Copy No. 6

PROCESS FOR THE PRODUCTION OF

AROCLORS, PYRANOLS, ETC.

AT THE ANNISTON AND AT THE WM. G. KRUMMRICH PLANT

April 1955 ---------- E. Mather

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TABLE OF CONTENTS

(See also the subject index at the end of the book).

INTRODUCTORY NOTE

When the MCC Aroclor process was studied in 1947 in preparation for the erection of a plant in Britain, an excellent report "Dept.246: Aroclor Process" Lyles, Soffranko and Becker, September 1946, describing the Plant "B" (now the Krummrich plant) process, was available. Supplementary information was collected into a report "Notes on the Plant 'B' Manufacturing Process for Aroclors" by E. Mather, April 1947, and soon afterwards the report "Notes on the Anniston Aroclor Plant", E. Mather and D. S. Havercroft, September, 1947, was written.

More information came to hand from time to time, including the "Process Description for Aroclor, Dept. 246", C. H. Schwarting, J. F. Neff and J. W. Graves, November 1953, also a revised version of this by Schwarting and Schwartz, March 15, 1955, and visits were paid to the Anniston and Krummrich plants for the purpose of revision early in 1955.

Havercroft and Mather briefly reviewed the Pyranol process at plant "B" in 1947, finding nothing to add to the MCC report, "Dept. A-246, Pyranol Process" Lyles, Soffranko and Miller, March 24, 1947. Subsequently the MCC report "Process Description for Pyranols and Inerteens", Schwarting, Neff and Graves, October 1953, appeared. Numerous research reports etc. are listed at the end of section VII, below.

The two reports of 1953 are much more condensed than the 1947 reports which they replace; they were drawn up to give just the essentials of the process work.

The present report is intended to condense all the process information up to about March 1955, into the form used for MCL process reports. The Pyranol process has been included in the same report because it dovetails so closely into the Aroclor process, and the writing of a separate report for Pyranols would have involved a good deal of repetition.

I. SYNOPSIS OF THE PROCESS

Aroclors are made by chlorination of diphenyl or of "diphenyl high boilers" (either crude or refined), or of mixtures of diphenyl with high boilers. The chlorination is done with temperature control in the presence of iron as a catalyst.

Chlorination is continued until the product contains the amount of chlorine corresponding to the grade of Aroclor required.

The crude chlorination product is usually air-blown to remove dissolved gases, and then either is sold as crude Aroclor, or it is worked up into distilled Aroclor. The distilled product is usually treated with Attapulgus earth. The following table shows which of these steps are involved in making each of the different grades of Aroclors.

All the operations are done batchwise, except that some of the distillation is done in a semi-continuous manner.

SYNOPSIS OF THE PROCESS, contd.

Grade	Diphenyl Crude HB Distilled HB used	% Chlorine	Air-blown	Distilled	Earth Treated	Nature of Finished Product
1142	DP	42	yes	no	no	Dark oil
1242	DP	42	yes	yes (vac)	yes	Clear oily liquid
1148	DP	48	yes	no	no	Dark oil
1248	DP	48	yes	yes (vac)	yes	Yellow oily liquid
1154	DP	54	yes	no	no	Dark viscous liquid
1254	DP	54	yes	yes (vac)	yes	Yellow viscous liquid
1160	DP	60	yes	no	no	Dark viscous sticky mass
1260	DP	60	yes	yes (vac)	yes	Lt.yellow sticky mass
1162	DP	62	yes	no	no	Dark sticky mass
1262	DP	62	yes	yes (vac)	yes	Lt.sticky resin
1262M is 90% Aroclor 1262 by weight, 10% Toluene						
1168	DP	68	yes	no	no	Dark crystalline solid
1268	DP	68	yes	yes (vac)	no	Dark crystaline solid
1169	DP	69	no	no	no	Dark crystalline solid
1269	DP	69	no	yes,atm.pr.	no	Flaked
1170	DP	70	no	no	no	Cryst. solid
1270	DP	70	no	yes, atm.pr.	no	Flaked
1171	DP	71	no	no	no	
1271	DP	71	no	yes, atm. pr.	no	Flaked
2565	75% DP 25% CHB	65	yes	no	no	Black brittle resin
4065	60% DP 40% DHB	65	yes	no	no	Black brittle resin
4465	60% DP 40% DHB	65	yes	yes, (vac)	no	Clear lt.yellow brittle resin
5060	DHB	60	yes	no	no	Black brittle resin
5460	DHB	60	yes	yes, (vac)	no	Clear lt.yellow brittle resin

SYNOPSIS OF THE PROCESS, contd.

Aroclors up to and including 1262 are usually spoken of as "liquid Aroclors"; those from 1267 upwards are called "solid Aroclors".

If the first digit of the code number is "1" it signifies that the Aroclor has been made from diphenyl. If the second digit is "1" the product has not been distilled, if it is "2" the product is a distilled grade. In all cases, the third and fourth digits give the percentage chlorine content of the product. Thus 1260 is a distilled grade, made from diphenyl, and containing 60% of chlorine.

The manufacture of materials with more than 68% of chlorine was discontinued in 1949, and the equipment for making them was taken down in 1952. This stage of the process required a special gas-heated agitated chlorinator, and a special still.

MCL research report 416A, 52/1/6, September '53, describes the production of sample amounts of higher Aroclors, but reports that the Newport plant is not suitable for making anything above 1268. Report 1151, NR 53/97/5 February 1954 describes the "topping" of 1254 for a special customer, and 25/11/54 describes the preparation of resinous Aroclors for a special customer.

The distillation residues may be sold as "Montars", various grades of these having been set up as shown on page 17 section III, below.

The off-gas (HCl etc.) coming from the chlorinators is cooled, and in some cases scrubbed with light Aroclor, then either is passed to a department using HCl gas, or else is absorbed in water to give muriatic acid, which may then be treated with carbon to bring it to "food grade" quality.

The preparation of Pyranols (Pyroclors, Inerteens, Askarels) involves at the most only a blending operation to arrive at the compositions as set out in the following table.

SYNOPSIS OF THE PROCESS, contd.

	w/w %					
	Tri Chl.bz.	Tri-tetra Chl. bz.	Aroclor 1254	Aroclor 1260	Tin Tetra - phenyl	Glycidyl-phenyl ether
Pyranol 1467	40	-	-	60	0.125	
1476	-	55	100	45	0.125	
1478	100	-	-	-	- -	
1481	25	-	75	-	- -	
1482	-	-	-	100	- -	
1488	40	-	-	60	- -	
1495	10	-	-	90	- -	
Inerteen PPO- * TCP-mixture	40	-	-	60	- -	0.20
Inerteen PPO-TTCB-mixture	-	55		45	- -	0.20
MCL Research Progress report 347D, 51/141/12, June 1952 describes certain variants of these.						

Pyranols are products for the (American) General Electric Company. Pyroclors are the MCL products corresponding to the Pyranols.

Inerteens are made for Westinghouse.

Askarel is the MCC trade mark.

The trichloro-benzene and the tri-tetra chloro are used to increase the fluidity of the Arcolors without seriously harming their electrical qualities. MCL Research Progress Report 1151 NR 53/97/8 suggests that TCB increases the solubility of tin-tetra-phenyl in the Aroclor.

The tin-tetra-phenyl, and glycidyl phenyl ether are "chloride scavengers" as discussed in section VII below.

* PPO denotes phenoxy propene oxide, a synonym for glycidyl phenyl ether $C_6H_5 \cdot O \cdot CH_2 \cdot CH \cdot O \cdot CH_2$

II. CHEMISTRY OF THE PROCESS

The main reaction, in the production of Aroclors, is a simple replacement of hydrogen by chlorine, but the product of the chlorination contains trace amounts of halogen-containing bye-products, which are less stable than the direct ring-substitution products; for example, they will give off HCl on being heated in the presence of metallic aluminium. (See the "Inorganic Chlorides" test, pages 77 - section IX, below. See also the discussion on pages 6 - section VII.)

Taking the atomic weights: H = 1.008
C = 12.01
Cl = 35.455 we have for direct substitution:

		M.wt.	% Cl	Compare
Diphenyl	$C_{12}H_{10}$	154.20		
Trichlorodiphenyls	$C_{12}H_7Cl_3$	257.54	41.3	Aroclor 1142, 1242 (42%Cl)
Tetra	$C_{12}H_6Cl_4$	291.99	48.6	Aroclor 1148, 1248 (48%)
Penta	$C_{12}H_5Cl_5$	326.44	54.3	Aroclor 1154, 1254 (54%)
Hexa	$C_{12}H_4Cl_6$	360.88	59.0	Aroclor 1160, 1260(60%)
Hepta	$C_{12}H_3Cl_7$	395.33	62.8	Aroclor 1162, 1262 (62%)
Octa	$C_{12}H_2Cl_8$	429.77	66.0	
Ennea	$C_{12}HCl_9$	464.22	68.8	Aroclor 1168, 1268 (68%)
Decachlorodiphenyl	$C_{12}Cl_{10}$	498.67	71.2	Aroclor 1171, 1271 (71%)
Terphenyl	$C_{18}H_{14}$	230.29		
Decachloroterphenyls	$C_{18}H_4Cl_{10}$	574.76	61.8	Aroclor 5060, 5460 (60%)
Undecachloro	$C_{18}H_3Cl_{11}$	609.21	64.0	

CHEMISTRY OF THE PROCESS, contd.

The conditions of chlorination (high temperature, and local excess of chlorine) are such that, for example, Aroclor 1268 containing 60% of chlorine, corresponding about to $C_{12}H_4Cl_6$, will certainly contain considerable amounts of $C_{12}H_5Cl_5$, $C_{12}H_3Cl_7$, etc. Further, there will probably be a very large number of isomers of, for example, $C_{12}H_4Cl_6$.

This complexity of composition has the advantage of depressing the melting points of the various components, so that materials of high molecular weight are obtained which have low vapour pressures, but which are still fluid at ordinary temperatures.

It is evident, however, that, though blending, say 2 parts of Aroclor 1260 with one part of Aroclor 1242, would give a product containing 54% of chlorine, such a product would not necessarily be identical with Aroclor 1254 made by direct chlorination of diphenyl to 54% chlorine content.

This is of practical importance because partial crystallization of liquid Aroclors, which has occured, and which has given rise to customer complaints, has been ascribed to preponderance of one component which chanced to be sparingly soluble in the general mixture. See pages 11- section VII, below.

Chloride Scavengers.

See the discussion of the action of scavengers in Section VII, below, pages 6-.

Heat of Reaction (chlorination).

Judging by literature information on similar reactions, the heat evolution in the chlorination must be around 40 kg.cal. per g. atom of chlorine combined, (i. e. about 20 kg. cal. per g. atom fed in).

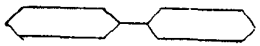
The heat of evaporation of Aroclors.

Trouton's rule (g. cal/g.mol = 21 x B.Pt. °C. abs) gives, for example, for Aroclor 1260 of M.Wt. about 360 and mid B.Pt. about 395°C.(668°abs) 14,028 g. cals. per g mol, or 39 g. cals per gram.

III. PHYSICAL AND CHEMICAL DATA

Physical Constants of Diphenyl

Compiled by W. P. Metzner
Date: February 12, 1943
Corrected: June 22, 1943
Corrected: Jan. 11, 1944



Beilstein V-576

NAME		SOURCE	DATE
Formula	$C_6H_5C_6H_5$		
Appearance	Monoclinic	Chem.Rubber handbook	1941 1942
Molecular Weight	154.20	Chem. Rubber handbook	
Boiling point at 760 mm	255°C.	Int.Crit.Tables	1928
Boiling point at 10.4 mm	118°C.	Swann file #137*	1935
Crystallizing point	69.0°C.	Swann curves *	1934
Melting point	69°C.	Int.Crit.Tables	1928
Solution point			
Specific Gravity at 77/4°C.	0.9896	Int.Crit.Tables	1928
Specific Gravity at 20/4°C.	1.041	Int.Crit.Tables	1928
Refractive index at 77.1°C.	D = 1.58822	Int.Crit.Tables	1928
Viscosity at 100°C.	28.8 Say. Sec.	Swann File#137A*	1930
Surface tension at 129.2°C.	29.5 dyne/cm	Int.Crit.Tables	1928
Solubility in water at 80°C.	Insoluble	Dow	1943

*Note: Files of W. P. Metzner, St. Louis

III. PHYSICAL AND CHEMICAL DATA, contd.

Physical Constants of Diphenyl, contd.

NAME		SOURCE	DATE
Viscosity at 70°C.	31.2 Say. Sec.	Swann File #137A*	1930
Solubility in Abs. Alc% Alc.at 19.5°C.	9.98 g./100g.solvent	Seidell	1941
Solubility in 100% Methanol at 19.5°C.	6.57 g./100g.solvent	Seidell	1941
Benzene at 27.9°C.	137g./100g.solvent	Seidell	1941
Heptane at 26.5°C.	25.1 g./100g.solvent	Seidell	1941
Flash point °F.	235°F. - closed cup 255°F. - open cup	Ind. & Eng.Chem. 32,882	1940
Fire point °F.	124°C.	Dow	1943
Heat of combustion Kg-cal.(15°)	per gm.mol=1504.4	Int.Crit.Tables	1928
Heat of formation			
Latent heat of fusion	28.8 gm-cal.per gm. (53.1 BTU/lb.)	Lange Handbook	1937
Latent heat of vap. at 69.2°C.	190.9 Btu.per lb.	Swann File #137A*	1928
Specific heat at 40°C. (solid)	0.387 cals/g 1.61 joule/gm. /	Int.Crit.Tables	1928
Critical temperature	-	-	-
Critical pressure	-	-	-
Dissociation constant	-	-	-
Pounds per U.S.gal.at 77°C	8.23 lbs.per gal.	Swann File #137A	1930
Toxicity (1) Dr.McLester,Anniston Plant Report (2) Percy May "Chemistry of Synthetic		Drugs" 3rd.Ed.p.19	1937

*NOTE: Files of W. P. Metzner,St.Louis.

III. PHYSICAL AND CHEMICAL DATA

1. Diphenyl

Specific Gravity: 0.992 at 75°C/4°C.

The Swann Chemical Company bulletin of 1930 gave thermal data on diphenyl

Viscosity of Diphenyl (from Anniston files)

°F.	Centipoises
160	1.439
170	1.309
180	1.208
190	1.114
200	1.034
220	0.901
250	0.752
300	0.574
350	0.459
400	0.374
450	0.313
482	0.283

Specific Heat (from Anniston files)

°F.	B.t.U./lb.
200	0.416
300	0.470
400	0.547
500	0.616
600	0.658
700	0.679
800	0.686
900	0.690
1000	0.696

III.PHYSICAL AND CHEMICAL DATA, contd.

1. Diphenyl, contd.

Another record gives the following values for liquid diphenyl:

°C.	Sp. Heat cals./gram
100	0.360
150	0.380
200	0.412
250	0.460
300	0.518
350	0.575
400	0.610
450	0.625

Vapour Cp = $\frac{0.460}{250°C.}$

$\frac{0.625}{450°C.}$ (D. A. Roper, Design Data for Units 4 & 5)

Density of Liquid Diphenyl (from Anniston files)

°C.	grams./cc
75	0.9885
100	0.9695
150	0.9294
200	0.8885
250	0,8470
300	0.8010
350	0.7510
400	0.6918
450	0.6180

Heat of Vaporization:

65.4 g. cal./g. at 258.5°C.

III. PHYSICAL AND CHEMICAL DATA, contd.

2. Distilled High Boilers

Santowax·R

contains about 1% diphenyl
10% ortho terphenyl
46% meta terphenyl
21% para terphenyl
22% triorthophenylene, quaterphenyls etc.
(Anniston Final Report 1942)

Upper hold point	137-143°C.	Changed)	137-145°C.
Lower hold point	45-60°C.	Sept.23/47)	45-63°C.

The density of the Santowax may be assumed to be the same in both the crude and distilled form. Viscosity also is the same.

Specific Gravity of Crude Santowax:

140°C.	1.026
175	1.000
215	0.98
300	0.964 (est. from curve of other points).

Viscosity of Santowax R

155°C.	32.4 Saybolt Universal Seconds
225	28.5 " " "

Molten Santowax flows about like water at 25°C.
(A.M.Ellenburg's letter January 6, 1949).
(Probably 250°C. was intended).

Specific Heats (estimated)

Solid	0.4
Liquid	0.5

Latent Heats (estimated)
Fusion 63 B. t. u./lb.
Vaporisation 140 B.t.u./lb.

III. PHYSICAL AND CHEMICAL DATA, contd.

2. Distilled High Boilers, contd.

Santowax R, contd.

Boiling Range: 360-450°C.

Flash Point: Cl.open cup 191°C.

Flame Point: Cl.open cup 238°C.

Another account gives Flash Point 190°C; Flame point 220°C.

Values assumed in the design of the Santowax still at Anniston:

Specific Heat	-	0.5
Heat of Vaporization	-	140 B.t.U./lb.
Viscosity at 250°C.	-	5 centipoises *
Viscosity at 350°C.	-	2 centipoises *

* (Mr. Ellenburg said these values were too high).

Terphenyls	"Hold Point"	B.Pt. at 30 mm
Crude ortho (Santowax O)	53 - 55°C.	205°C.
Crude meta (Santowax M)	83 - 85°C.) mixture	240°C.
Crude para (Santowax P)	209 -213°C.)	
m - p eutectic	84.8°C.	
Ternary eutectic	about 74°C.	

See also Monsanto Technical Bulletin #P-103, January 4, 1947 issue, for the properties of the separated crude isomers sold.

3. Trichlorobenzene

Dr. Jenkins, January 27, 1954, refers to Ind. Eng. Chem. 39 (1947) p. 517 for vapour pressure data.

III. PHYSICAL AND CHEMICAL DATA, cont'd.

Arochlors

The main characteristics of the Arochlors are given in the following table:

Grade	M. Pt. °C.	Specific Gravity	Sp. Gr. coeff. per °C.	Viscosity Saybolt Seconds	Distilling Range °C. 10 - 90%	Pour Point °C.	Hold Point °C.	Softening Point ASTM
1142		1.348 to 1.353 65/15.5°C.	0.0009					
1242		1.338 to 1.348 65/15.5°C.	0.0009	50 to 53 at 54.5°C.				
1148		1.412 to 1.415 65/15.5°C.	0.0009					
1248		1.404 to 1.414 65/15.5°C.	0.0009	73 to 80 at 54.5°C.				
1154		1.500 to 1.510 65/15.5°C.	0.00095					
1254		1.495 to 1.505 65/15.5°C.	0.00095	44.5 to 47.5 at 98.9°C.	365 to 390	8-12		
1160		1.558 to 1.563 90/15.5°C.	0.0010					
1260		1.550 to 1.560 90/15.5°C.	0.0010	72 to 77 at 98.9°C.	385 to 415	26-34		
1162		1.582 to 1.587 90/15.5°C.	0.0010					
1262		1.572 to 1.583 90/15.5°C.	0.0010	88 to 100 at 98.9°C.				
1262M								
1168							145-165	
1268							135-160	
1169							225-250	
1269							225-255	
1170								
1270							285-300	
1171								
1271	304°C. min.							
2565								66.0 to 72.0
4065								66.0 to 72.0
4465				160 to 210 at 130°C.				
5060								110.0 to 115.0
5460								100 to 105.5

See also Phosphate Division report 171-102 "Highly chlorinated quater phenyls" sent to Mr. Hamer, March 12, 1948.

III. PHYSICAL AND CHEMICAL DATA, contd.

Other properties are listed in Section IX, Finished Product Specifications. MCC Bulletin P-115 gives the following data not included in either of the foregoing lists: --

General description of the Aroclors: (non-oxidising, permanently thermoplastic, chemical inert, will not burn, compatible with most non-hydroxylic solvents, good electrical properties and fire resistance, adhesive, and non-drying).

Evaporation loss data, % loss in: 5 hours 163°C. and 6 hours 100°C.
Flash point
Fire point
Refractive index.
Corrosion data
Specific heat

Surface tension (Aroclor 1254)

°C.	dynes /sq.cm.
25	50.3
80	44.0
100	42.0

Thermal conductivities
Solubility data for many compounds
Vapour pressures (semi-log chart)
Viscosity variation with temperature
(use of V.I. improvers suggested).
Variation of Dielectric constant with temperature
Compatibility with lacquer components, structural materials, etc.
Suggested uses.

The paper by Baxter, Vodden and Davies of Ruabon, J. Appl. Chem. 3 October 1953, page 477, gives a method of determination of thermal conductivity of liquids in general.

MCL Res. Prog. Rpt. 355E, 51/91/2, October 1951, makes comparison between Aroclor 1248 and mineral oil as cooling media.

Heat of vapourisation is discussed on page 2, Section II, above, also the Heat of Chlorination of Diphenyl.

III. PHYSICAL AND CHEMICAL DATA, contd.

Vapour pressure curves for some of the Aroclors are given on pages 72 - 75 of the report by Lyles, Soffranko and Becker, November 1946. More recently vapour pressures for samples of Aroclors 1242, 1248 and 1254, measured by an effusion method at temperatures below 100°C. were given in a report from the Southern Research Institute, Birmingham, Alabama. (Report No. 1, project 526, to MCC February 4, 1954). All these data are shown in logarithmic form on pages III-9a&b below.

Dr. Roebuck, then at Ruabon, in a letter dated June 8, 1953, recommended that vapour pressure measurements should be made by a static method, rather than by effusion, because the Aroclors are mixtures of compounds of different volatilities, and the effusion method might cause some fractionation.

MCC progress reports under Job 171-1089, dealing with the vapour pressures of the different Aroclors, and with the measurement of the concentration of Aroclors in air, were sent to MCL at various times, for example June 17, August 13, and September 17, 1953. A final report under this job number was promised July 16, 1953.

Newport Plant Technical Committee, December, 1953, recommended investigation into combustion methods of determining the concentration of Aroclors in air. Mr. Benignus, of St. Louis, September, 1953, discussed the dangers of using Aroclors in indoor paints.

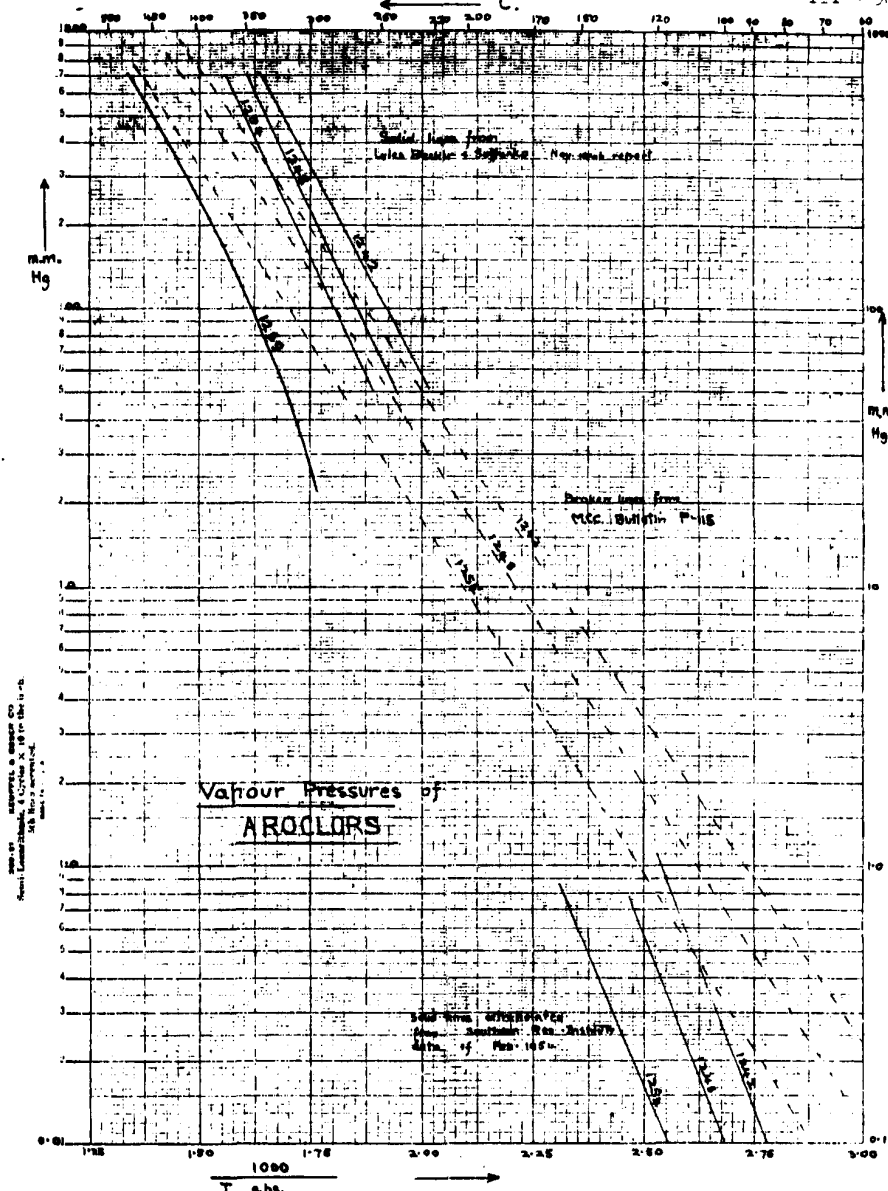
Dr. Newman at Newport, September, 1953, had files on the question of Aroclors in air. See also under "Hazards" in Section XI, below.

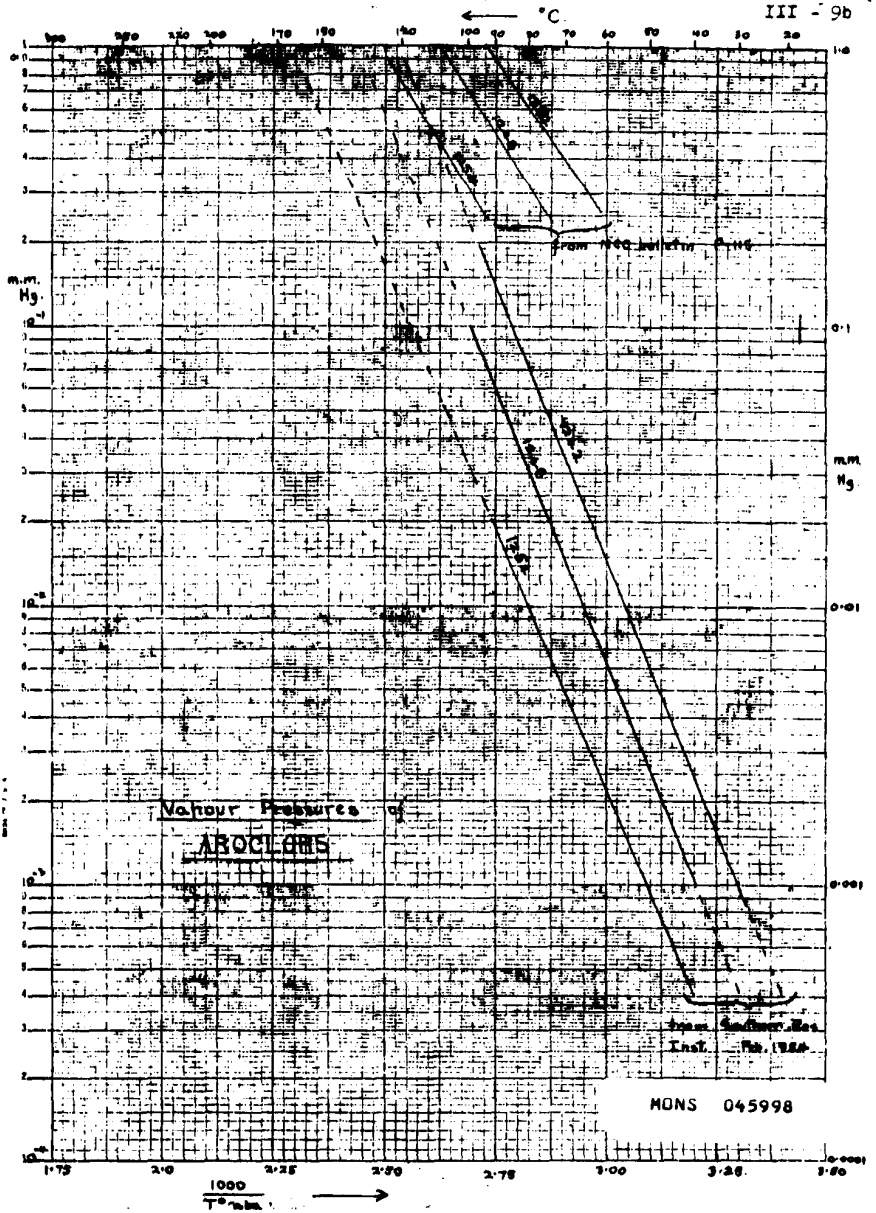
Dr. Jenkins, January 27, 1954 said that for the Pyranols containing 33%, or more, of Trichlorobenzene, the vapour pressure of the Pyranol might be taken as equal to that of TCB alone up to about 150°C. Above that temperature the vapour pressure of the Aroclor would begin to show up.

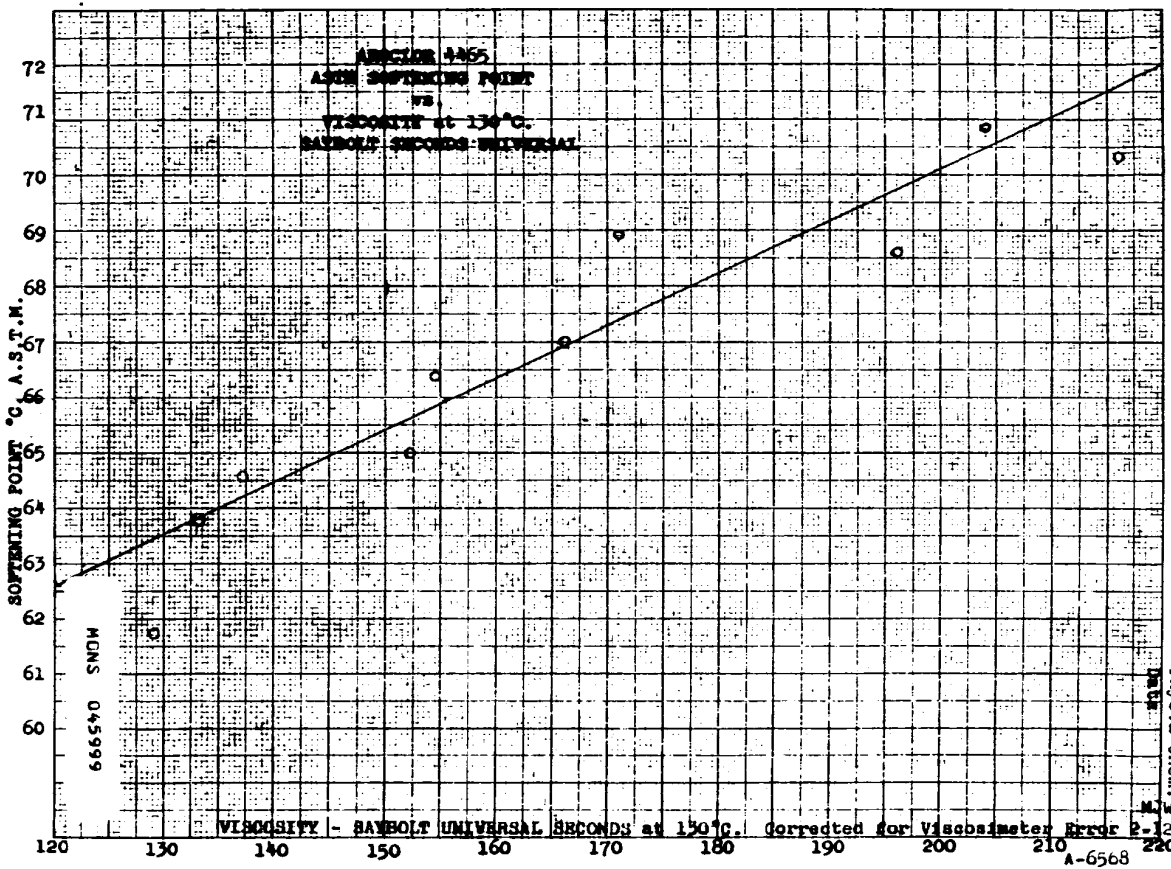
Viscosity-temperature data for Aroclors are given on pages III-10, and III-11, below.

MCL progress report 1152, DF 54/16/9, September, 1954, reports the viscosity of Aroclor 2565 from 100 to 130°C.

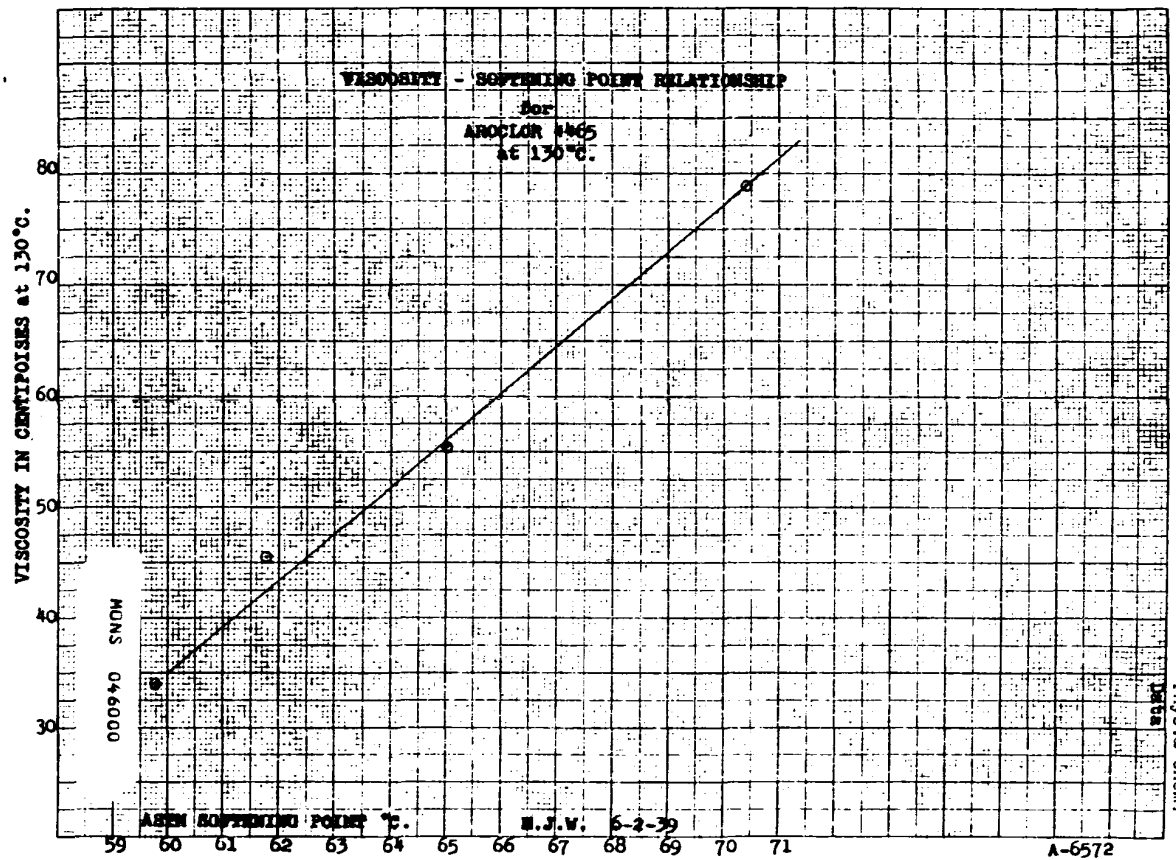
Report 1152, DF 52/26/6, August, 1954, discusses the viscosity, and the viscosity index of a tintetraphenyl blend of Pyranol.


°C.
m.m. Hg
Solid lines from Lewis Blacet & Sullivan
Broken lines from M.C.C. Bulletin P-115
Vapour Pressures of
AROCLORS
Solid lines extrapolated from Southern Res. Institute data of Nov. 1945
1000/T abs.
MONS 045997


°C.
m.m. Hg.
mm Hg.
Vahour Pressures of
AROCLORS
from Monsanto Res. Inst. Feb. 1954
MONS 045998
1000 / T°abs.



III-10
Phys.& Chem.


VISCOSITY - SOFTENING POINT RELATIONSHIP
for
ARACLOR #1465
at 130°C.
VISCOSITY IN CENTIPOISES at 130°C.
80
70
60
50
40
30
MONS 046000
ASTM SOFTENING POINT °C.
59 60 61 62 63 64 65 66 67 68 69 70 71
M.J.W. 6-2-39
A-6572

III. PHYSICAL AND CHEMICAL DATA, contd.

Specific Gravity-temperature data are given on page 11 of MCC Bulletin P-115, also on pages 13, 14 and 15, of Section III, below.

The coefficients of fall of specific gravities of the Pyranols with increasing temperature in the range between 45 and 85°C. are as follows: --

1467 and 1488 - 0.001 per 1°C.
1495 0.00285 per 1°C.

(Lyles, Soffranko & Miller page 43)

III. PHYSICAL AND CHEMICAL DATA, contd.

Specific Gravity of Aroclors
at Various Temperatures

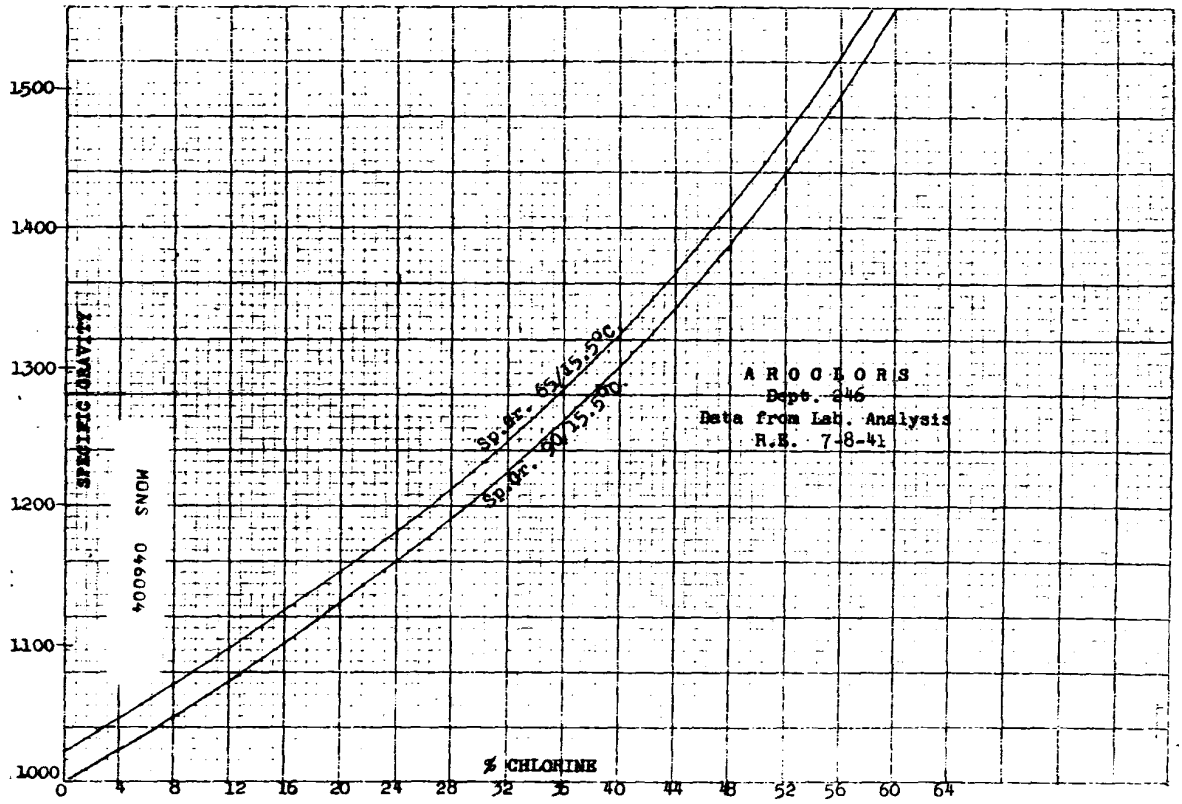
For	Temp. °C.	Sp. Gr.	Lbs. per Gallon (US)
1242	60	1.347	11.23
	65	1.344	11.20
	70	1.338	11.16
	75	1.335	11.13
	80	1.332	11.10
	85	1.329	11.07
	90	1.324	11.03
	95	1.319	11.00
	100	1.315	10.96
1248	60	1.413	11.78
	65	1.410	11.75
	70	1.403	11.71
	75	1.400	11.68
	80	1.397	11.65
	85	1.394	11.62
	90	1.390	11.58
	95	1.385	11.55
	100	1.381	11.51
1254 or OE 1476	60	1.505	12.55
	65	1.500	12.50
	70	1.495	12.46
	75	1.490	12.42
	80	1.485	12.38
	85	1.480	12.34
	90	1.475	12.30
	95	1.470	12.26
	100	1.465	12.21

contd...

III. PHYSICAL AND CHEMICAL DATA, contd.

Specific Gravity of Aroclors
at Various Temperatures
(contd.)

For	Temp. °C.	Sp. Gr.	Lbs. per Gallon(US)
1260 or	60	1.584	13.21
GE 1482	65	1.580	13.17
	70	1.575	13.13
	75	1.570	13.09
	80	1.565	13.05
	85	1.560	13.01
	90	1.555	12.96
	95	1.550	12.92
	100	1.545	12.88
1262	60	1.604	13.40
	65	1.600	13.36
	70	1.597	13.32
	75	1.593	13.28
	80	1.588	13.24
	85	1.582	13.19
	90	1.577	13.15
	95	1.572	13.11
	100	1.568	13.07


SPECIFIC GRAVITY
1500
1400
1300
1200
1100
1000
0
4
8
12
16
20
24
28
32
36
40
44
48
52
56
60
64
% CHLORINE
Sp. Gr. 60/15.5°C.
Sp. Gr. 65/15.5°C.
A R O C L O R S
Dept. 646
Data from Lab. Analysis
R.E. 7-8-41
MONS 046004

III. PHYSICAL AND CHEMICAL DATA, contd.

Solubility Data

MCC Bulletin P-115 includes data on the compatibility of Aroclors with numerous solvents. MCC report 2949, October 28, 1953, deals with the compatibility of Aroclors etc. with mineral oils.

Newport complaint N 464, January 1954, arose from the alleged incomplete solubility of 4465 and 5460 in Dutch Shell Co.'s Risella oil. See also the discussion of "Partial Crystallization" in Section VII, below.

The solubility of gases in Aroclors is of importance in connection with the use of Aroclors as lubricants in compressors. Reference is made to Monsanto Bulletin P-128 on this use of Aroclors.

MCC Central Research Report 543 "Studies on Light Stability etc. of Aroclors", White and Ruehrwein, January 1949, states that the solubility of oxygen in 1254 at 25°C. is about 1 x 10^{-6} moles of oxygen per gram of Aroclor.

On December 29, 1953, Dr. Gardner enquired for data on the solubility of gases in Aroclor up to 7000 psig, but MCC could not supply any data. The following MCL Research Reports deal also with questions of compatibility: --

Job 1152, 51/141/20 etc. "Effect of Pyroclors on electrical wire insulation" -- softening and loss of break-down strength -- Pyroclors unharmed. See also under "Gaskets" in Section XII, below.

Job 1151, DF 53/86/1, September 1953. "Solubility of methyl anthraquinones etc. in Aroclors".

Job 1151, NR 53/97/8 "Solubility of TTP in Aroclor 1248"

Also MCC report 2272, "Solubility of Aroclor 5460 in various solvents", Wade, December 1948.

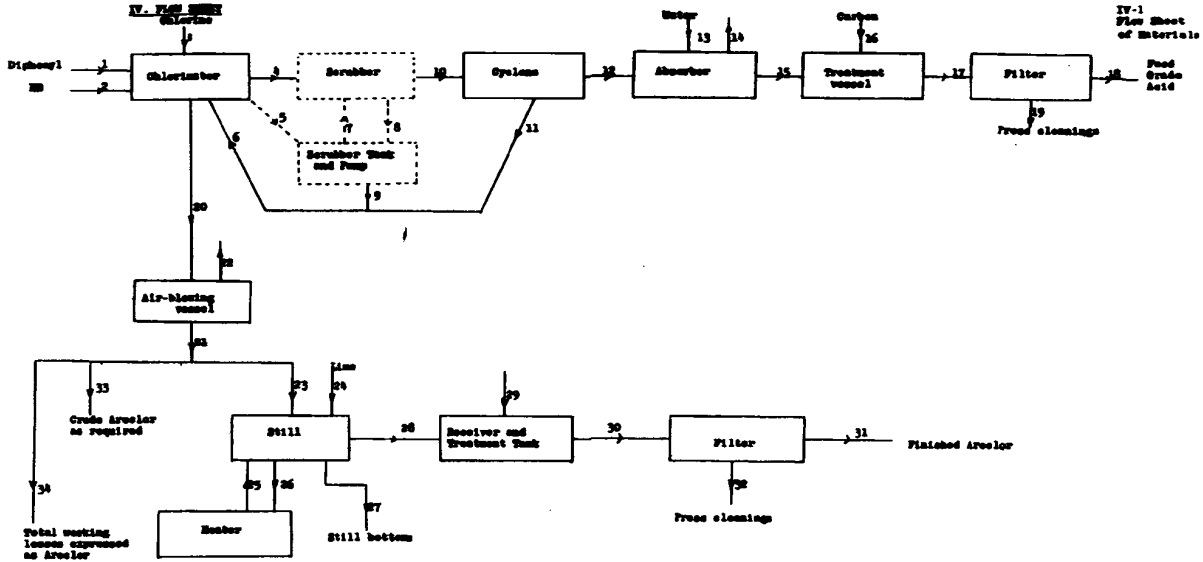
Variation of Resistivity with Temperature

It was stated in a report on Complaint NJ/21, November 11, 1954, that a volume resistivity of 17 to 47 x 10^9 at 120°C. corresponds to about 85 x 10^9 at 100°C. (Aroclor 1254 - - MCL Specification 500 x 10^9 ohm/cm^3 minimum at 100°C.)

III. PHYSICAL AND CHEMICAL DATA, contd.

MONTARS (1946 data)								
Grade	Softening Point ASTM °C	Flash Point °C.	Fire Point °C.	Penetration 250g.weight at 75°C.	Chlorine %	Iron %	Calcium oxide %	Acidity mg NaOH per gram
1	-	-	-	-	-	-	-	-
2	-	-	-	-	-	-	-	-
3	130-160	298	none at 343	-	51.6	0.31	3.47	Sl. basic
4	191-208	-	-	0	-	-	-	-
5	165-180	-	-	2 cm x 10^{-2} per minute	57-59	0.12 max.	1.6 max.	-
6	168	-	-	-	599	0.13	2.0	-
9	140-160	257	304	-	0.18*	0.21	0	0.104
10	139	-	-	47 cm x 10^{-2} per 5 secs.	-	-	-	-

* Montar #9 is the residue from the diphenyl process; data on it included here only for convenience. No data found on Montar #7.


IV. FLOW SHEET
Chlorine
Diphenyl
MB
Chlorinator
Scrubber
Cyclone
Absorber
Water
Carbon
Treatment vessel
Filter
Fused Crude Acid
Press cleanings
Scrubber Tank and Pump
Air-blowing vessel
Crude Aroclor as required
Still
Lime
Receiver and Treatment Tank
Filter
Finished Aroclor
Press cleanings
Total wasting losses expressed as Aroclor
Heater
Still bottom
IV-1
Flow Sheet of Materials

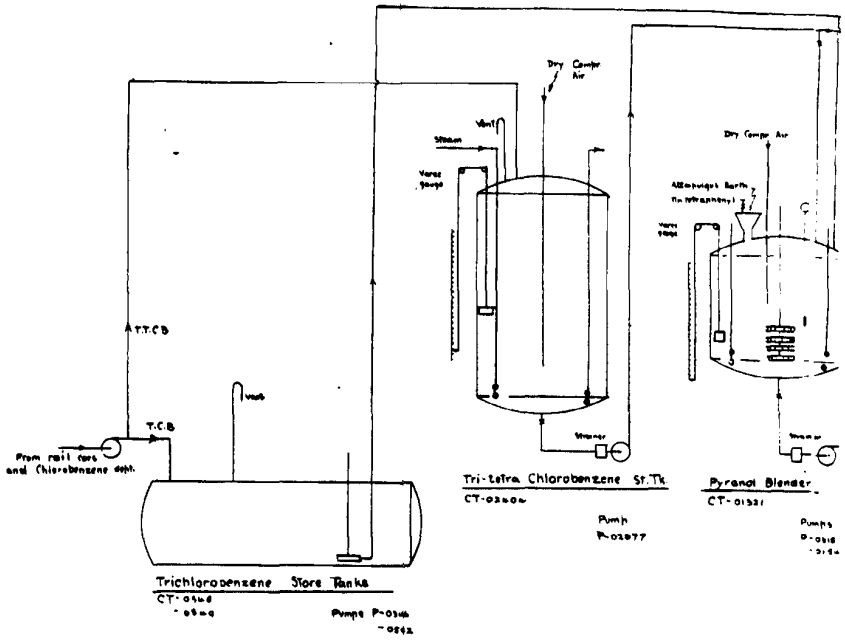
IV. FLOW SHEET

Quantities per 100 lbs. of Aroclor 1242 (150 lbs. of Aroclor 1142).
(Approximate quantities, not hard and fast process instructions.)

(Quantities for other Aroclors may be calculated in the same way from the data in Section X below).

Stream	1	3	4	6	10	12	13	14	15	16	17	18	19	20	21	22	23	24	27	28	29	30	31	32	34
Diphenyl	63.85																								
Chlorine		92.1	Trace		Trace	Trace		Trace																	
HCl			47.4		47.4	47.4		6.1	41.3		41.3	40.3	1.0	Trace		Trace									
Aroclor			5.0	5.0										108.55	108.55		102.5		2.4	100.1		100.1	0.1	100.0	6.05
Water							94.8	12.2	82.6		82.6	80.6	2.0												
Carbon										2.0	2.0		2.0												
Attapulgus earth																					0.05	0.05	0.05		
Lime																		0.5	0.5						
TOTAL	63.85	92.1	52.4	5.0	47.4	47.4	94.8	(*) 18.3	123.9	2.0	125.9	120.9	5.0	108.55	108.55		102.5	0.5	(#) 2.9	100.1	0.05	100.15	0.15	100.0	(x) 6.05

(*) Total loss from absorption system.
(#) Tapped at intervals.
(x) Total working losses of Aroclor; about 2.5 in the crude stage and 3.55 in the refining stage.


Dry Compr Air
Vent
Steam
Vapour Sleeve
Dry Compr Air
Vapour Sleeve
T.T.C.B
T.C.B
From rail cars and Chlorobenzene dehl.
Vent
Trichlorobenzene Store Tanks
CT-0146
Pumps P-0146
Tri-tetra Chlorobenzene St.Tk.
CT-02604
Pump
P-02077
Pyranol Blender
CT-01521
Pumps

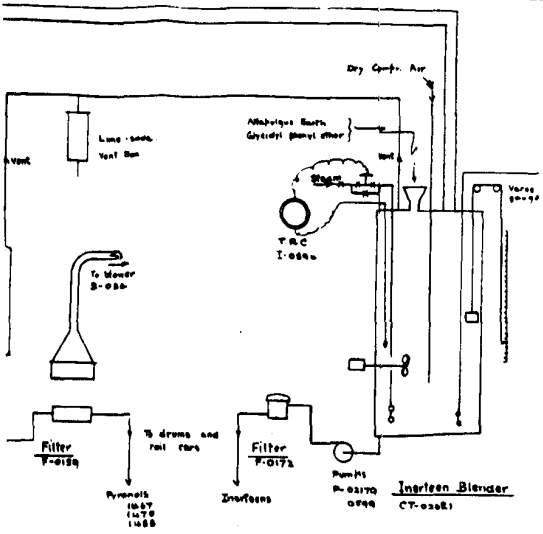
MONS 046009

FLOW SHEET OF EQUIPMENT

PYRANOLS and INERTEENS

Roughly 1" = 5'-0"

Ed May 1951



The Aroclor blender CT-0779 hump P-01757 and filter F-0238 are used for Pyranol 1481

Ground level

MONS 046010

VII. COMMENTS ON THE PROCESS, contd.

List of Reports, contd.

MCC RESEARCH PROGRESS REPORTS

Job. No.	Report No.		Date Copies sent to MCL
117-790	2673	Use of dilute chlorine December 10, 1951	January 30, 1952
117-1987		Method of determination of stability of TCB Munch and Ashworth	October 16, 1950
117-1987	part I	Infra-red studies, TCB and Pyranol 1467	May 7, 1948
117-2032		Tentative Amendment to Process for TCB, Hubbard and Steahly	October 16, 1950
117-4013		Aroclor 5460 - toluene solutions	
171-718	2771	Effect of exposure of Aroclors to steel	April 27, 1953
171-719	2772	Containers for Aroclor 1242	April 27, 1953
171-1027		A series of reports on minor inquiries from Sales	April 14, 1953
171-1028	2790	A series of reports on expansion of Aroclor manufacture, increase in rate of chlorination etc.	April 14, 1953 April 27, 1953 etc.

VII. COMMENTS ON THE PROCESS, contd.

List of Reports, contd.

MCC RESEARCH PROGRESS REPORTS, contd.

Job No.	Report No.		Date Copies sent to MCL
171-1029	2788	Use of Aroclors with sec. butyl acetate and a series of reports on assistance to the Aroclor Dept.	April 14, 1953 April 27, 1953 etc.
171-1054		A series of reports on low-temperature dielectrics	July 20, 1953
171-1075	2887	Minor investigation for sales; examination of competitive products	July 20, 1953
171-1076		Assistance to Aroclor Department	July 20, 1953
171-1077	2883	Aroclor 5460 from Davis "Tarophen"	July 20, 1953
171-1088	2881	Differences between Anniston and Krummrich Plants	July 20, 1953
171-1089	2892	Determination of Aroclor concentration in gases	June 7, 1954
171-4015		Aroclor 5460 - toluene solutions	
171-4019		Customer contacts.	

VII. COMMENTS ON THE PROCESS, contd.

List of Reports, contd.

MCC RESEARCH PROGRESS REPORTS, contd.

Job No.	Report No.		Date Copies sent to MCL
729-1573		Improve flexibility of operation of Krummrich plant Semi continuous distillation. Will quicker chlorination give more highboilers?	
729-1664		Series: Maximum production rate with minor changes in equipment; Krummrich plant.	April 27, 1953 etc.

VII. COMMENTS ON THE PROCESS, contd.

List of Reports, contd.

MCC CENTRAL RESEARCH REPORTS		Date sent to MCL
Report 543	January 1949. Studies on Light Stability of Electrical Resistivity of Aroclor 1254. White and Ruehrwein (Gives data on the solubility of oxygen in the Aroclor)	

MCC REPORTS		
1877	Standard Process Report on Aroclor 4465 Ellenburg	June 7, 1946
2215	Anniston Aroclor Data book Final Report	January 3, 1951
2272	Solubility of Aroclor 5460 in various solvents. December 1948.	
2492	Formation of precipitate in organic solutions of Aroclors. May 1950.	
2689	Distillation of Aroclors at higher pressures.	
2694	High rate of distillation of Aroclors.	
2949	Compatibility of Aroclors with mineral oils. October 1953.	

VII. COMMENTS ON THE PROCESS, contd.

List of Reports, contd.

PUBLICATIONS

J. Appl. Chem. 3
October 1953,
page 477
"Method of determination of thermal conductivity"
Baxter, Vodden and Davies

Am. Soc. Mech. Eng. 58
(1936) p. 719
"Thermal conductivity of Aroclors"

Ind. Eng. Chem. 39
(1947) p. 517
"Vapour Pressure of Trichlorobenzene"

See also MCL Research Progress Report 1151, 53/58/3, 54/26/13, DF 54/27/9 and 10, December 1954, and January 1955 on preparations of material for an Aroclor booklet.

See also letter, E. Mather to Dr. Newman, January 8, 1952, reviewing journal information on Aroclor toxicity.

Report No. 1, project 526, February 4, 1954, W. M. Nolan, Southern Research Institute, Brimingham, Alabama, "Vapour Pressures of Aroclors below 100°C."

VII. COMMENTS ON THE PROCESS, contd.

List of Reports, contd.

MCC BULLETINS

		Date sent to MCL
	"Askarel" Sales bulletin July 24, 1953	September 9, 1953
	Indirect Heater for Unit Chemical Operations ------ McArdle (re-issued as bulletin P-130)	
	Physical Properties of Aroclors	
P-115	Properties of Aroclors (replaced the foregoing report.)	
P-128	Aroclor Incombustible Lubricant	January 17, 1952
P-130	Indirect Aroclor Heater for Unit Chemical Operations	Requested July 7, 1954
P-137	Die casting	January 28, 195[illegible]

VII. COMMENTS ON THE PROCESS, contd.

List of Reports, cont'd.

LONDON CENTRAL ENGINEERING REPORTS

Preliminary Report.
March 1949.

Periodical reports on running-in the
Newport Plant.
July - November 1951.

Final Report.
Aroclor Plant Performance.
September 1951.

Operating Instructions for Aroclors
September 1951

Design Book.
Newport Project 41.
Chemical Engineering Design.
January 1950.

MCL REPORT

Market Research.
N. G. H. Thomas
November 1947.

Tentative Process and Analytical Methods
February 1951.

VII. COMMENTS ON THE PROCESS, contd.

List of Reports, contd.

MCL RESEARCH REPORTS

Job No.			
347E	50/175/2	Nov. 50	Electrical tests on TCB. Effect of insulating materials on Aroclors.
	51/16/1	Jan.51 etc.	Tests on TCB. Cell for dielectric tests. Equipment for high voltage break-down tests.
	51/59/1	Feb.51 etc.	Aroclors in PVC. Power factor curve. Resistivity.
	51/129/1	Aug. 51	High voltage breakdown equipment constructed.
	51/141/1	Aug. 51	Resistivities, power factors, dielectric constants of Aroclors. Electric tests on nitrated Aroclor. Routine electrical tests on Aroclors. Choice of containers. Competitive Aroclors. Literature survey of uses of Aroclors in the electrical industry. Properties of Montars. Nitrated Aroclors.
	52/47/1	Sept.52	Capacitator paper.
	52/48/1	Sept.52	Literature survey on uses for Aroclors.
	Final Reports	Dec. 52	Alternating current characteristics of Aroclors.
		Feb. 53	Literature Survey of Uses.
		Jun. 53	Electrical Testing Methods.

VII. COMMENTS ON THE PROCESS, contd.

List of Reports, contd.

MCL RESEARCH REPORTS, contd.

Job. No.			
355E	50/193/1	Nov.50	Examination of TCB samples
	51/91/1	Aug.51	Gasket materials. Aroclor 1248 as cooling medium, compared with mineral oil.
416A	52/1/1	Jan.52	Solubility of 4465 and 5460 in white spirit. Removal of green colour. Crystal formation in 1232. Acetone solubility of resinous Aroclors. Preparation of solid Aroclors. Mild steel effect on Aroclor. Evaluation of TCB.
	52/19/1	Mar.52	Evaluation of TCB.
454E	FR52/41/3	Apr.52	Aroclor as coolant for internal combustion engines. Effect of high temperatures. Corrosion by Aroclor.
	FR52/41/5	Sep.52	
	FR52/71/1		Aroclors in hydraulic pumps. Corrosion very slight.
1151	52/1/12	May 53	Bakelite Co.'s (air current) method of testing stability of Aroclor.
	FR52/17/4	Mar.53	Aroclor as hydraulic fluid.
	FR52/71/3		Aroclor in die casting.
	FR52/90/6		Use of Aroclors in silicate paints.
	FR53/34/4	May 53	Aroclors in die casting fluids.
	FR53/58/3	Jun.53	Breakdown strength after earth treatment. Capacitors. Effect of metals on Aroclors. Material for publication.

VII. COMMENTS ON THE PROCESS, contd.

List of Reports, contd.

MCL RESEARCH REPORTS, contd.

Job No.			
1151	DF53/85/4	Nov.53	Blowing of fuses in Aroclors.
	DF53/86	Sep.53	Anthraquinone derivatives as stabilizers. Joint box compound. Storage in steel. Activated alumina in refining competitive materials.
	NR53/97/1	Jul.53	Stability up to 300°C. Crystal deposition in 1232. (Torque conveyors). Blending of 1221 and 1242. Topping of 1254. Fuller Earth. Alumina etc. in refining. Solubility of TTP in Aroclor. Determination of Aroclor in Air. Resinous Arcolors.
	DF54/16/3	Mar.54	Exposure of plastics etc. to Pyroclors.
	DF54/26/3	Jun.54	Studies for hydraulic fluids. Metal exposure. Synthetic rubbers, Gaskets, mixtures of Aroclors with organic phosphates etc. Running tests. Rust inhibitors added. Resistant paints sought. Burn point. Autogenous ignition. V.I. improvers. Material for publication.
	DF54/27/1	Apr.54	Electrical tests. Montars. Paints for piping etc. Reclamation of arced Aroclors. Various applications studies. Gaskets. Test cell. Aroclor capacitors. Publications.

VII. COMMENTS ON THE PROCESS, contd.

List of Reports, contd.

MCL RESEARCH REPORTS, contd.

Job No.			
1152	51/141/20	Jan.53	Technical service. Effect on insulation of wire. Examination of arced material. Effect of Aroclor on Shellac.
	53/60/1	Jun.53	Effect on insulation of wire.
	DF53/85/1	Sep.53	Technical service. Test cell. Effect on insulating materials, processed wood etc. Silicon glass coated wire. Reclamation of used Pyroclor.,
	DF53/89/5	May 54	Use in polishes.
	NR53/103/1	Jun.54	Fuller's Earth removes TTP. Other absorbents tried. Determination of TTP in Pyroclors.
	DF54/16/5	Apr.54	Technical service. Effect of exposure of insulating materials to Aroclors. Gaskets. Test cell. Damage by arcing. Customer's test method corrected. Viscosity of 2565. Resistivity of 1254 against time. Silicone glass fibre etc.
1161	NR54/77/1	Nov.54	Hydrolysis of Aroclors.

VII. COMMENTS ON THE PROCESS, contd.

The early research work on Aroclors was done mainly at Anniston, from about 1929 onwards, under the Phosphate division, and the reports are to be assimilated into the Organic Division collection of reports. Work has been done, for example, on the isolation of the individual chlor-diphenyls, and on infra-red analysis of mixtures of chlor-diphenyls, also on the investigation of numerous uses suggested for Aroclors, and on application work.

Most of the basic data on Aroclors, as given in the MCC bulletins, is derived from this work.

No attempt is made here at a complete survey of the early work.

VIII. CONTROL TESTS.

Determination of Sticky and Gummy Stages of Aroclors 1168, 1169, 1170, 1171

1. Shortly after a specific gravity of 1.570 at 100°C. is passed, the Aroclors will pass into the sticky stage which lasts for a period of 2-4 hours.

2. Place a small amount of the Aroclor on a smooth metal surface, cool slightly and test between the thumb and forefinger, while wearing a rubber glove. If the material is stringy it is in the sticky stage.

3. If the material can be rolled into a ball it is in the gummy stage. The gummy stage follows the sticky stage and lasts 2 - 4 hours.

Determination of Crystallizing pr Hold Points of Aroclors 1168, 1169, 1170, 1171

1. After passing the gummy stage the Aroclors, when tested on a metal surface, solidify immediately into a sort of gray crystalline mass. Hold points can then be run on the chlorinator batch.

2. To run the hold point obtain a sample in a Pyrex test tube and heat to about 200°C. over a gas flame. Agitate with a 0-360°C. thermometer.

3. Insert the test tube into a Dewar tube which is mounted in a large mouth bottle packed with asbestos or rags.

4. Stopper the test tube with a cork stopper through which is inserted the thermometer.

5. Cool slowly, at the rate of 4 - 5°C. every 20 seconds, until the hold point is reached.

6. The temperature will continue to drop until a period is reached where there is no change in temperature for 2 - 3 minutes. If the temperature increases after this period, then the highest

VIII. CONTROL TESTS, contd.

Determination of Crystallizing or Hold Points of Aroclors 1168, 1169, 1170, 1171, contd.

6. contd.
temperature attained will be the hold point. However, if there is no increase in temperature, then the point where it held steady is the hold point.

7. CAUTION: Wear goggles and asbestos gloves when obtaining sample of the solid Aroclors in the molten stage.

Softening Point (SP) Determination of Aroclors 2565, 4065, 4465, 5060, 5065
ASTM Method

The sample shall be melted and stirred thoroughly, to avoid having air bubbles in the mass and then poured into the ring so as to leave an excess on cooling. The ring, while being cooled, should rest on a metal plate. After cooling, the excess material shall be cut off cleanly on top with a slightly heated knife.

Fill the 600 cc. beaker to a depth of 3-1/4" with glycerine at room temperature. Suspend the ring containing the sample and ball in the glycerine so that the lower surface of the ring is exactly 1" above the bottom of the beaker and the upper surface 2" below the upper surface of the glycerine. Suspend the thermometer so that the bottom of the bulb is level with the bottom of the ring and within 1/4" but not touching the ring.

Apply the heat in such a manner that the temperature of the glycerine is raised 5°C. each minute.

The temperature as read on the thermometer at the instant that the melted material from the ring touches the bottom of the beaker will be the softening point.

Run a standard sample along with the chlorinator sample for comparison and make corrections as follows: Suppose the S.P. of the standard sample is 112°C. and drops at 110°C., which indicates too rapid heating, then add 2°C. to the S.P. of the chlorinator sample. It is important to follow the setting of the ring and the thermometer as well as the rate of heating.

VIII. CONTROL TESTS, contd.

Hold Point

A little more detail of the "hold point" test was given by Mather, November 6, 1948:

> A portion of the sample is melted in a 3½" x 1" Pyrex test tube which is then closed with a cork carrying a thermometer, and the tube then hung in the neck of an unsilvered Pyrex vacuum vessel which in turn hangs in the neck of a wide-mouthed glass bottle, forming a support. The thermometer is watched as the temperature falls, then rises as the material crystallizes again and the highest temperature reached in the "hold point".
>
> Samples of finished product are taken before filtration through the Sparkler, and most of the passing out tests are made, on them to save time in packing the O.K. material.

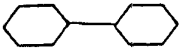
The following sample of material in process has been sent to MCL:

Distilled untreated Aroclor 1254, June 23, 1949

IX. SPECIFICATIONS AND TEST METHODS

Specifications for BIPHENYL (DIPHENYL) (XENENE) (Raw Material)

Code No. 611 Date: 4-6-51

Chemical Formula  Molecular Weight: 154.200

Supersedes Specifications of 8-6-40 Approved WGN SMK
TWD DBH
NB ATL

PROPERTY	SPECIFICATION	METHOD NUMBER
Appearance and Color	Light yellow crystalline solid	10,252-41
Crystallizing Point	68.7°C., Min.	10,298-40
Distilling Range*		
First Drop	252.5°C., Min.	10,299-40
100% (First Drop to Dryness)	1.5°C., Max.	
Dry Point	256.5°C., Max.	

SAMPLE FOR ANALYSIS - 1 x 16 oz. W. M. Bottle

Notes: Above specifications cover Monsanto Biphenyl as made at Anniston.

This Raw Material will be analyzed for the Distilling Range on special request only. However, a complete analysis will be made of the material purchased from outside sources.

High Boilers, Crude and Distilled.

No specifications; used at the Krummrich plant as delivered from Anniston.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for TOLUENE (Raw Material)

PROPERTY	SPECIFICATIONS	METHOD NUMBER
Appearance and Colour	Clear, practically colourless liquid, 10 APHA max.	11220-52
Sulphur	Trace, max.	119-40
Paraffin %	0.5 max.	S-529-3
Barrett acid wash	No. 2 max.	11222-52
Colour of unquenched Sulphonchloride	light brownish, but not opaque	S-529-2
Distilling range °C.		
1st drop	110.0 min.	
97%	within 0.6	
dry point	111.0 max.	
Aliphatics %	0.10 max.	11,799-52

"Aliphatics" and "paraffins" are defined as being measured by tests 11799 and S-529-3 respectively, rather than in their usual chemical sense.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Hydrated Lime (Raw Material)

PROPERTY	SPECIFICATIONS	METHOD NUMBER
Appearance and Colour	Fine white or slightly grey powder, free from lumps or gritty material.	10,188-40
Sulphate, as SO_3 %	0.30 max.	10,189-50
Mg $(OH)_2$ %	0.50 max.	10,192-40
Ca CO_3 %	3.0 max.	10,193-50
Ca $(OH)_2$	92.0 min.	10,191-41

Chlorine Gas.

No specifications. Snift gas from the chlorine cells appears to be satisfactory.

Specifications for Attapulgus Earth. (Raw Material)

The supplier's specification is: --

	Krummrich Plant Method No.
Through 200 mesh screen 95% minimum	12,304
Volatile matter at 950°C. 15% maximum	12,305

Deliveries are not tested except that the screen test is occasionally made.

Moisture content up to, say, 250°C., with a much lower allowance than 15% would appear to be more in line with the requirements of the process.

Cliff Char.

MONS 046126

No MCC specifications.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Tin-tetraphenyl (Raw Material)

No formal Monsanto specification has yet been drawn up, but the material is being purchased on the maker's specification, as follows:

M. Pt.	226 ± 2°C.
Colour	White
Purity	97% based on the reactivity of the material with HCl
Condition	Free from fibres, dirt and paper
Inorganic Chlorides	0.000080% max.* by method 11568-52
Hot water extract	Neutral to phenol phthalein

*Dropped to 50 ppm by agreement with Hooker. Normally only the inorganic chloride test is performed on deliveries to the Krummrich plant.

Material containing 50 ppm of inorganic chlorides is usable, but earlier deliveries, containing perhaps ten times this amount, were purified at the Krummrich plant as follows: (D. B. Hosmer and C. Barbre, January 1950).

1. Each container of tin-tetraphenyl received from the Hooker Company is transferred to a larger Fiberpak container where it is mixed thoroughly.

2. A sample taken from (1) above is tested for its suitability for use in making Pyranol No. 1467 by adding approximately 0.130% by weight of T.T.P. to Pyranol No. 1488 which has been found to be free of chlorides by a previous test. The resulting pilot batch of Pyranol No. 1476 is tested for chloride content by the regular analytical procedure. If the pilot batch is found to contain less than 0.00001% chlorides, the T.T.P., is approved for use. If the pilot batch contains more than 0.00001% chlorides, the T.T.P. is set aside for reprocessing.

3. T.T.P.,which is acceptable for use,is tested again before it is added to a plant batch, by making a second pilot batch from

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Tin-tetraphenyl, contd.

3. contd.
the T.T.P. to be used and a sample of Pyranol No. 1488 from the plant batch.

Our procedure for purifying T.T.P. consists of washing a monochlorbenzene solution of contaminated T.T.P. with city water followed by recovery of the T.T.P. by recrystallization. (See report on Job 115-1740 Refining of T.T.P. by L. A. Hertling. A copy of this report is to be sent to you under separate cover).

This treatment diminishes the inorganic chloride content to about 5 - 10 ppm.

A sample of suitable T.T.P. was sent from the Krummrich plant to Ruabon in January 1950.

Specifications for Glycidyl phenyl ether, Phenoxy propene oxide

$C_6H_5.O.CH_2.CH.OCH_2$

Supplier's specification	(Shell)
Epoxy content	98% w/w minimum
Colour, Hazen Pt. Co. standard	30 max.
Sp. Gr. 20/20 °C.	1.105 - 1.115
Total chlorine as Cl^1	40 ppm max.

Note: A batch showing 0.68% total chlorine gave bad results in use. Another sample showed 0.25%, but only 0.75 ppm inorganic chloride. Another again showed 0.85% total chlorine, but only 1.50 ppm inorganic chlorides.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications of Trichloro benzene, (Raw Material)

(Pyranol 1478 GEC)
Specification H 5228222

PROPERTY	SPECIFICATIONS	METHOD NUMBER
Appearance and Colour	Colourless mobile liquid APHA 10 max.	10,105-53
Condition	Clear	10,105-53
Acid number	0.014 max.	10,087-53
Sp.Gr. 15.5/15.5°C.	1.460 to 1.477	10,106-53
Inorganic chlorides	0.000010% max.	10,195-40
Viscosity, 100°F. SUS	28 - 32	10,086-40
Dielectric Strength, 25°C.	30 K.V.	10,122-40
Refractiv index 25°C.	1.5680 to 1.5715	10,125-40
Distillation range		10,108-42
1st drop	205.0 min.	
5%	210.0 min.	
50%	213.0 min.	
90%	215.0 max.	
Crystalliz.. point	10.0°C. max.	10,101-53
H_2O	0.0075% max.	10,620-41
Corrosion test 6 hours at 210°C.		
Change in weight	nil	10,126-53
Acidity after heating	0.014 max.	
Inorganic chlorides after heating	0.000010 max.	
Condition	clear	
colour	200 APHA max.	

MONS 046129

Specially processed material to make it suitable for electric grades. See also the finished product specifications for Pyranol 1478, page 35 Section IX, below.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Tri-Tetrachlorobenzene blend (Raw Material)

Chemical Formula: Mixture
Sample for Analysis: 3 x 5 pt. Amber Bottles

UNCLASSIFIED SPECIFICATIONS
Routine Tests

PROPERTY	SPECIFICATION	METHOD NUMBER
Color	APHA 15, max.	10,105-53
Condition	Clear	10,105-53
Sp. Gr. at 15.5/15.5°C.	1.510-1.522	10,114-53
Acidity (mg. KOH/g.)	0.014, max.	10,109-53
Moisture (H_2O)	75 ppm., max.	10,620-53
Refractive Index at 25°C.	1.5775-1.5790	10,125-53
Free Chlorides	0.10 ppm., max.	10,118-53
Crystallizing Point	-15°C., or lower	12,035-53
Last Crystal Point	0°C., max.	12,033-53
Dielectric Strength at 25°C.	30 KV, min.	11,605-54
Distillation Range (ASTM D-850, Modified)		10,108-52
First Drop	210°C., min.	
5% by Volume	215°C., min.	
65% by Volume	228°C., min.	
90% by Volume	255°C., max.	
95% by Volume	265°C., max.	
Dry Point	278°C., max.	
Corrosion Test, 6 hrs. at 210°C. with bright aluminum foil		10,126-53
Change in weight of Al	0.0%	
Color	APHA 100, max.	
Condition	Clear	
Acidity (mg. KOH/g.)	0.014, max.	
Free Chlorides	0.10 ppm., max.	
Chemical Composition		12,036-53
Trichlorobenzene	58.5%, min.	
1,2,3,4 Tetrachlorobenzene	26.0-29.0%	
1,2,4,5 Tetrachlorobenzene	3.0% max.	
Pentachlorobenzene	5.5% max.	

General Tests (made only on request)

Fire Point	None to Boiling Pt.	10,123-53

General Information

Dielectric Constant 1000 cycles, 100°C.	4.8 - 5.1	
Viscosity at 100°F.	28-32 SUS	

IX. SPECIFICATIONS AND TEST METHODS, contd.

Comments on Raw Materials.

Diphenyl
See the comments under "Excess Colour", page 10, Section VII, above. Anniston report 171-1077 2883 discusses the use of Dow's "Tarophen" as raw material.

Chlorine.
See the comments on the use of dilute chlorine on page 2, Section VI, above, and on possible impurities in the chlorine on page 10, Section VII.

See also MCC research report 117-790, 2673, December 10, 1951, mentioned on page 25 Section VII, above.

The suggested possible formation of traces of chlorphenols by air dilution is mentioned on page 10, Section VII.

At one time the chlorine gas was filtered, but this seems unnecessary in view of the use of iron as a catalyst in the chlorinator.

Trichlorobenzene.
The Krummrich plant laboratory, February 26, 1951, gave a limited approval of a French sample of trichlorobenzene submitted by MCL.

Copies of the following reports, concerning the suitability of trichlorobenzene supplies have been sent to MCL, May 7, 1948.

"A Method of Determining the Stability of Trichlorobenzene" 117-1987, Munch and Ashworth, April 25, 1948

"Trichlorobenzene and Pyranol 1467 (Infrared Section)" 117-1987, Part I, Sherman, February 23, 1950.

"Tentative Amendment to the Plant Process on TCB" 117-2032, Hubbard and Steahly, February 26, 1948.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Comments on Raw Materials, contd.

Trichlorobenzene, contd.
On November 3, 1950, Dr. Jenkins, in reply to a request for information on history of trouble with the chloride limit, stated that TCB, made at the Krummrich plant around 1948, had shown poor stability, and the cause had been found to be shortage of catalyst in the chlorination stage of manufacture of the TCB.

MCL examination of samples of TCB is discussed in Research Progress reports

347E	50/175/2	Nov.50	(unsatisfactory on electrical tests).
	51/16/1	Jan.51	examination of research samples
355E	50/193/1	Nov.50	examination of Prodelec Co. sample
and 416A	52/19/1	Mar.52	French TTB sample failed on tests but gave passable Pyroclor.
	52/1/9 etc.	Nov.52	I.C.I. sample satisfactory except on chlorides. Attapulgus earth treatment did not correct it.

The following samples, representing raw materials, have been sent to MCL at various times

Attapulgus Earth	August 23, 1949
Tin-Tetraphenyl	January 9, 1950 (Dec.13, 1952)
Benzene as used for making diphenyl at Anniston	January 7, 1948
Diphenyl as made at Anniston	January 7, 1948
Trichlorobenzene	December 13, 1950
Sample G-4072 of Diphenyl G-4073 of Santowax R (distilled highboilers) G-4074 of bottoms of diphenyl still (crude highboilers)	sent from Anniston to Ruabon October 13, 1947
Repeated as I-146-C, I-146-B, I-146-D	January 25, 1949

MONS 046132

IX. SPECIFICATIONS AND TEST METHODS, contd.

Comments on Raw Materials, contd.

Trichlorobenze, contd.

A letter from Mr. Ellenburg, July 14, 1947, discusses the use of higher grade benzene for making TCB, also the quality of bought dichlorobenzene used as raw material.

A Krummrich plant report, September 23, 1947, discusses the stabilization of TCB by heating and by treatment with Attapulgus Earth.

A MCC teletype message, July 13, 1954, is on file, questioning the chloride content of butyl acetate.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - Aroclor 1142 Crude, Commercial

ISSUED DEC 21 1954
STANDARDS DEPARTMENT
Wm. G. Krummrich Plant
By

PRODUCT CODE NO. 246 METHOD IDENTITY --- SALES CODE 1040-130-04-11-03/09

NAME AROCLOR 1142 CRUDE, COMMERCIAL

CHEMICAL FORMULA Mixture MOL. WT. ---

SUPERSEDES SPECIFICATIONS OF 1/6/42 (Anniston) 11/18/40 (WGK) SPECIAL MFG. JOB --

SAMPLE FOR ANALYSIS 1 qt. Bottle (Anniston) 2 x 16 oz. W.M. Bottles (WGK)

UNCLASSIFIED SPECIFICATIONS

Routine Tests

Property	Specification	Method Number	
		Anniston	WGK
Appearance and Color	Brown to black oily liquid	---	10,084-53
Specific Gravity 25°/15.5°C	1.384-1.397	14-49-54	10,114-53
Acid Number (mg. KOH/g.)	0.7, max.	14-46-54	10,087-53

RESTRICTED SPECIFICATIONS

Acid Number of the crude, which is stored in non-lacquered metal containers shall be 0.2 mg. KOH/g. max.

EXTERNAL SPECIFICATIONS

None

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - Aroclor 1148

ISSUED APR 11 1955
STANDARDS OF PERFORMANCE

PRODUCT CODE	METHOD IDENTITY	SALES CODE
246	---	1040-140-04/11-03/09

PRODUCT (Trade name) AROCLOR 1148 TYPE

PRODUCT (Chemical name) TYPE

CHEMICAL FORMULA

Mixture

MOL WT. ---

USE Sales

SUPERSEDES SPECS OF	SPECIAL JOB NO.	SAMPLE FOR ANALYSIS
11/18/40	---	2 x 16 oz. W. M. Bottles

UNCLASSIFIED SPECIFICATIONS

Routine Tests

Property	Specification	Method Number	
		WGK	Anniston
Appearance	Oily liquid	10,084	---
Color	Brown to black	10,084	---
Specific Gravity at 65/15.5°C	1.410-1.420	10,114	14-4-52
Acid Number (mg. KOH/g.)	0.7, max.	10,087	14-42-54

RESTRICTED SPECIFICATIONS

Acid Number of the crude which is stored in non-lacquered metal containers shall be 0.2 mg. KOH/g., max.

ISSUED DEC 21 1954
STANDARDS DEPARTMENT
Wm. G. Krummrich Plant
By

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - Aroclor 1154 Crude, Commercial

PRODUCT CODE NO. 246 METHOD IDENTITY -- SALES CODE 1040-160-04/11-03/09

NAME AROCLOR 1154 CRUDE, COMMERCIAL

CHEMICAL FORMULA Mixture MOL. WT. --

SUPERSEDES SPECIFICATIONS OF 7/2/41 (Anniston) 11/18/40 (WGK) SPECIAL MFG. JOB -- SAMPLE FOR ANALYSIS 1 qt. Bottle (Anniston) 2 x 16 oz. W.M. Bottles (WGK)

UNCLASSIFIED SPECIFICATIONS

Routine Tests

Property	Specification	Method Number	
		Anniston	WGK
Appearance and Color	Brown to black viscous liquid	---	10,084-53
Specific Gravity 65°/15.5°C	1.500-1.510	14-49-54	10,114-53
Acid Number (mg. KOH/g.)	0.7, max.	14-46-54	10,087-53

RESTRICTED SPECIFICATIONS

Acid Number of the crude, which is stored in non-lacquered metal containers shall be 0.2 mg. KOH/g., max.

EXTERNAL SPECIFICATIONS

None

ISSUED DEC 21 1954
STANDARDS DEPARTMENT
Wm. G. Krummrich Plant
By

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - Aroclor 1160 Crude, Commercial

PRODUCT CODE NO. 246 METHOD IDENTITY -- SALES CODE 1040-170-04/11-03/09

NAME AROCLOR 1160 CRUDE, COMMERCIAL

CHEMICAL FORMULA Mixture MOL. WT. --

SUPERSEDES SPECIFICATIONS OF 1/6/42 (Anniston) 11/18/40 (WGK) SPECIAL MFG. JOB

SAMPLE FOR ANALYSIS 1 pt. can (Anniston) 2 x 16 oz. W.M. Bottles (WGK)

UNCLASSIFIED SPECIFICATIONS

Routine Tests

Property	Specification	Method Number	
		Anniston	WGK
Appearance and Color	Brown to black viscous sticky mass	---	10,084-53
Specific Gravity 90°/15.5°C	1.558-1.572	14-49-54	10,114-53
Acid Number (mg. KOH/g.)	0.7, max.	14-46-54	10,087-53

RESTRICTED SPECIFICATIONS

Acid Number of the crude, which is stored in non-lacquered metal containers shall be 0.2 mg. KOH/g., max.

EXTERNAL SPECIFICATIONS

None

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - Aroclor 1162

ISSUED APR 11 1955
STANDARDS DEPARTMENT
Wm. G. Krummrich Plant
By

PRODUCT CODE	METHOD IDENTITY	SALES CODE
246	---	1040-180-04/11-03

PRODUCT (Trade name) AROCLOR 1162 TYPE

PRODUCT (Chemical name) TYPE

CHEMICAL FORMULA

Mixture

MOL WT. ---

USE Sales

SUPERSEDES SPECS OF	SPECIAL JOB NO.	SAMPLE FOR ANALYSIS
11/18/48	---	2 x 16 oz. W. M. Bottles

UNCLASSIFIED SPECIFICATIONS

Routine Tests

Property	Specification	Method Number
Appearance	Viscous liquid	10,084
Color	Brown to black	10,084
Specific Gravity at 90/15.5°C	1.577-1.587	10,114
Acid No. (mg. KOH/g.)	0.7, max.	10,087

RESTRICTED SPECIFICATIONS

Acid Number of the crude which is stored in non-lacquered metal containers shall be 0.2 mg. KOH/g., max.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Crude Solid Aroclors (Finished Products)

	1168	1169	2565	4065	5060	Method Number
Appearance and Colour	Gray to black cryst. powder	Grey to black cryst. powder	Black brittle resin	Black brittle resin	Black brittle resin	10,084-40
Acid number	--	--	0,3 to 1,0	---	--	10,870-40
Hold Point °C.	145 to 165	225 to 250	--	--	--	10,127-40
ASTM Softening Point °C.	--	--	66.0 to 72.0	66.0 to 72.0	110.0 to 115.0	10,085-40

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - Montars 3 and 4

ISSUED APR 11 1955
STANDARDS DEPARTMENT
Wm. G. Krummrich Plant
By

PRODUCT CODE	METHOD IDENTITY	SALES CODE
246	---	*

PRODUCT (Trade name): MONTARS 3 AND 4 — TYPE

PRODUCT (Chemical name) — TYPE

CHEMICAL FORMULA: Mixture

MOL WT.: ---

USE: Sales

SUPERSEDES SPECS OF	SPECIAL JOB NO.	SAMPLE FOR ANALYSIS
	---	2 x 16 oz. W. M. Bottles

UNCLASSIFIED SPECIFICATIONS

Routine Tests

Property	Specification	Method Number
Appearance	Black solid	11,321

*Montar 3 6405-003-04-03/09
Montar 4 6405-004-04-03/09

GENERAL INFORMATION

Physical Properties (typical values)	Montar 3	Montar 4
Softening Point	120-170°C	140-200°C
Acidity	Slightly basic	Slightly basic
Chlorine Content	50-52%	54-56%
CaO	5-10%	5-10%
Fe	0.3-0.6%	0.5-1.0%

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - Aroclor 1242 (Pyranol 1499)

PRODUCT CODE NO. 27 246 METHOD IDENTITY -- SALES CODE 1040-240-04/11-03/09

NAME AROCLOR 1242 (PYRANOL 1499) ISSUED FEB 14 1955 STANDARDS DEPARTMENT Wm. G. Krummrich Plant By

CHEMICAL FORMULA Mixture MOL. WT. --

SUPERSEDES SPECIFICATIONS OF 8/22/41 SPECIAL MFG. JOB -- SAMPLE FOR ANALYSIS 3 x 5 pt. Amber Bottles

UNCLASSIFIED SPECIFICATIONS

Routine Tests

Property	Specification	Method Number	
		WGK	Anniston
Color	APHA 100, max.	10,084-53	14-44-54
Condition	Clear	10,084-53	---
Specific Gravity at 25/15.5°C	1.381-1.392	10,114-53	14-49-54
Acidity (mg KOH/g.)	0.010, max.	10,087-53	14-42-54
Moisture (H_2O)	35 ppm., max.	10,620-53	14-53-54
Refractive Index at 25°C	1.6245-1.6265	10,125-53	14-34-54
Free Chlorides	0.10 ppm., max.	10,118-53	14-48-54
Pour Point	-14°C or lower	10,115-53	14-47-54
Dielectric Constant 1000 cycles, 100°C	4.7-4.9	11,608-54	14-36-54
Resistivity at 100°C 500 VDC and 0.1" gap	500 x 10^9 ohm-cm., min.	11,604-54	14-35-54
Corrosion Test, 6 hrs. at 210°C with bright aluminum foil		10,126-53	14-55-54
Change in weight of Al	0.0%		
Color	APHA 150, max.		
Condition	Clear		
Acidity (mg KOH/g.)	0.010, max.		
Free Chlorides	0.10 ppm., max.		

General Tests (made only on request)

Dielectric Strength at 25°C	35 K.V., min.	11,605-53	---
Sulfates	None	12,169-54	---
Fire Point	None	10,123-53	14-28-54
Viscosity at 100°F	82-92 SUS	10,086-54	14-4-52
Distillation Range (ASTM D-20, Corrected)		10,117-54	14-31-54
10%	325°C, min.		
90%	366°C, max.		

(over)

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - Aroclor 1242 (Pyranol 1499) contd.

GENERAL INFORMATION

Specific Heat at 25°C.	0.30
Coefficient of Expansion (25 - 65°C.)	0.00068 cc/cc/°C.
Fixed Chlorine	41.5 - 42.5 %

NOTE: Mr. Harden, August 1950, reporting on his visit to the Anniston plant, gave a run of test results on Aroclor 1242

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - Aroclor 1248

PRODUCT CODE NO. 246 METHOD IDENTITY --- SALES CODE 1040-260-04/11-03

NAME Aroclor 1248

CHEMICAL FORMULA Mixture MOL. WT. --Dielectric Grade Material - 3 x 5 pt. Amber Bottles

SUPERSEDES SPECIFICATIONS OF 8/22/41 SPECIAL MFG. JOB --+ SAMPLE FOR ANALYSIS All other uses - 3 x 16 oz. W. M. Bottles

UNCLASSIFIED SPECIFICATIONS

Routine Tests

Property	Specification	Method Number
Appearence and Color	Colorless to light yel.-green liquid; APHA 100, max.	10,084-53
Condition	Clear	10,084-53
Specific Gravity 65/15.5°C	1.405-1.415	10,114-53
Acid Number (mg. KOH/gram)	0.010 mg., max.	10,087-53
Moisture (H_2O)	35 ppm, max.	10,620-53
Viscosity at 54.4°C (SUS)	73-80 seconds	10,086-53

The Aroclor 1248 - Dielectric Grade Material (as Pyranol 1498) shall meet the following additional specifications:

Refractive Index at 25°C	1.6285-1.6305	10,125-53
Inorganic (free) Chlorides	0.10 ppm, max.	10,118-53
Pour Point	0°C, max.	10,115-53
Dielectric Constant at 100°C - 1000 cps	4.5-4.7	11,608-54
Resistivity at 100°C, ohm-cm x 10^9 minimum at 500 V-DC, 0.1 "Gap"	500	11,607-54

Corrosion Stability Test — 10,126-53

(After a sample containing one gram of aluminum foil, 0.003" thick, is heated for 6 hours at 210°C with Aroclor, the aluminum must not show corrosion by change of appearance or weight and the sample shall conform to the following requirements):

MONS 046143

Corrosion of Aluminum Foil	None	10,126-53

(over)

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - Aroclor 1248, contd.

The Aroclor used in the corrosion test should meet the following specifications after the test:

Acid Number (mg. KOH/gram)	0.010 mg., max.	10,087-53
Inorganic (free) Chlorides	0.10 ppm. max.	10,118-53
Color	APHA 150, max.	10,084-53
Condition	Clear	10,084-53

General Tests (for Dielectric Grade Material only - to be run on request):

Sulfates	None	12,169-54
Distilling Range (corrected for stem and barometric pressure):		10,117-54
10% Distilled by weight	345°C., min.	
90% Distilled by weight	385°C., max.	
Fire Point, °C., min.	None to Boiling Point	10,123-53
Dielectric Strength at 25°C.	35 KV, min	10,605-53

RESTRICTED SPECIFICATIONS

None

EXTERNAL SPECIFICATIONS

None

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - Aroclor 1254 (Pyranol 1476)

PRODUCT CODE NO. 27 246 METHOD IDENTITY --- SALES CODE 1040-280-04/11-03/09

NAME AROCLOR 1254 (PYRANOL 1476)

ISSUED FEB 14 1955
STANDARDS DEPARTMENT
Wm. G. Krummrich Plant
By

CHEMICAL FORMULA Mixture MOL. WT. ---

SUPERSEDES SPECIFICATIONS OF 8/27/41 SPECIAL MFG. JOB --- SAMPLE FOR ANALYSIS 3 x 5 pt. Amber Bottles

UNCLASSIFIED SPECIFICATIONS

Routine Tests

Property	Specification	Method Number	
		WGK	Anniston
Color	APHA 100, max.	10,084-53	14-44-54
Condition	Clear	10,084-53	---
Specific Gravity at 65/15.5°C	1.495-1.505	10,114-53	14-49-54
Acidity (mg KOH/g.)	0.010, max.	10,087-53	14-42-54
Moisture (H_2O)	35 ppm., max.	10,620-53	14-53-54
Refractive Index at 25°C	1.6370-1.6390	10,125-53	13-34-54
Free Chlorides	0.10 ppm., max.	10,118-53	14-48-54
Pour Point	7-12°C	10,115-53	14-47-54
Dielectric Constant 1000 cycles, 100°C	4.15-4.35	11,608-54	14-36-54
Resistivity at 100°C 500 VDC and 0.1" gap	500 x 10^9 ohm-cm., min.	11,604-54	14-35-54
Corrosion Test, 6 hrs. at 210°C with bright aluminum foil		10,126-53	14-55-54
Change in weight of Al	0.0%		
Color	APHA 150, max.		
Condition	Clear		
Acidity (mg KOH/g.)	0.010, max.		
Free Chlorides	0.10 ppm., max.		

General Tests (made only on request)

Dielectric Strength at 25°C	35 K.V., min.	11,605-53	---
Sulfates	None	12,169-54	---
Fire Point	None	10,123-53	14-28-54
Viscosity at 210°F	44-48 SUS	10,086-54	14-4-52
Distillation Range (ASTM D-20, Corrected)		10,117-54	14-31-54
10%	366-378°C		
50%	372-383°C		
90%	383-396°C		

MONS 046145

(over)

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - Aroclor 1254 (Pyranol 1476), contd.

GENERAL INFORMATION

Specific Heat at 25°C.	0.27
Coefficient of Expansion (25-65°C)	0.00066 cc/cc°C.
Fixed Chlorine	54.5 - 55.5%

EXTERNAL SPECIFICATIONS

Cornell-Dubilier

Resistivity at 100°C. (500 VDC and 0.1" gap)	1500 x 10^9 ohm - cm	11,604-54 14-35-54

Paint Customers

Specific Gravity at 65/15.5°C.	1.495-1.505	10,114-53 14-49-54
Acidity (mg KOH/g.)	0.010, max.	10,087-53 14-42-54
Color	APHA 100, max.	10,084-53 14-44-54
Moisture (H_2O)	35 ppm., max.	10,620-53 14-53-54
Viscosity at 210°F.	44-48 SUS	10,086-54 14-4-52
Flash Point	None	10,123-53 14-28-54

NOTE: Mr. Harden, August 1950, reporting on his visit to the Anniston plant, gave a run of test results on Aroclor 1254.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - Aroclor 1260 (Pyranol 1482)

PRODUCT CODE NO. 27 246 METHOD IDENTITY --- SALES CODE 1040-290-04/11-03/09

NAME AROCLOR 1269 (PYRANOL 1482) ISSUED FEB 14 1955

STANDARDS DEPARTMENT
Wm. G. Krummrich Plant
By

CHEMICAL FORMULA Mixture MOL. WT. ---

SUPERSEDES SPECIFICATIONS OF 8/27/41 SPECIAL MFG. JOB --- SAMPLE FOR ANALYSIS 3 x 5 pt. Amber Bottles

UNCLASSIFIED SPECIFICATIONS

Routine Tests

Property	Specification	Method Number	
		WGK	Anniston
Color	APHA 150, max.	10,084-53	14-44-54
Condition	Clear	10,084-53	---
Specific Gravity at 90/15.5°C	1.555-1.566	10,114-53	14-49-54
Acidity (mg KOH/g.)	0.014, max.	10,087-53	14-42-54
Moisture (H_2O)	35 ppm., max.	10,620-53	14-53-54
Viscosity at 210°F	72-78 SUS	10,086-54	14-4-52
The Aroclor 1260 Dielectric Grade Material (as Pyranol 1482) shall meet the following additional specifications:			
Refractive Index at 25°C	1.6455-1.6470	10,125-53	14-34-54
Free Chlorides	0.10 ppm., max.	10,118-53	14-48-54
Pour Point	25-34°C	10,115-53	14-47-54
Distillation Range (ASTM D-20, Corrected)		10,117-54	14-31-54
10% by weight	385-398°C		
50% by weight	390-404°C		
90% by weight	400-420°C		
Corrosion Test, 6 hrs. at 210°C with bright aluminum foil		10,126-53	14-55-54
Change in weight of Al	0.0%		
Color	APHA 150, max.		
Condition	Clear		
Acidity (mg KOH/g.)	0.014, max.		
Free Chlorides	0.10 ppm., max.		

(over)

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - Aroclor 1260 (Pyranol 1482) contd.

General Tests (made only on request)

Dielectric Strength at 50°C.	30 K.V., min.	11,605-53	---
Resistivity at 100°C. 500 VDC and 0.1" gap	500 x 10^9 ohm-cm., min.	11,607-53	14-35-54
Fire Point	350°C., min.	10,123-53	14-28-54
Fixed Chlorine	59.5% - 60.5%	10,088-53	14-13-54

GENERAL INFORMATION

Dielectric Constant 1000 cycles, 100°C.	3.6 - 3.8	11,608-54	14-36-54
Evaporation Loss after 6 hours at 100°C.	0.2%, max.	10,116-53	14-32-54
Stability test - after 30 days at 100°C. in glass sealed in air.	No liberation of chlorine or chlorides	----	-----

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - Aroclor 1262

ISSUED APR 11 1955
STANDARDS DEPARTMENT
Wm. G. Krummrich Plant
By

PRODUCT CODE	METHOD IDENTITY	SALES CODE
246	---	1040-300-04/11-03

PRODUCT (Trade name): AROCLOR 1262 — TYPE:

PRODUCT (Chemical name): — TYPE:

CHEMICAL FORMULA: Mixture

MOL WT.: ---

USE: Sales

SUPERSEDES SPECS OF	SPECIAL JOB NO.	SAMPLE FOR ANALYSIS
8/22/41	---	2 x 16 oz. W. M. Bottles

UNCLASSIFIED SPECIFICATIONS

Routine Tests

Property	Specification	Method Number
Appearance	Viscous liquid	10,084
Color	APHA 150, max.	10,084
Specific Gravity at 90/15.5°C	1.577-1.587	10,114
Acid No. (mg. KOH/g.)	0.014, max.	10,087
Viscosity at 210°F	86-100 SUS	10,086

General Information

Pour Point (ASTM)	35-38°C
Distilling Range	390-425°C
Total Chlorine	61.5-62.5%
Specific Gravity t coeff (85-130°C)	0.001/°C

MONS 046149

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - Aroclor 1260 - 10% Toluol

PRODUCT CODE	METHOD IDENTIFY	SALES CODE
246	---	1040-295-04/11-03

PRODUCT (Trade name) AROCLOR 1260 - 10% TOLUOL TYPE

PRODUCT (Chemical name) TYPE

ISSUED APR 11 1955
STANDARDS DEPARTMENT
Wm. G. Krummrich Plant
By

CHEMICAL FORMULA

Mixture

MOL WT. ---

USE Sales

SUPERSEDES SPECS OF	SPECIAL JOB NO.	SAMPLE FOR ANALYSIS
11/18/40	---	2 x 16 oz. W. M. Bottles

UNCLASSIFIED SPECIFICATIONS

Routine Tests

Property	Specification	Method Number
Appearance	Syrupy liquid	10,084
Color	Amber	10,084
Composition:		10,194
Aroclor 1260	90.5-89.5%	
Toluol	9.5-10.5%	

GENERAL INFORMATION

Physical Properties - Aroclor 1260

Specific Gravity at 90/15.5°C	1.555-1.566
Specific Gravity t coeff. (75-130°C)	0.001/°C

Physical Properties - Toluol

Specific Gravity at 15.5/15.5°C	0.870
Specific Gravity t coeff. (15-30°C)	0.00089/°C

Physical Properties - 1260 Toluol Mix

Specific Gravity at 40/15.5°C	1.475-1.495

MONS 046150

ISSUED APR 11 1955
STANDARDS DEPARTMENT
Wm. G. Krummrich Plant
By

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - Aroclor 1262 - 10% Toluol

PRODUCT CODE	METHOD IDENTITY	SALES CODE
246	---	1040-310-04/11-03

PRODUCT (Trade name): AROCLOR 1262 - 10% TOLUOL — TYPE:

PRODUCT (Chemical name): — TYPE:

CHEMICAL FORMULA: Mixture

MDL DT.: ---

USE: Sales

SUPERSEDES SPEC DT	SPECIAL JOB NO.	SAMPLE FOR ANALYSIS
7/7/40	---	2 x 16 oz. W. M. Bottles

UNCLASSIFIED SPECIFICATIONS

Routine Tests

Property	Specification	Method Number
Appearance	Syrupy liquid	10,084
Color	Amber	10,084
Composition:		10,194
Aroclor 1262	90.5-89.5%	
Toluol	9.5-10.5%	

GENERAL INFORMATION

Physical Properties - Aroclor 1262

Specific Gravity at 90/15.5°C	1.577-1.587
Specific Gravity t coeff. (85-130°C)	0.001/°C

Physical Properties - Toluol

Specific Gravity at 15.5/15.5°C	0.870
Specific Gravity t coeff.	0.00089/°C

Physical Properties - 1262 Toluol Mix

Specific Gravity at 40/15.5°C	1.497-1.516

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - Aroclor 1254 - 10% Secondary Butyl Acetate

ISSUED MAR 10 1955
STANDARDS DEPARTMENT
Wm. G. Krummrich Plant
By

PRODUCT CODE	METHOD IDENTITY	SALES CODE
246	---	1040-285-04/11-03

PRODUCT (Trade name) TYPE

PRODUCT (Chemical name) TYPE

AROCLOR 1254 - 10% SECONDARY BUTYL ACETATE

CHEMICAL FORMULA

Mixture

MOL WT. ---

USE Sales

SUPERSEDES SPEC'S OF	SPECIAL JOB NO.	SAMPLE FOR ANALYSIS
New	---	2 x 16 oz. W. M. Bottles

UNCLASSIFIED SPECIFICATIONS AND INFORMATION

Routine Tests

Property	Specification	Method Number
Color	APHA 200, max.	12,215-54
Composition:		12,208-54
Aroclor 1254	90.0-91.0%	
Secondary Butyl Acetate	9.0-10.0%	

General Information

Specific Gravity of Aroclor 1254 at 65/15.5°C	1.495-1.505 (changes 0.00095/°C
Specific Gravity of Secondary Butyl Acetate at 25/15.5°C	0.866
Specific Gravity Range of Mixture at 29/15.5°C	1.423-1.449

IX. SPECIFICATIONS AND TEST METHODS. contd.

Specifications for Finished Goods - Distilled Solid Aroclors

	1268	1269	1270	1271	4465	5460	Method No.
Appearance and Colour	white to yellow crystalline powder	white to grey or light yellow crystalline powder	white crystalline powder	practically white light fluffy powder	clear light yellow brittle resin 1.5 NPA (max.)	clear light yellow brittle resin 2.0 NPA (max.)	10,084-40
Hold Point °C.	135 to 160	225 to 255	285 to 300				10,127-40
Acid No. mg. NaOH/g. (max.)	0.05				0.035	0.05	10,087-40
Colour, 5% in toluene (max.)			80 APHA	80 APHA			10,579-41
Moisture % (max.)				0.50			10,577-41
Melting Point °C. (min)				304			10,578-41
Foreign Odour			None			to pass	10,510-40
Total Chlorine %			69.5 to 70.5			59.0 to 60.6	10,088-40
Softening Point °C.						100.0 to 105.5	10,085-40
Viscosity Saybolt Seconds at °C.					160 to 210 130°		10,086-40
Crystallinity							10,089-40

Mr. Harden, August 1950, reporting on his visit to the Anniston plant, gave a run of test results on 5460

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - 60% Aroclor 1260 - 40% Trichlorobenzene (Pyranol 1467)

ISSUED JAN 28 1955
STANDARDS DEPARTMENT
Wm. C. Krumm

PRODUCT CODE NO. A-246 METHOD IDENTITY -- SALES CODE 1050-100-11-03

NAME 60% AROCLOR 1260 - 40% TRICHLOROBENZENE (PYRANOL 1467)

Chemical Formula - Mixture Mol. Wt. ----------

Supersedes Specification of 8-27-541 Special Mfg. Job ---- Samples for Analysis 3 x 5 pt. amber bottles

UNCLASSIFIED SPECIFICATIONS

Routine Tests

Property	Specification	Method Number
Color	APHA 150, max.	10,084-53
Condition	Clear	10,084-53
Specific Gravity at 15.5/15.5°C	1.560-1.568	10,114-53
Acidity (mg KOH/g.)	0.014, max.	10,087-53
Moisture (H_2O)	30 ppm., max.	10,620-53
Refractive Index at 25°C	1.6137-1.6147	10,125-53
Free Chlorides	0.10 ppm., max.	10,118-53
Dielectric Strength	35 KV, min.	11,605-53
Resistivity at 100°C 500 VDC and 0.1" gap	100 x 10^9 ohm-cm., min.	10,604-54
Viscosity at 100°F	52-56 SUS	10,086-54
Pour Point	+32°C, max.	10,115-53
Tin Tetraphenyl	0.115-0.135% by wt.	11,533-53
Distillation Range: (ASTM D-20, Corrected)		10,117-54
First Drop	200°C, max.	
Below 270°C	40%, max.	
90% by wt.	395-415°C	
Corrosion Test, 6 hrs. at 210°C with bright aluminum foil		10,126-53
Change in weight of Al	0.0%	
Color	APHA 200, max.	
Condition	Clear	
Acidity (mg KOH/g.)	0.014, max.	
Free Chlorides	5.0 ppm., max.	

MONS 046154

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - 60% Aroclor 1260 - 40% Trichlorobenzene (Pyranol 1467), contd.

GENERAL TESTS (made only on request)

Dielectric Constant 1000 cycles, 100°C.	3.7-4.0	10,608-54
Fixed Chlorine	59.1% min.	10,088-53
Fire Point	None to Boiling Point	10,123-53
Arc Formed Combustible Gases	1.0%, max.	10,711-
Electrical Stability (Aged 96 hours at 100°C. covered jar, 15% air).		10,121-54
Decrease in resistivity	10% max.	

Analysis of each 10 ml. fraction of a fractional distillation

Chlorine/Hydrogen Ratio	1, min.
Arc Formed Combustible Gases	1.5% max.

GENERAL INFORMATION

Dielectric Constant 1000 cycles, 25°C.	4.0 - 4.3

Inerteen PPO-TCB has the same specification as Pyranol 1467, but it contains 0.20% of glycidyl phenyl ether instead of 0.125% of tin-tetraphenyl.

The fire point is reported every time for Inerteen PPO-TCB.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - 45% Aroclor 1260
55% Tri-Tetrachlorobenzene Blend (Pyranol 1470)

ISSUED JAN 28 1955
STANDARDS DEPARTMENT
Wm. G. Krummrich Plant
By

PRODUCT CODE NO. A-246 METHOD IDENTITY -- SALES CODE 1050-110-11-03

NAME 45% AROCLOR 1260 - 55% TRI-TETRACHLOROBENZENE BLEND (PYRANOL 1470)

CHEMICAL FORMULA Mixture MOL. WT. --

SUPERSEDES SPECIFICATIONS OF New SPECIAL MFG. JOB -- SAMPLE FOR ANALYSIS 3 x 5 pt. Amber Bottles

UNCLASSIFIED SPECIFICATIONS

Routine Tests

Property	Specification	Method Number
Color	APHA 150, max.	10,084-53
Condition	Clear	10,084-53
Specific Gravity at 15.5/15.5°C	1.563-1.571	10,114-53
Acidity (mg KOH/g.)	0.014, max.	10,087-53
Moisture (H_2O)	30 ppm., max.	10,620-53
Refractive Index at 25°C	1.6075-1.6085	10,125-53
Free Chlorides	0.10 ppm., max.	10,118-53
Dielectric Strength at 25°C	35 K.V., min.	11,605-53
Resistivity at 100°C, 500 VDC and 0.1" gap	100 x 10^9 ohm-cm., min.	10,604-54
Viscosity at 100°F	41-45 SUS	10,086-54
Pour Point	-44°C, max.	10,115-53
Tin Tetraphenyl	0.115-0.135% by wt.	11,533-53
Distillation Range: (ASTM D-20, Corrected)		10,117-54
First Drop	210°C, min.	
35% by volume	240-256°C	
55% by volume	290-330°C	
65% by volume	385-400°C	
90% by volume	395-415°C	
Corrosion Test, 6 hrs. at 210°C with bright aluminum foil		10,126-53
Change in weight of Al	0.0%	
Color	APHA 200, max.	
Condition	Clear	
Acidity (mg KOH/g.)	0.014, max.	
Free Chlorides	5.0 ppm., max.	

(over)

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - 45% Aroclor 1260 - 55% Tri-Tetrachlorobenzene Blend (Pyranol 1470), contd.

GENERAL TESTS (made only on request)

Dielectric Constant 1000 cycles, 100°C.	3.8 - 4.2	10,608-54
Fixed Chlorine	60.0 - 61.0	10,088-53
Fire Point	None to Boiling Point	10,123-53
Arc Formed Combustible Gases	1.0% max.	10,711-
Electrical Stability (Aged 96 hours at 100°C. covered jar, 15% air)		10,121-54
Decrease in Resistivity	10%, max.	

GENERAL INFORMATION

Dielectric Constant 1000 cycles, 25°C.	4.5 - 4.9

Inerteen PPO - TTCB has the same specification as Pyranol 1470, but it contains 0.20% of glycidyl phenyl ether instead of 0.125% of tin-tetraphenyl.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - Trichlorobenzene (Pyranol 1478)

PRODUCT CODE NO. B-233 METHOD IDENTITY -- SALES CODE 8510-000-05-03

NAME TRICHLOROBENZENE (PYRANOL 1478) ISSUED JAN 28 1955
STANDARDS DEPARTMENT

CHEMICAL FORMULA Mixture* MOL. WT. -- Wm. G. Krummrich Plant
BY

SUPERSEDES SPECIFICATIONS OF 8/27/41 SPECIAL MFG. JOB -- SAMPLE FOR ANALYSIS 3 x 5 pt. Amber Bottles

UNCLASSIFIED SPECIFICATIONS

Routine Tests

Property	Specification	Method Number
Color	APHA 15, max.	10,105-53
Condition	Clear	10,105-53
Specific Gravity at 15.5/15.5°C	1.460-1.477	10,114-53
Acidity (mg KOH/g.)	0.014, max.	10,109-52
Moisture (H_2O)	75 ppm., max.	10,620-53
Refractive Index at 25°C	1.5680-1.5715	10,125-53
Free Chlorides	0.10 ppm., max.	10,118-53
Crystallizing Point	10°C, max.	10,107-53
Dielectric Strength at 25°C	30 K.V., min.	11,605-54
Distillation Range: (ASTM D850, Modified) (Corrected for Stem and Barometric Pressure)		
First Drop	205°C, min.	
5% by volume	210°C, min.	
50% by volume	213°C, min.	
90% by volume	215°C, min.	
Corrosion Test, 6 hrs. at 210°C with bright aluminum foil		10,126-53
Change in weight of Al	0.0%	
Color	APHA 200, max.	
Condition	Clear	
Acidity (mg KOH/g.)	0.014, max.	
Free Chlorides	0.10 ppm., max.	

GENERAL TESTS (made only on request)

Fire Point	None to Boiling Point	10,123-53
Fixed Chlorine	58.5%, min.	10,088-53

GENERAL INFORMATION

Dielectric Constant 1000 cycles, 100°C.	3.8 - 4.3	
Viscosity at 100°C.	28 - 32 SUS	

MONS 046158

*This is a mixture of the various isomers of Trichlorobenzene plus

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Gopds - 75% Aroclor 1254
25% Trichlorobenzene (Pyranol 1481)

PRODUCT CODE NO. A-246 METHOD IDENTITY -- SALES CODE 1050-120-11-03

NAME 75% AROCLOR 1254 - 25% TRICHLOROBENZENE (PYRANOL 1481)

CHEMICAL FORMULA Mixture MOL. WT. --

SUPERSEDES SPECIFICATIONS OF New SPECIAL MFG. JOB -- SAMPLE FOR ANALYSIS 3 x 5 pt. Amber Bottles

UNCLASSIFIED SPECIFICATIONS

Routine Tests

Property	Specification	Method Number
Color	APHA 150, max.	10,084-53
Condition	Clear	10,084-53
Specific Gravity at 15.5/15.5°C	1.525-1.535	10,114-53
Acidity (mg KOH/g.)	0.010, max.	10,087-53
Moisture (H_2O)	35 ppm., max.	10,620-53
Refractive Index at 25°C	1.6205-1.6215	10,125-53
Free Chlorides	0.10 ppm., max.	10,118-53
Dielectric Constant 1000 cycles, 100°C	4.1-4.6	10,608-54
Resistivity at 100°C, 500 VDC and 0.1" gap	100 x 10^9 ohm-cm., min.	10,604-54
Viscosity at 100°F	70-82 SUS	10,086-54
Pour Point	-15°C or lower	10,115-53
Corrosion Test, 6 hrs. at 210°C with bright aluminum foil		10,126-53
Change in weight	0.0%	
Color	APHA 200, max.	
Condition	Clear	
Acidity (mg KOH/g.)	0.010, max.	
Free Chlorides	0.10 ppm., max.	

GENERAL TESTS (made only on request)

Fire Point	None to Boiling Point	10,123-53
Distillation Range: (ASTM D-20, Corrected)		10,117-54
1st Drop	205°C, min.	
Below 270°C	25%, max.	
90%	380-395°C	

GENERAL INFORMATION

MONS 046159

Fixed Chlorine	55.5%, min.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - 60% Aroclor 1260
40% Trichlorobenzene

PRODUCT (Trade name)	TYPE	PRODUCT CODE
PYRANOL 1488		A-246
PRODUCT (Chemical name)	TYPE	METHOD IDENTITY
60% AROCLOR 1260 - 40% TRICHLOROBENZENE		---
CHEMICAL FORMULA		SALES CODE
Mixture		1050-130-11-03
		MDL DT.

		USE
		Sales

SUPERSEDES SPECS OF	SPECIAL JOB NO.	SAMPLE FOR ANALYSIS
8/27/41	---	3 x 5 pt. Amber Bottles

UNCLASSIFIED SPECIFICATIONS

Routine Tests

Property	Specification	Method Number
Color	APHA 150, max.	10,084-53
Condition	Clear	10,084-53
Specific Gravity at 15.5/15.5°C	1.560-1.568	10,114-53
Acidity (mg KOH/g.	0.014, max.	10,087-53
Moisture (H_2O)	35 ppm., max.	10,620-53
Refractive Index at 25°C	1.6137-1.6147	10,125-53
Free Chlorides	0.10 ppm., max.	10,118-53
Dielectric Strength at 25°C	35 KV, min.	11,605-53
Resistivity at 100°C 500 VDC and 0.1" gap	100 x 10^9 ohm-cm., min.	10,604-54
Viscosity at 100°F	52-56 SUS	10,086-54
Pour Point	-32°C, max.	10,115-53
Distillation Range (ASTM D-20, corrected)		10,117-54
First Drop	200°C, min.	
Below 270°C	40%, max.	
90% by wt.	395-415°C	
Corrosion Test, 6 hrs. at 210°C with bright aluminum foil		10,126-53
Change in weight of Al	0.0%	
Color	APHA 200, max.	
Condition	Clear	
Acidity (mg. KOH/g.)	0.014, max.	
Free Chlorides	0.1 ppm., max.	

MONS 046160

General Tests (Made only on request)

Dielectric Constant 1000 cycles, 100°C	3.7-4.0	10,608-54
Fire Point	None to boiling point	10,123-53
Fixed Chlorine	59.1%, min.	10,088-53
Arc Formed Combustible Gases	1.0%, max.	10,711-

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specifications for Finished Goods - 60% Aroclor 1260
40% Trichlorobenzene, contd.

Electrical Stability (aged 96 hours at 100°C., covered jar, 15% air) Decrease in Resistivity	10%, max.	10,121

EXTERNAL SPECIFICATIONS

Wagner Electric Company

Power Factor (60 at 100°C.)	2%, max.	11,608
Resistivity at 100°C. 500 VDC and 0.1" gap	3000 x 10^9 ohm-cm min.	11,607

General Information

Dielectric Constant 1000 Cycles, 25°C.	4.0 - 4.3

IX. SPECIFICATIONS AND TEST METHODS, contd.

Finished Products

Specifications for Muriatic Acid 20° Bé. (American) Commercial.

PROPERTY		SPECIFICATION	Method No.
Appearance and Colour		Colourless to light yellow liquid 1.5 NPA max.	10,070-40
Turbidity		Practically clear	10,071-40
Sulphates as H_2SO_4 %	(max.)	0.01	10,073-40
As_2O_3 %	(max.)	0.0001	10,074-40
Assay (HCl) %	(min.)	31.45	10,072-40

IX. SPECIFICATIONS AND TEST METHODS, contd.

Finished Product Specifications and Test Methods.

Close cooperation with the General Electric Company has cleared up certain misunderstandings about the specifications and test methods for electrical grade materials, and there is now very little trouble in meeting G. E.'s requirements. Sometimes, for example, in the past, the acidity figures had been set in terms of mg. NaOH per gram of sample, and sometimes in terms of mg. KOH, and the numerical value of the specification limit had not always been changed to suit, and such points have now been corrected. There had also been confusion between "corrected" and "uncorrected" temperatures in the distillation range test.

G. E.'s main plant blend their own Pyranols from MCC Aroclor, and purchased chlorobenzenes, but MCC do the blending for G. E.'s Canadian and West Coast plants, and for G. E.'s licencees. G. E. still receive advance samples of components intended for their use, and samples of finished blends before despatch, though they are coming to rely more on MCC control test work. For example, the "advance" samples of components may be sent along with the sample of the finished blend.

A set of the specifications and test methods,as finally established about a year ago between MCC and the customers,is being obtained for MCL. Both G. E. and Westinghouse give the blends a light treatment with earth to improve the electrical properties; the resistivity, in particular, falls during packing, and during transit of the goods from MCC to the customer.

It will be noted that a mixture of tri-tetrachlorobenzene is largely replacing the trichlorobenzene. This is a change originated by G.E. mainly on grounds of cost.

Recently a blend of Aroclor with secondary butyl acetate has come into use (not for electrical purposes). While the butylacetate is being handled, the same fire precautions are observed as with toluene. The butyl acetate mixtures are made in the finished Aroclor vessels CT-0780 and CT-01400.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 8-9-54 Material: General Method Method No. 10,006-54
WGK G.M. No. 1

By: SMK:HOH Test: Distilling Range

The distillation test method as used by WGK Plant in testing its various products and raw materials is an adaptation of the method as outlined by the Barrett Company for the "Distillation of Nitration - and Industrial Pure Benzol.

A. Flasks: - The flask shall be the Standard Barrett Benzol Flask of 200-ml. capacity. For testing materials boiling above 100°C. the flask will be covered with a layer of asbestos paper, cutting into the latter a circular hole 1½ inches in diameter, with the center of the hole coincident with the bottom of the flask, and leaving the glass completely bare. The sidearm is not covered.

Two rectangular slits are also cut in the asbestos covering the neck of the flask, one on each side of the neck, and the center of each located 90° from a vertical plane bisecting the sidearm of the flask. The rectangular slits shall be 3/8" wide, and 2-½" long. The top and the bottom of each slit shall be 7/8" from the top and bottom of the flask neck. These serve to determine the thermometer bulb position, and the progress of the refluxing procedure.

Two circular holes are cut in the asbestos covering the bulb of the flask, one on each side, each 1-1/2" in diameter, and the center of each located in the center of the side of the flask bulb. These enable observation of the dry point to be made.

For testing materials boiling below, or including, 100°C., uncovered Standard Barrett Benzol Flasks of 200 ml. capacity are used.

B. Corks: - Select a new, sound #10 cork, free from holes of grooves which might cause escape of vapor. Choose a sharp cork borer of slighly smaller diameter than the thermometer to be used, and employing a slicing action rather than excessive pressure, bore the cork about half through from the bottom, then complete the hole from the top of the cork. The hole must be exactly centered when viewed at each end. Gently

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Distilling Range, Method No. 10,006-54, contd.

increase the hole-size with a rat-tail file, to fit the thermometer snugly. Discard corks which have split, even partly.

Using a similar procedure, bore a hole in a #5 cork to fit the sidearm of the flask snugly.

C. Condenser: - The distillate is condensed in a straight glass tube 0.5 inch I.D. and 24" long set at an angle of 75° to the vertical.

For materials boiling below, or including, 100°C., the tube will be cooled in a trough filled with ice-water mixture having a temperature of less than 5°C. (measured) throughout the distillation. The trough will be filled so that the entire length of glass in the trough is covered.

For materials boiling 100-200°C., the trough will be filled to the same extent, but with water only. Distillates which crystallize in the tube must be kept fluid by the use of sufficiently hot water.

For materials boiling above 200°C., the condenser tube will be air-cooled only. Any crystals must be kept from forming in the tube by playing a flame over its length as needed.

D. Receivers: - The receiver shall be either a standard 100-ml. graduated Pyrex cylinder, or one of the formed type distillation receivers. These are made up specially and are graduated from 0-10 ml. and 90-100 ml. in 0.5 ml. divisions along a relatively narrow glass tube, and 10-90 ml. in 10-ml. divisions along the bulb. The overall height is about 11 inches. The formed receivers are used for materials normally liquid at room temperatures. The standard 100 ml. Pyrex cylinders are used for materials which crystallize at room temperatures or above.

E. Heat Source: - Only the Bunsen type burner may be used, the entire flame being blue. The Meker types must not be used. A burner shield about 6" in diameter and sufficiently high to fully protect the flame shall be used.

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Distilling Range, Method No. 10,006-54, contd.

F. Thermometers: - The thermometer shall be an instrument of suitable range for the material being tested. If an accurate thermometer is used, it shall be checked against a U.S. Bureau of Standards Standardized thermometer and the proper corrections applied to the readings.

Normally the accurate thermometers used have a range of 35°, are graduated in 0.1°C. and are capable of being read to 0.05°C.

Select, if possible, a thermometer which will indicate the anticipated range desired in its upper two-thirds of graduation, so as to prevent the cork or flask from interferring with readings.

The usual accurate thermometer will be scaled for three inch immersion so no stem correction need be applied. If, however, a full immersion type thermometer is used, the stem correction is calculated and applied:

The calculation is: --

°Correction =

(No. of degree graduations exposed)(Temp.diff.in°)(0.00158)

The exposed graduations are read from the top of the cork. The temperature difference is the reading of the thermometer minus the temperature of the stem midway of the exposed mercury column as taken by an auxiliary thermometer. The correction shall be added.

G. Transite Boards: - The transite board used should be ca. 6" square, 3/16" thick and shall have a hole with a diameter corresponding to the following:

Materials boiling below 100°C. -	½" diameter hole
100-150°C. -	1" diameter hole
150-200°C. -	1½" diameter hole
200-250°C. -	2" diameter hole
250-300°C. -	2½" diameter hole
above 300°C. -	2½" diameter hole

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Distilling Range, Method No. 10,006-54, contd.

The flasks shall not be set directly on the transite boards during distillations, but shall be set upon a sheet of asbestos paper having a hole whose diameter is slightly smaller than the hole of the transite board. This provides a better seal, to prevent the flame from touching the sides of the flask and superheating the vapours.

H. Manipulation: - Swab out the condenser with a piece of dry cloth and a wooden ramrod.

1. Measure 100 ml. of the liquid sample in the receiver and transfer to the flask, being careful not to get any sample into the sidearm.

2. Seat the cork and thermometer, which is at room temperature, in the neck of the flask, adjusting the thermometer so that the top of the expansion bulb, or the top of the upper expansion bulb, if there are two, is level with the bottom of the sidearm of the flask. Be sure the thermometer bulb is in the center of the neck of the flask, and not inclined to the side.

3. Clamp the flask in place on the asbestos sheet, which rests on a transite board having the proper hole-size previously designated.

4. Connect the flask to the condenser in such a manner that the sidearm extends 2" into the condenser. The neck of the flask and the thermometer shall be in a truly vertical position.

5. Place the receiver in position at the lower end of the condenser tube without further draining or drying.

6. Place water, ice-and-water, or hot water in the trough, as indicated previously, if any is required.

7. The flask is then gently heated until the liquid begins to boil. Use only a Bunsen burner and an all blue flame. Center the flame, while heating, in the middle of the exposed glass at the bottom of the flask.

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Distilling Range, Method No. 10,006-54, contd.

8. As the vapours rise in the neck of the flask, a ring of condensed liquid forms. When this ring of condensed liquid envelopes the bulb of the thermometer and approaches the sidearm outlet, tilt the burner back until the ring subsides back into the flask, and the mercury in the thermometer recedes.

9. Restore the burner to position and repeat this "refluxing" procedure, which allows the thermometer time to reach the proper temperature.

10. Repeat once more, and when this is accomplished, restore the burner to position and quickly adjust the flame height to that which experience dictates will conduct the distillation at a rate of 2 drops per second unless otherwise specifically noted.

11. Thermometer readings are taken and recorded at the following intervals: 1st drop - the instant the 1st drop of distillate falls from the end of the condenser tube into the receiver; 1 ml., 3 ml., 5, 10, 20, 30, 40, 50, 60, 70, 80, 90, and then in 1 ml. intervals on up to the dry point. The dry point temperature is read at the instant the last drop of liquid material is vapourized from the bottom of the flask. At this same time, the burner is turned off. Any distillation showing less than 96 ml. recovered must be re-run.

12. The temperatures are all recorded at first as uncorrected. The barometer is read during the distillation procedure. The net correction incorporating the barometric correction, thermometer correction and stem correction (if required) is calculated and applied to the readings to obtain the corrected readings which are reported.

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Distilling Range, Method No. 10,006-54, contd.

Corrections for deviation of the barometric pressure from the normal are applied using the following formula:

$$\triangle T_B = (0.00012\ T_B)\quad(\triangle_P)$$

in which $\triangle T_B$ = Change in boiling point

T_B = Normal boiling point in degrees absolute

P = Change in pressure in mm. Hg (MacDougall "Thermodynamics and Chemistry", p.133.)

This correction is added if the Barometer is below 760 mm. and subtracted if the Barometer is above 760 mm. For your convenience, these corrections are already tabulated in a chart at the front entrance to the Laboratory.

NOTES: After the completion of a distillation test, the flask shall be disconnected from the condenser, and the condenser tube immediately wiped dry by means of a cotton patch and a wooden ramrod.

The thermometer is allowed to remain in the flask until the mercury has contracted into the thermometer's expansion chamber, if there is one. Never cool a hot thermometer suddenly.

The flasks and receivers are cleaned with the proper reagents and finally rinsed with distilled water and then alcohol. They are then placed in an oven at 110-150°C. to dry. Before using, the flasks and receivers are blown with filtered air for several minutes.

See specific method for details.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 3/25-53 Material: General Method Method No. 10,007-53

By: SMK,NPA:SMK Test: Color - APHA (Hazen) WGK G.M. No. 2

Introduction:

The colour of WGK Plant products for which the tints (hues) are not very pronounced are estimated by comparison with standards in 50-ml. tall form Nessler tubes. The standards are made up in accordance with the directions as given in the APHA Standard Methods of Water Analysis 7th Ed. 1933, pp. 9, as worked out by A. Hazen.

Preparation of the APHA Stock Solution:

1. Weigh accurately on an analytical balance 1.245 gms. potassium chloroplatinate (K_2PtCl_6), containing 0.5 gm. Platinum, and 1.000 g. cobaltous chloride ($CoCl_2.6H_2O$) containing about 0.25 g. Cobalt.

2. Add both components to one liter flask with 100 ml. C.P. HCl and distilled water.

3. Make up to one liter with distilled water and mix well. This is the APHA stock solution. It has a colour APHA - 500.

4. Keep the stock solution in a black painted bottle.

NOTE: In the absence of a reliable supply of potassium chloroplatinate, chloroplatinic acid may be substituted as follows:

Dissolve 0.5 g. metallic platinum in aqua regia; remove nitric acid by repeated evaporation to dryness after adding an excess of hydrochloric acid. Dissolve this product together with 1 gram of cobalt chloride as above directed.

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Colour APHA (Hazen), Method No. 10,007-53, contd.

Preparation of the APHA Standards:

1. To prepare standards, add the proper volume of the stock solution according to the following chart, to a set of matched tall-form, 50-ml. Nessler tubes.

APHA Std.	ml. Stock Sol'n.	APHA Std.	ml. Stock Sol'n.
3	0.3	30	3.0
5	0.5	40	4.0
8	0.8	50	5.0
10	1.0	60	6.0
15	1.5	80	8.0
20	2.0	100	10.0
25	2.5	200	20.0

NOTE: For every unit of APHA, 0.1 ml. of the stock solution is used.

2. Dilute with distilled water contents of each Nessler tube to the mark.

IMPORTANT: The prepared standards should be kept in the closed boxes provided for them to prevent excess evaporation and contamination.

Procedure for Colour Determination:

1. Fill a standard 50-ml. tall-form Nessler tube to the mark (to a height equal to that in the APHA Std. tubes) with the material to be examined.

2. Compare the tube with standards. The intensity of light passing through the sample shall be observed and compared with that of the standard.

IX, SPECIFICATIONS AND TEST METHODS, contd.

General Method, Colour APHA (Hazen), Method No. 10,007-53, contd.

Procedure for Colour Determination, contd.

3. Make the observation by looking vertically downward through the tubes upon a white surface placed in such a position that non-glaring daylight is reflected through the column of liquid. the regular daylight lamps can be used for night work.

IMPORTANT: Since our products vary in hue, the property sought is light intensity, rather than color (hue, tint).

This method is good only up to a color intensity of about 100. The APHA suggests dilution to read the higher values, but we have found that not all materials can be treated in this manner.

The results are reported as so many units of Colour APHA -- "A.P.H.A. 25", or in some cases as "Colour - Hazen 25".

Report the result to the nearest APHA standard, or by interpolation between standards.

Remarks: The standard Nessler tube (refer to Sargent Catalog #S - 21035) is approximately 12" long with 3/4" O.D., graduated at 50-ml. mark.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 8-19-53 Material: General Method Method No. 10,052-51

By: DKL:SMK Test: Melting Point WGK G.M. No. 3

Apparatus:

The melting point apparatus used is the modified Thiel type described in Drawing No. D-779 by Dept. 116 of Monsanto Chemical Company, J. F. Queeny Plant. The bath liquid is Dow-Corning #D.C-550, and sufficient is used in the apparatus so that the liquid level is coincident with the top inside of the crossarm when the liquid is at the temperature of the test and the thermometer and stirrer are inserted. The bath is heated electrically by means of Nichrome ribbon heating elements. The temperature is controlled with a variable transformer or other suitable voltage controlling device. A lighted magnifier is placed before the bath window to facilitate observation.

The melting point tubes are unwashed Pyrex glass capillary tubes, sealed at one end 1.20 ± 0.15 mm O.D. and 0.75 ± 0.15 mm I.D. and ca. 125 mm long. These tubes are to be stored in clean, and screw capped bottles to protect them from dust.

The thermometers, unless otherwise specified are standardized instruments of 35°C. range, graduated in 0.2°C. increments and capable of being read to 0.05°C. and of the 3-inch or 75 mm immersion types.

Manipulation:

1. Punch enough of the well mixed powdered sample (ca. 200 mesh) into the open end of the capillary tube so that when the tube is righted and tapped and/or gently rubbed with a file, the sample falls to the sealed end to a depth of ca. 0.75 inch.

2. Attach the capillary tube(s) (no more than four) to the proper range thermometer by means of small rubber bands positioned above the liquid level so that the sample(s) lie adjacent to the bulb (reservoir) of the thermometer.

3. Place the thermometer and sample(s) in the center of the (sample side) arm of the melting point bath at such a depth

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Melting Point, Method No. 10,052-51, contd.

3. contd.

that the thermometer will be properly immersed at the test temperature and the sample is clearly visible when viewed through the window with the lighted magnifier.

4. Start the motor-driven stirrer.

5. Adjust the voltage control for the heating element so that the temperature rises rapidly until it is within 10-20°C. below the anticipated melting point, and then adjust the rate of heating to 0.1°C. per minute (± 10 seconds) (unless otherwise directed) until the melting point is reached. The rate of heating is very important. Tests run at other rates must be repeated.

The following points may be read:

Softening Point:
The point at which the material seems to pull away from the side of the tube and becomes mushy.

Melting Point:
(First Meniscus): The point at which there is first seen a portion of liquid that is perfectly clear extending from wall to wall in the capillary.

Complete Melting Point:
The point at which the last traces of solid disappears into the liquid melt.

Report the points required to the nearest 0.1°C. after applying the necessary thermometer corrections.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 8-12-54	Material: General Method	Method No. 10,056-44	
By: SMK, DKL, NPA, GWM:WJG	Test: Total Chlorine in Organics - Parr Sodium Peroxide Bomb Method	WGK	G.M. No. 4

The chlorine content of chlorinated organic materials may be determined by means of the Parr Sodium Peroxide Bomb method which converts the organic chlorine to NaCl that is determined by means of a Volhard titration.

1. Scrub a Parr steel (30 Ni) Na_2O_2 Bomb thoroughly with a test tube brush in hot water. Do not use acid. Rinse thoroughly with distilled water and dry on a hot plate or in an oven at 110°C. Cool to room temperature.

   IMPORTANT: Discard any bomb that is badly etched or shows other obvious mechanical defect.

2. A sample of suitable size to give chlorine equivalent to approximately 40 ml. of 0.1 N $AgNO_3$ (0.14 g. Cl) is used. If the material being tested is a solid, the sample must be ground to pass a 60 mesh sieve. If the material to be tested is a liquid, it is weighed onto 2.0 g. of sodium carbonate A.R. contained in the fusion cup. In some instances it is necessary to add some powdered sugar to give sufficient heat for a good fusion.

   CAUTION: In no case should more than 1.00 g. total organics be fused in this manner, unless experience has shown it to be safe. PUT ON SAFETY GLASSES !

3. Add one scoop (ca. 14 g.) Na_2O_2 to the bomb.

4. Stir the mixture in the bomb with a clean dry stirring rod (1/8-in x 6-in.) until thoroughly mixed. Push any lumps of material below the surface of the material.

   CAUTION: Goggles must be worn during this operation, inasmuch as premature ignition of the charge may take place, especially when the humidity is high. It is imperative to

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Total Chlorine in Organics, - Parr Sodium Peroxide Bomb Method, Method No. 10,056, contd.

CAUTION: contd.
hold the bomb away from the operator and not to hold the head directly above the cup in order to see into it.

5. Wipe the stirring rod thoroughly with a small piece (1/2" x 1") of ashless filter paper. Drop the paper into the bomb and press lightly into the fusion mixture.

6. Cap the bomb. Put it into the jacket and tighten with wrenches provided.

7. Tap the bomb several times on the bench top to compact the fusion mass. DO NOT MIX BY SHAKING.

CAUTION: Prior to lighting the fusion burner, always blow a stream of air around and through the shield to remove any gas which may have accumulated in the area.

8. Ignite the mixture for exactly two minutes within the shield provided for the purpose with the burner set so that the apex of the inner core is just below the bottom of the bomb.

9. Allow the bomb to cool in the air for one minute.

10. Place the bomb in the water bath and allow it to cool to room temperature (ca. 5 min.).

11. Remove the jacket and rinse the outside of the bomb with distilled water.

12. Remove the cap from the bomb and place the bomb on its side in a clean 800 ml. beaker.

13. Wash the cap with distilled water, catching the washings in the beaker.

14. Cover the beaker with a watch glass and add enough distilled water rapidly (between the watch glass and the beaker) to cover the bomb.

15. Heat slowly to boiling.

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Total Chlorine in Organics, - Parr Sodium Peroxide Bomb Method, Method No. 10,056, contd.

CAUTION: Be careful that the solution does not proceed so vigorously that mist is carried out of the beaker. The addition of a few carborundum chips will prevent excessive bumping.

16. When the fusion mass has dissolved, remove the bomb with a clean pair of stainless steel tongs, and wash the bomb and tongs thoroughly, receiving the washings in the beaker. Inspect the bomb carefully for presence of undissolved fusion mass. If any remains, boil further until dissolved completely.

17. Put a stirring rod with a rough end in the beaker to aid in boiling later. Cool to room temperature in the water bath.

18. Neutralize the solution with conc. HNO_3 to litmus paper (ca. 30 ml.). Add 5 ml. excess.

19. Heat the solution to boiling.

20. Add 50.00 ml. standard 0.1 N $AgNO_3$ solution with stirring.

21. Boil gently until the AgCl has coagulated and the supernatant liquid is clear (ca. 15 min.).

22. Cool to room temperature in the water bath.

23. Add one ml. ferric alum indicator and back titrate with standard 0.1 N NH_4CNS to first appearance of faint pink colouration. When the titration is within ca. 0.5 ml. of the end point, stir gently and only within 1 inch of the bottom of the beaker so that the coagulated AgCl is not carried up into the body of the solution, obscuring the end point.

24. Make a blank determination in duplicate each time new reagents are used. Carry out the blank determination except omit the sample using 0.800 g. of sugar in its place and only 10.00 ml.

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Total Chlorine in Organics, - Parr Sodium Peroxide Bomb Method, Method No. 10,056-54, contd.

24. contd.

standard 0.1 N $AgNO_3$ (Step 20). If the duplicates do not check within 0.005 milliequivalents, re-run the blank determination. If the blank is greater than 0.025 milliequivalents, use fresh reagents.

Calculations:

(1) Blank Milliequivalents =

$$\text{ml. 0.1 N } AgNO_3 \times N - \text{ml. 0.1 N } NH_4CNS \times N$$

(2) % Cl =

$$\frac{(\text{ml. 0.1 N } AgNO_3 \times N) - (0.1 \text{ N } NH_4CNS \times N) - \text{Blk} \times 0.035457 \times 100}{\text{Sample Weight}}$$

The method is precise to ± 0.05%.

Report the results to the nearest 0.1%.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 8-12-54 Material: General Method Method No. 10,060-54

By: LG, WJG:SMK Test: Odour WGK G.M. No. 5

The odour of Monsanto Products is quite frequently listed under the specifications for the products. The odour is generally tested in the sample bottle by the sense of smell. The intensity of foreign odour, if any, is estimated by the analyst and a supervisor or shift leader. If necessary, the opinion of a third person should be asked. As a guide for the estimation of the intensity, the terminology given by the American Public Health Association as applicable to water analysis is used, in part, with some slight modifications.

We use only 5 intensities as indicated below.

Term	Definition
None	No foreign odour perceptible.
Very Faint	An odour that would not be detected ordinarily by the average consumer, but that could be detected in the Laboratory by an experienced observer.
Faint	An odour that the consumer might detect if his attention were called to it, but that would not attract attention otherwise.
Distinct	An odour that would be detected readily and might cause the material (water) to be regarded with disfavor.
Strong	An odour of such intensity that the material (water) would be absolutely unfit to use (drink). A term used only in extreme cases.

The interpretation of these terms must necessarily be left to the individual analyst; however, it has been found that fair agreement may be reached. In all cases, any detectable foreign odour should be characterized as to some familiar type of odour.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 2-11-53 Material: Aroclors, Pyranols Inerteens, and Tri-Tetrachlorobenzene Blend Method No.10,084-53 WGK Dept. No. 246

By: HOH,NPA:SMK Test: Appearance, Colour and Condition (Turbidity and Residue)

I. APPEARANCE (Crude or distilled liquid or solid material):

1. Examine the material in the sample bottle and describe its appearance. Report as colourless, light yellow, yellow, tan, light brown, brown or black mobile liquid, viscous liquid, sticky solid mass, brittle resinous mass, or otherwise if appropriate.

II. COLOR (Distilled liquid material):

a. If the colour of the sample is APHA 250 or less proceed as follows:

1. Fill a 50-ml. tall-form Nessler tube to the mark.

NOTE: If the material is of high viscosity, warm up the material carefully until it flows readily.

2. Determine the colour of the sample in terms of APHA units by comparing with the APHA standards in similar tubes. Follow General Method No. 2 (Method No. 10,007).

3. Report the colour to the nearest 5 units.

b. If the colour of the product is APHA 200 to 500, proceed as follows:

1. Pour a 50-ml sample into a 250-ml. Erlenmeyer flask and compare with the APHA standards in similar flasks.

c. If the colour of the material is greater than APHA 500, use the following procedure:

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test on Appearance, Colour and Conditions of Aroclors, Pyranols, Inerteens, and Tri-Tetrachlorobenzene Blend, Method No. 10,084-54, contd.

c. contd.

1. Fill 1-7/16" x 5" NPA tube half full of sample.

2. Place the tube in the ASTM Union Colorimeter and turn on the light at the back of the instrument.

3. Determine colour of the sample according to General Method No. 11 (Method No. 11,732) by matching the sample and standard coloured glasses through the observation hole.

4. Match the sample to the nearest half increment of colour.

The method is precise to ± 1/2 colour increment on all three sets of standards.

Report the result to the nearest 1/2 NPA unit.

III. CONDITION (Turbidity and residue):

1. Observe the sample in the sample bottle.

Report the condition (both turbidity and residue) as clear, very slight, slight, moderate, or considerable.

2. Indicate the presence (if any) and describe the character of any foreign matter present, e.g., black specks, white lint, rust, etc.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 2-26-53 Material: Aroclors (Solid) and Montars Method No. 10,085-53
By: AKB,NPA:SMK Test: Softening Point WGK Dept. No. 246

Introduction:

The softening point of solid Aroclors or Montars (Monsanto Pitch) is determined according to the "Ring-and Ball method (ASTM Designation: D36 - 26, reapproved in 1949) for Softening Point of Bituminous Materials" as described in 1949 Book of ASTM Standard, Part 3, p. 1091.

The apparatus is essentially the same as that illustrated in the ASTM procedure with the exception that two rings and balls are in our assembly instead of one.

Procedure:

1. To prepare the sample melt the material under test and stir it thoroughly.

   CAUTION: While stirring the material, avoid overheating or incorporating air bubbles in the mass.

2. Place a ring on a square of tinned sheet metal and pour molten material into the ring so as to leave an excess on cooling.

3. After the sample has cooled, cut off the excess material and trim the edges of the ring with a heated knife.

4. Prepare another ring in the same manner (Step 1 to 3) using a Standard Aroclor which has been provided for this purpose.

   NOTE: For Montars prepare another ring of the material under test. There is no standard Montar.

5. Fill a 600 ml. beaker to a depth of 3.25 inches with freshly boiled distilled water.

IX. SPECIFICATION AND TEST METHODS, contd.

Test for Softening Point of Aroclprs (Solid) and Montars, Method No. 10,085-53, contd.

6. Place the rings in the support and suspend them 1 inch above the bottom of the beaker.

7. Insert a -2 to 80°C. range, ASTM Softening Point thermometer into a support and adjust it so that the bottom of the mercury bulb is level with the rings and within 1/4 inch of both of them.

8. Place the balls on the bottom of the beaker and maintain the temperature of water at 5 (± 0.5)°C. for 15 minutes.

9. Place the balls in the center of the upper surface of each ring, thus completing the assembly.

   NOTE: Use a suitable forceps for this operation.

10. Apply heat in such a manner that the temperature of the water is raised at a rate of 5°C. per minute.

    NOTE: This rate of rise should be uniform within 0.5°C. after first 3 minutes of heating, otherwise the test shall be rejected.

11. Continue heating until both samples have softened to such a degree that the Aroclor (Montar) touches the bottom of the beaker. Record the temperature at this instant.

12. Correct the temperature reading (Step 11) by applying to it the difference between the observed softening point of the standard Aroclor and the known softening point which is on the label, thus allowing for the variations in the technique.

    NOTE: No temperature corrections are made on Montar "Softening Point" determinations.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Softening Point of Aroclors (Solid) and Montars, Method No. 10,085-53, contd.

13. Report corrected temperature as the "Softening Point" of the Aroclors.

NOTE: For Montar report the observed temperature.

The method is precise to ± 0.5°C.

Report the results as follows:

For Aroclors to the nearest 0.5°C.

For Montars to the nearest 1.0°C.

NOTE: For Aroclors (Montars) with the Softening Point above 80°C. use a slighly modified technique. The modifications are as follows:

a. Use Dow Corning Fluid instead of water in a bath..

b. Suspend the ring apparatus off the center of the container and place the burner midway between the center and the edge of the beaker away from the rings.

c. Use an ASTM High Softening Point Thermometer of a 0° - 360°C. range.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 9-13-54 Material: Aroclor, Pyranol, Inerteen, Tri-Tetra Chlorobenzene Method No. 10,086-54

By: SE,DKL,NPA, WWK: WJG Test: Viscosity WGK Dept. 246

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1. Determine the viscosity according to General Method No. 25 (Method No. 11,433) Part B - Kinematic Viscosity.

2. For the products listed below use the following temperatures and series number of Ostwald Viscosimeter tubes:

Product	Temperature	Tube Series Number
1242 Aroclor (Distilled)	100°F. (37.8°C.)	200
1248 Aroclor	130°F. (54.4°C.)	200
1254 Aroclor (G.E. 1476)(Distilled)	210°F. (98.9°C.)	100
1260 Aroclor (G.E. 1482)(Distilled)	210°F. (98.9°C.)	200
1262 Aroclor (Distilled)	210°F. (98.9°C.)	200
1467 and 1488 Pyranol and Inerteen PPO	100°F. (37.8°C.)	100
1470 Pyranol	100°F. (37.8°C.)	100
1478 Pyranol (TCB)	100°F. (37.8°C.)	50 or 100
1481 Pyranol	100°F. (37.8°C.)	200

Calculation:

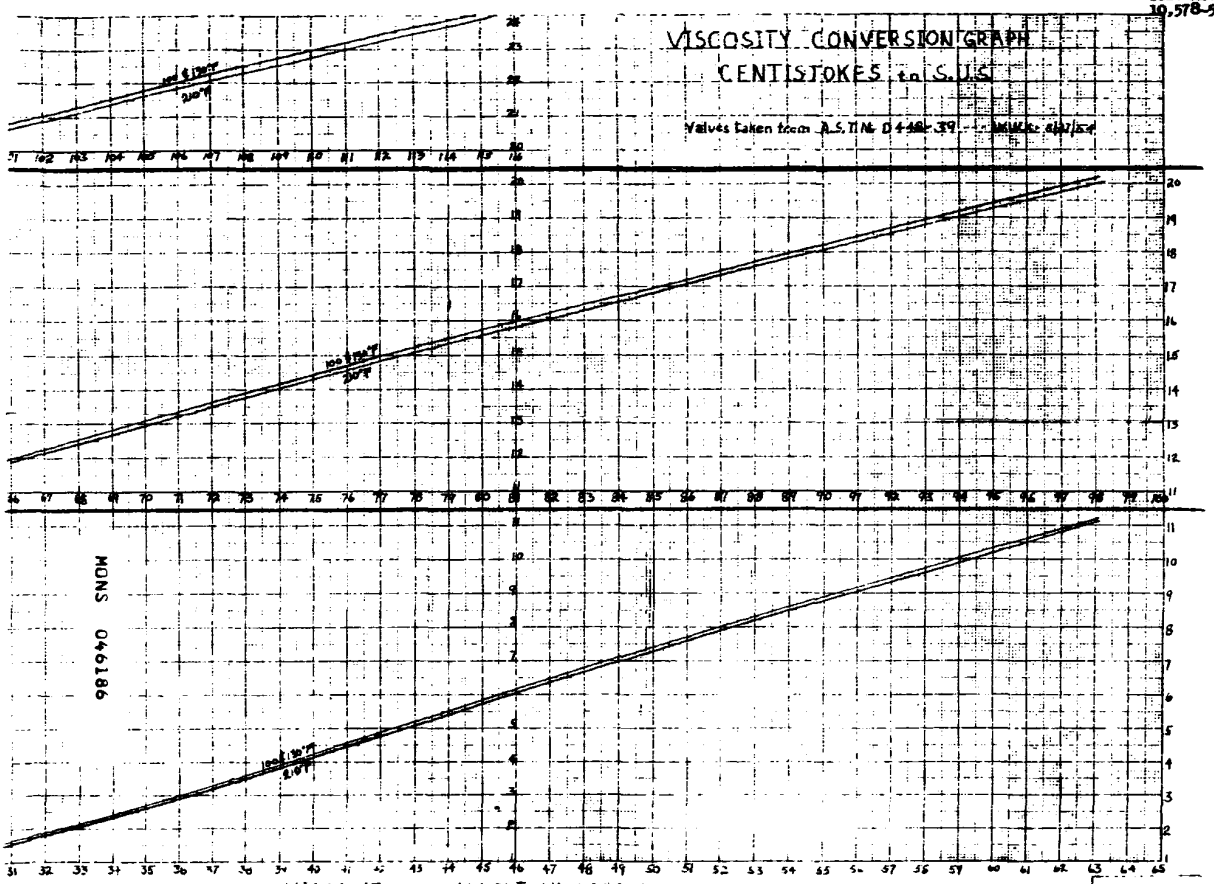
$V = Ct$

Where V = Viscosity in centistokes at T°F.
C = Tube constant (accompanying each tube) at T°F., and
t = efflux time in seconds at T°F.

Determine V to the nearest 0.05 centistokes.

Convert to Saybolt Universal Seconds using Chart No. B-7781

Report to the nearest 0.1 S.U.S.


VISCOSITY CONVERSION GRAPH
CENTISTOKES to S.U.S.
Values taken from A.S.T.M. D 446-39
VISCOSITY in SAYBOLT UNIVERSAL SECONDS

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 3-11-53 Material: Distilled Aroclors, Pyranols, Inerteens and Tri-Tetrachlorobenzene Blend — Method No. 10,087-53

By: DKL, NPA: SMK Test: Acid Number (G.E.Acidity) — WGK Dept. No.233/246

(For the products "as received" and after the "Corrosion Test").

Procedure:

1. Place 100 ml. of benzene, 100 ml. of Methanol (Measure both by a graduate), and 0.5 ml. (by pipette) of Phenol Red indicator into one of two clean, dry 500-ml. Erlenmeyer flasks.

2. Neutralize carefully with the 0.01 N. KOH (to the first definite pink colour).

3. Pour the mixture back and forth between the two flasks several times. If the solution is still neutral, divide it equally between the two flasks, if not, repeat Steps 2 and 3.

4. Weigh on a beam balance into one of the flasks a 75 (± 5.0) g. sample of the material under test.

5. Titrate with 0.01 N. KOH until the sample matches the blank (second 500-ml. flask).

NOTE: For Aroclors 1270 and 1271, toluene is used as the solvent and 300 to 400 ml. is required in order to dissolve the sample. Also, the sample shall be reduced to 10 (± 0.05) grams.

Calculation:

Acid Number (Acidity of G. E. Specifications) =

$$\text{mg. KOH/gram sample} = \frac{\text{ml. 0.01 N KOH} \times 0.56}{\text{Sample Weight}}$$

C

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Acid Number (G.E.Acidity) on Distilled Aroclors, Pyranols, Inerteens, and Tri-Tetrachlorobenzene Blend, Method 10,087-53 contd.

Calculation, contd.

To convert mg. KOH/gram to mg. NaOH/gram (on request only)

mg. NaOH/gram - (mg. KOH/gram) x 0.715

The method is precise to ± 0.002 for acid numbers below 0.01 and to ± 0.01 in the range of 0.1 to 0.01.

Report the results to the nearest 0.001 if they are below 0.1, otherwise to the nearest 0.01.

Reagents:

1. Benzene - Nitration Grade: This may be secured from reserve samples of benzene which have been analyzed for Depts. 223 and 233.

2. Methanol - Anhydrous.

3. Phenol Red: A saturated solution of phenol sulfonphthalein (Phenol Red) in methanol (approx. 0.1%).

4. A 0.01 N KOH solution in methanol: This solution is prepared by the Service Section (see "Procedure Manual for the Service Section", Part 2, page 6).

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 3-11-53 Material: Aroclors, Pyranols, Method No. 10,088-53
By: AEB, DKL, Inerteens WGK Dept. No. 246
NPA:SMK Test: Total Chlorine (Fixed Chlorine)

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1. Determine the total chlorine content of Aroclors, Pyranols, and Inerteens by the Parr Sodium Peroxide Bomb fusion method according to General Method No. 4 (Method No. 10,056) with the following modifications.

2. Use the quantities of sample under test and reagents to be used as listed in the following table:

Chlorine Content Expectancy in a Sample	Sample Weight (Use analytical balance)	Sugar Required (Use analytical balance)	Required Ml.0.1 N $AgNO_3$ (By burette)
0 - 30%	0.5 (±0.0100)g.	0.3(±0.01)g.	50.00 ml.
30 - 45%	0.4 (±0.0100)g.	0.4(±0.01)g.	50.00 ml.
45 - 58%	0.3 (±0.0100)g.	0.5(±0.01)g.	50.00 ml.
58 - 66%	0.25(±0.0100)g.	0.6(±0.01)g.	50.00 ml.
66 - 70%	0.22(±0.0100)g.	0.6(±0.01)g.	50.00 ml.

3. If the sample is liquid, in addition to the above reagents use 2 (± 0.05) g. Na_2CO_3 (weigh accurately on a beam balance), to be placed in the bomb before weighing the sample.

Calculation:

% Total (Fixed) Chlorine (as % Cl) =

$$\frac{\left[(\text{ml. } AgNO_3 \times \text{Normality} - \text{ml. } NH_4CNS \times \text{Normality}) - \text{Blank}^*\right] \times 0.035457 \times 100}{\text{Sample Weight}}$$

* Blank milliequivalents = (ml. $AgNO_3$ x Normality)-(ml.NH_4CNS x N)

The method is precise to ± 0.2%.

Report the result to the nearest 0.1%.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 9-3-54 Material: General Method Method No. 10,100-54

by:DKL,NPA:WJG Test: Water - Traces WGK G.M. No. 7

Introduction:

Water may be determined in most organic and many inorganic compounds by means of a titration with Karl Fischer (K. F.) Reagent, using either the "Dead Stop" or visual end point.

A. "DEAD STOP" Method:

1. Place 100 to 300 ml. of dry methanol in the titration flask (see note at the end of method).

   IMPORTANT: The stopper, which closes the charging opening of the titration flask, must be firmly in place before any attempt is made to proceed with the test. This applies, of course, to both the "blank" and the sample.

2. Start the stirring motor and adjust its speed so that effective agitation is achieved without splashing.

3. Snap the toggle switch on the front of the electron-ray indicator panel to the "ON" position and allow the instrument to warm up for 3 - 4 minutes.

4. Turn the "SOLVENT SELECTOR SWITCH" knob to the position corresponding to the type of solvent (or mixture) used as a solubilizing medium.

   NOTE: The switch positions are: "NONE" (when no other solvent but methanol is used); BENZENE (for benzene-methanol mixtures); TOLUENE (for methanol-toluene mixtures); and ACETIC ACID (for methanol-acetic acid mixtures).

5. Slowly add the K. F. reagent to the titration flask. Observe that the shadows on the indicator tube open momentarily and then close, the length of the "open" time increasing as more

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Water - Traces, Method No. 10,100-54, contd.

5. contd.
K. F. reagent is added. The solvent is "blanked" when the tube shadows remain fully open (ca. 100° each) for 30 seconds. Refill the burette with the K. F. reagent.

6. Weigh accurately (to three significant figures), by difference, a sample containing 0.03 to 0.06 grams of water, but not more than ca. 200 grams. Transfer quickly to the titration flask.

NOTE: Use a powder funnel if the sample is a powdered solid.

7. Allow the material in the flask to be agitated until all the sample is in solution.

8. Titrate the solution with K. F. reagent to the end point described in Step 5. Record the volume of K. F. reagent used.

Calculation:

$$\% H_2O = \frac{\text{ml. K.F. Reagent} \times H_2O \text{ factor} \times 100}{\text{Sample Weight}}$$

The method is precise to ± 5% of the water present.

Report the result to the nearest 5 in the third significant figure.

B. VISUAL METHOD

1. Place 100 - 300 ml. of dry methanol (see note) in a dry 500 ml. g.g.s. Erlenmeyer flask.

2. Titrate the solvent with K. F. reagent to the visual end point, i. e. the first change from the yellow to reddish orange that persists for 30 seconds. Refill the burette.

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Water - Traces, Method No. 10,100-54, contd.

B. VISUAL METHOD, contd.

3. Weigh accurately (to three significant figures), by difference, a sample containing 0.03 - 0.06 grams H_2O into the flask.

4. Stopper and shake until the sample is in solution.

5. Titrate the solution with K. F. reagent to the end point described in Step 2. Record the volume of K. F. reagent used.

CALCULATION:

$$\% H_2O = \frac{\text{ml. K. F. Reagent} \times H_2O \text{ factor} \times 100}{\text{Sample Weight}}$$

The method is precise to $\pm$ 5% of the water present.

Report to the nearest 5 in the third significant figure.

NOTE: Dry benzene or other suitable dry solvents may be used together with methanol in order to render some samples soluble. Dry glacial acetic acid must be added in excess when amines are present in the sample to prevent interference in the water determination.

References:

Mitchell, J. and Smith, D. M. Chemical Analysis, Vol. 5, Aquametry, Interscience Publishers Inc., N. Y. (1948).

Frederickson's Report to Hehner, May 23, 1950.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 3-25-53 Material: Trichlorobenzene Method No. 10,105-53

By: AEB,NPA:SMK Test: Appearance, Colour and Turbidity WGK Dept. No. 233

Trichlorobenzene (TCB) is usually a clear, colourless to light yellow liquid.

Appearance:

1. Examine the sample and describe its appearance noting any unusual characteristics. Report as a clear, colourless liquid, or otherwise, if appropriate.

2. Observe the sample for residue (visible foreign bodies), such as rust, dark specks, lint, etc. Report as none, very slight, slight, moderate, or considerable.

Describe appearance of impurities briefly.

Colour:

1. Determine the colour of this product in terms of APHA units by comparing 50 ml. of the sample to the APHA Standards. Follow General Method No. 2 (Method No. 10,007).

Report the colour to the nearest 5 units.

Turbidity:

1. For information of the manufacturing supervision, determine the turbidity of TCB. Apply General Method No. 15, (Method No. 11,788).

Report the result to the nearest General Turbidity Standard Number.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 3-26-53 Material: Trichlorobenzene Method No. 10,106-53

By: CCS, NPA: Test: Specific Gravity
SMK 15.5/15.5°C. WGK Dept. No. 233

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1. Determine the specific gravity of Trichlorobenzene (TCB) by means of the hydrometer. Apply General Method No. 13 (Method No. 10,497), with the following modifications:

2. Use a 1.400 - 1.600 range hydrometer.

3. Use a 0 - 110°C. range paper scale thermometer.

4. Read the hydrometer scale at a point directly in line with the lower meniscus.

5. Use the temperature correction of 0.0011 per°C.

6. Add this correction to any reading obtained above 15.5°C. and subtract it from readings obtained below 15.5°C.

7. Then make the hydrometer correction, if one is necessary.

The method is precise to ± 0.001 unit.

Report the Specific Gravity 15.5/15.5°C. to the nearest 0.001 unit.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 2-13-52 Material: Trichlorobenzene (Pyranol 1478) Method No.10,108-52
By: DKL:SMK Test: Distilling Range WGK Dept. No.233-246

Determine the distilling range of the sample according to General Method No. 1 (Method No. 10,006-50) using a 190 - 230°C. thermometer and a transite board with an opening 2" in diameter.

Report the following for 1,2,4 TCB:

First Drop
1 - 96 ml. (95% range)
100% (First Drop to Dry Point)
Dry Point

The method is precise to ± 0.1°C., however, if appreciable quantities of low or high boiling material are present, the precision is of the order of ± 0.3°C.

Report the results to the nearest 0.1°C.

Report the following for Technical TCB or Pyranol 1478:

First Drop
5%
50%
90%
Dry Point

The method is precise to ± 0.1°C., however, if appreciable quantities of low or high boiling material are present, the precision is of the order of ± 0.3°C.

Report the results to the nearest 0.1°C.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 2-11-53 Material: Aroclors, Pyranols Inerteen, and Tri-Tetrachlorobenzene Blends Method No. 10,114-53

By: RWS,RAC,SE NPA:SMK Test: Specific Gravity (by Hydrometer) WKG Dept. No. 246

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1. Determine the specific gravity of Aroclors, Pyranols, Inerteen, and Tri-Tetrachlorobenzene Blend according to procedure described in General Method No. 13 (Method No. 10,497) with the following modifications:

   NOTE: The temperaturesat which the specific gravities of the named products are taken vary with the viscosity of the individual material.

2. Find in the table, attached at the end of this method, the temperature at which the gravity is to be taken for the particular product under test and heat the sample to 10°C. above that temperature.

3. Pour the hot sample into the steam-jacketed hydrometer jar provided for this test.

4. Immerse the bulb of a hydrometer of a suitable range in the liquid and stir well with a paper scale thermometer.

5. Adjust the temperature to the desired point by controlling the steam feed to the jacket. Continue stirring until the temperature remains constant for half a minute.

6. Note the temperature, quickly withdraw the thermometer and allow the hydrometer to sink into the liquid at the center of the cylinder.

7. Read the hydrometer scale at the point of the lower meniscus and record with the temperature.

   NOTE: Due to the opaque nature of crude Aroclors, it is necessary to estimate this reading. Usually it is one scale division below the highest visible part of the scale.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Specific Gravity Test (by Hydrometer) for Aroclors, Pyranols, Inerteen, and Tri-tetrachlorobenzene Blend, Method No. 10,114-53, contd.

8. Correct the specific gravity reading from the observed temperature to a temperature required by the specification for the product under test. Use the following table.

Material	Sp. Gr. Correction	Temperature Range
Aroclor 1142-1242	0.00090/°C.	50 - 105°C.
Aroclor 1148-1248	0.00090/°C.	50 - 100°C.
Aroclor 1154-1254	0.00095/°C.	55 - 105°C.
Aroclor 1160-1260	0.00100/°C.	75 - 130°C.
Aroclor 1162-1262	0.00100/°C.	85 - 130°C.
Pyranol 1478-1470	0.00110/°C.	11 - 30°C.
Pyranol 1488-1467	0.00110/°C.	11 - 30°C.
Pyranol 1481	0.00110/°C.	11 - 30°C.
Pyranol 1495	0.00100/°C.	--
Inerteen P.O.P.O.	0.00110/°C.	11 - 30°C.
Tri-Tetrachloro-benzene Blend	0.00110/°C.	11 - 30°C.
Toluene Mixtures	0.00100/°C.	Taken at 40°C.

The determination is precise to ± 0.001 unit (1/2 scale division).

Report the Specific Gravity to the nearest 0.001 unit.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 2-26-53 Material: Aroclors, Pyranols and Inerteens Method No. 10,116-53

By: AEB,NPA:SMK Test: Loss on Heating at 100°C. WGK Dept. No. 246

Introduction:

This test is a modification of the test ASTM Designation D6-39T described in 1949 Book of ASTM Standard, part III, p. 1075-1077.

Procedure:

1. Weigh accurately on an analytical balance a 40 (± 0.00005) g. sample of Aroclor (Pyranol, Inerteen) into a tared (on the same balance) 3 ounce Gill style, flat bottom, seamless ointment box (jar).

   NOTE: If it is necessary to sample the material hot, upon sampling, cool the box and its contents to room temperature in a desiccator before making the weight determination.

2. Place the box with the sample in an oven maintained at 100°C.

3. Keep the sample in the oven at the same temperature for 6 hours.

4. Then cool the sample in a desiccator and reweigh on an analytical balance. Record weight.

Calculation:

$$\% \text{ Loss on Heating (Loss due to volatilization)} = \frac{[\text{Sample wt. (Step 1)} - \text{Sample wt. (Step 4)}] \times 100}{\text{Sample weight (Step 1)}}$$

Report the loss on heating to the nearest 0.01%.

IX. SPECIFICATIONS AND TEST METHODS, cont'd.

Date: 2-4-54	Material: Aroclors, Pyranols, and Inerteens	Method No. 10,117-54
By: AEB, LJW		
NPA,WWK:SMK	Test: Distilling Range	WGK Dept. No. 246

Introduction:

Distillation tests on Aroclors, Pyranols, and Inerteens are made essentially in accordance with the ASTM procedure D20-30 for the "Distillation of Tar Products Suitable for Road Treatment".

Apparatus:

Standard ASTM apparatus is used throughout with the following modifications:

Thermometer: Use a calibrated, 3-inch immersion, Palmer 0-400°C. range thermometer. If a full immersion thermometer is used, correction must be made for emergent stem.

Condenser: Electrically heated condenser controlled by a variac to an internal temperature of 125 ± 10°C. The variac settings, necessary for the desired temperature, for all available condensers are attached to the respective condensers. These settings were established using a variac that delivered 100 volts at a setting of 100. When variacs are changed, the voltage out-put at a setting of 100 must be checked with an A. C. voltmeter. If the voltmeter reading is not within 2 volts of 100, make the necessary setting correction for the stated variac settings. The voltage correction is assumed to be linear over the entire range of the variac.

Heat Source: Electrical-rheostat control.

Material:	Point at which condenser heat should be applied:
Aroclor 1254	At beginning
Aroclor 1260	At beginning
Pyranol 1467	When a pot temperature of 250°C. is reached
Inerteen PPO	
Pyranol 1470	When the distillate volume is 40 ml.
Pyranol 1481	When a pot temperature of 250°C. is reached.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Distilling Range of Aroclors, Pyranols, and Inerteens, Method No. 10,117-54, contd.

Procedure:

1. Weigh and transfer a 100 (± 0.05) g. sample of the product under test to the distilling flask. Take 100 ± 0.5 ml. for Pyranol 1470. Assemble the apparatus as described under ASTM D20-30.

   NOTE: The electrical condenser will be a permanent set-up so that no connection will be necessary.

2. Insert a 3-inch immersion thermometer into a cork so that the three inch (or 76 mm) line is at the bottom of the cork. Place thermometer and cork in the flask making sure the thermometer is in a vertical position.

3. Turn on the heat source of the flask and the condenser heat when indicated above.

4. Turn the rheostat control, for the heat source to the flask, to ca. 120. Leave in this position until exposed mercury column is within 20°C. of the approximate range of distillation.

5. When the distillation starts, reduce the rheostat (on the still-pot) reading until the rate of distillation averages 50-70 drops/minute.

6. Collect the distillate into a tared flask set on a beam balance.

   NOTE: Use a graduated cylinder to collect distillate for Pyranol 1470.

7. Record the temperature at the weight percentages indicated in the individual specifications for all Aroclors, Pyranols, and Inerteens with the exception of Pyranol 1470.

8. Collect distillate of Pyranol 1470 "by volume percent" instead of "Percent by weight". For this Pyranol, record the temperatures as follows: --

IX. SPECIFICATIONS AND TEST METHODS, contd.

Distilling Range of Aroclors, Pyranols, and Inerteens, Method No. 10,117-54, contd.

8. contd.

First Drop
35% by volume
55% " "
65% " "
90% " "

9. Make the following thermometer corrections (to be added algebraically to observed temperatures):

A. Barometric Pressure:

$$C = 0.00012\ (760-P)\ (273 + T_c)$$

Where P = observed pressure in mm.
T_c = observed temperature in °C.

B. Emergent Stem Correction:

If 3 inch immersion thermometer is used there is no emergent stem correction needed.

If a full immersion thermometer is used then the positive emergent stem correction is:

$$C_e = L\ (T_e - T_o)\ \ 0.000154$$

Where L = °C. on emergent stem.
T_e = observed temperature in °C.
T_o = temperature of emergent stem at mid-point in °C.

NOTE: The use of a full-immersion thermometer is not recommended because of the difficulties involved in obtaining an accurate measurement of T_o.

Report the temperatures (corrected) to the nearest 1°C.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 3-6-53 Material: Aroclors, Pyranols, Inerteens, and Tri-Tetrachloro-benzene Blend Method No. 10,118-53

By: RAC, NPA:SMK Test: Inorganic(G.E.Free)Chlorides WGK Dept. No. 246

(For the products "As received" and after the "Corrosion Test").

1. Thoroughly rinse two separatory funnels with chloride-free water three or four times. Then take an aliquot from each funnel in a test tube which has been rinsed with chloride-free water. Test these aliquots for Tyndall beams by adding 3 - 5 drops of $AgNO_3$ and allowing 45 seconds for full beam to evolve. Absolutely no dust or chloride beam should be present.

   Note: If beam is present rinse all equipment with dilute HNO_3 (1:1) and repeat Step 1.

2. When funnels are beam-free, drain out all the water except 50-ml. in one and 25-ml. in the other. Heat the water in both funnels to boiling.

   NOTE: Hold stopper while heating as steam may cause stopper to fail.

3. Transfer 50-ml. of the sample from the sample bottle at a temperature of 95-100°C. into the separatory funnel containing the 50-ml. of boiling distilled water.

   NOTE: As a precautionary measure pour some of the sample from the sample bottle into a waste beaker before adding the 50-ml. to the funnel.

4. Stopper the funnel and shake vigorously for at least 1 minute, venting frequently through the stopcock.

   CAUTION: Care must be exercised at all times to touch neither the lower part of the funnel stem nor the ground part of the stopcock.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Inorganic (G.E.Free)Chlprides in Aroclors, Pyranols, Inerteens, and Tri-Tetrachlorobenzene Blend, contd.

5. Allow the layers to separate and drain off the sample into the second funnel containing the 25 ml. of boiling, distilled, water. It may be necessary to heat the sample when transferring the sample to the second funnel; e. g. Aroclor 1260.

   NOTE: As previously (in Step 3, Note), drain off a few ml. of the sample into a waste beaker before draining the sample into the second separatory funnel.

6. Repeat Step 4 and allow the layers to separate. Then drain off the sample into a waste beaker.

7. Combine both water extracts in one funnel and shake thoroughly.

8. Take approximately a 10 ml. aliquot of the water extract out through the bottom of the funnel into a 3/4" x 6" test tube. Again first allow a few ml. to drain out before taking the aliquot.

   NOTE: The test tube used should be rinsed with distilled, chloride-free water several times prior to using.

9. Add an equal (approx.) portion of chloride-free ether.

   CAUTION: Test ether for chlorides before using in the following way.

   a. Shake a portion of the ether with chloride-free distilled water.

   b. Separate water layer and test it for Tyndall beam at the end of 45 seconds.

   c. If the beam is present, wash ether (as in a and b) several times with chloride-free water until washings show no beam after adding 3 - 5 drops $AgNO_3$.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Inorganic (G.E.Free)Chlorides in Aroclors, Pyranols, Inerteen, and Tri-Tetrachlorobenzene Blend, Method No. 10,118-53, contd.

10. Shake the ether-water mixture until the emulsion in the sample disappears and the water layer is completely beam free before adding $AgNO_3$. If emulsion is difficult to break, add sample dropwise through the ether and then shake.

11. Add 3 - 5 drops of 10% $AgNO_3$ solution and test for chloride beam for 45 seconds exactly. If no beam is present at the end of 45 seconds, report a <0.1 ppm. The very faintest of beams is considered 0.1 ppm. If beam is stronger it will be necessary to compare with standards of 0.15, 0.20 up to 1.0 ppm, adding the 3 - 5 drops of $AgNO_3$ and comparing at the end of 45 seconds.

The method is precise to the nearest 0.1 ppm.

Report results to the nearest 0.1 ppm.

Reagents, Solutions and Accessories:

The following standards are prepared by the Service Section of the W.G.K. plant laboratory:

Standard Chloride Solution:

(See "Procedure Manual for the Service Section Dated 2/27/52, Part 3, page 59).

The following standards are made up:

A primary standard of 100.0 ppm by weighing 0.1648 g. C.P. NaCl into a chloride-free 1-liter volumetric flask. Dilute to the mark with chloride-free distilled water and mix thoroughly.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Inorganic (O.E.Free)Chlorides in Aroclors, Pyranols, Inerteens, and Tri-Tetrachlorobenzene Blend, Method No.10,118-53,contd.

A 10.0 ppm Standard:

Dilute 100 ml. of the primary standard to 1-liter and mix well.

0.1 ppm. 0.2 ppm etc. Standards:

For every 0.1 ppm of a standard, dilute 10 ml. of the 10.0 ppm Standard to one liter and mix well.

NOTE: A 0.1 ppm beam is considered the very faintest beam perceptible to the eye between 15 to 45 seconds after the addition of the $AgNO_3$ solution. If the beam intensity is not visible at all, or if it is easily visible (too strong) discard the solutions and make new standards.

10% Silver Nitrate Solution:

Weigh on a beam balance 20 ($\pm$ 0.05) g. C. P. $AgNO_3$ into a chloride-free dark bottle. Add 20 ml. C. P. HNO_3 (chloride free). Dilute to 200 ml. with water.

NOTE: All solutions should be freshly prepared every two weeks and stored in glass-stoppered Pyrex bottles.

Source of light:

Use the 2-battery Penlite flashlight, having a 3-4 mm light and aperture. New batteries (use "Eveready" No. 915, size AA; "Burgess" No. Z, chrome protected; or comparable batteries)must be used frequently in order to perceive beams properly.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 7-23-54	Material: Aroclors, Pyranols and Inerteens	Method No. 10,121-54
By: AEB, NPA, GWM:WJG	Test: Electrical Stability (or Aging Test)	WGK Dept. No. 246

Introduction:

Electrical stability is determined by measuring the resistivity of Aroclors, Pyranols, or Inerteens at 100°C. before and after exposing the same sample to air at 100°C. for 96 hours.

PROCEDURE:

1. Allow the Electrode assembly (Method No. 11,607) containing the product under test, from the initial resistivity determination to remain in the oven at 100°C. for 96 hours.

   NOTE:

   For this test it is permissible, when convenient, to use either the same oven where the resistivity test has been conducted or any other suitable oven regulated at 100°C. However, care should be taken to see that material under test is not contaminated in any way during the aging process.

2. At the end of the 96 hour period merely attach the leads from the megohm bridge to the electrodes (top lead) (+) on the bridge to inner electrode and other lead to outer electrode) and measure the resistivity again. Follow the procedure described in Method No. 11,607.

   Report the resistivity at the end of 96 hours and indicate the change, if any, from the initial resistivity.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Composition by Distillation of Tri-Tetrachlorobenzene Blend, Method No. 12,036-53, contd.

Calculation:

$$\% \text{ Trichlorobenzene} = \frac{(\text{wt. of Fract. 1} + \tfrac{1}{2} \text{ wt. Fract. 2}) \times 100}{\text{Sample Weight (Step 2)}}$$

$$\text{Total Tetrachlorobenzene (Tetra) (gms)} = \text{wt. of Fract. 3} + \tfrac{1}{2} \text{ wt. Fract. 2} + \tfrac{1}{2} \text{ Fract. 4)}$$

$$\% \text{ 1,2,3,4-Tetra isomer} = \frac{\text{total wt. of Tetra} \times \text{percent 1,2,3,4 isomer (Step 8)}}{\text{Sample Weight (Step 2)}}$$

$$\% \text{ 1,2,4,5-Tetra isomer} = \frac{\text{total wt. of Tetra} \times \text{percent 1,2,4,5 isomer (step 8)}}{\text{Sample Weight (Step 2)}}$$

Example: A 700 g. sample of the blend has been distilled. Total Tetra Fraction weight was 70 g. It was found from Dwg. A-5538 that isomers were present in the following percentages: 1,2,3,4-isomer 90.0% and 1,2,4,5-isomer 10.0%. Then the percent of each isomer in the blend will be as follows:

$$\% \text{ 1,2,3,4-Tetra isomer} = \frac{70 \times 90}{700} = 9.0\% \text{ by wt. of sample (Step 2)}$$

$$\% \text{ 1,2,4,5-Tetra isomer} = \frac{70 \times 10}{700} = 1.0\% \text{ by wt. of sample (Step 2)}$$

$$\% \text{ High Boilers (as Pentachlorobenzene)} = \frac{\left[\text{wt. of Fract. 5} + \tfrac{1}{2} \text{ wt. of Fract. 4} + \tfrac{1}{2} \text{ Residue (Step 9)}\right] \times 100}{\text{Sample Weight (Step 2)}}$$

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Composition by Distillation of Tri-Tetrachlorobenzene Blend, Method No. 12,036-53, contd.

% Loss on distillation =

$$\frac{\text{[wt. of Sample (Step 2) - (combined wt. of Fracts. + wt. Residue (Step 9)]} \times 100}{\text{Sample Weight (Step 2)}}$$

Report all components, residue, and loss on distillation to the nearest 0.1%

Apparatus:

1 - A 2-liter, ℬ 24/40 neck, round bottom distilling flask (pot).

1 - A four foot stainless steel packed column.

1 - An insulated and electrically heated distilling head with a thermometer (ℬ 10/30 joint).

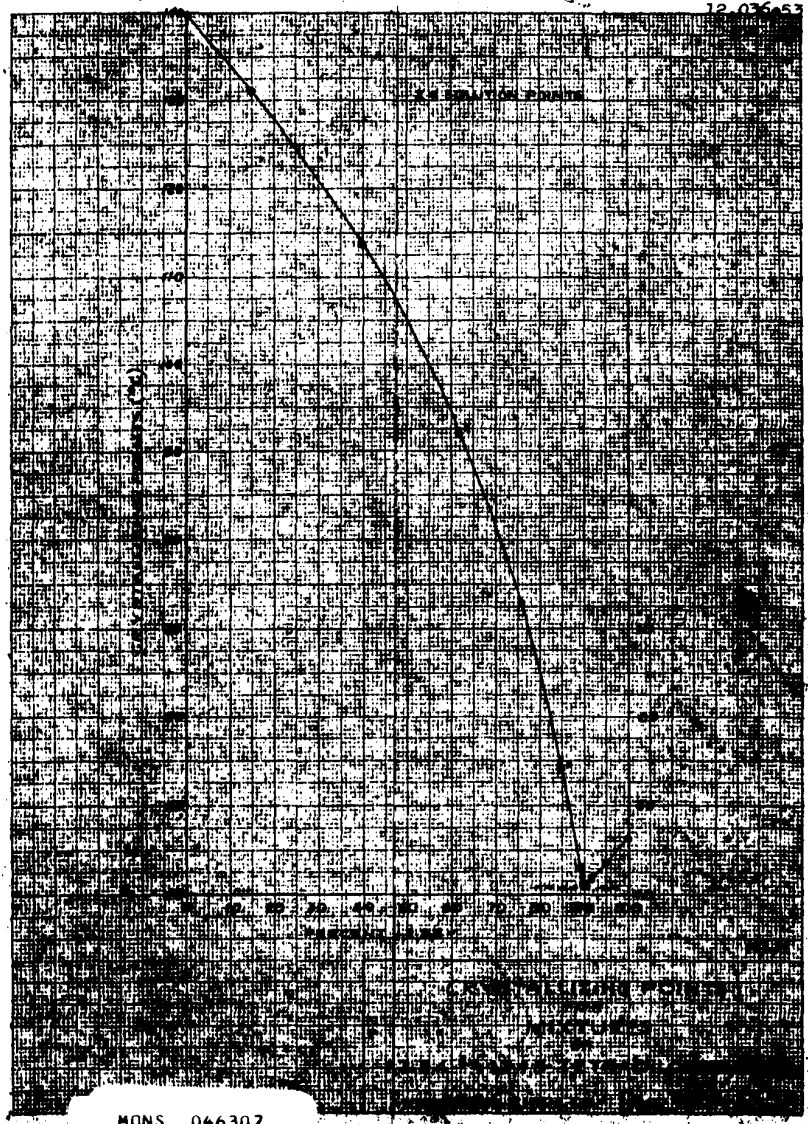
1 - A 500-ml. "glass-col" heating mantle.

3 - Variacs (to regulate individual heating of the distilling head, the column, and the pot).

1 - A 0° - 360°C. range, ℬ 10/30, thermometer (corrected)for the distilling head.

5 - Receivers:

2 x 500-ml. and
3 x 250-ml. Erlenmeyer flasks.



MONS 046307

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IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 9/23/54	Material: Aroclors, Pyranols and Inerteens	Method No. 12,169-54
By:WWK:WJG	Test: Sulfates	WGK Dept. No. 246

Scope:

This method of test is intended for the qualitative determination of inorganic sulfates, by precipitation with $BaCl_2$, in chlorinated biphenyl products.

Apparatus:

250-ml. separatory funnel, 1 x 6 inch test tube, and a burette or 5-ml. pipet.

Reagents:

10% (by wt.) aqueous $BaCl_2$ solution and C.P. HCl

Procedure:

1. Place 75 ml. of distilled water in a clean 250-ml. separatory funnel.
2. Heat the water to boiling by means of a Bunsen flame.
3. Add 100 ml. of sample to the funnel.
4. Shake contents of the funnel thoroughly, venting occasionally through the stopcock, for ca. 1 minute.
5. Allow the layers to separate.
6. Discard the lower or sample layer.
7. Drain 15 ml. of the water layer into a clean 1 x 6 inch test tube.
8. Heat to boiling over a Bunsen flame.
9. Add 5 drops of C.P. conc. HCl, then slowly add 5 ml. of $BaCl_2$ solution from a burette or pipette, and shake to mix.
10. A white precipitate reveals the presence of sulfates. Report as follows:

    If no precipitate is noted immediately report as "None".
    If a precipitate is noted report as "present".

Reference: ASTM-D117-53T and D878-49.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 8/20/54 Material: Aroclor-Butylacetate Mixtures Method No. 12,208-54

By:GWM:WJG Test: Composition WGK Dept. No. 246

1. Ascertain that the lot number of the Aroclor 1254 (used in the mixture) is recorded on the label of the sample bottle.

2. Determine the Specific Gravity of the mixture at 29°C./15.5°C. using General Method No. 13 (Method No. 10,497) with the following modifications:

   a. DO NOT heat the material to greater than 35°C.
   b. Use a 1.400 to 1.600 range hydrometer.
   c. Use a 0° to 50°C. range paper scale thermometer.

3. Apply the hydrometer correction (if any).

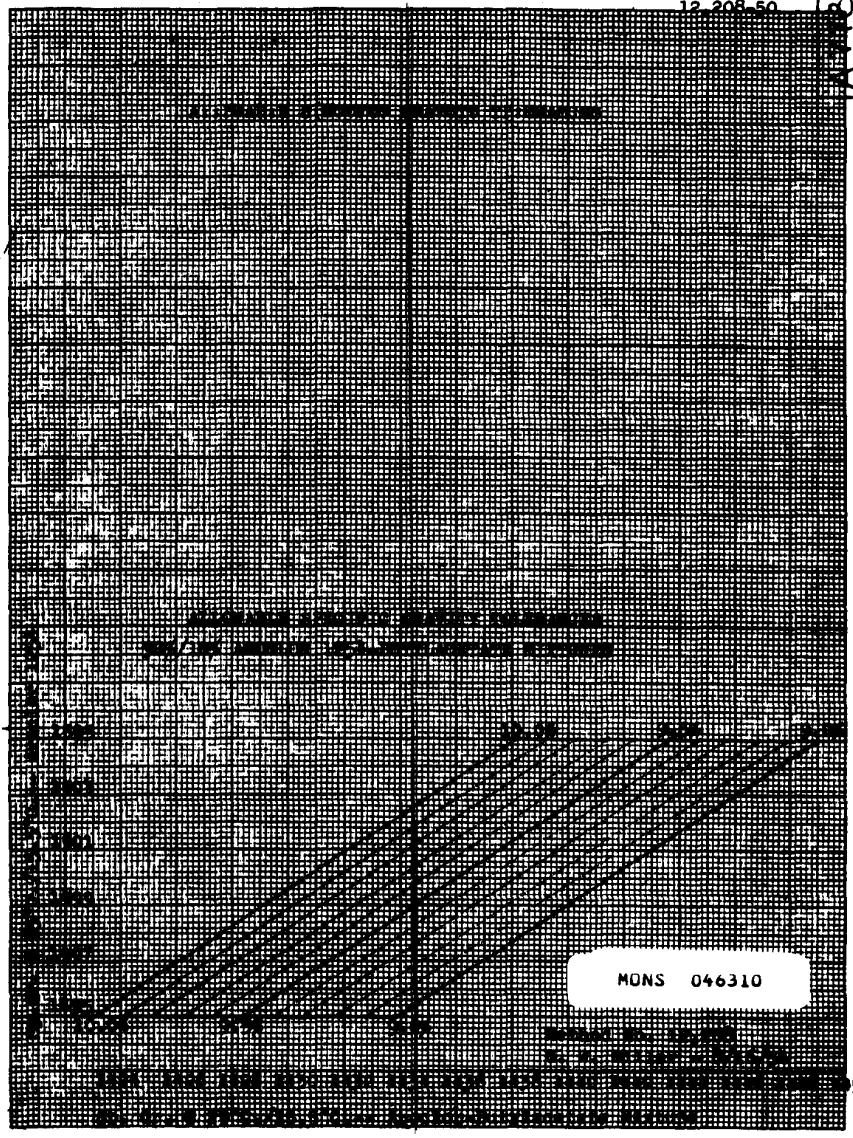
4. From chart A 7780 on the following page, IX - 176, determine if the corrected Sp. Gr. of the Mix at 29°/15.5°C. falls within the shaded portion of the chart at a point corresponding to the Specific Gravity* at 65°/15.5°C. of Aroclor 1254 used in the blend.

Report only the Specific Gravity of the mixture at 29/15.5°C. and indicate whether or not the material passes the specification limits. (Whether it falls within the shaded area on the chart or not.)

Remarks:

The diagonal lines on the chart indicate the % Butylacetate present in the mix at the corresponding coordinates of specific gravity of the mix at 29°/15.5°C. and the specific gravity of the Aroclor 1254 at 65°/15.5°C.

* This Specific Gravity may be obtained from the analytical ticket file.



IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 8/24/54 Material: 1254 Aroclor Butyl Acetate Mixture Method No.12,213-54
By: TT:WJG Test: Refractive Index WGK Dept. No. 246

1. Determine the Refractive Index of the mixture at 25°C. using General Method No. 28 (Method No. 11,784).

NOTE: Do not leave the sample bottle unstoppered, since possible volatilization of the butyl acetate would change the composition of the mixture.

Date: 8/24/54 Material: 1254 Aroclor Butyl Acetate Mixtures Method No. 12,214-54
By: TT:WJG Test: % Butyl Acetate WGK Dept. No. 246

1. Tare on an analytical balance a clean dry aluminum dish. Record the weight.
2. Weigh accurately on an analytical balance a 5 (± 0.1000) g. of the mixture into the dish and record weight of dish and sample.
3. Subtract weight of Step 1 from weight in Step 2. (This is the weight of the sample).
4. Place the dish into a 108 ± 1°C. oven for 3 hours.
5. Cool in a desiccator.
6. Reweigh and record weight of dish and contents.

Calculation:

$$\% \text{ Butyl Acetate} = \frac{\text{Original sample and dish (Step 2)} - \text{Final Wt.(Step 5)}}{\text{Sample Weight (Step 3)}} \times 100$$

Report to the nearest 0.1%.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 8/24/54	Material: 1254 Aroclor Butyl Acetate Mixtures	Method No.12,215-54
By:TT:WJG	Test: Color	WGK Dept. No. 246

1. Determine the color of the mixture in terms of APHA units. Refer to General Method No. 2 (Method No. 10,007).

Date: 9/29/54	Material: Aroclors, Pyranols, Inerteens	Method No. 12,220-54
By: WWKWJG	Test: Light Transmission	WGK Dept. No. 246

Scope:

The purpose of this method is to measure the percent light transmission through 246 products as compared to distilled water which has a transmission of 100%.

Equipment:

1. The Fisher Electrophotometer.
2. An empty plastic filter holder (no filter).
3. Round, 23-ml., matched absorption cells.

Procedure:

1. Determine the Light Transmission according to General Method No. 18 (Method No. 10,723-51) with the following modifications:

   A. Use an empty plastic filter holder.
   B. Use "A" light intensity.
   C. Use distilled water as a reference liquid.

2. Read the % Light Transmission on the "B" scale.

   Report results to the nearest 0.1%. MONS 046312

Reference: National Cash Register Co.'s Method 2021.55 (9/24/52).

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 3/7/55 Material: Aroclors and Pyranols Method No.12,306-55

By: WWK:WJG Test: Parlin # 10 Viscosity WGK Dept. No. 246

IMPORTANT: Conduct this test in a room having a temperature of 23-27°C. and away from drafts.

1. Support the cup in a level position on a ring stand high enough to permit the placing of a 50-ml. graduate and your finger under the cup.

2. Center a clean, dry 50-ml. graduate under the cup orifice.

3. Strain about 100 ml. of sample thru a funnel containing a 100 mesh or less screen into a clean, dry 250-ml. beaker.

4. Adjust the temperature of the sample to 25 ± 0.5°C.

5. Close the orifice by placing your finger under the cup.

6. Pour the sample into the cup, avoiding air bubbles, until it is level full.

   NOTE: The cup must be exactly level full with the surface of the liquid being neither convex nor concave.

7. Remove your finger and measure the time, by stopwatch, it takes for exactly 50 ml. of sample to be collected.

   NOTE: Start timing the instant you remove your finger from the orifice.

Report the viscosity in seconds to the nearest 0.1 second.

Duplicate results should check within 1 (one) second.

Reference: Geisman's memo to Schwartz, 8/5/54.

This method is the same as that used by Du Pont at their plant in Parlin, New Jersey.

IX. SPECIFICATIONS AND TEST METHODS, contd.
Note on the Inorganic Chlorides Test.
See also the comments on pages 12 - 17 in Section VII, above.

It will be noted that the chlorides content is expressed in parts chlorine ion per million. The specification limit of 0.1 parts chloride per million calls for very special control technique, especially in a plant area where the air is liable to be contaminated with traces of HCl gas etc.

In order to get repeatable results, it was found necessary, at the Krummrich plant laboratory, to set up a special test room with a supply of clean air, and to keep the pressure in the room a little above that of adjacent rooms to prevent the entrance of contaminated air. Full details of this special room were sent to Mr. Haywood at Newport, October 24, 1950, with drawings, and suggestions for improvement arising out of Krummrich plant experience.

Mr. Harden reported on visit to the Krummrich Plant control laboratory in August 1950, and Mr. Pennington visited the laboratory late in the same year to pick up the technique of the tests.

Mr. Haywood, November 19, 1951, reported that Newport had installed such a special room.

On February 2nd, 1953, Mr. Beauregard told us that G. E. in America no longer had "wide open" liaison with B. T. H. in England, but that MCL would, nevertheless, be in order in giving details of the G. E. test to B. T. H. MCC consider that the Munch test, in which the "volatile chlorides", formed during heating, are aspirated over into silver nitrate solution, gives a better estimate of the stability of the sample, but that MCC have not yet enough background of experience in the use of the test, and in the figures normally to be expected, to be in a position to recommend a change to the Munch test.

MCL Research Progress Report 1151 - 52/1/12, May 1953, deals with Bakelite Co.'s test of the same type as Dr. Munch's test.

The faintly opalescent suspensions of AgCl are unstable, and the turbidity measurements have to be made on a rigid time schedule, using visual comparison in Tyndall beam. Mr. Barbre suggested washing the condenser back into the sample, and using a Lumetron or similar

IX. SPECIFICATIONS AND TEST METHODS, contd.

Note on the Inorganic Chlorides Test, contd.

presumably because the tintetraphenyl, performing its function of removing HCl formed during the heating, has formed $SnCl_2$ or $SnCl_4$. The G. E. specification allows up to 5 ppm, but Mr. Kuster reports that MCC figures rarely exceed 2 ppm. While allowing the relaxation to 5 ppm, G. E. still insist that the separate main component shall show not more than 0.1 ppm.

The same 5 ppm tolerance has been extended to "Inerteens" which contain glycidyl phenylether in place of tintetraphenyl, but experience indicates that this tolerance is unnecesary.

Presumably the glycidyl phenyl ether removes the HCl by the opening of the alkene oxide ring:

giving products

which volatilise away, or at least do not react with the silver nitrate.

The very dilute sodium chloride standards required for the inorganic chlorides test require very special care in handling, otherwise they increase in strength by picking up HCl etc. from the air. In practice they are not actually made up, except when a new operator is being trained. An experienced operator works from a visual memory of the Tyndall opacity given by the standard (actually it is about the faintest discernible opacity).

Occasionally a high "chlorides after heating" figure on Inerteen has been traced to contamination with traces of tintetraphenyl picked up in the plant.

MCL Research Progress report 1152 NR 52/103/3, September 1954, deals with a method of determining tin tetraphenyl in Pyranols.

Report 1151 52/1/12, May 1953, shows that Aroclor 1262 is stable at 160°C. in air.

Report 1151, NR 53/97/1, July 1953, showed some darkening of 1262 in steel at 300°C. in air.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Note on Electrical Test Methods.

A great deal of work has been done by MCL in the development of electrical test methods and equipment, in consultation with customers, in testing of raw material supplies, and finished products, on the effect of stabilizers, effect of contamination by contact with insulating materials or gaskets, in customer service work generally, and in assembling data for various publications.

See the list of reports at the end of Section VII, above.

Finished Product Samples sent to MCL.

A complete set of samples of Aroclors 1142 to 5460 was requested April 1, 1947, also of all Montars, and of Pyranols 1467, 1488, 1496, with analytical data on each sample. (Aroclors 1169, and 1269 had been abandoned in favour of 1170 and 1270).

MCC report 171-1088, 2881, May 1953, compares Anniston and Krummrich plant 1260.

Competitor's Products.

Dr. Jenkins, February 20, 1952, reported a statement to the effect that Bayer's equivalent of Aroclor 1254 had a resistivity of 100 to 900 x 10^9.

MCC report 171-1075, 2887, discusses an Italian sample of "Fenclor".

Ruabon Development Division Annual Report, 1951, page 4, discusses ICI's "Cereclors" as plasticisers for PVC. Aroclors were better for this purpose.

Certain MCL research reports under job 1151, discuss competitive products, for example, 1151 DF 53/86/5, March 1954, on French "Pyralene".

X. PLANT OPERATING DATA

Plant Capacity

In 1946 the working rates of the different units at the Krummrich Aroclor plant were as set out in the table below.

TIME TABLE - AROCLOR PRODUCTION

Operation	Liquid Aroclors 1242-1248-1254-1260-1262		Solid Aroclors 1268-1269-1270-1271		Resinous Aroclors 2565-4465-5460	
	Time-hours	Equivalent Lbs.of fin. Aroclor	Time-hours	Equivalent Lbs. of Fin. Aroclor	Time-hours	Equivalent Lbs.of Fin. Aroclor
Melting Charge CMB and DMB					12.00*	
Pumping Diphenyl	0.40*		0.40*		0.40*	
Charging Chlorinator	0.35		0.35		1.25	
Chlorination	1242-13.00	6100	1268-23.00	7000	2565-24.00	6500
	1248-15.00	5700 **	1269-24.00	7200	4465-24.00	6900
	1254-19.00	7200	1270-26.00	6800	5460-28.00	6200
	1260-20.00	7800	1271-28.00	-		
	1262-21.00	8500				
Transfer to aerating tk	0.35				0.35	
Aerating	4.00 #				4.00 #	
Drawing to drums					2565- 3.00	
Distillation	12.00 ∮		1268-16.00		4465-14.00 ∮	
			1269-24.00		5460-16.00 ∮	
			1270-24.00			
			1271-24.00			
Drawing to drums			1268- 3.00			
Blending & treating	4.00 e		8.00*		4.00 e	
Filling containers			6.00*			
Filtering	5.00 e				5.00 e	
Filling containers					4.00*	
Dropping to storage	2.00*					
Cleaning press	3.00*				3.00*	

* These operations are performed at such times during the operating cycle that no production time is lost.

\# When the production rate is low, the aerating operation is performed similarly to (*) without any lost time in production. However, during a high rate of production the chlorination and distillation operations are frequently held up until the aeration operation is completed, which results in a loss of production.

∮ Approximately one and one-half chlorinator batches make one still charge.

e Normally these charges are equivalent to approximately three chlorinator batches.

** The figures on page VI-5 would indicate a figure of about 6300 lb. for finished 1248.

X. PLANT OPERATING DATA, contd.

Plant Capacity, contd.

See also page 5 of Section VI, above, for chlorination time cycles.

With four main chlorinators, one air-blowing tank, and two vacuum stills, 9,624,188 lbs. of liquid Aroclors, and 121,812 lbs. of solids were made in 1944.

The best month up to 1947 had produced over 1 million pounds. The capacity in 1954 (five main chlorinators, two air-blowing vessels and two vacuum stills) was given as 1½ million pounds per month.

In 1954 the capacity for Aroclors was given as 1½ million pounds per month.

The Pyranols plant was rated at 1½ million pounds a month in 1953. Typical operating times for Pyranols were given in 1947 as follows:

Pump Aroclor to mixer	8.0 hours
Pump TCB to mixer	5.0 hours
Mix and sample	2.5 hours
*Adjust composition and mix again	3.0 hours
Add tin-tetraphenyl, mix and sample	2.0 hours
Clean the press	0.5 hours
	21.0 hours

* This period is required for each separate adjustment found necessary

The elapsed time from starting a batch to finishing and loading was about 5 to 6 days. It took about 5 hours to load an 8,000 U.S. gallon rail tank.

Labour.

At Anniston one operator per shift looks after six to seven chlorinators, and another looks after the two stills. The chief operator of the area helps as necessary, and extra labour is available for drumming off and similar occasional heavy loads.

See page 25 Section VI, above, for the labour needed for the distillation of solid Aroclors.

One man, per shift, borrowed from the Aroclor department, does practically all the Pyranol work at the Krummrich plant.

X. PLANT OPERATING DATA, contd.

Yields

The departmental monthly returns are complicated because of the number of finished products, each with its own requirements of raw materials, and of services. Another difficulty is that the receipts of chlorine into the department are not measured in any way; the "consumption" of chlorine is reckoned back from the "make" of Aroclors. In the Krummrich plant Aroclor department, receipts of other raw materials, and despatches of finished goods, are totalled for the month in the usual way, and the month-end inventory made as described in Section VI, above, pages 47-.

True theoretical yield figures cannot easily be set up because the products are quite complex mixtures, but what are called "Theoretical Conversion Factors as Obtained from Experience", are tabulated on page X-13 below, each crude Aroclor being lumped with its corresponding distilled product, and each month the "Theoretical" requirements of chlorine and Diphenyl are calculated from the month's production figures for the different Aroclors as set out below. See also the letter, E. Mather to L. D. Stuart, June 2, 1947, discussing the origin and use of these "theoretical" factors.

DETERMINATION OF THEORETICAL YIELDS

Type Aroclor	Pounds Aroclor Produced	* Cl_2 Factor	Diphenyl Factor	Cl_2 Required To Finish	Diphenyl Required To Finish
1148 + 1248	32,685	0.9664	0.5304	31,587	17,336
1154 + 1254	187,407	1.0972	0.4669	205,623	87,500
1260	97,147	1.20	0.4038	116,576	39,228
1262M	(1)1,800	1.2270	0.3930	2,209	707
1271	3,000	1.4190	0.3200	4,257	960
TOTAL	322,039			360,252	145,731
(2000-1800)+ 200	322,239	(1262 mix)			

(1) Use 90% of total 1262 mix produced, or 1800#.

* From page X-13.

X. PLANT OPERATING DATA, contd.

Yields, contd.

"Standard Conversion Factors" also have been established as set out in the table on page X - 12b, in this case the crude and the distilled products being taken separately. Each month the "Standard" requirements of chlorine and diphenyl are calculated back from the month's production figure for each crude and distilled Aroclor separately (except that each crude Aroclor is bracketed with its corresponding distilled Aroclor where one is made, and the factor used for each pair is a weighted average of the single factors) as set out in the example below.

DETERMINATION OF STANDARD YIELDS

Type Aroclor	Produced	Cl_2 Factor	Diphenyl Factor	Cl_2 Required	Diphenyl Required
1148 + 1248	32,685	1.040	0.5687	33,992	18,588
1154 + 1254	187,407	1.181	0.5011	221,328	93,910
1260	97,147	1.2952	0.4375	125,825	42,502
1262M	(1) 1,800	1.3225	0.4210	2,381	758
1271	3,000	1.4995	0.3438	4,499	1,031
TOTAL	322,039			388,025	156,789
2000 - 1800 =	200	(1262 mix)			
	322,239	gross total production			

1) Use 90% of total 1262 mix produced or 1800 #.

The gross total production is then compared with the "theoretical", "standard", and "actual" consumptions of chlorine and diphenyl, as set out in the example on the following page (X-5).

The HCl production is compared with half the chlorine received, (i. e. used).

X. PLANT OPERATING DATA, contd.

Yields, contd.

Dept. 246 Monthly Inventory 8 A.M. - June 1, 1946

CHLORINE:

Theory $\frac{322\ 239}{360\ 252}$ ■ x 100 = 89.45, taken as 100%

Standard $\frac{322\ 239}{388\ 025}$ ø x 100 = 83.05 $\frac{83.05}{89.45}$ x 100 = 92.85%

Actual $\frac{322\ 239}{387\ 025}$ * x 100 = 83.26 $\frac{83.26}{89.45}$ x 100 = 93.08%

DIPHENYL:

Theory $\frac{322\ 239}{145\ 731}$ ■ x 100 = 221.12, taken as 100%

Standard $\frac{322\ 239}{156\ 789}$ ø x 100 = 205.42, $\frac{205.42}{221.12}$ = 92.95%

Actual $\frac{322\ 239}{156\ 289}$ * x 100 = 206.18, $\frac{206.18}{221.12}$ = 93.24%

100% HCl:

$$\frac{\text{Short tons 100\% HCl produced}}{\text{Short tons } Cl_2 \text{ received} \div 2} \times 100 = \frac{88.34}{94.99}\ ■ \times 100 = 93.00$$

$$\frac{93.00}{102.84} \times 100 = 90.43\ \%\quad \text{Note } 102.84 = \frac{HCl\ (36.5)}{Cl\ (35.5)} \times 100$$

■ These figures are the totals from page X-3.

ø These figures are the totals from page X-4.

* These figures are the totals from page X-7.

X. PLANT OPERATING DATA, contd.

The combined departmental monthly return covering all Aroclors on one statement is then set up as shown on pages X-7 and 8, and summary sheets are made out comparing the current month with the last three months and with the previous year, and giving a forecast of production for the next month, as on pages X-10a, b, c, and d.

The inventory methods in the Pyranol department have no special features.

X. PLANT OPERATING DATA, contd.

MONTHLY STOCK REPORT

May 11, 1946

CRUDE MATERIALS USED

	Cl_2 Gas	Cl_2 Liquid	Cl_2	Toluene	Lime	Att. Earth	Di-phenyl
1. No. of Material	#231	#232	Total	#529	#565A	#608	#611 (5)
2. Stock First of Month	6,400# (1)	0#	6,400#	400#	1000#	225#	159,178#
3. Received +	375,973 (2)	4,000 (3)	379,973	0	2450 (4)	200 (4)	100,000 (6)
4. Total	382,373	4,000	386,373	400	3450	425	259,178
5. Stock Last of Month -	2,348 (1)	0	2,348	200	1500	300	102,889
6. Difference	380,025	4,000	384,025	200	1950	125	156,289
7. Required to Finish Previous Month -	2,000	0	2,000	0	150	0	0
8. Difference	378,025	4,000	382,025	200	1800	125	156,289
9. Required to + Finish This Month	5,000	0	5,000	0	100	0	0
10. Total	383,025	4,000	387,025	200	1900	125	156,289
11. Credit -							
12. Net Used	383,025	4,000	387,025	200	1900	125	156,289
13. Per 100 lbs. Finished Goods	118.86	1.24	120.10	10.00	0.59	0.04	48.50

REMARKS: (1) In scrubber liquor.
(2) From Dept. 231 (Chlorine)
(3) From Dept. 232 (Chlorine)
(4) From Dept. 363 (Warehouse)
(5) 1178# in scrubber liquor.
(6) Purchased.

X. PLANT OPERATING DATA, contd.

May 1946

MONTHLY STOCK REPORT

	High Boiler		Labor			
	Crude	Distilled	Operating	Repair	Packing	Total
1. No. of Material	618	619				
2. Stock First of Month	0#	0#	0	0	0	0
3. Received +	0	0	1150	120	130	1400
4. Total	0	0	1150	120	130	1400
5. Stock Last Month -	0	0	0	0	0	0
6. Difference	0	0	1150	120	130	1400
7. Required to Finish Previous Month -	0	0	200	0	0	200
8. Difference	0	0	950	120	130	1200
9. Required to finish this month +	0	0	150	0	0	150
10. Total	0	0	1100	120	130	1350
11. Credit -					0	0
12. Net Used	0	0	1100	120	130	1350
13. Per 100 Lbs. Finished Goods			0.34	0.04	0.04	0.42

X. PLANT OPERATING DATA, contd.

MONTHLY STOCK REPORT

PRODUCTION, Dept. 246 May 1946

1. Products or By-Products	Aroclor	Montar #4	By-Product 100% HCl
2. Stock Last of Month	(1) 31,479#	0#	0 tons
3. Delivered to Packing Room or shipped	560,385	0	0
4. Del'd to 363 Storage	250,131	0	0
5. Delivered to Departments	(2) 52,109	0	(3) 93.00
6. Total 2, 3, 4 and 5	894,104	0	93.00
7. Rec'd from Packing Room	0	0	0
8. Rec'd from 363 Storage	540,000	0	0
9. Rec'd from Departments	0	0	(4)4.66
10. Stock first of Month	(1) 31,865	0	0
11. Total 7, 8, 9 and 10	571,865	0	4.66
12. Produced	322,239	0	88.34
13. Per Cwt. Main Product			

(1) Process Goods Stock

(2) To Dept. A-246 (Pyranol Dept.) 48,509#
To Dept. D-246 (Permasan Dept.) 3,600
Total ..... 52,109#

(3) To Dept.D-218 (Muriatic Acid Dept.)90.00 tons
To Dept. 217 (Chlorsulphuric Acid Dept.) 3.00 tons
93.00 tons

(4) From Dept. C-248. (Santolube)

X. PLANT OPERATING DATA, contd.

MONTHLY STOCK REPORT

YIELDS, DEPARTMENT 246 May 1946

Products or By-Products	Aroclors			HCl
Yield on chlorine	83.26	(93.08%)	93.00	(90.43%)
Yield Last Month	81.93	(92.80%)	92.79	(90.22%)
Standard Yield	83.05	(92.85%)	92.56	(90.00%)
Theoretical Yield	89.45	(100.00%)	102.84	(100.00%)
Yield on 611	206.18	(93.24%)		
Yield Last Month	208.87	(92.98%)		
Standard Yield	205.52	(92.95%)		
Theoretical Yield	221.12	(100.00%	Aroclors	Produced
Yield on			1148	200#
Yield Last Month			1248	32,485
Standard Yield			1154	300
Theoretical Yield			1254	187,107
			1260	97,147
			1262M	2,000
			1271	3,000
			Total.....	322,239

X. PLANT OPERATING DATA, contd,

SUMMARY OF MONTHLY REPORTS — Dept. 246

	Month: ----	March	April	May
Production:				
Products	1. Aroclors Liquid	400,000#	350,000#	319,239#
	2. Aroclor Solid	0	0	3,000
	3. Aroclor Total	400,000	350,000	322,239
	4. Muriatic Acid 100%			
Yields:				
Raw Materials	1. Diphenyl	94.20%	93.28%	93.24%
	2. Chlorine	94.50	93.43	93.08
	3. Chlorine (HCl)	91.00	90.75	90.43
Inventory:				
Raw Materials	1. Diphenyl	150,000#	220,000#	102,889#
	2. Chlorine	5,000	2,000	2,348
Process Goods	1. Aroclor 1254	3,000#	25,500#	0
	2. Aroclor 1260	7,500	0	31,479#
	3. Aroclor 1271	10,000	0	0
Finished Goods	1. Aroclor	0	0	0
	2. Muriatic Acid	0	0	0
Liquidation Reserve:				
Raw Materials	1. Diphenyl	5,000#	1,000#	3,000#
	2. Chlorine	2,000	3,000	1,000
Process Goods	1. Aroclor	0	0	2,631#
	2. Muriatic Acid	0	0	0
Goods of "No Value":				
	1. —	—	—	—
	2. —	—	—	—
	3. —	—	—	—

COMMENTS: (See attached report for detailed remarks.) Signed:_____

Operating Supt._____

Brief Comments Inserted.

X. PLANT OPERATING DATA, cont'd.

OPERATION REPORT
Dept. 246
May 1946

Rate of Operation

Year	1945	1	9	4	6
Month	12	5 to date	April	May	June Est.
Liquid Aroclors	5,400,000#	1,500,000#	300,000#	319,239#	500,000#
Solid Aroclor	300,000	50,000	0	3,000	50,000
Total Aroclors	5,700,000	1,550,000	300,000	322,239	550,000
100% by Produce HCl	2,200.0 tons	500.0 tons	90.0 tons	88.34 t.	180.0 t.

Brief Comments On:

(1) Number of days operated.
(2) Reason for rate of production.
(3) Reason for estimated rate of production.

Crude Materials and Yields

Crude Materials/cwt Aroclors

Year	1945	1	9	4	6
Month	12	5 to date	April	May	
#231	118.0	117.0	117.5	118.86	
#232	4.0	3.5	4.2	1.24	
Total Chlorine	122.0	120.5	121.7	120.10	
#611	47.2	47.0	46.9	48.50	
#618	0.2	0.2	0.2	0	
#6179	0	0	0	0	
#565A	0.5	0.6	0.4	0.59	
#529/cwt 1262 mix	11.6	11.0	11.5	10.00	
#608	0.03	0.03	0.03	0.04	
Hours labor pkg.	0.03	0.02	0.02	0.04	
Hours labor oper.	0.34	0.32	0.38	0.34	
Hours labor repair	0.02	0.02	0.02	0.04	
Hours labor total	0.42	0.39	0.45	0.42	

X. PLANT OPERATING DATA, contd.

OPERATION REPORT, Dept. 246, May 1946, contd.

Percent Yields (Theory 100%)

Year	1945	1	9	4	6
Month	12	5 to date	April	May	
Aroclor					
on Chlorine	93.24%	92.74%	92.80%	93.08%	
on #611	92.17	94.46	92.98	93.24	
100% by-prod.HCl					
on Chlorine	94.38	95.51	97.61	90.43	

Brief Comments on Yield.

Service Charges/cwt. Aroclor

Year	1945	1	9	4	6
Months	12	5 to date	April	May	
Steam M lbs.	0.22	0.42	0.33	0.33	
KWH Power	2.14	3.09	2.40	2.47	
KWH Lights	1.00	1.40	1.27	1.55	
KWH Total	3.14	4.49	3.67	4.02	
Comp.Air M Cu.Ft.	3.01	0.14	0.10	0.11	
City Water Cu.Ft.	0.11	21.90	24.50	24.00	
Well Water M Gals.	26.00	0.42	0.44	0.47	
Fuel Gas M Cu.Ft.	0.05	0.56	0.03	0.42	

Stocks

Stocks as of	5-1-46	6-1-46
Warehouse	532,000	100,000
Department	571,000	250,131
	1,103,000	350,131

Brief comments on present stock and amount shipped.

X. PLANT OPERATING DATA, contd.

OPERATION REPORT, Dept. 246, May 1946, contd.

Operation

Brief comments on:

(1) Operating difficulties.

(2) Operating changes or unusual occurrences.

(3) Major repairs.

(4) Future changes or major repairs contemplated.

Personnel

Note changes in personnel.

X. PLANT OPERATING DATA, contd.

The month's yield figure for the Aroclors, as explained above, is a composite one, depending to some extent on the ratios between the different Aroclors made. With this restriction, the following data give the results for 1946 at the Krummrich plant.

Raw Materials Consumption		lbs. per 100 lbs. of Aroclor
	Chlorine	122.22
	Diphenyl	47.22
	Lime	0.44
	Attapulgus Earth	0.02
also	Toluene	11.67 (lbs.per 100 lbs. 1262 M blend)

Crude and distilled high boilers are not included because the amounts used were small.

Krummrich plant standards, for 1954 were, per 100 lbs. Aroclor produced: --

	1248	1254	1260 mixture	1262 mixture	1260	1262
Diphenyl consumed	56.50	49.75	39.54	38.02	41.78	43.45
Chlorine consumed	103.25	117.56	117.21	119.47	131.29	128.80
Lime consumed	0.50	0.50	0.46	0.46	0.50	0.50
Attapulgus Earth consumed	0.04	0.04	0.04	0.04	0.04	0.04
Toluene consumed	-	-	1.40 US gallons	1.40 US gallons	-	-
Muriatic Acid produced	46.35	52.75	52.02	53.60	58.94	57.82

X. PLANT OPERATING DATA, contd.

Krummrich plant standards, per 100 lbs. Aroclor, for 1954, contd.

	925,000 lbs./month production rate	1,250,000 lbs./month production rate
Labour, man hours	1,622	1,622
Steam, lbs.	1,850,000	2,500,000
Electricity KWH	27,800	37,500
Compr.Air, cu.ft.	925,000	1,250,000
City water, U.S.gal.	278,000	376,000
Plant water, cu.ft.	46,300,000	62,500,000

The report "Process Description for Aroclor, Dept. 246", Schwarting and Schwartz, March 15, 1955, gives more recent figures.

Yields of Aroclor (as % of the "theoretical figures on page X - 13).

On chlorine consumed 93.24% (93.25)
On diphenyl consumed 92.17% (94.73)

(The figures in brackets are for the first 9 months of 1953.)

Yield of HCl

On chlorine consumed 94.58% (90.12) of the "theoretical" figure.

X. PLANT OPERATING DATA, contd.

Services Consumed Per 100 lbs. Aroclor

(Production at the rate of 336,100 lbs. of solid Aroclor
plus 6,753,775 lbs. of liquid Aroclor
7,089,875 lbs. of Aroclor per year.)

Steam 1000 lbs.	0.228
Power KWH	2.141
Lighting KWH	1.003
Comp. Air, 1000 cu.ft.	0.110
City water, cu.ft.	25.990
Well water, 1000 U.S. galls	0.464
Fuel gas, 1000 cu.ft.	0.049
Labour, man hours	0.025 packing 0.235 operating

See also the report "Process Description for Aroclor, Dept. 246" Schwarting and Schwartz, March 15, 1955, pages17- for more recent figures.

X. PLANT OPERATING DATA, contd.

Department 246 Standard Conversion Factors (1946)

AROCLOR	CHLORINE	DIPHENYL	DISTILLED HIGH BOILER	CRUDE HIGH BOILER	100% HCl
1142	0.875	0.607$			0.3829
1148	0.992	0.547$			0.4341
1154	1.130	0.482$			0.4945
1160	1.235	0.416$			0.5404
1162	1.253	0.406$			0.5485
1168	1.369	0.362$			0.5991
1169	1.433	0.345$			0.6271
1170	1.505	0.348$			0.6586
1171	1.565				0.6880
1242	0.921	0.638$			0.4030
1248	1.044	0.576$			0.4568
1254	1.186	0.508$			0.5192
1260	1.300	0.438$			0.5689
1262	1.327	0.426?			0.5809
1262M					
1268	1.331	0.390$			0.5827
1269	1.740	0.406$			0.7614
1270	1.828	0.410$			0.7999
1271	1.908				0.8350
2565	1.345	0.286$		0.095	0.5886
4065	1.320	0.240$	1.164		0.5776
4465	1.466	0.266?	0.180		0.6415
5060	1.290	--	0.418		0.5645
5460	1.573	--	0.510		0.6883

A somewhat different set of factors, in use in 1953, is given on the following pages X - 13 and 14.

Molten diphenyl stocks (at about 90°C.) taken at 8 lbs. per U.S. gallon.

X. PLANT OPERATING DATA, contd.

DEPARTMENT 246 - CONVERSION FACTORS

Theoretical Factors as Obtained from Experience (1946).

AROCLOR	CHLORINE	DIPHENYL	DISTILLED HIGH BOILER
1142-1242	0.8492 *	0.5874	
1148-1248	0.9664	0.5304	
1154-1254	1.0972	0.4669	
1160-1260	1.2000	0.4038	
1162-1262	1.2270	0.3930	
1168-1268	1.3330	0.3523	
1169-1269	1.3760	0.3314	
1170-1270	1.4190	0.3200	
1171-1271	1.4500	0.3100	
4065-4465	1.2682	0.2303	
2565	1.2950	0.2781	
5060-5460	1.2800	.---	0.437

These factors were still in use in 1953.

Aroclors are not pure compounds, but are mixtures of two or more compounds, consequently, they do not have a definite composition, and for this reason we cannot calculate the theoretical factors.

* See the Note by E. Mather marked * on the following page.

X. PLANT OPERATING DATA, contd.

DEPARTMENT 246 - CONVERSION FACTORS, contd.

* Note by E. Mather - May 1955

Theoretically 100 lbs. of an Aroclor containing x% of chlorine should give a chlorine conversion factor of

$$\frac{2x}{100}$$

and a diphenyl or high boiler conversion factor of

$$1 - \frac{34.5x}{350}$$

Thus

% chlorine in Aroclor	conversion factor	
	chlorine	diphenyl or high boiler
42	0.84	0.591
48	0.96	533
54	1.08	475
60	1.20	416
62	1.24	397
65	1.30	368
68	1.36	339
69	1.38	330
70	1.40	320
71	1.42	310

By this test the Krummrich plant figures for chlorine for 4065 seems to be impossibly low, and so do the "diphenyl" figures for 4065 and 2565 (taken as representing total hydrocarbon used).

X. PLANT OPERATING DATA, contd.

The % yield figures (on theory) at the Krummrich plant in 1954 for Pyranols, etc. were:

	Pyranols				Inerteens	
	1467	1470	1481	1488	PPO	PPO-TTCB mix
TCB standard	99.0%	-	99.0%	99.0%	99.0%	
actual 1953	99.85	-	98.51	99.85	99.85	
TTCB standard	-	98.0	-	-	-	98.0%
actual 1953	-	98.81	-	-	-	98.81
Aroclor 1254 std.	-	99.0	-	-	-	-
actual 1953	-	99.0	-	-	-	-
Aroclor 1260 std.	99.0	99.0	-	99.0	99.0	99.0
actual 1953	99.0	99.0	-	99.0	99.0	99.0
The consumption figures were						
Aroclor 1254	-	-	75.75	-	-	
Aroclor 1260	60.60	45.45	-	60.60	60.60	45.45
TCB	40.40	-	25.25	40.40	40.40	-
TTCB	-	55.56	-	-	-	55.56
TTP	0.128	0.128	-	-	-	-
Attapulgus earth	0.02	0.02	0.02	0.02	0.02	0.02
Glycidyl ph. ether	-	-	-	-	0.220	0.220

X. PLANT OPERATING DATA, cont'd.

	PYRANOLS	
	936,000 lbs./month	1,250,000 lbs./month
Labour, man hours	813	813
Steam, lbs.	278,000	374,000
Electricity, KWH	3,700	5,000
Comp. air. cu.ft.	-	-
City water	-	-
Plant water	-	-

Figures for earlier years are given by Lyles, Soffranko & Miller, March 24, 1947, pages 51-58.

Krummrich Plant records for Aroclors up to 1946 are summarised on the following pages

Dept. 246

October 2, 1947

PRODUCTION REPORT SUMMARY YEARLY

Year	Gaseous chlorine #231 Lbs.	#/cwt	Liquid chlorine #232 Lbs.	#/cwt	Total Cl 2 Lbs.	#/cwt	Diphenyl #611 Lbs.	#/cwt
			Initial production on September 4, 1937					
1936	18150	2.18	1067944	128.00	1086094	130.18	361095	43.28
1937	4002818	51.32	1386016	3.53	5388834	54.85	1735788	44.21
~~1938~~	~~2881774~~	~~115.17~~	~~512689~~	~~2.049~~	~~3394463~~	~~135.66~~	~~1073472~~	~~42.90~~
1939	3526099	114.67	406623	13.22	3932722	127.99	1322914	43.02
1940	6447110	126.27	181650	3.556	6628760	161.83	2235099	43.78
1941	7387766	126.94	227748	3.913	7615514	166.07	2546476	43.75
1942	9415836	116.94	1034700	12.86	10450536	129.84	3517935	43.71
1943	10665174	116.57	693980	7.585	11359154	124.16	4243212	46.37
1944	11338840	116.38	531680	5.457	11870520	121.84	4580000	47.01
1945	8264386	116.57	338400	4.773	8602786	122.82	3307987	47.22
1946		119.20		4/05		123.25		

Note: The cwts are 100 lbs.

MONS 046340

CRUDE MATERIALS

Year	#1148 Lbs.	#1148 #/cwt	#1160 Lbs.	#1160 #/cwt	Hydrated Lime #565A Lbs.	Hydrated Lime #565A #/cwt	Crude High Boiler #618 Lbs.	Crude High Boiler #618 #/cwt
1936	4550	.545	10432	125.03	7050	.845	5400	.647
1937	0	-	0	-	62445	1.591	26500	.675
1938	0	-	0	-	45002	1.799	9000	.360
1939	0	-	0	-	19700	.641	17187	.559
1940	0	-	0	-	57225	1.121	6000	.118
1941	0	-	0	-	53780	.924	0	-
1942	0	-	0	-	30600	.378	0	-
1943	0	-	0	-	23650	.258	0	-
1944	0	-	0	-	38600	.396	1800	.193
1945	0	-	0	-	41850	.445	2000	.180
1946	0	-	0	-	-	-	-	-

CRUDE MATERIALS

Year	Distilled High Boiler #619		Toluene #529		Attapulgus Earth #608	
	Lbs.	#/cwt	Lbs.	#/cwt	Lbs.	#/cwt
1936	9609	1.152	0	-	0	-
1937	19000	.484	3427	.087	1115	.0283
1938	47890	1.914	1684	.067	1050	.042
1939	54579	1.775	2554	.083	1231	.040
1940	13200	0.259	4587	.0898	4952	.097
1941	0	-	5176	.089	6257	.108
1942	0	-	475	.006	2725	.034
1943	0	-	2039	.223	3125	.034
1944	1800	.193	19446	19.96	2285	.024
1945	2000	.180	7534	11.67	1770	.024
1946						

Dept. 246

October 7, 1947

YIELDS

Year	On Diphenyl #611 #/cwt	On Diphenyl #611 %Th	On Chlorine #232 #/cwt	On Chlorine #232 %Th	HCl - 100% #/cwt	HCl - 100% % Th
1936	231.05		78.12		112.89	109.77
1937	226.18	98.19	72.86	81.27	98.55	95.83
1938	233.09	97.97	73.71	91.02	89.10	86.64
1939	232.45	98.13	78.19	91.10	93.02	90.45
~~1940~~	~~228.44~~	~~94.91~~	~~77.02~~	~~91.24~~	111.65	102.57
1941	228.55	94.08	76.42	91.28	106.64	103.70
1942	228.80	93.06	77.02	91.90	104.54	101.65
1943	215.64	96.16	80.44	92.83	102.20	99.38
1944	212.73	94.12	81.73	92.08	104.68	101.79
1945		92.17		93.24		94.38
1946		95.60		92.80		94.46

X - 20
Operating
Data

MONS 046343

Dept. 246

October 7, 1947

Man Hours

Year	Operating Hours	Operating Hrs./cwt	Repair Hours	Repair Hrs./cwt	Packing Hours	Packing Hrs./cwt	Total Hours	Total Hrs./cwt	Training Hours
1936	7016	.841					7016	.841	
1937	25115	.640					25115	.640	
1938	14364	.574					14364	.574	
1939	12200	.397					12200	.397	
~~1940~~	~~20286~~	~~.897~~					~~20286~~	~~.397~~	
1941	17444	.300			937	.016	18381	.327	
1942	18698	.232			1799	.022	20497	.255	
1943	20660	.226			2240	.024	22900	.250	
1944	22529	.231			2778	.029	25307	.260	
1945	18077	.235			2140	.025	18817	.260	
1946		.31				.04		.39	

Added March 14, 1951: See Dr. M. B. Dyson's notes on his 1950 trip to St. Louis for some notes on the manning of the department.

Dept. 246

October 7, 1947

Prod. & Yields

Year	Total Liquid Aroclor	Total Solid Aroclor	Total Liquid and Solid	By Prod. HCl Tons
1936			834324	209.60
1937			3926076	1176.76
1938			2502161	778.90
~~1939~~			~~3075121~~	~~914.54~~
1940			5105790	1501.90
1941			5819864	1785.39
1942	7954018	94974	8048992	2499.18
1943			9149940	2778.63
1944	9624196	121812	9743008	2839.84
1945	6753755	336110	7089875	2120.60
1946	3404795	229624	3634415	1092.81

Dept. 246

October 7, 1947

AROCLORS PRODUCED

Year	1119	1142	1148	1154	1160	1162	1168	1169
1936	0	8700	14400	6000	0	0	0	0
1937	20	38975	1000	0	0	1458	254	50
1938	0	13436	0	5900	0	1445	100	80
1939	0	11792	0	2360	0	9065	500	0
1940	0	4885	1543	8000	72	19	8490	0
1941	0	1537	0	0	627	1465	24089	0
1942	0	0	0	0	0	0	0	0
1943	0	25	485	125	0	0	0	0
1944	0	723	0	500	0	500	0	0
1945	0	467	0	0	0	0	0	0
1946	0	0	0	0	0	0	0	0

Dept. 246

October 7, 1947

AROCLORS PRODUCED

Year	1170	1171	1242	1248	1254	1260
1936	0	0	28593	13000	231584	264400
1937	15	0	43044	53362	1299822	1196468
1938	55	0	49735	25363	639878	842622
1939	0	0	22250	59313	801662	1542462
1940	0	0	30431	83013	1329442	2910460
1941	0	0	0	79779	361063	4202815
1942	0	0	12166	100242	0	7727813
1943	0	0	48875	95820	4124646	4333299
1944	0	0	74784	38784	6082785	2776688
1945	0	0	31556	100914	3609168	2858994
1946						

MONS 046347

Dept. 246

October 7, 1947

AROCLORS PRODUCED

Year	1262	1262 Mix	1268	1269	1270	1271
1936	39640	0	34514	78124	0	0
1937	165223	10068	51419	668517	0	0
1938	122074	5000	47375	179887	240124	0
1939	88216	0	500	0	97990	0
1940	140678	41232	8490	0	179891	0
1941	230056	40594	24089	0	319959	0
1942	121506	4457	0	0	94974	0
1943	453236	16524	0	0	0	0
1944	464012	182120	0	0	20657	0
1945	100444	52222	0	0	330310	5800
1946						1271 is expected to reach 86,000 lb/yr. (textile use).

MONS 046348

Dept. 246

October 7, 1947

AROCLORS PRODUCED

Year	2565	4065	4465	5060	5460	300	301
1936	57429	0	57710	0	0	0	0
1937	240124	0	115589	500	14048	0	0
1938	97990	40	148919	484	43460	0	0
1939	179891	200	193196	0	38850	1	66
1940	0	0	74451	0	0	0	0
1941	0	0	0	0	0	0	0
1942	0	0	0	0	0	0	0
1943	0	0	0	0	0	0	0
1944	20657	0	0	0	0	0	0
1945	0	0	0	0	0	0	0
1946							

MONS 046349

X. PLANT OPERATING DATA, contd.

Cost sheets for the Krummrich plant Aroclors Dept. were sent to London, February 1946, and those for Anniston were sent February 24, 1947. Anniston cost sheets for crude Aroclors, distilled Aroclors, and Muriatic Acid for August 1947, as included in the visit report, Mather and Havercroft, September 1947, are repeated on the next 3 pages.

In these, all the Aroclors are bulked together; records relating to single Aroclors are put on to "budget sheets" which are really forecasts of cost, the total cost figures being arbitrarily split between the separate Aroclors on the basis of previous experience. (Copies sent to London, see letter Mather to Durgin, May 24, 1945). Copies of the Anniston monthly returns for August 1947, as given in the report of Havercroft and Mather just mentioned, come a little nearer to giving consumptions of materials for each Aroclor; the services consumed are, however, bulked for the whole of the Aroclors.

Raw material consumption figures for Anniston in the early part of 1947, are set out on page X-31 below, and the Anniston monthly returns are given on pages X-31 - 36 below.

X - 28
Operating
Data

MONSANTO CHEMICAL COMPANY — **COST REPORT** — Anniston PLANT

PRODUCT: Crude Aroclors ... DEPT. NO. ______ MONTH: August 19[illegible]

PRODUCTION THIS MONTH: 409 9[illegible]0 — YEAR TO DATE: 7,[illegible] — COST UNIT: 100 lb

RATED CAPACITY ______ % OPERATED ______ DAYS OPERATED AT ______ % OF ______ BRACKET

PERCENT YIELD ON ______ STANDARD ______ ACTUAL ______ M. & E. INVESTMENT ______

PERCENT YIELD ON ______ STANDARD ______ ACTUAL ______ M. & E. DEP. RESERVE ______

Material	Actual Quantity Used	Standard Quantity Used	Material Unit	Standard Material Unit Price	Inventory Standard	Actual This Mo.	Actual Yr. to Date	Last Year
Diphenyl	2[illegible] 2[illegible]	2[illegible] 1[illegible]	lb	05252	[illegible]	[illegible]	[illegible]	[illegible]
Crude Santowax			lb				[illegible]	[illegible]
Santowax R			lb				[illegible]	[illegible]
Chlorine	[illegible]	[illegible]	lb	02315	[illegible]	[illegible]	[illegible]	[illegible]

Year to date prices
Diphenyl [illegible]
Chlorine [illegible]
[illegible]

STEAM
ELECTRICITY
COMPRESSED AIR
WATER PURCHASED
WATER - PLANT
FUEL GAS, COAL, OIL

Statement of Production Cost	Actual Cost	Inventory Standard Cost	Variance	Current Month Actual	Current Month Inventory Standard	Year to Date Actual	Last Year Actual
Diphenyl	14 539 34	14 [illegible] 79	[illegible]	2 [illegible]	[illegible]	2 092	2 [illegible]
Crude Santowax							
Santowax R						[illegible]	011
Chlorine	17 [illegible] 93	17 [illegible]	[illegible]	2 [illegible]	2 940	3 096	2 [illegible]
Total	32 130 [illegible]	32 [illegible]	[illegible] 25	[illegible]	[illegible]	5 [illegible]	[illegible]
Credit							
Muriatic Acid	[illegible]	2 [illegible]	-108 9[illegible]	.336	[illegible]	[illegible]	[illegible]
NET TOTAL MAT'LS	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
STEAM	[illegible] 62	[illegible]	100 [illegible]	.067	[illegible]	[illegible]	[illegible]
ELECTRICITY	62 [illegible]	[illegible]	119 52	.010	.030	[illegible]	[illegible]
COMPRESSED AIR							
WATER PURCHASED							
WATER PLANT	[illegible] 01	[illegible]	[illegible]	.007	.008	.005	[illegible]
FUEL GAS COAL OIL	[illegible]	[illegible]	27 [illegible]	.001	[illegible]	[illegible]	.004
LABOR MFG	941 [illegible]	609 31	- 332 53	.137	.105	.110	[illegible]
SUPERVISION MFG	79 60	147 19	37 97	.012	[illegible]	.010	.011
S S TAX, COMP INS PENSIONS	66 37	[illegible] 11	- 17 66	.012	.008	.009	0 06
REPAIRS MINOR	310 71	[illegible] 62	93 92	.058	[illegible]	.065	.035
REPAIRS MAJOR	22[illegible] 2[illegible]	2[illegible]	57 81	.037	.047	.040	.035
SUPPLIES	10[illegible] [illegible]	75 27	- 31 3[illegible]	.017	.012	.012	.007
LABORATORY	3 33	65 92	61 [illegible]		.011	.005	.012
CLOTHING AND LAUNDRY		[illegible] 35	24 35		.004	.004	.005
REFRIGERATION & ICE							- .013
EXP TO FINISH OPERATION							
TOTAL DIRECT CONV	2 [illegible] 63	2 [illegible] 61	1[illegible]	.371	[illegible]	.346	.362
DEPRECIATION	24[illegible] 71	24[illegible]	[illegible]	.040	[illegible]	.037	.037
CONTROLLABLE F I E	[illegible]	[illegible]	[illegible]	.085	.069	.060	.067
NON-CONTROLLABLE F I E	112 26	109 [illegible]	- [illegible]	.019	.017	.010	.012
TOTAL INDIRECT CONV	70[illegible]	72[illegible]	[illegible]	.115	.127	.077	.116
TOTAL MFG. COST	3[illegible]	3[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	5 113
TOTAL PACKED COST						.391	.306
Total [illegible] F.O.B. Cost						[illegible]	[illegible]

MONS 046351

X - 29
Operating
Data

MONSANTO CHEMICAL COMPANY — **COST REPORT**

PRODUCT [illegible] — 6-1-73C DEPT NO. — [illegible] PLANT

PRODUCTION THIS MONTH [illegible] — YEAR TO DATE [illegible] — MONTH [illegible] — NO. [illegible]

RATED CAPACITY — % OPERATED — DAYS OPERATED AT — % OF — COST UNIT [illegible] — BRACKET

PERCENT YIELD ON — STANDARD — ACTUAL — M. O. E. INVESTMENT

PERCENT YIELD ON — STANDARD — ACTUAL — M. O. E. DEP. RESERVE

MATERIAL AND UTILITY VOLUMES

	ACTUAL QUANTITY USED	STANDARD QUANTITY USED	MATERIAL UNIT	STANDARD MATERIAL UNIT PRICE	INVENTORY STANDARD	QUANTITY USED PER UNIT PRODUCED: THIS MO.	ACTUAL YR. TO DATE	LAST YEAR
[illegible] Crude [illegible]	[illegible]	[illegible]	lb	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]				[illegible]			[illegible]	
[illegible]				[illegible]			[illegible]	[illegible]
[illegible]							[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	lb	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]								[illegible]
[illegible]								[illegible]
[illegible] hydrated lime				[illegible]			[illegible]	
[illegible] Crude [illegible]				[illegible]			[illegible]	
STEAM				[illegible]				
ELECTRICITY				[illegible]				
COMPRESSED AIR				[illegible]				
WATER - PURCHASED				[illegible]				
WATER - PLANT				[illegible]				
FUEL - GAS, COAL, OIL				[illegible]				

ORGANIC DIVISION

STATEMENT OF PRODUCTION COST	ACTUAL COST	INVENTORY STANDARD COST	VARIANCE	UNIT COSTS: CURRENT MONTH ACTUAL	CURRENT MONTH INVENTORY STANDARD	YEAR TO DATE ACTUAL	LAST YEAR ACTUAL
Crude [illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]						[illegible]	[illegible]
[illegible]						[illegible]	[illegible]
[illegible]							[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]							[illegible]
[illegible]							[illegible]
[illegible] hydrated lime						[illegible]	
[illegible] Crude [illegible]						[illegible]	
NET TOTAL MAT'LS	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
STEAM	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
ELECTRICITY	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
COMPRESSED AIR							
WATER—PURCHASED							
WATER—PLANT	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
FUEL—GAS, COAL, OIL	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
LABOR—MFG.	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
SUPERVISION—MFG	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
S. S. TAX, COMP. INS., PENSIONS	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
REPAIRS MINOR	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
REPAIRS MAJOR	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
SUPPLIES	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
LABORATORY	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
CLOTHING AND LAUNDRY		[illegible]	[illegible]		[illegible]		
REFRIGERATION & ICE							
EXP. TO FINISH OPERATION							
TOTAL DIRECT COST	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
DEPRECIATION	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
CONTROLLABLE—F. I. E.	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
NON-CONTROLLABLE—F. I. E.	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
TOTAL INDIRECT COST	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
TOTAL MFG. COST	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
TOTAL PACKING COST	[illegible]			[illegible]		[illegible]	[illegible]
Total Cost F.O.B. [illegible]				[illegible]		[illegible]	[illegible]

MONS 046352

ORGANIC DIVISION

MONSANTO CHEMICAL COMPANY — COST REPORT — Anniston PLANT

PRODUCT: Muriatic acid (scrubber) 20° Be' — DEPT. NO. Code 6-1526 — MONTH: Aug. 19[illegible]

PRODUCTION—THIS MONTH: 883 329 — YEAR TO DATE: 1,529,[illegible] — COST UNIT: 100 lb

RATED CAPACITY ____ % OPERATED ____ DAYS OPERATED AT ____ % OF ____ BRACKET

PERCENT YIELD ON ____ STANDARD ____ ACTUAL ____ M.A.S. INVESTMENT ____

PERCENT YIELD ON ____ STANDARD ____ ACTUAL ____ M.A.S. DEP. RESERVE ____

Materials and Utilities	Actual Quantity Used	Standard Quantity Used	Material Unit	Standard Material Unit Price	Current Standard	Actual This Mo.	Actual Yr. to Date	Last Year
Muriatic acid	5 993 329	993 329	lb	.00225	100	103	101	[illegible]
Steam								
Electricity								
Compressed air								
Water—purchased								
Water—plant								
Fuel—gas, coal, oil								

Statement of Production Cost	Actual Cost	Inventory Standard Cost	Variance	Unit Costs Current Month Actual	Current Standard	Year to Date Actual	Last Year Actual
Muriatic acid	2 234 99	2 234 99	-	.225	.225	.225	.225
Net total matls	2 234 99	2 234 99		.225	.225	.225	.225
Steam							
Electricity							
Compressed air							
Water—purchased		9 94	9 90		.001	.002	.001
Water—plant	164 98	264 20	99 22	.019	.027	.024	.024
Fuel—gas, coal, oil							
Labor—mfg	302 10	227 [illegible]	- 75 [illegible]	.032	.025	.039	[illegible]
Supervision—mfg	48 02	46 67	- [illegible]	.005	.005	.004	.015
S.S. tax, comp. ins., pensions	22 30	19 57	- 3 [illegible]	.002	.002	.004	[illegible]
Repairs minor	150 [illegible]	238 [illegible]	138 [illegible]	.016	[illegible]	.009	.12
Repairs major	103 38	238 40	135 [illegible]	[illegible]	[illegible]	.017	.002
Supplies	130 36	47 [illegible]	- [illegible]	.13	.005	.017	.139
Laboratory	15 30	39 73	[illegible]	.002	[illegible]	.57	.001
Clothing and laundry		9 93	9 93		.001		.001
Refrigeration & ice							
Chg. to finish operation							
Total direct cost	900 99	1 172 13	230 [illegible]	.098	.118	.111	.183
Depreciation	456 98	408 93	- 39 95	.049	[illegible]	[illegible]	.051
Controllable—F.I.E.	251 [illegible]	264 33	- 2 81	.028	.028	.027	.023
Non-controllable—F.I.E.	153 [illegible]	49 33	- [illegible]	.016	.010	.012	.08
Total indirect cost	881 71	9[illegible] 50	- 87 12	.093	.01	.096	.095
Total mfg. cost	[illegible] 069 57	[illegible]	10[illegible] 12	[illegible]	[illegible]	[illegible]	.393
Total finishing cost	179 13			.019		.021	.036
Total cost 2.02 [illegible]				[illegible]		.495	.503

X. PLANT OPERATING DATA, contd.

Month of Aug.1947				Year to Date 1947
%	Pounds	Production	Pounds	%
0		Aroclor 1142		
0.98	6,000	1148	6,000	.08
65.12	396,500	1154	4,935,373	67.95
33.90	206,400	1160	1,178,200	16.22
		1162		
		1164		
		1168		
		2565	324,223	4.46
		4065	258,000	3.55
		5042		
		5060	561,600	7.74
100.00	608,900	Crude Aroclor Total	7,263,396	100.00
	6,728	" " lbs/Chlor-Day	6,719	
Per Lb.	Pounds	Raw Materials Used	Pounds	Per Lb.
		(1) Diphenyl		
		1142		
0.600	3,600	1148	3,600	0.600
0.472	187,200	1154	2,329,200	0.472
0.419	86,400	1160	493,200	0.419
		1162		
		1164		
		1168		
		2565	87,750	0.271
		4065	58,500	0.227
		(2) Santowax C		
		2565	29,250	0.090
		(3) Santowax R		
		4065	39,000	0.151
		5042		
		5060	234,000	0.417
		(4) Santowax Isomers		
		(5) Chlorine		
		1142		
1.000	6,000	1148	6,000	1.000
1.110	440,000	1154	5,525,000	1.119
1.226	253,000	1160	1,463,700	1.242
		1162		
		1164		
		1168		
		2265	431,000	1.329
		4065	342,000	1.326
		5042		
		5060	692,000	1.232

X. PLANT OPERATING DATA, contd.

Month of Aug.1947				Year to Date 1947
Per Lb.	Quantity	Services	Quantity	Per Lb.
1.117	680,000	Steam - Lbs.	7,466,000	1.028
		Water - City Cu.Ft.		
0.246	150,000	Water-Recovered,Cu.Ft.	1,512,000	208
0.064	39,000	Gas - Cu.Ft.	1,234,000	.170
0.008	5,000	Electricity - KWH	100,500	.014
Lbs.M.H.	Man Hours	Labour	Man Hours	Lbs. M.H.
825	738	Operating	7,571	959
2,859	213	Maintenance	2,201	3,300
		Packing	218	1,491
		Shipping	148	2,102
%	Chlor.Hrs.	Operating Time	Chlor.Hrs.	%
100.00	4,464	Hours-Calendar	34,992	100.00
48.95	2,185	Hours - Shut Down	8,027	22.94
51.05	2,279	Hours-Gross Operated	26,965	77.06
	107	Hours - Delay	1,017	
	2,172	Hours - Net Operated	25,948	74.15
4.70		Delays % of Gross Oper.		3.77
		Delays:		
		Out of Chlorine		
		Waiting on Charge		
		Clean Chlorine Lines	15	
		Clean HCl Lines	8	
		Clean Circ. Lines	15	
		Change Cooking Coil	169	
		Change Distributor	10	
		Change Chlorine Valve	15	
	19	Repair Circ. Pump	155	
	14	Repair Chlorine Lines	70	
	12	Repair Acid Towers	28	
		Waiting on Blowing Tank		
		Low Steam Pressure		
	3	Repair Water Line	95	
	27	Miscellaneous	152	
	32	Repair HCl Line	166	
		Repair Charging Line	13	
		Change Water Line	58	
		Repair Cone Traps	48	
Pounds		Shipped and Packed		Pounds
12		Shipped		311,980
12		Packed		325,033
		Repacked		1,867
Containers	Pounds	Containers Used	Pounds	Containers
		40 Gal.Black Steel Used	343,934	661
		40 Gal.Black Destroyed		1
		33 Gal. Fibre Dms-Used		
		" Destroyed		
		100# Fibre Drums Used	650	7
		" Destroyed		
		Textile Cans - Used	250	2
		" " Destroyed		
		50 Gal.Galv.Drums	1000	2

X. PLANT OPERATING DATA, contd.

Month of Aug.1947				Year to Date 1947
	Pounds	Production	Pounds	
		Aroclor - 1242		
		Aroclor - 1248		
79.25	450,109	Aroclor - 1254	4,882,430	73.10
20.75	117,868	Aroclor - 1260	1,066,967	15.98
		Aroclor - 1262		
		Aroclor - 1264		
		Aroclor - 1268		
		Aroclor - 4465	240,581	3.60
		Aroclor - 5442	11	
		Aroclor - 5460	488,608	7.32
100.00	567,977	Dist.Aroclor - Total	6,678,597	100.00
	17,476	Dist.Aroclor lbs/Still Day	19,948	
% Recovery	Pounds	Raw Materials Used	Pounds	% Recovery
		Aroclor 1142		
		Aroclor 1148		
96.05	468,598	Aroclor 1154	5,014,936	97.36
98.80	119,299	Aroclor 1160	1,091,108	97.78
		Aroclor 1162		
		Aroclor 1164		
		Aroclor 1168		
		Aroclor 4065	259,998	92.53
		Aroclor 5042		
		Aroclor 5060	559,601	87.31
Per Pound	Quantity	Services	Quantity	Per Pound
1.160	659,000	Steam Lbs.	6,856,000	1.026
-	-	Water-City,Cu.Ft.	-	-
1.144	650,000	Water-Recov'd Cu.ft.	6,518,000	.976
0.751	427,000	Gas - Cu. Ft.	4,641,000	.695
0.026	15,000	Electricity KWH	82,000	.012
Lbs/M.H.	Man Hours	Labor	Man Hours	Lbs/M.H.
666	853	Operating	8,103	824
1,993	285	Maintenance	1,206	5,538
2,927	225	Packing	1,520	4,241
10,349	76	Shipping	1,074	5,580

DISTILLED AROCLOR REPORT

X. PLANT OPERATING DATA, contd.

Month of Aug.1947				Year to Date 1947
%	Still-Hours	Operating Time	Still Hours	%
100.00	1,488	Hours-Calendar	11,664	100.00
44.76	666	Hours-Shut Down	2,989	25.63
55.24	822	Gross Hrs. Operated	8,675	74.37
	42	Hours-Delay	638	
	780	Net Hours Operated	8,037	68.90
5.11		Delays % of Gross Oper.		7.35
		Delays:		
		Out of Crude		
		Clean Goosenecks	17	
		Clean Still	62	
		Change Coil	21	
	7	Change Pump	79	
		Change Cotton	15	
		Change Cake		
		Drain Scrubber	13	
	2	Drain Trap	2	
	5	Repair Ejector	19	
	27	Wait on Blend.Tank	56	
	1	Low Steam Pressure	266	
		Misc.	45	
		Install New Steam Jets	32	

Pounds		Shipped and Packed		Pounds
		Shipped Arocolor 1242		1,500
		1248		4,603,798
567,619		1254		948,895
106,599		1260		
		1262		
		1264		1,627
		1268		99,893
4,999		4465		607
		5442		389,371
107,282		5460		5,992,681
786,499		Total		6,445,898
655,890		Packed - Total		17,644
2,580		Repacked		

Containers	Pounds	Containers Used	Pounds	Containers
		Drums:		
		55 Gal.Galv.Locktop Used		
		" Destroyed		
213	106,000	40 Gal.Galv.Bung Used	234,252	470
		" Destroyed		5
101	59,725	50 Gal.Galv.Bung Used	1,333,930	2,288
		" Destroyed		
		50 Gal.Galv.Used	467,115	939
		" Destroyed		33
		33 Gal.Fibre-Used	150	1
		" Destroyed		
9	970	100# Fibre-Used	3,960	38
		" " Destroyed		
		Slack Barrels-Used	7,268	30
		" " Destroyed		
19	1,125	Textile Cans-Used	3,[illegible]	64
		" " Destroyed		
		Paper Liners-Used		
		" " Destroyed		
5	490,320	Tank Cars (1254)	3,364,800	34
1	106,000	Tank Cars (1260)	942,315	9

X. PLANT OPERATING DATA, contd.

Month of Aug.1947				Year to Date 1947
Pounds		Production		Pounds
19,311		Muriatic Acid - Total Pounds Day		174,610
% Recovery	Pounds	Raw Materials Used	Pounds	% Recovery
		Anhydrous HCl		
		-Equiv.18°Bé Acid		
		-Equiv.20°Bé Acid		
100.00	17,143	Muriatic Acid, 20°Bé	155,009	100.00
Per Pound	Quantity	Services	Quantity	Per Pound
		Water City - Cu. Ft.		
		Water Recovered-Cu.Ft.		
		Electricity - KWH		
Lbs.M.H.	Man Hours	Labor	Man Hours	Lbs.M.H.
1,207	16	Man Hours - Operating	75	2,328
		Man Hours - Maintenance	24	7,275
568	34	Man Hours - Packing	650	268
1,315	12	Man Hours - Shipping	37	4,695
%	Tower-Hrs.	Operating Time	Tower Hrs.	%
		Hours - Calendar		
		Hours-Shut Down		
		Gross Hours Operated		
		Hours - Delay		
		Net Hours Operated		
		Delays % of Gross Oper.		
		Delays:		
		Out of Chlorine		
		Clean Acid Lines		
		Clean HCl Lines		
		Repair Acid Lines		
		Repair HCl Lines		
		Repair Vent Lines		
		Repair Drowning Tower		
		Repair Absorber		
		Repair Coke Scrubber		
		Work on Chlorinators		
		Storage Full		
Pounds		Used and Wasted		Pounds
		Used in 18° Be'Acid		
		Used in AlCl3		
		Wasted		
Pounds		Shipped and Packed		Pounds
15,777		Shipped		173,698
19,311		Packed		174,040
Containers	Pounds	Containers Used	Pounds	Containers
		Tank Cars - Monsanto		
		Tank Cars - Foreign	74,700	1
138	15,732	Carboys "A" - Used	99,522	873
		Carboys "A" destroyed		
		Carboys - Stranger		

X. PLANT OPERATING DATA, contd.

Month of Aug.1947				Year to Date 1947
Pounds		Production		Pounds
993,329		Muriatic Acid - Total		8,529,464
63,066		Pounds Day		48,282
% Recovery	Pounds	Raw Materials Used	Pounds	% Recovery
	349,000	Anhydrous HCl	4,228,850	
		Equiv.18°Be' Acid		
90.37	1,099,213	Equiv. 20°Be Acid	13,334,941	63.96
		Muriatic Acid, 28° Be'		
	89,960	Limestone	640,160	
Per Pound	Quantity	Services	Quantity	Per Pound
		Water City - Cu.Ft.	292,000	0.034
0.580	576,000	Water Recovered-Cu.ft.	6,572,000	0.771
		Electricity - KWH		
Lbs.M.H.	Man Hours	Labor	Man Hours	Lbs. M.H.
4,415	225	Man Hours - Operating	2,847	2,996
41,389	24	Man Hours-Maintenance	274	31.129
36,324	26	Man Hours-Packing	319	25,595
28,707	24	Man Hours-Shipping	180	44,241
%	Tower-Hrs.	Operating Time	Tower Hrs.	%
100.00	744	Hours - Calendar	5,832	100.00
47.85	356	Hours-Shut Down	1,191	20.42
52,15	388	Gross Hours Operated	4,641	79.58
	10	Hours - Delay	401	
	378	Net Hours Operated	4,240	72.70
0.026		Delays % of Gross Oper.		8.64
		Delays:		
		Out of Chlorine		
		Clean Acid Lines		
		Clean HCl Lines		
	4	Repair Acid Lines	13	
	4	Repair HCl Lines	10	
		Repair Vent Lines		
		Repair Drowning Tower		
		Repair Absorber	360	
		Repair Coke Scrubber		
	2	Work on Chlorinators	14	
		Storage Full		
Pounds		Used and Wasted		Pounds
17,143		Used in 18° Be' Acid		155,009
		Used in AlCl3		
107,458		Wasted		4,801,323
Pounds		Shipped and Packed		Pounds
688,976		Shipped		7,963,458
944,436		Packed		8,164,674
Containers	Pounds	Containers Used	Pounds	Containers
10	671,420	Tank Cars - Monsanto	6,187,314	91
		Tank Cars - Foreign	1,695,570	23
154	17,556	Carboys "A" Used	80,612	708
		Carboys "A" destroyed		
		Carboys - Stranger		

RAW MATERIAL PRACTICE IN THE CHLORE ARACLOR

Batch Charge			Biphenyl		Santowax C or R		Chlorine		Distilled Aroclor Recovery	Still Charge	No. of Runs before Removing Bottoms
Aroclor	Biphenyl	Santowax C or R	Theory	Actual	Theory	Actual	Theory	Actual			
1242	4000 lbs.	---	.592	---			.84	---	---	1135 gals.	5 charges
1248	4000	---	.534	---			.96	---	---	"	5
1254	3600	---	.466	.472			1.10	1.122	97.57	"	5
1260	3600	---	.417	.419			1.20	1.246	97.83	"	5
1262	3600	---	.398	---			1.24	---	---	"	5
1264	3000	---	.380	---			1.28	---	---	1047	3
1268	3000	---	.348	---			1.36	----	---	"	3
2565	2250	750 lbs. Sant. C	.276	.282	.092	.091	1.30	1.320	Not distilled --		--
4065	1950	1300 lbs. Sant. R	.222	.230	.147	.152	1.30	1.328	90.72	1047	3
5042	---	4000 lbs. Sant. R	.---	---	.592	---	.84	---	---	"	3
5060	---	3000 lbs. Sant. R	---	---	.417	.417	1.20	1.251	85.0	"	3

These practice figures are for the period January - June 1947 in most instances.

X. PLANT OPERATING DATA, contd.

Mr. J. E. Crouch, in a letter dated May 18, 1955, shows that the theoretical figures for chlorine consumption at Anniston are on a different basis from those given on page X-3 above for the Krummrich plant. As an example of the Anniston methods:

Aroclor 1268 should contain 68% of chlorine, therefore at theoretical yield 100 lb. of Aroclor should be made from

$$2 \times 68 = 136 \text{ lbs. of chlorine.}$$

The theoretical yield on chlorine, therefore, should be

$$\frac{100 \times 100}{136} = 73.5\%$$

The Krummrich plant Theoretical Conversion Factors obtained from Experience are a little different in most cases.

The Anniston Production Standards for 1955 for Aroclors per 100 lbs. product are:

	lbs. Diphenyl	lbs. Chlorine		
1221	86.1	46		
1232	74.1	70		
1242	63.20	92.10		
1254	49.20	115.20		
1260	43.70	127.8		
1262	41.70	133.50		
1268	50.1	165.8		
			Santowax C	Santowax R
2565*	28.60	132	9.50	--
4465	26.10	151.5	--	17.40
5460	--	148.20	--	50.2
5442	--	104.5	--	69.2
1248	55.0	101.5	--	--

*Mr. J. E. Crouch in his letter of May 18, 1955 (mentioned at the top of this page) suggests that this figure is rather low. Only small amounts of 2565 had been made at Anniston, so good data were not available.

X. PLANT OPERATING DATA, cont'd.
Anniston

The standards for utilities, services, etc. are grouped for all the Aroclors, as follows:

Crude Aroclors:

Steam 106 lb./100 lb. crude Aroclor
Electricity 1.21 K.W.H.
Plant Water 18.69 cu. ft.
Fuel 11.84 cu. ft. (natural gas)
Laboratory 0.01 unit
Labour 0.06 man hour
Maintenance 12¢
Supplies 1¢

Distilled Aroclors (from Crude):

Steam 109.5 lb. per 100 lbs. distilled Aroclor.
Electricity 0.69 K. W. H.
Plant Water 76.95 cu. ft.
Fuel 67.09 cu. ft. (natural gas)
Laboratory 0.1 unit (4¢)
Labour 0.1 man hour
Repairs 8¢
Supplies 2¢

Mr. Ellenburg, January 20, 1948, gave 0.66 cu. ft. gas/lb. Aroclors the gas being about 1060 B.t.u./cu.ft.

XI. HAZARDS.

Toxicity

There are many literature references* to harmful effects of the type of "chlor acne" resulting from exposure to chlorinated diphenyls, especially in cases where people working with small electrical components have been exposed to the fumes of hot, highly chlorinated, Aroclors. Chlor acne is sometimes accompanied by gastric troubles, and there are literature references to liver troubles.

There was some trouble of this kind among the production workers at Anniston in the early days of the development of the Aroclors. At that time highly chlorinated Aroclors were being made from diphenyl which had come from low grade benzene. Since good benzene has been used, the same Aroclors have been made without trouble. In March 1955 it was reported that the Anniston diphenyl and Aroclor plants had run 12 years without lost time accident.

From the start of Aroclor manufacture at the Krummrich plant the operators have been supplied a clean change of clothes every day, and time has been allowed at the end of the shift for bathing. Operators are advised to wash hands and face before eating. The Anniston operators do not have the same issue of clean clothes.

* Percy May, "Chemistry of Synthetic Drugs" , 3rd edition, page 19, The "Chemist Analyst", September 1947, Volume 36, No.2 page 33, and a report by Dr. M. C. Lester, Anniston plant, 1937, give data on the toxicity of diphenyl.

MCC Bulletin P-115 (new edition) mentions 0.5 to 1.0 mg. per cbm in air as the highest safe concentration of higher Aroclors, and 10 mg. for more highly chlorinated Aroclors.

H. B. Edkins "Chemistry of Industrial Toxicology", page 149 gives 0.5 to 1.0 mg. as the allowable limit in working rooms.

The MCC Bulletin "Physical Properties of Aroclors" mentions systemic effects arising from the oral injection of Aroclors.

See the letters, E. Mather to P.J.C.Haywood, December 17, 1951, and Mather to Newman, January 8, 1952, reviewing the literature on the toxicity of Aroclors.

See also correspondence relating to the article in Chem. & Eng. News May 17, 1954, pages 2038 -, and the warning labels mentioned on page VI-34 above.

XI. HAZARDS, contd.

St. Louis, Main Office

cc: Mr. W. E. Hamer, Ruabon
Mr. W. H. Ritchie, Ruabon
Dr. H. R. Newman, Ruabon
Mr. S. M. Kulifay, Krummrich
Dr. D. S. Weddell, St. Louis
Mr. F. T. Marshall, St. Louis

December 11, 1951

Mr. P. J. C. Haywood

Newport (Airmail)

AROCLORS::TOXICITY

Since writing to you on December 3rd I have come across a letter of mine to Mr. W. M. Cooper, London, June 24, 1948, (copies to W.D.S., W.H.G., and others), calling attention to an article in the J. T. Baker Company's "Chemist Analyst", Volume 26, No. 2, page 33, September 1947.

I attach a copy of this article, and I think you will agree that the warning relates only to really bad exposure, not such as should occur in ordinary analytical work.

On October 27, 1947 Mr. Barbre of the Krummrich plant sent a copy of the Journal to Dr. Jenkins at Anniston for comment. Dr. Jenkins did not make any comments at the time, but the following extract from Mr. Barbre's letter is interesting:

> "During the 11 years of production here only one man evidenced dermatitis. He had also shown similar symptoms in several other departments. When making Aroclor #1270 which is flaked, a noticeable amount of fine fume solidifies in the air. If men are exposed to it during hot humid weather, the skin is irritated, but we have not had any difficulty in curing cases of dermatitis from it".

MONS 046364

E. Mather

XI - HAZARDS, contd.

The Chemist Analyst, Vol. 36, No. 2, page 33,
J. T. Baker Chemical Co., Phillipsburg, N. J.
September 1947

ON THE TOXICITY OF THE "AROCHLORS" (sic)

Robert M. Brown, Chief
Industrial Hygiene Section, Division of Health
Dept. of Public Welfare, City of St.Louis, Mo.

A recently published article (Maglio, M. Martin, Chemist Analyst, 35 94 (1946)), has recommended the substitution of one of the "Arochlors" as the melting-point bath liquid in preference to the customary sulphuric acid. As stated in that article "Arochlors" are a group of chlorinated diphenyls produced by the Monsanto Chemical Company.

There is need therefore to give warning. For the toxicity of these compounds has been repeatedly demonstrated, both from the standpoints of their absorption from the inspired air, as well as from their effects in producing a serious and disfiguring dermatitis when allowed to remain in contact with the skin. Since these effects have been repeatedly observed, industrial hygienists have taken care to see that the proper controls have been established wherever these products are used. For example, the maximum allowable concentration of chlorinated diphenyl for an 8-hour working day is 1 milligram per cubic meter of air.

It is probable that nothing like an uncontrolled industrial exposure will occur in the laboratory. However, whether an individual is subjected to a possible acute exposure to chlorinated diphenyl, and its serious consequences, will depend upon the size of the melting-point bath, the caution with which it is used, and the temperature to which it may be heated. Likewise, with careless handling of the material and the resulting contamination of the skin, clothing, laboratory towels, work table surfaces, etc. the way is left open for the producing of dermatitis. Scrupulous cleanliness must be insisted upon wherever this material is handled.

The foregoing remarks have been prompted by the belief that in recommending the use of a material with which is associated a potential hazard from the health standpoint, it is very important that the possible consequences be presented together with recommendations for correct handling.

XI. HAZARDS, contd.

Mr. A. C. W. Pennington of Newport, reporting on his American tour, December 29, 1950, page 5, writes:

"HEALTH AND SAFETY":

"At Anniston, no special protective clothing is provided for the Diphenyl and Aroclors operators. A daily change of clothing was provided in the past but this practice ceased before the war. Gauntlet leather gloves and face shields are, of course, available as required on the plant.

" Tins of cold cream ointment, theatrical quality, are to hand in the building, but the application of the cream is left to the operator's decision of the job in question. The men are expected to take a bath, in their own time, at the end of the shift. A good quality soap, and alcohol for rubbing down purposes, are provided. The operators are sufficiently trained in the need for personal cleanliness that a record of bath taking is not warranted.

"Emergency showers are provided on each floor of the Diphenyl building and safety notices are widely used.

"At St. Louis, Plant B, the Aroclors building is rated a toxic department. Each operator is provided with a complete set of clothing comprising hat, coat, trousers, combination underclothes, socks and rubber shoes. A clean change of clothing, except shoes, is placed in the operator's locker in time for the following shift. Men working extra shifts are given a clean set of clothing. Canvas gloves and goggles are provided and 'Ply' hand barrier cream is available for use when necessary.

"Twenty minutes' paid time is allotted for bathing at the end of the shift but, again, no record is kept that baths are actually taken. Theoretically, food is not allowed to be eaten within the Aroclors building. Instructions are issued that hands and face should be washed well before eating.

"Employees in toxic departments are given an annual medical examination and a lung X-ray every three years."

XI. HAZARDS, contd.

Toxicity, contd.

A good deal of work has been done on determination of the vapour pressure of the Aroclors, and on the determination of the concentration of Aroclors in air. See pages 9-, Section III, above, for a summary of this work.

In 1950 (letter April 20, N. F. Rapps to E. Mather) the Admiralty expressed interest in the determination of micro-quantities of Aroclors. MCC have a method for the determination of traces in air by means of the "Halidometer".

Mr. Ellenburg, March 15, 1954, mentions work done by H. B. Richards Jr., of MCC, June 17, 1953, on the determination of the concentration of Aroclors in air. Mr. Ellenburg also states:

> "------- work on the safe limits of Aroclor vapour concentration in air is being carried on by the Medical Department at the Kettering Laboratories in Cincinnati. This work on animals is well under way, and will help us to know a little better just what is the safe limit of Aroclor Vapours that one might work in."

A question has been asked about the possible harm to plants if Aroclor-containing paints are used in greenhouses. The reply is that most paints are somewhat harmful, and there is no evidence that Aroclors make them worse. Aroclors have been used as rabbit repellants, without doing any harm to vegetation.

Care is needed in laboratories etc. where Aroclors are used in heating baths; there should be very positive ventilation.

The vapours of hot Aroclors are distinctly irritating to eyes and nose above a concentration of about 3 mg per cbm in air. Newport plant report for December 1951 records burns by hot Aroclor -- a case of a splash into a man's eye -- without serious damage.

Packages of Aroclors leaving the Krummrich plant bear a label calling attention to possible toxic effects.

XI. HAZARDS, contd.

Fire Hazards

Diphenyl, of course, will burn, and reasonable care is needed in handling it. Precautions with toluene are mentioned also on pages VI-29, and under "Special Risks", below.

The Aroclors are handled as if non-inflammable. Drums of material are melted in the middle of the operating building, by open gas flames, and open gas flames are freely used to heat pipe lines.

An instruction, dated April 28, 1948, calls for a daily inspection of safety showers, fire extinguishers and gas masks, a check list being provided.

Flanged joints on Aroclor heating system can be a source of fume. See the notes on gaskets, page 2, Section XII, below.

Phosgene may be generated in electrical flashes and fires in transformers containing Aroclors, but this is not thought to be a major risk.

Safety Equipment

Each operator in the Krummrich Aroclor plant is supplied with goggles, rubber covered gloves, canvas gloves, rubber shoes and fume respirator. See also page XI-1. A spare suit of clothes is kept in a case in the department. The department has the usual first aid cabinet, gas masks, (chlorine), fire extinguisher, (toluene, diphenyl), stretcher, safety showers, drinking fountain, eye bath.

Special Risks

The usual care is necessary in dealing with chlorine, in lighting the gas furnaces, and in handling hot materials, steam lines etc. Diphenyl might start to burn in chlorine gas if conditions in the chlorinator were badly out of line.

A Davis "Vapotester", model M-1, Type A, (Division of Davis Emergency Equipment Co., Inc., Newark, N.J.) is held in the department for use in testing the air of the working building after toluene has been used, and for testing vessels before men are permitted to enter, and before "flame certificates" are given. The instrument contains a

XI. HAZARDS, contd.

Special Risks, contd.

catalytic cell electrically heated to a fixed temperature, and the air to be tested is aspirated through it by means of a "squeeze ball". If combustible vapours are present, the temperature of the cell rises, and the indicating thermocouple registers on a calibrated scale.

The instrument has a zero adjustment, and it is checked twice a month by the instrument department. Flame certificates must give the number of the Vapotester used, also the data of its last calibration by the instrument department. The instrument has been useful in detecting gas escapes, and in tracing spills of gasoline, etc., from a neighbouring oil refinery into a public sewer which runs under Monsanto territory.

Mr. Benignus, of St. Louis, September 1953, discussed the dangers of using Aroclors in indoor paints. He discounted the possible dangers from phosgene formed by flash discharges in transformers charged with Aroclors.

See the references to safe handling of glycidyl phenyl ether on pages IX-161 etc.

XII. EQUIPMENT LIST

The following details are largely drawn from pages 18 - of the report by Lyles, Soffranko and Becker, November 1946, and they relate to the plant "B" (now Krummrich Plant) equipment, unless otherwise stated. A list of Aroclor equipment at the Anniston plant was sent to MCL June 7, 1946.

General Note

Since diphenyl and some of the Aroclors become solid, or viscous, at ordinary temperature, attention has to be paid to steam tracing or jacketing of pipe lines, and to the provision of gas flames for local heating where necessary. With due care, because of the presence of diphenyl and in some cases of toluene and butyl acetate, naked flames are quite freely used in the plant. (See operating instructions, Section XV, below).

Pipelines used for Aroclors need heat insulation, also proper allowance for expansion where high temperatures are involved.

Flanged crosses are freely used to assist in locating and clearing blockages in the pipe lines. Lines for diphenyl and heavy Aroclors should be blown clear after use. This also refers to outdoor water and steam lines, for example rail tank coils, in cold weather at the Krummrich plant.

Schwarting and others, "Process Description -- for Aroclors" November 12, 1953, page 3 state "Since iron has a deleterious effect on Aroclor electrical properties, the distilled product is handled in monel, monel-clad, zinc or tin-sprayed, and 304 stainless equipment. Galvanized iron or stainless steel is used for the process piping beyond the distillation stage -----".

XII. EQUIPMENT LIST, contd.

Gaskets and Packings

The following gaskets have been found suitable for use in the Aroclor process(1948):

#901 Garlock - 1/16" 1" - 1½" - 2" - 3" and 4".
Used for all crude material transfer lines.

#900 - Yellow Garlock 1/8"and 1/16", sheet Gasket.
Used for finished material transfer lines.

Metallic Asbestos Goetze gaskets 1" - 2" and 3".
Used for solid Aroclor line flanges; will stand heat.

Goetze Aluminium Asbestos. 16" I.D. 21½" I.D. and 9" I.D.
Used for Pyranol Car heads.

Goetze Metallic Asbestos 2" flange gaskets used inside, top and bottom of 18" sparkler press.

#262. 1/2"x 3/8" square Garlock gasket. Used in flange face of Sweetland press.

Gum rubber flange gaskets used in HCl valves and lines.

#900 Garlock gaskets used on all Chlorine flanges, tank manhole covers, inspection plates, inspection plates, and between all pump and tank flange connections.

Gum or Garlock red rubber gasket material used on all HCl tank flange connections.

Samples of Goetze corrugated copper-asbestos gaskets were sent to MCL, April 9, 1947, and again June 24, 1948. The 2" I.D. x 4" gaskets at that time cost $0.189 each.

XII. EQUIPMENT LIST, contd.

Gaskets and Packings, contd.

See also the London Engineering Dept. Final Report on the start-up of the Newport plant, for a discussion of gland packings for use against hot Aroclors.

See also MCL Research reports on packings and gaskets for hydraulic fluids, for example: --

347	50/175/2	November 1950
355E	51/91/1-	August 1951
1151	DF 54/26/3-	June 1954 -
	DF 54/27/1-	April 1954 -
1152	DF 54/16/5	April 1955

Some gaskets failed under test, others damaged the goods.

Samples of the washers used for the bungs on Aroclor drums were sent to MCL, April 9, 1947. The 1" size cost $0.004 each.

XII. EQUIPMENT LIST, contd.

PUMP PACKING LIST

EQUIP.NO. PUMPS	SERVICE	SIZE PACK.	TYPE AND NO. OF PACKING
P-0629	#1 Chlor Pump	5/16 inch	#234 Garlock
" 578	#2 " "	"	" "
" 579	#3 " "	"	" "
" 1056	#4 " "	"	" "
" 731	#1 Scrubber Pump	3/8	" "
" 575	#2 " "	"	" "
" 604	#3 " "	"	" "
" 573	#4 " "	"	" "
" 574	#1A " "	"	" "
" 580	#1 Blow Tank Pump	5/16	" "
" 1509	#2 " " "	"	" "
" 594	#1 Still Pump	1/2	" "
" 1277	#2 " "	"	" "
" 565	#1 Still Rec. Pump	3/8	Palmetto
" 1258	#2 " " "	"	"
" 378	City Water Pump to Tourrill	1/8	#117 Garlock
" 1054	" " " " Tantalum	"	" "
" 1260	18" Sparkler Press. Pump	3/16	" "
	18" " Head Gasket	1/2 x 3/8	#262 "
" 585	#1 Diphenyl Stg.Tk. Pump	1/2	#234 "
" 592	#2 " " " "	"	" "
" 584	#3 Aroclor Stg. Tank Pump	"	" "
" 583	#4 " " " "	"	" "
" 929	#5 " " " "	"	" "
" 617	CTO347 - Diphenyl Stg. Pump	3/8	Palmetto
	Flaker Drum	1/2	#234 Garlock
	1170 Stg. Pot Agitator	3/4	#234 Garlock
P-0595	#1 Still Condenser Pump	1/8	#117 Garlock
	D 218 Dept.		
P-0427	Inhibitor Acid Pump	1/4	#234 Garlock
P-0943	Wilfley Pump at #1 HCL Stg.	--	Do not use pckg.
P-01119	" " " HCl Tr.Tk.	--	" " " "
	Dept. A-246		
P-0725	#7 Sweetland & GE Press Pump	3/8"	Cutno or Palmetto
P-0346	#6 Stg. Tank Pump	--	No Pckg. req'd.
P-01111	TCB Unloading Pump	3/8"	-- Palmetto

XII. EQUIPMENT LIST, contd.

Cocks and Valves

A 2" Crane all Iron Gate Valve #475½ Durimet Stems. New Style	For all 2" Chlorine lines.
2" Merco cock - Cast Iron) 1" " " " ")	For all crude Aroclor lines, Blow Tanks, chlorinators, scrubbers.
Powell, Rising Stem flanged 1½" Stainless Steel Valves.	For all finished Aroclor lines, also for all Pyranol lines.
Clip gate valves. Brass or all iron.	For all city water lines.
Barstock Needle Valves.	On all Manometers for chlorine and vacuum.
3" Crane #475½ New Style with Durimet stems.	On all HCl off gas lines between scrubbers and HCl absorbers.

NOTE: Mr. D. G. Furzey, December 5, 1951, reported that cast steel valves were used throughout for the hot process liquors.

Merco lubricated cocks with special high temperature grease are widely used. Monel or bronze valves, or cocks, are used wherever their use appears to be desirable.

See also the note on the use of Teflon lined cocks for chlorine at the Anniston plant, page 3, Section VI, above.

XII. EQUIPMENT LIST, contd.

Blowers

B-034 Blower from American Blower Company. Type 3V. Used to exhaust fumes from around the Pyranol filter press, F-0159. On roof of CR. Motor M-01752.

B-094 Blower from American Blower Corp. Serial #5364. Print G-46215. #30-CCW-BHD-FH type E fan with LS wheel. Welded steel pedestal for motor. Drive thru couplings. Clockwise rotation, horizontal bottom discharge. Order B7377 on 4/14/36, Req. 69122 on 4/14/36. Price $104.21. Driven by M-0832-3HP-1220 RPM In lean-to on second level platform. On flaker D-012.

B-0105 Clarage Blower purchased from Anniston. Fan #47361, Type CI #7 with outboard sleeve bearing. "V" belt drive to M-0904. Used with cyclone CT-0852. On platform in lean-to. Driven by M-0904. On flaker D-012.

B-0148 Blower from American Blower Co. Size #1-3/4 Sirocco utility blower. Direct connected counter clockwise up blast discharge. Includes supports of steel. Order B1776 on 1/22/40, Req. 5788 on 1/22/40 Charged out $154.55 on 3/29/40.

At CT-0763, Bldg. CR.
Direct connected to M-01710.
On melter and still tapping drums.

All blowers are of ordinary steel construction (EM, March 10, 1947).

Drawing D-6945 shows the fume exhaust system at plant "B".

XII. EQUIPMENT LIST, contd.

Closed Tanks

CT-0347 Tank 40' dia. x 16' 3½" high, 1st course 1/2" metal, 2nd course 3/8" top ¼" sheet - 2-6" CI nozzles on bottom, 4-4" CI noz. on top. 1-20" CI manhole on side shell near bottom, 1-20" th. steel hatch on roof. May 1927. Drawgs. D-978 and C-1117 (tank and foundations) (diphenyl store).

CT-0743 Jacketed vaporizer. 2 parallel vertical 10" dia. x 4'4" tanks surrounded by a 2'0" ID x 4'0" jacket. Use XHy pipe - jacket of ¼" plate, Drwg. C-818. Order B9038 on 5/6/36, Req. 69670. Price $180.00. 2nd floor 12' level south side Bldg. CR.

CT-0744 Middle chlor. tank made by Nooter. 3' ID x 16' lg. with flg. head, shell 3/8" and head 7/16" thick, 3/16" jacket over lower half of tank. 3/16" inner shell and 2 sets of 1" XHy pipe coils. Order B9037 on 5/6/36, Req. 69669. Price $716.00. Same as CT0745.

In 1947 there were four chlorinators in use, CT-0744, -0745, -0833 and -01283, all essentially similar, and later CT-01432 was installed, only slightly different.

Each chlorinator is a vertical steel vessel 3'0" ID, 16'0" high on the straight side, with bulged bottom and flanged cover. Drawings E 880, E 6209. Relief connection drawing C-822. Chlorine inlet, drawing B 659.

The body of each chlorinator has the following fittings: --

1. A jacket for water and steam, starting about 18" up the straight side and ending below the level of the charge. This arrangement leaves the bottom of the vessel available for flame heating, and it leaves room for a simple manlid low down on the straight side, for the chlorine inlet fitting described below. There is a spiral baffle in the jacket space, to increase the cooling effect, but the baffles favoured the accumulation of mud in the jackets, and at the

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0744, contd.

1. contd.
overhaul in November 1948 most of the spiral was removed, leaving only enough to preserve the spacing between jacket and liner. It was also planned to put two 4" nozzles, low down on the jacket, to facilitate cleaning. City water is used rather than well water, and the jackets are cleaned occasionally by means of inhibited acid. The used cooling water goes to a collecting drum CT-0752. There does not appear to be any trouble from flashing of water into steam in the jacket, in spite of the high temperatures used. See* however, the note below on the bursting of a jacket due to trapping of water in it at high temperature. The walls of the chlorinators, above the jackets, are thermally insulated, partly for safety and partly to keep down the temperature of the building.

2. Formerly (but now abandoned at the Krummrich plant except in the new chlorinator CT-01432) an internal coil for steam and water.

3. An internal cylinder, about 24" ID, with perforated bottom, and a retaining grid on the top, containing about 8' depth of iron-turnings. See page 1, Section VII, above, for a description of the turnings.

4. A bottom outlet, with cock, to the circulating pump, P-0578 etc. Also formerly, from No. 1 chlorinator, to the gas heated chlorinator CT-0808, used for making the higher Aroclors. A piece of heavy gauze is placed over the outlet to retain stray pieces of metal, and at the Anniston plant there is a small catch pot in the line to the pump, for the same purpose. The lines are arranged for easy clearing, and have steam tracing. (Aroclor jackets at Anniston).

* See page XII-10.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0744, contd.

5. A manlid very nearly at the bottom of the straight side, carrying the chlorine inlet fitting. This fitting consists of a shallow round box, set horizontally below the internal cylinder of iron turnings, and having about 240 round holes 3/16" diameter on its upper side and a 3/4" drain hole on the under side. The chlorine enters the box horizontally from the chlorine inlet control valve. It is advisable to have a spare inlet box, because the holes slowly become enlarged, the corrosion being a little faster on the side further from the inlet valve. Messrs. Hosmer and Soffranko, October 15, 1948 describe a distributor of cast nickel. Cast monel has also been mentioned. Nickel cast iron is the more usual material.

   The chlorine inlet line is thermally insulated with asbestos paste to prevent the use of flames intended to clear blockages from the line. Overheating at this point may cause very rapid corrosion, and organic matter might even take fire in the chlorine.

   The cover of each chlorinator has the following fittings:

6. An inlet, with sight glass for diphenyl from the measuring tank CT-0767, and formerly for high boilers from the melter CT-0763.

7. An inspection cover, carrying the return inlet from the circulating pump. This inlet is carried through to the centre line of the chlorinator, above the internal cylinder.

8. A vertical 10" central pipe for the off gas, leading to the bottom of one of the scrubbing columns CT-0759 etc. and so to the HCl absorber. This exit pipe has a hand hole with cover, on the side, for cleaning, and has a

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0744, contd.

8. contd.
relief disc of 4 lb. lead at one connection. (See sketch on page 10a, Section VI.)

If the relief disk bursts, the gas will go directly to the HCl absorption unit. The disks rupture at about 25 psig, but they usually last for at least several years. There is a mercury manometer on the 10" pipe to give warning of blockage in the off-gas system.

9. A return line from the scrubbers CT-0759 etc. goes into No. 1 chlorinator.

10. A vent valve, hand controlled, to a drum outdoors.

11. A thermo well, reaching down into the charge.

The fifth chlorinator, CT-01432, which has been installed at the Krummrich plant, is of the same type as the previous four, with jacket and internal coil, but the coil has been retained, whereas the coils had been removed from the older chlorinators. (At the Krummrich plant, the internal coils had been abandoned as being more liable to put water into the batch, but at Anniston it was the jackets that were abandoned, because on one occasion a jacket had burst when water was trapped in it when the chlorinator temperature reached a high value). To give still more cooling capacity, the stream of liquid circulated from and to the chlorinator, during the chlorination, can now be passed through a homemade cooler consisting of two vertical lengths of jacketed pipe in parallel. (2" pipes in 3" shells). There is a thermometer well (glass thermometer) in the top of each length of jacketed pipe. The water goes up one jacket then down the other, and so out to waste, no special provision being made to keep the second jacket flooded.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0744, contd.

The old uncooled circulation line has been left in position, so that this external cooler can be byepassed. The circulation pump, therefore, delivers to a horizontal header which has four outlet cocks facing upwards, plus a sample cock facing downwards over a drip vessel (see sketch).* One of the four cocks will direct the flow of liquid to the air blowing tanks, one will byepass it directly to the top of the chlorinator, and the other two will direct half the stream through each of the external coolers, and so into the top of the chlorinator.

This same No. 5 chlorinator has been equipped with a flow recorder-controller on the chlorine feed. An orifice plate with a sulphuric acid U tube is used on the chlorine line, as with the older chlorinators, and the gas feed to the chlorinator can be hand controlled, just as with the older units, but in addition, the same orifice plate is piped up to a differential pressure cell which operates the flow recorder-controller on the instrument panel. That instrument in turn actuates a 2" diaphragm valve on the chlorine line (see sketch). The valve has steel working parts, but a tantalum stem, and it has given very satisfactory service. Similar recorders would be obtained for the other chlorinators, except that the expense cannot be justified at present on any saving of manpower, or on any necessary increase in production.

These two changes both make for shorter average time cycles in the chlorination. Research work indicates that faster chlorination (at the same temperature) gives essentially the same product, though infra-red analysis suggests a slightly different isomer ratio. This mainly concerns the higher Aroclors, and a suggestion has beenmade that more rapid chlorination might increase

*page 10a, Section VI.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0744, contd.

the proportion of (waste) highboiling material produced. Not much experience has yet been gathered on the chlorination speed which may be attained, but 800 to 900 lbs. chlorine have been fed per hour on occasion, and a batch of 1260 has been run in 14 hours, and one of 1248 in 10 hours.

The Krummrich plant has not been pressed for output, and the present scheme of working is to run the chlorinators at a moderate speed, using each in rotation, overlapping the cycles of the different units, so as to maintain a fairly steady consumption of chlorine. This suits the chlorine department, which has no outlet for snift gas when the Aroclor department is not taking chlorine.

The off-gas from each of the older chlorinators leaves by a 10" pipe running about 12' up from the centre of the cover of the chlorinator, but the new chlorinator has only a 4" exit pipe with a very small cyclone, draining back to the vessel. It has its own Raschig tower with Aroclor circulation, then a cyclone, etc., just like the older units.

Aroclors above 1262 are no longer made at the Krummrich plant, so there is much less chance of getting much chlorine in the off-gas, and the off-gas scrubbers, consequently, are of less importance. They are still used, and they give a cleaner HCl for the absorption unit, but if they should be out of action for a time, no particular harm results.

The Aroclors involving the use of diphenyl high boilers are no longer made at the Krummrich plant, and the melter-blow case, CT-0763, for handling the highboilers, has been taken out. A second air-blowing tank, CT-02086, like CT-0750, has been installed for the ordinary Aroclors.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0744, contd.

At the Anniston plant there are eight chlorinators, similar in shape to those at the Krummrich plant. They have internal coils for steam and water, but the water jackets have been put out of use. The chlorinators are located in an open-sided steel structure, outdoors, and hang from brackets about 30" below their rims, so that they are free to expand downwards as they become hot. This expansion distorted the connections to the pumps, which at first were rigidly mounted, so that pumps were given spring mountings on slotted clips. More recently the pumps on two of the chlorinators have been mounted on rubber pads on steel frames hung from the vessels themselves, so eliminating the distortion due to the expansion of the vessels.

The new pumps are larger ones, Taber horizontal centrifugal pumps, direct coupled to 5 h.p. motors, 1730 RPM. All the chlorinator pumps have jackets for heating by Aroclor. The hot Aroclor comes from a standard gas-fired unit in a building near the chlorinators, passes through the pump jackets, then through the jackets of the pipes and of the cock on the bottom outlet of the chlorinator, then through the heating "pad" on the bottom of the chlorinator, and so back to the heating unit. The other parts of the circulation system on the chlorinators are steam jacketed, and the pad was formerly used for steam. The return stream of process material from the circulating pump enters the chlorinator by a pipe projecting a little way through the cover. This location of the return pipe perhaps favours entrainment of liquid in the off-gas, but if the pipe goes further down, it may dip in the charge, and permit siphoning at an inconvenient time. There is a heavy steel grating over the bottom outlet of the chlorinator, and there is a heavy stool, of steel grating, with a perforated plate on it, to support the catalyst basket. The top of the basket has a screen welded over it to confine the catalyst, an arrangement which prevents easy "topping up" of the catalyst, but by the time that

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0744, contd.
the topping up is needed, there is usually some other repair to be done inside the vessel. There is a screen over the bottom outlet, and a small catch pot, both intended to trap stray metal.

The connections to the inlets and outlets of the steam and water coils in the chlorinators go in through the sides of the vessels.

The following notes on an overhaul of the Krummrich plant chlorinator, November 1948, are of interest: --

1. Chlorinator cover was taken off and all old catalyst was removed and discarded. It was found that a solid mass of catalyst, mud, residue, etc. was in the full length of the annular space between the inner sleeve and chlorinator. This solid mass was also present to some extent within the inner sleeve. In addition, there was a hole approximately 8" in diameter the entire length of the catalyst bed.

2. The sleeve was removed from the chlorinator and the top half was renewed with 3/16" steel. Although the top half of the sleeve did not have holes, it was considerably thinner than the lower half.

3. "Ears" were welded on the sleeve to make for easy removal next time.

4. Installed new screen on top of sleeve, between sleeve and shell to keep catalyst from falling into annular space. Screen had 4 rows of 1/2" holes and was 1/2" thick.

5. A new cast iron chlorine distributor was installed. The old distributor was discarded because many of the holes were enlarged and beyond repair.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0744, contd.

6. A new grating, above the distributor was installed to hold the catalyst in place. The grating made of ¼" x 1" strips, was purchased as such.

7. A new monel screen plate was put over the bottom outlet of chlorinator to keep catalyst from going into pump.

8. Welded corroded portion on inside, lower flange of 12" off-gas line. Corrosion was at the weld where the pipe and flange are welded.

9. 7 x 55 gallon drums of catalyst, which filled the basket of the chlorinator were put in.

Below is listed additional information that may be of interest and serve as information for future use.

	Chlorinator Number		
	2	3	4
Outage measurement of top of sleeve	92"		92"
Outage after charging 3600# Biphenyl	76"	68"	85"
Outage of finished 1160 batch	18"	13"	37"

All measurements taken from top flange of shell and not the manhole.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0744, contd.

The performance of the chlorinator was improved as evidenced by the following figures:

	Before Overhauling Chlorinator	After Overhauling Chlorinator
Time required to chlorinate batch.	30 - 35 hours	20 - 24 hours
Sp.Gr. increase per hour after reaching 1.450 at 90°C.	0.012 - 0.015	0.018 - 0.026
Chlorine pressure entering chlorinator during last 6 hours of chlorination.	10-11 lbs.gauge	7-8 lbs.gauge
Valve in cooling water line to jacket	wide open after 4 hours of chlorination	½-2 turns open during entire chlorination
Chlorinator temperature toward the end of chlorination	150 - 165°C.	140 - 150°C.
Scrubber liquor build up.	Fast (Every 4-8 days)	Normal (15-25 days)
Still bottoms produced, for all chlorinators, after every 4 still batches	2500 - 3000#	1000 - 1500#

The above data are only approximate but do show a very definite improvement after overhauling the chlorinator.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0744, contd.

Number 3 chlorinator was overhauled January 24, 1949 with similar results. In this chlorinator the chlorine distributor was found to be fit for re-use and the new grating installed to support the catalyst was ordinary floor grating. The water jacket was found to be reasonably clean, but two 4" nozzles were welded on at the bottom of the jacket, to serve as cleaning openings.

There are no deflectors in that jacket.

The measurements down from the flange of the shell were:

	Before overhauling	After overhauling
To top of sleeve	92"	92"
To level of 3600# biphenyl charge	68"	88"
To level of finished 1160 batch	13"	31"
To level of finished 1154 batch		46"
To level of finished 1148 batch		56"

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0745 West chlor. tank same as CT-0744. On Dept. struct. steel MCS-0650. #1 chlorinator.

CT-0748 Diphenyl storage tank made by Graver Drwg. C609, 10' dia. x 26' long straight side, dished heads 9/16", shell 1/2", welded const. 2" xHy pipe coils, 1" risers, manhole covers, outlets and sump. Pump P-0585. Order B7700 on 4/17/36, Req. 69220. Price $1000.00. drwg. 609. Underground west of CR, 1st from south. Same as CT-0749.

CT-0748 was originally installed for general use, mainly on Aroclor brought from Anniston for use in the scrubbers CT-0759 etc., and CT-0749 was piped up to receive material, in emergency, from the vacuum stills. Both 0748 and 0749 are now used to store diphenyl.

CT-0749 Aroclor 1148 and 1142 Aroclor stg. tank. Same as CT-0748.

Horizontal boilers in pits outside the shed.

Submerged pumps actuated by remote control from near the scrubbers. Molten diphenyl in from rail cars. Return overflow lines from the scrubbers. (disused).

Vent lines protected against weather and fire.

Steam coil.

One point of the thermo recorder is in CT-0748. The submerged pumps are carried on the manlid covers. Return line from overflow connections of diphenyl head tank, CT-0767.

CT-0750 Blow tank made by Graver. Dwg. E-863. 7' dia. x 7' str. sides, dished heads ½", shell 3/8". 5 turn coil of 2" xHy pipe supported on brackets welded. Order B7704 on 4/17/36, Req. 69224. Cost $325.00. On steel MSC-0650 dept. steel, 12' level, 2nd floor level Bldg. CR. 8/10/54:Network of pipe with 1/8" holes. Air jet in stack.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0750 The cover of Blow tank CT-0750 carries the following connections: -

Inlet from chlorinator pumps, with sight glass.
Float with indicator scale.
Compressed air in, to a grid of perforated piping.
Connections to steam coil.
Haveg vent column through the roof.

There is an angle thermometer on the side.
Bottom outlet to Pump P-0580 for delivery to the stills S-043 and S-062, or to drum filling on the floor immediately below the tank.
Side manhole.
The float has a simple brass chain passing over two pulleys to an indicator hanging in front of a scale.
Drawing D-01292 replaced E-863.

CT-0751 Cyclone collector purchased from Graver. Dwg. B645.
48" dia. x 27" on str. side.
3'6" long and tangential inlet near top of side.
3/16" steel throughout, of welded construction.
Order B7712 on 4/17/36, Req. 69232. Price $84.00
On 2nd floor level, lean-to of CR.

CT-0752 Vertical steel closed tank made by Graver. Dwg. C-804.
4' dia. x 5' high with flat bottom and dished flanged head.
Sides, bottom and top of 5/16" flange ½", welded const. with outlets.
Order B7709 on 4/17/36, Req. 69229, Price $120.00
On 1st floor, west side of CR. (Hot Well).

This tank for recovered hot water from the chlorinators.

Formerly received the barometric legs of ejectors MSC-0760 etc..

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0753 (also CT-01396) Coke scrubber on the vacuum still system.
Scrubber tank made by Graver. Dwg. C-807.
Steel, closed tank, 3' dia. x 10' long with dished bottom and flgd. dished head, shell 5/16", bottom 3/8", head 5/16" with 1" thick flanges.
Price $155.00
On 3rd floor elevation bldg. CR.
Coke scrubber on #1 still.

Vapours from the still receivers CT-0771 and 01377 enter at the top and the unabsorbed gases leave at the bottom via a cock, to the ejectors, MSC-0760, 01392, and 01686.

The scrubbers are filled with coke, which is flushed with caustic soda solution a few times a year. There is no history of serious corrosion trouble in the ejectors.

Mr. D. G. Furzey reported, December 5, 1951, that cotton wool in the scrubbers was renewed every few years.

Wide hand hole at the top for charging with coke and for flooding with caustic soda solution. Drain to sewer.

It is convenient to divide the distillation system into sections by means of isolating cocks and to have a manometer connection to each section to assist in locating vacuum leaks.

Mr. Soffranko points out the need for an inspection cover on the top of the scrubber.

There is a layer of cotton wool on top of the coke in the scrubbers. The scrubbers are needed mainly for the distillation of high boiling Aroclors, because there is a little HCl formed by decomposition, but they also trap water which might come back from the ejectors. Mr. Pemberton, December 1950, reported that Anniston gave up the use of caustic soda in the coke scrubbers after the Karbate ejectors were installed. The cotton wool becomes glazed over with heavy Aroclors and the vacuum lines may choke.

See also the comments on pages VII - 4 and 5.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0754 Scrubber liquor pump tank made by Graver. Dwg. C-800, 4'6" dia. x 5'6" on str. side with dished heads, shell 3/8" thick. Heads 1/2" thick, welded seams, pads and nozzles. Coil 3 turns of 1½" xHy pipe.

Order B7703 on 4/17/36, Req. 69223, Price $208.00. Tank mounted on steel, east tank on 38' level. Same as CT-0755 and 0756.

Of the five scrubber liquor pump tanks, three are coil heated and the two newer ones are jacket heated. Jacket heating is preferred as being less liable to put water into the Aroclor circulation system.

These tanks were formerly charged with Aroclor (coming originally from Anniston) stored in vessel CT-0749, and pumped from it by its submerged pump.

It is now found more convenient to charge them from a partly chlorinated charge in #4 chlorinator, pumped up by means of the chlorinator pump. With slow chlorination, especially in the early stages of chlorination, the scrubbers tend to pick up diphenyl, and the liquor becomes less dense. With faster chlorination, the circulation liquor tends to increase in density. When the liquor needs to be renewed it is dropped to the melter CT-0763 for return to one of the chlorinators. The foreman calculates from the density what volume should be taken to give the equivalent of a fresh diphenyl charge to the chlorinator.

See page 10 Section VI, for the method of calculating the equivalent.

See letter E.M. to M. B., May 17, 1948 on the handling of the scrubber charges.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0754 contd.

The cover of the scrubber liquer pump tank carries the following connections:

Vent line with hand operated valve normally open to a drum outdoors.

Steam coil, in and out unless a jacket is used.

Aroclor line in from CT-0749.

Aroclor line in from chlorinator.

Return line from tower and cyclone, with sight glass.

Dipping hole.

Submerged pump with its delivery line.

There is a thermometer well in the side of the vessel above the steam jacket.

An angle thermometer is used. Exact temperature control is not needed.

Bottom outlets to chlorinators and to melt tank CT-0763, and to a levelling line by which material may be passed from one scrubber tank to another

Overflow (disused) to CT-0748 and 0749.

CT-0755 Scrubber liquor pump tank. Center tank. Same as CT-0754 and 0756.

CT-0756 #1 scrubber liquor pump tank. West tank. Same as CT-0754 and 0755.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0759 #4 East Aroclor scrubber, used on CT-0754. Made by Graver. Steel tubular tank, 12" dia. x 8' high, removable head. Pkgd. with 6' of Raschig rings. Chg. out $84.00. Drg. C-798 and B-660. 15" dia. cyclone.

Gas in at bottom by wide descending pipe from the top of the chlorinator off gas pipe (the HCl manometer branch comes off the top of this line) and out at the top to the cyclone CT-01259,(01279, 01280, 01281 and 01282.)

Liquor in at the top from the submerged pump in the corresponding circulation pump tank, and out at the bottom by seal bend with sight glass to the same tank.

There are four water-cooled chlorinators and one gas-fired one and for these there are five scrubber systems all alike except that the one on the fired chlorinator can be gas heated. The off-gas from a water-cooled chlorinator ascends in a wide pipe to the bottom of a packed column with Aroclor circulation. From the top of the column the gas goes via a cyclone to the header leading to the absorption system of the Aroclor plant or to that of the chlorbenzol plant as required.

The Aroclor for each column is circulated by a submerged pump in a scrubber liquor pump tank, and the liquor draining from the tower, plus any trapped in the cyclone, returns to the liquor tank via a deep seal bend and a sight glass. There is a sampling cock at the bottom of the seal bend, and there is a thermo-well near the S. G.

The gas from the gas-fired chlorinator goes to the monel gas chamber CT-01258 where it deposits most of its entrained solid Aroclor, and thence by wide steel lines, with cleaning flanges, to a scrubber system which is similar to the others, but which has arrangements for gas heating. (The gas-fired chlorinator went out of use in 1949).

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0759 Only chlorinator #1 is normally used to feed the gas-fired chlorinator and #1 is not recharged until the gas-fired chlorinator is empty. Nevertheless, there is a scrubber system for #1 chlorinator as well as for the gas-fired chlorinator. The gas from #1 can be put to either of these scrubbers. It can in emergency be vented to atmosphere.

Mr. D. G. Furzey reported, December 5, 1951, that the scrubbers and separators sometimes block with solid which has to be melted out with blow torches.

A scrubber was installed at Newport, but after some experience the Raschig rings were taken out of it and the circulation was stopped.

It was found that the off-gases from high boiler chlorination batches deposited rather viscous material in the empty tower. This raises the question of the contamination of diphenyl liquors in the scrubbers, with high boiler products, but the contamination appears to be unimportant.

The towers were finally bye-passed, very little material was collected in the cyclones which came next in line of flow, and there was no evidence of trouble due to organic matter in the HCl absorption unit.

At Krummrich plant the increase in volume per scrubber came to about 10 Imperial gallons per week.

The cyclone at Newport corroded through. The seal bend was removed from its drain line as being unnecessary.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0760 #2 Center Aroclor scrubber. Same as CT-0759.

CT-0761 #3 scrubber tank made by Graver. Steel tubular tank 12" dia. x 8' high, removable head. Packed with 6' of Raschig rings. On CT-0968. Chg. out $84.00. Drg. C-798.

CT-0762 Vent line scrubber stored in CR lean-to. Made by Graver. Steel tubular tank, 12" dia. x 8' high (50# std. pipe 3/8") removable head. Dwg. C-798. Packed with 6' of Raschig rings. Drg. C-798. Supports B-663.

CT-0763 Melt and feed tank made by Graver. Dwg. C-810. Closed steel tank 4'6" dia. x 5'0" on str. side, shell 3/8", dished bottom and bolted dished head all ½". 2" xHy pipe coil, 10 turns with 3 supports welded to head. Tk. test 90# and coil 300# Hydro. Press. Order B7710 on 4/17/36, Req. 69230. Price $316.00. ½ way in floor, south side of CR.

The cover carries the following fittings: --

Manhole and cover, with handhole for admission of broken lumps of diphenyl highboiler.

Steam coil in and out.

C. air connection and vent (Dry air from FC-046.)

Inlet from all scrubbers CT-0759 etc.

Outlet to drums and to header leading to chlorinators.

There is a hand operated hoist with lifting hooks for drums over the manhole, and a ventilation hood connected to blower B-0148. The melter is set in the ground floor of the crude room.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0764 North Hopper Storage Bin made by Graver. Dwg. D-512. Vertical, 6' x 6' x 10'8½" high overall, with sloping bottom and bolt angles. 3/16" plate throughout with reinforced flat bolted on cover with trap door. Entire inside of hopper and underside of cover and trap door sprayed, 1st with .005" zinc and then .003" tin.

Order B7713 on 4/17/36, Req. 69233, Price $288.00
Southwest end of CR lean-to. Same as CT-0765.

CT-0765 South hopper storage bin. Same as CT-0764.

CT-0766 Circulating cooling tank made by Graver. On No. 1 Still. Dwg. B644. Steel tank 2'6" dia. x 3'0" str. side with dished head and welded on pipe legs, shell ¼", heads 3/8". Coil of 1½" dia. xHy pipe, 4 turns on supports welded to inside of shell. Test tk. 225# and coil 300# Hydro press. Safety Valve. Gauge glasses.

Order B7706 on 4/17/36. Req. 69226. Price $110.00.
On 38' floor level, Bldg. CR.

I-0510 Taylor Fulscope temperature indicating controller, 0-110°C., with steel diaphragm valve on the steam inlet to the coil in CT-0766. Operated from the temperature in CT-0766.

CT-0767 Diphenyl measuring tank made by Graver. Dwg. C-799. Steel tank 4'6" ID x 5'6" str. side, dished heads, shell 3/8, top head 3/8 and bottom head 3/4" with ½" jacket over it and extending 9" up on tank side. Welded throughout. Test tk. 60# and jacket 105#. Hydro. press. Return line to store tank.

Order B7701 on 4/17/36. Req. 69221, Price $329.00.
At 38' level, Bldg. CR.

Drg. C-799.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0767 contd.

Steam traced vent to drum outdoors.
Manlid. Dipping hole with cap.
Fixed overflows at 4 levels back to diphenyl store tank CT-0748 by steam traced line with sight glass and right angle cross pieces with blank flanges.

Lower part of vessel jacketed for steam from the 70 lb. main. Angle thermometer 30" long through side of vessel. Bottom outlet to header leading to the 4 chlorinators. S. G. at each chlorinator.

CT-0768 # 3 Aroclor 1260 finished product storage tank. Made by Graver. Dwg. C-609. Horiz. steel 10' dia. x 26' long on straight side, dished heads, shell ½", heads 9/16". Test tank 60# and coil 300# Hydro. Press. Coil 2" xHy pipe with 1" xHy risers. Welded construction throughout. All inside and flg. surfaces, coils, supports, etc., to be sprayed first .005" zinc and then .003" tin. Same as CT-0769. Order B7711 on 4/17/36, Req. 69231. Chg. out $1385.00. $CaCl_2$ vent trap.

CT-0769 # 4 Aroclor 1254 Finished Product Storage Tank. Same as CT-0768.

CT-0770 Tubular heat exchanger made by Nooter. Dwg. B651, Plant A, Size 12" dia. x 5'0" lg. having 19 x 2" OD 11 ga. Monel Tubes, Monel tube sheets, Monel reducer and carbon steel jacket and back-up rings on flanges.
Order B8848 on 5/1/36. Req. 69605. Price $660.00
Above No. 1 still SO43, supported by 3rd floor steel.

Plant "B" (Krummrich Plant) continue to recommend monel for the condensers on the vacuum stills. See also item CT-01378.

Only monel in contact with the goods on the "descending" side of the condensing system.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0770, contd.

Mr. Thrift, December 5, 1949, raised the question of the necessity of using monel for the vacuum still condensers and subsequent vessels. Mr. Ellenburg of Anniston replied, 15th December, 1949, stating that iron contamination of the Aroclors would have a bad effect on the resistivity and the power factor of the products. Steel also hastens the darkening of Aroclors at temperatures of 80°C. or higher.

Messrs. Hodges and Barbre of the Krummrich plant replied that monel was satisfactory for the condensers and receivers but that there were no reasons to believe steel would not be satisfactory. Although General Electric Company report that steel is satisfactory for storage and treatment vessels, Krummrich plant do not care to risk the use of steel because of the high standards required by G. E. for Aroclors.

CT-0771 Monel vacuum receiver made by Nooter. Dwg. C-795. (Plant A). Size 5'6" dia. x 5'6" str. side, 3/8" shell, ½" bottom and removable head, 4 support brackets on outside of tank of steel. 2" IP size xHy Monel coil with 2" inlet and 1" outlet with Monel bars and U bolts. 3 turns 5' dia. along side and 3 turns spiral on bottom. 60# Hyd. test on tank and 300# on coil.
Order B7379 on 4/14/36, Req. 69118. Price $3240.00.
On second floor level, 12' level, Bldg. CR.
No. 1 still. Return line, for spoilt material, to the still of the vacuum still receivers
The covers carry the following connections:

Inlet from condensers CT-0770, 01378 via sight glasses.

The cylindrical surface of the sight glass fitting is steam jacketed and so is the pipe from the condenser to the sight glass.

Manlid with hand hole and dipping opening.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0771 contd.

Dial vacuum gauge and mercury U-gauge.

Thermo recorder well.

Vent cock.

Steam coil in and out. This coil goes well down into the bottom dish of the receiver.

Vacuum line to the top of coke scrubber CT-0753 and -01396.

Bottom outlet to pump P-0984 for delivery to the Sparkler press and to Blackmer pumps, P-0565, -01037, for delivery to the blending tank, CT-0779.

Electric strip heaters to drawing C-847, formerly used on these vessels have been abandoned, so no copy of the drawing has been requested for MCL. Material 1268 requires H. P. steam in the coil to keep it molten.

There is only one receiver for each vacuum still in the crude room,no fractionation being attempted.

There is no gauge glass or level indicator, only a gauging hole.* A receiver will hold a full batch from the still.

CT-0779 #1 blending tank made by Graver. Dwg. E-862. Welded steel tank 7' dia. x 7' high dished heads ½", shell 3/8". All inside surfaces and nozzles sprayed .005 zinc then .003 tin. 2" xHy pipe coil and supports. Yoke and drive supports. Cast bronze turbine stirrer and bronze shaft. Test tank 60# and coil 300#, hydrostatic Pressure.

Order B7707 on 4/17/36, Req. 69227, Chg. out $599.00. On 2nd floor level, 12' level, Bldg. CR. Same as CT-0780

*But see page 21, section VI.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0779 contd.
See also CT-01399, #2 Blender and CT-0780, Filtrate Receiver.

Drawings E-862 and E-6838 are essentially the same. Drawings A-459 and A-8853 of the bearing have been requested. Drawings D-2076, 2077, show the agitator rotor and stator.

Anniston, December 15, 1949, state that their blenders are of steel, sprayed with zinc then with tin and that the agitator in one case is of Tobin bronze (stated to be 60% Cu. 39.25% Zn, 0.75 Sn), and in the other case is of steel, sprayed with zinc then with tin.

The sprayed agitator is said to be satisfactory. Plant "B", January 5, 1950, state 0.005" Zn then 0.003" Sn for the tanks, and their agitators are of bronze of unknown composition, probably "ordinary commercial bronze". Anniston and Plant "B" agree in distrusting plain steel for these vessels. Anniston suggest Aluminium.

CT-0780 #1 filtrate receiver tank. Dwg. E-862. Made by Graver. Welded steel tank 7' dia. x 7' high, dished heads ½", shell 3/8". Same as CT-0779.

Materials 1260 and 1254 can be run from the receivers CT-0780 and 01400 to bulk store tanks. Other materials are filled off into drums.

To save time, most of the control tests are done on samples taken before filtration.

Material 1268 needs H.P. steam to keep it fluid.

The filtrate receivers are like the blenders CT-0779 and 01399 except that the filtrate receivers have no agitators.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0785 Middle acid tank south of CR on foundation. Made by Goodrich. Dwg. C-823. "Vulcalok" lines - steel tk. 7'0" ID x 8'6", shell 5/16", dished head 5/16", bottom 7/16", 4" outlet for safety plug on bottom. 18" manhole and cover, 5 - 5" flgd. outlets in head, 3/16" Triflex lining, rubber covered float and card for measuring device. Head with 6 - 1½" couplings for railings. Order B9983 on 5/19/36, Req. 69966. Chg. out $945.65. South of CR on foundation. Same as 0784 and 0786.

CT-0786 West acid tank, same as CT-0784 and 0785.

CT-0808 Storage tank and chlorinator with stirrer and gas burner. (Gas fired chlorinator). Made by Kilpatrick and Sons Fdy. Co. Dwg. A324, Cast Iron, 6' ID x 4'11" deep inside, wall 2" thick, bottom 3" thick and dished in 3". 3" outlet at lower rim. Marked S-459, weight 12,675#. Machining outside of top flange but without holes and machining outlet in rim including drilling and tapping holes. Cost of machine work $36.00. Order B8162 on 4/24/36, Req. 69398, Cost $517.65. On 12" level SW corner Bldg. CR. Drawing C830 gas heater on exit-line.

Subsidiary drawings:

Pr. relief door on 6'0" CI storetank	B-613
Clamp for electric strip heater	B-684
Vent for Cl tank	B-686
Shear pin unit -- for drive on agitator	C-605
Brick setting	F-867
Drive for stirrer	E-888
8'3" x 10'11" steel shell for tank	E-881
Vent stack for still heater	B-689
Still heater	E-886

This gas-heated chlorinator is also called S-0459. The chlorine enters the gas-fired chlorinator through a perforated pipe ring, and there is a separate inlet pipe

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0808, contd.
with a simple open end for use when the perforated pipe becomes choked.

The cover carries a wide neck through which material recovered from the monel catch vessel CT-01258 can be returned by means of a wide steel funnel. There is another neck, wide enough to admit a sampling bottle - also used as a gauging hole. Inlet from #1 chlorinator CT-01283 by a gas-heated gravity line or via pump P-0907 through lines with H. P. steam tracing.

9" monel duct, with cleaning flanges, rises at 45° from the chlorinator cover, then descends at 45° to the monel gas chamber CT-01258. From the monel chamber onwards the ducting to the scrubber is of steel with cleaning flanges.

Compressed air connection into the chlorine inlet for clearing (not very successful).

Agitator with water cooled stuffing box. Thermo recorder well. Bottom outlet with gas flame heating, to the retort vessels R-026 and R-027 in an adjoining lean-to building.

See Drawing E-867 for the furnace setting,
E-881 for the steel casing of the setting
E-888 for the drive
C-605 for shear pin for drive
B-613 for pressure relief door on furnace
E-886 for vent stack,
F-324 which shows a shallower vessel; the chlorinator pot is similar but is 4'11" deep.

The final chlorination to high chlorine content is more easily done in this separate chlorinator because higher temperatures are needed to keep the charge molten.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0808 contd.
Drawings B-684 strip heater
B-686 clamp for strip heater
B-689 vent stack for furnace

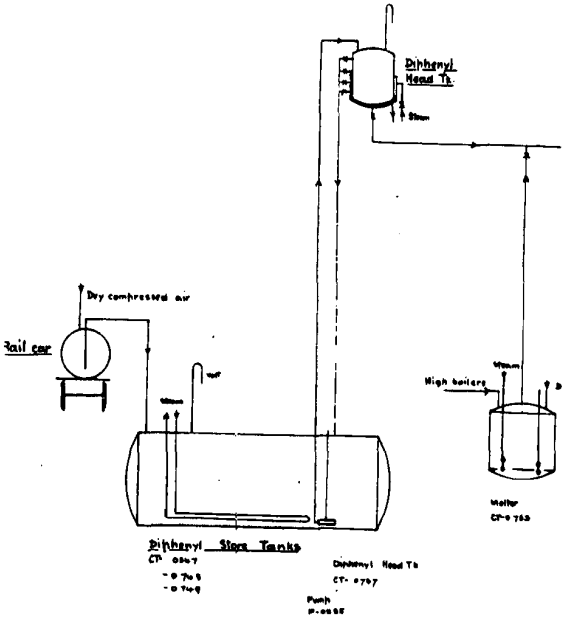
have not been requested as they are not of interest.
Strip heaters are not now used.

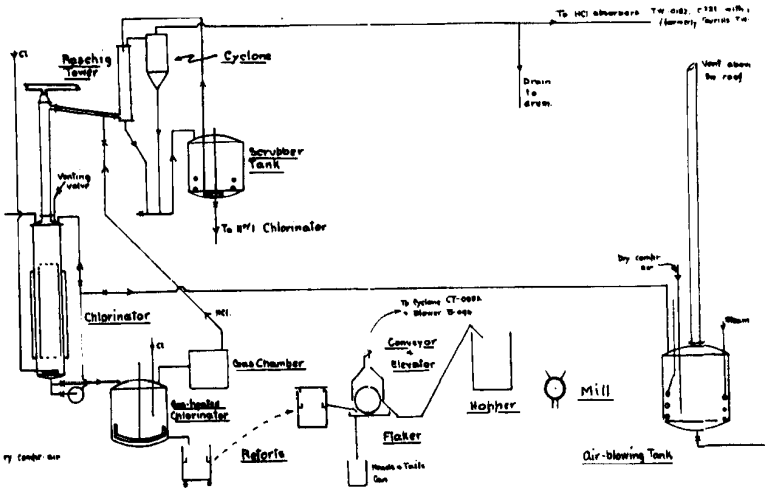
CT-0831 Water condenser made by Heine Boiler Company. Dwg. B483 (Plant A), used as temperature control for Aroclor condenser CT-0770. 12 tubes, 1" OD - 10 GA seamless steel tubing 10'-0½" long, 10'0" overall length of 6" std. pipe shell single pass. Order B8773 on 5/11/36, Req. 69576, Price $98.00.
Used on #1 vacuum still.
Safety valve on shell.

CT-0833 Chlorinator puchased from Leader Iron Works. Size 3' dia. x 16' made up complete including coils, inner shell, and grating. Dwg. E-880 and B-659. Order B-2813 on 2/5/37, Req. 76475 on 1/26/37, chg. out $790.00 on 3/31/37.
East chlorinator. #3 chlorinator. See CT-0744.

CT-0848 TCB stg. Tank. Made by Graver.
Tank dwg. E 4413
Coil C4412 for steam
General E4366
Tank 8' ID x 25'6" long welded. 3/8" shell, 7/16" dished heads. 13-26" and 2-24" dia. manhole openings on top.
2-1" xHy. W. S. pipe coil, 71' approx.
Order B3634 on 2/17/37, Req. 76931 on 2/12.
Price $920.00 West of CR underground. Pyranol Plant.

CT-0852 Raymond 2 stage cyclone dust collector sent to B by Anniston Plant. Each cyclone approx. 20" dia. - 21" str. section 32" depth of cone, steel construction, used with B-0105. In lean-to of building CR. No drawing.


Diphenyl Head Tk.
Dry compressed air
Rail car
High boilers
Diphenyl Stges Tanks
Diphenyl Head Tk
CT- 0707
Pump



Water

Hopper CT-0766 -0768

Mill ML-001

Air Blowing Tks CT-0780 CT-02068

Chlorinators
CT-0740
0748
0753
0758
0763
Chlorinator Pumps
P-0816
0819
-0824
-0827

Reactors
Columns T16-0806
CT-0798
-0780
-0781
-0782
Gas-heated
Chlor' CT-0800
Gas Chamber CT-0899

Cyclones
CT-01278
-01281
-01284
-01287

Scrubber Tks
CT-0784
0788
0792
0815
0760
Reforte
R-001, -007
Scrubber Tk. Pumps
P-0875
0878
0879
0880

Flaker D-012

Conveyor V-0046 [also V-0047 - goes to weighing tank]
Elevator L-001

MONS 046012

Removes CT-0766, -0766, -0768

FLOW SHEET OF EQUIPMENT, AROCLORS

N-3 Flow Sheet of Equipment

Ejector

Water Condenser

Coke Scrubber

Direction of steam in reverse to oil vapor

Steam

Still

Water Tk.

Still Receiver

Treatment Tk.

Filter

Toluene & Butyl acetate

Finished Batch Tk

To drums

Steam

Aroclor Store Tks. CT-0768, -0769, -01310

Batch Tks CT-0768, -01308

Pumps P-0892, -0893, -0894

Crude Aroclor St. Tks CT-0769, -0768 (formerly, now for withdrawal)

Still P-0893, -0894

Coil Furnace FC-0898, -0899

Condensers CT-0770, -01376

Still Receivers CT-0771, -01377

Still Pumps P-0869, -0897

Still Cyclone CT-0865

Water Tks. CT-0766, -01386

Water Pump P-0888

Pump P-0896

Coke Scrubbers CT-0765, CT-01396

Pump P-0871, -01787

Water Condensers CT-0931, -01981

Ejectors HSC-0760, -01968, -0970

Treatment Tks CT-0779, -01389

Filter F-0896

MONS 046013

V. PLANT LAY-OUT

The equipment at the Krummrich plant (formerly plant "B") had been fitted into a tall narrow building remaining from the 1914-1918 war period. The still and flaker for solid Aroclors, and some of the units of equipment for Pyranols, were accomodated in a lean-to building against the West wall of the main building. Store tanks, and the HCl absorption and treatment equipment were located in the open air alongside the building.

The following drawings, which were sent to London, April 25, 1947, relate particularly to the lay-out of the plant.

F 370
F 373
F 374
F 386
C 815
E 877
E 6838
C 6942
C 6943
E 6952
D 6956
D 10292

The building had been constructed with most of its structural steel outside the brick shell -- a special war-time requirement --, and the external steel work is now very badly corroded, also the roof of cement slabs has corroded away near the vent stacks, letting in the rain.

While the interior of the building was dry, there was very little corrosion of steel equipment and internal platforms, but the moist conditions now prevailing appear to be having a very damaging effect. A proposal has been made to roof the building with translucent plastic, and to do away with the windows in the side walls, because they require so much upkeep.

A fourth chlorinator was installed since those drawings were made, and more recently a fifth one has gone in.

V. PLANT LAY-OUT, contd.

The equipment for making solid Aroclors fell into disuse in 1949, and was pulled down in 1952.

The equipment at the Anniston plant is laid out in, and around, a rather lower broader building, with the chlorinators and their pumps in an open (roofed) structure nearby. Here again, most of the store tanks are outdoors.

The HCl absorber also is outdoors, at some distance from the Aroclor building. There is no equipment for making solid Aroclors at Anniston.

Both plants have railway tracks alongside the main building.

VI. PROCESS IN DETAIL

Handling of Diphenyl and High Boilers

Diphenyl and the Diphenyl High Boilers are made at Anniston and the Anniston Aroclor plant is supplied mainly by inter-departmental transfers. The Krummrich plant is supplied mainly from Anniston, but has been supplied to some extent also by purchases.

Diphenyl arrives at the Krummrich plant in rail cars, which are fitted with internal steam coils, and on arrival the material has to be melted with the usual precaution of first melting a hole down to the bottom of the tank by means of a bayonet or vertical coil to prevent development of pressure from the main coil of the tank, (note the possibility of development of full mains pressure in the shell of the rail tank if the coil should leak, or if there should be water on the bottom of the tank, note also that the diphenyl expands on being melted).

The molten diphenyl is then discharged over the top of the tank (by the use of dry compressed air) into storage tanks CT-0748, -0749 and -0347, which are underground in the open air. These tanks are automatically maintained by steam heat at about 90°C., and their connecting pipes and vent fittings are steam traced. Their temperatures are automatically recorded.

The diphenyl is pumped from them by submerged pumps P-0585, -0592, to the overhead feed and measuring tank CT-0767. This tank is steam jacketed to keep the contents at 85°C. or a little higher, by hand control, and it has overflow lines, at the 1800 lb., 2800 lb., 3600 lb., and 4000 lb. levels. The overflow lines are steam traced and the overflow can be directed to whichever store tank is in use. From CT-0767 the diphenyl charges can go by gravity to any of the five chlorinators CT-0744, -0745, -0833, -0183 and -0143 or (No. 2, 1, 3 4 and 5 respectively).

At Anniston the arrangements for measuring the diphenyl charges are less convenient; the charges are simply pumped from a tank on the floor of the building, the amount being measured by dip in the tank. The same tank has to be used alternatively for Santowax charges, so that it has frequently to be drained out into shallow trays, a procedure that is troublesome, and not very safe in view of the inflammable nature of the vapours.

VI. PROCESS IN DETAIL, contd.

Handling of Diphenyl and High Boilers, contd.

The crude and refined diphenyl high boilers, when formerly used at the Krummrich plant, arrived there in lumps in 75 lb. paper bags, or as material which had been melted into 550 lb. barrels. The barrels were broken open and the block of material cut up with axes as needed to get it into the melter CT-0763.

The melted material was blown from there, by air pressure, to one of the chlorinators. As far as possible, only the chlorinator CT-0745 was used for the higher Aroclors, to prevent cross contamination of the different kinds of Aroclors.

Before a chlorinator was changed to the production of Aroclor 5060 or 5460, it had to be rinsed through with distilled high boilers.

Throughout the handling of diphenyl, care must be taken to have the pipe lines hot (M. Pt. 69°C.) and to make sure that the tank vents are not blocked with sublimed material.

If the charge in the diphenyl measuring tank is allowed to cool, crystallization may occur on the walls of the vessel, resulting in incorrect charging of the chlorinator.

Handling of Chlorine

At the Krummrich plant, the Aroclor department is supplied partly with revapourised liquid chlorine (the chlorine simply being allowed to evaporate into the chlorine main, from the liquid chlorine store vessels which are sprayed with cold water; the vapourizer CT-0743 is no longer used), but the main supply is direct cell gas, dried, and compressed to about 30 psig. Actually, the Aroclor plant is able to take all the "snift" gas, and low-grade chlorine generally, without ill effect, though any considerable proportion of air in the chlorine may confuse the operator because the chlorinators will not show a heat evolution corresponding to the flow of gas. Air dilution also is detrimental in the HCl absorbers.

VI. PROCESS IN DETAIL, contd.

Handling of Chlorine, contd.

Conditions are similar at the Anniston plant, though the liquid chlorine used there is purchased material, which is passed, as liquid, into a simple vapourizer, made from old gas cylinders set vertically in a water bath automatically controlled at about 95°C.

The cell gas at Anniston is compressed only to about 15 psig, which is inconveniently near to the back pressure of a full chlorinator, so that the gradual increase of head of material in the chlorinator as the volume and density of the charge increase during the chlorination, necessitates close attention to the regulation of the flow of chlorine, and to the sharing of the chlorine between up to eight chlorinators, some at different stages of chlorination.

Ordinary chlorine valves have been used for this regulation, but more recently, "Durco" cocks, with Teflon lined bodies, and Durimet plugs, have been found more convenient at Anniston.

There are no flow meters on the chlorine feeds at Anniston, and the operator has to be guided mainly by the temperature behaviour of each chlorinator.

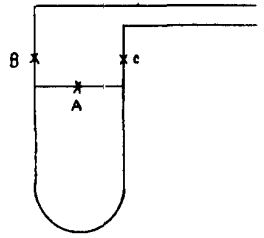
At the Krummrich plant there is a branch from the chlorine main for each of the five main chlorinators (formerly also one for the gas-fired chlorinator). Each branch starts with a rising stem control valve, followed by an orifice plate (1.1" round opening) with an U gauge containing 60° Bé H_2SO_4, then there is a pressure gauge on a branch line. The main pipe then rises to a level above the top of the chlorinator, and falls again to the inlet fitting, low down on the side of the chlorinator, where there is another rising stem valve.

The U tube of the H_2SO_4 manometer has a cross connection near the upper ends of the two legs, this connection being closed by a stop cock (A) when the manometer is in use. There are also two stop cocks (B & C) one at each end of the U tube. When the flow of

VI. PROCESS IN DETAIL, contd.

Handling of Chlorine, contd.

chlorine is first to be started, B and C are closed and A opened, otherwise the acid may be jerked out of the U. Then B and C are opened, and A gradually closed. Between batches, B and C are left closed, and A open.



See also the description of the flow controller to No. 5 chlorinator on page XII-5 below.

Chlorination

The chlorination procedure is essentially the same for all Aroclors up to 68% chlorine content, so there is no need to give a separate description for each grade. The charge of diphenyl has to be a little less for the more highly chlorinated grades, because the increase in volume during chlorination is greater, also the time temperature schedule, and the end point of the chlorination have to be varied, all as set out in the table on page 5, Section VI, below. Note, however, that chlorination is often started at as low a temperature at 100°C. gaining a little cooling effect, and so saving a little time.

CHLORINATION BATCH DATA

These figures refer to the standard chlorinators 3'0" I.D. 16'0" deep.
Taken mainly from Lyles, Soffranko and Becker, November 6, 1946 and from Schwarting and others, 1953.

Grade of Aroclor	Charge			Chlorination Time, Hours	Chlorine Consumption, lbs.	Temperature, °C.	Final Sp. Gr. at °C.		Weight Produced	
	Diphenyl lbs.	High Boiler Crude lbs.	High Boiler Distilled lbs.						Crude lbs.	Distilled lbs.
1221	4000				4000 x 86/85.1 *					
1232	4000				4000 x 70/74.1 *					
1242, 1242	4000			12 - 14	5735	120 to 135	1.342 to 1.346	65	6483	6165
1248, 1248	4000			14 - 16	6890	120 to 135	1.410 to 1.414	65	6600	6336
1254, 1254	3600			17 - 20	8550	120 to 150	1.502 to 1.506	65	7571	7200
1260, 1260	3600			18 - 22	10160	120 to 150	1.558 to 1.562	90	8227	7845
1262, 1262	3600			20 - 24	11089	120 to 150 or higher	1.568 to 1.572	100	8850	8517
1268, 1268	2800				10678	120 to 150 but always 20-50° above the hold point	1.570 then to hold point of 145 - 155°C.	100	7800	7000
1269, 1269	2800				11466				8100	7200
1270, 1270	2800				11950	as 1268 then finally to a hold point of 185 - 195°C.			8100	6800
1271, 1271	2800			(a)						
2565	1800	600			8849	120 to 175	1.500 then to softening point 68 - 70°C.	100	6560	
4065, 4465	1800		1200		10205	120 to 175	1.500 then to softening point 72 - 74°C.		7725	6950
5060, 5460			3600		10062	120 to 210	1.500/110°C. then to softening point 108 to 110°C.		7800	6200
							See page VI - 26 for slight variation in some of these limits.			

The 1954 operating instructions give about 2 hours less chlorination time (external cooling of the circulation stream).

Continue chlorinating in agitated chlorator to 235-245°C., 24 hrs. 220-325°C.

Continue chlorinating in agitated chlorator to 290-295°C., 24 hrs. 220 - 325°C.

Continue chlorinating in agitated chlorator to 305°C. minimum, 24 hrs. 220-325°C.

* Calculated from Anniston Standards

(a) The foremen, October 1947, stated that chlorination to reach a hold point of 150° took 16 hrs. then a further chlorination in the agitated pot, to 1171 end point, required 8 hrs. more. Overall time cycles for the Aroclors generally are given in the report of Lyles, Soffranko, Becker, November 6, page 164. --- At the Krummrich plant the operators are given a table of Sp. Gr. ranges a little narrower than those of the specifications (section IX B) so as to leave a little margin for safety.

VI. PROCESS IN DETAIL, contd.

Chlorination, contd.

Before a fresh charge is put into a chlorinator, which has been emptied after a preceeding batch, the chlorinator is allowed to cool to 150°C., or lower, this, of course, to diminish sublimation into the gas exit. The charge is admitted from the diphenyl head tank, or from the high boiler melter, or both, as required, (see the table on page 5, Section VI, above), and the temperature in the chlorinator is adjusted to 120°C. (Recent instructions say 100°C.)

Circulation of Aroclor is started on whichever of the scrubbers CT-0759 etc. corresponds to the chlorinator concerned, and the feed of water to the HCl absorber is adjusted to correspond to the expected flow of HCl, also cooling water as needed is put on to the absorber, then chlorine is admitted into the chlorinator. Usually the circulation of light Aroclor over the scrubber is not interrupted between batches unless a long delay is expected, but the valve in the line between the scrubber and the HCl absorber is kept closed until the diphenyl has been charged.

Circulation of the charge in the chlorinator itself is not started until the chlorination has been running for about 4 hours, this delay being practised in the hope of diminishing sublimation of the diphenyl into the off-gas line. Chlorine is passed in as fast as possible, having regard to the chlorine availability, the capacity of the HCl absorber, and the cooling capacity of the chlorinator. 2" - 3" H_2SO_4 column differential on the 1.1" orifice is a good starting rate, to be increased as circumstances permit. See also the table on the next page.

VI. PROCESS IN DETAIL, contd.

Chlorination, contd.

lb.Cl_2 /hr.	Gauge pressure lb./sq. in.								
300	1.7	1.6	1.5	1.3	1.2	1.2	1.1	1.0	0.9
400	3.0	2.8	2.6	2.4	2.2	2.0	1.9	1.8	1.7
500	4.7	4.3	3.9	3.6	3.4	3.1	2.9	2.7	2.5
600	6.8	6.3	5.8	5.3	4.9	4.5	4.2	3.9	3.7
700	9.3	8.5	7.8	7.2	6.7	6.1	5.8	5.3	5.0
800	12.1	11.1	10.2	9.4	8.7	8.0	7.5	7.0	6.5
900	15.3	14.1	12.9	12.0	11.0	10.2	9.5	8.8	8.2
1000	18.9	17.4	16.0	14.7	13.6	12.6	11.7	10.8	10.1
				1.1" orifice 60° Be H_2SO_4 Gauge					

The gauge readings are not recorded. The mercury manometer readings, which appear in the column "Chlor. Back Press. PSIG" on the daily record sheets, refer to the off-gas pressure, as discussed below, and, therefore, reflect the working of the HCl absorbers, rather than that of the chlorinators. Recent Krummrich plant instructions call for a chlorine feed rate of about 400 lbs./hour for the first 4 hours, then 600-700 lbs./hr., with water cooling on the external circulation line.

If Aroclor should get back into the feed line, as a result of some variation of pressure conditions, it should be quickly purged, by means of a short shot of chlorine, otherwise there may be local overheating and

VI. PROCESS IN DETAIL, contd.

Chlorination, contd.

severe corrosion around the gas inlet valve. There is a pressure test branch on the off-gas line, with a connection to a mercury manometer located near the chlorine control valve, so that the operator can control the feed to give not more than 6" Hg pressure in the off-gas line, i. e. he can keep within the limited capacity of HCl absorption equipment.

The temperatures must not exceed those set out in the table on page VI-5, above. After four hours, the circulation is started, and samples are drawn from the circulation stream, at intervals of 2 hours, for tests of specific gravity, hold point, or softening point, as set out in the table. (Test methods are detailed in Section VIII, below).

If the chlorination is carried beyond about the 1162 level, the product becomes first sticky, then gummy, and finally solid (at room temperature). The density of the scrubber liquor increases much more rapidly during this chlorination to higher values, because the efficiency of absorption in the chlorinator is less, and the chlorine passes through into the scrubber, where it is absorbed by the lighter Aroclor used for scrubbing.

Chlorination beyond 1162 was confined, as far as possible, to No. 1 chlorinator to prevent contamination of liquid Aroclors with solid Aroclors.

As the required test limit is approached, the control tests are made at shorter intervals, and when the correct end-point is reached, the chlorine feed is stopped, the chlorinator vent line is opened, the line to the HCl absorber closed, and the stream from the circulation pump diverted to the aeration vessel CT-0750 (for Aroclors up to and including 1162). Aroclor 1168 went directly to the vacuum still, the bottom outlet line from the chlorinators being heated with gas flames, and formerly, when Aroclors containing more than 68% of chlorine were made, chlorination would be carried to a hold point of 185 to 195°C. in the main chlorinator, then the batch dropped to the "chlorinator pot" CT-0808, for further chlorination, as indicated in the table on page VI-5, above.

VI. PROCESS IN DETAIL, contd.

Chlorination, contd.

Especial care is needed to prevent freezing during transfer of the higher Aroclors.

Care must be taken that the chlorinator vent is open and free when the chlorinator is being emptied, otherwise the batch may be contaminated with liquid sucked back from the scrubbing system. The mercury manometer on the off-gas line will give an indication of any drop in pressure in the chlorinator.

Typical record sheets are given at the end of this section.

Chlorination beyond 68%, i.e. for Aroclors 1169, 1170 and 1171

These products are no longer made, but the following description of the former method of production may be of interest.

Chlorination was carried on in two stages, the first in one of the ordinary chlorinators as for Aroclor 1168, and the second stage in the gas heated, agitated chlorinator CT-0808.

As the chlorination in the main chlorinator passed beyond the 1168 stage, external gas flames on the chlorinator body and on the circulating lines were needed to keep the charge always 25°C., or so, above the "hold" point, and the chlorination was continued in the No. 1 chlorinator to a hold point of 185-195°.

Towards the end of this stage, the gravity lines from No. 1 chlorinator to CT-0808 were checked to make sure they were clear, and -0808 checked to make sure there was room in it, then at the proper moment the charge was dropped to -0808, and the external flames kept on for about 5 minutes to drain the main chlorinator and the gravity lines. No. 1 chlorinator would not be recharged until it was certain that the gas heated chlorinator would be empty again in time to receive the next charge, partly because the same scrubbing system served No. 1 chlorinator and the gas heated chlorinator.

VI. PROCESS IN DETAIL, contd.

Chlorination beyond 68%, contd.

Chlorine was passed into CT-0808, by way of a dip pipe, quickly enough to raise the hold point by about 15-20°C. per hour, the charge always being agitated and kept 25-50°C. above the hold point, until the final test was attained as set out on the table on page VI-5 for the different Aroclors. The heat of reaction would usually maintain the temperature even with the furnace doors open, and the gas burners off. When the end point was reached, the burners would be put on to keep the charge molten, until it could be run off into finished product packages, or into the distillation pots of the solid Aroclor stills R-026 and 027, described in Section XII, below.

The off-gases from the gas fired chlorinator went through interceptor CT-01258 to trap subliming material, and then through the Aroclor scrubbers belonging to No. 1 chlorinator and so to the HCl absorber. The interceptor was cleaned after each second run, the cleanings being put into CT-0808.

Solid Aroclors

The gas fired chlorinator, CT-0808, and its ancillary vessels, have been taken out, also the still R-026 etc., the flaker D-012, and the mill, ML-045. The lean-to part of the building, which formerly housed most of these units, is now mainly used as storage space for full and empty drums.

Scrubbing of the Off-gas from the Chlorinators

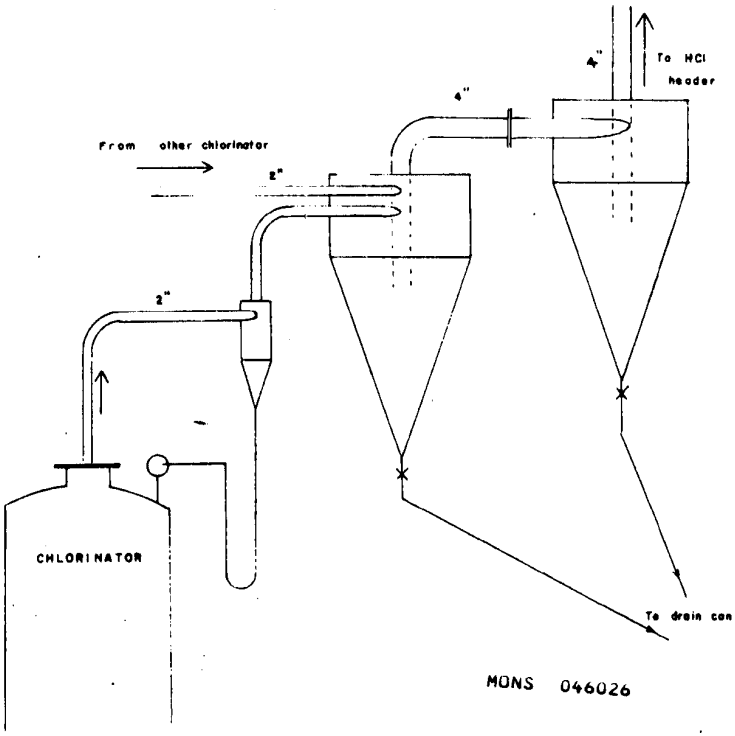
In the early part of the chlorination, the off-gas, which leaves the chlorinator, carries the saturation amount of diphenyl, or light Aroclors, at about 150°C.; as the chlorination proceeds, there is less of these materials in the vapour, but there will be heavier Aroclors, and as the chlorination approaches a high value, free chlorine will begin to come over.

At the Anniston plant, the off-gas handling is simpler than at the Krummrich plant, but it appears to be quite satisfactory. Usually there are two steel cyclones in series for each pair of chlorinators, (see sketch).* The cyclones can be drained at intervals into open-topped drums, and the material recycled.

*page VI-9a.

CYCLONES on the CHLORINATOR OFF-GAS SYSTEM (Aroclors)

Anniston, March, 1955



MONS 046026

Roughly to scale ½" = 1'-0"

VI. PROCESS IN DETAIL, contd.

Scrubbing of the Off-gas from the Chlorinators, contd.

At the Krummrich plant the off-gases are scrubbed with a light Aroclor, at a moderate temperature, to trap the volatilizing materials and any unused chlorine, and to purify the HCl gas.

The scrubbing is done by passing the off-gas up one of the Raschig columns CT-0759, -0760, -0761, 01014, or TW-0206, counter current to a stream of light Aroclor circulated from one of the pump tanks CT-0754, -0755, -0756, 0968, -01013. See the sketch on page 10a Section VI. About a 3/4" stream of liquid is used.

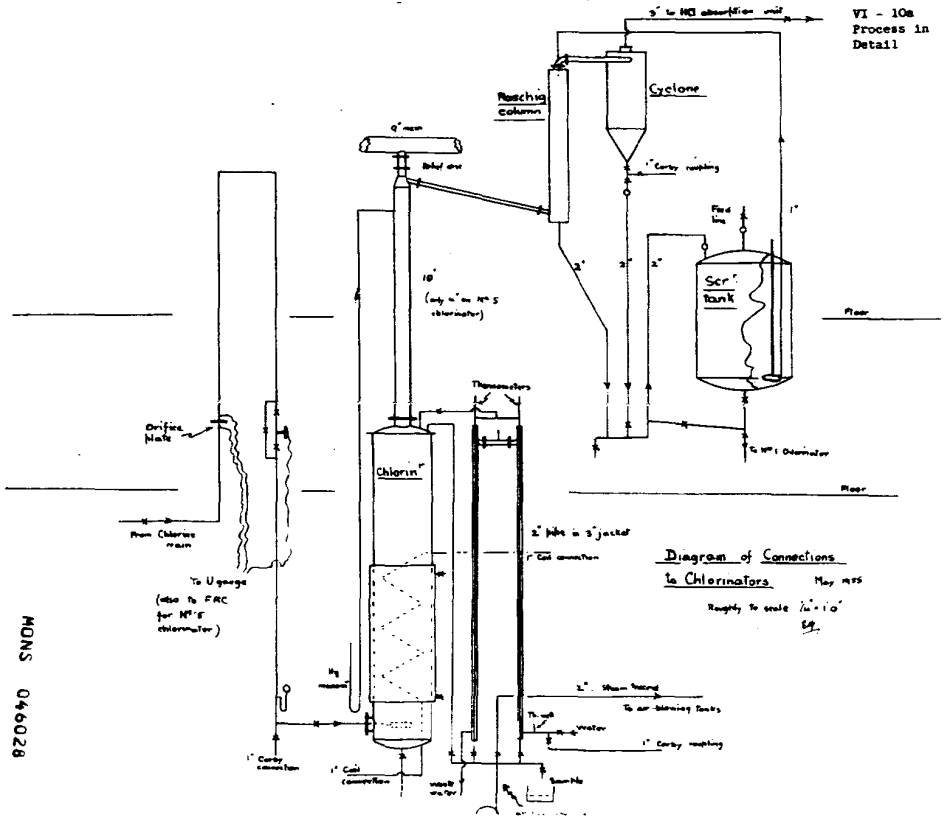
If diphenyl is picked up by the circulating liquid, then the density of the liquid falls, but if heavy Aroclor, or chlorine, is absorbed, the density rises, and the liquid may become too viscous for easy pumping. The density, therefore, is tested at intervals, and if it falls outside the limits 1.200 and 1.400 at 65°C. (more recently the limits have been given as 1.325 - 1.450 at 65°C.), or if the volume increases too much, a note is made of the volume of liquid in the scrubber circulation tank, a sample is taken for specific gravity test, and the liquor is dropped to chlorinator No. 1, and the scrubbing system recharged with fresh crude Aroclor of medium specific gravity. The weight of the discarded liquor is calculated, and the % chlorine content read from the curve of specific gravity vs. chlorine content on page III-15, see also pages VI-48 and 49.

The equivalent amount of diphenyl is calculated(assuming theoretical chlorination), and the charge made up to the equivalent of a normal chlorination batch by the addition of fresh diphenyl. Pages VI-10b,c,d give a ready reckoner for the calculation of the gross weight and the diphenyl equivalent of the liquor.

In recent years at the Krummrich plant the heavier Aroclors have not been made, and the general tendency has been for the scrubber liquor to fall in density during use.

The scrubbing liquor is not heated while in use, unless it becomes too viscous. The gas leaving the scrubber passes through one of the cyclones CT-01279, -01280, -01281 etc. and so into a main leading to the HCl absorbers, which are just outside the south wall of the building. Any liquor collecting in the HCl main drains to a drum outdoors.

VI - 10a
Process in
Detail



VI. PROCESS IN DETAIL, contd.

Dept. 246 Scrubber Liquor Diphenyl (8-13-46)

The table below lists the approximate range of Sp. Gr. of Aroclors that may be found in scrubber liquor. In addition to the Sp. Gr. the equivalent Aroclor as well as the pounds of solution per gallon is shown. The top No* represents the total pounds of liquor and the lower figure represents the equivalent pounds of diphenyl in the scrubber liquor.

Sp. Gr. at 65°C.		1200	1225	1250	1275	1300	1.325	1.350	1.375	1.400
Equivalent Aroclor		1126	1129	1132	1135	1138	1140	1142	1145	1147
Wt.Lb./U.S.Gal.		10.00	10.21	10.42	10.63	10.84	11.05	11.26	11.46	11.67
Scale on Side Sheet	Approx. No. of US Gal.									
Heel	73	730 540	745 529	760 517	776 504	791 490	806 484	822 477	837 460	852 452
1"	83	830 614	847 601	865 588	882 573	900 558	917 550	935 542	951 523	969 513
2	93	930 688	949 673	970 659	988 642	1009 626	1028 616	1048 607	1065 586	1086 574
3	103	1030 762	1051 745	1075 730	1094 711	1118 694	1139 682	1161 672	1179 649	1203 635
4	113	1130 836	1153 817	1180 801	1200 780	1227 762	1250 748	1274 737	1293 712	1320 696
5	123	1230 910	1255 889	1285 872	1306 849	1336 830	1361 814	1387 802	1407 775	1437 757
6	133	1330 984	1357 961	1390 943	1412 918	1445 898	1472 880	1500 867	1521 838	1554 818
7	143	1430 1058	1459 1033	1495 1014	1518 987	1554 966	1583 946	1613 932	1635 901	1671 879
8	153	1530 1132	1561 1105	1600 1085	1624 1056	1663 1034	1694 1012	1726 997	1749 964	1788 940
9	163	1630 1206	1663 1177	1705 1156	1730 1125	1772 1102	1805 1078	1839 1062	1863 1027	1905 1001
10	173	1730 1280	1765 1249	1810 1227	1836 1194	1881 1170	1916 1144	1952 1127	1977 1090	2022 1062
11	183	1830 1354	1867 1321	1915 1298	1942 1263	1990 1238	2027 1210	2065 1192	2091 1153	2139 1123
12	193	1930 1428	1969 1393	2020 1369	2048 1332	2099 1306	2138 1276	2178 1257	2205 1216	2256 1184

* on each panel

VI. PROCESS IN DETAIL, contd.

Dept. 246 Scrubber Liquor Diphenyl, contd. (8-13-46)

13	203	2030 1502	2071 1465	2125 1440	2154 1401	2208 1374	2249 1342	2291 1322	2319 1279	2373 1245
14	213	2130 1576	2173 1537	2230 1511	2260 1470	2317 1442	2360 6408	2404 1387	2433 1342	2490 1306
15	223	2230 1650	2275 1609	2335 1582	2366 1539	2426 1510	2471 1474	2517 1452	2547 1405	2607 1367
16	233	2330 1724	2377 1681	2440 1653	2472 1608	2535 1578	2582 1540	2630 1517	2661 1468	2724 1428
17	243	2430 1798	2479 1753	2545 1724	2578 1677	2644 1646	2693 1606	2743 1582	2775 1531	2841 1489
18	253	2530 1872	2581 1825	2650 1795	2684 1746	2753 1714	2804 1672	2856 1647	2889 1594	2958 1550
19	263	2630 1946	2683 1897	2755 1866	2790 1815	2862 1782	2915 1738	2969 1712	3003 1657	3075 1611
20	273	2730 2020	2785 1969	2860 1937	2896 1884	2971 1850	3026 1804	3082 1777	3117 1720	3192 1672
21	283	2830 2094	2887 2041	2965 2008	3002 1953	3080 1918	3137 1870	3195 1842	3231 1783	3309 1733
22	293	2930 2168	2989 2113	3070 2079	3108 2022	3189 1986	3248 1936	3308 1907	3345 1846	3426 1794
23	303	3030 2242	3091 2185	3175 2150	3214 2091	3298 2054	3359 2002	3421 1972	3459 1909	3543 1855
24	313	3130 2316	3193 2257	3280 2221	3320 2160	3407 2122	3470 2068	3534 2037	3573 1972	3660 1916
25	323	3230 2390	3295 2329	3385 2292	3426 2229	3516 2190	3581 2134	3647 2102	3687 2035	3777 1977
26	333	3330 2464	3397 2401	3490 2363	3532 2298	3625 2258	3692 2200	3760 2167	3801 2098	3894 2038
27	343	3430 2538	3499 2473	3595 2434	3638 2367	3734 2326	3803 2266	3873 2232	3915 2161	4011 2099
28	353	3530 2612	3601 2525	3700 2505	3744 2436	3843 2394	3914 2332	3986 2297	4029 2224	4128 2160
29	363	3630 2686	3703 2597	3805 2576	3850 2505	3952 2462	4025 2398	4099 2362	4143 2287	4245 2221
30	373	3730 2760	3805 2669	3910 2647	3956 2574	4061 2530	4136 2464	4212 2427	4257 2350	4362 2282
31	383	3830 2834	3907 2741	4015 2718	4062 2643	4170 2598	4247 2530	4325 2492	4371 2413	4479 2343

VI. PROCESS IN DETAIL, contd.

Dept.246 Scrubber Liquor Diphenyl (8-13-46), contd.

32	393	3930	4009	4120	4168	4279	4358	4438	4485	4596
		2908	2813	2789	2712	2666	2596	2557	2476	2404
33	403	4030	4111	4225	4274	4388	4469	4551	4599	4713
		2982	2885	2860	2781	2734	2662	2622	2539	2465
34	413	4130	4213	4330	4380	4497	4580	4664	4713	4830
		3056	2957	2931	2850	2802	2728	2687	2602	2526
35	423	4230	4315	4435	4486	4606	4691	4777	4827	4947
		3130	3029	3002	2919	2870	2794	2752	2665	2587
36	433	4330	4417	4540	4592	4715	4802	4890	4941	5064
		3204	3101	3073	2988	2938	2860	2817	2728	2648
37	443	4430	4519	4645	4698	4824	4913	5003	5055	5181
		3278	3173	3144	3057	3006	2926	2882	2791	2709
38	453	4530	4621	4750	4804	4933	5024	5116	5169	5298
		3352	3245	3215	3126	3074	2992	2947	2854	2770
39	463	4630	4723	4855	4910	5042	5135	5229	5283	5415
		3426	3317	3286	3195	3142	3058	3012	2917	2831
40	473	4730	4825	4960	5016	5151	5246	5342	5397	5532
		3500	3389	3357	3264	3210	3124	3077	2980	2892
41	483	4830	4927	5065	5122	5260	5357	5455	5511	5649
		3574	3461	3428	3333	3278	3190	3142	3043	2953
42	493	4930	5029	5170	5228	5369	5468	5568	5625	5766
		3648	3533	3499	3402	3346	3256	3207	3106	3014
43	503	5030	5131	5275	5334	5478	5579	5681	5739	5883
		3722	3605	3570	3471	3414	3322	3272	3169	3065
44	513	5130	5233	5380	5440	5587	5690	5794	5853	6000
		3796	3677	3641	3540	3482	3388	3337	3232	3126
45	523	5230	5335	5485	5546	5696	5801	5907	5967	6117
		3870	3749	3712	3609	3550	3454	3402	3295	3187
46	533	5330	5437	5590	5652	5805	5912	6020	6081	6234
		3944	3821	3783	3678	3618	3520	3467	3358	3248
47	543	5430	5539	5695	5758	5914	6023	6133	6195	6351
		4018	3893	3854	3747	3686	3586	3532	3421	3309
48	553	5530	5641	5800	5864	6023	6134	6246	6309	6468
		4092	3965	3925	3816	3754	3652	3597	3484	3370
49	563	5630	5743	5905	5970	6132	6245	6359	6423	6585
		4166	5037	3996	3885	3822	3718	3662	3547	3431
50	573	5730	5845	6010	6076	6241	6356	6472	6537	6702
		4240	4109	4067	3954	3890	3784	3727	3610	3492
51	583	5830	5947	6115	6182	6350	6467	6585	6651	6819
		4314	4181	4138	4023	3958	3850	3792	3673	3553
52	593	5930	6049	6220	6288	6459	6578	6698	6765	6936
		4388	4253	4209	4092	4026	3916	3857	3736	3614
53	603	6030	6151	6325	6394	6568	6689	6811	6879	7053
		4462	4325	4280	4161	4094	3982	3922	3799	3675

VI. PROCESS IN DETAIL, contd.

HCl absorption.

The Aroclor department at the Krummrich plant had a set of Silica tourills and a Tantalum unit, used, more or less, in parallel. The feed of city water was hand controlled, through a Rotameter, to give 20.2°Bé acid measured at 65°C.

The tourills were cooled by being sprinkled externally with well water, and the Tantalum unit had well water through its jacket.

Gas is sometimes passed to another department, and at other times, gas is received from other departments, for absorption in the Aroclor department units. The operation of the chlorinators has to be regulated to some extent to suit the capacity of the various recovery units, and close cooperation is needed between the different departments.

The tourills on the HCl off-gas line have been taken out, and a second tantalum-Karbate unit, TW-0312, installed alongside the old one. The HCl treatment vessels are of Haveg, and the store tank for finished acid is rubber-lined steel.

About 50 lbs. of Cliffchar carbon are used for 3000 U. S. gallons of acid, agitated by air for half an hour (off-gas to a drowning tower) then left 24 hours, or more, to settle, and the clear acid decanted, by means of a Karbate pump, to finished product storage. The treatment tanks are cleaned out to the sewer about once a month, and the system is working surprisingly well. A project has been set up, however, to install a small clean-up filter for the settled acid. The old Haveg sand filter has been taken out; it had cracked, and the acid-soaked Haveg could not be cemented. The various vessels have Pneumercator depth gauges, with the indicators in a tall glass-fronted cupboard.

Off-gas Treatment at Anniston (March 1955)

At Anniston, the gas streams, from all the cyclones on the chlorinator exits, combine into a header leading to a coke tower 4'0" diameter, 7'6" high. A little organic matter lodges in the tower, but there is no trouble due to the presence of organic matter in the absorption unit which follows. In particular, a rubber-lined store tank for muriatic acid gave good service and was taken out for repairs only after 18 years. The coke tower is outdoors, at the end of a long outdoor header, and the gas is quite cold by the time it arrives at

VI. PROCESS IN DETAIL, contd.

Off-gas Treatment at Anniston, contd.

the tower. The coke in the tower is in about fist-sized lumps, and it is renewed about once a year.

The HCl absorber now in use at Anniston is a Karbate unit, somewhat modified since first installed. It has a vertical tubular Karbate heat exchanger-contactor, followed by a Karbate tail-gas scrubber, packed with 1" porcelain Rasohig rings. The gas goes down the tubular unit, then up the tail-gas unit. The water enters the top of the tail-gas unit, then flows through a seal bend to the top of the tubular unit, down the unit, concurrently with the gas, and so to the catch tank.

There is a Brown ratio controller, which automatically proportions the flow of feed water to the flow of HCl. It is manually pre-set to give about 20.8°Bé, which leaves a little in hand, above the strength desired.

When the absorption unit was first installed, it had provision for recycling the solution through the heat-exchanger section, so that the tubes would still be properly wetted when the flow of gas, (and consequently the feed of fresh water), was very low. The unit was designed for 2500 lbs. HCl per hour, and below about one quarter load, the wetting was inefficient without recycling. However, when the recycling pump failed, it was not replaced, though the exit of the unit smokes somewhat at low loads.

Some of the muriatic acid made is sold without further treatment, but some is treated with carbon, to give the "food grade" quality. For this treatment there is a rubber-lined pre-coat tank, a rubber lined treatment tank, an Adams filter with 18 tubes, a Karbate Wilfley pump, and various rubber-lined store tanks. Saran lining has not been so satisfactory. 10 to 15 pounds of Celite are suspended in muriatic acid (or in water) in the pre-coat tank, pumped on to the tubes in the filter, then the carbon-treated acid is pumped in without interrupting the flow, so that the Celite does not drop off the tubes.

Any acid produced in excess of requirements is put, untreated, to the sewer, by way of a limestone pit, which also takes the spent carbon.

VI. PROCESS IN DETAIL, contd.

Changing from One Aroclor to Another.

The crude Aroclors fall into four main groups: --

1. Liquid Aroclors from Diphenyl (1142 to 1162)

2. Solid Aroclors from Diphenyl (1168, and formerly 1169 to 1171)

3. Aroclor from crude or distilled high boilers (5060)

4. Aroclors from mixtures of diphenyl and highboilers, (2265 and 4065), and in addition the material from the scrubbers on the off-gas has to be worked up through the chlorinators. It may contain some products derived from highboilers.

In changing to 5060 from any of the other Aroclors, it is necessary to give the chlorinator two (or more) successive washes with distilled high boilers, using 2,000 lbs. each time, and circulating the(hot) material for 10 - 12 hours each time to give thorough washing of the catalyst. The first washing can be re-used several times, then used for making 2265 or 4065. The second washing, if its density indicates less than 0.3% chlorine content, can be used for making 5060. Washing should be continued down to the 0.3% chlorine level. Rinsing is not thought necessary at other changes.

See also the comments on page VI-25.

Air-blowing of Chlorinator Charges.

At the Krummrich plant, the crude Aroclors are air-bleem for 4 hours in CT-0750 or CT-02086, both of which are vertical cylindrical steel vessels with dish bottom and flanged covers. They have steam coils for maintaining the temperature of the charge, by hand control, and "spargers" for dry air from the "Lectro-dryer." The off-gases are simply vented by wide Haveg stacks to the open air above the roof of the building. At Anniston there are 3 air-blowing vessels, and the vent stacks are off-set from the air-blowing vessels in two cases,so that nothing can drip back from them into the charges.

The blowing vessel at the Krummrich plant is large enough to contain 3 chlorinator batches at one time, and it may be necessary to put

VI. PROCESS IN DETAIL, contd.

Air-blowing of Chlorinator charges, contd.

unblown charges on top of blown material, in which case the air-blowing must be repeated. The steam coils are not used during the air-blowing of Aroclors up to and including 1148, but the higher Aroclors need temperatures of 140°C., or more, depending on their softening points. 80 psig steam is used in the coils for 2565 and 4065, but 200 psig was needed for 1168 and 5060.

A sufficient flow of air is used to cause the contents of the blowing tank to "roll". A flickering of the needle of the pressure gauge on the air-inlet line will indicate that air is bubbling into the vessel.

Aroclor 2565 is not distilled, but is sampled in the air-blowing vessel, checked for acid number and softening point, and if correct, is dropped directly into cans for sale.

Distillation

There are two vacuum stills for Aroclors at the Krummrich plant, both being vertical steel cylinders 6'0" I.D., with bulged tops and bottoms, as detailed in section XII below. S-043 is 5'6" deep on the straight side, and S-062 is 11'-10".

Each still has the following fittings: --

1. An external heating coil, set in a gas-fired furnace.

2. A submerged pump to circulate the charge through the coil and back to the still. The pump shaft has a water-cooled stuffing box, and there is an indicating ammeter on the driving motor.

3. An inlet with sight-feed coupling, for charges from the air-blowing vessels CT-0750 and CT-02086.

4. An inlet from the chlorinators, normally blanked off.

5. A hand hole with a screw cap, for charging hydrated lime.

VI. PROCESS IN DETAIL, contd.

Distillation, contd.

6. (Formerly) Emergency lines to and from Aroclor store vessel CT-0749.

7. A Manlid low down on the side wall, for maintenance work.

8. A thermo-recorder well through the side wall, about 7' below the rim of S-062 (disused).

9. A bottom outlet which will drain the still.

10. An outlet which will leave in a "heel" of about 80 U.S. gallons, just filling the bottom dish of the still. These outlets are equipped for gas heating.

11. A vapour outlet, to one of the condensers CT-0770 and 01378. The vapour duct has a dial vacuum gauge, a mercury manometer, and a thermo-recorder well. Each duct has a spray separator (different in each case as detailed in Section XII below.)

The condensing systems have only monel exposed on the descending sides. At the Krummrich plant, the condenser is cooled by distilled water, which is allowed to boil off into heat exchangers CT-083 and 01491; the condensate from the exchangers returning to the Aroclor condensersby way of one of the buffer tanks CT-0766 and -01396. Wastage of the distilled water is made up by bleeding steam into the system at a convenient time. Formerly, for the higher boiling Aroclors the distilled water was replaced by Aroclor 1242, circulated through the same system by pump P-0595. Each buffer tank has an internal coil for water and steam. The whole system, whether used in water or in Aroclor is at atmospheric pressure, though it was designed to be worked, if necessary, as a closed system under pressure.

The Aroclor circulation system was out of use in 1955, because higher Aroclors were no longer being made at the Krummrich plant.

The Aroclor condensers lead to the vacuum receivers CT-0771, -01377, which have the usual fittings, including a connection to the 3-stage vacuum jets MSC-0760, -01392, -01685 by way of the coke scrubbers CT-0753 and -01396.

VI. PROCESS IN DETAIL, contd.

Distillation, contd.

Although still S-062 at the Krummrich plant is about twice as deep as S-043, it is given only the same charge (about 10,000 pounds) of crude Aroclor. The greater depth of S-062 was designed only to give more disengagement space for the vapour. See the comments on this feature under item S-043 in Section XII below. The charge is sucked to either still, or is pumped by P-0580 from the air-blowing vessel CT-0750, then two cocks on the transfer line are closed, to prevent leakage of crude material into the still during the distillation.

The internal pump in the still is started, if it is not already running, and 50 lbs. of lime, (formerly 30 lbs.), hoisted in bags to the operating platform, are added through the charging nozzle on the cover of the still. The amount of lime may be increased, up to 100 lbs., if the crude Aroclor is believed to contain an unusually large amount of HCl.

The vent on the still is closed, and the vacuum jet started to reduce the pressure in the still to 7 mm (or less) Hg. absolute pressure.

The pilot lights, and a low flame, are usually kept going in the gas furnace between batches, and when the charge is seen to be circulating correctly, the main burners are adjusted to give a temperature rise of about 20°C. in the material as it passes through the heating coil. The operator has a table of gas pressures at the burners, corresponding to different operating requirements, but he must also use judgment in controlling the distillation. The higher Aroclors require considerably more gas. Cooling of the condenser is not started until the distillation has well commenced, because over-cooling of the first distillate might cause blockage with frozen, or very viscous, material. Judgment is required throughout the distillation, to give sufficient cooling without cooling too far, and the rate of distillation is limited so that dark material is not entrained into the distillate. The whole distillate goes into one receiver, no cuts being attempted unless the distillate is seen to be off colour for a time. Typical record charts are given at the end of this section.

VI: PROCESS IN DETAIL, contd.

Distillation, contd.

The end of the distillation is signalled by a quicker rise in temperature of the material (at a steady pressure, around 6 mm Hg. absolute), and by a greater temperature "spread" between the coil inlet and outlet, or between the coil inlet and the vapour outlet from the still; about a 40°C. spread through the coil is taken as an indication to stop. Any darkening of the distillate stream would also be an indication.

Mr. Pemberton, reporting on his visit to Anniston in 1950, mentioned a colour standard, which the operator may use to check the colours of samples of distillate drawn from the sampler between the condenser and the receiver.

Distillation should in any case be stopped before the volume of bottoms has dropped to a point at which the internal pump will be "starved". This is liable to happen with the first batch in a clean still. If the bottoms become too viscous, the pump will "kick out", and the furnace coil will become overheated, and either damaged, or at least blocked with coke. If the ammeter indicates overloading, some light Aroclor should be put into the still to prevent trouble.

The internal pump is left running, but the gas flames are cut down to a point just sufficient to keep the "heel" of residue hot enough to circulate freely without coking in the coil; while there is any heel in the still, the internal pump must be kept running (there is an alarm which sounds if the pump shaft stops), and the gas burners kept on their low setting. Mismanagement at this stage may cause a freeze-up of the whole system, or may cause coking in the coil. It is important, also, so to arrange the work, that there will be a fresh charge waiting to go in on top of the heel, so that the residue in the still is immediately diluted with more mobile material.

After about four batches, more or less, depending on circumstances, the bottoms are drained off through the upper drain opening, leaving a heel of about 80 U.S. gallons in the still. To stop the distillation, a little air is admitted to the still.

VI. PROCESS IN DETAIL, contd.

Distillation, contd.

The outlet cock is heated, in good time, with a gas flame, a little air sucked in through the cock to make sure the way is clear, then air is let into the system by way of the vent cock on the receiver, and the residue is run out into open-topped cans. The residue is very hot, and gives off an irritating vapour, so the cans are placed on small wheeled trucks, under a canopy with a good exhaust system. Some little of the residue is run into packages for sale as Montar, but the greater amount is allowed to go solid in the open-topped cans, then broken out, and thrown away. If the material is not to be sold, the cooling may be hastened in some cases by using a fine mist of water from a distance of about 15 feet, to cause the upper layer of the material to freeze. Some discretion, however, is needed in doing this, because if the water sinks into the hot material, it will vaporise explosively. For this reason, the water spray is not used for residues from 1242, or 1248, which have a lower density.

It is not even used for residues from 1254, 1260 or 1262, unless the batch has been well worked down in the still (temperature spread in the coil at least 35°C.). If water is not used, the drums are lightly covered during cooling.

As already mentioned, the still should be recharged without delay, to thin out the residue left in it. While the new charge is circulating, but the furnace burners are on the idle setting, the thermo couples at the inlet and outlet of the coil should show the same temperature -- this checks them one against the other, so that the temperature "spreads" observed during the distillation will be dependable even if the readings are not correct in an absolute sense.

If the still is completely drained, through the lower discharge cock, so that the pump circulation must stop, the heating coil must be sucked empty. To facilitate this, two cocks have been installed on the highest part of the circuit with a 1" branch with cock and screw cap between them. With this arrangement, air can be pulled into the still in either direction along the pump circuit.

VI. PROCESS IN DETAIL, contd.

Distillation, contd.

The shallower still, S-043, is being worked semi-continuously at the Krummrich plant, but there is very little to add to the description of continuous working, as given in my report of March 29, 1955* on the Anniston visit. The material is continuously moved from the air-blowing vessel to the still by the vacuum in the still. A sight glass has been necessary in the cover of the still receiver. A feature worthy of note is that the intake of each of the submerged pumps in the stills has been fitted with a shallow basket, about 10" diameter, 2½" deep, of steel plate, with ½" round perforations set nearly as closely as possible over the whole surface. The purpose of this is to trap stones etc. which might get in with the lime. The pumps are still of the 2" delivery size, and the furnace coils are of 2" pipe. It is doubted if the Anniston 3" coils offer more surface, because, of course, the turns of the 2" coils can be at closer pitch. The furnace setting with a central steel dummy would appear to be better than that with the central fire-brick dummy, because it will retain less heat. The gas burners are interlocked, to shut off if the pump shaft stops, but it is suggested that a more rational arrangement would be to have the stoppage worked from an orifice plate, in the circulation line, and so set as to cut off the gas as soon as the flow fell below a certain level. This could be better since it would shut down the gas if, for example, the circulation line should become partly blocked.

The stills at the Krummrich plant are worked at the highest available vacuum. A small operating trick is mentioned. As a still batch approaches the end, as judged by the temperature "spread" between the inlet and outlet of the coil, the burners should not be cut back until the end point is reached. If they are, then the temperature spread falls correspondingly, and there is risk of working the bottoms too far, in an attempt to attain this specified spread, and the bottoms may become too thick to tap easily. The last receiver-full, when the bottoms of a continuous run are being worked down, will be rather heavier Aroclor, and will need blending off into the bulk of distillate.

* See also pages VI-20- of the present report.

VI. PROCESS IN DETAIL, contd.

Distillation, contd.

At Anniston there are two vacuum stills, each of the shallower type, 6'0" in diameter, 5'-6" deep on the straight side, like S-043 at the Krummrich plant, and the heating and condensing arrangements are generally similar to those at the Krummrich plant (vertical monel condensers, cooled only by recycled distilled water).

The submerged pumps now used in the stills are Taber pumps with 3" outlets, and the furnace coils are of 3" piping, nine turns at 5" pitch, 24" diameter, to centres. Each coil stands on four short legs on the bottom of the furnace, and there are short steel distance pieces welded between the turns of coil at four points on the circumference (see sketch).* Each furnace is brick lined and has a central hollow cylindrical steel core to confine the flue gases. Stainless steel gives better service as the core. Each furnace has four burners for natural gas, directed horizontally and tangentially, just below the lowest turn of coil, the four burners being equally spaced around the circumference. The furnace is set as close as is possible to the still body, without hindering removal of the still cover; the stream of Aroclor from the submerged pump in the still goes by a short horizontal pipe to the bottom of the coil, upwards through the coil, then back to the still, by a pipe projecting just through the cover. The lowest turn of the coil is the only part which burns out.

The bearings on the shafts of the submerged pumps gave a lot of trouble, and various changes were tried. Finally the master mechanic suggested trying thinner shafts ($1\frac{1}{4}$"), and these give trouble-free service for a year, or so, before the bearings need attention.

The bearings are lubricated by Aroclor fed back from the pump stream, (see sketch).

Note Newport's use of a universal coupling on the pump shaft.

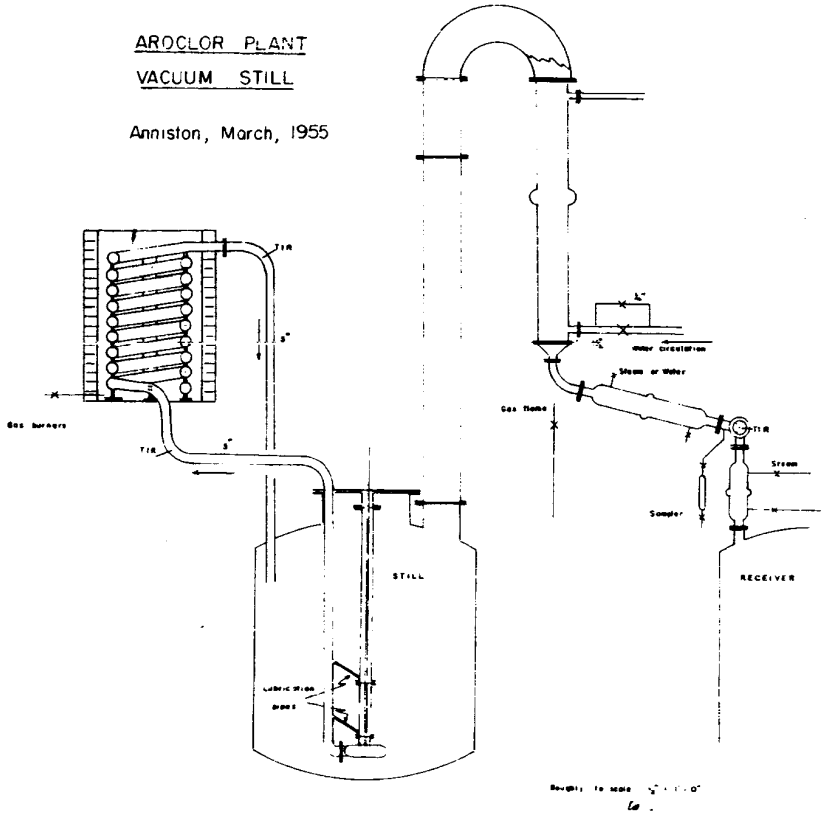
From the still cover, the vapour rises about 12'0" in 12" pipe, then turns downwards through a tubular condenser, 7'0" long, by about 10" O.D., set vertically. From the bottom of the condenser, the distillate goes by a sloping jacketed line to a sight glass. (see sketch).*

* page VI - 20a.

VI - 20a
Process in
Detail

AROCLOR PLANT
VACUUM STILL

Anniston, March, 1955



MONS 046042

VI. PROCESS IN DETAIL, contd.

Distillation, contd.

The 12' pipe from the still cover is of steel, but beyond that point only monel is used.

The cooling water for the condenser is distilled water coming from a vented head tank high in the building. The water enters the bottom of the condenser shell by a 1½" valve bye-passed by a ¼" line having a ¼" valve for fine control. The water, (and/or steam), leaving the top of the shell, returns to the head tank by way of a heat exchanger which can cool or heat the return stream, as required. Any make-up water required for the circulating stream, is supplied by bleeding steam into the stream just before the heat exchanger.

The water circulation system had been planned to work under pressure, with a pop valve on the head tank to relieve excess pressure, but it has been found more convenient to run it under atmospheric pressure. Even so, some skill is required to get proper condensation of the Aroclor without cooling so far as to freeze the heavier Aroclors in the condensers.

Each of the two vacuum still receivers at the Anniston plant has a sight glass in the cover, through which the operator, by using a flash lamp, can see the level of liquid. The condensate can be run off to drums under a draught hood, or it may be run to a Blackmer pump, on the ground floor, about 11'0" below the bottom of the receiver. This pump moves the distillate to the treatment tank, or elsewhere as required, and it will work, though not very well, with the receiver under full vacuum. The pump and connections are steam traced, and the same pump serves the receivers of both stills.

By using this pump without stopping the still, it is possible to work the still in a semi-continuous manner, instead of purely batch-wise, so a level controller has been fitted through the cover of each still, and the crude Aroclor is fed, by steam jacketed lines, continuously into the still, keeping the level in the still at about 55" outage. The level controller is of the differential bubbling type. The arrangement is working very well, and distillation can

VI. PROCESS IN DETAIL, contd.

Distillation, contd.

be continued until the equivalent of up to 13 normal batches has been distilled, before the accumulation of bottoms begins to cause darkening of the distillate stream.

The feed is then stopped, and the bottoms distilled down until the distillate stream becomes too dark for blending, then the still is tapped to open-ended drums under a draught canopy.

The open-ended drums, into which the still bottoms are tapped, are placed on the ground floor, under a steel canopy, about 8'0" diameter, coming down to about 3'0" from the ground, and connected to a powerful exhaust fan. There is a canopy under each of the two stills, and smaller canopies at the various drum-filling points.

Naturally, it is not always possible to continue for the equivalent of 13 batches, because the manufacturing programme may have to be changed before that. The semi-continuous working saves time and labour in tapping and recharging, and it probably permits better recovery of distillate from the bottoms.

The dosage of lime, per equivalent still batch, varies with the grade of Aroclor being worked. Aroclors 1242, 1248, 1250 and 1260 take 30 lbs. per "batch", and the same amount is probably right for 1262 (not recently worked at Anniston). 4465 takes 50 lbs. and so does 5442 (rarely worked). 5460 takes 100 lbs., this high amount being found to improve the colour.

If semi-continuous working is planned, lime is added in ratio to the total amount of Aroclor it is intended to work, the whole of the lime being added at the start. Thus for 13 batch equivalents of 1260, the amount would be 390 lbs. corresponding to 13 batches at 30 lbs. each.

At one time it was thought best to run the stills at as good a vacuum as could be obtained with the four-stage jet, but at

VI. PROCESS IN DETAIL, contd.

Distillation, contd.

Anniston it has been found better, and more economical in steam, to use only a two-stage jet for some of the Aroclors, for example: --

Aroclor	m.m. Hg in the receivers recommended
1260	30
1250	30
1242	30
1248 (probably)	30
1268	3 - 5
4465	3 - 5
5442	3 - 5
5460	3 - 5

Compare the figures reported April 1952 by Mr. Harden for Anniston 20 m. m. in the receivers for liquid Aroclors, and 10 m. m. for 5460.

The temperature "spread" between the inlet and outlet of the heating coil, of course, determines the heat input to the still, for any given rate of pumping. Under the present conditions at Anniston, the spread is about 8 to 12°C., widening towards the end of a run. For the continuous working, the spread usually runs between 8 and 10°C.

VI. PROCESS IN DETAIL, contd.

Distillation of Solid Aroclors.

See also Lyles, Soffranko and Becker, November 1947, pages 61-and 97..

In spite of its high melting point, when Aroclor 1268 was made, the distillation was done in the ordinary vacuum stills, as with the lower Aroclors, but with greater precautions against freezing in the pipe lines etc.

1169, 1170 and 1171, as formerly made, were kept hot in the gas fired chlorinator CT-0808, and drawn off in portions into retort pots R-026. and 027. The pots were placed in turn, on a rail cart, on a weighing scale, under the bottom outlet of the chlorinator, the outlet line heated with a gas flame, 30 lbs. of lime put in the pot, and about 500 lbs. (600 lbs. in 1947) of the hot Aroclor added, a (vented) lid clamped on with C clamps, then the pot moved to a spot under a crane for hoisting into position on one of the two furnaces. A thermocouple was inserted through the cover of the retort. Each retort pot was equipped with a channel about half-way up the inside wall, to collect Aroclor, which distilled up on to the upper part of the wall. A pipe connection from this channel, into the pan of the flaker D-012, was made after the pot was in position.

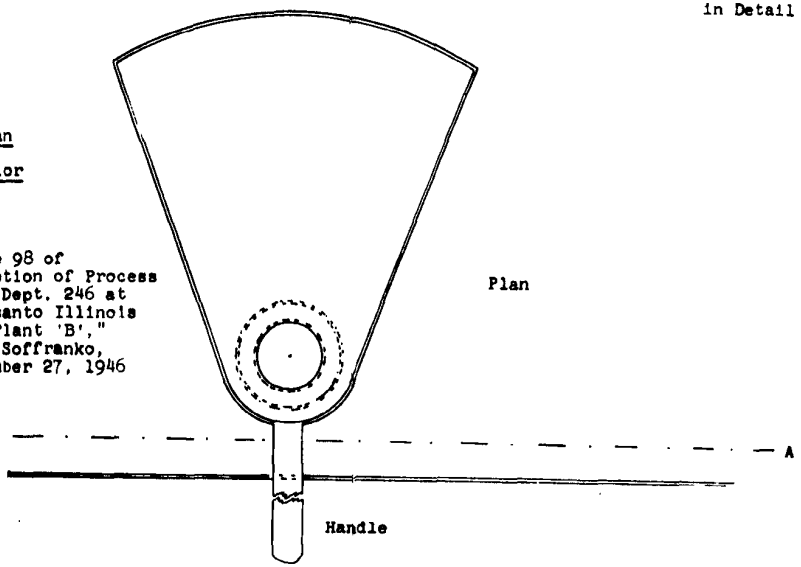
The pots were gas heated, a pre-run cut being diverted to a drum, * then the main cut taken to the flaker pan, then a finishing cut, to the drum again (judged on colour). The tops and tails were recycled partly to the succeeding batch in the still, and partly back to the chlorinator, and the main cut was flaked, the flaking roll being warmed at first with low pressure steam, but later as the system warmed up, the drum was cooled with water. The flaked material was moved by a conveyor and an elevator L-031 to a finished product hopper. The adjustment of the knife had to be varied as the temperature of the roll altered, and the flaker tray was heated by gas flames.

The end of the distillation was indicated by the evolution of a little fume caused by decomposition. The whole system was served by an exhaust fan with cyclone separators. The retorts were removed to a concrete slab, sprayed with water to cool them, then they were cleaned for re-use.

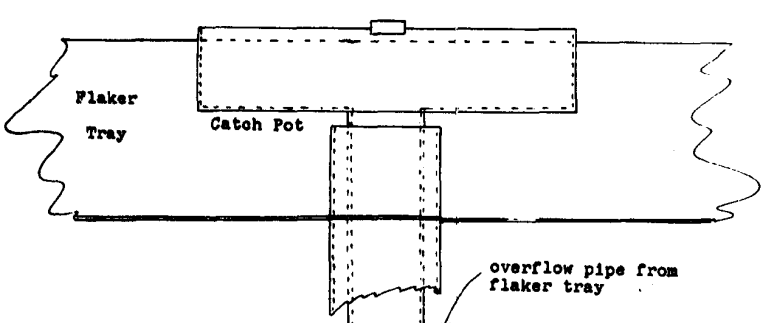
*see sketch, page VI-24a.

Catch Pan
for Aroclor
Flaker

See page 98 of
"Description of Process
used in Dept. 246 at
the Monsanto Illinois
Plant, Plant 'B'."
Lyles & Soffranko,
September 27, 1946



Section A-A



VI. PROCESS IN DETAIL, contd.

Distillation of Solid Aroclors, contd.

There were spare retorts, so that the residues could be cleaned out without delaying the operation.

The finished product in the hopper would be mixed by recycling it round the elevator, then sampled for the control lab. When approved, it would be run off to barrels for sale, or for transfer to the mill ML-045, by means of an electric hoist. The mill required a steady feed of "dry-ice" to keep the product cold enough to be brittle and not sticky.

A white operator, with a coloured operator to clean the pots, could distill 5 pot fulls in an 8-hour shift.

Change from one Aroclor to another.

See also page VI-13.

In changing from 1268 to any of the other Aroclors, it is necessary to drain the still and the receivers, then to put in a 2000 lb. charge of one of the lower Aroclors, and distill it through to clear the system. The distillate is retained for chlorination to 1168, and the still is drained again to drums, inspected, and, if necessary cleaned by hand.

Apart from this, at the Krummrich plant, the heel now is usually left in the still, even if a different Aroclor is to be distilled, the first two chlorinations being carried just a little further, if a heavy Aroclor is to be charged on the bottoms from a light Aroclor, and vice versa, as set out in the following table on page VI-26.

In batch working, Anniston prefer to work the still down to a very low level, and drain off the bottoms, whenever a change is to be made to a different Aroclor, and in any case, to drain the still after every 5th run of 1254 or 1260, and after 2nd run of 5460. Before a long stoppage, the still and coil are rinsed through with about 50 gallons of 1154, mainly to clean the coil.

Beyond this, no rinsing of the still is ordinarily considered necessary at either plant, and there is no cleaning of the air-blowing tank.

VI. PROCESS IN DETAIL, contd.

Changing from One Aroclor to Another, contd.

AROCLOR TO BE MADE:	1142	1148	1154	1160	1162
Heel remaining from	Specific Gravity to be attained				
1142	usual	+ 0.003	+ 0.002	+ 0.003	+0.005
1148	usual	usual	+ 0.001	+ 0.001	+0.003
1154	usual	- 0.001	usual	usual	+0.001
1160	-0.001	- 0.003	- 0.001	usual	usual
1162	-0.003	-0.005	-0.003	- 0.003	usual
Temperature at which Sp. Gr. is measured	65°C.	65°C.	65°C.	90°C.	100°C.

A + sign means that the chlorination should be carried a little further than usual, and

A - sign means that the chlorination should be stopped a little short, for example

+ 0.005 above means that the 1162 should be finished at 1.574 to 1.576 instead of at 1.570 as given on page VI-5, below.

and - 0.005 means that the 1148 should be finished at 1.406 to 1.408, instead of at 1.410 to 1.414.

VI. PROCESS IN DETAIL, contd.

Treatment of the Distilled Aroclors.

From the still receivers at the Krummrich plant, the Aroclors up to and including 1262 are moved by pumps P-0565 and -01037, to the blending tanks CT-0779 and 01399 for treatment with Attapulgus earth, mainly to remove traces of HCl. If the distillate contains moisture, it is warmed and air-blown to remove the moisture, before the earth is added. The Attapulgus earth is dried before use, in a vertical steam heated vessel with a cone bottom. (Sketch sent to MCL in 1947). This dryer does not carry the moisture content down to a very low level, and supplies of dried earth are sometimes drawn from Anniston, where the drying is done in an electrically heated tray dryer. The normal dosage of earth is 10 pounds for the distillate from 2 distillation batches, each of 10,000 pounds of crude Aroclor, but the amount of earth may be increased if found necessary to lower the chloride test on the distilled Aroclor. The earth is added, by hand, from a bucket or scoop.

The Aroclor and the earth are agitated together for 4 hours, the temperature being raised to 150°C. at which temperature the filtering characteristics of the Aroclor are good. The steam tracing on the lines and the steam jacket on the filter are put in action, and the batch is recycled through filter F-0238 (formerly the smaller filter F-0172) until clear, (about ½ hour), then the stream of the filtrate is turned to one of the filtrate receivers CT-0780 and -01400, a half gallon sample of filtrate being taken in a hot dry sample jar for the control laboratory. Filtration of a batch takes about 5 hours. A sample of the filter paper was sent to MCL by Mr. Harden, August 1950.

In May 16, 1955 it was noted that a 30" Sparkler filter, F-0238, (steam jacketed) had been installed for use on Aroclors, and the 18" one, F-0172 moved for use on Pyranols. A small "Fulflow" filter was being installed for use on the toluene-Aroclor blends. These blends are made up in the Aroclor finished product vessels CT-0780 or CT-01400, rather than in the Pyranol mixers, see page VI-29. The Krummrich plant were depending on supplies of thoroughly dried Attapulgus Earth from the Anniston electrically heated oven.

VI. PROCESS IN DETAIL, contd.

Treatment of the Distilled Aroclors, contd.

It does not appear necessary to empty the earth from the filters after each batch. If satisfactory on all ordinary tests, not including special G. E. approval, the filtered Aroclor may be moved to finished product containers, or to store tanks CT-0768, 0769, 01310. If G. E. approval is required, a fresh sample is taken from the bulked material in the store tank.

At Anniston a Sparkler filter 20" diameter, has been installed, for the earth treatment of the Aroclors, but it has turned out to be less convenient to clean than the old plate and frame press, so the old press is mostly used. It has bronze plates and frames, 23 frames, 12" square, with open delivery to a trough which leads back to the treatment tank or forward to the filtrate tank, as required. The press is set, under a draught hood, in a drip tray which drains back to the treatment tank. The feed lines are steam jacketed. Lines for the finished Aroclor are mostly of galvanized steel.

The filter papers are dried, (not above 85°C.), before use, in an oven, and there is one compartment in the oven for storing the dipper and funnel used for sampling Aroclors, also the sample bottles.

The sample bottles are used, just as delivered by the maker, simply rinsed out 3 times with the material being sampled, then blown out with dry air or nitrogen, then filled with the product to be sampled. Aroclor stored in sunlight in a colourless glass bottle will drop in resistivity, and will begin to smell of HCl. Amber glass bottles are better. See page VII-14.

At Newport a "Steller" candle filter was provided for the earth treatment of the Aroclors, and it appears to have been satisfactory. There is, however, a record of leakage from the jacket, where an air pipe passes through into the interior. This suggests that steam tracing, as formerly used at the Krummrich plant, may have advantages over a jacket.

The bronze dome of the Steller press was replaced by a galvanized steel one, to meet insurance requirements.

VI. PROCESS IN DETAIL, contd.

Treatment of the Distilled Aroclors, contd.

Aroclors 4465 and 5465 are sampled from the still receivers. When the end of the distillation of these products appears to have been reached, the gas flames are cut down, and the contents of the still receiver are "roused" by admitting air through the bottom inlet of the receiver, then the vacuum is released and a large sample of distillate drawn off into a drum. The softening point is checked, and if it is too low, the distillation is recommenced to drive over more of the heavier back ends. When the contents of the receiver show the correct softening point, the product is dropped directly into packages for sale, a sample for the control laboratory being taken during the filling off.

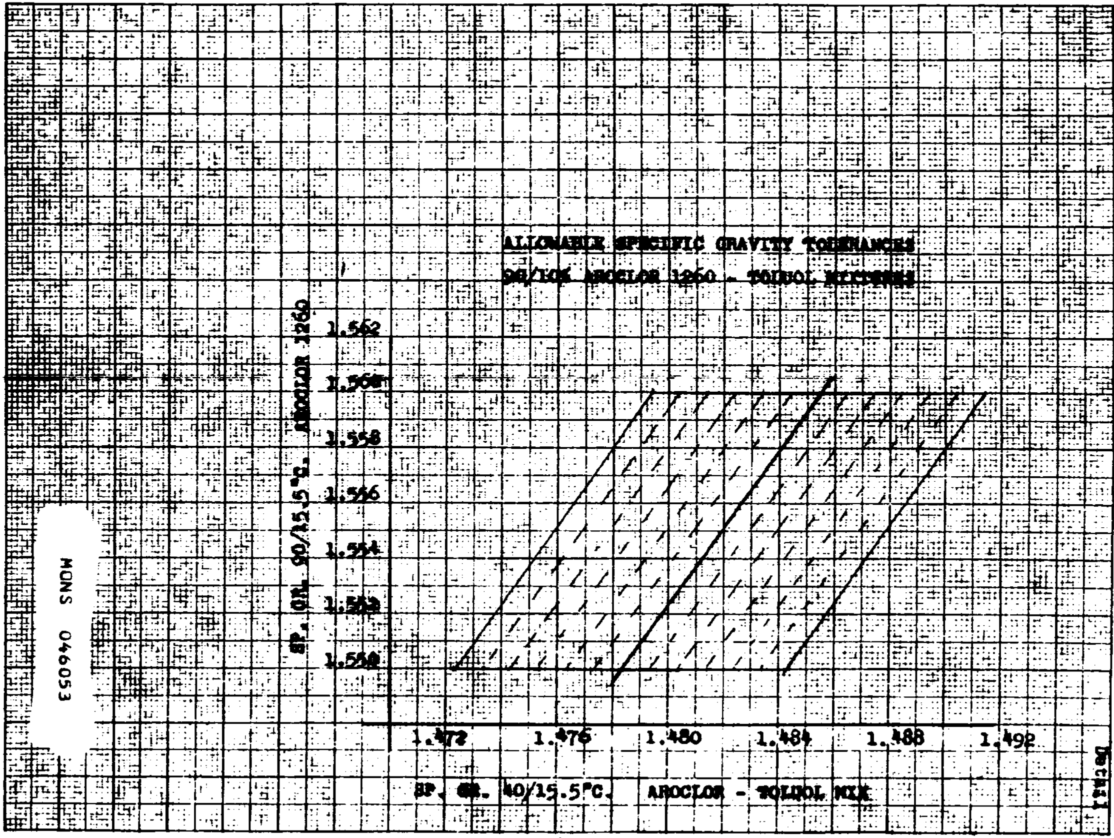
Blending of Aroclor "1260 mix" or "1262 mix".

These are blends of 90% by weight of the Aroclor with 10% by weight of toluene. The blending is done either in finished product drums or in one of the blenders CT-0780 or -01400, so for the time that these blending operations are in hand, the use of flames etc. in the building has to be considerably restricted. Earth wires are attached to the toluene drums, and the nearer of the two still furnaces is shut down.

When the blending is completed, the air of the building is tested for inflammable vapour before normal working is resumed. (See page 6, Section XI, for a note on the Davis Vapotester). The blend is sampled for a determination of specific gravity, and the composition adjusted, if necessary, as indicated by the following charts, pages VI-30 and VI-31. See also pages IX-89, 90 and 90a.

The mixing tank needs to be steamed out before it can be used again for ordinary Aroclors.

See also MCC report 171-4013, "Aroclor 5460 - Toluene mixture".



ALLOWABLE SPECIFIC GRAVITY TOLERANCES
90/10% AROCLOR 1260 - TOLUOL MIXTURES
SP. GR. 90/15.5°C. AROCLOR 1260
1.562
1.560
1.558
1.556
1.554
1.552
1.550
1.472
1.476
1.480
1.484
1.488
1.492
SP. GR. 40/15.5°C. AROCLOR - TOLUOL MIX

VI. PROCESS IN DETAIL, contd.

Blending for Pyranols.

The compositions of the different Pyranols (Pyroclors, Inerteens, Askarels) are given on page 4, Section I, above. Pyranol 1467 is the main product, 1481 is blended only in relatively small batches (2100 U. S. gallons).

The main components are Aroclor 1254 or 1260, and trichloro benzene, or a tri-tetra chlorobenzene mixture. The Aroclors used are made by MCC, and so is most of the trichlorobenzene, but the tri-tetra mixture is bought from the Hooker Chemical Company.

The "chloride scavengers", tin-tetraphenyl and glycidyl phenyl either, are purchased, but the tin-tetraphenyl may have to be purified by MCC, as described on page 4, Section IX, below.

Before a blend is made, great care is taken that all the components are of the correct quality, especially in respect of the inorganic chlorides and corrosion tests discussed in Sections VII and IX, below.

Bulk quantities of the Aroclor and the chlorobenzene concerned are collected, sampled and put through all the routine tests set out in section IX, below. Excess moisture may be removed, down to about 10 to 15 p.p.m., by passing dry air through these materials at about 70 to 80°C. (or even through the materials after blending).

The Aroclor may be treated again with Attapulgus Earth to lower the chloride test figure.

Air-blowing after blending may disturb the composition of the blend unless the temperature is kept down, because the chlorobenzenes are more volatile than the Aroclors. When satisfactory tests have been obtained, the raw material may be sampled for submission to the customer (General Electric Company), as an additional precaution, before blending is done.

When approval is complete, the Aroclor and the chlorobenzene components are blended, and a sample of the blend is checked for density and refractive index, and small adjustments made if necessary in the

VI. PROCESS IN DETAIL, contd.

Blending for Pyranols, contd.

proportion of Aroclor to tri- or tri-tetra-chlorbenzene. Chloride scavenger is not added at this stage, but when all tests are known to be satisfactory, a portion of the blend is made up into a pilot batch, using chloride scavenger from the batch which is to be used for the main blend, full tests are made on the pilot batch, and if everything is correct, the main batch is treated with the scavenger, agitated 4 hours, circulated through the filter press for 1/4 hour, and sampled for the final test, an extra sample being taken, if necessary, for submission to the customers. For blending with tin-tetraphenyl, it is usual to agitate the batch at about 50°C. to make sure the TTP goes into solution. When the bulk is known to be satisfactory, it is filtered into the drums, or rail tanks, for sale, a final sample being taken. In some cases the consignment is held pending the customer's acceptance of the sample.

In all this work, care must be taken to exclude moist air, HCl fumes and dirt. The filter paper used is catalog 73 and 852, size 12, for the General Electric press type FP-30. It is said to remove traces of moisture and inorganic chlorides, even without the help of Attapulgus Earth.

All possible care should be taken to have the batch correct before any scavenger is added, and to check the scavenger itself, because any further treatment with Attapulgus earth, which might be necessary, after addition of the scavenger, will remove more or less of the scavenger. The presence of scavenger also complicates the "corrosion" test as discussed in Section VII, pages 12-, and Section IX, page 181.

Traces of Attapulgus earth which have carried through to the finished product amy interfere with the test for inorganic chlorides, see page IX-181.

VI. PROCESS IN DETAIL, contd.

Packing

The finished Aroclors are run off into drums, a piece of diaper cloth being tied on the pipe outlet for the lighter Aroclors, and a piece of fine gauze for the heavier Aroclors, which have to be handled hot.

This, of course, is to arrest stray bits of pipe scale etc., but the precaution is now omitted at the Krummrich plant when materials are being filled directly from the delivery of the filter. Newport arrangements for filling and weighing drums are discussed in London Engineering Final Report, September 1951, page 1D8 and 1 F3-10, also in the start-up reports, July 24, and September 27, 1951.

The drums must be clean and dry, especial care being needed with electrical grades. Newport complaint NF/61, November 1951, of turbidity in 1248 was traced to the presence of water in the drums. Apparently, in presence of water, the Aroclor reacted on the galvanizing of the drum. Newport plant report, December 1951, mentions the installation of equipment for drying the drums.

Care is taken not to do any packing if there is HCl fume in the air.

The gaskets for the bungs of the drums have to be correctly chosen. A sample of the gaskets supplied by the Melrath Supply & Gasket Co. to the Krummrich plant was sent to MCL, April 1947. See also the discussion of gaskets on page 2, Section XII, below.

Mr. Pemberton, December 1950, collected samples of the labels used at the Krummrich plant; one a patent list label, and the other a warning label on toxicity.

The effect of exposure of Aroclors to metal contact is discussed in Section VII, comments, page 17 below. Generally speaking, the distilled Aroclors are packed in galvanized drums, the undistilled ones in black steel drums. Flaked or milled solids were packed in barrels or Leverpac containers, bulk solid or resinous Aroclors go out in friction type lid drums, (galvanized for the distilled grades). Storage and blending of the distilled Aroclors is done in steel vessels sprayed with tin or zinc, or aluminum lined.

VI. PROCESS IN DETAIL, contd.

Packing, contd.

Since the Aroclors are such effective solvents , they damage, and are damaged by, ordinary paints, lacquers, insulating varnishes, etc. This feature is important in connection with hydraulic fluids containing Aroclors. See, for example, MCL Research Progress reports 1151, DF 54/26/7 and DF 54/277/6 and 1152 DF 4/16/7 and 8 July 1954, etc., also the Section III, Physical & Chemical data, above. Drums for electrical grades of Aroclors are not painted.

Newport Plant Technical Committee meeting, April 14, 1953, page 2, reported some promising tests with lacquered drums for electrical grades. It was decided not to use lacquered drums for resinous Aroclors on a returnable basis, because the lacquer suffered at the high temperatures likely to be used in melting the material from the drums.

Aroclors also are dispatched in rail cars. At the Krummrich plant the manhole of the car is protected by a tarpaulin tent during filling. Newport plant report for July 1954 mentions the installation of tank-filling facilities. The Krummrich plant operating instructions, August 10, 1954 mention that rail cars for Pyranols and Aroclors are insulated and have either a steam coil or a steam jacket. They are lined with Aluminium or else sprayed with tin and zinc. Only dry air is used for unloading. MCC have a "Christmas Tree" fitting, a one piece fitting carrying air valve, dip pipe, release valve and pressure gauge, for use on rail tanks. It is important to drain the coil or jacket of the rail tank in cold weather. Galvanized or lacquered drums are used for Pyranols.

VI, PROCESS IN DETAIL, contd.

Operating Records.

The following pages, VI-37 to VI-46, show typical Krummrich plant Operating Records.

Chlorination Records are given on pages VI-37 to VI-39. Examples of Dept. 246 Still Records can be seen on pages VI-40 to VI-44. Also shown in this section is a sheet giving Dept. D-218 Muriatic Acid, Acid Receiving, Treatment and Filtration Batch Data, on page VI-45, and page VI-46 is an example of a Pyranol Batch Record Sheet.

See also Operating Records, pages 25b-, of the "Notes on the Plant 'B' Process", Mather, April 1947.

A list of transfers(chlorinators to air-blowing etc. etc.) also is made each day.

CHLORINATION RECORD - Dept - 246

Batch No.

Arrlar No.		Chlorinator No.	Operator	Time	am	pm	Date
1154	223	3	Started Boland	8:30		✓	3-31-55
			Finished Tennyson	7:00		✓	4-1-55

Charge

Cl_2

Biphenyl 3600 #

Total Hrs 23.50 Ins. to _____ Tank _____ @ _____ °C

DH.B. _____

Gas Hrs _____ Ins. in Scrubber _____ @ _____ °C

Time	Cl_2 Gas Press. PSIG	T°C	Cl_2 Feed Rate #/Hr.	Chlor* Back Press. PSIG	Chlor Feed Rate # / Hr.	Sp. Gr. ___ °C Hold Pt. Soft Pt.	Acid Be° 60 °F #1 Unit	Acid Be° 60 °F #2 Unit	Remarks
10:00	26	120	3	6	350	---			
11:00	27	112	3	6	350	---			
12:00	27	112	3	6	300	---			
1:00	27	115	3	6	300	---			
2:00	27	115	3	6	300	1176 - 65			
3:00	27	114	3	6	300				
4:00	24	114	3	9	300	1210-- 65			
5:00	27	114	3	9	350				
6:00	27	114	3	9	250	1256 - 65			
7:00	23	116	3	8	400				
8:00	23	116	3	8	400	1295 - 65			
9:00		Busy	scrubbers						
10:00	24	120	3	8	400	1338 - 65			
11:00	24	121	3	8	400				
12:00	27	123	3½	8	450	1374 - 65			
1:00	25	134	3½	9	475				
2:00	25	136	3½	9	475	1410 - 65			
3:00	26	135	3½	9	475				
4:00		Busy							
5:00	27	144	3½	10	500				
6:00	26	150	3½	11	500	1485 - 65			

MONS 046060

* See page VI-6a.

CHLORINATION RECORD - Dept - 246

Batch No.

Arclor No.		Chlorinator No.	Operator	Time am pm	Date
1148	165	3	Started Robinson	9:15 ✓	5-10-55
			Finished Edmonds - Wyatt	10:30 ✓	5-11-55

Charge

Biphenyl 3600# is _____

U.H.B. _____

Cl_2

Total Hrs 12.25 Ins. to _____ Tank _____ @ _____ °C

Gas Hrs _____ Ins. in Scrubber _____ @ _____ °C

Time	Cl_2 Gas Press. PSIG	T°C	Cl_2 Feed Rate #/Hr	Chlor Back Press. PSIG	Chlor Feed Rate # / Hr.	Sp. Gr. °C Hold Pt. Soft Pt.	Acid Be' 60 °F #1 Unit	Acid Be' 60 °F #2 Unit	Remarks
11:00	20	135	3½	9	450				
12:00	16	130	3½	8	450				
1:00	B	u s	y						
2:00	18	126	4	8	475	1.176 - 90			
3:00	20	130	4	8	475				
4:00	20	138	4	8	475	1.240-90			
5:00	22	135	4	8	475				
6:00	22	135	4	8	475	1.288 - 90			
7:00	B	u s	y						
8:00	20	134	4	8	475	1.333 - 90			
9:00	24	140	4	8	475				
10:00	24	139	4	8	475	1.380 - 90			
10:30	F	i n i s h e d at 10:30				1.416 - 65			
		Switched to 1148. Finished at 10:30.							
		Holding for room.							

MONS 046061

VI - 5
Process
Detail

CHLORINATION RECORD - Dept - 246

Batch No.

Arclor No.		Chlorinator No.	Operator		Time	am	pm	Date
1160	414	5	Started	Robinson	4:30	/		4-26-55
			Finished	Boland	11:00	/		4-27-55

Charge

Biphenyl 3600 # @ _____

TNCB _____

Cl_2

Total Hrs _____ Ins. to _____ Tank _____ @ _____ °C

Gas Hrs _____ Ins. in Scrubber _____ @ _____

Time	Cl_2 Gas Press. PSIG	T°C	Cl_2 Feed Rate #/Hr	Chlor Back Press. PSIG	Chlor Feed Rate # Lbs.	Sp. Gr. @ °C Hold Pt. Soft Pt.	Acid No. @ 60 °F #1 Unit	Acid Be @ 60 °F #2 Unit	Remarks
6:00	26	124	4						
7:00	26	125	4						
8:00	27	126	4						
9:00	27	120	4						
10:00	27	127	4		1.246 - 90				
11:00	25	137	4						
12:00	25	136	4		1.309 - 90				
1:00	22	138	4						
2:00	22	145	4		1.367 - 90				
3:00	19	140	4						
4:00	Busy								
5:00	21	141	4						
6:00	16	135	4						
7:00	18	135	4						
8:00	22	133	4		1.505 - 90				
9:00	22	137	4						
10:00	22	140	4		1.538 - 90				
11:00	21	150	4		~				
					Finished				

MONS 046062

KRUMMRICH PLANT -- BATCH DISTILLATION

DEPT 246 STILL RECORD Arclor No. 1242 Batch No. 16 Still No. 1

		TIME	DATE
Started By	Robinson	1:00 [illegible]	4 - 17 - 55
Finished By	Carnial	4:00 [illegible]	4 - 17 - 55

CHARGE

INS FROM BLOW TK	STILL OUTAGE IN.	TOTAL HRS	STILL BOTTOMS LBS	NO BTCHS after DRG RES	LIME LBS
46" @ °C	@ °C	15.00			

psig

	mm Hg Vacuum		steam		Burners				Temperatures °C					
Time	Rec	Still	Ejector	Flow	1	2	3	4	Rec	Vapor	Inlet	Outlet	Cond. Water	REMARKS
4:00	6	8	175	25	5	5	5	5	50	160	166	171	on	
5:00	✓	✓	✓	✓	✓	✓	✓	✓	68	163	172	177	✓	
6:00	✓	✓	✓	✓	✓	✓	✓	✓	58	165	175	180	✓	
7:00	✓	✓	✓	✓	✓	✓	✓	✓	58	166	178	183	✓	
8:00	✓	✓	✓	✓	✓	✓	✓	✓	58	168	180	185	✓	
9:00	✓	✓	✓	✓	✓	✓	✓	✓	58	169	182	187	✓	
10:00	✓	✓	✓	✓	✓	✓	✓	✓	58	171	183	188	✓	
11:00	✓	✓	✓	✓	✓	✓	✓	✓	56	170	183	188	✓	
12:00	✓	✓	✓	✓	✓	✓	✓	✓	55	170	184	189	✓	
1:00	✓	✓	✓	✓	✓	✓	✓	✓	53	175	186	194	✓	
2:00	✓	✓	✓	✓	✓	✓	✓	✓	53	175	187	197	✓	
3:00	✓	✓	✓	✓	✓	✓	✓	✓	52	180	192	207	✓	
4:00	F	i n	i s	h	e	d			51	184	201	237	off	

MONS 046063

KRUMMRICH PLANT -- Semi-continuous Distillation

DEPT 246 STILL RECORD Arclor No. 1242 Batch No. 18 Still No. 1

	TIME	DATE
Started By Edmonds - Wyatt	3:00 am/pm	4 - 20 - 55
Finished By	8:35 am/pm	4 - 24 - 55

CHARGE				
INS FROM BLOW TK	STILL OUTAGE IN.	TOTAL HRS	STILL BOTTOMS LBS	NO BTCHS after DRG RES	LIME LBS
30" @ 1 °C	@ °C	89.50			200

psig

Sheet No. I (of 4 sheets)

Time	mmHg Vacuum Rec	Still	steam Ejector	Flow	Burners 1	2	3	4	Temperatures °C Rec	Vapor	Inlet	Outlet	Cond. Water	REMARKS
3:00	6	8	175	30	10	6	6	5	52	167	180	187	on	
6:00	✓	✓	✓	✓	✓	✓	✓	✓	61	165	178	185	✓	
7:00									58	165	178	185	✓	
8:00	✓	✓	✓	✓	✓	✓	✓	✓	54	165	178	185	✓	
9:00									53	165	178	185		I
10:00	✓	✓	✓	✓	✓	✓	✓	✓	52	165	178	185	✓	1242
11:00	✓	✓	✓	✓	✓	✓	✓	✓	50	167	180	186	✓	
12:00	✓	✓	✓	✓	✓	✓	✓	✓	50	167	180	186	✓	
1:00	✓	✓	✓	✓	✓	✓	✓	✓	50	167	180	186	✓	
2:00	✓	✓	✓	✓	✓	✓	✓	✓	50	168	181	187	✓	Pumped rec. Away
3:00	✓	✓	✓	✓	✓	✓	✓	✓	50	168	181	187	✓	
4:00	✓	✓	✓	✓	✓	✓	✓	✓	50	168	181	187	✓	
5:00	✓	✓	✓	✓	✓	✓	✓	✓	49	169	182	188	✓	
6:00	✓	✓	✓	✓	✓	✓	✓	✓	48	170	183	189	✓	
7:00	✓	✓	✓	✓	✓	✓	✓	✓	48	170	183	189	✓	
8:00	✓	✓	✓	✓	✓	✓	✓	✓	49	170	182	189	✓	
9:00	✓	✓	✓	✓	✓	✓	✓	✓	49	171	183	189	✓	
10:00	✓	✓	✓	✓	✓	✓	✓	✓	49	171	184	190	✓	
11:00	✓	✓	✓	✓	✓	✓	✓	✓	49	170	184	189	✓	
12:00	✓	✓	✓	✓	✓	✓	✓	✓	49	170	184	189	✓	
1:00	✓	✓	✓	✓	✓	✓	✓	✓	49	170	184	189	✓	
2:00	✓	✓	✓	✓	✓	✓	✓	✓	50	171	184	189	✓	
3:00	✓	✓	✓	✓	✓	✓	✓	✓	50	171	184	189	✓	
4:00	✓	✓	✓	✓	✓	✓	✓	✓	50	171	183	191	✓	
5:00	✓	✓	✓	✓	✓	✓	✓	✓	50	171	185	191	✓	
6:00	✓	✓	✓	✓	✓	✓	✓	✓	50	171	185	191	✓	
7:00	✓	✓	✓	✓	✓	✓	✓	✓	50	171	185	191	✓	

WOK 662 rev DEPT 246 STILL RECORD Arclor No._____ Batch No._____ Still No._____

	TIME	DATE
Started By	am pm	
Finished By	am pm	

CHARGE

INS FROM BLOW TK	STILL OUTAGE IN.	TOTAL HRS	STILL BOTTOMS LBS	NO BTCHS after DRG RES	LIME LBS
@ °C	@ °C				

SHEET NO. II (of 4 sheets)

	Vacuum				Burners				Temperatures °C					
Time	Rec	Still	Ejector	Flow	1	2	3	4	Rec	Vapor	Inlet	Outlet	Cond. Water	REMARKS
8:00	6	8	175	30	10	6	6	5	50	171	185	190	on	
9:00	✓	✓	✓	✓	✓	✓	✓	✓	49	170	184	189	✓	
10:00	✓	✓	✓	✓	✓	✓	✓	✓	49	170	184	189	✓	
11:00	✓	✓	✓	✓	✓	✓	✓	✓	49	170	184	189	✓	
12:00	✓	✓	✓	✓	✓	✓	✓	✓	48	170	184	189	✓	
1:00	✓	✓	✓	✓	✓	✓	✓	✓	47	170	185	190	✓	I
2:00	✓	✓	✓	✓	✓	✓	✓	✓	47	170	184	190	✓	1242
3:00	✓	✓	✓	✓	✓	✓	✓	✓	47	170	184	190	✓	Pumped rec. Away
4:00	✓	✓	✓	✓	✓	✓	✓	✓	47	170	184	190	✓	
5:00	✓	✓	✓	✓	✓	✓	✓	✓	47	170	184	190	✓	
6:00	✓	✓	✓	✓	✓	✓	✓	✓	47	170	184	189	✓	
7:00	✓	✓	✓	✓	✓	✓	✓	✓	47	170	184	190	✓	
8:00	✓	✓	✓	✓	✓	✓	✓	✓	48	170	184	190	✓	
9:00	✓	✓	✓	✓	✓	✓	✓	✓	48	171	185	191	✓	
10:00	✓	✓	✓	✓	✓	✓	✓	✓	48	171	185	191	✓	
11:00	✓	✓	✓	✓	✓	✓	✓	✓	48	171	185	190	✓	
12:00	✓	✓	✓	✓	✓	✓	✓	✓	45	171	185	191	✓	
1:00	✓	✓	✓	✓	✓	✓	✓	✓	45	171	187	192	✓	
2:00	✓	✓	✓	✓	✓	✓	✓	✓	46	171	186	191	✓	
3:00	✓	✓	✓	✓	✓	✓	✓	✓	45	172	187	192	✓	
4:00	✓	✓	✓	✓	✓	✓	✓	✓	48	172	187	193	✓	
5:00	✓	✓	✓	✓	✓	✓	✓	✓	49	172	187	193	✓	
6:00	✓	✓	✓	✓	✓	✓	✓	✓	50	172	187	193	✓	
7:00	✓	✓	✓	✓	✓	✓	✓	✓	49	172	187	193	✓	
8:00	✓	✓	✓	✓	✓	✓	✓	✓	49	172	187	193	✓	
9:00	✓	✓	✓	✓	✓	✓	✓	✓	49	172	189	195	✓	
10:00	✓	✓	✓	✓	✓	✓	✓	✓	49	172	189	195	✓	
11:00	✓	✓	✓	✓	✓	✓	✓	✓	49	172	189	195	✓	

WORK SHEET DEPT 246 STILL RECORD Arclor No. 1242 Batch No. 18 Still No. 1

	TIME	DATE
Started By	am pm	
Finished By	am pm	

CHARGE

INS FROM BLOW TK	STILL OUTAGE IN.	TOTAL HRS	STILL BOTTOMS LBS	NO BTCHS after DRG RES	LIME LBS
@ °C	@ °C				

SHEET NO. III (of 4 sheets)

Time	Vacuum Rec	Vacuum Still	Vacuum Ejector	Flow	Burners 1	2	3	4	Temperatures °C Rob	Vapor	Inlet	Outlet	Cond. Water	REMARKS
12:00	6	8	175	25	5	5	5	5	49	172	189	195	on	
1:00	✓	✓	✓	✓	✓	✓	✓	✓	49	173	189	195	✓	
2:00	✓	✓	✓	✓	✓	✓	✓	✓	49	189	189	195	✓	Pumped rec. Away
3:00	✓	✓	✓	✓	✓	✓	✓	✓	49	172	189	195	✓	
4:00	✓	✓	✓	✓	✓	✓	✓	✓	50	172	189	195	✓	
5:00	✓	✓	✓	✓	✓	✓	✓	✓	50	172	189	195	✓	
6:00	✓	✓	✓	✓	✓	✓	✓	✓	51	172	189	195	✓	1242
7:00	✓	✓	✓	✓	✓	✓	✓	✓	51	172	190	196	✓	
8:00	✓	✓	✓	✓	✓	✓	✓	✓	51	172	190	196	✓	
9:00	✓	✓	✓	✓	✓	✓	✓	✓	50	172	190	196	✓	
10:00	✓	✓	✓	✓	✓	✓	✓	✓	49	172	190	196	✓	
11:00	✓	✓	✓	✓	✓	✓	✓	✓	49	172	190	196	✓	
12:00	✓	✓	✓	✓	✓	✓	✓	✓	49	172	190	196	✓	
1:00	✓	✓	✓	✓	✓	✓	✓	✓	49	172	190	196	✓	
2:00	✓	✓	✓	✓	✓	✓	✓	✓	49	172	190	196	✓	
3:00	✓	✓	✓	✓	✓	✓	✓	✓	48	172	191	197	✓	
4:00	✓	✓	✓	✓	✓	✓	✓	✓	50	172	191	197	✓	
5:00	✓	✓	✓	✓	✓	✓	✓	✓	50	172	192	195	✓	
6:00	✓	✓	✓	✓	✓	✓	✓	✓	50	172	190	195	✓	
7:00	✓	✓	✓	✓	✓	✓	✓	✓	48	170	190	195	✓	
8:00	✓	✓	✓	✓	✓	✓	✓	✓	48	170	190	195	✓	
9:00	✓	✓	✓	✓	✓	✓	✓	✓	48	169	189	195	✓	
10:00	✓	✓	✓	✓	✓	✓	✓	✓	48	169	190	196	✓	
11:00	✓	✓	✓	✓	✓	✓	✓	✓	48	169	190	196	✓	
12:00	✓	✓	✓	✓	✓	✓	✓	✓	48	170	190	196	✓	
1:00	✓	✓	✓	✓	✓	✓	✓	✓	48	171	196	196	✓	
2:00	✓	✓	✓	✓	✓	✓	✓	✓	48	171	191	196	✓	
3:00	✓	✓	✓	✓	✓	✓	✓	✓	48	171	191	196	✓	

DEPT 246 STILL RECORD Archer No. 1242 Batch No. 18 Still No. 1

	TIME	DATE
Started By	am pm	
Finished By Boland	8:35 am pm	4-24-55

CHARGE

INS FROM BLOW TK	STILL OUTAGE IN.	TOTAL HRS	STILL BOTTOMS LBS	NO BTCHS after DRG RES	LIME LBS
@ °C	@ °C				

SHEET NO. IV (of 4 sheets)

	Vacuum				Burners				Temperatures °C					
Time	Rec	Still	Ejector	Flow	1	2	3	4	Rec	Vapor	Inlet	Outlet	Cond. Water	REMARKS
4:00	6	8	175	25	5	5	5	5	47	172	192	197	on	Pumped away
5:00	✓	✓	✓	✓	✓	✓	✓	✓	47	172	192	198	/	at 4:30 a.m.
6:00	✓	✓	✓	✓	✓	✓	✓	✓	52	175	200	208	/	auto. off
7:00	✓	✓	✓	✓	✓	✓	✓	✓	52	177	206	212	/	
8:00	✓	✓	✓	✓	✓	✓	✓	✓	49	186	230	240	/	
8:35	F	1	h 1	s	h	e		d	49	190	246	255	off	

MONS 046067

DEPT. D-218 MURIATIC ACID

ACID RECEIVING, TREATMENT AND FILTRATION

BATCH DATA

Date: May 10, 1955

CARBON BATCH NO. 2
ACID BATCH NO. 318

Time flow started to # 2 Receiver 8:10 pm. Operator: Maclin

A. Hourly Readings

TIME	INCHES	OPERATOR	TANKS WASHED BY	OPER.	TIME
10:00 pm	26"	Maclin			
12:00 mn	35"	Moesser			
2:00 am	45"	Moesser			
4:00 am	55"	Moesser			
6:00 am	69"	Moesser	carbon added		
8:00 am	69" ??	Hall			
10:00 am	79"	Hall			
12:00 pm	86"	Hall			
2:00 pm	89" by stick	Hall			
4:00 pm	89" by stick	Hall			
6:00 pm	90"	Hall			
8:00 pm	93"gage	Hall			
9:00 pm	96"gage	Hall			

Time Start Stirring 9:00 PM Oper. Hall End 9:30 PM Oper. Hall

Settling Time 9:30(5-11-55) PM Time Finished 9:30(5-12-55) PM

Time Start Filtering 9:30PM (5-12-55) Oper. _____ Fin. PM Oper. _____

ACID ANALYSIS AND ADJUSTMENT

Be at 60°F. ✓ After ✓ Gals. Water added. Be ✓ Color ✓

Lab. Report & Final analysis - Color ✓ AM Be ✓ Color ✓

Time Pumping to # ✓ Stg. _____ PM Oper. ✓ Oper. ✓

Time finished ✓ AM PM Oper. ✓

MONS 046068

PYRANOL BATCH RECORD SHEET

	TIME	DATE	OPERATOR
Batch No. 27 Iner PPO Lot No. R-1813			
Pumped ToB to Mix Tk.#1 To 107"Out	7:45 pm	May 10/55	"Mac"
From Car No. _____			
From #6 Stg. B.P. 38 A.P.14"			
Pumped Aroclor 1260 to Mix Tk.#1 to 60 "out			
No. 5 Stg. B.P. 72" A.P. 36½	11:30 pm	May 10/55	"Mac"
1st. Sample to Lab at _____	1:45 am	May 10/55	Moeser
Sp. Gr. Ref.2nd H_2O CL			
1.567 1.6160 .0010 L - 10	PPO is OK Moeser		
Added ____ "1260 at _____			
No. 6 Stg. B.P. _____ A. P. ____			
Added 2 "ToB at 4:45 am			Moeser
#6 Stg. B.P. 14"A.P. 10"			
Mix Tk B.P. ___ A.P. ____			
Sample to Lab at 7:00 am			Moeser
Sp. Gr. Ref. 2nd N_2O CL ____			
_____ _____ _____ _____	Added 1 bucket of Att. earth 10#		
No. _____ Stg. B.P. ____ A.P. ____			
Mix Tk B.P. _____ A.P. ____			
Added _____ "ToB at _____			
#6 Stg. B.P. _____ A.P. ____			
Mix Tk. B.P. _____ A.P. ____			
Sample to Lab at _____			
Sp. Gr. Ref.2nd. H_2O CL ____			
Added _____ lbs.Tin Tet to Mix Tk.at	P.P.O. added 9:30 am 5-11-55		Hall
_____ lbs. from Drum No. added			
_____ lbs. from Drum No. P.P.O.			
Mix Tk. Sampled by Lab. at 11:30 am	11:30 am 5-11-55		Hall

VI. PROCESS IN DETAIL, contd.

Process Records, Krummrich Plant

Daily Inventory

At 7:00 a.m. each working day a "Daily Inventory Report" is made covering the main items of process material, giving for example the gauge, density, batch number where applicable, in the following places:

HCl in store tanks
treatment tanks
rail cars

Diphenyl in store tanks
head tank
rail cars

High boilers in melt tank

Aroclor in store tanks
chlorinators
airblowing tanks
stills
blending tanks
rail cars

Pyranol in blender
rail cars

Materials in drums, or bags, to the nearest whole package.

Readings of the "utilities" meters, and deliveries of HCl gas to other departments, or receipts of gas from other departments, are reported on the same form, also the past day's repairs and any special operating difficulties, and a list of samples ready for test at 8:00 a.m.

VI. PROCESS IN DETAIL, contd.

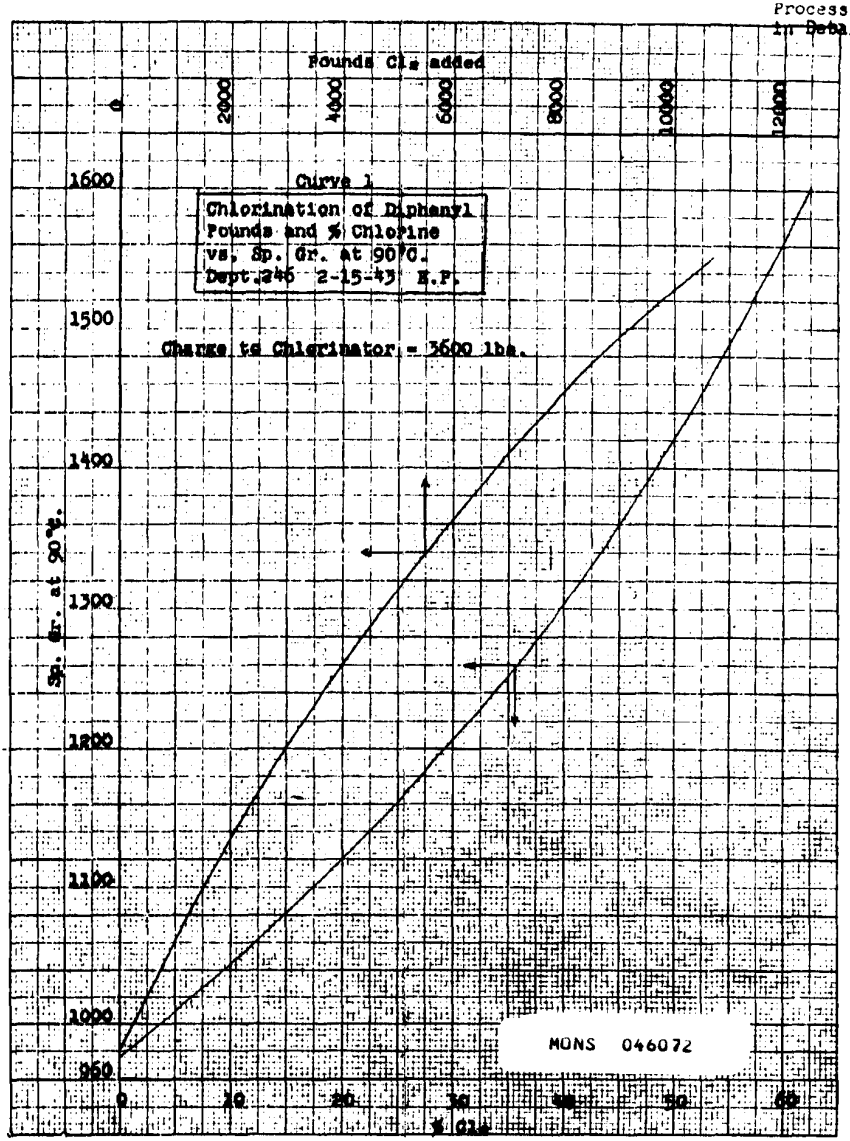
Monthly Inventory

The usual measurements of tank contents, temperatures, densities etc. are made.

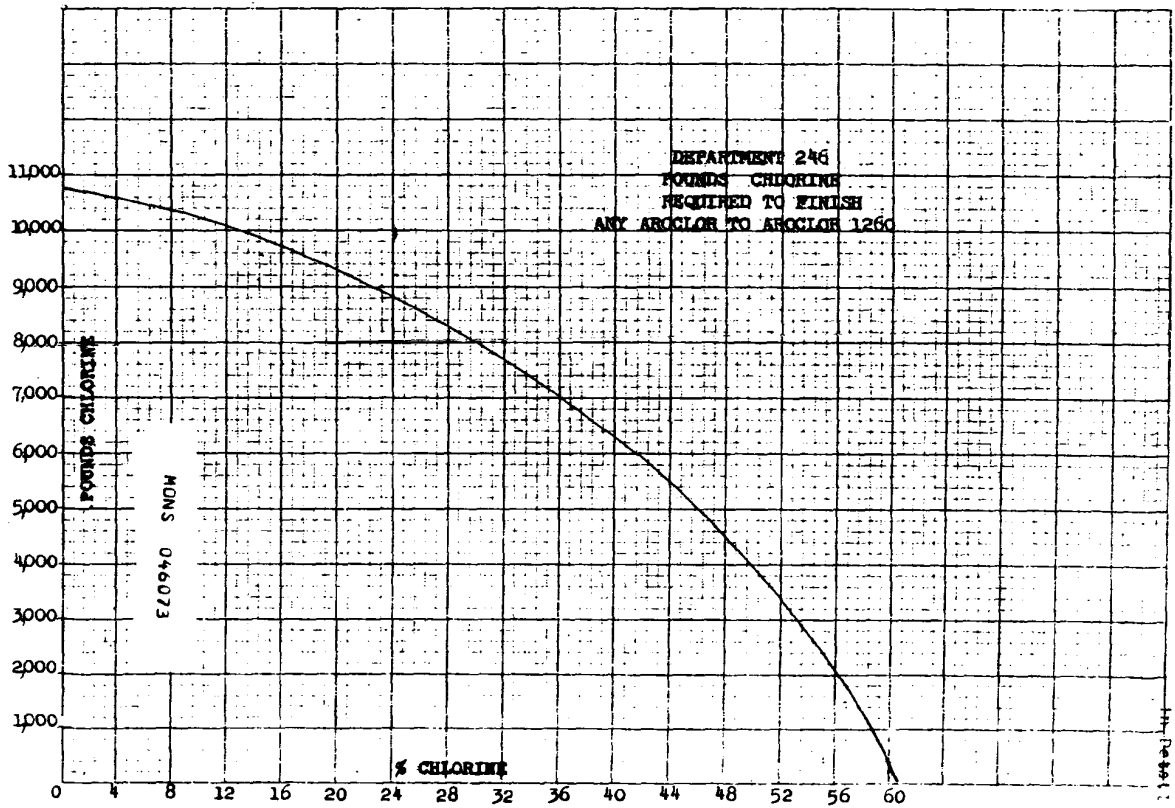
From the densities of the chlorinator batches at the time the inventory is taken, it is possible to calculate from the curves on pages III-15 and VI-48 and VI - 49, how much chlorine is still required to finish the batches, and the batches are then evaluated as if their contents of diphenyl had been carried through to crude Aroclor, with a deduction for the chlorine still needed. Other materials in process are similarly evaluated, vacuum still batches being taken at the values of the crude charges issued to them.

Examples of the inventory calculations are given on pages 55 - of Section VI, taken from the pages 137- of the report by Lyles, Soffranko and Becker, November 1946.

The Inventory is used to prepare monthly departmental returns, as described in Section X, below.


Pounds Cl₂ added
0
2000
4000
6000
8000
10000
12000
Curve 1
Chlorination of Diphenyl
Pounds and % Chlorine
vs. Sp. Gr. at 90°C.
Dept.246 2-15-43 E.P.
Charge to Chlorinator = 3600 lbs.
Sp. Gr. at 90°C.
1600
1500
1400
1300
1200
1100
1000
950
0
10
20
30
40
50
60
% Cl₂
MONS 046072

A-8393


DEPARTMENT 246
POUNDS CHLORINE
REQUIRED TO FINISH
ANY AROCLOR TO AROCLOR 1260
POUNDS CHLORINE
% CHLORINE
MONS 04073
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
4
8
12
16
20
24
28
32
36
40
44
48
52
56
60

VI. PROCESS IN DETAIL, contd.

Dept. 246 Monthly Inventory 8 A.M.

Goods in Process - Aroclor

Chlor. No.	Batch No.	Diphenyl pounds Charged	Sp. Gr.	Temp. °C.	Equiv. Aroclor	Finished Aroclor to be	Lbs. Cl_2 required Finish	Equiv. Lbs. of Finished Aroclor
1	Empty						(1)	(2)
2	641	3600	1.366	90	1145 (45.6 %Cl)	1260	5000	7800
3	Empty							3600 / 0.4385 x 95%* recovery
4	Empty							

(1) From curves attached, pages III-15, and VI-49.
(2) From table attached, page X-12b.
* Note: Mr. Furzey reported Dec.11/51 that the distillation yield was about 95% at the Krummrich plant and a little higher at Anniston.

Tank No.	Batch No.	Type Aroclor	Ins. Out-age	U.S. Gals.	Sp. Gr. Temp. °C.	Lbs. Per US Gallon	% Recov-ery	Total Lbs. Recovered
Blow Tank	640	1160	23" in,	651	1.525 at 120	12.7	95	7854
Old Still	Empty							
New Still	894	1160	114	784	1.535 at 110	12.8	95	9533
Old Blend Tank	Empty							
New Blend Tank	893	1160	73	706	1.545 at 100	12.9	98	8923
							Total	26,310
							Less 10% contingency	2,631
							Difference	23,679
							In Chlorinator	7,800
							Total goods in process	31,479

VI. PROCESS IN DETAIL, contd.

Dept. 246 Monthly Inventory 8 A.M.

Finished Goods - Aroclor

Tank	Type Aroclor	Sp.Gr.	Temp. °C.	Lbs. Per. Gal.	Inches in Tank	U.S. Gallons	Lbs.Finished Aroclor
Old F.P.Tk	Empty						
New F.P.Tk	1260	1.555	90	12.96	30" outage	1725	22,356
#3 Stg.	1260	1.555	90	12.96	53	6492	84,136
#4 Stg.	1254	1.495	70	12.46	52	6587	82,073
#5 Stg.	Empty						
Cars	MONX 809 Empty						

(Solid Aroclors)

Tank	Batch No.	Lbs. Diphenyl charged	Hold Pt.	Equiv. Aroclor	Fin. Aroclor To Be	Lbs.Cl$_2$ Req'd.To Finish	Lbs. Crude	% Rec'd.	Lbs. Fin. Aroclor
#1 Chlor.	Empty								
Stg.Pot	Empty								

Still Pot - Empty
Pre Runs - None

Finished Goods - Aroclor

North Hopper

Empty Inches out - _____ Cu.Ft. x _____ #1 Cu.Ft. _____ #

South Hopper

Empty Inches out - _____ Cu.Ft. X _____ #1 Cu.Ft. = _____ #

Lots 0 No.Bags 50 No.Lbs. _____ Lbs. in container

VI. PROCESS IN DETAIL, contd.

Dept. 246 Monthly Inventory 8 A.M.

Raw Materials

Diphenyl

In Scrubber Liquor (See page VI-53)	8401
In Measuring Tank	3600
In #1 Storage Tank	
82 inches in. = 11736 U.S.Gals. x 8#/U.S.Gal.	93888
In #1 Storage Tank	
0 Inches in = _____U.S.Gals.x_____#/Gal.	
In Cars - None	
	105889
Less Reserve	3000
Reported Total	102889

Chlorine

	Pounds
Liquid in cylinders - 0	
Gas in Scrubber liquor (See Sheet following this page)	

Crude High Boilers 0

Distilled High Boilers 0

Aroclor Trappings 0

Lime 30 bags at 50# each = 1500#

Attapulgus Earth 6 bags at 50# each = 300 #

Toluol 1 drum = 200#

VI. PROCESS IN DETAIL, contd.

Dept. 246 Monthly Inventory 8 A.M.

Scrubber Liquor

Scrubber No.	Inches Innage	U.S. Gals.	Sp.Gr.	Temp. °C.	Equiv.* Aroclor	U.S. #/Gal.	Lbs. Cl_2 (1)	Lbs. Diphenyl (2)	Lbs. Total
1	10	172	1.200	65	1126	10.00	447	1273	1720
2	10	172	1.230	65	1130	10.25	529	1234	1763
3	20	271	1.200	65	1126	10.00	706	2004	2710
4	20	271	1.230	65	1130	10.25	833	1945	2778
1A	20	271	1.230	65	1130	10.25	833	1945	2778
							3348	8401	11749
					Less Reserve		1000	0	
					Total Reported		2348	8401	

Note:
(1) Lbs. total x % Cl_2 = lbs. Cl_2
(2) Lbs. total - lbs. Cl_2 = lbs. diphenyl

* corresponding to the % chlorine from the curve on page III-15.

See also the ready reckoner on pages 10b, c and d, Section VI.

MONS 046077

VI. PROCESS IN DETAIL, contd.

Dept. 246 Monthly Inventory 8 A.M.

CRUDE AROCLOR

1142	Lot	R-2	= 1	part	drum	258#
1148	"	T-2	= 1	"	"	485
1154	"	T-1	= 1	"	"	492
1160	"	T-1	= 1	"	"	530
1162	"	T-1	= 1	"	"	597
1168	0					
1169	0					
1170	Lot	R-1	= 1	part	drum	170#
1171	"	T-1	= 1	"	"	220
4065	0					
5060	0					
2565	0					
300	0					
301	0					

VI. PROCESS IN DETAIL, contd.

Dept. 246 Monthly Inventory 8 A.M. 6-1-46

Aroclor	On Hand 5-1-46	Produced	Net Recd.	Total To Account For	To 363 Net	To A-246	Total Transfer	On Hand 6-1-46 by Diff.	On Hand 6-1-46 by Inventor
1119	0	0	0	0	0	0	0	0	0
1132	0	0	0	0	0	0	0	0	0
1142	260	0	0	260	2	0	2	258	258
1148	285	200	0	485	0	0	0	485	485
1154	194	300	0	494	2	0	2	492	492
1160	530	0	0	530	0	0	0	530	530
1162	598	0	0	598	1	0	1	597	597
1168	0	0	0	0	0	0	0	0	0
1169	0	0	0	0	0	0	0	0	0
1170	170	0	0	170	0	0	0	170	170
1171	220	0	0	220	0	0	0	220	220
4065	0	0	0	0	0	0	0	0	0
5060	0	0	0	0	0	0	0	0	0
2565	0	0	0	0	0	0	0	0	0
									2752
1242	400	0	0	400	0	0	0	400	400
1248	415	32,485	0	32,900	11,800	0	11,800	21,100	21,100
1254	230,818	187,107	0	417,925	325,352	0	325,352	92,573	92,573
1260	307,296	97,147	0	404,443	217,963	48,509	266,472	137,971	137,971
1262	440	0	0	440	148	0	148	292	292
1262M	205	2,000	0	2,205	2,000	0	2,000	205	205
1268	0	0	0	0	0	0	0	0	0
1269	0	0	0	0	0	0	0	0	0
1270	116	0	0	116	116	0	116	0	0
1270S	0	0	0	0	0	0	0	0	0
1271	143	3,000	0	3,143	3,001	0	3,001	142	142
4465	0	0	0	0	0	(1) 0	0	0	0
5460	29,775	0	0	29,775	0	3,600	3,600	26,175	26,175
300	0	0	0	0	0	0	0	0	0
301	0	0	0	0	0	0	0	0	0
TOTAL	571,865	322,239	0	894,104	560,385	52,109	612,494	281,610	281,610
							In process - Finished		31,470 250,131#

(1) To Dept. D-246

VI. PROCESS IN DETAIL, contd.

Emergency Measures

Power Failure

Chlorinators

1. Shut off the chlorine feed.
2. Shut off the cooling water.
3. Shut off the feed water and cooling water on HCl absorbers.

Stills

4. Shut of still furnaces.
5. Suck out still coils.
6. For a long stoppage close off, or vent, the still. Stop the steam on the ejectors, then the water.
7. Drain the still if only a heel is left in it.

Aroclor Filter

8. Blow the press contents to the blending tank.

HCl Handling

9. Close valves below all acid tanks.
10. Replace all plugs and manlids.

General

11. Throw all motor switches to the "off" position.
12. Turn off all gas burners.
13. Leave rail cars in safe condition.

Air Failure

1. Affect tank unloading, air-blowing of batches, air jets in the stacks of air blowing tank and acid mixing tank.

2. Distillation will have to stop when the supplied aerated crude is exhausted.

VI. PROCESS IN DETAIL, contd.

Emergency Measures, contd.

Plant Water Failure

1. Chlorination would have to stop because of lack of cooling for the HCl absorber.
2. Ejector interstage cooling could fail.
3. Water supply to the heat exchangers on the stills could fail.
4. Chlorinator pot gland, water cooling would fail, also the cooling of the flaker (solid Aroclor plant no longer in use).
5. Water supply to various pump glands would fail.

City Water Failure

The following units depend on city water: --

1. Chlorinator jackets.
2. HCl absorbers (feed water).
3. Safety showers, toilets, drinking fountains, wash bowls.

Natural Gas Failure

Natural gas serves: --

1. Chlorinator circulation line etc.
2. Chlorinator pot.
3. Still furnaces.
4. General local heating.

VI. PROCESS IN DETAIL, contd.

Emergency Measures

Low Pressure Steam Failure

Low Pressure Steam serves:

1. Steam tracing
2. Air-blowing tank coils
3. Chlorinator jackets
4. Scrubber liquor tank coils and jackets
5. Under-ground store tanks
6. Blending tank coils
7. Flaker drain
8. Condenser make-up water
9. Rail-car heating.

High Pressure Steam Failure

High Pressure Steam serves:

1. Tracing from No. 1 chlorinator to the still.
2. Tracing from high boiler melt-tank to the chlorinators
3. High boiler melt tank coil
4. Still receiver coils
5. Ejectors

If gas and power are available, but other services lacking for a short period, the circulation should be kept up through the still coils with a minimum of furnace heat.

VII. COMMENTS ON THE PROCESS

See also Section II, "CHEMISTRY OF THE PROCESS".

See also Section XI, "Hazards".

Chlorination Stage.

The iron turnings used inside the chlorinator bring about more intimate contact between gas and liquid, as well as providing the catalyst for the reaction, and probably diminishing the corrosion of the vessel itself.

Coarse iron turnings are best, and before being used, they are placed in a heap on the floor, and any oil they may contain is burnt off with a gas flame, the heap being turned over with a fork, so that small pieces drop out and can be discarded.

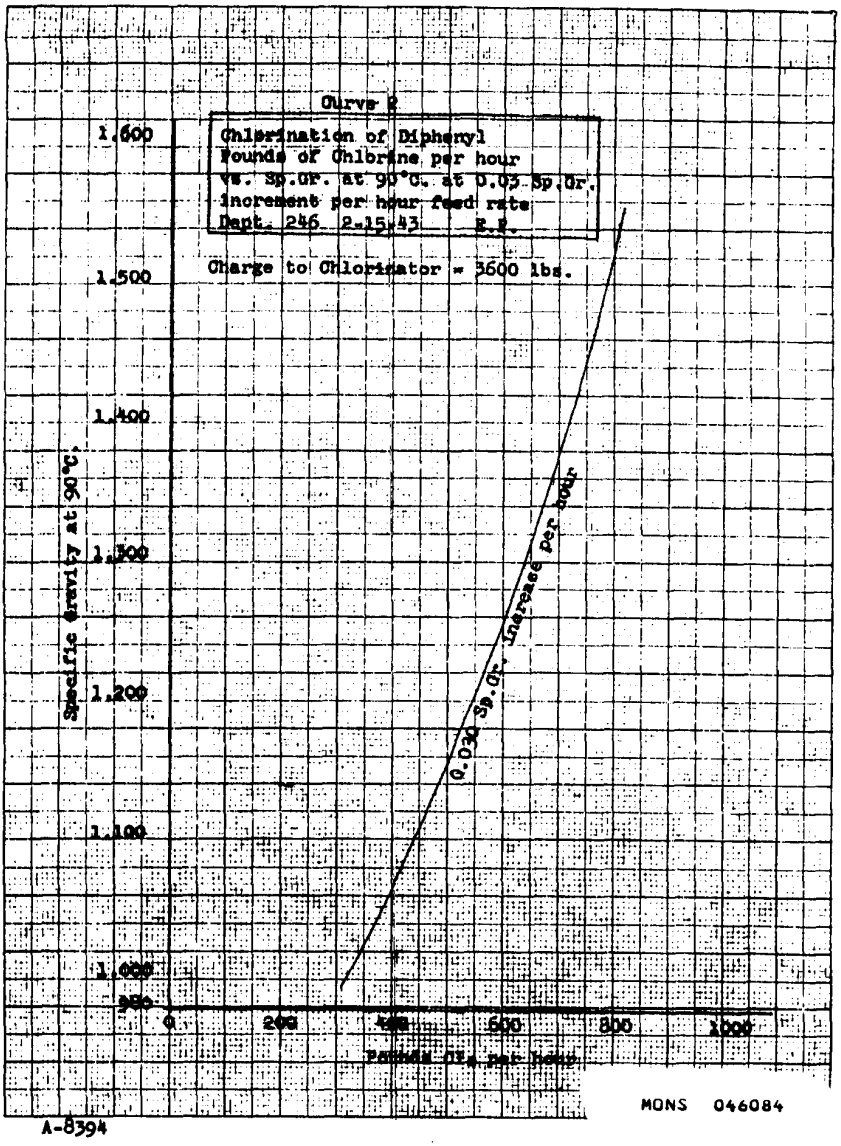
A sample of suitable turnings was sent to MCL by Mr. Harden, in August 1950.

See Section VII, page 10, for a suggestion that foreign metals in the turnings may cause discolouration of the Aroclors.

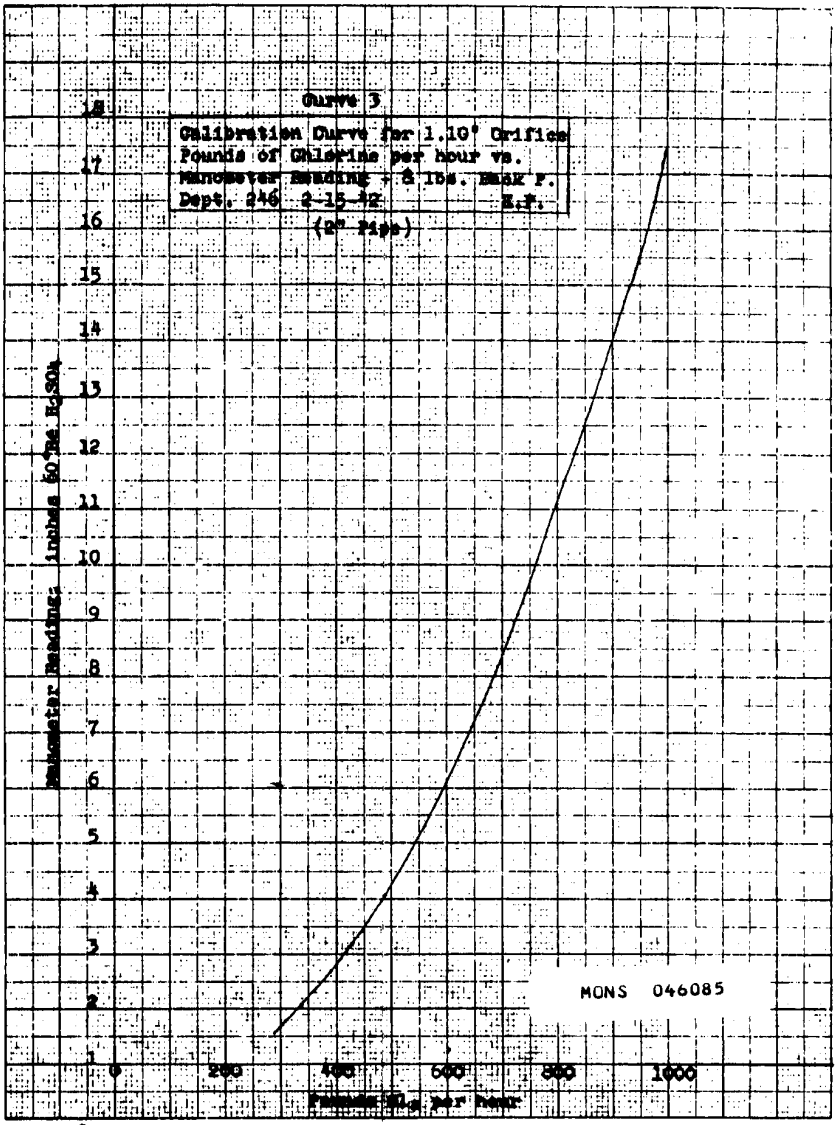
The turnings are slowly consumed in use, and their level in the chlorinator should be checked about once a month. High chlorine content in the off-gas is an indication that the bed of catalyst may be getting thin, or may have a hole through it, or be in some other way out of order. The presence of moisture, as from a leak in the chlorinator coil or jacket, naturally increases the rate of consumption of the iron. Normally the wastage is about 200 - 300 pounds in 3 to 4 months for each chlorinator.

Chart A 8393, page VI-48, showing the relation between density, chlorine content, and chlorine usage in the chlorination of diphenyl, and chart A 8394, page VII-1a, connecting the rate of feed of chlorine with the rate of increase in density, may be of use in judging the progress of chlorinations. Chart A 8394 is used also in calculating the inventory values of unfinished chlorination batches.

Chart A 8395, page VII-1b, is a calibration curve for the flow of chlorine through the 1.1" orifice as used on the lines to the Krummrich Plt.


Curve 2
Chlorination of Diphenyl
Pounds of Chlorine per hour
Vs. Sp.Gr. at 90°C. at 0.03 Sp.Gr.
increment per hour feed rate
Dept. 246 2-15-43 E.E.
Charge to Chlorinator = 3600 lbs.
1.600
1.500
1.400
1.300
1.200
1.100
1.000
Specific Gravity at 90°C.
0.030 Sp.Gr. Increase per hour
0
200
400
600
800
1000
Pounds Cl2 per hour
MONS 046084

A-8394


Curve 3
Calibration Curve for 1.10" Orifice
Pounds of Chlorine per hour vs.
Manometer Reading + 8 lbs. Mark F.
Dept. 246 2-15-42 E.F.
(2" Pipe)
Manometer Readings inches 60°Be H2SO4
Pounds Cl2 per hour
0
200
400
600
800
1000
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
MONS 046085

A-8395

VII. COMMENTS ON THE PROCESS, contd.

Chlorination Stage, contd.

chlorinators. Note that the curve refers to gas at 8 psig, but the actual pressure at the orifice varies with the rate of flow of the gas, and with the depth and density of the charge in the chlorinator.

The use of low grade chlorine appears to be harmless, as mentioned on page 2, Section VI. MCC Short form report 2673, 171-790, December 10, 1951, deals with the use of dilute chlorine.

A few points from the early experience at Newport are worth noting. If the circulation line is blocked at its upper end, samples drawn from the sample point at the bottom of the line may be misleading, and chlorination may be carried too far. The circulation lines were re-arranged for easier clearing, with a thermo couple in the delivery end, to guard against this at Newport. Silver relief discs 12" diameter 0.003" thick, calibrated to burst at 25 psig were used on the chlorinators, but failed by pinholing. Later, similar discs failed under pressure when the gas outlet line became blocked. Silicone grease served well on the chlorine cocks. Chlorinated Santowax proved rather difficult to pump. The lines needed high pressure steam (or even heated Aroclor) jackets, and more powerful pumping had to be arranged.

A chlorinator inlet fitting of nickel cast iron was installed -- said to be the same as used at Anniston. The cooling coils were lengthened in 1953, giving a 33% increase in chlorination rate.

Crystallization trouble was encountered with 5460 batches, but washing of the chlorinator with Santowax, before use, gave improvement.

A suggestion has been made at Anniston to work two chlorinators together by crossing the pump connection. This might save a little time, since twice the amount could be handled at each charging and discharging.

Some thought also has been given to setting up continuous working through a cascade of chlorinators, drawing off the different grades at different points. The production rate is not yet thought to justify this.

VII. COMMENTS ON THE PROCESS, contd.

Chlorination Stage, contd.

In a report on a visit to Anniston, March 31, 1952, Mr. Dalton mentions discussion of the effect of quicker chlorination. It was thought that the higher rate might disturb the isomer ratio particularly with the lower Aroclors, or might give a higher proportion of polychloro diphenyl, leading to a higher proportion of still bottoms. Higher temperatures might have similar effects, and might increase the rate of corrosion of the chlorination vessel.

Scrubbing of the Chlorination Off-gases.

At a starting temperature of 120°C. the diphenyl would have a vapour pressure of about 12 mm, so that one mole of HCl would carry away something less than 1/60th mole of diphenyl during chlorination to mono-chlor. Similarly, mono-chlor diphenyl at about 135°C. would have about the same vapour pressure, so that about 1/60th of a mole more would be carried away during chlorination to dichlor, and similarly with chlorination at 150° to tri-chlor. After that, the vapour pressures drop more quickly than corresponds to the batch temperature increases, so that vapourization losses become almost negligible in the later stages, and the total vapourisation loss cannot exceed about 5% of the batch.

Cooling the off-gas to something below 100°C. would condense almost all of this vapour, so the Anniston cyclones, with the long outdoor gas main, and the coke towers, are probably as efficient in this respect as are the scrubbers etc. at the Krummrich plant. Reaction with unused chlorine is another matter, and the Anniston arrangement does not include any provision for this. Havercroft and Mather, 1947 reported a smell of chlorine at the Anniston HCl absorber exit, and so did Harden in 1950, but in the latter case the explanation was given that the chlorinators were running rather cold. When the higher Aroclors are being made, there is a less depth of diphenyl etc. in the chlorinator during the early stages, than when lighter Aroclors are being made, and towards the end of the chlorination of the reaction presumably becomes quite slow, in spite of the higher temperatures used. Nevertheless, Anniston

VII. COMMENTS ON THE PROCESS, contd.

Scrubbing of the Chlorination Off-gases, contd.

report fairly good chlorine efficiencies, (see Section X below). With the Anniston off-gas arrangements, any material volatilising from higher Aroclors can fairly easily be kept apart from any coming from lower Aroclors.

Air-blowing of Crude Aroclors.

It is desirable to use thoroughly dried air because it helps to remove moisture from the product, and probably this makes the HCl easier to remove. London Engineering Dept. report, September 1951, (page 107), on the start-up of the Newport plant, reported that a dense cloud of HCl was formed when air-blowing started, and a simple lime scrubber was installed to prevent trouble with the Alkali Acts. The amount of HCl was about 2 lbs./batch. Resinous Aroclor required longer aeration -- up to 6 hours. Page 1P3 - 12 stated that the receiver for the air-blown batches needed an agitator.

The Newport start up report of July 12, 1951 mentions a stoneware off-gas pipe carried above the roof level. A letter, Mather to Thrift, May 2, 1949, mentions a Haveg stack at the Krummrich plant, and states that there was no fume nuisance there. The moister air at Newport could cause the HCl to be more noticeable.

The crude, before air-blowing, on occasional tests, showed 0.5 to 1.0 mg KOH, equivalent per gram of sample, corresponding to about 2½ to 5 lbs. HCl per chlorination batch.

Anniston in 1947 were using compressed air from the plant main, not dried otherwise than by compression and cooling.

Distillation

Operating difficulties may arise from poor operation of the ejectors, or from freezing of the condenser, or from plugging of the vacuum lines. The cotton wool, or the upper layers of coke, in the scrubbers on the vacuum line, may become plugged with Aroclor, and with pipe scale, in which case the top cover of the scrubber is removed, the screen taken out, and the upper layers of coke removed. The coke in

VII. COMMENTS ON THE PROCESS, contd.

Distillation, contd.

the tower is impregnated with 50% caustic soda solution once or twice a year. The scrubber is filled with the solution, left 24 hours, then drained through the bottom outlet.

Later reports state that the caustic soda has been given up at Anniston and at the Krummrich plant. At the Krummrich plant the scrubbers are heated externally by gas flames about each 10 days, and drained to cans.

The hydrated lime used in the still presumably contains some free water, and any reaction with HCl will produce more water. Most of this water will escape during the distillation, and the remainder will be taken up by the Attapulgus earth in the subsequent treatment of the distilled Aroclor.

The London Engineering Department Final Reports on the start-up of the Newport plant record the troubles encountered with the circulation pump, which was located in a secondary vessel below the main still. See also page 79, Section XII. The tube plate of the condenser leaked at high temperature. Steam leaked into jacketed lines, and gave colour trouble. It appeared advantageous to direct the return stream from the heating coil tangentially into the still. It was easier to keep a steady vacuum at 12 mm than when working at 3 - 4 mm was attempted.

There was discussion of the relative advantages of a Hagen separator inside the still, and a cyclone separator outside it on the vapour stream.

Mr. Furzey reported, December 5, 1951, that the return lines from the coils at Newport had been carried down below the level of the charges in the still. This arrangement, however, did not give any better coloured distillate, so it was abandoned.

The return section, in particular, should be arranged for easy rodding, to clear it. At the Krummrich plant, Merco cocks are used on the circulating line.

VII. COMMENTS ON THE PROCESS, contd.

Distillation, contd.

The stills have to be cleaned out about once a year, and any incrustation of lime etc. chipped out. As the pumps become less efficient by wear or corrosion, or the velocity of circulation of liquid through the heating coil becomes slower for other reasons, there is more liability for the coil to become choked with coke etc.

The Anniston stills have a cyclone on the vacuum line from the receivers.

Early experience at Newport brought out the danger of leakage of water or steam from jacketed lines, and showed the need for some means of determining the level of material in the still receivers. The temperature spread in the heating coil naturally depends on the rate of circulation of the liquid, and, therefore, is not in itself a fully reliable warning of the end of the distillation; the delivery capacity of the pump falls as the charge in the still diminishes. Directing the return stream of liquor up through the bottom of the still appears to be bad practice, also the sucking out of the coil at the end of a run is more difficult. Aroclor 5460 tended to block the vacuum connections from the still receivers.

A closed feeder for the lime to the vacuum still was installed at Newport, to save losing the vacuum between batches, except when the still had to be emptied.

Treatment of Distilled Aroclors

Exclusion of atmospheric moisture is naturally important where high electrical resistivity is required, and, of course, the Attapulgus earth needs to be well dried.

The earth treatment diminishes the "inorganic chloride" content of the Aroclor. Schwarting and others, in the Process report of October 15, 1953, page 6, state that 10 - 20 pounds of Attapulgus earth in a batch (of 12,000 lbs.) of Pyranol, will reduce the chloride figure from 0.3 ppm to 0.1 ppm. A larger amount of earth may be useful in some cases, but a badly contaminated batch would be returned to the air-blowing stage and then redistilled.

VII. COMMENTS ON THE PROCESS, contd.

Treatment of Distilled Aroclors, contd.

Mr. Ellenburg, December 1949, said that finished Aroclors should not be maintained above 80°C., especially in the presence of iron.

The earth treatment may be omitted if the products are not required for electrical purposes.

MCL have investigated the use of alternative materials in place of Attapulgus earth, for example, activated alumina, acid-treated Fuller's earth, and KN4 grade of Fuller's earth, but have not found anything to equal the Attapulgus earth either in respect of removal of chlorides (and moisture), or in respect of speed of filtration.

MCL Research Progress Reports 1151 DF 53/86/4 February 1954
DF 53/97/6 March 1954
1152 DF 53/85/7 December 1953
NR 53/103/12 July 1954 etc.
deal with this question. The removal of tin-tetraphenyl by earth treatment of Pyroclors was confirmed.

The importance of thorough drying of the Attapulgus earth was confirmed at Newport. The steam-heated dryer installed there would bring the moisture content of the earth down only to about 2%, so electrical heating was installed to give a drying temperature of 250°C. The drier earth so obtained gave shorter working cycles in the treatment of the Aroclors.

Aroclor - Toluene blends.

The curves on pages VI-30 & IX-89 show the change in density to be expected from the addition of toluene, and show the range of variation which is allowable in the density of the finished blend.

Blending of Pyranols.

The necessary precautions to ensure electrical grade quality have been set out on pages VI-32 and VI-33 above, and all that need be added here is that this has been one of the most troublesome quality questions ever met at the Krummrich plant.

VII. COMMENTS ON THE PROCESS, contd.

Note on "Chloride Scavengers"

Mr. Stickley, September 1, 1953, quoting Dr. Jenkins, wrote: --

> "The use of glycidyl phenyl ether as a chloride scavenger in transformer Askarels is covered by a Monsanto patent, and one of our customers, namely, Westinghouse, has been making use of this scavenger. They did this in order to avoid payment of royalty to G. E. on G. E.'s scavenger, which is tin-tetra phenyl. I understand that G. E. has either abolished royalty payments, or so lowered them, that Westinghouse is now going back to tin-tetraphenyl --. The only reason for using glycidyl phenyl ether, is that it is more soluble in the Askarel. It is our understanding, that in practical use it isn't quite so efficient."

(There is history of actual trouble with pump blockages due to the separation of tin-tetra phenyl in the customer's plant during cold weather, and deposits of tin-tetra phenyl have several times been seen in rail tanks returned to the Krummrich plant for refilling).

Mr. Stickley adds that the glycidyl phenyl ether is supplied by Shell Petroleum Corporation, and there is difficulty in getting the ether sufficiently low in chlorides. The ether is made via chlorhydrin. A possible alternative discussed at MCL R & D meeting, October 1949, was dibutyl diphenyl tin, but that material may be covered by the General Electric patents.

See also the discussion of the action of scavengers under "Excess Inorganic Chlorides", page 12-, Section VII, below.

Note on the use of tri-tetra chlorobenzene.

Mr. Stickley, February 28, 1953, quoting Dr. Jenkins, wrote: --

> "--- the use of tri-tetra chlorobenzene was a G. E. development. MCC make the blend, for G. E. licencees and use a tri-tetra blend supplied by Hooker. Most of G. E.'s licencees are expected to go over to the new formulation containing 45% Aroclor 1260 and 55% tri-tetra chlorobenzene mixture."

VII. COMMENTS ON THE PROCESS, contd.

Stabilizers

MCC Central Research report 543, "Studies on Light Stability of the Electrical Resistivity of Aroclor 1254" White and Ruehrwein, deals with this question, incidentally discussing the solubility of oxygen in Aroclors.

MCL Research Progress report 1151, DF 53/86/1, September 1953, deals with the solubility, in Aroclor, of certain anthraquinone derivatives which had been proposed for use as stabilizers.

About the end of 1951 MCL were asked if they had a method of estimating beta-chlor anthraquinone in Pyranols, and enquiry revealed that stabilizers are sometimes added by the users, to Aroclor 1252 used in capacitors (see letters Ellenburg to Mather, December 28, 1951, and Mather to Quartly, January 8, 1952).

Rosalla J. Ulm, of Dayton, in a letter to R. J. Good, March 12, 1951, discussed the isolation of substituted anthraquinones by chromatographic methods, and their identification by ultraviolet spectroscopy.

Quality of the Raw Materials.

See Section IX for comments on Raw Material Quality.

Quality of the Finished Products.

The finished products have at times been unsatisfactory for different reasons, as set out below: ---

1. Excess Moisture

Possible causes are (a) imperfect drying of the blowing air (activated alumina not correctly regenerated);

(b) imperfect drying of the Attapulgus earth;

(c) entry of moisture from rain, or moist air, especially during sampling. Batches may be blown with dry air (not above 80°C. for the lower Aroclors) to remove excess moisture.

VII. COMMENTS ON THE PROCESS, contd.

Quality of the Finished Products, contd.

2. Excess Colour.

Around 1950-51 the distilled Aroclors at the Krummrich plant sometimes showed yellow to green colours. In the running-in period at Newport, also, there was trouble with colour; the distillate might be brownish or pale green. The green colour was not removed by re-distillation, even in glass. The origin of the colour was never ascertained, though at Newport the occurence of colour appeared to coincide with leakages of water into the distillation system.

Details of construction of the still -- direction of entry of the return stream from the heating coil -- use of Hagen separator or cyclone on the vapour exit, also may play a part -- also the frequency of tapping the still bottoms. Air leak through the bottom cock on the still may be harmful.

Very high vacuum may actually be disadvantageous because of the very high linear velocity of the vapours which results. Note also that a tubular condenser, once fouled, may not quickly clear itself in use. Re-starting after a long shut-down may give colour trouble for a time.

Mr. Ellenberg, December 21, 1951, called attention to the fact that Ferric chloride has an appreciable vapour pressure at the temperature of the still and that the (dry) hydrated lime may not be efficient in "fixing" the ferric chloride. It has also been suggested that copper or nickel salts from non-ferrous metals in the turnings used in the chlorinators might cause colour. MCL Research Report 416A, 52/1/1, January 17, 1952 indicated that lime treatment would remove the green colour.

Dr. Jenkins, January 8, 1952 mentioned that Aroclors sometimes contained traces of chlorophenols. Possibly air in the chlorine may favour the formation of phenols. Mr. Ellenberg, December 21, 1951, suggested that the chlorine gas may carry other impurities. See Anniston report 2673 in the list at the end of this section. Diphenyl sometimes shows a yellow or pinkish colour, but colourless Aroclor has been made from such Diphenyl, and it is not thought that the colour of the Aroclors is in any way related to that of the Diphenyl.

VII. COMMENTS ON THE PROCESS, contd.

Quality of the Finished Products, contd.

3. Partial Crystallization, (possibly after admixture with solvents).

At Anniston, the trouble with crystallization in Aroclor 5460 is attributed to the presence of diphenyl in the Santowax used. This may come about by cross-contamination in the Aroclor plant, as well as by incomplete fractionation in the diphenyl still. Attention to this point has been more effective than dropping the setting point of the Aroclor.

The setting points are now in the range 98.5° to 105½°C., instead of 100 to 105½°C. as formerly. There are some indications that Santowax from "low-conversion" runs of the diphenyl units, will tend to give crystallization in the 5460.

MCL research reports under job 1152, August - October 1953, etc. cover work done in the blending of 1221 and 1242 to give material which would match 1232, but which would have less tendency to crystallize, also work on the blending of Aroclors with materials thought likely to diminish the tendency of the Aroclors to deposit crystals. A blend of two Aroclors to make a third may match on density etc. but still be different in other respects, as discussed in Section II above.

Complaints of deposition of solid in solutions of Anniston Aroclor 5460 in white spirit were ascribed to the presence of hexachloro 1,4 -diphenyl benzene. See also MCC reports 2492 "Formation of precipitate in organic solutions of Aroclors", May 1950, and 2272 "Solubility of Aroclors 5460 in various solvents", Wade, December 1948, (detailed in the list at the end of this section). 1248 has also shown incipient crystallization.

An acetone solubility test was developed, to detect the tendency to crystallize, but it was not completely satisfactory, though it was improved by the use of the technique of seeding the test solution with a little of the insoluble product. See MCL Research Report 416 A, 52/1/4, April 11, 1952 etc.

VII. COMMENTS ON THE PROCESS, contd.

Quality of the Finished Products, contd.

3. Partial Crystallization, contd.

Refrigeration to remove the crystallizable materials has been proposed, but not put into effect. A suggestion was made that the insoluble matter might be tin-tetra phenyl, but though this might occur in some cases, the usual cause is the presence of sparingly soluble chlor-diphenyls etc. as discussed above.

MCL Research Progress Report 1151, NR 53/97/8, June 1954 reports determinations of solubility of TTP in 1248 and suggests that TCB increases the solubility.

Various aspects of the occurrence of this partial crystallization are discussed in MCL research reports under Job 416 A.

High diphenyl content of the diphenyl highboilers appeared to be the cause of trouble in 4465 and 5460. The crystals occurred in the low and the high boiling fraction of 4465.

The crystals depositing in 1232 appeared to be 4-4' dichlorodiphenyl.

Processing 4465 to a lower softening point helped a little.

4. Excess Inorganic Chlorides

A sample of the Aroclor being tested is washed with hot water, the water extract clarified by centrifugation in a laboratory machine, washed with ether, then tested with acidified silver nitrate solution. Any turbidity produced, in a Tyndall beam, is ascribed to "inorganic chlorides" in the product. The turbidity is matched by use of standard NaCl dilutions, and expressed as parts chloride per million on the original sample.

Another portion of the Aroclor is heated with a strip of aluminum foil for 6 hours at about 210°C. under a condenser, cooled,

VII. COMMENTS ON THE PROCESS, contd.

Quality of the Finished Products, contd.

4. Excess Inorganic Chlorides, contd.

and the chloride estimation repeated, care being taken to side-track anything which has collected on the walls of the condenser. Details of the test methods are given in Section IX, below.

These tests arise from the use of the Aroclors in electrical equipment. The first chloride test covers possible corrosive effects of the impurities in the Aroclor as received, and the second one is intended to cover stability of the Aroclor, and the development of corrosive materials when the Aroclor is kept in contact with metals at temperatures liable to arise in everyday electrical use.

The material collecting in the condenser in the second test is side-tracked because the General Electric Company distinguish between "volatile" and"non-volatile" chlorides. The volatile chlorides are supposed to go off from the transformers, etc. in which Pyranol is used, without corroding the equipment, but the non-volatile chloride figure is supposed to represent the amount of corrosion to be expected. It would appear that in the test any reflux stream would tend to carry the "volatile" chlorides back into the test flask, confusing them with the "non-volatile".

See also the notes on the test method itself, Section IX, pages 77-, and 181-.

Dr. Munch and Mr. Ashworth at St. Louis have developed a test in which the "HCl" liberated during the heating is continuously aspirated over into silver-nitrate solution, a method which would appear to give a better measure of the stability of the material.

There is a long history of trouble with the very stringent specification laid down by users in respects of the chlorides tests on Aroclors for the electrical industry.

VII. COMMENTS ON THE PROCESS, contd.

Quality of the Finished Products, contd.

4. Excess Inorganic Chlorides, contd.

In the first place, quite special precautions have to be observed in taking samples and in making the tests, especially in factory conditions where the air may contain, for example, traces of HCl. Tanks should not be opened for sampling etc. when HCl fumes are being released nearby.

Mr. Clegorn of Anniston has developed a satisfactory method of taking samples from rail cars and factory tanks. New, dry, sample bottles, not cleaned or washed in any way, are used, and plunged well below the surface of the liquid to be sampled, and washed out at least three times with the material, blown out with clean dry air or nitrogen, then filled and closed. This procedure can make the difference between acceptance, or rejection of a batch.

Aroclor stored in sunlight in a colourless glass bottle will drop in resistivity, and will begin to smell of HCl. Amber coloured glass bottles are better.

Laboratory arrangements necessary for the chloride test are described in Section IX, below.

The direct test, for chlorides in the sample before heating, will estimate free HCl and metallic chlorides such as might result from corrosion, say of the still condensers. If chlorides get through to the "clean" side of the filter, they may be very troublesome. Air-blowing of the warm material under warm conditions, will bring the HCl down to a low limit, and reaction with tin-tetra phenyl or glycidyl phenyl ether will remove traces of HCl.

If the T.T.P. reacts to precipitate the chlorine as $SnCl_4$, which is then removed by filtration with Attapulgus earth, then its effect is understandable, but since the tetraphenyl is used in excess, there would appear to be a chance of the formation of materials such as $SnClPh_3$ which would carry through into the finished product, and so spoil the chloride test. Gradual

VII. COMMENTS ON THE PROCESS, contd.

Quality of the Finished Products, contd.

4. Excess Inorganic Chlorides, contd.

addition of the tin-tetra phenyl might precipitate all the reactive chlorine as $SnCl_4$, so preventing the formation of the hypothetical $SnClPh_3$ etc. In practice, the tetra phenyl is the last ingredient to go into the blender, so it may be that the first portion of it to enter the Pyranol picks up all the chlorides.

Treatment with Attapulgus earth is the standard method of improving batches which fail to pass the inorganic chlorides test, but sometimes quite heavy treatment is needed.

If blends such as Pyranols 1467 and 1470, which are required to contain Tin-tetraphenyl, are heavily treated with earth, a good deal of the T.T.P. will be removed by the earth, and the amount so lost will have to be made up again.

Possible causes of high "direct" chloride test are:

1. Contamination of the system with metallic chlorides already mentioned.

2. Insufficient removal of HCl by air-blowing.

3. Insufficient amount of lime used in the distillation.

4. Entrance of HCl from the factory atmosphere.

5. Entrance of $CaCl_2$ from the air breather on the tank vent.

6. Moisture (in the Arochlor or Pyranol or in the Attapulgus Earth) will delay the removal of HCl by air-blowing, and will promote the formation of chlorides which will remain in the batch.

7. High chloride content of the chloride "scavenger" used.

VII. COMMENTS ON THE PROCESS, contd.

Quality of the Finished Products, contd.

4. Excess Inorganic Chlorides, contd.

General Electric (1950) reported that they could improve the electric properties of Pyranol 1467 by a comparatively light treatment with heat-treated Attapulgus earth without a significant change in the T.T.P. content of the material. The Krummrich plant have been unable to duplicate this result in the plant or in the laboratory. Possibly some chlorides are more easily removed than others.

A batch of 45,000 lbs. of Pyranol 1467 made at the Krummrich plant early in 1951 was found to test 0.15 ppm chlorides, and was, therefore, returned from the rail car to the plant for re-treatment. By this time it showed 0.8 ppm. It was treated with small amounts of Attapulgus earth, and filtered through the Sweetland press, but with little effect. The amount of earth was increased, two treatments finally being given, each with 600 lbs. of earth. The total amount of earth used was 1400 lbs. The first 600 lb. treatment diminished the chlorides to 0.2 ppm, and the second to 0.10 ppm. By this time, most of the T.T.P. had also disappeared.

Instability of the Aroclors, as indicated by the inorganic chlorides test after the sample has been heated in the corrosion test, presumably arises from the presence of traces of moderately stable organic chlorine compounds, rather than from any instability of the chlorodiphenyls themselves, since good batches show really high stability. Ring substitution of chlorine for hydrogen in the diphenyl nucleus should yield stable compounds; the unstable impurities may be ring addition compounds, or they may be side-chain substitution products derived from traces of toluene etc. in the benzene used for diphenyl. The crude diphenyl also contains traces of styrene, and no doubt other hydrocarbons, which may form unstable chlorine derivatives.

Little exact information on these points appears to be available, and it might be thought that the unstable materials would be destroyed during the prolonged heating of the charges in the vacuum stills, except for any which might distill over in the early part of the batch.

VII. COMMENTS ON THE PROCESS, contd.

Quality of the Finished Products, contd.

4. Excess Inorganic Chlorides, contd.

Similar considerations apply to tri- and tetra- chlorobenzenes. Trouble with Pyranols in 1947 was traced to the instability of trichlorbenzene due to the use of too little catalyst in the manufacture of the TCB, resulting presumably in the formation of small amounts of ring addition products.

Trouble also arose from the presence of "chlorides" in the purchased tin-tetra phenyl. Some deliveries contained as much as 500 ppm, and MCC found it expedient to purify the T.T.P. as discussed in Section IX, pages 4-.

Trouble with Inerteen in 1953 was ascribed to instability of the glycidyl phenyl ether used.

5. Unsatisfactory Electrical Properties.

Moisture and inorganic chlorides naturally are objectionable, and it has been stated that storage of Aroclors in steel containers will harm the electrical properties (Anniston report 2771, in the list on page VII-25). The choice of containers is discussed in Anniston report 2772. See also MCL research progress reports 1151 51/141/ 24 etc. 1953 and 416A 52/1/8, October 1952 (Aroclor 1254 not discoloured in plain steel, 1 week at 60°C.). Packages are discussed on pages 34 and 35 of Section VI, above.

Report 1151 RR 53/57/2, May 1953, shows very little deterioration in electrical properties under severe conditions of exposure to steel, and even less with aluminium.

Mixtures containing TCB or TTCB have a more powerful solvent effect than straight Aroclors, and this has caused trouble, for instance, in cases where jointing materials, Glyptal "dopes" on pipe joints, etc. have been attacked in the customer's plant, with detriment to the electrical properties of the Pyranol.

VII. COMMENTS ON THE PROCESS, contd.

Uses for the Products; Applipation Work.

The MCC Bulletins "Physical Properties of Aroclors" and P-115, "The Aroclors, Physical Properties and Suggested Applications", mention the following possible uses: --

Adhesives (adhere to metals) -- low vapour pressures advantageous.

Electrical fluids and solids.

Expansion media in instruments.

Hydraulic fluids; high densities are a favourable feature. Working fluids for die casting (see the special bulletin P-137).

Lubricants in high pressure work -- use in air compressors, because non-combustible (See the special bulletin P-128).

Lubricants for plug cocks on chlorine lines. (MCL Res. Prog. Rpt. 291 51/92/1, April 1951).

Using in cutting oils.

Heat transfer media (See also the special bulletin P-130).

Plasticisers in PVC, nitrocellulose, chlorinated rubber etc. -- can be used to "extend" more costly plasticisers. (Special bulletins).

Water-proofing paper.

Vehicle for pigments in decorations of fired glass articles.

Use in paints, lacquers (flame resistance), but note possibility of toxic vapours). Good penetration of stucco and road surfaces (traffic paints).

(See Phys. & Chem. Data, Section III, above, for references to lists of compatibilities of materials with Aroclors.)

VII. COMMENTS ON THE PROCESS, contd.

Uses for the Products ; Application Work, contd.

Extender for the costly Carnauba wax in polishes.

Prevent sticking of drawers in furniture.

Wood treatment (along with pentachlorophenol).

For many of these uses, gaskets and packing materials, which are resistant to Aroclors are needed. On this question see Section XII, page 2, below.

See also MCL research reports, 1152 DF 54/16/7 and 8, July and August 1954.

Aroclor 2565 has been used to make non-flame roof sheeting, (Anniston visit report 1947, page 3).

Properties favouring the use of Aroclors are:

1. The wide range of properties available.
2. Low vapour pressure.
3. Very low corrosive effect.
4. Compatibility with numerous materials.
5. Chemical inertness and thermal stability.
6. Waterproof.
7. Non-inflammable.
8. The solid Aroclors are odourless and tasteless.
9. Good electrical properties.

On the other hand, the Aroclors are not quite above question on toxicity, and they have a very high viscosity index, i. e. a large drop in viscosity with a small rise in temperature. Viscosity

VII. COMMENTS ON THE PROCESS, contd.

Uses for the Products; Application Work, contd.

index improvers have been suggested, but their use may harm the products in other ways. On this point see MCL Research Progress reports, 1151, D 54/26/5, July 1954, etc.

The following reports deal with electrical uses of the Aroclor group.

MCC Bulletin "Askarels", July 24, 1953, use in transformers etc.

MCL Research Progress Reports 1151 RR 53/58/3 June 1953, and 1151 DF 54/26/13, Feb.1955	Material for publications on application in capacitors etc.
347 52/47/1, Sept. 1952, and the corresponding Final Report, Mar.3/53	Literature survey on electrical uses

Final report 347D, June 1953, Methods of testing dielectrical properties.

Research Progress report 347 51/141/17, November 1952, "Electrical properties of Montars," and 1151 DF 53/86/4, February 1954 "Montars in junction boxes."

Work on uses in hydraulic fluids is described in MCL Research Progress Reports.

1151 FR 52/71/3 etc.	Testing in die casting Machines
DF 53/34/4 May 1953 etc.	Pump tests, burn points
DF 54/26/5 etc. July 1954	Autogenous ignition V. I. improvers

See also reports under Job 454 E in 1952.

VII. COMMENTS ON THE PROCESS, contd.

Uses for the Products; Application Work, contd.

Work on uses as plasticizers is described in: --

MCL Research Progress Report 347 51/59/1 etc. "Aroclors in PVC".

Work on uses in paints, and polishes etc. is described in: --

1152 DF 53/89/5 May 1954, 1242 improves solvent polishes
FR 52/90/6 Jan.1953, No gloss imparted to silicate paints
DF 54/27/1 etc. Apr. 1954 Montars in paints for pipe lines, and battery boxes.

(Montars raise the softening point of asphalts).

Agricultural uses:

Animal repellant. See effect on vegetation; toxicity.
The use with Lindane etc. was discussed in correspondence, January 26, 1955.

Miscellaneous Uses:

Use as rust inhibitor is described in DF 54/26/11, January 1955.

Use in lithographic inks has been suggested in correspondence, (May 3, 1954).

Aroclors also have possibilities as chemical intermediates.

See MCL Res. Prog. Rpt. 347D - 51/141/ June 1952 etc. for nitrated Aroclors.

The MCL Progress Research Department report for November 1952, page 4, mentions the preparation of Aroclor derivatives for trial as insecticides.

Report 1161 RR 54/77/1, November 1954, discusses the hydrolysis of Aroclors to hydroxydiphenyl.

Mr. Stark of the Krummrich plant, September 24, 1954, suggested that Montars would make a good sealing compound to cover the cast lead which holds the carbon anodes in Hooker chlorine cells.

VII. COMMENTS ON THE PROCESS, contd.

Patent Position.

Lyles, Soffranko and Becker, November 6, 1946, page 167, state: --

> "In 1935 the Swann Chemical Company was acquired by Monsanto, which gave the latter access to the patent rights for the production of Aroclors".

MCL Research and Development Dept. report for August 1949, page 26, states that the market for Aroclors in the electrical industry was largely dominated (in Britain) by B.T.H. patents up to about 1946. These patents had run out, but the B.T.H. still held protection for the use of tin-tetraphenyl. MCL would try to come to a license arrangement with B.T.H.

The September 1949 report, page 27, stated that B.T.H. were willing to grant MCL a license, under their patents, to sell Aroclor compositions containing tin-tetraphenyl, for use in transformers. B.T.H. needed the British source of supply.

The October report, page 30, indicated that discussions were still in progress with B.T.H. Meanwhile the Anniston alternative material, dibutyl diphenyl tin would be considered, although it probably comes under the B.T.H. patents.

See also the discussion under "Stabilizers", page VII-9, above.

VII. COMMENTS ON THE PROCESS, contd.

List of Reports relating to Aroclors and Pyranols

	PROCESS REPORTS	Date Copies sent to MCL
1.	Dept. 246; Aroclor Process Lyles Soffranko and Becker November 1946	April 26, 1947 and October 24, 1950
2.	Notes on No. 1 above. E. Mather April 1947	April 25, 1947
3.	Notes on Anniston Aroclor Plant Havercroft and Mather September 1947.	October 7, 1947
4.	Dept. A-246; Pyranol Process Lyles Soffranko and Miller March 24, 1947	October 15, 1947
5.	Process Description for Aroclors, Dept. 246 Schwarting, Neff and Graves November 1953	March 26, 1954
6.	Revised version of No. 5 above Schwarting and Schwartz March 15, 1955	May 17, 1955
7.	Process Description for Pyranols and Inerteens Schwarting, Neff & Graves October 1953	
8.	Aroclors Operating Instructions Dept. 246 Ainsley and Schwartz August 10, 1954	May 23, 1955

VII. COMMENTS ON THE PROCESS, contd.

List of Reports, contd.

		Date Copies sent to MCL
9.	Anniston Equipment list for Aroclors, Ellenburg	June 7, 1946
10.	Notes on a Visit to the Anniston Plant, Harden August 5, 1950	
11.	Notes on a Visit to the Anniston Plant, Pemberton January 22/ 1951	
12.	Notes on a Visit to the Anniston Plant, Mather March 29, 1955	March 29, 1955
13.	Anniston Process Improvement reports	routine
14.	Anniston Monthly Operation Reports	routine
15.	Anniston Plant Investigation Reports	routine
16.	Krummrich plant Monthly Operation Reports	routine
17.	Krummrich Plant Investigation Reports	routine

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 3-10-53 Material: Aroclors, Pyranols, Inerteens, and Tri-Tetrachlorobenzene Blends Method No.10,123-53

By: AEB, NPA: SMK Test: Flash and Fire (Burn) Point WGK Dept. No.246/233

Introduction:

The flash and fire (burn) point of Aroclors, Pyranols, Inerteens, and Tri-Tetrachlorobenzene Blend shall be determined in the Cleveland Open Cup Tester, following the procedure (ASTM Designation D92-46) described in 1949 Book of ASTM Standards, part 5, p. 716-718.

For our purposes, however, it is not usually necessary to make the complete test (Complete Procedure) as given below (A). On routine sample of Pyranols 1476 and 1482 merely use the abbreviated test (Abbreviated Procedure) for Fire (Burn) Point described under (B) at the end of the method.

A. Complete Procedure:

1. Fill the cup to about 3/8" from the top with the sample.

2. Insert a 20-760°F. range ASTM Open Flash (1 inch immersion) thermometer so that the bulb is 1/4" off the bottom and midway between the center and the back edge.

3. Adjust the flame so that the rise of the sample's temperature will be at the rate of 9 - 11°F. per minute.

4. Adjust the testing flame so that it will be produced through a blow-pipe with an opening 1/32". This flame should be only a bead 3/32" in diameter.

   CAUTION: The test should be carried out under a hood where there are no drafts. This is very important as is the rate of temperature rise.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Flash and Fire (Burn) Point Test for Aroclors, Pyranols, Inerteens, and Tri-Tetrachlorobenzene Blends, Method No. 10,123-53, contd.

A. Complete Procedure, contd.

5. When visible vapors start to rise from the product under the test carry across the surface of material the small bead flame (Step 4). The test flame should never be allowed to get any lower than a height of the top rim of the cup.

6. Carry the flame directly across the product, as in Step 5, every five degrees Fahrenheit until the rising vapors flash back to the material under the test.

7. Record the temperature at this time as the Flash Point.

8. Continue to pass the flame bead across the sample (Step 5) ever five degrees Fahrenheit until the vapors flash down to the material and continue to burn for at least 5 seconds.

9. Record this temperature as the Fire (Burn) Point.

   NOTE: Always read the temperature before testing as the flash tends to increase the temperature.

10. Convert the reading to centigrade units. Report the Flash and Fire (Burn) Points to the nearest 1°C.

B. Abbreviated Procedure:

1. Fill the cup with the sample to within 3/8" of the top and heat to boiling under a well ventilated hood.

2. As the material comes to a boil pass a test flame (see Step 4, Complete Procedure), across the top of the cup (Step 5, Complete Procedure).

3. Note if the sample burns for five seconds or more.

4. If the material does not burn for 5 seconds, report the Fire (Burn) Point as > B.P. (above boiling point).

5. If a positive result (the sample burns for 5 seconds or more) was obtained, a test for Flash and Fire (Burn) Point should be run on the same material using the procedure described above under "Complete Procedure".

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 2-26-53 Material: Aroclors, Pyranols, Inerteens, and Tri-Tetrachloro-benzene Blends Method No. 10,124-53

By: AEB,NPA:SMK Test: Iron (as Fe) WGK Dept.No.233/246

Iron is determined in Aroclors, Pyranols, Inerteens, and Tri-Tetrachlorobenzene Blend on occasional samples only.

1. Weigh on a beam balance a 50 (± 0.05) g. sample in a casserole.

2. Add 20 ml. C. P. H_2SO_4 and digest the mixture on the hot plate.

3. Ignite the carbonaceous mass in the muffle at 800°C. for 1 hour, burning off the carbon residue.

4. Add to the residue 1-3 ml. H_2SO_4 and warm to dissolve the iron.

5. Heat up the sample until SO_3 fumes will appear, to insure complete solution of Iron in the ignited sample.

6. Cool the sample and dilute it to 100 ml.

7. Transfer an aliquot to a 100-ml. low form Nessler tube.

8. Add 3 drops H_2O_2, 10 ml. N NH_4SCN, dilute with distilled water to 100 ml. and mix thoroughly.

9. Compare the intensity of the color with that of a standard containing the same quantities of H_2SO_4, H_2O_2 and NH_4SCN. Follow the procedure described in General Method No. 8 (Method No. 10,199). Add enough Standard Fe solution to match the color of the sample.

Calculation:

$$\% \text{ Iron (Fe)} = \frac{(\text{ml. Std. Fe solution} \times 0.000025) \times 100}{\text{Weight of Sample (Step 1)} \times \frac{\text{Aliquot}}{100}}$$

Report the results to the nearest 0.0001%

MONS 046209

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 2/26/53 Material: Aroclors, Pyranols Inerteens, and Tri-Tetrachlorobenzene Blends Method No. 10,125-53

By: AEB, NPA SMK Test: Refractive Index WGK Dept. No. 233/246

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1. Determine refractive index of Aroclors, Pyranols, Inerteens, and Tri-Tetrachlorobenzene Blend according to General Method No. 28 (Method No. 11,784) with the following modifications:

2. Take refractive index at 25°C.

3. Clean the Abbe prisms by flushing with Trichlorobenzene. Then follow with benzene and "Mersol" washes.

NOTE:

DO NOT TOUCH THE POLISHED SURFACES OF THE ABBE PRISMS!

Report Refractive Index

$$(N_d^{25})$$

to the nearest 0.0001 unit.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 3-9-53 Material: Aroclors, Pyranols Inerteens, Tri-Tetra-chlorobenzene Blend Method No. 10,126-53

By: SE,NPA:SMK Test: Corrosion and Chemical Stability WGK Dept. No. 233/246

Procedure:

1. Roll a rectangular (2" x 4") piece of aluminum foil so that it will pass through a ☒ 24/40 joint of the corrosion test flask.

   CAUTION: Be careful that after rolling the specimen does not touch itself at any point.

2. Wash the aluminum foil (Step 1) scrupulously with acetone, distilled water, acetone, benzene, and chloride-free ether.

3. Then place the foil on a clean watch-glass and dry in an oven at 110°C. for 30 minutes. After cleaning handle the specimen with tongs or forceps only.

4. Weigh accurately on an analytical balance the specimen (Step 3) at room temperature.

5. Drop the weighed aluminum foil into the chloride-free corrosion flask of the "G. E. Corrosion Apparatus". Rinse out flask with sample and rinse end of condenser with sample.

6. Add 200 ml. of the product under test to the aluminum foil (Step 4 and 5).

7. Set the corrosion flask in the corrosion test apparatus.

8. Attach a 12-inch straight-tube air-cooled condenser, the outside of which is painted with aluminum.

9. Cover the exposed part of the flask with aluminum foil.

10. Heat the flask for 6 (± 0.1) hours at 210 (± 5)°C.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Corrosion and Chemical Stability on Aroclors, Pyranols, Inerteens, Tri-Tetrachlorobenzene Blends, Method No. 10,126-53, contd.

11. At the end of the heating period, detach condenser from the flask before removing it from the hot plate.

12. Remove the flask from the hot plate and cover all of the flask with aluminum foil (when the flask is not on the hot plate).

13. Without removing the aluminum foil covering of the flask, analyze the product (Step 6) remaining in the corrosion apparatus for:
    a. Appearance, Color, and Condition - Use Method No. 10,084
    b. Inorganic (Free) Chlorides - Apply Method No. 10,118
    c. Acidity (Acid Number) - Follow Method No. 10,087

14. With a pair of clean, straight nichrome tongs remove the aluminum foil specimen (Step 5), wash thoroughly, dry and weigh accurately on an analytical balance in the same manner as before (Steps 2, 3, and 4).

Report the corrosion as loss or gain in weight to the nearest 0.0001 g. and the Chemical Stability as indicated by the analysis of the products "After Corrosion Test", in the same way as reported for the original (as received) material.

Apparatus:

G. E. Corrosion Apparatus consists of the following:

a) A Corrosion Flask - It is a 300-ml. Pyrex flask with a 19 g.g. 24/40 joint equipped with a 12-inch straight tube as an air-cooled condenser. The air-condenser is painted on the outside with aluminum.

b) The Corrosion Apparatus: A transite box 32" long x 8" wide x 5" deep. The top of the box represents a split transite board with holes cut to fit the flasks. The box is heated by two 500-watt, 15 volt G.E. Strip heaters with off-set terminals at one end (23.5" overall length). The heating length of the heating element is covered by a copper strip 19-1/2" long x 4" wide x 1/4" thick.

The temperature is controlled by an automatic thermostat with temperature setting indicator.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 2-26-53 Material: Aroclors(Crystalline) Method No. 10,127-53

By:EAB,NPA:SMK Test: Hold(Crystallizing)Point WGK Dept. No. 246

Introduction:

The cyrstallizing point of the crystalline Aroclors is defined as the highest temperature noted after crystallization begins when the sample is cooled slowly in an insulated container.

This is also referred to as the "Hold Point".

Procedure:

1. Fill a 1" x 8" test tube to within 2 inches of the top with molten Aroclor and heat sufficiently to insure complete fluidity.

2. Insert an 0-360°C. range, 3-inch immersion, thermometer through a cork and place in position in the center of the tube with the lowest part of the mercury bulb about 1" from the bottom of the test tube.

3. Place the test tube in a suitable Dewar flask (tube form-evacuated).

4. Allow the sample to cool to about 3°C. below the expected crystallizing point without stirring. After the temperature is 3 to 5°C. below the expected crystallizing point (material supercooled 3-5°C.) start to stir the Aroclor carefully with the thermometer and note the highest temperature rise of the Aroclor. This point is taken as the Hold (Crystallizing)Point.

NOTE: If a material of unknown quality is to be tested and the approximate cyratallizing point is not known, a preliminary test is made in order to determine at what temperature the stirring of material should start.

CAUTION: The material must not be supercooled more than 3 - 5°C. below the expected crystallizing point.

Duplicate tests should check within 1°C.

Report the Crystallizing (Hold) point to the nearest 1°C.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 4-5-55 Material: Aroclor-Toluene Mixtures Method No. 10,194-55

by: WWK:WJG Test: Composition WGK Dept. No. 246

Introduction:

Certain customers require a mixture of 90% by weight of Aroclor (1254, 1260, or 1262) with 10% Toluene. A rapid method of determining whether or not the required amount of Toluene is present in the sample of the mixture has been devised and is described below.

Procedure:

1. Examine the sample bottle label to ascertain whether the mix is a 1254, 1260, or 1262 - Toluene mix, and the Lot number of the Aroclor used.

2. From the supervisor's file obtain the specific gravity of the Aroclor used in the mix.

3. Warm the sample to about 50°C. by placing it next to a hot plate.

4. Pour the sample into a clean, dry hydrometer jar and determine the specific gravity at 40/15.5°C. according to General Method No. 13 (Method No. 10,497).

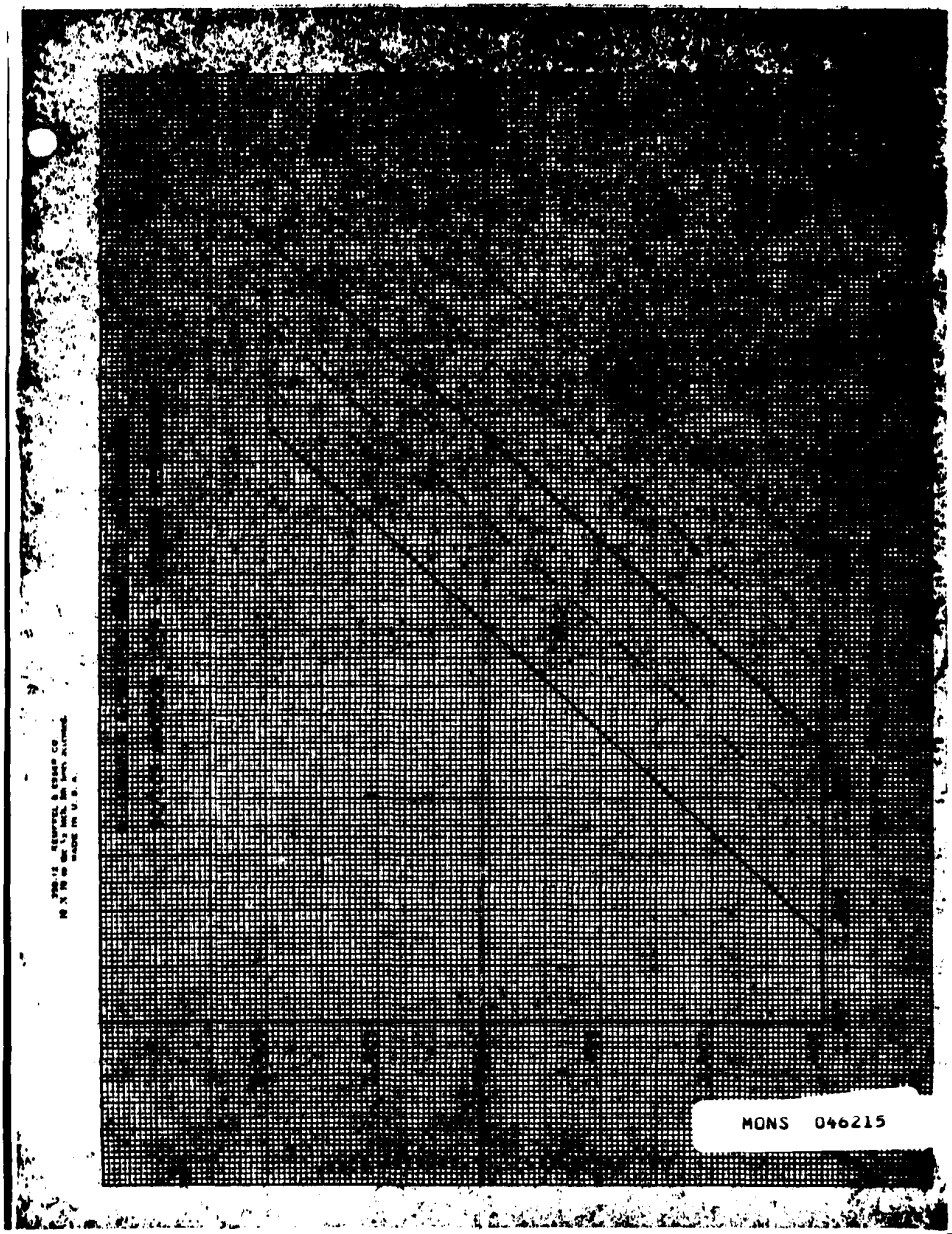
   NOTE: The gravity must be taken at exactly 40°C. since no gravity-temperature coefficients are available for these mixes.

5. Use a 1.400 - 1.600 hydrometer.

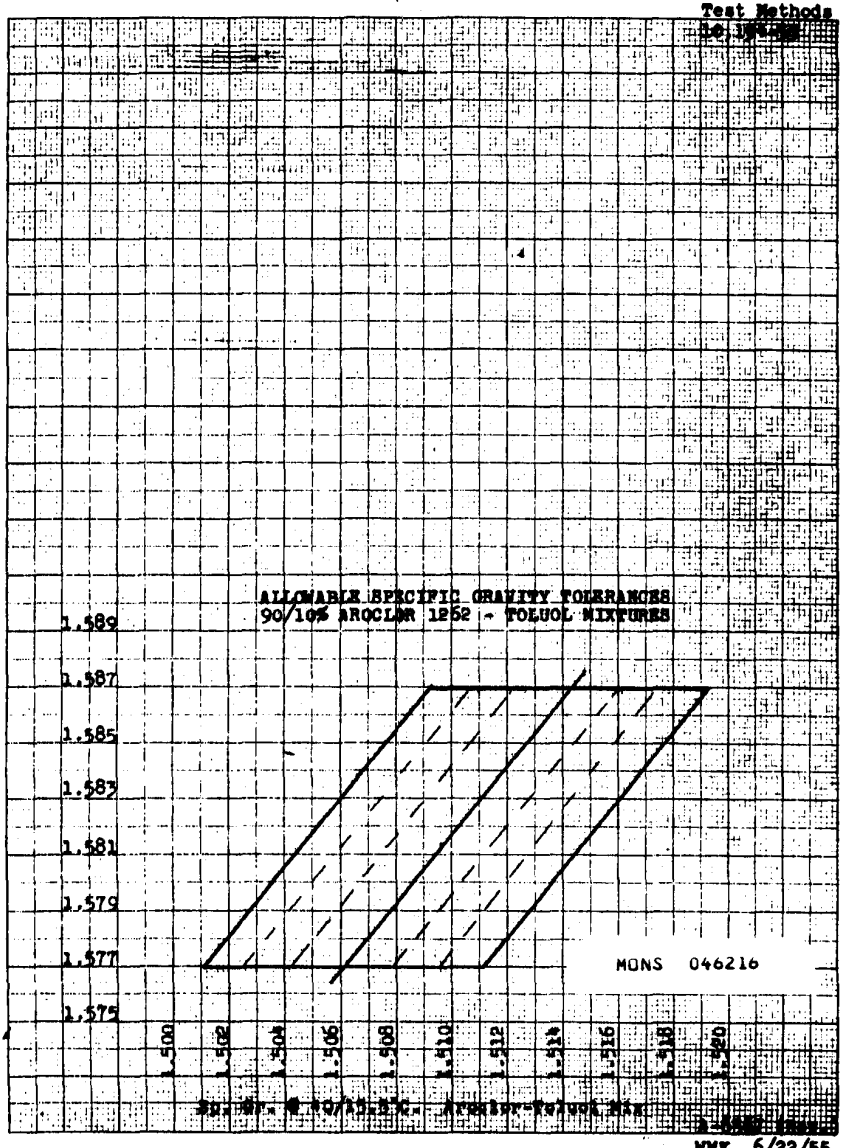
6. The gravity of the mix is the abscissa and the gravity of the Aroclor used in the mix the ordinate on the following graphs on pages IX-89, IX-90, and IX-90a.

7. Using the appropriate chart (TS-A-139 for 1254, A-11144 for 1260, or A-6567 for 1262) determine the point where the abscissa and ordinate intersect on the graph. If this point falls within the shaded portion of the chart, the mixture contains 9.5 - 10.5% Toluene and is acceptable. If the point falls outside this area, the sample is rejected.

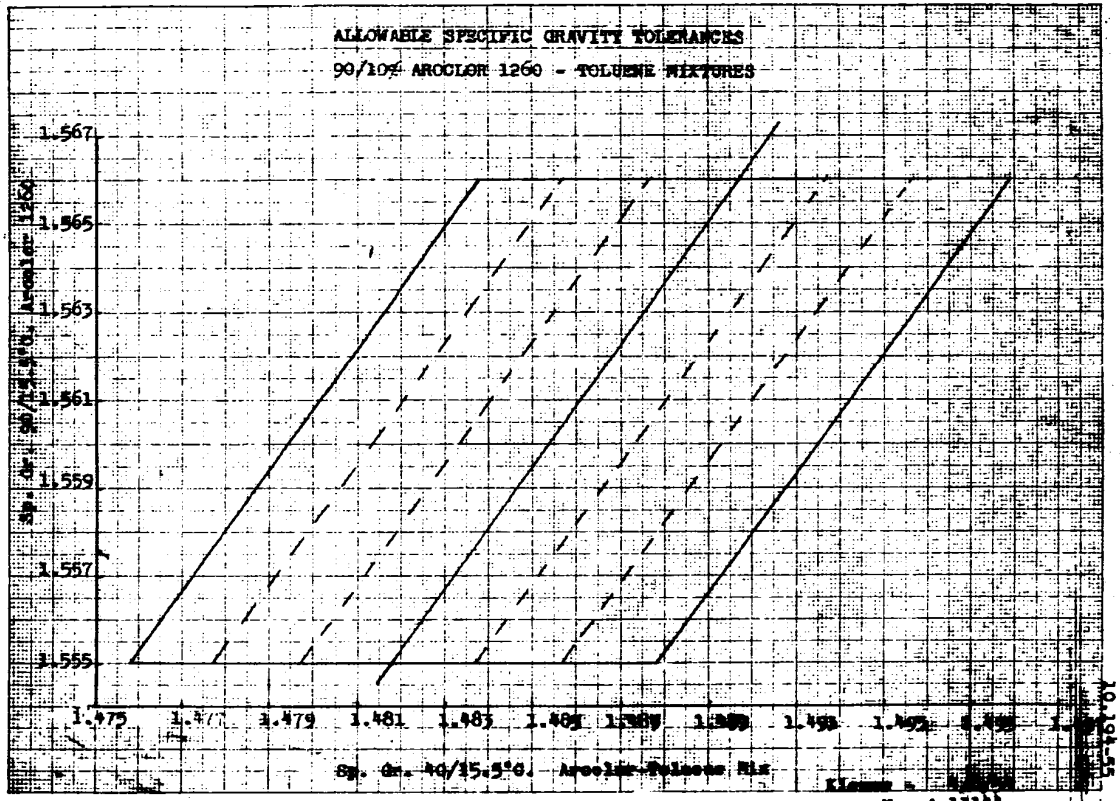
Report only the specific gravity of the mix at 40/15.5°C. and whether or not the material passes the specification limits. Report the gravity to the nearest 0.001 unit.



MONS 046215


ALLOWABLE SPECIFIC GRAVITY TOLERANCES
90/10% AROCLOR 1252 - TOLUOL MIXTURES
1.589
1.587
1.585
1.583
1.581
1.579
1.577
1.575
1.500
1.502
1.504
1.506
1.508
1.510
1.512
1.514
1.516
1.518
1.520
MONS 046216

WWK 6/22/55


ALLOWABLE SPECIFIC GRAVITY TOLERANCES
90/10% AROCLOR 1260 - TOLUENE MIXTURES
1.567
1.565
1.563
1.561
1.559
1.557
1.555
1.475
1.479
1.481
Sp. Gr. 40/15.5°C. Aroclor-Toluene Mix

MONS 046217

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 8-25-54 Material: General Method Method No. 10,199-54

By: AEB,NPA:WJG Test: Iron (Fe) Traces (Colorimetric) WGK G.M. No. 8

Introduction:

Quite frequently iron is present in Monsanto products as an impurity to an extent of from 0.0001 to approximately 0.02%. In these instances it is advisable to test for iron colorimetrically if interfering ions are absent. This method determines only ferric iron. It is based on the fact that ferric iron and ammonium thiocyanate in an acid solution give a red color, the intensity of which is proportional to the quantity of iron present.

Important: Silver, copper, cobalt and mercuric chloride interfere with iron determination. Nitric Acid also gives a color that may be mistaken for iron.

Procedure:

Carry out preparation of the sample in a clean 100 ml. low-form Nessler tube which has previously been rinsed with C.P. HCl.

If the material to be tested is a solid:

1. Weigh accurately on an analytical balance a 1 to 10 grams (more for extremely low Fe content).

2. Dissolve the sample in dilute H_2SO_4.

3. Oxidize the iron by adding 3 - 4 drops of 3% H_2O_2 solution.

If the material to be tested is a liquid:

1. Acidify the sample with C.P. H_2SO_4 or C. P. HCl.

2. Oxidize the iron by adding 3 - 4 drops of 3% H_2O_2 solution.

The procedure is continued for solid and liquid material in the following way: --

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Iron (Fe) Traces (Colorimetric), Method No. 10,199-54, contd.

A. Add to the prepared sample (Step 3 - solid material: Step 2 - liquid product) 10 ml. N NH_4SCN solution and make up the 100-ml. mark.

B. Prepare "blank" in a second Nessler tube of the same type. Use the same quantities of acid and reagents as used in the sample.

C. Add to the "blank" standard Fe solution until the tints of the two tubes match. Record the number ml. of Standard Fe solution required to make the match.

Calculations: $\% \text{ Iron (as Fe)} = \frac{\text{ml. Std. Fe Sol'n.} \times 0.000025 \times 100}{\text{Weight of Sample}}$

The test will easily detect 0.00001% Fe.

Important: In the event that a sample contains sufficient iron to require a titration of more than 10 ml. Standard Iron (Fe) solution, the prepared sample should be diluted to a larger volume (500 or 1000 ml.) and the iron (Fe) should be determined on a suitable aliquot.

Reagents Used:

N NH_4SCN Solution: Dissolve 76.1 g. NH_4SCN in distilled water and dilute to one liter.

3% H_2O_2 Solution: - U.S.P.

Iron Stock Solution: Add to 0.2500 gm. pure iron wire* 10 ml. C. P. HCl, 25 ml. distilled water, and 2 drops Bromine water. Cover and place on steam bath to effect solution. When iron is completely dissolved, dilute to one liter. This solution contains 0.00025 g.Fe/ml.

Standard Iron (Fe) Solution: Pipet 100 ml. of Iron Stock Solution into a liter volumetric flask and dilute to the mark with distilled water. Mix well. This solution contains 0.000025 g. Fe/ml.

* Clean wire with emery cloth to remove any oxide and wipe clean before weighing.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 7-23-52 Material: Biphenyl (Diphenyl) Method No. 10,252-52

By: AEB,NPA:SMK Test: Appearance and Colour WGK Dept. No. 246

Biphenyl (Diphenyl: Xenene) is a light yellow crystalline solid. The melt of this material is a straw coloured liquid. It should be clear and free of residue.

NOTE:

Biphenyl is not manufactured at WGK Plant but it is purchased from Anniston or outside suppliers for use in production of various biphenyl derivatives.

1. Examine the sample and describe its appearance and colour noting any unusual characteristics. Report as light yellow crystalline solid, or otherwise if appropriate.

2. Examine the sample in molten and solid state for residue (visible foreign bodies) such as rust, foreign particles, etc. Report observations as none, very slight, slight, moderate, or considerable. Describe appearance of impurities briefly.

NOTE:

Any off colour dirty gray or deep brown material or material which contains more than a slight amount of residue in the melt is to be rejected.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 7-23-52 Material: Biphenyl (Diphenyl) Method No. 10,298-52

By: AEB, NPA Test: Crystallizing Point WGK Dept. No. 246
SMK

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1. Melt completely the sample. Avoid overheating.

2. Fill a 1" x 8" test tube to within one inch of the top with the molten material.

3. Place the test tube in a Dewar vacuum jacketed tube.

4. Insert a 55° - 90°C. range, 3" immersion thermometer and a plungertype metal stirrer.

5. Cool the material with constant stirring to crystallization. After slight supercooling of the molten sample, the temperature will rise sharply and remain at equilibrium for a few minutes.

6. Record the highest point of the temperature rise as the crystallizing point of the material.

The method is precise to $\pm$ 0.1°C.

Report the results to the nearest 0.1°C.

The crystallizing point of this material is approximately 68 - 69°C.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 7-24-52 Material: Biphenyl (Diphenyl) Method No. 10,299-52

By: AEB,NPA: WGK Dept. No. 246
SMK Test: Distilling Range:

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1. Determine distilling range of Biphenyl in the manner described in General Method No. 1 (Method No. 10,006) with the following changes:

2. Measure 100 ml. of the molten sample into an asbestos covered benzene distilling flask.

3. Use a 230° - 265°C. 3" immersion thermometer and set up the distillation apparatus using a transite board having a two inch opening.

4. Use an air cooled condenser. Be sure to warm up the condenser before the first drop to prevent crystallization of the distillate.

Report the following results along with the complete distillation to the nearest 0.1°C.

First Drop

Dry Point

100% Range (First Drop to Dry Point)

The results are precise to ± 0.1°C.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 11-17-53	Material: General Method	Method No. 10,497-53
By:LG,DKL,NPA:SMK	Test: Specific Gravity by Hydrometer	WGK G.M. No. 13

1. Rinse each piece of equipment with a portion of the sample.

2. Fill a hydrometer jar with the well-mixed liquid or molten sample near the specified temperature to a height sufficient to float a specific gravity hydrometer of appropriate range.

   IMPORTANT: Pour the sample into the cylinder (jar) without splashing, so as to avoid formation of air bubles. Remove any air bubbles adhering to the surface by touching them with a glass stirring rod. Select a location for the test that is free from air currents. Place the jar on a level surface.

3. Insert a 0 to 110°C. range thermometer and a hydrometer of suitable range.

4. Holding the thermometer and hydrometer together in the hand, stir the contents of the cylinder (Jar), being careful to avoid formation of air bubbles. Stir until a uniform temperature is reached throughout the liquid. Use a vertical stroke which will prevent thermal stratification.

5. If the Specific Gravity coefficient is known, record this temperature (Step 4) and withdraw the thermometer. If this coefficient is not known heat or cool the liquid under the test to the specified temperature. In either case, wipe the stem of the hydrometer, allow it to settle back gently into the liquid and to come to rest floating freely away from the walls of the cylinder.

6. Read the Specific Gravity as the point at which the surface of the sample apparently cuts the hydrometer scale. Record the reading after applying the hydrometer correction (if any).

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Specific Gravity by Hydrometer, Method No.10,497-53 contd.

IMPORTANT: Make this observation by placing the eye slightly below the level of the liquid and slowly raise the eye until the surface of the sample first seen as a distorted ellipse seems to become a straight line (lower meniscus) cutting the hydrometer scale. The reading should not be taken until the liquid and hydrometer are free from air bubbles and are at rest.

7. If the specific gravity coefficient is not known, record the value obtained in Step 6. If the specific gravity coefficient is known take the numerical difference between the specified and the observed temperature.

Calculations:

CAUTION: These calculations apply only to those hydrometers whose specific gravity scales are numerically higher at the bottom of the scale than at the top of the scale.

Specific Gravity temperature correction =

(Difference between the specified and the observed temperature) x Specific Gravity Coefficient

Apply the specific-gravity correction as follows:

a. If the observed temperature is above the specified temperature:

Sp.Gr. at specified temperature = Sp.Gr. at observed temp.+ Sp. Gr. temp. correction.

b. If the observed temperature is below the specified temperature:

Sp.Gr. at specified temperature = Sp.Gr. at observed temperature - Sp.Gr.temperature correction

Report the specific gravity at the specified temperature to the nearest 0.001 units.

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Specific Gravity by Hydrometer, General Method 10,497-53 contd.

The method is precise to ca. ± 0.001 units.

Material specifications often specify different temperatures at which specific gravity shall be measured. For example:

Specific Gravity 15.5/15.5°C.
Specific Gravity 25/15.5°C.
Specific Gravity 60/60°F.
Specific Gravity 20/4°C.

The meaning of the expression given in this example can be easily seen from the following definition of specific gravity and of density:

A Specific Gravity is the ratio of the weight (in air) of a given volume of the material at a stated temperature to the weight (in air) of an equal volume of gas-free distilled water taken as a standard at a stated temperature.

Density: The weight of any substance per unit volume at any definite temperature.

Then in the expression "Specific Gravity 25/15.5°C. the upper temperature is referred to the material under the test and the lower one to the distilled water of corresponding density.

It should be noted that the specific gravities at x/x°C. and at x/y°C. are inversly proportional to the absolute densities of water at x°C. and y°C.

NOTE: Absolute densities of water at different temperatures are tabulated in Lang's Handbook of Chemistry; page 1221, 8th Edition, 1952.

If it is desirable, therefore, to report the specific gravity at x/y°C. instead of x/x°C., the following calculation is sufficient:

$$\text{Specific Gravity } x/y\,^{\circ}C. = (\text{Sp.Gr. } x/x\,^{\circ}C.) \times \frac{\text{density of } H_2O \text{ at } x\,^{\circ}C.}{\text{density of } H_2O \text{ at } y\,^{\circ}C.}$$

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 6-7-54 Material: Aroclor 1270 Method No. 10,510-54

By: AEB, NPA: Test: Foreign Odour
WKG W.G.K. Dept. No. 246

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1. Place approximately 2 or 3 grams of the material in a large metal spoon.

2. Apply heat to the bottom of the spoon for about 5 seconds with a Bunsen flame.

3. Upon removing the spoon from the flame dense white fumes are given off. The fumes should have the odour of burnt rubber and should be free of sharp, penetrating odour of the lower chlorinated Aroclors.

4. Material showing sharp odours will be rejected.

Report the Foreign Odour according to the General Method No. 5 (Method No. 10,060) definitions of odours (none, very faint, faint, distinct, or strong).

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 6-7-54 Material: Aroclors 1270 and 1271 Method No.10,511-54

By: AEB, NPA
WJG Test: Colour of 5% Toluene Sol'n. WGK Dept. No. 246

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1. Weigh accurately on an analytical balance a 2.5 (± 0.0100) g. sample of Aroclor.

2. Transfer to a 250-ml. beaker containing 50 ml. of filtered toluene.

3. Place on a hotplate and stir until all the Aroclor has dissolved.

   Do not overheat the solution.

4. Transfer the hot solution to a warm 50-ml. tall form Nessler tube.

5. Determine colour of the material according to General Method No. 2 (Method No. 10,007).

The method is precise to ± 5 units.

Report the APHA Colour to the nearest 5 units.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 6-18-54 Material: Aroclor 1271 Method No. 10,576-54

By: AEB, NPA
WJG Test: Appearance and Colour WGK Dept. No. 246

Aroclor 1271 (Decachlorodiphenyl) is usually a fine, practically white, fluffy powder. Normally the material is pure white, but on standing in the presence of small amount of moisture if may take on a very pale blue cast. The material is a redistilled product, and the best material is that which is free of jagged crystals when examined under a microscope.

1. Examine the sample and describe its appearance noting any unusual characteristics.

   Report as fine, white, fluffy powder or otherwise if appropriate.

2. Observe the sample for visible foreign bodies (residue), such as rust, wood, lint, black specks etc.

   Report observations as none, very slight, slight, moderate, or considerable.

   Describe appearance of impurities briefly.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 6-18-54 Material: Aroclor 1271 Method No. 10,577-54

By: AEB, NPA:
WJG Test: %Moisture (H_2O) WGK Dept. No. 246

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1. Tare accurately on an analytical balance a wide mouth drying bottle. Record tare.

2. Weigh accurately on the same balance a 10(± 0.0005) g. sample into the tared bottle. Record weight of the sample.

3. Place the bottle in an oven at 120°C. for 24 hours.

4. Cool the sample in a desiccator.

5. Reweigh the bottle. Record loss in weight of the sample. Calculate percent moisture.

Calculation:

$$\% \text{ Moisture } (H_2O) = \frac{\text{Loss in weight (Step 5) x 100}}{\text{wt. of Sample (Step 2)}}$$

The method is precise to ± 0.01%

Report the result to the nearest 0.01%.

IX. SPECIFICATIONS AND TEST METHODS, cont'd.

Date: 3-26-52 Material: Aroclor 1271 Method No. 10,578-52

By: RK,WJG:SMK Test: Melting Point WGK Dept. No. 246

1. Fill a 400 ml. tall-form beaker with Dow Corning Fluid.

2. Fill one capillary melting point tube to a height of 3/4" with sample and another with the standard Aroclor 1271. Attach both capillary tubes to a 300-335°C. 5" immersion thermometer with two short lengths of thin wire, adjusting the lower end of the tubes level with the mercury bulb.

3. Immerse the assembly in the bath and clamp the thermometer stem in position 3/4" from the side of the beaker. (A desk lamp placed near the bath will facilitate observations). Adjust the initial bath temperature to 250-60°C.

4. After the apparatus is in position, heat at such a rate that the bath temperature will increase 0.5 ± 0.1°/min. The rate of rise in temperature is important and strict observance of the above limits is necessary.

5. Record the temperature at which the first meniscus appears and the temperature of complete melting of both the sample tested and the du Pont standard. The first meniscus is taken as the temperature at which the first liquid meniscus in the tube is observed. The complete melt is taken as the temperature at which all crystals have disappeared.

The method is precise to ± 0.2°C.

Report the melting point (M.P.) (first meniscus) and the complete melt (C.M.) on both the standard and sample tested to the nearest 0.10.°C.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 3-4-41 Material: Aroclor 1271 Method No. 10,579-41

By: AEB,MJW Test: 5% Toluol Solution Color WGK Dept. No. 246

Weigh 2.50 grams of sample and transfer to a 250 ml. beaker containing 50 ml. of filtered toluol. Place on a hot plate and stir until all the Aroclor has dissolved. Now transfer the hot solution to a warm 50 ml.tall form Nessler tube and compare the colour with APHA Standards contained in similar tubes, employing the procedure described in General Method 2 (10,007).

Report the APHA Colour to the nearest 5 units.

The method is accurate to ± 5 units.

Date: 6-24-52 Material: Aroclor 1271 Method No. 10-580-52

By:AEB,WJG:SMK Test: Crystal Structure WGK Dept. No. 246

1. Spread a small amount of the sample on a glass slide and place it on the stage of a microscope which magnifies from 75 to 300 diameters.

2. When the sample has been brought into the field and is in focus, slowly move the slide from side to side and observe the kinds of crystals present and the proportion of each.

3. Estimate roughly the percent of jagged crystals.

Report the amount of sharp edged crystals to the nearest 5%.

Two analysts observing the same material will usually agree within 10%.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 3-9-53 Material: Aroclors, Pyranols Inerteens, Tri-Tetra-chlorbenzene blends Method No. 10,620-53

By:SE,NPA:SMK WGK Dept.No.233/246

Test: Moisture (H_2O Water Content)

Introduction:

Because of the varying solubility of Aroclors, Pyranols, Inerteens, and Tri-tetrachlorobenzene Blend, different proportions of a mixed solvent are required. However, the analysis itself is the same for all listed products.

Procedure:

1. Determine moisture (water content) in Aroclors, Pyranols, Inerteens, and Tri-tetrachlorobenzene Blend using Karl Fischer (K. F.) reagent. Apply the "Dead Stop" method described in General Method No. 7, (Method No. 10,100) with the following modifications:

2. Place the solvents, in the proportions indicated below, into the titration flask:

Material	Anhydrous Benzene	Anhydrous Methanol
Pyranol 1478	0 ml.	300 ml.
Pyranol 1488	100 ml.	200 ml.
Pyranol 1467	100 ml.	200 ml.
Pyranol 1470	100 ml.	200 ml.
Pyranol 1481	100 ml.	200 ml.
Pyranol 1495	100 ml.	200 ml.
Inerteen P.O.P.O.	100 ml.	200 ml.
Tri-Tetrachlorobenzene blend	100 ml.	200 ml.
All Aroclors	110 ml.	190 ml.

3. Titrate the solvent with the K. F. reagent until it is "blanked".

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Moisture (H_2O Water Content) for Arcolors, Pyranols, Interteens, Tri-Tetrachlorobenzene Blends, Method No. 10,620-53. contd.

NOTE: The solvent is "blanked" when the tube shadow remains fully open (ca. 100°) for 30 seconds.

4. Refill the burette with K. F. reagent to the zero mark.

5. Weigh on a beam balance a 100 (± 0.05) g. sample by difference, into the "blanked" solvent and allow the material in the flask to mix well.

6. Titrate the solution with K. F. reagent to the end-point described in Step 5. Record the volume of K. F. reagent used.

Calculation:

$$\% \text{ Moisture } (H_2O)^* = \frac{\text{ml K. F. Reagent} \times H_2O \text{ factor} \times 100}{\text{Sample Weight}}$$

The method is precise to ± 5% of the water present.

Report the result to the nearest 0.0005% or equivalent p.p.m.

NOTE: 1 ppm is equivalent to 0.0001%.

* If required by a specification, report as "Water Content" in p.p.m. equivalent to percent moisture.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 4-13-53 Material: Pyranols 1488 and 1495 Method No. 10,670-53

By: DBH,NPA:SMK Test: Trichlorobenzene (TCB) WGK Dept. No. 246

Introduction:

Aroclor 1260 (G.E. Compound No. 1482) and Trichlorobenzene (TCB) (G. E. Pyranol No. 1478) are blended together to make transformer Pyranols for the General Electric Company. Two blends are made: Pyranol 1488 (60% Aroclor - 40% TCB) and Pyranol 1495 (90% Aroclor - 10% TCB).

Sixteen ounce samples of the Aroclor 1260 and the TCB used for blending in the department are brought to the laboratory for the analysis.

Procedure:

1. Tare accurately on an analytical balance two conical ground glass stoppered weighing bottles.

2. Add 10 (± 0.0005) g. of warm (60° - 70°C.) Aroclor 1260 to each bottle.

3. Cool and reweigh on the same balance.

4. Add TCB to each weighing bottle from an eyedropper until the desired composition is obtained.

   NOTE: The following proportions are used:

Pyranol No.	TCB Added Sample No. 1	Sample No. 2
1488	39%	41%
1495	9%	11%

5. Reweigh the material accurately on the same balance.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Trichlorobenzene (TCB) in Pyranols 1488 and 1495, Method No. 10,670-53, contd.

6. Put the stoppers in the weighing bottles and place them on a covered steam bath.

   Swirl the materials occasionally to facilitate thorough mixing.

7. After the blends are thoroughly mixed, determine the refractive index at 25°C. with an Abbe Refractometer according to Method No. 10,125.

8. Plot ND_{25} versus % TCB on coordinate paper and connect the two points obtained with a straight line.

9. Determine the refractive index at 25°C. of the sample under test according to Method No. 10,125.

10. Determine the percent T.C.B. present from the graph (step 8).

Report T.C.B. to the nearest 0.1%.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 5/16/51 Material: General Method Method No. 10,723-51
By: HOH,DKL:SMK Operation of Fisher
Test: Electrophotometer WGK G.M. No.18

PRECAUTIONS:

a. Remember that this is a precision instrument, and that for best results it must be handled with care. Certain adjustments have been made for optimum operation of the instrument. If in doubt as to proper adjustment of functioning of the instrument consult your supervisor.

b. Be certain that the proper filter is in the instrument before depressing the lamp button and that the filter is clean.

c. Always adjust the galvanometer needle at the beginning of a series of measurements.

d. See that the intensity control is in the proper position.

e. Use only absorption cells in the instrument. The matched pairs will be engraved with the same number by the Service Section. Be sure the cells are clean and free from fingerprints.

f. In filling the absorption cells, always add sufficient solution to bring the meniscus safely above the path of the light beam when in the operating position.

g. Always fill the cells before placing them in the adapter; never fill them while they are in the instrument.

h. Always be sure to have the label of the cells facing front (towards the operator) when placed in the adaptor.

i. Always close the compartment when making measurements or setting the initial null.

j. In changing the position of the sliding platform, push the platform gently but firmly against the proper stop. Do not slam the platform against the stop.

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Operation of Fisher Electrophotometer, Method No. 10,723-51, contd.

PRECAUTIONS: contd.

k. Do not release the lamp button during the interval required to move the platform, exchanging in the light path the reference cell and the cell containing the unknown material.

l. Do not keep the lamp button depressed longer than necessary.

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1. Place the proper filter in position.

2. Set the intensity control (lower left switch) to "ADJ. GAL" and turn the "ADJUST ZERO" knob (above the galvanometer) so that the galvanometer needle coincides exactly with the index line.

3. Set the intensity control to that position designated by the letter engraved on the handle of the filter holder.

4. Place one of the matched pair of carefully cleaned 23 mm Fisher cylindrical absorption cells containing the reference solution in the rear position in the adapter platform in such a manner that the label faces front (towards the operator) and place the other cell of the pair, containing the unknown solution in the front position in the adapter holder with its label also facing front.

5. Close the cell compartment door.

6. Pull the platform knob forward until the catch is in firm contact with the front stop in order to bring the rear absorption cell containing the reference solution into the light path.

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Operation of Fisher Electrophotometer, Method No. 10,723-51, contd.

7. Adjust the dial control to zero on Scale A. Depress the lamp button (in the center of the dial control) and after ten seconds adjust the "INITIAL NULL" knob to bring the galvanometer needle back to the index line.

8. Without releasing the lamp button, push the platform knob until the platform is firmly in contact with the rear stop to place the absorption cell with the unknown solution in the light path.

9. Still holding down the lamp button, bring the galvanometer needle back to the index line by turning the dial control.

10. Record the A-Scale or B-Scale reading as indicated in the specific method.

11. Without releasing the lamp button, recheck the initial null adjustment as before (Steps 6 and 7). If not in adjustment, repeat steps 6 thru 11 until there is no change in null adjustment.

12. Empty the cells, rinse thoroughly, fill with distilled water, and return them to the adapter.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 5/21/53 Material: Boiler Water Method No. 10,733-53

By: HOH,NPA:SMK Test: Chloride WGK Dept. No. 350

1. Filter, using Whatman No. 42 filter paper, about 125 ml. of the sample.

2. Place 100 ml. of filtered sample into a 250-ml. Erlenmeyer flask. Then add 10 ml. of 3% H_2O_2 solution and mix well.

3. Add 3 drops phenolphthalein, then follow with 0.5 N H_2SO_4 until pink color just disappears.

   NOTE: If the sample is acid to phenolphthalein, add 0.5 N NaOH until pink, then 0.5 N H_2SO_4 dropwise until the color just disappears.

4. Add 1 ml. Chromate indicator ($Na_2C_2O_4$) and titrate with standard Silver Nitrate solution (0.0005 g. Cl/ml) to the first appearance of a permanent red coloration. Record ml. $AgNO_3$ used.

5. Prepare and make a blank determination as follows:

   a. Place 100 ml. distilled water into a 250 ml. Erlenmeyer flask, add 10 ml. 3% H_2O_2 solution and mix well.

   b. Add 3 drops phenolphthalein and 0.5 N NaOH until pink.

   c. Add carefully 0.5 N H_2SO_4 until the color just disappears.

   d. Add 1 ml. Chromate indicator and titrate with standard Silver Nitrate solution to the same end-point as for the sample. Record ml. $AgNO_3$ used.

Calculation:

$$\text{Chloride (as ppm Cl)} = \left[\text{ml. } AgNO_3 \text{ for sample (Step 4)} - \text{ml. } AgNO_3 \text{ for blank (Step 5;)}\right] \times 5$$

The method is precise to ± 5 ppm.

Report the chloride to the nearest 1 ppm.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 2-27-53 Material: Aroclors, Pyranols Inerteens, TCB, and Tri-Tetrachlorobenzene Blends Method No. 11,147-53

By: SE, NPA: SMK Test: Sampling Procedure WGK Dept. No. 233/246

Samples of the listed products are taken in the following way:

A. Batch Samples (taken by men in Dept.)

Three 16-oz. wide-mouth, screw cap bottles for each batch.

B. Storage Tank (S.T.) Lot, Car and Drum-Lot (D-Lot) are sampled by the Laboratory personnel according to the following:

For S.T. Lot, Car, and D-Lot samples use only 5-pt. narrow-mouth (narrow-neck), amber coloured, Bakelite-screw-cap, bottles.

Samples to be taken as per table below:

Material	Reserve Sample	Advance Sample*
Pyranol 1467	S.T. Lot - 3 x 5-pt. Bottle	1 x 5-pt. Bottle
	Car - 2 x 5-pt. "	
	D. Lot - 2 x 5-pt. "	
Pyranol 1470	S.T.Lot - 3 x 5-pt. "	1 x 5-pt. "
	Car - 2 x 5-pt. "	
	D. Lot - 2 x 5-pt. "	
G.E.Compound No.1476 (Aroclor 1254)	S.T.Lot - 3 x 5-pt. "	1 x 5-pt. "
	Car - 2 x 5-pt. "	
	D. Lot - 2 x 5-pt. "	
Pyranol No. 1478 (Trichlorobenzene)	S.T. Lot - 3 x 5-pt. "	1 x 5-pt. "
	Car - 3 x 5-pt. "	
G.E.Compound No.1482 (Aroclor 1260)	S.T.Lot - 3 x 5-pt. "	1 x 5-pt. "
	Car - 2 x 5-pt. "	
	D. Lot - 2 x 5-pt. "	
Pyranol No. 1488 (60% Aroclor - 40%TCB	S.T.Lot - 3 x 5-pt. "	1 x 5-pt. "
	Car - 2 x 5-pt. "	
Tri-Tetrachlorobenzene Blend	Car(only) - 3 x 5-pt. "	1 x 5-pt. "
Inerteens	S.T.Lot - 3 x 5-pt. "	1 x 5-pt. "
	Car - 2 x 5-pt. "	
	D. Lot - 2 x 5 pt. "	

MONS 046240

* Advance samples to be sent on request only.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Sampling Procedure for Aroclors, Pyranols, Inerteens, TCB, and Tri-Tetrachlorobenzene Blends, Method No. 11,147-53, contd.

General Information:

1. As soon as the amber (brown) 5-pt. bottles (see "Caution" on the last page of the method) are received, the carton is opened, each bottle is capped, and the bottles are returned to the carton until needed.

2. When ready to sample, remove a bottle from the carton and wipe off with a clean, dry towel (see Note 1 at the end of method).

Procedure for Car Sampling:

1. Attach bottle to the clean stainless steel sampling rod (see Note 2 at the end of method).

NOTE: The bottle is attached firmly so that it can not swing, to one end of the rod, with the neck toward the handle of the rod.

2. After the department man has opened the car dome, remove the cap from the bottle and immediately plunge the bottle, neck up, at least 12 to 18 inches beneath the surface of the material in the dome. Allow the bottle to fill completely.

3. Withdraw the bottle and empty the contents into a bucket.

NOTE: The bucket content is later thrown away.

4. Repeat the filling of the bottle from the car and emptying into the bucket three times (Step 2 and 3).

5. Plunge the bottle again into the car, fill it completely with the material, remove from the car, pour out few milliliters of its contents to rinse the inside of the bottle cap, allowing the drippings to fall into the bucket.

6. Cap the bottle with the rinsed cap and remove from the rod, wipe clean and label.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Sampling Procedure for Arcclors, Pyranols, Inerteens, TCB, and Tri-Tetrachlorobenzene Blends, Method No. 11,147-53, contd.

Procedure for Storage Tank (S.T.) Lot Sampling:

1. Use the same procedure as described for car sampling with the following modifications:

2. Substitute by a specially designed sample holder the sampling rod used for car sampling.

3. Ascertain that a sampling line of the storage tank has been drained and thoroughly flushed with the material to be sampled.

Procedure for Drum-Lot (D-Lot) Sampling:

1. Sample directly from drums designated by the department. Take a composite sample of the lot.

2. Use for transfer of the material a syphon with a rubber bulb as an aspirator.

3. Cap the sample bottle with the rinsed cap, clean and label.

CAUTION: USE NO SUBSTITUTE BOTTLES for the standard 5-pint, narrow-neck, amber sample bottle fitted with the factory metal-lined screw cap.

NOTE: 1: In Dr. R. L. Jenkins' memo to Mr. D. L. Eynon, 9/26/47, in which Clegorn's method of sampling is included, "diaper cloth" is used. Mr. J. F. Stickley has approved the method as here modified.

NOTE: 2: In Dr. R. L. Jenkins' memo to Mr. D. L. Eynon, 9/26/47, in which Clegorn's method of sampling is included, "a clean hardwood stick about 1" x 1" x 4'" is used. Mr. J. F. Stickley has approved the method as here modified.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 2-25-54 Material: Montars Method No. 11,321-54

By:SE,NPA:SMK Test: Appearance WGK Dept. No. 246

Montar is usually brown to black lumps or chunks.

1. Examine the contents of the sample bottle and describe its appearance. Report as black chunks or otherwise if appropriate.

2. Observe the sample for visible foreign bodies, such as rust, wood, lint, etc. Report as none, very slight, slight, moderate, or considerable. Describe appearance of impurities briefly.

Date: 2-25-54 Material: Montars Method No. 11,323-54

By:SE,NPA:SMK Test: Non-Volatile Residue at 800°C. WGK Dept. No. 246

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1. Weigh accurately on an analytical balance a 5(±0.0100)g. sample in a large porcelain crucible which previously has been cleaned and heated in the muffle at 800°C., cooled in a desiccator and weighed on an analytical balance. Record weight of the sample and the tare of the crucible.

2. Smolder the sample over a burner until volatile matter is no longer given off.

3. Place the crucible in the muffle and heat at 800°C. for 45 minutes.

4. Cool the sample in a desiccator and reweigh accurately on an analytical balance. Record weight of the crucible.

Calculation:

% Non-Volatile Residue at 800°C =

$$\frac{\text{Weight of Crucible (Step 4) - Tare (Step 1)} \times 100}{\text{Weight of Sample (Step 1)}}$$

Report the result to the nearest 0.01%.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 9-21-54 Material: General Method Method No. 11,433-54

By: WMK, LJW, WJG
WWK:WJG Test: Viscosity WGK G.M. No. 25

Part A: Saybolt Universal Viscosity by Saybolt Viscosimeter.

1. Take a scrupulously clean, dry 60-ml. viscosity receiving flask from the oven and allow it to cool to room temperature while the viscosimeter is being cleaned. (Use only receiving flasks which have been standardized and found to be within ± 0.05 ml. of 60.00 ml.)

2. Clean the viscosimeter tube as follows:

   A. Insert the cork, which should be clean and in good condition, in place in the bottom of the tube.

   B. Carefully pour enough benzene into the tube so that the latter is filled and the liquid overflows into the gallery. Do not allow any benzene to spill down into the oil bath, as the vapours will affect later viscosities.

      CAUTION: While using benzene for cleaning the viscosimeter, be sure all circuits of the instrument are turned off.

   C. Using a thermometer fitted with a holder to prevent it touching the bottom of the tube, stir the mixture well so that any adhering material is dissolved. Be sure the entire inner surface of the tube and gallery is wet.

      Pull the cork to drain the tube. With a clean withdrawal tube draw the excess liquid from the gallery and discharge it directly into a beaker -- not into the viscosity tube.

   D. Repeat Steps A through C inclusive.

   E. Permit the viscosimeter tube to drain and dry. ABSOLUTELY DO NOT INTRODUCE A CLOTH OR KLEENEX INTO THE TUBE OR GALLERY FOR BLOTTING UP RESIDUAL LIQUID.

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Viscosity, Method No. 11,433-54, contd.

F. Flush ca. 80 ml. of the well-shaken (and heated if needed to insure fluidity) sample to be tested through the apparatus and follow Step C above.

G. Drain this out, and again flush with a fresh portion of sample to make certain any last drops of previous material have been rinsed out.

H. Shine a pen-light up through the orifice while looking down into the tube. The orifice should be free of fibers or any other obstruction. If these are present, they should be removed by flushing with sample only, not by use of wires. If flushing with sample fails, inform the supervisor.

ADDITIONAL PRECAUTIONS:

A. See that the bath oil, when hot, is not less than 1/4" above the level of the overflow rim of the gallery of the tube.

B. The bath oil should be a grade of light-coloured mineral oil which has a viscosity of 40 ± 5 Saybolt seconds at 210°F. and should be replaced when it displays a definite darkened colour.

C. For viscosities to be run at 100°F. the bath oil temperature shall not exceed 100.25°F (± 0.05°F. for 10 Min.). For viscosities determined at 130°F., the bath temperature shall not exceed 130.50°F. (± 0.05°F. for 10 Min.). For viscosities determined at 210°F, the bath shall not exceed 212.0°F. (± 0.10°F. for 10 Min.)

D. Use only thermometers standardized to the nearest 0.01°F. against a National Bureau of Standards thermometer.

E. Use only the stopwatch provided for timing purposes, which should be accurate to within 0.1 percent when tested over a 60 minute period. Electrical timers must not be used unless the available power source is known to be of sufficiently

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Viscosity, Method No. 11,433-54, contd.

E. Contd.
accurate frequency. The stopwatch must be left in the holder provided, since variations in its position can cause error.

F. Never use the plunger, commonly provided, for cleaning the instrument and never expose the viscosimeter to a draft during a determination.

3. Blot the cork dry with a lint-free cloth and insert it in the tube. Pour ca. 150 ml. of sample into a scrupulously clean beaker which has been rinsed with sample, and heat the contents to not over 7°F. hotter than the temperature of test. Do not use a sample which has overheated, even if it is then cooled to within the prescribed range.

4. Pour enough heated sample into the tube that it ceases to overflow into gallery. All samples must be strained through the 100 mesh screen which has been carefully flushed with sample.

5. Stir the sample with the standardized thermometer (with attached holder) until its temperature has remained constant within ± 0.02°F. of the desired temperature for one full minute (with constant stirring).

NOTE: Stirring is a very critical point, especially in the case of more viscous liquids such as Aroclor 1260. Use the exact technique described as follows:

Stir the sample with the thermometer, continuously in the same direction, at a measured rate of three revolutions per second. Alternately sweep the thermometer against the walls of the tube for five revolutions and then stir in the center of the tube for three revolutions. Do not deviate from the prescribed rate of stirring or the practice of stirring five revolutions at the walls of the tube, three revolutions at the center, five at the walls, three at the center and so on for the one-minute period specified.

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Viscosity, Method No. 11,433-54, contd.

5. contd.

Regulate the temperature of the sample while stirring, by adjusting the oil bath until the desired reading holds constant within ± 0.02°F. throughout the required interval. Do not stir by moving the thermometer up and down unless you desire to cool the sample, and under no circumstances stir in this manner during the one-minute timed interval.

6. Remove the surplus sample from the gallery, with the scrupulously clean withdrawal tube which is inserted at one point in the gallery without touching the over-flow rim, and which removes sufficient sample that the level of sample in the gallery is below the level of sample in the oil tube proper. Do not rotate the withdrawal tube around the gallery.

7. Place the receiving flask in position so that the stream of oil from the outlet tube strikes the neck of the flask.

8. Snap the cork from its position, and at the same instant start the stopwatch.

9. Stop the stopwatch at the same instant that the bottom of the meniscus of the sample reaches the mark in the neck of the receiving flask.

10. Examine the end of the cork to see if it has become moistened with sample. This would denote an incomplete seal and a premature seepage of sample though the orifice which would produce erroneous results.

The time in seconds, after applying the proper calibration correction of the tube is the Saybolt Universal Viscosity.

Different operators in different laboratories should agree within 0.5% on all readings.

Report the results to the nearest 0.1 seconds.

Reference: ASTM D-88-44

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Viscosity, Method No. 11,433-54, contd.

OPERATION OF THE TAG VISCOSIMETER

The viscosimeter is heating when the lamp is off. Do not go away and leave the "quick heat" on. The "quick heat" should be turned off when the temperature of the bath is within 1 degree of the desired bath temperature. The viscosimeter should be allowed 30 minutes to come to temperature equilibrium before making a test. This will allow the metal parts of the viscosimeter to come to temperature equilibrium with the bath. Note: Every now and then, trouble will be found in getting the bath to hold the proper temperature. This is usually due to dirty contact points. They are found in a small housing between the motor and rheostat. The points may be cleaned by taking off the housing cover and removing any carbon deposits with tissue paper. If possible, this job should be performed by Service Section personnel.

Part B: Kinematic Viscosity by Modified Ostwald Viscosimeter.

1. Clean the viscosimeter tube as follows:

   A. Connect the large side arm of the viscosimeter, by means of a piece of clean rubber tubing, to a vacuum source.

   B. Apply gentle suction.

   C. Place ca. 100 ml. of benzene or TCB in a 150-ml. beaker, place the capillary arm of the tube in the beaker, and suck the solvent through the tube.

   D. Repeat Step C using acetone.

   E. Dry the tube by sucking a gentle stream of air through it or placing in a steam cabinet.

2. Adjust the constant temperature bath to the desired temperature, holding the temperature within $\pm$ 0.05°F.

3. Place a 1 x 6 inch test tube inside a 500-ml. suction flask. Insert a medium porosity Selas glass crucible into the rubber adapter. Place the adapter in the top of the flask with the

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Viscosity, Method No. 11,433-54, contd.

3. contd.
glass tube extending into the test tube. Filter, by suction, enough of the sample to fill the 1 x 6 inch test tube ca. 3/4 full. Have the sample hot enough to be fluid.

4. Select the proper Ostwald tube series number (see chart below) and fill the tube as follows:

   A. To the large side arm of the Ostwald tube, connect a piece of clean rubber tubing which is connected to a vacuum source.

   B. Invert the tube and place the capillary side into the filtered sample contained in the test tube.

   C. Using gentle suction draw enough filtered sample to fill both bulbs and into the capillary up to the mark etched on it.

   D. After loading, the tube is turned right side up, vacuum removed, and the excess sample is wiped off the viscosimeter tube.

Modified Ostwald Viscosimeter	Approximate Viscosity Range	
	Centistokes	Saybolt Universal Seconds
Series 50	0.8 - 3	----------
Series 100	3 - 10	35 - 65
Series 200	10 - 70	60 - 325
Series 300	25 - 175	120 - 800
Series 400	120 - 850	550 -4000
Series 500	800 -5600	3600 - 25,000

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Viscosity, Method No. 11,433-54, contd.

5. Supporting the tube in a rubber stopper, immerse the viscosimeter in the constant temperature bath to such a level that the top bulb is completely immersed. Adjust the tube to a vertical position. This can be best accomplished by either visual examination or using a small plumb bob consisting of a piece of solder and a thread placed in the large side arm of the Ostwald. The plumb bob should not touch the walls of the tube.

6. Allow the sample and tube to come to temperature equilibrium in the bath. 10 minutes will be the minimum time required for this.

7. After the sample has attained bath temperature, apply vacuum to the capillary arm and draw the sample up to a point ca. 5 mm. above the mark between the bulbs.

8. By means of a stop watch, measure the time required for the meniscus to pass from the upper to the lower mark. This is efflux time, t, in seconds.

9. A check determination can be made by re-drawing the sample back above the upper mark and proceeding as in Step 8. It is not necessary to refill the viscosimeter.

10. Calculate the viscosity of the sample as follows:

$$V = CT$$

Where V = Viscosity of sample in centistokes at T°F,
C = Calibration constant for tube at T°F, and
t = Efflux time in seconds for sample at T°F.

11. A calibration constant is determined on the viscosimeter as follows:

A. Adjust the constant temperature bath to 100 $\pm$ 0.05°F.

B. For the standard oil use API or NBS oil.

C. Repeat Steps 3 through 9 above.

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Viscosity, Method No. 11,433-54, contd.

D. Using the viscosity (V), in centistokes, marked on the standard oil lable, calculate the tube constant (C) as follows:

$$C = \frac{V}{t}$$

where C = Calibration constant at 100°F.for the tube tested.
V = Viscosity of the oil in centistokes, and
t = Efflux time in seconds.

E. Determine the constant in duplicate. Duplicate efflux times should be within 0.2 seconds.

F. To determine the calibration constant on a tube at other temperatures use the following equations:

Calibration constant at 210°F.(98.89°C.) = 0.996 x calibration constant at 100°F. (37.78°C.)
Calibration constant at 130°F.(54.44°C.) = 0.999 x calibration constant at 100°F. (37.78°C.)
Calibration constant at 60°F.(15.56°C.) = 1.001 x calibration constant at 100°F. (37.78°C.)

12. To convert from kinematic viscosity in centistokes to Saybolt viscosity in Saybolt Universal Seconds consult either the attached graph B-7781* or ASTM D-446-39.

13. The following equivalents are frequently used in connection with viscosity conversions:

Poise = c.g.s. unit of absolute viscosity
Centipoise = 0.01 poise
Stoke = c.g.s. unit of kinematic viscosity
Centistoke = 0.01 stoke
Centipoise = centistokes x density (at temp. under consideration)
Reyn (1 lb. sec. per sq. in.) = 69 x 10^5 centipoises.

Reference: ASTM D-445-46-T and D-446-39.

*See page IX - 61b, above.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 9-23/52 Material: Trichlorobenzene Method No. 11,503-52

By: LDF, DKL, NPA:SMK Test: Stability WGK Dept. No. 223-246

1. Rinse a clean, dry, 300-ml. g.g.s wide-mouth Erlenmeyer flask twice with a few ml. of the Trichlorobenzene (TCB) sample. Weigh on a beam balance the flask and add 290 (± 0.05) g. of the sample. Set the dip-pipe into the flask making sure the dip-pipe is not touching the bottom of the flask.

2. Place the flask fitted with its dip-pipe in position by slipping it through the opening provided in the bath lid section and clamp firmly in place. The absorber flask is rinsed with distilled, chloride-free water, and about 20 ml. of chloride-free water is added, to seal effectively the opening of the side-arm bulb. Place rubber stopper tightly on the absorber flask. Seat absorber male joint into female joint of dip-pipe, remembering that the ground glass joint formed and the inside of its glass tube must be dry; this is accomplished by the use of a clean tissue of Kleenex. Complete the assembly by connecting the dip-pipe with the rubber tube from the bubbler.

3. The Dewar bottle space is filled with dry ice and methanol around the condenser trap. Use ca. 3" of methanol, then crushed dry ice to the top. Check the dry-ice trap before each run. If water or ice is present anywhere inside the condenser trap, clean the trap with distilled water and alcohol and place in the oven to dry before using. Always blow the dried trap with air before using.

4. Immediately turn on the air by releasing the pinch clamp which is between the dry ice trap and pressure regulator.

   CAUTION: Do not connect the sample to air line before dry ice has been added to trap, or the cooling air in the trap will suck the sample from the flask into the air line. If this should ever occur, replace the rubber tubing with new Tech. Prod. Co. amber translucent rubber tubing, 3/16" I.D. 1/16" wall -- do not attempt to clean out soiled tubing, clean out glass capillary tube with methanol, and blow dry with air, clean and dry the bubblers and put in fresh tricresyl phosphate.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Stability Test for Trichlorobenzene, Method No. 11,503-52, contd.

5. Cover the parts of the assembly (excepting the absorber bulb) that are above the bath top with aluminum foil to exclude light, which has proven detrimental. Make certain there is a water seal in the side arm bulb with a water head rising well into the bulb. This assures complete absorption of chlorides.

6. Turn on the variac operating the main bath-heat source and adjust heat to approximately 210°C. with this source alone. The stirring motor for the bath is also turned on and allowed to circulate the fluid during the heating. When the proper bath temperature has been reached, the thermo-regulator-relay-immersion heater system is plugged in and the bimetallic thermo-regulator is adjusted as follows: Loosen locking screw in the base and rotate the Bakelite head until the pointer indicates the temperature desired on the graduated scale. Relock the head by tightening this locking screw. At the instant the desired temperature is obtained, the red contact screw-governing setting should be turned so that it just touches its contact arm button. Usually only a small part of a turn of the adjuster will be sufficient.

   NOTE: This thermo-regulator is a very delicate and accurate mechanism which is easily broken if mishandled. In no case should it be removed or tampered with except for the setting instructions mentioned above. This adjustment will normally be made by the Instrument man.

7. The manometer in the air line should show a pressure differential of 24-26 mm. of mercury. Markings for the proper reading have been made on the manometer. The rate of air flow should be regulated to a value of 44 ml. air/minute by means of a rotameter applied to the outlet. This flow rate can be regulated by means of a screw clamp on the tubing before the capillaries. The bleed-off valve can also be used to make small changes. Usually, however, it should not be necessary to change the setting of the regulator.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Stability Test for Trichlorobenzene, Method No. 11,503-52, contd.

8. Clean out the mercurous sulfate electrode by allowing some solution in it to drain around the loosened glass plug. (First read instructions appearing later in this method on use of electrode, care, and sources of trouble). Occasionally (every 2 or 3 days) it is necessary to clean the silver wire electrode by dipping it in dilute nitric acid and sanding with fine sand paper.

   CAUTION: Do not use emery cloth. Emery cloth contains impurities which will affect the electrode potential.

9. Rinse off electrodes by placing a 100-ml. beaker of distilled water under electrodes and turning on agitator.

10. Before titrating the sample, run a blank on the methanol by putting 20 ml. of distilled water in a 100-ml. beaker and adding 50-75 ml. of the methanol, placing the beaker under the electrodes, turning on the agitator, and reading voltages on the previously balanced potentiometer. The voltage should be 60 millivolts, but it might be higher due to contamination. If 0.02 ml. or less of 0.005 N $AgNO_3$ reduce the potential to 60 mv. or less, and methanol is good enough to use. Discard the beaker contents.

    NOTE: If the methanol is not satisfactory, obtain a fresh supply from the solvent vault. If this fresh supply is not satisfactory, the methanol will have to be purified by distillation as described at the end of this method.

11. The length of run for each sample is two 8-hour periods. At the end of the first 8-hour period, remove the first absorber without turning off the air, and pour its contents into a 100-ml. beaker which has been cleaned by rinsing with 1:4 HNO_3, then distilled water. Rinse into beaker, adding a total of 50-75 ml. of methanol, and titrate to a potential of 60 mv., using 0.005 N. $AgNO_3$.

12. The absorber must be rinsed well with distilled water, 20 ml. of water then being added to the flask, and water removed from joints with tissue, as before. The absorber is then placed back on the dip-pipe for the second 8 hour period.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Stability Test for Trichlorobenzene, Method No. 11,503-52, contd.

13. The contents of the other absorbers are then treated as described in Step 11.

14. At the end of the second 8 hour period, repeat Step 11 for each absorber.

15. At the end of the stability test, allow flasks to drain free of bath fluid and wipe remainder free with Kleenex, empty the flasks, rinse with alcohol, flush with distilled water and dry in oven. Do not put foils in flasks. Also clean the dip-pipes with alcohol and distilled water and place in the oven to dry. The Dow Corning fluid level should be maintained 1 inch from the top of the bath.

    During any run where only one or two flasks and assemblies are in use, the remaining positions are occupied by dummy flasks to maintain the proper bath level.

Calculation:

Parts per million chlorides = ml. 0.005 N $AgNO_3$ x 0.61

In the range 0.1 to 0.5 ppm agreement between duplicate samples must be 100% of one another. In the range 0.6 to 1.0, the agreement must be 50% of one another.

Report results to the nearest 0.1 ppm.

NOTE: If the chlorides on batch samples are greater than 1.0 ppm determine also Stability by Method No. 10,126 (G.E.) on the sample filtered through Attapulgus earth.

Preparation of Solution and Apparatus:

0.005 N $AgNO_3$: The silver nitrate solution is made up by adding 50.00 ml. 0.01 N $AgNO_3$ from a 50-ml. volumetric flask (painted black), to a 100-ml. volumetric flask (also painted), and diluting to the mark with distilled water. This solution is prepared by the Analytical Section.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Stability Test for Trichlorobenzene, Method No. 11,503-52, contd.

Checking the electrode system:

Standard chloride solution may be prepared to check the electrode system in case the cell exhibits abnormal behavior.

To make 1.00 ppm standard, add 1.49 ml. of 0.55 N HCl to a 1000-ml. volumetric flask and dilute to the mark. Dilute a 10-ml. aliquot with 50-75 ml. of methanol in a 100-ml. beaker, and titrate with 0.005 N $AgNO_3$. Calculate the parts per million chlorides indicated by the titration and compare with the standard value.

Fresh standards should be prepared from time to time. Calculations for 1.00 ppm std: ml. $AgNO_3$ req'd. = $\frac{1.00}{0.61}$ ppm = 1.64 ml.

Use of mercurous sulfate electrode:

1. Do not place electrode too near the heating bath. Evaporation changes the normality of the K_2SO_4 solution.

2. Before refilling electrode with potassium sulfate from the volumetric flask, pour a small amount over the lip to avoid contamination.

3. Remove all air bubbles from electrode arm by closing stop-cock, tightening rubber stopper, and turning upside down, allowing glass plug to slide down arm of electrode, forcing the bubble to the surface. Fill arm with potassium sulfate solution from eyedropper.

4. Always close stopcock before removing rubber stopper. Otherwise the suction created will draw air bubbles into the electric arm.

5. Before using the electrode, loosen glass plug and allow some solution to drain out. Sometimes potassium sulfate crystallizes around the plug, preventing good electrical contact. Remove rubber stopper before loosening plug to allow solution to drain freely.

6. Make certain mercury in outer arm is in contact with platinum wire which connects outer arm with inner electrode system.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Stability Test for Trichlorobenzene, Method No. 11,503-52, contd.

7. Always keep stop-cock open while electrode is being used.

8. Always keep rubber stopper in place when making a run, and never leave stopper off very long. The atmosphere may contaminate the electrode.

9. Always leave electrode tip in a beaker of distilled water when not in use.

NOTE: A second electrode has been prepared in case the first one should be broken. This electrode is kept in a labeled box in the Aroclor Room. It is advisable to drain out the K_2SO_4 solution and refill the new electrode with fresh solution before using.

Preparation of a New Mercurous Sulfate Electrode:

Before filling the new electrode, clean thoroughly with dilute nitric acid and rinse with distilled water followed by potassium sulfate solution.

Reagent grade mercury is added to outer arm with an eyedropper. Make sure the mercury is in contact with platinum wire at the bottom of arm.

Punch a hole in a rubber policeman and fit over arm of electrode. Insert a piece of clean copper wire about 3 inches long. One lead wire from potentiometer is connected to wire. Other lead wire is connected to silver metal electrode.

Add enough reagent grade mercury to inner electrode bulb to cover the platinum wire. Add about 1/4 inch of mercurous sulfate, reagent grade, which has previously been washed with normal potassium sulfate solution. Fill the electrode with normal potassium sulfate.

CAUTION: Mercuric sulfate is a poison to the electrode. Mercuric salts can be reduced by leaving in prolonged contact with mercury and K_2SO_4 solution.

Methanol Still: Using methanol from the can, fill a large round-bottom flask 3/4 full. Add ca. 5 grams Sodium Hydroxide pellets.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Stability Test for Trichlorobenzene, Method No. 11,503-52, contd.

Methanol Still: contd.

Use a distilling column packed with glass helices and a water-jacketed condenser. All joints are glass-to-glass. Do not use rubber. A bulb-shaped funnel on receiver end of condenser protects methanol from air contamination. The funnel fits into a two-hole cork stopper which fits into a receiver bottle. The glass tube from the other cork stopper hole leads into an empty trap which is connected to a "breather" bottle containing caustic solution.

Set heating mantle under flask. Discard first 100 ml. distillate. Run a blank test on the next 50 ml. of methanol; 0.02 ml. or less of 0.005 N $AgNO_3$ should be required to reach a potential of 60 mv.

Leave a sizeable heel in the distilling flask (300 - 400 ml.)

Fill receiver bottle close to neck, as bad air in the bottle will spoil the methanol. Use either a receiver bottle with a screw cap or a glass jointed cap. It has been found necessary to add one drop of very dilute nitric acid (about 0.01 N) to each bottle of methanol to give sharper breaks at the end point of the titration.

When bottle is full, cover cap with aluminum foil.

When bottle of distilled methanol is put into use, use siphon arrangement on shelf in Aroclor Room. The system consists of two empty traps with "breather" bottle between containing alcoholic caustic to absorb chlorides and TCB vapors. It is necessary to occasionally refill the alcoholic caustic bottle with fresh solution. If good distilled methanol is not available, undistilled reagent-grade methanol from Mallinckrodt Chemical Company can be used. The methanol must be tested before use, however.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Stability Test for Trichlorobenzene, Method No. 11,503-52, contd.

A. Bath
B. Stirring Motor
C. Main Heat Variac, 20 amp. cap.
D. Dewar flask containing cold trap.
E. Thermoregulator
F. Bubbler (Air)
G. Flask, 300 ml.
H. Dip Pipe
I. Absorber Bulb
J. Relay (Aminco # 4-37 OB)
K. Reserve D-C Fluid
L. Mercury Manometer
M. Clamp adjustment for air flow.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 5/24/54 Material: Aroclors: 1142, 1148 Method No.11,532-54

By:LDF,NPA,GWM:
WJG Test: Acid Number WGK Dept. No. 246

This method is used for all dark-coloured Aroclors (mainly crude Aroclors).

1. Weigh accurately on a beam balance a 10(± 0.05)g. sample by difference into a clean, dry 400-ml. beaker. Record weight of the sample.

2. Add 100 ml. of Carbon Tetrachloride (CCl_4) and 100 ml. distilled water, previously just neutralized with 0.01 N KOH, using Phenol Red indicator.

3. Stir the solution with a glass rod until all the Aroclor is in solution. Transfer the contents of the beaker to a 250-ml. separatory funnel. Shake and allow the layers to separate.

4. Draw off the lower (Aroclor-CCl_4) layer into another 250-ml. separatory funnel, and the water layer (containing free acid) into a clean, dry 400-ml. beaker.

5. Add another 100 ml. of neutralized (as above Step 2) distilled water to the second separatory funnel, shake and collect the water layer in the same beaker as before (Steps 3 and 4). Combine water extracts.

6. Titrate the combined water extracts with 0.01 N KOH solution to Phenol Red end-point. Record ml. KOH solution used.

CALCULATION:

$$\text{Acid Number (mg. KOH/gram of sample)} = \frac{\text{ml. 0.01 N KOH (Step 6)} \times 0.56}{\text{Sample Weight (Step 1)}}$$

Upon request only, convert Acid Number to mg.NaOH/g. in the following way:

mg. NaOH/gram = mg. KOH/gram x 0.715

Report the results to the nearest 0.001 if they are below 0.1, otherwise to the nearest 0.01.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 2-26-53 Material: Pyranol 1467 and 1470 Method No.11,533-53

By:Se,NPA:SMK Test: Tin Tetraphenyl WGK Dept. No. 246

CAUTION: This method is not applicable to product containing more than 0.0035% water. Determine results on two separately weighed samples. Blow the funnel with dry air 10 minutes.

Procedure:

1. Weigh on a beam balance by difference a 15 (± 0.05) gram sample (approx. 10 ml.) at 25°C. from a 10-ml. graduate into a 125-ml. separatory funnel. Bubble dry air through the sample 10 minutes.

2. Bubble dry HCl through a glass tube of small bore into the sample for 10 minutes. Occasionally rotate the separatory funnel so that any spatter drops are washed down.

   NOTE: A tube drawn to an almost capillary tip is desirable for the bubbling.

3. Leave the capillary tip in the funnel and bubble dry air through the sample for 30 min. to sweep out excess HCl. Dry the air over Drierite. Occasionally rotate the separatory funnel so that any spatter drops are washed down. Check for complete removal of HCl by odour. Do not remove air line from funnel.

4. Pipet 25 ml. of distilled water into the funnel, allow to blow for 1 - 2 minutes and then withdraw air line.

5. Stopper funnel and shake vigorously for 2 - 3 minutes.

6. Allow separation of layers and decant bulk of water layer into a clean, dry 100-ml. beaker.

7. Pipet into the funnel another 25-ml. portion of distilled water, shake vigorously as before, allow to separate, discard the Pyranol layer and combine the water extracts.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Tin Tetraphenyl in Pyranol 1467 and 1470, Method No.11,533-53 contd.

8. Mix the water extracts and pipet a 20-ml. portion into a 250-ml. Erlenmeyer Flask.

9. Add 50 ml. distilled water and 3-5 drops of alcoholic phenolphthalein indicator solution (1 g. indicator to 100 ml. 95% ethanol) and titrate with 0.01 N NaOH until pink colour remains for 5 - 10 seconds.

10. Determine a blank titration with 0.01 N NaOH on 70 ml. of distilled water.

NOTE: The distilled water used for the blank and samples must be taken from the same containers.

Calculation:

$$\% \text{ Tin Tetraphenyl} = \frac{(\text{ml. NaOH-Step 9} \times \text{Normality}) - (\text{ml. NaOH-Step 10} \times \text{Normality}) \times 40 \times 2.5}{\text{Sample Weight (Step 1)} \times \text{Empirical Factor (last page of method)}}$$

Report the result to the nearest 0.001%

Determination of Empirical Factor for Tin Tetraphenyl Analysis in Pyranols 1467 and 1470.

Introduction:

In the process of blending Pyranol 1467 (or Pyranol 1470), the manufacturing department will send a 1/2-gallon sample of Pyranol 1488 (or Tri-Tetrachlorobenzene Blend-Aroclor 1260 mixture in the case of Pyranol 1470) and a sample of Tin Tetraphenyl to the Laboratory to be used in this determination.

NOTE: Inasmuch as the laboratory preparation is in fact a pilot run for the manufacturing department, all materials must be the same as proposed to use in the plant production.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Tin Tetraphenyl in Pyranol 1467 and 1470, Method No.11,533-53
contd.

Procedure:

1. Weigh on a beam balance a clean, dry 16 oz. wide-mouth bottle.

2. Rinse the bottle twice with about 100 ml. of Pyranol 1488 (or Tri-Tetra-Aroclor 1260 Mixture for Pyranol 1470) sample.

3. Fill the bottle approximately two-thirds full.

4. Reweigh the bottle with the sample and obtain the weight of Pyranol 1488 (or Tri-Tetra-Aroclor 1260 Mixture) being used. Record the weight.

5. Calculate the necessary amount of Tin Tetraphenyl powder to give a 0.130% solution of Pyranol 1467 (or Pyranol 1470).

Tin Tetraphenyl (grams) = g. of Pyranol 1488(or Tri-Tetra-Aroclor 1260 Mixture) x 0.00130

Record calculated weight of Tin Tetraphenyl.

6. Weigh accurately on an analytical balance, using a watch glass, the amount of Tin Tetraphenyl calculated in Step 5.

7. Brush the powder from the watch glass into the bottle containing the Pyranol 1488 (or Tri-Tetra-Aroclor 1260 Mixture).

8. Effect complete solution by heating to approx.60°C. with constant agitation. This should take about 1 hour and 15 minutes.

9. Analyze Pyranol 1467 (or Pyranol 1470) prepared by the above procedure, for Inorganic (Free) Chlorides according to Method No. 10,118. Report the result to Department A-246.

10. Analyze the Pyranol 1467 (or Pyranol 1470) in the regular manner (Procedure at the beginning of this method) as for Tin Tetraphenyl content.

Calculation: $\text{Empirical factor} = \frac{\text{ml. NaOH x Normality}}{\text{Sample Weight used x } 0.130} \times 100$

Report the result to the nearest 0.001.

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IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 4-22-54 Material: Tin Tetraphenyl Method No. 11,565-54

By: SMK,NPA:WJG Test: Appearance and Colour WGK Dept. No. 246

Tin Tetraphenyl is usually a white to light cream colored powder.

1. Examine the sample and describe its appearance noting any unusual characteristics. Report as white powder or otherwise if appropriate.

2. Examine the sample for visible foreign bodies such as rust, lint, wood, etc. Report as none, very slight, slight, moderate, or considerable. Describe appearance of impurities briefly.

3. Report presence of lumps and moisture also.

Date: 4-22-54 Material: Tin Tetraphenyl Method No. 11,566-54

By: SMK,NPA
WJG Test: Melting Point WGK Dept. No. 246

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1. Place 2 g. of the sample in an agate mortar and grind to a fine powder.

2. Fill a melting point tube, which has been sealed at one end, with the sample to a height of 3/4 inch.

3. Place the melting point tube in the melting point apparatus, fastening the tube to a 195 to 235°C. thermometer.

4. Determine melting point according to General Method No. 3 (Method No. 10,052) with the following modifications:

5. Regulate the temperature to give a rise of 0.25°C. per minute.

6. Report Softening Point, Melting Point, and Complete Melt.

7. Duplicate determinations should check to within ± 0.25°C.

Report the result to the nearest 0.5°C.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 10-6-54	Material: Tin Tetraphenyl	Method No. 11,568-54
By: WMK,WJG,NPA		
PLD,RFS:WJG	Test: Chlorides	WGK Dept. No. 246

1. Weigh a 20 ± 0.1 g. sample into a 500 ml.g.g.s.Erlenmeyer flask.

2. Add 350 ml. CP monochlorobenzene and heat on the steam bath until the sample dissolves. Occasional swirling of the contents will speed solution.

3. Transfer solution to hot, 80 - 100°C., 1-liter separatory funnel.

4. Rinse flask with 100 ml. hot water into funnel. Shake for one minute venting carefully through stopcock.

5. Transfer bottom layer (monochlorobenzene - TTP) to second hot 1-liter separatory funnel.

6. Add 50 ml. hot water and extract as before.

7. Draw off bottom layer and discard.

8. Combine H_2O extracts and transfer to 250-ml. beaker.

9. Cool to room temperature, add 3 drops conc. HNO_3 and 50 ml.2-B alcohol.

10. Chill in ice bath and titrate potentiometrically using 0.0333 N $AgNO_3$ from a 5-ml. microburette.

    NOTE: Add $AgNO_3$ in 0.10 ml. increments until potential approaches 200 millivolts. Then add in increments of 0.05 ml. until potential falls below 125 mv. Resume 0.10 ml. increments until a potential of less than 100 mv. is reached. Record all readings.

11. Make a blank determination following steps 1 - 10 except omitting sample.

12. Determine the equivalence point from the plot of ml. 0.0333 N $AgNO_3$ vs. emf (mv) as follows (see Fig.1):

IX. SPECIFICATIONS AND TEST METHODS, contd.

Chlorides Test for Tin Tetraphenyl, Method No.11,568-54, contd.

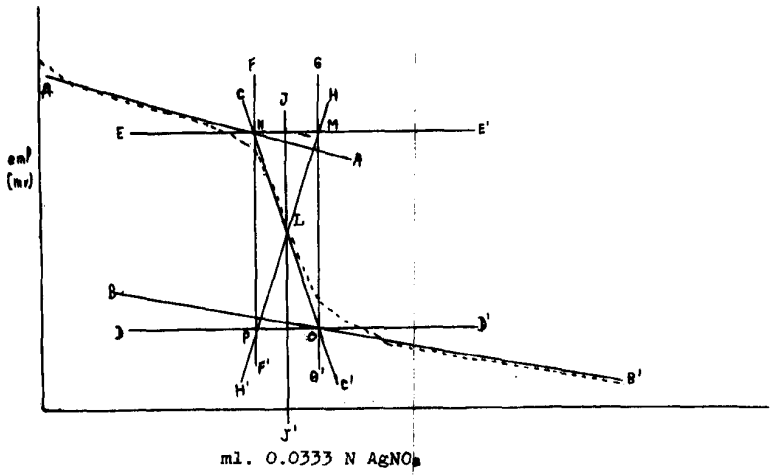
12. contd.

a. Draw line AA' through the top plateau.
b. Draw line BB' through the bottom plateau.
c. Draw line CC' through the step.
d. Draw lines DD' and EE' through points O - N and parallel to the horizontal axis.
e. Draw lines FF' and GG' through points O and N and parallel to the vertical axis.
f. Draw line HH' through points M and P.
g. Draw line JJ' through point of intersection L and parallel to the vertical axis.
h. Read the volume of 0.0333 N $AgNO_3$ from the intersection of line JJ' with the horizontal axis.

Calculation:

$$\text{ppm Chlorides} = \frac{(\text{ml.}AgNO_3\text{(sample)} - \text{ml.}AgNO_3\text{(blank)}) \times N \times 0.035457 \times 10^{6*}}{20}$$

* 10^6 = 1,000,000



IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 10/2/54 Material: Aroclor, Pyranols, Inerteen, Tri-tetra-chlorobenzene Blend Method No. 11,605-54

By: CM, LJW, NPA WWK:WJG Test: Dielectric Strength WGK Dept. No.246/233

Introduction:

Consult Method No. 11,752 for the sequence of electrical measurements and the terms used with the determinations.

The dielectric strength of an insulating oil is of importance as a measure of its ability to withstand electric stress without failure. It may also serve to indicate the presence of contaminating agents such as water, dirt, or conducting particles in the oil. One or more of these may be present simultaneously when low dielectric strength values are found by test.

However, a high dielectric strength is not a certain indication of the absence of all contaminants.

Procedure:

1. Ascertain that temperature of the material under test is 25 (±0.5) °C. Exception: For Aroclor 1260 only the temperature of the material under test will be 50 ± 1°C.

   NOTE: Testing at other temperature is likely to give variable results which may be misleading.

2. Shake the sample container so as to thoroughly mix the oil before filling the test cup.

   NOTE: This operation is even more important with used than new oil as the impurities may settle to the bottom and the test may be misleading.

3. Rinse the oil testing cup three times with small portions of the sample to be tested.

4. Immediately after final rinse, fill the cup to a height of not less than 20 mm. (0.787 in.) above the top of the electrodes.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Dielectric Strength of Aroclor, Pyranols, Inerteen, Tri-tetrachlorobenzene bland, Method No. 11,605-54, contd.

5. Rock the cup a few times in order that any entrapped air may escape. Close cover over oil test cup.

6. Allow to stand 3 minutes. CAUTION: THIS IS IMPORTANT!

7. Turn main toggle switch on front panel to "ON" (or up) position. Both green and amber pilot lights on the top at either side of the voltmeter will now glow.

   NOTE: The green signal light is connected across the 115 volt in-put and denotes that line voltage has been applied to operating control circuit.

   Amber light is connected in series with sensitive switch located under high voltage contactors and connected to its armature. It indicates that high voltage contactor is in its rest position and away from contacts energizing auto transformer.

8. Turn voltage control (large knob on front) to extreme counter-clockwise position.

9. Depress red botton on front. This energizes high voltage transformer and circuit breaker is indicated by amber light going out and the red light directly over voltmeter will light.

10. Watch the voltmeter and, while holding the button "IN", turn the voltage control at such speed that will cause voltage as indicated on voltmeter to rise at a rate of 3 K.V. per second.

11. Note and Record the voltmeter reading at breakdown.

12. Repeat the test until two successive breakdowns occur on each of two fillings of the test cup which do not differ by more than 10%.

Report the average value of these two readings (Step 12) as the Dielectric Strength.

If the limit of the instrument is reached before breakdown, report the Dielectric Strength as $>$ 50 K.V. at 25°C. or $>$ 50 KV. at 50°C. for 1260 Aroclor.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Dielectric Strength of Aroclor, Pyranols, Inerteen, Tri-tetrachlorobenzene blend, Method No. 11,605-54, contd.

Cleaning of the test cup:

1. After the test is completed, drain the cup.

2. Flush the cup with benzene. Use warm benzene after the 1260 test.

3. Then fill with Aroclor 1248 and let stand until the next analysis.

NOTE: An exception, when the samples of oil from the plant are brought in for test, the cup must be thoroughly cleaned with benzene and carbon tetrachloride before and after running the test.

The electrodes:

The oil testing cup has two electrodes. Both electrodes are movable and have twenty threads to the inch with index notches on both the electrodes and the lock nuts.

To set the Gap:

1. Arrange one of the electrodes and the lock nuts with the index marks in line.

2. Move the other electrode until it comes in firm contact with the first electrode and lock it.

3. Now unscrew the electrode with the index marks in line (Step 1) two complete turns and lock it.

This will leave a gap of 0.1 inch between faces of the electrodes.

Cleaning of the electrodes and the test cup free of carbon coating:

The following ASTM method of cleaning shall be followed when it is apparent from visual inspection that the electrode discs of the cup are coated with carbon.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Dielectric Strength of Aroclor, Pyranols, Inerteen, Tri-tetrachlorobenzene blend, Method No. 11,605-54, contd.

1. Wipe clean with dry calendered tissue paper the electrodes and the test cup.

   CAUTION: It is important to avoid touching the electrodes with the finger or with portion of the tissue paper which have been in contact with hands.

2. Rinse the electrodes and cup with dry lead-free gasoline, Stoddard Solvent (or dry, water white Kerosene) until they are entirely clean. Care should be taken not to touch the electrodes or the inside of the cup after cleaning so as to avoid possible contamination.

Apparatus:

General Information:

The electrical equipment necessary to provide high voltage to permit the determination of dielectric strength of liquid dielectric at commercial power frequencies is basically quite simple.

The equipment was assembled in the W.G.K. Plant Laboratory and consists of a high voltage transformer of good design and with a current capacity of 2.43 KVA and with equipment for control of the voltage and a means of measuring the voltage and to provide safety for the operator.

It is enclosed in a steel grey crackle finished cabinet measuring 42" high, 22" wide 17" deep and set on truck casters for easy mobility.

Protective equipment incorporated in this apparatus prevents the application of high voltage unless all safeguards are complied with. The door on rear of cabinet must be closed. The cover over the oil must be all the way down and the voltage control must be at 0 position. Failure to comply with these requirements will prevent any action when the red button is depressed.

The test cup: Transformers, Voltmeters, and Accessories

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Dielectric Strength of Aroclor, Pyranols, Inerteen, Tri-tetrachlorobenzene Blend, Method No. 11,605-54, contd.

The oil testing cup type No. 224809 supplied by General Electric Co. is mounted on the top rear of the cabinet. It is protected by a heavy plastic cover, hinged at the rear for accessability to the receptical.

It is equipped with safety contactor so placed that the circuit energizing the high voltage contactor can not be completed unless the protective cover is completely lowered and in place. It is impossible for the operator or anyone else to touch the testing cup when high voltage is applied.

The High Voltage Transformer manufactured by the Kelly-Koett Manufacturing Co. is of the closed core, oil immersed, shell type design. Rated at 81,000 volt at 400 millampers. It was recovered from a used X-ray machine, purchased quite inexpensively.

An auto transformer from the same X-ray machine is connected so as to limit the output voltage of the high voltage secondary to 50,000 volts.

The primary of the auto transformer is connected to the secondary of a 2½ KVA powerstat variable auto transformer supplied by the Superior Electric Company.

Power to the powerstat is controlled by a 4 contact 30 amp. solenoid circuit breaker.

The voltmeter mounted on top near front edge is connected across the powerstat secondary and is calibrated to read directly in kilovolts in the range of 0 - 50 K.V.

The overload circuit breaker consists of a small relay connected between one side of the high voltage transformer secondary center tap and ground. It is adjusted to break contact on a current drain of about 50 milliampers. The circuit for the coil of the solenoid circuit breaker is wired through the contacts of this relay.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Dielectric Strength of Aroclor, Pyranols, Inerteen, Tri-tetrachlorobenzene Blend, Method No. 11,605-54, contd.

Safety and Operating Controls:

(a) Door interlock switch located on rear door.

(b) Oil test cup cover interlock switch.

(c) Powerstat switch mounted on rear of unit arranged so that high voltage contactor can not be closed unless powerstat is at zero position.

(d) Main power switch on front panel.

(e) Powerstat voltage control on front panel.

(f) High voltage contactor push button on front panel, (red).

(g) Signal lamps mounted on top around voltmeter. Purpose and operation described in method of use.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 5-24-54	Material: Aroclors, Pyranols and Inerteens	Method No.11,607-54
By: CM, LJW, NPA GWM:WJG	Test: Resistivity	WGK Dept.No.246/233

Introduction:

Consult Method No. 11,752 for the sequence of electrical measurements and the terms used in connection with the determinations.

The Resistivity of an electrical insulating oil is an index of its insulating properties. High resistivity indicates high resistance to the passage of electrical current and normally, but not necessarily, establishes the absence of conducting contaminants.

Procedure:

1. Assemble the test cell. Place the recently cleaned (within the last 8 hours -- see Method No. 11,751, Step 4) electrodes in an 800-ml. beaker.

2. Measure the capacitance of the test cell (C_o) according to Method No. 11,608 (Dielectric Constant and Power Factor measurements).

3. Fill the cell assembly until the liquid level is 3/4 inch above the top of the electrodes.

4. Heat the assembly on the hot plate to 100 ($\pm$ 0.5)°C.

5. Place the assembly inside of the testing oven.

6. Attach top lead (+) on the megohm bridge to inner electrode.

7. Attach other lead to outer electrode.

8. Throw the three switches at the top of the meghom bridge to "ON" position.

9. Allow 10 minutes for assembly to reach temperature equilibrium inside the oven.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Resistivity of Arocolors, Pyranols, and Inerteens, Method No. 11,607-54, contd.

9. contd.
DANGER: Make sure control knob is in "CHECK" position. Otherwise painful shock will result.

10. Bring the galvanometer pointer to zero by turning the "ZERO ADJUST" knob in the direction in which the pointer of the galvanometer should move.

11. Turn the control knob to "CHARGE" position for one minute.

12. Turn the control knob to "OPERATE" position and return the galvanometer pointer to zero by adjustment of the "MULTIPLY BY" switch and the megohm dial.

13. Read after one minute.

Calculation:

Resistivity* - megohm dial reading (Step 12) x "Multiply By" reading(Step 11) x capacitance of cell (Step 2) in uuf. x 11.29 x 0.001

Report the result in units of 10^9 ohm/cm^3.

NOTE: It is important that the product under test, electrodes, and beaker be at uniform temperature for this determination. Temperature variations in different parts of the sample will cause the galvanometer zero to change constantly and give misleading results.

CAUTION: In as much as measurements must be made at a potential of 500 volts DC a shock hazard exists in the handling of this apparatus. With the control knob in the charge and operate position full voltage of the bridge (500 volts) is applied to the + and low terminals and through the test leads to the electrodes. Do not attempt to handle the electrodes or the test leads unless the control knob is in the "CHECK" position. Possible penalty for failure to observe this precaution -- Painful Shock.

* Values of resitivity are qualified by designation of temperature and voltage. These are for this test, 100°C. and 500 volts DC.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Resistivity of Aroclors, Pyranols, and Inerteens, Method No. 11,607-54, contd.

Apparatus:

General Radio Co. Megohm Bridge Type 544-B. This is a combination of Wheatstone bridge and vacuum tube voltmeter for indicating null. The direct measurement of resistance up to 1,000,000 megohms is made possible by the use of a vacuum tube detector which absorbs negligible amount of power.

The voltage applied to the unknown resistor is held approximately constant, regardless of the value of the unknown resistance. This condition is necessary to measure resistance properly.

The accuracy of the instrument in the range encountered in the measurement of Aroclor resistivity, 100 to 1000 megohm is $\pm$ 6%.

The instrument is equipped with a 115 volt A.C. power supply which supplies all operating voltages for the bridge indicating circuits and in addition supplies 500 V DC for application to the material under test. The instrument is completely enclosed in a waxed finish shielded oak cabinet measuring 8½" wide, 22½" long and 8" high. Approximate weight --- 26 pounds.

Test Electrodes:

Two concentric nickel cylinders with feet, obtained from General Electric Company. The inner electrode has outside diameter of 2.8" and a height of 3.25" with area of 184 sq. cm. The outer electrode has inside diameter of 3" and height of 3.25" with area of 198 sq. cm. The distance between electrodes is, therefore, 0.1" or 0.254 cm.

By theory, electrode constant (K) area/length is 191/0.254 or 752 where average area is 191 sq. cm.

Also $K = 36\pi \times 10^{11} \times C$ (in farads with air as dielectric) = 11.29 x C (in uuf. with air as dielectric).

Glass Plate: Pyrex about 3½" diameter with concentric grooves to assist in spacing electrodes. Obtained from General Electric Company.

Heating Unit: Assembled in the laboratory and is the same unit described in Dielectric Constant Apparatus (see Method No. 11,608; equipment).

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 5/17/54 Material: Aroclors, Pyranols, Inerteens, and Tri-Tetra-chlorobenzene Blends — Method No. 11,608-54

By: CM,NPA,GWM:WJG — WGK Dept. No. 246

Test: Dielectric Constant and Power Factor

I. Adjustment of Controls on Electrical Apparatus

A. On Panel No. 1 (Top Panel, Amplifier and Null Detector)

a. Turn the 4-way (main power) switch on the upper right hand side to the #3 position to determine the Dielectric Constant at 1000 cycles. Turn this switch to the #2 position for measurements at 60 cycles.

b. Allow the equipment to warm up 10 minutes.

c. Turn "GAIN CONTROL" to 6.

d. Depress "NULL DET." button.

e. Depress "INPUT > 0.03V." button

B. On Panel No. 2 (Oscilloscope)

a. Turn "INTEN." to about the 12 o'clock position.

CAUTION: Do not allow a high intensity spot to remain stationary on the screen for any length of time.

b. Using "HOR. POSITION" and VERT. POSITION" control center the image on the screen.

c. Adjust "FOCUS" for sharp line.

d. Turn "FREQ. SELECTOR" to 100 KC.

e. Turn "FREQ VERNIER" to 80.

f. Turn "VERTICAL GAIN" to 5.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Dielectric Constant and Power Factor of Aroclors, Pyranols, Inerteens, and Tri-Tetrachlorobenzene Blends, Method No. 11,608-54, contd.

g. Turn "VERTICAL INPUT" to "10 VOLT MAX.".

h. Turn "HORIZONTAL GAIN" to about 20.

i. Turn "SYNCHRONIZING" to ± 20.

j. Turn "SYN. to "EXT. SYN."

k. Turn "GEN." to "SWEEP GEN."

C. On Panel No. 3 (Capacitance Bridge)

a. Turn "RANGE SELECTOR" switch to "100c" for 60 cycle measurements and to 1 KC" for 1000 cycle measurements.

b. Turn "METHOD SWITCH" to direct.

c. Turn "DISSIPATION FACTOR" selector switch to "0".

D. On Panel No. 4 (Oscillator)

a. Disregard this panel for measurements at 60 cycles.

b. On 1000 cycle measurements, depress the No. 10 "MULTIPLY BY" button.

c. Set "FREQUENCY DIAL" to 100.

d. Turn "OUTPUT" dial so that pointer is at the end of the arrow.

e. Depress the "UNBAL. 5000 OHMS" button.

When all of the above adjustments are made, the electrical apparatus is ready for measurement of Dielectric Constant and Power Factor.

IX. SPECIFICATIONS AND TEST METHODS, cond.

Test for Dielectric Constant and Power Factor of Arcolors, Pyranols, Inerteens, and Tri-Tetrachlorobenzene Blends, Method No. 11,608-54, contd.

II. Determination of Constants for the Apparatus.

1. Carefully assemble the cell which has been cleaned and dried within the last 8 hours. Refer to Method No. 11,751 for the procedure to use in cleaning the cells.

2. Place the cell assembly in the Fisher oven which has been adjusted to 25°C.

3. Connect the back wire inside the oven to the lead on the inner cylinder of the cell and the front wire to the lead on the outer cylinder of the cell.

4. Connect the cable from the capacitance bridge to the terminals on top of the oven so that the inner wire of the cable goes to the back terminal and the outside mesh casing of the cable (the ground) goes to the front terminal.

5. Remove the thermometer from the top of the oven before going on with the test. This is important.

6. Make all adjustments on the electrical apparatus as directed in Part I of this method.

7. Balance the bridge by rotating the "CAPACITANCE" and "DISSIPATION FACTOR" dials on Panel No. 3 until the wide vertical band on the oscilliscope is adjusted to a minimum width.

8. Record the sum of the readings on the "CAPACITANCE" dial and vernier and call this value A.

9. Remove the beaker containing the cell from the oven and fill it with C. P. benzene to a level 0.737 inches (ca. 3/4 inch) above the top of the concentric cylinders of the cell.

10. Adjust the temperature of the benzene to 25°C. while stirring with a thermometer.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Dielectric Constant and Power Factor of Aroclors, Pyranols, Inerteens, and Tri-Tetrachlorobenzene Blends, Method No. 11,608-54, contd.

11. Replace the cell in the oven (at 25°C.) and make the same electrical connections as in Steps 3 and 4. DO NOT interchange connections.

12. Balance the bridge again as in Step 7.

13. Record the sum of the readings on the "CAPACITANCE" dial and vernier and call this value B.

14. Calculate the cell constant by the following equation:

$$\text{Cell Constant, } K = \frac{B + A}{2.27 - 1.0}$$, (this is usually around 70 uuf).

15. Remove the cell from the oven and balance the bridge as in Step 7 with the "CAPACITANCE" and "DISSIPATION FACTOR" dials.

16. Record the sum of the readings on the "CAPACITANCE" dial and vernier and call this value F. (capacitance of connecting cable).

17. Calculate the Cell Lead Capacitance by the following equation:

$$\text{CELL LEAD CAPACITANCE, } G = A - F - K$$ (this is usually around 3 uuf.)

WHERE:

A = CAPACITANCE OF ENTIRE SYSTEM IN AIR (SYSTEM CONSTANT)

G = CAPACITANCE OF THE CELL LEADS (CELL LEAD CONSTANT)

F = CAPACITANCE OF CABLE AND WIRES WHICH CONNECT THE CELL AND CELL LEADS TO THE BRIDGE. (CONNECTOR CONSTANT).

K = CAPACITANCE OF THE CELL ALONE (THE CELL CONSTANT).

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Dielectric Constant and Power Factor of Aroclors, Pyranols, Inerteens, and Tri-Tetrachlorobenzene Blends, Method No. 11,608-54, contd.

Tabulate the System Constant (A), the Cell Lead Constant (G), the Connector Constant (F), and the Cell Constant (K) on a piece of stiff paper and post them near the instrument where they can be easily referred to for comparison and calculations.

These constants must be checked at least once every three months and in all cases where the Dielectric Constant and/or Power Factor are out of specification.

III. Measurement of Dielectric Constant and Power Factor on Aroclors, Pyranols, Inerteens, and Tri-Tetrachlorobenzene Blends.

A. Test Run on Cell to Determine Whether it is Clean and Properly Aligned.

1. Carefully assemble a cell which has been cleaned and dried within the past 8 hours.

   NOTE: Refer to Method No. 11,751 for procedure to use in cleaning cells.

2. Adjust the oven control to hold at a temperature of 100°C. for all materials except Tri-Tetra Blends. If a Tri-Tetra blend is to be tested, adjust the oven to hold a temperature of 25°C.

3. Place the empty cell assembly in the oven and connect the back wire inside the oven to the lead on the inner cylinder of the cell, and connect the other (front) wire to the lead on the outer cylinder.

4. Connect the cables from the capacitance bridge to the terminals on top of the oven so that the inner wire of the cable goes to the back terminal and the outside metal casing (ground) goes to the front terminal.

5. Allow 15 minutes for the cell to reach temperature equilibrium inside the oven.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Dielectric Constant and Power Factor of Aroclors, Pyranols Inerteens, and Tri-Tetrachlorobenzene Blends, Method No. 11,608-54, contd.

6. Remove the thermometer from the top of the oven before taking any measurements on the bridge. This is important.

7. Make all the adjustments on the electrical apparatus as directed in Part I of this method.

8. Balance the bridge by rotating the "CAPACITANCE" and "DISSIPATION FACTOR" dials on Panel No. 3 until the wide vertical band on the oscilloscope screen is adjusted to a minimum width.

9. Record the sum of the readings on the "CAPACITANCE" dial and vernier and compare this value with the SYSTEM CONSTANT determined in Part II of this method.

   IMPORTANT: If the value obtained in Step 9 does not agree with the System Constant A (Part II) within 5 uuf, the cell must be re-cleaned, re-dried, re-assembled, and the test run for the System Constant must be repeated.

   NOTE: Although the above test run must be made prior to each analysis, the value obtained in Step 9 is not to be used in calculations but is to be used only as a check on the cleanliness and alignment of the cell.

B. Procedure for Testing Materials.

10. Remove the cell from the oven and fill the beaker with the material to be tested to a level 0.737 inches (ca 3/4 inch) above the cylinders of the cell.

11. Adjust the temperature of the sample to 100°C. (use hot plate) for all materials except Tri-Tetra blends. For Tri-Tetra blends, adjust the temperature of the sample to 25°C. using an ice-water bath if necessary.

    NOTE: Stir sample continuously with a thermometer while adjusting the temperature.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Dielectric Constant and Power Factor of Aroclors, Pyranols Inerteens, and Tri-Tetrachlorobenzene Blends, Method No. 11,608-54, contd.

12. Place the cell and sample in the oven and make the same connections from the cell to the bridge as in Steps 3 and 4, DO NOT interchange connections.

13. Allow fifteen minutes for the cell to reach temperature equilibrium inside the oven.

14. Remove thermometer from the oven before taking a measurement. This is important.

15. Make the adjustment of controls on the electrical apparatus as directed in Part I of this method.

16. Balance the bridge by rotating the "CAPACITANCE" and "DISSIPATION FACTOR" dials on Panel No. 3 until the wide vertical band on the oscilloscope screen is adjusted to a minimum width.

17. Record the sum of the readings on the "CAPACITANCE" dial and vernier, and call this value X.

18. Record the sum of the readings on the "DISSIPATION FACTOR" dial and switch. Call this value D.

Calculations:

$$\text{Dielectric Constant} = \frac{X - F - G}{K}$$

Where:

X = Capacitance reading from Step 17.
F = Connector Constant (Determined in Part II)
G = Cell Lead Constant (Determined in Part II)
K = Cell Constant (Determined in Part II)

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Dielectric Constant and Power Factor of Aroclors, Pyranols Inerteens, and Tri-tetrachlorobenzene Blends, Method No. 11,608-54, contd.

$$\% \text{ Power Factor} = \frac{f}{f_o} \times D$$

Where:

f = Test Frequency (60 cycles or 1000 cycles)
f_o = frequency of "Range Selector" on Panel No.3
D = Dissipation Factor reading from Step 18.

NOTE: When D (dissipation factor) is less than 0.1, the dissipation and power factors differ by less than 0.0005. Therefore, for our measurements, power factors and dissipation factors are equal.

Precision: (Reference General Radio Manual for Model 716-C Capacitance Bridge).

a. Capacitance readings are precise to ± 2 uuf x multiplier reading (± 0.2% of full scale for each range) when the dissipation factor is less than 0.01.

b. Dissipation Factor (Power Factor) readings are precise to ± 0.0005 or ± 2% of the dial reading whichever is larger, for values less than 0.1 for D (Dissipation Factor).

IV. Apparatus

A. Oscilloscope: Heathkit Model O - 6.

B. Constant temperature Heating Unit: Fisher Isotemp oven. Model 13-245A, modified to include inter-wall connectors.

C. A. C. Generator: General Radio type 1302-A.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Dielectric Constant and Power Factor of Aroclors, Pyranols, Inerteens, and Tri-tetrachlorobenzene Blends, Method No. 11,608-54, contd.

IV. Apparatus, contd.

D. Amplifier and Null Detector: General Radio type 1231-B with type 1261-A power supply.

E. Capacitance Bridge: General Radio Company Capacitance Bridge type 716-C.

F. Test Cells: G. E. type, concentric cylinder electrodes Catalog # 1,559,663.

G. Class B driver transformer: This is used for 60 cycle measurements to excite the bridge directly from the domestic power line.

It has 50 volts output with a 4800 ohm resistor in series.

H. Tuned Circuit Filters: General Radio Type 1231-P2 (400 and 1000 cycle) and 1231-P3 (60 cycle).

These filters aid in obtaining a more accurate frequency for the measurements by removing harmonics, noise, hum, etc.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 4/22/54 Material: General Method Method No. 11,732-54

By: DKL, NPA:WJG Test: Color (N. P. A. scale) WOK G.M. No. 11

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1. Fill a 1-5/16" x 5" N. P. A. glass test jar (N. P. A. tube) half full of the liquid (or molten) sample.

2. Place the jar in the right hand opening in the A. S. T. M. Union Colorimeter and a similar tube half full of distilled water in the left hand opening.

3. Observe the sample and standard colored glasses through the observation opening (hole).

4. Turn the knob that brings the various colored glass standards into view.

5. Match the sample as nearly as possible with the color standards.

The method is precise to ± 1/4 N. P. A. units.

Report the color to the nearest 1/4 N. P. A. unit.

Reference: ASTM Union Colorimeter and the N. P. A. Color Standards are given in ASTM Designation: D-155-45T (see 1952 Book of ASTM Standards, part 5, pages 86 to 90 inclusive).

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 2/26/53 Material: Aroclors, Pyranols, Inerteens, and Tri-Tetrachloro-benzene Blends Method No. 11,751-53 WGK Dept.No. 233/246

By: CM, HOH, LJW,WJG, NPA: SMK Test: Procedure for Cleaning of Electrodes, G. E. Cell, and Accessories

A. The Electrodes Cleaning Procedure:

1. Remove the electrodes from the tested material while hot and allow them to drain.
2. Place the electrodes in hot TCB for 10 minutes.
3. Wash with unheated TCB.
4. Rinse twice with methanol and twice with tap water.
5. Place the electrodes in hot 10% T. S. P. solution. Soak and heat for 10 minutes.
6. Wash thoroughly with tap water.

CAUTION: After Step 6 -- DO NOT TOUCH THE ELECTRODES WITH HANDS!

7. Wash with distilled water twice.
8. Dry in drying oven for at least two hours at 120°C.

B. The G. E. Cell Cleaning:

1. Reclean the Cell before use, if more than 8 hours have elapsed since the previous cleaning.
2. Follow the procedure for the electrodes starting at Step 4.

C. Cleaning of the Accessories:

MONS 046286

1. Apply the same cleaning procedure as given for the electrodes (Steps 1 to 8) to prepare the glass spacer and beaker for next test.
2. Clean the thermometer in the same manner as the electrodes, except for Step 8.
3. Place the wet thermometer (after Step 7) directly in position in the Temperature Heating Unit (Modified Fisher Isotemp Oven) and allow to dry.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 2/26/53 Material: Aroclors, Pyranols, Inerteens, and Tri-Tetrachloro-benzene Blends Method No. 11,752-53

By: CM, LJW Test: Electrical Measurements WGK Dept. No. 233/246

Introduction:

Some of the Aroclors, Pyranols, Inerteens, and Tri-Tetrachloro-benzene Blends have important industrial uses based on their electrical properties. Therefore, tests for certain electrical properties are made on these products.

General Information:

The tests should be run in the following order:

Test		Method No.
1.	Dielectric Constant	11,608
2.	Power Factor (if needed)	11,608
3.	Resistivity	11,607
4.	Dielectric Strength	11,605

The terms, which are used in connection with the above listed electrical measurements, are defined as follows:

1. Dielectric:

A dielectric is an insulating or non-conducting material.

2. Dielectric Constant:

The dielectric constant is the ratio of the equivalent parallel capacitance of a capacitor in which the material

IX. SPECIFICATIONS AND TEST METHODS, contd.

Electrical Measurements for Aroclors, Pyranols, Inerteens, and Tri-Tetrachlorobenzene Blends, Method No. 11,752-53, contd.

2. Dielectric Constant, contd.

is the dielectric, measured at a specified frequency, to the capacitance of the same capacitor with a vacuum as the dielectric, and is represented by the symbol K. The K of air may be considered equal to that of a vacuum.

3. Capacitor:

A capacitor consists of an electrical conductor upon which an electric charge can be stored. In practice, a condenser consists of two or more metallic plates separated by a dielectric.

4. Resistivity:

In the metric system, the volume resistivity of a material is the resistance between two electrodes which cover opposite faces of a centimeter cube. It is expressed in ohms/cm^3.

5. Dielectric Strength:

Dielectric Strength is the rupturing strength of an insulating material when subjected to voltage stress, under specific conditions and expressed in volts/milliampere. Breakdown varies with the shape of the electrodes and does not increase in proportion to the thickness of the dielectric.

6. Power Factor:

The power factor of a dielectric is the ratio of the energy loss in the dielectric to the "apparent power" in the dielectric.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 10/7/53 Material: General Method Method No. 11,784-53
By: WRB,DKL
DTM:SMK Test: Refractive Index WGK G. M. No. 28

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1. Study the attached diagram to familiarize yourself with the principal parts of the Refractometer which will be referred to throughout the method.

   CAUTION: The glass from which the prisms in this instrument are constructed is necessarily somewhat unstable and is very easily scratched or corroded. It is, therefore, necessary to exercise extreme caution in using them by following these 3 rules:

   a. Do not wipe the glass with any rough material or with anything in which there may be specs or grit.

   b. Never touch the surfaces of the Abbe Prisms.

   c. Clean the prisms only by flushing them with alcohol, Xylol, TCB or water.

2. Make sure that the surfaces of the prisms, between which the fluid is to be placed, are perfectly clean. This can be accomplished by flushing them with alcohol and allowing to dry in air.

3. Turn on the stirring motor in the constant temperature bath by flipping the toggle switch marked "Line" to the up position.

4. Adjust the prisms to 25°C. or other specified temperature as registered on the thermometer. This is done as follows: If the temperature is above 25°C., turn on the valve back of the constant temperature bath which allows cooling water to circulate in the bath. If the temperature is below 25°C., flip the toggle switch in the upper right corner to the "Medium" position. This should bring the temperature to 25°C. or other level, as pre-set and maintain it at that point. If it does not, an adjustment is required. Report this to your supervisor. DO NOT PERFORM ANY ADJUSTMENTS YOURSELF.

5. Place several drops of pure distilled water on the auxiliary (lower) prism and immediately bring the prisms together and clamp them in position.

IX. SPECIFICATIONS AND TEST METHODS, contd.

General Method, Test for Refractive Index, Method No. 11,784-53, contd.

NOTE: Be sure prism is completely covered with water.

6. Turn on the light back of the refractometer and adjust the mirror until light is reflected into the lower prism.

7. Release the "Hand Positioning Control Clamp" and move the lever arm slowly by hand until a position on the scale, at which the lower part of the field is dark and the upper part light, is obtained.

8. Clamp the movable arm in this position by means of the hand positioning control clamp.

9. Move the mirror slightly. If the dividing line moves it is spurious. The mirror should be moved to clear the spurious dividing line from the field and the true dividing line sought again.

10. The border between the light and dark portions of the field will usually be coloured. This may be corrected by turning the "Compensator Control Knob" so that a sharp black and white edge is obtained.

11. Finally adjust the "Tangential Screw Mechanism" until the black edge just crosses the intersection of the cross-wires.

12. The refractive index is then read by observing the scale of the "Alidade Arm" through the "Scale Magnifier", the fourth decimal place being estimated.

13. If the reading of the distilled water at 25°C. varies from 1.3325 (the true index of distilled water at 25°C.)record the correction and apply this same correction to the sample reading when it is observed.

NOTE: Corrections at other temperatures than 25°C. may be made with suitable conversion -- see your supervisor.

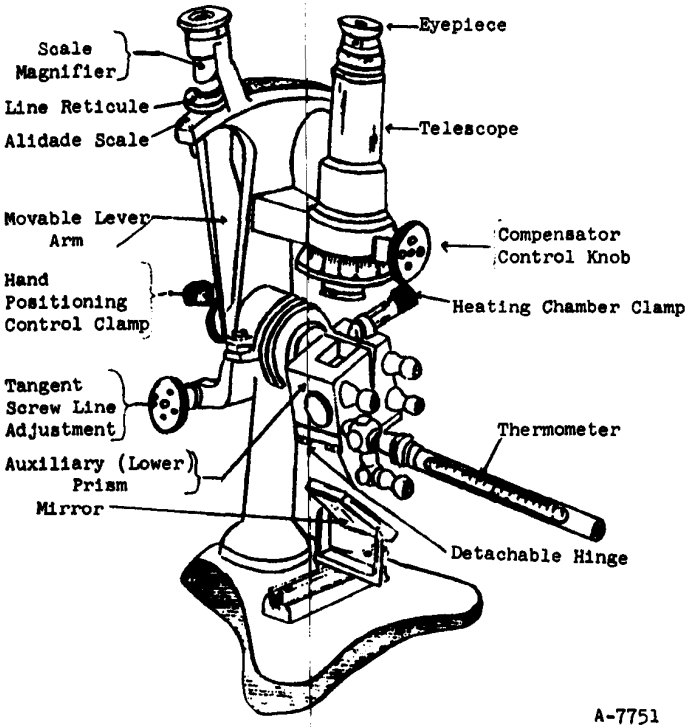
14. Repeat Steps 2 to 12 inclusive substituting the sample in place of the distilled water in Step 5.

15. Apply the correction from Step 13 and record the reading.

16. Repeat Steps 2 to 15 excluding Step 14 twice more, using fresh sample each time. The readings should agree within 0.0001 units. Report Refractive Index at 25°C. to the nearest 0.0001 unit.

IX. SPECIFICATIONS AND TEST METHODS, contd.

ABBE TYPE REFRACTOMETER

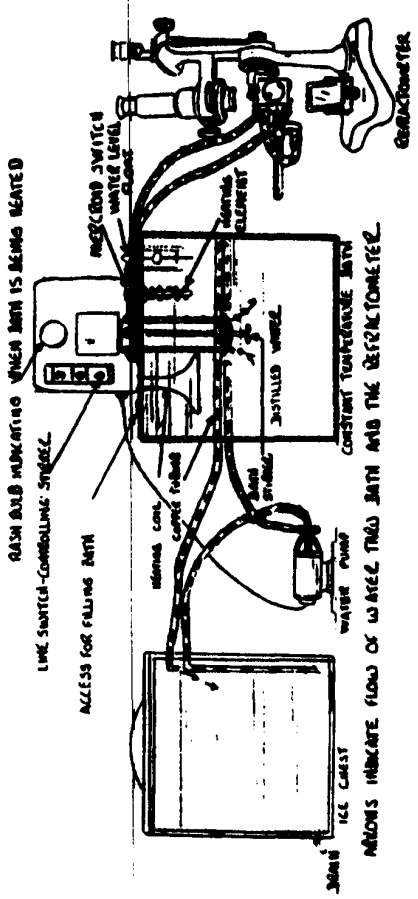


A-7751

IX. SPECIFICATIONS AND TEST METHODS, contd.

A-7752

CONSTANT TEMPERATURE BATH



IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 7/30/53 Material: General Method Method No. 11,788-53

By:DKL,WJG:SMK Test: Turbidity WGK G.M. No. 15

The turbidity of WGK Plant Products and Raw Materials is determined by comparison of a sample of the material with a set of ten turbidity standards representing the normal range of turbidity encountered.

1. Shake the sample for 15 seconds.

2. Compare the sample immediately to the appropriate turbidity standards in sample bottles of the same size and similarly shaken. Report the turbidity to the nearest standard number, e. g. Turbidity, = Standard No. 5.

NOTE: A helpful procedure is to compare the ease which one might see a pencil or window frame through the sample and standards, or to compare the haze in the sample and standards against a well lighted white back-ground.

Turbidity Equivalents:

Standard Number	$\approx H_2SO_4$ Std. No.	$\approx H_3PO_4$ Std. No.	APHA Turbidity ppm Suspended Matter
1	------	------	2
2	------	------	4
3	------	1	8
4	½	2	16
5	1	4	30
6	2	8	60
7	3	18	125
8	4	------	250
9	------	------	500
10	5	------	1000

Preparation of Standards:

Turbidity stock solution, 1000 ppm Fuller's Earth in accordance with APHA 6th Edition, obtained from the Hartman-Leddon Co. (Harleco) Philadelphia, Pa.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Turbidity, General Method, Method No. 11,788-53, contd.

The stock solution is Standard No. 10.

The standards are prepared by dilution of the well shaken stock solution as follows:

Standard	ml. Stock Solution diluted to one liter
10	1000
9	500
8	250
7	125
6	60
5	30
4	15

Dilute 100 ml. of the well shaken Stock Solution to one liter and dilute as follows:

Standard	ml. Stock Solution diluted to one liter
3	80
2	40
1	20

Add 50 ml. of a 5% mercuric chloride solution containing 1% HCl per liter of final standard before dilution. The mercuric chloride serves as a preservative to prevent mold growth in the standards.

For verification, the newly-prepared standards may then be compared with reference turbidity standards (kept by the Service Section) from the Hartman Leddon Company representing the A. P. H. A. turbidity values listed in the Turbidity Equivalents table.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 5/6/54 Material: Phenoxypropene Oxide Method No. 11,861-54
(Glycidyl Phenyl Ether)
By:WGM,NPA:WJG Test: Crystallizing Point WGK Dept. No. 246

CAUTION: Phenoxypropene Oxide (Glycidyl Phenyl Ether) is a toxic irritant. Handle with care and avoid contact with the skin. Wear your Safety Glasses.

1. Prepare a dry ice-acetone bath in a 400-ml. beaker.

2. Prepare an ice-water bath in a second 400-ml. beaker.

3. Fill a 1" x 4" test tube 3/4 full with the sample.

4. Insert a -15° to 20°C. range thermometer and place the test tube (supported by a lead disc provided for this purpose) in the dry ice-acetone bath. Stir constantly with the thermometer.

5. Upon cooling of the sample to -2° to 5°C. remove it from the bath and seed with previously prepared crystals of Phenoxypropene Oxide (PPO).

6. Replace the sample in the dry ice-acetone bath. Continue stirring scratching repeatedly the sides of the test tube, until the temperature begins to rise or until the sample cools to -15°C.

   IMPORTANT: DO NOT COOL BELOW -15°C.

7. Immediately place the sample in the ice-water bath and continue stirring until the temperature reaches a maximum point. Record this temperature.

8. This maximum temperature is the crystallizing point.

The method is precise to ± 0.1°C.

Report the result to the nearest 0.1°C.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 5/3/54 Material: Phenoxypropene Oxide (PPO) Method No. 11,862-54

By:NN,NPA:WJG Test: Distilling Range WGK Dept. No. 246

CAUTION: Glycidyl Phenyl Ether (Phenoxypropene Oxide: P.P.O.) is a toxic irritant. Handle with care and avoid contact with the skin. Wear Your Safety Glasses.

1. Determine the distilling range of PPO according to General Method No. 1 (Method No. 10,006) with the following modifications:

2. Insert a 230 to 260°C. range, 3" immersion thermometer.

3. Use a transite board with a 2-inch opening.

4. Distill at the rate of 2 drops per second.

5. Apply the thermometer and barometric correction.

Report the following temperatures:

First drop

95% (1-96 ml.) range

100% (First Drop to Dry Point) range

Dry point

Report the results to the nearest 0.1°C.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 5/3/54 Material: Phenoxypropene Oxide Method No.11,863-54

By: NN,NPA:WJG Test: REFRACTIVE INDEX AT 25°C. WGK Dept. No. 246

CAUTION:

Glycidyl Phenyl Ether (Phenoxyprppene Oxide: P. P. O.) is a toxic irritant. Handle with care and avoid contact with the skin. Wear Your Safety Glasses.

1. Determine the refractive index of PPO at 25°C. according to General Method No. 28 (Method No. 11,784) with the following modifications.

2. Clean the Abbe prisms by flushing with Trichlorobenzene. Then follow with benzene and "Mersol" washes.

IMPORTANT:

Do Not Touch the Polished Surfaces of the Abbe Prisms!

Report the Refractive Index (N_d^{25}) to the nearest 0.0001 unit.

/————————————/

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 7/23/54 Material: Phenoxypropene Oxide (PPO) Method No.11,864-54

By: NN, NPA, GWM:WJG Test: Specific Gravity WGK Dept. No.246

CAUTION: Glycidyl Phenyl Ether (Phenoxypropene Oxide: PPO) is a toxic irritant. Handle with care and avoid contact with the skin. Wear Your Safety Glasses.

1. Determine the Specific Gravity 25/15.5°C. according to General Method No. 13 (Method No. 10,497) with the following modifications:

2. Use a 1.000 to 1.200 range hydrometer.

3. Use a 0° to 50°C. range thermometer.

4. Adjust the temperature of PPO between 20 - 30°C.

5. Apply (if any) the hydrometer correction.

6. Use Specific Gravity Coefficient 0.0008 unit/°C.

Calculation:

A. For Specific Gravity at 15.5°C./15.5°C.

Temperature Correction = (Temp.of Sample - 15.5°C.) x 0.0008

Add this correction to the reading obtained if the temperature of the sample was above 15.5°C., subtract if below 15.5°C.

B. For Specific Gravity at 25°C./15.5°C.

Temperature Correction = (Temp.of Sample - 25°C.) x 0.0008

Add this correction to the reading obtained if the temperature of the sample was above 25°C. subtract if below 25°C.

The method is precise to ± 0.001 unit.

Report the Specific Gravity 15.5°C./15.5°C. and 25°C./15.5°C. to the nearest 0.001 unit.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 7/8/52 Material: Transformer Inerteen (PPO) Method No. 11,865-52

By: NN, TT:SMK Test: (Phenoxypropene Oxide) WGK Dept. No. 246

NOTE: Run this analysis in duplicate

Preparation of Reagents:

Indicator - Mix 1 part of a 0.1% water solution of cresol red with 3 parts of a 0.1% water solution of thymol blue.

Pyridinium Chloride Reagent - Completely mix 16 ml. of conc. HCl with 1 liter of Baker's C. P. pyridine.

1. Weigh (± 0.05 g.) a 100 ± 5 g. sample into a 500 ml. Erlenmeyer flask.

2. Add by pipette 20 ml. of the pyridinium chloride reagent.

3. Attach to a reflux condenser and reflux for thirty minutes.

   NOTE: Measure reflux time from the start of boiling.

4. Turn offthe heat at the end of the reflux period, and add 50 ml. of distilled water at the top of the condenser.

5. Detach the flask from the condenser and wash the end of the condenser with a few ml. of distilled water.

6. Cool the flask and contents to room temperature in ice water or under the tap.

7. Add 1 ml. of the cresol red-thymol blue indicator.

8. Swirl the flask rapidly so that there is a thorough and violent mixing of the contents while titrating with 0.1 N NaOH to a distinct blue colour.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for (Phenoxypropene Oxide)in Transformer Inerteen (PPO), Method No. 11,865-52, contd.

NOTE: If any doubt exists about the end point allow the layers to separate completely after each addition of NaOH where close to the end point. The end point has been reached when the upper aqueous layer is blue.

9. Blank:
   Into a 500 ml. Erlenmeyer flask, weigh, on a beam balance, a 100 ± 5 g. sample of Transformer Inerteen which contains no PPO.

10. Repeat Steps 2 to 8.

Calculations:

A = ml. 0.1 N NaOH for blank.

B = ml. 0.1 N NaOH for sample

$$\% \text{ Phenoxypropene Oxide (PPO)} = \frac{(A - B)(\text{Normality NaOH})(15.0)}{\text{Gram Sample}}$$

Report to the nearest 0.001%.

Reference:

Westinghouse Research Report No. 60-94602-9-48.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 5/12/53	Material: Tri and Tetrachloro - benzene Blend	Method No.12,033-53
By:NPA:SMK	Test: Last Crystal Point	WGK Dept. No. 246

1. Fill Pour-Point test jar with sample to a depth of two inches.

2. Insert the thermometer-stirrer assembly so that the mercury bulb of the thermometer is near the center of the sample.

3. Cool, while stirring, until sample is at about -15°C.

4. Seed with a few crystals of 1,2,4,5 Tetrachlorobenzene and continue stirring and cooling until the sample crystallizes forming a slurry.

5. Continue stirring at the rate of one to three strokes per second, while exposing jar and contents to room temperature.

   NOTE: As the last crystal point approaches, the mercury meniscus of the thermometer under the liquid is invisible, but it is within a range of 0.2 - 0.3°C. from surface of the sample.

6. Record temperature at which the mercury column of the thermometer just becomes clearly visible. This is the Last Crystal Point.

   NOTE: The visibility does not appreciably change during the next 0.5°C. rise in temperature.

Report the result to the nearest 0.1°C.

Apparatus:

A Pour-Point test jar (see Section 3 a, ASTM D97).

A -15 to 20°C. range thermometer accurately calibrated and having 0.1°C. subdivisions.

A loop stirrer of a heavy gauge wire.

A rubber stopper with holes to accomodate thermometer and stirrer.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 2/6/53	Material: Tri-Tetrachloro-benzene Blend	Method No. 12,034-53
By:NPA:SMK	Test: Distilling Range	WGK Dept. No. 233/246

1. Determine the distilling range of the sample according to General Method No. 1 (Method No. 10,006) with the following modifications:

2. Use a transite board with a 2" opening.

3. Use a 0 - 360°C. range 3-inch immersion thermometer (corrected at 200° and 250°C.) with 1°C. subdivisions.

4. Use an air-cooled condenser. Any crystals must be kept from forming in the tube by playing a flame over its length as needed.

5. Report the following (corrected) temperatures to the nearest 1°C.

First Drop

5% by volume

65% by volume

90% by volume

95% by volume

Dry Point.

NOTE:

The distillate is collected "by volume" and not "by weight" as in the case of Pyranol distillations.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Date: 2/10/53 Material: Tri-Tetrachloro-benzene Blend Method No. 12,036-53
WGK Dept. No. 233/246
By: TT,NPA:SMK Test: Composition by Distillation

Use a beam balance for all weights.

1. Weigh a "heel" of 200 (± 0.05) g. of Aroclor 1248 into a 2-liter still-pot containing three large Berl saddles and a glass "Perculator" tube.

2. Weigh into the same (tared) pot a 700 (± 0.05) g. sample of the Tri-Tetra-blend to be tested.

3. Connect the still-pot to the distillation unit.

4. Turn on the electrical current to heat up the distillation assembly (DO NOT APPLY HEAT ON THE HEAD AT THIS TIME).

5. Carry out the distillation at an atmospheric pressure and take fractions as follows:

   a. Fraction 1 (Trichlorobenzene Fract.): Collect distillate from 204° to 220°C. (the distilling head temperature), into a 500-ml. Erlenmeyer flask (tared).

   b. Fraction 2 (1st Intermediate Fract. - Tri-Tetrachlorobenzene): When the distilling head temperature reaches 220°C. remove the receiver with 1st Fraction and replace it with a dry, tared 250-ml. Erlenmeyer flask. Collect the intermediate fraction until the head temperature reaches 245°C.

      NOTE: To prevent possible "Freezing" during this stage of the distillation, turn on electrical current in the circuit to the distilling head when the head temperature reaches ca. 230°C.

   c. Fraction 3 (Tetrachlorobenzene Fract.): Collect the distillate at the head temperature 245-255°C. into a dry, tared 500 ml. Erlenmeyer flask.

IX. SPECIFICATIONS AND TEST METHODS, contd.

Test for Composition by Distillation of Tri-Tetrachlorobenzene Blend, Method No. 12,036-53, contd.

NOTE: An additional heat on the distilling head may be required during part of this fraction.

d. Fraction 4 (2nd Intermediate Fract.) - Tetra-Pentachlorobenzene Fract.): Collect all distillate from 255-275°C. in a dry, tared 250-ml, Erlenmeyer flask.

NOTE: If the Pentachlorobenzene content is not required, the distillation may be terminated at this point. If the "Penta" is desired then proceed as follows:

e. Fraction 5 (Pentachlorobenzene Fract.): When the head temperature reaches 275°C., replace the fourth fraction receiver with a dry, tared 250-ml. Erlenmeyer flask and collect all distillate until the head temperature is 277°C.

6. Stop the distillation. Turn off electric current from the pot heater only. Allow half an hour for the head and column to drain. Turn off the current on all units.

7. Weigh and record all the fractions collected.

8. Upon weighing, determine a crystallizing point of Fract. 3 using a 20-55°C. range, 3" immersion thermometer. Record the highest temperature reached after crystallization has taken place. Determine from Dwg. No. A-5538 on page IX - 173 below the 1,2,3,4- and 1,2,4,5-Tetrachlorobenzene isomeric content of this fraction corresponding to the crystallizing point of the fraction. Record percent for each of the isomers.

9. After the still-pot cools down, determine weight of the residue as follows:

wt. of Residue (gms) = [wt. of Pot - (wt. of "Heel" + still-pot Step 1)]

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-0968 Scrubber liquor pump tank purchased from Nooter. Dwg. C-4616. Steel vertical jacketed, size 4'6" ID x 5'6" long with jacket. Order B15673 on 8/2/37. Req. 81458 on 8/2/37. Price $575.00.

CT-01013 #1A scrubber liquor pump tank made by Leader. Dwg. C-4616. Size 4'6" ID x 5'6" vertical, steel, flange quality, all welded. Jacketed. Order B 4164 on 4/29/38, Req. 87029 on 3/9/38. Price $582.00 On 38' level SE corner CR.

CT-01014 Scrubber tank made by Heintz. Dwg. C-4845. Steel 12" dia. std. wrought steel pipe 10'10" high, ½" dished end and other end fitted with 3/4" faced and drilled flg. with 5/8" cover plate. Steel plate, and distributor. Use 5 cu. ft. 1" CI Raschig rings pkg., Order B3933 on 3/4/38, Req. 86921 on 3/4/38. Price $139.00. On top of CT-01013, #1A scrubber.

CT-01162 Scrubber tank made by Heintz. Dwg. C5743. Welded steel scrubber, size 12" ID x 5'. Order B19519 on 9/27/39, Req. 1977 on 9/19 Price $102.00. Vent line to CT-0848. (*)

CT-01163 Catch Tank 55 gal. steel drum. For CT-0762. Mate to CT-01164. Chg. out $11.40. 11/27/39. (*)

(*) Both CT-01162 and CT-01163 are on vents of CT-01321 and 02621, Pyranol Plant.

CT-01164 Catch Tank. Same as CT-01163.

For CT-01162, Pyranol Plant.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-01258 Monel gas chamber made by Nooter. Dwg. C-5825. 4' dia. x 4' high constructed of Monel. Order B7835 on 4/3/40, Req. 8061 on 3/27/40, Req. 8061 on 3/27/40. Chg. out $867.00. On gas exit from fire heated chlorinator.

This monel vessel is at the lowest point of the gas exit line from the gas-heated chlorinator. Solid Aroclor which collects in it is removed by hand and returned to the chlorinator with a subsequent batch.

CT-01259 Cyclone Tank made by Heintz. Dwg. B5821. 2'6" dia. x 6'7" high constructed of 3/16" steel plate. Order B7345 on 4/27/40. Req. 8062 on 3/27/40. Price $92.00. Hang from roof of Bldg. on 38' level.

CT-01259,01279, 01280, 01281, 01282, cyclones on chlorinator off-gas, follow the scrubbing towers CT-0759 etc. Drain through S. G's to the return seal pipes between towers and circulation tanks. Off-gas to the main leading to the HCl absorption systems.

See drawing B-5821 (This drawing not yet found April 5, 1947). See drawing B-660.

CT-01263 Monel condenser made by Nooter. Dwg. C-6144. 42" dia. x 63" high cone bottom condenser constructed of 3/16 Monel metal welded construction throughout. Order B-8364 on 4/9/40. Req. 8198 on 4/1/40. Price $928.00

This vessel is obsolete.

CT-01279 Cyclone separator made by Nooter Dwg. 5821. 2' dia. x 6'7" high cyclone separator made of steel and welded throughout. Order B13177 on 6/12/40. Req. 10232 on 6/5/40. Chg. out $66.25. For use at CT-0968.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-01280 Cyclone separator made by Nooter. Dwg. B-5821. 2' dia. x 6'7" high cyclone separator made of steel and welded throughout. Order B13177 on 6/12/40. Req. 10232 on 6/5/40. Price $66.25/ At CT-0762 east scrubber.

CT-01281 Cyclone separator. Same as CT-01279 and 1282. At CT-0759. 2nd from E scrubber.

CT-01282 Cyclone separator. Same as CT-01279 - 1280 - 1281. Used at CT-0760. 3rd from E scrubber.

CT-01283 Chlorinator made by Leader Iron Works, Inc. Dwg. E6209, 3' x 16' steel vertical chlor. welded const. Order B-14119 on 6/21/40, Req. 10408 on 6/11/40. Price $720.00 East of CT-0833.
#4 chlorinator. See CT-0744.

CT-01310 #5 Aroclor 1260 Storage Tank made by Leader Iron Works. Dwg. C-609. Horizontal 10' OD x 26' long on str. shell, incld. coils, etc. Omitted 2 - 3" nozzles changed 18" noz. to 24" and added covers for 24" noz. Tanks, coils, coil supports, inside of noz. and manholes and manholes and noz. and inside covers must be sprayed with zinc 0.005" thick followed by a sprayed coat of 0.003" tin. Order B13240 on 6/11/40, Req. 10432 on 6/11/40 Price $2200.00. Underground S of CT-0848. Pump P-0929.

CT-01321 Pyranol Mixing Tank made by Nooter.
Drawing D6455.
14' OD x 10' high steel tank with 3/8" shell, 1/2" std. knuckle radius heads, entire interior sprayed with min. thickness of .015" tin.
42" Quadruplex turbo mixer complete including Turbo adaptor assembly -- off set from centre.

(1) Order B20976 on 9/27/40. Chg. out $2930.00
(2) Order B21762 on 10/8/41, Req. 13664, Cost $740.00

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-01321 contd.

Driven by MSC-01288, Motor M01718.
1½" steam coil, 45 turns, 18" diameter, through the cover.
9600 Imp. gallons. Outdoors. Everdur or galvanized piping. Bronze valves.

Bottom outlet to Taber pump P-01540 delivering to filter press F-0159. Capped inlet on cover for Attapulgus Earth. Manlid. Steam coil. Dial thermo. Vent to soda-lime vent box. Heat insulation (gone).

CT-01377 Still receiver tank made by Nooter. Dwg. C-795, B-7082, like CT-0771. PA #145. 5'6" x 5'6" Monel metal receiver, complete with cover, coil, etc. Glass wool insulation 2" and ½" finished coat.
Order B4137 on 6/27/41, Req. 18315 on 2/17/41.
Price $3295.00. NE corner of 12' level platform in Bldg. CR. Is used on #2 still S-062.

CT-01378 Condenser tank made by Nooter. Dwg. B651.
12" ID x 5' long. Monel metal.
Order B4137 on 10/20/41. Req. 18315 on 2/17/41.
Price $650.00 W of CT-01413 on 20'6" level platform, Bldg. CR. Like CT-0770. Used on #2 still.

CT-01396 Vacuum scrubber purchased from Combustion Engineering Co. Drawing C-807. 3' ID x 10' high, steel construction including grating and screen.
Order B8360 on 4/4/41, Req. 19989 on 4/4/41
Price $260.00. N one on 29' platform.
Like CT-0753. On #2 still, coke scrubber.

CT-01397 Water heat exchanger made by Leader Iron Works.
Dwg. B-483. 6" dia. x 10' long, 12 tube.
Order B8324 on 4/4/41, Req. 19993, Price $119.00
North of CT-0831. On #2 still. Spare.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-01398 Cooler tank made by Leader. Dwg. B644.
2'6" ID x 3' high steel cooler tank complete with coil.
Order B8324 on 4/4/41, Req. 19993. Price $152.00
South one on 29' platform
Used on #2 still. Vented.

CT-01399 #2 blending tank made by Nooter. Dwg./D-10290, general arrangement. 7' ID x 7' high, complete with coil, agitator, and drive support, zinc and tin lined, seams to be chipped and ground smooth. E-6839 and
Order B8325 on 4/4/41, Req. 19990 on 4/4/41
Chg. out $950.00. Driven by M-02170. Middle one of 3, north end of 12' level platform.
Vertical bearing for agitator shaft, A-6853.
Compare CT-0779.
Newport experience shows the need for adequate heating and agitation in blending higher Aroclors.

CT-01400 #2 filtrate receiver, made by Nooter.
7' ID x 7' high incl. coil and supporting legs.
Inside of tank zinc sprayed, all seams chipped and ground. 1" glass wool insulation 2½" fin. coat.
Order B8323 on 4/4/41, Req. 19992, Price $925.00
West one of 3 north end of 12' level platform, CR.
Dwg. C-6863. See CT-0780.

CT-01413 Separator tank made by Heintz Steel. Used on #2 still.
Dwg. C6957. 3' ID x 6' high tangential separator with integral vapor pipe complete.
Order B10722 on 5/9/41, Req. 20429 on 5/1/41.
Chg. out $382.50. North one on 29' level platform Bldg. CR.

CT-01432 5th chlorinator. See CT-0744. Nooter. Drawing E6209.

CT-01491 Heat exchanger tank made by Leader Iron. Dwg. B483, CABI #217. 6" dia. x 10' long, all steel, 12 tubes
Order B21479 on 9/19/41, Req. 24894 on 9/19/41.
Chg. out $140.00. Used on #2 vacuum still system.

XII. EQUIPMENT LIST, contd.

Closed Tanks, contd.

CT-02086 Air blowing tank. Like CT-0750.

CT-02404 This store tank has been set up outdoors to hold the tri-tetra-chloro benzene mixture, which is bought in rail cars and unloaded by pump P-01111. The tank is a vertical cylindrical vessel 13'0" ID, 20'0" high on straight side with dome top and bottom.
Total capacity 20,000 U.S. gallons. It has a bottom outlet, with a strainer box, to pump P-02077 delivering to either of the Pyranol blending tanks. The tank has a Varec depth indicator with stainless steel exposed parts, a vent pipe, a dip pipe for air blowing, an internal steam coil, and external heat insulation.

NOTE: Mr. Barbre, 1947, said that a batch tank to hold filtered Pyranols, pending final approval, would be a convenience.

CT-02621 An additional blender, CT-02621 has been installed outdoors for making Pyranols. It is a vertical cyclindrical steel tank 10'6" ID 23'0" high with flat top and bottom. Total capacity 14,800 U.S. gallons. The bottom slopes 2" to the bottom outlet, which leads through a strainer box to a Taber pump P-02179, delivering to the filter press, F-0172.

The tank has a 7½ H.P. Lightnin mixer installed horizontally low down through the straight side, the exposed parts of the mixer being metallized with 0.015" of aluminium. The vessel also has a Varec depth gauge with stainless steel float etc, a vent pipe to a soda lime vent box, an internal steam coil with T.I.C., item I-01894, set at 50°C. (tin-sprayed thermo well), and external heat insulation. There is also a dip pipe through which dry air from the Lectrodryer FC-046 can be blown by a hose connection with Corby coupling to agitate and dry the contents of the tank. Attapulgus earth is introduced through a funnel fitted to an opening (with screw cap) in the top of the tank.

XII. EQUIPMENT LIST, contd.

Note: In MCC manuals the CT and OT items of equipment are listed under "T", e. g. Closed Tanks and Open Tanks.

XII. EQUIPMENT LIST, contd.

Dryers

D-012 Flaker, Devine, 48" dia. x 30" long, drum 5/8" thick, rolled steel, machined and polished. Housing to be of #16 ga. monel.
Order B 7378 on 4/14/36, Req. 69124 on 4/14/36.
Price $1668.00 quot. 3/17/36.
In lean-to on floor on found. of concrete.
Driven by M-0827 - 3 HP Wagner motor thru MSC-0639.
Jones Speed Reducer. The pan is heated by gas flames.

Sketch showing drum side-knife, dip pan and essential dimensions on 48" diam. x 30" long steel drum, monel hood flaker -- drg. B-675, chutes for finished product. Drg. D-518.

See drawing B-374. General arrangement, and C-827, weighing truck.

Dryer for Attapulgus Earth.

In 1946 a vertical steel tank with internal steam coils was installed at the Krummrich plant. It was still the only equipment for drying earth at the Krummrich plant in 1955, but reliance was placed mainly on supplies drawn from Anniston. With the Krummrich plant dryer, the earth was fed in through a hand-hole in the cover, and withdrawn, as required, by means of a twin damper arrangement on the wide bottom outlet.

See hand sketch sent to MCL April 1947.

At Anniston the earth is screened, 20 mesh, then dried in trays about 3" deep, place in an electrically heated cupboard. Each tray contains a weighed amount, so that the correct charge of earth can be put into the treatment tank directly from the dryer.

XII. EQUIPMENT LIST, contd.

Dryers, contd.

Dryer for Attaplugus Earth, contd.

In 1947 at the Krummrich plant the Attapulgus earth was being dried in a vertical cylindrical steel vessel, with cone bottom, and an internal steam coil. The earth was fed in through a hand hole in the cover and withdrawn as wanted through twin dampers in the bottom. See the sketch sent to MCL with the drawings collected in 1947.

The cover of this dryer had a vapour vent, but there was no positive movement of air through the vessel, and the electrically heated shelf dryer used at Anniston (see page 5 of the report "Notes on the Anniston Aroclor plant" Havercroft and Mather, September 1947), would appear more likely to give thorough drying. The steam heated dryer was still in use at the Krummrich plant March 31, 1954. The reference of Schwarting and others, November 12, 1953, page 10, to an electrically heated dryer refers to a still earlier unit which had been abandoned.

XII. EQUIPMENT LIST, contd.

Filters

F-0106 Steel filter purchased from Nooter. Dwg. C-3775, Size 12" dia. x 3'10-3/4" high. Leg supports, glass wool packing. Order B3511 on 2/19/36. Req. 67729 on 2/14/36. Price $56.00 quot. 2/5/36.

Formerly used on chlorine gas, but used in 1946 to filter out iron etc. impurities from the HCl gas coming from other departments through a long steel line.

F-0113 Sperry 24" bronze filter press. Cover for electric oven heater for press --- Drg. B-685. Replaced by Sparkler press F-0172.

Steel support and pan for Sperry filter B-7121. 3/4" pipe coil for filter catch pan -- A-7136.

F-0159 General Electric Filter Press. Serial #6186968. Type FP30. Accessories included. 3 HP motor and 4 compartment drying oven. Form G, Cap. 25/30 gal. per min. Size 12", 20 chambers. Heated and insulated steel cover* with pullies, cable, counter weight and strip heaters, Pyranol plant.

Order B18935 on 9/21/39. Req. 1991 on 2/20/39. Chg. out $984.00 on 11/30/39.

F-0172 Sparkler filter. Serial 484. Model 18-d12 all bronze construction with improved Scavenger plate, plates to be arranged so entire set can be removed. Steam tracing inside the lagging, then a metal shield over all. Steel legs and floor flanges for bolting to floor. Served by pump P-0984.
Order B10302 on 4/2/41, Req. 20430 on 4/30/41.
Price $830.00 per quot. 3/19/41.
On ground floor center of Bldg. CR.

Installed for Aroclors, but later transferred to use on Pyranols. Chain hoist, MSC-01495, provided for lifting out the nest of plates.

* MSC-02108.

XII. EQUIPMENT LIST, contd.

Filters, contd.

F-0172, contd.

Samples of filter paper for the Sparkler press F-0172, which was then being used on Aroclors, bought under the reference "#21-18 Filter paper", $2.755 per 100 sheets, Sparkler Mfg. Co. Mundelein, were sent to London, April 9, 1947,

F-0172 was moved to the Pyranol department and replaced by a 33" Sparkler. See Operating Instructions 8/10/54, and Mr. Harden's notes of August 1950.

F-0238 Sparkler Filter, 330-12 cell type, 304 Stainless steel, with jacket for 30 psig.

33" Sparkler press for Aroclors installed in place of F-0172, F-0172 being used for Pyranols.

F-0239 Sweetland, Size 10 RH, 54 leaves. 523 sq.ft. C.I. body, lined with thick nickel. Nickel valves and outlet line, with pump P-01757. Out of use in 1955. (Pyranol plant).

XII. EQUIPMENT LIST, contd.

Furnaces

FC-045 Gas heater for No. 1 still. Steel shell 3/16" x 3'9" dia. x 4'10½" long, lined with insulating fire brick, etc. 1 coil 2" xHy pipe, 12 turns formed into a 2' dia coil with spare blocks and legs. Drawing E-886. Req. 69859 on 5/14/36, Order B9665 on 5/29/36. Used to heat material in S-043, pumped thru coil by P-0593.

Vent stack drawing B-689 (could not be found in 1946.) Piping details, drawing C-815, was sent to London April 24, 1947.

At plant B in 1946, there were two natural gas burners aimed tangentially into the bottom of each furnace, and there was some evidence of damage due to overheating of the lowest part of the coil. The central "dummy" inside the coil was made as a solid cylinder of firebrick and plastic refractory material built up on a circular plate suspended on a central steel lifting bar. Head room and a suspension hook are required over each furnace, for maintenance work.

The material from the still is pumped downwards through the coil in the furnace of the new still, but upwards in the old still. The main reason for this is that this arrangement enables the coil of the deep still to be on the same level as that of the shallow one.

At Anniston the material is pumped upwards because this puts the coldest Aroclor in at the bottom where the coil is most in danger of overheating. This can have little advantage, except towards the end of the run, when the temperature spread between the inlet and the outlet is greater.

There is a thermo-recorder pocket in the inlet of each coil, and another in the outlet. There are valves on the circulation lines, by which the coil can be sucked empty. See Section VI, page 18. These also help in locating any blockage in the circulation system.

XII. EQUIPMENT LIST, contd.

Furnaces, contd.

FC-045 contd.
Mr. Furzey reported, December 5, 1951, that the coils last about 12 months, but may require cleaning every 3 or 4 months to remove plugs.

At Newport the still coil is interchangeable with that of the diphenyl department still.

A temperature difference recorder controller was installed on the heater coil at Newport, and a CO_2 connection made to the furnace for emergency use.

At Anniston there is a 12-point Micromax temperature recorder on the chlorination system, and one for each of the two vacuum stills.

FC-046 Electric, Air Drying Furnace purchased from Lectrodryer Corp. Serial 270, Size BWC-250, dual-absorber type. 40# pressure. 440 volts, single phase, 60 cy. 4-8 hours operation. 300 amp. heater on each side thermostatically controlled, at 270°F. See T-014. Order B7403 on 4/14/36, Req. 69119 on 4/14/36.
Price $1,350.00 on 5/30/36. Has blower with Motor M-01486 for scavenging. Serves the air-blowing tanks CT-0750 and CT-02086, and the melter CT-0763, in the latter case at pressure enough to blow the charge to the chlorinator. Brings the dew point of the air to between - 10°F. and + 10°F.

Lyles, Soffranko and Miller (Pyranol report March 24, 1947, page 60) state that the dry air should be checked twice a month for its moisture content, and that the alumina needs renewal about once a year.

FC-066 Steel Heater Furnace for No. 2 still. Drawing C-3479. Brick lining, coil, burners and stack. S. W. corner of the 20'6" level platform, Bldg. CR.
Generally like FC-045.
Drawing C-3479 (missing April 1946).

XII. EQUIPMENT LIST, contd.

Instruments

I-016 Taylor Temperature Controller. Drawing D4417, Size 1, #36JR323. Direct acting, self acting, with std. double seated valve incl. steam strainer. Range 110°F. to 170°F., Set 122°, 25' tubing 3/4" union hub connection placed 7' from bulb end.
Order B3443 on 3/10/37, Req. 76979 on 2/15/37.
Chg. out 3/31/31, $75.57.
On CT-0848, Pyranol blender.

I-0108 Rotameter Instrument purchased from Economy Equipment Co. Serial #10166. For scrubber TW-0182. No. 6, Fig. 25 bronze with stainless steel float calibrated for max. cap. of 300 gph water.
Order B19959 on 11/10/37, Req. 83079. Price $66.70

I-0204 Foxboro. DP cell. Stainless Steel body.
Ni diaphragm.

I-0244 TIC, L & N.
2 point Micromax. Range changed to 0-600°C. Used to replace MSC-0668.

I-0381 Palmer Supercar Dial Thermometer, 8" cone Mercury actuated. Range 0-150°C., 25' stainless steel connecting tubing with adj. clamp, flg. to be located 9' from end of bulb.
Order B18385 on 8/23/40, Req. 12533 on 8/23/40.
On 1269 stg. tk. CT-01310. Price $56.50

I-0398 Fulton Sulphon Temperature regulator #931 with 6A bulb and 20' of capillary - Bronze body stainless steel trim, to be used on steam at 65# pressure. Range No. 76K-190F. to 250°F. Size 1, press N. G. on CT-0748 diphenyl stg. tk. Order B23310 on 10/28/40, Req. 14583 on 10/28/40. Price $69.05

I-0441 L & N 6 point temperature recorder. Serial #389329, #40352M, Model S, micromax strip chart temperature recorder. Type DC potentiometer - multi point curve printing, record numbered and color dot for each

XII. EQUIPMENT LIST, contd.

Instruments, contd.

I-0441 contd.
thermocouple. Time cycle - 30 sec. normal for each point.
Order B13260 on 6/18/41, Req. 21072 on 6/17/41.
Price $395.00.
In second floor control room, Bldg. CR.

I-0443 Watt hour meter instrument purchased from General Electric.
Serial 21-220-743. #85 x 905. Type V3A, 220 to 5 CT use.
Order B 9118 on 4/15/41.
Req. 20313 on 4/14/41. Chg. out $51.40 on 6/30/41.
In cabinet outside E wall Bldg. CR.

I-0507 Palmer Dial Thermometer. CABI #217, Range 0-150°C.
in Pyranol Mixing Tank CT-01321. 15 ft. stainless steel
special armor flexible tubing plain bulb. Order B20438
on 12/1/41, Req. 24425 on 9/9/41.
Chg. out $49.59 on 12/27/41.

I-0835 Bailey Integrating and Recording Flow Steam Flowmeter.
Serial #13996R. Order B11589 on 9/25/45, Req. 67905 on
5/11/45. Price $348.00
In control room E wall. For steam from the 200 lb. main.
Supplies the ejectors, the high pressure coils in the
air-blowing tanks, and melter. See MSC-0682 for the low
pressure steam meter.

Taylor Fullscope recording controller on the No. 1 still
vacuum system.

Chlorine flow recorder controller on No. 5 chlorinator.

See also MSC-0663, page 61.
0668, page 61.
0675, page 61.
0682, page 63.
0705, page 63.

I-01118 Foxboro steam flow controller, 0 - 2000 lbs. per hour.

XII. EQUIPMENT LIST, contd.

Instruments, contd.

I-01547 Taylor PR, 2 pen, with alarm and shut off valve. Chlorine lines. 0 - 100 psig.

I-01894 Taylor Fulscope TI C. 0-150°C. working at 50°C. Tin-sprayed thermo well. On blender CT-02621. Controls Fisher diaphragm valve on steam to coil in CT-02621. Pyranol blender.

I-01941 Foxboro. D.P. cell for chlorine liquid in store tank.

I-02019 Taylor Fulscope LRC.

I-02045 Worthington meter, gallons, well water.

See also MSC - 0663
0668
0675
0682
0705
0770
01393

XII. EQUIPMENT LIST, contd.

Elevators

L-031 Bucket elevator from General Conveyor & Mfg. Co. Drawing E-870. Lg. to be 22'4-11/16" OA c to c take up shafts and 20'6" casing and head section 3/16" and boot 1/4". 6" belting, 4 ply R covered pinched 10-7/32" for buckets of 4½" x 3" chrome steel. Order B10455 on 5/15/36, Req. 70140 on 5/25/36. Price $491.20. Driven by M-0834. Drive drawing B-661. Solid Aroclors (dismantled 1952).

See also V44, 45, 46 conveyors from flaker D-012 to elevator L-031, and chain hoist MSC-01495 over the Sparkler filter E-0172, and electric hoist MSC-0655 for solid Aroclors.

A chain hoist is used also to lift bags of lime to the still operating platform.

XII. EQUIPMENT LIST, contd.

Motors

Note: The motors are changed to other duties from time to time, but the following details will serve to indicate the types of motors used.

M-0790 Westinghouse Motor 3 HP-1750 RPM. Serial 7435. Type CS, sq. cage induction motor, horiz., frame W225, 220/440 V, 3 ph., 60 cy., 8/4 amp., 55°C. temp., rise, style 801068Y, class 1, total enclosed, ball bearing. Rec'd. 8/20/35. (#6058-5077) $78.96. On pump P-0346.

M-0827 Motor 3 HP 1200 RPM from Wagner Electric Co. Serial #1718199, horizontal induction motor, totally enclosed, fan cooled, ball bearing, 3 ph, 60 cy, 220/440 volts, 8.2/4.1 amp., type CPI, frame 254, model W425198. Order B8184 on 4/23/36, Req. 69376 on 4/23/36. Chg. out $94.16 Also drives V-046 thru second chain on link belt; drives D-012 flaker thru MBC-0639 speed reducer and chain.

M-0830 Motor 2 HP 1800 purchased from General Electric. Serial HK-1407, Horizontal induction, totally enclosed, fan cooled ball bearing, frame 225, 2HP 1740 RPM, 220/440 volts, 5.61/2.81 amp., type K, 3 ph., 60 cy., model SK-225-A715. Order B8169 on 4/23/36, Req. 69377 on 4/23/36. Chg. out $73.84 on 6/30/36. Drives P-0565.

M-0832 Motor 3 HP 1200 RPM purchased from Wagner Electric. Serial 1714954. Horizontal induction, totally enclosed, fan cooled, ball bearing, 220/440 volts, frame 254, 3 ph., 60 cy., type CPI, model W42J198, 8.2/4.1 amp. Order B8178 on 4/23/36, Req. 69378 on 4/23/36. Chg. out $94.16 on 6/30/36. On blower B-094.

M-0834 Geared head motor 2 HP 1200 RPM purchased from Master Electric Co. Serial HE-975, air jacketed, totally enclosed, fan cooled, 1140 to 220 RPM single reduction parallel geared head, 220/440 volts, 3 ph., frame 254, type PA, style 47476, 55° cont. rating, 6.2/3.1 amp. Drives L-031. Order B8156 on 4/24/36. Req. 69373. Chg. out $109.14.

XII. EQUIPMENT LIST, contd.

Motors, contd.

M-0835 Reeves vertical motodrive 1½ HP 1800 RPM purchased from Bates Co. Motor serial 1914691, motodrive unit serial MD-1136. Size 4253-E-18, model V 34J223, 3ph, 60 cy., 1.9 amp., frame 224, 4 to 1 speed variation and 50 to 1 gear reduction, hand chain control and chain in place of standard hand wheel.
Order B 7376 on 4/14/36, Req. 69123.
Charged out $300.96. Drives V-044.

M-0836 Reeves vertical motodrive, same as M-0835. Drives V-045.

M-0841 Geared head vertical mounted motor 5 HP purchased from Master Electric Co. Serial HE-988. 220/440 volts, 3 ph., 60 cy., totally enclosed, fan cooled, ball bearing, type PA, frame 11230 # 254 DP - 1725 to 80.2 RPM
Style 47478 - cont. 55°C.
Order B 8155 on 4/24/36, Req. 69375 on 4/23/36.
Chg. out $211.84 on 6/30.
Drives stirrer in blending tank CT-0779.

M-0846 Vertical mounted induction motor. 1 HP 1800 purchased from General Electric. Serial EP-2773. Totally enclosed fan cooled, ball bearing, thrust type with ring base and drip cover, 3 ph., 60 cy., 220/440 volts, 1720 RPM.
Model 5K204A1550. Frame 204Y.
Order B 8175 on 4/23/36, Req. 69371.
Chg. out $55.44. Drives P-0573.

M-0847 Vertical mounted induction motor 1 Hp 1800 purchased from General Electric Co. Serial EP-2779. Totally enclosed, fan cooled, ball bearing, etc. same as M-0846.
Drives P-0909.

M-0848 Vertical mounted induction motor 1 HP 1800 purchased from General Electric Co. Serial EP-2777. Totally enclosed, fan cooled, ball bearing, thrust type with ring base and drip cover, 3 ph., 60 cy. 220/440 volts, 1720 RPM, model 5K204A1550, frame 204Y.
Order B8175 on 4/23/36, Req. 69371. Chg. out $55.44
Drives P-0674.

XII. EQUIPMENT LIST, contd.

Motors, contd.

M-0849 Wagner horiz. induction motor 5 HP 1800 Serial #1714362, totally enclosed, fan cooled, ball bearing, type CPI, frame 254, model W-30J195, 3 ph., 60 cy., 220/440 volts 1750 RPM, 12.6/6.3 amp.
Order B-8177, Req. 69372, chg. out $97.08 6/30/36
Drives P-0578. On pump P-0578, on No. 2 chlorinator.

M-0850 Horizontal induction Wagner motor 5 HP 1800 Serial 1718376. Same as M-0849 and 0851. Drives P-0579. On pump P-0579 on #3 chlorinator.

M-0851 Motor same as M-0849 and 0850. Serial 1718377.

M-0853 Vertical mounted induction motor 3 HP 1800 G. E. Makers. Serial FP-1104. Totally enclosed, fan cooled, ball bearing, thrust type with ring base and drip cover, 3 ph., 60 cy., 220/440 volts, 1730 RPM, model 5K225A2430, 8.54/4.27 amp.,frame 225Y, type K.
Order B 8174 on 4/23/36, Req. 69367.
Chg. out $95.92. Drives P-0583

M-0854 Motor Serial FP-3898 same as M-0852 and 3, on CT-0768.

M-0856 Vertical mounted induction motor 3 HP 1800. Made by G. E. Serial FP-2457. Thrust type and drip cover, ring base, class 1, group d, underwriters #5474727. Explosion proof, ball bearing, 1720 RPM, 3 ph., 60 cy., model 5K225A2446, 220/440 volts, 8.42/4.21 amp., frame 225Y, type K
Order B8172 on 6/26/36, Req. 69368 on 4/23/36.
Chg. out $115.58 on 7/13/36. Drives P-0585.

M-0871 Vertical induction motor 5 HP - 1750 RPM from G. E. Serial FP-3177. Totally enclosed, fan cooled, ball bearing, thrust type with ring base and drip cover. 220/440 volts, 13.9/6.95 amp., frame 254 Y, type K, 60 cy., 3 ph., model 5K254B1930.
Order B8173 on 4/23/36, Req. 69369. Chg. out $124.21 on 7/31/36. Still pump drive, drives P-0593.

XII. EQUIPMENT LIST, contd.

Motors, contd.

M-0873 Vertical mounted induction motor 5 HP - 1750 RPM. Purchased from G. E. Serial FP-3107. 220/440 volt, 60 cy., 3 ph., totally enclosed, fan cooled, ball bearing, thrust type with ring base and drip cover. Model 5K254B1930, 13.9/6.95 amp., frame 254Y, type K. Order B8176 on 4/23/36, Req. 69370. Chg. out $124.21 on 7/31/36. Drives P-0592 in tank outside.

M-0875 Horizontal induction motor 1 HP 1200 made by G. E. Serial DP-5227, totally enclosed, fan cooled, 60 cy., 1130 RPM, model 5K204A1036, 220/440 v., 3.45/1.73 amp., frame 204, type K, 3 phase. Order B13188 on 7/2/36, Req. 71152, chg. out 7/21. Drives P-0595.

M-0885 Vertical mounted induction motor 1 HP 1800 purchased from Continental Equipment Co. Serial 50498. Totally enclosed, fan cooled, ball bearing, thrust type with ring base and drip cover. 2.9 amp. 3 ph., 60 cy., 220/440 v., 1750 RPM, type NCU20. Ordered for spare. Drives P-0604. Price $48.00. Order B-13196, Req. 71147 on 7/1/36.

M-0904 Motor 3 HP - 1200 purchased from Continental Electric Co. Serial 51704. Totally enclosed, fan cooled, normal starting current, squirrel cage induction motor, 3 ph., 60 cy., 220/440 volts, 1150 RPM, 55° cont. rise, type NF 254, 8.4/4.2 amp. Order B17431 on 9/1/36, Req. 72637, Chg. out $90.40 on 10/24. Drives B-0105 in Bldg. CR.

M-0914 Blackmer motor 7½ HP 1800 made by Louis Allis. Serial 217543. Totally enclosed, fan cooled, type JX, 3 ph, 60 cy., 220/440 volts, 1750 RPM, frame 284, class 53d, form B, 18.4/9.2 amp. Order B21241 on 10/22/36, Req. 74034, Chg. out $133.12 Drives Naphthalene pump P-0617 for CT-0347.

XII. EQUIPMENT LIST, contd.

Motors, contd.

M-0955 Portable "Lightnin-Mixer" 1/4 HP 1725 purchased from Mixing Equipment Co. Mixer Serial 37108, Motor Serial HK-4536. CABI 106/1936. Model C-4, 3" folding monel metal propeller monel shaft, 4/2 amp., 110/220 volts, single phase, 60 cy., class 1, group d, explosion proof, underwriters F-519372, extension cord and attachment plug.
Order B1340 on 1/19/37, Req. 76212 on 1/18.
Chg. out $109.42 on 2/27/37. Used for drum shipments.

M-0975 Motor 5 HP 1800 purchased from Wagner Electric Co. Serial 1725358. Totally enclosed, fan cooled, ball bearing, induction motor, type CPI, frame 254, model W30J247, 3 ph, 60 cy. 220/440 volts, 12.6/6.3 amp. cont. rating 55°C.
Order B2834 on 2/9/37, Req. 76765 on 2/5/37.
Cost $95.61. On P-0629. On No. 1 chlorinator pump P-0629.

M-0985 Wagner Vertical Motor 7½ HP - 1800 RPM. Serial #1728961. Totally enclosed, fan oooled, ball bearing, type CP-2, frame 234Y, 440V, 60 cy., 3 ph., 18.4/9.2 amp. 1750 RPM, model W40V143.
Order B3323 on 2/12/37 and Req. 76927.
Chg. out $133.98. Inst. to drive submerged pump in TCB stg. tank CT-0848. On P-0346.

M-01229 Vertical mounted induction motor 1 HP 1800 purchased from Louis Allis. Serial 269797. Totally encl. fan cooled, ball bearing, thrust type with ring base and drip cover, weatherproof conduit connection box, type IS, frame 204V, class L, form B. 220/440 volts, 3 ph, 60 cy., 3/1.5 amp. cont. 55°C., 1730 RPM. Order B3853 on 3/4/38, Req. 86889 on 3/3/38.
Chg. out $55.20 on 4/29/38.
On pump P-0731.

XII. EQUIPMENT LIST, contd.

Motors, contd.

M-01377 G. E. Motor 2 HP - 1800 RPM. Serial MR-8969. Totally enclosed, fan cooled, sq. cage, induction motor, type K, ball bearing, 220/440 V, 60 cy., 3 ph., frame 254, 1760 RPM, model 5K254B91. 5.44/272 amp. Order B15955 on 9/26/38, Req. 91711, Chg. out $75.00. On P-01111.

M-01456 Westinghouse horizontal motor 3/4 HP 1200 Serial 4139. Totally encl. fan cooled, low starting current, 440 volts, 3 ph., 60 cy., 1160 RPM. Type CS, frame 204, 2.6/1.3 amp., style 1707136. 55°C. rise, Chg. out mach. $41.03 on 12/29/39 Order B17367 on 12/18/39. Req. 1357 on 8/31/39 on Blower B-0148.

M-01469 G. E. Motor 3 HP-1200. Serial #KT-10671. Totally encl. fan cooled, model 5K254A1024, frame 254, type K, 220/440V, 3 ph., 60 cy., 1165 RPM, 9.06/4.53 amp., cent. 55°C. rise, Order B18935 on 9/21/39, Req. 1991 on 9/20/39. Chg. out $72.47 on 11/30/39. On gear pump to F-0159.

M-01486 General Electric Motor 1/4 HP 1800, totally encl., model 5 KH47AB984, type KH, 110 volts, single phase, 3.4 amp., 55° Cont. Order B17923 on 10/17/39, Req. 1531 on 9/8/39. Cost $24.25. On Lectrodyer FC-046.

M-01501 Replaced by M-01706.

M-01573 Motorized car spotter 5 HP 1640 RPM purchased from Link Belt Co. motor serial 307939, spotter serial 5252. 220/440 volts, 60 cy., 3 ph, type IK, frame 225 VY, class 3, form B, 17.2/8.6 amp. 55°C. Order B26741 on 12/29/39, Req. 5128 on 12/29/39. Chg. out $373.86. On track 11 at load dock.

XII. EQUIPMENT LIST, contd.

Motors, contd.

M-01622 General Electric motor 7½ HP, 3600 RPM, Serial BU7108. Totally enclosed fan cooled, type KF, model 5KF254C25, frame 254, 220/440 v., 3 ph., 60 cy., 3435 RPM, Drives ML-045. Order B4118 on 2/20/40, Req. 6737 on 2/20. Chg. out $114.96.

M-01629 Vertical motor 7½ HP purchased from Turbo. Made by Westinghouse. Serial 2640. Totally enclosed, fan cooled, frame 284, class 1, RPM at full load 1735, cont. rating at full load 55°C. rise, 220/440 volts. 3 ph, 60 cy., 18.8/9.4 amp., style 1071192. Order B8337 on 5/20/40, Req. 8441 on 4/9/40, chg. out $112.66. On MSC-01205. Driving agitator in CT-0808, final chlorinator.

M-01650 Louis Allis motor 5 HP, serial 258334, 1750 RPM, type JS, frame 254, class 43N, form B, 220/440 v., 3 ph, 60 cy., 13/6.5 amp., full load 55°C. cont.
Order B26263, Req. 35432 on 12/24/37.
On Sparkler press pump.

M-01656 Wagner Electric Motor 5 HP 1800 RPM Serial 1758959. Horizontal totally enclosed, fan cooled, ball bearing, less base and pulley, type CPI, frame 254, 220/440 v., 3 ph., 60 cy., 1750 RPM, cont. rating 55°CP, model 1B8J247B, 13.2/6.6 amp. Order B12802 on 7/8/40, Req. 10231 on 6/5/40, chg. out $87.92.
Drives P-01056 Taber Horizontal pump.

M-01660 General Electric Motor 1 HP 1800 RPM purchased from Taber Serial GU1902. Totally enclosed, fan cooled, vertical mounted, ball bearing, thrust type, with ring base and drip cover, 3 ph, 60 cy., 220/440 volts, 1720 RPM, model 5K204A1550, frame 204Y, type K, 3.11/1.56 amp., cont. 55°C. rise
Order B13111 on 6/10/40, Req. on 6/10/40, chg. out $50.19. Drives new Taber sub. pump P-0909.

XII. EQUIPMENT LIST, contd.

Motors, contd.

M-01697 Motor made by Wagner 1/3 HP, CABI 169, 30" dia. type S-60WS, style 1249-4622 portable circulating fan, single ph., 60 cy., 115 volts, complete with guards, floor column, and cord. B16528 on 7/26/40. Req. 11797 on 7/26/40. Chg. out $60.58. Used on operating platform near chlorinator.

M-01706 Vertical mounted induction motor 3 HP 1800 made by G. E. serial JU3498. Totally enclosed, fan cooled, ball bearing, thrust type with ring base and drip cover, model 5K225A4264, frame 225VY, type K, 220/440 v., 3 ph., 60 cy., 1730 RPM, 8.54/4.27 amp., cont. 55°C. rise.
Order B18443 on 8/23/40, Req. 12560 on 8/23/40.
Chg. out $92.24 on 10/31/40. On P-0929 Taber.
Replaced by M-01501.

M-01718 Louis Allis Motor 7½ HP-1800 RPM, Serial 335985. Totally enclosed, fan cooled, weatherproof conduit connection box. Type JX, frame 284, 220/440V, 3 ph., 60 cy., 1750 RPM, Class D, form R, 19.6-9.8 amp., full load temp. rise 55°C. trine hours cent. Order B21071 on 9/30/40, Req. 13674.
Chg. out $113.00 on 10/31/40.
Drives (Falk Reducer MSC-01288) on Turbo Mixer for CT-01321.

M-01752 Motor 1 HP-1800. Serial 77640.
Totally enclosed, ball bearing, squirrel cage, weather-proof threaded type connection box, type CS, frame W-204, 220/440 v., 3 ph., 60 cy., 1750 RPM.
Order B26350 on 12/4/40 and Req. 15836.
Chg. out $36.98. On B-034.

M-01772 Lewis Motor 1/2 HP 3600 made by G. E. Serial J33, CABI 169, totally encl., model 5K49BG756A, type K, rise 55°C., 440 v., 3 ph., 3450 RPM, amp. 7.
Chg. out $25.00, Req. 16663 on 12/30/40.
Use with P-0378 on OT-0640.

XII. EQUIPMENT LIST, contd.

Motors, contd.

M-01811 Louis Allis Motor 2 HP 1800 RPM Serial 385335.
Totally enclosed, fan cooled, normal starting current, CI conduit connection box, less base and pulley. 220/440 volts, 3 ph., 60 cy., 1750 RPM, type JS, frame 225, class M, form R, code H, 5.6/2.8 amp.
Order B4136 on 3/7/41, Req. 18313 on 2/17/41
Chg. out $65.36 on 6/30/41.
On ground floor N. E. of F-0172 on P-01037.

M-01850 Louis Allis Motor 1 HP 1800 RPM Serial 395205
Totally enclosed, fan cooled, ball bearing, 220/440 v., 3 ph., 60 cy., 1740 RPM, type IS, frame 204, class L, form B, 3.1/1.55 amp.
Order B7411 on 3/26/41, Req. 19634 on 3/26/41.
Chg. out $36.98. On ground floor.

M-01856 Louis Allis motor 5 HP 1800 RPM Serial 391009.
Totally enclosed, fan cooled, vertical flgd. mounted (CI conduit connection box) normal starting current, 3 ph, 60 cy., 220/440 v., 1750 RPM, type JS, frame 254 VB, class N, form R, code H, 1.3/6.5 amp. full load temp. rise 55°C.
Order B6380 on 3/13/41, Req. 19213 on 3/13/41.
Chg. out $109.74 on 6/30/41. On P-0997 top of S-062.

M-01859 Unit heater from Am. Blower Corp. Serial 1939.
Order B8936 on 4/11/41 and Req. 20240.
Chg. out $48.73.
In MSC-01393.

M-01995 Motor 1/3 HP 1800 RPM made by G. E.
Explosion Proof, class 1, group d, underwriters No. SA352-094. Totally enclosed, fan cooled, ball bearing, type K, frame 45A, model 5K45AC1024A, temp. rise 55°C. trine rating cont., 440 v., 3 ph, 60 cy, 1725 RPM, 55 amp.
Order B4625 on 2/21/41, Req. 18520 on 7/21/41.
Chg. out $29.75 on 9/30/41. On P-01054.

XII. EQUIPMENT LIST, contd.

Motors, contd.

M-02170 Geared head motor 5 HP 1800 RPM. Made by Master Elec. Serial PD-9. Parallel, air jacketed, fan cooled, totally enclosed, frame # 254, 80 RPM on mixer shaft, barrel mounting for vertical operation, style 103543, 220/440 v., 3 ph., 60 cy., 1725 RPM, type PA, amp. 13/6.5, 3 ph, trine hours cont. FLTR 55°C. Order B3580 on 4/8/42, Req. 30060 on 2/10/42. Chg. out $259.25. On CT-01399, #2 blender.

M-02175 Office Fan.

M-02877 Drives P-01111 at loading dock - 5 HP.

M-03133 Drives P-01540.

M-04592 Drives Side entering agitator in #2 mix tank CT-02621.

M-04691 Drives P-02077 at TNCB storage.

M-04817 Exhaust fan - located in pyranol press room.

M-04025 Drives P-02077.

M-0514 Drives P-02251.

XII. EQUIPMENT LIST, contd.

Mills

ML-045 "Helix-Seal" impact mill purchased from Williams P. C. & Pul. Co. Serial #9394. Size #1.

Less drive pulley. Driven at 5500 RPM thru V belt drive furnished by Monsanto.

Order B2371 on 2/1/37. Req. 76578. Price $420.00. West lean-to.

Hand fed.

For solid Aroclors.

Dismantled 1952.

XII. EQUIPMENT LIST, contd.

Miscellaneous Equipment

MSC-0639 Speed reducer made by W. A. Jones Foundry, Serial 85957, #105DW, assembly #1, style 4, 36 to 1 ratio, low speed shaft to be 7½" instead of 4" extension.
Order B8242 on 4/24/36, Req. 69409 on 4/24/36,
Price $156.00
Driven by M-0827, drives D-012. OR, lean-to.

MSC-0655 Shepard Electric Liftabout Hoist. Serial 36306.
440 volts, AC, 3 ph, 60 cy., single speed control with motor drive trolley. Hoist speed 18' per min., lift 19'. Hoist motor 3 HP. Travel motor 3/4 HP.
Order B7405 on 4/14/36, Req. 69120.
Chg. out $906.70.

MSC-0663 Multiple temperature recorder 40112-291 Micromax Model S strip chart, 2 prints, surface panel $300.00.
Made by Leeds and Northrup, Serial 276758.
Order B9975 on 5/19/36, Req. 69970.
In lean-to, Bldg. CR.

MSC-0668 L & N Temp. Recorder made by Leeds and Northrup.
Dwg. B-666, Serial 276807.
(1) #40354-291 Micromax model S strip chart, multiple temp. recorder, 4 point. (Multiple colored dots instead of numbers on print wheel). Surface panel mounting $465.00.
(2) Extra for high contact for operating alarm $20.00
Order B9976 on 5/19/36, Req. 69969, Chg. out $491.82.
South wall, west instrument, Bldg. CR.(See note on next pg.)
Replaced by I-0244.

MSC-0675 Temperature Recorder purchased from Leeds and Northrup. Serial 276784. #40353-291 Micromax, model S, strip chart multiple point temperature recorder for 3 prints. (Multi-colored dots instead of numbers on print whsel.) Surface panel mounting $400.00. Extra for high contact for operating alarm, $20.00. 3 thermocouples to fit wheels. Our Dwg. B-666.
Order B9977 on 5/19/36, Req. 69968, Chg. out $324.05.
South wall, east instrument, Bldg. CR.
(See note on top of next page.)

XII. EQUIPMENT LIST, contd.

Miscellaneous Equipment, contd.

MSC-0668
-0675

Note:

Record the following temperatures:

Batch temperatures in chlorinators: CT-0744
0745
0833
01283
0808

Goods temperature in diphenyl store tank CT-0748

Coil inlet temperature on the stills S-043 and 062.

" outlet " " " " " " "

Vapour temperature entering the condenser.

Goods temperature in receiver.

Goods temperature in blending tank, CT-0779.

Goods temperature in finished product receiver, CT-0780.

Drawing B-666 shows thermo-couple wells.
Drawing B-6868 shows monel thermo-couple wells as used in distilled Aroclors.

MCS-0676

Gas Electric Water Cooler for drinking water. Made by G. E. Compressor 6541356, cabinet 6600807. Model RM-51, pressure type, 110 v., single phase, 60 cy.
Order B14045 on 7/14/36, Req. 71432.
Chg. out 7/31/31, $176.00. On 12' level, Bldg. CR.

XII. EQUIPMENT LIST, contd.

Miscellaneous Equipment, contd.

MSC-0681 Rail truck purchased from Nutting Truck Company. Dwg. C-827. Welded construction, wheels equipped with Hyatt Roller Bearings. Used as weigh truck for still pots on SC-095. Includes track.
Order B11134 on 6/3/36, Req. B70395.
In lean-to Bldg. CR.

MSC-0682 Brown mech. flowmeter. Serial 115969. Recording and integrating steam at 65# ga. press. Dry sat. orifice monel in 4" std. horiz. line. (used on 80 lb. main). Max. flow 4000#/hr. Model 2221-x40. 2 lgs. 25' of ½" copper tubing and connectors - Monometer valves. Elec. clock for 110 v - 60 cy.
Order B14604 on 7/22/36, Req. 71548 on 7/20, chg. out $280.00

MSC-0705 Palmer Industrial Thermometer CABI #89. #249 - 45° reclining industrial therm. 12" scale, steel separable socket - 30" long with 1" std. pipe thread. Range 0-200°C. No nickel plating, case painted reg. lacquer.
Order B18261, chg. out $28.05 on 10/31/36.
On Buffer tank CT-0750.

MSC-0718 Lab and Office of Dept. CABI #89 - 1936.
20' x 10' x 10' high includes partition and elec. lights on NW corner of CR.

MSC-0725 Laboratory testing equipment. See reports on G. E. testing.

MSC-0734 Electric drying oven. CABI 89-1936. Cat. 7764, 3 heat oven with one shelf and 9525 thermometer for 110 v., AC current. Belongs to the Pyranol Dept.
Order B23080 on 11/16/36, Req. 74628. Chg. out $26.60.

XII. EQUIPMENT LIST, contd.

Miscellaneous Equipment, contd.

MSC-0760 Vacuum jet made by Worthington.
#2½ JFD-S std. 3 stage steam jet ejector with 2nd stage condensing and fitted with jet type intercooler. Jet to be CI bronze nozzles, heads, monel nozzles.
Order B7404 on 4/14/36, Req. 69211 on 4/14.
Chg. out $796.00 on 6/30/36.
N of MSC-01392 Bldg. CR.

Ejectors MSC-0760, 01392, and 01686 are in a row running north and south in the building, and they are interconnected so that the centre one can serve as a spare for either of the other two, to serve the vacuum stills S-043 and 062. Cast iron bodies with monel jets. Karbate ejectors were to be tried. (Anniston were using ejectors with two stages of Karbate jets, then a wet condenser, and then two more Karbate stages.)

Faulty behaviour, due to erosion or corrosion, or to wet or dirty steam, is very objectionable as allowing water to get back into the goods. Karbate units have to be very carefully supported to prevent strains from the pipe lines.

More recently procelain units have been installed at the Krummrich plant.

MSC-0770 Super Tank Gauge made by Varec.
Fig. 68, Gastight. Use on tank according to B/P E-4366, Monsanto Drawing.

All material to be standard, aluminum float, stainless steel tape and float guide wires.
Order B3569 on 2/16/37, Req. 77035. Chg. out $174.80. On CT-0848. TCB store tank.

MSC-0903 Steel cabinet made by Medart. CABI #139. 1 unit 48" wide and 24" deep x 6'3" high 9" ledge,bin fronts, dividers and 2 doors on lower part with lock.
Order B3013 on 2/18/38, Req. 86579 on 2/17/38.
Chg. out $50.51.

XII. EQUIPMENT LIST, contd.

Miscellaneous Equipment, contd.

MSC-0971 10 steel box lockers made by Medart. CABI 139. Olive green steel arranged 2 wide x 5 high. Complete with locks, 2 keys for each lock x 3 master keys common to all. Lockers 12 x 12 x 15" deep.
Order B15781 on 9/23/38, Req. 91603 on 9/21/38. Cost $25.75.

MSC-01061 Two loading ramps made by Monsanto. For one ton chlorine cylinders, using I-beam supports and concrete footings with I-beam rails. East side, outside Bldg. CR.

MSC-01094 Drying Oven purchased from G. E.
Used with F-0159 in the Pyranol plant.
4 compartment electric drying oven with 115-230 volt heating elements.
3 HP motor.
Order B18935 on 9/21/39, Req. 1991 on 9/20/39.

MSC-01191 Metal Desk and Stool. Desk 36-5/32" wide, 42" high front, 54" high back, 31" deep. Stool 30" high.
Order B5169 on 3/4/40, Req. 7148 on 2/29/40.

MSC-01205 Speed reducer made by Falk. Serial M0127-928.
Size 28DZX, RPM 1750 to 180, ratio 9.7.
Order B8337 on 5/20/40, Req. 8941 on 4/9/40. Chg. out $169.94. Driven by M-01629 on CT-0808.

MSC-01288 Falk Motor Reducer 7½ HP. Serial M0131-842.
#44DZX, output speed 1750 to 41.9 RPM, service factor 1¼, ratio 41.9.
Order B21071 on 9/30/40, Req. 13674 on 9/30/40.
Chg. out $341.94 on 10/31/40. On CT-01321, Pyranol blender.

MSC-01392 #2½ Worthington vacuum jet 3 stage. Type 9-10-11.
North of MSC-0760.
Same as MSC-01686.

XII. EQUIPMENT LIST, contd.

Miscellaneous Equipment, contd.

MSC-01393 Control Room. Drawing C-9646.
Structural steel, floor plate, Bldg. tile 12 x 12 x 3.
Pyrobar roof, door and instrument panel for I-0441.

MSC-01495 ½ ton Peerless hoist and trolley. Model C, 11' lift.
Model J trolley.
Order B17508 on 8/6/41, Req. 23095 on 8/6/41.
Chg. out $72.74 on 11/27/41.
Used to remove plates of filter F-0172 for cleaning.

MSC-01686 #2½ Worthington 3 stage vacuum jet. Type 9-10-11.
North of MSC-01392. Same as MSC-01392.

MSC-01699 Lockers (4) made by Medart. CABI 152,1939.
2 only 15 x 18 x 60 dark green. #14 and #15.
2 only 15 x 15 x 36 double tier. #12 and #13.
Order B3439 on 3/8/39.

MSC-01877 Canvas tent for tank car. Approx. 7' dia. x 7' high with swing boom, to raise and lower tent into a wooden box along west wall of CS. To be located at load dock at track #11, SW corner CS. Also elec. light in tent. order B20696 on 9/7/43, Req. 48441 on 9/4/43.

MSC-01879 South dock, wood, muriatic loading. Also swing plank and wood decking.
On track 11 west of CS. 5' x 5'6". 1943.

MSC-01916 North load dock made of steel with swing plank. 5' x 7' long, 9'6" high. 5 x 5 angle columns with 6" channels to support ¼" steel decking. 1½" pipe railings.
North of MSC-01879. SW of CS on track 11. 1936.

MSC-02071 Stoneware sink.

MSC-02108 Steel Hood and Duct. 2' x 4' counter balanced with 6" dia. steel duct to outside of Bldg. Hood located over F-0159 G. E. Filter Press.

XII. EQUIPMENT LIST, contd.

Miscellaneous Equipment, contd.

MSC-02426 Ducts, hoods, dampers.

MSC-03417 Air cond. unit.

MSC-03523 Vac. ejector. Worthington, Porcelain, Karbate. 10" Porcelain condenser. 10 lb. hr. air at 2 mm cbs. 150 psig steam. 65°F. well water.

MSC-03611 Duct and hood.

MSC-03689 Haveg sink. 4' 8" x 19" x 18".

MSC-03706 Water cooler. Drinking water.

MSC-03741 1/2 T chain hoist above F-0172. } Pyranol plant

MSC-03835 1/2 T chain hoist - #2 mix tank. }

MSC-04018 Work bench

MSC-04109 Lightnin mixer (only - not motor) 7½ HP side entering metallized with 0.015" Al. See CT-02621.

MSC-04132 Water cooler.

MSC-04442 Hand hoist.

Heaters for plant office and for instrument and control room.
Gas supply and metering.
Water hose with steam mixing jet for cleaning the floor.
Desks.
Clothes lockers.
Test bench.
Phone.
Clock.
Framed instructions for special pieces of equipment.
Drinking water supply.
First aid cabinet.
Fire alarm & extinguishers. Sprinklers installed in all rooms.
Hose connections for casual gas heating.
Waste cans.
Used water collection.
Lighting.

XII. EQUIPMENT LIST, contd.

Open Tanks

OT-0640 Drum, 110 gal. ICC5, CABI #217-1941. With float valve on city water line. Connect 3 GPM pump P-0378. To pump from drum through rotometer to acid absorption system.
Black Iron
$18.30 as trans from Dept. 360.

Seal cans for barometric legs of the ejectors.
MSC-0760, -01292, -01685. On the ground floor.

OT-0751 Steel vessel 4'0" I.D. x 6'0" high, for city water, to conform to state law.

XII. EQUIPMENT LIST, contd.

Pumps

Note: The pumps are changed around from one duty to another, in the course of ordinary maintenance work, but the details given below will serve to show what type of pump is used for any given duty. Oversized pumps may be used in some cases to diminish the number of types required in the department.

P-0346 Swaby Submerged Pump made up 11/18/29. Size #1½, Cast Iron. On CT-0848. In TCB store tank, CT-0848.

P-0378 Chas. Lewis pump Serial 4597, Type BA-481, cast iron vertically split shell cent. pump. CI bed plate connected with flex. coupling, direct to 1/4 HP 3600 RPM motor. GE M-0466, 3 gal. PM - 35' head, 3450 RPM. Order B693 on 1/15/31. Price $68.00 incl.motor, M-01772. Boosts city water to HCl absorber.

P-0427 Horizontal cast iron pump 1½ HP Monsanto Design. Direct connected, cold rolled steel shaft, includes base, coupling and c guard.

P-0565 Blackmer pump, Serial 197033, type PA-50L bronze rotary pump, bronze lining, bronze steam jacketed heads, monel shaft, CI base, 1.65 sp. gr., temp. 350 to 500°F. - 50 gal. Order B-8170 on 4/23/36. Chg. out $155.45 on 6/30/36. Distillate transfer pump.

P-0571 Oberdorfer gear pump for filter press. Motor M-01650.

P-0573 Vertical submerged centrifugal pump purchased from The Engineering Equipment Co. Serial 33007, Size L-0B with 1½" inlet and 1" outlet. Compare P-0604, which is a Taber pump on #4 scrubber CT-0759.

P-0574 Ditto.

P-0575 Taber submerged pump. Serial 33009. Order B7517 on 4/16/36. Req. 9161. Chg. out $177.80 on 6/30. Direct to vertical M-0848 in CT-0756 on #1 scrubber. Same as P-0573 and 0574.

XII. EQUIPMENT LIST, contd.

Pumps, contd.

P-0578 Taber horizontal centrifugal pump. Serial 33040. Type L-2, 2½" inlet 2" outlet connections. Semi-steel with monel shaft and water jacketed stuffing boxes. Bed plate ready to receive motor. 20' static head, rate 80 g.p.m. 1.5 sp. gr. at 500°F. Uses M-0849. On #2 chlorinator, CT-0744.
Order B8237 on 4/24/36, Req. 69401.
Chg. out $152.92 on 6/30.

There are four pumps P-0578, 0579, 0629 and 0907, one for each main chlorinator.

Liquor in from chlorinator bottom outlet and out back to the chlorinator. The charge can be pumped, if necessary, from one chlorinator to another, or to the air-blowing vessels CT-0750 and CT-2086.

No. 1 chlorinator can be emptied by gravity to the gas-fired chlorinator CT-0808, and there is also a pump line for the same service (disused). The charge from No. 1 chlorinator can be pumped directly to No. 1 vacuum still S-043.

There is a line by which partly chlorinated diphenyl can be pumped from a chlorinator to any scrubbing tank, CT-0759, -0760, -0761 or 01014.

There is a sampling cock on each pump delivery, with a small box to hold the used sample material, and to deliver it back to the pump intake.

P-0579 Taber horizontal centrifugal pump. Serial 33041. Same as 0578-80. For middle chlorinator CT-0744, uses M-0850. On #3 chlorinator CT-0833.

P-0580 Taber horizontal centrifugal pump. Serial 33042. Same as 0578-79. From blow tank CT-0750. Uses M-0851.

XII. EQUIPMENT LIST, contd.

Pumps, contd.

P-0582 Taber vert. submerged centrifugal pump. Serial 35048., Fig. 1940, size C-3, 2½" inlet, 2" outlet connections, bronze except shaft which is monel metal. 11'6" length of column pipe. Spare pump for underground tanks. Order B7514 on 4/16/36, Req. 69158. Chg. out $409.20. Direct to M-0852. Same as P-0583 and 0584. Used in Aroclor 1260 store tank.

Submerged pump in CT-0768 TCB store.

P-0583 Taber vert. subm. cent. pump. Serial 33049. In CT-0769, direct to M-0853. Same as P-0582-0584. Aroclor storage.

P-0584 Taber vert. sub. cent. pump. Serial 33050. In CT-0768, 1254 store tank, direct to M-0854. Same as P-0582 and 0583.

P-0585 Taber vert. sub. cent. pump. Serial 33069, Dwg. A387, Type C-3, fig. 1940, 2" inlet and outlet. CI body, renewable bronze bearings, bronze impellor mounted on nickel steel shaft. Length 11'6", sump cover plate, cap. 50 gal. p.m., 60' static head, sp. gr. 1.18 at temp. 165°-170°F. Driven by M-0856 in CT-0748, Diphenl store tank. Order B7518 on 4/16/36, Req. 69162. Chg. out $257.62.

P-0592 Taber vert. sub. cent. pump. Serial 33113, type C-3, Fig. 1940, 2" inlet and outlet. All iron construction on nickel steel shaft length 11'6". Cap. 50 g.p.m., 60' static head, sp. gr. 1.368 at 70° to 90°F. In CT-0749* driven by M-0873. Order B7516 on 4/16/36. Req. 69160, chg. out $255.41.

* general service and diphenyl tank.

P-0593 Taber vert. sub. cent. pump. Serial 33111. Type L-2, fig. 1940, 2½" inlet and 2" outlet, casing, yoke, impellor made of steel shaft nickel steel, column supporting intermediate bearing, wrought iron. Length 7'9".

XII. EQUIPMENT LIST, contd.

Pumps, contd.

P-0593 contd.
Cap. 75 GPM, static head 15', sp. gr. 1.8 at temp. 700°F.
Driven by M-0871. On Number 1 still.
Order B7515 on 4/16/36, Req. 69159. Chg. out $382.91.
In still S-043. Same as P-0574. Sump plate.
Drawing A-493.

P-0594 Spare motor same as P-0593. Serial 33112.
Driven by M-0872.

P-0595 Oberdorfer gear pump. Serial 2835. #7AXZ unloader pump, all bronze gear type. 10-15 GPM at 20# pressure. Used to circulate Aroclor on No. 1 still cooling system.
Order B13188 on 7/2/36, Req. 71152. Cost $78.80.

P-0604 Vert. sub. cent. pump. Taber. Serial 33207, Size L-0B, 1½" inlet and 1" outlet. All iron const., open impellor on nickel steel shaft. Same as P-0573-4 and 5. In CT-0754.
Order B13195 on 7/2/36, Req. 71148 on 7/1/36. On #3 scrubber, CT-0761.

P-0615 Blackmer Pump Serial #201747.
Size 502, 50 gallon per min. right hand rotary gear pump. Bronze with bronze liner and monel shaft. Steam jacketed heads.
Order B20818 on 10/16/36. Req. 73912 on 10/16/36
Chg. out $148.27 on 11/30
Direct to 3 HP 1800 M-01449.
On filter press F-0159.

P-0617 Blackmer pump. Serial 201902. Size 100L, all iron, jacketed right hand pump, base plate for motor, and flex. coupling, direct connected (M-0914). Order B21241 on 10/22/36, Req. 74034. Chg. out $145.20.
Spare pump.

P-0629 Taber horiz. cent. pump. Serial 33708. Type L-2, 2½" inlet and 2" outlet, Size 103. Order B2833 on 2/9/37. Req. 76766 on 2/5/37. Chg. out $152.52. Direct to M-0975. On No. 1 chlorinator.

XII. EQUIPMENT LIST, contd.

Pumps, contd.

P-0674 On #1A scrubber, CT-01013, driven by motor M-0848 on No. 1 chlorinator.

P-0731 Taber sub. cent. pump. Serial 84722, Size L-OB, 1½" inlet and 1" outlet. Order B3854 on 3/4/38, Req. 86891 on 3/3/38. Chg. out $174.25.
In CT-01013.

P-0907 Taber horiz. cent. pump. Serial 36622. Size 103, type L-2, 2½" inlet and 2" outlet connections, semi-steel through out with monel shaft and water jacketed stuffing boxes, with bed plate ready to receive motor, 20' static head, rate 80 GPM, 1.5 sp. gr. at 500°F.
Order B12801 on 6/5/40, Req. 10230 on 6/5/40.
Chg. out $155.28, driven by M-01656 to circulate liquor for chlorinator CT-01283, #4 chlorinator.

P-0909 Taber sub. pump. Serial 36676. Size L-OB with 1½" inlet and 1" outlet, all iron const., open impeller on nickel steel shaft. Order B13110 on 6/10/40. Req. 10366 on 6/10/40. Chg. out $193.75. On CT-0968 scrubber liq. tank. Also on CT-0760.

P-0929 Taber sub. pump. Serial 36853. Size C-3, fig. 1940, vertical 2½" inlet and 2" outlet. Bronze except shaft to be monel metal, lg. of col. pipe 11'6". Order B18442 on 8/23/40. Req. 12559 on 8/23/40. Chg. out $404.59. On stg. tank CT-01310.
Driven by M-01706.

P-0932 Roper cast iron pump. RIBI #169. Size 1" rotary, geared, complete with base, gears, pinion, big stand and adapter plate for motor mounting.

P-0984 Roper pump. All iron series "O" rotary pump for 15 GPM. 1.6 sp. gr. 50' head pump, spiral gears pkgd. stuffing box with non-graphitic white asbestos pkg. built in by-pass and complete with base, drive gears.
Order B4118 on 2/17/41, Req. 18322 on 2/17/41. For filter in CR. On Sparkler filter F-0172. Replaced by P-0571.

XII. EQUIPMENT LIST, contd.

Pumps, contd.

P-0997 Taber vert. sub. pump. Serial 37682. Type CL4, 2½" inlet and 2" discharge for 75 GPM against 25' head. Pump of dynamo steel with wrought iron pipe column and nickel steel shaft, water cooled, stuffing box.
Order B6379 on 3/13/41. Req. 19214 on 3/13/41.
Chg. out $446.53. In S-062.
Driven by M-01856. Shaft has two intermediate C.I. bearings.

P-01037 Blackmer pump. Serial 246058. 5WG3202 bronze, size 51L with base and relief valve. Still receiver to blender.
Order B16097 on 7/21/41. Req. 22429 on 7/21/41.
Chg. out $177.56. On gr. fl. NE of F-0172.

P-01054 Pump made by Weil. Serial 13132. Type DSI, size 1". Uses city water. Order B4625 on 2/21/41. Req. 18520 on 2/21/41. Cost $65.00. On TW-0221. Driven by M-01995.

P-01056 Taber horiz. cent. pump. Serial 38318. Type L-2, size 103, 2½" inlet 2" outlet complete with base. Order B19730 on 9/3/41, req. 24125 on 9/2/41.
Chg. out $157.44 on 4/31/44. On #4 chlorinator CT-01283.

P-01111 Blackmer Pump Serial 249113. #5 OL all iron steam jacketed pump with CI base plate and gear drive ready to receive our 3 HP 1800 RPM motor size 511. Order B16349 on 7/23/41, Req. 22597. Chg.out $105.05. Driven by M-02877. For discharging rail tanks.

P-01258 Blackmer pump, Serial 269500, Size 1202-50. Fig. 3203 complete with relief valve. No base. #2 still receiver to blending tanks.
Order B29303 on 1/28/43. Req. 40312 on 12/30/42.
Chg. out $134.63.
Bronze.

XII. EQUIPMENT LIST, contd.

Pumps, contd.

P-01277 Taber vert. sub. cent. pump. Serial 40890. Type CL-4, 2½" inlet, 2" outlet, size 384. Pump to be dynamo steel with wrought iron pipe, column cap. of pump 75 GPM at 25' head for liquid 1.8 sp. gr.
Order B8875 on 7/12/43, Req. 43713 on 4/16/43.
Chg. out $434.00 on 12/27/43.
At S-062 Spare. Same as P-0997.
#2 still pump (spare).

P-01509 2" Taber pump. 80 GPM. 130 ft. TDH. Steel with monel shaft. Flexible coupling. M-03014.
S. G. 1.5 500°F.

P-01540 G. E. Filter pump - N. W. corner of lean-to. Taber pump 304 stainless steel, 2" suction, 1½" discharge. Located indoors. Pumps from old Pyranol blender CT-01321 to filter presses.

P-01757 On Sweetland filter F-0239. Now used on F-0238. 304 stainless steel 2", 100 gpm. Motor M-03718.

P-02052 2" Taber. Semi-steel, monel shaft, water jacketed stuffing box. 1.5 S. G.,500°F.

P-02077 Blackmer pump, from TTCB store to blenders. Bronze lined, with monel shaft. N.W. of Bldg. CR.

P-02078 Taber 3" x 2½" 150 gpm. 80 ft. head. Horizontal impeller.

P-02179 Taber Pump from new blender CT-02621 to filters. Motor M-04025.

P-02251 Viking pump on Aroclor mixers. M-0514. Pyranol plant.

XII. EQIPMENT LIST, contd.

Pumps, contd.

Note:

London Engineering Dept. Final Report on the start-up of the Newport Aroclor plant states that Hamworthy pumps were not found satisfactory in use. Good jacketing is needed.

There was trouble from corrosion of core plugs in some pumps, leading to leakage of steam into the goods.

Steel end covers were used later.

The same reports discuss the difficulties encountered with the still pump, which was located in a small secondary vessel, below the vacuum still.

XII. EQUIPMENT LIST, contd.

Retorts

R-026 Aroclor 1269 Retort assembled and erected by Monsanto.
Pots from Nooter. $516.00 for 4.
Jacket from Graver. Req. B69906.
Burners from Surface Combustion. Req. 69374.
Burners - gas fired.
Jacket, steel, firebrick lined, holding 3'0" OD x 3'8" lg. over dished bottom still pot with cover. Equipped with 2 gas burners and stack. Still pot 1/2" fire box steel, removable so residue can be dumped.

R-027 Aroclor 1269 Retort - Same as R-026.

Drawings: B-646 Retort.
E-865 Setting.
B-374 General arrangement of solid Aroclor plant.
C-830 Gas heater for discharge line.
C-821 Platform for retorts.

Mr. Soffranko in 1947 commented that the retorts would be better with thicker bottoms than shown in drawing B-646, and that spare retorts should be held.

XII. EQUIPMENT LIST, contd.

Stills

S-043 No. 1 Vacuum still purchased from Graver. Plant A Drawing C-809. Size 5' dia. x 5'6" long on straight side, 1/2" shell, dished head 5/8" supporting brackets 60# Hydro press test.
Order B-7705 on 4/17/36, Req. 69225, Chg. out $336.00 on 2nd floor, 12' level.

S-043, No. 1 still, at the Krummrich plant, is of the shallower type, like the Anniston stills, and like them it has a vertical vapour pipe (with Hagan separator inside the still), leading to the top of a vertical condenser, CT-0770, whereas No. 2 still is of the deeper type, and has a cyclone, CT-01413 in place of the vertical vapour pipe and the separator. The cyclone returns a considerable amount of darkish liquid, through a sight glass, to a return line going nearly to the bottom of the still body. Mr. Furzey, December 11, 1951 reported that the cyclone gave better coloured distillate than did the Hagan separator. Anniston, January 1950, reported satisfactory performance of their Hagan separators. Newport have the internal Hagan separator.

In a report dated March 31, 1952, Mr. Dalton of the Krummrich plant mentions a modification to the Hagan separator at the Krummrich plant. The Aroclor colour was better after the change, but it was not certain that the alteration really caused the improvement.

There is no attempt at fractionation, and the condensing systems were planned to give the lowest pressure drop attainable, consistent with removal of coloured entrainment particles. The deeper pattern of still S-062 was chosen to give more disengagement space for vapour, but it does not appear to have given any better coloured distillate, and it has the disadvantage of requiring a longer shaft etc. for the submerged pump. The condenser on No. 1 still delivers to receiver CT-0771

XII. EQUIPMENT LIST, contd.

Stills, contd.

S-043 contd.

No. 2 still has heat exchanger CT-01491, head tank CT-01398 with gravity flow (of cooling water only) to condenser CT-01378. CT-01398 has an internal coil (not used) and there is no temperature control instrument .

The distillate goes to receiver CT-01377.

The temperature of the distillate is roughly controlled by hand adjustment of the flow of water from CT-01398 to the condenser CT-01378.

The Newport vacuum still was provided with a subsidiary vessel, located at a lower level than the main vessel, and containing the submerged pump. With this arrangement, it was hoped to diminish the liability of cavitation of the pump, and to permit the use of a much shorter shaft for the pump impellor.

The arrangement, however, was unsatisfactory, largely because the pump gland was exposed to hot liquid Aroclor (See the London Engineering Dept. Final Report on the start-up of the Newport plant).

Numerous different gland packings were tried, the gland was given a lantern ring, fed with liquid Aroclor etc. etc., but finally a long vertical steam jacketed pipe was fitted on top of the secondary vessel, to carry the stuffing box above the level of the charge in the main still. This location also put the driving motor into cooler surroundings, and the new arrangement has been made to work satisfactorily, though it brought back the troubles of the long shaft.

In the pump assembly, the pump is hung from a manlid on the still cover, on a wide pipe down the centre of which the pump shaft runs. The delivery pipe from the pump runs independently up through the same manlid on the cover of the still (welded through). Differential expansion may distort the alignment of the parts, and may

XII. EQUIPMENT LIST, contd.

Stills, contd.

S-043 contd.

displace the impeller axially within its casing. Another trouble which appeared was that air leaking in through the gland could travel down the central pipe around the shaft and cause cavitation in the pump. The remedy for this is to have holes in the pipe, communicating with the main vapour space in the vessel.

The stuffing box is water cooled; the bearings inside the still may give trouble, this clearance must be just right. Carbon bushings have shown some promise. It has to be remembered that the charge contains a fair amount of solid (lime) etc. which might lodge to some extent in the still instead of flowing to the secondary vessel.

The internal bearings are lubricated by Aroclor bled off the pump delivery line, but they may become choked by the solid matter in the charge.

See also the description of the Anniston still and pump on pages 20-, Section VI, above, and comments on the distillation process, pages 4-, Section VII.

From the bottom of the condenser at the Krummrich plant, the distillate passes through a jacketed line to a jacketed sight glass with sampling fitting, and so to one of the receivers CT-0771 or CT-01377. There is a ring of gas burners around each condenser exit, to deal with the heavier Aroclors, but the burners are not much used.

Condenser CT-0770 can be cooled by Aroclor circulated by pump P-0595 through the condenser shell space then through heat exchanger, CT-0831 (cooled with well water) and through a sight glass to head tank CT-0766, and so back to the pump. This system is alternative to the cooling by distilled water used on this condenser, but it has gone out of use since the Krummrich plant gave up the production of the higher Aroclors.

XII. EQUIPMENT LIST, contd.

Stills, contd.

S-043 contd.

There is a steam and water coil in the head tank to keep the Aroclor at around 110-120°C. There is a Taylor Fullscope temperature controller I-0510 controlling a valve on the steam inlet to the coil. This Aroclor cooling is more convenient for higher Aroclors, but the water system is preferred for the lower Aroclors.

The system normally works at about atmospheric pressure though the vent (on the top of heat exchanger CT-0831 is usually kept closed, and there is a pop valve at the same point).

The sketch on page XII - 82 will serve to show the arrangement and to indicate certain minor features not described above.

Each still has a bottom outlet which will completely empty the still, also an outlet at about the same level of the top of the bottom dish, which will leave about 80 U.S. gallons of bottoms in the still. Both deliver to open topped cans on small wheeled "dollies" under a hood ventilated by suction fan B-0148. Rising stem gate valves are used on these outlets and gas flames are freely used to heat them for some time before the still is to be tapped.

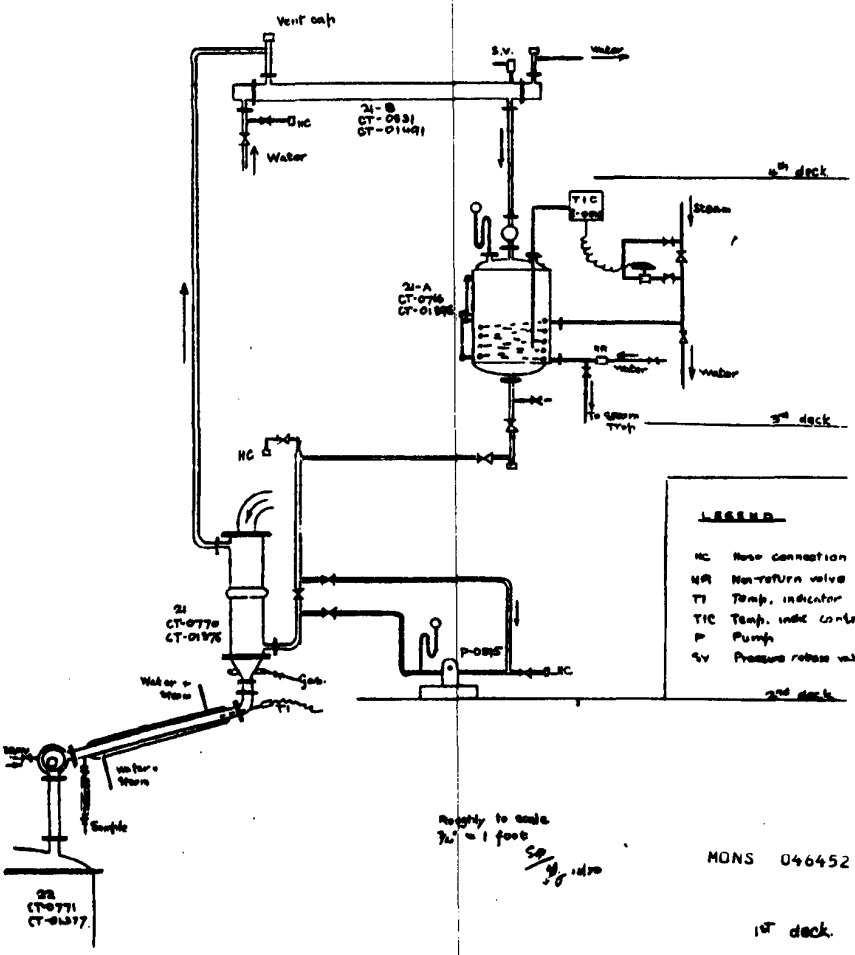
Bottom outlet also to emergency tank CT-0749, but this vessel is now used as extra storage for diphenyl.

The deeper still has a thermo well obliquely through the side wall, about 7'6" below the cover, but this well was not in use May 1955. Each still body has a BS & B relief disc of AlPb, 8" diameter, vacuum supported, calibrated to rupture at 25 psig. The receivers do not have relief discs.

XII - 82
Equipment
List

COOLING SYSTEM, AROCLOR CONDENSERS.

Plant 'D', July, 1930.



Roughly to scale.
¾" = 1 foot

MONS 046452

1st deck.

XII. EQUIPMENT LIST, contd.

S-043, contd.

Drawings.

B 483	Water-cooled condenser on No. 1 still	CT-0831, -01397, -01491
A 493	Sump plate for submerged pump	S-043
B 644	Head tank for cooling medium	CT-01398
B 651	Tubular condenser	CT-0770, -01378
B 662	Vapour exit pipe	S-042
C 795	Receiver	CT-0771
C-807	Coke tower	CT-0753
C 809	No. 1 Still	S-042
C 815	Piping of vacuum still - FC-045,CT-0770,-01377	
C 864	like B 483, on No. 2 still	S-062
C 6942	Piping diagram, No. 2 still	S-062
C 6943	Piping diagram, No. 2 still	S-062
D 6944	No. 2 still	S-062
E 6952	General layout No. 2 still	S-062
D 6956	General layout, No. 2 still with cyclone, receiver, and coke scrubbers.	
D 6957	Cyclone on No. 2 still	S-062
B 7082	Monel condenser on No. 2 still	CT-01378

XII. EQUIPMENT LIST, contd.

Stills, contd.

S-062 #2 Vacuum still purchased from Nooter. Serial #52. Dwg. D-6944, 6' ID x 11'10" str. shell, internal pipes, etc. Std. dished heads. Glass wool insulation 3" with 1/2" finish coat.
Order B10730 on 5/12/41, Req. 20421 on 4/28/41.
Chg. out $950.00. Suspended between 20'6" and 12' platform in CR.

S-0459 is another number for C-0808, gas fired chlorinator.

XII. EQUIPMENT LIST, contd.

Scales

SC-094 Howe Portable Scale #822, Serial 1363205, all iron platform 30½" x 30½". Cap. 1500 lb.
Order B9986 on 5/20/36, Req. 69955, Pyranol Plant.

SC-098 Howe scale. Serial 1364566. #1420 Skeleton type, ball bearing, dormant scale, cap. 2500# with double beams. Platform 46 x 38. All metal incl. steel shaft. Order B14351 on 7/20/36. Req. 71536 on 7/17/36.
Price $213.85.

SC-0100 Scale purchased from The Exact Weight Scale Company. Serial 39992. Style 1226, cap. 200# on ratio of 10 to 1. Dial with indicator travel 12½" under wgt, and 2½" over wgt. Fitted with bag holder for 100 to 200# bags. All on swinging rack.

Order B17906 on 9/9/36. Req. 72843. Chg. out $289.41.

In lean-to near V-044 and V-045.

SC-0246 1000 lbs. capacity, Pyranol Plant.

XII. EQUIPMENT LIST, contd.

Transformer

T-014 Transformer made by Westinghouse. Serial 530335. 200 KVA, type SK, now used 440 volt primary 110/220 volt secondary. On the heater of the Lectrodryer FC-046. Cost $10.00. Outside SW corner Bldg. CR.

Also used formerly for strip heaters in the still receivers, etc.

XII. EQUIPMENT LIST, contd.

Towers

TW-0166 Fused Silica Tower purchased from Amercel Co. Inc. 1 "U" bend trap. 12 absorbers sections B/P G111 8" ID x 6'6" C to center. Order B8161, Req. 69399. Same as TW-0165. Regarded as obsolete design.

TW-0182 Scrubbing HCl tower made by Haveg. Drawing C-4705. "41" std. 2'6" dia. 10'2" deep, 5/8" thick walls x 3/4" thick bottom, machined dished head, and connecting material, 2" thick Haveg drilled plate with necessary supports 4-3", 1-8" and 1-10" std. Haveg nipple type outlets with flgs. Order B20284 on 10/5/37, Req. 83234 on 10/5/37.
Chg. out $668.56 on 12/31/37.
On 38' level steel struct. south side of CR.

TW-0206 Scrubbing Tower purchased from Nooter. Drawing B-5815 support, drawing C-5814 tower. 17-18" ID x 8' welded steel const. tower, welded steel scrubber support for tower.
Order B7346 on 3/27/40. Req. 8063 on 3/27/40.
Chg. out $277.00 on 4/30/40.
Above CT-0756, scrubber by pump tank.

TW-0221 HCl absorption tower purchased from Fan Steel Metallurgical Corp. Serial 955-1941. Drawing D-4300 foreign. PA #147. Type AL-400. The tower, fittings and piping made of Karbate. Gas line of Haveg. Cap. to produce 205° Baume HCl at 4620# per hour.
Order B4625 on 2/21/41. Req. 18520 on 2/21/41.
Chg. out $5321.13 on 8/21/41.
On steel struct. S of CR.

Schwarting and others "Process Description --- For Aroclors", November 12, 1953, state that the Fansteel off-gas absorbers are constructed of Haveg 60, and have Tantalum heat exchange surfaces.

TW-0312 Absorption tower. Fansteel AL-300. Haveg 60 parts. Tantalum tubes, (some tantalum parts salvaged from an older unit.)

XII. DRAWING LIST

Note:

Some of the earlier drawings were made at the JFQ plant, and the later ones at the Krummrich plant. As the two plants have independent numbering systems it is necessary to specify which plant is concerned. The drawings marked "A" in column 2 on the following pages are from the JFQ plant, whereas those marked "B" are from the Krummrich plant.

In column 3, on the following pages, the letter

"L" indicates that prints of drawings were sent to Mr. Cooper, London, by air mail, April 24, 1947.

"S" indicates that prints of drawings were sent to Mr. Cooper, London, by surface mail, April 24, 1947.

"N" indicates that the drawings in question do not appear to be of interest to MCL.

"M" indicates that the drawings were not available April 23, 1947.

"F" indicates that the drawings were found July 28, 1950, and that prints were sent to Mr. Buis, London.

XII. EQUIPMENT LIST, contd.

Conveyors

V-044 Screw conveyor made by Essmueller MF Co. Drawing C-816. Size 6", 6'8½" trough length with outlet at each end. Cover of galvanized iron. CI trough ends with zinc and tin sprayed. Req. 69909. On bottom CT-0764 - North Conveyor. From stg. bin to bagging scale and back to boot of elevator, driven by M-0835.

Layout - E-877.
Flow Sheet - B-640

V-045 Screw conveyor made by Essmueller MF Co. Drwg. C-816. Size 6", 6'8-½" trough length with outlet at each end - cover of galv. iron. CI trough ends with zinc and tin sprayed.

Req. 69909. On bottom of CT-0765 south conveyor. From storage bin to bagging scale and back to boot of elevator, driven by M-0836.

V-046 Screw conveyor made by Essmueller MF Co. Drawing C-816. Same as V-044 and 045. From flaker discharge to elevator boot. Driven by chain on M-0827.

All these were part of the equipment for Solid Aroclors: dismantled in 1952

XII. DRAWING LIST

Drawing Number	*	*	Title	Equipment Number
A-324	A	L S	Gas-heated chlorination pot(see E-888)	CT-0808
E-346		M	Muriatic treatment vessel	CT-0861
F-352		N	Fractionating column(not Aroclor plant)	
F-370	A	L S	General Layout	Layout
F-373	A	L S	Ducts for process fumes,solid Aroclors	D-012 etc.
F-374	A	L S	Layout, solid Aroclors	Layout
F-375		N	Structural steel	
F-376		N	Steel and foundation	
F-380		N	Electrical layout	
F-381		N	Electrical conduits	
F-382		N	Lighting	
F-383		N	Service lines	
F-384		N	Foundation details	
F-385		N	Structural steel, Muriatic tower	
F-386	A	L S	Piping layout	Piping
F-389		N	Electrical details	
F-394		N	Gen. arrt. HCl absorption	
A-459	A	L S	Bearing for blender shaft	CT-0779

* * See note on page 86 in this section.

XII. DRAWING LIST, contd.

Drawing Number	*	*	Title	Equipment Number
B-483	A	L S	Water-cooled condenser for vac.still	CT-0831 CT-01307
A-493	A	L S	Sump plate for submerged pump in vac. still	CT-01491 S-043
A-501		N	Lead-lined tray for HCl absorber	
C-502B		M	Sand filter for muriatic acid	
D-512	A	L S	Storage hopper for solid Aroclor	CT-0764 CT-0765
D-518	A	L S	Chute for Aroclor flake	D-012
C-605	A	L S	Shear pin on drive of gas-heated chlorinator	CT-0808
D-609	B	M	Store tanks	CT-0748 CT-0749 CT-0768 CT-0769 CT-01310
B-613	A	L S	Relief door gas-fired chlorinator furnace	CT-0808
B-640	A	L S	Flow sheet	Flow sheet
B-643		N	Separator, not installed	
B-644	A	L S	Cooler on vac. still system	CT-0766 CT-01398
B-645	A	L S	Cyclone on solid Aroclor plant	CT-0751
B-646	A	L S	Still pots for solid Aroclor	R-026 R-027
B-651	A	L S	Heat exchanger on vacuum still	CT-0770 CT-01378

* * See note on page 86 in this section.

XII. DRAWING LIST, contd.

Drawing Number	*	*	Title	Equipment Number
B-653	A	L S	Stack for solid Aroclor plant	CT-0751
B-654	A	L S	Support for stack	CT-0751
B-658		N	Floor gratings	
B-659	A	L S	Chlorine inlet	CT-0745 CT-0833
B-660	A	L S	Cyclone - not identified	
B-661	A	L S	Elevator drive, solid Aroclors	L-031
B-662	A	L S	Vacuum still vapour pipe	S-042
B-663	A	N	Supports for vent line scrubber	CT-0762
B-664	A	N	Vac. still furnace vent pipe	S-042
B-666	A	L S	Thermocouple wells	
B-675	A	L S	Hood, etc. of flaker	D-102
B-684	A	obsol-ete	Strip heater clamp for chlorinator	CT-0808
B-685	A	obsol-ete	Heater for Sperry press	F-0172
B-686	A	N	Vent stack	FC-045
B-689		N	Vent stack	FC-045
C-795	A	L S	Vacuum still receiver	CT-0771 CT-01377
C-798	A	L S	$CaCl_2$ breather	CT-0762
C-799	A	L S	Diphenyl head tank	CT-0767
C-800	A	L S	Scrubber liquor tank	CT-0754 CT-0755 CT-0756

* * See note on page/in this section.

XII. DRAWING LIST, contd.

Drawing Number	*	*	Title	Equipment Number
C-804	A	N	Hot water collection tank	CT-0752
C-807	A	L S	Coke scrubber	CT-0753 CT-01396
C-809	A	L S	#1 vacuum still	S-043
C-810	A	L S	Melt tank for high boilers etc.	CT-0763
C-815	A	L S	Piping of vacuum still	PC-045 CT-0770 CT-01378
C-816	A	L S	Aroclor conveyor	V-044
C-818	A	L S	Chlorine vapourizer	CT-0743
C-821		N	Platform for still pots	R-026, R-027
C-822	A	L S	Chlorinator relief connection	CT-0744
C-823	A	L S	Muriatic acid store tanks	CT-0784 CT-0785 CT-0786 CT-0795
C-827	A	L S	Weighing truck, solid Aroclor plant	MSC-0681
C-829		N	List of motors	
C-830	A	L S	Gas-heater for discharge of gas-heated chlorinator	CT-0808
F-838		N	18" fractionating column, not in Aroclor plant	
C-847		N	Holder for strip heater	CT-0771

* * See note on page 86 in this section.

XII. DRAWING LIST, contd.

Drawing Number	*	*	Title	Equipment Number
C-848		N	Holder for strip heater	CT-0771
E-876		N	Floor grating	
E-862		L S	#1 Blending tank and #1 Filtrate receiver	CT-0779 CT-0780
E-863		L S	Buffer tank (air blowing tank)	CT-0750
C-864	A	M	Heat exchanger for #2 vacuum still (see drawing B-483)	S-062
E-865	A	L S	Setting for stills for solid Aroclor	R-026, R-027
E-867	A	L S	Setting for fireheated chlorinator	CT-0808
E-870		N	Conveyor for Aroclor flake	L-031
E-877		L S	Conveyor for Aroclor flake (layout)	V-044
E-880	A	L S	Chlorinator	CT-0744 CT-0745 CT-0833
E-881	A	L S	Shell for gas-fired chlorinator furnace	CT-0808
E-886		L S	Shell for above and brick setting	S-043 F-045
E-888		L S	Drive for gas-fired chlorinator	CT-0808
E-889		N	HCl absorption tower	
E-896		N	Floor grating, HCl tower	
D-978	B	L S	Diphenyl store tank	CT-0347

* See the note on page 86 in this section.

XII. DRAWING LIST, contd.

Drawing Number	*	*	Title	Equipment Number
C-1117	B	N	Foundation of D-978	
D-2076	B	L S	Turbo agitator for blending tank	CT-0779
D-2077	B	L S	Stator for above	CT-0779
C-3479	B	M	Heating furnace for coil of #2 vacuum still	FC-066
C-3775	B	L S	Filter for HCl gas	F-0106
D-4252	B	L S	Muriatic tank	CT-01260
D-4300		M	Fansteel drawing HCl absorber	
E-4366	B	F M	Varec. Gauge on Pyranol mixer	CT-01321
E-4413	B	L S	T.C.B. Store tanks	
D-4459	B	L S	Rubber-lined treatment tank with agitator	CT-0861
C-4616	B	L S	Scrubber liquor pump tank	CT-0968 CT-01013
E-4679		N	Obsolete, Layout for HCl absorber	
E-4689		N	Obsolete, Piping of HCl absorber	
C-4705	B	L S	Haveg tower (obsolete) for HCl abs.	TW-0182
C-4845	B	L S	Scrubber liquor tank	CT-0104
E-C5711		M	Goodrich rubber-lined tanks	CT-01575
C-5743	B	L S	Scrubber in Pyranol plant	CT-01162
C-5814	B	L S	Obsolete, HCl tower	TW-0206

* * See the note on page 86 in this section.

XII. DRAWING LIST, contd.

Drawing Number	*	*	Title	Equipment Number
B-5815	B	N	Obsolete, HCl tower support	
B-5821		F	Cyclone	CT-01259 CT-01279 CT-01280 CT-01282
C-5825	B	L S	Monel gas chamber on fireheated chlorination system	CT-01258
C-6144	B	M F	Monel condenser	CT-01263
E-6209	B	L S	Chlorinator	CT-0744 CT-01283
D-6455	B	L S	Pyranol mixer	CT-01321
E-6838	B	L S	Assembly of blending tank	CT-0779 CT-01399
A-6853		N	Agitator in blending tank	CT-01399
B-6868		N	Thermocouple wells	
C-6863	B	L S	Filtrate receiver	CT-01400
E-6941		N	Platform of still	
C-6942	B	L S	Piping diagram for #2 vac. still	S-062
C-6943	B	L S	Monel piping diagram for #2 vac.still	S-062
D-6944	B	L S	#2 Vac. Still	S-062
D-6945			Exhaust System,mentioned on p.XII-6.	
D-6946		M F	Control room	S-062
E-6952	B	L S	General layout #2 still	S-062

* * See the note on page 86 in this section.

XII. DRAWING LIST, contd.

Drawing List	*	*	Title	Equipment Number
D-6956	B	L S	General layout #2 still	S-062
C-6957	B	L S	Cyclone on #2 vac. still	S-062
B-7082	B	L S	Monel condenser #2 vac. still	S-062
B-7121		N	Support for Sperry press	F-0113
A-7136		N	Pipe coil under Sperry press	F-0113
A-8853		M	Bearing for blending tank (See drg. A-459)	
D-10292	B	L S	Piping of air blowing tank	
Hand sketch	B	L S	Dryer for Attapulgus Earth (See corresp. file Mar. 25/47)	
Anniston Drawings				
9C-8000			Layout of chlorinators } to MCL, June 7, 1946	
9C-8245			#2 Aroclor still }	
C-6836-1			Detail of chlorinator (Swan Chemical Co.) } to MCL March 12, 1947	

* * See the note on page 86 of this section.

XIV. HISTORY OF THE PROCESS IN MONSANTO.

In the years 1927 - 1928, the Swann Chemical Company developed a process for making diphenyl, but as the expected demand for it did not continue, other outlets were sought for the diphenyl. Aroclors were produced in the laboratory in 1929, and as they showed good electrical properties, fire resistance, and general inertness, the General Electric Company became interested, and developed the use of Aroclors in transformers and capacitators.

A plant was installed to produce 3000 lbs./day at Anniston.

In 1935 Monsanto acquired the Swann Company, so gaining access to their Aroclor patents. As the demand for Aroclors increased, additional production equipment was installed, located at the Monsanto Illinois factory (then plant "B" and now called the Krummrich plant). This equipment came on stream in September 1936.

The Krummrich plant site was chose partly because of the availability of chlorine there, but when chlorine consumption overtook the production, and chlorine had to be purchased, proximity to the diphenyl production at Anniston had more weight, and additional capacity, largely designed on the plant "B" model, was installed at Anniston in 1941.

By 1946 both plants had doubled their capacity, to give a total production capacity approaching 2 million pounds a month. About 1954 chlorine cells were installed at Anniston.

The Newport plant was designed, mainly on Krummrich plant information, but to a less extent on Anniston information, under Newport Project No. 12 of the London Central Engineering Department.

Chlorination started at Newport in June 1951, Mr. Clegorn from Anniston spending some time at Newport to assist in starting the diphenyl and Aroclor plants there.

The first production of Pyranol by MCC was in 1936, when Pyranol 1488 was made for the General Electric Company, the blending being done at first in rail tanks. Pyranol 1495 was made in July 1938.

The use of tin-tetraphenyl started in 1944, since when the main production has been of 1467 (TCB - 1260 mixture plus tin-tetraphenyl).

XV. OPERATING INSTRUCTIONS.

Operating instructions were given in the report of Lyles, Soffranko and Becker, November 1947, pages 84 - and a revised version was put out by the Krummrich Plant Standards Department, August 10, 1954, (copy sent to Newport, May 23, 1955).

Points calling for special mention are: --

1. Care in melting diphenyl received in cars. The first move is to melt a hole, from the surface, down to the bottom of the tank to prevent development of pressure from the heat of the tank coil or jacket.

2. Usual rail track routine in handling cars.

3. Exclusion of rain etc.

4. Proper use of tracing on pipe lines. Lines to be blown clear after use where necessary.

5. Tank vents and overflow lines to be maintained above the melting point of diphenyl

6. Charges in the diphenyl measuring tank to be kept hot.

7. Fire precautions in handling diphenyl, toluene, and butyl acetate and mixtures containing them.

8. Chlorinator charges kept as cool as possible, and not circulated in the early stage of chlorination.

9. Watch inlet pressure on chlorine line to chlorinator.

10. Care in emptying chlorinators not to suck back the scrubber charge.

11. Keep chlorinator charge out of the chlorine feed line.

12. Watch gravity and temperature of scrubber for indication of poor absorption of chlorine in the chlorinators.

XV. OPERATING INSTRUCTIONS, contd.

13. Control air-blowing temperature to suit the Aroclor being handled.

14. Check dryness of air from Lectrodryer at proper intervals.

15. Guard against burning or coking the still coils.

16. The usual care in lighting gas furnaces.

17. Care in dealing with hot material tapped from the stills.

18. Cleanliness in handling finished Aroclors.

19. With higher Aroclors take special care against blockage of lines.

20. Safety precautions as discussed under "Hazards", Section XI, above.

Pyranols

Operating Instructions relating specifically to the Krummrich plant are given on pages 33- of the report "Dept. A-246 Pyranol Process", Lyles, Soffranko and Miller, March 24, 1947, (copy #8 sent to MCL October 15, 1947).

Points mentioned are:

1. Checking the moisture content of incoming cars of trichlorobenzene. Air blowing at about 50°C. to remove excess moisture.

2. Sampling of materials at various stages for submission to General Electric Company.

3. Exclusion of moisture, HCl fume and dirt.

4. As a working rule, in adjusting blends of TCB with Aroclor 1260, 1 part of 1260 added to 80 parts of the blend will raise the Sp. Gr. about 0.001 and the refractive index about 0.0001.

XV. OPERATING INSTRUCTIONS, contd.

Pyranols, contd.

5. Blends containing tin-tetraphenyl are to be warm (about 80°C.) when filtered, otherwise some TTP may come out of solution.

6. Waterproof cloth over the domes of the filtered rail tanks to exclude moisture and dirt while travelling.

7. Avoidance of delay in dealing with samples because of the numerous testing stages required.

ACKNOWLEDGMENT

To Mr. J. Soffranko who was in charge of the Krummrich plant Aroclor Dept. in 1947, and to the foreman, Mr. Errol Smith.

The report "Dept. 246, Aroclor Process" by Lyles, Soffranko and Becker, November 6, 1946 was of very great help in assembling information for the Newport project, also the companion report "Dept. A-246, Pyranol Process" Lyles, Soffranko and Miller, March 24, 1947.

The reports by Schwarting and others, 1953 and 1954, have been used in the present revision of the process details.

Acknowledgment is due also to other Krummrich plant people who helped then and later.

Dr. E. E. Hardy was of great help at Anniston in 1947 in explaining the process and locating information.

To Messrs. Ellenburg and Wood for guidance around the Anniston plant, and to Messrs. Baker and Dunlop for analytical information.

Mr. Clegorn was very helpful during a visit in March 1955.

I N D E X