rec 3/15/7[illegible]

Process for Interim
Quantities of
Monochloroethylene

Job # 93-000-76922-1016005

written by
D. R. Coos

MONS 058365

Distribution List

1 Tech Reports Library Permanent Copy
2 " " " Loan "
3 " " " " "
4 Circulate to MTCC Re Mgr/Library
5 W.R. Richard
6 A.E. Thompson
7 R.H. Munch
8 J.F. Guinn
9 D.R. Cova
10 J.R. Savage - Manufact Dept
11 J.E. Maurer / W.H. Dubley - Patent Dept.
12 ~~[illegible]~~ T.L. Hossage / C. Paton - Marketing Dept.
13 K.E. Boucher - TSD
14 Harry Johnson - Manufact Dept
15 W.C. Engman - TSD
16 A.E. Leisy - Manufact Dept
17 D.R. Lynch - WGK

MONS 058366

Introduction

Monochlorobiphenyl (MCBP) consists mostly of ~~the ortho~~ O-CBP (o-chlorobiphenyl). Its use is as an intermediate ~~[illegible]~~ raw material for Phenylphenols.

It was made in drum quantities in the Monsanto Research Corporation pilot plant in Dayton, Ohio. The purpose of this report is to describe the procedure for manufacture of interim quantities at the W. G. Krummrich Plant.

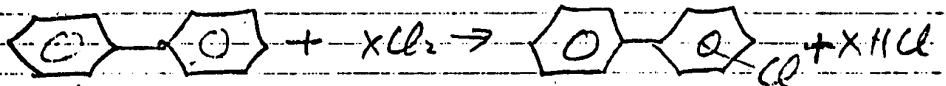
This process was written to cover specifically the case of a short plant run with ~~[illegible]~~ only one fractionation batch. It is for this reason that raw material requirements are high. With an extended run non-product streams would be recycled to the process, and the raw material requirements would be lower.

MONS 058367

Synopsis of the Process

Biphenyl is chlorinated to Aroclor 1111. This material is then fractionated through a 30-tray Oldsum. This is a semi-continuous fractionation in which three cuts are taken. First is a large forecut, then an intermediate cut, and finally the product fraction. The pentachlorobiphenyl is distilled in a straight takeover still and then converted to Aroclor 1254. The forecut is recycled to the chlorination area, and the intermediate fraction is held for recycle to be distilled in future ACB Pounds.

Chemistry of Process



MONS 058368

Raw Material Requirements

The raw material requirements for this process are higher than normal, because it is written for only one still batch. In continued operation, recycling of non-product streams would reduce raw material requirements considerably. (Solvent would be recycled to chlorination and the intermediate fraction to the subsequent fractionation batch). The requirements for 100 lbs. of MCBP are as follows:

171.8 lbs. Biphenyl
0.14 lbs $FeCl_3$
115.1 lbs Chlorine
1.5 lbs Lime
6.0 lbs Aroclor 1242

The residue from MCBP fractionation is converted to Aroclor 1254. The following requirements for each 100 lbs of Aroclor 1254

61.5 lbs. Distilled Residue
84.0 lbs. Chlorine
0.02 lbs. $FeCl_3$
0.3 lbs. Lime

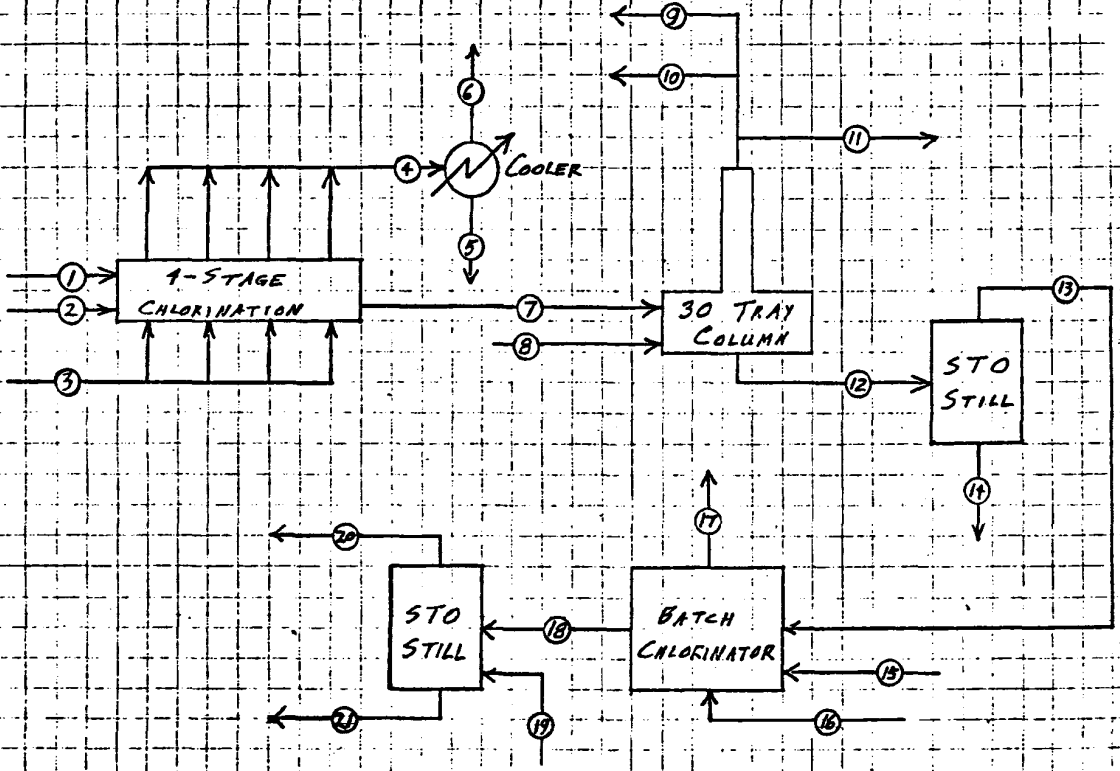
MONS 058369

Lab I

Stream / Component / Concentration	1 Methanol Feed	2 Catalyst	3 Chlorine	4 HCl Effluent	5 Effluent Chlorine	6 Crude Product	7 Crude III	8 Lime Slurry	Effluent	9 Carbonate Reactor	10 Product Reactor	11 MCBP Reactor	12 Distilled Residue	13	14 Final Wash Solution	15 Catalyst	16 Chlorine	17 Ex Gases	18 Aerator Liquid	19 Lime	20 Aerator Effluent	21 Residue
Dichloryl	100			23.4	76.6	3.7	17.1			94.4	32.2	.5	.06	.06								
o-MCBP				4.3	15.1	.25	22.0			5.6	67.6	95.6	2.1	2.2								
2,2'-DCBP				.12	.4	4×10^{-3}	1.5				.0	.4	5.3	5.4								
m-MCBP				.36	1.3	.01	3.8				.1	3.9	11.0	11.2								
p-MCBP				1.7	6.1	.06	17.7				.1	9.5	58.0	57.2								
Dichloro				.12	.4	2×10^{-3}	4.2						19.4	15.9								
Trichloro							.3						1.0	1.1								
Tetrachloro							.4						1.4	1.4								
$FeCl_3$		100					.03						.1		5.6	100			.03			1.2
Chlorine			100.0														100					
HCl				70.1		96.0												100				
Lime								20					1.1		37.8					100		12.2
Aerolor 1192								80.					1.4	2.1	56.6							
Aerolor 1249																			100		100	86.6
Stream Weight, lbs.	425 171.8	.14	115 115.1	844	22.8	61.6	502.4	7.5	111.5	52.1	100.0	136.5	133.8	2.7	.04	182.8	94.1	222.5	.7	145.7 217.6	5.6	
Temp, °C	100	Ambient	Ambient	120	80	80	150	180	80	80	80	270	100	270	Ambient	Ambient	80	180	Ambient	100	290	
Vol, gal	584	—	63.6 ft³	813 ft³	2.6	783 ft³	60.2	0.7	26.9	5.8	10.8	16.3	14.1	?	—	1010 ft³	1200 ft³	19.3	—	17.8	?	

MONS 058370

FIG. 1
FLOW SHEET FOR P.C.F.P



Process in Detail

Chlorination to Aroclor 1111

471.8 lbs of biphenyl is chlorinated to Aroclor 1111 in the 4-stage continuous chlorinator system. ~~[illegible]~~ Cl2 catalyst is added to the biphenyl in a concentration of 0.03%. Chlorine A total of 85.1 lbs of chlorine is fed to the system, 8.8 lbs to each stage. Stage temperatures are held at 120°C in the first 3 stages and 130°C in the last stage. Specific gravities of the liquid effluent from each stage are as follows:

MONS 058372

Stage no.	1	2	3	4
Specific Gravity at 85°C	1.002	1.022	1.039	1.055

A total of 89.4 lbs of HCl is given off. The gas stream is passed through a cooler where its temperature is reduced from 128°C down to 80°C. As a consequence of this, 22.8 lbs of biphenyl and chlorinated biphenyls is condensed out and collected for further conversion to

Aroclor 1254. The cooled off gas, containing about 4% organics, is passed ~~on to~~ through the ~~HCl so present~~ existing HCl gas scrubber before it is fed to the chlorosulfonic acid department.

The crude Aroclor 1111 from the chlorinator is mixed with 7.5 lbs of lime slurry (20% lime in Aroclor 1142). This ~~ga~~ gives concentration of 0.3% lime in the crude. The purpose of lime addition is to react with dissolved HCl in the Aroclor leaving the chlorinator.

MONS 058373

B. Fractionation

291.5 lbs of limed Aroclor 1111 is charged to the stillpot of the 30-tray distillation column. The system pressure is reduced to 330 Torr and the system is brought to reflux. Head and stillpot temperatures will be about 220 and 246°C, respectively. After the system has lined out at total reflux, distillate flow is started forward to the forecut receiver at a reflux ratio of 3/1. At the same time, limed Aroclor 1111

is fed into the stillpot at the same rate as distillate is drawn off. This is continued until a rise in head temperature occurs (this will probably occur when 117. lbs of distillate has been collected) The reflux ratio is now increased to 3/1, and distillate flow is continued into the forecut receiver. The feed of lined crude to the stillpot is continued until a total of 218.6 lbs has been fed. During this period, should the head temperature rise again, the reflux ratio is increased to 5/1. When this amount of lined crude has been fed, the feed is stopped and the reflux ratio is increased to 15/1. Distillate flow to the forecut receiver is continued until the head temperature begins to increase. This indicates the end of the forecut. ~~The cut~~ 221.5 lbs of forecut will have been collected at this point. Distillate flow is now diverted to the intermediate receiver. The forecut is recycled

MONS 058374

to distillation for conversion to heavier products.

~~Distillate~~

The intermediate cut is taken until 520.1 lbs ~~of~~ has been collected. This will occur when the head temperature has leveled out again (indicating ~~the~~ pure σ-CBP at the column head). Head and still pot temperatures will be about 290 and 264°C, respectively at a Biphenyl concentration in the distillate at this time should be 2.0% or less. If it is not, continue distilling into the intermediate cut receiver until it is. The intermediate fraction is held separately for future use (sample to the next MCBP batch). The reflux ratio is now reduced to 0/1, and distillation is continued until 100.0 lbs of product has been collected. Towards the end of the ~~main~~ product fraction samples of the accumulated product should be analyzed to make certain that the m-CBP and Dichlorobiphenyl specifications are not exceeded.

After the product fraction has been distilled off, ~~the~~ fractionation is stopped. Head and still pot temperatures will ~~now~~ have risen to about 253 and 275°C, respectively. 136.5 lbs of residue are pumped to residue storage.

MONS 058375

C. Residue Disposal

The residue from fractionation is

straight-takeover distilled at 40 Torr to remove lime and solder solids. Distillation is continued to a stillpot temperature of 270°C. At this point, 133.8 lbs of distilled residue will have been collected. The remaining final residue (2.7 lbs) is disposed of by incineration.

The distilled residue is chlorinated based on Aroclor 1254. $FeCl_3$ catalyst is added in 0.03% concentration. The chlorination is carried out at 150°C. 182.9 lbs of chlorine is added to ~~~~ form Aroclor 1154. Specific gravity of the finished crude should lie within the range, 1.498-1.508 at 65°C.

MONS 058376

After chlorination is finished, lime is added in the concentration of 0.3%, and the material is then straight takeover distilled at a system pressure of 40 Torr. Distillation is ended at a stillpot temperature of ~~~~ 290°C or less.

At this point, 217.6 lbs of Aroclor 1254 will have been produced. This material is treated with Prorad as is presently done and is then pumped to storage. The residue remaining in the stillpot (5.6 lbs) is disposed of by incineration.

risen to 251 and 274° C., respectively, at this point. Distillate flow to the product receiver is continued at this reflux ratio until a total of 100.00 lbs. of product has been collected. The fractionation is then stopped. At this point, the head and stillpot temperatures will have risen to 265 and 281° C., respectively. The crystallizing point of the distillate will have risen to 61-62° C. by the end of the distillation.

The fractionation residue (33.98 lbs.) is transferred to a straight takeover (STO) still.

4. Distillation of Column Residue

33.98 lbs. of column residue are charged to a STO still. The pressure in the still is reduced to 40 mm. Hg. The still is heated and distillate begins to boil over at a temperature of 190-205° C. The distillation is stopped when the stillpot temperature reaches 360° C. 31.95 lbs. of distillate is collected. This material is recycled for further chlorination to make Aroclor 1254 or 1260. The residue (2.03 lbs.) which consists of tars, $FeCl_3$ catalyst and lime is transferred hot to an incinerator.

DISCUSSION OF IMPORTANT VARIABLES

A. Chlorination to Aroclor ~~1262~~ 1111

1. Chlorination Temperature and Catalyst Concentration

Laboratory experiments have shown that a 0.03% ferric chloride charge (based on biphenyl) is entirely adequate for this chlorination. In fact, earlier work by Helmut Merten on the preparation of Aroclor 1242 has shown that a catalyst concentration of 0.005% is sufficient provided the biphenyl temperature is above 110° when initial chlorination is commenced. For this reason we recommend a minimum chlorination temperature of ~~110°~~ 120°C and a catalyst concentration of 0.03%.

2. Chlorine Purity

Calculations of off-gas volumes, etc., are based on a feed of pure chlorine. In practice, the chlorine feed may contain air (perhaps 25 volume %). A low chlorine content does not hurt the quality of Aroclor 1111 produced, but the presence of excess inert gas contributes to loss of organics in the off-gas stream.

3. Chlorination Level

The object of producing Aroclor 1111 is ultimately to obtain monochlorobiphenyl with a minimum of di or higher

chlorinated biphenyls. In order to hold down formation of the higher chlorobiphenyls, it is necessary to limit the amount of chlorination in the distillation feedstock. Chlorination levels of about ~~5-10~~ 10-12 % have been found to be about optimum. Chlorination below 10% requires excessive biphenyl recycle.

With an Aroclor feedstock of greater than ~12% chlorine content, difficulty is encountered in separating sufficient ~~some~~ p-chlorobiphenyl from the closely boiling dichlorobiphenyls (specifically, 2,2'-dichlorobiphenyl). Disposition of polychlorobiphenyls ~~may also influence the feedstock chlorination level~~. The higher the chlorination level, the greater will be the quantity of ~~di and~~ polychloro biphenyls left in the still pot residue. These must be disposed of either by further chlorination to a higher Aroclor (1154 for example) or incinerated. If outlets for higher Aroclors do not permit conversion of the residue to useful material then the residue should be held to a minimum to avoid excessive incineration expenses.

B. Fractionation

1. Effect of Variations in Degree of Chlorination

The tentative process material balance is based on a laboratory fractionation of Aroclor 1111 produced batchwise in the laboratory. The crude analyzed 11.2 ~~10.9~~ weight % chlorine (~~both by combustion and by~~ by calculation from chromatographic analysis).

Differences in the degree of chlorination will affect crude composition, and hence also the fractionation material balance. A higher degree of chlorination would give a decreased biphenyl concentration and increased concentrations of the chlorinated components, in particular the polychlorinated compounds. This would cause the biphenyl forefraction to be smaller, and the column residue to be larger. This latter effect is undesirable since residue must be either converted to the higher Aroclors or else incinerated if the market for these Aroclors is insufficient. ~~Another undesirable effect is that it will become increasingly harder to maintain the p-chlorobiphenyl level in the product above the minimum specification without exceeding the specification level limit of polychlorobiphenyls in the product. Thus, when the chlorine content of the crude is above 15% it is impossible by this fractionation technique to meet both the above specifications.~~ A lower level of chlorination would reverse the above effects.

If the fractionation is controlled by material balance, the above variations in crude composition could have

quite an adverse effect on product quality and yield. The possibility of crude composition variation must be taken into account. The next section, on control, discusses this point further.

2. Control of Fractionation

There are six points in the fractionation at which action must be taken. The first two are changes in reflux ratio (from 3/1 to 5/1 and from 5/1 to 8/1). These are signaled by rise in head temperature. Chromatographic analysis may also be used, an increase in CBP concentration ~~[illegible]~~ showing the need for an increase in reflux ratio.

The third action point occurs when 218.6 lbs of limed crude has been fed. At this point, the feed is stopped, and the reflux ratio is increased to 15/1.

The fourth point is the end of the forecut and the start of the intermediate cut. This is signaled by a rise in head temperature. It can also be detected by an

increase in o-C.B.P. concentration as shown by chromatographic analysis or by a decrease in overhead boiling point (see Fig. 4 in the Appendix).

The first point is the end of the initial fraction and the start of the product fraction. Reflux ratio is returned to 8/1 at the same time. This point is signaled by a leveling off of head temperature. It is essential that the distillate be sampled at this time and analyzed by chromatography. Biphenyl concentration should be 2% or less before distillate flow is sent to the product receiver. It may be desirable to set the column on total reflux while awaiting analytical results. This would maximize the rigs of the product fraction.

The last action point is the end of the product fraction and the end of fractionation. This point can but be determined by chromatographic analysis of the accumulated product in the golden

~~receiver~~ storage tank. ~~The column could~~
It will be essential that the storage tank
be completely mixed before sampling, so
that a representative sample can be obtained.
It will be necessary when this cut point is near
to place the column on total reflux while
awaiting analytical results. The small ~~c~~
distillate receiver can be used to good advantage
by filling it with distillate and keeping it
separate from the material in product storage.
~~The material~~ Analyses of the material in
the receiver and of the accumulated product in
the storage tank are combined through a
simple calculation to decide whether or not to
pump the receiver material to product storage.

nitrogen would be sufficient to lower the HCl concentration of 510 lbs. of HCl-saturated crude Aroclor 1105 from 4.5 meq.HCl/100g. down to 0.6 meq HCl/100g. It is essential that the nitrogen flow be at least 3.62 lbs./510 lbs. of crude and that the contacting be adequate. Otherwise, the HCl concentration of the sparged material may be too high for the lime treatment step. This would allow HCl to enter the distillation system and cause corrosion problems. It is also necessary that the sparging temperature be at least 150°C. Otherwise the nitrogen will not be as efficient in removing HCl. For example, it can be calculated that at 120°C, the same amount of nitrogen (3.62 lbs/510 lbs of crude) would leave 1.0 meq HCl/100g. in the sparged Aroclor.

3). Lime Treatment

The purpose of the lime treatment is to ~~remove the last bit of~~ HCl ~~from~~ the ~~sparged~~ Aroclor before feeding it into the distillation system.

The lime does not react stoichiometrically with the HCl. Apparently only the exposed surface of the lime particles reacts to form $CaCl_2$ while the interior of the lime particles remains unchanged. Lime requirements can best be expressed as grams lime/meq. HCl. Laboratory tests have shown that 0.15 g. lime/meq. HCl is not quite sufficient to react all the HCl and that 0.20 g. lime/meq. HCl is more than sufficient. The tentative process balance, ~~is based on the higher lime usage.~~

Figure 3 depicts the fall in HCl concentration with time during batchwise treatment with Krummrich plant lime. Reaction is rapid until either the HCl or the reactive portion of the lime is used up. The reaction is virtually complete after 6 minutes. It can be seen from these curves that a lime usage of 0.15 g. lime/meq. HCl is not quite adequate to remove all the HCl while 0.20 g. lime/meq HCl is sufficient to remove all the HCl. From these plots, it is apparent that with the proper amount of lime, a retention time of at least one hour should be sufficient to remove all HCl in a continuous process.

It is essential that the treated Aroclor entering the distillation system be checked for the absence of HCl. Analytical methods are presented in the appendix. Analysis of the crude entering the lime treatment will help ensure that an adequate amount of lime is being fed to the process. If analysis indicates that higher than normal concentrations of HCl are present, the lime feed rate should be increased to keep a ratio of 0.2 g. lime/meq. HCl.

Mixing in lime treatment must be sufficient to keep the lime suspended and to ensure adequate contacting between lime and crude Aroclors.

4). Slurrying of Lime in Aroclor 1142

Lime is fed into the ~~system~~ Aroclor 1104 stream as a concentrated slurry (~~of~~ 25% by weight of lime) in Aroclor 1142. Aroclor 1142 is used rather than Aroclor 1104 because of safety considerations (Aroclor 1142 has a higher flashpoint). Unfortunately Aroclor 1142 contains 17 to 20% dichlorobiphenyls. These are undesirables in MCBP ~~MCS 1209~~ (specification is less than 1.0% polychlorobiphenyls). For this reason the amount of Aroclor 1142 used should be kept at a minimum. The tentative process balance calls for 6.7 lbs. of Aroclor 1142. ~~The largest amount used in the laboratory was 11.8 lbs. (on the same basis). More than this amount of Aroclor 1142 should definitely not be used.~~

5). Crystallizing Point of Distillate

The crystallizing point of distillate was found to range from 70°C. at the start of distillation when almost pure biphenyl was coming over, down to near room temperature when the distillate was high in o-chlorobiphenyl concentration, and up again to 55°C. towards the end of fractionation. Distillate lines and receivers will have to be heated to prevent solidification.

6). Corrosion

The material of construction of the distillation column is stainless steel. Stainless steel is prone to stress corrosion cracking in the presence of inorganic chlorides. For this reason it is essential that crude entering the distillation system be free of HCl. HCl is removed from the crude by ~~stripping with nitrogen gas to remove the bulk of it, then~~ treating with hydrated lime to remove the residual HCl chemically.

It should be mentioned at this point that the stillpot is constructed of steel. Steel should be impervious to the calcium chloride formed in the lime treatment step.

~~11). Blending~~

~~The composition of the distillate will vary during fractionation. To ensure a uniform product, each batch of MCS 1109 should be thoroughly mixed.~~

7.) Fractionation Requirements

Experiments in the Laboratory were designed to simulate fractionation in the [illegible] tray column in the Aroclor Department. The same number of trays was used, and the column holdup-to-charge ratio was approximate the same. In following the reflux ratio program it is recommended that lower reflux ratios not be used. Higher reflux ratios should cause no problem.

8.) Product Storage

Product in storage should be kept above the freezing point of o-CBP which is 32.4 °C. This will prevent the formation of solids. In the laboratory (at about 20 °C) samples of MCBP precipitated out solids after several days of sitting.

~~Tentative~~ Specifications

Tentative specifications for Aroclor 1111 have been included below as a guide to aid in manufacture of MCBP, strictly.

AROCLOR 1111
(Tentative Specifications)

The following specifications have been derived from laboratory analyses and experiments. Deviations from these specifications may cause deterioration of yield and quality of the final product.

Biphenyl Concentration	≤48%
o-Chlorobiphenyl	≥26 1/2%
m-Chlorobiphenyl	~~0-30~~ o – 3.5 %
p-Chlorobiphenyl	≥15%
~~Polychlorobiphenyls~~	~~≤7 1/2%~~
~~HCl~~	~~≤6.5 meq/100-g~~
~~Temperature~~	~~110-120° C~~

2,2' Dichlorobiphenyl < 2 %

Monsanto
ORGANIC CHEMICALS DIVISION

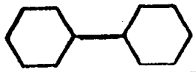
RAW MATERIAL SPECIFICATION

82601	DIPHENYL (BIPHENYL)
PLANT: WGK	USING DEPTS.: 246
DATE EFFECTIVE: 10/23/70	SUPPLIER: Anniston Plant

APPROVALS

PRODUCTION: A. E. Leisy	RESEARCH: W. R. Richard
PROCUREMENT: J. Leyerle	QUALITY CONTROL (Chief Chemist): R. Blowers

GENERAL DESCRIPTION



mol wt. 154.20

CHEMICAL FORMULA

SUPERSEDES SPEC DF: 2/6/69	SAMPLE FOR ANALYSIS: 2 x 16 oz. W.M. Bottles	ISSUED BY: S. Shaulter/G. Stito

CHARACTERISTIC		LIMITS	METHOD
UNRESTRICTED INFORMATION			
(R)	Appearance and Color	Light Yellow Cryst. Solid	10,252
(R)	Crystallizing Point	68.7°C., min.	10,298
(R)	H_2O	250 ppm max.	13,155
GENERAL INFORMATION			
	Distillation Range:		
	First Drop to	2.5°C., max.	10,299
	Dry Point	Must include 255°C. within range	

This specification is the property of Monsanto Company and is for internal use only.

(R) Routine Analysis. All others Analyzed by request only.

Monsanto
ORGANIC CHEMICALS DIVISION

RAW MATERIAL
SPECIFICATION

MATERIAL CODE	MATERIAL
38800	Lime, Hydrated
PLANT	USING DEPTS.
W.G.K.	287, 750, 246
DATE EFFECTIVE	SUPPLIER
4/7/70	Mississippi Lime Co.

APPROVALS

PRODUCTION	RESEARCH
A. E. Leisy T. W. Dalton	W. R. Richard
PROCUREMENT	QUALITY CONTROL (Chief Chemist)
J. Leyerle	R. Blowers

GENERAL DESCRIPTION

CHEMICAL FORMULA

$Ca(OH)_2$

SUPERSEDES SPECS OF	SAMPLE FOR ANALYSIS	ISSUED BY
8/13/69	1 x 16 oz. W.M. Bottle	

CHARACTERISTIC	LIMITS	METHOD
Appearance	Fine white or slightly gray powder, free from lumps or gritty material.	10,188
Assay ($Ca(OH)_2$)	92.00%, min.	10,191
Carbonates as ($CaCO_3$)	3.00%, max.	10,193
Magnesium	0.50%, max.	10,192
Sulfates (as SO_3)	0.30%, max.	10,189

Supplier's certificate of analysis to be sent on day of shipment to:

Chief Chemist, Monsanto, Sauget, Illinois

MONS 058388

(R) Routine Analysis, All others Analyzed by request only.

WGK-514

ORGANIC CHEMICALS DIVISION
W. G. Krummrich Plant
RAW MATERIAL
SPECIFICATION

33500 | Ferric Chloride Anhydride

PLANT: WGK | USING DEPTS.: 233

DATE EFFECTIVE: Jan. 31, 1969 | SUPPLIER: McKesson & Robbins

APPROVALS

PRODUCTION	RESEARCH
A. E. Loisy	W. R. Richard
PROCUREMENT	QUALITY CONTROL (Chief Chemist)
J. Loyerle	R. Blowers T. Bell

GENERAL DESCRIPTION

CHEMICAL FORMULA

$FeCl_3$

mol wt. 162.21

SUPERSEDES SPECS OF	SAMPLE FOR ANALYSIS	ISSUED BY
1/7/66	1 x 8 oz. W.M. Bottle	Higgerson/Stites

UNRESTRICTED INFORMATION

CHARACTERISTIC	LIMITS	METHOD
		J.F.O.
Appearance	Black powder or Granules	16-A
Assay	97.0%, min.	22-570
Sulfates	0.05%, max.	120-570
Ferrous Salts	Trace, max.	S-570-1
Solubility:		112-B
10:10 of Water	Complete to slightly turbid, max.	
10:10 of Formula 30 Alcohol	Complete to slightly turbid, max.	

MONS 058389

(R) Routine Analysis, All others Analyzed by request only.

MONSANTO CHEMICAL COMPANY

INORGANIC CHEMICALS DIVISION

RAW MATERIAL SPECIFICATION

Chlorine

FOR USE IN	MATERIAL NO.
WGK	81059 Inter 18850 External

GRADE: Liquid

DATE EFFECTIVE	ISSUE NO.	SUPERSEDES
12/7/67	103	7/11/66

SUPPLIERS

Monsanto - WGKrummrich Plant

Pittsburgh Plate Glass — Diamond Alkali

APPROVALS

PRODUCTION (Plant Manager)	RESEARCH (Asst. Director)
O. D. Dressel	R. Perry, Jr.
PROCUREMENT (Procurement Manager)	QUALITY CONTROL (Chief Chemist)
R. F. Neunuebel	W. J. Gresham

AUTHORIZED (Quality Control Director)

J. S. Metcalf

GENERAL DESCRIPTION

A clear, heavy liquid, amber to greenish yellow in color. For use in the manufacture of ACL products.

CHARACTERISTICS	LIMITS	PREPARED	METHOD NO.
Appearance	A clear amber to greenish-yellow heavy liquid		10,748
Assay - Cl_2	99.5% Min.		----
Iron as $FeCl_3.6\ H_2O$	0.005%, max.		10,688
as Fe	10 ppm max.		
Non-Volatile Matter.	40 ppm Max.		10,660
Moisture, H_2O	40 ppm, max.		12,793

MONS 058390

Monsanto
ORGANIC CHEMICALS DIVISION
W. G. Krummrich Plant
RAW MATERIAL
SPECIFICATION

MATERIAL CODE	MATERIAL
81059	Chlorine
PLANT	USING DEPTS.
W.G.K.	236, 237
DATE EFFECTIVE	SUPPLIER
2/8/72	*

APPROVALS

PRODUCTION	RESEARCH
T. W. Dalton	D. R. Dill
PROCUREMENT	QUALITY CONTROL (Chief Chemist)
	R. M. Blowers

GENERAL DESCRIPTION

CHEMICAL FORMULA

Cl_2

SUPERSEDES SPEC OF	SAMPLE FOR ANALYSIS	ISSUED BY
NEW		R.Sorrell/S.Shaulte

CHARACTERISTIC	LIMITS	METHOD
Color	Amber to Greenish Yellow	10,748
Chlorine	95%, Min.	
Moisture (H_2O)	75 ppm, Max.	12,793
Sulfate	10 ppm, Max.	
Carbon Tetrachloride	500 ppm, Max.	

* No current vendors specification identified by Research.

Supplier's certificate of analysis to be sent on day of shipment to: Chief Chemist, Monsanto., Sauget, Illinois 62201.

MONS 058391

(R) Routine Analysis, All others Analyzed by request only.

~~Raw Material Specifications~~

~~Control Analysis~~

Analytical Procedures

A chromatographic procedure for analyzing mixtures of biphenyl and monochlorobiphenyls is included in the appendix. This method will be required to analyze Aroclor 1111 and the final product, MCBP, as well as process streams from fractionation. Also in the appendix is a procedure for measuring acidity in timed Aroclor 1111.

For all other specifications analytical method numbers are given on the specification sheets.

~~Product Specifications~~

MONS 058392

Tentative Specifications
for Monochlorobiphenyl

Biphenyl 0.6% Max
o-chlorobiphenyl 80.0% Min.
m-chlorobiphenyl 4.0% Max
p-chlorobiphenyl Remainder
Polychlorobiphenyls 1.0% Max

MONS 058393

Monsanto
ORGANIC CHEMICALS DIVISION
W. G. Krummrich Plant
FINISHED PRODUCT SPECIFICATION

PRODUCT: Aroclor 1254 Electrical Grade | 246
PLANT: W. G. K. | PRODUCT CODE: 811.03 | USE: Sales to 810
DATE EFFECTIVE: 9/8/72 | SALES CODE: 1040-281-16-03

APPROVALS

PRODUCTION: A. E. Leisy
RESEARCH: W. R. Richard
SALES: C. Paton
QUALITY CONTROL (Chief Chemist): H. J. Horner

GENERAL DESCRIPTION

CHEMICAL FORMULA

Also called Pyranol 1476.

MOL WT.

SUPERSEDES SPECS. OF	SAMPLE FOR ANALYSIS	ISSUED BY
11/4/68	3 x 4 Pt. Amber Bottles	R. McCutchan/S.Shaulte

CHARACTERISTIC	LIMITS	METHOD
UNRESTRICTED INFORMATION		
(R) Color, APHA	50, Max.	10,084
(R) Condition	Clear	10,084
(R) Sp. Gr. @ 65/15.5°C.	1.495 - 1.505	10,114
(R) Acidity (mg. KOH/gm.)	0.010, Max.	10,087
(R) Moisture (H_2O)	35 ppm., Max.	10,620
(R) Refractive Index @ 25°C.	1.6375 - 1.6415	10,125
(R) Inorganic (Free) Chlorides	0.05 ppm., Max.	12,741
(R) Pour Point	7 - 12°C.	10,115
(R) Dielectric Constant (60 cycles @ 100°C.)	4.25 - 4.55	T-2273
(R) Hydrolysis Stability Test (As Chlorides)	1.0 ppm., Max.	12,672
(R) Monsanto Stability Test (As Chlorides)	0.5 ppm., Max.	11,503
(R) Distillation Range (ASTM D-20, Corrected)		10,117
10% Distilled b y Wt.	366 - 378°C.	
50% Distilled by Wt.	371 - 383°C.	
90% Distilled by Wt.	379 - 394°C.	
Sulfates	None	12,169
Dielectric Strength @ 25°C.	35 KV, Min.	11,605
Flash Point	None	10,123
Fire Point	None	10,123
Visc. @ 210°F.	44 - 48	10,086
Corrosion Test (6 hrs. @ 210°C. with bright Aluminum Foil.)	MONS 058394	10,126
Change in wt. of Al (%)	0.0	
A.C. Color, APHA	60, Max.	10,084
A.C. Condition	Clear	10,084
A.C. Acidity (mg. KOH/g.)	0.010, Max.	10,087
A.C. Inorganic (Free) Chlorides	0.05 ppm., Max.	10,118

		Internal	Customer	
(R) Power Factor @ 100°C., 60 cycles, Max.	Bulk	0.5%	1.0%	T-2273
	Drums	2.0%	4.0%	

(R) Routine Analysis, All others Analyzed by request Only

CONTINUED PAGE 2

Characteristics	Limits	Methods
GENERAL INFORMATION		
Specific Heat @ 25°C.	0.27	
Coefficient of Expansion (25 - 65°C.)	0.00066 cc/°C	
Fixed Chlorine	54.5 - 55.5%	

Physical Constants Data

Specific gravities of MCBP were measured. The results were:

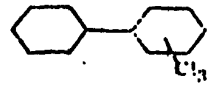
Temp (°C)	Specific Gravity
20 ½	1.161
59	1.133
70	1.120

MONS 058396

PHYSICAL CONSTANTS DATA

AROCLOR 1142 (Technical)

STRUCTURAL FORMULA



Rev. by A. M. Ellenburg

DATE 1947 7/5/57

NAME		SOURCE & DATE
Formula $C_{12}H_7Cl_3$	Total Cl_2 41.5 - 42.5%	Specs 12-21-54
Molecular Weight		
Appearance	Brown to black oily liquid	Specs 12-21-54
Boiling Point at 760 mm		
Boiling Point at ___ mm		
Boiling Point at ___ mm		
Boiling Point at ___ mm		
Crystallizing Point		
Melting Point		
Solution Point		
Specific Gravity 65/15.5°C	1.348-1.360	Specs 12-21-54
Specific Gravity 25/15.5°C	1.384-1.397	
Pounds per Gal. 25 °C	10.5	
Coefficient of Expansion		
Refractive Index n δ		
Infra Red Spectrum (Reference)		
Ultra Violet Spectrum (Reference)		
Viscosity		
Viscosity		
Surface Tension		
Solubility in Water		
Solubility in		
Solubility in		
Sp. Gr. Temp. Coeff. (50-105°C)	0.0009/°C	Specs 12-21-54
Flash Point °F (Method) C.O.C.	350	Aroclor Bull. [illegible]
Fire Point °C	None	
Heat of Combustion		
Heat of Formation		
Thermal Stability (cal./kg/min.)		
Latent Heat of Fusion		
Latent Heat of Vap.		
Specific Heat		
Critical Temperature		
Critical Pressure		
Dissociation Constant		
Other Constants		
		MONS 058397
Toxicity (Give Reference)	Schwartz, U.S. Pub. Health Service Bull. #[illegible] "Industrial Poisons, Hazards and Solvents" by Jacobs Vol. I, p. 463-464, 1941.	

NOTE: These data should not be released outside of Monsanto Chemical Company. Use additional sheets if necessary.

PHYSICAL CONSTANTS (cont'd.)

PHYSICAL AND CHEMICAL DATA

Diphenyl

Specific Gravity: 0.992 at 75°C/4°C.

The Swann Chemical Company bulletin of 1950 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

PHYSICAL AND CHEMICAL DATA (Cont'd.)

Diphenyl (Cont'd.)

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.

APPENDIX E

"Safety and Toxicity Data"

Taken in part from the Standard Manufacturing Process for the Manufacture of Aroclors, in Dept. 246, W. G. Krummrich Plant. New material has been added, and some changes were made in the old material.

Safety Equipment

The following is a list of safety equipment (personal and departmental) required in department 246.

1. Fire extinguishers:

   A. Dry Chemical (3) - Southwest corner Bldg. OR - Main Floor

   - West wall at top of stairs of Second Floor.

   B. Carbon Dioxide Snow - Southwest corner of Bldg. OR - ground level.

2. Chlorine Gas Masks (4) - Southwest corner, second floor

   - Control room, second floor

   - East side, outside control room

   - Wall, Southwest corner of sprinkler house, main floor.

3. Scott Air Masks - West of Lean-To

4. Safety Showers (2) - Southeast corner of Bldg. OR - ground level.

   - Southeast corner of Bldg. OR - 2nd Floor.

5. Davis Vapotester

6. Eye Baths

7. Sprinkler System

Safety Precautions

The following are safety precautions followed in the operation of department 246.

1. Safety Equipment of Employee

   Each employee has the following safety equipment: (1) Hard hat, (2) Safety glasses, (3) Goggles, (4) Rubber and canvas gloves, (5) Rubber overshoes. Special equipment may be issued as necessary for performing a particular job in a safe manner.

2. Sampling of Crude Aroclor

   The samples of Aroclor taken during the chlorination reaction are hot (150° C.). Precaution must be taken to avoid skin contact of samples and inhalation of Hydrogen Chloride, Chlorine and Aroclor vapors.

3. Fires

Fires may result from the ignition of leaking gas, Xylene, Diphenyl, rags, paper, wood, etc. Automatic fire extinguishers and sprinklers have been installed to combat fires.

4. Explosions

Explosions may be caused from accumulated pockets of natural gas or a rapid burning of Diphenyl in a closed container such as a Chlorinator. Chlorinators are equipped with a 10" diameter pressure relief line with a rupture disc made to burst at 22 psi in case Diphenyl within the Chlorinator ignites.

5. Toxicity

Toxicity does not present a serious problem. However, a complete daily change of clean clothes is supplied to each person and time is allotted at the end of each shift to take a bath. Instructions are also issued to avoid actual skin contact with Biphenyl and Aroclor as much as possible and to wash hands and face before eating.

6. Miscellaneous

There are many accidents that can occur that may be classified as health and safety hazards. These accidents may result from such things as broken process lines, acid leaks, steam leaks, etc.

The worst inherent hazards are as follows:

a. Free Chlorine from ruptured line or major leak.
b. HCl Gas from ruptured line or major leak.
c. Dropping bottoms from stills.
d. Hot Aroclor and Diphenyl.

CRUDE AROCLOR 1111

Toxicity: This material has a crystallizing point of 35 °C. Since it is an Aroclor, it should have a medium toxicity range for liquid ingestion and high toxicity for vapor inhalation. The maximum allowable concentration (M.A.C.) is 1 mg/cubic meter.

Fire Hazard: Since this material contains high concentrations of biphenyl, it is very flammable and will burn when exposed to heat or flame or oxidizing materials. It has a flash point of 121° C.

Other Hazards: This material can cause dermatitis, systemic poisoning from the fumes, and yellow atrophy of the liver. There are skin, mucous membranes and eye hazards encountered in handling this material.

Treatment and Antidotes: Call a physician and remove patient from fumes.

Storage and Handling: Protect the skin, lungs, and eyes by wearing approved protective clothing, respirator, and goggles. People who handle this material at elevated temperatures should have medical supervision, and the air should be sampled to insure compliance with M.A.C. There are no special storage restrictions under normal conditions except store in a ventilated area.

Shipping: There are no special shipping restrictions.

FERRIC CHLORIDE ($FeCl_3$)

Toxicity: This material is a solid under normal conditions and is nontoxic or has a rating as low.

Fire Hazard: There is no fire hazard under normal conditions, but it will emit toxic fumes at high temperatures.

Other Hazards: Some people are allergic to this material and may develop skin rash, and there is an eye hazard.

Treatment and Antidotes: Call a physician.

Storage and Handling: Protect the skin with approved protective clothing, avoid the breathing of $FeCl_3$ dust, wear approved respirator with adequate cartridge, and protect the eyes with approved goggles. There are no storage regulations.

Shipping: There are no shipping restrictions.

DIPHENYL ($C_6H_5C_6H_5$)

Toxicity: This material is a solid at normal conditions and is medium toxic. The M.A.C. of vapor is 2 mg/cubic meter of air. If ingested the lethal dose is 2.4 g/Kg of body weight. The fumes are not dangerously deadly, but they are poisonous over a prolonged time.

Fire Hazard: Diphenyl is very flammable and will burn when exposed to heat or flame or oxidizing materials.

Other Hazards: Diphenyl is not too corrosive to skin, but it will affect the eyes.

Treatment and Antidotes: Wash eyes with water and get the patient to fresh air. Try to get patient to vomit, and call a physician.

Storage and Handling: This material can be stored in steel containers away from fire hazards and oxidizing agents. Wear suitable clothes and approved goggles when handling diphenyl.

Shipping: Ship in cars or other suitable containers with a red warning label of flammability.

LIME $Ca(OH)_2$

Toxicity: Lime is not dangerously toxic, and it is regarded as more of a nuisance. This material is a solid or slurry.

Fire Hazard: This material is not flammable.

Other Hazards: Lime is very hazardous to the eyes. It will burn the skin also, and the dust will affect the mucous membrane.

Treatment and Antidotes: Flood eyes and skin with large quantities of water. If lime is ingested, get patient to vomit and call physician.

Storage and Handling: Lime can be stored in steel containers, and people should wear protective clothing, respirators, and rubber or chemical safe goggles when handling lime.

Shipping: There are no special restrictions for shipping, but the containers should have a white label, a caustic warning.

HYDROGEN CHLORIDE (HCl)

Toxicity: This material is a gas under normal conditions and is very toxic. The M.A.C. is 5-10 ppm and a concentration of 1500-2000 ppm would be fatal to humans in a few minutes.

Fire Hazard: There is no fire hazard under normal conditions, but wet gas reacts with some metals to form hazardous hydrogen.

Other Hazards: This material is hazardous to teeth, skin, eyes, and mucous membranes.

Treatment and Antidotes: Wash profusely with water, if injested, eat chalk, magnesium hydroxide, egg whites, and milk, and also administer oxygen. Call a physician.

Storage and Handling: This material should be stored in a cool, well ventilated area away from oxidizing agents. This material can be stored in rubber lined, glass, or tantalum vessels. People handling this material should wear suitable clothing like rubber goggles or chemical safe grades and approved pack or canister gas masks.

Shipping: Shipping containers must have a white warning label and a non-flammable label. Ship in appropriate containers such as rail car or carboy.

CHLORINE (Cl_2)

Toxicity: This material is a gas under normal conditions and is very toxic. The M.A.C. is 0.35 to 2 ppm. A concentration of 1000 ppm would be rapidly fatal. Least concentration with detectable odor is 3.5 ppm.

Fire Hazard: There is no fire hazard under normal conditions, but there is an explosion hazard if chlorine is mixed with a strong reducing agent such as hydrogen or hydrocarbons at high temperature.

Other Hazards: This material causes eye, skin, and mucous membrane irritation.

Treatment and Antidotes: The patient should be given oxygen immediately and treated for shock. Call a physician.

Storage and Handling: This material should be stored in a ventilated and cool place away from fire hazards. When handling, wear a gas mask or approved respirator, clothing and rubber or chemical safe goggles. In strong chlorine concentrations, wear canister gas masks, Scott air pack, or oxygen breathing apparatus.

Shipping: The shipping containers must have a green gas label and a non-flammable label on. Ship in steel cars or cylinders.

~~MCB-1104~~ (Monochlorobiphenyl)

Toxicity: This material is a liquid under normal conditions. It is an Aroclor and so should have a medium toxicity range for liquid ingestion and high toxicity for vapor inhalation. The maximum allowable concentration is 1 mg/cubic meter.

Fire Hazard: This material has some flammability. It has a flash Point (open cup) of ~~170~~ 171° C.

Other Hazards: This material can cause dermatitis, systemic poisoning from the fumes, and yellow atrophy of the liver. There are skin, mucous membranes and eye hazards encountered in handling this material.

Treatment and Antidotes: Call a physician and remove patient from fumes.

Storage and Handling: Protect the skin, lungs, and eyes by wearing approved protective clothing, respirator, and goggles. People who handle this material at elevated temperatures should have medical supervision, and the air should be sampled to insure compliance with M.A.C. There are no special storage restrictions under normal conditions except store in a ventilated area.

Shipping: There are no special shipping restrictions.

AROCLORS 1242, and 1254, 1142, and 1154

Toxicity: This material is a liquid under normal conditions and has a medium toxicity range for liquid ingestion and high toxicity for vapor inhalation. The maximum allowable concentration (M.A.C.) is 1 mg/cubic meter.

Fire Hazard: There is no fire hazard under normal conditions. Aroclor mixes (Xylene, Toluene, Butyl Acetate, etc.) may burn and explode at high temperatures when exposed to flames.

Environmental Protection

There are a total of ~~six~~ seven waste streams from this process. Two of these are HCl off gas streams. These can be handled as ~~and~~ is presently done by passing the gases through ~~an~~ a scrubbing tower where the gases are scrubbed with residue from Aroclor 1016 fractionation. ~~These~~ This will condense out most of the chlorinated biphenyls ~~[illegible]~~ present in the gases. The gases can then ~~be~~ be passed on ~~to~~ to the Chlorosulfonic Acid Department.

There are two residue streams ~~from~~ from straight takeover stills, 2.7 lbs of final MCF Residue and 5.6 lbs of Aroclor 1254 residue. ~~This~~ These materials, both of which contain lime and iron salts should be incinerated.

MONS 058406

The last ~~two~~ three waste streams to be considered are vent losses from various stills.

The vent loss from the fractionation column has been estimated to be less than 0.5 lbs/hr of organics (see page 11 in P-1621, Tentative Process on Refining of MCS-1109 by D. R. Cox). This would be mostly biphenyl and monochlorobiphenyls. More highly chlorinated material would not appear in this stream except in extremely minute quantities. The vent loss ~~to~~ from the Aroclor 1251 still would be no worse than is presently experienced in Aroclor 1251 manufacture. Vent loss from the straight takeover still for the ~~[illegible]~~ MCB Presidue is estimated at 0.5 lbs of ~~chlorine~~ chlorinated biphenyls per hour (assuming a 10 lb/hr air leak ~~at~~ with a temperature and pressure of at the vent of 80°C and 10 Torr, respectively).

References

1.) Thompson, Q. E., Tentative Process for the Preparation of Grade 1105, Report No. P-1620, June 15, 1971

2.) Cows, D. R., Tentative Processes Refining of MCS-1109, Report No. P-1621, Jan 15, 1971.

3.) Cows, D. R. and Q. E. Thompson, Tentative Process for the Manufacture of MCS-1160 at Newport, England, Report No. P-1626, August 2, 1971.

MONS 058408

Appendix

Method 73-9
Job No. 1005122

GAS CHROMATOGRAPHIC DETERMINATION OF MONOCHLOROBIPHENYL ISOMERS IN MCS 1547

PURPOSE OF ANALYSIS

To determine whether all components in MCS 1547 are within specifications.

SAMPLE PREPARATION

None (add benzene to liquify if the sample is solid).

CHROMATOGRAPHIC CONDITIONS

Instrument: Varian Aerograph Series 1200

Type Detector: Flame Ionization

Column: 50' X 0.02" I.D. Support Coated Open Tubular (SCOT) coated with Poly-I-110 polyimide.

Column Temperature: 200°C

Detector Block Temperature: 300°C

Injection Port Temperature: 300°C

Sample Volume Injected: 0.05 µℓ (without split)

Flow Rates

Helium: 6 ml/min.

Auxiliary Gas: 40 ml/min.

Hydrogen: 45 ml/min.

Air: 350 ml/min.

RETENTION TIMES (UNCORRECTED) OF COMPONENTS SEPARATED (CHROMATOGRAM ATTACHED)

Biphenyl (3.6'); o-Chlorobiphenyl (4.5'); 2,2'-Dichlorobiphenyl (6.2'); m-Chlorobiphenyl (6.8'); p-Chlorobiphenyl (7.1').

CALCULATION TECHNIQUE

Normalization with Area Correction Factors.

PRECISION

No determination of precision was made. The variability of a single GC analysis at the 95% confidence limits is usually ±0.5 to 1% relative.

db

Monsanto Industrial Chemicals Company
Applied Sciences
St. Louis, Mo.

1/14/74 - G. M. Gasser, E. M. Emery

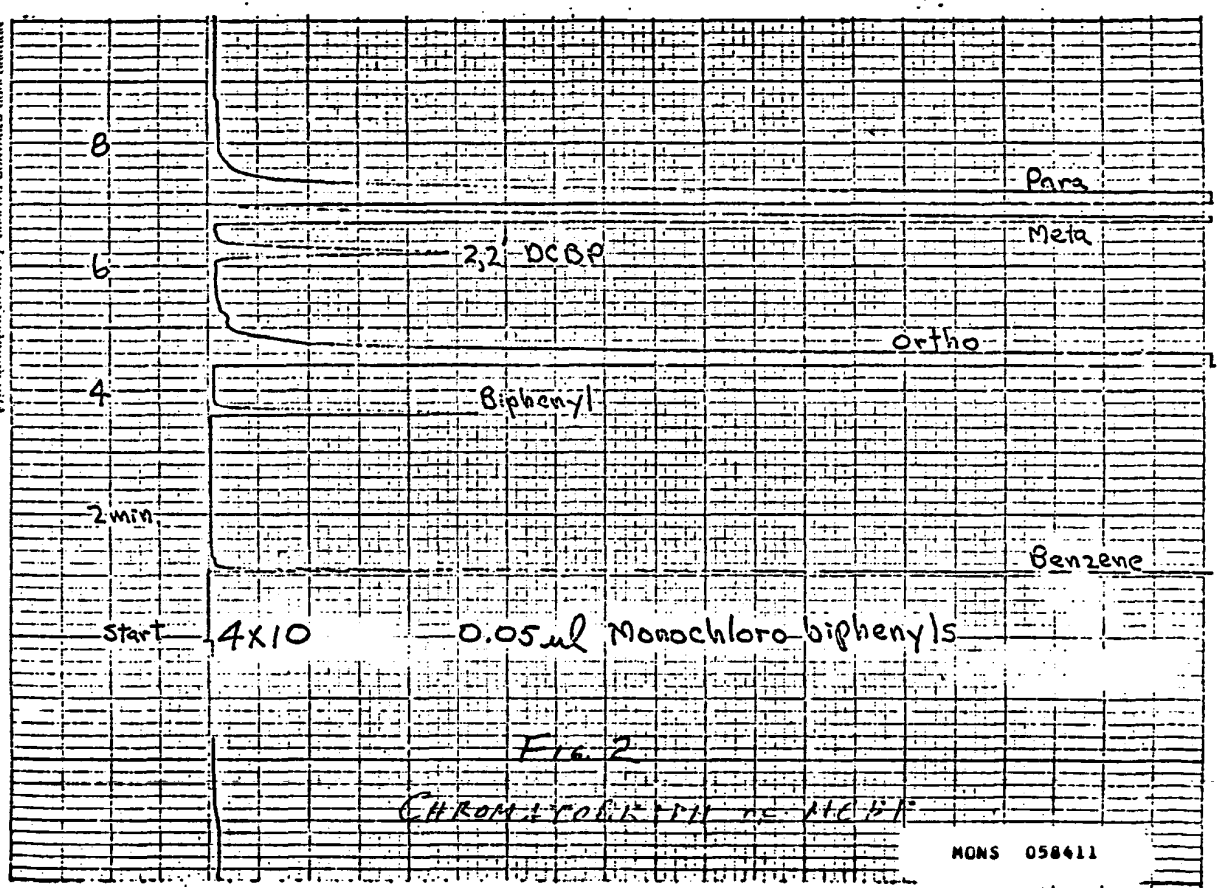


FIG. 2

CHROMATOGRAM OF MCBP

MONS 058411

Determination of HCl in Aroclor ~~1105~~ 1111

Scope

This method measures HCl concentrations from 0.01 meq/100g. to 6.0 meq/100g.

Principle

This method is intended for the determination of acidity attributable to HCl in the Aroclor 1111. It involves a total acid titration with standardized NaOH.

Reagents

1. Toluene, ASTM Grade
2. Sodium Hydroxide, 0.1N.
3. Hydrochloric Acid, 0.1N.
4. Nitrogen

Apparatus

1. 2-Burets, graduated in 0.1 ml divisions
2. Separatory funnels, 250 ml.
3. 150 ml, coarse, ASTM 40-60, Pyrex glass sintered funnels
4. Erlenmeyer Flasks, 250 ml.
5. No. 13-1/2 Rubber stopper with one hole
6. Pan Balance
7. Small glass funnels
8. Cotton

Procedure

1. Add by buret 10.0 ml. 0.1N NaOH to the separatory funnel.
2. Add 100 ml toluene to the separatory funnel.
3. Get total tare weight on funnel, toluene, and NaOH.
4. Pour or drain (not pipet) approx. 50 gm of Aroclor into the glass sintered funnel which is placed in the separatory funnel.
5. Place rubber stopper over glass sintered funnel and force the Aroclor thru the filter with nitrogen.
6. Reweigh separatory funnel to get the weight of the sample.
7. Extract sample with the NaOH layer.
8. Filter extract through cotton plug into the Erlenmeyer flask.
9. Wash organic layer with 50 ml. distilled H_2O.
10. Filter extract again and repeat H_2O wash and filter.
11. Add methyl red indicator.
12. If solution is red, titrate with 0.1N NaOH to methyl red end-point.
13. If solution is yellow, titrate with 0.1N HCl to methyl red end-point.

Calculations

if 0.1N HCl is used:

$$\text{meq/100g acid} = \frac{(10.0 - \text{ml 0.1N HCl used})\ (N)\ (100)}{\text{sample weight}}$$

if 0.1N NaOH is used:

$$\text{meq/100g acid} = \frac{(10.0 + \text{ml 0.1N NaOH used})\ (N)\ (100)}{\text{sample weight}}$$

Notes

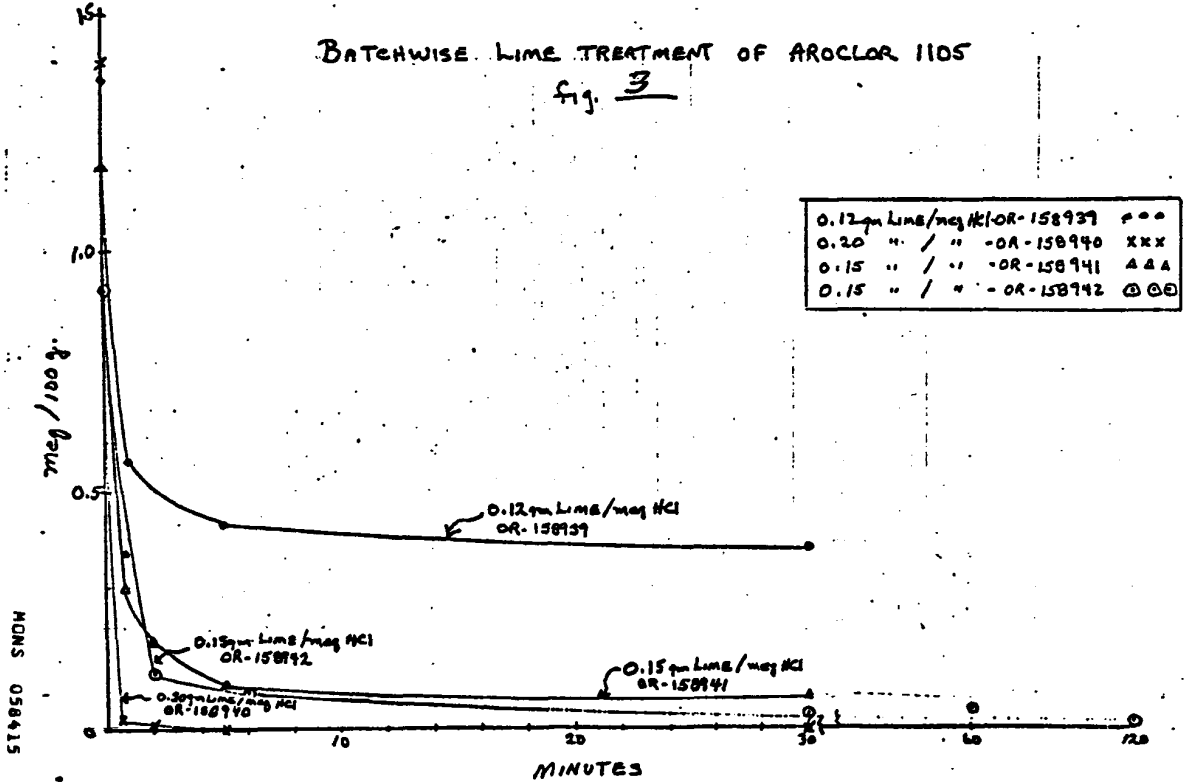
The sample cannot be drawn with a pipet because the vacuum will draw off the HCl.

A qualitative test for HCl can be used with this procedure also. Alkacid test paper can be exposed to the fumes above the sample. Trace amounts of HCl can be detected by this method, down to 0.1 meq/100g.

Sample must be kept above 54°C. Otherwise, solids will form.

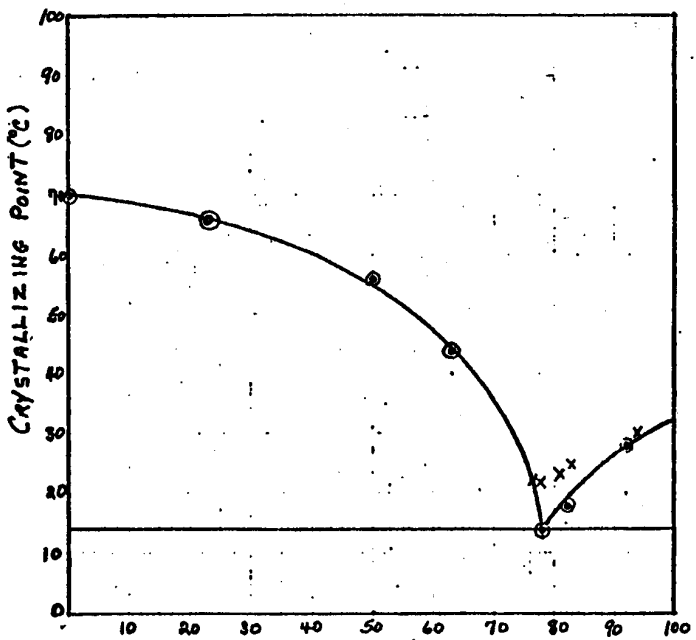
BATCHWISE LIME TREATMENT OF AROCLOR 1105

fig. 3



HONS 058415

EQUILIBRIUM DIAGRAM
BIPHENYL-O-CHLOROBIPHENYL
FIG 4



● = BIPHENYL + O'-CHLOROBIPHENYL

X = DISTILLATE W/MINOR AMOUNTS OF M + P CHLORO BIPHENYL

MONS 058416

MONSANTO COMPANY

ST. LOUIS, U.S.A.

VAPOR PRESSURES

Fig. 5

