R. M. Kountz - General Offices

December 19, 1968

Biphenyl Reclamation

H. S. Bergen, Jr.
J. O. Bright
R. E. Howard
W. R. Richard

Mr. W. A. Kuhn

Several weeks ago you requested information as to the state of Monsanto's knowledge on recovery of biphenyl from "petroleum streams". You also requested an estimate of time and manpower to fully evaluate biphenyl recovery.

In searching engineering and research files and discussing the project with Research, I have put together the following information which is attached:

a) Research notebook pages 77969-70-71-72 and 95 showing the 1961 results of fractionating, recovery of main fractions, chlorinating main fractions, distillation of aroclor 1242 and aroclor test analyses from Signal Oil material.

b) Research progress report on (a).

c) A 1961 engineering analysis by Dr. Dmytryszyn for a plant designed to recover the biphenyl. This analysis was based on the results of Item (a).

d) A series of letters provided by Dr. Richard indicates work done by Gulf Oil and lists potential sources of material which may have a substantial biphenyl level.

As you will see, research work was done only on a Signal Oil sample. It seems logical, if the entire recovery program is to be evaluated, someone must determine the composition of material from other sources. Potentially, Research would need to fractionate samples from each source to determine the feasibility of an adequate recovery level.

Since other groups in addition to engineering are required to fully evaluate biphenyl recovery from petroleum streams, I have not attempted to estimate the time and manpower for the evaluation.

I suggest your review of the attached data after which an overall program should be developed for the pursuit.

If I can further assist, please call.

R. M. Kountz

DSW 200494

at/

(a)

MONSANTO CHEMICAL COMPANY
ORGANIC CHEMICALS DIVISION - RESEARCH DEPARTMENT

SUBJECT: [illegible] SL: Nº 77969

JOB NO. 3659 | PREPARED BY (SIGNATURE) [illegible] | DATE 9/20/[illegible]

Fractionate sample of Signal Oil Refinery, Conn. same material as on pg 77964, but this is another sample for the purpose of obtaining enough material for other analyses could be made therefrom, then analyzed.

Apparatus: Same as on pg 77965 except, larger flask 5 lit dist flask

Charge: 5078 / 1178 wt of col ed. Stainless Steel (Ring type) packing / 3900 gms; Signal Oil Refinery, Conn. Sa 61085 #1+2 A+B

TIME	°C LIQUID	°C VAP.	VAC mmHgABS	FRACT NO.	APPROX SAMPLE WT.	WT OF FRACT.	TOTAL REFLUX RATIO	REMARKS
1:00	175	122	50	1	0		12:1	Heat on.
3:00	181	130	50		15		12:1	
3:30	182	145	50		30		12:1	
3:50	184	153	50		40	58.7	1.5 12:1	about 50°C
10:00	178	153	50	2	0		12:1	Est. Ktals suspended in mixture
10:30	181	156	50		20		12:1	
		156.5	50			81.4	12:1	
11:00		156.5	50		30		2.1/36 12:1	60 - 66.4°C
11:05		156.5	50	3	35		12:1	
11:15	185	156.6	50		40	51.9	1.5/60 12:1	60 - 66.9°C
11:25	180	156.6	50	4			12:1	
11:30		156.6	50			37.9	1/50 12:1	60 - 67.4°C
11:40	174	156.6	50	5			12:1	
11:50	174	156.5	50			55.6	1.4/75 12:1	66.8°C
	174	156.5	50	6			12:1	
1:15	175	156.5	50			117.9	3.5/100 12:1	67.9°C
				7			12:1	
2:00	176	156.5	50			96.7	1.5/100 12:1	68.2°C
2:15	179	156	50	8		61.9	1.5/100 12:1	68.2°C
4:15		156.5	50	9		293.5	7.5/100 12:1	68.2°C
10:50	180	123.5	50	Ended run, retaining [illegible] coming over.				

READ AND UNDERSTOOD BY [illegible] DATE

DSW 200495

STLCOPCB4058559

MONSANTO CHEMICAL COMPANY
ORGANIC CHEMICALS DIVISION - RESEARCH DEPARTMENT

SUBJECT Biphenyl

SL: Nº 77970

JOB NO.	PREPARED BY (SIGNATURE)	DATE
3659	J. Stalm	Sept 25 '61

Continued from pg. 77969

TIME	TEMP °C STILL	TEMP °C VAP	PRESS ABS	FRACT NO	APPROX RMS DIST	WT FRACT	TOTAL % DIST	REFLUX RATIO	
11[illegible]	170	156	100 50	10	0				4 68.3 °C
1[illegible]	173	1	100 50			148.7		11:1	
1 15	174	156	100 50	11 A+B					
2 45	185	156	100 50						
3 [illegible]	194	156	100 50			1378.8		12:1	CP = 68.3 °C
8 15	194	156	100 50	12					note: Approx 80% of this fract boiled at 156° (Thermo-watch became defective)
12 [illegible]	198	156	100 50						
4 [illegible]	197	157	100 50			504.1		12:1	CP. 64.4 °C
4 30	196	161	50	13					
7 45	194	166	50			59.2		12:1	> 45 °C

Residue 918.0 gms.
Cold Trap 2.8 gms
Total Recovery 3898.8 gms
Dist loss. 1.2 gms.

Fractions 8, 9, 10 & 11 were combined as main fract. 1826.0 gms.

* main fract $\frac{1826.0}{3900.0} \times 100$ gms = 46.8% of chge.

* But if 80% of fract 12 is included

$\frac{1826 + (504.1 \times 80)}{3900} \times 100 = 57.2\%$ chge.

$\frac{57}{7}$

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DSW 200496

STLCOPCB4058560

MONSANTO CHEMICAL COMPANY
ORGANIC CHEMICALS DIVISION - RESEARCH DEPARTMENT

SUBJECT: Biphenyl - Corrosion Prob. SL: № 77971

JOB NO.	PREPARED BY (SIGNATURE)	DATE
3659	J.H. Hahn	10/29/61

Purpose: Chlorinate the main fract from 77969 - to Arochlor 1242 ...

Procedure: As outlined on pg 77958 (same as 77961)

Charge: 3844
2344 Flask, distrib. stirrer ring (1)
1500 g Biphenyl from main fract comp 77969
1.0 Gm Fe Cl3 from W.D.K.

TIME	TEMP	Chl added	Sp gr	*Cuft Chl/hr	Remarks
9:30	137	0.		2.0/3.0	Start Chl.
2:30	150	1086	>1,240	3.0	
4:30	148	640/1728	1,280@25°	3.0/0	off for night - agitation off boiling static
8:40 AM	132	1728	1,280@25	3.0	← start Chl.
11:40	148		1,350@25	3.0/2.0	
12:40	148		1.392@25°	2.0	Shut down

Air Blowing

1:10	102	1.5	Start air
2:20	92	1.5	Air off

*Wt of reaction product 2578 gms. incl cat.
W of mixture in off gas trap 8.8 gms

* 42% chlorinated = 2580 = 100% theory - next page

* not correct for Chl. - (relative to air)

READ AND UNDERSTOOD BY	DATE

DSW 200497

STLCOPCB4058561

MONSANTO CHEMICAL COMPANY
ORGANIC CHEMICALS DIVISION - RESEARCH DEPARTMENT

SUBJECT Biphenyl - Signal SL: No. 77972

JOB NO.	PREPARED BY (SIGNATURE)	DATE
3659	J.B. Dahm	10/6/61

Purpose Distill aroclor from preceding page

Equipment Same as on pg 77960

Charge: ~~3460~~ 3460
884 gms. ± lack, ring (1), thermo, shaves, stopper.
2576 gms. aroclor, crude from preceding page
14 gms. Hydrated lime

TIME	STILL TEMP	VAP TEMP	VAC H_2 ABS	WTS	REMARKS
8:00	25		40		Heat, vac, & agitation
10:00	222	199/205	37		yield main fract (theory 42% chlorinated by sp. gr.)
10:05	224	210	40		~~2578~~
1:15	233	218	40		
2:30	237	222	40		$\frac{2496 \text{ VPO}}{2598}$ = 95.4% of theory
3:37	243	225	40		
4:25	255	235	40		
4:35	274	236	40	2496	Shut down

Residue
90.0 gms.
− 14.0 gms.
80.0 gms. Higher residue than lab
− 1.0 gm ± salt and plant (77962 for comparison)
79.0

Earth Treatment

The main fract from distillation was treated at 60-80°C for 2 hrs (with vig agitation) with 0.2% Attapulgus Earth (dried at 200°C)

Filter cake = 32.6 gms.

To Dunnerton 10/10/61 for analysis See pg. 77995

READ AND UNDERSTOOD BY	DATE

DSW 200498

STLCOPCB4058562

MONSANTO CHEMICAL COMPANY

ORGANIC CHEMICALS DIVISION - RESEARCH DEPARTMENT

SUBJECT Preliminary Evaluation of Oxychlor catalysts from Ammiston

SL: No 77995

JOB NO. 3659

PREPARED BY (SIGNATURE)

DATE 10/10/61

ANAL	CODE APHA	ACIDITY MG. NaOH/g	REF. IND @ 25°C	INORGANIC FREE CHLORIDE	HYDROLYSIS STABILITY TEST ppm	THERMAL ppm CHEMICAL TEST	MONSANTO STABILITY TEST ppm	I.C. (ITM) (CONSTANT) TOTAL Cl %	10% DIST	90% DIST RANGE °C
SPEC'S. 77962	50 max	0.010	1.6240 1.6260	0.05	1.0 max		0.4 max	31.5 32.5	326	360 max
STANDARD	10	0.003	1.6245	0.027	0.18	0.14	0.17	30.3	331	351
CS_2 250 ppm 77976	<10	0.002	1.6247	0.017	0.10	0.15	0.18	0.05 %	331.5	351.5
77972 SIGNAL	<10	0.004	1.6247	0.038	0.25	0.25	0.21	0.00	333	357
150 ppm CS_2 77985	10	0.001	1.6259		0.10	0.15	0.19	0.35	330	351
250 ppm CS_2 (77989 INTER. ADDED)	10	0.001	1.6257	0.055	0.52	1.055	0.12	0.2	336	351
250 CS_2 B2806	10	0.002	—	0.009	0.14	0.16	0.10		332	353

	Hue APHA	Viscosity @ 100°C	Refract. Index @ 100°C	Acidity Soluble Chloride	Sp. Gr.	Freeze Point °F	Pour Pt.	Power Fac. 100°C			
SPEC'S. 77962	35	92	4.7/4.9	500 max	1.361 1.364	338-35	45 max				
STAND.	2	2	2	2	2	338					
CS_2 77976	21	86	4.89	355 ppm	1.386	338					
SIGNAL 77972	40		4.84	715 ppm	1.392	355	-15°C	1.03%			
77985	54	85.0	4.89	1300 ppm	1.396	360	-17°C	0.04			
77989	30	85.3	4.91	5105 ppm	1.396	365	-17°C	0.06			
B2806	29		4.86	4005 ppm	1.404	378	-17°C	0.06			

See (NH file) Plant analysis for comparison.

PAPER REMOVED AND ATTACHED TO BACK OF BOOK

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(b)

EVALUATION OF CRUDE BIPHENYL-BY-PRODUCT OF HYDRODEALKYLATION UNITS

Apparatus

1. Distillation of Crude Biphenyl Concentrates

a. Five liter distillation flasks equipped with stirrer, thermometer, still head including a magnetic forward flow control (activated by a Flexipulse timer to assure exact reflux ratios), Oldershaw type, 20 actual sieve plate fractionating column (electrically heated). Approx. 10 theoretical plates. All ground glass joints.

b. Same still as described immediately foregoing, but with a more efficient fractionating column - two 24 inch sections, vacuum jacketed, silvered, 1" I.D., containing 1/8 x 1/4" Cannon extruded packing. Made by H. S. Martin Co. Approx. 30 theoretical plates.

2. Chlorination Equipment

Two liter, "Ace" glass reactor equipped with stirrer, coarse grade fritted glass subsurface chlorine distributor, Fischer flow meter, off-gas absorber (water type), thermometer, electric heating, all ground glass joints.

3. Aroclor Still

Three liter, 3 neck flask equipped with stirrer, thermometer, 6" punched section (20 mm dia.), still head, vacuum facilities, electric heating. All ground glass joints.

4. Aroclor Attapulgus Earth Treating

Three liter, 3 neck flask equipped with stirrer, thermometer, electric heating. All ground glass joints.

Typical Laboratory Experiment

Large batch sizes were required because about 2 liters of Aroclor 1242 was needed for analyses of the important chemical and electrical properties. The laboratory experiment from which data was obtained for cost evaluation follows (Exp. No. 77969).

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1. Fractionation

Charge to the still 3900 grams of biphenyl concentrate, Sample No. A & B (or 1 and 2) from Signal Oil Co. (representative of plant by-product). Table No. 10 summarizes the results of the fractionation.

The percent head fraction shown in the table represents the composite of seven fractions, five of which boil at the same temperature as the main fraction (156°C./50 mm.) but below the desired crystallizing point of 68.6°C., at which the cut is made into main fraction in present biphenyl plant. The cryst. points were 50, 66.4, 66.9, 67.4, 66.8, 67.9, 68.2°C., respectively.

The main fraction was a composite of four fractions (8, 9, 10 and 11) but the crystallizing point remained constant at 68.3°C. - never reaching the crystallizing point of Monsanto biphenyl specifications. The percentage of main fraction shown in the table, however, includes 80% of fraction No. 12 which boiled at the same temperature.

The tail fraction CF (No. 13) was less than 45°C. and was quite yellowish.

2. Chlorination

Fifteen hundred grams of biphenyl main fraction from the foregoing fractionation was charged to the chlorinator and 1.0 gram of ferric chloride (dry) catalyst.

The chlorination was carried out according to plant methods for Aroclor 1242 preparation (Ref. memo, Headworth to Robinson, Aug. 21, 1961, Aroclor Manufacture and Analyses for Aroclor 1242). Heat and agitation was started and when the temperature reached 137°C., chlorine was fed subsurface at the rate of about 2 to 3 cu. ft. per hour. The reaction was held between 145-150°C. Eleven hours were required to chlorinate to about 42% chlorine as measured by the specific gravity of 1392 at 25°C. taken at intervals by means of a regular gravity spindle. The batch was then cooled to 100°C. and air was bubbled through the batch for two hours at 1.5 cu. ft. per hour. Regular laboratory supply of compressed air was used. The crude product, minus the catalyst was equal to 99.5% of theory for 42% chlorination. Weight of liquid in the off-gas trap was 8.8 gms. which was not included in the subsequent distillation.

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3. Distillation

Charge to the still was 2576 gms. of crude Aroclor containing the catalyst and 14 gms. of hydrated lime. The distillation was carried out at 40 mm abs. pressure following plant procedures outlined in the memo referred to under chlorination. The distillate boiling between 199 and 236 at 40 mm. was equal to 95.4% of theory for 42% chlorination or 97% of the still charge.

4. Attapulgus Earth Treatment

The distillate from the foregoing distillation was treated with 0.2% Attapulgus Earth (dried at 200°C.) at 60-80°C. for two hours with vigorous agitation. The treated product was filtered at 60°C.

Analyses

The finished product, Aroclor 1242, was sent to Anniston for analyses of all the important properties.

Discussion

1. Fractionation

The laboratory fractionations of samples of crude biphenyl concentrates using several different fractionating columns are summarized in Table No. 29. Fractionation equal to about 10 theoretical plates (Experiments 77814 and 77966) was considered inadequate, because the main fraction which was a composite of 5 fractions boiling at the same temperature had crystallizing points of 65.6, 65.8, 66.6, 63.8, 65.0°C., respectively. Furthermore, a distinct vapor temperature rise was not detectable. Recovery of the available biphenyl (80% by V.P.C. anal.) as main fraction was only about 52%.

Fractionation equal to about 30 theoretical plates (Exp. 77969) appears to be satisfactory from an operational standpoint in that discernible temperature rises for cutting into and out of the main fraction were achieved. Although the CP of the main fraction was lower than specifications, 68.3°C. compared with 68.9°C. from Monsanto biphenyl as in Exp. 77955, recovery of the available biphenyl in the crude biphenyl concentrate (76% by V.P.C. anal.) as main fraction was 75%.

A sample of by-product crude biphenyl concentrate from Schenectedy Varnish Co. (phenol process) was also fractionated, but since it was discovered that this material was not representative of large scale production, no further work was done (Exp. 77980).

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Exp. 77955 represents the laboratory standard of comparison for ease of fractionation and quality of biphenyl (C.P., 68.9°C.) and Aroclor 1242 made therefore. Regular plant crude biphenyl was fractionated in the laboratory under simulated plant conditions, but intermediates from previous batches were not recycled.

2. Vapor Phase Chromatographic Analyses

The V.P.C. analyses of crude biphenyl concentrates charged to the still and the main fractions from the fractionation are shown in Table No. 30. Components are listed in the order of elution. Since only a few compounds were identified by our Physical Chemistry Group in carrying out these analyses, samples were submitted to the Lion Oil Research Department for further V.P.C. and Mass Spectrographic Analyses. Apparently, the chief contaminant in the fractionated biphenyl main fraction is methyl-biphenyls. Many other compounds have been identified in the crude concentrates as indicated in the reprint of the following memo from W. R. Knox to F. E. Rosenberger, dated October 12, 1961:

"We analyzed these things to get a little more light on their potential for recycle to our HDA plant. Organic has also run them on their programmed temperature chromatograph which gave considerably more resolution than the system used by us. However, they have not identified any components other than biphenyl and terphenyls so that it is difficult to get a direct comparison. Their results are at least qualitatively similar to ours. We ran the biphenyl concentrate on low voltage mass only. Signal sample Number 1 was trapped peak by peak on the chromatograph and low voltage data taken for qualitative identification using our previous confirmation U.V. data for mass number identification of this kind of a sample.

Peak identification of Signal Oil Sample Number 1 was used for Nos. 56 and 60185 A and B. The somewhat questionable assumption of constant ratio of biphenyl to one unseparated methyl biphenyl isomer for 56, 60185 A and B was made to expedite this job. The several different isomer peaks of methyl biphenyl and dimethyl biphenyl are lumped by molecular weight in the following table.

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Sample	Organic Fractionated Biphenyl (From #56)	Signal No. 1	Signal No. 56	Signal 60185A	Signal 60185B
Components					
"Lights"		(2.56)[1]	(1.10)		
Naphthalene		5.7 (6.1)	3.1 (2.5)	0.5	0.6
2 Unidentified		0.2 (1.05)	0.2 (0.65)	--	--
Biphenyl	98.6 (98.3)	64.0 (60.0)	61.4 (54.7)	77.4 (75.2)	77.6 (77.6)
Mebiphenyl	1.4 (1.4)	14.4	15.8	16.6	14.9
Dimebiphenyl		0.9	1.3	0.7	0.6
Fluorene		11.1 (11.2)	14.7	7.0	8.1
2 Unidentified		0.9 (0.93)	0.4	0.1	0.2
o-Terphenyl		1.5 (1.6)	2.3 (2.1)	2.1 (2.2)	2.1 (2.3)
3 Unidentified		0.4	--	Trace	Trace
m-Terphenyl		0.5 (0.71)	0.4 (0.7)	1.1 (1.3)	1.1 (1.3)
p-Terphenyl		0.4 (0.29)	0.4 (0.30)	0.7 (0.72)	0.8 (0.63)
t Unidentified					
Minor comp.					
"Heavys"		0.58	0.96		

[1] All terms in brackets are figures from Organic Research.

The discrepancy in quantitative data is due at least in part to the small percent of low boilers which our constant temperature instrument did not isolate from solvent. Over-all, however, these results appear to give good agreement considering the difference in techniques and lack of refinements in both techniques."

The V.P.C. analyses of Aroclor 1242 produced from Monsanto biphenyl (77955, Table No. 29) compared with Aroclor 1242 made from Signal Oil biphenyl purified by fractionation (77969, Table No. 29) is also shown in Table No. 30. Although there is a slight difference in some of the components, essentially they are about the same.

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TABLE NO. 29

FRACTIONATION OF CRUDE BIPHENYL CONCENTRATES

Experiment Number (Notebook Page Number)	% Head Fract.	B. Pt. °C.	Reflux Ratio	% Main Fract.	B. Pt. °C.	Reflux Ratio	% Tail Fract.	B. Pt. °C.	Reflux Ratio	% Residue	Pressure Hg. Abs.
67814 - Signal Oil #56 (Oldershaw Co. About 10 Theo. Plates)	5.0	78 to 115	10-20:1	42.2 Assay 97.1%	114 to 116 biphenyl	2:1	46.9	116 to 157	10-20:1	5.9	8
77966 - Signal Oil #56 (Cannon Protuded packing. About 30 Theo. Plates)	12.6	104 to 177	4:1	42.2 Assay 98.3%	117 biphenyl	4-6:1	39.0	177 to 193	12:1	6.2	100
77969 - Signal Oil #60185 A and B (About 30 Theo. Plates)	15.8	122 to 156	12:1	57.2 Assay 96.9%	156 biphenyl	12:1	1.5	156 to 166	12:1	25.5	50
77980 - Schenectady Varnish (About 30 Theo. Plates) Semi Works Sample	12.2	103 to 156	20:1	56.8	156	5-20:1	4.5	156 to 172	4:1	24.0	50
77955 - Monsanto Crude Biphenyl - Anniston (About 10 Theo. Plates)	3.8	190 to 245	10:1	75.0	250	4:1	0.8	250 to 300	10:1	20.5	700

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TABLE NO. 30

VAPOR PHASE CHROMATOGRAPHIC ANAL. - COMPONENTS LISTED IN THE ORDER OF ELUTION

	Components not listed are un-identified	Crude Biphenyl: Semi-works Crude Biphenyl Conc.	Crude Biphenyl: Lot # 60185 Part A Crude Biphenyl Conc.	Crude Biphenyl: Lot # 60185 Part B Crude Conc.	Crude Biphenyl: Anniston Plant Crude Biphenyl	Main Fract.: Biphenyl Main Fract. Refined Lot # 56 (Signal)	Main Fract.: Biphenyl Main Fract. Refined 60185 A and B (Signal)	Main Fract.: Biphenyl Main Fract. Refined Anniston Plant Crude		Aroclor 1242: Components not listed are un-identified	Aroclor 1242 From Main Fract. (Signal) 60185	Aroclor 1242 From Main Fract. Anniston Crude
A									1	Biphenyl		
B		1.38			0.36				2	o-Terphenyl		
C		1.03							3	p-Terphenyl		
D		0.55			0.14				4			
E		0.58							5	2,2'-Di-chlor-biphenyl	2.3	3.2
F									6		0.2	0.2
G		0.32							7		0.6	1.0
H		0.06							8	2,4'-Di-chlor-biphenyl	5.7	7.4
I		0.08							9		1.0	1.0
J									10	4,4'-Di-chlor-biphenyl	13.0	12.5
K									11		4.2	5.3
L									12		0.5	0.6
M									13		7.1	7.5
N		4.4	0.62	0.73					14		2.0	2.3
O									15			
P									16		18.1	17.8
Q		0.18	0.12	0.13		0.10	0.09	1.0	17			
R		0.05	0.05						18		8.1	8.1
S	Biphenyl	80.6	75.2	77.16	78.5	98.3	96.9	98.8	19		3.9	3.9
T		0.91				1.4	2.0		20		0.9	0.7
U									21		3.7	3.0
V		2.4	7.3	7.4	0.06	0.17	1.0	0.15	22			
W									23		2.9	2.3
X		0.27	0.06						24		1.9	2.1
Y			0.12						25		3.5	3.0
Z		1.06							26		3.9	3.7
AA									27		3.4	2.9
BB		1.33	8.9	8.0	0.07			0.05	28		0.7	0.6
CC		0.09							29		0.1	0.1
DD		0.12	0.32	0.32	0.04				30			
EE		0.28							31		4.8	4.1
FF									32			
GG									33		3.3	2.9
HH									34		0.1	0.1
II	o-Terphenyl	0.50	2.2	2.3	4.8				35		3.0	2.3
JJ		0.25	0.24	0.1					36			
KK		0.05							37		0.3	0.3
LL		0.37			0.04				38		0.1	0.1
MM					0.05				39			
NN									40		0.1	0.1
OO	m-Terphenyl	1.2	1.3	1.3	8.1				41		0.2	0.2
PP	p-Terphenyl	0.74	0.72	0.63	4.7				42			
QQ		0.09			0.7				43		0.5	0.4
RR		0.08	0.14	0.13					44			
SS			0.29	0.18					45			
TT		0.89	1.2	0.85					46			
UU			0.48	0.32	0.50				47			
VV		0.16	0.21	0.03	0.92				48		0.1	0.2
WW			0.20		0.16				49			
XX					0.10				50			
YY			0.27		1.5				51			
ZZ					1.1				52		0.1	0.2
1					0.27							
2					0.50							
3												

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TABLE NO. 31

ANALYSES OF AROCLOR 1242

Property	Specifications	(Standard) Fractionation Exp. 77955	From Signal Oil Lot #60185 Fractionation Exp. 77969
Color (APHA)	50 max.	10	<10
Acidity (mg. KOH/gram)	0.01 max.	0.003	0.002
Ref. Ind. 25°C.	1.624 to 1.660	1.6245	1.6247
Inorg. Free Chloride	0.05 ppm	0.027 ppm	0.028 ppm
Hydrolyses Stab. Test	1.0 ppm max.	0.18 ppm	0.25 ppm
Thermal Chem. Test	No Spec's.	0.14 ppm	0.25 ppm
Monsanto Stab. Test	0.4 ppm max.	0.17 ppm	0.21 ppm
Item C (Dif. H.S.T. and T.C.T.)	No Spec's.	----	0.00 ppm
Dist. Range	325°C., min. 360°C., max.	331°C. 351°C.	333°C. 351°C.
H_2O	35 ppm	----	46
Viscosity 100°F.	82-92	----	----
Dielectric Constant 1,000 cycles - 100°C.	4.7-4.9	----	4.8
Resistivity, 500 VDC at 100°C. as OHM-CM x 10^9	500 min.	----	795
Sp. Gr. 25°C.	1.381-1.392	----	1.392
Flash Point	338-392°F.	----	355°F.
Pour Point	-14°C. or lower	----	-18
Power Factor 100°C.	----	----	0.03
Sulfur	----	11	1

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3. Aroclor 1242 Quality and Analyses

One of the most important factors involved in determining the acceptability of polyphenyl raw materials from another source is the quality of Aroclor 1242 produced. Aroclor sold to General Electric Co. for use in condensers, transformers, etc. must meet the rigid electrical properties and stability tests to be acceptable.

Table No. 31 summarizes the results of the analyses comparing the quality of Aroclor 1242 produced from rectified Signal Oil Co. crude biphenyl concentrate with a laboratory standard of Aroclor made from biphenyl purified from Monsanto plant crude. All the properties are well within specifications except moisture. Some analyses were not made because not enough sample was available. All the analyses were carried out in the Anniston laboratory.

The results have been given to Organic Engineering (M. Dmytryszyn) for economic evaluation of plant production possibilities. No further work is planned awaiting the results of the evaluation. Also, further evaluation of the biphenyl concentrate from Schenectady Varnish's phenol process is being held up (1962) until samples representative of large scale production can be obtained and, depending upon the results of the economic study.

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