Aroclor 246

PROGRESS REPORT
ORGANIC CHEMICALS DIVISION
TECHNOLOGY DEPARTMENT

REPORTED BY	FOR	DETAILS	REPORT NO.	DATE
D. R. Cova K. W. Holbert	10/1/73- 1/30/74	Yes	2	2/15/74

LOCATION	JOB NOS
Research Center - St. Louis	1005105

FROM WITHIN SCOPE OF THIS WORK REGULATIONS PLEASE RETURN

This document is the property of MONSANTO COMPANY. It contains CONFIDENTIAL INFORMATION which must not be reproduced, revealed to unauthorized persons or sent outside the Company without proper authorization. The recipient is responsible for its safekeeping and destruction.

DISTRIBUTION	*WITH DETAILS
W. R. Richard	J. R. Savage
A. Leisy	R. M. Kountz
*J. F. Quinn	*D. K. Lynch
*R. H. Munch	*V. Brawley
C. W. Roos	L. B. Skatoff
*K. E. Boucher	*G. Johnson

PROJECT TITLE

MONOCHLOROBIPHENYL

PROJECT OBJECTIVE

Development of a process for monochlorobiphenyl.

SUMMARY

The reflux ratio program for monochlorobiphenyl (MCBBP) fractionation was optimized. Distillate composition curves were developed for comparison with plant-scale fractionation. Material balances were developed for MCBP fractionation for product containing either high or low levels of p-chlorobiphenyl. Dow's preference at this point is for low-para MCBP. This makes for easier fractionation control but increases the size of the residue fraction to be disposed of.

Residue from MCBP fractionation was converted to Aroclors 1016, 1242, and 1254. Electrical properties were all satisfactory. Infrared analysis and chromatographic analysis showed differences from the standard products due to changes in isomer concentrations. Aroclor 1016 and 1242 (from MCBP residue) formed crystals on holding at low temperatures. Aroclor 1254 from residue also formed crystals at low temperatures, but when mixed with trichlorobenzene (for transformer use) it appears to be equivalent to standard Aroclor 1254 from the W. G. Krummrich Plant with respect to crystal formation at low temperatures.

D. R. Cova

FUTURE WORK

MONS 058270

1. Continue work on residue disposal.
2. Write Process for Interim Quantities.
3. Obtain additional toxicity and enviromental information on MCBP.

DETAILS

I. Fractionation to Make MCBP

A. General Procedure

This fractionation is carried out in semi-continuous fashion in a 30-tray column under vacuum. The 30-tray column was chosen to simulate the Aroclor 1016 column at the W. G. Krummrich Plant. Column head pressure was set to give stillpot pressure equivalent to that expected in the plant. Likewise the ratio of column holdup to stillpot charge (about 0.05) was maintained close to that experienced in the plant.

After a period at total reflux, distillate flow is started at a relatively low reflux ratio. At the same time Aroclor 1110 is fed into the stillpot at the same rate as distillate is taken off. At some point the Aroclor feed is cut off, and the intermediate cut from the previous run is fed in. At some point during this time, reflux ratio is increased to keep the o-chlorobiphenyl (o-CBP) concentration low in the distillate. When all of the intermediate fraction has been fed in, the reflux ratio is increased to 15/1. From here on, fractionation is in the batch mode. An increase in head temperature signals the end of the forefraction and the beginning of the intermediate fraction. The intermediate fraction is collected separately and is held for recycle to the next fractionation batch. The forecut can be recycled back to the chlorination reactor. Leveling off of the head temperature indicates depletion of biphenyl from the head of the column. This signals that the end of the intermediate fraction is near at hand. When the biphenyl concentration has fallen sufficiently, the distillate flow is diverted to the main fraction receiver and the reflux ratio is lowered (generally to 8/1). Main fraction is taken off until the level of m-chlorobiphenyl (m-CBP) in the main fraction approaches 4.0%. At this point, distillation is stopped. In our laboratory experiments additional small fractions are taken around each cut point. After all these fractions have been analyzed they are mixed in the proper order and amount to make up the four fractions called for, forecut, intermediate cut, main fraction, and residue.

B. Feed/Charge Ratio and Reflux Ratio Program

The purpose of the semi-continuous mode of operation is to reduce time spent in charging the stillpot, batch

heatup, establishing the concentration gradients at total reflux, and dumping residue. For this reason, it is desirable to maximize the feed/charge ratio. As the fractionation proceeds in the semi-continuous mode, biphenyl is selectively removed from the system. Column feed must be stopped before the biphenyl is depleted. Otherwise, o-CBP will reach the head of the column and be taken over in the forecut. The optimum feed/charge weight ratio was found to be 0.63 (the feed here consists of both Aroclor 1110 and the intermediate cut from the previous batch).

The reflux ratio program was optimized, and the final program decided upon is as follows. After a total reflux period to establish the column gradients, distillate flow is started to the forecut receiver at a reflux ratio of 3/1. At the same time Aroclor 1110 is fed to the stillpot at the same rate as distillate is taken off. After 91.4 lbs. of distillate has been collected, the reflux ratio is increased to 5/1. After 102.1 lbs. of Aroclor 1110 has been fed, its flow is stopped, and the intermediate cut from the previous run is fed (on the virgin batch where there is no intermediate fraction to be fed, an additional 40.3 lbs. of Aroclor 1110 is fed to the stillpot). When 40.3 lbs. of intermediate fraction has been fed, the feed is stopped and the reflux ratio is increased to 15/1. After a total of 155.3 lbs. of forecut has been collected, the distillate flow is diverted to the intermediate cut receiver. After 40.3 lbs. of intermediate fraction has been collected, the distillate flow is diverted to the product receiver and the reflux ratio is lowered to 8/1. 100 lbs. of MCBP is distilled off at this reflux ratio. At the end of this period the fractionation is stopped, and 71.4 lbs. of residue is pumped from the stillpot to the residue receiver.

The forecut contains 4.9% o-CBP. Increasing the reflux ratios during this cut to 5/1 and 8/1 (instead of 3/1 and 5/1) would reduce this concentration to about 2-1/2%. Since the forecut is returned to the chlorination step where it is converted to Aroclor 1110, most of the o-CBP is recovered (except for that converted to dichlorobiphenyl). It is estimated that operation at the lower reflux ratios will mean a loss in yield of only 0.4%.

The reflux ratio of 15/1 at the end of the forecut and through the intermediate is essential to reduce the size of the intermediate fraction and to maximize the size of the product fraction.

MONS 058272

A reflux ratio of 8/1 is recommended for the main fraction. It was found that a lower reflux ratio (5/1), while it gave an adequate product (though higher in p-CBP), gave an increased residue. The increase was about 8 lbs. per 100 lbs. of MCBP.

C. Instantaneous Distillate Concentrations

In two of our fractionations, sets of small (1-2 cc) samples were taken. These represented instantaneous distillate samples which should be very useful in comparisons with fractionations in the plant column. In plant scale fractionations, instantaneous samples are easily had whereas composite samples are more difficult to obtain and are necessarily of a historical nature. Figure 1 in the appendix depicts the fall of biphenyl concentration in the distillate through the intermediate fraction and into the main fraction. The concentrations are plotted against percent of the charge plus total feed distilled. On the semi-logarithmic plot the curve is a straight line with a break at low concentrations. This break may represent analytical error, a small degree of decomposition in the stillpot to form biphenyl, or biphenyl holdup in an area infrequently washed by distillate.

Figure 2 depicts the changes in concentration through the main fraction and beyond it if the fractionation is continued further. Concentration curves for m-CBP, p-CBP, and 2,2'-dichlorobiphenyl (2,2'-DCP) are depicted.

Figure 3 shows o-CBP concentration in distillate through most of the forecut during the time that the stillpot is being fed.

D. Material Balances for Plant Runs

Material balances proposed for plant runs are given in Tables I and II. The balance in Table I deals with the first fractionation run when there is no intermediate cut to be fed to the stillpot. As would be expected, raw material requirements and the forecut and residue are higher than normal. On any succeeding runs where intermediate fraction is available to feed the stillpot, the Aroclor 1110 requirements and the forecut and residue sizes will be normal as in Table II.

II. Product Analyses, Specifications and Environmental Considerations

A. Product Compositions and Tentative Specifications

In the past three months, more fractionations of Aroclor 1110 were carried out for the purpose of generating residue for residue disposal studies and also to make samples of high-para MCBP. The original MCS 1547 product specifications gave a product containing 11% or less p-chlorobiphenyl (p-CBP). Three months ago Dow requested MCBP containing at least 14% p-CBP. In order to meet this specification it was necessary to distill further into the residue. This necessitated loosening specifications to the following:

	Specifications	Typical Product
Biphenyl	0.6% max.	0.47%
o-Chlorobiphenyl	75.0% min.	78.3%
m-Chlorobiphenyl	5.5% max.	4.64%
p-Chlorobiphenyl	14.0% min.	15.6%
Total Polychlorobiphenyl	1.3% max.	0.85%

In the second column of the above table is a typical analysis of laboratory product. Table III in the appendix presents a material balance for the high-para process.

More recently, Dow have had another change of heart and have decided that they prefer the low-para MCBP. Low-para MCBP, which is identical with MCS-1547, has the following specifications:

	Specifications	Typical Product
Biphenyl	0.6% max.	0.50%
o-Chlorobiphenyl	80.0% min.	85.75%
m-Chlorobiphenyl	4.0% max.	3.08%
p-Chlorobiphenyl	Remainder	10.25%
Total Polychlorobiphenyl	1.0% max.	0.41%

The second column of the above table gives an analysis of typical laboratory product. Table II in the appendix presents a material balance for the low-para product. Comparing material balances for the low- and high-para products shows that the low-para product generates more residue per 100 lbs. of product (15.5 lbs. more residue per

100 lbs of MCBP). Therefore, the low-para process increases the problem of residue disposal.

B. Analyses of MCBP for Trace Components

Table IV in the appendix gives analyses (by J. P. Mieure) for trace components in the first lab sample of MCS-1547, of low-para MCBP, and of high-para MCBP. It is evident that the preponderance of polychlorobiphenyls in these three MCBP samples consists of 2,2'-dichlorobiphenyl which ranged in concentration from 0.15 to 0.85% in the samples. All the remaining trace components shown in Table IV add up to no more than 500 ppm (or 0.05%).

C. Environmental Considerations

An acute toxicity screen was carried out on MCS-1547 (a sample of MCBP). The results showed no unusual acute toxicological properties of the material (see memo, Johannsen, F. R. to D. R. Cova, 11/5/73, in the appendix). Studies have been started on tissue accumulation in rats, acute fish toxicity, rat inhalation, and a 90-day feeding study for sub-acute toxicity.

III. Residue Disposal

Aroclor 1016 was made from the residue of low-para MCBP. The MCBP residue was straight takeover distilled to remove lime. This distillate was chlorinated to Aroclor 1142 and then fractionated semi-continuously in a 30-tray column to give Aroclor 1016. Analysis of this product at the W. G. Krummrich Analytical Laboratory gave 0.30% High-Boiling Homologs (HBH). The specification on HBH is 0.40%. Infrared analysis showed that the same compounds were present as in standard Aroclor 1016, but the various concentrations were different. On cooling the sample down, a white solid formed between -5 and -10°C. This solid (which is 4,4'-dichlorobiphenyl) did not redissolve on warming up to room temperature.

Aroclor 1016 was also made from the residue of high-para MCBP. The procedure was the same as above. This product was placed in a freezer whose temperature cycled between -14 and -20°C. After two weeks the white solid had formed. In standard Aroclor 1016 from the W. G. Krummrich Plant no solid formed. The conclusions from infrared analysis of the product were the same as with the previous sample.

HBH analysis gave 0.38% Electrical properties were good (0.00066 dissipation factor, 4.80 DK, and 344 x 10^6 resistivity). Table V in the appendix compares chromatographic analysis of this product to standard Aroclor 1016. This material contains twice the concentration of 4,4'-dichlorobiphenyl as does standard Aroclor 1016. In general, those compounds containing para-chlorine are increased in concentration, while those having no para chlorine are reduced in concentration. This is as expected since MCBP residue contains over 50% p-CBP.

In an attempt to resolve the problem of solids formation at low temperatures, Aroclor 1016 from MCBP was diluted down to 30% and 40% concentrations with standard Aroclor 1016. After two weeks in the freezer both mixtures gave solids formation. After two more weeks the 40% mixture still had solids, but none were detected in the 30% mixture.

Aroclor 1242 was made from high-para MCBP residue by straight takeover distilling the residue from the Aroclor 1016 fractionation above and mixing it with the 1016 in aliquot portions. Part of this Aroclor 1242 mixture was placed in the freezer where, after 3 weeks, crystals were detected in it. The remainder of the mixture was converted to Aroclor 1154 by further chlorination, then to Aroclor 1254 by straight takeover distillation. This Aroclor 1254 sample was placed in the freezer. After nine days crystals were found in the material. This material was then diluted 50/50 with standard Aroclor 1254 from the W. G. Krummrich Plant and placed in the freezer along with the standard Aroclor 1254. After two weeks neither sample had formed crystals.

The above Aroclor 1254 sample (undiluted with standard 1254) and standard Aroclor 1254 were each diluted to 70% and 60% concentration with trichlorobenzene (TCB). These four samples were placed in a freezer held at -40°C. The purpose of this test was to duplicate transformer conditions in a cold climate. After six days both samples that contained 40% TCB showed crystals. The other samples containing only 30% TCB contained no crystals although the mixture made with standard Aroclor 1254 was cloudy. Apparently, TCB is the material solidfying.

The undiluted Aroclor 1254 sample tested out satisfactorily for electrical properties (0.00054 dissipation factor and 4.36 decay). Infrared analysis of the sample showed it to

be similar to standard Aroclor 1254 with small concentration differences. At this point Aroclor 1254 from MCBP residue appears to be equivalent to standard Aroclor 1254 for transformer use. As expected, chromatographic analysis shows changes in isomer concentration from standard Aroclor 1254.

IV. Miscellaneous Experiments

Aroclor 1016 from MCBP residue was isomerized over $AlCl_3$ to see if the isomerized product would be more similar to standard Aroclor 1016. It was not. The isomerization appeared to consist of concentrating most of the chlorine on one ring.

In one experiment the MCBP fractionation was carried further to remove the p-CBP from the residue as a tail cut. This tail cut was chlorinated to 30% chlorine, then mixed with the MCBP residue in aliquot portionsand fractionated to make MCS-1043. The product was not suitable, because it contained too much 4,4'-dichlorobiphenyl.

The p-CBP concentration of MCBP was varied in an attempt to reduce the crystallizing point sufficiently low to make a suitable solvent for tolylxylylsulfone. The lowest crystallizing point found was 9°C, and this concept was dropped.

APPENDIX

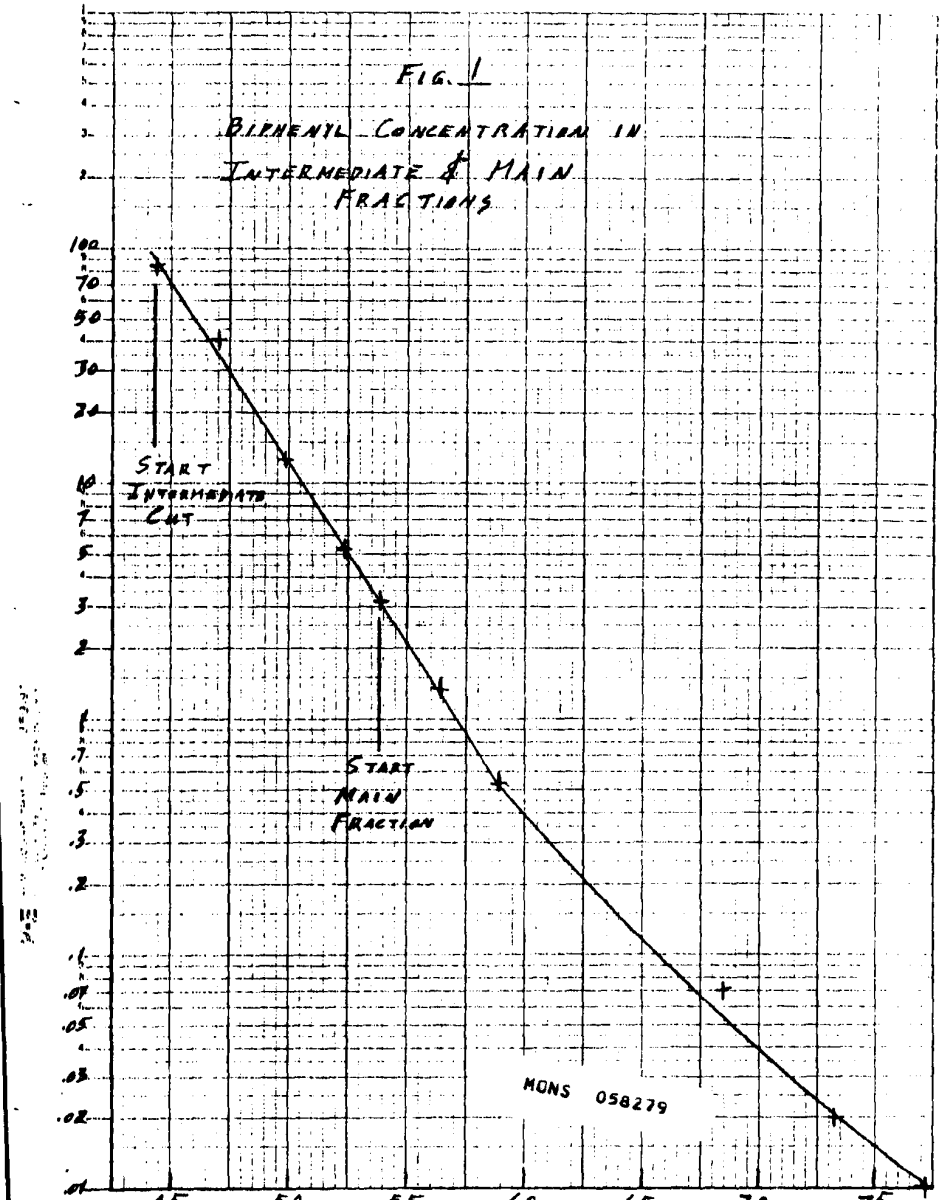
FIG. 1
BIPHENYL CONCENTRATION IN INTERMEDIATE & MAIN FRACTIONS
100
70
50
30
20
10
7
5
3
2
1
.7
.5
.3
.2
.1
.07
.05
.03
.02
.01
START INTERMEDIATE CUT
START MAIN FRACTION
MONS 058279
45
50
55
60
65
70
75
% OF CHARGE + FEED DISTILLED

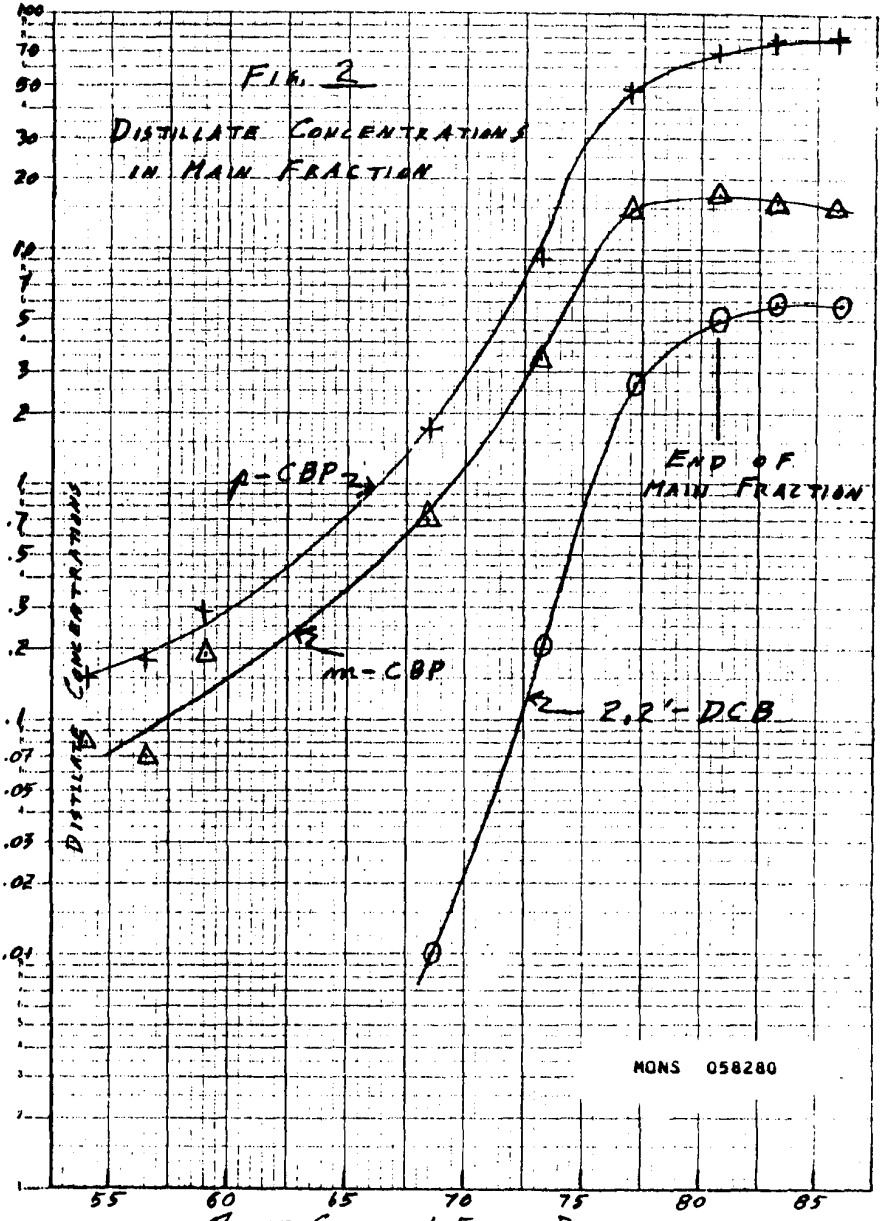
FIG. 2
DISTILLATE CONCENTRATIONS IN MAIN FRACTION
DISTILLATE CONCENTRATIONS
p-CBP
m-CBP
2,2'-DCB
END OF MAIN FRACTION
MONS 058280
% OF CHARGE + FEED DISTILLED
55
60
65
70
75
80
85

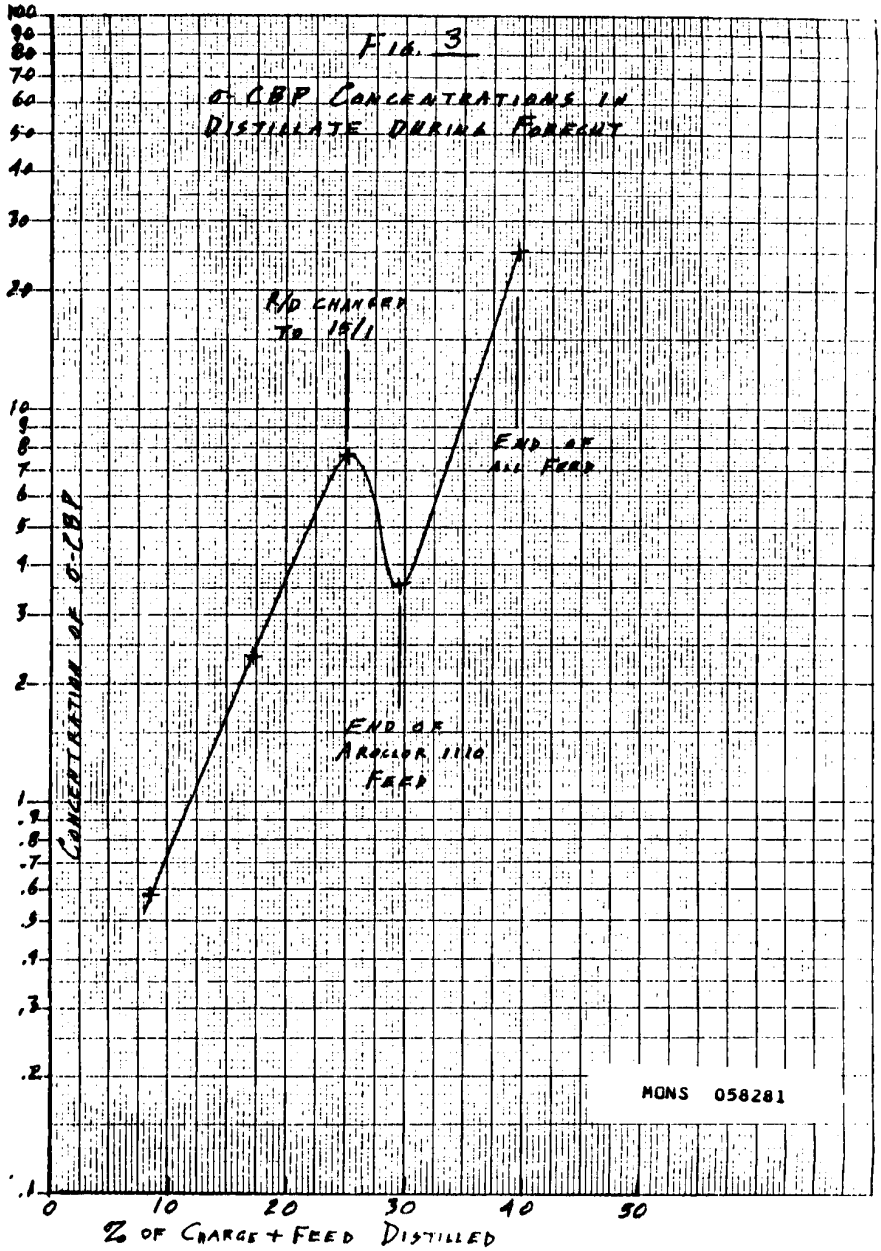
FIG. 3
α-CBP CONCENTRATIONS IN DISTILLATE DURING FORECUT
R/D CHANGED TO 15/1
END OF ALL FEED
END OF AROCLOR 1110 FEED
CONCENTRATION OF α-CBP
% OF CHARGE + FEED DISTILLED
MONS 058281

TABLE I

LOW-PARA MCBP, FIRST FRACTIONATION RUN

	Charge*	Crude Feed*	Fore Fraction	Intermed. Fraction	Main Fraction	Residue
Biphenyl	45.08	45.08	94.45	32.25	.48	.06
Ortho	27.02	27.02	5.48	67.62	85.56	2.26
2,2'-Dichlor	1.52	1.52	.00	.00	.45	5.59
Meta	3.77	3.77	.01	.04	3.87	11.65
Para	17.66	17.66	.03	.08	9.52	61.35
Dichlors	4.22	4.22	.00	.00	.00	16.45
Trichlors	.28	.28				1.11
Tetras	.37	.37				1.46
TOTAL	287.22	215.41	221.52	52.09	100.0	129.02

MONS 058282

*Charge and feed analysis was calculated from the compositions and weights of distillate cuts and residue.

TABLE II

MATERIAL BALANCE FOR LOW-PARA MCBP

	Charge*	Crude Feed*	Intermed. Feed	Fore Fraction	Intermed. Fraction	Main Fraction	Residue
Biphenyl	45.37	45.37	31.82	95.13	31.82	.44	.05
Ortho	28.16	28.16	67.75	4.87	67.75	83.78	.90
2,2'-Dichlor	1.49	1.49				.60	6.00
Meta	3.01	3.01	.03		.03	3.65	8.67
Para	15.45	15.45	.08		.08	11.24	54.94
Dichlors	5.52	5.52				.02	25.25
Trichlors	.30	.30					1.39
Tetras	.28	.28					1.28
TOTAL	224.6	102.1	40.3	155.3	40.3	100.0	71.4

* The analysis of the charge and crude feed was calculated from the analyses and weights of all the other streams entering and leaving the system.

MONS 058283

TABLE III

MATERIAL BALANCE FOR HIGH-PARA MCBP

	Charge*	Crude Feed*	Intermed. Feed	Fore Fraction	Intermed. Fraction	Main Fraction	Residue
Biphenyl	46.3	46.3	33.1	97.5	33.1	0.41	0.10
Ortho	28.3	28.3	66.4	2.5	66.4	79.3	0.6
2,2'-Dichlor	1.3	1.3				0.89	5.6
Meta	3.0	3.0				4.50	8.1
Para	14.9	14.9	0.1		0.1	14.6	53.1
Dichlors	5.0	5.0				0.04	26.8
Trichlors	0.4	0.4					1.9
Tetras	0.2	0.2					1.2
TOTAL	202.1	92.9	44.6	139.1	44.6	100.0	55.9

* The analysis of the charge and crude feed (same as thecharge) was calculated from the analyses and weights of all the other streams entering and leaving the system.

MONS 058284

TABLE IV

TRACE COMPONENT ANALYSIS IN MCBP SAMPLES

Peak No.	Peak Identity	First Lab Sample of MCS 1547 MIC 207152 Blend 2	MIC 207162 Low-Para MCBP	MIC 207175 High-Para MCBP
1-10	?	20-30 ppm total	∿10 ppm total	<10 ppm total
11	Biphenyl	-	-	-
12	?	20 ppm	40 ppm	50 ppm
13	?	50 ppm	100 ppm	40 ppm
14	o-Chlorobiphenyl	-	-	-
15	?	30 ppm	30 ppm	30 ppm
16	?	50 ppm	70 ppm	80 ppm
17-19	m & p-chlorobiphenyl and 2,2'-di-chlorobiphenyl	-	-	-
20	a dichlor	65 ppm	90 ppm	190 ppm
21	?	<5 ppm	<5 ppm	<3 ppm
22	a dichlor	22 ppm	30 ppm	40 ppm
23	a dichlor	35 ppm	30 ppm	40 ppm
24	?	6 ppm	3 ppm	<3 ppm
25	?	3 ppm	<3 ppm	<3 ppm
26	?	4 ppm	<3 ppm	<5 ppm
27	4,4'-dichlor	80 ppm	<10 ppm	<5 ppm
28	?	4 ppm	} probably less than 50 ppm	} Very low
29-34	?	∿50 ppm total		

Note: Peaks 1 through 28 were determined with a 4 meter x 1/8" dia. Santicizer 409 column at 200°C and a helium flow of 40 ml/min. Peaks 29 through 34 were detected using an OV-17 column 4 meters x 1/8" dia. at 260°C. Flowrate was 20 ml/min. The dtector was a flame ionization unit in both cases.

Monsanto

FROM (NAME & LOCATION): Medical, A2SC

DATE: November 5, 1973

CC:

SUBJECT: MCS 1547 - Acute Toxicity Screen

REFERENCE: Y-73-182

TO:

D. R. Cova
T3C

The attached toxicity report characterizes the results of the acute screening studies with MCS 1547. The following data summarize those results:

OLD_{50} (mg/Kg)	DLD_{50} (mg/Kg)	Skin Irr. (max=8.0)	Eye Irr. (max=110.0)	Vapor Inhalation (mg/L)	mortality
2,370	$\geq$2,000	2.4	7.6	0.0	0/6

This material appears to possess no unusual acute toxicological properties that would require special handling practices other than the good hygienice practices employed with any industrial chemical.

Frederick R. Johannsen

/bkp

att.

YOUNGER LABORATORIES

INCORPORATED

123 CLIFF CAVE ROAD SAINT LOUIS, MO., 63129

PHONE: (314) 487-6663

Toxicologic Investigation of: MCS 1547

Lot Number 001-NBP207155

Date 10/2/73

To: Monsanto Company
St. Louis, Missouri 63166

Project No. Y-73-182

ACUTE ORAL TOXICITY

Species Sprague-Dawley Albino Rats

LD_{50} 2,370 Mg/Kg 95% Confidence Limits 2250 - 2490 Mg/Kg

CONDITIONS Fed Undiluted - Single Oral Dose

	Dosage Mg/Kg	Average Initial Weight Male	Average Initial Weight Female	Mortalities/Dosed Male	Mortalities/Dosed Female	Mortalities/Dosed Combined	Time of Mortality
1	2000	220	215	0/2	1/3	1/5	One to three days
2	2510	220	225	1/3	2/2	3/5	
3	3160	240	225	2/2	1/3	3/5	
4	3980	210	220	2/3	2/2	4/5	
5							
6							

Signs of Intoxication: Reduced appetite and activity (one to three days in survivors), increasing weakness, collapse, and death.

Gross Autopsy Decedents: Lung and liver hyperemia and acute gastrointestinal inflammation.

Survivors (7 Days) Viscera appeared normal

ACUTE DERMAL TOXICITY

Species New Zealand Albino Rabbits

LD_{50} > 2,000 Mg/Kg 95% Confidence Limits --- Mg/Kg

CONDITIONS Applied Undiluted - 24 hours exposure

	Dosage Mg/Kg	Initial Weight Male	Initial Weight Female	Mortalities/Dosed Male	Mortalities/Dosed Female	Mortalities/Dosed Combined	Time of Mortality
1	1260	2.5	---	0/1	---	0/1	
2	2000	---	2.4	---	0/1	0/1	
3	3160	2.6	---	1/1	---	1/1	1 Day
4	5010	---	2.3	---	1/1	1/1	2 Days
5							
6							

Signs of Intoxication: Reduced appetite and activity (one to two days in survivors), increasing weakness, collapse, and death.

Gross Autopsy Decents: Hemorrhagic areas of the lungs and liver, slightly enlarged gall bladder, and gastrointestinal inflammation.

MONS 058287

Survivors (14 Days) Viscera appeared normal.

The material in this report is to be used in development of the product and may be given to responsible sales contacts, but it is not to be used by them in advertising copy. The source of this material is not to be divulged until it appears in formal publications. No exceptions to the established rule may be made without the approval of the Medical Department in St. Louis. Customers' inquiries regarding matters of toxicity are to be referred as before to the Monsanto Company Medical Department in St. Louis for reply.

TEST MATERIAL MCS 1547
LOT NUMBER 001-MBP207155
PROJECT NO. Y-73-182

ACUTE EYE IRRITATION

Species New Zealand Albino Rabbits

Irritation Score (24, 48, 72 Hour Avg.) 7.6 Classification Slight *

Dose and Conditions 0.1 Ml. applied undiluted /F.H.S.A./ 24-Hours Exposure

* F.H.S.A.: NOT classed as an eye irritant

Time of Reading Hours	STRUCTURE	SCORES ANIMAL NUMBER 1	2	3	4	5	6	Mean Score (X/110)	Comments
24	- CORNEA	0	0	0	0	0	0		Immediate: Discomfort was slight
	- IRIS	0	0	0	0	0	0	7.6	1-Hr.: Slight to moderate erythema, very slight edema copious discharge
	- CONJUNCTIVAE	8	10	6	8	8	6		
48	- CORNEA	0	0	0	0	0	0		24-Hr.: Slight to moderate erythema, very slight edema slight to moderate discharge
	- IRIS	0	0	0	0	0	0	2.3	
	- CONJUNCTIVAE	2	4	2	2	2	2		
72	- CORNEA	0	0	0	0	0	0		48-Hr: Slight to moderate erythema
	- IRIS	0	0	0	0	0	0	1.2	
	- CONJUNCTIVAE	2	2	2	0	2	0		72-Hr.: Slight erythema
168	- CORNEA	0	0	0	0	0	0		
	- IRIS	0	0	0	0	0	0	0.0	
	- CONJUNCTIVAE	0	0	0	0	0	0		

PRIMARY SKIN IRRITATION

Species New Zealand Albino Rabbits

Irritation Score (24, 72 Hour Avg.) 2.4 Classification Moderate *

Dose and Conditions 0.5 Ml. applied undiluted /F.H.S.A./ 24-Hours Exposure

* F.H.S.A.: NOT classed as a primary skin irritant

Animal Number	HOUR / SKIN	ERYTHEMIA 4 I	4 A	24 I	24 A	48 I	48 A	72 I	72 A	168 I	168 A	EDEMA 4 I	4 A	24 I	24 A	48 I	48 A	72 I	72 A	168 I	168 A
1		0	0	1	1	1	1	1	1	0	0	0	0	2	2	1	2	1	1	0	0
2		0	0	1	1	1	1	1	1	0	0	0	0	3	3	2	2	1	1	0	0
3		1	1	1	1	1	1	0	0	0	0	0	0	2	2	2	2	1	1	0	0
4		1	1	1	1	1	1	1	1	0	0	0	0	2	2	2	2	1	1	0	0
5		0	0	1	1	1	1	0	0	0	0	0	0	2	2	1	2	1	1	0	0
6		0	0	1	1	1	1	1	1	0	0	0	0	2	2	2	2	1	1	0	0

Comments: Slight defatting effect - flaked off in seven to ten days. No injury in depth.

INHALATION TOXICITY - AMBIENT TEMPERATURE

Species Sprague-Dawley Albino Male Rats

Exposure period 6 Hours Mortality 0/6 Vapor concen. 0.0 Gram / M³

Avg. chamber temp 26 °C. Avg. chamber rel. humidity 85 %

Chamber capacity 35 L. Air flow rate 4.0 L./min.

Initial sample size 112.0 Grams Recovered sample size 112.0 Grams

Sample vaporized 0.0 Gram Recovered from condenser 0.0 Gram

Signs of Intoxication None

Ten day observation period uneventful

Gross Autopsy Decedents MONS 058288

Survivors(10 Days) Viscera appeared normal

Test Method Reference A.T.S. 8/73 Submitted by: MELVIN D. BIRCH

comp.	cl no.	Aroclor 1016 from MCBP residue	Aroclor 1016 from USGS
1 Biphenyl		—	0.02
2 Ortho	1	0.10	0.70
3 2,2'	2	0.74	1.6
4 Meta	1	—	0.46
5 Para	1	0.03	0.20
6 2,5	2	0.16	0.69
7 2,4+2,6	2	0.10	0.41
8 2,3'	2	1.0	3.0
9	3		
10 2,3	2		0.22
11 2,4'	2	9.2	8.8
12 2,5,2'	3	4.5	10.4
13	3	4.1	4.2
14	3	0.3	0.9
15 2,3,2'	3	1.2	4.4
16	3	3.0	2.5
17 3,3'	2		
18 3,4'	2		0.06
19	2	0.30	1.1
20	4	0.11	0.15
21 4,4'	2	4.91	2.4
22	3	1.2	3.1
23	3	1.3	1.3
24	4	—	—
25 2,5,4'	3	11.1	8.5
26 2,4,4'	3	17.8	8.8
27 3,4,2'	3	7.3	6.2
28 2,5,2',5'	4	1.3	4.1
29 2,3,4'	3	5.2	3.5
30	4	3.9	4.7
31	4	1.4	1.1
32	4	1.6	4.2
33	4	0.1	0.4
34	4	1.2	1.7
35	4	1.6	2.0
36	4	2.0	2.8
37	5	0.43	0.44
38	5	—	0.07
39	5	0.12	
40	5	0.26	0.12
41	4	0.21	0.04
42 3,4,4'	3	4.9	1.24
43	4	3.3	0.70
44	4		
45	4	1.6	0.20
46 2,4,5,2',5'	5	0.17	
47	5	—	
48	5	0.02	
49	4	0.15	
50	4	0.10	
51	5		
52 2,3,4,2',5'	5		
53	5		
54	5		
55	5		
56			
57			
58			
59 Alone			

TABLE V

COMPARISON OF STANDARD AROCLOR 1016 AND THAT FROM MCBP RESIDUE

MONS 058289

TABLE VI

DESCRIPTION OF EXPERIMENTS

Page Number	Description
207165	Fractionation of Aroclor 1142 from chlorinated MCBP residue to make Aroclor 1016.
207167-8	Fractionation of Aroclor 1110, fourth in a recycle series.
207169-70	Fifth fractionation of Aroclor 1110 in the recycle series.
207171	Blending of cuts from 207165 to make Aroclor 1016 made from the residue from low-para MCBP.
207172	Composition of standards and area correction factors for MCBP fractionation.
207173	Sixth fractionation of Aroclor 1110 in the recycle series.
207174	Distillation of MCBP residue by STO distillation.
207175	Making up of high-para MCBP from cuts from 207167.
207176	Blending of tail cuts from 207173, 207169, and 207167 for chlorination.
207177	Chlorination of mixture from above to 32% chlorine level.
207178	Analyses by J. P. Mieure of various MCBP samples for trace components.
207179	Fractionation in a 30-tray column of a mixture of the chlorinated material from 207177 and distilled residue from 207174 in aliquot portions to make MCS-1043. The 4,4'-chlorobiphenyl content was too high.
207181	Chlorination of an aliquot mixture of blend from 207176 and distilled residue from 207174 (representing the residue from high-para MCBP distillation) to make Aroclor 1142.
207182	Fractionation of material from 207181 in a 30-tray column to make Aroclor 1016.

Table VI - Description of Experiments (Cont'd.)

Page Number	Description
207183	Blending of cuts from 207182 to make Aroclor 1016. This product formed a small amount of solids on standing a couple of weeks in a freezer.
207184	Isomerization of Aroclor 1016 from MCBP residue (207183) over $AlCl_3$ catalyst.
207185	Addition of more p-chlorobiphenyl to high-para MCBP to see if crystallization temperature would be appreciably lowered. It was not.
207186	STO distillation of isomerized Aroclor from 207184. This product looked even more dissimilar from Aroclor 1016 than did that from MCBP residue.
207187	STO distillation of residue from Aroclor 1016 fractionation on 207182.
207188	Dilution of Aroclor 1016 from high-para MCBP residue down to 40% and 30% with standard Aroclor 1016 before storing in the freezer. After 3 weeks a few crystals formed.
207189	Blending of distillate from 207187 with cuts from Aroclor 1016 distillation on 207182 to make Aroclor 1242. This material was placed in the freezer and precipitated out some crystals after 3 weeks.
207190, 207193	Seventh fractionation in the recycle series. In this run a lower reflux ratio was used in the forecut and instantaneous samples were taken throughout the distillation for the first time.
207191	Chlorination of Aroclor 1242 from 207189 to Aroclor 1154 to simulate production of Aroclor 1254 from MCBP residue.
207192	STO distillation of Aroclor 1154 from 207191 to make Aroclor 1254. This material was placed in the freezer and crystals formed after 1-1/2 weeks.
207194	STO distillation of residue from Aroclor 1016 fractionation on 207165.
207195	Analyses of instantaneous samples from 207190, 207193.

Table VI - Description of Experiments (Cont'd.)

Page Number	Description
207196	Aroclor 1016 and 1254 samples placed in freezer.
207197	Eighth fractionation in the recycle series. A low reflux ratio was again used in the fore-fraction and the feed/charge ratio was reduced slightly.
207198	Analyses of samples from 207197.
207199	Freezer tests of Aroclor 1254 diluted with trichlorobenzene.