SUMMARY OF OPERATING CONDITIONS AND ANALYSIS FOR SEPARATION OF MONOCHLOROBIPHENYL FROM AROCHLOR

Project Number
1661

MONSANTO RESEARCH CORPORATION

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for

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Specialty Products Group
Sauget, Illino.s

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PURPOSE

The purpose of this work was to separate monochlorobiphenyl from Arochlor for DOW Chemical. Specifications for the product (designated MCS 1547) are:

	Biphenyl	0.6% max.
(O⁻)	2-chlorobiphenyl	80.0% min.
(M⁻)	3-chlorobiphenyl	4.0% max.
(P⁻)	4-chlorobiphenyl	14.4% max.
	Dichloro- and higher biphenyls	1.0% max.

EQUIPMENT

Equipment used for the distillation was a 750 gallon SS gas fired vacuum still system with a 20" dia. 45' packed column.

OPERATING CONDITIONS

In each of 5 batches, thirteen drums of feed and 1/2% by weight of lime were initially charged to the still. The still was brought up to relux and the product collected in one drum increments.

In batches 1 and 2, after three drums of product were collected, three drums of feed were charged to the still. After the sixth drum was collected three additional drums of feed were charged and after the eighth drum was collected, two drums of feed were added.

In batches 3 through 5 a combination of feed and intermediate fractions from previous batches were recycled to still after the initial charge of thirteen drums of feed. See Data.

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The still was operated with a pot temperature of nominally 230°C at a pressure of approximately 20" Hg. The vapor temperature generally ranged from 165 - 185°C. Pressure drop across the column was maintained between 5-7" Hg and the reflux ratio was varied from 4/1 to 16/1. Column head pressure ranged from 30 to 100mm of Hg.

DATA

A summary of the weight (and %) of material collected at each reflux ratio is presented in Table 1 along with the analysis of the drummed material collected at the start (and the end) of the period during which the still was operating at a given reflux ratio. Raw data sheets showing the weight collected and the analysis (when made) for each drum are attached. Analyses obtained from St. Louis (Dario Cova) were used for blending 11 drums of distillate to constitute Lot 001 and 9 drums for Lot 002. Table 2 shows the analyses of the individual drums blended and the analysis of the resultant blend.

In general, analytical data generated by MRC and Monsanto - St. Louis were in reasonably good agreement, however, appreciable differences in the % di- and higher biphenyls were reported by MRC and Monsanto - St. Louis. Somewhat smaller variances were noted in the ortho and para data.

Table 1

DATA SUMMARY MCB DISTILLATION

Batch No.	Weight #	% of Total Wt.	Reflux Ratio	Analysis* - - Start - -				- - End - -			
				ϕ	O-	M+P	Di-	ϕ	O-	M+P	Di-
1	3900	44	4/1	100				50			
	2245	25	8/1	75	25			3	96		
	900	10	6/1		89	10			60	40	
	1800	20	8/1		32	63				85	15
2	1800	23	4/1	99+				99+			
	450	6	6/1	99+				99+			
	1350	17	8/1	99+				83	16		
	1350	17	10/1	71	29			34	65		
	3150	40	16/1	9	91				1	93	6
3	1350	17	4/1								
	450	6	6/1								
	1350	17	8/1	85	14			88	11		
	1350	17	10/1	55	45			8	89	3	
	3420	43	16/1	3	96				5	91	5
4	2250	29	4/1								
	1350	18	8/1	96	4			80	20		
	900	12	10/1	80	20			34	65		
	3150	41	16/1	10	90				83	15	3
5	1800	25	4/1								
	1800	25	8/1								
	900	12	10/1								
	2700	38	16/1	11	89				8	88	4

* Monsanto-St. Louis Data

Table 2

ANALYSIS* OF DRUMS USED FOR BLENDING LOT 001 AND LOT 002

	Values in Percent				
Lot 001	ϕ	O-	P	M	Di-
B1-14	3.0	95.9	0.7	0.3	0.02
B1-15	0.66	92.2	4.8	2.1	0.02
B1-16	0.15	61.3	27.9	9.1	0.2
B2-14	0.39	99.1	0.2	0.1	0
B2-15	0.11	99.3	0.1	0.3	0
B2-17	0.06	6.4	81.4	10.8	1.2
B3-12	0.46	98.8	0.3	0.1	0
B3-14	0.05	99.5	0.2	0.1	0
B3-15	0.04	99.5	0.2	0.1	0
B4-13	0.34	98.5	0.9	0.2	0
B4-14	0.72	96.4	2.6	0.8	0.1
Analysis of Blend	0.48	86.8	8.6	3.4	0.6
Lot 002					
B2-16	0.07	88.5	6.9	4.2	0.2
B3-13	0.11	99.5	0.2	0.05	0
B3-16	0.02	73.3	16.8	9.3	0.6
B4-12	1.2	98.6	0.2	0	0
B4-15	0.47	95.4	1.3	3.1	0.1
B5-12	1.57	98.1	0.2	0.1	
B5-13	0.19	99.7	0.1	0.03	
B5-14	0.06	98.8	0.7	0.3	0.0
B5-15	0.07	74.7	16.0	8.5	0.6
Analysis of Blend	0.44	91.0	5.5	3.0	0.1

* Monsanto-St. Louis Data

BATCH 2

ANALYST MRC

um No.	Wt.	%	% Q	% O	% P+m	% M	% Di.	L/D	Cum. %
1	400		99+					4/1	
2	↓		↓					↓	
3	↓		↓					↓	
4	450		↓					↓	
5	↓		↓					↓	
6	↓		88.9	11.1				↓	
7	↓		84.4	15.6				↓	
8	↓		86.4	13.6				↓	
9	↓		48.5	49.5	2.0			↓	RECYCLED BATCH #3
10	↓		73.7	25.8	0.5			8/1	
11	↓		53.0	46.6	0.4			↓	RECYCLED BATCH 3
12	↓		35.3	63.7	1.0			↓	
13	↓		14.6	83.5	1.9			↓	RECYCLED 3
14	445		3.2	95.6	1.2			↓	USED IN LOT 001
15	450		0.8	89.2	10.1			6/1	" " "
16	↓		0.1	60.4	39.5			6/1	" " "
17	↓		0.1	32.1	62.6		5.2	8/1	
18	↓		0.04	9.2	81.3		9.4	↓	
19	↓		0.06	1.8	84.2		13.9	↓	
20	↓		0.08	0.4	85.3		14.2	↓	
	8845								

BATCH 6

ANALYST MRC

Sum No.	Wt.	%	% Q	% O	% P+M	% M	% Di.	I/D	Cum. %
1	450		99+					4/1	
2									
3									
4									
5								6/1	
6			99					8/1	
7			98.9	1.1	0.2				
8			83.4	16.4	0.2				
9			70.5	29.4	1.1			10/1	
10			89.3	10.4	0.3			10/1	
11			33.6	64.8	1.0			16/1	[redacted]
12			9.0	90.8	0.2				[redacted]
13			2.7	97.3	0.1				RECYCLED BATCH #3
14			0.4	99.9	0.1				USED IN LOT 002
15			0.2	98.6	1.2				" " "
16			0.2	75.9	23.4		0.7		USE IN LOT 002
17			0.03	20.0	74.3		5.7		USE IN LOT 001
18	235		0.1	0.8	93.5		6.5		
	7885								

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BATCH 3

ANALYST MRC

um No.	Wt.	%	% Q	% O	% P+M	% M	% DI.	L/D	Cum. %	
1	450							4/1		
2	↓							4/1		
3								4/1		
4			98.9	1.1				6/1		RECYCLED DRUMS
5			84.6	13.7	1.7			8/1		B1-9, B1-11 & B1-12
6	466		90.1	8.4	1.5			↓		
7	450		88.3	11.1				↓		RECYCLED DRUMS
8	↓		55	45				10/1		B1-13, B2-12 & B2-13
9			32.2	66.8				↓		[redacted]
10			7.9	89.4	2.7			↓		
11			3.4	96	0.6			16/1		
12			0.4	99	0.6			↓		USED IN Lot 001
13			0.06	99.5	0.5			↓		USED IN LOT 002
14				100	T			↓		USED IN LOT 001
15				100				↓		" "
16	↓			76.2	23.8			↓		USED IN LOT 002
17				4.7	90.8		4.5	↓		
18	270							↓		
	7936									

BATCH 4

ANALYST MRC

Run No.	Wt.	%	% Q	% O	% P+m	% M	% Di.	L/D	Cum. %	
1	450							4/1		
2										
3										
4										
5										
6			96	4.0	0.03		0	8/1		
7			91.8	8.2	0.06		0			RECYCLED DRUMS
8			79.6	20.0	0.04		0			B3-8, B3-9 & B3-10
9			79.7	20.0	0.3		0	10/1		
10			34.1	64.8	1.1		0	10/1		
11			10.3	89.7	0.03		0	16/1		
12			1.2	98.6	0.2		0			USED IN LOT 002
13			0.2	99.1	0.7		0			USED IN LOT 001
14			0	97.4	2.6					" "
15			0.05	95.9	4.1					USED IN LOT 002
16			0.01	28.2	68.6		3.2			
17			0.29	83.4	15.2		0.64			
	7650									

BATCH 5

ANALYST MRC

um No.	Wt.	%	% Q	% O	% P+M	% M	% Di.	L/D	Cum. %
1	450							4/1	
2	↓							↓	
3									
4								↓	
5								8/1	
6								↓	
7									RECYCLED DRUMS
8								↓	B4-10 & B4-11
9				53.9	46.1			10/1	
10				23.4	76.4	0.2		16/1	
11				10.6	89.3	0.1		16/1	
12				1.1	98.7	0.2		↓	USED IN LOT 002
13				0.1	99.9	T			" " "
14				T	99.2	0.8			" "
15				0.08	77.1	22.3		0.6	" "
16	↓			T	8.0	87.6		4.4	↓
	7200								

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