ANALYTICAL RESEARCH AND DEVELOPMENT REPORT No. 2565, October 10, 1974

OTHER LOCATIONS	GENERAL OFFICE	W. G. KRUMMRICH PLANT
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TLE: Dept. 246 - Monochlorobiphenyl Profile of Chlorinators During HCS 1111 Production.

RSONNEL:

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OBLEM:

Determine composition of the chlorination mass at the various chlorinators during HCS 1111 production.

STIFICATION:

Aid in studying the chlorination of biphenyl and to determine the best distribution of chlorine feed to each of the chlorinators.

MMARY:

Each of the four chlorinators were sampled at two different rates. The composition of the chlorinator mass was determined by gas chromatography. The HCl storage was also sampled shortly after the start of the chlorination and at the 90% level. The analytical data and analytical procedures are attached.

Reference - HCK Notebook 1S929-30.

L. H. Horcher

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Details

We washed 5 to 10 ml of the sample with 2-10 ml portion of distilled water to remove any HCl. One gram of the washed sample was taken up in 10 ml of benzene. A half a microliter of the solution was analyzed by GC using a flame detector and a 50ft. X .02 inch ID FFAP SCOT column at 200°C isothermal. The values were calculated by the area normalization with factors technique. Our results are shown below.

Date	9/20	9/20	9/20	9/20	9/20	9/2
Chlorinator	6	6	7	7	8	8
Rate,%	55	70	55	70	55	70
Sp.Gr.100°C/15.5°C	0.986	0.980	1.010	0.996	1.016	1.020
Time	12 Mid	12 am	12 Mid	12 am	12 Mid	12 [illegible]
BiØ	88.03	88.53	75.91	75.64	66.45	63.[illegible]
3 MeBiØ	0.05	0.04	0.04	0.04	0.03	0.03
4 MeBiØ	0.01	0.01	0.01	0.01	0.01	-
o-ClBiØ	6.84	6.51	13.38	13.57	18.36	19.[illegible]
mCl BiØ	0.75	0.73	1.46	1.49	2.00	2.12
p ClBiØ	3.76	3.65	7.43	7.42	10.00	10.[illegible]
2,2'DiClBiØ	0.13	0.13	0.43	0.44	0.77	0.[illegible]
Other Di's	0.42	0.41	1.36	1.40	2.39	2.[illegible]4
Tri's or higher	-	-	-	-	-	0.05

Date	9/20	9/20	9/21 (1)	9/21 (1)
Chlorinator	9	9	(2)	(3)
Rate	55%	70%	70%	70%
Sp.Gr.100°C/15.5°C	1.041	1.044	-	-
Time	12 Mid	12 am	12.40 pm	[illegible]
BiØ	50.02	48.41	59.46	41.08
3 MeBiØ	0.02	0.02	0.04	0.05
4 MeBiØ	-	-		
o ClBiØ	25.73	26.28	16.84	16.60
m ClBiØ	2.75	2.86	1.64	1.25
p ClBiØ	13.82	14.16	8.51	6.81
2,2'DiClBiØ	1.85	1.95	1.64	2.99
Other Di's	5.74	6.27	3.54	6.02
Tri's or higher	0.08	0.10	8.02	24.62

(1) these values are area normalization values but this should not make much difference because the factors are fairly close relative to the dichlorobiphenyls

(2) HCl stg. after rising from 17 to 56% ← this sample

(3) HCl stg at 90% level at pumpout ← [illegible]

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