Analyses

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

C. Discussion

1. Fractionation

The laboratory fractionations of samples of crude biphenyl concentrates using several different fractionating columns are summarized in Table No. 10. 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).

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 therefrom. 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. 11. 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

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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.

Sample	Org. 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
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 unidentified		0.4	--	trace	trace
m-terphenyl		0.5 (0.71)	0.4 (0.7)	1.1 (1.3)	1.1 (1.
p-terphenyl		0.4 (0.29)	0.4 (0.30)	0.7 (0.72)	0.8 (0.
t unidentified minor comp.					
"Heavys"		0.58	0.96		

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[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. Overall, however, these results appear to give good agreement considering the difference in techniques and lack of refinements in both techniques."

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