WGK ANALYTICAL RESEARCH AND DEVELOPMENT REPORT No. 2457, May 16, 1974

OTHER LOCATIONS	GENERAL OFFICE	W. G. KRUMMRICH PLANT
T. Bell - Anniston T. Brown - Anniston	J. R. Savage - B2SL (Copies sent to R.Munch, W.R.Richard - G.O. and V.R. Haupt - Anniston 5/28/74)	W. C. Engman A. E. Leisy K. E. Boucher G. Johnson G. A. Hippe J. R. Kuster File

TITLE:

Dept. 246 - Aroclors. Biphenyl Quality

PERSONNEL:

L. H. Horcher (H. J. Horner)

PROBLEM:

Develop semi-routine method for determining methyl biphenyls in biphenyl. Analyze samples of biphenyl made from 'good' and high toluene benzene.

JUSTIFICATION:

The quality of benzene used for biphenyl production at Anniston may be of lower quality and could increase the methyl biphenyl level in the biphenyl. Methyl biphenyls are thought to affect stability of the Aroclors.

SUMMARY:

We developed a procedure using a Varian Model 1440 FID GC to analyze the biphenyl for the various impurities. We can detect impurities down to 10 ppm without any serious problems. Biphenyl samples produced with 'good' benzene and material made during the Anniston 'Toluene Test' (500 ppm) run were analyzed. Results of these samples, as well as chromatograms of a standard containing the probable impurities in biphenyl and of a typical sample are attached. The biphenyl produced during the 'Toluene Test' showed an increase in the methyl biphenyl by 1.5 to 3.5 times that in normal production.

Ref. WGK Notebook 16380-16393.
18246-18260.

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Details

I. Background

Past work has indicated that high methyl biphenyl levels in biphenyl cause stability problems in Aroclors. Benzene shortages may force our Anniston Plant to use lower quality benzene (higher in toluenes). A procedure is necessary to determine methyl biphenyls and other pertinent impurities in biphenyl. Recently Anniston made a production run using benzene containing 500 ppm toluene. We have received two samples taken from their biphenyl receivers, as well as samples of two cars loaded from the storage tank containing some of the biphenyl made during this period. These samples and several reserve samples from past production are to be analyzed to determine the change if any, in methyl biphenyl levels.

II. Method Development

In January of 1972 we did some analytical work on biphenyl quality. Based on this work we set up an instrument with conditions similar to those used at that time. A standard containing the major impurities found at that time was made up to check and optimize instrumental conditions. Oven temperatures, flowrates, split ratios and sample concentrations were varied until we felt we had good resolution of the pertinent components. A shortened version of the original column combination was sufficient.

Since the analysis requires that we analyze only the minor components of a sample that is essentially 99+% biphenyl, we employed the internal standard technique. Normal C-12 was chosen as the internal standard since it elutes before the components of interest and is well clear of the solvent tail. None of the reagents with the exception of specially purified napthalene and biphenyl were pure. The reagents were all assayed to enable us to prepare a reasonably accurate calibration standard. The area correction factors vs n C_{12} of naphthalene, 2-, 3-, and 4-methyl biphenyl were determined. A factor for 2,3 dimethylnapthalene was also determined and used for all the mono and dimethylnapthalenes. A standard containing the probable impurities in biphenyl was run and a chromatogram is attached.

III. Analytical Work

The samples were taken up in benzene (solvent) containing 503 ppm n C12 (internal standard). The sample solvent concentration was 1:1 on a w/w basis. Four samples of biphenyl made during the last quarter of 1973, the two Anniston Biphenyl receiver samples and the two cars loaded after the recent 'Toluene Test' run were analyzed in this manner. The results and a typical chromatogram are attached.

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IV. Conclusions

In all the samples from the Toluene Test the methyl biphenyl levels were higher than that produced in the last quarter of 1973.

The receiver samples were more than 3.5 times higher and the car samples which were diluted by the good biphenyl in storage were up by 1.5 times.

The impurity labeled as Unknown (1) which we tentatively identified by retention time as diphenyl methane increased somewhat but the increase was not as significant as the methyl biphenyls.

We can analyze biphenyl for methyl biphenyl on a spot basis. The instrument and columns are not available on a daily routine basis. We also need to determine the area correction factors more rigorously if the procedure is to be used routinely.

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(1) This unknown tentatively identified by retention time as diphenyl methane.

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RESULTS

Biphenyl Quality, Last Quarter 1973

Lot No.	AC-168	AC-156	AC-183	GATX 94513 (2)
Date	11/5/73	10/4/73	12/8/73	11/27/73
Napthalene	39 ppm	12 ppm	14 ppm	19 ppm
Unknowns	36	7	29	5
2 Methyl Mapthalene	-	4	5	9
1 Methyl Napthalene	8	-	4	14
2 Methyl Biphenyl	121	84	157	260
Unknown (1)	430	394	537	776
Di Methyl Napthalenes	18	-	-	23
3 Methyl Biphenyl	904	474	948	821
4 Methyl Biphenyl	179	63	120	150
Unknown (High Boilers)	-	78	-	-
Total Impurities	1735	1116	1814	2077
Total Methyl Biphenyls	1204	621	1225	1231

Biphenyl Quality - 'Toluene Test'

Lot No.	GATX 93194	GATX 94529	Anniston Receivers	
Date	5/3/74 (2)	5/8/74 (2)	4/19/74(2)	4/26/74
Napthalene	14 ppm	10 ppm	16 ppm	17 ppm
Unknowns	-	4	-	120
2 Methyl Napthalene	-	-	2	-
1 Methyl Napthalene	23	29	12	11
2 Methyl Biphenyl	208	211	173	209
Unknown (1)	656	792	799	816
Di Methyl Napthalenes	10	5	-	-
3 Methyl Biphenyl	1364	1587	2623	2599
4 Methyl Biphenyl	249	307	554	545
Unknown(High Boilers)	62	6	7	-
Total Impurities	2586	2951	4184	4317
Total Methyl Biphenyls	1821	2105	3350	3353

(1) This unknown tentatively identified by retention time as diphenyl methane.

(2) These results are the average of duplicate runs.

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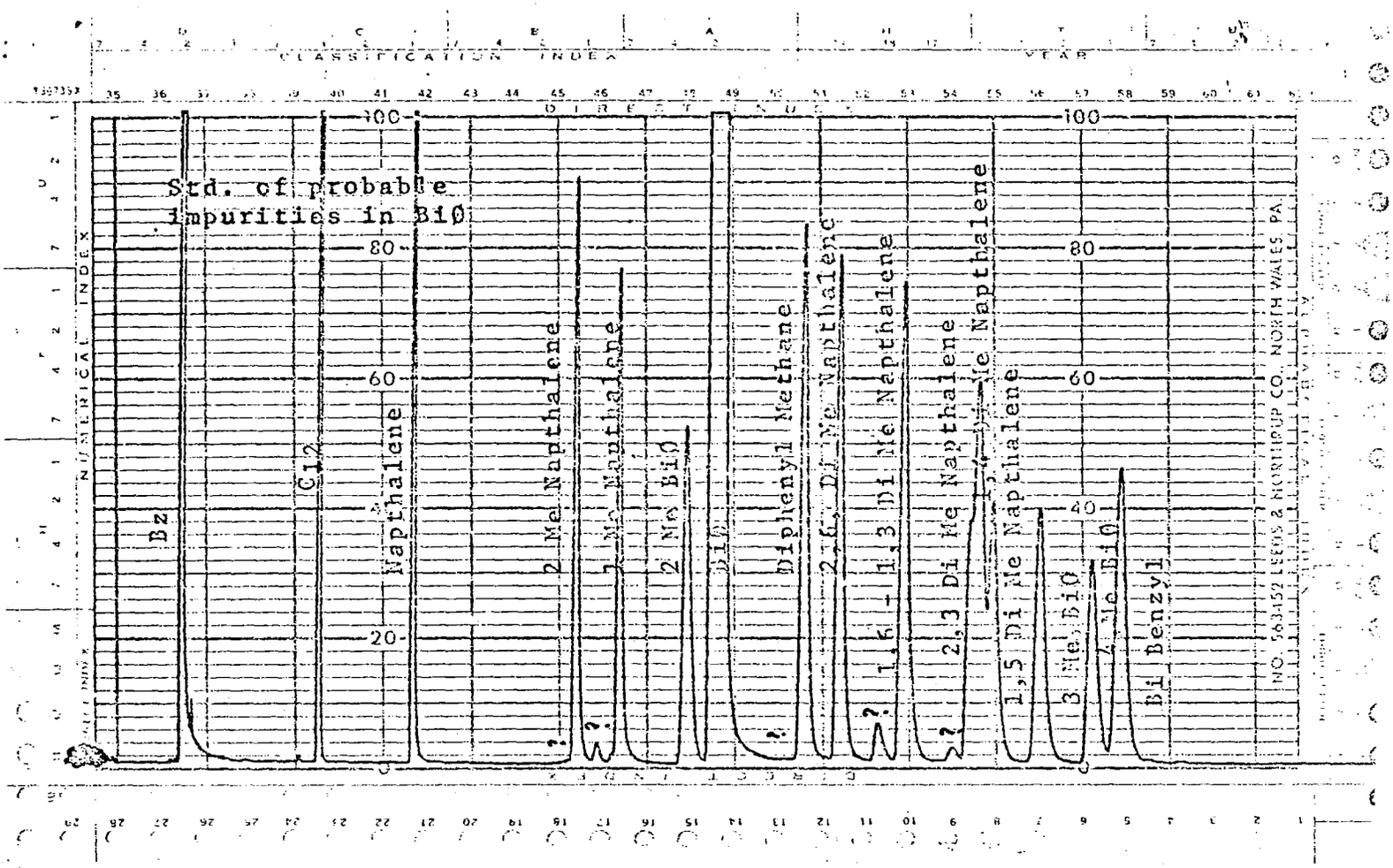

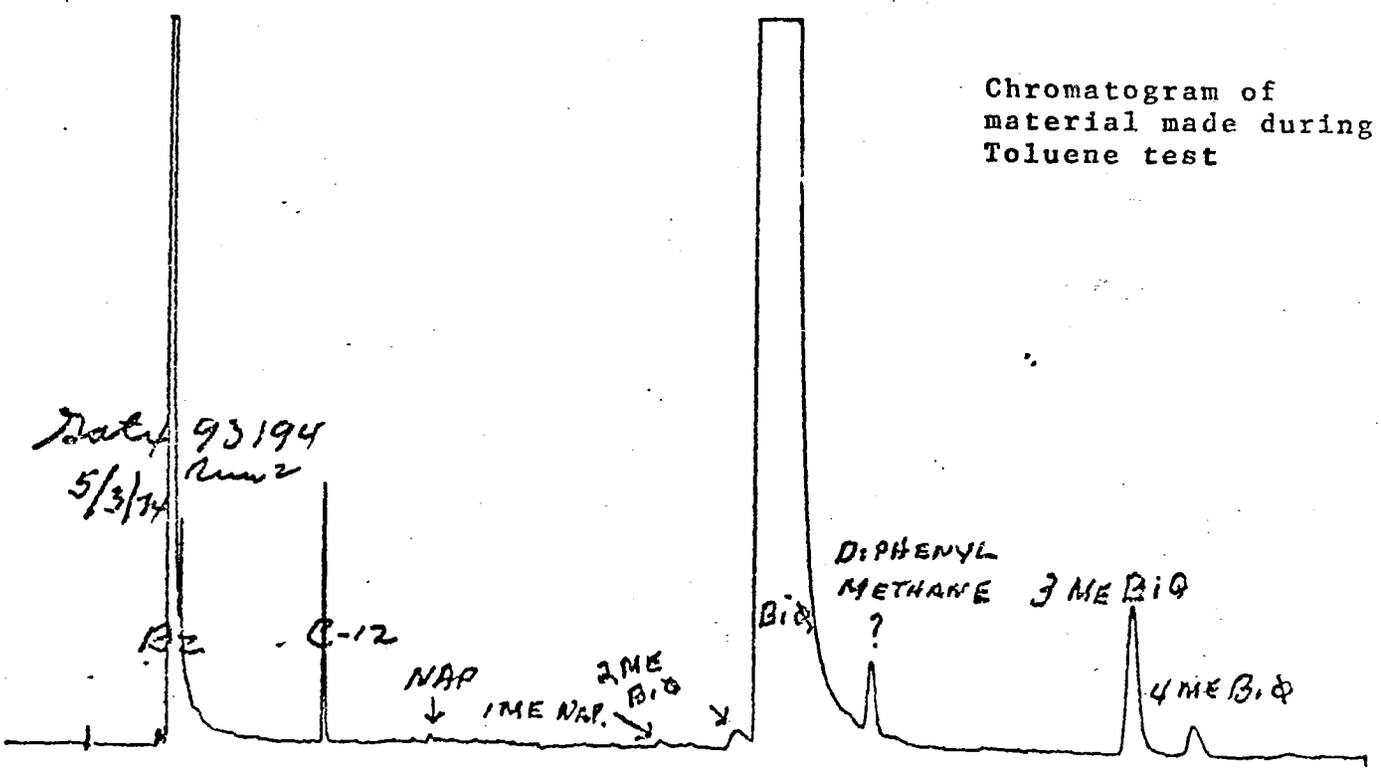

Chromatogram of material made during Toluene test

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