Physical Chemistry
Method No. 71-11
Job No. 1348522

GAS CHROMATOGRAPHIC DETERMINATION OF PENTA- AND HEXA-CHLOROBIPHENYLS IN AROCLOR 1242 AND MCS 1016

PURPOSE OF ANALYSIS

This is a rapid, simplified method to be used to determine the level of high boilers (mainly pentachlorobiphenyls) in MCS 1016 obtained from the fractionation of Aroclor 1142. It is not intended for general analysis of chlorinated biphenyl mixtures. Physical Chemistry Method No. 70-20 should be used for that purpose.

CHROMATOGRAPHIC CONDITIONS

Instrument: Perkin-Elmer 800, Hewlett-Packard 7624A or Equivalent

Type Detector: Flame Ionization

Column: 50' X 0.02" I.D. FFAP S.C.O.T. (Support-Coated Open Tubular) Capillary (Perkin-Elmer Part No. 008-0804)

		Flow Rates	
Column Temperature:	220°C	Column Helium:	8 ml/min (7 PSIG)
Detector Block Temperature:	300°C	Inlet Splitter:	210 ml/min
Injection Port Temperature:	320°C	Auxiliary Helium:	50 ml/min
Sample Volume Injected:	0.005 ml Split 25 to 1	Hydrogen:	50 ml/min
		Air:	400 ml/min

RETENTION TIMES (UNCORRECTED) OF COMPONENTS SEPARATED (CHROMATOGRAMS ATTACHED)

Biphenyl, Mono-, Di-, Tri- and Most Tetra-Chlorobiphenyls (1.0 to 5.0 min); High Boilers (Mostly Penta- and Hexa-Chlorobiphenyls) (5.5 to 11.2 min).

CALCULATION TECHNIQUE

Peak area normalization. The lack of pure knowns prevents the accurate determination of response factors (area correction factors) for the individual components. Therefore, the specification for MCS 1016 is simply based on area percent of total high boilers.

PRECISION

Reference samples* of MCS 1016 (Drum 2) and Aroclor 1242 (Lot KK 491) were analyzed in duplicate on the two instruments listed using both the Disc chart integrator and an electronic integrator (Infotronics CRS-100 on the Perkin-Elmer 800 chromatograph and Hewlett-Packard 3370A on the Hewlett-Packard 7624A chromatograph). The same column was used in both instruments. Results reported as Area % High Boilers:

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MCS 1016 (Drum 2):

	Method of Integration		
Chromatograph	Disc Chart	Infotronics CRS-100	Hewlett-Packard 3370A
Perkin-Elmer 800	0.30, 0.27	0.23, 0.21	-
Hewlett-Packard 7624A	0.30, 0.30	-	0.27, 0.28
Aroclor 1242 (Lot KK 491):			
Perkin Elmer 800	5.88, 5.78	6.22, 5.94	-
Hewlett-Packard 7624A	6.14, 6.32	-	6.63, 6.50

*Portions of these samples are available to those Monsanto laboratories that are setting up this method for control of MCS 1016 manufacture.

db

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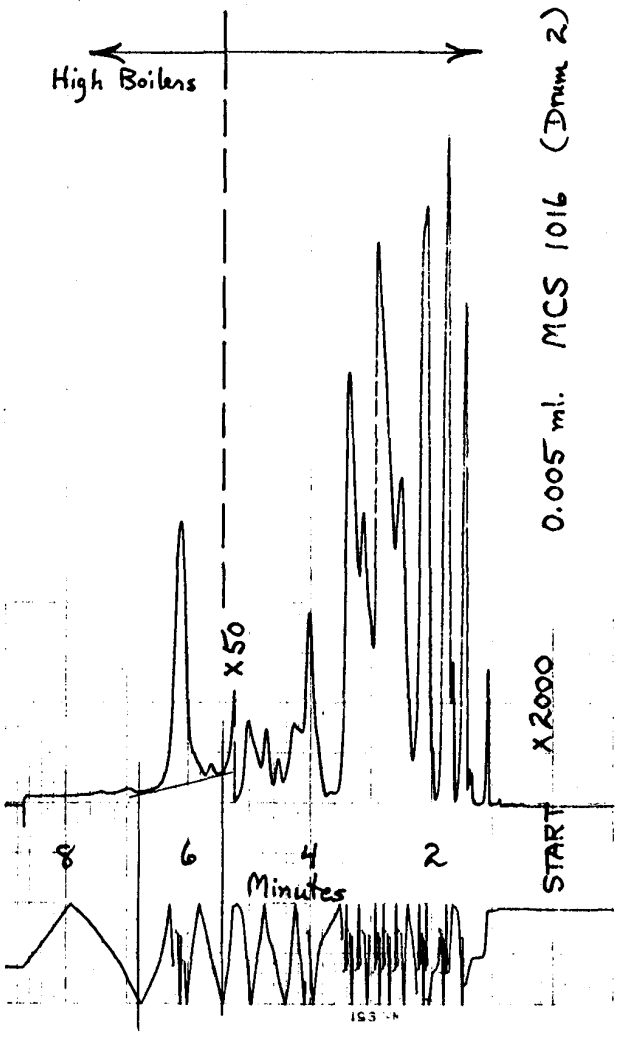
Perkin-Elmer 800



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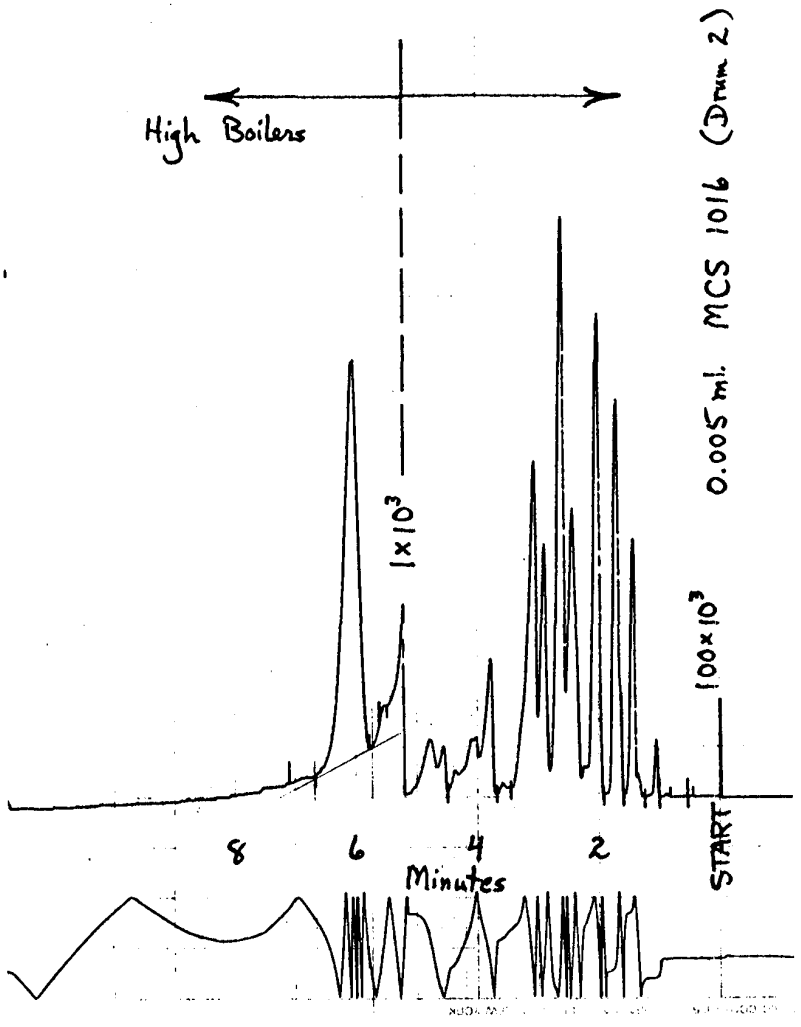
Hewlett-Packard 7624A



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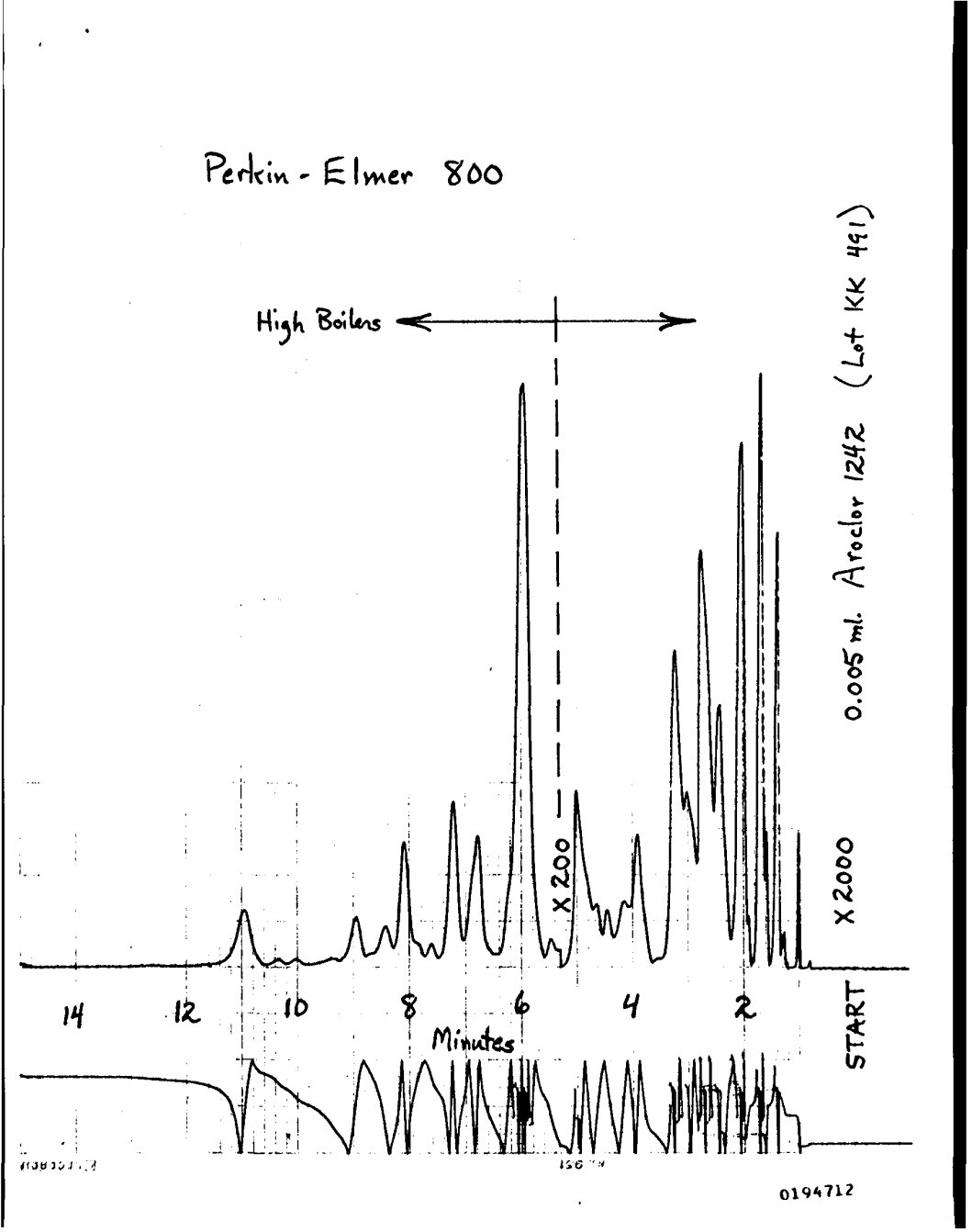

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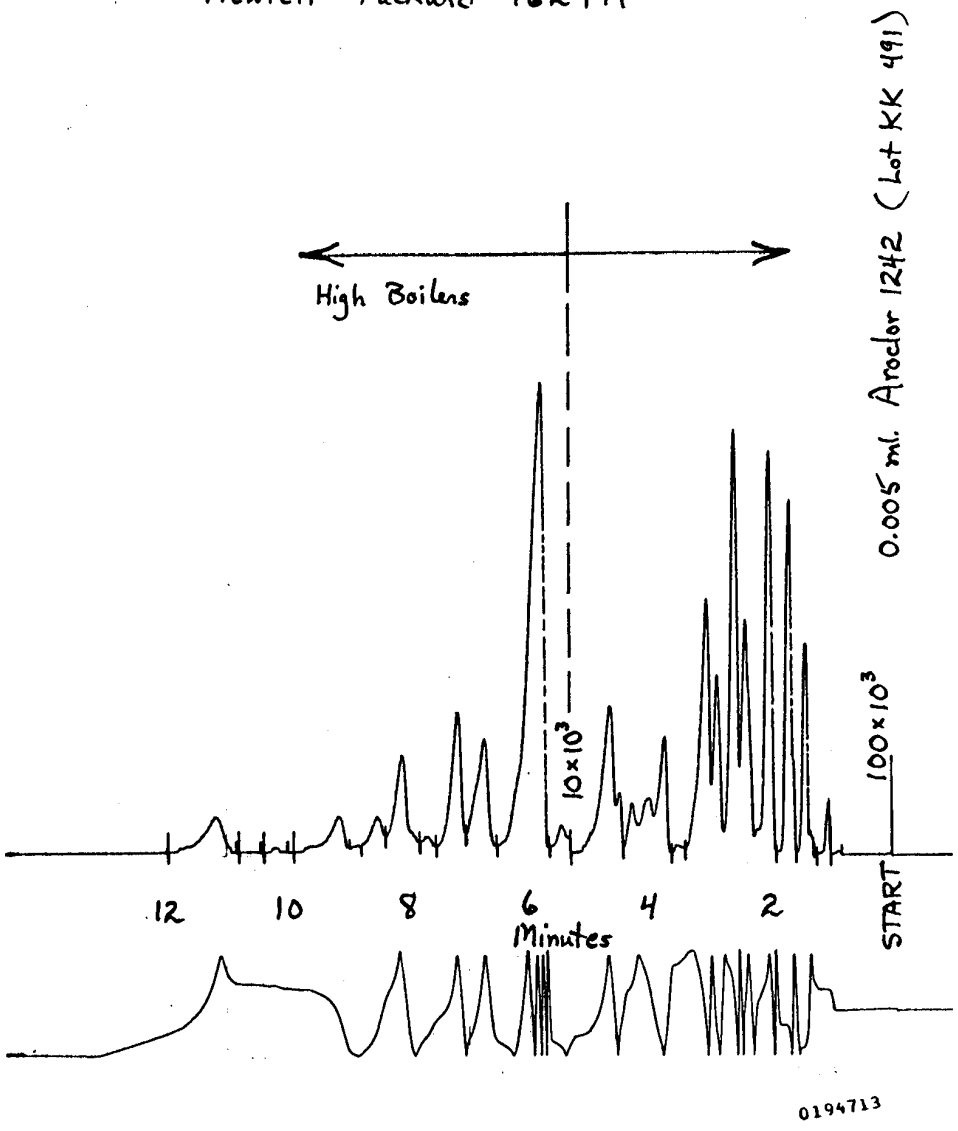
Hewlett-Packard 7624A



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