EPA-600/2-81-055b
April 1981

EVALUATION OF PCB DESTRUCTION EFFICIENCY IN AN INDUSTRIAL BOILER: AUDIT REPORT

by
P. F. Collins and G. F. Hunt

Research Triangle Institute
P.O. Box 12194
Research Triangle Park, NC 27709

EPA Contract No. 68-02-3146
Task No. 129

EPA Task Officer
D. Sanchez

Industrial Environmental Research Laboratory
Office of Environmental Engineering and Technology
Research Triangle Park, North Carolina 27711

Prepared for

U.S. ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF RESEARCH AND DEVELOPMENT
WASHINGTON, D.C. 20460

ABSTRACT

Systems audits and an evaluation of the quality of data obtained by GM and GCA in the analysis of a test burn oil for PCB were conducted by two members of the Environmental Quality Assurance Department of the Research Triangle Institute during the week of August 18, 1980. Audits were conducted by inspection of available documentation and records, discussion of analytical methodology and data with personnel of the organization being audited, and independent data reduction. The analytical data reported by GM and GCA were subsequently confirmed by separate analyses performed by the Analytical Chemistry Branch of EPA/HERL-RTP. The results are reported in Appendix A.

This report was submitted in fulfillment of Task Directive No. 129 on EPA Contract No. 68-02-3146 by the Research Triangle Institute. This report covers the period August 6, 1980, to September 30, 1980, and work was completed as of October 1980.

CONTENTS

FIGURES

TABLES

SECTION 1
INTRODUCTION

In May 1980, a verification test burn for destruction of polychlorinated biphenyls (PCB), was conducted to evaluate the PCB destruction efficiency of the No. 3 industrial boiler at the Chevrolet plant in Bay City, Michigan. The verification burn was conducted by General Motors Corporation, with GCA Corporation serving as a contractor to the U.S. Environmental Protection Agency (USEPA) to provide and operate the flue gas and ambient air sampling equipment and to perform the necessary analytical work.

The verification burn fired a reclaimed oil that had been prepared (spiked) by GM to contain approximately 500 ppm PCB as Aroclor 1242. Analysis of this test burn oil for PCB was conducted by both GM and GCA, and the results of these analyses were in questionable agreement. Consequently, the Research Triangle Institute (RTI) was requested by USEPA to perform systems audits and to establish the quality of data obtained by GM and GCA in the analysis of the test burn oil for PCB.

During the week of August 18, 1980, two auditors from the Environmental Quality Assurance Department of RTI visited the Central Office, Chevrolet Motor Division, General Motors Corporation, Warren, Michigan, and the Technology Division of GCA Corporation in Bedford, Massachusetts, to perform the systems audits. This report includes a description of the audit procedures used, the audit results, and conclusions and recommendations.

Subsequent to RTI's systems audit and in response to the audit's conclusions and recommendations, analyses of the PCB reference standard material and the split samples of the test burn oil were performed by the Applied Chemistry Branch of the Health Effects Research Laboratory, EPA-RTP. The results of these analyses are presented in Appendix A.

The assistance and cooperation of the personnel at GM and GCA are acknowledged with appreciation.

SECTION 2
SUMMARY

Systems audits and an evaluation of the quality of data obtained by GM and GCA in the analysis of a test burn oil for PCB were conducted by two members of the Environmental Quality Assurance Department of the Research Triangle Institute during the week of August 18, 1980. Audits were conducted by inspection of available documentation and records, discussion of analytical methodology and data with personnel of the organization being audited, and independent data reduction. A checklist was used as a guide in conducting the audits.

An examination of the available documentation regarding sampling and sample custody indicated no records were available at either facility for the test burn oil sample from the time it was taken at the Bay City plant until it was received in the laboratories.

The analytical results obtained by GM and GCA are summarized in Table 1, along with the Aroclor 1242 used for calibration or as a reference standard for each quantitation. In some cases, the analytical result was calculated by the RTI auditors, using data supplied by the organizations. It was concluded that the analytical methodology used at each organization is acceptable and is not primarily responsible for the interlaboratory difference in the results. Rather, the differences are most likely attributable to the different Aroclor 1242 materials used for calibration or as reference standards. This can only be confirmed by examination by one laboratory of all the Aroclor 1242 reference materials used in the analyses at GM and GCA.

Subsequent to the audit, the reference materials and sample of test burn oil from each organization were reanalyzed by EPA/HERL-RTP, as described in Appendix A. Those results for the test burn oil were intermediate between results obtained by GM and GCA, suggesting that the difference between results by GM and GCA is due to analytical variability, which is often encountered in the analysis of a complex mixture such as Aroclor 1242.

PB81-187270

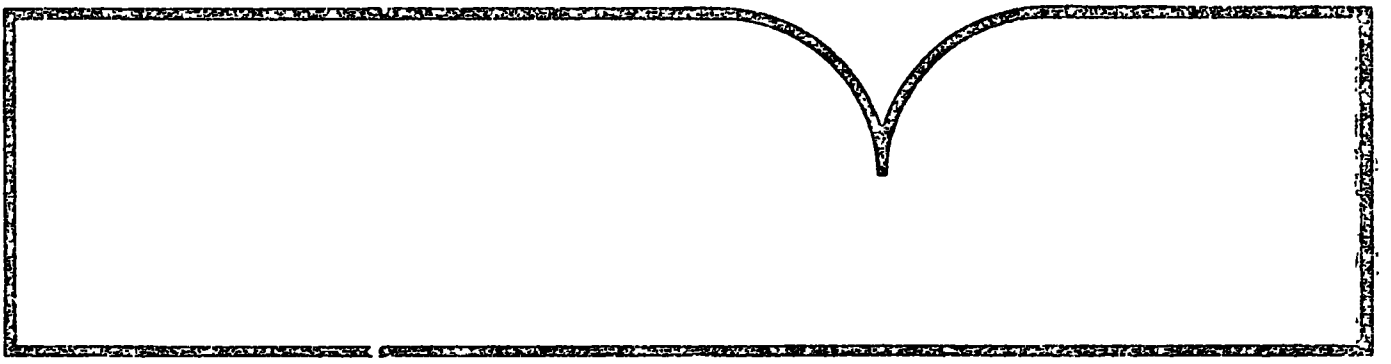
Evaluation of PCB Destruction Efficiency in an Industrial Boiler: Audit Report

Research Triangle Inst.
Research Triangle Park, NC

Prepared for

Industrial Environmental Research Lab.
Research Triangle Park, NC

Apr 81



U.S. Department of Commerce
National Technical Information Service
NTIS

United States
Environmental Protection
Agency

EPA-600/2-81-055b

April 1981

EPA

Research and Development

EVALUATION OF PCB DESTRUCTION EFFICIENCY IN AN INDUSTRIAL BOILER: AUDIT REPORT

Prepared for

Office of Pesticides and Toxic Substances

Prepared by

Industrial Environmental Research
Laboratory
Research Triangle Park NC 27711

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1 REPORT NO EPA-600/2-81-055b	2	3 RECIPIENT'S ACCESSION NO
4 TITLE AND SUBTITLE Evaluation of PCB Destruction Efficiency in an Industrial Boiler: Audit Report		5 REPORT DATE April 1981 6 PERFORMING ORGANIZATION CODE
7 AUTHOR(S) P.F. Collins and G.F. Hunt		8 PERFORMING ORGANIZATION REPORT NO
9 PERFORMING ORGANIZATION NAME AND ADDRESS Research Triangle Institute P.O. Box 12194 Research Triangle Park, North Carolina 27709		10 PROGRAM ELEMENT NO C1YLIB 11 CONTRACT/GRANT NO 68-02-3146, Task 129
12 SPONSORING AGENCY NAME AND ADDRESS EPA, Office of Research and Development Industrial Environmental Research Laboratory Research Triangle Park, NC 27711		13 TYPE OF REPORT AND PERIOD COVERED Task Final; 8-9/80 14 SPONSORING AGENCY CODE EPA/600/13

15 SUPPLEMENTARY NOTES IERL-RTP project officer is David C. Sanchez, Mail Drop 62, 919/541-2547.

16 ABSTRACT The report gives results of systems audits and an evaluation of the quality of data obtained by GM and GCA in the analysis of a test burn oil for PCB conducted by Research Triangle Institute. Audits included inspection of documentation and records, discussion of analytical methodology and data with personnel of the organization being audited, and independent data reduction. The analytical data reported by GM and GCA were subsequently confirmed by separate analyses by EPA's Health Effects Research Laboratory (RTP) and are reported in Appendix A.

17 KEY WORDS AND DOCUMENT ANALYSIS

a DESCRIPTORS	b IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Pollution	Pollution Control	13B
Chlorine Aromatic Compounds	Stationary Sources	07C
Biphenyl	Polychlorinated Biphenyls	21D,11H
Fuel Oil		21B
Combustion		05A
Auditing		14B
Analyzing		

18 DISTRIBUTION STATEMENT Release to Public	19 SECURITY CLASS (This Report) Unclassified 20 SECURITY CLASS (This page) Unclassified	21 NO OF PAGES 32 22 PRICE

EPA Form 2220-1 (9-73)

1.b

RESEARCH REPORTING SERIES

Research reports of the Office of Research and Development, U S Environmental Protection Agency, have been grouped into nine series These nine broad categories were established to facilitate further development and application of environmental technology Elimination of traditional grouping was consciously planned to foster technology transfer and a maximum interface in related fields The nine series are:

1. Environmental Health Effects Research
2. Environmental Protection Technology
3. Ecological Research
4. Environmental Monitoring
5. Socioeconomic Environmental Studies
6. Scientific and Technical Assessment Reports (STAR)
7. Interagency Energy-Environment Research and Development
8. "Special" Reports
9. Miscellaneous Reports

This report has been assigned to the ENVIRONMENTAL PROTECTION TECHNOLOGY series This series describes research performed to develop and demonstrate instrumentation, equipment, and methodology to repair or prevent environmental degradation from point and non-point sources of pollution This work provides the new or improved technology required for the control and treatment of pollution sources to meet environmental quality standards.

EPA REVIEW NOTICE

This report has been reviewed by the U S. Environmental Protection Agency, and approved for publication. Approval does not signify that the contents necessarily reflect the views and policy of the Agency, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.

This document is available to the public through the National Technical Information Service, Springfield, Virginia 22161.

EPA-600/2-81-055b
April 1981

EVALUATION OF PCB DESTRUCTION EFFICIENCY IN AN INDUSTRIAL BOILER: AUDIT REPORT

by
P. F. Collins and G. F. Hunt

Research Triangle Institute
P.O. Box 12194
Research Triangle Park, NC 27709

EPA Contract No. 68-02-3146
Task No. 129

EPA Task Officer
D. Sanchez
Industrial Environmental Research Laboratory
Office of Environmental Engineering and Technology
Research Triangle Park, North Carolina 27711

Prepared for

U.S. ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF RESEARCH AND DEVELOPMENT
WASHINGTON, D.C. 20460

ABSTRACT

Systems audits and an evaluation of the quality of data obtained by GM and GCA in the analysis of a test burn oil for PCB were conducted by two members of the Environmental Quality Assurance Department of the Research Triangle Institute during the week of August 18, 1980. Audits were conducted by inspection of available documentation and records, discussion of analytical methodology and data with personnel of the organization being audited, and independent data reduction The analytical data reported by GM and GCA were subsequently confirmed by separate analyses performed by the Analytical Chemistry Branch of EPA/HERL-RTP. The results are reported in Appendix A.

This report was submitted in fulfillment of Task Directive No. 129 on EPA Contract No. 68-02-3146 by the Research Triangle Institute. This report covers the period August 6, 1980, to September 30, 1980, and work was completed as of October 1980.

CONTENTS

FIGURES

TABLES

SECTION 1
INTRODUCTION

In May 1980, a verification test burn for destruction of polychlorinated biphenyls (PCB), was conducted to evaluate the PCB destruction efficiency of the No. 3 industrial boiler at the Chevrolet plant in Bay City, Michigan. The verification burn was conducted by General Motors Corporation, with GCA Corporation serving as a contractor to the U.S. Environmental Protection Agency (USEPA) to provide and operate the flue gas and ambient air sampling equipment and to perform the necessary analytical work.

The verification burn fired a reclaimed oil that had been prepared (spiked) by GM to contain approximately 500 ppm PCB as Aroclor 1242. Analysis of this test burn oil for PCB was conducted by both GM and GCA, and the results of these analyses were in questionable agreement. Consequently, the Research Triangle Institute (RTI) was requested by USEPA to perform systems audits and to establish the quality of data obtained by GM and GCA in the analysis of the test burn oil for PCB.

During the week of August 18, 1980, two auditors from the Environmental Quality Assurance Department of RTI visited the Central Office, Chevrolet Motor Division, General Motors Corporation, Warren, Michigan, and the Technology Division of GCA Corporation in Bedford, Massachusetts, to perform the systems audits. This report includes a description of the audit procedures used, the audit results, and conclusions and recommendations.

Subsequent to RTI's systems audit and in response to the audit's conclusions and recommendations, analyses of the PCB reference standard material and the split samples of the test burn oil were performed by the Applied Chemistry Branch of the Health Effects Research Laboratory, EPA-RTP. The results of these analyses are presented in Appendix A.

The assistance and cooperation of the personnel at GM and GCA are acknowledged with appreciation.

SECTION 2
SUMMARY

Systems audits and an evaluation of the quality of data obtained by GM and GCA in the analysis of a test burn oil for PCB were conducted by two members of the Environmental Quality Assurance Department of the Research Triangle Institute during the week of August 18, 1980. Audits were conducted by inspection of available documentation and records, discussion of analytical methodology and data with personnel of the organization being audited, and independent data reduction. A checklist was used as a guide in conducting the audits.

An examination of the available documentation regarding sampling and sample custody indicated no records were available at either facility for the test burn oil sample from the time it was taken at the Bay City plant until it was received in the laboratories.

The analytical results obtained by GM and GCA are summarized in Table 1, along with the Aroclor 1242 used for calibration or as a reference standard for each quantitation. In some cases, the analytical result was calculated by the RTI auditors, using data supplied by the organizations. It was concluded that the analytical methodology used at each organization is acceptable and is not primarily responsible for the interlaboratory difference in the results. Rather, the differences are most likely attributable to the different Aroclor 1242 materials used for calibration or as reference standards. This can only be confirmed by examination by one laboratory of all the Aroclor 1242 reference materials used in the analyses at GM and GCA.

Subsequent to the audit, the reference materials and sample of test burn oil from each organization were reanalyzed by EPA/HERL-RTP, as described in Appendix A. Those results for the test burn oil were intermediate between results obtained by GM and GCA, suggesting that the difference between results by GM and GCA is due to analytical variability, which is often encountered in the analysis of a complex mixture such as Aroclor 1242.

TABLE 1. SUMMARY OF PCB CONCENTRATIONS FOUND IN TEST BURN OIL BY GM AND GCA*†

Organization and analysis method	Aroclor 1242 reference material	Aroclor 1242 found, µg/g (ppm)
GM· GC/EC	Monsanto Aroclor 1242	496† (493)
GM· GC/MS	Monsanto Aroclor 1242	529, 539‡
GM: GC/MS, standard addition	Monsanto Aroclor 1242	510‡ (486)
GCA· GC/EC	Supelco C/N 4-4803	-- (471)
GCA: GC/EC	EPA/RTP Reference, Lots 8937, 8758 (Monsanto Aroclor 1242)	750 (738)
GCA: GC/EC	Applied Science, Lot #478	750 (731)
GCA: GC/EC	EPA/EMSL-Ci QC Samples, WP679, Conc. 7 and 8	-- (538-590)

*Following the audit, the sample of test burn oil that each organization had analyzed was reanalyzed for Aroclor 1242 by EPA/HERL-RTP, as described in Appendix A. Results were· GM sample, 614 ppm; and GCA sample, 640 ppm.

†Values in parentheses were calculated by RTI auditors from data supplied by the applicable organization.

‡Data provided by GM indicate that results would be 8 percent higher if referenced to EPA Reference Aroclor, Lot 5041, from EPA/HERL-RTP.

SECTION 3
AUDIT PROCEDURES

The procedures used for the audits of the analytical data obtained by GM and GCA for PCB in the test burn oil were designed to evaluate all aspects of the data gathering process, including sampling, sample custody, sample analysis, and data reduction. As a guide in conducting the audit, the checklists shown in Figure 1 (GM) and Figure 2 (GCA) were completed (as applicable). Each audit included inspection of the documentation, discussion of analytical methodology and data, and independent data reduction, which served to verify the calculations. Each analytical laboratory was also visited. At the conclusion of each audit, a debriefing session was held with representatives of the organization.

COMPOUND: Test Burn Oil
Plant/City/State: Central Offices, Chevrolet Motor Division (GM)
Sample Point Location: Unknown
Date Sampled: Unknown Date Analyzed: Unknown

		Yes	No
(1)	Was chain of custody procedure followed: No written records, hand carried to lab. sample control in hands of analyst after receipt in lab.	—	✓
(2)	Analysis Procedure:		
(a)	Were sample preparation steps described?	✓	—
(b)	Were instrument parameters documented? std. procedure - parameters remain same. Inst. dedicated to this analysis	✓	—
(c)	Was compound identified on chromatogram?	✓	—
(d)	How was data compiled?		
(1)	Manually?	✓	—
(2)	Electronically?	—	✓
(e)	How was data quantitated?		
(1)	Manually?	✓	—
(2)	Electronically?	—	✓
(f)	Did retention times of sample, standard and spiked sample for compound agree?	✓	—
(g)	Was compound analyzed by GC/MS?	✓	—
	If so, does data agree with GC data?	✓	—

Figure 1. Checklist used at GM.

		Yes	No
(3)	Standardization Procedures:		
	(a) Was purity of standard compound verified? From EPA - Ref. Std. - 1242 From Monsanto - 1242 (See note 2.	✓	—
	(b) Was a ~~five~~ three-point calibration curve prepared?	✓	—
	(c) Was instrument calibration checked daily?	✓	—
	If not, at what interval?		
	(d) Did calibration curve bracket sample concentration?	✓	—
(4)	Recording Procedures:		
	(a) Were data sheets and notebooks signed and dated?	—	✓
	(b) Were data entered properly?	—	✓
	(c) Were sample calculations shown with proper unit designations?	✓	—
	(1) Injection volume corrections made?	✓	—
	(2) Dilution and/or concentration corrections made?	✓	—
	(d) Was data reviewed?	—	✓
	If so, how frequently?	—	—

COMMENTS

Figure 1 (continued)

COMPOUND: Test Burn Oil
Plant/City/State: Technology Division of GCA Corporation
Sample Point Location: Unknown
Date Sampled: Unknown Date Analyzed: 6-12,13-80

		Yes	No
(1)	Was chain of custody procedure followed: After sample was received in lab.	✓	
(2)	Analysis Procedure:		
(a)	Were sample preparation steps described?	✓	
(b)	Were instrument parameters documented?	✓	
(c)	Was compound identified on chromatogram?	✓	
(d)	How was data compiled?		
	(1) Manually?		✓
	(2) Electronically?	✓	
(e)	How was data quantitated?		
	(1) Manually?	✓	
	(2) Electronically?		✓
(f)	Did retention times of sample, standard and spiked sample for compound agree?	✓	
(g)	Was compound analyzed by GC/MS?		✓
	If so, does data agree with GC data?		

Figure 2. Checklist used at GCA.

	Yes	No
(3) Standardization Procedures:		
(a) Was purity of standard compound verified? Checked against EPA ref. materials.	—	—
(b) Was a ~~five~~-point calibration curve prepared? three	✓	—
(c) Was instrument calibration checked daily? If not, at what interval?	✓	—
(d) Did calibration curve bracket sample concentration?	✓	—
(4) Recording Procedures:		
(a) Were data sheets and notebooks signed and dated?	✓	—
(b) Were data entered properly?	✓	—
(c) Were sample calculations shown with proper unit designations?	✓	—
(1) Injection volume corrections made?	✓	—
(2) Dilution and/or concentration corrections made?	✓	—
(d) Was data reviewed?	✓	—
If so, how frequently?	—	—

COMMENTS

Figure 2 (continued)

SECTION 4
AUDIT RESULTS

The results of the systems audits for data quality evaluation conducted at GM on August 18-19, 1980, and at GCA on August 20-21, 1980, are presented in this section.

4.1 AUDIT OF GM DATA

4.1.1 Sampling

Documented information concerning the sampling of the test burn oil was not available to the auditors. From discussions with GM and GCA personnel, it appears that the oil was sampled by GM-Bay City, with one portion of the sample given to the onsite GCA field crew and one portion transported to the GM-Warren Laboratory.

4.1.2 Sample Custody

Documentation concerning custody of the oil sample prior to receipt in the GM laboratory was not available to the auditors. Once the sample was received in the laboratory, it was logged into a notebook and then maintained under the custody of the analyst. A work order number was assigned to the sample that referenced a computer file containing the receiving date. It was stated that the sample would be retained for 6 months before disposal.

4.1.3 Sample Analysis

4.1.3.1 Analysis by GC/EC--

The test burn oil sample was initially analyzed by GM using gas chromatography with an electron capture detector (GC/EC). The documentation of the procedure given to the auditors was a hand-written outline of GC operating conditions and instructions for standard preparation. No sample cleanup was used; the oil sample was simply weighed and diluted with hexane prior to injection into the chromatograph. A 0.5 µg/mL solution of Monsanto Aroclor 1242 in hexane was used as the standard.

The sample was quantitated using the peak height of the major peak in the chromatogram; this was measured using a valley baseline correction. Instead of using a calibration curve for data calculation, the intent was to adjust the injection volumes of the sample and standard to obtain approximately the same peak heights and then to calculate the sample concentration by ratio. This was achieved in the analyses of the test burn oil where the peak heights were 6.7 and 6.75 cm for the standard and sample, as measured by the auditors. When the peak height of the standard is considerably different from that of the samples error may be introduced by using the ratio method of calculation. To evaluate this, the auditors requested that a calibration curve be prepared. It was found that the peak heights are proportional to the amount of Aroclor 1242 injected over the range of 0.5 to 2.5 ng, but the calibration curve does not pass through the origin, as is assumed in the ratio method of calculation. However, this error is not present in the analytical data for the test burn oil.

As noted above, the standard was prepared from Aroclor 1242. At the request of the auditors, a standard so prepared was compared to a standard prepared from EPA Reference Aroclor 1242, Lot No. 5041, obtained on February 10, 1976. Using the chromatograms, the auditors calculated that the 0.50-ppm standard used by GM was 0.54 ppm relative to the EPA reference material.

The results obtained for the test burn oil sample using the Monsanto standard are shown in Table 2. The result obtained by GC/EC is from a single analysis.

4.1.3.2 Analysis by GC/MS--

The test burn oil was also analyzed by GM using gas chromatography/mass spectrometry (GC/MS), again using a standard prepared from Monsanto Aroclor 1242. Mass 256 was used to monitor Aroclor 1242 and mass 172 used to monitor the 2-fluorobiphenyl, which was added as an internal standard. A standard addition experiment was conducted in which 250 and 500 ppm of Aroclor 1242 (on the initial oil basis) was added to the sample. The results of the GC/MS analysis are included in Table 2.

4.1.4 Preparation of Test Burn Oil

The data on the preparation of the test burn oil were examined to determine if the analytical results were consistent with the amount of PCB added.

TABLE 2. RESULTS OBTAINED BY GM FOR PCB IN TEST BURN OIL*

Method	Aroclor 1242 Found (µg/g)†
GC/EC	496 (493)
GC/MS	529, 539
GC/MS, standard addition	510 (486)

*Values in parentheses were calculated by RTI auditors from data supplied by GM.

†Values were obtained using Monsanto Aroclor 1242 as the standard. Based on a comparison of standards prepared from Monsanto and EPA Reference Aroclor 1242, Lot No. 5041, the results would be 8 percent higher if the EPA material had been used as the standard.

The preparation of the test burn oil involved initial analysis of a lot of reclaimed oil for PCB and then addition, in two stages, of capacitor fluid that contained 93 percent Aroclor 1242, based on GM analysis. This mixture was analyzed using GC/EC. The data are summarized in Table 3.

4.2 AUDIT OF GCA DATA

4.2.1 Sampling

No documented information was made available concerning the sampling of the test burn oil. After examining the sample container, GCA personnel concluded that the sample had been taken by GM and then transferred to the on-site GCA field crew.

4.2.2 Sample Custody

No records relating to the custody of the sample prior to receipt at the GCA facility in Bedford were available. Upon receipt at Bedford, the sample was logged in, and from this point, sample custody appears satisfactory with adequate documentation.

4.2.3 Sample Analysis

The test burn sample was analyzed by GCA using gas chromatography with electron capture detection. Prior to analysis, a portion of the sample was weighed, diluted with hexane, and treated using a sulfuric acid cleanup procedure. The cleaned, diluted sample was then injected into the chromatograph. Adequate documentation of the analysis procedure was provided. The standards used initially were prepared from an Aroclor 1242 stock solution obtained from Supelco, Inc.

Quantitation of the chromatograms was done by summing the areas of five major peaks. A calibration curve was prepared daily relating total area to concentration of Aroclor 1242 injected. Data examined by the auditors yielded calibration curves of satisfactory linearity and the sample was bracketed by standards.

Quality control measures used in this analysis included spiking of No. 6 fuel oil with Aroclor 1242, blind checks using Aroclor 1242 reference materials from EPA/HERL-RTP, Lots 8758 and 8937, and the use of quality control check samples procured from EPA/EMSL-Ci. A stock solution of Aroclor 1242 from

TABLE 3. GM DATA RELATED TO PREPARATION OF TEST BURN OIL

Sample	Aroclor 1242, µg/g	
	Found	Expected
Initial reclaimed oil, (430 gal, 1,489 kg)	132.5	---
Oil after initial spike with 408 g of Aroclor 1242*	370	407
Oil after second spike with 103 g of Aroclor 1242*	496	476

*Weight of added Aroclor 1242 calculated using a specific gravity of 1.3695 and a value of 98 percent Aroclor 1242 in the capacitor fluid.

Applied Science Labs, Inc., in addition to that obtained from Supelco, was used to prepare standards

These quality control checks by GCA verified the analytical methodology in that Aroclor 1242 could be satisfactorily recovered (92 to 96 percent) when added to No. 6 fuel oil at the level of 50 μg/g. The results using the various commercial and EPA Aroclor 1242 materials, however, were inconsistent, indicating discrepancies in assigned concentrations. Only the neat EPA/HERL-RTP reference Aroclor 1242 lots and the Applied Science Labs, Inc., standards were in agreement. Examination of the chromatograms indicated similar patterns for all of the materials. The discrepancies have not been resolved by GCA or by the RTI auditors.

The results obtained relative to the various Aroclor materials found by GCA for the test burn oil are presented in Table 4 Only a single analysis of the test burn oil was conducted by GCA. Some of these results were calculated by the auditors using data supplied by GCA. As noted above, the test burn oil was analyzed using Supelco, Inc., standards; however, data were made available by GCA interrelating all of the Aroclor reference materials and these data were used to calculate the values for the test burn oil compared to the various reference materials.

TABLE 4. RESULTS OBTAINED BY GCA
FOR PCB IN TEST BURN OIL*

Reference material	Aroclor 1242 found in test burn oil (μg/g)
Supelco, Inc., C/N 4-4803 (Kit PCB-A-21)	--- (471)
EPA/HERL-RTP Reference Aroclor 1242, Lots 8937, 8758	750 (738)
Applied Science Labs, Inc. Lot #478	750 (731)
EPA/EMSL-Ci QC Samples WP679, Conc. 7 and 8	--- (538-590)

*Values in parentheses were calculated by RTI auditors from data supplied by GCA.

SECTION 5
CONCLUSIONS AND RECOMMENDATIONS

5.1 CONCLUSIONS

1. The recordkeeping and procedural documentation related to the sampling of the test burn oil and to sample custody prior to receipt of the samples in the laboratories was inadequate, thus jeopardizing the analytical data.
2. The analytical methodologies used by each of the laboratories, though different, appear acceptable and comparable in the case of the test burn oil. The possible effect of the sample cleanup is not known. The difference between organizations in the method of quantitation used to reduce the GC/EC data does not appear to be a major cause of any discrepancy in results. This was indicated by reduction of GCA data (Supelco standard and test burn oil) using the GM method of peak height measurement, which gave a value for the test burn oil within 5 percent of that obtained by the GCA peak area summation method.
3. The GM laboratory documentation and recordkeeping related to the test burn oil sample do not appear adequate for EPA project work. It should be noted that GM analyzed the oil for internal purposes only and did not expect that their results would be used as part of the EPA project data.
4. The difference in results for the concentration of PCB in the test burn oil both between laboratories and within GCA appeared, from the audit, to be primarily attributable to the different standards that were used. Both GM and GCA results are traceable to various lots of EPA/HERL-RTP reference Aroclor 1242 (GM, Lot 5041; GCA, Lots 8758 and 8937) and information obtained following the audit indicated that these various lots all originated from the same batch of Aro-

clor 1242 obtained from Monsanto. Subsequent to the audit, EPA/HERL-RTP analyzed a sample of the test burn oil from each laboratory and also reference materials that had been used (GM, Monsanto Aroclor 1242 and GCA, a solution of EPA/HERL-RTP reference 1242, Lot 8937). The results indicated that there was no significant difference between the standards, and the results for 1242 in the test burn oil were 614 ppm for the GM sample and 640 ppm for the GCA sample. Thus, the differences among the laboratories appear to represent the analytical variability that is inherent in the analysis for complex mixtures such as Aroclor 1242.

5. The analytical data obtained at GM and GCA are consistent with the data obtained in the preparation of the test burn oil.

5.2 RECOMMENDATIONS

1. It is recommended that in any future projects involving PCB analysis, the analytical procedures and standards to be used be established before analysis is begun. Also, it is desirable to analyze critical samples more than once.
2. It is recommended that in projects involving sampling and analysis, all related activities should be fully documented and that valid sample chain-of-custody procedures be instituted

APPENDIX A

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

DATE September 10, 1980

SUBJECT Results of Analyses of PCB Standards

FROM Merrill Jackson, Research Chemist
ACB/ETD/HERL (MD-69)

TO David Sanchez, IERL (MD-62)

THRU: Dr. Robert G. Lewis, Chief
Analytical Chemistry Branch/ETD/HERL (MD-69)

This is to report to you the results of our analyses of PCB standards and PCB contaminated oil used in the General Motors PCB boiler burn.

On Sept. 3, 1980, Dr. Peter Collins of RTI delivered to me an Arochor 1242 standard which he had received from GM and a sample of the diluted 1242 standard used by GCA in their analyses. I requested a new 1242 standard from the EPA repository and one from lot 9224 was delivered on this date. On Sept. 8, 1980, Dr. Collins delivered two PCB contaminated fuel samples, one each from those analyzed by GM and GCA.

I prepared stock solutions of the EPA and GM 1242 standards. The GCA 1242 standard was labeled as 6.7 ng/µl. Final dilutions of all three standards were prepared so that the concentration was 1 ng/µl. (This was the concentration needed for my gas chromatographic conditions.)

I did not perform any cleanup on the fuel oils. They were simply diluted with hexane to the final concentration of 1 ng/µl of 1242 based on a 500 ppm initial concentration.

Quantitation of the chromatograms was by totaling the peak heights of all peaks present. Under our conditions Arochor 1242 has 14 major peaks.

However, the diluted fuel had a major interference which blocked out the last 4 peaks, therefore on the fuel analyses only the first 10 peaks were measured and compared to the first 10 peaks of the standards.

We found that the GM Arochor 1242 standard was 1.03 ng/µl and the GCA was 0.97 ng/µl when compared to the EPA standard. Repeated GC analyses (injections) of the EPA standard solution gave a result of 1.00 ± 0.03 ng/µl, therefore there were no analytical differences between any of the 1242 standards. We found 614 ppm of 1242 in the GM oil and 640 ppm in the GCA oil. Again we believe that the differences are analytical and that an average of the two is valid.

I have attached copies of the chromatograms in the order in which they were ran.

CC: Peter Collins, RTI
D. E. Gardner

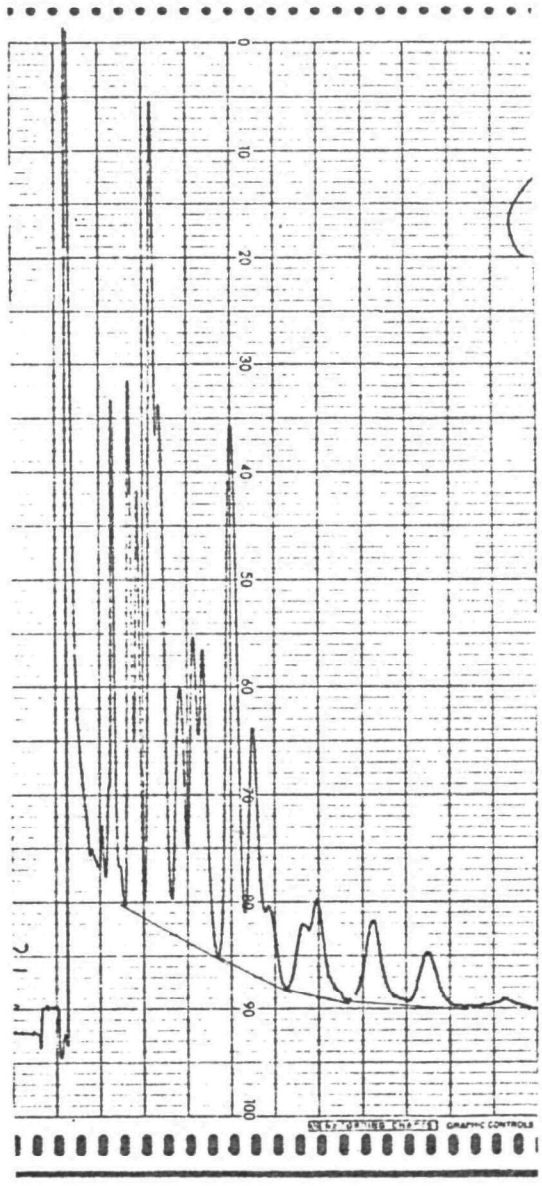


Figure A-1. Chromatogram of 3.1 µL of EPA-1242, 1 µg/mL.

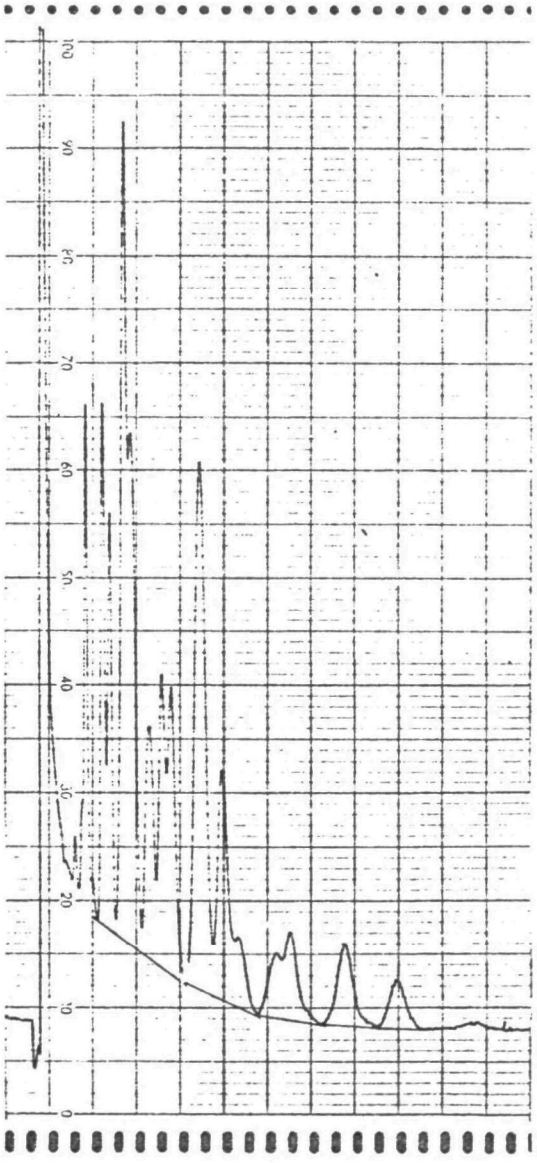


Figure A-2. Chromatogram of 3.1 µL of EPA-1242, 1 µg/mL.

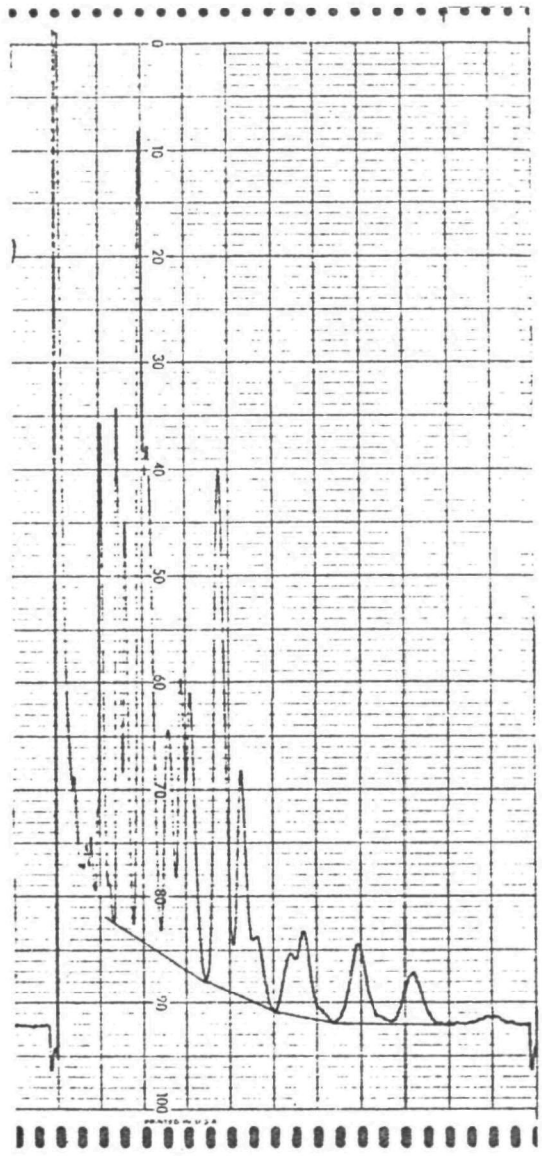


Figure A-3. Chromatogram of 3.1 µL of GCA standard, 1 µg/mL.

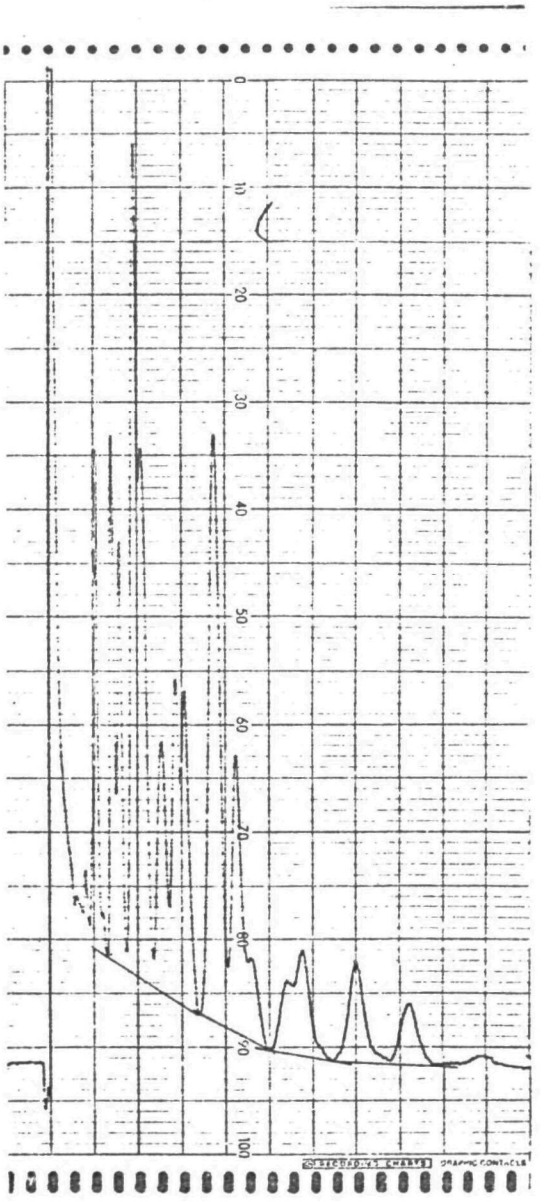


Figure A-4. Chromatogram of 3.1 µL of GM standard, 1 µg/mL.

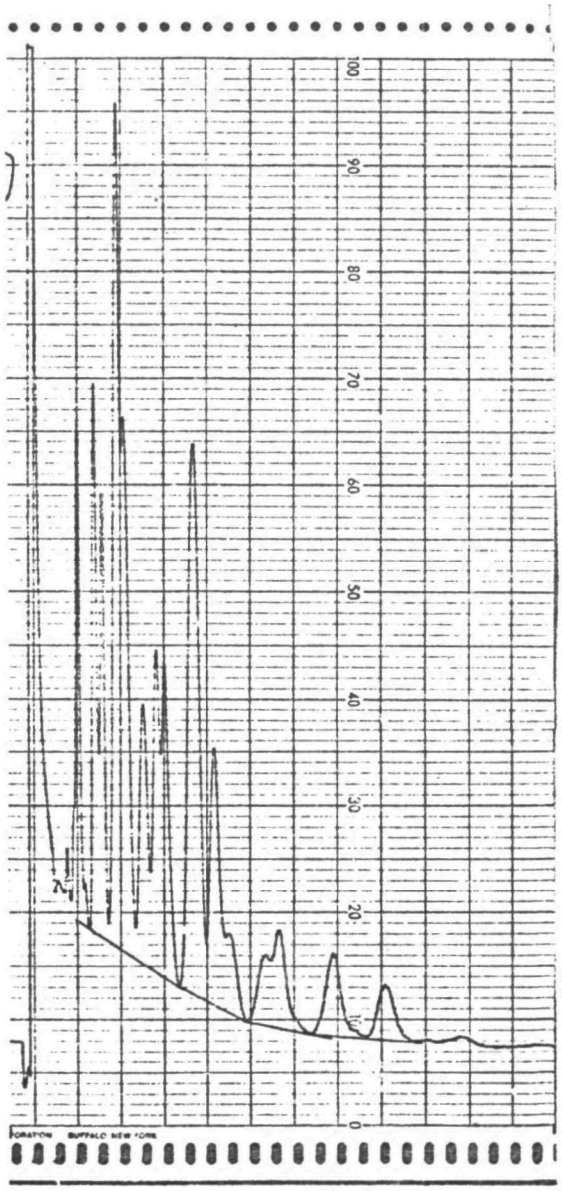


Figure A-5. Chromatogram of 3.1 µL of EPA-1242, 1 µg/mL.

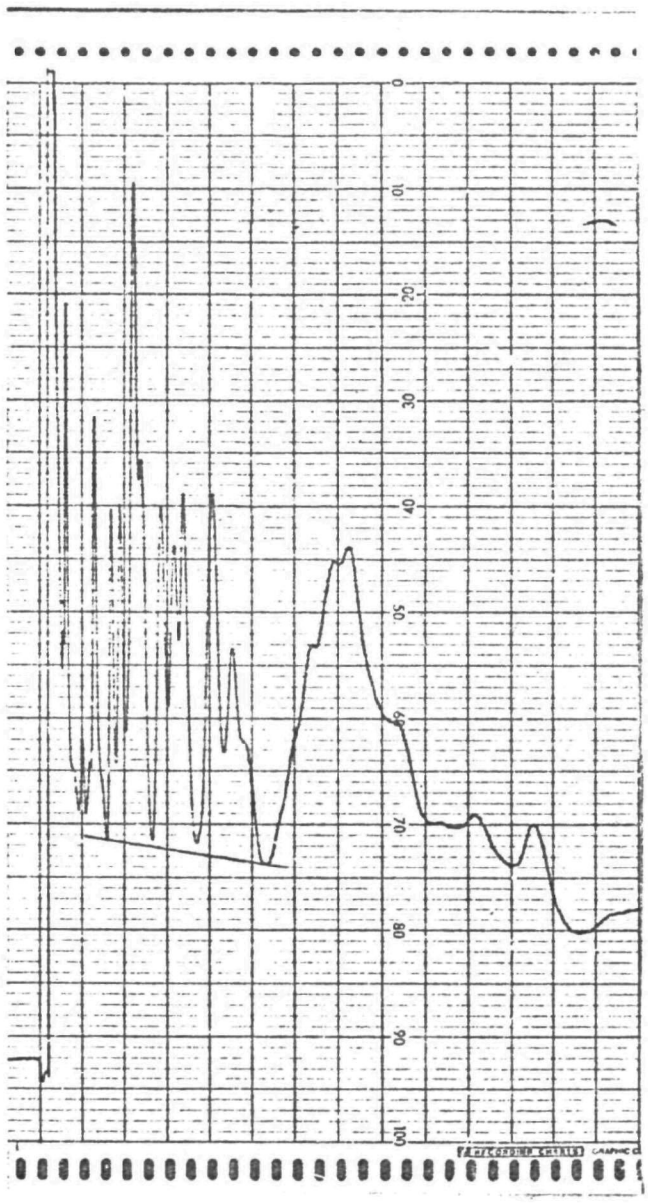


Figure A-6. Chromatogram of 2.1 µL of diluted GCA sample of test burn oil.

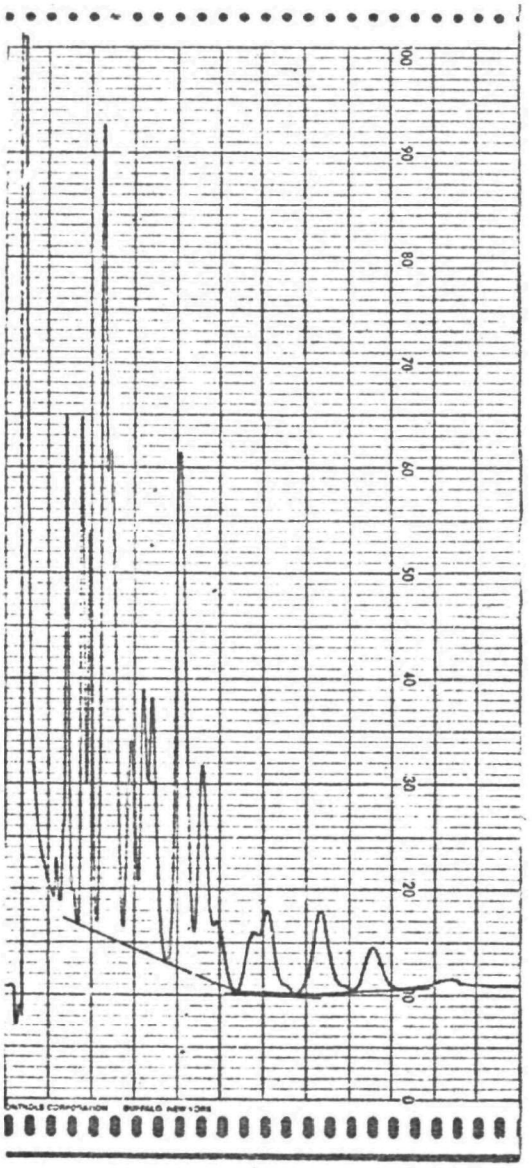


Figure A-7. Chromatogram of 3.1 µL of EPA-1242, 1 µg/mL.

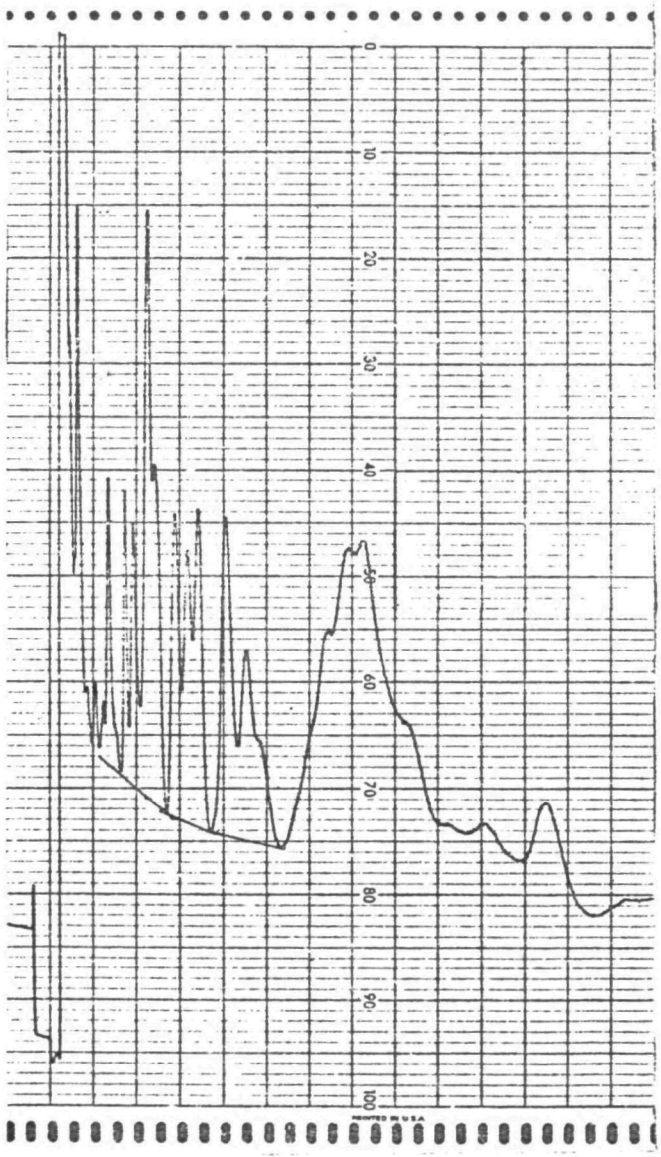


Figure A-8. Chromatogram of 2.1 µL of diluted GM sample of test burn oil.