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TO B. KONKOSKI
MASSENA OPERATIONS

1992-12-01

ANALYTICAL CHEMISTRY DIVISION FINAL REPORT

RE: ANALYSIS FOR PCBs IN RPM/GALBESTOS COATED SIDING
Letter Report No. 08-92-100

ANCH Job Order #: 92-102108
Shop Order #: 78M39508
Analyst: V. L. Bastecki

Data Quality Objectives:
In the evaluation of RPM/Galbestos siding coatings, a sample preparation and analysis method is required that results in a representative characterization of the PCB content in these materials.

Quality Control:
Sample preparation and analysis was performed in duplicate.

Case Narrative:
To obtain a more representative determination of PCB content in asphalt-based coated siding samples, a 10 cm x 10 cm panel was extracted. Section 7.1.2 of ANCH Method 3042-001 (copy attached) was used to prepare samples for the analysis reported here.

Sample Identification:
LSN 234847
Customer Identification N-40 Lower Level Sidewall Galbestos

RESULTS:

PCB Concentration:	81,000 mg/kg (ppm) Run 1
	73,000 mg/kg (ppm) Run 2
Aroclor Type:	1268
Limit of Detection:	5 mg/kg (ppm)

Lab Reference: 2562:174-177

ALCOA

Analytical Method:
ANCH 3042-001 (Rev 92/11), Determination of Polychlorinated Biphenyl (PCBs) in Oils and Miscellaneous Organic Materials

Conclusion:
The PCB data reported here is in concentration units in the extracted coating. In addition to the asphalt-based coating, this extract may include metal fines, rust, asbestos felt and any other components removed from the metal panel during the solvent extraction process.

In the previous preliminary analysis of this sample (J. O. # 92-092320, LSN 231066) approximately 20 mg of coating scraped from the surface of the siding panel was used for the analysis; 46,000 mg/kg (ppm) Aroclor 1268 was the calculated PCB concentration for that determination.

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ANALYTICAL CHEMISTRY DIVISION
ALCOA TECHNICAL CENTER

Determination of Polychlorinated Biphenyls (PCBs) in Oils and Miscellaneous Organic Materials

METHOD:	3042-001
REVISED BY:	J. P. Auses, V. L. Bastecki
REVISION DATE:	1992 November
ANALYTE:	PCBs, Aroclor
METHOD TYPE:	Gas Chromatography, Fused Silica Capillary
ANCH DIVISION AREA:	302
AGENCY CROSS REFERENCE:	ASTM Method D4059, EPA Method 608, EPA SW-846 Method 8080
SAMPLE CLASS:	Oil, Tar, Miscellaneous Organic Matrices
RELATED ANCH METHODS:	3042

1.0 SUMMARY:

This method employs fused silica capillary gas chromatography (GC) with electron capture detection to analyze for Aroclor PCBs in a variety of organic matrices.

2.0 SCOPE:

The method provides for the determination of Aroclor PCBs in transformer oils, hydraulic fluids, asphalt-based building siding coatings and miscellaneous organic matrices. It employs solvent dissolution of the sample, a concentrated sulfuric acid clean-up procedure and fused silica capillary gas chromatography with electron capture detection. Aroclor PCBs are identified by pattern matching of the chromatograms with those of standard Aroclor mixtures. Quantitation of the Aroclors is accomplished by measuring peak areas for selected retention time ranges, based on the suspect Aroclors.

3.0 APPARATUS/EQUIPMENT:

3.1 Laboratory glassware including beakers, graduated cylinders, volumetric flasks and the following:

- Graduated receiver tube, 10 mL, with ground glass stopper (Kontes Glass Part Nos. K-570050 (size 1025) and K-850500 (stopper 19/22) or equivalent).
- Autosampler vials for gas chromatograph, (Hewlett-Packard Part No. 5080-8712 or equivalent).
- 10 uL liquid syringe

3.2 Hewlett-Packard 5840A Gas Chromatograph with capillary injection system and electron capture detector; J & W DB-5 30 meter fused silica capillary column, or equivalent.

4.0 REAGENTS/CHEMICALS:

4.1 Aroclor PCB Standards:
- Aroclor 1016, 1221, 1232, 1242, 1248, 1254, 1260, 1268.

4.2 Isooctane (2,2,4 trimethylpentane), pesticide grade or equivalent.

4.3 Concentrated sulfuric acid.

4.4 Methylene chloride (dichloromethane).

5.0 SAFETY/HAZARDS:

5.1 Flammable, toxic solvents.

5.2 Protective Clothing: Laboratory safety glasses with sideshields, plastic gloves, laboratory apron and/or labcoat.

5.3 Perform sample preparation in fume hood (including sample dissolution, solvent evaporation/concentration and sulfuric acid clean-up).

* SPECIAL NOTE FOR COATED SIDING SAMPLES

5.4 Take proper precautions to avoid exposure to asbestos which may be present.

6.0 QUALITY ASSURANCE:

6.1 Analysis of Aroclor standard solutions required with each batch of samples. Blanks and spikes to be analyzed as required to assure acceptable recovery and to identify interferences and degree of weathering of sample matrices.

6.2 Performance evaluation samples to be analyzed on a regular basis.

7.0 PROCEDURE:

7.1 Sample Preparation

7.1.1 OILS

7.1.1.1 10-20 mg of sample is weighed to the nearest 0.01 mg in a graduated receiver tube. The sample is then diluted to 1 mL with isooctane. For clean-up, 1 mL concentrated sulfuric acid is added to the diluted sample. The tube is then shaken for 1 minute and the supernatant solvent layer is withdrawn and placed in an autosampler vial for analysis. Vial is sealed and loaded into gas chromatograph autosampler tray.

7.1.1.2 If necessary, appropriate dilutions of samples are made in isooctane.

7.1.2 ASPHALT-BASED SIDING COATINGS

7.1.2.1 Coating is removed from metal siding panel by scraping with lab spatula (Remove coating in fume hood; wear proper personal protective equipment). If coating is homogeneous, 10-20 mg of sample weighed to the nearest 0.01 mg in a graduated receiver tube. Sample dilution, clean-up and transfer continue as in 7.1.1.1-2 above.

7.1.2.2 If coating is non-homogeneous or if a surface area-based measurement is required, the following procedure is used:

7.1.2.2.1 Measure and cut a 10 cm x 10 cm piece of coated metal siding. Weigh and record weight to the nearest 0.1 gram.

7.1.2.2.2 Holding this piece with lab tongs or suitable clamp over a 1 liter beaker, rinse both sides of sample with isooctane and/or methlyene chloride, collecting all solvent rinsate in beaker. Use spatula to assist in removal of coating from metal surface. Continue rinsing until all coating has been removed from the metal.

7.1.2.2.3 After removal of coating bring metal panel to constant temperature and weigh. Record weight of metal panel to nearest 0.1 gram. Subtract this weight from weight obtained in 7.1.2.2.1 to determine coating weight.

7.1.2.2.4 After rinsing with solvent remove felt pieces or large particulate from solvent rinsate. If possible solvent may be filtered through a coarse filter. (Coating does however tend to blind even coarse screen filters.)

7.1.2.2.5 Dilute solvent rinse into appropriate volume of isooctane solvent or evaporate solvent to dryness. If allowed to dry, transfer 10-20 mg of dry residue to graduated concentrator tube and add 1 mL of isooctane or hexane. To the 1 mL aliquot of diluted sample, carry out acid clean-up step and transfer into GC autosampler vial as in 7.1.1.1-2 above. Analyze by gas chromatography. After initial GC analysis, re-dilute as needed to result in an on-scale chromatogram, and re-analyze by GC.

7.2 Gas Chromatograph Instrument Conditions

7.2.1 Column conditions:

7.2.1.1 Column: 0.25 mm ID x 30 m, J&W Fused Silica Capillary Column.

7.2.1.2 Stationary Phase: Durabond DB-5 (polymethyl (5% phenyl) siloxane).

7.2.1.3 Carrier gas: Helium

7.2.1.4 Carrier gas velocity: 30 cm/sec

7.2.1.5 Make-up gas: 5% methane in argon

7.2.1.6 Make-up gas flow rate: 35-40 mL/min
7.2.1.7 Capillary split ratio: 10:1 or splitless injection
7.2.1.8 Injection volume: 1 uL (Autosampler injection recommended)

7.2.2 GC operating conditions:
7.2.2.1 Initial column temperature: 150°C
7.2.2.2 Initial hold time: 0 minutes
7.2.2.3 Temperature program rate: 10°C/min
7.2.2.4 Maximum temperature: 270°C
7.2.2.5 Maximum temperature hold time: 15 minutes
7.2.2.6 Injection port temperature: 280°C
7.2.2.7 Electron Capture Detector temperature: 325°C
7.2.2.8 Slope sensitivity: 0.1
7.2.2.9 Peak area rejection minimum: 0

7.3 Analysis
7.3.1 Aroclor standards are analyzed by GC (approximate concentration 0.001 mg/mL) before each batch of samples. Chromatograms of each Aroclor standard and the reports that include their peak area count summaries are printed out on integrator.
7.3.2 Samples are analyzed by GC with reports generated as noted immediately above.
7.3.3 To identify Aroclor PCBs present, the pattern of peaks in the chromatograms of each sample are compared with those of the standards. Where distinct patterns of peaks are present in the sample chromatograms, identification of specific Aroclors is made. Summation of the peak areas associated with pattern retention times are used to quantitate Aroclors present.
7.3.4 Where distinct Aroclor patterns are not present in the sample or where multiple Aroclors overlap, selected ranges of peaks which match the standards are used for quantitation.

8.0 CALCULATIONS

8.1 The concentration of Aroclor PCBs present in oil sample or coating is calculated as follows:

$$\frac{\text{mG/mL Aroclor standard}}{\text{Standard area summation}} \text{ X Sample area summation X Dilution}$$

$$\text{X}\frac{1 \times 10^6}{\text{mG sample/mL}} = \text{mG/KG (ppm) PCB}$$

8.2 The area-based quantity of Aroclor PCBs present on coated siding samples is calculated as follows:

$$\frac{\text{mG/mL Aroclor standard}}{\text{Standard area summation}} \text{ X Sample area summation X Dilution}$$

X 1000 uG/mG = µG PCB on 10 cm x 10 cm panel
(both sides extracted; 200 cm^2 surface area)