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JOB PROJECT NO.: 483.5420

DATE: October 27, 1983

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TITLE: ANALYSIS OF LULING INCINERATION RESIDUE

AUTHORS: B. M. Hughes

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ABSTRACT: A sample of mixed DCA heads and residue was analyzed for the possible PCB content. The results of the analyses are shown in this report.

Technical Approval:

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Approved by:

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Approval date: 10-27-83

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ACKNOWLEDGEMENT

Contributions of the following Dayton Laboratory personnel to this project are gratefully acknowledged:

Analyses: B. M. Hughes
D. McKenzie
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ANALYSIS REPORT

DATE: October 24, 1993

LOG NUMBER: 1-83-10-04-02

PROJECT NO: 483.54218

TO:

Bob Ivory/St. Louis (Luline Incineration Residue)

FROM:

Environmental Sciences Center
Monsanto Research Corporation
1515 Nicholas Road
Dayton, Ohio 45418

Phone No: (513) 265-2411

Title: Analysis of PCBs in DCA Heads (60%) and Residue (40%)

Standard Sampling Method No(s): N/A

Standard Extraction Method No(s): PCEX01

Standard Analysis Method No(s): ECAN01

Final Report File No(s): A54218

SAMPLE EXTRACTION: One sample labelled AM-148 DCA Heads (60%) and Residue (40%) was extracted in duplicate by placing 1.0 g in a 14-ml Pierce vial with teflon-lined lids, spiking with 40 ug 1,2-dichlorobenzene-d4, and adding 10 ml hexane. Extracts were then washed with sulfuric acid and eluted through a column of silica gel impregnated with potassium permanganate, potassium hydroxide and sulfuric acid and an activated alumina column. The PCB-containing fraction was eluted with 5% methylene chloride in hexane and concentrated to 1.0 ml.

SAMPLE ANALYSIS AND RESULTS: The above extracts were analyzed using capillary GC/MS and capillary GC/EC techniques. From GC/MS analyses, one tetrachloro-biphenyl isomer was detected in the duplicate samples at concentrations of 1.8 and 1.9 ug/g. Recoveries of CL2-benzene-d4 were measured to be 16 and 26 %, respectively. Capillary GC/EC analyses of the

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above two extracts yielded values of 3.2 and 4.0 $\mu\text{g/g}$ of the CL4-biphenyl isomer. To assure that good recoveries were being obtained for PCB isomers, separate analyses were also conducted in which the sample was spiked with Aroclor 1254 at concentrations of 12 and 24 $\mu\text{g/g}$. Recoveries of Aroclor 1254 in these two spiked samples were 92 and 88 % and concentrations of the CL4-biphenyl isomer of 5.0 and 5.3 $\mu\text{g/g}$ were measured. Therefore the value of 2.0 $\mu\text{g/g}$ CL4-biphenyl isomer, measured from the GC/MS analyses is consistent with the capillary GC/EU results.

Capillary GC/EC analyses were conducted with levels of detection of PCB isomers approximately two orders of magnitude lower than GC/MS levels of detection. Therefore 14 other components, which have retention times identical to major components in Aroclor 1254, could also be quantified, assuming CL4-biphenyl electron capture response factors. The total concentration of these additional components were measured to be approximately 14 and 13 % of the major CL4-biphenyl isomer concentration. These 14 components are also thought to be PCB isomers, but at levels too low to unequivocally identify from complete mass spectra. They are also at low enough concentrations so that their quantitation should not be required for the future studies on incineration of these still bottoms.

The results reported above are for one single sample supplied by Dr. John Rapin in St. Louis. These results indicate that, based upon this one analysis, the Luling waste can be treated as not containing PCBs (since the total PCB content is less than 50 $\mu\text{g/g}$). However if there is wide variability of the PCB concentrations in other lots of dichloro-aniline heads and residues, the actual sample to be incinerated may approach or even exceed the 50 $\mu\text{g/g}$ criterion. Therefore it is very important to determine PCB content for the heads and residues which will actually be incinerated to assure that this incinerated sample is still below the 50 $\mu\text{g/g}$ total PCB concentration requirement.

ANALYSTS: R. M. HUGHES, D. MCENZIE AND C. TRANG

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The following pages describe Standard Extraction Methods and Standard Analysis Methods used for this project.

ESC Standard Extraction Method (SEM)
TITLE: PCBs in Various Matrices

ESC Standard Extraction Method No:
PCEX01

MATRIX (IES):

Still bottoms, soils, beef fat,
beef serum, whole milk, beef
feces

TYPICAL SAMPLE
SIZE
(g)

0.5 - 5.0

TYPICAL EXTRACT
VOLUME
(ml)

1

TYPICAL ANALYTES: Aroclors 1232,
1248, 1254, 1260, 1262, trichloro-
biphenyl isomers through octachloro-
biphenyl

ESC Standard Analysis Method Nos:
ECAN01, ECAN02

INTERFERENCES: Any electron capturing
species in the retention time region
of trichlorobiphenyl isomers through
octachlorobiphenyl

TYPICAL
SURROGATES

TYPICAL
RECOVERIES (%)

He-chlorobenzene

50 - 120

2,4,6-trichloro-
biphenyl

50 - 120

APPARATUS AND MATERIALS:

Burdick and Jackson he-xane, benzene and methylene chloride for sample
extraction.

Concentrated sulfuric acid for sample digestion or sample clean-up.

Silica gel columns impregnated with potassium permanganate, sulfuric acid,
and potassium hydroxide.

Activated basic alumina.

Disposable glassware.

LIMITATIONS: Method applies only to matrices which do not contain non-hydro-
lizable electron capturing species in the trichlorobiphenyl through octa-
chlorobiphenyl retention time region.

SAFETY PRECAUTIONS: See PCB handling protocols in laboratory safety manual.

DATES OF METHOD CREATION AND UPDATES: 10/25/83

ESC Standard Analysis Method (SAM)
TITLE: PCBs in Solvents

ESC Standard Analysis Method No:
ECAN01

ANALYTICAL INSTRUMENT AND MODE OF OPERATION: HP-5890 Capillary GC/EC interfaced to an HP-3356-LAS Laboratory Data System

TYPICAL ANALYTE	TYPICAL INSTRUMENT LEVEL OF DETECTION (LOD) (ug/ml)	TYPICAL PRECISION @ 10X LOD (%)	TYPICAL ACCURACY @ 10X LOD (%)
Trichlorobiphenyl isomers through octachlorobiphenyl isomers	0.01	30	30

INTERFERENCES AND LIMITATIONS: Any electron capturing species in the retention time region of trichlorobiphenyl isomers through octachlorobiphenyl are potential interferences. Exact retention times of most likely PCB isomers can be obtained from the analysis of Aroclor 1249, Aroclor 1254 or Aroclor 1260 formulations.

CHROMATOGRAPHIC COLUMN: J&W 16-5 (0.25 um) on fused silica wide bore (0.32mm)

COLUMN TEMPERATURE PROGRAM: 130(1)/4/300

INJECTION TYPE: Splitless

CALIBRATION AND STANDARDIZATION: Use internal standard quantitation technique and calibrate instrument response factors from authentic PCB isomer standards.

CALCULATIONS: $\text{PCB isomer concentration} = (\text{isomer area})/(\text{isomer RRF})$

where: RRF is the relative response factor for the appropriate PCB isomer.

SAFETY PRECAUTIONS: See PCB handling protocols in laboratory safety manual.

DATES OF METHOD CREATION AND METHOD UPDATES: 10/25/83