

November 18, 2009

003-09155-08 transmitted via email only

Ms. Carmen Santos U.S. Environmental Protection Agency, Region 9 Mail Code WST-5 75 Hawthorne Street San Francisco, CA 94105

Subject: Conditional Approval of the Toxic Substance Control Act Self-Implementing Cleanup

Notification and Certification, Former Pacific Electric Motors Facility, 1009 66th

Avenue in Oakland, California

Dear Ms. Santos:

The property owner, Aspire Public Schools (Aspire) and LFR Inc., an Arcadis Company (LFR) would like to thank the staff of the U.S. Environmental Protection Agency (USEPA) for the letter providing the conditional approval of the Self-Implementing Cleanup Plan (SICP; dated October 23, 2009) with conditions at the former Pacific Electric Motors Facility 1009 66th Avenue in Oakland, California (the "Site" [Figure 1] letter dated, November 13, 2009; the "EPA Letter"). The excavation of the polychlorinated biphenyl (PCB) affected soil began at the Site in accordance with the SICP and the EPA Letter on November 4, 2009 (Figure 2).

Aspire and LFR's intention to comply with the parameters of the conditional approval are provided as follows:

1. Certification Signed by LFR & Aspire

A revised certification for this project signed by representatives of both Aspire and LFR is attached.

2. Pre-Demolition and Post-Demolition PCB survey

The pre-demolition and post-demolition sampling plan for building materials is provided as an attachment to this letter.



3. Sampling & Analysis Plan

As acknowledged in the EPA Letter, LFR transmitted a Sampling and Analysis Plan for the Site (the "SAP") on November 5, 2009. This SAP focused on the objectives, methods, procedures associated with the soil samples to be collected and analyzed in conjunction with pre-demolition soil characterization and post-remediation soil sampling. As requested, the pre-demolition and post-demolition sampling plan for building materials is provided as an attachment of this letter.

4. Sequence of Pre-Cleanup PCB Soil Characterization; Pre-Demolition Sampling; Soil Remediation; Soil Clean-Up Verification

Aspire and LFR will complete the project under the following sequence of work:

- Pre-Cleanup PCB Soil Characterization The scope of this work was completed in accordance with the SAIC and the SAP
- Pre-Demolition Sampling this sampling was completed in accordance with the building materials SAP provided above (in this letter).
- Soil Remediation Site remedial actions are taking place at the Site in accordance with the Revised Corrective Action Plan, the SAIC, and the SAP
- Soil Clean-Up Verification and Post-Demolition Sampling Soil clean up verification and post-demolition sampling will be conducted in accordance with the procedures provided in the CAP, SAP, and SAIC. As provided in the SAIC, post-demolition soil sampling regarding the removal of the sewer pipelines at the Site will take place by collecting soil samples adjacent to the sanitary and storm sewer pipelines that are to be abandoned as part of the redevelopment of the Site. If material (liquid or solid) is present in the sewer pipes, samples will be collected for PCB analysis (EPA test method 8082) so that the material may be disposed of in accordance with the procedures provided in the EPA letter (see item 5 below).
- Following the demolition of the large warehouse building, soil samples will be collected from the ground surface (surface soil samples) at areas of the Site that were unpaved during demolition activities. Soil samples will be collected on a 75-foot grid in the unpaved areas. Samples will be collected and analyzed using methods provided in the SAP.
- In addition to samples of material from in the sewer pipeline(s) and as provided in the SAIC, soil samples will be collected every approximately 50 feet of sewer line approximately 1 to 2 feet below the pipeline invert. The soil samples will be analyzed for PCBs in accordance with the SAP. If soil containing greater than 0.13 milligrams per kilogram (mg/kg) is detected in the soil samples, additional soil will be removed and the additional confirmation soil samples will be collected for analysis in accordance with the SAP.



5. PCB Remediation Waste

Aspire has the following EPA identification number for this property: CAC002647778. Aspire and LFR will dispose of the soil in accordance with the procedures provided in the EPA letter. As such (porous and non-porous) building materials will be disposed of in accordance with the following regulations:

§ 761.61 PCB Remediation Waste

Bulk PCB remediation waste may be sent off-site for decontamination or disposal in accordance with this paragraph, provided the waste is either dewatered on-site or transported offsite in containers meeting the requirements of the DOT Hazardous Materials Regulations (HMR) at 49 CFR parts 171 through 180. (1) Removed water shall be disposed of according to paragraph (b)(1) of this section.

- (2) Any person disposing off-site of dewatered bulk PCB remediation waste shall do so as follows:
- (i) Unless sampled and analyzed for disposal according to the procedures set out in § 761.283, 761.286, and 761.292, the bulk PCB remediation waste shall be assumed to contain \geq 50 ppm PCBs.
- (ii) Bulk PCB remediation wastes with a PCB concentration of < 50 ppm shall be disposed of in accordance with paragraph (a) (5) (v) (A) of this section.
- (iii) Bulk PCB remediation wastes with a PCB concentration \geq 50 ppm shall be disposed of in a hazardous waste landfill permitted by EPA under section 3004 of RCRA, or by a State authorized under section 3006 of RCRA, or a PCB disposal facility approved under this part.

Analytical results of soil samples collected from soil boring 4B located in proposed excavation area PCB-EXC1, contained PCBs at a concentration of greater than 50 mg/kg (see Figure 2). Based on theses analytical results, soil excavated from this area will be transported off-site and disposed of at Waste Management's Kettleman Hills Landfill.

Analytical results of soil samples collected from soil borings located in proposed excavation areas PCB-EXC2, PCB-EXC3, and PCB-EXC4 of the Site contained PCBs at a concentration of less than 50 mg/kg (see Figure 2). Based on theses analytical results, this soil will be transported offsite and disposed of at Republic Services' Vasco Road Landfill located in Livermore, California.

§ 761.62 Disposal of PCB Bulk Product Waste

(b) Disposal in solid waste landfills. (1) Any person may dispose of the following PCB bulk product waste in a facility permitted, licensed, or registered by a State as a municipal or non-municipal non-hazardous waste landfill.



Based on the analytical results of samples collected from the various building materials at the Site, the building materials from the Site generated from demolition activities will be transported off-site and disposed of at Republic Services' Vasco Road Landfill located in Livermore, California.

6. Measures to Prevent Exposure of the Neighboring Community to Air Borne Particulates

In accordance with the SICP, the following provides the details regarding the air monitoring plan for the proposed excavation and demolition activities that are proposed for the Site.

Air Monitoring and Dust Control Measures

Real-time aerosol monitoring devices (mini-RAM) will be used to monitor total dusts generated during site work. If dust in excess of background levels (greater than 0.25 milligram per cubic meter [mg/m³] above background levels) is observed for a sustained period of time (greater than 5 minutes), appropriate dust suppression measures (e.g., spraying soil with water) will be undertaken.

A total dust action level of 0.25 mg/m³ above background levels that is sustained for 15 minutes would be conservative for the various COPCs detected on the Site that would be likely to adhere to windblown dust and protective of the on-site workers and members of the surrounding community.

Field staff will obtain and document total dust readings from the mini-RAM throughout each work day when affected soil excavation activities are occurring on the Site. These readings will be obtained from air monitoring stations established along the Site's perimeters (a total of 5 stations; see Figure 2).

In addition to monitoring for total dust using at least four fixed air monitors, equipped with a mini-RAM, Personal Air Monitors (PAMs) used to collect air samples. The air samples will be collected on cassettes (media) that will be submitted to a laboratory for analysis of PCBs, arsenic, lead, and benzene. The air samples will be collected each work day when affected soil excavation activities and site demolition activities are occurring on the Site. Air monitoring stations will be at locations illustrated on Figure 2 (attached).

Air samples to be analyzed for PCBs will be collected on laboratory supplied filter tubes equipped with a solid sorbent material comprised of 13-mm glass fiber and Florisil. The samples media will be provided by and the samples will be an analyzed by EMSL Analytical, Inc. located in Westmont, New Jersey. Details regarding the collection and analytical methods for the air sample samples are provided in the attached documentation.



Public Notification

The public participation document mailed by the Alameda County Environmental Health has been laminated and is posted in two places along the fence that is adjacent to the public right-of way along 66th Avenue.

7. Revised Clean-up Level for PCBs in Soil

Aspire and LFR will remove soil containing PCBs at concentrations exceeding 0.13 mg/kg. If soil containing concentrations of PCBs greater than 0.13 mg/kg cannot be removed from the Site that area will be documented as described under item 9. Risk Management Plan and Deed Notice below.

8. Cap for Site

In accordance with the development plan for the Site, the entire property will be capped with either building structures, asphalt, or concrete. Prior to developing the Site, a minimum of 2 feet of imported fill will be placed and compacted as backfill in areas where affected soil has been previously removed from the Site. In addition, areas of the Site that will be redeveloped for vehicular traffic or structures, 8 to 12 inches of base rock will be imported to meet the geotechnical requirements of the redevelopment project.

9. Risk Management Plan and Deed Notice

A risk management plan will be prepared for the Site and a notice will be placed on the deed in accordance with item 9 of the EPA Letter.

10. Record Keeping and PCB Clean-Up Report

Documentation associated with the remediation of the PCB-affected soil and building materials will be retained and the PCB Clean-Up Report will be prepared in accordance with item 9 of the EPA Letter.

11. Restoration of the Site

The Site will be restored in accordance with the CAP, the SICP, and the EPA letter.



Following your review of this letter, please do not hesitate to contact me if you have any questions or require additional information.

Sincerely,

Alan D. Gibbs, P.G., C.HG.

Vice President/Principal Hydrogeologist

Ron Goloubow, P.G.

Senior Associate Geologist

Attachments

Figures 1 and 2 Certification

Sampling Plan for Building Materials

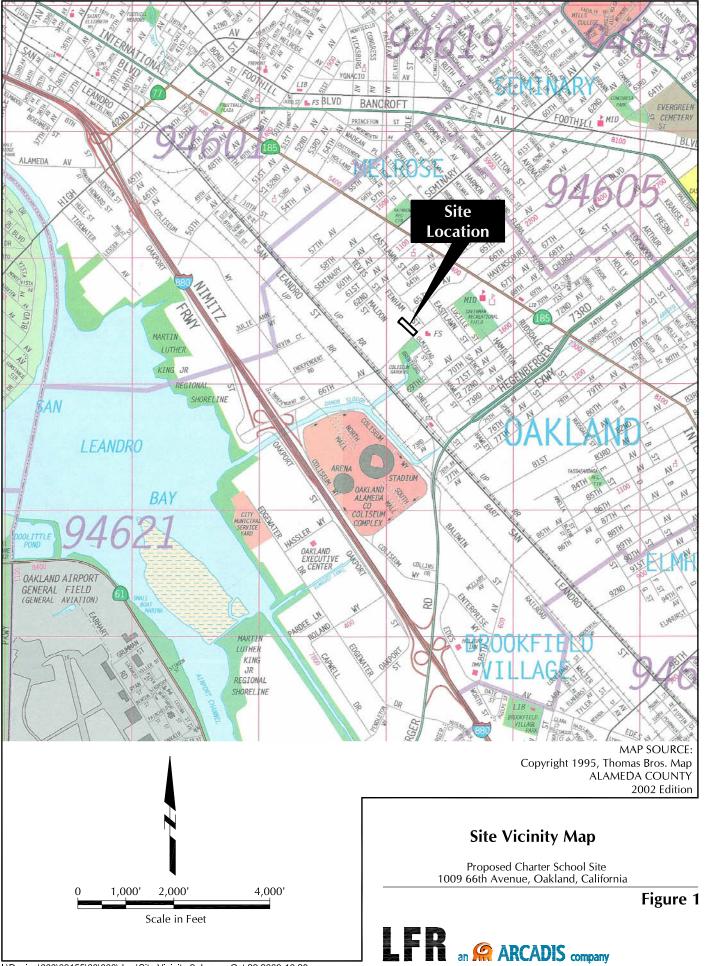
Air Monitoring; Sample Analysis Methods

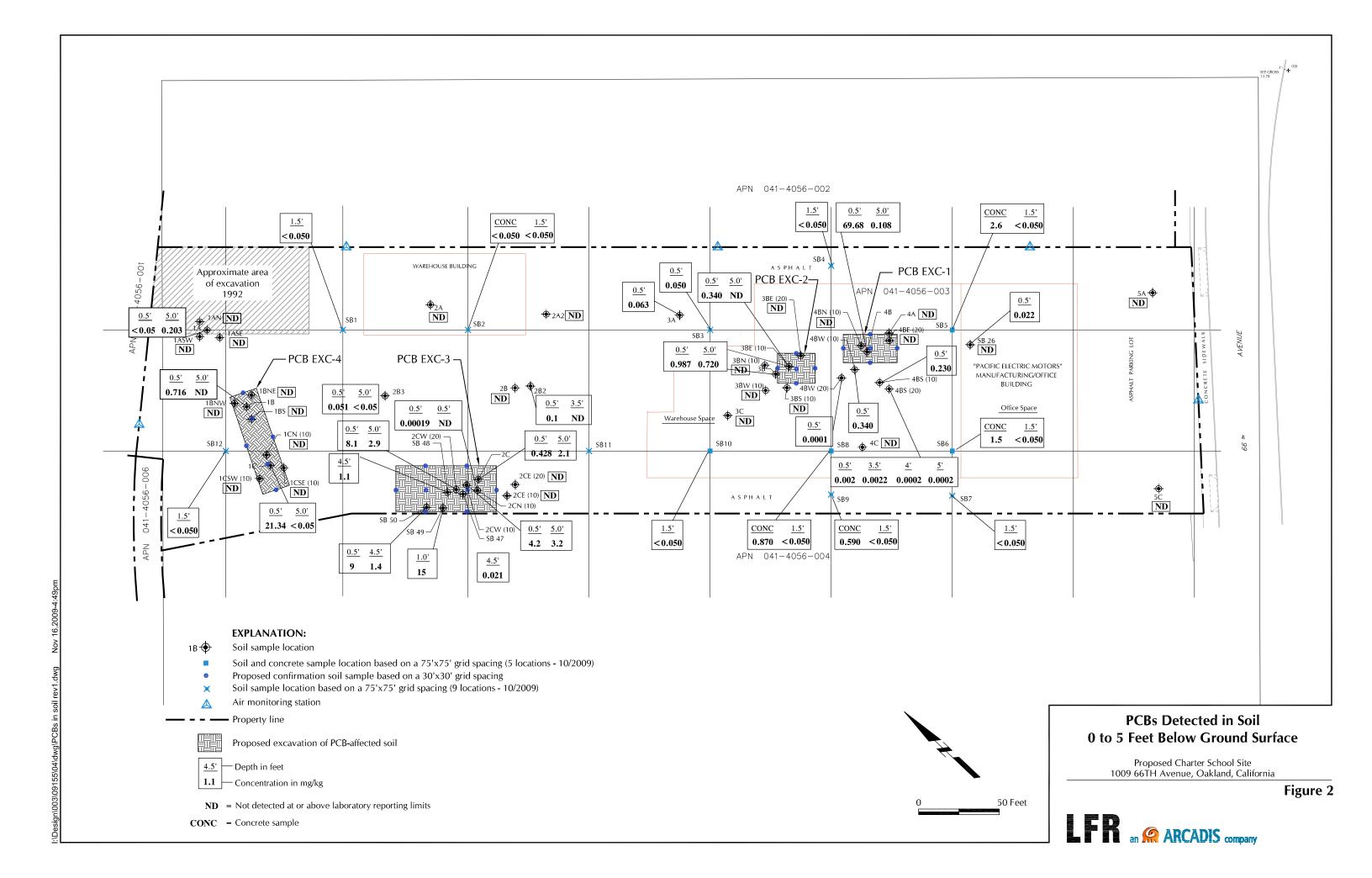
cc: Mr. Mike Barr- Aspire Charter Schools

Charles Robitaille - Pacific Charter Schools

Paresh Khatri - Alameda County Department of Environmental Health

FIGURES





TSCA CERTIFICATION

Certification Statement

Owner: Aspire Public Schools

Parties Conducting Cleanup: Arcadis and Innovative Construction Solutions Project: Former Pacific Motors Facility – 1009 66th Avenue, Oakland, CA

In accordance with 761.61(a)(3)(i)(E); I, Michael Barr, hereby certify, that all sampling plans, sample collection procedures, sample preparation procedures, extraction procedures, and instrumental/chemical analysis procedures used to assess or characterize the presence, concentrations, and extent of polychlorinated biphenyl- (PCB) impacted media for Former Pacific Motors Facility – 1009 66th Avenue, Oakland, CA are on file and available for USEPA review at the following location:

LFR Inc. an Arcadis Company Contact: Ron Goloubow

Under civil and criminal penalties of law for the making or submission of false or fraudulent statements or representations (18 U.S.C. 1001 and 15 U.S.C. 2615), I certify that the information contained in or accompanying this document is true, accurate, and complete. As to the identified section(s) of this document for which I cannot personally verify truth and accuracy, I certify as the company official having supervisory responsibility for the persons who, acting under my direct instructions, made the verification that this information is true, accurate, and complete.

By:

Michael Barr - Aspire Public Schools

Date

11/9/09

By:

Ronald E. Goloubow - LFR Inc. An Arcadis company

BUILDING MATERIALS SAMPLING PLAN

Building Materials Sampling Plan Former Pacific Electric Motors Facility 1009 66th Avenue in Oakland, California

On behalf of Aspire Public Schools (Aspire) LFR Inc. an Arcadis company (LFR) has prepared this Building Materials Sampling Plan (BMSP). The BMSP provides the methods used by LFR to assess the presence of polychlorinated biphenyls (PCBs) in the building materials in the two buildings at located at the former Pacific Electric Motors Facility located at 1009 66th Avenue in Oakland, California. The purpose of the BMSP was to provide data regarding the presence of PCBs in the building materials at the Site. The data collected will be used to assess disposal methods for the building materials following demolition of the two buildings currently located at the Site.

During the survey, LFR attempted to identify and collect samples of the building materials that may contain PCBs in preparation for the demolition of the buildings. The building materials survey was conducted to comply a request from us U.S. Environmental Protection Agency (U.S. EPA) to determine if the building materials in the buildings at located at the Site contain PCBs.

The survey encompassed visible and accessible interior areas of the two subject buildings. To assess the presence of PCBs in the building materials LFR collected representative samples of the following materials:

Window Caulk
Paint (or painted surfaces)
Concrete
Concrete Caulk
Roofing material

Sample Collection, Handling and Documentation

Sample procedures described in this section will be used for sample collection, shipping, analysis, and disposal. Each sample of the building materials will be collected using hand tools and the sample will be placed in a laboratory supplied glass jar. Sample containers will be 4 or 8 ounce laboratory supplied glass jars, and no preservative will be used. The sample container will be labeled with the sample identification, the time and date of collection, the analysis requested, and the initials of the sampler. The samples will be stored in an ice-chilled cooler and submitted to the laboratory under strict chain-of-custody protocols. The sample identification will reference the type of building material and location that the sample was collected (i.e. window caulking-building 1). The location of the sample and the sample identification will be recorded on a map at the time of collection. LFR shall coordinate with the laboratory for the delivery of collected soil samples under chain-of-custody protocols for chemical analysis.

Concrete Sample Collection Methods

In accordance with EPA Site Revitalization Guidance, proposed concrete samples will be collected by drilling a nominal one-inch diameter hole using a rotary impact hammer drill to generate a fine concrete powder suitable for analysis. The powder is to be placed in a laboratory supplied sample container for laboratory analysis. The procedure can be used to collect concrete samples within the upper 6 inches of concrete at each proposed location. As with the soil samples, the concrete samples submitted to the laboratory will be labeled with the sample identification number, the time and date of collection, the analysis requested, and the initials of the sampler. The samples will be stored in an ice-chilled cooler and submitted to the laboratory under strict chain-of-custody protocols. LFR shall coordinate with the laboratory for the delivery of collected soil samples under chain-of-custody protocols for chemical analysis.

Analytical Methods

The samples of the building materials will be submitted for PCB analyses using USEPA SW-846 Method 8082.

Sampling Equipment Decontamination

Sampling equipment cleaning procedures are described in this section. Specifications for standard cleaning materials referred to in this section are as follows:

- <u>Soap</u> will be a standard brand of phosphate-free laboratory detergent such as Liquinox. Use of other detergent must be justified and documented in the field logbooks.
- <u>Tap water</u> may be used from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.
- Organic/analyte free water is defined as tap water that has been treated with activated carbon and deionizing units.

Improperly handled cleaning solutions may easily become contaminated. Storage and application containers must be constructed of the proper materials to ensure their integrity. Following are acceptable materials used containing the specified cleaning solutions:

- <u>Soap</u> must be kept in clean plastic, metal, or glass containers until used. It should be poured directly from the container during use.
- <u>Tap water</u> must be kept in clean tanks, hand pressure sprayers, and squeeze bottles, applied directly from a hose.
- <u>Analyte free water</u> must be stored in clean glass, stainless steel, or plastic containers that can be closed prior to use. It can be applied form plastic squeeze bottles.
- Organic/analyte free water must be stored in clean glass, Teflon®, or stainless steel containers prior to use. It may be applied using Teflon® squeeze bottles.

Sampling Equipment Decontamination Procedure

The following procedures are to be used for all sampling equipment (hand tools or power tools). When appropriate disposable equipment (one time use) will be used :

- 1. Clean with tap water and soap using a brush if necessary to remove particulate matter and surface films.
- 2. Rinse thoroughly with tap water
- 3. Cover the equipment with plastic. Equipment stored overnight should be wrapped in aluminum foil and covered with clean, unused plastic.

AIR SAMPLE ANALYTICAL METHODS

FORMULA: Table 1 CAS: Table 1 RTECS: Table 1 MW: Table 1

METHOD: 1501, Issue 3 **EVALUATION: Full** Issue 1: 15 August 1990 Issue 3: 15 March 2003

OSHA: Table 2 **PROPERTIES:** Table 1

NIOSH: Table 2 ACGIH: Table 2

SYNONYMS: Group A: benzene toluene ethylbenzene o-xylene m-xylene p-xylene

(Synonyms

-MAX:

BIAS:

OVERALL PRECISION (Ŝ,T):

Table 3

in Table 1) Group B: cumene p-tert-butyltoluene α-methylstyrene B-methylstyrene stvrene

> **SAMPLING MEASUREMENT**

> > ANALYTE:

INJECTION

SAMPLER: SOLID SORBENT TUBE **TECHNIQUE:** GAS CHROMATOGRAPHY, FID

(coconut shell charcoal, 100 mg/50 mg)

FLOW RATE: Table 3 **DESORPTION:**

1 mL CS₂, stand 30 min with agitation VOL-MIN: Table 3

VOLUME: 1 μL (Group A: split 5:1;

SHIPMENT: Group B: split 1:1) Routine

SAMPLE **TEMPERATURE**

Table 3

-INJECTION: 250 °C STABILITY: 30 days @ 5°C 300 °C -DETECTOR:

BLANKS: -COLUMN: Group A: 40 °C (10 min) to 230°C 10% of samples

(10 °C/min)

Hydrocarbons listed above

Group B: 35°C (8 min) to 225°C

(10°C/min)

CARRIER GAS: He @ 2.6 mL/min **ACCURACY**

COLUMN: Capillary, fused silica **RANGE STUDIED:** Table 3

Group A: 30m x 0.32-mm ID; 1-µm film

100% PEG or equivalent Table 3

Group B: 30m x 0.53-mm ID; 3-µm film crossbonded® 35% diphenyl 65% dimethyl polysiloxane or equivalent

ACCURACY: Table 3 **CALIBRATION:** Solutions of analytes in CS2

> RANGE: Table 4 ESTIMATED LOD: Table 4

PRECISION (S,): Table 4

APPLICABILITY: This method is for peak, ceiling, and TWA determinations of aromatic hydrocarbons. Interactions between analytes may reduce breakthrough volumes and affect desorption efficiencies. Naphthalene, originally validated in S292 [4], failed to meet acceptable desorption efficiency recovery and storage stability criteria at the levels evaluated in this study. However, the application of this method to naphthalene levels at or near the REL/PEL continues to meet acceptable recovery criteria. Styrene failed to meet acceptable recovery criteria at the two lowest levels evaluated in this study (highest level to meet the criteria was 181 µg/sample).

INTERFERENCES: Under conditions of high humidity, the breakthrough volumes may be reduced. Other volatile organic compounds such as alcohols, ketones, ethers, and halogenated hydrocarbons are potential analytical interferences.

OTHER METHODS: This method updates NMAM 1501 issued on August 15, 1994 [1] which was based upon P&CAM 127 (benzene, styrene, toluene, and xylene) [2]; S22 (p-tert-butyltoluene) [3]; S23 (cumene) [3]; S29 (ethylbenzene) [3]; S26 (α-methylstyrene) [3]; S30 (styrene); S311 (benzene) [4]; S343 (toluene) [4]; and S318 (xylenes) [4].

REAGENTS:

- 1. Carbon disulfide*, low benzene, chromatographic quality.
- 2. Analytes, reagent grade.
- 3. Helium, prepurified and filtered.
- 4. Hydrogen, prepurified and filtered.
- 5. Air, prepurified and filtered.
 - * See SPECIAL PRECAUTIONS

EQUIPMENT:

- Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, flame-sealed ends, containing two sections of activated coconut shell charcoal (front = 100 mg, back = 50 mg) separated by a 2-mm urethane foam plug. A silylated glass wool plug precedes the front section and a 3-mm urethane foam plug follows the back section. Tubes are commercially available.
- 2. Personal sampling pump, 0.01 to 1.0 L/min (Table 3), with flexible connecting tubing.
- 3. Gas chromatograph, FID, integrator, and columns (page 1501-1).
- 4. Autosampler vials, glass, 1.8 mL, with PTFE-lined caps.
- 5. Pipets, 1-mL, and pipet bulb.
- 6. Syringes, 10-μL, 25-μL, and 250-μL.
- 7. Volumetric flasks, 10-mL.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and extremely flammable (flash point = -30°C), benzene is a suspect carcinogen. Prepare standards and samples in a well ventilated hood.

SAMPLING:

- 1. Calibrate each personal sampling pump with a representative sampler in line.
- 2. Break the ends of the sampler immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
- 3. Sample at an accurately known flow rate between 0.01 and 0.2 L/min for a total sample size as shown in Table 3.
- 4. Cap the samplers with plastic (not rubber) caps and pack securely for shipment.

SAMPLE PREPARATION:

- 5. Place the front and back sorbent sections of the sampler tube in separate vials. Include the glass wool plug in the vial along with the front sorbent section.
- 6. Add 1.0 mL eluent to each vial. Attach crimp cap to each vial immediately.
- 7. Allow to stand at least 30 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

- 8. Calibrate daily with at least six working standards from below the LOD to 10 times the LOQ. If necessary, additional standards may be added to extend the calibration curve.
 - a. Add known amounts of analytes to carbon disulfide solvent in 10-mL volumetric flasks and dilute to the mark. Prepare additional standards by serial dilution in 10-mL volumetric flasks.
 - b. Analyze together with samples and blanks (steps 11 through 12).
 - c. Prepare calibration graph (peak area of analyte vs. µg analyte per sample).

- 9. Determine desorption efficiency (DE) at least once for each batch of charcoal used for sampling in the calibration range (step 8).
 - a. Prepare three tubes at each of five levels plus three media blanks.
 - b. Inject a known amount of DE stock solution (5 to 25 µL) directly onto front sorbent section of each charcoal tube with a microliter syringe.
 - c. Allow the tubes to air equilibrate for several minutes, then cap the ends of each tube and allow to stand overnight.
 - d. Desorb (steps 5 through 7) and analyze together with standards and blanks (steps 11 and 12).
 - e. Prepare a graph of DE vs. µg analyte recovered.
- 10. Analyze a minimum of three quality control blind spikes and three analyst spikes to insure that the calibration graph and DE graph are in control.

MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 1501-1. Inject a 1-µL sample aliquot manually using the solvent flush technique or with an autosampler. Note: If peak area is above the linear range of the working standards, dilute with solvent, reanalyze, and apply the appropriate dilution factor in the calculations.

Analyte	Approximate Retention Time (min)
benzene ^a	3.52
toluene ^a	6.13
ethylbenzene ^a	10.65
<u>o</u> -xylene ^a	12.92
<u>m</u> -xylene ^a	11.33
<u>p</u> -xylene ^a	11.04
cumene ^b	18.61
<u>p</u> -tert-butyltoluene ^b	21.45
α-methylstyrene ^b	19.99
β-methylstyrene ^b	20.82
styrene ^b	18.33

^a Separation achieved using a 30-m Stabilwax fused silica capillary colum.

12. Measure peak areas.

CALCULATIONS:

13. Determine the mass, μg (corrected for DE) of analyte found in the sample front (W_f) and back (W_b) sorbent sections, and in the average media blank front (B_f) and back (B_b) sorbent sections.

NOTE: If W_b > W_f/10, report breakthrough and possible sample loss.

14. Calculate concentration, C, of analyte in the air volume sampled, V (L):

$$C = \frac{(W_f + W_b - B_f - B_b)}{V}, mg / m^3$$

NOTE: $\mu g/L = mg/m^3$

^b Separation achieved using a 30-m Rtx-35 fused silica capillary column.

EVALUATION OF METHOD:

The desorption efficiency, at levels ranging from 5 times the LOQ to 0.1x the REL, was determined for each analyte by spiking known amounts (in CS_2) on coconut shell charcoal tubes. Both groups of analytes (A and B) were spiked together on the charcoal sorbent tubes. All analytes, with the exception of styrene and naphthalene, exhibited acceptable desorption efficiency recovery results at all five levels evaluated. Styrene failed to meet the 75% recovery criteria at the 18.1 μ g and 90.6 μ g levels. Naphthalene failed to meet the 75% criteria at all levels evaluated ranging from 48.8 μ g to 976.0 μ g.

Each analyte, at a level approximately 0.05x REL/PEL, was evaluated for its storage stability @ 5°C after 7, 14, and 30 days. All analytes, with the exception of naphthalene, had acceptable recoveries after 30 days storage.

REFERENCES:

- [1] NIOSH [1984]. Hydrocarbons, Aromatic: Method 1501. In: Eller PM, ed. NIOSH Manual of Analytical Methods. 4th rev. ed. Cincinnati, OH: U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control, National Institute for Occupational Safety and Health, DHHS (NIOSH) Publication No. 94-113.
- [2] NIOSH [1977]. NIOSH Manual of Analytical Methods, 2nd. ed., V. 1, P&CAM 127, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A.
- [3] Ibid, V. 2, S22, S23, S25, S26, S29, S30, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
- [4] Ibid, V. 3, S292, S311, S318, S343, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).
- [5] NIOSH [1977]. Documentation of the NIOSH Validation Tests, S22, S23, S25, S26, S29, S30, S292, S311, S318, S343, U.S. Department of Health, Education, and Welfare; Publ. (NIOSH) 77-185.

METHOD WRITTEN BY:

Stephanie M. Pendergrass, NIOSH/DART

TABLE 1. SYNONYMS, FORMULA, MOLECULAR WEIGHT, PROPERTIES

Name/Synonyms	Empirical Formula	Molecular Weight	Boiling Point (°C)	Vapor Pressure @ 25 °C (mm Hg)	(kPa)	Density @ 20 °C (g/mL)
benzene						
CAS #71-43-2						
RTECS CY1400000	C_6H_6	78.11	80.1	95.2	12.7	0.879
<u>p-tert</u> -butyltoluene CAS #98-51-1						
RTECS XS8400000						
1-tert-butyl-4-methylbenzene	$C_{11}H_{16}$	148.25	192.8	0.7	0.09	0.861
cumene						
CAS #98-82-8						
RTECS GR8575000	0.11	400.00	450.4	4.7	0.00	0.000
isopropylbenzene	C_9H_{12}	120.20	152.4	4.7	0.63	0.862
ethylbenzene						
CAS #100-41-4						
RTECS DA0700000	C_8H_{10}	106.17	136.2	9.6	1.28	0.867
α-methylstyrene						
CAS #98-83-9						
RTECS WL5075300						
isopropenylbenzene						
(1-methylethenyl)-benzene	C_9H_{10}	118.18	165.4	2.5	0.33	0.909
0 " 1 "						
β-methylstyrene						
CAS #873-66-5 RTECS DA8400500	СП	118.18	175.0			0.911
RTECS DA8400500	C_9H_{10}	110.10	175.0	_	_	0.911
toluene						
CAS #108-88-3						
RTECS XS5250000	0.11	00.44	440.0	20.4	0.70	0.007
methylbenzene	C_7H_8	92.14	110.6	28.4	3.79	0.867
xylene°	C ₈ H ₁₀	106.17				
CAS #1330-20-7	(<u>ortho</u>)		144.4	6.7	0.89	0.880
RTECS ZE2100000	(<u>meta</u>)		139.1	8.4	1.12	0.864
dimethylbenzene (p-xylene)	(<u>para</u>)		138.4	8.8	1.18	0.861
styrene						
CAS #100-42-5						
RTECS WL3675000				• •		
vinylbenzene	C ₈ H ₈	104.15	145.2	6.1	0.81	0.906

TABLE 2. PERMISSIBLE EXPOSURE LIMITS, PPM

		NIOSH			AC		
Substance	OSHA TWA	TWA	С	STEL	TLV	STEL	mg/m³ per ppm
benzene	1	0.1ª	1		10 ^b		3.19
<u>p-tert</u> -butyltoluene	10	10		20	1		6.06
cumene	50 (skin)	50 (skin)			50 (skin)		4.91
ethylbenzene	100	100		125	100	125	4.34
α-methylstyrene	100	50		100	50	100	4.83
β-methylstyrene	100	50		100	50	100	4.83
toluene	200	100		150	50 (skin)		3.77
<u>o</u> -xylene	100	100°		150	100	150	4.34
<u>m</u> -xylene	100	100			100	150	4.34
<u>p</u> -xylene	100	100			100	150	4.34
styrene	100	50		100	50	100 (skin)	4.26

^a Potential carcinogen

TABLE 3. SAMPLING FLOWRATE^a, VOLUME, CAPACITY, RANGE, OVERALL BIAS AND PRECISION

	Sampling				kthrough ne @	Range at	Overall		
Substance	Flowrate (L/min)	<u>Volume^b (L)</u> MIN MAX		Concentration (L) (mg/m³)		VOL-MIN (mg/m³)	Bias (%)	Precision (Ŝ,,)	Accuracy (±%)
benzene	≤0.20	5	30	>45	149	42 - 165	-0.4	0.059	11.4
<u>p-tert</u> -butyltoluene	≤0.20	1	29	44	112	29 - 119	-10.3	0.071°	20.7
cumene	≤0.20	1	30	>45	480	120 - 480	5.6	0.059	15.2
ethylbenzene	≤0.20	1	24	35	917	222 - 884	-7.6	0.089 ^c	17.1
α-methylstyrene	≤0.20	1	30	>45	940	236 - 943	-7.6	0.061 ^c	16.9
β-methylstyrene	≤ 0.20	1	30	>45	940	236 - 943	-7.6	0.061	16.9
toluene	≤0.20	1	8	12	2294	548 - 2190	1.6	0.052	10.9
xylene (o-,m-,p-)	≤0.20	2	23	35	870	218 - 870	-1.2	0.060	12.2
styrene	<1.00	1	14	21	1710	426 - 1710	-7.9	0.058 ^c	16.7

^b Suspect carcinogen

^c Group I Pesticide

 $^{^{\}rm a}$ Minimum recommended flow is 0.01 L/min. $^{\rm b}$ V $_{\rm Min}$ = minimum sample volume @ OSHA TWA; V $_{\rm Max}$ = maximum sample volume @ OSHA TWA $^{\rm c}$ Corrected value, calculated from data in Reference 5.

TABLE 4. MEASUREMENT RANGE AND PRECISION^a

	_	Measurement			
Substance	LOD (µg/sample)	Range (mg)	Precision (\hat{S}_r)		
benzene	0.5	0.004-0.35	0.013		
<u>p-tert</u> -butyltoluene	1.1	0.013-1.09	0.017ª		
cumene	0.6	0.039-3.46	0.017		
ethylbenzene	0.5	0.045-8.67	0.015		
α-methylstyrene	0.6	0.036-3.57	0.014		
β-methylstyrene	0.6	0.036-0.728	0.014		
toluene	0.7	0.024-4.51	0.022		
o-xylene	0.8	0.044-10.4	0.014		
m-xylene	0.8	0.043-0.864	0.013		
p-xylene	0.7	0.043-0.861	0.015		
styrene	0.4	0.181-8.49	0.014		

^a Corrected value, calculated from data in [5].

POLYCHLOROBIPHENYLS

mixture: $C_{12}H_{10-x}CI_x$ MW: ca. 258 (42% CI; $C_{12}H_7CI_3$); CAS: Table 1 RTECS: Table 1

[where x = 1 to 10] ca. 326 (54% CI; $C_{12}H_5Cl_5$)

0.5 mg/m³ (54% CI)

METHOD: 5503, Issue 2 EVALUATION: PARTIAL Issue 1: 15 February 1984

Revision #1: 15 August 1987 Issue 2: 15 August 1994

OSHA: 1 mg/m³ (42% CI); PROPERTIES: 42% CI: BP 325 to 366 °C; MP -19 °C;

d 1.38 g/mL @ 25 °C;

NIOSH: 0.001 mg/m 3 /10 h (carcinogen) VP 0.01 Pa (8 x 10 5 mm Hg;

ACGIH: 1 mg/m³ (42% CI) (skin) 1 mg/m³) @ 20 °C

0.5 mg/m³ (54% CI) (skin) 54% CI: BP 365 to 390 °C; MP 10 °C; d 1.54 g/mL @ 25 °C; VP

0.0004 Pa (3 x 10⁻⁶ mm Hg; 0.05 mg/m³) @ 20 °C

SYNONYMS: PCB; 1,1'-biphenyl chloro; chlorodiphenyl, 42% CI (Aroclor 1242); and 54% CI (Aroclor 1254)

SAMPLING MEASUREMENT SAMPLER: FILTER + SOLID SORBENT TECHNIQUE: GAS CHROMATOGRAPHY, ECD (63Ni) (13-mm glass fiber + Florisil, 100 mg/50 mg) ANALYTE: polychlorobiphenyls FLOW RATE: 0.05 to 0.2 L/min or less **DESORPTION:** filter + front section, 5 mL hexane; back section, 2 mL hexane VOI -MIN-1 L @ 0.5 mg/m³ -MAX: 50 L **INJECTION** VOLUME: 4-µL with 1-µL backflush SHIPMENT: transfer filters to glass vials after sampling TEMPERATURE-INJECTION: 250 to 300 °C SAMPLE -DETECTOR: 300 to 325 °C -COLUMN: 180 °C STABILITY: unknown for filters; 2 months for Florisil tubes [1] **CARRIER GAS:** N₂, 40 mL/min **BLANKS:** 2 to 10 field blanks per set COLUMN: glass, 1.8 m x 2-mm ID, 1.5% OV-17/1.95% QF-1 on 80/100 mesh Chromosorb WHP **ACCURACY** CALIBRATION: standard PCB mixture in hexane **RANGE STUDIED:** not studied RANGE: 0.4 to 4 µg per sample [2] BIAS: none identified OVERALL PRECISION (\$,T): not evaluated ESTIMATED LOD: 0.03 µg per sample [2] ACCURACY: not determined

APPLICABILITY: The working range is 0.01 to 10 mg/m³ for a 40-L air sample [1]. With modifications, surface wipe samples may be analyzed [3,4].

PRECISION (Š,): 0.044 [1]

INTERFERENCES: Chlorinated pesticides, such as DDT and DDE, may interfere with quantification of PCB. Sulfur-containing compounds in petroleum products also interfere [5].

OTHER METHODS: This method revises methods S120 [6] and P&CAM 244 [1]. Methods S121 [7] and P&CAM 253 [8] for PCB have not been revised.

REAGENTS:

- 1. Hexane, pesticide quality.
- Florisil, 30/48 mesh sieved from 30/60 mesh. After sieving, dry at 105 °C for 45 min. Mix the cooled Florisil with 3% (w/w) distilled water.
- 3. Nitrogen, purified.
- Stock standard solution of the PCB in methanol or isooctane (commercially available).*
 - * See SPECIAL PRECAUTIONS.

EQUIPMENT:

- 1. Sampler: 13-mm glass fiber filter without binders in a Swinnex cassette (Cat. No. SX 0001300, Millipore Corp.) followed by a glass tube, 7 cm long, 6-mm OD, 4-mm ID containing two sections of 30/48 mesh deactivated Florisil. The front section is preceded by glass wool and contains 100 mg and the backup section contains 50 mg; urethane foam between sections and behind the backup section. (SKC 226-39, Supelco ORBO-60, or equivalent) Join the cassette and Florisil tube with PVC tubing, 3/8" L x 9/32" OD x 5/32" ID, on the outlet of the cassette and with another piece of PVC tubing, 3/4" L x 5/16" OD x 3/16" ID, complete the union.
- Personal sampling pump, 0.05 to 0.2 L/min, with flexible connecting tubing.
- 3. Tweezers.
- 4. Vials, glass, 4- and 7-mL, with aluminum or PTFE-lined caps
- 5. Gas chromatograph, electron capture detection (⁶³Ni), integrator and column (page 5503-1).
- 6. Volumetric flasks, 10-mL and other convenient sizes for preparing standards.
- 7. Syringe, 10-µL.

SPECIAL PRECAUTIONS: Avoid prolonged or repeated contact of skin with PCB and prolonged or repeated breathing of the vapor [9-11].

SAMPLING:

- 1. Calibrate each personal sampling pump with a representative sampler in line.
- 2. Break the ends of the Florisil tube immediately before sampling. Connect Florisil tube to Swinnex cassette and attach sampler to personal sampling pump with flexible tubing.
- 3. Sample at an accurately known flow rate between 0.05 and 0.2 L/min for a total sample size of 1 to 50 L.
 - NOTE: At low PCB concentrations, the sampler was found to be efficient when operated at flow rates up to 1 L/min, for 24 hours [4]. Under these conditions, the limit of detection was 0.02 µg/m³.
- 4. Transfer the glass fiber filters to 7-mL vials. Cap the Florisil tubes with plastic (not rubber) caps and pack securely for shipment.

SAMPLE PREPARATION:

- 5. Place the glass wool and 100-mg Florisil bed in the same 7-mL vial in which the filter was stored. Add 5.0 mL hexane.
- In a 4-mL vial, place the 50-mg Florisil bed including the two urethane plugs. Add 2.0 mL hexane
- 7. Allow to stand 20 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

- 8. Calibrate daily with at least six working standards over the range 10 to 500 ng/mL PCB.
 - a. Add known amounts of stock standard solution to hexane in 10-mL volumetric flasks and dilute to the mark.
 - b. Analyze together with samples and blanks (steps 11 and 12).
 - c. Prepare calibration graph (sum of areas of selected peaks vs. ng PCB per sample).
- 9. Determine desorption efficiency (DE) at least once for each lot of glass fiber filters and Florisil used for sampling in the calibration range (step 8). Prepare three tubes at each of five levels plus three media blanks.
 - a. Remove and discard back sorbent section of a media blank Florisil tube.
 - b. Inject known amounts of stock standard solution directly onto front sorbent section and onto a media blank filter with a microliter syringe.
 - c. Cap the tube. Allow to stand overnight.
 - d. Desorb (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
 - e. Prepare a graph of DE vs. µg PCB recovered.
- 10. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and DE graph are in control.

MEASUREMENT:

- 11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 5503-1. Inject sample aliquot manually using solvent flush technique or with autosampler.
 - NOTE 1: Where individual identification of PCB is needed, a procedure using a capillary column may be used [12].
 - NOTE 2: If peak area is above the linear range of the working standards, dilute with hexane, reanalyze and apply the appropriate dilution factor in calculations.
- 12. Sum the areas for five or more selected peaks.

CALCULATIONS:

- 13. Determine the mass, μg (corrected for DE) of PCB found on the glass fiber filter (W) and in the Florisil front (W_f) and back (W_b) sorbent sections, and in the average media blank filter (B) and front (B_f) and back (B_b) sorbent sections.
 - NOTE: If $W_b > W_b/10$, report breakthrough and possible sample loss.
- 14. Calculate concentration, C, of PCB in the air volume sampled, V (L):

$$C = \frac{(W + W_f + W_b - B - B_f - B_b)}{V}, mg/m^3.$$

EVALUATION OF METHOD:

This method uses 13-mm glass fiber filters which have not been evaluated for collecting PCB. In Method S120, however, Aroclor 1242 was completely recovered from 37-mm glass fiber filters using 15 mL isooctane [8,13,14]. With 5 mL of hexane, Aroclor 1016 was also completely recovered from 100-mg Florisil beds after one-day storage [1]. Thus, with no adsorption effect likely on glass fiber filters for PCB, 5 mL hexane should be adequate to completely extract PCB from combined filters and front sorbent sections. Sample stability on glass fiber filters has not been investigated. Breakthrough volume was >48 L for the Florisil tube at 75% RH in an atmosphere containing 10 mg/m ³ Aroclor 1016 [1].

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METHOD REVISED BY:

James E. Arnold, NIOSH/DPSE; S120 originally validated under NIOSH Contract 210-76-0123.

Table 1. General Information.

Compound	CAS	RTECS
Polychlorinated Biphenyls	1336-36-3	TQ1350000
Chlorobiphenyl	27323-18-8	DV2063000
Aroclor 1016 (41% CI)	12674-11-2	TQ1351000
Aroclor 1242 (42% CI)	53469-21-9	TQ1356000
Aroclor 1254 (54% CI)	11097-69-1	TQ1360000

Table 2. Composition of some Aroclors [15].

Major Components	Aroclor 1016	Aroclor 1242	Aroclor 1254
Biphenyl	0.1%	<0.1%	<0.1%
Monochlorobiphenyls	1	1	<0.1
Dichlorobiphenyls	20	16	0.5
Trichlorobiphenyls	57	49	1
Tetrachlorobiphenyls	21	25	21
Pentachlorobiphenyls	1	8	48
Hexachlorobiphenyls	<0.1	1	23
Heptachlorobiphenyls	none detected	<0.1	6
Octachlorobiphenyls	none detected	none detected	none detected

ELEMENTS by ICP (Nitric/Perchloric Acid Ashing)

MW: Table 1 CAS: Table 2 RTECS: Table 2

METHOD: 7300, Issue 3 EVALUATION: PARTIAL Issue 1: 15 August 1990 Issue 3: 15 March 2003

OSHA: Table 2 PROPERTIES: Table 1

NIOSH: Table 2 ACGIH: Table 2

SAMPLE

STABILITY:

ELEMENTS: aluminum* calcium lanthanum

vanadium* antimony* chromium* lithium* potassium tellurium arsenic cobalt* magnesium phosphorus tin yittrium barium thallium copper manganese* selenium zinc beryllium* iron molybdenum* silver titanium zirconium*

nickel

strontium

tungsten*

cadmium lead*

stable

*Some compounds of these elements require special sample treatment.

SAMPLING MEASUREMENT

SAMPLER: FILTER TECHNIQUE: INDUCTIVELY COUPLED ARGON

(0.8-µm, cellulose ester membrane, or 5.0-µm, polyvinyl chloride membrane) PLASMA, ATOMIC EMISSION SPECTROSCOPY (ICP-AES)

FLOWRATE: 1 to 4 L/min ANALYTE: elements above

VOL-MIN: Table 1

-MAX: Table 1 REAGENTS: conc. HNO₃/ conc. HClO₄ (4:1), 5 mL; 2mL increments added as needed

SHIPMENT: routine

CONDITIONS: room temperature, 30 min; 150 °C to near

FINAL

dryness

BLANKS: 2 to 10 field blanks per set

LANKS: 2 to 10 field blanks per set

WAVELENGTH: depends upon element; Table 3

ACCURACY BACKGROUND CORRECTION:

CORRECTION: spectral wavelength shift

RANGE STUDIED: not determined CALIBRATION: elements in 4% HNO₃, 1% HClO₄

BIAS: not determined RANGE: varies with element [1]

OVERALL PRECISION (\hat{S}_{rt}): not determined **ESTIMATED LOD**: Tables 3 and 4

ACCURACY: not determined PRECISION (S): Tables 3 and 4

APPLICABILITY: The working range of this method is 0.005 to 2.0 mg/m³ for each element in a 500-L air sample. This is simultaneous elemental analysis, not compound specific. Verify that the types of compounds in the samples are soluble with the ashing procedure selected.

INTERFERENCES: Spectral interferences are the primary interferences encountered in ICP-AES analysis. These are minimized by judicious wavelength selection, interelement correction factors and background correction [1-4].

OTHER METHODS: This issue updates issues 1 and 2 of Method 7300, which replaced P&CAM 351 [3] for trace elements. Flame atomic absorption spectroscopy (e.g., Methods 70XX) is an alternate analytical technique for many of these elements. Graphite fumace AAS (e.g., 7102 for Be, 7105 for Pb) is more sensitive.

REAGENTS:

- 1. Nitric acid (HNO₃), conc., ultra pure.
- 2. Perchloric acid (HClO₄), conc., ultra pure.*
- Ashing acid: 4:1 (v/v) HNO₃:HCIO₄. Mix 4 volumes conc. HNO₃ with 1 volume conc. HCIO₄.
- Calibration stock solutions, 1000 μg/mL.
 Commercially available, or prepared per instrument manufacturer's recommendation (see step 12).
- Dilution acid, 4% HNO₃, 1% HCIO₄. Add 50 mL ashing acid to 600 mL water; dilute to 1 L.
- 6. Argon.
- 7. Distilled, deionized water.
 - * See SPECIAL PRECAUTIONS.

EQUIPMENT:

- Sampler: cellulose ester membrane filter, 0.8-µm pore size; or polyvinyl chloride membrane, 5.0-µm pore size; 37-mm diameter, in cassette filter holder.
- 2. Personal sampling pump, 1 to 4 L/min, with flexible connecting tubing.
- Inductively coupled plasma-atomic emission spectrometer, equipped as specified by the manufacturer for analysis of elements of interest.
- 4. Regulator, two-stage, for argon.
- 5. Beakers, Phillips, 125-mL, or Griffin, 50-mL, with watchglass covers.**
- 6. Volumetric flasks, 10-, 25-,100-mL., and 1-L**
- 7. Assorted volumetric pipets as needed.**
- 8. Hotplate, surface temperature 150 °C.
 - ** Clean all glassware with conc. nitric acid and rinse thoroughly in distilled water before use.

SPECIAL PRECAUTIONS: All perchloric acid digestions are required to be done in a perchloric acid hood. When working with concentrated acids, wear protective clothing and gloves.

SAMPLING:

- 1. Calibrate each personal sampling pump with a representative sampler in line.
- 2. Sample at an accurately known flow rate between 1 and 4 L/min for a total sample size of 200 to 2000 L (see Table 1) for TWA measurements. Do not exceed a filter loading of approximately 2 mg total dust.

SAMPLE PREPARATION:

- 3. Open the cassette filter holders and transfer the samples and blanks to clean beakers.
- 4. Add 5 mL ashing acid. Cover with a watchglass. Let stand 30 min at room temperature. NOTE: Start a reagent blank at this step.
- 5. Heat on hotplate (120 °C) until ca. 0.5 mL remains.
 - NOTE 1: Recovery of lead from some paint matrices may require other digestion techniques. See Method 7082 (Lead by Flame AAS) for an alternative hotplate digestion procedure or Method 7302 for a microwave digestion procedure.
 - NOTE 2: Some species of Al, Be, Co, Cr, Li, Mn, Mo, V, and Zr will not be completely solubilized by this procedure. Alternative solubilization techniques for most of these elements can be found elsewhere [5-10]. For example, aqua regia may be needed for Mn [6,12].
- 6. Add 2 mL ashing acid and repeat step 5. Repeat this step until the solution is clear.
- 7. Remove watchglass and rinse into the beaker with distilled water.
- 8. Increase the temperature to 150 °C and take the sample to near dryness (ca. 0.5 mL).
- 9. Dissolve the residue in 2 to 3 mL dilution acid.
- 10. Transfer the solutions quantitatively to 25-mL volumetric flasks.
- 11. Dilute to volume with dilution acid.
 - NOTE: If more sensitivity is required, the final sample volume may be held to 10 mL.

CALIBRATION AND QUALITY CONTROL:

12. Calibrate the spectrometer according to the manufacturers recommendations.

NOTE: Typically, an acid blank and 1.0 µg/mL multielement working standards are used. The following multielement combinations are chemically compatible in 4% HNO₃/1% HCIO₄:

- a. Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, La, In, Na
- b. Ag, K, Li, Mg, Mn, Ni, P, Pb, Se, Sr, Tl, V, Y, Zn, Sc
- c. Mo, Sb, Sn, Te, Ti, W, Zr
- d. Acid blank
- 13. Analyze a standard for every ten samples.
- 14. Check recoveries with at least two spiked blank filters per ten samples.

MEASUREMENT:

- 15. Set spectrometer to conditions specified by manufacturer.
- 16. Analyze standards and samples.

NOTE: If the values for the samples are above the range of the standards, dilute the solutions with dilution acid, reanalyze and apply the appropriate dilution factor in the calculations.

CALCULATIONS:

- 17. Obtain the solution concentrations for the sample, C_s (µg/mL), and the average media blank, C_b (µg/mL), from the instrument.
- 18. Using the solution volumes of sample, V_s (mL), and media blank, V_b (mL), calculate the concentration, C (mg/m³), of each element in the air volume sampled, V (L):

$$C = \frac{CsVs - CbVb}{V}, mg / m^3$$

NOTE: $\mu g/L \equiv mg/m^3$

EVALUATION OF METHOD:

Issues 1 and 2

Method, 7300 was originally evaluated in 1981 [2,3]. The precision and recovery data were determined at 2.5 and 1000 μg of each element per sample on spiked filters. The measurements used for the method evaluation in Issues 1 and 2 were determined with a Jarrell-Ash Model 1160 Inductively Coupled Plasma Spectrometer operated according to manufacturer's instructions.

Issue 3

In this update of NIOSH Method 7300, the precision and recovery data were determined at approximately 3x and 10x the instrumental detection limits on commercially prepared spiked filters [12] using 25.0 mL as the final sample volume. Tables 3 and 4 list the precision and recovery data, instrumental detection limits, and analytical wavelengths for mixed cellulose ester (MCE) and polyvinyl chloride (PVC) filters. PVC Filters which can be used for total dust measurements and then digested for metals measurements were tested and found to give good results. The values in Tables 3 and 4 were determined with a Spectro Analytical Instruments Model End On Plasma (EOP)(axial) operated according to manufacturer's instructions.

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METHOD REVISED BY:

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Method originally written by Mark Millson, NIOSH/DART, and R. DeLon Hull, Ph.D., NIOSH/DSHEFS, James B. Perkins, David L. Wheeler, and Keith Nicholson, DataChem Labortories, Salt Lake City, UT.

TABLE 1. PROPERTIES AND SAMPLING VOLUMES

	Proper			
Element	Atomic		Air Volume	e, L @ OSHA PEL
(Symbol)	Weight	MP, °C	MIN	MAX
Silver (Ag)	107.87	961	250	2000
Aluminum (AI)	26.98	660	5	100
Arsenic (As)	74.92	817	5	2000
Barium (Ba)	137.34	710	50	2000
Beryllium (Be)	9.01	1278	1250	2000
Calcium (Ca)	40.08	842	5	200
Cadmium (Cd)	112.40	321	13	2000
Cobalt (Co)	58.93	1495	25	2000
Chromium (Cr)	52.00	1890	5	1000
Copper (Cu)	63.54	1083	5	1000
Iron (Fe)	55.85	1535	5	100
Potassium (K)	39.10	63.65	5	1000
Lanthanum	138.91	920	5	1000
Lithium (Li)	6.94	179	100	2000
Magnesium (Mg)	24.31	651	5	67
Manganese (Mn)	54.94	1244	5	200
Molybdenum (Mo)	95.94	651	5	67
Nickel (Ni)	58.71	1453	5	1000
Phosphorus (P)	30.97	44	25	2000
Lead (Pb)	207.19	328	50	2000
Antimony (Sb)	121.75	630.5	50	2000
Selenium (Se)	78.96	217	13	2000
Tin (Sn)	118.69	231.9	5	1000
Strontium (Sr)	87.62	769	10	1000
Tellurium (Te)	127.60	450	25	2000
Titanium (Ti)	47.90	1675	5	100
Thallium (TI)	204.37	304	25	2000
Vanadium (V)	50.94	1890	5	2000
Tungsten (W)	183.85	3410	5	1000
Yttrium (Y)	88.91	1495	5	1000
Zinc (Zn)	65.37	419	5	200
Zirconium (Zr)	91.22	1852	5	200

TABLE 2. EXPOSURE LIMITS, CAS #, RTECS

Element (Symbol)	CAS#	RTECS	Exposi OSHA	ure Limits, mg/m³ (Ca = c NIOSH	carcinogen) ACGIH
Silver (Ag)	7440-22-4	VW3500000	0.01 (dust, fume, metal)	0.01 (metal, soluble)	0.1 (metal) 0.01 (soluble)
Aluminum (AI)	7429-90-5	BD0330000	15 (total dust) 5 (respirable)	10 (total dust) 5 (respirable fume) 2 (salts, alkyls)	10 (dust) 5 (powders, fume) 2 (salts, alkyls)
Arsenic (As)	7440-38-2	CG0525000	varies	C 0.002, Ca	0.01, Ca
Barium (Ba)	7440-39-3	CQ8370000	0.5	0.5	0.5
Beryllium (Be)	7440-41-7	DS1750000	0.002, C 0.005	0.0005, Ca	0.002, Ca
Calcium (Ca)	7440-70-2		varies	varies	varies
Cadmium (Cd)	7440-43-9	EU9800000	0.005	lowest feasible, Ca	0.01 (total), Ca 0.002 (respir.), Ca
Cobalt (Co)	7440-48-4	GF8750000	0.1	0.05 (dust, fume)	0.02 (dust, fume)
Chromium (Cr)	7440-47-3	GB4200000	0.5	0.5	0.5
Copper (Cu)	7440-50-8	GL5325000	1 (dust, mists) 0.1 (fume)	1 (dust) 0.1 (fume)	1 (dust, mists) 0.2 (fume)
Iron (Fe)	7439-89-6	NO4565500	10 (dust, fume)	5 (dust, fume)	5 (fume)
Potassium (K)	7440-09-7	TS6460000			
Lanthanum	7439-91-0		-	-	
Lithium (Li)	7439-93-2				
Magnesium (Mg)	7439-95-4	OM2100000	15 (dust) as oxide 5 (respirable)	10 (fume) as oxide	10 (fume) as oxide
Manganese (Mn)	7439-96-5	OO9275000	C 5	1; STEL 3	5 (dust) 1; STEL 3 (fume)
Molybdenum (Mo)	7439-98-7	QA4680000	5 (soluble) 15 (total insoluble)	5 (soluble) 10 (insoluble)	5 (soluble) 10 (insoluble)
Nickel (Ni)	7440-02-0	QR5950000	1	0.015, Ca	0.1 (soluble) 1 (insoluble, metal)
Phosphorus (P)	7723-14-0	TH3500000	0.1	0.1	0.1
Lead (Pb)	7439-92-1	OF7525000	0.05	0.05	0.05
Antimony (Sb)	7440-36-0	CC4025000	0.5	0.5	0.5
Selenium (Se)	7782-49-2	VS7700000	0.2	0.2	0.2
Tin (Sn)	7440-31-5	XP7320000	2	2	2
Strontium (Sr)	7440-24-6	-	-	-	
Tellurium (Te)	13494-80-9	WY2625000	0.1	0.1	0.1
Titanium (Ti)	7440-32-6	XR1700000			
Thallium (TI)	7440-28-0	XG3425000	0.1 (skin) (soluble)	0.1 (skin) (soluble)	0.1 (skin)
Vanadium (V)	7440-62-2	YW240000		C 0.05	
Tungsten	7440-33-7	-	5	5 10 (STEL)	5 10 (STEL)
Yttrium (Y)	7440-65-5	ZG2980000	1	N/A	1
Zinc (Zn)	7440-66-6	ZG8600000	_		
Zirconium (Zr)	7440-67-7	ZH7070000	5	5, STEL 10	5, STEL 10

TABLE 3. MEASUREMENT PROCEDURES AND DATA [1]. Mixed Cellulose Ester Filters (0.45 µm)

Element (a)	wavelength nm	Est. LOD μg/ Filter	LOD ng/mL	Certified 3x LOD (b)	% Recovery (c)	Percent RSD (N=25)	Certified 10x LOD (b)	% Recovery (c)	Percent RSD (N=25)
Ag	328	0.042	1.7	0.77	102.9	2.64	3.21	98.3	1.53
ΑI	167	0.115	4.6	1.54	105.4	11.5	6.40	101.5	1.98
As	189	0.140	5.6	3.08	94.9	2.28	12.9	93.9	1.30
Ва	455	0.005	0.2	0.31	101.8	1.72	1.29	97.7	0.69
Ве	313	0.005	0.2	0.31	100.0	1.44	1.29	98.4	0.75
Ca	317	0.908	36.3	15.4	98.7	6.65	64.0	100.2	1.30
Cd	226	0.0075	0.3	0.31	99.8	1.99	1.29	97.5	0.88
Co	228	0.012	0.5	0.31	100.8	1.97	1.29	98.4	0.90
Cr	267	0.020	0.8	0.31	93.4	16.3	1.29	101.2	2.79
Cu	324	0.068	2.7	1.54	102.8	1.47	6.40	100.6	0.92
Fe	259	0.095	3.8	1.54	103.3	5.46	6.40	98.0	0.95
K	766	1.73	69.3	23.0	90.8	1.51	96.4	97.6	0.80
La	408	0.048	1.9	0.77	102.8	2.23	3.21	100.1	0.92
Li	670	0.010	0.4	0.31	110.0	1.91	1.29	97.7	0.81
Mg	279	0.098	3.9	1.54	101.1	8.35	6.40	98.0	1.53
Mn	257	0.005	0.2	0.31	101.0	1.77	1.29	94.7	0.73
Mo	202	0.020	0.8	0.31	105.3	2.47	1.29	98.6	1.09
Ni	231	0.020	0.8	0.31	109.6	3.54	1.29	101.2	1.38
Р	178	0.092	3.7	1.54	84.4	6.19	6.40	82.5	4.75
Pb	168	0.062	2.5	1.54	109.4	2.41	6.40	101.7	0.88
Sb	206	0.192	7.7	3.08	90.2	11.4	12.9	41.3	32.58
Se	196	0.135	5.4	2.3	87.6	11.6	9.64	84.9	4.78
Sn	189	0.040	1.6	0.77	90.2	18.0	3.21	49	21.79
Sr	407	0.005	0.2	0.31	101.0	1.55	1.29	97.3	0.65
Te	214	0.078	3.1	1.54	102.0	2.67	6.40	97.4	1.24
Ti	334	0.050	2.0	0.77	98.4	2.04	3.21	93.4	1.08
TI	190	0.092	3.7	1.54	100.9	2.48	6.40	99.1	0.80
V	292	0.028	1.1	0.77	103.2	1.92	3.21	98.3	0.84
W	207	0.075	3.0	1.54	72.2	10.1	6.40	57.6	14.72
Υ	371	0.012	0.5	0.31	100.5	1.80	1.29	97.4	0.75
Zn	213	0.310	12.4	4.60	102.2	1.87	19.3	95.3	0.90
Zr	339	0.022	0.9	0.31	88.0	19.4	1.29	25	57.87

⁽a) Bold values are qualitative only because of low recovery.

⁽b) Values are certified by Inorganic Ventures INC. at 3x and 10x the approximate instrumental LOD

⁽c) Values reported were obtained with a Spectro Analytical Instruments EOP ICP; performance may vary with instrument and should be independently verified.

TABLE 4. MEASUREMENT PROCEDURES AND DATA [1]. Polyvinyl Chloride Filter (5.0 μ m)

Element	wavelength nm	Est. LOD µg per	LOD ng/mL	Certified 3x LOD	% Recovery	Percent RSD	Certified ¹⁷ 10x LOD	% Recovery	Percent RSD
(c)		filter		(b)	(a)	(N=25)	(b)	(a)	(N=25)
Ag	328	0.042	1.7	0.78	104.2	8.20	3.18		18.9
ΑI	167	0.115	4.6	1.56	77.4	115.24	6.40		20.9
As	189	0.140	5.6	3.10	100.7	5.13	12.70		3.2
Ва	455	0.005	0.2	0.31	102.4	3.89	1.270		2.0
Ве	313	0.005	0.2	0.31	106.8	3.53	1.270		2.1
Ca	317	0.908	36.3	15.6	68.1	12.66	64.00		5.3
Cd	226	0.0075	0.3	0.31	105.2	5.57	1.27		2.8
Co	228	0.012	0.5	0.31	109.3	4.67	1.27	102.8	2.8
Cr	267	0.020	8.0	0.31	109.4	5.31	1.27	103.4	4.1
Cu	324	0.068	2.7	1.56	104.9	5.18	6.40	101.8	2.4
Fe	259	0.095	3.8	1.56	88.7	46.82	6.40	99.1	9.7
K	766	1.73	69.3	23.4	96.4	4.70	95.00	99.2	2.2
La	408	0.048	1.9	0.78	45.5	4.19	3.18	98.8	2.6
Li	670	0.010	0.4	0.31	107.7	4.80	1.27	110.4	2.7
Мg	279	0.098	3.9	1.56	54.8	20.59	6.40	64.5	5.7
Mn	257	0.005	0.2	0.31	101.9	4.18	1.27	99.3	2.4
Мо	202	0.020	8.0	0.31	106.6	5.82	1.27	98.1	3.8
Ni	231	0.020	8.0	0.31	111.0	5.89	1.27	103.6	3.2
Р	178	0.092	3.7	1.56	101.9	17.82	6.40	86.5	10.4
Pb	168	0.062	2.5	1.56	109.6	6.12	6.40	103.2	2.9
Sb	206	0.192	7.7	3.10	64.6	22.54	12.70	38.1	30.5
Se	196	0.135	5.4	2.30	83.1	26.23	9.50	76.0	17.2
Sn	189	0.040	1.6	0.78	85.7	27.29	3.18	52.0	29.4
Sr	407	0.005	0.2	0.31	71.8	4.09	1.27	81.2	2.7
Te	214	0.078	3.1	1.56	109.6	7.49	6.40	97.3	3.8
Ti	334	0.050	2.0	0.78	101.0	9.46	3.18	92.4	5.5
TI	190	0.092	3.7	1.56	110.3	4.04	6.40	101.9	2.0
V	292	0.028	1.1	0.78	108.3	3.94	3.18	102.5	2.6
W	207	0.075	3.0	1.56	74.9	15.79	6.40	44.7	19.6
Υ	371	0.012	0.5	0.31	101.5	3.63	1.27	101.4	2.5
Zn	213	0.310	12.4	4.70	91.0	68.69	19.1	101.0	9.6
Zr	339	0.022	0.9	0.31	70.7	54.20	1.27		42.1

⁽a) Values reported were obtained with a Spectro Analytical Instruments EOP ICP; performance may vary with instrument and should be independently verified.

⁽b) Values are certified by Inorganic Ventures INC. at 3x and 10x the approximate instrumental LOD [12].

⁽c) Bold values are qualitative only because of low recovery. Other digestion techniques may be more appropriate for these elements and their compounds.