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**EVALUATION OF WEAPONS' COMBUSTION PRODUCTS
IN ARMORED VEHICLES**

Final Report

Appendix A: Sampling and Analysis Methods

Appendix B: Analytical Data

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The U.S. Army Biomedical Research and Development Laboratory defined an extensive research program to address the generation of potentially toxic propellant combustion products in crew compartments of armored vehicles during weapons firing. The major objectives of the research were (1) to determine the presence and concentration of propellant combustion products, (2) to determine potential crew exposure to these combustion products, and (3) to assess the efficacy of field monitoring in armored vehicles. To achieve these goals, air monitoring was conducted in selected armored vehicle types, i.e., M109, M60, M3, M1, at several Army installations. Auxiliary information concerning the specific munitions fired and the Training and Doctrine Command (TRADOC) or Forces Command (FORSCOM) firing scenarios was collected so that a comparison of pollutant concentrations generated by specific weapons both within vehicle types and between vehicle types could be made.				
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19. ABSTRACT (continued)

The characterization of the airborne combustion products in armored vehicles during weapons firing exercises was facilitated by the use of optimized sampling and analysis methods to permit the collection of large sample volumes and thus enhance the ability to identify and quantify trace pollutants. Inorganic gases and members of several compound classes were found in one or more armored vehicles during firing:

WEAPON POLLUTANTS

Carbon Monoxide	Vapor Phase Organics
Ammonia	Aldehydes
Carbon Dioxide	Polycyclic Aromatic Hydrocarbons (PAHs)
Hydrogen Cyanide	Nitro-PAHs
Hydrogen Sulfide	Particulates (Total, Respirable)
Nitrogen Oxides	Metals
Sulfur Dioxide	

On a few occasions, carbon monoxide was observed to exceed the NRC recommended emergency and continuous exposure limit, which is 1500 ppm, for up to 40 minutes in tanks (M1 and M60). Carbon monoxide was observed to exceed 2000 ppm for shorter periods in all vehicles except the M3, where the peak level was 1300 ppm. Mean carbon monoxide concentrations ranged from 3.6 to 4.7 ppm in the non-tank vehicles (M3 and M109) and from 35 to 43 ppm in the tanks. With few exceptions, the maximum concentrations of all other pollutants in all vehicles were less than their respective threshold limit values and short-term emergency exposure levels.

The peak instantaneous concentrations of pollutants generated during weapon firing, and to which crewmen such as the ammunition loader are exposed, may exceed 500 times the average concentrations inside vehicles. These peak excursions are very localized and short-lived. Carbon monoxide, which is a major combustion product, is observed at statistically significantly higher mean and peak concentrations in tanks (M1; M60) compared to non-tank vehicles (M3; M109). All other pollutants are generally observed at higher levels in tanks than non-tank vehicles, although the statistical significance of this observation is affected by sample size and variability.

The rigor and complexity of field sampling in armored vehicles during firing exercises can be successfully dealt with if proper planning and careful limitation of the duration of sampling is followed. The use of sampling vests for breathing zone measurements is feasible although subject to failure due to the activity of the subject.

Classification/_____
Priority Codes_____
and/or_____
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A-1

APPENDIX A
SAMPLING AND ANALYSIS METHODS

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FORMULA: Table 1

POLYNUCLEAR AROMATIC HYDROCARBONS

M.W.: Table 1

METHOD: 5506

ISSUED: 5/15/85

OSHA: proposed for B[a]P: 0.2 $\mu\text{g}/\text{m}^3$
ACGIH: suspect carcinogen (B[a]P)

PROPERTIES: Table 1

COMPOUNDS:	acenaphthene	benzo[ghi]perylene	fluorene
	acenaphthylene	benzo[a]pyrene	indeno[1,2,3-cd]pyrene
	anthracene	benzo[e]pyrene	naphthalene
	benz[a]anthracene	chrysene	phenanthrene
	benzo[b]fluoranthene	dibenz[a,h]anthracene	pyrene
	benzo[k]fluoranthene	fluoranthene	

SYNONYMS: PAH; PNA; also see Table 2.

SAMPLING	MEASUREMENT
SAMPLER: FILTER + SORBENT (2- μm , 37-mm PTFE + washed XAD-2, 100 mg/50 mg)	METHOD: HPLC, FLUORESCENCE/UV DETECTION ANALYTE: compounds above
FLOW RATE: 2 L/min	EXTRACTION: 5 mL organic solvent appropriate to sample matrix (step 7)
VOL-MIN: 200 L -MAX: 1000 L	COLUMN: 15 cm x 4.6 mm, reverse phase, 5- μm C ₁₈
SHIPMENT: transfer filters to culture tubes; wrap sorbent and culture tubes in Al foil; ship @ 0 °C	INJECTION VOLUME: 10 to 50 μL MOBILE PHASE: H ₂ O/CH ₃ CN gradient @ ambient temperature
SAMPLE STABILITY: unknown; protect from heat and UV radiation	FLOW RATE: 1.0 mL/min
FIELD BLANKS: 10% (>3) of samples MEDIA BLANKS: 6 to 10	DETECTORS: UV @ 254 nm; fluorescence @ 340 nm (excitation), 425 nm (emission)
AREA SAMPLES: 8 replicates on preweighed filters for solvent selection	CALIBRATION: external standards in CH ₃ CN RANGE, LOD AND PRECISION (s_p): EVALUATION OF METHOD
ACCURACY	
RANGE STUDIED, BIAS, AND OVERALL PRECISION (s_p): not measured	

APPLICABILITY: The working range for B[a]P is 1 to 50 $\mu\text{g}/\text{m}^3$ for a 400-L air sample. Specific sample sets may require modification in filter extraction solvent, choice of measurement method, and measurement conditions (see EVALUATION OF METHOD).

INTERFERENCES: Any compound which elutes at the same HPLC retention time may interfere. Heat, ozone, NO₂, or UV light may cause sample degradation.

OTHER METHODS: This revises P&CAM 206 and 251 [1]. The spectrophotometric methods, P&CAM 184 and 186 [1], have not been revised. Also see Method 5515 (GC).

5/15/85

REAGENTS:

1. Filter extraction solvent: benzene,* cyclohexane, methylene chloride, or other appropriate solvents, pesticide grade grade (step 7).
2. Water, distilled, deionized, degassed.
3. Acetonitrile, HPLC grade, degassed.
4. PAH reference standards,* appropriate to the PAH-containing matrix sampled.
5. Calibration stock solution, 0.25 mg/mL.* Check purity of each PAH reference standard by GC/FID, HPLC/fluorescence and/or melting point. Purify, if necessary, by recrystallization. Weigh 25 mg of each PAH into a 100-mL volumetric flask; dilute to volume with acetonitrile. Stable six months if refrigerated and protected from light.

*See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler:
 - a. Filter. PTFE-laminated membrane filter, 2- μ m pore size, 37-mm diameter (ZEFLOUR, Membrana, Pleasanton, CA or equivalent), backed by a gasket (37-mm OD, 32-mm ID) cut from a cellulose support pad, in cassette filter holder.
NOTE 1: If sampling is to be done in bright sunlight, use opaque or foil-wrapped cassettes to prevent sample degradation.
NOTE 2: Take filters to be preweighed from the filter package and allow to equilibrate 24 hrs with laboratory atmosphere before taring.
 - b. Sorbent tube, connected to filter with minimum length PVC tubing. Plastic caps are required after sampling. Washed XAD-2 resin (front = 100 mg; back = 50 mg) (Supelco ORBO 43 or equivalent). Pressure drop at 2 L/min airflow 1.6 to 2 kPa (15 to 20 cm H₂O).
2. Personal sampling pump capable of operating for 8 hrs at 2 L/min, with flexible connecting tubing.
3. Aluminum foil.
4. Vial, scintillation, 20-mL, glass, PTFE-lined cap.
5. Refrigerant, bagged.
6. Culture tubes, PTFE-lined screw cap, 13-mm x 100-mm.
7. Forceps.
8. Filters, 0.45- μ m, PTFE or nylon (for filtering sample solutions).
9. Pipet, 5-mL.
10. Syringe or micropipets, 1- to 100- μ L.
11. Ultrasonic bath.
12. HPLC, with gradient capability, fluorescence (excitation @ 240 nm, emission @ 425 nm) and UV (254 nm) detectors in series, electronic integrator, and column [HC-ODS-SILX (Perkin-Elmer Corp.), Vydac 201TP (The Separations Group) or equivalent; see page 5506-1].
13. Volumetric flasks, 10- and 100-mL.
14. Lighting in laboratory: incandescent or UV-shielded fluorescent.
15. Kuderna-Danish extractor.

SPECIAL PRECAUTIONS: Treat benzene and all polynuclear aromatic hydrocarbons as carcinogens. Neat compounds should be weighed in a glove box. Spent samples and unused standards are toxic waste. Regularly check counter tops and equipment with "black light" for fluorescence as an indicator of contamination by PAH.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Take personal samples at 2 L/min for a total sample size of 200 to 1000 L. Take a concurrent set of eight replicate area samples at 2 to 4 L/min on preweighed, 2- μ m PTFE filters in an area of highest expected PAH concentration.
NOTE: The area samples are needed for solvent selection (step 7).
3. Immediately after sampling, transfer the filter carefully with forceps to a scintillation vial. Hold filter at edge to avoid disturbing the deposit. Cap the scintillation vial and wrap it in aluminum foil.
NOTE: This step is necessary to avoid loss of analytes due to sublimation and degradation by light.
4. Cap the sorbent tube and wrap it in aluminum foil.
5. Ship to laboratory in insulated container with bagged refrigerant.

SAMPLE PREPARATION:

NOTE: UV light may degrade PAH. Use yellow, UV-absorbing shields for fluorescent lights or use incandescent lighting.

6. Refrigerate samples upon receipt at laboratory.
7. Determine optimum extraction solvent.
 - a. Allow the preweighed area filter samples to equilibrate 24 hrs with the laboratory atmosphere.
 - b. Weigh the area filters. Determine total weight collected on each.
 - c. Extract the first pair of area filters with acetonitrile, the second with benzene, the third with cyclohexane, and the fourth with methylene chloride, according to step 8.
NOTE: Use alternate solvents, if appropriate. PAH of interest may be entrained within, and adsorbed by, particulate matter collected on the filter. It is necessary to determine the solvent which maximizes recovery of the PAH from each sample matrix. For example, methylene chloride [2,3] and benzene:ethanol (4:1 v/v) [4] have been recommended for extraction of PAH from diesel exhaust particulate.
 - d. Analyze the extracts for the PAH of interest (steps 10 through 18). Normalize the total mass of PAH found to the mass of sample collected.
 - e. Choose the solvent which gives the highest recovery of PAH of interest. Use the solvent chosen to extract the personal filter samples.
8. Extract filters.
 - a. Add 5.0 mL of the solvent chosen in step 7 to each scintillation vial containing a filter. Start media and reagent blanks at this step.
 - b. Cap and let sit 15 to 20 min in an ultrasonic bath.
NOTE 1: Soxhlet extraction may be required when large amounts of highly adsorptive particulate matter (e.g., fly ash or diesel soot) are present.
NOTE 2: The sample must be dissolved in acetonitrile for chromatography. If needed, perform solvent exchange as follows:
CAUTION: To avoid loss of volatile components, do not allow the sample to go to dryness at any time.
 - (1) After filtration (step 10), take the sample to near dryness in a Kuderna-Danish extractor.
 - (2) Add ca. 1 mL acetonitrile, take to near dryness, and adjust final volume to 1.0 mL with acetonitrile and filter again.
9. Desorb PAH from sorbent.
 - a. Score each sorbent tube with a file in front of the front (larger) sorbent section. Break tube at score line.

- b. Transfer glass wool plug and front sorbent section to a culture tube. Discard the foam plug. Transfer back sorbent section to a second culture tube.
 - c. Add 5.0 mL acetonitrile to each culture tube. Cap the culture tubes.
 - d. Allow samples to sit for 30 min. Swirl occasionally.
10. Filter all sample extracts through an 0.45- μ m membrane filter.

CALIBRATION AND QUALITY CONTROL:

11. Calibrate daily with at least five working standards.
 - a. Dilute aliquots of calibration stock solution with acetonitrile in 10-mL volumetric flasks (e.g., to 2.5, 0.5, 0.1, 0.02, and 0.002 μ g/mL).
 - b. Intersperse working standards and samples in the measurements.
 - c. Prepare calibration graphs (peak area vs. μ g of each PAH per sample).
12. Recovery and desorption efficiency.
 - a. Determine recovery (R) from filters and desorption efficiency (DE) from sorbent tubes at least once for each lot of filters and sorbent tubes used in the range of interest.
 - (1) Filters. Using a microliter syringe or micropipette, spike four filters at each of five concentration levels with a mixture of the analytes. Allow the filters to dry in the dark overnight. Analyze the filters (steps 8, 10, and 14 through 16). Prepare graphs of R vs. amounts found.

NOTE: This step may not be used for some highly adsorptive particulate matrices for which calibration by the method of standard additions may be more accurate.
 - (2) Sorbent tubes. Transfer an unused front sorbent section to a culture tube. Prepare a total of 24 culture tubes in order to measure DE at five concentration levels plus blanks in quadruplicate. Using a microliter syringe or micropipette, add calibration stock solution directly to sorbent. Cap culture tubes and allow to stand overnight. Analyze (steps 9, 10, and 14 through 16). Prepare graphs of DE vs. amounts found.
 - b. Check R and DE at two levels for each sample set, in duplicate. Repeat determination of R and DE graphs if checks do not agree to within $\pm 5\%$ of DE graph.
13. Analyze at least three field blanks for each sample medium.

MEASUREMENT:

14. Set HPLC according to manufacturer's recommendations and to conditions on page 5506-1. Equilibrate column at 60% CH₃CN/40% H₂O at 1.0 mL/min for 15 min before injecting first sample.
15. Inject sample aliquot. Start mobile phase gradient:
 - a. Linear gradient 60% CH₃CN to 100% CH₃CN, 20 min.
 - b. Hold at 100% CH₃CN for 20 min.

NOTE: Hold longer if necessary to prevent carryover of background, e.g., from coal dust.

 - c. Linear gradient to initial condition, 5 min.
16. Measure peak areas.

NOTE 1: Approximate retention times appear in Table 3.

NOTE 2: If peak area is above the calibration range, dilute with appropriate solvent, reanalyze, and apply dilution factor in calculations.

NOTE 3: If sample has many interferences, additional sample cleanup may be necessary. Many cleanup procedures have been published. Liquid-liquid partitioning between cyclohexane and nitromethane [5,6] is widely used, but other techniques may be more appropriate for specific samples.

CALCULATIONS:

17. Read the mass, μg (corrected for R or DE) of each analyte found on the filter (W) and front sorbent (W_f) and back sorbent (W_b) sections, and on the average media blank filter (B) and front sorbent (B_f) and back sorbent (B_b) sections from the calibration graphs.
18. Calculate concentration, C ($\mu\text{g}/\text{m}^3$), in air as the sum of the particulate concentration and the vapor concentration using the actual air volume sampled, V (L).

$$C = \frac{(W - B + W_f + W_b - B_f - B_b) \cdot 10^3}{V}, \mu\text{g}/\text{m}^3$$

NOTE: W_f and W_b include analyte originally collected on the filter as particulate, then volatilized during sampling. This can be a significant fraction for many PAH (e.g., fluoranthene, naphthalene, fluorene, anthracene, phenanthrene).

EVALUATION OF METHOD:

The fluorescence detector used in this method is both sensitive and selective. The detector can "see" as little as 50 pg of many PAH injected on the column. LODs for the 17 analytes range from 50 to 350 ng per sample. It does not respond to non-fluorescent molecules such as aliphatics. The method is, therefore, most amenable to determination of trace amounts of PAH in mixtures of aliphatic compounds. Successful applications include: aluminum reduction facilities, asphalt fume, coal gasification plants, coal liquefaction plants, coal tar pitch, coke oven emissions, creosote treatment facilities, diesel exhaust, graphite electrode manufacturing, petroleum pitch, and roofing tearoff operations.

This method has been evaluated by analyzing spiked filters, spiked sorbent tubes, and complete spiked sampling trains through which were drawn 500 L of air [7]. Each of the three groups was spiked with each analyte at two concentration levels in sextuplicate. Particular note should be made that the effect of particulate matter has not been evaluated, and every sampling matrix is unique. The data on the following page were obtained on spiked samplers stored refrigerated in the dark for three months followed by measurement with HPLC.

COMPOUND	CALIBRATION RANGE (μg per sample)	LOO (μg per sample)	MEASUREMENT PRECISION	
			SPIKED ^a	SPIKED + AIR ^b
1. ACENAPHTHENE	2.0 - 13	0.8	.058 S	.093 (50)
2. ACENAPHTHYLENE	1.0 - 100	0.35	.032 S	.075 (100)
3. ANTHRACENE	0.4 - 13	0.05	.039 S	.037 (5)
4. BENZ[a]ANTHRACENE	0.4 - 13	0.15	.032 F	.084 (5)
5. BENZO[b]FLUORANTHENE	0.4 - 12	0.1	.027 F	.028 (10)
6. BENZO[k]FLUORANTHENE	0.4 - 13	0.15	.025 F	.027 (1)
7. BENZO[ghi]PERYLENE	0.5 - 25	0.2	.031 F	.029 (10)
8. BENZO[a]PYRENE	0.4 - 14	0.2	.027 F	.029 (5)
9. BENZO[e]PYRENE	0.5 - 13	0.2	(c)	(c)
10. CHRYSENE	0.4 - 12	0.15	.039 F	.024 (5)
11. DIBENZ[a,h]ANTHRACENE	0.5 - 25	0.2	.026 F	.029 (10)
12. FLUORANTHENE	0.4 - 13	0.15	.026 S	.050 (10)
13. FLUORENE	0.7 - 13	0.25	.031 S	.090 (10)
14. INDENO[1,2,3-cd]PYRENE	0.5 - 12	0.2	.044 F	.032 (10)
15. NAPHTHALENE	0.6 - 13	0.25	.041 S	.125 (50)
16. PHENANTHRENE	0.4 - 13	0.1	.036 S	.070 (2)
17. PYRENE	0.5 - 13	0.2	(c)	(c)

^aRSO for filter (F) where volatilization is nil or for sorbent (S) where substantial volatilization may occur during sampling.

^bRSO determined at the μg level shown in parenthesis for a spiked filter followed by a sorbent tube. After spiking, laboratory air was drawn through the sampling train at 2 L/min for 4 hrs.

^cNot determined.

REFERENCES:

- [1] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).
- [2] Breuer, G. M. Anal. Lett., **17**(A11), 1293-1306 (1984).
- [3] Zweidinger, R. B., S. B. Tejada, D. Dropkins, J. Huisingsh, and L. Claxton. "Characterization of Extractable Organics in Diesel Exhaust Particulate," paper presented at Symposium on Diesel Particulate Emissions Measurement Characterization, Ann Arbor, MI (1978).
- [4] Swarin, S. J. and R. L. Williams. "Liquid Chromatographic Determination of Benzo[a]pyrene in Diesel Exhaust Particulate: Verification of the Collection and Analytical Methods," Polynuclear Aromatic Hydrocarbons: Physical and Biological Effects, Bjorseth, A. and Dennis, Eds., Battelle Press, 771-790 (1980).
- [5] Wise, S. A., et al. "Analytical Methods for the Determination of Polycyclic Aromatic Hydrocarbons on Air Particulate Matter," Polynuclear Aromatic Hydrocarbons: Physical and Biological Chemistry, Cooke, Dennis and Fisher, Eds., Battelle Press, 919-929 (1982).
- [6] Novotny, M., M. L. Lee and K. D. Bartle. J. Chromatog. Sci., **12**, 606-612 (1974).
- [7] Backup Data Report for Method 5506, Analytical Report for NIOSH Sequence 4170 (NIOSH, unpublished, March 16, 1984).
- [8] Studt, P., Liebigs Ann. Chem., 528 (1978).
- [9] Clar, E. Polycyclic Hydrocarbons, Academic Press (1964).
- [10] Handbook of Chemistry and Physics, 62nd ed., CRC Press (1982).

METHOD REVISED BY: B. R. Belinky and E. J. Slick, NIOSH/DPSE.

Table 1. Formulae and physical properties.

COMPOUND (by M.W.)	EMPIRICAL FORMULA	MOLECULAR WEIGHT	DETECTOR	MELTING POINT (°C)	BOILING POINT (°C)*	REF.
1. NAPHTHALENE	C ₁₀ H ₈	128.17	UV	80	218	[9]
2. ACENAPHTHYLENE	C ₁₂ H ₈	152.20	UV	92-93	265-275	[10]
3. ACENAPHTHENE	C ₁₂ H ₁₀	154.21	UV	96.2	279	[10]
4. FLUORENE	C ₁₃ H ₁₀	166.22	UV	116	293-295	[9]
5. ANTHRACENE	C ₁₄ H ₁₀	178.23	UV	218	340	[9]
6. PHENANTHRENE	C ₁₄ H ₁₀	178.23	UV	100	340	[9]
7. FLUORANTHENE	C ₁₆ H ₁₀	202.26	FL	110	—	[9]
8. PYRENE	C ₁₆ H ₁₀	202.26	FL	156	399	[9]
9. BENZ[a]ANTHRACENE	C ₁₈ H ₁₂	228.29	FL	158-159	—	[9]
10. CHRYSENE	C ₁₈ H ₁₂	228.29	UV	255-256	—	[9]
11. BENZO[b]FLUORANTHENE	C ₂₀ H ₁₂	252.32	FL	168	—	[9]
12. BENZO[k]FLUORANTHENE	C ₂₀ H ₁₂	252.32	FL	217	480	[10]
13. BENZO[a]PYRENE	C ₂₀ H ₁₂	252.32	FL	177	—	[9]
14. BENZO[e]PYRENE	C ₂₀ H ₁₂	252.32	FL	178-179	—	[9]
15. BENZO[ghi]PERYLENE	C ₂₂ H ₁₂	276.34	FL	273	—	[9]
16. INDENO[1,2,3-cd]PYRENE	C ₂₂ H ₁₂	276.34	FL	161.5-163	—	[8]
17. DIBENZ[a,h]ANTHRACENE	C ₂₂ H ₁₄	278.35	FL	262	—	[9]

*Many of these compounds will sublime.

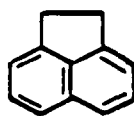
Table 2. Synonyms.

COMPOUND (alphabetically)	SYNONYMS
1. ACENAPHTHENE	CAS# 83-32-9
2. ACENAPHTHYLENE	CAS# 208-96-8
3. ANTHRACENE	CAS# 120-12-7
4. BENZ[a]ANTHRACENE	1,2-benzanthracene; benzo[b]phenanthrene; 2,3-benzophenanthrene; tetraphene; CAS# 56-55-3
5. BENZO[b]FLUORANTHENE	3,4-benzofluoranthene; 2,3-benzofluoranthene; benz[e]acephenanthrylene; B[b]F; CAS# 205-99-2
6. BENZO[k]FLUORANTHENE	11,12-benzofluoranthene; CAS# 207-08-9
7. BENZO[ghi]PERYLENE	1,12-benzoperylene; CAS# 191-24-2
8. BENZO[a]PYRENE	3,4-benzopyrene; 6,7-benzopyrene; B[a]P; BP; CAS# 50-32-8
9. BENZO[e]PYRENE	1,2-benzopyrene; 4,5-benzopyrene; B[e]P; CAS# 192-97-2
10. CHRYSENE	1,2-benzophenanthrene; benzo[a]phenanthrene; CAS# 218-01-9
11. DIBENZ[a,h]ANTHRACENE	1,2,5,6-dibenzanthracene; CAS# 53-70-3
12. FLUORANTHENE	benzo[jk]fluorene; CAS# 206-44-0
13. FLUORENE	CAS# 86-73-7
14. INDENO[1,2,3-cd]PYRENE	2,3-phenylenepyrene; CAS# 193-39-5
15. NAPHTHALENE	naphthene; CAS# 91-20-3
16. PHENANTHRENE	CAS# 85-01-8
17. PYRENE	benzo[def]phenanthrene; CAS# 129-00-0

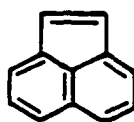
Table 3. Approximate PAH retention times.

<u>COMPOUND</u>	<u>RETENTION TIME (min)*</u>
1. NAPHTHALENE	2.4
2. ACENAPHTHALENE	2.8
3. ACENAPHTHENE	3.6
4. FLUORENE	3.9
5. PHENANTHRENE	4.7
6. ANTHRACENE	5.8
7. FLUORANTHENE	6.8
8. PYRENE	7.7
9. BENZ[a]ANTHRACENE	11.2
10. CHRYSENE	12.1
11. BENZO[e]PYRENE	14.0
12. BENZO[b]FLUORANTHENE	14.8
13. BENZO[k]FLUORANTHENE	16.5
14. BENZO[a]PYRENE	17.3
15. DIBENZ[a,h]ANTHRACENE	20.0
16. BENZO[ghi]PERYLENE	20.0
17. INDENO[1,2,3-cd]PYRENE	21.2

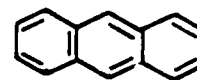
*NOTE: Determined with a Perkin-Elmer HC-ODS-SILX column. Actual retention times will vary with individual columns and column age.



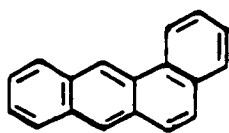
ACENAPHTHENE



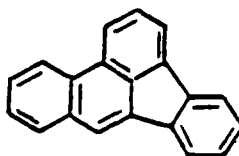
ACENAPHTHYLENE



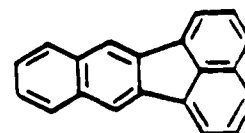
ANTHRACENE



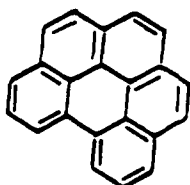
BENZ(a)ANTHRACENE



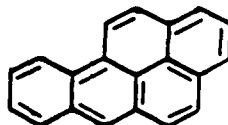
BENZO(b)FLUORANTHENE



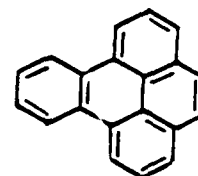
BENZO(k)FLUORANTHENE



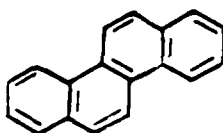
BENZO(g h i)PERYLENE



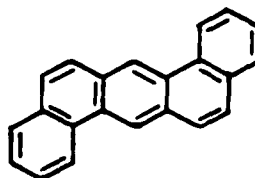
BENZO(a)PYRENE



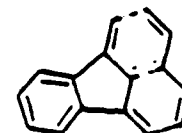
BENZO(e)PYRENE



CHRYSENE



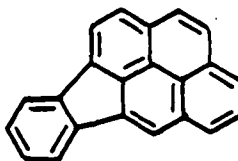
DIBENZ(a,h)ANTHRACENE



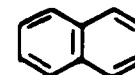
FLUORANTHENE



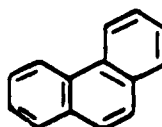
FLUORENE



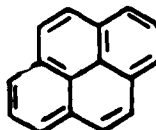
INDENO(1,2,3-c d)PYRENE



NAPHTHALENE



PHENANTHRENE



PYRENE

Figure 1. Structures of PAH.

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ORGANICS SAMPLING AND ANALYSIS METHOD

INSTRUCTIONS FOR USING GC/MS TUBES

A. PREPARATION OF GC/MS AIR SAMPLE TUBES

1. Cut 6 mm O.D. Pyrex tubing into $7 \pm 1/16$ inch lengths.
2. Fire polish both ends of each length.
3. Check to ensure that each length will fit into the appropriate concentrator. Glass tubing is not perfectly uniform, so some lengths may fit and others may not. Before inserting the tubes into the concentrator, wipe them clean with a Kimwipe®.
4. Using 65 mg (1 1/2 inch) of 20/35 mesh Tenax® GC and 140 mg (1 inch) of Ambersorb® 340 (Rohm and Haas) fill the tube as indicated by the following diagram:

	GS	AMERSORB®	GW	TENAX®	GW	
--	----	-----------	----	--------	----	--

The Ambersorb must be at least 2 inches from the end of the tube. the adsorbents are retained and separated in the tube using silanized (DMCS) treated glass wool plugs. The glass wool (GW) prevents any shifting of the adsorbents at sampling flow rate.

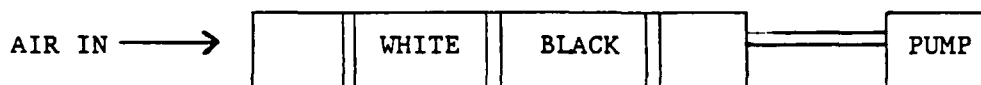
5. Check the pressure drop across each tube and pack the glass wool such that the pressure drop across each tube is approximately equal.
6. Condition each tube for at least 2 hours at $270 \pm 10^{\circ}\text{C}$ while passing 25 ± 5 mL/minute of nitrogen or helium (ultra high purity) through the tube in the direction from Ambersorb® to Tenax®.

7. After the tube is conditioned, care must be taken in handling the tube. Use a Kimwipe® when handling; do not handle the tube by hand. Also, avoid exposing it to organic vapors, e.g., solvents, cigarette smoke, hand lotion, etc.
8. After disconnecting the tube from the oven chamber, insert either end into a clean, dry 200 mm long, screw cap test tube. Two sheets of 5x8 1/2" Kimwipe® should first be inserted into the test tube and compressed to the bottom of the tube to provide a snug fit of the air sample tube and test tube after the screw cap is tightened.
9. These sample tubes can be reused. Before they are reused, make sure there are no gaps in the adsorbents, check step 4, and then repeat steps 5, 6, 7, and 8.

B. SAMPLING

1. Mark several tubes as "FIELD BLANK - DO NOT USE." These are control tubes used to indicate contamination in handling or storage and should not be opened or used in sampling.
2. Use one tube as a "CALIBRATION TUBE." This tube can be used for calibrating pumps. Also, do not use the tube for actual sampling of air streams.
3. "SAMPLE TUBE." Do not handle the glass portion of the tube by hand. Use a Kimwipe® or equivalent for handling.
 - a. Obtain three samples per individual using a low flow air sampling pump and triple variable flow controller manifold. Adjust the manifold to provide 50 mL/min at each tube holder (each manifold inlet can be adjusted and calibrated independently).

- b. Keep the sample tubes in the screw cap test tube until just before sampling and return them to the test tube as soon as possible after sampling. Close the screw cap tightly. Affix labels identifying the tubes on the outer screw cap tube rather than the actual GC/MS tube.
- c. It is essential to attach the tubes to the pump with the dark adsorbent adjacent to the pump as shown below:



4. While GC/MS tubes are a powerful tool for detecting a large number of organic compounds in air, these tubes are not universal samplers. Certain compounds such as formaldehyde, carbon monoxide, and hydrochloric acid for example cannot be collected and/or detected with these tubes.

C. GC/MS PROCEDURE FOR ANALYSIS OF AIR SAMPLE TUBES

1. The tubes are kept refrigerated until analysis.
2. The tubes are taken one at a time from the refrigerator. One microliter of a 3-component spike mixture is added to the Tenax GC adsorbent using a syringe and the tube is inserted into a concentrator unit. (The spike mixture assures that the analysis is performed satisfactorily.)
3. The tube is thermally desorbed at 270°C and products given off the tube are directed to a capillary gas chromatographic column programmed from 20°C to 280°C at 5°C/min.
4. The column effluent is passed to a mass spectrometer/data system capable of scanning a 20-400 amu mass range each second and storing the mass spectral information for subsequent data analysis.

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ELEMENTS (ICP)

ELEMENTS (ICP)

M.W.: Table 1

METHOD: 7300
ISSUED: 2/15/84

OSHA/NIOSH/ACGIH: Table 1

PROPERTIES: Table 1

ELEMENTS: aluminum	cobalt	manganese	silver	tungsten
arsenic	copper	molybdenum	sodium	vanadium
beryllium	iron	nickel	tellurium	yttrium
cadmium	lead	phosphorus	thallium	zinc
calcium	lithium	platinum	tin	zirconium
chromium	magnesium	selenium	titanium	

SYNONYMS: vary depending upon the compound.

SAMPLING	MEASUREMENT
SAMPLER: FILTER (0.8- μ m, cellulose ester membrane)	TECHNIQUE: INDUCTIVELY COUPLED ARGON PLASMA, ATOMIC EMISSION SPECTROSCOPY
FLOW RATE: 1 to 4 L/min	ANALYTE: elements above
VOL-MIN: Table 1 -MAX: Table 1	ASHING REAGENTS: conc. HNO_3 , 4 mL; and conc. HClO_4 , 1 mL CONDITIONS: room temperature, 30 min; 150 °C to near dryness
SHIPMENT: routine	FINAL SOLUTION: 4% HNO_3 , 1% HClO_4 , 10 mL
SAMPLE STABILITY: stable	WAVELENGTH: depends upon element; Table 2
BLANKS: 2 to 10 field blanks per set	BACKGROUND CORRECTION: spectral wavelength shift
ACCURACY	CALIBRATION: elements in 4% HNO_3 , 1% HClO_4
RANGE STUDIED: not studied	RANGE: 2.5 to 1000 μ g per sample [1]
BIAS: none identified	ESTIMATED LOD: 1 μ g per sample [1]
OVERALL PRECISION (s_p): not evaluated	PRECISION (s_p): Table 2

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 this ashing procedure.

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 [1,2].

OTHER METHODS: This method replaces P&CAM 351 [2] for trace elements. Atomic absorption spectroscopy (e.g., Methods 70XX) is an alternate analytical technique for many of these elements.

REAGENTS:

1. Nitric acid, conc.
2. Perchloric acid, conc.*
3. Ashing acid: 4:1 (v/v) HNO_3 : HClO_4 .
Mix 4 volumes conc. HNO_3 with
1 volume conc. HClO_4 .
4. Calibration stock solutions,
1000 $\mu\text{g/mL}$. Commercially available,
or prepared per instrument
manufacturer's recommendation (see
step 12).
5. Dilution acid, 4% HNO_3 , 1% HClO_4 .
Add 50 mL ashing acid to 600 mL
water; dilute to 1 L.
6. Argon.
7. Distilled, deionized water.

*See Special Precautions.

EQUIPMENT:

1. Sampler: cellulose ester membrane filter,
0.8- μm pore size, 37-mm diameter; in cassette
filter holder.
2. Personal sampling pump, 1 to 4 L/min, with
flexible connecting tubing.
3. 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- and 100- mL.*
7. Assorted volumetric pipets as needed.*
8. Hotplate, surface temperature 150 $^{\circ}\text{C}$.

*Clean all glassware with conc. nitric acid and
rinse thoroughly in distilled water before use.

SPECIAL PRECAUTIONS: Perform all perchloric acid digestions in a perchloric acid hood.

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 $^{\circ}\text{C}$) until ca. 0.5 mL remains.
NOTE: Some species of Li, Mn, Mo, Sn, W, and Zr will not be completely solubilized by this
procedure. Alternative solubilization techniques for most of these elements can be
found elsewhere [2,3,4,5,6,7].
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 $^{\circ}\text{C}$ and take the sample to dryness.
9. Dissolve the residue in 2 to 3 mL dilution acid.
10. Transfer the solutions quantitatively to 10-mL volumetric flasks.
11. Dilute to volume with dilution acid.

CALIBRATION AND QUALITY CONTROL:

12. Calibrate the spectrometer according to the manufacturers recommendations.

NOTE: Typically, an acid blank and 10 $\mu\text{g/mL}$ multielement working standards are used. The
following multielement combinations are chemically compatible in 4% HNO_3 /1% HClO_4 :

- a. Ag, Ca, Co, Mn, Pb, V, Zn;
- b. Al, Be, Cd, La, Li, Ni, Ti;
- c. As, B, Ba, Mg, Mo, P, Sn;

Table 1. Properties and sampling volumes.

Element (Symbol)	Properties		Permissible Exposure Limits, mg/m ³ TWA OSHA/NIOSH/ACGIH	Air Volume @ OSHA, L	
	Atomic Weight	MP, °C		MIN	MAX
Silver (Ag)	107.87	961	0.01/ — / 0.1	250	2000
Aluminum (Al)	26.98	660	— / — / 10.	5 (g)	100 (g)
Arsenic (As)	74.92	817*	0.5/C 0.002/ 0.2	5	2000
Beryllium (Be)	9.01	1278	0.002/ 0.0005/ 0.002	1250	2000
Calcium (Ca)	40.08	842	5 (b)/ — / 2 (b)	5	200
Cadmium (Cd)	112.40	321	0.2/ 0.04/ 0.05	13	2000
Cobalt (Co)	58.93	1495	0.1/ — / 0.1	25	2000
Chromium (Cr)	52.00	1890	1.0 (c)/ 0.025/ 0.5 (c)	5	1000
Copper (Cu)	63.54	1083	1.0/ — / 1.0	5	1000
Iron (Fe)	55.85	1535	10 (b)/ — / 5 (b)	5	100
Lithium (Li)	6.94	179	0.025 (d)/ — / 0.025 (d)	100	2000
Magnesium (Mg)	24.31	651	15 (b)/ — / 10 (b)	5	67
Manganese (Mn)	54.94	1244	C 5/ — / C 5	5	200
Molybdenum (Mo)	95.94	651	15 (e)/ — / 10 (e)	5	67
Sodium (Na)	22.99	98	2 (f)/ C 2 (f)/ C 2 (f)	13	2000
Nickel (Ni)	58.71	1453	1/ 0.015/ 1 (c)	5	1000
Phosphorus (P)	30.97	44	— / — / 0.1	25 (g)	2000 (g)
Lead (Pb)	207.19	328	0.05/ 0.1/ 0.15	50	2000
Platinum (Pt)	195.09	1769	0.002 (a)/ — / 1 (c)	1250	2000
Selenium (Se)	78.96	217	0.2/ — / —	13	2000
Tin (Sn)	118.69	232	2/ — / 2 (c)	5	500
Tellurium (Te)	127.60	450	0.1/ — / 0.1	25	2000
Titanium (Ti)	47.90	1675	— / — / 10 (b)	5	100
Thallium (Tl)	204.37	304	0.1 (a)/ — / 0.1 (a)	25	2000
Vanadium (V)	50.94	1890	C 0.5/ 1 (c)/ 0.05 (V ₂ O ₅)	5	2000
Tungsten (W)	183.85	3410	— / 5 (e)/ 5 (e)	5 (g)	200 (g)
Yttrium (Y)	88.91	1495	1/ — / 1	5	1000
Zinc (Zn)	65.37	419	5 (b)/ 5 (b)/ 5 (b)	5	200
Zirconium (Zr)	91.22	1852	5/ — / 5	5	200

(a) soluble

(b) oxide

(c) metal

(d) hydride

(e) insoluble

(f) hydroxide

(g) at the ACGIH TLV

- d. Cu, Fe, Na, Pt, Sr, Te, Y;
- e. Cr, K, Sb, Se, Ti, Zr; and
- f. Si, W (distilled water only)

- 13. Analyze a standard for every ten samples.
- 14. Check recoveries with at least two spiked media blanks 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 ($\mu\text{g/mL}$), and the average media blank, C_b ($\mu\text{g/mL}$), from the instrument.
- 18. Using the solution volumes of sample, V_s (mL), and media blank, V_b (mL), calculate the concentration, C (mg/m^3), of each element in the air volume sampled, V (L):

$$C = \frac{C_s V_s - C_b V_b}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

Method P&CAM 351 was evaluated in 1981 [1,2]. The precision and recovery data were determined at 2.5 and 1000 μg of each element per sample on spiked filters. The precision and recovery data, instrumental detection limits, sensitivity, and analytical wavelengths are listed in Table 2. The values in Table 2 were determined with a Jarrell-Ash Model 1160 ICP operated according to manufacturer's instructions.

REFERENCES:

- [1] Hull, R.D. "Multielement Analysis of Industrial Hygiene Samples," NIOSH Internal Report, presented at the American Industrial Hygiene Conference, Portland, Oregon (May 1981).
- [2] NIOSH Manual of Analytical Methods, 2nd ed., V. 7, P&CAM 351, U.S. Department of Health and Human Services, Publ. (NIOSH) 82-100 (1981).
- [3] Ibid, S341 (Lead).
- [4] Ibid, V. 2, S5 (Manganese), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
- [5] Ibid, V. 4, P&CAM 271 (Tungsten), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-175 (1978).
- [6] Ibid, V. 5, P&CAM 173 (Metals by Atomic Absorption), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 79-141 (1979).
- [7] Ibid, V. 3, S183 (Tin), S185 (Zirconium), and S376 (Molybdenum), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).

METHOD REVISED BY: R. DeLon Hull and Mark Millson, NIOSH/DPSE.

Table 2. Measurement procedures and data (a).

Element	Wavelength (nm)	Instrumental LOD (ng/mL)	Sensitivity (Intensity/ $\mu\text{g/mL}$)	Recovery (%)		Precision (s_r) (N = 3)	
				@ 2.5 $\mu\text{g}/$ filter (b)	@ 1000 $\mu\text{g}/$ filter	@ 2.5 $\mu\text{g}/$ filter	@ 1000 $\mu\text{g}/$ filter
Ag	328.3	26	0.65	111	91	0.02	0.075
Al	308.2	14	0.23	93	100	0.092	0.023
As	193.7	13	0.57	103	99	0.062	0.026
Be	313.0	1.5	1.29	107	90	0.040	0.034
Ca	315.9	10	0.49	99	95	0.036	0.014
Cd	226.5	1.6	0.83	107	99	0.032	0.020
Co	231.2	7.4	0.38	101	95	0.040	0.005
Cr	205.6	1.3	0.50	98	106	0.053	0.016
Cu	324.8	2.1	0.72	98	99	0.036	0.022
Fe	259.9	3.9	0.13	94	97	0.068	0.016
Li	670.8	2.8	0.48	89	95	0.171	0.043
Mg	279.6	24	0.22	105	106	0.084	0.027
Mn	257.6	0.4	0.74	84	93	0.062	0.035
Mo	281.6	7.0	0.18	94	88	0.023	0.049
Na	589.0	10	0.76	(c)	101	(c)	0.045
Ni	231.6	3.4	0.41	105	97	0.027	0.020
P	214.9	22	0.17	(c)	91	(c)	0.056
Pb	220.4	17	0.42	105	95	0.060	0.011
Pt	203.7	15	0.69	106	91	0.041	0.075
Se	190.6	21	0.28	105	97	0.068	0.049
Sn	190.0	64	0.43	74	67	0.33	0.16
Te	214.3	29	0.41	102	94	0.050	0.063
Ti	334.9	1.2	0.55	96	108	0.051	0.029
Tl	190.9	17	0.22	103	99	0.043	0.017
V	310.2	3.2	0.88	99	94	0.043	0.014
W	207.9	13	2.58	35	23	0.053	0.60
Y	371.0	0.8	2.35	99	100	0.015	0.013
Zn	213.9	0.6	0.60	101	94	0.013	0.013
Zr	339.2	1.9	0.88	75	98	0.049	0.008

(a) Values reported were obtained with a Jarrell-Ash Model 1160 ICP; performance may vary with instrument and should be independently verified.

(b) 2.5 $\mu\text{g}/\text{filter}$ corresponds to 5 $\mu\text{g}/\text{m}^3$ for a 500-L air sample.

(c) Blank levels too high to make accurate determinations

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GLUTARALDEHYDE

FORMULA: $\text{OCH}(\text{CH}_2)_3\text{CHO}$

GLUTARALDEHYDE

MW: 100.13

METHOD:

ISSUED:

ACGIH: 0.7 mg/m^3 (Ceiling)

PROPERTIES: liquid; BP 187-189d

SYNONYMS: 1,5-Pentanedial; Glutaric Dialdehyde

SAMPLING

ANALYSIS

SAMPLER: Sorbent Tube
(7 cm x 4 mm ID)
Two sections
(100-150 mg/50-75 mg)
with 5% dinitrophenyl-
hydrazine hydrochloride

FLOW RATE: 0.2-1.0 LPM

VOL-MIN: 3 L
VOL-MAX: < 24 L

SHIPMENT: Blue ice

SAMPLE STABILITY: At least 7 days
at 4°C

BLANKS: 2 to 10 field blanks per set

BULK SAMPLE: Not required

ACCURACY

RANGE STUDIED: $0.130\text{--}4.80 \text{ mg/m}^3$

BIAS: -5.5%

OVERALL PRECISION: Not available

METHOD: HPLC/UV

ANALYTE: Glutaraldehyde dinitro-
phenyl hydrazone (DNPH)

PREPARATION: Desorb in
acetonitrile

ANALYSIS: Column-Zorbax ODS
Mobile Phase - 60% acetonitrile/
40% water → 15 min
90% acetonitrile/10% water
Flow Rate - 1.3 mL/min
Detection - UV 365 nm

ANALYTICAL RANGE: 1.5-95 ug/sample

ESTIMATED LOD: 0.3 ug/sample

ANALYTICAL PRECISION:
8.3% at 1.5 ug
2.1% at 25 ug

APPLICABILITY: The method is very specific for glutaraldehyde among other aldehydes, in the range of 0.130 to 4.80 mg/m^3 for a 3-20 liter sample.

INTERFERENCES: Other aldehydes and ketones react with DNPH but can be resolved from glutaraldehyde by using gradient HPLC conditions.

GLUTARALDEHYDE

METHOD:

REAGENTS:

1. Glutaraldehyde, 25 wt % solution in water (Aldrich G400-y or equivalent)
2. 2,4-Dinitrophenylhydrazine (Aldrich Chemical 20% moist or equivalent)
3. Hydrochloric Acid (Reagent Grade, ACS)
4. Water - Distilled, deionized
5. Dichloromethane (HPLC grade)
6. 2,4-Dinitrophenylhydrazine Hydrochloride Solution - The solution is prepared by adding 2.5 g dry DNPH to 1.0 L of 2N HCl. The suspension must be placed on a magnetic stirrer for 1-2 hours to allow complete solution of the DNPH. The solution is then extracted three times with 25 mL dichloromethane.
7. Acetonitrile (HPLC grade)
8. Ethanol (HPLC grade)
9. XAD-2 Resin (Supelpak 20 or equivalent)
10. DNPH·HCl XAD-2 Chemosorbent- Prepare DNPH·XAD-2 chemosorbent by coating DNPH·HCl onto the surface of the XAD-2 polymer resin. Prepare the DNPH·HCl by dissolving 2,4-dinitrophenylhydrazine in boiling 4M HCl. When the DNPH has dissolved completely, cool the solution in an ice bath. Collect the yellow crystalline precipitate by filtration through a glass fritted crucible. Recrystallize the precipitate from fresh, hot 4M HCl and dry in a desiccator for about eight hours.
11. Clean the XAD-2 by extracting with dichloromethane in a Soxhlet apparatus.

EQUIPMENT:

Solid Sorbent Collection

1. Glass tubing (6.0 mm OD, 4.0 mm ID)
2. DNPH·HCl-coated XAD-2 (see Reagent Section)
3. Glass wool
4. Rotary evaporator
5. Water bath
6. Ice bath

Solid Sorbent Sample Preparation

7. 3.0 mL pipet, Class A
8. Sample filter (see Sample Prep. Section)
9. Sample vials (see Sample Prep. Section)

HPLC Apparatus

10. Column - DuPont Zorbax ODS 5 μ m
11. UV Detector - Waters Associates Model 450 Absorbance Detector (or equivalent), 365 nm
12. Varian 5000 LC equipped with a Varian 8055 autosampler.
13. Injector - Rheodyne Model 7126 with 20 μ L loop.
14. Integrator - Spectra Physics Minigrator (or equivalent)
15. Recorder - Hewlett Packard 7133A (or equivalent)

12. Coat the DNPH·HCl onto the XAD-2 in a rotary evaporator. Weigh XAD-2 and place in a distillation flask of the rotary evaporator. Weigh sufficient DNPH·HCl for a 5% coating on the XAD-2 and dissolve it in a 9:1 ethanol:hydrochloric acid (12 M) mixture. Add the yellow solution to the distillation flask with the XAD-2. Attach the distillation and solvent receiving flask to the rotary evaporator. Place a water bath (100°C) under the distillation flask and an ice bath (0°C) under the receiving flask. Apply a vacuum to the evaporator and remove the solvent from the sorbent mixture. Store the sorbent in a sealed container, protected from light. The sorbent appears to be stable for at least 4-5 months.

SAMPLING

Sample Collection and Handling

1. Clean the glass sample tube with water, followed by methanol, and then dichloromethane. Allow it to dry.
2. Clean the glass wool by Soxhlet extraction (12 hours) with dichloromethane.
3. Cut glass tubing in 7.0-10.0 cm lengths.
4. Pack the tubes in the following manner:
 - glass wool plug at the front of the tube
 - 150 mg 5% DNPH·HCl/XAD-2 or 100 mg 5% DNPH·HCl/Supelpak 20
 - glass wool plug
 - 75 mg backup section 5% DNPH·HCl/XAD-2 or 50 mg 5% DNPH·HCl/Supelpak 20
 - glass wool plug at back of tube
5. Flame seal or cap the tubes until ready for use. Calibrate each personal sampling pump with a representative sampler in line.
6. Collect solid sorbent samples at a flow rate of 0.2 L/min or up to 1.0 L/min for at least 15 minutes. Overloading of the tube can often be visually verified by a color change of pale yellow 2,4-dinitrophenylhydrazine·hydrochloride to the respective derivative.
7. After sampling, tightly cap the tubes and cover to protect from exposure to light. Aluminum foil is useful if the tube ends have been capped with Teflon and/or a plastic cap.

SAMPLE PREPARATION

8. For each sample, break the sorbent tube in the area of the glass wool plug separating the front and back sorbent sections to facilitate the emptying of the sorbent tube for analysis. It is desirable to have the tube broken cleanly at the point where the front sorbent section and middle glass wool plug meet. Empty the sorbent and glass wool plug from the front section into a vial followed by the two glass wool plugs and backup section. (Note: The front and back sections can be analyzed separately if desired.) A wooden applicator stick may be used to force the sorbent out if necessary.
9. Pipet 3.0 mL (or whatever volume is necessary) HPLC-grade acetonitrile into the vial.
10. Allow the sample to desorb in the acetonitrile for one hour.
11. If necessary, filter the sample through a 0.5 μ m Teflon filter with a syringe with Swinex adaptor. Store the filter solution in a vial which is capped with a Teflon-lined, self-sealing septum. NH_4Cl may precipitate out of the filtered samples after approximately 24 hours. The precipitate does not appear to affect the chromatography of the compounds of interest. Precipitation is prevented by pH neutralization of the samples with a dilute NaOH solution prior to the filtration.

CALIBRATION AND STANDARDIZATION

12. Prepare calibration standards of glutaraldehyde derivative (i.e., 2,4-dinitrophenylhydrazone). Prepare the derivative by direct combination of the pure aldehyde with an acidic solution of 2,4-dinitrophenylhydrazine. Add the aldehyde in excess to assure that no underivatized DNPH remains. Extract the derivative with dichloromethane. Remove the dichloromethane under vacuum. Recrystallize the 2,4-dinitrophenylhydrazone from hot ethanol several times, until an acceptable melting point range is determined.
13. Prepare chromatographic standards by dissolving known masses of the hydrazone derivative in acetonitrile. A stock standard mixture of 400 $\mu\text{g/mL}$ is appropriate. Prepare other standards by dilution. The linearity of the detector must be determined over the full available range of detector sensitivities. A concentration range of up to two orders of magnitude is appropriate.
14. Assemble the necessary high pressure liquid chromatographic apparatus and establish operating parameters equivalent to those indicated in Table 1. By injecting calibration standards, establish the sensitivity limit of the detectors and the linear range of the analytical systems.

TABLE 1
HPLC PARAMETERS

Column:	DuPont Zorbax ODS 250 mm x 4.6 mm ID reverse phase
Mobile Phase:	60% CH ₃ CN/40% H ₂ O 15 min → 90% CH ₃ CN/10% H ₂ O
Flow Rate:	1.3 mL/min
Ultraviolet Detector:	λ 365 nm Range: 0.04 AUFS
Recorder:	Speed: 0.5 cm/min Range: 10 mV
Injections:	20 uL
Instruments:	Varian 5000 LC with autosampler Waters 450 UV absorbance detector Spectra physics minigrater Hewlett Packard recorder
Retention Time:	600 seconds
Detection Limit:	0.3 ug/sample

GLUTARALDEHYDE

METHOD:

15. Before processing any samples, the analyst should demonstrate, through the analysis of a solvent blank, that all glassware and reagents are interference-free. Each time a new set of samples is analyzed or there is a change in reagents, a solvent blank should be processed as a safeguard against chronic laboratory contamination.
16. Standard quality assurance practices should be used with this method. Laboratory replicates should be analyzed to validate the precision of analysis. Spiked samples should be analyzed to validate the accuracy of the analysis.

ANALYTICAL PROCEDURE

17. Table 1 summarizes the recommended HPLC column materials and operating conditions for the instrument. Included in the table are retention time and sensitivities that should be achieved by this method. An example of the separation achieved by this column is shown in Figure 1 of the Backup Data Report. Calibrate the system daily with standards.
18. Inject 20 uL of the sample extract with a high pressure syringe or a sampling loop. Record the volume injected to the nearest 0.05 uL, and the resulting peak size, in area units.
19. If the peak area exceeds the linear range of the system, dilute the extract and reanalyze.
20. If the peak measurement is hindered by the presence of interferences, other chromatographic conditions may be required.

CALCULATIONS

21. Determine the concentration of glutaraldehyde present in the sample atmosphere as follows:

$$\text{Concentration (ug/L)} = C_e V_e (100)/(460) V_s$$

where C_e = concentration of hydrazone in sample extract (ug/mL)

V_e = volume of extract (mL)

100 = molecular weight of glutaraldehyde (g/mole)

460 = molecular weight of glutaraldehyde 2,4-dinitrophenyl-hydrazone (g/mole)

V_s = volume of air sampled (L)

24.45 = molar volume of air (L) @ 25°C; 760 mm Hg

$$\text{Concentration (ppm)} = C(\text{ug/L}) \times 24.47/M_c$$

EVALUATION OF METHOD

This method was developed and validated with laboratory samples at Arthur D. Little, Inc. The relative standard deviation was determined to be 2.1 to 8.3 percent over the range 0.56 to 1.26 mg/m³.

REFERENCES

- [1] Menzies, K.T., K.J. Beltis and C.M. Wong. "Development of Sampling and Analytical Methods for Toxicants in Diesel Exhaust Streams," Final Report. Bureau of Mines Contract J0308005. March 1983.
- [2] Kuwata, D., M. Vebori and Y. Yamasaki. "Determination of Aliphatic and Aromatic Aldehydes in Polluted Airs as their 2,4-Dinitrophenylhydrazones by HPLC." J. Chromat. Sci. 17:264-268, 1979.
- [3] Andersson, G., K. Andersson, C. Nilsson and J. Levin. "Chemosorption of Formaldehyde in Amberlite XAD-2 Coated with 2,4-Dinitrophenylhydrazine." Chemosphere 10:823-827, 1979.
- [4] Andersson, K., C. Hallgren, J. Levin and C. Nilsson. "Solid Chemosorbent for Sampling Sub-ppm Levels of Acrolein and Glutaraldehyde in Air." Chemosphere 10:275-280, 1981.

METHOD WRITTEN BY: K.T. Menzies, K.J. Beltis, A.C. Roche
Arthur D. Little, Inc.

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FORMALDEHYDE

FORMULA: $\text{H}_2\text{C=O}$; CH_2O

FORMALDEHYDE

M.W.: 30.03

METHOD: 2502

ISSUED: 2/15/84

OSHA: 3 ppm; C 5 ppm; peak 10 ppm
NIOSH: lowest feasible level [1]
ACGIH: 1 ppm; STEL 2 ppm
(1 ppm = 1.23 mg/m³ @ NTP)

PROPERTIES: gas; BP -19.5 °C;
vapor density 1.067 (air = 1);
explosive range 7 to 73% v/v in air

SYNONYMS: methanal; CAS #50-00-0; Formalin (aqueous 30 to 50% w/v HCHO).

SAMPLING	MEASUREMENT
SAMPLER: SOLID SORBENT TUBE (2-(benzylamino)ethanol on Chromosorb 102 or XAD-2, 120 mg/60 mg)	TECHNIQUE: GAS CHROMATOGRAPHY, FID
FLOW RATE: 0.01 to 0.05 L/min	ANALYTE: 3-benzylloxazolidine
VOL-MIN: 1 L @ 3 ppm -MAX: 15 L	DESORPTION: 2 mL isooctane; ultrasonic bath 45 min or shake 4 hr
SHIPMENT: routine	INJECTION VOLUME: 1 µL, splitless; split vent time 30 sec
SAMPLE STABILITY: 4 weeks @ 25 °C	TEMPERATURE-INJECTION: 210 °C -DETECTOR: 220 °C -COLUMN: 70 °C for 1 min; 10 °C/min; hold @ 200 °C for 11.5 min
BLANKS: 2 media blanks and 2 field blanks per set of 10; 6 unopened tubes for DE determination (same lot as samples)	GASES-CARRIER: He, 100 kPa, ca 0.5 cm ³ /min; makeup flow, 29 cm ³ /min
ACCURACY	COLUMN: fused silica capillary, 25 m x 0.2 mm; Carbowax 20M
RANGE STUDIED: 0.55 to 4.7 mg/m ³ [2]	CALIBRATION: solutions of 3-benzylloxazolidine in isooctane
BIAS: not significant [2]	RANGE: 4 to 60 µg per sample
OVERALL PRECISION (s_p): 0.061 [2]	ESTIMATED LOD: 1 µg per sample [3]
	PRECISION (s_p): 0.055 [2]

APPLICABILITY: The working range is 0.3 to 5 mg/m³ (0.25 to 4 ppm) for a 12-L air sample.

INTERFERENCES: Phenol has a retention time close to that of 3-benzylloxazolidine but is baseline-resolved. Acid mists may inactivate the sorbent leading to inefficient collection of formaldehyde.

OTHER METHODS: This method was formerly designated P&CAM 354 [4]. It has improved sample stability and ease of personal sampling compared to Methods 3500 and 3501. Method 3500 (chromotropic acid) is the most sensitive.

2/15/84

REAGENTS:

1. Water, distilled, deionized.
2. Eluent: Isooctane, chromatographic grade, containing 0.025% (v/v) hexadecane or other suitable internal standard.
3. Formalin solution, 37%.*
4. Sulfuric acid, 0.02 N.
5. Sodium hydroxide, 0.01 N.
6. Sodium sulfite, 1.13 M.
7. Toluene, distilled in glass.
8. 2-(benzylamino)ethanol, distilled, 100 to 130 °C at 130 Pa (1 mm Hg).
9. 3-Benzylloxazolidine (see Appendix).
10. Formaldehyde stock solution, 1 mg/mL (see Appendix). Stable at least 3 months.
11. Helium, purified.

*See Special Precautions.

EQUIPMENT:

1. Sampler: glass tube, 10 cm x 4 mm ID, containing a 120-mg front section and 60-mg backup section of 2-(benzylamino)ethanol on either Chromosorb 102 or XAD 2. Sorbent sections are retained and separated by small plugs of glass wool. Pressure drop ca. 0.2 kPa (0.8 inch water) at 50 cm³/min airflow. Tubes are commercially available or may be prepared according to the Appendix.
2. Personal sampling pump, 0.01 to 0.05 L/min, with flexible connecting tubing.
3. Gas chromatograph, capillary column capability, FID, integrator (page 2502-1).
4. Vials, 4-mL, with plastic screw caps.
5. Ultrasonic bath or mechanical shaker.
6. Pipettes, volumetric, 1-, 5- and 10-mL, with pipet bulb.
7. Flasks, volumetric, 10-mL and 1-L.
8. Burettes, 50-mL.
9. pH meter.
10. Disposable pipettes, 2-mL.
11. Syringe, 10-μL, readable to 0.1 μL.

SPECIAL PRECAUTIONS: Formaldehyde is viewed as a potential occupational carcinogen by NIOSH [1].

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 0.05 L/min for a total sample size of 1 to 15 L.

NOTE 1: Sampling rate is limited by the speed of reaction of formaldehyde with the sorbent coating. At 0.10 L/min, appreciable formaldehyde (ca. 25%) is found on the backup section, possibly invalidating the sample. At higher flow rates, formaldehyde concentrations will be grossly underestimated.

NOTE 2: The presence of acid mists or gases may interfere indirectly by reacting with the 2-(benzylamino)ethanol to form amine salts which are not reactive with formaldehyde. If a sufficient amount of the 2-(benzylamino)ethanol is consumed by the acid, formaldehyde concentrations found with the method may be lower than the true concentrations.

4. Cap the samplers with plastic (not rubber) caps and pack securely for shipment.

SAMPLE PREPARATION:

5. Score each sampler with a file in back of the rear sorbent section.
6. Break sampler at score line. Remove and place glass wool plug and rear sorbent section in a vial.
7. Transfer front section with the remaining glass wool plugs to a vial.

8. Add 2.0 mL eluent to each vial. Screw cap tightly onto each vial.
9. Agitate vials in an ultrasonic bath for at least 45 min or in a shaker for 4 hr.

CALIBRATION AND QUALITY CONTROL:

10. Calibrate daily with at least five working standards.
 - a. Add known amounts of 3-benzylloxazolidine to eluent in 10-mL volumetric flasks and dilute to the mark.
NOTE: Prepare standard solutions for splitless injection in the range 1 to 50 $\mu\text{g/mL}$; for split injection, in the range 1 to 400 $\mu\text{g/mL}$.
 - b. Analyze together with samples and blanks (steps 13 and 14).
 - c. Prepare calibration graph (ratio of peak area or height of analyte to peak area or height of internal standard vs. μg 3-benzylloxazolidine) for the injection technique used.
11. Determine desorption efficiency (DE) at least once for each batch of charcoal used for sampling in the calibration range (step 10). Prepare three tubes at each of five levels plus three media blanks.
 - a. Remove and discard back sorbent section of a media blank sampler.
 - b. Inject a known amount of formaldehyde stock solution directly onto front sorbent section with a microliter syringe.
 - c. Cap the tube. Allow to stand overnight.
 - d. Desorb (steps 5 through 9) and analyze together with working standards (steps 13 and 14).
 - e. Prepare a graph of DE vs. μg 3-benzylloxazolidine recovered.
12. Analyze three quality control blind spiked and three analyst spikes to insure that the calibration graph and DE graph are in control.

MEASUREMENT:

13. Set gas chromatograph to conditions given on page 2502-1. Set air and hydrogen flows on the flame ionization detector to manufacturer's specifications. Inject 1- μL sample aliquot via the splitless injection technique. $t_r = 11.5$ min for these conditions.
NOTE: If sample amount of 3-benzylloxazolidine overloads the column (> 50 ng/ μL for 0.2 mm ID column), either dilute sample with eluent or inject via split injection technique, reanalyze, and apply appropriate volume correction factor in calculations. Column overloading is indicated by a plateau on the calibration graph at high concentrations. If split injection is required, the following conditions are typical:

Column temperature program:	150 $^{\circ}\text{C}$ for 7 min; 10 $^{\circ}\text{C}/\text{min}$; hold at 200 $^{\circ}\text{C}$
Split flow rate:	10 cm^3/min He
Retention time:	5.9 min

14. Measure peak area or height. Divide the peak area or height of analyte by the peak area or height of internal standard on the same chromatogram.

CALCULATIONS

15. Determine the mass, μg (corrected for DE) of 3-benzylloxazolidine 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 1: If $W_b > W_f/10$, report breakthrough and possible sample loss.
NOTE 2: A blank level of 1 to 7 μg HCHO is typical. Measure sufficient media blanks (at least 2 per 10 samples) to determine a representative mean value.

16. Multiply by the desorption volume (2 mL) and the conversion factor (0.184) from 3-benzoyloxazolidine to formaldehyde to calculate concentration, C, of formaldehyde in the air volume sampled, V (L):

$$C = \frac{(W_f + W_b - B_f - B_b) \cdot 2 \text{ mL} \cdot 0.184}{V} \cdot \text{mg/m}^3.$$

EVALUATION OF METHOD

Side-by-side comparisons of this method using laboratory-prepared samplers with a 2,4-dinitrophenylhydrazine-coated silica gel tube method [5] were done in a formaldehyde production facility. Means of the two methods were not significantly different [6]. Lab testing with spiked samplers and atmospheres generated by syringe pump/air dilution [2]; verified by 2,4-dinitrophenylhydrazine-coated silica gel tubes [5]. Breakthrough volume of laboratory-prepared samplers (80% RH, 6 mg HCHO/m³, 0.05 L/min) was greater than 16 L; DE (10.5, 37.5, 76.0 µg per sample) = 99%; recovery after storage (0.85 µg per sample) = 94.3% after two weeks at 25 °C; precision and accuracy as given on page 2502-1 (24 samples). When acetaldehyde was present as a cocontaminant, 5% breakthrough volume was 16 L (80% RH, 10 mg HCHO/m³, 10 mg acetaldehyde/m³, 0.05 L/min). Sampling rate influences reaction of formaldehyde with the sorbent coating. Rates above 0.05 L/min give low results with laboratory-prepared samplers.

In a breakthrough study done using commercially-available tubes, the breakthrough volume was found to be greater than 73 L at 8.7 mg/m³ and greater than 58 L at 28 mg/m³ of formaldehyde. These atmospheres were sampled at 0.078 L/min.

An atmosphere of 0.36 mg/m³ formaldehyde, as determined by P&CAM 125 [7], was sampled with sets of six tubes at ca. 0.08 L/min for 70 min, 285 min and 482 min. The average amount indicated by these tubes was 0.32 mg/m³ with the relative standard deviation less than 10% in all cases. The tube loadings for these sampling periods were 1.6, 6.3 and 10.8 µg. This information indicates that concentrations as low as 0.1 mg/m³ should be measurable with a sampling rate of 0.08 L/min and a sampling time of 8 hrs.

REFERENCES:

- [1] NIOSH Current Intelligence Bulletin 34, "Formaldehyde: Evidence of Carcinogenicity," U.S. Department of Health and Human Services, Publ. (NIOSH) 81-111 (1981).
- [2] Kennedy, E. R. and R. H. Hill, Jr. *Anal. Chem.*, **54**, 1739-1742 (1982).
- [3] User check, UBTL, NIOSH Sequence #3990-Y (unpublished, November 21, 1983).
- [4] NIOSH Manual of Analytical Methods, 2nd ed., V. 7, P&CAM 354, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 82-100 (1982).
- [5] Beasley, R. K., C. E. Hoffmann, M. L. Reuppel and J. W. Worley. *Anal. Chem.*, **52**, 1110-1114 (1980).
- [6] Smith, D. L., M. Bolyard and E. R. Kennedy. *Am. Ind. Hyg. Assoc. J.*, **44**, 97-99 (1983).
- [7] NIOSH Manual of Analytical Methods, 2nd ed., V. i, P&CAM 125, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977). P&CAM 125 appears as Method 3500 in this Manual.

METHOD WRITTEN BY: Eugene R. Kennedy, Ph.D., and Robert H. Hill, Jr., Ph.D., NIOSH/DPSE.

APPENDIX:

SAMPLING TUBE PREPARATION

Extract Chromosorb 102 or XAD-2 with a 50/50 (v/v) mixture of acetone/methylene chloride in a soxhlet apparatus for 4 hrs using a 30-min cycle time. Vacuum dry [1 mm Hg (133 Pa)] the sorbent at ambient temperature overnight. To a slurry of the dried, extracted sorbent (10 g in 100 mL toluene), add 1 g distilled 2-(benzylamino)ethanol in 10 mL toluene. Allow to stand for 1 hr with occasional swirling. Remove the solvent by rotary evaporation and vacuum dry [1 mm Hg (133 Pa)] at ambient temperature overnight. For each batch of the coated sorbent, desorb several 100-mg portions with isooctane and analyze. If the background is greater than 7 µg 3-benzylloxazolidine/100 mg coated sorbent, discard the batch.

PREPARATION OF 3-BENZYLOXAZOLIDINE

Add a solution of 1.51 g (10 mmole) 2-(benzylamino)ethanol in 10 mL toluene dropwise to a solution of 1 mL 37% formalin (0.37 g formaldehyde, 12.3 mmole) in 25 mL toluene. Stir 1 hr. Remove the solvent at reduced pressure by rotary evaporation. The product is a yellow viscous oil. Vacuum distill at 58 to 62 °C at 1 mm Hg (133 Pa); yields 3-benzylloxazolidine as a clear, colorless oil, stable at room temperature for several months in a closed vial.

PREPARATION AND STANDARDIZATION OF FORMALDEHYDE STOCK SOLUTION (ca. 1 mg/mL)

Dilute 2.7 mL 37% formalin solution to 1 L with distilled, deionized water. This solution is stable at least three months. Standardize as follows:

Place 5.0 mL 1.13 M sodium sulfite solution in a beaker, stirred with a magnetic stirrer. Adjust pH to between 7 and 9 with base or acid. Record the pH. Add 10.0 mL stock formaldehyde solution. The pH should now be about 12. Titrate the solution back to its original pH with 0.02 N sulfuric acid (1 mL acid = 0.600 mg HCHO; about 17 mL acid needed). If the endpoint pH is overrun, back titrate to the endpoint with 0.01 N sodium hydroxide. Calculate the concentration, C_s (mg/mL), of the formaldehyde stock solution:

$$C_s = \frac{30.0 \cdot [(N_a \cdot V_a) - (N_b \cdot V_b)]}{V_s}$$

where: 30.0 = 30.0 g/equivalent of formaldehyde

N_a = normality of sulfuric acid

V_a = volume of sulfuric acid (mL) used for titration

N_b = normality of NaOH

V_b = volume of NaOH (mL) used for back titration

V_s = volume of HCHO stock solution (10.0 mL).

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NITROGEN DIOXIDE - BREATHING ZONE

FORMULA: NO₂

NITROGEN DIOXIDE

M.W.: 46.01

METHOD: 5700

ISSUED: 2/15/84

OSHA: C 5 ppm

NIOSH: 1 ppm/15 min [1]

ACGIH: 3 ppm; STEL 5 ppm

(1 ppm = 1.881 mg/m³ @ NTP)

PROPERTIES: dark brown fuming liquid or gas;

BP 21 °C; MP -11 °C

SYNONYMS: nitrogen peroxide, CAS #10102-44-0.

SAMPLING	MEASUREMENT
SAMPLER: PASSIVE (Palmer tube with three triethanolamine-treated screens [2])	TECHNIQUE: VISIBLE ABSORPTION SPECTROPHOTOMETRY ANALYTE: nitrite ion (NO ₂ ⁻)
SAMPLING TIME-MIN: 15 min @ 5 ppm -MAX: 8 hr @ 10 ppm	REAGENT: aqueous solution of sulfanilamide, H ₃ PO ₄ and N-1-naphthylethylene- diamine dihydrochloride
SHIPMENT: routine	WAVELENGTH: 540 nm
SAMPLE STABILITY: use sampler within 1 month after preparation; analyze within 1 month after sampling	PATHLENGTH: 1 cm CALIBRATION: solutions of NaNO ₂ in reagent
BLANKS: 5 field blanks per sample set	RANGE: 0.13 to 8.5 µg NO ₂ per sample [3] ESTIMATED LOD: 0.01 µg NO ₂ per sample
ACCURACY	PRECISION (s _r): 0.05 [3]
RANGE STUDIED: 1.2 to 80 ppm-hrs (0.13 to 8.5 µg NO ₂ per sample) [3]	
BIAS: complete conversion of nitrogen dioxide to nitrite (Saltzman factor = 1) [2]; slightly lower collection efficiency at lower pressure (-7% at 5,500 m altitude) [4]	
OVERALL PRECISION (s _r): 0.06 [5]	

APPLICABILITY: The working range is 1.2 to 80 ppm-hrs [3].

INTERFERENCES: In very dusty environments, particles may deposit on the inside surface of the samplers. Resuspension of the dust in the analytical reagent can give a positive bias in the spectrophotometric reading.

OTHER METHODS: Short-term, long-term, and passive indicator tubes, and various other passive samplers and electrochemical instruments are used. P&CAM 231 [6] and S320 [8] are active solid sorbent sampling methods using similar color development; P&CAM 108 [7] uses a bubbler.

REAGENTS:

1. Absorbing reagent. 1 volume triethanolamine (TEA) diluted in 7 volumes analytical grade acetone.
2. Sulfanilamide solution. 2 g sulfanilamide + 5 mL conc. H_3PO_4 diluted to 100 mL with distilled H_2O .
3. N-1-naphthylethylenediamine dihydrochloride (NEDA) solution. 70 mg NEDA dissolved in 50 mL distilled H_2O .
4. Combined reagent. 1 volume sulfanilamide solution + 1 volume water + 1/10 volume NEDA solution. Stable ca. one month if protected from light and refrigerated.
5. Sodium nitrite stock solution, 0.05 M. Dissolve 0.345 g (accurately weighed) NaNO_2 (reagent grade) in distilled water to make 100 mL solution. Protect from light and keep refrigerated. Stable 90 days.
6. Calibration stock solution. Dilute sodium nitrite stock solution with distilled water. Prepare fresh just before use. For example, a 1:50 dilution yields 1 nanomole $\text{NO}_2/\mu\text{L}$.

EQUIPMENT:

1. Sampler: See APPENDIX (potential sources of equipment given in reference [2]):
 - a. Acrylic tubing, 3/8 inch (9.5 mm) ID.
 - b. Stainless steel screen, 40 x 40 mesh/inch (16 x 16 mesh/cm).
 - c. Polyethylene cap, 1/2 inch (12.7 mm, unflanged).
 - d. Polyethylene cap, 1/2 inch (12.7 mm, flanged).
 - e. Pen clips, 0.48 inch (12.2 mm) I.D.
 - f. Electrical tape, plastic.
 - g. Stopcock grease.
2. Spectrophotometer, reading at 540 nm, with 1-cm cuvettes.
3. Volumetric flasks and pipets for preparation of standards.
4. Mixer, vibration or vortex (optional).

SPECIAL PRECAUTIONS: None.

SAMPLING:

1. Attach the sampler with flanged cap down. Start sampling by removing flanged cap. Estimate appropriate sampling time such that the amount of NO_2 collected is in the range 1.2 to 80 ppm-hrs (0.13 to 8.5 $\mu\text{g NO}_2$).
2. Terminate sampling by replacing flanged cap.

CALIBRATION AND QUALITY CONTROL:

3. Calibrate daily.
 - a. Prepare a series of working standards just before use over the range 0 to 40 nanomoles (0 to 1.84 μg) NO_2 per 2.1 mL combined reagent.
 - b. Allow 10 min for color development.
 - c. Transfer an aliquot of the working standard to a cuvette and analyze (steps 6 through 8).
4. Plot absorbance at 540 nm against NO_2 mass in nanomoles.

NOTE: The absorbance of 40 nanomoles NO_2 is ca. 1 absorbance unit.

5. Check dimensions of the sampler. If cross-sectional area divided by length (A_t/L) of the sampler tube differs significantly from 0.10 cm, recalculate the diffusive collection rate (step 9).

MEASUREMENT:

6. Remove flanged cap from samplers. Add 2.1 mL combined reagent directly into samplers.
NOTE: If 2.1 mL is not sufficient to completely cover the exit slit of the spectrophotometer, a larger volume can be used provided the same volume is used for both standards and unknowns.
7. Recap the samplers and mix manually or with a mixer. Allow 10 min for the color to develop.
8. Transfer the solution to a cuvette and read the absorbance at 540 nm within 30 min from time reagent was added.
NOTE: If sample reads beyond calibration graph, dilute sample with combined reagent or extend calibration range.

CALCULATIONS:

9. From calibration graph, read nanomoles NO_2 collected by the sampler. Divide by 2.3 nanomoles/ppm-hr (the diffusive collection rate [2]) and the sample exposure time, t (hr), to obtain time-weighted average concentration, C (ppm NO_2), of NO_2 :

$$C = \frac{\text{nanomoles NO}_2}{2.3 t}$$

NOTE: Use $2.3 \cdot (\text{actual } A_t/L [\text{cm}] \div 0.1 \text{ cm})$ nanomoles/ppm-hr as the diffusive collection rate if sampler dimensions are different from those specified in the APPENDIX.

EVALUATION OF METHOD:

Analytical precision and useful range was estimated from a laboratory evaluation conducted by NIOSH (1982) [3]. Overall precision ($s_r = 0.06$) was estimated from side-by-side replicate samples collected in a underground salt mine [5]. In a laboratory study, this method gave results averaging $94 \pm 4\%$ (mean $\pm s_r$) of a reference method over the range 1.3 to 79 ppm-hrs [3]. A field study found results for this method of $109 \pm 9\%$ (mean $\pm s_r$) vs. a reference method in the range 12 to 19 ppm-hrs [5]. Sampling errors may exist in this method when the concentration is not constant in time and the sampling period is short [9,10]. For example, the value of s_r associated with estimating the TWA of an isolated random 10-sec concentration pulse within a 15-min sampling period may be calculated [9] to equal 0.5. Secondly, reference [9] reports a specific set of real-time concentration data measured in an industrial environment. For these data, the error s_r in making 15-min TWA estimates is calculated to equal 0.12. Although these values are large, similar sampling errors due to time variations are expected to be better controlled for longer sampling periods as the variance of the sampling error varies inversely with the sampling period.

REFERENCES:

- [1] Criteria for a Recommended Standard...Occupational Exposure to Oxides of Nitrogen (Nitrogen Dioxide and Nitric Oxide), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 76-149 (1976).
- [2] Palmes, E. D., A. F. Gunnison, J. DeMattio and C. Tomczyk. Personal Sampler for Nitrogen Dioxide, Am. Ind. Hyg. Assoc. J., **37**, 570-577 (1976).
- [3] Woebkenberg, M. L. A Comparison of Three Personal Passive Sampling Methods for NO_2 , Am. Ind. Hyg. Assoc. J., **43**, 553-561 (1982).

- [1] Lindenboom R. and E. D. Palmes. Effect of Reduced Atmospheric Pressure on a Diffusional Sampler, Am. Ind. Hyg. Assoc. J., 44, 105-108 (1983).
- [2] Jones W., E. D. Palmes, C. Tomczyk, and M. Millson. Field Comparison of Two Methods for the Determination of NO₂ Concentration in Air, Am. Ind. Hyg. Assoc. J., 40, 437-438 (1979).
- [3] NIOSH Manual of Analytical Methods. 2nd. ed., V. 1, P&CAM 231, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).
- [7] Ibid, P&CAM 108.
- [8] Ibid, V. 4, S320, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-175 (1978).
- [9] Bartley, D. L., L. J. Doemeny and D. G. Taylor. Diffusive Monitoring of Fluctuating Concentrations, Am. Ind. Hyg. Assoc. J., 44, 241-247 (1983).
- [10] Hearl, F. J. and M. P. Manning. Transient Response of Diffusive Dosimeters, Amer. Ind. Hyg. Assoc. J., 41, 778-783 (1980).

METHOD DEVELOPED BY: E. D. Palmes, New York University [2].

METHOD REVISED BY: William Jones and Frank Hearl, NIOSH/DRDS; Mary Lynn Woebkenberg, NIOSH/DPSE.

APPENDIX:

PREPARATION OF SAMPLER

1. Measure the average cross-sectional area of a length of 3/8 inch (9.5 mm) ID acrylic tubing.
 - a. Cap one end of the tubing. Pour in a known volume, v (cm^3), of water to nearly fill the tubing (e.g., 100 mL water for a 180-cm (6-foot) length of tubing).
 - b. Measure the height, h (cm), of the water column in the tubing.
 - c. Determine the average cross-sectional area, A_t (cm^2), of the tubing.

$$A_t = \frac{v}{h}$$

2. Cut the tubing into lengths, L (ca. 7.1 cm), such that $A_t/L = \text{exactly } 0.1 \text{ cm}$.
NOTE: The collection rate is directly proportional to A_t/L . For $A_t/L = 0.1 \text{ cm}$, the collection rate is 2.3 nanomoles/ppm-hr [2].
3. Cut circular portions, 13/32 inch (10.3 mm) to 7/16 inch (11.1 mm) in diameter, from stainless steel screen using a 13/32 inch (10.3 mm) paper punch or other suitable means.
4. Clean the tubes, screens and caps with detergent solution in an ultrasonic bath. Rinse with distilled water. Air dry.
5. Dip the screens in absorbing reagent.
6. Using forceps, place the screens on absorbent paper. Press the screens momentarily with the forceps tips to blot. Allow the acetone to evaporate.
7. Stack three treated screens in the bottom of an unflanged cap. Insert the acrylic tube into the unflanged cap securing the screens (see the figures).
8. Slide the pen clip onto the acrylic tube touching the unflanged cap. Secure the pen clip and unflanged cap with a piece of electrical tape.
9. Apply a small amount of stopcock grease to the outside of the uncapped end of the acrylic tube and slide the flanged cap into place.

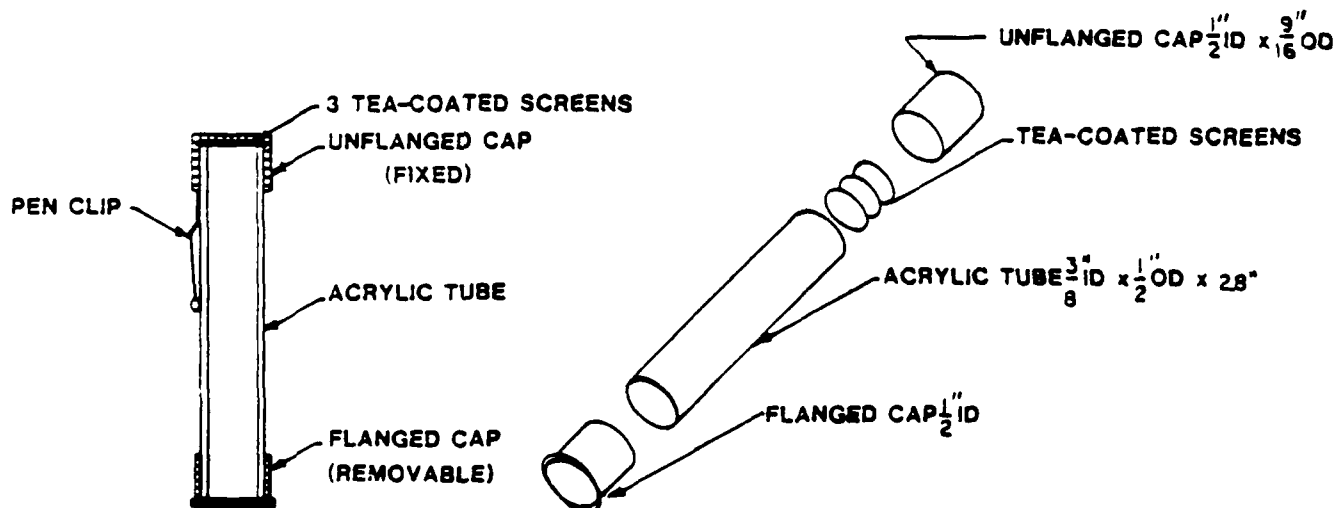


Figure 1. Assembled view (left) and exploded view (right) of sampler.

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AMMONIA

Ammonia

Analyte:	Ammonia	Method No.: S347
Matrix:	Air	Range: 17-68 mg/cu m
OSHA Standard:	50 ppm (35 mg/cu m)	Precision ($\overline{CV}_T$): 0.062
Procedure:	Adsorption on sulfuric acid-treated silica gel, desorption with 0.1 N sulfuric acid, ammonia specific electrode	Validation Date: 11/25/77

1. Principle of the Method

- 1.1 A known volume of air is drawn through a glass tube containing sulfuric acid-treated silica gel to trap ammonia vapors. The sampling tube is connected in series to a prefilter to collect particulate ammonium salts.
- 1.2 Ammonia is desorbed from the silica gel with 0.1 N sulfuric acid, and the sample is analyzed using an ammonia specific electrode.

2. Range and Sensitivity

- 2.1 This method was validated over the range of 16.9-67.6 mg/cu m at an atmospheric temperature of 24°C and atmospheric pressure of 759 mm Hg, using a 30-liter sample. This sample size is based on the capacity of the sulfuric acid-treated silica gel to collect vapors of ammonia in air at high relative humidity. The method is capable of measuring much smaller amounts if the desorption efficiency is adequate. Desorption efficiency must be determined over the range used.
- 2.2 The upper limit of the range of the method is dependent on the adsorptive capacity of the sulfuric acid-treated silica gel. This capacity varies with the concentrations of ammonia and other substances in the air. Breakthrough is defined as the time that the effluent concentration from the collection tube (containing 200 mg of sulfuric acid-treated silica gel) reaches 5% of the concentration in the test gas mixture. Breakthrough was not observed after 310 minutes at an average sampling rate of 0.209 liter/minute and relative humidity of 85% and temperature of 25°C. The breakthrough test was conducted at an average concentration of 68.6 mg/cu m.

3. Interferences

- 3.1 When interfering compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.
- 3.2 Methyl amine and ethyl amine are known interferences of the analytical method. Other volatile amines may also interfere in the analytical method.
- 3.3 Particulate contaminants such as ammonium salts are removed by the prefilter.

4. Precision and Accuracy

- 4.1 The Coefficient of Variation ($\overline{CV_T}$) for the total analytical and sampling method in the range of 16.9-67.6 mg/cu m is 0.062. This value corresponds to a 2.2 mg/cu m standard deviation at the OSHA standard level. Statistical information can be found in Reference 11.1. Details of the test procedures are found in Reference 11.2.
- 4.2 On the average, the concentrations obtained in the laboratory validation study at 0.5X, 1X, and 2X the OSHA standard level were 2.4% lower than the "true" concentrations for 18 samples. Any difference between the "found" and "true" concentrations may not represent a bias in the sampling and analytical method, but rather a random variation from the experimentally determined "true" concentration. The Coefficient of Variation is a good measure of the accuracy of the method since the recoveries and storage stability were good and would not contribute to a bias in a determined concentration. Storage stability studies on samples collected from a test atmosphere at a concentration of 33.8 mg/cu m indicate that collected samples are stable for at least 7 days.

5. Advantages and Disadvantages of the Method

- 5.1 The sampling device is small, portable, and involves no liquids. The tubes are analyzed by means of a quick, instrumental method.
- 5.2 One disadvantage of the method is that the amount of sample that can be taken is limited by the number of micrograms that the tube will hold before overloading. When the amount of ammonia found on the backup section of the sulfuric acid-treated silica gel tube exceeds 25% of that found on the front section, the probability of sample loss exists.
- 5.3 The precision of the method is limited by the reproducibility of the pressure drop across the tubes. This drop will affect the flow rate and cause the volume to be imprecise, because the pump is usually calibrated for one tube only.

6. Apparatus

- 6.1 Prefilter Unit: The prefilter unit, which is used to remove particulate interferences, consists of a 37-mm diameter cellulose ester membrane filter with a pore size of 0.80 micrometer contained in a 37-mm two-piece cassette filter holder. The filter is supported in the holder by a stainless steel screen.
- 6.2 Personal Sampling Pump: A calibrated personal sampling pump whose flow rate can be determined within 5% at the recommended flow rate.
- 6.3 Sulfuric Acid-Treated Silica Gel Sampling Tubes: Glass tube with both ends unsealed and fire-polished, 6.0-cm long with a 6-mm O.D. and a 4-mm I.D. containing two sections of 20/40 mesh sulfuric acid-treated silica gel (Section 8.2) separated by a 2-mm portion of glass wool. The adsorbing section of the tube contains 200 mg of sulfuric acid-treated silica gel and the backup section contains 100 mg. A plug of silylated glass wool is placed at the ends of the tube. The pressure drop across the tube must be no greater than 13 inches of water at a flow rate of 0.2 liter/minute. The glass tubes should be rinsed and dried with acetone before packing. The tubes are capped with plastic caps.
- 6.4 Orion Model 95-10 ammonia gas sensing electrode, or equivalent.
- 6.5 Orion Model 407 specific ion meter, or equivalent. A pH meter with a millivolt readout can also be used.
- 6.6 Scintillation vials, 20 mL.
- 6.7 Magnetic stirrer and stirring bars.
- 6.8 Pipets: Delivery type of convenient sizes.
- 6.9 Volumetric Flasks: 1-liter and 50-mL and other convenient sizes for preparing standard solutions.
- 6.10 Beakers, 250 mL.
- 6.11 Gas-tight syringes: 2- and 5-mL for preparing spiked samples.
- 6.12 Stopwatch.
- 6.13 Manometer.

7. Reagents

Whenever possible, reagents used must be ACS Reagent Grade or better.

- 7.1 Lecture bottle of ammonia gas, reagent grade.
- 7.2 Ammonium chloride, reagent grade.
- 7.3 Sulfuric acid, reagent grade in the following concentrations: 0.1 N and 0.4 N.

- 7.4 Prepare a 1000 micrograms/mL ammonia stock standard by weighing 3.1476 g ammonium chloride in a 1-liter volumetric flask. Make to volume with deionized water.
- 7.5 Prepare a 10,000 micrograms/mL ammonia stock standard by weighing 31.476 g ammonium chloride in a 1-liter volumetric flask. Make to volume with deionized water.
- 7.6 Sodium hydroxide solution, 10 N.
- 7.7 Silica gel, 20/40 mesh from SKC, Inc.

8. Procedure

- 8.1 Cleaning of Equipment. All glassware used for the laboratory analysis should be detergent washed, thoroughly rinsed with tap water and distilled water, and dried.
- 8.2 Preparation of Sulfuric Acid-Treated Silica Gel
 - 8.2.1 Place 6 g of 20/40 mesh silica gel in a 250-mL beaker.
 - 8.2.2 Add 15 mL of 0.4 N sulfuric acid to the beaker. Stir the mixture, and cover the beaker with a watch glass.
 - 8.2.3 Heat the silica gel-acid mixture in a fume hood with a Bunsen burner to a very gentle boil. Evaporate approximately one-half of the liquid.
 - 8.2.4 Place the covered beaker in a drying oven at 120°C until the remainder of the water has been evaporated.
 - 8.2.5 The prepared acid-treated silica gel should flow freely and not adhere to the beaker. Store the silica gel in a desiccator until ready for use.
- 8.3 Calibration of Sampling Pumps. Each personal sampling pump must be calibrated with a representative sampling tube and prefilter cassette unit in the line to minimize errors associated with uncertainties in the volume sampled.
- 8.4 Collection and Shipping of Samples
 - 8.4.1 Assemble the filter in the cassette holder and close firmly. The filter is backed up by a stainless steel screen rather than a filter pad. Secure the cassette holder with tape or shrinkable band.
 - 8.4.2 Immediately before sampling, remove the caps from the ends of the sulfuric acid-treated silica gel tube. Remove the filter holder plugs and attach the outlet of the filter holder to the inlet of the sampling tube with a short piece of flexible tubing.

- 8.4.3 The smaller section of sulfuric acid-treated silica gel is used as a backup and should be positioned nearer the sampling pump.
- 8.4.4 The tube should be placed in a vertical direction during sampling to minimize channeling through the sulfuric acid-treated silica gel.
- 8.4.5 Air being sampled should not pass through any hose or tubing before entering the prefilter cassette.
- 8.4.6 A sample size of 30 liters is recommended. Sample at a flow rate between 0.1 and 0.2 liter/minute. Record the sampling time, flow rate, and type of sampling pump used.
- 8.4.7 The temperature, pressure, and relative humidity of the atmosphere being sampled should be recorded. If pressure reading is not available, record the elevation.
- 8.4.8 The sampling tube should be capped with plastic caps immediately after sampling. Under no circumstances should rubber caps be used.
- 8.4.9 The filter should be removed from the cassette filter holder and discarded. The cassette holders and stainless steel screens should be cleaned and saved for future use.
- 8.4.10 With each batch of ten samples, submit one tube from the same lot of tubes used for sample collection. This tube must be subjected to exactly the same handling as the samples except that no air is drawn through it. This tube should be labeled as the blank. A minimum of 18 extra sulfuric acid-treated silica gel tubes should be provided for desorption efficiency determinations.
- 8.4.11 Capped tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping.

8.5 Analysis of Samples

The meter used in the analysis of samples must be calibrated before samples are analyzed. The procedure for calibration of the specific ion meter or pH/millivolt meter is discussed in Section 9. Proceed to Section 9 before sample analysis.

- 8.5.1 Preparation of Samples. Remove the plastic cap from the inlet end of the sampling tube. Remove the glass wool plug and transfer the first (larger) section of sulfuric acid-treated silica gel to a 20-mL scintillation vial. Remove the separating section of glass wool and transfer the backup section of sulfuric acid-treated silica gel to another scintillation vial. Analyze these two sections separately. Firm tapping of the tube may be necessary to effect complete transfer of the sulfuric acid-treated silica gel.

- 8.5.2 Desorption of Samples. Prior to analysis, 10 mL of 0.1 N sulfuric acid is pipetted into each vial. Cap and shake the sample vigorously. Desorption is complete in 45 minutes. Analyses should be completed within one day after the ammonia is desorbed.
- 8.5.3 Pipet an 8-mL aliquot of the desorbed sample into a clean 20-mL scintillation vial. Add 6 mL of deionized water to the vial.
- 8.5.4 Add 1 mL of 10 N sodium hydroxide to the vial to make the solution basic. The total volume in the vial should be 15 mL. Add a magnetic stirring bar. After addition of base, samples should be analyzed immediately.
- 8.5.5 Lower the ammonia specific electrode into the solution, taking care not to trap air under the electrode. If using a specific ion meter, record the meter reading on the logarithmic scale. This reading is the sample concentration in micrograms/mL. If a pH/millivolt meter is used, record the millivolt reading and refer to the calibration curve prepared in Section 9 to determine the sample concentration.
- 8.5.6 If the sample falls outside of the range of analysis, recalibrate the meter in the range of interest.
- 8.6 Determination of Desorption Efficiency
- 8.6.1 The desorption efficiency of a particular compound can vary from one laboratory to another. Thus, it is necessary to determine the fraction of the specific compound that is removed in the desorption process.
- 8.6.2 Extra sampling tubes containing sulfuric acid-treated silica gel are used to prepare spiked samples for desorption efficiency determinations. Spiked samples are prepared by drawing air through the tubes and spiking the air upstream of the tube with the appropriate amount of ammonia gas. Ammonia gas is spiked upstream using gas tight syringes. Volumes of 0.755, 1.51, and 3.02 mL of ammonia gas represent the amount present at 0.5X, 1X, and 2X the OSHA standard levels, respectively. The amount spiked is equivalent to that present in a 30-liter air sample at the selected level.
- Six tubes at each of three levels (0.5X, 1X, and 2X the OSHA standard) are prepared in this manner and allowed to stand for at least overnight to ensure complete adsorption of the ammonia onto the sulfuric acid-treated silica gel. These tubes are referred to as the samples. A parallel blank tube should be treated in the same manner except that no sample is added to it. The sample and blank tubes are desorbed and analyzed in exactly the same manner as the sampling tube described in Section 8.5.

The desorption efficiency (D.E.) equals the average weight in micrograms recovered from the tube divided by the weight in micrograms added to the tube, or

$$\text{D.E.} = \frac{\text{Average Weight recovered (micrograms)} - \text{Blank}}{\text{Weight added (micrograms)}}$$

The desorption efficiency is dependent on the amount of ammonia collected on the sulfuric acid-treated silica gel. Plot the desorption efficiency versus weight of ammonia found. This curve is used in Section 10.5 to correct for adsorption losses.

9. Calibration and Standards

9.1 Prepare standard solutions containing 10 micrograms/mL, 100 micrograms/mL, and 1000 micrograms/mL as described below:

- 9.1.1 10 micrograms/mL: Using the 1000 micrograms/mL stock solution (Section 7.4), pipet a 5-mL aliquot into a 50-mL volumetric flask and bring to volume with deionized water. From this solution, pipet another 5-mL aliquot into a clean 50-mL volumetric flask, and add 20 mL 0.1 N sulfuric acid, 2 mL of 10 N sodium hydroxide, and bring to volume with deionized water. This final solution is the 10 micrograms/mL standard. Cap the solution after preparation.
- 9.1.2 100 micrograms/mL: Pipet a 5-mL aliquot from the 1000 micrograms/mL stock solution into a clean 50-mL volumetric flask. Add 20 mL 0.1 N sulfuric acid, 2 mL 10 N sodium hydroxide, and bring to volume with deionized water. Cap the solution after preparation.
- 9.1.3 1000 micrograms/mL: Pipet a 5-mL aliquot from the 10,000 micrograms/mL stock solution (Section 7.5) into a clean 50-mL volumetric flask. Add 20 mL 0.1 N sulfuric acid, 2 mL 10 N sodium hydroxide, and bring to volume with deionized water. Cap the solution after preparation.

Note: These standards are good for approximately 2 hours if kept tightly capped.

Additional standards may be prepared in order to accommodate the range of samples to be analyzed. Prepare additional standards over the range of interest using the 1000 micrograms/mL stock standard solution.

9.2 The specific ion meter must be calibrated over the range of interest using standard solutions prepared as described above. The meter is calibrated over a 10-fold concentration range.

To calibrate the specific ion meter in the range of 10-100 micrograms/mL, use the following procedure:

- 9.2.1 Place the electrode in the 10 micrograms/mL standard. Turn the function switch to X^- and adjust the meter needle to "10" on the logarithmic scale with the calibration control. Use magnetic stirring throughout the procedure.
 - 9.2.2 Rinse the electrode and place in the 100 micrograms/mL standard and stir thoroughly. Turn the temperature compensator knob until the meter needle reads "100" on the logarithmic scale. The meter is now calibrated in the range of 10-100 micrograms/mL.
 - 9.2.3 Recalibration of the meter is necessary in order to analyze samples outside of this range. Repeat the calibration procedure for the range of 100-1000 micrograms/mL.
- 9.3 If a pH/millivolt meter is used, the standards described above can be used to prepare a standard calibration curve. The curve is prepared on semi-log paper by plotting millivolt versus concentration in micrograms/mL. The concentration should be plotted on the logarithmic scale.

10. Calculations

- 10.1 Read the concentration, in micrograms/mL, corresponding to each meter reading.
- 10.2 Corrections for the blank must be made for each sample.

$$\text{micrograms/mL} = \text{micrograms/mL sample} - \text{micrograms/mL blank}$$

where:

$$\text{micrograms/mL sample} = \text{micrograms/mL found in front section of sample tube}$$

$$\text{micrograms/mL blank} = \text{micrograms/mL found in front section of blank tube}$$

A similar procedure is followed for the backup sections.

- 10.3 Determine the micrograms/sample by making the following volume correction.

$$\text{Micrograms/sample} = \text{micrograms/mL} \times 15 \text{ mL} \times \frac{10 \text{ mL}}{8 \text{ mL}}$$

- 10.4 Add the weights found in the front and backup sections to determine the total weight of the sample.

- 10.5 Read the desorption efficiency from the curve (see Section 8.6.2) for the amount found in the front section. Divide the total weight by this desorption efficiency to obtain the corrected micrograms/sample.

$$\text{Corrected micrograms/sample} = \frac{\text{Total weight}}{\text{D.E.}}$$

- 10.6 For personal sampling pumps with rotameters only, the following correction should be made.

$$\text{Corrected Volume} = f \times t \left(\sqrt{\frac{P_1}{P_2} \times \frac{T_2}{T_1}} \right)$$

where:

- f = flow rate sampled
- t = sampling time
- P₁ = pressure during calibration of sampling pump (mm Hg)
- P₂ = pressure of air sampled (mm Hg)
- T₁ = temperature during calibration of sampling pump (°K)
- T₂ = temperature of air sampled (°K)

- 10.7 The concentration of ammonia in the air sampled can be expressed in mg/cu m.

$$\text{mg/cu m} = \frac{\text{Corrected micrograms (Section 10.5)}}{\text{Corrected air volume (liters) (Section 10.6)}}$$

- 10.8 Another method of expressing concentration is ppm.

$$\text{ppm} = \text{mg/cu m} \times \frac{24.45}{\text{M.W.}} \times \frac{760}{P} \times \frac{T + 273}{298}$$

where:

- P = pressure (mm Hg) of air sampled
- T = temperature (°C) of air sampled
- 24.45 = molar volume (liter/mole) at 25°C and 760 mm Hg
- M.W. = molecular weight of ammonia
- 760 = standard temperature (°K)
- 298 = standard temperature (°K)

11. References

- 11.1 Documentation of NIOSH Validation Tests, National Institute for Occupational Safety and Health, Cincinnati, Ohio (DHEW-NIOSH-Publication No. 77-185), 1977. Available from Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., Order No. 017-033-00231-2.
- 11.2 Backup Data Report for Ammonia, prepared under NIOSH Contract No. 210-76-0123.

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SULFATES, SULFITES AND SULFUR DIOXIDE

SULFATES, SULFITES AND SULFUR DIOXIDE

Measurements Research Branch

Analytical Method

Analyte:	Sulfates, Sulfites and Sulfur Dioxide	Method No.:	P&CAM 268
Matrix:	Air	Range:	Sulfates: 0.1-10 mg/m ³ Sulfites: 0.1-10 mg/m ³ SO ₂ : 0.04-4 ppm (200-L air sample)
Procedure:	Particulate sulfates and sulfites collected on filter; SO ₂ on treated filter; analysis by ion chromatography	Precision:	5% (Analytical)
Date Issued:	7/2/79		
Date Revised:		Classification:	E (Proposed)

1. Synopsis

A known volume of air is drawn through a filter train consisting of a cellulose ester membrane filter followed by an impregnated cellulose filter containing potassium hydroxide. Particulate matter, including sulfates and sulfites, is collected on the first filter, while sulfur dioxide passes through the first filter and is collected on the second filter.

The filters are extracted with deionized water and the extracts are analyzed by anion-exchange chromatography. The following quantities are obtained:

SO₂ concentration: calculated from the sulfite peak on the impregnated cellulose filter chromatogram.

Total sulfates concentration (sulfuric acid plus soluble metal sulfates): from the sulfate peak on the untreated cellulose ester membrane filter chromatogram.

Particulate sulfites concentration: from the sulfite peak on the untreated cellulose ester membrane filter chromatogram.

2. Working Range, Sensitivity, and Detection Limit

- 2.1 The working range for a 200-L air sample is 0.1-10 mg SO_4^{2-} or $\text{SO}_3^{2-}/\text{m}^3$, and 0.04-4 ppm SO_2 (0.1-10 mg SO_2/m^3). This corresponds to 20-2000 μg of sulfate, sulfite or sulfur dioxide per sample.
- 2.2 The sensitivity at 30 μmho full scale is 5 μg sulfate, sulfite, or sulfur dioxide per sample per mm chart deflection. The sensitivity may be improved by using scale expansion on the readout and by using a smaller volume than 10 mL to desorb the sample.
- 2.3 The detection limit is approximately 0.5 μg SO_4^{2-} or $\text{SO}_3^{2-}/\text{mL}$ in the solution injected, corresponding to 5 μg sulfate, sulfite, or sulfur dioxide per sample.

3. Interferences

- 3.1 Oxidation of particulate sulfite on the sample filters results in a positive bias for sulfates and a negative bias for particulate sulfites.
- 3.2 Sulfur trioxide gas, if present in dry atmospheres, gives a positive bias in the sulfur dioxide determination.
- 3.3 Nitrate or phosphate ions may give similar retention times to sulfite. Identity of the sulfite peak may be established by spiking the samples with known amounts of sulfite and analyzing with at least two different eluents (e.g., the eluent in Section 7.14 and 0.003 M NaCO_3 /0.001 M NaHCO_3).
- 3.4 Insoluble sulfates collected on the first filter will not be measured unless special care is taken to dissolve them.

4. Precision and Accuracy

- 4.1 The relative standard deviation of the analytical method is 5% or less in the range 50-1000 μg SO_3^{2-} or SO_4^{2-} per sample, corresponding to 0.25-5 mg/m³ SO_2 , sulfites, or sulfates.
- 4.2 A major factor affecting accuracy is the tendency of particulate sulfites and absorbed sulfur dioxide to oxidize. Because of this, a negative bias which has not been thoroughly investigated occurs.

5. Advantages and Disadvantages

- 5.1 The sampling device uses only filters and involves no liquids.
- 5.2 Oxidation of a significant fraction of the particulate sulfites and sulfur dioxide in the sample is unavoidable.
- 5.3 Because identification is based on retention time, interferences may not be easily identified (see Section 3.3).

6. Apparatus

- 6.1 The apparatus for the collection of personal air samples consists of:
 - 6.1.1 Filter holder, 3-piece cassette, polystyrene, 37-mm diameter.
 - 6.1.2 Shrinkable cellulose band.
 - 6.1.3 Mixed cellulose ester membrane filter, 0.8 micrometer pore size, 37-mm diameter, supported by a cellulose backup pad.
 - 6.1.4 Cellulose filter, Whatman-40 or equivalent, impregnated with potassium hydroxide-glycerine solution, supported by a cellulose backup pad. To prepare the filter, saturate it with filter impregnating solution on a clean glass plate or watch glass and dry at 100°C for 20-30 minutes.
 - 6.1.5 Personal sampling pump whose flow can be calibrated in line with a representative loaded filter holder to an accuracy of $\pm 3\%$ at the recommended flow rate.
 - 6.1.6 Thermometer
 - 6.1.7 Manometer
 - 6.1.8 Stopwatch
 - 6.1.9 Screw cap, glass bottles, such as scintillation vials.
 - 6.1.10 Tweezers
- 6.2 Ion-exchange chromatograph, equipped with electrical conductivity detector and recorder or integrator.
- 6.3 10-mL pipette
- 6.4 10-mL plastic syringe with male Luer fitting
- 6.5 In-line filter with Luer fitting, 25 mm diam (0.8 μ m membrane filter).
- 6.6 Volumetric flask, 100 mL

7. Reagents

All reagents used should be ACS Reagent Grade or better.

- 7.1 Deionized, filtered water. Conductivity-grade deionized water with a specific conductance of 10 μ mho/cm or less is needed for preparation of eluents and other solutions which will be used on the ion chromatograph. The water should be filtered through a membrane filter (0.45-0.8 μ m pore size) before use to avoid plugging valves on the chromatograph.

- 7.2 Potassium hydroxide, KOH (pellets)
- 7.3 Glycerol
- 7.4 Sodium carbonate, Na_2CO_3
- 7.5 Sodium bicarbonate, NaHCO_3
- 7.6 Sodium sulfite, Na_2SO_3
- 7.7 Sodium sulfate, Na_2SO_4
- 7.8 Nitrogen gas
- 7.9 Filter impregnating solution. Dissolve 20 g KOH in about 50 mL deionized water, add 10 mL glycerol and dilute with deionized water to 100 mL.
- 7.10 Sulfite stock standard (1000 ppm $\text{SO}_3^{=}$). Add 5 mL glycerol to a 100 mL volumetric flask and dissolve in approximately 75 mL deionized water which has been heated to 100°C and cooled under nitrogen to remove dissolved oxygen. Add 0.1575 g Na_2SO_3 and dilute to 100 mL with deionized water. This standard should be prepared fresh weekly.
- 7.11 Sulfite working standard (100 ppm $\text{SO}_3^{=}$). Pipette 10.0 mL of 1000 ppm sulfite stock standard into a 100 mL³ volumetric flask and dilute to 100 mL with a solution containing 2% (v/v) glycerol. Prepare fresh daily.
- 7.12 Sulfate stock standard (1000 ppm $\text{SO}_4^{=}$). Dissolve 1.4792 g Na_2SO_4 in deionized water and dilute to 1 liter.
- 7.13 Sulfate working standard (100 ppm $\text{SO}_4^{=}$). Dilute 10.0 mL of the sulfate stock standard to 100 mL with deionized water.
- 7.14 Eluent ($0.003 \text{ M CO}_3^{=} / 0.003 \text{ M HCO}_3^{-}$). Dissolve 1.27 g Na_2CO_3 and 1.01 g NaHCO_3 in 4 liters of deionized, filtered water.

8. Procedure

- 8.1 Cleaning of Equipment. Glassware, including screw cap bottles, should be washed in detergent and rinsed in dilute (1-5%) nitric acid, followed by thorough rinsing with distilled or deionized water.
- 8.2 Collection and Shipping of Samples
 - 8.2.1 Each personal sampling pump must be calibrated with a representative filter cassette in line to assure accurately known sample volumes.

- 8.2.2 Assemble the filter cassette as follows: First, place a backup pad in place in the rear section of the cassette. On top of this place a treated cellulose filter (Sec. 6.1.4) and then put the center retaining ring of the cassette in place. Next, put another backup pad on top of the retaining ring, place a mixed cellulose ester membrane filter (Sec. 6.1.3) on top of the backup pad, and put the front section of the cassette in place. A shrinkable band should be used to seal the cassette.
- 8.2.3 Collect the sample at 1.5 liters per minute. The air being sampled should not pass through any hose or tubing before entering the cassette. A sample size of 200 liters is recommended.
- 8.2.4 If significant amounts of sulfuric acid are suspected in the sample, the cellulose ester membrane filter must be transferred to a clean, glass bottle within 4 hours of sampling to avoid low recovery of sulfate. Handle the filter with tweezers to avoid contamination. Reclose the cassette containing the treated cellulose filter.
- 8.2.5 Carefully record the sample identity and all pertinent sampling data. With each batch of up to 10 samples submit appropriate blank filters for analysis.

8.3 Analysis of Samples

- 8.3.1 Put the two filters from the cassette into two separate, clean, screw-top glass bottles. Add 10.0 mL eluent (Sec. 7.14) to each bottle and let stand, with occasional vigorous shaking, for 30 minutes.
- 8.3.2 Pour the contents of the bottle into a syringe fitted with an in-line filter and collect the filtrate in a second syringe.
- 8.3.3 Inject the filtered sample onto the chromatograph and record the sample identity and instrumental conditions. Typical operating conditions are:
- sensitivity: 30 μ mho full scale (for 5-100 ppm sulfate and sulfite)
 - eluent: 0.0030 M Na_2CO_3 , 0.0030 M NaHCO_3
 - flow rate: 138 mL/hr
 - separator column: 3 mm I.D. x 500 mm (anion exchanger), preceded by a precolumn
 - suppressor column: 6 mm I.D. x 250 mm (cation exchanger)

- SO_3^- retention time: 6-7.5 min (depending on eluent)
- SO_4^- retention time: 9-10.5 min (depending on eluent)

8.3.4 Measure and record the peak height or peak area of each sulfite and sulfate peak. If interfering substances (e.g., nitrate or phosphate) are present, establish positive identity of sulfite and sulfate peaks by adding known amounts of standard solutions and by changing eluent concentration for better separation, if necessary.

9. Calibration and Standardization

- 9.1 From the 100 ppm working standards, prepare 5, 10, 15, 20, 30, 50, and 80 ppm sulfate and sulfite standards by diluting, respectively, 0.5, 1.0, 1.5, 2.0, 3.0, 5.0, and 8.0 mL to 10 mL with deionized water. These standard solutions should be prepared fresh daily.
- 9.2 With each set of samples analyzed, a complete calibration curve should be constructed, using the standards prepared in 9.1 or additional standards as needed. Plot peak height or peak area vs. concentration for both sulfite and sulfate. A sulfite standard with nominal concentration C_n (ppm) will give two peaks: a sulfite peak, C , and a sulfate peak, C_s (ppm). The relationship between these is $C = C_n - C_s \times 0.8334$.

10. Calculations

- 10.1 From the calibration curves obtained in Sec. 9.2, read the concentrations of sulfite and sulfate ions in each sample in ppm. Designate whether the ions originated on the cellulose ester membrane filter or the treated cellulose filter. Thus, four concentrations will be obtained.

C_1 = concentration, ppm, of sulfite from cellulose ester membrane filter

C_2 = concentration, ppm, of sulfate from cellulose ester membrane filter

C_3 = concentration, ppm, of sulfite from treated cellulose filter

C_4 = concentration, ppm, of sulfate from treated cellulose filter

10.2 Calculate the concentrations in the air sample using the formulae:

$$\text{Total particulate sulfite (mg/m}^3\text{)} = \frac{C_1 \times 10}{V}$$

$$\text{Total particulate sulfate (mg/m}^3\text{)} = \frac{C_2 \times 10}{V}$$

$$\text{Sulfur dioxide (mg/m}^3\text{)} = \frac{(C_3 \times 10 \times 0.08002) + (C_4 \times 10 \times 0.6669)}{V}$$

$$\text{Sulfur dioxide (ppm)} = 0.3817 \times \text{sulfur dioxide (mg/m}^3\text{)} \times \frac{760 \times T}{298 \times P}$$

where V is the volume (liters) of air sampled.

T is the absolute temperature (°K = °C + 273) at which the sample was taken.

P is the pressure (mm Hg) at which the sample was taken.

11. References

- 11.1 Mulik, J.D., R. Puckett, D. Williams, and E. Sawicki: Analysis of Nitrate and Sulfate in Ambient Aerosols. Anal. Lett. 9: 653(1976)
- 11.2 Pate, J.B., Lodge, and M.P. Neary: The Use of Impregnated Filters to Collect Traces of Gases in the Atmosphere. Anal. Chim. Acta 28: 341 (1963)

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Section

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TOTAL SUSPENDED PARTICULATES

DEFINITION: Total aerosol mass

NUISANCE DUST, TOTAL

METHOD: 0500
ISSUED: 2/15/84

OSHA: 15 mg/m³
NIOSH: no standard
ACGIH: 10 mg/m³, total dust less than
1% quartz

PROPERTIES: quartz less than 1% [1]

SYNONYMS: boron oxide (CAS #1303-86-2) and nuisance dusts [1] including alumina (CAS #1344-28-1), calcium carbonate (CAS #1317-65-3), cellulose (paper fiber; CAS #9004-34-6), glycerin mist (CAS #56-81-5), limestone (CAS #1317-65-3), etc.

SAMPLING	MEASUREMENT
SAMPLER: FILTER (tared 37-mm, 5-µm PVC filter)	! TECHNIQUE: GRAVIMETRIC (FILTER WEIGHT) !
FLOW RATE: 1.5 to 2 L/min	! ANALYTE: airborne particulate material !
VOL-MIN: 25 L @ 15 mg/m ³ -MAX: 133 L @ 15 mg/m ³	! BALANCE: 0.01 mg sensitivity or better; use same ! balance before and after sample ! collection !
SHIPMENT: routine	! CALIBRATION: National Bureau of Standards ! Class M weights !
SAMPLE STABILITY: indefinitely	! RANGE: 0.3 to 2 mg per sample !
BLANKS: 2 field blanks per 10 samples	! ESTIMATED LOD: 0.2 mg per sample !
BULK SAMPLE: none required	! PRECISION: 0.08 mg per sample [3] !
ACCURACY	!
RANGE STUDIED: 8 to 28 mg/m ³	!
BIAS: not significant	!
OVERALL PRECISION (s _p): 0.056 [2]	!

APPLICABILITY: The working range is 3 to 20 mg/m³ for a 100-L air sample. This method is nonspecific and determines the total dust concentration to which a worker is exposed. It may be applied, e.g., to gravimetric determination of fibrous glass [4] in addition to the other ACGIH nuisance dusts [1].

INTERFERENCES: Organic and volatile particulate matter may be removed by dry ashing [4].

OTHER METHODS: This method is similar to the criteria document method for fibrous glass [4] and Method 5000 for carbon black. This method replaces Method S349 [5]. Impingers and direct-reading instruments may be used to collect total dust samples, but these have limitations for personal sampling.

EQUIPMENT:

1. Environmental chamber at constant temperature and humidity (e.g., $20\text{ }^{\circ}\text{C} \pm 0.3\text{ }^{\circ}\text{C}$ and $50\% \pm 5\% \text{ RH}$).
2. Sampler: 37-mm PVC, 2- to 5- μm pore size membrane or equivalent hydrophobic filter and cellulose supporting pad in 37-mm cassette filter holder.
3. Personal sampling pump, 1.5 to 2 L/min, with flexible connecting tubing.
4. Microbalance, capable of weighing to 0.01 mg.
5. Vacuum desiccator.
6. Static neutralizer: e.g., Po-210; replace nine months after the production date.

SPECIAL PRECAUTIONS: None.

PREPARATION OF FILTERS BEFORE SAMPLING:

1. Dry filters and backup pads under vacuum in the vacuum desiccator for at least 15 min.
2. Release the vacuum, remove the desiccator cover and equilibrate the filters in the environmental chamber for at least 1 hr.
3. Number the backup pads with a ballpoint pen and place them, numbered side down, in filter cassette bottom sections.
4. Weigh the filters in the environmental chamber. Record the filter tare weight, W_1 (mg).
 - a. Zero the balance before each weighing.
 - b. Handle the filter with forceps (nylon forceps if further analyses will be done).
 - c. Pass the filter over an antistatic radiation source. Repeat this step if filter does not release easily from the forceps or if filter attracts balance pan. Static electricity can cause erroneous weight readings.
5. Place the weighed filters on top of the backup pads in the filter cassette bottom sections and allow to stand an additional 8 to 16 hrs in the environmental chamber.
6. Reweigh the filters. If this tare weight differs by more than 0.01 mg from the first tare weight obtained in step 4 above, discard the filter.

NOTE: Insert a rod through the outlet hole of the filter cassette bottom section to raise the backup pad and filter so that the filter can be grasped with forceps.
7. Assemble the filter in the filter cassettes and close firmly so that leakage around the filter will not occur. Place a plug in each opening of the filter cassette. Place a cellulose shrink band around the filter cassette, allow to dry and mark with the same number as the backup pad.

SAMPLING:

8. Calibrate each personal sampling pump with a representative sampler in line.
9. Sample at 1.5 to 2 L/min. Do not exceed a total filter loading of approximately 2 mg total dust.

SAMPLE PREPARATION:

10. Wipe dust from the external surface of the filter cassette with a moist paper towel to minimize contamination. Discard the paper towel.
11. Remove the top and bottom plugs from the filter cassette. Place the filter cassettes in a vacuum desiccator under vacuum for at least 15 min, followed by equilibration for at least 1 hr in the environmental chamber.
12. Remove the cassette band, pry open the cassette and remove the filter. Handle the filters very gently by the edge to avoid loss of dust.

NOTE: If the filter sticks to the underside of the cassette top, very gently lift away by using the dull side of a scalpel blade. This must be done carefully or the filter will tear.

CALIBRATION AND QUALITY CONTROL:

13. Zero the microbalance before all weighings. Use the same microbalance for weighing filters before and after sample collection. Maintain and calibrate the balance with National Bureau of Standards Class M weights.
14. Take two to four replicate samples for every batch of field samples for quality assurance on the sampling procedures. The set of replicate samples should be exposed to the same dust environment, either in a laboratory dust chamber [6] or in the field. The quality control samples must be taken with the same equipment, procedures and personnel used in the routine field samples. The relative standard deviation calculated from these replicates should be recorded on control charts and action taken when the precision is out of control.

MEASUREMENT:

15. Weigh each filter, including field blanks. Record this post-sampling weight, W_2 (mg), beside its corresponding tare weight. Record anything remarkable about a filter (e.g., overload, leakage, wet, torn, etc.).

CALCULATIONS:

16. Calculate the concentration of total nuisance dust, C (mg/m^3), in the air volume sampled, V (L):

$$C = \frac{(W_2 - W_1) + B}{V} \cdot 10^3, \text{ mg}/\text{m}^3$$

where: W_1 = tare weight of filter before sampling (mg)

W_2 = post-sampling weight of sample-containing filter (mg)

B = mean change in field blank filter weights between tare and post-sampling (mg)
(+ or -).

EVALUATION OF METHOD:

Lab testing with blank filters and generated atmospheres of carbon black was done at 8 to 28 mg/m^3 [2,6]. Precision and accuracy data are given on page 0500-1.

REFERENCES:

- [1] TLVs - Threshold Limit Values for 1983-84, Appendix D, ACGIH, Cincinnati, OH (1983).
- [2] This Manual, Method 5000.
- [3] Unpublished data from Non-textile Cotton Study, NIOSH/DRDS/EIB.
- [4] NIOSH Criteria for a Recommended Standard ... Occupational Exposure to Fibrous Glass, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-152, 119-142 (1977).
- [5] NIOSH Manual of Analytical Methods, 2nd ed., V. 3, S349, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).
- [6] Documentation of the NIOSH Validation Tests, S262 and S349, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).

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RESPIRABLE SUSPENDED PARTICULATES

FORMULA: The respirable fraction of the dust mass, as specified by the American Conference of Governmental Industrial Hygienists [1]

NUISANCE DUST, RESPIRABLE

METHOD: 0600
ISSUED: 2/15/84

OSHA: 5 mg/m³
NIOSH: no standard
ACGIH: 5 mg/m³

PROPERTIES: Penetrates the non-ciliated portions of the lung; quartz less than 1%

SYNONYMS: boron oxide (CAS #1303-86-2) and nuisance dusts [2], including alumina (CAS #1344-28-1), calcium carbonate (CAS #1317-65-3), cellulose (paper fiber; CAS #9004-34-6), glycerin mist (CAS #56-81-5), limestone (CAS #1317-65-3), etc.

SAMPLING	MEASUREMENT
SAMPLER: CYCLONE + FILTER (10-mm Dorr-Oliver cyclone + tared 5- μ m PVC membrane)	TECHNIQUE: GRAVIMETRIC (FILTER WEIGHING) ANALYTE: mass of respirable dust fraction
FLOW RATE: 1.7 L/min	BALANCE: 0.01 mg sensitivity or better; use same balance before and after sample collection
VOL-MIN: 75 L @ 5 mg/m ³ -MAX: 1000 L @ 5 mg/m ³	CALIBRATION: National Bureau of Standards Class M weights
SHIPMENT: routine	RANGE: 0.3 to 2 mg per sample
SAMPLE STABILITY: indefinitely	ESTIMATED LOD: 0.2 mg per sample
BLANKS: 2 to 10 field blanks per set	PRECISION: 68 μ g with 0.01-mg sensitivity balance [5]
ACCURACY	
RANGE STUDIED: 0.5 to 10 mg/m ³ (lab and field)	
BIAS: depends on dust size distributions [3]	
OVERALL PRECISION (s_p): 0.043 to 0.145 (lab); 0.144 to 0.227 (field) [4]	

APPLICABILITY: The method measures the mass concentration of any non-volatile respirable dust. Besides inert dusts [1], the method is recommended for respirable coal dust, which has an OSHA PEL = 2.4 mg/m³. The method may be biased where the respirable fraction is defined by the British Medical Research Council's criteria or the MRE horizontal elutriator [4].

INTERFERENCES: Larger than respirable particles (over 10 μ m) have been found in some cases by microscopic analysis of cyclone filters. Over-sized particles in the sample are known to be caused by inverting the cyclone assembly. Heavy dust loadings, charged particles, fibers and water-saturated dusts also interfere with the cyclone's size-selective properties.

OTHER METHODS: This method is based on and replaces Sampling Data Sheet #29.02 [6].

EQUIPMENT:

1. Sampler:
 - a. Filter: 37-mm diameter, 5.0- μ m pore size, polyvinyl chloride filter or equivalent hydrophobic membrane filter supported with backup pad in a two-piece, 37-mm cassette filter holder held together by tape or cellulose shrink band.
 - b. Cyclone: 10-mm Dorr-Oliver nylon cyclone.
 - c. Sampling head holder: this holder must keep the cassette, cyclone and coupler together rigidly so that air enters only at the cyclone inlet.
 2. Personal sampling pump, 1.7 L/min $\pm$ 5%, with flexible connecting tubing.
NOTE: Pulsation in the pump flow must be within $\pm$ 20% of the mean flow.
 3. Balance, analytical, with sensitivity of at least 0.01 mg. A more sensitive balance will be necessary for substances with PEL's below 1 mg/m³.
 4. Static neutralizer, e.g., Po-210; replace nine months after the production date.
 5. Environmental chamber for balance, e.g., 20 °C $\pm$ 0.3 °C and 50% $\pm$ 5% RH.
 6. Vacuum desiccator.
-

SPECIAL PRECAUTIONS: None.

PREPARATION OF SAMPLERS BEFORE SAMPLING:

1. Dry filters and backup pads under vacuum in the vacuum desiccator for at least 15 min.
2. Release the vacuum, remove the desiccator cover, and equilibrate the filters in the environmental chamber for at least 1 hr.
3. Number the backup pads with a ballpoint pen and place them, numbered side down, in filter cassette bottom sections.
4. Weigh the filters in the environmental chamber. Record the filter tare weight, W_1 (mg).
 - a. Zero the balance before each weighing;
 - b. Handle the filter with forceps (nylon forceps if further analyses will be done); and
 - c. Pass the filter over an antistatic radiation source. Repeat this step if filter does not release easily from the forceps or if filter attracts balance pan. Static electricity can cause erroneous weight readings.
5. Place the weighed filters on top of the backup pads in the filter cassette bottom sections and allow to stand an additional 8 to 16 hrs in the environmental chamber.
6. Reweigh the filters. If this tare weight differs by more than 0.01 mg from the first tare weight obtained in step 4 above, discard the filter.
NOTE: Insert a rod through the outlet hole of the filter cassette bottom section to raise the backup pad and filter so that the filter can be grasped with forceps.
7. Assemble the filters in the filter cassettes and close firmly so that leakage around the filter will not occur. Place a plug in each opening of the filter cassette. Place a cellulose shrink band around the filter cassette, allow to dry, and mark with the same number as the backup pad.
8. Remove the cyclone's grit cap and vortex finder before use and inspect the cyclone interior. If the inside is visibly scored, discard this cyclone since the dust separation characteristics of the cyclone might be altered. Clean the interior of the cyclone to prevent reentrainment of large particles.
9. Assemble the sampler head. Check alignment of filter holder and cyclone in the sampling head to prevent leakage.

SAMPLING:

10. Calibrate each personal sampling pump to 1.7 L/min with a representative sampler in line.
11. Sample at 1.7 L/min for 45 min to 8 hrs (76 to 816 L). Do not exceed 5 mg dust loading on the filter.

NOTE: Do not allow the sampler assembly to be inverted at any time. Turning the cyclone to anything more than a horizontal orientation may deposit over-sized material from the cyclone body onto the filter.

SAMPLE PREPARATION:

12. Wipe dust from the external surface of the filter cassette with a moist paper towel to minimize contamination. Discard the paper towel.
13. Remove the top and bottom plugs from the filter cassette. Place the filter cassettes in a vacuum desiccator under vacuum for at least 15 min, followed by equilibration for at least 1 hr in the environmental chamber.
14. Remove the filter cassette band, pry open the filter cassette, and remove the filter by inserting a rod in the outlet hole of the filter cassette. Handle the filters very gently by the edge to avoid loss of dust.

NOTE: If the filter sticks to the underside of the cassette top, very gently lift away by using the dull side of a scalpel blade. This must be done carefully or the filter will tear.

CALIBRATION AND QUALITY CONTROL:

15. Zero the microbalance before all weighings. Use the same microbalance for weighing filters before and after sample collection. Calibrate the balance with National Bureau of Standards Class M weights.
16. Take two to four replicate samples for every batch of field samples for quality assurance on the sampling procedures. The set of replicate samples should be exposed to the same dust environment, either in a laboratory dust chamber [7] or in the field [8]. The quality control samples must be taken with the same equipment, procedures and personnel used in the routine field samples. Calculate precision from these replicates and record s_r on control charts. Take corrective action when the precision is out of control [7].

MEASUREMENT:

17. Weigh each filter, including field blanks. Record this post-sampling weight, W_2 (mg), beside its corresponding tare weight. Record anything remarkable about a filter (e.g., visible particles, overloaded, leakage, wet, torn, etc.).

CALCULATIONS:

18. Calculate the concentration of respirable nuisance dust, C (mg/m³), in the air volume sampled, V (L):

$$C = \frac{(W_2 - W_1) + B}{V} \cdot 10^3, \text{ mg/m}^3$$

where: W_1 = tare weight of filter before sampling (mg)

W_2 = post-sampling weight of sample-containing filter (mg)

B = mean change in field blank filter weights between tare and post-sampling (mg) (+ or -).

EVALUATION OF METHOD:

1. Bias. In respirable dust measurements, the bias in a sample is calculated relative to the appropriate respirable dust criterion. The theory for calculating bias is developed by Bartley and Breuer [3]. For this method, the bias, therefore, depends on the ACGIH criterion for respirable dust, the cyclone's penetration curve at 1.7 L/min flow rate, and the size distribution of the ambient dust. Based on the cyclone's penetration curves for non-pulsating flow measured with a monodisperse aerosol by Caplan, Doemeny and Sorenson [9], the bias in this method is shown in Figure 1.

For dust size distributions in the shaded region, the bias in this method lies within the ± 0.10 criterion established by NIOSH for method validation. Bias larger than ± 0.10 would, therefore, be expected for many workplace aerosols, especially those with small mass median diameters. However, bias within ± 0.20 would be expected for dusts with geometric standard deviations greater than 2.0, which is the case in most workplaces.

Bias can also be caused in a cyclone by the pulsation of the personal sampling pump. Bartley, et al. [10] showed that cyclone samples with pulsating flow can have negative bias as large as -0.22 relative to samples with steady flow. The magnitude of the bias depends on the amplitude of the pulsation at the cyclone aperture and the dust size distribution. For pumps with instantaneous flow rates within 20% of the mean, the pulsation bias is less than -0.02 for most dust size distributions encountered in the workplace.

Electric charges on the dust and the cyclone will also cause bias. Briant and Moss [11] have found electrostatic biases as large as -50%, and show that cyclones made with graphite-filled nylon eliminate the problem.

2. Precision. In a recent review [4], the overall cyclone precision is shown to be most sensitive to two factors: the analytical precision and the sampling procedures, particularly the quality control system used in the maintenance and calibration of samplers. Theoretically, the variance for the overall precision is the sum of the variances from the sampling and analysis. The analytical variance depends on the dust loading on the filter. For the dust loading in an 8-hr sample above 1.5 mg/m³, Bowman, et al. [4] find that the empirically determined sampling error dominates this analytical error.

Because of the effects of the environment, precision estimates for dust samplers are much more variable than those reported for gas and vapor sampling. In laboratory tests with 0.01 mg sensitivity balances, the overall precision of a single respirable dust sample has relative standard deviations (s_r) from 0.043 to 0.145 over concentrations ranging from 0.5 to 5 mg/m³. In the laboratory studies where the dust concentrations in the test chamber are more carefully controlled, the estimated s_r is less than 0.091, which is the target precision value for a bias equal to ± 0.10 in the NIOSH validation criteria.

In the field tests with 0.01 mg sensitivity balances, precision estimates range from 0.144 to 0.227 over concentrations ranging from 1 to 10 mg/m³. Whether the larger s_r values in field tests are due to sampler performance or to more inhomogeneous dust concentrations in the field tests cannot be determined from existing data.

REFERENCES:

- [1] TLVs - Threshold Limit Values for Chemical Substances and Physical Agents in the Work Environment with Intended Changes for 1983-84, 38, ACGIH, Cincinnati, OH (1983).
- [2] Ibid, Appendix D, 52.
- [3] Bartley, D. L. and G. M. Breuer. Analysis and Optimization of the Performance of the 10-mm Cyclone, Am. Ind. Hyg. Assoc. J., 43, 520-528 (1982).
- [4] Bowman, J. D., D. L. Bartley, G. M. Breuer and S. A. Shulman. The Accuracy of Sampling Respirable Coal Mine Dust, Draft NIOSH report (1983).
- [5] Parobeck, P., T. F. Tomb, H. Ku and J. Cameron. Measurement Assurance Program for the Weighings of Respirable Coal Mine Dust Samples, J. Qual. Tech., 13, 157 (1981).
- [6] NIOSH Manual of Sampling Data Sheets, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-159 (1977).
- [7] Bowman, J. D., D. L. Bartley, G. M. Breuer, L. J. Doemeny and D. J. Murdock. Accuracy Criteria Recommended for the Certification of Gravimetric Coal Mine Dust Personal Samplers, NIOSH report (in press, 1983).
- [8] Breslin, J. A., S. J. Page and R. A. Jankowski. Precision of Personal Sampling of Respirable Dust in Coal Mines, U.S. Bureau of Mines Report of Investigations #8740 (1983).
- [9] Caplan, K. J., L. J. Doemeny and S. Sorenson. Evaluation of Coal Mine Dust Personal Sampler Performance, Final Report, NIOSH Contract No. PH CPE-r-70-0036 (1973).
- [10] Bartley, D. L., G. M. Breuer, P. A. Baron and J. D. Bowman. Pump Fluctuations and Their Effect on Cyclone Performance, submitted to the Am. Ind. Hyg. Assoc. J. (1983).
- [11] Briant, J. K. and O. R. Moss. The Influence of Electrostatic Charge on the Performance of 10-mm Nylon Cyclones, American Industrial Hygiene Conference (1983).

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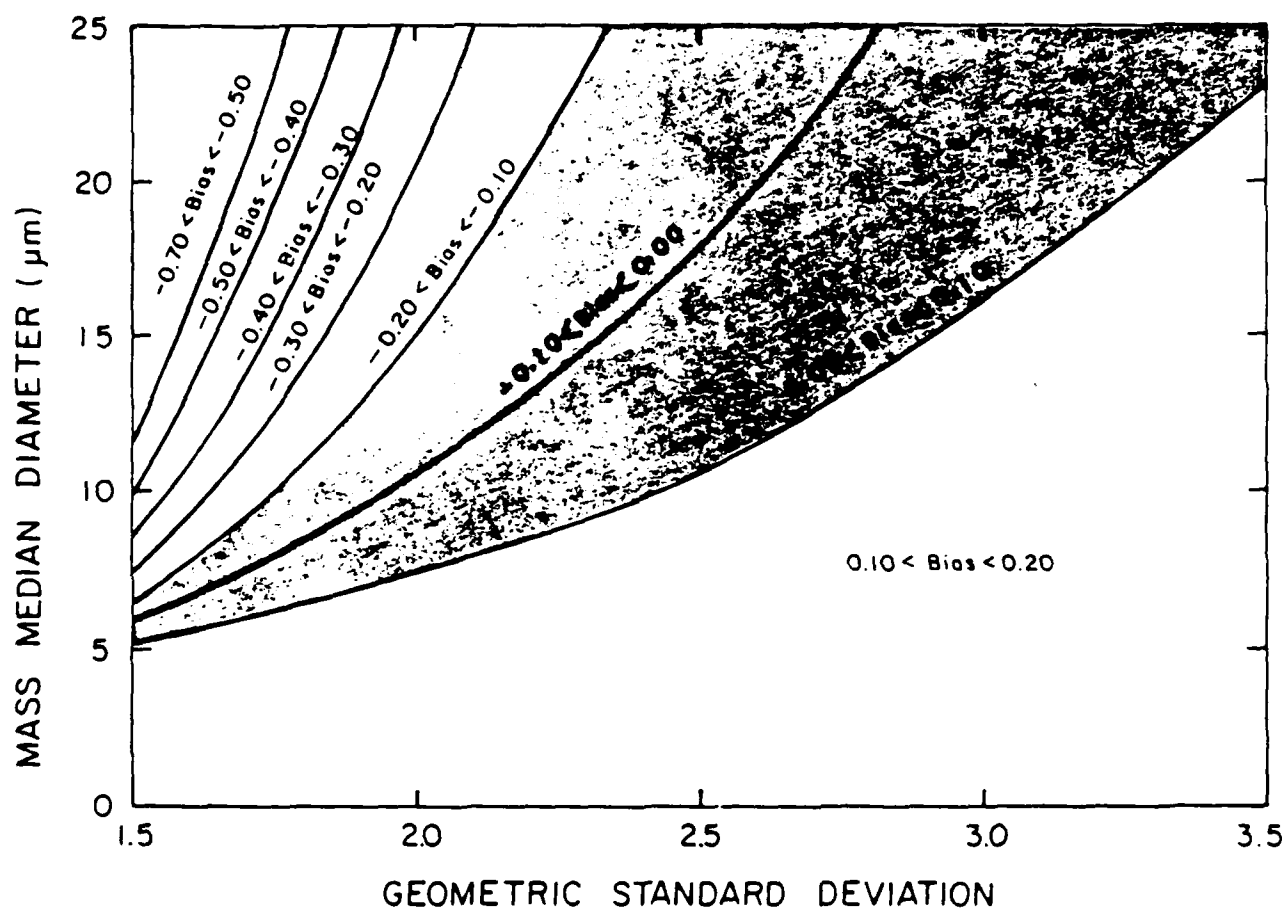


Figure 1. Bias in respirable dust determination.

2/15/84

NITRO-POLYCYCLIC AROMATIC HYDROCARBONS

ANALYTICAL PROCEDURES FOR NITRO-PAHS IN DIESEL PARTICULATE EXTRACTS

- Analytes:
 - 2-Nitrofluorene
 - 3-Nitro-9-fluorenone
 - 9-Nitroanthracene
 - 3-Nitrofluoranthene
 - 1-Nitropyrene
 - 2,7-Dinitrofluorene
 - 1,8-Dinitropyrene
 - 6-Nitrobenzo(a)pyrene
- Summary: Diesel particulate extracts in methylene chloride are separated into aliphatic and aromatic fractions using HPLC/UV. The aromatic fraction is analyzed for nitro-PAHs using GC/ECD.
- Sample Preparation:
 1. Dissolve the diesel extract in methylene chloride (to 5 mg/mL).
 2. Dilute 100 uL of the 5 mg/mL sample to 0.5 mL with 1% MeOH in C₆.
 3. Inject 200 uL of a nitro-PAH standard into HPLC system. HPLC conditions are as follows:
 - Column: uBondapak NH₂
7.8 mm x 30 cm
 - Mobile Phase: 10/90 CH₂Cl₂-C₆
1.5 mL/min
 - UV Detector: 254, x0.2 AUFS
 4. The results from the standard chromatogram determine where to fraction the diesel extracts. The nitro-PAHs typically elute between 12.5 and 18.75 minutes.

APPENDIX C (Continued)

5. Fraction a 200-uL aliquot of the MeOH/C₆ diesel sample. Collect only the 12.5-18.75 min fraction for analysis. The fractionation is done with the UV lamp off and is based on retention time. Keep the room as dark as possible.
6. Concentrate the sample to 0.5 mL in hexane using N₂ and a hot water bath. Add 1 uL of lindane² (0.75 ng/uL) as an internal standard.

• Sample
Analysis:

1. Inject 1 uL of the extract onto the GC. Chromatographic conditions are as follows:

Column: DB-5 30 m x 0.32 mm fused-silica capillary column

Carrier Gas: Helium, 2 mL/min

Makeup Gas: Nitrogen, 8-9 mL/min

Column Temp.: 40°C (1 min)
15°C/min → 150°C
5°C/min → 300°C (5 min)

Injector Temp.: 250°C

ECD Temp.: 315°C

2. Calibration standards containing 5-150 ppb of each analyte should be analyzed with the samples.

HYDROGEN CYANIDE

A personal sampling apparatus for monitoring CO , CO_2 , O_2 , NO_2 , HCl , HCN and particulate content. Two fire companies made ninety successful sample runs during structural fires. CO presented a potential acute hazard and particulate concentrations were high. HCN was detected at low levels in half the samples. HCl was detected in only eight samples but on two occasions exceeded 100 ppm. CO and NO_2 levels and O_2 depression do not appear to represent significant hazards.

Exposure of firefighters to toxic air contaminants

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Introduction

Despite considerable laboratory work and test data, little field data on the exposure of firefighters to toxic combustion gases are available.¹⁻³ In addition to acute hazards, significant long range health effects are implicated in firefighting. One study⁴ has determined that pulmonary function as measured by FVC and FEV₁ decreases twice as fast among firefighters as among the general population. The same study demonstrated correlation between frequency and estimated severity of exposure and accelerated loss of lung function among individual firefighters. Other work⁵ has identified heart disease as a special problem of firefighters, possibly arising from extensive stress including exposure to high levels of CO . These and other studies which implicate inhalation of combustion products as a significant factor in morbidity and mortality among firefighters point up the urgency of examining quantitatively the atmosphere to which firefighters are exposed on the job. This paper describes the development and use of personal sampling as a means for evaluating airborne contaminants encountered during structural firefighting operations by two units of the Boston Fire Department.

Six gases, O_2 , CO_2 , CO , NO_2 , HCl and HCN were monitored in this study. In addition, provision was later made to collect and measure total particulates. Oxygen was selected in order to determine whether depressed O_2 levels often

reported in experimental burns are a hazard in real fire situations. Previous field work in which CO and O_2 were monitored at real fires has not shown this to be the case.⁶ Carbon monoxide, a product of incomplete combustion of carbonaceous materials was selected for monitoring because it is ubiquitous at fires and is currently considered to represent the most dangerous acute exposure faced by firefighters.⁷⁻⁹ Carbon dioxide, the end product of complete combustion of carbon containing materials, has been reported in high concentration in experimental burns¹⁰ and was selected for study since it is considered by some workers to represent a major hazard. Nitrogen dioxide is a highly toxic gas whose presence at fires might be expected through fixation of atmospheric nitrogen¹¹ and, to a lesser extent from the oxidation of nitrogenous materials. The extreme toxicity of nitrogen dioxide and the fact that firefighters have at times suffered symptoms consistent with exposure to this gas, lead to its inclusion in the study. Hydrochloric acid could arise from the pyrolysis and combustion of PVC-containing plastics frequently encountered in structural fires.¹² Sources of hydrogen cyanide are wool and plastics containing urethanes, acrylonitriles or polyamides.^{13,14} Because of the abundance of plastics in home furnishings, vehicles, and aircraft, HCl and HCN could be significant hazards to firefighters, and were therefore also monitored.

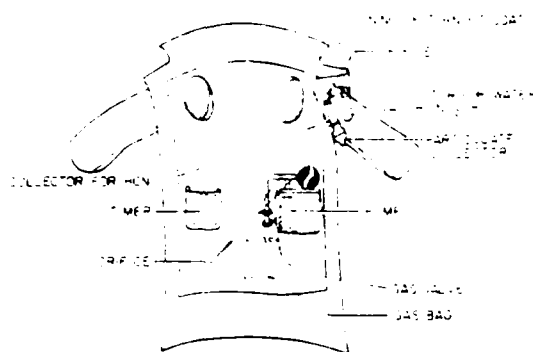


Figure 1 - Turnout coat equipped with sampling system

sampling system

During operations, Boston firefighters wear protective equipment weighing approximately sixty pounds. Any air sampling equipment must be compatible with this gear. To withstand severe mechanical stress during firefighting the sampler must be rugged and for safety reasons must in no way restrict the firefighter's movement. Since speed is of the utmost importance in firefighting, activation and shutdown of the system must be conveniently accomplished.

The sampling system is shown in Figure 1. All tubing in the system is polyethylene and all connections made with Swage Lok fittings. Tubing and wiring are concealed between the coat and liner and fastened to the liner at critical points. The external reagent tubes are secured below the collar by a fastener riveted to the coat and may be conveniently removed and exchanged by loosening the Swage Lok fittings. A 25 mm filter holder is fastened to one reagent tube by a rubber connector. A 2.5 l PVC grab sampling bag is suspended between the coat and liner. Upon completion of the sample run, the firefighter closes a 1/4-turn valve to retain the bag sample.

Orifices fashioned from 6 mm lengths of 23 ga. syringe needle soldered into the tees regulate the flow at approximately 0.3 L/min in all branches of the sampler. If the sampling period extends beyond the bag filling time (9 minutes) the flow through the reagent tubes decreases to 0.23 L/min, while the overflow sample is dumped through the open ended branch of the tee downstream from the pump.

The pump is an MSA Model G modified by removal of the flow control and rotameter and

placement of the switch in the rotameter recess. This placement allows easy operation of the pump while guarding against inadvertent operation of the switch. A timer is wired into the switch and records time directly in minutes.

The firefighters participating in the study were instructed in the design and use of the coat. They were asked to activate the pump at the immediate location of the fire and to shut down the sampler upon leaving the location. The firefighters filled out a questionnaire after each test. Two companies, Aerial Tower 2 and Engine 43, participated in the study; each made 45 sample runs.

analytical methods

Nitrogen dioxide. The analysis is based on a modified Saltzman method^{12,13} in which the NO_2 is trapped on 13X molecular sieves impregnated with triethanolamine (TEA). The sieves are contained in one of the reagent tubes, downstream from activated 13X sieves which serve to prevent condensation of water in the sample line (Figure 2 A). For the analysis, the sieves are thoroughly mixed and half are used for the NO_2 determination. The sieves are desorbed with 12 ml of a 0.1 M solution of TEA.¹² A 5 ml aliquot is removed for color development, with a final volume made up to 21 ml. For a 1000 ml gas sample, the detection limit is approximately 0.5 ppm.

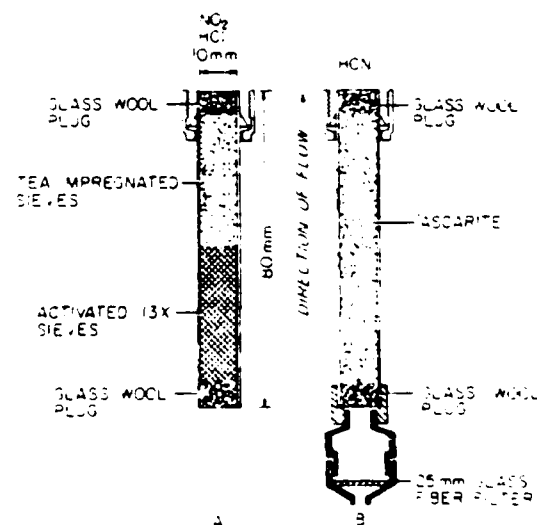


Figure 2 - Reagent tubes and filter for total particulates

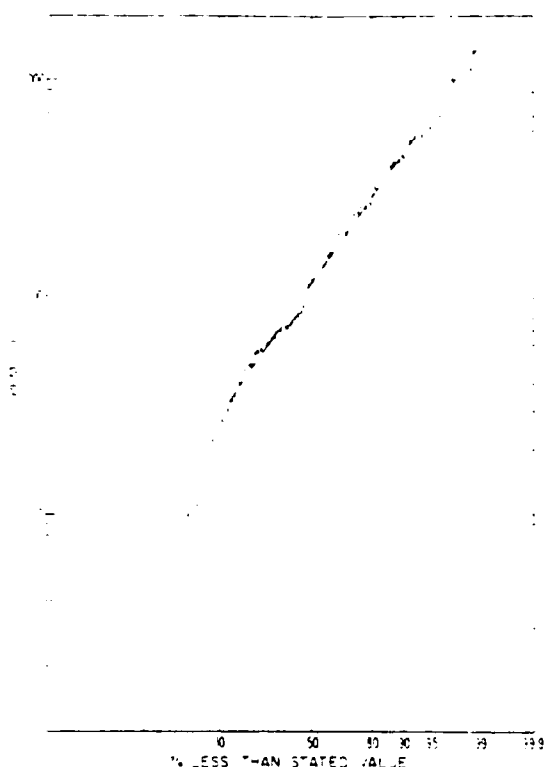


Figure 3 - Distribution of CO concentrations. Geometric mean: 110 ppm, geometric standard deviation: 3.0

Hydrogen chloride. The TEA impregnated sieves are an efficient trap for HCl and the remaining sieves are used for this determination. The mercuric thiocyanate method¹⁴ for chloride ion is employed. The sample is desorbed with 10 ml deionized water and a 5 ml aliquot removed for analysis. Final volume of the developed aliquot is 25 ml. Under these conditions the limit of detection is 20 ppm in a 1000 ml gas sample. TEA, acetic and formic acids, acetaldehyde and formaldehyde do not affect color development. More sensitive chloride determinations^{15,16} could not be adapted to the method of sample collection or to the batch type analytical operation required by the study.

Hydrogen cyanide. Hydrogen cyanide is collected on 30-60 mesh Ascarite in the second reagent tube (Figure 2 B) and determined colorimetrically by conversion to cyanogen chloride and oxidation of pyridine by cyanogen chloride to a dialdehyde which forms a chromophore with barbituric acid.¹⁷ The

Ascarite from the tube is dissolved in 25 ml distilled water, the solution filtered and a 10 ml aliquot of filtrate titrated with 4 N HCl to a phenolphthalein end point. The neutralized solution is treated with the colorimetric reagents and made up to a final volume of 25 ml. Sensitivity for a 1000 ml gas sample is approximately 0.09 ppm.

Carbon monoxide, oxygen and carbon dioxide. These three gases are determined in the bag sample at the fire station. CO is determined with an Ecolyzer Model 2400, O₂ is determined by a Beckman Model D paramagnetic oxygen analyzer and CO₂ by Bendix 2L CO₂ detector tubes.

Particulates. Particulates are collected on pretared 25 mm binderless glass fiber filters and determined gravimetrically. The filter cassette is attached to the Ascarite reagent tube (Figure 2 B).

discussion

CO, HCN, and particulate concentrations plotted in log-probability coordinates are

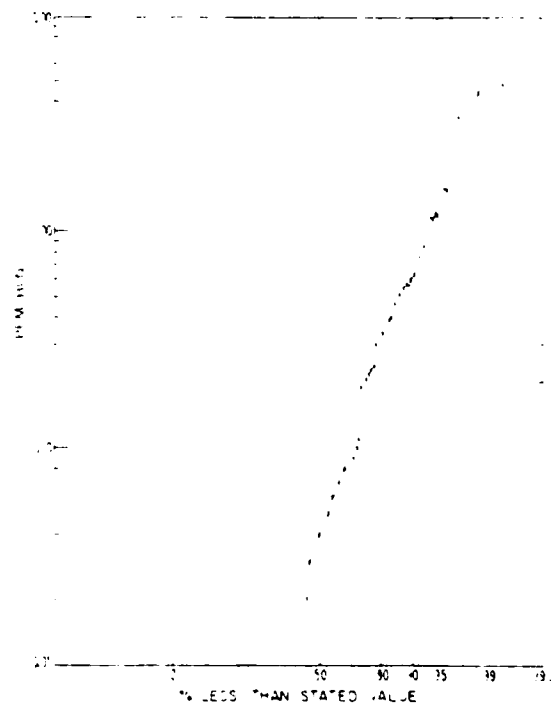


Figure 4 - Distribution of HCN concentrations. Geometric mean: 0.04 ppm, geometric standard deviation: 8.3

presented in Figures 3-5 with the best straight lines visually fitted to the data. The distributions appear to be lognormal, in conformance with much air sampling data.⁽¹⁹⁾ Data on O₂, CO₂, HCl, and NO₂ are summarized in Table I.

Sampling times were bimodally distributed around 7 and 9 minutes. CO was uniformly present at all fires at elevated levels. The highest concentrations were recorded at fires where there was general involvement of structures, furniture and trash and were not correlated with any specific materials. The median value for the CO samples was 110 ppm, with 3% exceeding 1000 ppm.

Particulates were also present in significant amounts, with a median concentration of 22 mg/m³ and 15% of the samples being in excess of 100 mg/m³. The highest particulate exposures occurred at fires involving the highest CO exposures.

Hydrogen cyanide was detected frequently, though at low levels. Of the 43 samples in which cyanide was detected, eleven were from fires that were confined to a few specific materials: one upholstered chair, five mattress, two tire and two vehicle fires and one fire involving butyl rubber and silicone rubber insulated wire in a curing oven. Of six incidents in which the HCN concentrations were over 1 ppm, three were mattress fires and one a vehicle fire.

Hydrogen chloride was detected in five fires. In all five cases there was general involvement of a room, its contents and an assortment of

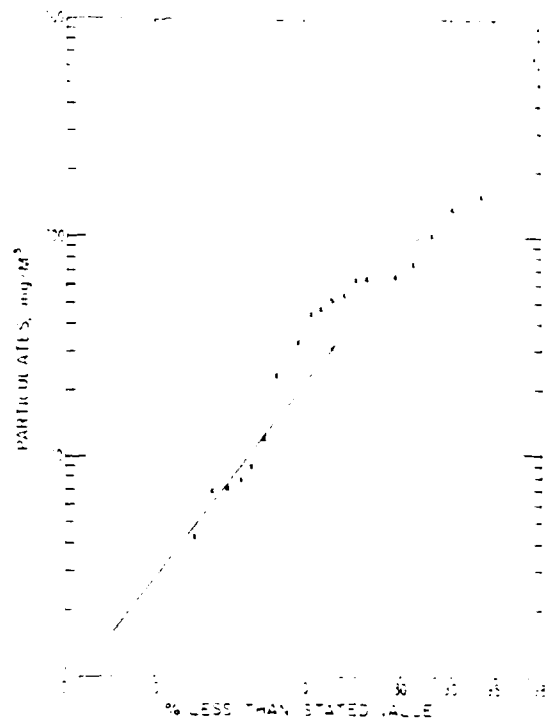


Figure 5 - Distribution of particulate concentrations. Geometric mean 21.5 mg/m³ geometric standard deviation, 4.7

rubbish. In two of the incidents "plastics" were specifically identified among the combustibles by firefighters. The maximum concentration recorded was 150 ppm.

Carbon dioxide concentrations never exceeded the lowest detectable limit of the

TABLE I

Summary of Data on O₂, CO₂, HCl and NO₂

Gas	No. samples taken	No. samples in which detected	Comments
O ₂	79	79	Depressed 0.5% in 7 samples 0.4% 4 0.3% 3 0.2% 12 0.1% 9
CO ₂	63		Never with certainty above 0.26%
HCl	10	5	Concentration (ppm): 18, 32, 75, 128, 150*
NO ₂	10	3	Concentration (ppm): 0.02, 0.29, 0.31, 0.37, 0.59*, 0.63, 0.64, 0.89

*Results questionable because of short sampling time

detector (0.26%), and oxygen levels below 20% were not recorded for any fire.

Nitrogen dioxide was detected on eight occasions, with 0.89 ppm being the highest concentration observed.

The data indicate that carbon monoxide is the one gas of those monitored that could involve a potential acute hazard for the firefighters of Aerial Tower 2 and Engine 43. Particulates may occur in high enough concentrations to have significant long term health effects. Although hydrogen cyanide was frequently detected, concentrations did not pose an acute hazard based on the Short Term Exposure Limit of 15 ppm.

The fire companies participating in this study are located in older, dilapidated residential sections of Boston. Structures are for the most part old and apt to contain fewer synthetic materials than those more recently constructed. Many structural fires appear to be the work of arsonists and include materials such as tires, gasoline and trash. The exposures experienced by firefighters in this study might therefore differ from those in newer residential or industrial areas. Hence, more widespread sampling is necessary to establish the general applicability of these results.

The data on CO and HCN concentrations collected by each of the companies were compared and found not to differ significantly at the 99% level of confidence.

On the basis of results to date, plans are to revise the sampling program to include monitoring of organic vapors, particularly aldehydes and acids.

acknowledgements

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Chloride and Fluoride with Chloranilic Acid. *Anal. Chem.* 30:202 (1958).

18. Lambert, J. L., J. Ramasamy and J. V. Mustelis. Stable Reagents for the Colorimetric Determination of Cyanide by Modified Konig Reactions. *Anal. Chem.* 47:916 (1975).

19. Hounam, R. F. An Application of the Lognormal Distribution to Some Air Sampling Results and

references

1. Burgess, W. A., R. Sidor, J. J. Lynch, P. Buchanan and E. V. Clougherty. Minimum Protection Factors for Respiratory Protective Devices. *Am. Ind. Hyg. Assoc. J.* 38:18 (1977).
2. Anon. Answers to Burning Questions. *Moscow J.* 2:3 (1973).
3. Peters, J. M., G. P. Theriault, L. J. Fine and D. H. Wegman. Chronic Effects of Fire Fighting on Pulmonary Function. *N. Engl. J. Med.* 291:1320 (1974).
4. Sammons, J. H. and R. L. Coleman. Firefighters Occupational Exposure to Carbon Monoxide. *J. Occup. Med.* 16:543 (1974).
5. Boettner, E. A. and B. Weiss. Combustion Products from the Incineration of Plastics. EPA-670/273049.
6. Dufour, R. E. Survey of Available Information on the Toxicity of the Combustion and Thermal Decomposition Products of Certain Building Materials Under Fire Conditions. *Underwriters Laboratories Bulletin of Research* No. 53, July 1963.
7. Tewarson, A. Some Observations on Experimental Fires in Enclosures. Factory Mutual Research Corp. Serv. No. 18305, July 1971.
8. Vodvarka, F. J. Urban Burns - Full Scale Field Studies. Engineering Mechanics Division, BT Research Institute. Project J6171, Jan. 1970.
9. Gordon, G. S. and R. L. Rogers. Project Monoxide. International Association of Fire Fighters. Washington, DC 20006 (1969).
10. Environmental Protection Agency. Air Quality Criteria for Nitrogen Oxides. Air Pollution Control Office. Washington, DC (1971).
11. Gross, D., J. J. Loftus, T. G. Lee and W. E. Gray. Smoke and Gases Produced by Burning Aircraft Interior Materials. Building Science Series 18. National Bureau of Standards, Washington, DC (1969).
12. Levaggi, D. A., W. Siu and M. Feldstein. A New Method for Measuring Average 24-Hour Nitrogen Dioxide Concentrations in the Atmosphere. *J. Air Poll. Contr. Assoc.* 23:30 (1973).
13. Blacker, J. H. Triethanolamine for Collecting Nitrogen Dioxide in the TLV Range. *Am. Ind. Hyg. Assoc. J.* 34:390 (1973).
14. Zall, D. M., D. Fisher and M. Q. Garner. Photometric Determination of Chlorides in Water. *Anal. Chem.* 28:1665 (1956).
15. Humphrey, R. E., R. R. Clark, L. Houston and D. J. Kippenburger. Measurement of Chloride Ion by Ultraviolet Absorption of Mercury Complexes. *Anal. Chem.* 44:1299 (1972).
16. Humphrey, R. E. and W. L. Hinze. Determination of Chloride by Spectrophotometric Measurement of Mercuric Chloride with Phenolphthalein Complexone or Xylenol Orange. *Anal. Chem.* 45:1747 (1973).
17. Bertolacini, R. J. and J. E. Barney. Ultraviolet Spectrophotometric Determination of Sulfate. Recommendations on the Interpretation of Air Sampling Data. Atomic Energy Research Establishment Report AERE/M 1469. Her Majesty's Stationary Office, London, England, 1965.
20. American Conference of Governmental Industrial Hygienists. Threshold Limit Values for Chemical Substances and Physical Agents in the Workroom Environment with Intended Changes for 1975.

CYANIDE, TOTAL

Method 335.2 (Titrimetric; Spectrophotometric)

STORET NO. 00720

1. Scope and Application
 - 1.1 This method is applicable to the determination of cyanide in drinking, surface and saline waters, domestic and industrial wastes.
 - 1.2 The titration procedure using silver nitrate with p-dimethylamino-benzal-rhodanine indicator is used for measuring concentrations of cyanide exceeding 1 mg/l (0.25 mg/250 ml of absorbing liquid).
 - 1.3 The colorimetric procedure is used for concentrations below 1 mg/l of cyanide and is sensitive to about 0.02 mg/l.
2. Summary of Method
 - 2.1 The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by volumetric titration or colorimetrically.
 - 2.2 In the colorimetric measurement the cyanide is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at a pH less than 8 without hydrolyzing to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-pyrazolone or pyridine-barbituric acid reagent. The absorbance is read at 620 nm when using pyridine-pyrazolone or 578 nm for pyridine-barbituric acid. To obtain colors of comparable intensity, it is essential to have the same salt content in both the sample and the standards.
 - 2.3 The titrimetric measurement uses a standard solution of silver nitrate to titrate cyanide in the presence of a silver sensitive indicator.
3. Definitions
 - 3.1 Cyanide is defined as cyanide ion and complex cyanides converted to hydrocyanic acid (HCN) by reaction in a reflux system of a mineral acid in the presence of magnesium ion.
4. Sample Handling and Preservation
 - 4.1 The sample should be collected in plastic or glass bottles of 1 liter or larger size. All bottles must be thoroughly cleansed and thoroughly rinsed to remove soluble material from containers.
 - 4.2 Oxidizing agents such as chlorine decompose most of the cyanides. Test a drop of the sample with potassium iodide-starch test paper (KI-starch paper); a blue color indicates the need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.06 g of ascorbic acid for each liter of sample volume.

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- 4.3 Samples must be preserved with 2 ml of 10 N sodium hydroxide per liter of sample ($\text{pH} \geq 12$) at the time of collection.
- 4.4 Samples should be analyzed as rapidly as possible after collection. If storage is required, the samples should be stored in a refrigerator or in an ice chest filled with water and ice to maintain temperature at 4°C .
5. Interferences
 - 5.1 Interferences are eliminated or reduced by using the distillation procedure described in Procedure 8.1, 8.2 and 8.3.
 - 5.2 Sulfides adversely affect the colorimetric and titration procedures. Samples that contain hydrogen sulfide, metal sulfides or other compounds that may produce hydrogen sulfide during the distillation should be distilled by the optional procedure described in Procedure 8.2. The apparatus for this procedure is shown in Figure 3.
 - 5.3 Fatty acids will distill and form soaps under the alkaline titration conditions, making the end point almost impossible to detect.
 - 5.3.1 Acidify the sample with acetic acid (1 + 9) to pH 6.0 to 7.0.

Caution: This operation must be performed in the hood and the sample left there until it can be made alkaline again after the extraction has been performed.
 - 5.3.2 Extract with iso-octane, hexane, or chloroform (preference in order named) with a solvent volume equal to 20% of the sample volume. One extraction is usually adequate to reduce the fatty acids below the interference level. Avoid multiple extractions or a long contact time at low pH in order to keep the loss of HCN at a minimum. When the extraction is completed, immediately raise the pH of the sample to above 12 with NaOH solution.
 - 5.4 High results may be obtained for samples that contain nitrate and/or nitrite. During the distillation nitrate and nitrite will form nitrous acid which will react with some organic compounds to form oximes. These compounds formed will decompose under test conditions to generate HCN . The interference of nitrate and nitrite is eliminated by pretreatment with sulfamic acid.
6. Apparatus
 - 6.1 Reflux distillation apparatus such as shown in Figure 1 or Figure 2. The boiling flask should be of 1 liter size with inlet tube and provision for condenser. The gas absorber may be a Fisher-Milligan scrubber.
 - 6.2 Microburet, 5.0 ml (for titration).
 - 6.3 Spectrophotometer suitable for measurements at 578 nm or 620 nm with a 1.0 cm cell or larger.
 - 6.4 Reflux distillation apparatus for sulfide removal as shown in Figure 3. The boiling flask same as 6.1. The sulfide scrubber may be a Wheaton Bubber #709682 with 29/42 joints, size 100 ml. The air inlet tube should not be fritted. The cyanide absorption vessel should be the same as the sulfide scrubber. The air inlet tube should be fritted.
 - 6.5 Flow meter, such as Lab Crest with stainless steel float (Fisher 11-164-59).
7. Reagents
 - 7.1 Sodium hydroxide solution, 1.25N: Dissolve 50 g of NaOH in distilled water, and dilute to 1 liter with distilled water.

- 7.2 Lead acetate: Dissolve 30 g of $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2) \cdot 3\text{H}_2\text{O}$ in 950 ml of distilled water. Adjust the pH to 4.5 with acetic acid. Dilute to 1 liter.
- 7.3 Sulfuric acid; 18N: Slowly add 500 ml of concentrated H_2SO_4 to 500 ml of distilled water.
- 7.6 Sodium dihydrogenphosphate, 1 M: Dissolve 138 g of $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$ in 1 liter of distilled water. Refrigerate this solution.
- 7.7 Stock cyanide solution: Dissolve 2.51 g of KCN and 2 g KOH in 900 ml of distilled water. Standardize with 0.0192 N AgNO_3 . Dilute to appropriate concentration so that 1 ml = 1 mg CN.
- 7.8 Standard cyanide solution, intermediate: Dilute 100.0 ml of stock (1 ml = 1 mg CN) to 1000 ml with distilled water (1 ml = 100.0 μg).
- 7.9 Working standard cyanide solution: Prepare fresh daily by diluting 100.0 ml of intermediate cyanide solution to 1000 ml with distilled water and store in a glass stoppered bottle. 1 ml = 10.0 μg CN.
- 7.10 Standard silver nitrate solution, 0.0192 N: Prepare by crushing approximately 5 g AgNO_3 crystals and drying to constant weight at 40°C. Weigh out 3.2647 g of dried AgNO_3 , dissolve in distilled water, and dilute to 1000 ml (1 ml = 1 mg CN).
- 7.11 Rhodanine indicator: Dissolve 20 mg of p-dimethyl-amino-benzalrhodanine in 100 ml of acetone.
- 7.12 Chloramine T solution: Dissolve 1.0 g of white, water soluble Chloramine T in 100 ml of distilled water and refrigerate until ready to use. Prepare fresh daily.
- 7.13 Color Reagent — One of the following may be used:
 - 7.13.1 Pyridine-Barbituric Acid Reagent: Place 15 g of barbituric acid in a 250 ml volumetric flask and add just enough distilled water to wash the sides of the flask and wet the barbituric acid. Add 75 ml of pyridine and mix. Add 15 ml of conc. HCl, mix, and cool to room temperature. Dilute to 250 ml with distilled water and mix. This reagent is stable for approximately six months if stored in a cool, dark place.
 - 7.13.2 Pyridine-pyrazolone solution:
 - 7.13.2.1 3-Methyl-1-phenyl-2-pyrazolin-5-one reagent, saturated solution: Add 0.25 g of 3-methyl-1-phenyl-2-pyrazolin-5-one to 50 ml of distilled water, heat to 60°C with stirring. Cool to room temperature.
 - 7.13.2.2 3,3'-Dimethyl-1, 1'-diphenyl-[4,4'-bi-2 pyrazoline]-5,5'-dione (bispyrazolone): Dissolve 0.01 g of bispyrazolone in 10 ml of pyridine.
 - 7.13.2.3 Pour solution (7.13.2.1) through non-acid-washed filter paper. Collect the filtrate. Through the same filter paper pour solution (7.13.2.2) collecting the filtrate in the same container as filtrate from (7.13.2.1). Mix until the filtrates are homogeneous. The mixed reagent develops a pink color but this does not affect the color production with cyanide if used within 24 hours of preparation.
- 7.14 Magnesium chloride solution: Weigh 510 g of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ into a 1000 ml flask, dissolve and dilute to 1 liter with distilled water.
- 7.15 Sulfamic acid.

8. Procedure

8.1 For samples without sulfide.

8.1.1 Place 500 ml of sample, or an aliquot diluted to 500 ml in the 1 liter boiling flask. Pipet 50 ml of sodium hydroxide (7.1) into the absorbing tube. If the apparatus in Figure 1 is used, add distilled water until the spiral is covered. Connect the boiling flask, condenser, absorber and trap in the train. (Figure 1 or 2)

8.1.2 Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately two bubbles of air per second enters the boiling flask through the air inlet tube. Proceed to 8.4.

8.2 For samples that contain sulfide.

8.2.1 Place 500 ml of sample, or an aliquot diluted to 500 ml in the 1 liter boiling flask. Pipet 50 ml of sodium hydroxide (7.1) to the absorbing tube. Add 25 ml of lead acetate (7.2) to the sulfide scrubber. Connect the boiling flask, condenser, scrubber and absorber in the train. (Figure 3) The flow meter is connected to the outlet tube of the cyanide absorber.

8.2.2 Start a stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately 1.5 liters per minute enters the boiling flask through the air inlet tube. The bubble rate may not remain constant while heat is being applied to the flask. It may be necessary to readjust the air rate occasionally. Proceed to 8.4.

8.3 If samples contain NO_3 and/or NO_2 add 2 g of sulfamic acid solution (7.15) after the air rate is set through the air inlet tube. Mix for 3 minutes prior to addition of H_2SO_4 .

8.4 Slowly add 50 ml 18N sulfuric acid (7.5) through the air inlet tube. Rinse the tube with distilled water and allow the airflow to mix the flask contents for 3 min. Pour 20 ml of magnesium chloride (7.14) into the air inlet and wash down with a stream of water.

8.5 Heat the solution to boiling. Reflux for one hour. Turn off heat and continue the airflow for at least 15 minutes. After cooling the boiling flask, disconnect absorber and close off the vacuum source.

8.6 Drain the solution from the absorber into a 250 ml volumetric flask. Wash the absorber with distilled water and add the washings to the flask. Dilute to the mark with distilled water.

8.7 Withdraw 50 ml or less of the solution from the flask and transfer to a 100 ml volumetric flask. If less than 50 ml is taken, dilute to 50 ml with 0.25N sodium hydroxide solution (7.4). Add 15.0 ml of sodium phosphate solution (7.6) and mix.

8.7.1 Pyridine-barbituric acid method: Add 2 ml of chloramine T (7.12) and mix. See Note 1. After 1 to 2 minutes, add 5 ml of pyridine-barbituric acid solution (7.13.1) and mix. Dilute to mark with distilled water and mix again. Allow 8 minutes for color development then read absorbance at 578 nm in a 1 cm cell within 15 minutes.

8.7.2 Pyridine-pyrazolone method: Add 0.5 ml of chloramine T (7.12) and mix. See Note 1 and 2. After 1 to 2 minutes add 5 ml of pyridine-pyrazolone solution

(7.13.1) and mix. Dilute to mark with distilled water and mix again. After 40 minutes read absorbance at 620 nm in a 1 cm cell.

NOTE 1: Some distillates may contain compounds that have a chlorine demand. One minute after the addition of chloramine T, test for residual chlorine with KI-starch paper. If the test is negative, add an additional 0.5 ml of chlorine T. After one minute, recheck the sample.

NOTE 2: More than 0.5 ml of chloramine T will prevent the color from developing with pyridine-pyrazolone.

8.8 Standard curve for samples without sulfide.

8.8.1 Prepare a series of standards by pipeting suitable volumes of standard solution (7.9) into 250 ml volumetric flasks. To each standard add 50 ml of 1.25 N sodium hydroxide and dilute to 250 ml with distilled water. Prepare as follows:

ML of Working Standard Solution (1 ml = 10 μ g CN)	Conc. μ g CN per 250 ml
0	BLANK
1.0	10
2.0	20
5.0	50
10.0	100
15.0	150
20.0	200

8.8.2 It is not imperative that all standards be distilled in the same manner as the samples. It is recommended that at least two standards (a high and low) be distilled and compared to similar values on the curve to insure that the distillation technique is reliable. If distilled standards do not agree within $\pm 10\%$ of the undistilled standards the analyst should find the cause of the apparent error before proceeding.

8.8.3 Prepare a standard curve by plotting absorbance of standard vs. cyanide concentrations.

8.8.4 To check the efficiency of the sample distillation, add an increment of cyanide from either the intermediate standard (7.8) or the working standard (7.9) to 500 ml of sample to insure a level of 20 μ g/l. Proceed with the analysis as in Procedure (8.1.1).

8.9 Standard curve for samples with sulfide.

8.9.1 It is imperative that all standards be distilled in the same manner as the samples. Standards distilled by this method will give a linear curve, but as the concentration increases, the recovery decreases. It is recommended that at least 3 standards be distilled.

8.9.2 Prepare a standard curve by plotting absorbance of standard vs. cyanide concentrations.

8.10 Titrimetric method.

8.10.1 If the sample contains more than 1 mg/l of CN, transfer the distillate or a suitable aliquot diluted to 250 ml, to a 500 ml Erlenmeyer flask. Add 10-12 drops of the benzalrhodanine indicator.

8.10.2 Titrate with standard silver nitrate to the first change in color from yellow to brownish-pink. Titrate a distilled water blank using the same amount of sodium hydroxide and indicator as in the sample.

8.10.3 The analyst should familiarize himself with the end point of the titration and the amount of indicator to be used before actually titrating the samples.

9. Calculation

9.1 If the colorimetric procedure is used, calculate the cyanide, in $\mu\text{g/l}$, in the original sample as follows:

$$\text{CN, } \mu\text{g/l} = \frac{A \times 1,000}{B} \times \frac{50}{C}$$

where:

A = μg CN read from standard curve

B = ml of original sample for distillation

C = ml taken for colorimetric analysis

9.2 Using the titrimetric procedure, calculate concentration of CN as follows:

$$\text{CN, mg l} = \frac{(A - B)1,000}{\text{ml orig. sample}} \times \frac{250}{\text{ml of aliquot titrated}}$$

where:

A = volume of AgNO_3 for titration of sample.

B = volume of AgNO_3 for titration of blank.

10. Precision and Accuracy

10.1 In a single laboratory (EMSL), using mixed industrial and domestic waste samples at concentrations of 0.06, 0.13, 0.28 and 0.62 mg/l CN, the standard deviations were ± 0.005 , ± 0.007 , ± 0.031 and ± 0.094 , respectively.

10.2 In a single laboratory (EMSL), using mixed industrial and domestic waste samples at concentrations of 0.28 and 0.62 mg/l CN, recoveries were 85% and 102%, respectively.

Bibliography

1. Bark, L. S., and Higson, H. G. "Investigation of Reagents for the Colorimetric Determination of Small Amounts of Cyanide", *Talanta*, 2:471-479 (1964).
2. Elly, C. T. "Recovery of Cyanides by Modified Serfass Distillation". *Journal Water Pollution Control Federation* 40:848-856 (1968).
3. *Annual Book of ASTM Standards*, Part 31, "Water", Standard D2036-75, Method A, p 503 (1976).
4. *Standard Methods for the Examination of Water and Wastewater*, 14th Edition, p 367 and 370, Method 413B and D (1975).
5. Egekeze, J. O., and Oehne, F. W., "Direct Potentiometric Determination of Cyanide in Biological Materials," *J. Analytical Toxicology*, Vol. 3, p. 119, May/June 1979.
6. Casey, J. P., Bright, J. W., and Helms, B. D., "Nitrosation Interference in Distillation Tests for Cyanide." Gulf Coast Waste Disposal Authority, Houston, Texas.

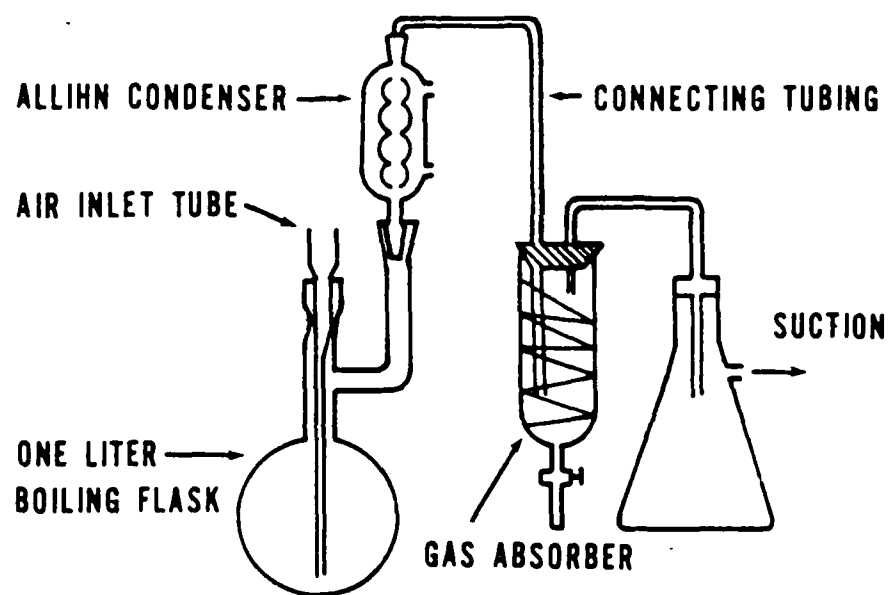


FIGURE 1
CYANIDE DISTILLATION APPARATUS

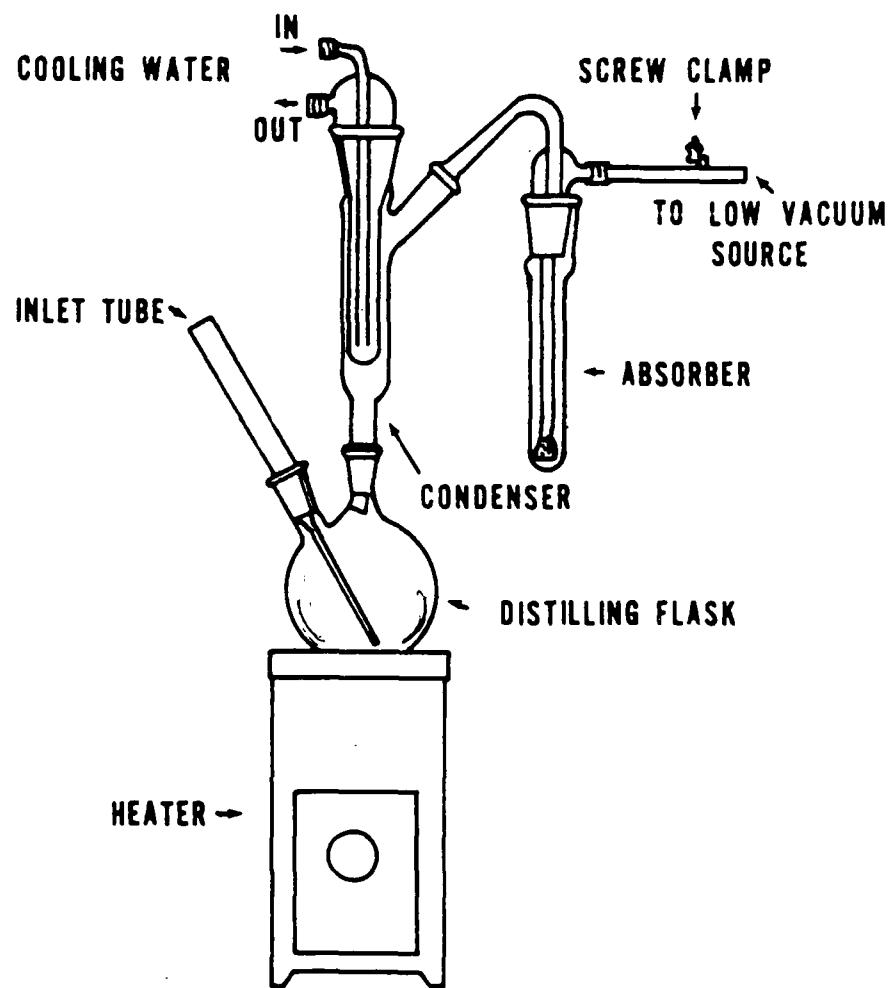


FIGURE 2
CYANIDE DISTILLATION APPARATUS

NITROGEN DIOXIDE - GENERAL AREA

NITROGEN DIOXIDE AND NITRIC OXIDE IN AIR

Measurements Support Branch

Analytical Method

Analyte: Nitrogen Dioxide
and Nitric Oxide

Method No.: P&CAM 231

Matrix: Air

Range: 0.8 to 30 ppm of NO₂
or NO in a 1-liter sample

Procedure: Solid sorbent
collection; triethanol-
amine extraction; spec-
trophotometry

Precision(CV_T): NO₂, 0.07 at
0.5 to 5 ppm; NO, 0.06 at
12.5 to 50 ppm

Classification: D (Operational)

Date Issued: 6/30/76

Date Revised:

1. Principle of the Method

Nitrogen dioxide (NO₂) and nitric oxide (NO) are collected from air in a three-section sorbent tube. The NO₂ is absorbed in the first section, which contains triethanolamine (TEA) impregnated on molecular sieve. The NO is converted to NO₂ by a proprietary oxidizer in the second section. The NO₂ thus formed from the NO is absorbed in the third section by another bed of TEA-impregnated molecular sieve. The first and third sections are desorbed with solutions of TEA in water and the nitrite in these solutions is determined spectrophotometrically by the Griess-Saltzman reaction. (Reference 11.1). The nitrite found in the first section is reported as NO₂ and the nitrite in the third section is reported as NO.

2. Range and Sensitivity

- 2.1 The linear range of the standard curve is from 0.5 to 18 µg of nitrite in 10 ml of desorbing solution, which corresponds in this method to a range of 0.8 to 30 ppm of NO₂ or NO in a 1-liter sample of air.
- 2.2 The sensitivity is 0.4 µg/10 ml for an absorbance of 0.04.
- 2.3 The upper limit of the range can be extended by taking smaller aliquots for analysis, or by diluting intensely colored solutions with water.

3. Interferences

- 3.1 Inorganic nitrites cause positive interference.

3.2 Nitric acid and nitrates do not interfere.

3.3 Ammonia does not interfere.

4. Precision and Accuracy

4.1 The average recovery for 22 samples in the range 0.5 to 5 ppm of NO_2 was greater than 96% and the coefficient of variation was 0.07.

4.2 For 18 samples the average recovery of NO varied with the amount of NO collected. The recovery was 100% at 12.5 ppm. At 25 ppm only 84% recovery was achieved, and at 50 ppm only 67%. However, the coefficient of variation over the range was only 0.06. The recovery may vary depending upon the sample flow rate and the properties of the particular lot of oxidizer used. Each laboratory should determine the efficiency of the sampling tubes employed.

4.3 The accuracy of the overall sampling and analytical method has not been determined.

5. Advantages and Disadvantages of the Method

5.1 Both nitrogen dioxide and nitric oxide are collected simultaneously.

5.2 This method is simple and convenient for field sampling.

5.3 Samples can be stored at ambient temperature for at least 10 days without any effect on the results.

5.4 At 50 ppm of NO the collection efficiency is poor (about 67%) because the oxidizer is consumed.

5.5 If high humidity or water mist is present, the breakthrough volume can be severely reduced. If water condenses in the tube, NO_2 and NO may not be collected quantitatively.

6. Apparatus

6.1 Sampling Equipment

6.1.1 Solid sorbent tubes are made in the following manner. Using a gas-oxygen torch, heat a section of 5-mm i.d., 7-mm o.d. Pyrex glass tubing and pull it

apart to form a tube approximately 15 cm long with a taper 2 cm long. Seal the tapered end of the tube in the flame. Allow it to cool, then insert a small plug of glass wool through the open end of the tube; push the glass wool through the open end of the tube with a thin wooden stick and pack gently. Weigh 400 mg of TEA sorbent and pour the material into the tube. (See Section 7.2) Gently tap the tube on the table top several times to ensure uniform packing. Insert another small plug of glass wool to keep the TEA sorbent in place. For the next section, pour 800 mg of oxidizer into the tube. (See Section 7.1.) Again tap the tube and insert a plug of glass wool; pack lightly. Insert another plug of glass wool, maintaining an air gap of 12 mm between these two plugs. Weigh 400 mg of TEA sorbent and pour the material into the tube. Carefully tap the tube and gently pack another glass wool plug without closing the 12-mm air gap. Seal the open end of the tube with the torch. See the figure on page 231-9.

- 6.1.2 A personal sampling pump that can provide a flow rate of 50 mL/min within 5% accuracy is required. The pump should be calibrated with a representative sorbent tube in the sampling line. A dry or wet test meter or glass rotameter that will determine the flow rate to within 5% may be used for the calibration.

6.2 Spectrophotometer capable of measurements at 540 nm.

6.3 Matched glass cells or cuvettes, 1-cm path length.

6.4 Assorted laboratory glassware: pipettes, glass-stoppered graduated cylinders, and volumetric flasks of appropriate sizes.

7. Reagents

7.1 **Oxidizer.** Proprietary material Number 1900277 from the Drägerwerk Company of West Germany, supplied through its U.S. distributor, National Mine Safety Company, or the equivalent.

7.2 **TEA Sorbent.** Place 25 g of triethanolamine in a 250-mL beaker; add 4 g of glycerol, 50 mL of acetone and sufficient distilled water to bring the volume up to 100 mL. To the mixture add about 50 mL of Type 13X, 30/40-mesh Molecular Sieve. Stir and let stand in a covered beaker for about 30 min. Decant the excess liquid, and transfer the molecular sieve to a porcelain pan. Place the pan under a heating lamp until most of the moisture has evaporated. Complete the drying in an oven at 110°C for 1 hr. The sorbent should be free flowing. Store it in a closed glass container.

- 7.3 **Desorbing Solution.** Dissolve 15.0 g of triethanolamine in approximately 500 mL of distilled water, add 0.5 mL of *n*-butanol, and dilute to 1 liter.
- 7.4 **Hydrogen Peroxide, 0.02%(v/v).** Dilute 0.2 mL of 30% hydrogen peroxide to 250 mL with distilled water.
- 7.5 **Sulfanilamide Solution.** Dissolve 10 g of sulfanilamide in 400 mL of distilled water. Add 25 mL of concentrated phosphoric acid, mix well, and dilute to 500 mL.
- 7.6 **NEDA Solution.** Dissolve 0.5 gm of N-(1-naphthyl)ethylenediamine dihydrochloride in 500 mL of distilled water.
- 7.7 **Nitrite Stock Standard Solution (100 µg/mL).** Dissolve 0.1500 g of reagent grade sodium nitrite in distilled water and dilute to 1 liter.

8. Procedure

- 8.1 **Cleaning of Equipment.** Wash all glassware with detergent solution, soak in nitric acid, rinse in tap water and distilled water, and then rinse thoroughly with double distilled water.
- 8.2 **Collection and Shipping of Samples**
- 8.2.1 Before sampling, break open the ends of the sorbent tube to provide an opening that is approximately one-half the internal diameter of the tube.
- 8.2.2 The air must flow through the 12-mm air space before it flows through the oxidizer. Therefore attach the end of the tube without the air gap between the oxidizer section and TEA sorbent section to the pump with a length of small diameter Tygon® tubing.
- 8.2.3 Mount the tube in a vertical position to avoid channeling.
- 8.2.4 The air being sampled should not pass through any hose or tubing before it enters the sorbent tube.
- 8.2.5 Turn on the pump to begin sample collection. Sample at a flow rate of 50 mL/min or less to obtain a maximum sample volume of 1 liter. Measure the flow rate and time, or volume, as accurately as possible. If a low flow rate pump is used, set the rate to an approximate value and record the initial and final stroke counter readings. Obtain the sample volume by multiplying the number of strokes by the stroke volume.
- 8.2.6 Measure and record the temperature and pressure of the atmosphere being sampled.

- 8.2.7 Cap the sorbent tubes with 7-mm i.d. plastic caps immediately after sampling. (Masking tape can be substituted for the plastic caps.)
- 8.2.8 With each batch of samples, submit one blank sorbent tube. This tube is handled in the same manner as the other tubes (break, seal, and transport) except that no air is drawn through it. When more than ten samples are submitted, include an additional blank for every ten samples.
- 8.2.9 Pack the capped sorbent tubes tightly and pad them to minimize breakage during shipping.

8.3 Analysis of Samples

- 8.3.1 With tweezers remove and discard the glass wool plugs from an exposed sorbent tube and transfer each TEA sorbent bed to separate, 25-ml glass-stoppered graduated cylinders. Label the graduated cylinder as to the location of the TEA sorbent with respect to the oxidizer section.
- 8.3.2 To each graduated cylinder add enough of the desorbing solution to make the volume up to 20 ml, and shake the mixture vigorously for about 30 sec.
- 8.3.3 Allow a few minutes for the solids to settle, and then transfer 10 ml to another 25-ml glass-stoppered graduated cylinder.
- 8.3.4 Develop the color of the solution for 10 min in the same manner as described for the preparation of the standard curve (Sections 9.4 to 9.6). From the standard curve determine the amount of nitrite in the 10-ml aliquot.

8.4 Determination of Collection and Desorption Efficiencies

- 8.4.1 **Importance of Determination.** The collection and desorption efficiencies of a given compound can vary from one laboratory to another and also from one batch of sorbent tubes to another. Thus, it is necessary to determine at least once the percentages of sample collected and then removed in the desorption process. Results indicate that the recovery of NO varies with the amount of NO collected, particularly at higher concentrations (for example, at 50 ppm).

8.4.2 **Procedure for Determining Collection and Desorption Efficiencies.** Sorbent tubes from the same batch as that used in obtaining samples are used in this determination. Known volumes of NO_2 and NO are injected into a bag containing a known volume of air. The bag is made of Tedlar (or another material that will not absorb NO_2 or NO) and should have a gas sampling valve and a septum injection port. The concentrations of NO_2 and NO in the bag may be calculated at room temperature and pressure. A measured volume is then sampled through a sorbent tube with a calibrated sampling pump. At least five tubes are prepared in this manner. These tubes are desorbed and analyzed in the same manner as the samples (Section 8.3).

8.4.3 **Calculation of Desorption Efficiency.** The desorption efficiency (D.E.) is the average concentration (corrected for the blank) of NO_2 or NO found by analysis of the sorbent tubes divided by the concentration of NO_2 or NO in the bag.

9. Calibration and Standards

9.1 Dilute 2 mL of the nitrite stock standard (100 $\mu\text{g}/\text{mL}$) to 100 mL with the desorbing solution to prepare a solution with a nitrite concentration of 2 $\mu\text{g}/\text{mL}$.

9.2 To a series of 25-mL glass-stoppered graduated cylinders add 1, 3, 5, 7, and 9 mL of the dilute standard solution.

9.3 Add enough of the absorbing solution to bring the volume in each cylinder up to 10 mL to prepare working standards with nitrite concentrations of 2, 6, 10, 14, and 18 $\mu\text{g}/10\text{ mL}$.

9.4 To each graduated cylinder, add 1 mL of the 0.02% hydrogen peroxide solution, 10 mL of the sulfanilamide solution, and 1.4 mL of the NEDA solution, with thorough mixing after the addition of each reagent.

9.5 Allow 10 min for complete color development.

9.6 Measure the absorbance of the solutions at 540 nm, using a reagent blank in the reference cell.

9.7 Prepare a standard curve by plotting absorbance *versus* weight of nitrite (in μg) in 10 mL of the desorbing solution.

10. Calculations

10.1 From the standard curve, read the weight of nitrite (in μg) in 10 ml of the desorbing solution corresponding to the absorbance of the sample solution. Multiply this weight by 2 to determine the total amount (in μg) of nitrite extracted with 20 ml of desorbing solution from the sorbent section being analyzed. The calibration procedure is based upon the empirical observation that 0.63 mole of sodium nitrite produces the same absorbance in the color-developed solution as 1 mole of NO_2 . (See Reference 11.2.) Divide the amount of nitrite desorbed from the sorbent material by 0.63 to determine the apparent amount of NO_2 collected in the sorbent section. These calculations are summarized in the following equation:

$$W = \frac{\mu\text{g NO}_2 \times 2}{0.63}$$

where: W = weight (in μg) of NO_2 found.

10.2 Correct the amount of NO_2 calculated in Section 10.1 for the amount of NO_2 , if any, found on the corresponding sorbent section of a blank tube to obtain the amount of NO_2 in the sample, as follows:

$$W_s = W - W_b$$

where: W_s = corrected weight (in μg) of NO_2 in sample.

W_b = weight (in μg) of NO_2 in the corresponding section of a blank tube.

10.3 The concentration of NO_2 in parts per million (ppm) by volume in the air sample is calculated as follows:

$$\text{ppm} = \frac{W_s}{V} \times \frac{24.45}{\text{M.W.}} \times \frac{760}{P} \times \frac{T+273}{298}$$

where: V = volume (liters) of air sampled.

M.W. = molecular weight.

24.45 = molar volume (liter/mole) at 25°C and 760 mm.Hg.

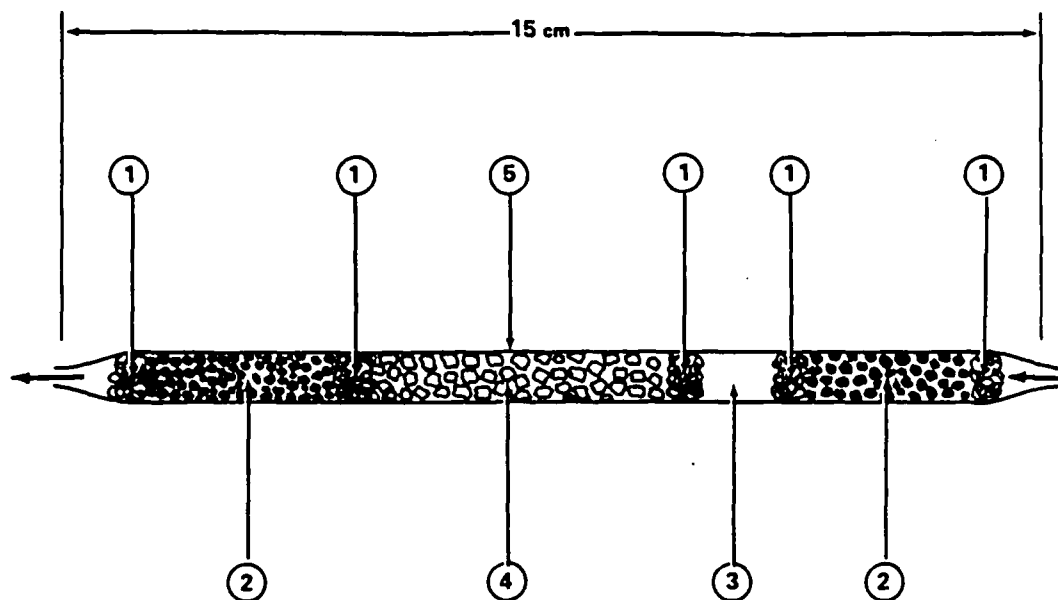
P = pressure (mmHg) of air sampled.

T = temperature (°C) of air sampled.

10.4 The ppm of NO_2 found in the third section (downstream from the oxidizer) is reported as ppm of NO .

11. References

- 11.1 Saltzman, B.E. "Colorimetric Microdetermination of Nitrogen Dioxide in the Atmosphere," *Anal. Chem.*, 26, 1949 (1954).
- 11.2 Blacker, J. H., "Triethanolamine for Collecting Nitrogen Dioxide in the TLV Range," *Am. Ind. Hyg. Assoc. J.*, 34, 390 (1973).
- 11.3 NIOSH Sampling Data Sheet No. 32.01, "NIOSH Manual of Sampling Data Sheets," Measurements Research Branch, Division of Physical Sciences and Engineering, National Institute for Occupational Safety and Health, December 22, 1975.
- 11.4 Willey, M.A., C. S. McCammon, Jr., and L. J. Doemeny, "A Solid Sorbent Personal Sampling Method for the Simultaneous Collection of Nitrogen Dioxide and Nitric Oxide in Air," presented at the American Industrial Hygiene Association Conference, Atlanta, Georgia, May 1976.



- 1. GLASS WOOL PLUGS
- 2. TEA SORBENT, 400 mg
- 3. AIR GAP, 12 mm
- 4. OXIDIZER, 800 mg
- 5. GLASS TUBE, 5 mm i.d.

SORBENT TUBE FOR NO_2 and NO

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APPENDIX B
ANALYTICAL DATA

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Carbon Monoxide.....	B-1
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Nitrogen Dioxide - Breathing Zone.....	B-19
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Sulfur Dioxide.....	B-27
Respirable Suspended Particulates.....	B-31
Total Suspended Particulates.....	B-33
Aldehydes.....	B-37

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CARBON MONOXIDE

SASOUT.CODATA

OBS	L	F	S	F	V	D	V	F	A	R	A	B	F	T	O	T	S	T	V	G	R	O	U	P
145	5620	FKA13BLC01	F	K	A	1	3	B	L	D	C01	1560	272	126.15	40	1	6	KM1	E					TANK
146	5622	FKA14ADC01	F	K	A	1	4	A	L	C01	140	118	102.90	10	1	2	3	KM1	E					TANK
147	5623	FKA14ALC01	F	K	A	1	4	A	L	C01	1940	118	102.90	10	1	3	4	KM1	E					TANK
148	5624	FKA14BCC01	F	K	A	1	4	B	C	C01	750	118	102.90	10	1	4	7	KM1	E					TANK
149	5626	FKA14BCC01	F	K	A	1	4	B	C	C01	1120	118	102.90	20	1	7	6	KM1	E					TANK
150	5627	FKA14BLC01	F	K	A	1	4	B	L	C01	2280	118	102.90	590	1	6	1	KM1	E					TANK
151	5631	FKA21ACC01	F	K	A	2	1	A	C	C01	2280	178	160.05	70	1	1	1	KM1	E					TANK
152	5632	FKA21ADC01	F	K	A	2	1	A	D	C01	1280	178	160.05	30	1	2	1	KM1	E					TANK
153	5633	FKA21ALC01	F	K	A	2	1	A	L	C01	1300	178	160.05	40	1	3	3	KM1	E					TANK
154	5634	FKA21BCC01	F	K	A	2	1	B	C	C01	1970	178	160.05	50	1	4	4	KM1	E					TANK
155	5635	FKA21BDC01	F	K	A	2	1	B	D	C01	1410	178	160.05	40	1	5	5	KM1	E					TANK
156	5636	FKA21BGC01	F	K	A	2	1	B	G	C01	1970	178	160.05	60	1	7	6	KM1	E					TANK
157	5637	FKA21BLC01	F	K	A	2	1	B	L	C01	1890	178	160.05	50	1	6	1	KM1	E					TANK
158	5639	FKA22ADC01	F	K	A	2	2	A	D	C01	1220	124	137.10	10	1	2	3	KM1	E					TANK
159	5640	FKA22ALC01	F	K	A	2	2	A	L	C01	1280	124	137.10	10	1	3	4	KM1	E					TANK
160	5641	FKA22BCC01	F	K	A	2	2	B	C	C01	2160	124	137.10	10	1	4	7	KM1	E					TANK
161	5643	FKA22BGC01	F	K	A	2	2	B	G	C01	960	124	137.10	0	1	7	6	KM1	E					TANK
162	5644	FKA22BLC01	F	K	A	2	2	B	L	C01	1920	124	137.10	20	1	6	1	KM1	E					TANK
163	5645	FKA23ACC01	F	K	A	2	3	A	C	C01	2280	125	142.80	30	1	1	1	KM1	E					TANK
164	5646	FKA23ADC01	F	K	A	2	3	A	D	C01	580	125	142.80	10	1	2	3	KM1	E					TANK
165	5647	FKA23ALC01	F	K	A	2	3	A	L	C01	1740	125	142.80	20	1	4	4	KM1	E					TANK
166	5648	FKA23BCC01	F	K	A	2	3	B	C	C01	2200	125	142.80	30	1	3	5	KM1	E					TANK
167	5649	FKA23BDC01	F	K	A	2	3	B	D	C01	600	125	142.80	10	1	4	7	KM1	E					TANK
168	5650	FKA23BGC01	F	K	A	2	3	B	G	C01	2310	125	142.80	30	1	7	6	KM1	E					TANK
169	5651	FKA23BLC01	F	K	A	2	3	B	L	C01	2300	125	142.80	30	1	6	1	KM1	E					TANK
170	5653	FKA24ADC01	F	K	A	2	4	A	D	C01	200	30	171.00	0	1	2	3	KM1	E					TANK
171	5654	FKA24ALC01	F	K	A	2	4	A	L	C01	640	30	171.00	20	1	3	4	KM1	E					TANK
172	5655	FKA24BCC01	F	K	A	2	4	B	C	C01	230	30	171.00	0	1	4	5	KM1	E					TANK
173	5656	FKA24BDC01	F	K	A	2	4	B	D	C01	230	30	171.00	0	1	5	7	KM1	E					TANK
174	5657	FKA24BGC01	F	K	A	2	4	B	G	C01	990	30	171.00	10	1	7	6	KM1	E					TANK
175	5658	FKA24BLC01	F	K	A	2	4	B	L	C01	1320	30	171.00	20	1	6	1	KM1	E					TANK
176	5700	FKT11ACC01	F	K	T	1	1	A	C	C01	320	552	155.48	20	1	1	1	KTAN	F					TANK
177	5701	FKT11ADC01	F	K	T	1	1	A	D	C01	450	552	155.48	20	1	2	3	KTAN	F					TANK
178	5702	FKT11ALC01	F	K	T	1	1	A	L	C01	560	552	155.48	20	1	3	5	KTAN	F					TANK
179	5704	FKT11BDC01	F	K	T	1	1	B	D	C01	390	552	155.48	40	1	5	7	KTAN	F					TANK
180	5705	FKT11BGC01	F	K	T	1	1	B	G	C01	1910	552	155.48	40	1	6	1	KTAN	F					TANK
181	5706	FKT11BLC01	F	K	T	1	1	B	L	C01	2290	552	155.48	90	1	6	1	KTAN	F					TANK
182	5707	FKT12ACC01	F	K	T	1	2	A	C	C01	1030	327	154.80	70	1	1	2	KTAN	F					TANK
183	5708	FKT12ADC01	F	K	T	1	2	A	D	C01	580	327	154.80	40	1	2	3	KTAN	F					TANK
184	5709	FKT12ALC01	F	K	T	1	2	A	L	C01	2290	327	154.80	90	1	3	4	KTAN	F					TANK
185	5710	FKT12BCC01	F	K	T	1	2	B	C	C01	1310	327	154.80	60	1	4	5	KTAN	F					TANK
186	5711	FKT12BDC01	F	K	T	1	2	B	D	C01	650	327	154.80	50	1	5	7	KTAN	F					TANK
187	5712	FKT12BGC01	F	K	T	1	2	B	G	C01	1640	327	154.80	70	1	7	6	KTAN	F					TANK
188	5713	FKT12BLC01	F	K	T	1	2	B	L	C01	1980	327	154.80	100	1	6	1	KTAN	F					TANK
189	5714	FKT13ACC01	F	K	T	1	3	A	C	C01	40	331	177.60	0	1	1	2	KTAN	F					TANK
190	5715	FKT13ADC01	F	K	T	1	3	A	D	C01	250	331	177.60	20	1	3	4	KTAN	F					TANK
191	5716	FKT13ALC01	F	K	T	1	3	A	L	C01	1610	331	177.60	30	1	4	7	KTAN	F					TANK
192	5717	FKT13BCC01	F	K	T	1	3	B	C	C01	2280	331	177.60	10	1	4	7	KTAN	F					TANK

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CARBON DIOXIDE

O	B	S	L	A	B	N	O	F	L	D	C	O	D	E	S	C	A	L	R	A	N	A	B	O	F	I	R	E	G	
49	4704	FK112ACC02	F	K	1	2	A	C	C02	>	>	>	402	327	154.8	659	1	1	1	1	1	1	1	1	1	1	1	1	1	TANK
50	4705	FK112ADC02	F	K	1	2	A	D	C02	>	>	>	326	327	154.8	595	1	1	1	1	1	1	1	1	1	1	1	1	1	TANK
51	4707	FK112ALC02	F	K	1	2	A	L	C02	>	>	>	249	327	154.8	535	1	1	1	1	1	1	1	1	1	1	1	1	1	TANK
52	4200	FSM11ACC02	F	S	M	1	1	A	C02				973	7	84.0	677	1	1	1	1	1	1	1	1	1	1	1	1	1	NON
53	4201	FSM11ADC02	F	S	M	1	1	A	D	C02			599	7	84.0	551	1	1	1	1	1	1	1	1	1	1	1	1	1	NON
54	4202	FSM11ALC02	F	S	M	1	1	A	L	C02			925	7	84.0	666	1	1	1	1	1	1	1	1	1	1	1	1	1	NON
55	4203	FSM12ACC02	F	S	M	1	2	A	C02			1220	12	144.0	778	1	1	1	1	1	1	1	1	1	1	1	1	1	1	NON
56	4204	FSM12ADC02	F	S	M	1	2	A	D	C02			890	12	144.0	646	1	1	1	1	1	1	1	1	1	1	1	1	1	NON
57	4206	FSM12ALC02	F	S	M	1	2	A	L	C02			976	12	144.0	675	1	1	1	1	1	1	1	1	1	1	1	1	1	NON
58	4207	FSM15ACC02	F	S	M	1	5	A	C02			1240	13	156.0	776	1	1	1	1	1	1	1	1	1	1	1	1	1	1	NON
59	4208	FSM15ADC02	F	S	M	1	5	A	D	C02			695	13	156.0	580	1	1	1	1	1	1	1	1	1	1	1	1	1	NON
60	4209	FSM15ALC02	F	S	M	1	5	A	L	C02			807	13	156.0	620	1	1	1	1	1	1	1	1	1	1	1	1	1	NON
61	4210	FSM23ACC02	F	S	M	2	3	A	C02			672	10	120.0	694	1	1	1	1	1	1	1	1	1	1	1	1	1	1	NON
62	4211	FSM23ADC02	F	S	M	2	3	A	D	C02			416	10	120.0	556	1	1	1	1	1	1	1	1	1	1	1	1	1	NON
63	4213	FSM23ALC02	F	S	M	2	3	A	L	C02			323	10	120.0	507	1	1	1	1	1	1	1	1	1	1	1	1	1	NON
64	4214	FSM24ACC02	F	S	M	2	4	A	C02			247	10	120.0	422	1	1	1	1	1	1	1	1	1	1	1	1	1	1	NON
65	4215	FSM24ADC02	F	S	M	2	4	A	D	C02			424	10	120.0	575	1	1	1	1	1	1	1	1	1	1	1	1	1	NON
66	4217	FSM24ALC02	F	S	M	2	4	A	L	C02			198	10	120.0	399	1	1	1	1	1	1	1	1	1	1	1	1	1	NON
67	4100	PSM11ACC02	P	3	1	A	C02	>	>	>	903	12	144.0	678	1	1	1	1	1	1	1	1	1	1	1	1	1	NON		
68	4101	PSM11ADC02	P	3	1	A	D	C02	>	>	658	12	144.0	583	1	1	1	1	1	1	1	1	1	1	1	1	1	NON		
69	4102	PSM11ALC02	P	3	1	A	L	C02	>	>	683	12	144.0	589	1	1	1	1	1	1	1	1	1	1	1	1	1	NON		
70	4104	PSM12ACC02	P	3	2	A	C02			612	16	192.0	569	1	1	1	1	1	1	1	1	1	1	1	1	1	1	NON		
71	4112	PSM23ACC02	P	4	3	A	C02			529	17	204.0	662	1	1	1	1	1	1	1	1	1	1	1	1	1	1	NON		
72	4113	PSM23ADC02	P	4	3	A	D	C02			554	17	204.0	678	1	1	1	1	1	1	1	1	1	1	1	1	1	NON		
73	4114	PSM23ALC02	P	4	3	A	L	C02			554	17	204.0	678	1	1	1	1	1	1	1	1	1	1	1	1	1	NON		
74	4116	PSM24ACC02	P	4	4	A	C02			1110	16	192.0	1150	1	1	1	1	1	1	1	1	1	1	1	1	1	1	NON		
75	4117	PSM24ADC02	P	4	4	A	D	C02			751	16	192.0	880	1	1	1	1	1	1	1	1	1	1	1	1	1	NON		
76	4118	PSM24ALC02	P	4	4	A	L	C02			848	16	192.0	960	1	1	1	1	1	1	1	1	1	1	1	1	1	NON		

HYDROGEN SULFIDE

SASOUT.H2SDATA

[illegible]

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SASOUT.H2SDATA

OBS	L	F	S	F	V	D	Y	P	H	T	R	A	N	B	A	H2S	F	J	R	E	C	O	N	C	L	S	T	O	M	S	T	C	N	C	T	V	G
49	8522	FCT21BDH2S	F	C	T	2	1	B	2	2	1	B	2	2	1	485.0	162	69.90	243.0	1	5	CTAN	C	TANK													
50	8523	FCT21BLH2S	F	C	T	2	1	B	2	2	1	B	2	2	1	442.0	162	69.90	221.0	1	6	CTAN	C	TANK													
51	8524	FCT22ACH2S	F	C	T	2	2	A	2	2	2	A	2	2	2	6120.0	166	92.70	1880.0	1	1	CTAN	C	TANK													
52	8525	FCT22ADH2S	F	C	T	2	2	A	2	2	2	A	2	2	2	927.0	166	92.70	285.0	1	2	CTAN	C	TANK													
53	8527	FCT22ALH2S	F	C	T	2	2	A	2	2	2	A	2	2	2	1070.0	166	92.70	330.0	1	3	CTAN	C	TANK													
54	8528	FCT22BCH2S	F	C	T	2	2	B	2	2	2	B	2	2	2	520.0	166	92.70	160.0	1	4	CTAN	C	TANK													
55	8529	FCT22BDH2S	F	C	T	2	2	B	2	2	2	B	2	2	2	440.0	166	92.70	135.0	1	5	CTAN	C	TANK													
56	8543	FCT22BGH2S	F	C	T	2	2	B	2	2	2	B	2	2	2	676.0	166	92.70	208.0	1	7	CTAN	C	TANK													
57	8530	FCT22BLH2S	F	C	T	2	2	B	2	2	2	B	2	2	2	2900.0	166	92.70	892.0	1	6	CTAN	C	TANK													
58	8533	FCT25ACH2S	F	C	T	2	5	A	2	2	5	A	2	2	2	1830.0	160	58.50	141.0	1	1	CTAN	C	TANK													
59	8534	FCT25ADH2S	F	C	T	2	5	A	2	2	5	A	2	2	2	530.0	160	58.50	40.8	1	2	CTAN	C	TANK													
60	8536	FCT25ALH2S	F	C	T	2	5	A	2	2	5	A	2	2	2	480.0	160	58.50	36.9	1	3	CTAN	C	TANK													
61	8602	FKA13ACH2S	F	K	A	1	3	A	1	3	3	A	1	3	2	70.7	272	126.15	51.5	1	1	KM1	E	TANK													
62	8603	FKA13ADH2S	F	K	A	1	3	A	1	3	3	A	1	3	2	75.1	272	126.15	54.7	1	2	KM1	E	TANK													
63	8605	FKA13ALH2S	F	K	A	1	3	A	1	3	3	A	1	3	2	131.0	272	126.15	95.1	1	3	KM1	E	TANK													
64	8606	FKA13BCH2S	F	K	A	1	3	B	1	3	3	B	1	3	2	150.0	272	126.15	122.0	1	4	KM1	E	TANK													
65	8607	FKA13BDH2S	F	K	A	1	3	B	1	3	3	B	1	3	2	73.3	272	126.15	59.5	1	5	KM1	E	TANK													
66	8608	FKA13BGH2S	F	K	A	1	3	B	1	3	3	B	1	3	2	80.2	272	126.15	65.1	1	7	KM1	E	TANK													
67	8609	FKA13BLH2S	F	K	A	1	3	B	1	3	3	B	1	3	2	77.1	272	126.15	61.6	1	6	KM1	E	TANK													
68	8610	FKA14ACH2S	F	K	A	1	4	A	1	4	4	A	1	4	2	226.0	118	102.90	151.0	1	1	KM1	E	TANK													
69	8611	FKA14ADH2S	F	K	A	1	4	A	1	4	4	A	1	4	2	111.0	118	102.90	74.5	1	2	KM1	E	TANK													
70	8613	FKA14ALH2S	F	K	A	1	4	A	1	4	4	A	1	4	2	113.0	118	102.90	75.4	1	3	KM1	E	TANK													
71	8614	FKA14BCH2S	F	K	A	1	4	B	1	4	4	B	1	4	2	113.0	118	102.90	75.4	1	4	KM1	E	TANK													
72	8615	FKA14BDH2S	F	K	A	1	4	B	1	4	4	B	1	4	2	113.0	118	102.90	75.4	1	5	KM1	E	TANK													
73	8616	FKA14BGH2S	F	K	A	1	4	B	1	4	4	B	1	4	2	113.0	118	102.90	75.4	1	7	KM1	E	TANK													
74	8617	FKA14BLH2S	F	K	A	1	4	B	1	4	4	B	1	4	2	113.0	118	102.90	75.4	1	6	KM1	E	TANK													
75	8619	FKA21ACH2S	F	K	A	2	1	A	2	1	2	A	2	1	2	105.0	178	160.05	74.6	1	1	KM1	E	TANK													
76	8620	FKA21ADH2S	F	K	A	2	1	A	2	1	2	A	2	1	2	60.2	178	160.05	42.6	1	2	KM1	E	TANK													
77	8622	FKA21ALH2S	F	K	A	2	1	A	2	1	2	A	2	1	2	64.9	178	160.05	45.9	1	3	KM1	E	TANK													
78	8623	FKA21BCH2S	F	K	A	2	1	B	1	2	2	B	1	2	2	48.5	178	160.05	34.3	1	4	KM1	E	TANK													
79	8624	FKA21BDH2S	F	K	A	2	1	B	1	2	2	B	1	2	2	54.1	178	160.05	38.2	1	5	KM1	E	TANK													
80	8625	FKA21BGH2S	F	K	A	2	1	B	1	2	2	B	1	2	2	56.2	178	160.05	39.8	1	7	KM1	E	TANK													
81	8626	FKA21BLH2S	F	K	A	2	1	B	1	2	2	B	1	2	2	60.2	178	160.05	42.6	1	6	KM1	E	TANK													
82	8627	FKA22ACH2S	F	K	A	2	2	A	2	2	2	A	2	2	2	75.2	124	137.10	52.8	1	1	KM1	E	TANK													
83	8628	FKA22ADH2S	F	K	A	2	2	A	2	2	2	A	2	2	2	77.3	124	137.10	54.3	1	2	KM1	E	TANK													
84	8630	FKA22ALH2S	F	K	A	2	2	A	2	2	2	A	2	2	2	43.4	124	137.10	30.5	1	3	KM1	E	TANK													
85	8631	FKA22BCH2S	F	K	A	2	2	B	2	2	2	B	2	2	2	108.0	124	137.10	76.2	1	4	KM1	E	TANK													
86	8632	FKA22BDH2S	F	K	A	2	2	B	2	2	2	B	2	2	2	78.3	124	137.10	55.0	1	5	KM1	E	TANK													
87	8633	FKA22BGH2S	F	K	A	2	2	B	2	2	2	B	2	2	2	64.8	124	137.10	45.5	1	7	KM1	E	TANK													
88	8634	FKA22BLH2S	F	K	A	2	2	B	2	2	2	B	2	2	2	352.0	124	137.10	248.0	1	6	KM1	E	TANK													
89	8702	FKT13ACH2S	F	K	T	1	3	A	1	3	3	A	1	3	2	45.8	331	177.60	36.2	1	1	KTAN	F	TANK													
90	8703	FKT13ADH2S	F	K	T	1	3	A	1	3	3	A	1	3	2	44.4	331	177.60	35.1	1	2	KTAN	F	TANK													
91	8705	FKT13ALH2S	F	K	T	1	3	A	1	3	3	A	1	3	2	46.1	331	177.60	36.9	1	3	KTAN	F	TANK													
92	8706	FKT13BCH2S	F	K	T	1	3	B	1	3	3	B	1	3	2	43.6	331	177.60	36.2	1	4	KTAN	F	TANK													
93	8707	FKT13BDH2S	F	K	T	1	3	B	1	3	3	B	1	3	2	44.7	331	177.60	36.5	1	5	KTAN	F	TANK													
94	8708	FKT13BGH2S	F	K	T	1	3	B	1	3	3	B	1	3	2	88.8	331	177.60	72.0	1	7	KTAN	F	TANK													
95	8709	FKT13BLH2S	F	K	T	1	3	B	1	3	3	B	1	3	2	49.4	331	177.60	39.9	1	6	KTAN	F	TANK													
96	8710	FKT14ACH2S	F	K	T	1	4	A	1	4	4	A	1	4	2	67.0	577	155.55	52.5	1	1	KTAN	F	TANK													

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SASOUT.H2SDATA

145	O	B	S	8118	PSM21ALH2S	P	S	M	4	1	A	L	H2S	<	599.0	16	192	412.0	1	3	SM10	B	NON
146				8120	PSM21BCH2S	P	S	M	4	1	B	C	H2S	<	85.0	16	192	59.5	1	4	SM10	B	NON
147				8121	PSM21BDH2S	P	S	M	4	1	B	D	H2S	<	70.8	16	192	49.6	1	5	SM10	B	NON
148				8122	PSM21BLH2S	P	S	M	4	1	B	L	H2S	<	65.9	16	192	46.1	1	6	SM10	B	NON
149				8123	PSM22ACH2S	P	S	M	4	2	A	C	H2S	<	224.0	18	216	162.0	1	1	SM10	B	NON
150				8124	PSM22ADH2S	P	S	M	4	2	A	D	H2S	<	229.0	18	216	166.0	1	2	SM10	B	NON
151				8125	PSM22ALH2S	P	S	M	4	2	A	L	H2S	<	217.0	18	216	157.0	1	3	SM10	B	NON
152				8127	PSM22BCH2S	P	S	M	4	2	B	C	H2S	<	52.9	18	216	38.3	1	4	SM10	B	NON
153				8128	PSM22BDH2S	P	S	M	4	2	B	D	H2S	<	181.0	18	216	131.0	1	5	SM10	B	NON
154				8129	PSM22BLH2S	P	S	M	4	2	B	L	H2S	<	71.6	18	216	51.8	1	6	SM10	B	NON

HYDROGEN CYANIDE

SASOUT.HCNDATA

O	B	S	L	A	B	N	O	F	L	D	C	O	D	E	S	F	O	R	T	V	E	H	T	P	D	A	Y	N	O	Z	A	R	E	A	N	A	B	O	A	N	A	A	L	Y	T	A	N	C	F	I	R	O	T	C	O	M	A	S	S	T	O	T	A	S	T	O	C	N	C	I	S	E	L	L	E	C	T	T	R	E	F	Y	P	V	G	R	O	U	P
1	7302	FB813ACHCN	F	B	B	B	1	3	A	A	D	360.0	239	5.082	79.9	1	1	BBFV	A	NON																																																																					
2	7303	FB813ADHCN	F	B	B	B	1	3	A	A	D	398.0	239	5.082	88.8	1	2	BBFV	A	NON																																																																					
3	7305	FB813ALHCN	F	B	B	B	1	3	A	A	D	400.0	239	5.082	88.8	1	3	BBFV	A	NON																																																																					
4	7306	FB814ACHCN	F	B	B	B	1	4	A	A	C	339.0	424	10.002	51.5	1	1	BBFV	A	NON																																																																					
5	7307	FB814ADHCN	F	B	B	B	1	4	A	A	D	365.0	424	10.002	55.1	1	2	BBFV	A	NON																																																																					
6	7309	FB814ALHCN	F	B	B	B	1	4	A	A	C	396.0	424	10.002	59.7	1	3	BBFV	A	NON																																																																					
7	7315	FB822ACHCN	F	B	B	B	2	2	A	A	C	411.0	463	12.350	80.6	1	1	BBFV	A	NON																																																																					
8	7316	FB822ADHCN	F	B	B	B	2	2	A	A	D	402.0	463	12.350	78.8	1	2	BBFV	A	NON																																																																					
9	7318	FB822ALHCN	F	B	B	B	2	2	A	A	C	494.0	463	12.350	96.9	1	3	BBFV	A	NON																																																																					
10	7321	FB825ACHCN	F	B	B	B	2	5	A	A	C	208.0	382	11.525	138.0	1	1	BBFV	A	NON																																																																					
11	7322	FB825ADHCN	F	B	B	B	2	5	A	A	D	193.0	382	11.525	129.0	1	2	BBFV	A	NON																																																																					
12	7324	FB825ALHCN	F	B	B	B	2	5	A	A	C	229.0	382	11.525	153.0	1	3	BBFV	A	NON																																																																					
13	7502	FC113ACHCN	F	C	T	T	1	3	A	A	C	1070.0	160	58.500	223.0	1	1	CTAN	C	TANK																																																																					
14	7503	FC113ADHCN	F	C	T	T	1	3	A	A	D	1080.0	160	58.500	230.0	1	2	CTAN	C	TANK																																																																					
15	7505	FC113ALHCN	F	C	T	T	1	3	A	A	C	1150.0	160	58.500	234.0	1	3	CTAN	C	TANK																																																																					
16	7506	FC114ACHCN	F	C	T	T	1	4	A	A	C	1120.0	163	75.600	103.0	1	1	CTAN	C	TANK																																																																					
17	7507	FC114ADHCN	F	C	T	T	1	4	A	A	D	1110.0	163	75.600	103.0	1	2	CTAN	C	TANK																																																																					
18	7509	FC114ALHCN	F	C	T	T	1	4	A	A	C	1190.0	163	75.600	110.0	1	3	CTAN	C	TANK																																																																					
19	7511	FC121ACHCN	F	C	T	T	2	1	A	A	C	1090.0	162	69.900	547.0	1	1	CTAN	C	TANK																																																																					
20	7512	FC121ADHCN	F	C	T	T	2	1	A	A	D	1170.0	162	69.900	583.0	1	2	CTAN	C	TANK																																																																					
21	7514	FC121ALHCN	F	C	T	T	2	1	A	A	C	951.0	162	69.900	476.0	1	3	CTAN	C	TANK																																																																					
22	7515	FC122ACHCN	F	C	T	T	2	2	A	A	C	701.0	166	92.700	216.0	1	1	CTAN	C	TANK																																																																					
23	7516	FC122ADHCN	F	C	T	T	2	2	A	A	D	1150.0	166	92.700	355.0	1	2	CTAN	C	TANK																																																																					
24	7518	FC122ALHCN	F	C	T	T	2	2	A	A	C	1070.0	166	92.700	330.0	1	3	CTAN	C	TANK																																																																					
25	7602	FKA13ACHCN	F	K	A	A	1	3	A	A	C	207.0	272	126.150	151.0	1	1	KM1	E	TANK																																																																					
26	7603	FKA13ADHCN	F	K	A	A	1	3	A	A	D	207.0	272	126.150	151.0	1	2	KM1	E	TANK																																																																					
27	7605	FKA13ALHCN	F	K	A	A	1	3	A	A	C	207.0	272	126.150	151.0	1	3	KM1	E	TANK																																																																					
28	7606	FKA14ACHCN	F	K	A	A	1	4	A	A	C	311.0	118	102.900	208.0	1	1	KM1	E	TANK																																																																					
29	7608	FKA14ADHCN	F	K	A	A	1	4	A	A	D	311.0	118	102.900	208.0	1	2	KM1	E	TANK																																																																					
30	7609	FKA14ALHCN	F	K	A	A	1	4	A	A	C	311.0	118	102.900	208.0	1	3	KM1	E	TANK																																																																					
31	7611	FKA21ACHCN	F	K	A	A	2	1	A	A	C	1280.0	178	160.050	905.0	1	1	KM1	E	TANK																																																																					
32	7612	FKA21ADHCN	F	K	A	A	2	1	A	A	D	919.0	178	160.050	650.0	1	2	KM1	E	TANK																																																																					
33	7615	FKA22ACHCN	F	K	A	A	2	2	A	A	C	448.0	124	137.100	314.0	1	1	KM1	E	TANK																																																																					
34	7616	FKA22ADHCN	F	K	A	A	2	2	A	A	D	599.0	124	137.100	420.0	1	2	KM1	E	TANK																																																																					
35	7618	FKA22ALHCN	F	K	A	A	2	2	A	A	C	146.0	124	137.100	152.0	1	3	KM1	E	TANK																																																																					
36	7624	FKA25ACHCN	F	K	A	A	2	5	A	A	C	216.0	77	154.150	103.0	1	1	KM1	E	TANK																																																																					
37	7702	FKT13ACHCN	F	K	T	T	1	3	A	A	C	123.0	331	177.600	96.7	1	1	KTAN	F	TANK																																																																					
38	7703	FKT13ADHCN	F	K	T	T	1	3	A	A	D	102.0	331	177.600	80.6	1	2	KTAN	F	TANK																																																																					
39	7705	FKT13ALHCN	F	K	T	T	1	3	A	A	C	177.0	331	177.600	142.0	1	3	KTAN	F	TANK																																																																					
40	7706	FKT14ACHCN	F	K	T	T	1	4	A	A	C	226.0	577	155.550	177.0	1	1	KTAN	F	TANK																																																																					
41	7707	FKT14ADHCN	F	K	T	T	1	4	A	A	D	102.0	577	155.550	79.0	1	2	KTAN	F	TANK																																																																					
42	7709	FKT14ALHCN	F	K	T	T	1	4	A	A	C	92.5	577	155.550	72.5	1	3	KTAN	F	TANK																																																																					
43	7202	FSM13ACHCN	F	S	M	1	3	A	A	C	44.5	12	1.440	18.5	1	1	SM10	B	NON																																																																						
44	7203	FSM13ADHCN	F	S	M	1	3	A	A	D	44.5	12	1.440	18.5	1	2	SM10	B	NON																																																																						
45	7205	FSM13ALHCN	F	S	M	1	3	A	A	C	44.5	12	1.440	18.5	1	3	SM10	B	NON																																																																						
46	7206	FSM14ACHCN	F	S	M	1	4	A	A	C	142.0	16	1.920	32.3	1	1	SM10	B	NON																																																																						
47	7207	FSM14ADHCN	F	S	M	1	4	A	A	D	193.0	16	1.920	45.4	1	2	SM10	B	NON																																																																						
48	7209	FSM14ALHCN	F	S	M	1	4	A	A	C	73.2	16	1.920	17.5	1	3	SM10	B	NON																																																																						

NITRIC OXIDE

9:11 TUESDAY, JANUARY 10, 1989

SASOUT.NODATA

OBS	L	F	S	F	V	D	H	T	P	E	N	A	R	A	B	F	I	O	T	A	S	E	T	V	G
49	10218	FSM21ADNO	F	2	1	2	2	1	1	1	1	1	1	1	1	487.0	10	1.20	248.0	1	2	1	2	SM10	NON
50	10220	FSM21ALNO	F	2	1	2	2	1	1	1	1	1	1	1	1	635.0	10	1.20	339.0	1	3	1	3	SM10	NON
51	10224	FSM22ACNO	F	2	2	2	2	2	2	2	2	2	2	2	2	667.0	8	0.96	357.0	1	1	1	1	SM10	NON
52	10225	FSM22ADNO	F	2	2	2	2	2	2	2	2	2	2	2	2	503.0	8	0.96	289.0	1	2	1	2	SM10	NON
53	10227	FSM22ALNO	F	2	2	2	2	2	2	2	2	2	2	2	2	470.0	8	0.96	258.0	1	3	1	3	SM10	NON
54	10233	FSM25ACNO	F	2	5	2	5	2	5	2	5	2	5	2	5	649.0	8	0.96	335.0	1	1	1	1	SM10	NON
55	10234	FSM25ADNO	F	2	5	2	5	2	5	2	5	2	5	2	5	630.0	8	0.96	320.0	1	2	1	2	SM10	NON
56	10102	PSM13ACNO	P	3	3	3	3	3	3	3	3	3	3	3	3	838.0	16	1.92	261.0	1	1	1	1	SM10	NON
57	10103	PSM13ADNO	P	3	3	3	3	3	3	3	3	3	3	3	3	118.0	16	1.92	36.1	1	2	1	2	SM10	NON
58	10104	PSM13ALNO	P	3	3	3	3	3	3	3	3	3	3	3	3	473.0	16	1.92	144.0	1	3	1	3	SM10	NON
59	10109	PSM14ACNO	P	3	4	3	4	3	4	3	4	3	4	3	4	163.0	16	1.92	52.2	1	1	1	1	SM10	NON
60	10110	PSM14ADNO	P	3	4	3	4	3	4	3	4	3	4	3	4	135.0	16	1.92	41.8	1	2	1	2	SM10	NON
61	10111	PSM14ALNO	P	3	4	3	4	3	4	3	4	3	4	3	4	182.0	16	1.92	58.5	1	3	1	3	SM10	NON
62	10116	PSM21ACNO	P	4	1	4	1	4	1	4	1	4	1	4	1	210.0	16	1.92	145.0	1	1	1	1	SM10	NON
63	10117	PSM21ADNO	P	4	1	4	1	4	1	4	1	4	1	4	1	286.0	16	1.92	197.0	1	2	1	2	SM10	NON
64	10118	PSM21ALNO	P	4	1	4	1	4	1	4	1	4	1	4	1	273.0	16	1.92	187.0	1	3	1	3	SM10	NON
65	10123	PSM22ACNO	P	4	2	4	2	4	2	4	2	4	2	4	2	107.0	18	2.16	77.2	1	1	1	1	SM10	NON
66	10124	PSM22ADNO	P	4	2	4	2	4	2	4	2	4	2	4	2	84.8	18	2.16	61.4	1	2	1	2	SM10	NON
67	10125	PSM22ALNO	P	4	2	4	2	4	2	4	2	4	2	4	2	88.6	18	2.16	64.1	1	3	1	3	SM10	NON

NITROGEN DIOXIDE - GENERAL AREA

O	B	S	L	A	R	N	A	B	A	R	I	I	O	T	O	T	S	F	T	Y	V	G	
1	10302	FB813ACN02	F	B	B	B	1	3	A	C	2240	239	5.082	498	1	1	1	1	1	1	BBFV	A	NON
2	10303	FB813ADN02	F	B	B	B	1	3	A	D	1380	239	5.082	307	1	2	1	1	1	1	BBFV	A	NON
3	10305	FB813ALN02	F	B	B	B	1	3	A	L	1780	239	5.082	395	1	3	1	1	1	1	BBFV	A	NON
4	10306	FB814ACN02	F	B	B	B	1	4	A	C	2100	424	10.002	320	1	1	1	1	1	1	BBFV	A	NON
5	10307	FB814ADN02	F	B	B	B	1	4	A	D	1190	424	10.002	180	1	2	1	1	1	1	BBFV	A	NON
6	10309	FB814ALN02	F	B	B	B	1	4	A	L	1310	424	10.002	198	1	3	1	1	1	1	BBFV	A	NON
7	10311	FB821ACN02	F	B	B	B	2	1	A	C	1160	467	12.750	250	1	1	1	1	1	1	BBFV	A	NON
8	10312	FB821ADN02	F	B	B	B	2	1	A	D	876	467	12.750	188	1	2	1	1	1	1	BBFV	A	NON
9	10314	FB821ALN02	F	B	B	B	2	1	A	L	1300	467	12.750	280	1	3	1	1	1	1	BBFV	A	NON
10	10315	FB822ACN02	F	B	B	B	2	2	A	C	894	463	12.350	175	1	1	1	1	1	1	BBFV	A	NON
11	10316	FB822ADN02	F	B	B	B	2	2	A	D	675	463	12.350	132	1	2	1	1	1	1	BBFV	A	NON
12	10502	FCT13ACN02	F	C	T	1	3	A	C	2890	160	58.500	602	1	1	1	1	1	1	1	CTAN	C	TANK
13	10503	FCT13ADN02	F	C	T	1	3	A	D	4290	160	58.500	913	1	2	1	1	1	1	1	CTAN	C	TANK
14	10505	FCT13ALN02	F	C	T	1	3	A	L	5170	160	58.500	1060	1	3	1	1	1	1	1	CTAN	C	TANK
15	10506	FCT14ACN02	F	C	T	1	4	A	C	1560	163	75.600	143	1	1	1	1	1	1	1	CTAN	C	TANK
16	10507	FCT14ADN02	F	C	T	1	4	A	D	2130	163	75.600	109	1	2	1	1	1	1	1	CTAN	C	TANK
17	10509	FCT14ALN02	F	C	T	1	4	A	L	2920	163	75.600	272	1	3	1	1	1	1	1	CTAN	C	TANK
18	10511	FCT21ACN02	F	C	T	2	1	A	C	3550	162	69.900	1740	1	1	1	1	1	1	1	CTAN	C	TANK
19	10512	FCT21ADN02	F	C	T	2	1	A	D	3090	162	69.900	1540	1	2	1	1	1	1	1	CTAN	C	TANK
20	10514	FCT21ALN02	F	C	T	2	1	A	L	3380	162	69.900	1690	1	3	1	1	1	1	1	CTAN	C	TANK
21	10515	FCT22ACN02	F	C	T	2	2	A	C	8430	166	92.700	2590	1	1	1	1	1	1	1	CTAN	C	TANK
22	10516	FCT22ADN02	F	C	T	2	2	A	D	2820	166	92.700	868	1	2	1	1	1	1	1	CTAN	C	TANK
23	10518	FCT22ALN02	F	C	T	2	2	A	L	2280	166	92.700	702	1	3	1	1	1	1	1	CTAN	C	TANK
24	10602	FKA13ACN02	F	K	A	1	3	A	C	267	272	126.150	195	1	1	1	1	1	1	1	KM1	E	TANK
25	10603	FKA13ADN02	F	K	A	1	3	A	D	249	272	126.150	182	1	2	1	1	1	1	1	KM1	E	TANK
26	10605	FKA13ALN02	F	K	A	1	3	A	L	288	272	126.150	210	1	3	1	1	1	1	1	KM1	E	TANK
27	10606	FKA14ACN02	F	K	A	1	4	A	C	561	118	102.900	375	1	1	1	1	1	1	1	KM1	E	TANK
28	10607	FKA14ADN02	F	K	A	1	4	A	D	540	118	102.900	361	1	2	1	1	1	1	1	KM1	E	TANK
29	10609	FKA14ALN02	F	K	A	1	4	A	L	561	118	102.900	375	1	3	1	1	1	1	1	KM1	E	TANK
30	10611	FKA21ACN02	F	K	A	2	1	A	C	202	178	160.050	143	1	1	1	1	1	1	1	KM1	E	TANK
31	10612	FKA21ADN02	F	K	A	2	1	A	D	164	178	160.050	116	1	2	1	1	1	1	1	KM1	E	TANK
32	10614	FKA21ALN02	F	K	A	2	1	A	L	219	178	160.050	155	1	3	1	1	1	1	1	KM1	E	TANK
33	10615	FKA22ACN02	F	K	A	2	2	A	C	532	124	137.100	373	1	1	1	1	1	1	1	KM1	E	TANK
34	10616	FKA22ADN02	F	K	A	2	2	A	D	351	124	137.100	247	1	2	1	1	1	1	1	KM1	E	TANK
35	10615	FKA22ALN02	F	K	A	2	2	A	L	234	124	137.100	164	1	3	1	1	1	1	1	KM1	E	TANK
36	10624	FKA25ALN02	F	K	A	2	5	A	L	264	77	154.150	186	1	3	1	1	1	1	1	KM1	E	TANK
37	10702	FKT13ACN02	F	K	T	1	3	A	C	348	331	177.600	275	1	1	1	1	1	1	1	KTAN	F	TANK
38	10703	FKT13ADN02	F	K	T	1	3	A	D	424	331	177.600	335	1	2	1	1	1	1	1	KTAN	F	TANK
39	10705	FKT13ALN02	F	K	T	1	3	A	L	466	331	177.600	372	1	3	1	1	1	1	1	KTAN	F	TANK
40	10706	FKT14ACN02	F	K	T	1	4	A	C	458	577	155.550	359	1	1	1	1	1	1	1	KTAN	F	TANK
41	10707	FKT14ADN02	F	K	T	1	4	A	D	461	577	155.550	357	1	2	1	1	1	1	1	KTAN	F	TANK
42	10709	FKT14ALN02	F	K	T	1	4	A	L	494	577	155.550	387	1	3	1	1	1	1	1	KTAN	F	TANK
43	10203	FCT113ACN02	F	C	T	1	3	A	C	309	12	1.440	128	1	2	1	1	1	1	1	SM10	B	NON
44	10205	FCT113ADN02	F	C	T	1	3	A	D	418	12	1.440	173	1	3	1	1	1	1	1	SM10	B	NON
45	10209	FCT113ALN02	F	C	T	1	3	A	L	731	16	1.920	166	1	1	1	1	1	1	1	SM10	B	NON
46	10210	FST14ACN02	F	S	T	1	4	A	C	603	16	1.920	142	1	2	1	1	1	1	1	SM10	B	NON
47	10212	FST14ADN02	F	S	T	1	4	A	D	596	16	1.920	143	1	3	1	1	1	1	1	SM10	B	NON
48	10217	FST21ACN02	F	S	T	2	1	A	C	829	10	1.200	464	1	1	1	1	1	1	1	SM10	B	NON

SASOUT.NO2DATA

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NITROGEN DIOXIDE - BREATHING ZONE

SASOUT.N02DATA

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1	20301																																				
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11	20311																																				
12	20312																																				
13	20313																																				
14	20314																																				
15	20500																																				
16	20501																																				
17	20515																																				
18	20502																																				
19	20503																																				
20	20504																																				
21	20516																																				
22	20505																																				
23	20506																																				
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25	20517																																				
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27	20509																																				
28	20510																																				
29	20518																																				
30	20511																																				
31	20600																																				
32	20601																																				
33	20602																																				
34	20603																																				

SASOUT.NO2DATA

[illegible]

FORMALDEHYDE

SASOUT.FORDATA

[illegible]

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AMMONIA

SULFUR DIOXIDE

RESPIRABLE SUSPENDED PARTICULATE

SASOUT.RSPDATA

OBS	L A B N O	F L D C O D E	S C A L E	F O R T	V E H T Y P	D A Y	V E H N O	F I X B Z	A R E A	B O O L E A N	F I R E C C O N C	T O T T C A L	T O T M A S S	T O T C O N C	S E L E C T	T R E A T	V T Y P F O R T	V L O C	G R O U P	
49	16209	FSM14ALRSP	F	S	M	1	4	A	L	RSP	<	741	16	192	177	1	3	SM10	B	NON
50	16211	FSM21ACRSP	F	S	M	2	1	A	C	RSP	<	851	10	120	476	1	1	SM10	B	NON
51	16212	FSM21ADRSP	F	S	M	2	1	A	D	RSP	<	848	10	120	432	1	2	SM10	B	NON
52	16214	FSM21ALRSP	F	S	M	2	1	A	L	RSP	<	836	10	120	446	1	3	SM10	B	NON
53	16215	FSM22ACRSP	F	S	M	2	2	A	C	RSP	<	946	8	96	506	1	1	SM10	B	NON
54	16216	FSM22ADRSP	F	S	M	2	2	A	D	RSP	<	913	8	96	524	1	2	SM10	B	NON
55	16218	FSM22ALRSP	F	S	M	2	2	A	L	RSP	<	882	8	96	484	1	3	SM10	B	NON
56	16102	PSM13ACRSP	P	S	M	3	3	A	C	RSP	<	760	16	192	238	1	1	SM10	B	NON
57	16103	PSM13ADRSP	P	S	M	3	3	A	D	RSP	<	372	16	192	114	1	2	SM10	B	NON
58	16104	PSM13ALRSP	P	S	M	3	3	A	L	RSP	<	520	16	192	158	1	3	SM10	B	NON
59	16106	PSM14ACRSP	P	S	M	3	4	A	C	RSP	<	407	16	192	131	1	1	SM10	B	NON
60	16107	PSM14ADRSP	P	S	M	3	4	A	D	RSP	<	398	16	192	123	1	2	SM10	B	NON
61	16108	PSM14ALRSP	P	S	M	3	4	A	L	RSP	<	364	16	192	117	1	3	SM10	B	NON
62	16110	PSM21ACRSP	P	S	M	4	1	A	C	RSP	<	364	16	192	253	1	1	SM10	B	NON
63	16111	PSM21ADRSP	P	S	M	4	1	A	D	RSP	<	329	16	192	227	1	2	SM10	B	NON
64	16112	PSM21ALRSP	P	S	M	4	1	A	L	RSP	<	488	16	192	335	1	3	SM10	B	NON
65	16114	PSM22ACRSP	P	S	M	4	2	A	C	RSP	<	371	18	216	269	1	1	SM10	B	NON
66	16115	PSM22ADRSP	P	S	M	4	2	A	D	RSP	<	338	18	216	245	1	2	SM10	B	NON
67	16116	PSM22ALRSP	P	S	M	4	2	A	L	RSP	<	358	18	216	259	1	3	SM10	B	NON

TOTAL SUSPENDED PARTICULATES

SASOUT.TSPDATA

OBS	L	F	S	F	V	F	A	B	A	I	T	O	S	T	Y	G
1	17302	FB813ACTSP	F	B	B	B	B	B	TSP	1360	239	5.08	303	1	1	NON
2	17303	FB813ADTSP	F	B	B	B	B	B	TSP	55600	239	5.08	12400	1	2	NON
3	17305	FB813ALTSP	F	B	B	B	B	B	TSP	1370	239	5.08	304	1	3	NON
4	17306	FB813BCTSP	F	B	B	B	B	B	TSP	1380	239	5.08	306	1	4	NON
5	17307	FB813BDTSP	F	B	B	B	B	B	TSP	1420	239	5.08	316	1	5	NON
6	17308	FB813BLTSP	F	B	B	B	B	B	TSP	2030	239	5.08	674	1	6	NON
7	17309	FB814ACTSP	F	B	B	B	B	B	TSP	2070	424	10.00	315	1	1	NON
8	17310	FB814ADTSP	F	B	B	B	B	B	TSP	1480	424	10.00	224	1	2	NON
9	17312	FB814ALTSP	F	B	B	B	B	B	TSP	4730	424	10.00	714	1	3	NON
10	17313	FB814BCTSP	F	B	B	B	B	B	TSP	2310	424	10.00	348	1	4	NON
11	17314	FB814BDTSP	F	B	B	B	B	B	TSP	1680	424	10.00	336	1	5	NON
12	17315	FB814BLTSP	F	B	B	B	B	B	TSP	2120	424	10.00	423	1	6	NON
13	17324	FB822ACTSP	F	B	B	B	B	B	TSP	2800	463	12.35	549	1	1	NON
14	17325	FB822ADTSP	F	B	B	B	B	B	TSP	3290	463	12.35	646	1	2	NON
15	17327	FB822ALTSP	F	B	B	B	B	B	TSP	3310	463	12.35	649	1	3	NON
16	17328	FB822BCTSP	F	B	B	B	B	B	TSP	2730	463	12.35	535	1	4	NON
17	17329	FB822BDTSP	F	B	B	B	B	B	TSP	3970	463	12.35	779	1	5	NON
18	17330	FB822BLTSP	F	B	B	B	B	B	TSP	2560	463	12.35	501	1	6	NON
19	17333	FB825ACTSP	F	B	B	B	B	B	TSP	2960	382	11.53	1970	1	1	NON
20	17334	FB825ADTSP	F	B	B	B	B	B	TSP	2390	382	11.53	1590	1	2	NON
21	17336	FB825ALTSP	F	B	B	B	B	B	TSP	6140	382	11.53	4090	1	3	NON
22	17337	FB825BCTSP	F	B	B	B	B	B	TSP	1750	382	11.53	1170	1	4	NON
23	17338	FB825BDTSP	F	B	B	B	B	B	TSP	1980	382	11.53	1320	1	5	NON
24	17339	FB825BLTSP	F	B	B	B	B	B	TSP	1850	382	11.53	1230	1	6	NON
25	17502	FCT13ACTSP	F	C	T	T	T	T	TSP	13200	160	58.50	2750	1	1	TANK
26	17503	FCT13ADTSP	F	C	T	T	T	T	TSP	5700	160	58.50	1210	1	2	TANK
27	17505	FCT13ALTSP	F	C	T	T	T	T	TSP	11200	160	58.50	2290	1	3	TANK
28	17506	FCT13BCTSP	F	C	T	T	T	T	TSP	6140	160	58.50	1390	1	4	TANK
29	17507	FCT13BDTSP	F	C	T	T	T	T	TSP	4160	160	58.50	913	1	5	TANK
30	17540	FCT13BGTSP	F	C	T	T	T	T	TSP	3400	160	58.50	739	1	7	TANK
31	17508	FCT13BLTSP	F	C	T	T	T	T	TSP	4950	160	58.50	1160	1	6	TANK
32	17509	FCT14ACTSP	F	C	T	T	T	T	TSP	10300	163	75.60	946	1	1	TANK
33	17510	FCT14ADTSP	F	C	T	T	T	T	TSP	4120	163	75.60	382	1	2	TANK
34	17512	FCT14ALTSP	F	C	T	T	T	T	TSP	7370	163	75.60	686	1	3	TANK
35	17513	FCT14BCTSP	F	C	T	T	T	T	TSP	6760	163	75.60	644	1	4	TANK
36	17514	FCT14BDTSP	F	C	T	T	T	T	TSP	6900	163	75.60	651	1	5	TANK
37	17541	FCT14BGTSP	F	C	T	T	T	T	TSP	3750	163	75.60	361	1	7	TANK
38	17515	FCT14BLTSP	F	C	T	T	T	T	TSP	4900	163	75.60	467	1	6	TANK
39	17517	FCT12ACTSP	F	C	T	T	T	T	TSP	2740	162	69.90	1370	1	1	TANK
40	17518	FCT12ADTSP	F	C	T	T	T	T	TSP	7190	162	69.90	3590	1	2	TANK
41	17520	FCT12ALTSP	F	C	T	T	T	T	TSP	2610	162	69.90	1300	1	3	TANK
42	17521	FCT12BCTSP	F	C	T	T	T	T	TSP	2630	162	69.90	1320	1	4	TANK
43	17522	FCT12BDTSP	F	C	T	T	T	T	TSP	2900	162	69.90	1450	1	5	TANK
44	17542	FCT12BGTSP	F	C	T	T	T	T	TSP	3750	162	69.90	1870	1	7	TANK
45	17523	FCT12BLTSP	F	C	T	T	T	T	TSP	2750	162	69.90	1380	1	6	TANK
46	17524	FCT22ACTSP	F	C	T	T	T	T	TSP	2480	166	92.70	762	1	1	TANK
47	17525	FCT22ADTSP	F	C	T	T	T	T	TSP	2890	166	92.70	888	1	2	TANK
48	17527	FCT22ALTSP	F	C	T	T	T	T	TSP	2750	166	92.70	845	1	3	TANK

SASOUT.TSPDATA

OBS	LABNO	F	L	D	C	O	D	E	S	C	A	L	E	F	I	R	E	C	O	C	N	A	L	T	O	T	M	A	S	T	O	S	T	R	E	A	T	V	T	Y	P	F	O	R	O	U	P
97	17205	FSM13AL	TSP	L	TSP	443	12	144	184	1	3	SM10	B	NON																																	
98	17206	FSM13BCT	TSP	C	TSP	876	12	144	363	1	4	SM10	B	NON																																	
99	17207	FSM13BD	TSP	D	TSP	818	12	144	339	1	5	SM10	B	NON																																	
100	17208	FSM13BL	TSP	L	TSP	393	12	144	163	1	6	SM10	B	NON																																	
101	17209	FSM14ACT	TSP	A	TSP	9000	16	192	2050	1	1	SM10	B	NON																																	
102	17210	FSM14AD	TSP	D	TSP	2240	16	192	526	1	2	SM10	B	NON																																	
103	17212	FSM14ALT	TSP	A	TSP	2240	16	192	526	1	3	SM10	B	NON																																	
104	17213	FSM14BCT	TSP	B	TSP	2080	16	192	524	1	4	SM10	B	NON																																	
105	17214	FSM14BD	TSP	D	TSP	2280	16	192	570	1	5	SM10	B	NON																																	
106	17215	FSM14BLT	TSP	L	TSP	1050	16	192	266	1	6	SM10	B	NON																																	
107	17217	FSM21ACT	TSP	A	TSP	846	10	120	474	1	1	SM10	B	NON																																	
108	17218	FSM21AD	TSP	D	TSP	1800	10	120	915	1	2	SM10	B	NON																																	
109	17220	FSM21ALT	TSP	A	TSP	851	10	120	454	1	3	SM10	B	NON																																	
110	17221	FSM21BCT	TSP	B	TSP	1710	10	120	834	1	4	SM10	B	NON																																	
111	17222	FSM21BD	TSP	D	TSP	848	10	120	413	1	5	SM10	B	NON																																	
112	17223	FSM21BLT	TSP	L	TSP	2200	10	120	1070	1	6	SM10	B	NON																																	
113	17224	FSM22ACT	TSP	A	TSP	1500	8	96	802	1	1	SM10	B	NON																																	
114	17225	FSM22AD	TSP	D	TSP	897	8	96	515	1	2	SM10	B	NON																																	
115	17227	FSM22ALT	TSP	A	TSP	905	8	96	497	1	3	SM10	B	NON																																	
116	17228	FSM22BCT	TSP	B	TSP	1190	8	96	684	1	4	SM10	B	NON																																	
117	17229	FSM22BD	TSP	D	TSP	1240	8	96	724	1	5	SM10	B	NON																																	
118	17230	FSM22BLT	TSP	L	TSP	1170	8	96	674	1	6	SM10	B	NON																																	
119	17102	PSM13ACT	TSP	A	TSP	662	16	192	207	1	1	SM10	B	NON																																	
120	17103	PSM13AD	TSP	D	TSP	369	16	192	113	1	2	SM10	B	NON																																	
121	17104	PSM13ALT	TSP	A	TSP	384	16	192	117	1	3	SM10	B	NON																																	
122	17106	PSM13BCT	TSP	B	TSP	380	16	192	178	1	4	SM10	B	NON																																	
123	17107	PSM13BD	TSP	D	TSP	586	16	192	268	1	5	SM10	B	NON																																	
124	17108	PSM13BLT	TSP	L	TSP	586	16	192	267	1	6	SM10	B	NON																																	
125	17109	PSM14ACT	TSP	A	TSP	1100	16	192	355	1	1	SM10	B	NON																																	
126	17110	PSM14AD	TSP	D	TSP	600	16	192	185	1	2	SM10	B	NON																																	
127	17111	PSM14ALT	TSP	A	TSP	399	16	192	128	1	3	SM10	B	NON																																	
128	17113	PSM14BCT	TSP	B	TSP	10600	16	192	5150	1	4	SM10	B	NON																																	
129	17114	PSM14BD	TSP	D	TSP	367	16	192	176	1	5	SM10	B	NON																																	
130	17115	PSM14BLT	TSP	L	TSP	388	16	192	191	1	6	SM10	B	NON																																	
131	17116	PSM21ACT	TSP	A	TSP	364	16	192	253	1	1	SM10	B	NON																																	
132	17117	PSM21AD	TSP	D	TSP	329	16	192	227	1	2	SM10	B	NON																																	
133	17118	PSM21ALT	TSP	A	TSP	439	16	192	302	1	3	SM10	B	NON																																	
134	17120	PSM21BCT	TSP	B	TSP	329	16	192	230	1	4	SM10	B	NON																																	
135	17121	PSM21BD	TSP	D	TSP	497	16	192	348	1	5	SM10	B	NON																																	
136	17122	PSM21BLT	TSP	L	TSP	376	16	192	263	1	6	SM10	B	NON																																	
137	17123	PSM22ACT	TSP	A	TSP	456	18	216	330	1	1	SM10	B	NON																																	
138	17124	PSM22AD	TSP	D	TSP	347	18	216	251	1	2	SM10	B	NON																																	
139	17125	PSM22ALT	TSP	A	TSP	351	18	216	254	1	3	SM10	B	NON																																	
140	17127	PSM22BCT	TSP	B	TSP	565	18	216	409	1	4	SM10	B	NON																																	
141	17128	PSM22BD	TSP	D	TSP	376	18	216	272	1	5	SM10	B	NON																																	
142	17129	PSM22BLT	TSP	L	TSP	462	18	216	334	1	6	SM10	B	NON																																	

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ALDEHYDES

CONCENTRATION (TOTCONC) OF ALDEHYDES (UG/M3)

LABNO	FLDCODE	ACETAL (UG/M3)	ACROLEIN (UG/M3)	CROTONAL (UG/M3)	BUTYRAL (UG/M3)	BENZAL (UG/M3)	HEXANAL (UG/M3)	TOTCAL (NO.)	TOTMASS (KG)
1101	PSM12AGALD	2.64E+01	< 3.00E-01	6.50E-01	2.65E+00	6.83E-01	< 3.83E-01	16	192.00
1102	PSM13ACALD	< 2.41E-01	< 3.10E-01	< 2.41E-01	< 6.38E-01	< 5.17E-01	< 3.97E-01	16	192.00
1103	PSM13ADALD	3.34E-01	< 3.34E-01	< 2.60E-01	< 6.86E-01	< 5.56E-01	< 4.27E-01	16	192.00
1104	PSM13ALALD	< 2.27E-01	< 2.92E-01	< 2.27E-01	< 6.00E-01	< 4.86E-01	< 3.73E-01	16	192.00
1105	PSM13AGALD	2.66E+01	< 2.92E-01	4.86E-01	2.97E+00	1.33E+00	< 3.73E-01	16	192.00
1106	PSM14ACALD	< 2.65E-01	< 3.41E-01	< 2.65E-01	< 7.01E-01	< 5.68E-01	< 4.36E-01	16	192.00
1107	PSM14ADALD	1.63E+00	< 3.85E-01	< 3.00E-01	< 7.92E-01	< 6.42E-01	< 4.92E-01	16	192.00
1108	PSM14ALALD	< 2.83E-01	< 3.63E-01	< 2.83E-01	< 7.47E-01	< 6.06E-01	< 4.64E-01	16	192.00
1109	PSM14AGALD	2.06E+00	< 3.63E-01	< 2.83E-01	< 7.47E-01	< 6.06E-01	< 4.64E-01	16	192.00
1110	PSM21ACALD	< 4.67E-01	< 6.01E-01	< 4.67E-01	< 1.23E+00	< 1.00E+00	< 7.67E-01	16	192.00
1111	PSM21ADALD	< 5.19E-01	< 6.68E-01	< 5.19E-01	< 1.37E+00	< 1.11E+00	< 8.53E-01	16	192.00
1112	PSM21ALALD	< 4.15E-01	< 5.34E-01	< 4.15E-01	< 1.10E+00	< 8.90E-01	< 6.82E-01	16	192.00
1113	PSM21AGALD	4.33E+01	< 7.38E-01	7.38E-01	1.25E+01	9.22E+00	1.80E+00	16	192.00
1114	PSM22ACALD	8.92E-01	< 6.69E-01	< 5.20E-01	< 1.37E+00	< 1.11E+00	< 8.54E-01	18	216.00
1115	PSM22ADALD	6.10E-01	< 6.86E-01	< 5.34E-01	< 1.41E+00	< 1.14E+00	< 8.77E-01	18	216.00
1116	PSM22ALALD	2.18E+00	< 5.45E-01	< 4.24E-01	< 1.12E+00	< 9.08E-01	< 6.96E-01	18	216.00
1200	FSM11AGALD	< 3.03E-01	< 2.42E-01	< 4.24E-01	< 5.14E-01	< 6.66E-01	< 5.75E-01	18	216.00
1202	FSM13ACALD	2.15E+00	< 1.74E-01	< 3.04E-01	< 3.69E-01	< 4.78E-01	< 4.13E-01	12	144.00
1203	FSM13ADALD	1.43E+01	< 1.74E-01	< 3.04E-01	< 3.69E-01	< 4.78E-01	< 4.13E-01	12	144.00
1205	FSM13ALALD	4.69E+00	< 1.79E-01	< 3.13E-01	< 3.80E-01	< 4.91E-01	< 4.24E-01	12	144.00
1206	FSM14ACALD	3.37E+00	< 2.08E-01	< 3.63E-01	< 4.41E-01	< 5.71E-01	< 4.93E-01	16	192.00
1207	FSM14ADALD	5.58E-01	< 2.35E-01	< 4.11E-01	< 5.00E-01	< 6.46E-01	< 5.58E-01	16	192.00
1209	FSM14ALALD	7.47E+00	< 2.13E-01	< 3.73E-01	< 4.53E-01	9.07E-01	< 5.07E-01	16	192.00
1211	FSM21ACALD	8.22E+00	< 5.48E-01	< 9.59E-01	< 1.16E+00	< 1.51E+00	< 1.30E+00	10	120.00
1212	FSM21ADALD	5.99E+00	< 5.32E-01	< 9.32E-01	< 1.13E+00	< 1.46E+00	< 1.26E+00	10	120.00
1214	FSM21ALALD	4.53E+00	< 5.84E-01	< 1.02E+00	< 1.24E+00	< 1.61E+00	< 1.39E+00	10	120.00
1215	FSM22ACALD	< 8.02E-01	9.63E-01	< 1.12E+00	< 1.36E+00	< 1.77E+00	< 1.52E+00	8	96.00
1216	FSM22ADALD	2.99E+00	1.06E+00	< 1.35E+00	< 1.64E+00	< 2.12E+00	< 1.83E+00	8	96.00
1218	FSM22ALALD	1.66E+00	< 6.03E-01	< 1.06E+00	< 1.28E+00	< 1.66E+00	< 1.43E+00	8	96.00
1221	FSM25ACALD	8.87E+00	< 6.45E-01	< 1.13E+00	< 1.37E+00	< 1.77E+00	< 1.53E+00	8	96.00
1222	FSM25ADALD	< 7.76E-01	< 6.21E-01	< 1.09E+00	< 1.32E+00	< 1.71E+00	< 1.47E+00	8	96.00
1224	FSM25ALALD	2.59E+02	< 7.66E-01	2.78E+00	< 1.63E+00	2.30E+00	< 1.82E+00	8	96.00
1302	FBB13ACALD	< 4.65E-01	< 3.72E-01	< 6.51E-01	< 7.90E-01	< 1.02E+00	< 8.83E-01	239	5.08
1303	FBB13ADALD	8.06E+00	< 3.79E-01	< 6.63E-01	1.71E+01	< 1.04E+00	< 9.00E-01	239	5.08
1305	FBB13ALALD	4.03E+00	< 3.84E-01	< 6.72E-01	< 8.16E-01	< 1.06E+00	< 9.12E-01	239	5.08
1306	FBB14ACALD	7.50E+00	< 2.86E-01	< 5.00E-01	9.64E+00	9.28E-01	7.14E-01	424	10.00
1307	FBB14ADALD	5.64E+00	< 2.51E-01	< 4.38E-01	1.16E+01	< 6.89E-01	< 5.95E-01	424	10.00
1309	FBB14ALALD	2.67E+00	< 2.46E-01	< 4.30E-01	< 5.22E-01	< 6.75E-01	< 5.83E-01	424	10.00

CONCENTRATION (TOTCONC) OF ALDEHYDES (UG/M3)

LABNO	FLDCODE	ACETAL (UG/M3)	ACROLEIN (UG/M3)	CROTONAL (UG/M3)	BUTYRAL (UG/M3)	BENZAL (UG/M3)	HEXANAL (UG/M3)	TOTCAL (NO.)	TOTMASS (KG)
1011	FBB21ACALD	5.47E+00	< 2.57E-01	< 4.50E-01	4.18E+01	9.97E-01	6.43E-01	467	12.75
1312	FBB21ADALD	3.50E-01	< 2.54E-01	< 4.45E-01	< 5.40E-01	< 6.99E-01	< 6.04E-01	467	12.75
1314	FBB21ALALD	1.54E+00	< 2.46E-01	< 4.30E-01	< 5.22E-01	8.61E-01	< 5.84E-01	467	12.75
1315	FBB22ACALD	1.17E+00	< 2.13E-01	< 3.73E-01	< 4.53E-01	< 5.87E-01	< 5.07E-01	463	12.35
1316	FBB22ADALD	< 2.85E-01	< 2.28E-01	< 3.98E-01	< 4.84E-01	< 6.26E-01	< 5.41E-01	463	12.35
1318	FBB22ALALD	< 2.86E-01	< 2.28E-01	< 4.00E-01	2.11E+00	< 6.28E-01	< 5.43E-01	463	12.35
1321	FBB25ACALD	2.77E+00	< 7.14E-01	< 1.25E+00	< 1.52E+00	< 1.96E+00	< 1.69E+00	382	11.53
1322	FBB25ADALD	< 8.80E-01	< 7.04E-01	< 1.23E+00	< 1.50E+00	< 1.94E+00	< 1.67E+00	382	11.53
1324	FBB25ALALD	5.13E+00	< 7.07E-01	< 1.24E+00	< 1.50E+00	< 1.94E+00	< 1.68E+00	382	11.53
1502	FCT13ACALD	4.73E+00	< 2.30E+00	< 1.79E+00	< 2.68E+00	< 3.32E+00	4.98E+00	160	58.50
1503	FCT13ADALD	8.06E+00	< 2.13E+00	< 1.66E+00	< 2.49E+00	< 3.08E+00	3.44E+00	160	58.50
1505	FCT13ALALD	6.29E+00	< 2.02E+00	< 1.57E+00	< 2.36E+00	< 2.92E+00	3.59E+00	160	58.50
1506	FCT14ACALD	4.18E+00	< 1.02E+00	< 7.91E-01	< 1.19E+00	< 1.47E+00	5.26E+00	163	75.60
1507	FCT14ADALD	9.25E+00	< 9.79E-01	< 7.61E-01	< 1.14E+00	< 1.41E+00	5.98E+00	163	75.60
1509	FCT14ALALD	5.20E+00	< 9.37E-01	< 7.29E-01	2.24E+00	< 1.35E+00	6.25E+00	163	75.60
1511	FCT21ACALD	1.13E+01	< 5.20E+00	< 4.04E+00	< 6.07E+00	< 7.51E+00	< 8.09E+00	162	69.90
1512	FCT21ADALD	1.71E+01	< 5.70E+00	< 4.43E+00	< 6.65E+00	< 8.23E+00	< 8.86E+00	162	69.90
1513	FCT21AGALD	1.54E+01	< 5.56E+00	< 4.32E+00	< 6.49E+00	< 8.03E+00	< 8.65E+00	162	69.90
1514	FCT21ALALD	1.91E+01	< 5.20E+00	< 4.04E+00	< 6.07E+00	< 7.51E+00	< 8.09E+00	162	69.90
1515	FCT22ACALD	7.20E+00	< 3.01E+00	< 2.34E+00	< 3.52E+00	< 4.35E+00	< 4.69E+00	166	92.70
1518	FCT22ALALD	5.97E+00	< 2.90E+00	< 2.26E+00	< 3.39E+00	< 4.19E+00	< 4.52E+00	166	92.70
1522	FCT25ADALD	7.04E-01	< 2.59E-01	< 2.01E-01	< 3.02E-01	6.47E-01	2.59E+00	160	58.50
1524	FCT25ALALD	2.46E+00	< 3.41E-01	< 2.65E-01	8.71E-01	7.00E-01	4.35E+00	160	58.50
1602	FKA13ACALD	1.46E+00	< 8.75E-01	< 8.17E-01	1.05E+00	6.42E-01	1.34E+00	272	126.15
1603	FKA13ADALD	1.28E+00	1.07E+00	< 7.47E-01	< 5.87E-01	1.07E+00	2.45E+00	272	126.15
1605	FKA13ALALD	1.80E+00	1.22E+00	< 6.81E-01	< 5.35E-01	7.78E-01	1.94E+00	272	126.15
1606	FKA14ACALD	1.53E+00	8.16E-01	< 4.97E-01	6.74E-01	4.97E-01	1.17E+00	118	102.90
1607	FKA14ADALD	8.51E-01	6.39E-01	< 4.97E-01	< 3.90E-01	< 3.19E-01	< 6.39E-01	118	102.90
1609	FKA14ALALD	1.06E+00	< 6.03E-01	< 4.97E-01	5.68E-01	3.90E-01	< 6.39E-01	118	102.90
1611	FKA21ACALD	1.86E+00	< 5.61E-01	< 4.91E-01	< 3.86E-01	3.51E-01	8.77E-01	178	160.05
1612	FKA21ADALD	1.80E+00	6.83E-01	< 4.35E-01	< 3.42E-01	5.28E-01	1.21E+00	178	160.05
1614	FKA21ALALD	1.42E+00	< 5.30E-01	< 4.64E-01	< 3.64E-01	< 2.98E+00	7.95E-01	178	160.05
1615	FKA22ACALD	1.90E+00	5.99E-01	< 3.00E-01	5.35E-01	4.07E-01	8.13E-01	124	137.10
1616	FKA22ADALD	8.44E-01	4.67E-01	< 3.11E-01	< 2.44E-01	< 2.00E-01	< 4.00E-01	124	137.10
1618	FKA22ALALD	7.01E-01	4.07E-01	< 3.17E-01	< 2.49E-01	< 2.04E-01	< 4.07E-01	124	137.10
1624	FKA25ALALD	1.73E+00	< 4.71E-01	< 4.39E-01	< 3.45E-01	5.02E-01	1.41E+00	132	142.10
1702	FKT13ACALD	3.38E+00	< 2.23E-01	< 3.91E-01	< 3.07E-01	7.25E-01	1.45E+00	331	177.60
1703	FKT13ADALD	< 5.61E-01	< 8.97E-01	< 7.85E-01	< 6.17E-01	7.29E-01	< 1.01E+00	331	177.60

CONCENTRATION (TOTCONC) OF ALDEHYDES (UG/M3)

LABNO	FLDCODE	ACETAL (UG/M3)	ACROLEIN (UG/M3)	CROTONAL (UG/M3)	BUTYRAL (UG/M3)	BENZAL (UG/M3)	HEXANAL (UG/M3)	TOTCAL (NO.)	TOTMASS (KG)
1704	PKT13AGALD	6.11E+00	1.23E+00	< 7.85E-01	< 6.17E-01	1.23E+00	1.63E+00	331	177.60
1705	PKT13ALALD	3.24E+00	< 5.12E-01	< 3.98E-01	< 3.13E-01	7.67E-01	1.19E+00	331	177.60
1706	PKT14ACALD	2.81E+00	4.77E-01	< 3.34E-01	< 2.62E-01	7.63E-01	1.36E+00	577	155.55
1707	PKT14ADALD	3.82E+00	7.91E-01	< 3.26E-01	2.79E-01	8.15E-01	1.16E+00	577	155.55

CONCENTRATION (FIRECONC) OF ALDEHYDES (UG/M3)

LABNO	FLDCODE	ACETAL (UG/M3)	ACROLEIN (UG/M3)	CROTONAL (UG/M3)	BUTYRAL (UG/M3)	BENZAL (UG/M3)	HEXANAL (UG/M3)	TOTCAL (NO.)	TOTMASS (KG)
1102	PSM13ACALD	< 7.77E-01	< 1.00E+00	< 7.77E-01	< 2.05E+00	< 1.67E+00	< 1.28E+00	16	192.00
1103	PSM13ADALD	1.09E+00	< 1.09E+00	< 8.44E-01	< 2.23E+00	< 1.81E+00	< 1.39E+00	16	192.00
1104	PSM13ALALD	< 7.46E-01	< 9.60E-01	< 7.46E-01	< 1.97E+00	< 1.60E+00	< 1.23E+00	16	192.00
1106	PSM14ACALD	< 8.26E-01	< 1.06E+00	< 8.26E-01	< 2.18E+00	< 1.77E+00	< 1.36E+00	16	192.00
1107	PSM14ADALD	5.26E+00	< 1.25E+00	< 9.70E-01	< 2.56E+00	< 2.08E+00	< 1.59E+00	16	192.00
1108	PSM14ALALD	< 8.81E-01	< 1.13E+00	< 8.81E-01	< 2.33E+00	< 1.89E+00	< 1.45E+00	16	192.00
1110	PSM21ACALD	< 6.74E-01	< 8.66E-01	< 6.74E-01	< 1.78E+00	< 1.44E+00	< 1.11E+00	16	192.00
1111	PSM21ADALD	< 7.53E-01	< 9.68E-01	< 7.53E-01	< 1.99E+00	< 1.61E+00	< 1.24E+00	16	192.00
1112	PSM21ALALD	< 6.05E-01	< 7.77E-01	< 6.05E-01	< 1.60E+00	< 1.30E+00	< 9.93E-01	16	192.00
1114	PSM22ACALD	1.23E+00	< 9.24E-01	< 7.19E-01	< 1.90E+00	< 1.54E+00	< 1.18E+00	18	216.00
1115	PSM22ADALD	8.43E-01	< 9.48E-01	< 7.37E-01	< 1.95E+00	< 1.58E+00	< 1.21E+00	18	216.00
1116	PSM22ALALD	3.01E+00	< 7.53E-01	< 5.85E-01	< 1.55E+00	< 1.25E+00	< 9.62E-01	18	216.00
1202	FSM13ACALD	5.19E+00	< 4.19E-01	< 7.33E-01	< 8.91E-01	< 1.15E+00	< 9.95E-01	12	144.00
1203	FSM13ADALD	3.46E+01	< 4.19E-01	< 7.33E-01	< 8.91E-01	< 1.15E+00	< 9.95E-01	12	144.00
1205	FSM13ALALD	1.13E+01	< 4.31E-01	< 7.54E-01	< 9.16E-01	< 1.19E+00	< 1.02E+00	12	144.00
1206	FSM14ACALD	1.48E+01	< 9.11E-01	< 1.60E+00	< 1.94E+00	< 2.51E+00	< 2.16E+00	16	192.00
1207	FSM14ADALD	2.37E+00	< 1.00E+00	< 1.75E+00	< 2.12E+00	< 2.75E+00	< 2.37E+00	16	192.00
1209	FSM14ALALD	3.12E+01	< 8.91E-01	< 1.56E+00	< 1.89E+00	< 3.79E+00	< 2.12E+00	16	192.00
1211	FSM21ACALD	1.47E+01	< 9.79E-01	< 1.71E+00	< 2.08E+00	< 2.69E+00	< 2.32E+00	10	120.00
1212	FSM21ADALD	1.18E+01	< 1.05E+00	< 1.83E+00	< 2.22E+00	< 2.88E+00	< 2.48E+00	10	120.00
1214	FSM21ALALD	8.49E+00	< 1.10E+00	< 1.92E+00	< 2.33E+00	< 3.01E+00	< 2.60E+00	10	120.00
1215	FSM22ACALD	< 1.50E+00	1.80E+00	< 2.10E+00	< 2.55E+00	< 3.30E+00	< 2.85E+00	8	96.00
1216	FSM22ADALD	5.20E+00	1.85E+00	< 2.35E+00	< 2.85E+00	< 3.69E+00	< 3.19E+00	8	96.00
1218	FSM22ALALD	3.02E+00	< 1.10E+00	< 1.92E+00	< 2.34E+00	< 3.02E+00	< 2.61E+00	8	96.00
1221	FSM25ACALD	1.72E+01	< 1.25E+00	< 2.18E+00	< 2.65E+00	< 3.43E+00	< 2.97E+00	8	96.00
1222	FSM25ADALD	< 1.53E+00	< 1.22E+00	< 2.14E+00	< 2.59E+00	< 3.36E+00	< 2.90E+00	8	96.00
1224	FSM25ALALD	4.24E+02	< 1.26E+00	4.55E+00	< 2.67E+00	3.77E+00	< 2.98E+00	8	96.00
1302	FBB13ACALD	< 2.09E+00	< 1.67E+00	< 2.93E+00	< 3.56E+00	< 4.60E+00	< 3.97E+00	239	5.08
1303	FBB13ADALD	3.62E+01	< 1.71E+00	< 2.99E+00	7.68E+01	< 4.69E+00	< 4.05E+00	239	5.08
1305	FBB13ALALD	1.82E+01	< 1.73E+00	< 3.03E+00	< 3.67E+00	< 4.75E+00	< 4.11E+00	239	5.08
1306	FBB14ACALD	4.93E+01	< 1.88E+00	< 3.29E+00	6.34E+01	6.10E+00	4.69E+00	424	10.00
1307	FBB14ADALD	3.73E+01	< 1.66E+00	< 2.90E+00	7.68E+01	< 4.56E+00	< 3.94E+00	424	10.00
1309	FBB14ALALD	1.77E+01	< 1.63E+00	< 2.85E+00	< 3.46E+00	< 4.47E+00	< 3.86E+00	424	10.00
1311	FBB21ACALD	2.55E+01	< 1.20E+00	< 2.10E+00	1.95E+02	4.65E+00	3.00E+00	467	12.75
1312	FBB21ADALD	1.63E+00	< 1.19E+00	< 2.08E+00	< 2.52E+00	< 3.26E+00	< 2.82E+00	467	12.75
1314	FBB21ALALD	7.17E+00	< 1.15E+00	< 2.01E+00	< 2.44E+00	4.02E+00	< 2.72E+00	467	12.75
1315	FBB22ACALD	5.98E+00	< 1.09E+00	< 1.90E+00	< 2.31E+00	< 2.99E+00	< 2.58E+00	463	12.35
1316	FBB22ADALD	< 1.45E+00	< 1.16E+00	< 2.03E+00	< 2.47E+00	< 3.19E+00	< 2.76E+00	463	12.35

CONCENTRATION (FIRECONC) OF ALDEHYDES (UG/M3)

LABNO	FLDCODE	ACETAL (UG/M3)	ACROLEIN (UG/M3)	CROTONAL (UG/M3)	BUTYRAL (UG/M3)	BENZAL (UG/M3)	HEXANAL (UG/M3)	TOTCAL (NO.)	TOTMASS (KG)
1318	FBB22ALALD	< 1.46E+00	< 1.17E+00	< 2.04E+00	1.08E+01	< 3.20E+00	< 2.77E+00	463	12.35
1321	FBB25ACALD	4.15E+00	< 1.07E+00	< 1.87E+00	< 2.27E+00	< 2.94E+00	< 2.54E+00	382	11.53
1322	FBB25ADALD	< 1.32E+00	< 1.06E+00	< 1.85E+00	< 2.24E+00	< 2.90E+00	< 2.51E+00	382	11.53
1324	FBB25ALALD	7.69E+00	< 1.06E+00	< 1.86E+00	< 2.25E+00	< 2.92E+00	< 2.52E+00	382	11.53
1502	FCT13ACALD	2.27E+01	< 1.10E+01	< 8.59E+00	< 1.29E+01	< 1.59E+01	2.39E+01	160	58.50
1503	FCT13ADALD	3.79E+01	< 1.00E+01	< 7.80E+00	< 1.17E+01	< 1.45E+01	1.62E+01	160	58.50
1505	FCT13ALALD	3.08E+01	< 9.90E+00	< 7.70E+00	< 1.16E+01	< 1.43E+01	1.76E+01	160	58.50
1506	FCT14ACALD	4.54E+01	< 1.10E+01	< 8.59E+00	< 1.29E+01	< 1.59E+01	5.70E+01	163	75.60
1507	FCT14ADALD	9.99E+01	< 1.06E+01	< 8.22E+00	< 1.23E+01	< 1.53E+01	6.46E+01	163	75.60
1509	FCT14ALALD	5.60E+01	< 1.01E+01	< 7.83E+00	2.41E+01	< 1.45E+01	6.71E+01	163	75.60
1511	FCT21ACALD	2.25E+01	< 1.04E+01	< 8.09E+00	< 1.21E+01	< 1.50E+01	< 1.62E+01	162	69.90
1512	FCT21ADALD	3.42E+01	< 1.14E+01	< 8.86E+00	< 1.33E+01	< 1.65E+01	< 1.77E+01	162	69.90
1514	FCT21ALALD	3.81E+01	< 1.04E+01	< 8.09E+00	< 1.21E+01	< 1.50E+01	< 1.62E+01	162	69.90
1515	FCT22ACALD	2.34E+01	< 9.79E+00	< 7.62E+00	< 1.14E+01	< 1.41E+01	< 1.52E+01	166	92.70
1518	FCT22ALALD	1.94E+01	< 9.44E+00	< 7.34E+00	< 1.10E+01	< 1.36E+01	< 1.47E+01	166	92.70
1522	FCT25ADALD	9.16E+00	< 3.36E+00	< 2.62E+00	< 3.92E+00	8.41E+00	3.36E+01	160	58.50
1524	FCT25ALALD	3.20E+01	< 4.43E+00	< 3.45E+00	1.13E+01	9.11E+00	5.66E+01	160	58.50
1602	FKA13ACALD	2.00E+00	< 1.20E+00	< 1.12E+00	1.44E+00	8.81E-01	1.84E+00	272	126.15
1603	FKA13ADALD	1.76E+00	1.47E+00	< 1.03E+00	< 8.06E-01	1.47E+00	3.37E+00	272	126.15
1605	FKA13ALALD	2.47E+00	1.67E+00	< 9.35E-01	< 7.34E-01	1.07E+00	2.67E+00	272	126.15
1606	FKA14ACALD	2.28E+00	1.22E+00	< 7.43E-01	1.01E+00	7.43E-01	1.75E+00	118	102.90
1607	FKA14ADALD	1.27E+00	9.55E-01	< 7.43E-01	< 5.84E-01	< 4.78E-01	< 9.55E-01	118	102.90
1609	FKA14ALALD	1.59E+00	< 9.02E-01	< 7.43E-01	8.49E-01	5.84E-01	< 9.55E-01	118	102.90
1611	FKA21ACALD	2.63E+00	< 7.94E-01	< 6.94E-01	< 5.46E-01	4.96E-01	1.24E+00	178	160.05
1612	FKA21ADALD	2.55E+00	9.66E-01	< 6.15E-01	< 4.83E-01	7.47E-01	1.71E+00	178	160.05
1614	FKA21ALALD	2.01E+00	< 7.49E-01	< 6.56E-01	< 5.15E-01	< 4.22E+00	1.12E+00	178	160.05
1615	FKA22ACALD	2.71E+00	8.53E-01	< 4.27E-01	7.62E-01	5.79E-01	1.16E+00	124	137.10
1616	FKA22ADALD	1.27E+00	7.00E-01	< 4.67E-01	< 3.67E-01	< 3.00E-01	< 6.00E-01	124	137.10
1618	FKA22ALALD	9.99E-01	5.80E-01	< 4.51E-01	< 3.54E-01	< 2.90E-01	< 5.80E-01	124	137.10
1624	FKA25ALALD	1.83E+00	< 5.00E-01	< 4.67E-01	< 3.67E-01	5.33E-01	1.50E+00	132	142.10
1702	PKT13ACALD	4.28E+00	< 2.83E-01	< 4.95E-01	< 3.89E-01	9.19E-01	1.84E+00	331	177.60
1703	PKT13ADALD	< 7.11E-01	< 1.14E+00	< 9.95E-01	< 7.82E-01	9.24E-01	< 1.28E+00	331	177.60
1705	PKT13ALALD	4.05E+00	< 6.39E-01	< 4.97E-01	< 3.91E-01	9.59E-01	1.49E+00	331	177.60
1706	PKT14ACALD	3.59E+00	6.09E-01	< 4.26E-01	< 3.35E-01	9.74E-01	1.73E+00	577	155.55
1707	PKT14ADALD	4.94E+00	1.02E+00	< 4.22E-01	3.61E-01	1.05E+00	1.51E+00	577	155.55

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