PB-297 783

# NIOSH MANUAL OF SAMPLING DATA SHEETS

## Supplement to the 1977 Edition

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August 1978

## DISCLAIMER

**DHEW (NIOSH) Publication No. 78-189**

**BIBLIOGRAPHIC DATA SHEET**

**1. Report No.** NIOSH 78-189

**2.**

**3. Recipient's Accession No.** PB297783

**4. Title and Subtitle**
NIOSH Manual for Sampling Data Sheets - Supplement to 1977 Edition

**5. Report Date** August 1978

**6.**

**7. Author(s)**
William A. Heitbrink

**8. Performing Organization Rept. No.** NIOSH 78-189

**9. Performing Organization Name and Address**
National Institute for Occupational Safety and Health
4676 Columbia Parkway
Cincinnati, Ohio 45226

**10. Project/Task/Work Unit No.**

**11. Contract/Grant No.**

**12. Sponsoring Organization Name and Address**
SAME

**13. Type of Report & Period Covered**

**14.**

**15. Supplementary Notes**

**16. Abstracts**

The supplement to the NIOSH Manual of Sampling Data Sheets is a collection of 46 Sampling Data Sheets (SDS's). Each SDS contains recommended sampling methodology for measuring occupational exposure to air contaminants. The SDS's are graded according to the quality and extent of supporting data.

**17. Key Words and Document Analysis. 17a. Descriptors**

**17b. Identifiers/Open-Ended Terms**

**17c. COSATI Field/Group**

**18. Availability Statement**

**19. Security Class (This Report)** UNCLASSIFIED

**20. Security Class (This Page** UNCLASSIFIED

**21.** ages

**22. Price** PCA07/MF A01

FORM NTIS-35 (REV. 10-73) ENDORSED BY ANSI AND UNESCO. THIS FORM MAY BE REPRODUCED USCOMM-DC 8265-P74

# PREFACE

This supplement is a continuation of the 1977 edition of the NIOSH Manual of Sampling Data Sheets.[1] This publication presents sampling methods developed since the publication of this manual. In addition, it contains errata for the 1977 edition of the NIOSH Manual of Sampling Data Sheets.

The Sampling Data Sheets (SDS's) are written as guides for sampling worker exposure to toxic air contaminants. Each data sheet is graded according to the quality and extent of supporting data. Details of the grading scheme are provided in the 1977 edition of the manual.

In the Contents, each SDS is identified as an SDS number. Starting with this supplement, the SDS number will have the same number as the corresponding sampling and analytical method described in the NIOSH Manual of Analytical Methods.[2,3] However, a few SDS's will not have corresponding sampling and analytical methods. Many SDS numbers are prefixed by the letter "S." This identifies SDS's that were produced as part of a follow-up to the Standards Completion Program. After the Contents, the SDS's appear in numerical order. The SDS's prefixed by the letter "S" follow the other SDS's.

Because an update of the Manual of Sampling Data Sheets is being planned, comments on the use and content of this manual are welcomed and encouraged. Such comments may be addressed to the Coordinator, NIOSH Manual of Sampling Data Sheets. For those wishing to make comments, a franked, pre-addressed envelope is attached to this publication. The mailing address for the Coordinator is:

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[1] NIOSH Manual of Sampling Data Sheets. 1977. DHEW (NIOSH) Publication No. 77-159. NIOSH, Cincinnati, Ohio. **(Not available from NIOSH. Order from NTIS or GP**

[2] NIOSH Manual of Analytical Methods. 1977. Volume 1 - DHEW (NIOSH) Publication No. 77-157A; Volume 2 - DHEW (NIOSH) Publication No. 77-157B; Volume 3 - DHEW (NIOSH) Publication No. 77-157C. NIOSH, Cincinnati, Ohio.

[3] Taylor, D. 1978. Volume 4 - NIOSH Manual of Analytical Methods - DHEW (NIOSH) Publication No. 78-1. NIOSH, Cincinnati, Ohio.

# ABSTRACT

This supplement to the NIOSH Manual of Sampling Data Sheets is a collection of 46 Sampling Data Sheets (SDS's). Each SDS contains recommended sampling methodology for measuring occupational exposure to air contaminants. The SDS's are graded according to the quality and extent of supporting data.

# CONTENTS

Sampling Data Sheets

# ACKNOWLEDGMENTS

Dr. Laurence J. Doemeny and Mr. William A. Heitbrink, Chief and Industrial Hygiene Engineer, respectively, of the Measurements Systems Section, Measurements Research Branch, are responsible for the development and content of this manual. Dr. Samuel P. Tucker, Dr. Donald Dollberg, Mr. John Palassis, Mr. Charles S. McCammon, and Dr. Denis L. Foerst prepared individual Sampling Data Sheets. The SDS's whose numbers are prefixed by the letter "S" were produced by SRI International and A.D. Little, Inc. under NIOSH Contract CDC 210-76-0123. Special thanks is due Ms. Grace A. Fannin for her patience and accuracy in the typing preparation of this manual.

# ERRATA TO 1977 EDITION OF THE NIOSH MANUAL OF SAMPLING DATA SHEETS

1. Page ix and xiv. The correct substance for Sampling Data Sheet S113 is chlorobromomethane instead of chlorobromoethane.

2. Page xvii. Under Silica, Crystalline add, "3.02" before S315.

3. Page 18-1. Under "Shipping" delete the second to last sentence about plastic caps supplied by NIOSH.

4. Page S73-1. The inside diameter of the silica gel tube is unlisted. This dimension is 4 mm. These tubes contain 150 mg of silica gel in the front section and 75 mg of silica gel in the backup section.

5. Page S135-1. The correct standard for o-Dichlorobenzene is a ceiling of 50 ppm (300 mg/cu m). The correct sample volume is 3 liters. Also, the word "class" is misspelled.

6. Page S155-1. The inside diameter of the charcoal tube is 4 mm. The front section contains 100 mg of charcoal and the backup section contains 50 mg.

7. Page S156-2. Replace the words "silica gel" with the word "charcoal" throughout the page.

# ALPHABETICAL LISTING OF
# SAMPLING DATA SHEETS

# NIOSH SAMPLING DATA SHEET #41.01

March 9, 1977 Class D

## Substance

Mercury Vapor

## Standard

8-Hour Time Weighted Average: 0.1 mg/m$^3$

## Analytical Method

When the monitor is exposed to contaminated air, mercury vapor diffuses through the monitor's barrier film and is adsorbed onto a gold film. The electrical conductivity of the film is measured before and after exposure. The change in conductivity is directly proportional to the mercury concentration.

## Sampling Equipment

A 3M Company Mercury Vapor Monitoring System is used to collect mercury vapor. The monitor, which resembles a small plastic badge, essentially consists of a gold foil covered by a barrier film. The monitor comes in a shipping bag which is used to return the monitor for analysis.

## Sampling Procedure

1. Remove the monitor from the supplied bag. Retain this bag for shipment.

2. Using either the monitor's pin or clip, place the monitor on the worker's garment in the breathing zone.

3. Record the date, monitor number, ambient temperature and pressure, time that the monitor's bag was opened, time that the monitor was sealed in the bag, and the employee's name or ID. Record this information on the bag supplied by 3M Company. If the ambient pressure is unavailable, record the elevation.

4. After sampling for eight hours, remove the monitor from the worker and reseal the monitor in its original bag. Ship the monitor to the manufacturer per his directions.

5. Return one monitor as a blank for every 20 to 30 samples. The blanks are never removed from their bags.

Special Considerations

1. The only tested interference is chlorine gas. Chlorine gas concentrations in excess of 5 ppm cause appreciable loss of mercury. Chlorine gas concentrations below 1 ppm have been shown not to affect the monitor. Other halides may interfere. The badge is not recommended for use in atmospheres containing strong oxidizers.

2. Because the amount of mercury adsorbed on the gold film is proportional to the diffusivity of mercury in air, the results are affected by ambient temperature and pressure. Consequently, this data must be recorded. The manufacturer should multiply the observed concentration by this factor:

$$\frac{T_a^{3/2}}{T_c^{3/2}} \qquad \frac{P_c}{P_a}$$

where,

$T_a$ = ambient absolute temperature

$T_c$ = absolute temperature during calibration

$P_a$ = ambient atmospheric pressure

$P_c$ = ambient atmospheric pressure during calibration

3. This Sampling Data Sheet describes a service of the 3M Company and is not to be construed as an endorsement of this service. This Sampling Data Sheet is merely one of serveral methods available for measuring worker exposure to elemental mercury.

User quality control for this analysis is based on the user's ability to expose badges to known concentrations of a mercury vapor.

## Shipping Instructions

Follow manufacturer's directions.

NIOSH SAMPLING DATA SHEET #225.02    Class D

January 27, 1976

Substance:

Kepone

Standards:

A standard has not been established.

Analytical Method:

Because Kepone may exist as an aerosol and a vapor, first the aerosols are collected on a filter. Then the vapors which penetrate the filter are collected in an impinger containing 0.1N sodium hydroxide. The collected material is extracted, separated by gas chromatogrphy, and the Kepone is detected by electron capture.

Interference:

The pesticide Mirex $^R$ is converted to Kepone in a 0.1N sodium hydroxide solution. The presence of this contaminant and the presence and identities of other suspected contaminants should be reported to the analytical chemist.

Sampling Equipment:

Pump: A calibrated personal sampling pump whose flow rate can be determined accurately, ± 5%, at the recommended flow rate. The pump must be calibrated with representative sampling train in line.

Filter Holder: Three-piece, 37-mm diameter filter holder. The filter holder must be held together by stretchable tape or shrinkable band.

Filter: 37-mm diameter, 0.8 micrometer pore size mixed cellulose ester membrane filter. The filter is mounted on a support pad.

Impinger: A midget impinger which contains 15 ml of 0.1N sodium hydroxide.

Trap: A midget impinger with a broken stem.

Glass Vial: A vial with a Teflon-lined, threaded cap.

Sample Size:

Sample for at least 2 hours at a flow rate of 1.0 lpm. Longer sampling times up to a full shift are recommended.

Sampling Procedure:

1. Assemble filter, support pad, and filter holder. Be sure the filter holder's center ring seals the edge of the filter. Examine the filter holder for a good seal. If the filter holder does not seal tightly, discard it.

2. Remove the plugs from the filter holder. A short piece of tubing is used to connect the outlet of the filter to the impinger's inlet. An adaptor may be needed to provide a tight connection between the tubing and filter holder. The impinger's outlet is connected to the pump or the pump's trap by tubing. During personal sampling, spillage of impinger solution occurs. To protect the pump, the spilled solution can be collected in a trap. The trap is placed in a holder that is attached to the pump. Connect the outlet of the trap to the pump's inlet with a short piece of tubing.

3. If personal sampling is being done, place midget impinger in its holder. Attach the holder to the worker's clothing in his breathing zone.

4. Turn the pump on and set the flow rate to 1.0 lpm according to the manufacturer's directions. Periodically check the flow rate and readjust as needed. Terminate sampling if you are unable to readjust. In addition, check the level of solution in the impinger. When less than 5 ml remain, add enough 0.1N sodium hydroxide to bring the level up to the 15 ml mark.

5. Record the ambient pressure and temperature. If the ambient pressure is unavailable, record the elevation.

6. After sampling, transfer solution in the impinger and trap to separate glass vials. Wash the impinger stem with 1-2 mg of unused distilled water and add the wash to the vial. Tightly screw on the cap and seal with vinyl electrical tape or shrinkable band.

7. Transfer the filter and support pad to another glass vial. Tightly screw the cap on and seal with vinyl electrical tape.

8. Blank: With each batch of samples, submit one filter and impinger to the same handling as the other samples except that no air is drawn through it. Label this as a blank.

Special Considerations:

Never return spilled solution collected in the trap to the impinger. Always ship the two liquids in separate vials.

Shipping Instructions:

The filter vials should be shipped in a container that prevents damage during shipment. Never ship filters and solution vials in the same container.

Sampling Data Sheet #250.01 Class D

Substance:

Respirable Zirconium Oxide

Standard:

No standard for respirable zirconium oxide

Analytical Method:

Airborne dust is collected on polyvinyl chloride filters, ashed in low-temperature asher, re-deposited on silver membrane filters and analyzed for zirconium oxide by X-ray powder diffractometry. The analytical range of the method is 0.5--15 mg/cu m.

Sampling Equipment:

1. Filter: polyvinyl membrane filters, 37-mm, 5-μm pore size. (Gelman VM-1 filters have been found to produce a high background in the ash). The filter is mounted on a cellulose back-up pad.
2. Filter Holder: A 2-piece, 37-mm filter holder held together by a shrinkable band.
3. Pump: A calibrated personal sample pump whose flow rate can be determined to an accuracy of ±5% at the recommended sampling rate of 1.7L/minute.
4. Cyclone: 10-mm nylon cyclone is used to separate respirable from non-respirable dust. Its outlet is connected to the filter holder inlet.

Sample Size:

1. Sample for one hour at a rate of 1.7 L/minute to obtain a 90L sample.
2. A maximum of 3.0 mg and a minimum of 0.2 mg of total particulate should be collected.

Sampling Procedure:

1. Assemble the filter, back-up pad, and two-piece filter cassette and close firmly to insure that the face cap seals against the filter. Examine the holder for a good filter seal. If the cassette will not seal tightly, it should be discarded. This filter holder should be held together by tape or a shrinking band.
2. Assemble the filter holder, cyclone, and sampling head. The sampling head rigidly holds together the cyclone and filter holder. The air outlet of this assembly is connected to the pump by a flexible piece of tubing.

3. Clip the sampling head to the worker's lapel.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record the ambient temperature and pressure. If ambient pressure is not available, record the elevation. If the personal sampler pump is used, position the middle of the rotameter ball to the proper calibration mark as accurately as possible. Since it is possible for the filter to become plugged by heavy particulate loading or by the presence of oil mists in the air, the pump flow rate should be checked and readjusted as needed. If unable to readjust, terminate sampling.

5. After sampling, place small plugs back in cassette's inlet and outlet.

6. Blank: With each batch of samples submit one filter which is subjected to exactly the same handling as for the samples, except that no air is drawn through it. Label this as a blank.

Special Consideration:

1. The 10-mm nylon cyclone interior must be inspected before use. The cyclone must be cleaned before use to prevent reentrainment of collected dust. If the interior of the cyclone is visibly scored, the cyclone should be discarded since its size-selective characteristics may be changed.

2. The cyclone is used to eliminate particles larger than 10 micrometers, because the precision of the analytical method is adversely affected by larger particles.

References:

1. Dollberg, D. 1978. P&CAM Method Number 250: Zirconium Oxide in Air. NIOSH, Measurements Support Branch, 4676 Columbia Parkway, Cincinnati, Ohio.

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## NIOSH SAMPLING DATA SHEET #266.01 Class D

December 7, 1977

Substance:

Vinylidene Chloride (1,1-dichloroethene)

Standard:

As of the date of this Sampling Data Sheet there is no standard for vinylidene chloride published by OSHA in 29 CFR 1910 nor is there a published NIOSH recommended standard for occupational exposure to vinylidene chloride. The American Conference of Governmental Industrial Hygienists has recommended an eight hour time-weighted average of 40 $mg/m^3$ for occupational exposure to vinylidene chloride.[1]

Analytical Method:

A known volume of air is drawn through a charcoal tube to trap the vinylidene chloride vapor present. The analyte is desorbed from the charcoal with carbon disulfide, and an aliquot of the solution is analyzed using a gas chromatograph with a flame ionization detector. The front and rear sections of the charcoal tube are analyzed as individual samples.[2] The rear section is used as a breakthrough indicator.

The supporting analytical method has been evaluated for sample loadings between 7 and 84 μg per charcoal tube. The complete sampling and analytical method has been tested against air containing 10 $mg/m^3$ vinylidene chloride at 23°C and 85% relative humidity.[3]

Sampling Equipment:

1. Personal sampling pump, the flow of which can be determined accurately (±5%) at the recommended sampling flow rates. The pump is calibrated at the recommended sampling flow rates with a recommended sampling tube in line.

2. Charcoal tube, 4-mm inside diameter, containing two sections of 20/40 mesh charcoal. The first section contains 100 mg of activated charcoal. The second section contains 50 mg of activated charcoal. A plug of silanized glasswool is placed in front of the first section. A 2-mm urethane foam spacer separates the two sections. A 3-mm urethane foam spacer is placed between the second section and outlet end of the tube. All charcoal tubes to be used should come from the same batch.

3. Tubing, Tygon, for connecting the charcoal tube to the pump.

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Sample Size:

A maximum sample volume of 7 liters is recommended. Sample at a flow rate between 10 and 200 ml/min.

Sampling Procedure:

1. Immediately before sampling, the ends of a tube should be broken so as to provide an opening about one-half the internal diameter of the tube.

2. The charcoal tube is positioned in the tube holder with the smaller backup section of charcoal being positioned nearest the pump. The tube should be in a vertical position during sampling to avoid channeling and subsequent premature breakthrough of vinylidene chloride.

3. Air being sampled should not be passed through any hose or tubing before entering the charcoal tube.

4. Set the flow rate on the predetermined setting. Record the temperature and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. If a low flow rate pump is used, set the flow rate and record the initial and final counter reading. The sample volume is obtained by multiplying the number of counter strokes by the cc/stroke factor.

5. The charcoal tubes should be capped with the supplied plastic caps immediately after sampling. Under no circumstances should rubber caps be used.

6. One charcoal tube (at least one) for every 10 samples should be handled in the same manner as the sample tubes except that no sample is taken. The ends are broken and the tube is immediately capped and prepared for shipping. This tube is labeled as a blank.

7. The desorption efficiency of charcoal can vary from batch to batch. Therefore, all the tubes used to collect a set of samples should contain charcoal from the same batch. At least six unused, unopened charcoal tubes should be sent to the analyst for desorption efficiency studies.

Special Considerations:

1. Where two or more compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.

2. The pumps should not be operated more than 8 hours continuously without fully recharging the battery.

3. High humidity can cause a reduction in breakthrough volume. However, this has been taken into account and as long as a maximum of 7 liters is sampled, no breakthrough should occur. If the charcoal becomes coated with water, the containinants will not be trapped quantitatively.

Bulk Samples:

If samples of material containing vinylidene chloride are shipped to the lab, they should be submitted in a glass container with a Teflon-lined cap. Do not transport these samples in the same container with charcoal tubes. CAUTION: Because vinylidene chloride has a low flash point, the shipment of bulk samples must comply with applicable DOT regulations concerning transportation of hazardous materials (49 CFR 100-199).

Shipping Instructions:

Capped charcoal tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping. Never transport, mail, or ship the bulk sample in the same container as the sample or blank tubes. When the samples are received by the laboratory, they should be stored under refrigeration to avoid any chance of loss of vinylidene chloride from the samples.

References:

1. American Conference of Governmental Industrial Hygienists. 1976. Threshold Limit Values for Chemical Substances and Physical Agents in the Work Environment with Intended Changes for 1976. Cincinnati, OH.

2. Foerst, D. 1977. Vinylidene Chloride in Air: P&CAM #266. Measurements Research Branch, NIOSH, Cincinnati, OH.

3. Foerst, D. 1977. A Sampling and Analytical Method for Vinylidene Chloride. (In preparation) Measurements Research Branch, NIOSH, Cincinnati, OH.

Sampling Data Sheet #281.01 Class D

June 21, 1978

Substance:

Ethylene thiourea

NIOSH Recommended Standard:

No standard has been established

Synopsis:

Ethylene thiourea is collected from air on a PVC or cellulose ester membrane filter. The filter is extracted with distilled water. Pentacyanoamminеferrate reagent is added to the extract to form a colored coordination complex. The absorbance of the solution is measured spectrophotometrically at 590 nm, and the concentration of ethylene thiourea is determined from a calibration curve. For a 100-L sample, the working range of the method is 0.03-1.5 $mg/m^3$. Reference 1 completely describes the sampling and analytical method.

Sampling Equipment:

1. Filters: PVC 5 μm in pore size, 37-mm in diameter or mixed cellulose ester filter 0.8 μm in pore size, 37-mm in diameter.
2. Filter Holders: Plastic three piece 37-mm holders (cassettes).
3. Pump: Use a personal sample pump whose flow rate can be determined accurately, ±5%, at the sampling rate of 1.5 liters per minute (Lpm).
4. Other Equipment: Flexible PVC tubing, filter back-up pads, shrinking bands or masking tape.

Sample Size:

Collect a sample of 200 L or more at a rate of 1.5 Lpm. Do not allow filter loading to exceed 2 milligrams per filter.

Sampling Procedure:

1. Assemble the filters in the filter holder so that the air being sampled passes first through the filter and then through the filter support. Close firmly to insure that the face cap seals against the filter. Examine the holder for a good filter seal. If the cassette will not seal tightly, it should be discarded. This filter holder should be held together by tape or a shrinking band. Remove the small plugs from the filter holder and connect the filter holder to the sampling pump by means of an adapter and a length of flexible PVC tubing.
2. Air being sampled should not be passed through any hose or tubing before entering the filter holder.

3. Clip the filter holder to the worker's lapel.

4. Set the flow rate at 1.5 Lpm as accurately as possible using the manufacturer's directions. Record the ambient temperature and pressure. If ambient pressure is not available, record the elevation. If the personal sampler pump is used, position the middle of the rotameter ball to the proper calibration mark as accurately as possible. Since it is possible for the filter to become plugged by heavy particulate loading or by the presence of oil mists in the air, the pump flow rate should be checked and readjusted as needed. If unable to readjust, terminate sampling.

5. After sampling, place small plugs back in cassette's inlet and outlet.

6. Blank: With each batch of 10 samples submit one filter which is subjected to exactly the same handling as for the samples, except that no air is drawn through it. Label this as a blank.

Special Considerations:

Where substances other than the analyte are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.

Bulk Samples:

A bulk sample of the suspected substance should be submitted to the laboratory in a glass container with a Teflon-lined cap. The label of the bulk sample should match air samples for identification purposes. The cap should be tightened and taped.

Shipping Instructions:

Use suitable containers designed to prevent damage of samples in transit. The bulk samples should be shipped separately from the filter samples or blanks.

Reference:

1. Palassis, J. 1978, NIOSH Method No. P&CAM 281: Ethylene Thiourea in Air. Measurement Support Branch, NIOSH, Cincinnati, Ohio.

## SAMPLING DATA SHEET 282.01 Class D

June 22, 1978

Substance:

Tetramethyl thiourea

NIOSH Recommended Standard:

No standard has been established

Synopsis:

Tetramethyl thiourea is collected from air in midget impingers containing 15 mL water. Pentacyanoammineferrate reagent is added to the sample to form a colored coordination complex. The absorbance of the solution is measured spectrophotometrically at 590 nm, and the concentration of tetramethyl thiourea is determined from a calibration curve. For a 100-L sample, the working range of the method is 0.15-1.5 mg/$m^3$. Reference 1 completely describes the sampling and analytical method.

Sampling Equipment:

1. Midget impingers: Filled with 15 mL distilled water. A second empty impinger may be used as a liquid trap to protect pump from splashover.
2. Pump: Use a personal sample pump whose flow rate can be determined accurately, ±5%, at the sampling rate of 2.8 liters per minute (Lpm).
3. Trap: A second empty bubbler or impinger is used to protect the pump from spillage.
4. Distilled water or deionized water.
5. Other Equipment: Flexible PVC tubing, "parafilm" or equivalent, Teflon stoppers.

Sample Size:

Collect a sample of 200 L or more at a rate of 2.8 Lpm.

Sampling Procedure:

1. Pour 15 mL of distilled water into the first impinger.
2. Assemble the sampling train. Put the first impinger in a suitable impinger holder. The outlet of this impinger is connected by tubing to the inlet of the trap. The outlet of the trap is connected by a short piece of tubing to the pump's inlet. The trap is in a suitable impinger holder which is attached to the pump. Liquid collected in the trap must never be returned to the first impinger.

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3. Air should not pass through any hose or tubing before entering the first impinger.

4. Set the flow rate at 2.8 Lpm as accurately as possible using the manufacturer's directions. Record all the necessary information to determine flow rate or volume and also record the initial and final sampling time. Record the temperature and pressure of the atmosphere being sampled. If pressure reading is not available, record the elevation.

5. After sampling, remove the impinger stem and tap the stem gently against the inside wall of the impinger bottle to recover as much of the sampling solution as possible. Rinse the impinger stem with 1-2 mL of distilled water into the midget impinger flask. Repeat this process for liquid collected in the trap. However, do not combine the two solutions in one impinger bottom. Be sure each impinger bottom is properly labeled. Seal the impinger with a hard, nonreactive stopper (preferably Teflon). Do not seal with rubber. The stoppers on the impingers should be tightly sealed to prevent leakage during shipping.

6. Attempt to minimize sample spillage. Do not allow the solution level in the first impinger to fall below 10 mL. Replace spilled solution with fresh distilled water. If spillage is not evidenced by liquid in the trap or in the tubing, evaporation has probably occurred. Use distilled water to bring the solution volume back up to 15 mL.

7. With each batch of ten samples submit one midget impinger containing 10 mL of distilled water from the same stock as that used for sample collection. This impinger must be subjected to exactly the same handling as the samples except that no air is drawn through it. Label this impinger as the blank.

Special Consideration:

1. When other air contaminants are known or suspected to be present, such information, including their suspected identities, should be transmitted with the sample.

2. If a significant amount of tetramethyl thiourea is found by the analyst in the trap or if less than 7 mL of solution remains in the first impinger, the sample should be considered invalid.

Bulk Samples:

A bulk sample of the suspected compound should be submitted to the laboratory in a glass container with a Teflon-lined cap. Use shrinkable bands or stretchable tape to keep the container tightly sealed. Label of the bulk sample should match air samples for identification purposes. Do not ship sample with the collected samples.

Shipping Instructions:

The impingers in which the samples are stored should be shipped in a suitable container designed to prevent damage or leakage in transit.

Reference:

1. Palassis, J. 1978. NIOSH Method No. P&CAM 282: Tetramethyl Thiourea. Measurement Support Branch, NIOSH, Cincinnati, Ohio.

Sampling Data Sheet No. S24

November 25, 1977

## Substance

Diphenyl (Biphenyl)

## Standard

8-hour time-weighted average: 0.2 ppm (1 mg/cu m)

## Analytical Method

A known volume of air is drawn through a tube containing 35/60 mesh Tenax GC to trap the biphenyl vapors present. The biphenyl is desorbed from the Tenax GC with carbon tetrachloride, and the sample is separated and analyzed using a gas chromatograph with a flame ionization detector. The method has been validated over the range of 0.642-2.35 mg/cu m for a 30-liter sample at 24.5°C and 760 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

Sampling equipment includes a calibrated personal sampling pump whose flow can be determined accurately (±5%) at a flow rate of 0.50 liter per minute. A tube (6.0 cm long with a 6-mm O.D. and a 4-mm I.D.), containing two sections of 35/60 mesh Tenax GC separated by a 2-mm portion of urethane foam, is used to collect the samples. The front section of the tube contains 20 mg of Tenax GC, and the backup section contains 10 mg. Immediately prior to packing, the tubes should be acetone rinsed and dried to eliminate the problem of Tenax GC adhering to the walls of the glass tubes. Prior to packing, the Tenax GC is prewashed in acetone. NIOSH Method S24 for Diphenyl describes the prewashing procedure.

## Sample Size

A sample size of 30 liters is recommended. Sample at a flow rate of 0.5 liter/minute or less. Do not sample at a flow rate less than 0.01 liter/minute.

## Sampling Procedure

1. Immediately before sampling, remove the caps from the ends of the tube. All tubes must contain Tenax GC from the same manufacturer's lot.

2 The smaller section of Tenax GC is used as a backup and should be positioned nearer the sampling pump. Air should flow through the larger front section before entering the smaller backup section.

The Tenax GC tube should be placed in a vertical position during sampling to avoid channeling and subsequent premature breakthrough of biphenyl.

3. Air being sampled should not be passed through any hose or tubing before entering the Tenax GC tube.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record the temperature, relative humidity, and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. Also report the type of sampling pump that is used.

5. The Tenax GC tube should be capped individually with plastic caps immediately after sampling. Masking tape is the only suitable substitute for sealing the tubes. Under no circumstances should rubber caps be used.

6. From the same lot of tubes used for sample collection, submit one blank Tenax GC tube and an additional blank tube for every ten samples. This tube must be subjected to exactly the same handling as the samples except that no air is drawn through it. Label this tube as a blank. Information on the batch number of the Tenax GC must be supplied. A minimum of 18 extra Tenax GC tubes should be provided for desorption efficiency determinations. This is necessary, because desorption efficiency may vary among different batches of Tenax GC and different laboratories.

## Special Considerations

1. When other compounds are known or suspected to be present in the air, such information, including their suspected identities should be transmitted with the sample.

2. Due to the high resistance of the Tenax GC tube, this sampling method places a heavy load on the sampling pump. Therefore, no more than eight hours of sampling should be done without first fully recharging the battery.

3. The volume recommended is based on high humidity breakthrough tests. Further reduction in sample volume due to high humidity should not be needed. If condensation of water occurs in the tube, the substance may not be trapped quantitatively.

## Bulk Samples

A bulk sample of the suspected compound should be submitted to the laboratory in a glass container with a Teflon-lined cap. Label of the bulk sample should match air samples for identification purposes.

## Shipping Instructions

Capped Tenax GC tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping. Never transport, mail, or ship the bulk sample in the same container as the sample or blank tube.

## Reference

Diphenyl, NIOSH Method No. S24.

# Sampling Data Sheet No. S57

## Substance

Hydroquinone

## Standard

8-hour time-weighted average: 2.0 mg/cu m

## Analytical Method

A known volume of air is drawn through a mixed cellulose ester membrane filter to trap the hydroquinone particulate present. The sample filters are stored in 1% acetic acid and the solution is analyzed by high pressure liquid chromatography using a variable wavelength UV detector set at 290 nm. The method has been validated over a concentration range of 0.84 - 4.1 mg/cu m at 20°C and 762 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

The following equipment is needed for sampling hydroquinone particulate: a calibrated personal sampling pump whose flow can be determined to an accuracy of ±5% at a flow rate of 1.5 liters per minute; a 37-mm three piece cassette filter holder held together by tape or shrinkable band; a 37-mm/0.8 micrometer mixed cellulose ester membrane filter (MCEF) supported by a cellulose backup pad; a 2 oz. ointment jar sealed with Teflon film and cap or any other suitable container capable of storing the cellulose filter and 10 ml of solution; a 1% acetic acid in distilled water solution; and a 10-ml graduate or any other device capable of adding roughly 10 ml of solution to the filter container.

## Sample Size

A sampling period of one hour is recommended. Sample at a flow rate of 1.5 liters per minute.

## Sampling Procedure

1. Assemble the filter and three-piece filter cassette and close firmly to insure that the center ring seals the edge of the filter. Examine the holder for a good filter seal. If the cassette will not seal tightly, it should be discarded. Secure the cassette holder together with tape or shrinkable band.

2. Remove the cassette plugs and attach to the personal sampling pump tubing. Clip the cassette to the worker's lapel.

3. Air being sampled should not be passed through any hose or tubing before entering the filter cassette.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record all the necessary information to determine flow rate or volume and record also the initial and final sampling time. Record the temperature and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. Since it is possible for the filter to become plugged by heavy particulate loading or by the presence of oil mists or other liquids in the air, the pump rotameter should be observed frequently, and readjusted as needed. If the flow rate cannot be adjusted to correct a problem, terminate the sampling.

5. Open the cassette filter holder and carefully remove the cellulose membrane filter from the holder and cellulose backup pad with the aid of appropriate tweezers. Transfer filter to a 2-oz. ointment jar. The filter should lay flat at the bottom of the jar.

6. Add 10 ml of 1% acetic acid to the ointment jar and seal using Teflon film as a gasket.

7. Carefully record sample identity and all relevant sample data.

8. Blank. With each batch of samples, submit one filter which is subjected to exactly the same handling as for the samples except that no air is drawn through it. Label this as a blank. Submit one blank for every ten samples.

## Special Considerations

Where two or more compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample. This sampling method is applicable for particulate hydroquinone only; in operations where significant vapor may also be present, this method will not apply.

## Bulk Sample

A bulk sample of the suspected material should be submitted to the laboratory in a glass container lined with a Teflon cap. Label of the bulk sample should match air samples for identification purposes.

## Shipping Instructions

The containers in which the sample filter and acetic acid are stored should be shipped in a suitable container designed to prevent damage or leakage in transit.

## Reference

Hydroquinone, NIOSH Method No. S57.

# Sampling Data Sheet No. S99

## Substance

Methyl Chloride

## Standard

8-hour time-weighted average: 100 ppm
Ceiling: 200 ppm
Peak: 300 ppm (5 minutes in any 3 hours)

## Analytical Method

A known volume of air is drawn through a tube containing activated coconut charcoal connected in series with a smaller backup charcoal tube to trap the methyl chloride vapors present. The methyl chloride is desorbed from the charcoal with methylene chloride, and the sample is separated and analyzed using a gas chromatograph with a flame ionization detector. The method has been validated over the range of 58.9-222 ppm for the T.W.A. concentration and 143.4-582 ppm for the peak concentration at 25°C and 760 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

Sampling equipment includes a calibrated personal sampling pump whose flow can be determined accurately (±5%). A charcoal tube, approximately 10-cm long with a 8-mm O.D. and a 6-mm I.D., containing two sections of 20/40 mesh activated coconut charcoal separated by a 2-mm portion of urethane foam, is used to collect the samples. The front section of the tube contains 400 mg of charcoal, and the back section contains 200 mg. A smaller charcoal tube, 7-cm long with a 6-mm O.D. and a 4-mm I.D., is used as a backup tube. This second tube contains 100 mg of charcoal in the front section and 50 mg in the back section.

## Sample Size

For measurement of the peak concentration, a sample size of 0.5 liter is recommended. Sample for 5 minutes at a flow rate of 0.10 liter per minute. For measurement of the T.W.A. concentration, a sample size of 1.5 liters is recommended. Sample at a flow rate of 0.025 liter/minute.

## Sampling Procedure

1. Immediately before sampling, the ends of the large and small charcoal tubes should be broken so as to provide an opening at least one-half the internal diameter of the tube.

2. Connect the larger charcoal tube in line with the smaller charcoal tube by connecting the backup section of the larger charcoal tube to the front section of the small charcoal tube with flexible vinyl tubing. The shortest length of tubing compatible with maintaining a leak-free connection should be used. The smaller charcoal tube is used as a backup and should be positioned nearer the sampling pump. Air should flow through the larger charcoal tube before entering the small charcoal tube. The charcoal tubes should be placed in a vertical position during sampling to avoid channeling and subsequent premature breakthrough of methyl chloride.

3. Air being sampled should not pass through any hose or tubing before entering the charcoal tube.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record the temperature, relative humidity, and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. Also report the type of sampling pump that is used.

5. The charcoal tubes should be separated and capped individually with plastic caps immediately after sampling. Masking tape is the only suitable substitute for sealing the tubes. Under no circumstances should rubber caps be used. Each set of tubes should be marked to identify the large charcoal tube with the corresponding backup charcoal tube.

6. With each batch of ten samples, submit one set of charcoal tubes (a larger charcoal tube and a smaller backup charcoal tube) from the same lot of tubes used for sample collection. These tubes must be subjected to exactly the same handling as the samples except that no air is drawn through them. Label this set of tubes as a blank. Information on the batch number of the charcoal must be supplied. A minimum of 18 charcoal tubes from the same batch of tubes should be provided to the analyst for desorption efficiency tests during sample analysis.

Special Considerations

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.

2. Due to the high resistance of the sampling train, this sampling method places a heavy load on the sampling pump. Therefore, no more than eight hours of sampling should be done without first fully recharging the battery.

3. The volume recommended is based on high humidity breakthrough tests. If condensation of water occurs in the tube, the substance will not be trapped quantitatively.

## Shipping Instructions

Capped charcoal tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping.

## Reference

Methyl Chloride, NIOSH Method No. S99.

Sampling Data Sheet No. S105
January 20, 1978

## Substance

Ethyl chloride

## Standard

8-hour time-weighted average: 1000 ppm (2600 mg/cu m)

## Analytical Method

A known volume of air is drawn through two charcoal tubes connected in series to trap the ethyl chloride gas present. The ethyl chloride is desorbed from the charcoal with carbon disulfide, and the sample is analyzed using a gas chromatograph with a flame ionization detector. The method has been validated over the range of 1586-6500 mg/cu m for a 3-liter sample at 24.5°C and 770.8 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

The equipment needed for sampling ethyl chloride includes a calibrated personal sampling pump whose flow can be determined to an accuracy of ±5%, at a flow rate of 0.05 liter per minute and a charcoal sampling tube series. The pump is calibrated with a representative sampling tube series in line.

The sampling train consists of two separate charcoal tubes connected in series. The tubes are glass tubes with both ends flame-sealed, 10-cm long with an 8-mm O.D. and a 6-mm I.D. The front tube contains 400 mg of 20/40 mesh activated coconut charcoal; the backup tube, 200 mg. A plug of silylated glass wool is placed at each end of the charcoal tubes. The pressure drop across the two tubes in series must be less than one inch of mercury at a flow rate of 1 liter per minute.

This sampling train can be prepared by modifying commercially available large charcoal tubes as follows: for the front tube--break off exit end of a large tube and using pointed tweezers remove urethane foam plug and the backup charcoal section; push in snugly remaining plug and charcoal to minimize channeling. For the backup tube--similarly break off inlet end of a second large tube and remove retaining plug and the front charcoal section; again push together remaining plug and charcoal. The front tube is connected to the backup tube with a minimal piece of plastic or rubber tubing.

Note: This sampling tube scheme is necessary to minimize probable sample migration during storage.

## Sample Size

A sample size of 3 liters is recommended. Sample at a flow rate of 0.02-0.05 liter per minute.

## Sampling Procedure

1. Immediately before sampling, the ends of the tubes should be broken so as to provide openings approximately one-half the internal diameter of the tubes (3-mm). Prepare and assemble tubes as described in sampling equipment section. Connect the front 400-mg tube to the 200-mg backup tube with a short piece of tubing.

2. The tube containing 200 mg of charcoal is used as a backup and should be positioned nearest the sampling pump. The charcoal tube series should be placed in a vertical position during sampling to avoid channeling and subsequent premature breakthrough of the analyte.

3. Air being sampled should not be passed through any hose or tubing before entering the front charcoal tube.

4. A low flow rate pump is used. Set the flow rate as accurately as possible using the manufacturer's directions. Record the necessary information to determine flow rate and also record the initial and final sampling time. Record the temperature and pressure of the atmosphere being sampled. If pressure reading is not available, record the elevation.

5. The charcoal tubes should be capped with the supplied plastic caps immediately after sampling. The two tubes must be separated and each tube recapped tightly. The tubes should be identified accordingly to distinguish the corresponding pair of front and backup sampling tubes.

6. One set of charcoal tubes (a 400-mg tube and a 200-mg backup tube) should be handled in the same manner as the sample tubes (break, seal, and transport), except for the taking of an air sample. This set of tubes should be labeled as a blank. Submit one blank for every batch of 10 samples.

7. Unused, capped charcoal tubes should accompany the samples. These tubes are used in desorption efficiency studies in conjunction with these samples because desorption efficiency varies from one batch of charcoal to another. Record the batch number of the charcoal used.

## Special Considerations

1. Where two or more compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.
2. Due to the high resistance of the charcoal tube, this sampling method places a heavy load on the sampling pump. Therefore, no more than 8 hours of sampling should be done without first fully recharging the battery.

## Shipping Instructions

Capped charcoal tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping.

## Reference

Ethyl chloride, NIOSH Method No. S105.

## Sampling Data Sheet No. S120

### Substance

Chlorodiphenyl, 42% chlorine

### Standard

8-hour time-weighted average: 1 mg/cu m - skin

### Analytical Method

A known volume of air is drawn through a glass fiber filter connected in series with a midget bubbler containing 15 ml of iso-octane to trap the chlorodiphenyl, 42% chlorine, present. The solution in the midget bubbler is transferred to a 20-ml scintillation vial and the filter is added. The analyte is separated and analyzed using a gas chromatograph with an electrolytic conductivity detector. The method has been validated over the range of 0.507-2.747 mg/cu m for a 235-liter sample at 23.5°C and 766 mm Hg atmospheric temperature and pressure.

### Sampling Equipment

Sampling equipment includes a calibrated personal sampling pump whose flow rate can be determined accurately (±5%) at 1.5 liters per minute, a 37-mm two-piece cassette filter holder held together by tape or shrinkable band, and a 37-mm diameter glass fiber filter connected in series with a midget bubbler. The filter must be free of organic binders. The filter holder should be made of polystyrene. Do not use filter holders made of Tenite. The filter is held in the filter holder supported by a stainless steel support screen. The sampling pump is protected from splash-over or solvent condensation by a 5-cm long x 6-mm I.D. glass tube loosely packed with a plug of glass wool and inserted between the exit arm of the bubbler and the pump. (One hundred milliliters of iso-octane should accompany each set of samples for rinsing the bubbler stems and transferring the contents of the bubblers to the scintillation vials.) The scintillation vials which are used for shipping should be marked at 15 ml.

### Sample Size

A sample size of 270 liters is recommended. Sample at a flow rate of 1.5 liters per minute.

### Sampling Procedure

1. Assemble the filter in the two-piece filter cassette holder and close firmly. Secure the cassette holder together with tape or a shrinkable band. Pipet 15 ml of the collection medium (iso-octane)

into each bubbler and mark the liquid level. The liquid level in the bubbler must be checked at least every 15 minutes. If the liquid level is lowered by an appreciable amount (to less than 10 ml) from evaporation, the bubbler must be refilled with additional iso-octane. The final refilling of iso-octane into the bubbler should be done at approximatley 15 minutes before the end of sampling. The final volume at the end of sampling should be 12 ml or less.

2. Remove the cassette plugs and attach the outlet of the filter cassette to the inlet arm of the midget bubbler using a short piece of flexible tubing. Connect the outlet arm of the midget bubbler with a 5-cm glass splashover tube (6-mm I.D.) containing the glass wool plug, then to the personal sampling pump, using short pieces of flexible tubing. The bubbler must be maintained in a vertical position during sampling.

3. Air being sampled should not pass through any hose or tubing before entering the filter cassette.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record the temperature and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. Also report the type of sampling pump used. Since it is possible for the filter to become plugged by heavy particulate loading or by the presence of oil mists or other liquids in the air, the pump rotameter should be observed frequently, and readjusted as needed. If the rotameter cannot be adjusted to correct a problem, terminate the sampling.

5. After sampling, remove the bubbler stem and tap the stem gently against the inside wall of the bubbler bottle to recover as much of the sampling solution as possible. Rinse the bubbler stem with 1 ml of iso-octane into the midget bubbler.

6. Transfer the contents of the midget bubbler to the scintillation vial which has been marked at 15 ml. Rinse the bubbler with 2 ml of iso-octane, adding rinse to the vial. Bring the volume in the vial to the 15-ml mark with iso-octane.

7. Remove the glass fiber filter from the cassette filter holder and place into the scintillation vial. Care must be taken to handle the filter only with clean tweezers. Cap the scintillation vial with an appropriate cap with a Teflon cap liner.

8. Carefully record the sample identity and all relevant sampling data.

9. With each batch of ten samples submit one filter and midget bubbler from the same lot of filters and bubblers used for sample collection. This filter and bubbler must be subjected to exactly the same handling as the samples except that no air is drawn through them. Label this filter and bubbler as the blank.

## Special Consideration

When other compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.

## Shipping Instructions

The scintillation vials in which the samples are stored should be shipped in a suitable container, designed to prevent damage in transit. These samples must not be sent through the mail. The Department of Transportation Regulations do permit the shipment of these samples in containers that will prevent breakage and leakage of these samples.

## Reference

Chlorodiphenyl, 42% chlorine, NIOSH Method No. S120.

Sampling Data Sheet No. S138
July 8, 1977

## Substance

n-Butylamine

## Standard

Ceiling: 5 ppm (15 mg/cu m)

## Analytical Method

A known volume of air is drawn through a sulfuric acid-treated silica gel tube to trap the n-butylamine vapors present. The n-butylamine is desorbed from the treated silica gel with 50% methanol, and the sample is analyzed using a gas chromatograph with a flame ionization detector. The method has been validated over the range of 8.09-35.5 mg/cu m for a 15-liter sample at 24°C and 759.4 mm Hg atmospheric temperature and pressure. It should be emphasized that this method is not valid when the relative humidity of the atmosphere being sampled is greater than 60%.

## Sampling Equipment

The sampling equipment needed consists of a sulfuric acid-treated silica gel sampling tube and a personal sampling pump calibrated with a representative silica gel tube in the line. The pump flow rate should be determined accurately, ± 5%, at 1.0 liter per minute. A suitable tube holder should be used to protect the worker from the sharp edges of the glass sampling tube.

The silica gel tube used to collect the sample consists of a glass tube flame sealed at both ends, 7-cm long with a 6-mm O.D. and a 4-mm I.D., packed with two sections of 20/40 mesh silica gel treated with sulfuric acid. The two sections include a front section containing 150 mg of treated silica gel and a backup section containing 75 mg. The two sections are separated by a plug of silylated glass wool and both the inlet and outlet ends of the tube are plugged with silylated glass wool. The pressure drop across the tube must be less than 25 mm Hg at a flow rate of 1 liter per minute.

The treated silica gel may be prepared as follows: A known amount of 20/40 mesh silica gel is placed in a drying oven at 125°C for one hour to activate the silica gel. The gel is then cooled to constant weight, W. Reagent grade concentrated sulfuric acid is added dropwise by means of a disposable pipet to 1.25W, or 25% by weight acid. A glass stirring rod is used to more evenly distribute the sulfuric acid. The treated silica gel is then returned to the drying oven for one hour with intermittent mixing. The treated silica gel should be stored in an airtight container.

## Sample Size

A sample size of 15 liters is recommended. Sample at a flow rate of 1.0 liter per minute for 15 minutes.

## Sampling Procedure

1. Immediately before sampling, the ends of the tubes should be broken so as to provide openings approximately one-half the internal diameter of the tubes.

2. The section containing 75 mg of treated silica gel is used as a backup and should be positioned nearer the sampling pump. The silica gel tube should be placed in a vertical position during sampling to avoid channeling and subsequent premature breakthrough of the analyte.

3. Air being sampled should not be passed through any hose or tubing before entering the front section of the treated silica gel tube.

4. Set the pump flow rate as accurately as possible using the manufacturer's directions. Record all the necessary information to determine flow rate or volume and also record the initial and final sampling time. Record the temperature and pressure of the atmosphere being sampled. If pressure reading is not available, record the elevation.

5. The treated silica gel tubes should be labeled properly and capped with the supplied plastic caps immediately after sampling. Under no circumstances should rubber caps be used.

6. One treated silica gel tube should be handled in the same manner as the sample tubes (break, seal, and transport), except for the taking of an air sample. This tube should be labeled as a blank. Submit one blank for every batch of ten samples.

7. In addition, several unused and sealed treated silica gel tubes should accompany the samples. These tubes are used in desorption efficiency studies in conjunction with these samples, because desorption efficiency may vary from one batch of treated silica gel to another.

## Special Considerations

1. Where two or more compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.

2. Due to the high resistance of the treated silica gel tube, this sampling method places a heavy load on the sampling pump. Therefore, no more than eight hours of sampling should be done without first fully recharging the battery.

3. This method is limited to sampling atmospheres where the relative humidity is less than 60%.

## Shipping Instructions

Capped silica gel tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping.

## Reference

n-Butylamine, NIOSH Method No. S138.

# Sampling Data Sheet No. S141

October 28, 1977

## Substance

Diisopropylamine

## Standard

8-hour time-weighted average: 5 ppm (20 mg/cu m)

## Analytical Method

A known volume of air is drawn through a midget impinger which contains 15 ml of 0.1N sulfuric acid. An aliquot of this sample solution is neutralized and injected into a gas chromatograph with a flame ionization detector. This method has been validated over the range of 8.5-37.4 mg/cu m for a 120-liter sample at 24°C and 766 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

The following equipment is needed for sampling diisopropylamine: a calibrated personal sampling pump whose flow can be determined to an accuracy of ±5% at a flow rate of 1.0 liter per minute, a standard midget impinger containing 15 ml of 0.1N sulfuric acid, a trap which is a second impinger or bubbler used to protect the pump from spillage, and an extra impinger solution and distilled water.

## Sample Size

A sample size of 120 liters is recommended. Sample at a flow rate of 1.0 liter per minute for 120 minutes.

## Sampling Procedure

1. Pour 15 ml of 0.1N sulfuric acid into the first impinger.

2. Assemble the sampling train. Put the first impinger in a suitable impinger holder. The outlet of this impinger is connected by tubing to the inlet of the trap. The outlet of the trap is connected by a short piece of tubing to the pump's inlet. The trap is in a suitable impinger holder which is attached to the pump. Liquid collected in the trap must never be returned to the first impinger.

3. Air should not pass through any hose or tubing before entering the first impinger.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record all the necessary information to determine flow

rate or volume and also record the initial and final sampling time. Record the temperature and pressure of the atmosphere being sampled. If pressure reading is not available, record the elevation.

5. After sampling, remove the impinger stem and tap the stem gently against the inside wall of the impinger bottle to recover as much of the sampling solution as possible. Rinse the impinger stem with 1-2 ml of 0.1N sulfuric acid into the midget impinger flask. Repeat this process for liquid collected in the trap. However, do not combine the two solutions in one impinger bottom. Be sure each impinger bottom is properly labeled. Seal the impinger with a hard, non-reactive stopper (preferably Teflon). Do not seal with rubber. The stoppers on the impingers should be tightly sealed to prevent leakage during shipping.

6. Attempt to minimize sample spillage. Do not allow the solution level in the first impinger to fall below 10 ml. Replace spilled solution with fresh 0.1N sulfuric acid. If spillage is not evidenced by liquid in the trap or in the tubing, evaporation has probably occurred. Use distilled water to bring the solution volume back up to 15 ml.

7. With each batch of ten samples submit one midget impinger containing 15 ml of 0.1N sulfuric acid prepared from the same stock as that used for sample collection. This impinger must be subjected to exactly the same handling as the samples except that no air is drawn through it. Label this impinger as the blank.

Special Consideration

1. When other air contaminants are known or suspected to be present, such information, including their suspected identities, should be transmitted with the sample.

2. If a significant amount of diisopropylamine is found by the analyst in the trap or if less than 7 ml of solution remains in the first impinger, the sample should be considered invalid.

Bulk Samples

A bulk sample of the suspected compound should be submitted to the laboratory in a glass container with a Teflon-lined cap. Use shrinkable bands or stretchable tape to keep the container tightly sealed. Label of the bulk sample should match air samples for identification purposes. Do not ship sample with the collected samples.

Shipping Instructions

The impingers in which the samples are stored should be shipped in a suitable container designed to prevent damage or leakage in transit.

## Reference

Diisopropylamine, NIOSH Method No. S141.

# Sampling Data Sheet No. S158

September 30, 1977

## Substance

2-Aminopyridine

## Standard

8-hour time-weighted average: 0.5 ppm (2.0 mg/cu m)

## Analytical Method

A known volume of air is drawn through two tubes in series each containing 35/60 mesh Tenax GC to trap the 2-aminopyridine vapors present. The 2-aminopyridine is thermally desorbed from the Tenax GC and the sample is separated and analyzed using a gas chromatograph with a flame ionization detector. The method has been validated over the range of 0.913-3.59 mg/cu m for a 12-liter sample at 24°C and 758 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

Sampling equipment includes a calibrated personal sampling pump whose flow can be determined accurately (±5%) in the range of 0.01 to 0.2 liter per minute. Two tubes (each tube is 13 cm long with a 6-mm O.D. and 4-mm I.D.) connected in series are used to collect the samples. The front tube contains 35 mg of Tenax GC, and the backup tube contains 17 mg. The Tenax GC is held in place in the sample tube with 3-mm plugs of glass wool. Immediately prior to packing, the tubes should be acetone rinsed and dried to eliminate the problem of Tenax GC adhering to the walls of the glass tubes. Before use of the tubes, each Tenax GC tube must be thermally desorbed for 3 minutes at 240°C with nitrogen to rid the Tenax GC of any interfering substances.

## Sample Size

A sample size of 12 liters is recommended. Sample at a flow rate between 0.01 and 0.2 liter/minute. Do not sample at a flow rate less than 0.01 liter/minute.

## Sampling Procedure

1. Immediately before sampling, remove the caps from the ends of the tube. All tubes must contain Tenax GC from the same manufacturer's lot.

2. The tube containing the smaller amount of Tenax GC is used as a backup and should be positioned nearer the sampling pump. The two tubes are connected with a small piece of flexible tubing. Air should flow through the front tube before entering the backup tube. The tubes should be placed in a vertical position during sampling to avoid channeling and subsequent premature breakthrough of 2-aminopyridine.

3. Air being sampled should not be passed through any hose or tubing before entering the Tenax GC tube.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record the temperature, relative humidity, and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. Also report the type of sampling pump that is used.

5. The Tenax GC tubes should be separated and capped individually with plastic caps immediately after sampling. Under no circumstances should rubber caps be used. Each set of tubes should be marked to identify the front Tenax GC tube with its corresponding backup tube.

6. With each batch of ten samples, submit one set of tubes (a front adsorbing tube containing 35 mg of Tenax GC and a backup tube containing 17 mg of Tenax GC) from the same lot of tubes used for sample collection. These tubes must be subjected to exactly the same handling as the samples except that no air is drawn through them. These tubes should be labeled as the blanks.

Special Considerations

1. When other compounds are known or suspected to be present in the air, such information, including their suspected identities should be transmitted with the sample.

2. Due to the high resistance of the Tenax GC tube, this sampling method places a heavy load on the sampling pump. Therefore, no more than eight hours of sampling should be done without first fully recharging the battery.

3. The volume recommended is based on high humidity breakthrough tests. Further reduction in sample volume due to high humidity should not be needed. If condensation of water occurs in the tube, the substance may not be trapped quantitatively.

Bulk Samples

A bulk sample of the suspected compound should be submitted to the laboratory in a glass container with a Teflon-lined cap or equivalent. Label of the bulk sample should match air samples for identification purposes.

## Shipping Instructions

Capped Tenax GC tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping. Never transport, mail, or ship the bulk sample in the same container as the sample or blank tube.

## Reference

2-Aminopyridine, NIOSH Method No. S158.

# Sampling Data Sheet No. S169

## Substance

Acetic Acid

## Standard

8-hour time-weighted average: 10 ppm (25 mg/cu m)

## Analytical Method

A known volume of air is drawn through a tube containing activated coconut charcoal to trap the acetic acid vapors present. The acetic acid is desorbed from the charcoal with formic acid, and the sample is separated and analyzed using a gas chromatograph with a flame ionization detector. The method has been validated over the range of 12.5-50.0 mg/cu m for a 173-liter sample at 22°C and 767 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

Sampling equipment includes a calibrated personal sampling pump whose flow can be determined accurately (±5%) at 1.0 liter per minute. A charcoal tube (7-cm long with a 6-mm O.D. and a 4-mm I.D.), containing two sections of 20/40 mesh activated coconut charcoal separated by a 2-mm portion of urethane foam, is used to collect the samples. The front section of the tube contains 100 mg of charcoal, and the back section contains 50 mg.

## Sample Size

A sample size of 168 liters is recommended. Sample at a flow rate of 1.0 liter per minute or less.

## Sampling Procedure

1. Immediately before sampling, the ends of the charcoal tube should be broken so as to provide an opening at least one-half the internal diameter of the tube. All tubes must be from the same manufacturer's lot.

2. The smaller section of charcoal is used as a backup and should be positioned nearer the sampling pump. The charcoal tube should be placed in a vertical position during sampling to avoid channeling and subsequent premature breakthrough of acetic acid.

3. Air being sampled should not be passed through any hose or tubing before entering the charcoal tube.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record the temperature, relative humidity, and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. Also report the type of sampling pump that is used.

5. The charcoal tubes should be capped individually with plastic caps immediately after sampling. Masking tape is the only suitable substitute for sealing the tubes. Under no circumstances should rubber caps be used.

6. With each batch of ten samples, submit one charcoal 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. Label this tube as a blank. Information on the batch number of the charcoal must be supplied. A minimum of 18 extra charcoal tubes should be shipped for desorption efficiency determinations.

## Special Considerations

1. When other compounds are known or suspected to be present in the air, such information, including their suspected identities should be transmitted with the sample.

2. Due to the high resistance of the charcoal tube, this sampling method places a heavy load on the sampling pump. Therefore, no more than eight hours of sampling should be done without first fully recharging the battery.

3. The volume recommended is based on high humidity breakthrough tests. If condensation of water occurs in the tube, the substance will not be trapped quantitatively.

## Shipping Instructions

Capped charcoal tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping. Never transport, mail, or ship the bulk sample in the same container as the sample or blank tube.

## Reference

Acetic Acid, NIOSH Method No. S169.

Sampling Data Sheet No. S181
September 30, 1977

## Substance

Quinone

## Standard

8-hour time weighted average: 0.4 mg/cu m

## Analytical Method

A known volume of air is drawn through an XAD-2 resin tube to trap the quinone vapors present. The quinone is desorbed from the resin with 20% ethanol in hexane, and the sample is analyzed by high pressure liquid chromatography with UV detection at 240 nm. The method has been validated over the range of 0.017-0.75 mg/cu m for a 24-liter sample at 25°C and 767 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

The sampling equipment needed consists of an XAD-2 resin sampling tube and a personal sampling pump calibrated with a representative resin tube in the line. The pump flow rate should be determined accurately to within ±5%, at 0.2 liter per minute. A suitable tube holder should be used to protect the worker from the sharp edges of the glass sampling tube.

The resin tube used to collect the sample consists of a glass tube, cap sealed at both ends, 10-cm long with a 6-mm O.D. and a 4-mm I.D., packed with two sections of (20/50) mesh XAD-2 resin. The two sections include a front section containing 100 mg of resin and a backup section containing 50 mg. The two sections are separated by a plug of silylated glass wool and both the inlet and outlet ends of the tube are also plugged with silylated glass wool. The pressure drop across the tube must be less than 25 mm of mercury at a flow rate of 1 liter per minute.

## Sample Size

A sample size of 24 liters is recommended. Sample at a known flow rate between 0.2 and 0.01 liter per minute.

## Sampling Procedure

1. Immediately before sampling, the plastic caps at the ends of the tubes should be removed.

2. The section containing 50 mg of resin is used as a backup and should be positioned nearest the sampling pump. The resin tube should be placed in a vertical position during sampling to avoid channeling and subsequent premature breakthrough of the analyte.

3. Air being sampled should not be passed through any hose or tubing before entering the front section of the resin tube.

4. A low flow rate pump is used. Set the flow rate as accurately as possible using the manufacturer's directions. Record all the necessary information to determine flow rate or volume and also record the initial and final sampling time. Record the temperature and pressure of the atmosphere being sampled. If pressure reading is not available, record the elevation.

5. The resin tubes should be labeled properly and capped with the supplied plastic caps immediately after sampling. Under no circumstances should rubber caps be used.

6. One resin tube should be handled in the same manner as the sample tubes (break, seal, and transport), except for the taking of an air sample. This tube should be labeled as a blank. Submit one blank for every batch of 10 samples.

7. Unused, capped sampling tubes should accompany the samples. These tubes are used in desorption efficiency studies in conjunction with these samples, because desorption efficiency can vary from one batch or resin to another. Record the batch number of the resin used.

## Special Considerations

1. Where two or more compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.

2. Due to the high resistance of the resin tube, this sampling method places a heavy load on the sampling pump. Therefore, no more than 8 hours of sampling should be done without first fully recharging the battery.

3. Arrange to have the samples analyzed within seven days. Minimize exposure to light and refrigerate samples.

## Shipping Instructions

Capped resin rubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping.

## Reference

Quinone, NIOSH Method No. S181.

## Sampling Data Sheet No. S199

July 8, 1977

### Substance

Mercury

### Standard

Ceiling: 0.1 mg/cu m

### Analytical Method

A known volume of air is drawn through a glass tube containing silvered Chromosorb P (AgCP) to trap the mercury vapors present. The mercury is thermally desorbed from the AgCP and passed through the absorption cell of a flameless atomic absorption spectrophotometer for analysis. The method was validated over the range of 0.0456-0.1800 mg/cu m for a 3-L sample at 23°C and 760 mm Hg atmospheric temperature and pressure.

### Sampling Equipment

Sampling equipment includes a calibrated personal sampling pump whose flow can be determined accurately (±5%) in the range of 0.01 to 0.2 liter/minute. A tube (2.0-cm long with a 6-mm O.D. and a 4-mm I.D.) containing 30 mg of specially prepared 30/60 mesh AgCP, is used to collect the samples. The sample tubes must be specially prepared as described in NIOSH Method No. S199, and thermally desorbed before use. Before and after sampling, these tubes are stored in clean capped glass vials. A 0.8-micrometer/37-mm mixed cellulose ester membrane prefilter is used to collect particulate mercury.

### Sample Size

A sample size of 3 liters is recommended. Sample at a flow rate between 0.01 and 0.2 liter/minute. Do not sample at a flow rate less than 0.01 liter/minute. Sample for 15 minutes.

### Sampling Procedure

1. White gloves or laboratory wipers should be used when handling the sampling tubes. After the sampling tubes have been prepared and stored, they should be stable indefinitely. Immediately before sampling, remove the sample tube from the vial.

2. The sample tube is preceded by a 0.8 micrometer/37-mm cellulose ester membrane filter to collect particulate mercury. The filter is placed in a styrene filter holder, and the filter is supported by a cellulose backup pad. The prefilter is connected to the sample tube with a short piece of flexible tubing.

3. The sample tube should be placed nearer the sampling pump and in a vertical position during sampling to avoid channeling through the AgCP.

4. Air being sampled should not be passed through any hose or tubing before entering the filter holder. A minimum amount of flexible tubing should be used to connect the filter holder to the sample tube.

5. Set the flow rate as accurately as possible using the manufacturer's directions. Record the temperature, relative humidity, and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. Also report the type of sampling pump that is used.

6. After sampling, disconnect the filter cassette from the sampling tube. Return the AgCP tube to the glass vial. Insert cotton into the vial to prevent breakage of the tube during shipping. Gloves or wipers should be used when handling the sample tube. The prefilter should be discarded after sampling, and the filter cassette holder should be cleaned for future use.

7. 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.

Special Considerations

1. To avoid contamination of the outer surfaces of the sampling tube, white gloves or laboratory wipers should be used when handling the tubes.

2. Loading the prefilter with excess particulate will increase air flow resistance through the sampler. This may overload the battery-operated sampling pumps.

3. It has been reported that AgCP absorbs methyl mercury chloride and that most other organomercury compounds are not retained.

4. Strong oxidizing vapors and gases, particularly chlorine, which attack silver, reduce the efficiency of the sampling tube, but do not interfere in the analysis of the sample. These interferences should not be a problem under normal sampling conditions.

5. When other compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.

Bulk Samples

A bulk sample of the suspected compound should be submitted to the laboratory in a glass container with a Teflon-lined cap. Label of the bulk sample should match air samples for identification purposes.

## Shipping Instructions

Vials containing the sample tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping. Never transport, mail, or ship the bulk sample in the same container as the sample or blank tubes.

## Reference

Mercury, NIOSH Method No. S199.

Sampling Data Sheet No. S203
February 18, 1977

## Substance

Cobalt Metal Dust and Fume

## Standard

8-hour time-weighted average: 0.1 mg/cu m

## Analytical Method

A known volume of air is drawn through a mixed cellulose ester membrane filter to trap the cobalt metal dust and fume present. The cobalt is solubilized by treating with aqua regia and the sample filters are digested with aqua regia followed by concentrated nitric acid. The quantity of analyte present is determined by atomic absorption spectrophotometry at 240.7 nm, using an air acetylene flame. The method has been validated independently, for dust over the concentration range of 0.04-0.26 mg/cu m for a 300-liter sample at 22-26 °C and 740-765 mm Hg atmospheric temperature and pressure, and for fume over the concentration range of 0.03-0.22 mg/cu m for a 270-liter sample at 18-22 °C and 750-760 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

The following items are needed: a calibrated personal sampling pump whose flow can be determined to an accuracy of ±5%, at a flow rate of 1.5 liters per minute; a 37-mm three piece filter holder held together by tape or shrinkable band; and a 37-mm diameter, 0.8 micrometer pore size mixed cellulose ester membrane filter (MCEF) supported by a cellulose backup pad.

## Sample Size

For the personal sample, a sample size of 300-liters is recommended. Sample at a flow rate of approximately 1.5 liters per minute for 200 minutes.

## Sampling Procedure

1. Assemble the filter and three-piece filter holder and close firmly to insure that the center ring seals the edge of the filter. Examine the holder for a good filter seal. If the filter holder will not seal tightly, it should be discarded. Secure the filter holder together with tape or shrinkable band.

2. Remove the filter holder plugs and attach to the personal sampling pump tubing. Clip the holder to the worker's lapel.

3. Air being sampled should not be passed through any hose or tubing before entering the filter holder.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record all the necessary information to determine flow rate or volume and record also the initial and final sampling time. Record the temperature and pressure of the atmosphere being sampled.

5. Since it is possible for the filter to become plugged by heavy particulate loading or by the presence of oil mists or other liquids in the air as evidenced by filter caking, the pump rotameter should be observed frequently, and readjusted as needed. If the flow rate cannot be readjusted to 1.5 liters per minute, terminate the sampling.

6. After sampling, filter holders should be firmly sealed with plugs in both the inlet and outlet.

7. Carefully record sample identity and all relevant sample data.

8. Blank. With each batch of samples, submit one filter which is subjected to exactly the same handling as for the samples except that no air is drawn through it. Label this as a blank. Submit one blank for every ten samples.

## Special Considerations

Where two or more compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.

## Shipping Instructions

The filter holders in which the samples are collected should be shipped in a suitable container, designed to prevent damage in transit.

## Reference

Cobalt metal dust and fume, NIOSH Method No. S203.

# Sampling Data Sheet No. S214

April 15, 1977

## Substance

Dinitrobenzene (all isomers)

## Standard

8-hour time-weighted average: 1 mg/cu m - skin

## Analytical Method

A known volume of air is drawn through a mixed cellulose ester membrane filter connected in series to a midget bubbler containing 10 ml of ethylene glycol to collect dinitrobenzene. The filter and bubbler are disconnected, and the filter is removed from the cassette holder and added to the bubbler flask. Dinitrobenzene is analyzed by high pressure liquid chromatography. The method has been validated over the range of 0.418-2.386 mg/cu m for a 90-liter sample at 23°C and 758 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

Sampling equipment includes a calibrated personal sampling pump whose flow rate can be determined accurately (±5%) at 1.5 liters per minute, a 37-mm two-piece cassette filter holder held together by tape or shrinkable band, and a 37-mm/0.8 micrometer cellulose ester membrane filter connected in series with a midget bubbler. The bubbler medium is reagent grade ethylene glycol, and it should be obtained from the supporting analytical laboratory. The filter is supported by a stainless steel screen. It is important that the screen be used rather than a backup pad, as dinitrobenzene vapors may be partially retained by the pad, and low results obtained. The pump can be protected from spillage or splashover by using a trap which is a second bubbler or impinger downstream of the midget bubbler. Teflon tubing (15 cm long x 7-mm I.D.) or Teflon plugs are needed for sealing the inlet and outlet of the bubbler stem before shipping.

## Sample Size

A sample size of 90 liters is recommended. Sample at a flow rate of 1.5 liters per minute.

## Sampling Procedure

1. Assemble the filter in the two-piece filter cassette holder and close firmly. Secure the cassette holder together with tape or a shrinkable band. Pour 10 ml of the ethylene glycol into each

bubbler. Be sure that the frit is completely immersed in the ethylene glycol. If necessary, adjust the liquid level to cover the frit.

2. Remove the cassette plugs and attach the outlet of the filter cassette to the inlet arm of the midget bubbler using a short piece of flexible tubing. Connect the outlet of the midget bubbler to either the pump's inlet or the trap's inlet. When a trap is used, it is attached to the pump by tape or a holder. The outlet of the trap is connected by tubing to the pump's inlet. Material collected in the trap must never be returned to the midget bubbler. After sampling, discard the material collected in the trap. The bubbler must be maintained in a vertical position during sampling.

3. Air being sampled should not pass through any hose or tubing before entering the filter cassette.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record the temperature and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. Also report the type of sampling pump used. Since it is possible for the filter to become plugged by heavy particulate loading or by the presence of oil mists or other liquids in the air, the pump rotameter should be observed frequently, and readjusted as needed. If the rotameter cannot be adjusted to correct a problem, terminate the sampling.

5. After sampling, disconnect the filter and bubbler. Remove the bubbler stem, and remove the filter from the filter cassette with clean tweezers and add it to the bubbler. Replace the bubbler stem. The inlet and outlet of the bubbler stem should be sealed by connecting a piece of Teflon tubing between them or inserting Teflon plugs in the inlet and outlet. Do not seal with rubber. The standard taper joint of the bubbler should be taped securely to prevent leakage during shipping. It is necessary to place the filter in the bubbler solution at this time, otherwise loss of dinitrobenzene from the filter by vaporization may occur.

6. Carefully record the sample identity and all relevant sampling data such as sample flow rate and collection time.

7. With each batch of ten samples submit one midget bubbler containing 10 ml of ethylene glycol and a blank filter from the same lot of filters and bubblers used for sample collection. This filter and bubbler must be subjected to exactly the same handling as the samples except that no air is drawn through them. Label this filter and bubbler as the blank.

## Special Consideration

When other compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.

## Shipping Instructions

The bubblers should be shipped in a suitable container, designed to prevent damage in transit. The samples should be shipped to the laboratory as soon as possible.

## Reference

Dinitrobenzene, NIOSH Method No. S214.

# Sampling Data Sheet No. S215

## Substance

Dinitrotoluene

## Standard

8-hour time-weighted average: 1.5 mg/cu m - skin

## Analytical Method

A known volume of air is drawn through a mixed cellulose ester membrane filter connected in series to a midget bubbler containing 10 ml of ethylene glycol to trap the dinitrotoluene present. Five milliliters of methanol are added to the bubbler flask, and the filter is added to the bubbler flask. Dinitrotoluene is separated and analyzed by high pressure liquid chromatography. The method has been validated over the range of 0.900-5.02 mg/cu m for a 90-liter sample at 20°C and 764 mm atmospheric temperature and pressure.

## Sampling Equipment

Sampling equipment includes a calibrated personal sampling pump whose flow can be determined accurately (±5%) at 1.5 liters per minute, a 37-mm two-piece cassette filter holder held together by tape or shrinkable band, and a 37-mm/0.8 micrometer cellulose ester membrane filter connected in series with a midget bubbler. The filter is supported by a stainless steel screen. It is important that the screen be used rather than a backup pad, as dinitrotoluene vapors may be partially retained by the pad. The sampling pump is protected from splashover or solvent condensation by a 5-cm long x 6-mm I.D. glass tube loosely packed with a plug of glass wool and inserted between the exit arm of the bubbler and the pump. Teflon tubing (15-cm long x 7-mm I.D.) or Teflon plugs are needed for sealing the inlet and outlet of the bubbler stem before shipping.

## Sample Size

A sample size of 90 liters is recommended. Sample at a flow rate of 1.5 liters per minute.

## Sampling Procedure

1. Assemble the filter in the two-piece filter cassette holder and close firmly. Secure the cassette holder together with tape or a shrinkable band. Pipet 10 ml of the collection medium (ethylene glycol) into each bubbler, and mark the liquid level. Be sure that the frit is completely immersed in the ethylene glycol.

2. Remove the cassette plugs and attach the outlet of the filter cassette to the inlet arm of the midget bubbler using a short piece of flexible tubing. Connect the outlet arm of the midget bubbler with a 5-cm glass splashover tube (6-mm I.D.) containing the glass wool plug, then to the personal sampling pump, using short pieces of flexible tubing. The bubbler must be maintained in a vertical position during sampling.

3. Air being sampled should not pass through any hose or tubing before entering the filter cassette.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record the temperature and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. Also report the type of sampling pump used. Since it is possible for the filter to become plugged by heavy particulate loading or by the presence of oil mists or other liquids in the air, the pump rotameter should be observed frequently, and readjusted as needed. If the rotameter cannot be adjusted to correct a problem, terminate the sampling.

5. After sampling, disconnect the filter and bubbler. The inlet and outlet of the bubbler stem should be sealed by connecting a piece of Teflon tubing between them or inserting Teflon plugs in the inlet and outlet. Do not seal with rubber. The standard taper joint of the bubbler should be taped securely to prevent leakage during shipping.

6. The filters are shipped in the cassette filter holder. Each filter should be marked to identify it with its corresponding backup bubbler.

7. Carefully record the sample identity and all relevant sampling data such as sample flow rate and collection time.

8. With each batch of ten samples submit one filter and midget bubbler containing 10 ml of ethylene glycol from the same lot of filters and bubblers used for sample collection. This filter and bubbler must be subjected to exactly the same handling as the samples except that no air is drawn through them. Label this filter and bubbler as the blank.

### Special Consideration

When other compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.

## Shipping Instructions

The cassette filter holders and bubblers should be shipped in a suitable container, designed to prevent damage in transit. The samples should be shipped to the laboratory as soon as possible.

## Reference

Dinitrotoluene, NIOSH Method No. S215.

Sampling Data Sheet No. S219
January 20, 1978

## Substance

Nitroethane

## Standard

8-hour time-weighted average: 100 ppm (310 mg/cu m)

## Analytical Method

A known volume of air is drawn through two XAD-2 tubes connected in series to trap the nitroethane vapors present. The nitroethane is desorbed from the XAD-2 with ethyl acetate, and the sample is analyzed using a gas chromatograph with a flame ionization detector. The method has been validated over the range of 147.4-604 mg/cu m for a 3-liter sample at 22°C and 777 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

The equipment needed for sampling nitroethane includes a calibrated personal sampling pump whose flow can be determined to an accuracy of ± 5% at a flow rate between 0.03 and 0.05 liter per minute and an XAD-2 sampling tube series. The pump is calibrated with a representative sampling tube series in line.

The sampling tube series consists of two separate XAD-2 tubes. The tubes are glass tubes with both ends flame-sealed, 10 cm long with a 10-mm O.D. and a 8-mm I.D. Each tube contains the appropriate amount of 20/50 mesh XAD-2. The XAD-2 must be prewashed with ethyl acetate and dried. The front tube contains 600 mg of XAD-2; the backup tube, 300 mg. A plug of silylated glass wool is placed at each end of the sorbent tube. The pressure drop across the tube must be less than one inch of mercury at a flow rate of 1 liter per minute.

Note that this sampling tube scheme is necessary to prevent sample migration upon storage.

## Sample Size

A sample size of 3 liters is recommended. Sample at a known flow rate between 0.03 and 0.05 liter per minute.

## Sampling Procedure

1. Immediately before sampling, the ends of the tubes should be broken so as to provide openings approximately one-half the internal diameter of the tubes (4-mm). Connect the front 600-mg tube to the 300-mg backup tube with a short piece of tubing.

2. The tube containing the 300 mg of XAD-2 is used as a backup and should be positioned nearest the sampling pump. The XAD-2 tube series should be placed in a vertical position during sampling to avoid channeling and subsequent premature breakthrough of the analyte.

3. Air being sampled should not be passed through any hose or tubing before entering the front XAD-2 tube.

4. A low flow rate pump is used. Set the flow rate as accurately as possible using the manufacturer's directions. Record the necessary information to determine flow rate, and also record the initial and final sampling time. Record the temperature and pressure of the atmosphere being sampled. If pressure reading is not available, record the elevation.

5. Immediately after sampling, the two XAD-2 tubes must be separated and each tube capped with the supplied plastic caps. The tubes should be identified to distinguish each corresponding pair of front and backup sampling tubes.

6. One set of XAD-2 tubes (a 600-mg tube and a 300-mg backup tube) should be handled in the same manner as the sample tubes (break, seal, and transport), except for the taking of an air sample. This set of tubes should be labeled as a blank. Submit one blank for every ten samples.

7. Unused XAD-2 tubes should accompany the samples. These tubes are used in desorption efficiency studies in conjunction with these samples because desorption efficiency may vary from one batch of XAD-2 to another. Record the batch number of the XAD-2 used.

## Special Considerations

1. Where two or more compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.

2. Due to the high flow resistance of the XAD-2 tube, this sampling method places a heavy load on the sampling pump. Therefore, no more than eight hours of sampling should be done without first fully recharging the battery.

## Shipping Instructions

Capped XAD-2 tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping.

## Reference

Nitroethane, NIOSH Method No. S219.

Sampling Data Sheet No. S228

November 25, 1977

## Substance

Picric acid

## Standard

8-hour time-weighted average: 0.1 mg/cu m

## Analytical Method

A known volume of air is drawn through a mixed cellulose ester membrane filter to trap the picric acid particulate present. The sample filters are extracted with 70% aqueous methanol and the solution is analyzed by high pressure liquid chromatography using a variable wavelength UV detector set at 360 nm. The method has been validated over a concentration range of 0.036 - 0.189 mg/cu m at 22°C and 772 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

The following equipment is needed for sampling picric acid particulate: a calibrated personal sampling pump whose flow can be determined to an accuracy of ±5% at a flow rate of 1.5 liters per minute; a 37-mm, three piece filter holder held together by tape or shrinkable band; a 37-mm diameter, 0.8 micrometer mixed cellulose ester membrane filter (MCEF) supported by a cellulose backup pad.

## Sample Size

A sampling period of two hours is recommended. Sample at a flow rate of 1.5 liters per minute.

## Sampling Procedure

1. Assemble the filter in the three-piece filter holder and close firmly to insure that the center ring seals the edge of the filter. The MCEF is held in place by a cellulose backup pad and the filter holder is held together by plastic tape or a shrinkable cellulose band. If the middle piece of the filter holder does not fit snugly into the bottom piece of the filter holder, sample leakage will occur around the filter. A piece of flexible tubing is used to connect the filter holder to the pump.

2. Remove the filter holder plugs and attach to the personal sampling pump tubing. Clip the filter holder to the worker's lapel.

3. Air being sampled should not be passed through any hose or tubing before entering the filter holder.

4. A sample size of 180 liters is recommended. Sample at a flow rate of 1.5 liters per minute. The flow rate should be known with an accuracy of at least ±5%.

5. Turn the pump on and begin sample collection. Set the flow rate as accurately as possible using the manufacturer's directions. Since it is possible for filters to become plugged by heavy particulate loading or by the presence of oil mists or other liquids in the air, the pump rotameter should be checked frequently and readjusted as needed. If the rotameter cannot be readjusted, terminate sampling.

6. Terminate sampling at the predetermined time and note sample flow rate, collection time and ambient temperature and pressure. If pressure reading is not available, record the elevation.

7. After sampling, holders should be firmly sealed with filter holder plugs in both the inlet and outlet.

8. Carefully record sample identity and all relevant sample data.

9. With each batch of samples, submit one filter which is subjected to exactly the same handling as for the samples except that no air is drawn through it. Label this as a blank. Submit one blank for every ten samples.

## Special Considerations

Where two or more compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample. This sampling method is applicable for particulate picric acid only; in operations where significant vapor may also be present, this method will not apply.

## Bulk Sample

A bulk sample of the suspected material should be submitted to the laboratory in a glass container lined with a Teflon cap. Label of the bulk sample should match air samples for identification purposes.

## Shipping Instructions

The filter holders should be shipped in a suitable container designed to prevent damage in transit.

## Reference

Picric acid, NIOSH Method No. S228.

# Sampling Data Sheet No. S243

## Substance

Stibine

## OSHA Standard

8-hour time weighted average: 0.1 ppm (5 mg/cu m)

## Analytical Method

A known volume of air is drawn through mercuric chloride-coated silica gel to trap the stibine gas. The antimony is extracted from the treated silica gel with concentrated hydrochloric acid and analyzed colorimetrically after formation of a Rhodamine B-antimony complex. The method has been validated over the range 0.119-1.008 mg/cu m for a 20-liter sample at 21°C and 769 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

The sampling equipment needed consists of a sampling tube filled with mercuric chloride-coated silica gel and a personal sampling pump calibrated with a representative gel tube in the line. The pump flow rate should be determined accurately to ± 5% at the sampling rate used. A suitable tube holder should be used to protect the worker from the sharp edges of the glass sampling tube.

The treated silica gel tube used to collect the sample consists of a glass tube, flame sealed at both ends, 2.5 in. long with an 8-mm O.D. and 6-mm I.D., packed with two sections of mercuric chloride-coated silica gel (14/20 mesh). The two sections include a front section containing 1.0 g of treated silica gel and a backup section containing 0.5 g. The two sections are separated by a plug of silylated glass wool and both the inlet and outlet ends of the tube are plugged with silylated glass wool. The pressure drop across the tube must be less than 25 mm of mercury at a flow rate of 1 liter per minute.

The mercuric chloride-coated silica gel is prepared as described. Dry one hundred grams of silica gel at 100°C for 2 hours. Meanwhile, prepare a 10% w/v mercuric chloride solution in methyl alcohol. Add the dried silica gel to the mercuric chloride solution and let sit for 15 minutes with occasional stirring. Drain the excess mercuric chloride solution and dry the remaining silica gel at 80°C for 3 hours. Cool the silica gel to room temperature in a covered beaker. Expose the tube to a humid atmosphere until the indicator turns pink.

## Sample Size

A sample size of 18 liters is recommended. Sample at a known flow rate of 0.2 liter per minute or less. Do not sample at a flow rate less than 0.01 liter per minute.

## Sampling Procedure

1. Immediately before sampling, the ends of the tube should be broken so as to provide an opening approximately one-half the internal diameter of the tube.

2. The section containing 0.5 g of treated silica gel is used as a backup and should be positioned nearest to the sampling pump. The silica gel tube series should be placed in a vertical position during the sampling to avoid channeling and subsequent premature breakthrough of the analyte.

3. Air being sampled should not be passed through any hose or tubing before entering the treated silica gel tube.

4. A low flow rate pump is used. Set the flow rate as accurately as possible using the manufacturer's directions. Record all the necessary information to determine the flow rate or volume and also record the initial and final sampling time. Record the temperature and pressure of the atmosphere being sampled. If pressure reading is not available, record the elevation.

5. The treated silica gel tubes should be capped with the supplied plastic caps immediately after sampling. Masking tape is the only suitable substitute for sealing the tubes. Under no circumstances should rubber caps be used.

6. One treated silica gel tube should be handled in the same manner as the sample tubes (break, seal, and transport), except for the taking of an air sample. This tube should be labeled as a blank. Submit one blank for every batch of 10 samples.

## Special Considerations

1. Other air contaminants may interfere with the analysis. These species have been reported as interferences: americium (III), gold, iron (III), thallium (I), and tin (II). The suspected presence of these elements and other air contaminants should be transmitted with the samples.

2. Sample for no more than eight hours without recharging the battery to avoid drainage of pump battery.

3. Because breakthrough experiments were conducted at a relative humidity of 85%, further reductions in sample volume should not be needed. However, when water condenses on the solid sorbent, the analyte may not be trapped quantitatively.

## Shipping Instructions

Capped treated silica gel tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping. Never transport, mail, or ship the bulk sample in the same container as the sample or blank tube.

## Reference

Stibine, NIOSH Method S243.

# Sampling Data Sheet No. S253

## Substance

Benzoyl Peroxide

## Standard

8-hour time-weighted average: 5 mg/cu m

## Analytical Method

A known volume of air is drawn through a cellulose ester membrane filter to trap benzoyl peroxide. Benzoyl peroxide is extracted from the filter with ethyl ether, and the sample is separated and analyzed using a high pressure liquid chromatograph with a uv detector. The method has been validated over the range of 3.12-19.10 mg/cu m for a 90-liter sample at 26°C and 764 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

Sampling equipment includes a calibrated personal sampling pump whose flow rate can be determined accurately (±5%) at 1.5 liter per minute, a 37-mm two-piece cassette filter held together by tape or shrinkable band, and a 37-mm/0.8-micrometer cellulose ester membrane filter supported by a cellulose backup pad.

## Sample Size

A sample size of 90 liters is recommended. Sample at a flow rate of 1.5 liter per minute. Larger sample volumes can be collected provided sample loading does not exceed 2 mg.

## Sampling Procedure

1. Assemble the filter and two-piece filter cassette and close firmly. Examine the holder for a good filter seal. The filter is supported by a cellulose backup pad. If the cassette will not seal tightly, it should be discarded. Secure the cassette holder together with stretchable tape or a shrinkable band.

2. Remove the cassette plugs and attach the cassette to the personal sampling pump tubing. Clip the cassette to the worker's lapel.

3. Air being sampled should not be passed through any hose or tubing before entering the filter cassette.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record the temperature and pressure of the atmosphere being sampled. If the pressure reading is not available, record the

elevation. Also report the type of sampling pump used. Since it is possible for the filter to become plugged by heavy particulate loading or by the presence of oil mists or other liquids in the air, the pump rotameter should be observed frequently, and the flow rates readjusted as needed. If the flow rate cannot be adjusted to correct the problem, terminate sampling.

5. Record sample identity and all relevant sample data such as sample flow rate and collection time.

6. With each batch of ten samples, submit one filter from the same lot of filters used for sample collection. This filter must be subjected to exactly the same handling as the samples except that no air is drawn through it. Label this filter as a blank.

## Special Considerations

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.

2. Upon receipt of the samples in the laboratory, the filters should be transferred to scintillation vials, extracted with ether, and the solutions stored in the refrigerator. If it is not feasible to extract the filters immediately, the filters should be refrigerated as soon as possible. The samples must be received by the laboratory within 5 days of collection.

## Shipping Instructions

The cassette filter holders in which the samples are stored should be shipped in a suitable container, designed to prevent damage in transit. The samples should be shipped to the laboratory as soon as possible.

## Reference

Benzoyl Peroxide, NIOSH Method No. S253.

Sampling Data Sheet No. S288
September 2, 1977

## Substance

Hydrogen cyanide

## Standard

8-hour time-weighted average: 10 ppm (11 mg/cu m)

## Analytical Method

A known volume of air is drawn through a mixed cellulose ester membrane filter connected in series with a midget bubbler containing 15 ml of 0.1N potassium hydroxide to trap the hydrogen cyanide present. The filter is used to collect the particulate interferences, and is discarded after sampling. The resulting solution is diluted to 25 ml and analyzed by direct potentiometry using a cyanide ion selective electrode. The method has been validated over the range of 5.2-21.0 mg/cu m for a 12-liter sample at 23°C and 753 mm Hg, atmospheric temperature and pressure.

## Sampling Equipment

The following equipment is needed for sampling hydrogen cyanide: a calibrated personal sampling pump whose flow rate can be determined accurately (±5%) at 0.2 liter per minute, a 37-mm, two-piece filter holder held together by tape or shrinkable band, a 37-mm/0.8 micrometer cellulose ester membrane filter connected in series with a midget bubbler, a trap which is a second impinger or bubbler used to protect the pump from spillage, and extra bubbler solution and distilled water.

## Sample Size

A sample size of 12 liters is recommended. Sample at a flow rate of 0.2 liter per minute.

## Sampling Procedure

1. Assemble the filter in the two-piece filter holder and close firmly. Secure the filter holder together with tape or a shrinkable band. Pipet 15 ml of 0.1N potassium hydroxide into the first bubbler.

2. Remove the holder plugs and attach the outlet of the filter holder to the inlet arm of the midget bubbler using a short piece of flexible tubing. The outlet of this bubbler is connected by tubing to the inlet of the trap. The outlet of the trap is connected by a short piece of tubing to the pump's inlet. The trap is in a suitable impinger holder which is attached to the pump. Liquid collected in the trap must never be returned to the first bubbler.

3. Air being sampled should not pass through any hose or tubing before entering the filter holder.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record the temperature and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. Also report the type of sampling pump used. Since it is possible for the filter to become plugged by heavy particulate loading or by the presence of oil mists or other liquids in the air, the pump rotameter should be observed frequently, and readjusted as needed. If the rotameter cannot be adjusted to correct a problem, terminate the sampling.

5. After sampling, remove the bubbler stem and tap the stem gently against the inside wall of the bubbler bottle to recover as much of the sampling solution as possible. Rinse the bubbler stem with 1-2 ml of 0.1N potassium hydroxide into the midget bubbler flask. Seal the bubbler with a hard, non-reactive stopper (preferably Teflon). Do not seal with rubber. The stoppers on the bubblers should be tightly sealed to prevent leakage during shipping. Repeat this process for the liquid in the trap. However, do not combine the two solutions.

6. Remove the filter from the filter holder. The filter can be discarded and the holders saved for future use.

7. Carefully record sample identity and all relevant sampling data such as sample flow rate and collection time.

8. Attempt to minimize sample spillage. Do not allow the solution level in the first bubbler to fall below 10 ml. Replace spilled solution with fresh 0.1N potassium hydroxide. If spillage is not evidenced by liquid in the trap or in the tubing, evaporation has probably occurred. Use distilled water to bring the solution volume back up to 15 ml.

9. With each batch of ten samples submit one midget bubbler containing 15 ml of 0.1N potassium hydroxide prepared from the same stock as that used for sample collection. This bubbler must be subjected to exactly the same handling as the samples except that no air is drawn through it. Label this bubbler as the blank.

Special Considerations

1. When other compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample. This is especially important for halides and sulfide which interfere with the cyanide electrode performance and for ions of metals that complex cyanide such as cadmium, zinc, silver, nickel, copper, iron, and mercury.

2. Particulate cyanides will be removed using a 0.8-micron cellulose ester membrane filter connected in front of the midget bubbler. Particulate halides, sulfides and metallic ions will be removed as well.

3. In humid atmospheres collection of cyanide particulate matter has been shown to produce hydrogen cyanide during the sampling process. Therefore, in humid atmospheres the interference from particulate cyanides is not completely removed by placing a filter before the bubbler.

4. Samples should be stored or shipped in containers which prevent exposure to light so that maximum stability is maintained.

5. If a significant amount of hydrogen cyanide is found by the analyst in the trap or if less than 7 ml of solution remains in the bubbler, the sample should be considered invalid.

## Shipping Instructions

The bubblers should be shipped in a suitable container designed to prevent damage in transit. The samples should be shipped to the laboratory as soon as possible.

## Reference

Hydrogen Cyanide, NIOSH Method No. S288.

Sampling Data Sheet No. S297
December 23, 1977

## Substance

Pentachlorophenol

## Standard

8-hour time-weighted average: 0.5 mg/cu m - skin

## Analytical Method

A known volume of air is drawn through a mixed cellulose ester membrane filter connected in series to a midget bubbler containing 15 ml of ethylene glycol to collect pentachlorophenol aerosols and vapors. The filter and bubbler are disconnected, and the filter is removed from the holder and added to the bubbler flask. Pentachlorophenol is separated and analyzed by high pressure liquid chromatography using a μBondapak $C_{18}$ column and a UV detector set at 254 nm. The method has been validated over the range of 0.265-1.130 mg/cu m for a 180-liter sample at 24°C and 761 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

Sampling equipment includes a calibrated personal sampling pump whose flow rate can be determined accurately ($\pm 5\%$) at 1.5 liters per minute, a 37-mm three-piece filter holder held together by tape or shrinkable band, and a 37-mm diameter, 0.8 micrometer cellulose ester membrane filter, connected in series with a midget bubbler using flexible Teflon or polyethylene tubing. Rubber tubing must not be used or low results will be obtained. The filter is supported by a stainless steel screen. It is of importance that the screen be used rather than a backup pad, as pentachlorophenol vapors may be partially retained by the pad and again low results obtained. The sampling pump may be protected from splashover by a trap which is a second bubbler inserted between the exit arm of the first bubbler and the pump. Teflon tubing (15-cm long x 7-mm I.D.) or Teflon plugs are needed for sealing the inlet and outlet of the bubbler stems before shipping.

## Sample Size

A sample size of 180 liters is recommended. Sample at a flow rate of 1.5 liters per minute.

## Sampling Procedure

1. Assemble the filter in the three-piece filter holder and close firmly. Secure the holder together with tape or a shrinkable

band. Pipet 15 ml of the collection medium (ethylene glycol) into the first bubbler. Be sure that the frit is completely immersed in the ethylene glycol. Mark the liquid level.

2. Connect the outlet arm of this bubbler to the inlet of the trap which may be a second empty bubbler Connect the outlet of the trap to the pump's inlet. Liquid collected in the trap must never be returned to the first bubbler.

3. Remove the filter holder plugs and attach the outlet of the holder to the inlet arm of the midget bubbler using a short piece of polyethylene or Teflon tubing. The bubbler and trap must be maintained in a vertical position during sampling.

4. Air being sampled should not pass through any hose or tubing before entering the filter holder.

5. Set the flow rate as accurately as possible using the manufacturer's directions. Record the temperature and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. Since it is possible for the filter to become plugged by heavy particulate loading or by the presence of oil mists or other liquids in the air, the pump rotameter should be observed frequently and readjusted as needed. If the rotameter cannot be adjusted to correct a problem, terminate the sampling.

6. After sampling, disconnect the filter and bubbler. Remove the bubbler stem, and remove the filter from the filter holder with clean tweezers and add it to the bubbler. Replace the bubbler stem. The inlet and outlet of the bubbler stem should be sealed by connecting a piece of Teflon tubing between them or inserting Teflon plugs in the inlet and outlet. Do not seal with rubber. The standard taper joint of the bubbler should be taped securely to prevent leakage during shipping. It is necessary to place the filter in the bubbler solution at this time, otherwise loss of pentachlorophenol from the filter by vaporization may occur.

7. Carefully record the sample identity and all relevant sampling data such as sample flow rate and collection time.

8. With each batch of ten samples submit one midget bubbler containing 15 ml of ethylene glycol and a blank filter from the same lot of filters used for sample collection. This filter and bubbler must be subjected to exactly the same handling as the samples except that no air is drawn through them. Label this filter and bubbler as the blank.

Special Considerations

1. When other compounds are known or suspected to be present in the air, such information, including their suspected identities, should

be transmitted with the sample.

2. If a significant amount of pentachlorophenol is found by the analyst in the trap or if less than 7 ml of solution remains in the bubbler, the sample should be considered invalid.

Bulk Sample

A bulk sample of the suspected material should be submitted to the laboratory in a glass container closed with a Teflon-lined cap. Label of the bulk sample should match air samples for identification purposes.

Shipping Instructions

The bubblers should be shipped in a suitable container, designed to prevent damage in transit. The samples should be shipped to the laboratory as soon as possible.

Reference

Pentachlorophenol, NIOSH Method No. S297.

Sampling Data Sheet No. S308

July 8, 1977

## Substance

Sulfur Dioxide

## Standard

8-hour time-weighted average: 5 ppm (13 mg/cu m)

## Analytical Method

A known volume of air is drawn through a mixed cellulose ester membrane filter connected in series with a midget bubbler containing 15 ml of hydrogen peroxide to trap the sulfur dioxide present. The filter is used to collect the particulate interferences, and is discarded after sampling. Isopropyl alcohol is added to the contents of the bubbler, and the pH of the sample is adjusted with dilute perchloric acid. The resulting solution is titrated with 0.005 M barium perchlorate using Thorin as the indicator. The method has been validated over the range of 6.55-26.8 mg/cu m for a 90-liter sample at 21°C and 757 mm Hg, atmospheric temperature and pressure.

## Sampling Equipment

Sampling equipment includes a calibrated personal sampling pump whose flow rate can be determined accurately (±5%) at the sampling rate; a 37-mm two-piece filter holder held together by tape or shrinkable band; a 37-mm, 0.8-micrometer cellulose ester membrane filter; and a midget bubbler containing 0.3 N hydrogen peroxide. The filter is supported by a stainless steel screen. The pump can be protected from spillage or splashover by using a second impinger or bubbler as a trap. One hundred milliliters of 0.3 N hydrogen peroxide should accompany each set of samples for rinsing the bubbler stems. The pump must be calibrated with a representative sampling train in line.

## Sample Size

1. At levels corresponding to the standard, a sample size of 90 liters is recommended. Sample at a known flow rate between 1.5 and 1.0 liter per minute.

2. At levels outside of range over which the method was validated, adjust the sample volume to collect between 0.6 and 2.3 milligrams of sulfur dioxide.

Sampling Procedure

1. Assemble the filter in the two-piece cassette holder and close firmly. Secure the cassette holder together with tape or a shrinkable band. Pipet 15 ml of hydrogen peroxide into each bubbler.

2. Remove the filter holder's plugs and attach the outlet of the filter holder to the inlet arm of the midget bubbler with a short piece of flexible tubing. The outlet of the midget bubbler is attached to the pump's inlet or a trap which may be used to protect the pump during personal sampling. The trap is a midget impinger or bubbler with the stem broken off which is used to collect spillage. The trap is attached to the pump with a metal holder. The outlet of the trap is connected to the pump by flexible tubing.

3. Air being sampled should not pass through any hose or tubing before entering the filter cassette.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record the temperature and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. Also report the type of sampling pump used. Since it is possible for the filter to become plugged by heavy particulate loading or by the presence of oil mists or other liquids in the air, the pump rotameter should be observed frequently, and readjusted as needed. If the rotameter cannot be adjusted to correct a problem, terminate the sampling.

5. After sampling, remove the bubbler stem and tap the stem gently against the inside wall of the bubbler bottle to recover as much of the sampling solution as possible. Rinse the bubbler stem with 1-2 ml of hydrogen peroxide into the midget bubbler flask. Replace the bubbler stem. The inlet and outlet of the bubbler stem should be sealed by connecting a piece of Teflon tubing between them or inserting Teflon plugs in the inlet and outlet. Do not seal with rubber. The standard taper joint of the bubbler should be taped securely to prevent leakage during shipping.

6. Remove the filter from the cassette filter holder. The filter can be discarded and the cassette holders and stainless steel screens cleaned and saved for future use.

7. Carefully record sample identity and all relevant sampling data such as sample flow rate and collection time.

8. With each batch of ten samples submit one midget bubbler containing 15 ml of 0.3 N hydrogen peroxide prepared from the same stock as that used for sample collection. This bubbler must be subjected to exactly the same handling as the samples except that no air is drawn through it. Label this bubbler as the blank.

9. Care should be taken at all times to minimize spillage or loss of sample by evaporation.

## Special Considerations

1. The presence and suspected identities of other air contaminants must be recorded and reported to the laboratory. Volatile phosphates will be a significant interference. Volatile metals can be converted to metallic ions in the bubbler. The presence of these and other air must be noted so that the analyst can pretreat the sample.

2. Particulate contaminants such as sulfate, sulfuric acid, and metals are removed by the prefilter.

3. Metal particulates will also be removed using the 0.8-micrometer cellulose ester membrane filter. Volatile metal interferences that are converted to metallic ions in the midget bubbler can be removed by passing the sample through a cation exchange resin.

4. Concentrations of phosphate ions greater than the sulfate ion concentration will cause appreciable interference. Particulate phosphates will be removed using the 0.8-micrometer cellulose ester membrane filter connected in front of the midget bubbler. Phosphates can also be removed by precipitation with magnesium carbonate.

## Shipping Instructions

The bubblers should be shipped in a suitable container designed to prevent damage in transit. The samples should be shipped to the laboratory as soon as possible.

## Reference

Sulfur Dioxide, NIOSH Method No. S308.

## Sampling Data Sheet No. S316

### Substance

Zinc Oxide Fume

### Standard

8-hour time-weighted average: 5 mg/cu m

### Analytical Method

A known volume of air is drawn through a membrane filter (copolymer of acrylonitrile and polyvinylchloride) to trap the zinc oxide fume present. The quantity of analyte present on the filter samples is determined by X-ray diffraction (XRD).

The method has been validated for zinc oxide fume using the secondary XRD peak over the concentration range of 2.4-9.9 mg/cu m for a 180-210 liter sample at an atmospheric temperature and pressure which varied within the range of 20-22°C and 760-767 mm Hg.

### Sampling Equipment

The following items are needed: a calibrated personal sampling pump whose flow can be determined to an accuracy of ± 5%, at a flow rate of 1.5 liters per minute; a 37-mm three-piece filter holder held together by tape or shrinkable band; and a 37-mm/0.8 micrometer pore size membrane filter (a copolymer of polyvinylchloride and acrylonitrile) supported by a cellulose backup pad.

### Sample Size

For the personal sample, a sample size of 180 liters is recommended. Sample at a flow rate of approximately 1.5 liters per minute for 120 minutes. Filter loading should not exceed 2 mg of zinc oxide. This will occur if the workplace concentration exceeds 2 times the OSHA standard. If this is suspected, collect a smaller sample such as 90 liters.

### Sampling Procedure

1. Assemble the filter and three-piece filter holder and close firmly to ensure that the center ring seals the edge of the filter. Examine the holder for a good filter seal. If the holder will not seal tightly, it should be discarded. Secure the holder together with tape or shrinkable band.

2. Remove the holder plugs and attach to the personal sampling pump tubing. Clip the holder to the worker's lapel.

3. Air being sampled should not be passed through any hose or tubing before entering the filter holder.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record all the necessary information to determine flow rate or volume and record also the initial and final sampling time. Record the temperature and pressure of the atmosphere being sampled.

5. Since it is possible for the filter to become plugged by heavy particulate loading or by the presence of oil mists or other liquids in the air as evidenced by filter caking, the pump rotameter should be observed frequently, and readjusted as needed. If the flow rate cannot be readjusted to 1.5 liters per minute, terminate the sampling.

6. After sampling, holders should be firmly sealed with plugs in both the inlet and outlet.

7. Carefully record sample identity and all relevant sample data.

8. Blank. With each batch of samples, submit one filter which is subjected to exactly the same handling as the samples except that no air is drawn through it. Label this as a blank. Submit one blank for every ten samples.

Special Considerations

Where two or more compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample. Serious interference would be caused by the compounds listed below and their possible presence should be reported. They include:

$Zn$

$\alpha\text{-}Fe_2O_3$

$(NH_4)_3ZnCl_5$

$(NH_4)_2Zn(SO_4)_2 \cdot 6H_2O$

$(NH_4)_2ZnCl_4$

$Zn(NH_3)_2Cl_2$

Shipping Instructions

The holders in which the samples are collected should be shipped in a suitable container, designed to prevent damage in transit.

Reference

Zinc Oxide Fume, NIOSH Method No. S316

# Sampling Data Sheet No. S319
August 5, 1977

## Substance

Nitric Acid

## Standard

8-hour time weighted average: 2 ppm (5 mg/cu m)

## Analytical Method

A known volume of air is drawn through a midget impinger containing 20 ml of distilled water to trap nitric acid vapors and aerosols present. The impinger solutions are analyzed by direct potentiometry using the nitrate ion specific electrode. The method has been validated over the range of 2.60-10.8 mg/cu m for a 180-liter sample.

## Sampling Equipment

Required sampling equipment includes a midget impinger containing 20 ml of distilled water and a personal sampling pump. The pump should be calibrated with a representative impinger in the line; the flow rate should be determined accurately to within ±5% at 1.0 liter per minute. The flexible tubing connecting the impinger to the pump should be loosely plugged with a piece of glass wool to protect the sampling pump from splashover and condensation.

## Sample Size

A sample size of 180 liters is recommended. Sample at a flow rate of 1.0 liter per minute for 180 minutes.

## Sampling Procedure

1. Pipet 20 ml of distilled water into a midget impinger.

2. Attach the impinger to the sampling pump with a short piece of flexible tubing. The tubing should be loosely plugged with a piece of glass wool to protect the sampling pump from splashover and condensation. The impinger must be maintained in a vertical position during sampling.

3. Air being sampled should not be passed through any hose or tubing before entering the impinger.

4. Set the pump flow rate as accurately as possible using the manufacturer's directions. Record all the information necessary to determine flow rate or volume and also record the initial and final sampling time. Record the temperature and barometric pressure of

the atmosphere being sampled. If pressure reading is not available, record the elevation.

5. Remove the impinger stem and tap the stem gently against the inside wall of the impinger bottle to recover as much of the sampling solution as possible. Rinse the impinger stem with 1 ml of distilled water and add the wash to the impinger bottle. Seal the impinger with a hard, non-reactive stopper (preferably Teflon). Do not seal with rubber. The stoppers on the impingers should be tightly sealed to prevent leakage during shipping.

6. With each batch of 10 samples submit one impinger containing 20 ml of distilled water. This impinger should be handled in the same manner as the samples except that no air is drawn through it. Label this impinger as the blank.

## Special Considerations

Where two or more air contaminants are known or suspected to be present in the air, such information, including their suspected identifies, should be transmitted with the sample. The following anions are known interferences: $Br^-$, $Cl^-$, $F^-$, $I^-$, $PO_4^{-3}$ and $NO_2^-$.

## Shipping Instructions

The sealed impinger bottles containing the sample solutions and blanks should be shipped in a suitable container, designed to prevent damage in transit.

## Reference

Nitric Acid, NIOSH Method No. S319.

## Sampling Data Sheet No. S320
October 28, 1977

### Substance

Nitrogen Dioxide

### Standard

Time-weighted average: 5 ppm (9 mg/cu m)

### Analytical Method

A known volume of air is drawn through a sampling tube containing triethanolamine-impregnated molecular sieve to collect the nitrogen dioxide. The nitrogen dioxide is desorbed with a triethanolamine solution and an aliquot treated with hydrogen peroxide, sulfanilamide and N-(1-naphthyl)ethylenediamine dihydrochloride. The reacted nitrite ion is measured spectrophotometrically at 540 nm. The method has been validated over the range of 3.074-11.49 ppm for a 3.9-liter sample at 19°C and 758 mm Hg atmospheric temperature and pressure.

### Sampling Equipment

The sampling equipment needed consists of a sampling tube containing triethanolamine-impregnated molecular sieve and a personal sampling pump calibrated with a representative sampling tube in the line. The pump flow rate should be determined accurately, ±5%, at 0.050 liter per minute. A suitable tube holder should be used to protect the worker from the sharp edges of the glass sampling tube.

The sampling tube used to collect the sample consists of a glass tube flame-sealed at both ends, with a 7-mm OD and a 5-mm ID, packed with a 400-mg section of triethanolamine-impregnated molecular sieve and a backup section of 200-mg triethanolamine-impregnated molecular sieve. Sections are separated by glass wool.

The triethanolamine-impregnated molecular sieve can be prepared as follows: Add 25 g triethanolamine to a 250-mL beaker; add 4 g glycerol, 50 mL of acetone and sufficient distilled water to bring up to 100 mL. To the mixture add about 50 mL Type 13X, 30-40 mesh molecular sieve. Stir and let stand in a covered beaker for about 30 minutes. Decant the excess liquid and transfer the molecular sieve to a porcelain pan which is then placed under a heating lamp until most of the moisture has evaporated. Complete the drying in an oven at 110°C for one hour. The sorbent should be free flowing. Store in a closed glass container.

## Sample Size

A sample size of 3.0 liters is recommended. Sample at a flow rate of 0.050 liter per minute for 60 minutes.

## Sampling Procedure

1. Immediately before sampling, the ends of the tubes should be broken so as to provide openings approximately one-half the internal diameter of the tubes.

2. The sampling tube should be placed in a vertical position during sampling to avoid channeling and subsequent premature breakthrough of the analyte.

3. Air being sampled should not be passed through any hose or tubing before entering the front section of the sampling tube.

4. A low flow rate pump is used. Set the flow rate as accurately as possible using the manufacturer's directions. Record all the necessary information to determine flow rate or volume and also record the initial and final sampling time. Record the temperature and pressure of the atmosphere being sampled. If pressure reading is not available, record the elevation.

5. The sampling tubes should be labeled properly and capped with the supplied plastic caps immediately after sampling.

6. One sampling tube should be handled in the same manner as the sample tubes (break, seal, and transport), except for the taking of an air sample. This tube should be labeled as a blank. Submit one blank for every batch of five or fewer samples.

## Special Considerations

1. Where two or more compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.

2. Due to the high resistance of the sampling tube, this sampling method places a heavy load on the sampling pump. Therefore, no more than eight hours of sampling should be done without first fully recharging the battery.

## Shipping Instructions

Capped sampling tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping.

## Reference

Nitrogen Dioxide, NIOSH Method No. S320.

Sampling Data Sheet No. S321
August 5, 1977

## Substance

Nitric oxide

## Standard

Time-weighted average: 25 ppm (30 mg/cu m)

## Analytical Method

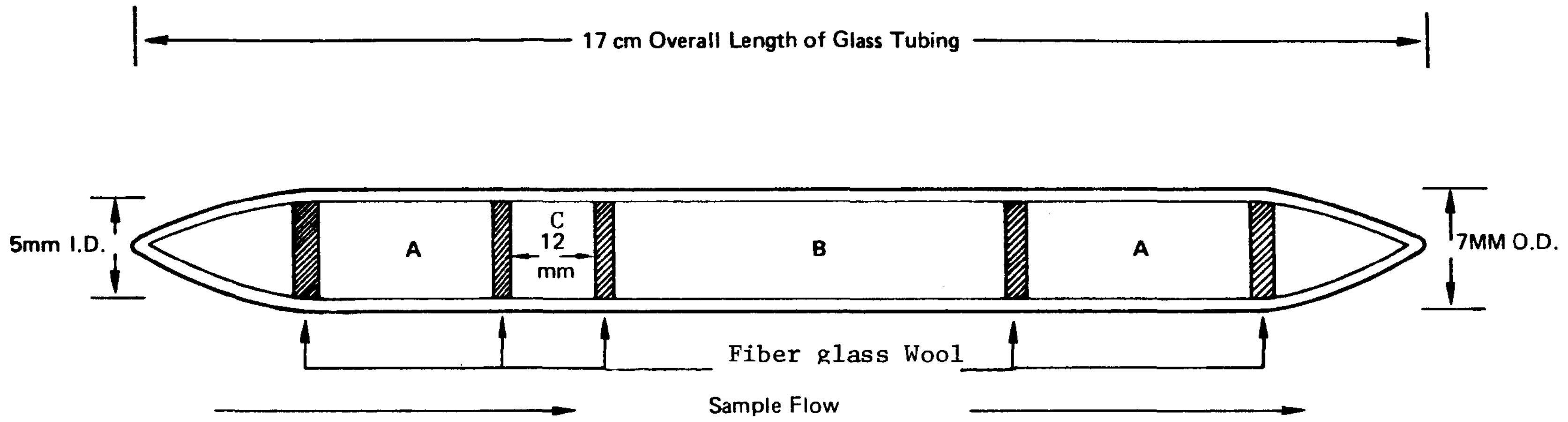
A known volume of air is drawn through a three-section sampling tube containing triethanolamine-impregnated molecular sieve and Draeger oxidizer to collect the nitric oxide after oxidation to nitrogen dioxide. The spectrophotometric method has been validated over the range of 11.14-47.7 ppm for a 1.5-liter sample at 19°C and 764 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

The sampling equipment needed consists of a three-section sampling tube containing triethanolamine-impregnated molecular sieve and Draeger oxidizer and a personal sampling pump calibrated with a representative sampling tube in the line. The pump flow rate should be set accurately, ±5%, at 0.025 liter per minute. A suitable tube holder should be used to protect the worker from the sharp edges of the glass sampling tube.

The sampling tube used to collect the sample consists of a glass tube flame-sealed at both ends, 17 cm long with a 7-mm OD and a 5-mm ID, packed with two 400-mg sections of triethanolamine-impregnated molecular sieve and one 1.2-g section of Draeger oxidizer. Sections are separated by glass wool and a 12-mm inner glass tube as shown in Figure S321-1. The pressure drop across the tube is approximately 3 cm of water at a flow rate of 0.025 liter per minute.

The triethanolamine-impregnated molecular sieve can be prepared as follows: Add 25 g triethanolamine to a 250-mL beaker; add 4 g glycerol, 50 mL of acetone and sufficient distilled water to bring up to 100 mL. To the mixture add about 50 mL Type 13X, 30-40 mesh molecular sieve. Stir and let stand in a covered beaker for about 30 minutes. Decant the excess liquid and transfer the molecular sieve to a porcelain pan which is then placed under a heating lamp until most of the moisture has evaporated. Complete the drying in an oven at 110°C for one hour. The sorbent should be free flowing. Store in a closed glass container.



A. 400 mg Triethanolamine – Impregnated Type 13 X, 30-40 Mesh Molecular Sieve

B. 1.2 g of Oxidation Material No. 1900277 from Draeger Company of Germany, supplied by National Mine Service Co.

C. 12 mm glass spacer tube, 4.0 mm O.D., 2.6 mm I.D.

NITRIC OXIDE SAMPLING TUBE

Figure S321-1

## Sample Size

A sample size of 1.5 liters is recommended. (See Special Considerations.) Sample at a flow rate of 0.025 liter per minute for 60 minutes.

## Sampling Procedure

1. Immediately before sampling, the ends of the tubes should be broken so as to provide openings approximately one-half the internal diameter of the tubes.

2. The 12-mm inner glass tube should be positioned farthest from the sampling pump. The sampling tube should be placed in a vertical position during sampling to avoid channeling and subsequent premature breakthrough of the analyte.

3. Air being sampled should not be passed through any hose or tubing before entering the front section of the sampling tube.

4. A low flow rate pump is used. Set the flow rate as accurately as possible using the manufacturer's directions. Record all the necessary information to determine flow rate or volume and also record the initial and final sampling time. Record the temperature and pressure of the atmosphere being sampled. If pressure reading is not available, record the elevation.

5. The sampling tubes should be labeled properly and capped with the supplied plastic caps immediately after sampling.

6. One sampling tube should be handled in the same manner as the sample tubes (break, seal, and transport), except for the taking of an air sample. This tube should be labeled as a blank. Submit one blank for every batch of five samples.

## Sampling Considerations

1. Where two or more compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.

2. Collection efficiency is not acceptable at a flow rate much in excess of 0.025 liter per minute if the concentration of nitric oxide approaches or exceeds twice the OSHA standard level. Therefore, the sampling rate must be set carefully.

## Shipping Instructions

Capped sampling tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping.

## Reference

Nitric oxide, NIOSH Method No. S321.

# Sampling Data Sheet No. S327

September 2, 1977

## Substance

Formaldehyde

## Standard

8-hour time-weighted average: 3 ppm

Ceiling: 5 ppm

Peak: 10 ppm for 30 minutes

## Analytical Method

A known volume of air is drawn through a midget bubbler containing 15 mL of buffered Girard T reagent to trap the formaldehyde present. The formaldehyde is collected and derivatized with the Girard T reagent and measured by polarography. This method was validated over the range of 1.4 - 6.2 ppm at an atmospheric temperature and pressure of 21°C and 759 mm Hg for a 18-L sample, representing an 8-hour time-weighted average collection. This method was also validated over the range of 4.62-19.84 ppm for 30-minute peak samples at an atmospheric temperature and pressure of 21°C and 757 mm Hg.

## Sampling Equipment

Sampling equipment includes a calibrated personal sampling pump whose flow rate can be determined accurately (±5%) at the sampling rate and a midget bubbler containing a solution of Girard T reagent buffered at a pH of 4.5. The Girard T reagent should be obtained from the supporting analytical laboratory. The pump can be protected from spillage or splash-over by using a second bubbler or an impinger as a trap.

## Sample Size

1. At levels corresponding to the 8-hour time-weighted average standard, a sample size of 18 liters is recommended. Sample at a flow rate of 0.2 liter/minute.

2. Levels corresponding to the peak standard should be collected for 30 minutes at a flow rate of 0.2 liter/minute.

## Sampling Procedure

1. Pipet 15 mL of Girard T reagent solution into each midget bubbler. Mark the liquid level on the bubbler with a china marker.

2. The outlet of the midget bubbler is attached to the pump's inlet or a trap which may be used to protect the pump during personal sampling. The trap is a midget impinger or bubbler with the stem broken off which is used to collect spillage. The trap is attached to the pump with a metal holder. The outlet of the trap is connected to the pump by flexible tubing.

3. Air being sampled should not pass through any hose or tubing before entering the midget bubbler.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record the temperature and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. Also report the type of sampling pump used.

5. Turn the pump on and begin sample collection. Terminate sampling at the predetermined time and record sample flow rate and collection time.

6. The inlet and outlet of the bubbler stem should be sealed by connecting a piece of Teflon tubing between them or inserting Teflon plugs in the inlet and outlet. Do not seal with rubber. The standard taper joint of the bubbler should be taped securely to prevent leakage during shipping. Care should be taken to minimize spillage or loss of sample by evaporation at all times.

7. With each batch of ten samples, submit one bubbler containing 15 mL of the collection medium prepared from the same stock as that used for sample collection. This bubbler must be subjected to exactly the same handling as the samples except that no air is drawn through it. Label this bubbler as the blank.

## Special Considerations

1. When other compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.

2. Other volatile aldehydes such as acrolein, crotonaldehyde, and benzaldehyde may cause significant interference.

3. The use of polyvinyl tubing preceding the midget bubbler should be kept to a minimum. Products from the tubing may interfere in the analytical method.

4. Do not return the material which is collected in the trap.

## Bulk Sample

A sample of the bulk material should be submitted to the laboratory in a glass container with a Teflon-lined cap or equivalent. This sample should not be transported in the same container as the bubblers.

## Shipping Instructions

The bubblers should be shipped in a suitable container designed to prevent damage in transit. The samples should be shipped to the laboratory as soon as possible.

## Reference

Formaldehyde, NIOSH Method No. S327.

## Sampling Data Sheet No. S334

November 25, 1977

### Substance

Phosphorus (yellow)

### Standard

8-hour time-weighted average: 0.1 mg/cu m

### Analytical Method

A known volume of air is drawn through a tube filled with Tenax-GC to trap the phosphorus vapor. The phosphorus is extracted from the resin using xylene. An aliquot of this extract is injected into a gas chromatograph equipped with a flame photometric detector. This method has been validated over the range of 0.056-0.244 mg/cu m for a 12-liter sample at 23°C and 763 mm Hg atmospheric temperature and pressure.

### Sampling Equipment

The sampling equipment needed consists of a Tenax-GC resin sampling tube and a personal sampling pump calibrated with a representative resin tube in the line. The pump flow rate should be determined accurately, ±5%, at 0.2 liter per minute. A suitable tube holder should be used to protect the worker from the sharp edges of the glass sampling tube.

The resin tube used to collect the sample consists of a glass tube flame sealed at both ends, 10-cm long with an 8-mm O.D. and a 6-mm I.D., packed with two sections of 35/60-mesh Tenax-GC resin. The two sections include a front section containing 100 mg of resin and a backup section containing 50 mg. The two sections are separated by a plug of silylated glass wool and both the inlet and outlet ends of the tube are plugged with silylated glass wool. The pressure drop across the tube must be less than 25 mm of mercury at a flow rate of 1 liter per minute.

### Sample Size

A sample size of 12 liters is recommended. Sample at a flow rate of 0.2 liter per minute or less. Do not sample at flow rates less than 0.01 liter per minute.

### Sampling Procedure

1. Immediately before sampling, the ends of the tube should be broken so as to provide an opening approximately one-half the internal diameter of the tube (3-mm).

2. The section containing 50 mg of resin is used as a backup and should be positioned nearest the sampling pump. The resin tube

series should be placed in a vertical position during sampling to avoid channeling and subsequent premature breakthrough of the analyte.

3. Air being sampled should not be passed through any hose or tubing before entering the Tenax-GC tube.

4. A low flow rate pump is used. Set the flow rate as accurately as possiable using the manufacturer's directions. Record all the necessary information to determine the flow rate or volume and also record the initial and final sampling time. Record the temperature and pressure of the atmosphere being sampled. If pressure reading is not available, record the elevation.

5. The Tenax-GC tubes should be capped with the supplied plastic caps immediately after sampling. Under no circumstances should rubber caps be used.

6. One resin tube should be handled in the same manner as the sample tubes (break, seal, and transport), except for the taking of an air sample. This tube should be labeled as a blank. Submit one blank for every batch of 10 samples.

7. Unused, capped sampling tubes should accompany the samples. These tubes are used in desorption efficiency studies in conjunction with these samples, because desorption efficiency can vary from one batch of resin to another. Record the batch number of the resin used.

Special Considerations

When two or more compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.

Bulk Samples

A bulk sample of the suspected compound should be submitted to the laboratory in a glass container with a Teflon-lined cap. Label of the bulk sample should match air samples for identification purposes.

Shipping Instructions

Capped Tenax-GC tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping. Do not transport, mail or ship the bulk sample in the same container as the sample or blank tube.

Reference

Phosphorus (yellow), NIOSH Method No. S334.

# Sampling Data Sheet No. S340

## Substance

Carbon Monoxide

## Standard

8-hour time-weighted average: 50 ppm (57 mg/cu m)

## Analytical Method

A known volume of air is collected in a five-layer gas sampling bag (Calibrated Instruments, Inc.) with the aid of a low flow rate personal pump capable of filling a bag. The sample is analyzed using an "Ecolyzer" instrument (Energetics Science Inc.). The method has been validated over the range of 25-115 ppm for a 4-liter sample at 23°C and 750 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

The sampling equipment needed includes a low flow rate personal sampling pump capable of filling a bag. The flow rate should be calibrated to within ±5%.

The 5-liter aluminized five layer gas sampling bag used to collect the sample is fabricated by Calibrated Instruments, Inc. (731 Saw Mill River Road, Ardsley, N.Y. 10502). The actual bag material composition is unknown. The bag is fitted with a metal valve and hose bib.

## Sample Size

A sample size of 3-4 liters is recommended. Sample at a flow rate of 0.05 liter per minute or less, but no less than 0.01 liter per minute.

## Sampling Procedure

1. Before sampling, the gas sampling bags should be flushed with $N_2$ or filtered air free of carbon monoxide and evacuated. The bag can be evacuated manually by pressing the bag until it is completely flat or by using a vacuum pump.

2. The gas sampling bag valve is attached to the outlet of the sampling pump with a short piece of Tygon or plastic tubing.

3. Air being sampled will pass through the pump before entering the gas sampling bag. No tubing is attached to the inlet of the pump.

4. Set the flow rate using the manufacturer's directions and determine flow rate to within ±5%. Record the information necessary to determine volume and also record the initial and final sampling time. Collect a total sample volume equal to only 80% the volume capacity of the bag to avoid over-filling.

5. The gas sampling bag should be labeled properly and the valve sealed tightly immediately after sampling.

## Special Considerations

1. Nitrogen oxide and sulfur dioxide have been reported as interferences. Information regarding the suspected presence of these bases and the identities of any other air contaminants must be transmitted with the samples.

2. Fully charge the pump battery before sampling and sample for no longer than 8 hours without recharging the battery.

## Shipping Instructions

Sealed gas sampling bags should be packed loosely and padded before they are shipped to minimize the danger of puncture during shipping.

## Reference

Carbon Monoxide, NIOSH Method No. S340.

# Sampling Data Sheet No. S346

## Substance

Allyl Glycidyl Ether

## Standard

Ceiling: 10 ppm (45 mg/cu m)

## Analytical Method

A known volume of air is drawn through a Tenax-GC resin tube to trap the allyl glycidyl ether vapors present. The allyl glycidyl ether is desorbed from the resin with diethyl ether, and the sample is separated and analyzed using a gas chromatograph with a flame ionization detector. The method has been validated over the range of 19-87 mg/cu m for a 3-liter sample at 17°C and 752 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

The sampling equipment needed consists of a Tenax-GC resin sampling tube and a personal sampling pump calibrated with a representative resin tube in the line. The pump flow rate should be determined accurately, ±5%, at 0.2 liter per minute. A suitable tube holder should be used to protect the worker from the sharp edges of the glass sampling tube.

The resin tube used to collect the sample consists of a glass tube flame sealed at both ends, 10-cm long with an 8-mm O.D. and a 6-mm I.D., packed with two sections of 35/60-mesh Tenax-GC resin. The two sections include a front section containing 100 mg of resin and a backup section containing 50 mg. The two sections are separated by a plug of silylated glass wool and both the inlet and outlet ends of the tube are plugged with silylated glass wool. The pressure drop across the tube must be less than 25 mm of mercury at a flow rate of 1 liter per minute.

## Sample Size

A sample size of 3 liters is recommended. Sample at a flow rate of 0.2 liter per minute for 15 minutes.

## Sampling Procedure

1. Immediately before sampling, the ends of the tubes should be broken so as to provide openings approximately one-half the internal diameter of the tubes.

2. The section containing 50 mg of resin is used as a backup and should be positioned nearest the sampling pump. The resin tube series should be placed in a vertical position during sampling to avoid channeling and subsequent premature breakthrough of the analyte.

3. Air being sampled should not be passed through any hose or tubing before entering the front section of the resin tube.

4. A low flow rate pump is used. Set the flow rate as accurately as possible using the manufacturer's directions. Record all the necessary information to determine flow rate or volume and also record the initial and final sampling time. Record the temperature and pressure of the atmosphere being sampled. If pressure reading is not available, record the elevation.

5. The resin tubes should be labeled properly and capped with the supplied plastic caps immediately after sampling. Under no circumstances should rubber caps be used.

6. One resin tube should be handled in the same manner as the sample tubes (break, seal, and transport), except for the taking of an air sample. This tube should be labeled as a blank. Submit one blank for every batch of 10 samples.

   In addition, three unused and sealed resin tubes should accompany the samples. These tubes are used in desorption efficiency studies in conjunction with these samples, because desorption efficiency can vary from one batch of resin to another. Record the batch number of the resin used.

## Special Considerations

1. Where two or more compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.

2. Due to the high resistance of the resin tube, this sampling method places a heavy load on the sampling pump. Therefore, no more than 8 hours of sampling per pump should be done without first fully recharging the battery.

3. The sample size recommended is less than two-thirds the breakthrough capacity at 90% relative humidity for a test atmosphere at a concentration twice the OSHA standard level. This breakthrough capacity was found to be 12 liters.

## Shipping Instructions

Capped resin tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping.

## Reference

Allyl Glycidyl Ether, NIOSH Method No. S346.

## Sampling Data Sheet No. S350

### Substance

n-Butyl Mercaptan*

### Standard

8-hour time-weighted average: 10 ppm (35 mg/cu m)

### Analytical Method

A known volume of air is drawn through a tube containing 60/80 mesh Chromosorb 104 to trap the n-butyl mercaptan vapors present. The n-butyl mercaptan is desorbed from the Chromosorb 104 with acetone and the sample is separated and analyzed using a gas chromatograph with a flame photometric detector. The method has been validated over the range of 16.8-74.2 mg/cu m for a 1.5-liter sample at 22°C and 759 mm Hg atmospheric temperature and pressure.

### Sampling Equipment

Sampling equipment includes a calibrated personal sampling pump whose flow can be determined accurately (±5%) in the range of 0.010 to 0.025 liter per minute. A tube (8.5-cm long with a 6-mm O.D. and a 4-mm I.D.), containing two sections of 60/80 mesh Chromosorb 104 separated by a 2-mm portion of urethane foam, is used to collect the samples. The front section of the tube contains 150 mg of Chromosorb 104, and the back section contains 75 mg. Immediately prior to packing, the empty tubes should be acetone rinsed and dried to eliminate the problem of Chromosorb 104 adhering to the walls of the glass tubes. Prior to packing, Chromosorb 104 is prewashed in acetone.

### Sample Size

A maximum sample size of 1.5 liters is recommended. Sample at a flow rate between 0.010 and 0.025 liter per minute. Do not sample at a flow rate less than 0.01 liter per minute.

### Sampling Procedure

1. Immediately before sampling, remove the caps from the ends of the tube. All tubes must contain Chromosorb 104 from the same manufacturer's lot. The Chromosorb 104 must be prewashed with acetone.

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*1-Butanethiol

2. The smaller section of Chromosorb 104 is used as a backup and should be positioned nearer the sampling pump. The Chromosorb 104 tube should be placed in a vertical position during sampling to avoid channeling and subsequent premature breakthrough of n-butyl mercaptan.

3. Air being sampled should not be passed through any hose or tubing before entering the Chromosorb 104 tube.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record the temperature, relative humidity, and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. Also report the type of sampling pump that is used.

5. The Chromosorb 104 tube should be capped individually with plastic caps immediately after sampling. Masking tape is the only suitable substitute for sealing the tubes. Under no circumstances should rubber caps be used.

6. With each batch of ten samples, submit one Chromosorb 104 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. Label this tube as a blank. Information on the lot number of the Chromosorb 104 must be supplied. A minimum of 18 extra Chromosorb 104 tubes should be provided for desorption efficiency determinations.

## Special Considerations

1. When other compounds are known or suspected to be present in the air, such information, including their suspected identities should be transmitted with the sample.

2. Due to the high resistance of the Chromosorb 104, this sampling method places a heavy load on the sampling pump. Therefore, no more than eight hours of sampling should be done without first fully recharging the battery.

3. The volume recommended is based on high humidity breakthrough tests. Further reduction in the sample volume due to high humidity should not be needed. If condensation of water occurs in the tube, the substance may not be trapped quantitatively.

## Bulk Samples

A bulk sample of the suspected compound should be submitted to the laboratory in a glass container with a Teflon-lined cap. Label of the bulk sample should match air samples for identification purposes. Because of postal and DOT regulations, the bulk sample should be shipped by surface mail.

## Shipping Instructions

Capped Chromosorb 104 tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping. Never transport, mail, or ship the bulk sample in the same container as the sample or blank tube.

## Reference

n-Butyl Mercaptan, NIOSH Method No. S350.

## Sampling Data Sheet No. S354

September 30, 1977

### Substance

Copper Fume

### Standard

8-hour time-weighted average : 0.1 mg/cu m

### Analytical Method

A known volume of air is drawn through a cellulose ester membrane filter to trap the copper fume present. The filter is digested with concentrated nitric acid to destroy the filter and any other organic material. The copper is then taken up in 1% nitric acid and aspirated into an atomic absorption spectrophotometer for determination of concentration. The method has been validated over the range of 0.0548-0.372 mg/cu m using a 480-liter sample at 22°C and 761 mm Hg atmospheric temperature and pressure. A detailed description of the sampling and analytical method is given in the method referenced below.

### Sampling Equipment

Sampling equipment includes a calibrated personal sampling pump whose flow can be determined to an accuracy of ±5% at approximately 2.0 liters per minute; a 37-mm two-piece cassette filter holder and a 37-mm/0.8 micrometer mixed cellulose ester membrane filter (MCEF) supported by a cellulose backup pad.

### Sample Size

A sample size of 480 liters is recommended. Sample at a flow rate of 2.0 liters per minute.

### Sampling Procedure

1. Assemble the filter and two-piece filter cassette and close firmly to insure that the center ring seals the edge of the filter. Examine the holder for a good filter seal. If the cassette will not seal tightly, it should be discarded. Secure the cassette holder together with tape or shrinkable band.

2. Remove the cassette plugs and attach to the personal sampling pump tubing. Clip the cassette to the worker's lapel.

3. Air being sampled should not be passed through any hose or tubing before entering the filter cassette.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record the temperature and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. Also report the type of sampling pump that is used.

5. Since the filter may become plugged or overloaded as evidenced by caking, the filter and pump's sampling rate should be checked periodically. When the filter becomes overloaded or when the pump's flow rate cannot be adjusted to 2.0 liters per minute, terminate sampling.

6. After termination of sample collection, the cassettes should be firmly sealed with the plugs in both the inlet and outlet.

7. Carefully record sample identity and all relevant sample data such as collection time and sampling flow rate.

8. With each batch of ten samples, submit one filter from the same lot of filters which was used for sample collection and which is subjected to exactly the same handling as the samples except that no air is drawn through it. Label this as a blank.

Special Considerations

1. Incomplete digestion of the filter or a high zinc/copper ratio in the atmosphere may lead to depression of the copper absorbance.

2. Any particulate material other than the fume containing copper or copper salts will also be collected on the filter and analyzed. This will result in a high value being reported as copper fume. If any of these compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.

Shipping Instructions

The cassettes in which the samples are collected should be shipped in a suitable container, designed to prevent damage in transit.

Reference

Copper Fume, NIOSH Method No. S354.

# Sampling Data Sheet No. S365

## Substance

Furfuryl Alcohol

## Standard

8-hour time-weighted average: 50 ppm (200 mg/cu m)

## Analytical Method

A known volume of air is drawn through a tube containing 50/80 mesh Porapak Q to trap the furfuryl alcohol vapors present. The furfuryl alcohol is desorbed from the Porapak Q with acetone and the sample is separated and analyzed using a gas chromatograph with a flame ionization detector. The method has been validated over the range of 117-469 mg/cu m for a 6-liter sample at 22°C and 765 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

Sampling equipment includes a calibrated personal sampling pump whose flow can be determined accurately (±5%) in the range of 0.01 to 0.05 liter per minute. A tube (8.5 cm long with a 6-mm O.D. and a 4-mm I.D.), containing two sections of 50/80 mesh Porapak Q separated by a 2-mm portion of urethane foam, is used to collect the samples. The front section of the tube contains 150 mg of Porapak Q, and the back section contains 75 mg. Immediately prior to packing, the tubes should be acetone rinsed and dried to eliminate the problem of Porapak Q adhering to the walls of the glass tubes. Prior to packing, the Porapak Q is prewashed in acetone.

## Sample Size

A sample size of 6 liters is recommended. Sample at a flow rate between 0.01 and 0.05 liter per minute. Do not sample at a flow rate less than 0.01 liter per minute.

## Sampling Procedure

1. Immediately before sampling, remove the caps from the ends of the tube. All tubes must contain Porapak Q from the same manufacturer's lot.

2. The smaller section of Porapak Q is used as a backup and should be positioned nearer the sampling pump. Air should flow through the larger front section before entering the smaller backup section. The Porapak Q tube should be placed in a vertical position during sampling to avoid channeling and subsequent premature breakthrough of furfuryl alcohol.

3. Air being sampled should not be passed through any hose or tubing before entering the Porapak Q tube.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record the temperature, relative humidity, and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. Also report the type of sampling pump that is used.

5. The Porapak Q tube should be capped individually with plastic caps immediately after sampling. Masking tape is the only suitable substitute for sealing the tubes. Under no circumstances should rubber caps be used.

6. With each batch of ten samples, submit one Porapak Q 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. Label this tube as a blank. Information on the batch number of the Porapak Q must be supplied. A minimum of 18 extra Porapak Q tubes should be provided for desorption efficiency determinations.

Special Considerations

1. When other compounds are known or suspected to be present in the air, such information, including their suspected identities should be transmitted with the sample.

2. Due to the high resistance of the Porapak Q tube, this sampling method places a heavy load on the sampling pump. Therefore, no more than eight hours of sampling should be done without first fully recharging the battery.

3. The volume recommended is based on high humidity breakthrough tests. Further reduction in sample volume due to high humidity should not be needed. If condensation of water occurs in the tube, the substance may not be trapped quantitatively.

Bulk Samples

A bulk sample of the suspected compound should be submitted to the laboratory in a glass container with a Polyseal* cap. Label of the bulk sample should match air samples for identification purposes.

Shipping Instructions

Capped Porapak Q tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping. Never transport, mail, or ship the bulk sample in the same container as the sample or blank tube.

Reference

Furfuryl Alcohol, NIOSH Method No. S365

*The Polyseal Corp., Baltimore, Md.

# Sampling Data Sheet No. S366

## Substance

Iron Oxide Fume

## Standard

8-hour time-weighted average: 10 mg/cu m

## Analytical Method

A known volume of air is drawn through a cellulose ester membrane filter to trap the iron oxide present. The filter is digested with nitric and hydrochloric acids to destroy the filter and any other organic material. The iron oxide is then solubilized in nitric acid and aspirated into an atomic absorption spectrophotometer for determination of concentration. The method has been validated over the range of 3.87-18.19 mg/cu m using a 145-liter sample at 23°C and 764 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

A calibrated personal sampling pump whose flow can be determined to an accuracy of ±5% at approximately 1.5 liters per minute; a 37-mm three piece cassette filter holder and a 37-mm/0.8-micrometer mixed cellulose ester membrane filter (MCEF) supported by a cellulose backup pad.

## Sample Size

A sample size of 150 liters is recommended. Sample at a flow rate of 1.5 liters per minute.

## Sampling Procedure

1. Assemble the filter and three-piece filter cassette and close firmly to insure that the center ring seals the edge of the filter. Examine the holder for a good filter seal. If the cassette will not seal tightly, it should be discarded. Secure the cassette holder together with tape or shrinkable band.

2. Remove the cassette plugs and attach to the personal sampling pump tubing. Clip the cassette to the worker's lapel.

3. Air being sampled should not be passed through any hose or tubing before entering the filter cassette.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record the temperature and pressure of the atmosphere being sampled. If the pressure reading is not available, record the

elevation. Also report the type of sampling pump that is used.

5. Since the filter may become plugged by oil mist or overloaded as evidenced by caking, the filter and pump's sampling rate should be checked periodically. When the filter becomes overloaded or when the pump's flow rate cannot be adjusted to 1.5 liters per minute, terminate sampling.

6. After termination of sample collection, the cassettes should be firmly sealed with the plugs in both the inlet and outlet.

7. Carefully record sample identity and all relevant sample data such as collection time and sampling flow rate.

8. With each batch of ten samples, submit one filter from the same lot of filters which was used for sample collection and which is subjected to exactly the same handling as the samples except that no air is drawn through it. Label this as a blank.

## Special Consideration

Because other iron compounds will be a positive interference, the presence of other iron compounds and their suspected identities should be recorded and transmitted with the samples.

## Shipping Instructions

The cassettes in which the samples are collected should be shipped in a suitable container, designed to prevent damage in transit.

## Reference

Iron Oxide Fume, NIOSH Method No. S366.

# Sampling Data Sheet No. S371

December 23, 1977

## Substance

Methoxychlor

## Standard

8-hour time-weighted average: 15 mg/cu m

## Analytical Method

A known volume of air is drawn through a glass fiber filter to trap methoxychlor. Methoxychlor is extracted from the filter with iso-octane, and the sample is separated and analyzed using a gas chromatograph equipped with an electrolytic conductivity detector. The method has been validated over the range of 7.7-31 mg/cu m for a 100-liter sample at 23°C and 761 mm Hg atmospheric temperature and pressure. Details of the sampling and analytical method are given in the method referenced below.

## Sampling Equipment

Sampling equipment includes a calibrated personal sampling pump whose flow can be determined accurately (±5%) between 1-1.5 liter/minute, a 37-mm two-piece cassette filter holder held together by tape or shrinkable band, and a 37-mm diameter glass fiber filter. The filter must be free of organic binders. The filter holder should be made of polystyrene. Do not use filter holders made of Tenite.

## Sample Size

A sample size of 100 liters is recommended. Sample at a flow rate of 1.5 liter/minute.

## Sampling Procedure

1. Assemble the filter and two-piece filter cassette and close firmly. Examine the holder for a good filter seal. The filter is supported by a backup pad. If the cassette will not seal tightly, it should be discarded. Secure the cassette holder together with tape or shrinkable band.

2. Remove the cassette plugs and attach the cassette to the personal sampling pump tubing. Clip the cassette to the worker's lapel.

3. Air being sampled should not be passed through any hose or tubing before entering the filter cassette.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record the temperature and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. Also report the type of sampling pump used. Since it is possible for the filter to become plugged by particulate loading of greater than 2 mg of dust per sample or by the presence of oil mists or other liquids in the air, the rotameter should be observed frequently, and the flow rates readjusted as needed. If the flow rate cannot be adjusted to correct the problem, terminate sampling.

5. After sampling, cap each end of the filter cassette firmly.

6. Record sample identity and all relevant sample data such as sample flow rate and collection time.

7. With each batch of ten samples, submit one filter from the same lot of filters used for sample collection. This filter must be subjected to exactly the same handling as the samples except that no air is drawn through it. Label this filter as a blank.

Special Considerations

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.

2. Technical formulations usually contain 6-12% of the o,p' isomer of methoxychlor, which may cause an interference in the analytical method.

Bulk Samples

A bulk sample of the methoxychlor should be submitted to the laboratory in a glass container with a Teflon-lined cap. Label of the bulk sample should match air samples for identification purposes.

Shipping Instructions

The cassettes in which the samples are collected should be shipped in a suitable container designed to prevent damage in transit. Never transport, mail, or ship the bulk sample in the same container as the sample or blank tube.

Reference

Methoxychlor, NIOSH Method No. S371.

Sampling Data Sheet No. S374

January 20, 1978

## Substance

Methylcyclohexanol*

## Standard

8-hour time-weighted average: 100 ppm (470 mg/cu m)

## Analytical Method

A known volume of air is drawn through a tube containing activated coconut charcoal to trap methylcyclohexanol vapors present. The methylcyclohexanol is desorbed from the charcoal with methylene chloride, and the sample is separated and analyzed using a gas chromatograph with a flame ionization detector. The method has been validated over the range of 215-920 mg/cu m for a 12-liter sample at 25.5°C and 761 mm Hg atmospheric temperature and pressure. A detailed description of the sampling and analytical method is given in the method referenced below.

## Sampling Equipment

Sampling equipment includes a calibrated personal sampling pump whose flow can be determined accurately (±5%) at 0.2 liter/minute. A charcoal tube (7-cm long with a 6-mm O.D. and a 4-mm I.D.), containing two sections of 20/40 mesh activated coconut charcoal separated by a 2-mm portion of urethane foam, is used to collect the samples. The front section of the tube contains 100 mg of charcoal, and the back section contains 50 mg.

## Sample Size

A sample size of 12 liters is recommended. Sample at a known flow rate of 0.01-0.2 liter/minute.

## Sampling Procedure

1. Immediately before sampling, the ends of the charcoal tube should be broken so as to provide an opening at least one-half the internal diameter of the tube. All tubes must be from the same manufacturer's lot.
2. The smaller section of charcoal is used as a backup and should be positioned nearer the sampling pump. The charcoal tube should be

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* 50:50 mixture of the meta and para isomers of methylcyclohexanol.

placed in a vertical position during sampling to avoid channeling and subsequent premature breakthrough of methylcyclohexanol.

3. Air being sampled should not be passed through any hose or tubing before entering the charcoal tube.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record the temperature, relative humidity, and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. Also report the type of sampling pump that is used.

5. The charcoal tubes should be capped individually with plastic caps immediately after sampling. Masking tape is the only suitable substitute for sealing the tubes. Under no circumstances should rubber caps be used.

6. With each batch of ten samples, submit one charcoal 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. Label this tube as a blank. Information on the batch number of the charcoal must be supplied. A minimum of 18 extra charcoal tubes should be shipped for desorption efficiency determinations.

## Special Considerations

1. When other compounds are known or suspected to be present in the air, such information, including their suspected identities should be transmitted with the sample.

2. Due to the high resistance of the charcoal tube, this sampling method places a heavy load on the sampling pump. Therefore, no more than eight hours of sampling should be done without first fully recharging the battery.

3. The volume recommended is based on high humidity breakthrough tests. If condensation of water occurs in the tube, the substance will not be trapped quantitatively.

## Bulk Sample

A bulk sample of the bulk material should be submitted to the laboratory in a glass container with a Teflon-lined cap.

## Shipping Instructions

Capped charcoal tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping. Never transport, mail, or ship the bulk sample in the same container as the sample or blank tube.

## Reference

Methylcyclohexanol, NIOSH Method No. S374.

# Sampling Data Sheet No. S375

## Substance

Methylcyclohexanone*

## Standard

8-hour time-weighted average: 100 ppm (460 mg/cu m) - skin

## Analytical Method

A known volume of air is drawn through a tube containing 50/80 mesh Porapak Q to trap the methylcyclohexanone vapors present. The methylcyclohexanone is desorbed from the Porapak Q with acetone and the sample is separated and analyzed using a gas chromatograph with a flame ionization detector. The method has been validated over the range of 213-852 mg/cu m for a 3-liter sample at 22°C and 759 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

Sampling equipment includes a calibrated personal sampling pump whose flow can be determined accurately (±5%) in the range of 0.01 to 0.05 liter per minute. A tube (8.5-cm long with a 6-mm O.D. and a 4-mm I.D.), containing two sections of 50/80 mesh Porapak Q separated by a 2-mm portion of urethane foam, is used to collect the samples. The front section of the tube contains 150 mg of Porapak Q, and the back section contains 75 mg. Immediately prior to packing, the tubes should be acetone rinsed and dried to eliminate the problem of Porapak Q adhering to the walls of the glass tubes. Prior to packing, Porapak Q is prewashed in acetone.

## Sample Size

A maximum sample size of 3 liters is recommended. Sample at a flow rate between 0.01 and 0.05 liter per minute. Do not sample at a flow rate less than 0.01 liter per minute.

## Sampling Procedure

1. Immediately before sampling, remove the caps from the ends of the tube. All tubes must contain Porapak Q from the same manufacturer's lot.

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* Methylcyclohexanone is a mixture of isomers of methylcyclohexanone, primarily 3-methylcyclohexanone and 4-methylcyclohexanone.

2. The smaller section of Porapak Q is used as a backup and should be positioned nearer the sampling pump. Air should flow through the larger front section before entering the smaller backup section. The Porapak Q tube should be placed in a vertical position during sampling to avoid channeling and subsequent premature breakthrough of methylcyclohexanone.

3. Air being sampled should not be passed through any hose or tubing before entering the Porapak Q tube.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record the temperature, relative humidity, and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. Also report the type of sampling pump that is used.

5. The Porapak Q tube should be capped individually with plastic caps immediately after sampling. Masking tape is the only suitable substitute for sealing the tubes. Under no circumstances should rubber caps be used.

6. With each batch of ten samples, submit one Porapak Q 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. Label this tube as a blank. Information on the batch number of the Porapak Q must be supplied. A minimum of 18 extra Porapak Q tubes should be provided for desorption efficiency determinations.

Special Considerations

1. When other compounds are known or suspected to be present in the air, such information, including their suspected identities should be transmitted with the sample.

2. Due to the high resistance of the Porapak Q tube, this sampling method places a heavy load on the sampling pump. Therefore, no more than eight hours of sampling should be done without first fully recharging the battery.

3. The volume recommended is based on high humidity breakthrough tests. Further reduction in sample volume due to high humidity should not be needed. If condensation of water occurs in the tube, the substance may not be trapped quantitatively.

Bulk Samples

A bulk sample of the suspected compound should be submitted to the laboratory in a glass container with a Polyseal* cap or equivalent. Label of the bulk sample should match air samples for identification purposes.

*The Polyseal Corp., Baltimore, MD.

## Shipping Instructions

Capped Porapak Q tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping. Never transport, mail, or ship the bulk sample in the same container as the sample or blank tube.

## Reference

Methylcyclohexanone, NIOSH Method No. S375.

Sampling Data Sheet No. S381
July 8, 1977

## Substance

Sodium Hydroxide, NaOH

## Standard

8-hour time-weighted average: 2 mg/cu m

## Analytical Method

A known volume of air is drawn through a polytetrafluoroethylene membrane filter to trap the sodium hydroxide particulate present. The sample filters are placed in water containing a known amount of excess hydrochloric acid. The amount of analyte present is determined by back titration of the remaining excess hydrochloric acid with standard sodium hydroxide. The method has been validated over the concentration range of 0.76-3.88 mg/cu m for a 360-liter sample at 23°C and 758 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

The following items are needed: a calibrated personal sampling pump whose flow rate can be determined to an accuracy of ± 5% at a flow rate of 1.5 liters per minute; a 37-mm two or three-piece filter holder held together by stretchable tape or shrinkable band; and a 37-mm diameter, 1.0 micrometer pore size polytetrafluoroethylene (PTFE) membrane filter supported by a cellulose backup pad.

## Sample Size

A sample volume of 360 liters is recommended. Sample at a flow rate of 1.5 liters per minute.

## Sample Procedure

1. Assemble the filter and three-piece filter cassette and close firmly to insure that the center ring seals the edge of the filter. Examine the holder for a good filter seal. If the cassette will not seal tightly, it should be discarded. Secure the cassette holder together with tape or shrinkable band.

2. Remove the cassette plugs and attach to the personal sampling pump tubing. Clip the cassette to the worker's lapel.

3. Air being sampled should not be passed through any hose or tubing before entering the filter cassette.

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record all the necessary information to determine flow rate or volume and record also the initial and final sampling time. Record the temperature and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. The pump rotameter should be observed frequently, and readjusted as needed. If the flow rate cannot be adjusted to correct a problem, terminate the sampling.

5. Collected sample cassettes should be firmly sealed with plugs in both the inlet and outlet.

6. Carefully record sample identity and all relevant sample data.

7. Blank. With each batch of samples, submit one filter which is subjected to exactly the same handling as for the samples except that no air is drawn through it. Label this as a blank. Submit one blank for every ten samples.

## Special Considerations

1. When other air contaminants are known or suspected to be present, such information, including their suspected identities, should be transmitted with the sample.

2. This method does not distinguish between sodium hydroxide and other alkaline species such as sodium carbonate and sodium bicarbonate.

## Shipping Instructions

The cassettes in which the samples are collected should be shipped in a suitable container, designed to prevent damage in transit.

## Reference

Sodium Hydroxide, NaOH, NIOSH Method No. S381.

Sampling Data Sheet No. S383
March 18, 1977

## Substance

Tetraethyl lead

## Standard

8 hour time weighted average: 0.075 mg/cu m (as Pb)

## Analytical Method

A known volume of air is drawn through a XAD-2 resin tube to trap the tetraethyl lead vapors present. The tetraethyl lead is desorbed from the resin with pentane, and the sample is separated and analyzed using a gas chromatograph with a photoionization detector. The method has been validated over the range of 0.045 - 0.20 mg/cu m (as Pb) for a 120-liter sample at 22°C and 767 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

The sampling equipment needed consists of a XAD-2 resin sampling tube and a personal sampling pump calibrated with a representative resin tube in the line. The pump flow rate should be determined accurately to within ±5%, at 1.0 liter per minute. A suitable tube holder should be used to protect the worker from the sharp edges of the glass sampling tube.

The resin tube used to collect the sample consists of a glass tube, capped sealed at both ends, 10-cm long with a 6-mm O.D., and a 4-mm I.D., packed with two sections of 20/50 mesh XAD-2 resin. The two sections include a front section containing 100 mg of resin and a backup section containing 50 mg. The two sections are separated by a plug of silylated glass wool and both the inlet and outlet ends of the tube are plugged with silylated glass wool. The pressure drop across the tube must be less than 25 mm of mercury at a flow rate of 1 liter per minute.

## Sample Size

A sample size of 120 liters is recommended. Sample at a flow rate of 1.0 liter per minute for 120 minutes.

## Sampling Procedure

1. Immediately before sampling, the plastic caps at the ends of the tubes should be removed.

2. The section containing 50 mg of resin is used as a backup and should be positioned nearest the sampling pump. The resin tube series

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should be placed in a vertical position during sampling to avoid channeling and subsequent premature breakthrough of the analyte.

3. Air being sampled should not be passed through any hose or tubing before entering the front section of the resin tube.

4. A low flow rate pump is used. Set the flow rate as accurately as possible using the manufacturer's directions. Record all the necessary information to determine flow rate or volume and also record the initial and final sampling time. Record the temperature and pressure of the atmosphere being sampled. If pressure reading is not available, record the elevation.

5. The resin tubes should be labeled properly and capped with the supplied plastic caps immediately after sampling. Under no circumstances should rubber caps be used.

6. One large resin tube should be handled in the same manner as the sample tubes (break, seal, and transport), except for the taking of an air sample. This tube should be labeled as a blank. Submit one blank for every batch of 10 samples.

   In addition, several unused and sealed resin tubes should accompany the samples. These tubes are used in desorption efficiency studies in conjunction with these samples, because desorption efficiency can vary from one batch of resin to another. Record the batch number of the resin used.

## Special Considerations

1. Where two or more compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.

2. Due to the high resistance of the resin tube, this sampling method places a heavy load on the sampling pump. Therefore, no more than 8 hours of sampling should be done without first fully recharging the battery.

## Shipping Instructions

Capped resin tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping.

## Reference

Tetraethyl lead (as Pb), NIOSH Method No. S383.

# Sampling Data Sheet No. S384

## Substance

Tetramethyl lead

## Standard

8-hour time-weighted average: 0.07 mg/cu m (as Pb)

## Analytical Method

A known volume of air is drawn through an XAD-2 resin tube to trap the tetramethyl lead vapors present. The tetramethyl lead is desorbed from the resin with pentane, and the sample is analyzed by gas chromatography using a photoionization detector. The method has been validated over the range of 0.040 - 0.18 mg/cu m (as Pb) for a 24-liter sample at 30°C and 760 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

The sampling equipment needed consists of an XAD-2 resin sampling tube and a personal sampling pump calibrated with a representative resin tube in the line. The pump flow rate should be determined accurately to within ±5%, at 0.2 liter per minute. A suitable tube holder should be used to protect the worker from the sharp edges of the glass sampling tube.

The resin tube used to collect the sample consists of a glass tube, capped sealed at both ends, 10-cm long with an 8-mm O.D. and a 6-mm I.D., packed with two sections of (20/50) mesh XAD-2 resin. The two sections include a front section containing 400 mg of resin and a backup section containing 200 mg. The two sections are separated by a plug of silylated glass wool and both the inlet and outlet ends of the tube are also plugged with silylated glass wool. The pressure drop across the tube must be less than 25 mm of mercury at a flow rate of 1 liter per minute.

## Sample Size

A sample size of 24 liters is recommended. Sample at a flow rate of 0.2 liter per minute or less.

## Sampling Procedure

1. Immediately before sampling, the plastic caps at the ends of the tubes should be removed.

2. The section containing 200 mg of resin is used as a backup and should be positioned nearest the sampling pump. The resin tube series should be placed in a vertical position during sampling to avoid channeling and subsequent premature breakthrough of the analyte.

3. Air being sampled should not be passed through any hose or tubing before entering the front section of the resin tube.

4. A low flow rate pump is used. Set the flow rate as accurately as possible using the manufacturer's directions. Record all the necessary information to determine flow rate or volume and also record the initial and final sampling time. Record the temperature and pressure of the atmosphere being sampled. If pressure reading is not available, record the elevation.

5. The resin tubes should be labeled properly and capped with the supplied plastic caps immediately after sampling. Under no circumstances should rubber caps be used.

6. One large resin tube should be handled in the same manner as the sample tubes (break, seal, and transport), except for the taking of an air sample. This tube should be labeled as a blank. Submit one blank for every batch of 10 samples.

7. Unused and sealed resin tubes should accompany the samples. These tubes are used in desorption efficiency studies in conjunction with these samples, because desorption efficiency can vary from one batch of resin to another. Record the batch number of the resin used.

## Special Considerations

1. Where two or more compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.

2. Due to the high resistance of the resin tube, this sampling method places a heavy load on the sampling pump. Therefore, no more than 8 hours of sampling should be done without first fully recharging the battery.

## Shipping Instructions

Capped resin tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping.

## Reference

Tetramethyl lead, NIOSH Method No. S384.

# Sampling Data Sheet No. S388

## Substance

Vanadium, $V_2O_5$ Fume

## Standard

Ceiling Value: 0.1 mg/cu m

## Analytical Method

A known volume of air is drawn through a mixed cellulose ester membrane filter to trap the vanadium oxide fume present. The sample filters are placed in 0.01N NaOH and the vanadium oxide is solubilized by heating at 50° C for 15 minutes. The quantity of analyte present is determined by flameless atomic absorption spectrophotometry at 318.4 nm, using a high temperature graphite atmomizer. The method has been validated over the concentration range of 0.06-0.29 mg/cu m for a 25-liter sample at 23°C and 761 mm Hg atmospheric temperature and pressure.

## Sampling Equipment

The following items are needed: a calibrated personal sampling pump whose flow can be determined to an accuracy of ±5% at a flow rate of approximately 1.5 liters per minute, a 37-mm three-piece cassette filter holder held together by tape or shrinkable band, and a 37-mm/ 0.8 micrometer mixed cellulose ester membrane filter (MCEF) supported by a cellulose backup pad.

## Sample Size

$V_2O_5$ fume has a ceiling value and therefore a maximum sampling period of 15 minutes is recommended. Sample at a flow rate of about 1.5 liters per minute for 15 minutes (22.5 liters).

## Sampling Procedure

1. Assemble the filter and three-piece filter cassette and close firmly to insure that the center ring seals the edge of the filter. Examine the holder for a good filter seal. If the cassette will not seal tightly, it should be discarded. Secure the cassette holder together with tape or shrinkable band.

2. Remove the cassette plugs and attach to the personal sampling pump tubing. Clip the cassette to the worker's lapel.

3. Air being sampled should not be passed through any hose or tubing before entering the filter cassette

4. Set the flow rate as accurately as possible using the manufacturer's directions. Record all the necessary information to determine flow

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rate of volume and record also the initial and final sampling time. Record the temperature and pressure of the atmosphere being sampled. If the pressure reading is not available, record the elevation. The pump rotameter should be observed frequently, and readjusted as needed. If the flow rate cannot be adjusted to correct a problem, terminate the sampling.

5. Collected sample cassettes should be firmly sealed with plugs in both the inlet and outlet.

6. Carefully record sample identity and all relevant sample data.

7. Blank. With each batch of samples, submit one filter which is subjected to exactly the same handling as for the samples except that no air is drawn through it. Label this as a blank. Submit one blank for every ten samples.

## Special Considerations

Where two or more compounds are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample. This method does not distinguish between $V_2O_5$ dust and fume.

## Shipping Instructions

The cassettes in which the samples are collected should be shipped in a suitable container, designed to prevent damage in transit.

## Reference

Vanadium, $V_2O_5$ Fume, NIOSH Method No. S388.

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