CRITERIA FOR A RECOMMENDED STANDARD

OCCUPATIONAL EXPOSURE
TO
XYLENE

ORGANIZATION RESOURCES COUNSELORS, INC.

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STUDY - ANALYSIS

There follows an ORC Occupational Safety and Health Standards Group Study-Analysis of Criteria for recommended standard - Occupational Exposure to Xylene, issued by National Institute for Occupational Safety and Health during the week of June 6, 1975.

An OSHA project officer has been assigned to study the Criteria Document. We expect an Advance Notice of Intended Rulemaking to appear in the Federal Register.

The "Recommendations for a Xylene Standard" comprising 11 pages of the Criteria Document is attached hereto.

(the following page and sections numbers are those of the Criteria Document).

The "Recommendations for a Xylene Standard" begins on page 1 of the Criteria Document; the Introduction is Section II beginning on page 12. That section states: - "The proposed standard applies only to the processing, manufacture, use of, or other occupational exposure to xylene as applicable under the Occupational Safety and Health Act of 1970."

On page 1 under the heading "I. Recommendations for a Xylene Standard", the second paragraph is: - "Regardless of the source of raw materials from which produced,'xylene' (also known as 'xylol' or 'dimethylbenzene') refers to any one of or combination of the isomers of xylene: ortho-, meta-, or para-dimethyl-benzene. The term 'xylene' will be used throughout this document. 'Exposure to xylene' is defined as exposure above half the recommended time-weighted average environmental limit. If 'exposure' to other chemicals also occurs, for example from contamination of xylene with benzene, provisions of any applicable standard for the other chemicals shall also be followed.

Major Issues Raised by the Criteria Document

1. Exposure Limits - No change is recommended in the 100 ppm twa from Table G-1 of 1910.93 except for setting a ceiling limit of 200 parts per million/10 minute sampling period and provisions relating to skin absorption.

2. Toxicity - Section II - Introduction states the primary problem to be its narcotic effects on workers. ("Therefore, the major problem of Xylene toxicity concerns its narcotic effects on workers, causing symptoms and signs such as muscular weakness, incoordination, and mental confusion which may pose a risk to both the worker and others.)
The historical research listed in the Criteria Document, however, indicates Xylene is not a carcinogen or co-carcinogen. It has only tenuous links to teratogenic effects; may provoke seizures in latent epileptics and possibly disturbs menstruation in women.

3. Format - A comparison to the Criteria Document for Benzene (ORC Study-Analysis, August 5, 1974) shows a marked similarity to the recommended standard for Xylene. Absent in the Xylene recommended standard, however, are the lengthy specifications on medical surveillance and the respirator requirements during an application for a variance. One would think that the joint OSHA/NIOSH standards completion project would have set the format for recommended standards in Criteria Documents as well. Such is not the case and this point will be raised strongly with NIOSH. Except as specifically noted our comments for Benzene apply to Xylene as well.

Section 1 - Environmental (Workplace Air), page 2

The definition of exposure, subpart (c) in the Benzene Criteria Document inexplicably appears instead in the Preamble to the Xylene recommendations.

Section 2 - Medical, page 2

As noted above, a substantial reduction in the specific requirements on medical surveillance for Benzene is apparent. It is not clear whether this represents the reduced toxicity of Xylene or a switch to a more performance approach.

Section 3 - Labeling (Posting), page 3

Unlike Benzene Section 3(a) stipulates labeling requirements for containers of Xylene in addition to the posting of warning signs.

Section 4 - Personal Protective Equipment, page 4

(b) Respiratory Protection

(1) Requires engineering controls "wherever feasible". This is duplicative of the requirements of Section 6 Work Practices (b) which also requires engineering controls. In addition it states that "such control equipment shall be spark proof". Substantial doubts have been raised by some as to the utility of this provision.

Section 5 - Informing Employees of Hazards from Xylene, page 7

(a) Emergency Procedures

This essentially duplicates the authority of the Act in Section 6(b)(7).

Subpart (b) recommends the required use of OSHA Form 20 Material Safety Data Sheets or "on a similar form approved by OSHA." No statutory or policy justification is evident for this provision.

Section 6 - Work Practices, page 7

Subpart (g) requires the grounding of all metal dispensing containers. This needs to be carefully scrutinized of cost and appropriateness.

Section 7 - Sanitation, page 9

(b) Smoking

The prohibition thereof has been moved from its position under work practices in the Benzene Criteria Document, again explicably.

Section 8 - Monitoring and Recordkeeping, page 9

Whereas the process of sampling and recording makes extensive specific requirements, no requirement is stated for length or retention for the records.

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OCCUPATIONAL EXPOSURE TO XYLENE

U.S. DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE
Public Health Service
Center for Disease Control
National Institute for Occupational Safety and Health
1975

I. RECOMMENDATIONS FOR A XYLENE STANDARD

The National Institute for Occupaticnal Safety and Health (NIOSH) recommends that employee exposure to xylene in the workplace be controlled by adherence to the following sections. The standard is designed to protect the health and safety of workers over a working lifetime; compliance with all sections of the standard should therefore prevent adverse effects of xylene on the health and safety of workers. The standard is measurable by techniques that are valid, reproducible, and available to industry and government agencies. Sufficient technology exists to permit compliance with the recommended standard. The criteria and standard will be subject to review and revision as necessary.

Regardless of the source of raw materials from which produced, "xylene" (also known as "xylol" or "dimethylbenzene") refers to any one of or combination of the isomers of xylene: ortho-, meta-, or para-dimethylbenzene. The term "xylene" will be used throughout this document. "Exposure to xylene" is defined as exposure above half the recommended time-weighted average environmental limit. If "exposure" to other chemicals also occurs, for example from contamination of xylene with benzene, provisions of any applicable standard for the other chemicals shall also be followed.

Section 1 - Environmental (Workplace Air)

(a) Concentration

Occupational exposure to xylene shall be controlled so that workers are not exposed to xylene at a concentration greater than 100 parts per million parts of air by volume (approximately 434 milligrams per cubic meter of air) determined as a time-weighted average (TWA) exposure for up to a 10-hour workday, 40-hour workweek, with a ceiling concentration of 200 parts per million parts of air by volume (approximately 868 milligrams per cubic meter of air) as determined by a sampling period of 10 minutes.

(b) Sampling, Collection, and Analysis

Procedures for collection and analysis of environmental samples shall be as provided in Appendices I and II or by any method shown to be equivalent in accuracy, precision, and sensitivity to the method specified.

Section 2 - Medical

Comprehensive preplacement and biennial (every 2 years) medical examinations should be provided for all workers subject to "exposure to xylene." The history and examination should be directed toward, but not limited to, the incidence of headaches, nausea or other gastrointestinal disturbance, dizziness, and of alcohol consumption. Particular attention should be focused on complaints and evidence of eye, mucous membrane, or skin irritation. Laboratory tests recommended at the time of the biennial examination include a complete blood count, a routine urinalysis, and appropriate liver function tests.

Section 3 - Labeling (Posting)

(a) Labeling

Containers of xylene shall carry a label stating:

XYLENE
(XYLOL, DIMETHYLBENZENE)

WARNING! FLAMMABLE
HARMFUL IF INHALED OR SWALLOWED
IRRITATING TO SKIN OR EYES

Keep away from heat, sparks, and open flame.
In case of fire, use foam, dry chemical, or CO2.
Avoid breathing vapor.
Avoid contact with eyes, skin, and clothing.
Keep container closed.
Use with adequate ventilation.
Do not use for washing hands, floors, or equipment.

First Aid: In case of eye or skin contact with liquid xylene, wash with plenty of water. If eye contact, call a physician. If swallowed, call a physician. Do not attempt to induce vomiting.

(b) Posting

Areas where there is occupational exposure to xylene shall be posted with a sign reading:

XYLENE

WARNING! FLAMMABLE
HARMFUL IF INHALED OR SWALLOWED
IRRITATING TO SKIN OR EYES

Keep out heat, sparks, or open flames.
No smoking permitted.
In case of fire, use fire extinguishers. (Give location)
Avoid breathing vapor.
Avoid contact with skin, eyes, and clothing.
Provide adequate ventilation.

This warning sign shall be printed both in English and in the predominant language of non-English-speaking workers, unless they are other-

wise trained and informed of the hazardous areas. All illiterate workers shall receive such training.

Section 4 - Personal Protective Equipment

(a) Protective Clothing

(1) Chemical safety goggles, face shields, or safety glasses with side shields shall be provided by the employer and shall be worn in any operation in which xylene may splash into the eyes.

(2) Appropriate protective clothing, including gloves, aprons, suits, boots, or face shields, shall be worn where needed to prevent repeated or prolonged skin contact.

(b) Respiratory Protection

(1) Engineering controls shall be used wherever feasible to maintain xylene concentrations below the prescribed limits. Such control equipment shall be sparkproof. Compliance with the permissible exposure limit may not be achieved by the use of respirators except:

(A) During the time period necessary to install or test the required engineering controls.

(B) For nonroutine operations such as a brief exposure to concentrations in excess of the permissible exposure limit as a result of maintenance or repair activities.

(C) During emergencies when air concentrations of xylene may exceed the permissible limit.

(2) When a respirator is permitted by paragraph (b)(1) of this Section, it shall be selected and used pursuant to the following requirements:

(A) For the purpose of determining the type of respirator to be used, the employer shall measure, when possible, the atmospheric concentration of xylene in the workplace initially and thereafter whenever process, worksite, climate, or control changes occur which are likely to increase the xylene concentrations; this requirement shall not apply when only atmosphere-supplying positive pressure respirators will be used. The employer shall ensure that no worker is being exposed to xylene in excess of the standard because of improper respirator selection, fit, use, or maintenance.

(B) A respiratory protection program meeting the requirements of 29 CFR 1910.134 as amended shall be established and enforced by the employer.

(C) The employer shall provide respirators in accordance with Table I-1 below and shall ensure that the employee uses the respirator provided.

(D) Respiratory protective devices described in Table I-1 shall be those approved under the provisions of 30 CFR 11 as amended.

(E) Respirators specified for use in higher concentrations of xylene may be used in atmospheres of lower concentrations.

(F) The employer shall ensure that respirators are adequately cleaned, and that employees are instructed on the use of respirators assigned to them, and on how to test for leakage.

(G) Where an emergency may develop which could result in employee injury from overexposure to xylene, the employer shall provide respiratory protection as listed in Table I-1.

TABLE I-1

RESPIRATOR SELECTION GUIDE FOR PROTECTION AGAINST XYLENE

Multiples of TWA Limit	Respirator Type
Less than 10x	Chemical cartridge respirator with full facepiece and organic vapor cartridge(s).
Less than 50x	Gas mask (full facepiece) with a chin-style organic vapor canister.
Less than 100x	(1) Gas mask (full facepiece) with front or back mounted organic vapor canister. (2) Type C supplied air respirator with a full facepiece operated in the demand (negative pressure) mode. (3) Type C supplied air respirator with a full facepiece operated in the pressure-demand (positive pressure) or continuous flow mode. (4) Self-contained breathing apparatus with a full facepiece operated in the demand (negative pressure) mode. (5) Combination Type C supplied air respirator with full facepiece operated in the demand (negative pressure) or continuous flow mode and an auxiliary self-contained air supply operated in the demand (negative pressure) mode.
100x or more CAUTION! The lower explosive limit is approximately 11,000 ppm	(1) Self-contained breathing apparatus with a full facepiece operated in the pressure-demand (positive pressure) mode. (2) Combination Type C supplied air respirator with full facepiece operated in the pressure-demand (positive pressure) or continuous flow mode and an auxiliary self-contained air supply operated in the pressure-demand (positive pressure) mode.
Unknown concentration	Self-contained breathing apparatus with a full facepiece operated in the pressure-demand (positive pressure) mode.
Escape	(1) Gas mask (full facepiece) with chin style or front or back mounted organic vapor canister. (2) Self-contained breathing apparatus with full facepiece operating either in the demand (negative pressure) or pressure demand (positive pressure) mode.

Section 5 - Informing Employees of Hazards from Xylene

(a) Each employee exposed to xylene shall be informed at the beginning of his employment or assignment to a xylene area of the hazards, relevant symptoms, appropriate emergency procedures, and proper conditions and precautions for safe use or exposure. Each employee shall be instructed as to the availability of such information which shall be kept on file. Information kept on file shall include that prescribed in (b) below and shall be accessible to the worker at each place of employment where xylene is involved in unit processes and operations.

(b) Information as specified in Appendix III shall be recorded on US Department of Labor Form OSHA-20, "Material Safety Data Sheet," or on a similar form approved by the Occupational Safety and Health Administration, US Department of Labor.

Section 6 - Work Practices

(a) Emergency Procedures

For all work areas in which there is a reasonable potential for emergencies, procedures as specified below, as well as any other procedures appropriate for a specific operation or process, shall be formulated in advance and employees shall be instructed in their implementation.

(1) Procedures shall include prearranged plans for obtaining emergency medical care and for necessary transportation of injured workers.

(2) Firefighting procedures shall be established and implemented. These shall include procedures for emergencies involving release of xylene vapor. In case of fire, xylene sources shall be shut off

or removed. Containers shall be removed or cooled with water spray. Chemical foam, carbon dioxide, or dry chemicals should be used for fighting xylene fires, and proper respiratory protection and protective clothing shall be worn.

(3) Approved eye, skin, and respiratory protection as specified in Section 4 shall be used by personnel essential to emergency operations.

(4) Nonessential employees shall be evacuated from exposure areas during emergencies. Perimeters of areas of hazardous exposures shall be delineated, posted, and secured.

(5) Personnel properly trained in the procedures and adequately protected against the attendant hazards shall shut off sources of xylene, clean up spills, and immediately repair leaks.

(b) Engineering controls such as process enclosure or local exhaust ventilation shall be used to maintain xylene concentrations within the recommended environmental limits. All such control equipment shall be sparkproof. Ventilation systems shall be designed to prevent the accumulation or recirculation of xylene in the workplace and to effectively remove xylene from the breathing zones of exposed workmen. Exhaust ventilation systems discharging to outside air must conform with applicable local, state, and federal air pollution regulations. Ventilation systems shall be subject to regular preventive maintenance and cleaning to ensure maximum effectiveness, which shall be verified by periodic airflow measurements.

(c) Containers of xylene shall be kept tightly closed at all times when not in use. Containers shall be stored in accordance with the

provisions of 29 CFR 1910, and shall be protected from heat, corrosion, mechanical damage, and sources of ignition.

(d) Washing of hands, equipment, or structures with xylene shall be prohibited.

(e) Any spills of xylene shall be promptly cleaned up.

(f) Prior to maintenance work, sources of xylene and xylene vapor shall be eliminated to the extent feasible. If concentrations below the workplace air limit cannot be assured, respiratory protective equipment shall be used during such maintenance work.

(g) All metal dispensing containers shall be properly grounded.

Section 7 - Sanitation

(a) Food Facilities

Food preparation, dispensing (including vending machines), and eating should be prohibited in xylene work areas.

(b) Smoking

Smoking shall not be permitted in areas where xylene is used, transferred, stored, or manufactured.

Section 8 - Monitoring and Recordkeeping

Workroom areas shall not be considered to have xylene exposure if environmental levels, as determined on the basis of a professional industrial hygiene survey or by the judgment of the compliance officer, do not exceed half of the recommended time-weighted average limit. Records of these surveys, including the basis for concluding that air levels are at or

below half of the time-weighted average limit, shall be maintained until a new survey is conducted. Surveys shall be repeated when any process change indicates a need for reevaluation or at the judgment of the compliance officer. Requirements set forth below apply to areas in which there is xylene exposure.

Employers shall maintain records of environmental exposures to xylene based upon the following sampling and recording schedules:

(a) In all monitoring, samples representative of the exposure in the breathing zone of employees shall be collected. An adequate number of samples shall be collected to permit construction of a time-weighted average (TWA) exposure for every operation or process. The minimum number of representative TWA determinations for an operation or process shall be based on the number of workers exposed as provided in Table I-2.

TABLE I-2

SAMPLING SCHEDULE

Number of Employees Exposed	Number of TWA Determinations
1-20	50% of the total number of workers
21-100	10 plus 25% of the excess over 20 workers
over 100	30 plus 5% of the excess over 100 workers

(b) The first environmental samples shall be completed within 6 months of the promulgation of a standard incorporating these recommendations.

(c) Environmental samples shall be taken within 30 days after installation of a new process or process change.

(d) Samples shall be collected at least quarterly in accordance with Appendix I for the evaluation of the work environment with respect to the recommended standard.

(e) Environmental monitoring of an operation or process shall be repeated at 30-day intervals when the xylene concentration has been found to exceed the recommended environmental standard. In such cases, suitable controls shall be initiated and monitoring shall continue at 30-day intervals until two consecutive surveys indicate the adequacy of these controls.

(f) Sampling records shall be maintained so that exposure information is available for individual employees and shall indicate the type of personal protective devices, if any, in use at the time of sampling. Each employee shall be able to obtain information on his own environmental exposure.

VII. APPENDIX I

SAMPLING FOR XYLENE

The sampling and analytical methods presented in Appendices I and II are based on those described by White et al, [95] Kupel and White, [130] and in Method No. 127 of the Physical and Chemical Analysis Branch of NIOSH. [100]

Atmospheric Sampling

Breathing zone samples representative of the individual worker's exposure shall be collected. A description of sampling location and conditions, equipment used, time and rate of sampling, and any other pertinent information shall be recorded at the time of sample collection. Enough samples shall be collected to permit calculation of a time-weighted average (TWA) exposure for every operation or location in which there is exposure to xylene.

(a) Equipment

The sampling train consists of a charcoal tube and vacuum pump.

(1) Charcoal tubes: Glass tubes, with both ends flame-sealed, 7-cm long with a 6-mm OD and a 4-mm ID, containing 2 sections of 20/40 mesh activated charcoal separated by a 2-mm portion of polyurethane foam. The primary section contains 100 mg of charcoal, the backup section, 50 mg. A 3-mm portion of polyurethane foam is placed between the outlet end of the tube and backup section. A plug of glass wool is placed in front of the primary section. The pressure drop across the tube must be

less than 1 inch of mercury at a flowrate of 1 liter/min. Tubes with the above specifications are commercially available.

(2) Pump: A battery-operated pump, complete with clip for attachment to the worker's belt, capable of operation at 1 liter/min or less.

(b) Calibration

Since the accuracy of an analysis can be no greater than the accuracy of the volume of air which is measured, the accurate calibration of a sampling pump is essential to the correct interpretation of the volume indicated. The frequency of calibration is dependent on the use, care, and handling to which the pump is subjected. Pumps should also be recalibrated if they have been misused or if they have just been repaired or received from a manufacturer. If the pump receives hard usage, more frequent calibration may be necessary. Regardless of use, maintenance and calibration should be performed on a regular schedule and records of these kept.

Ordinarily, pumps should be calibrated in the laboratory both before they are used in the field and after they have been used to collect a large number of field samples. The accuracy of calibration is dependent on the type of instrument used as a reference. The choice of calibration instrument will depend largely upon where the calibration is to be performed. For laboratory testing, primary standards such as a spirometer or soapbubble meter are recommended, although other standard calibrating instruments such as a wet test meter or dry gas meter can be used. The actual setups will be similar for all instruments.

Instructions for calibration with the soapbubble meter follow. If another calibration device is selected, equivalent procedures should be

used. The calibration setup for personal sampling pumps with a charcoal tube is shown in Figure X-1. Since the flowrate given by a pump is dependent on the pressure drop of the sampling device, in this case a charcoal tube, the pump must be calibrated while operating with a representative charcoal tube in line.

(1) The voltage of the pump battery is checked with a voltmeter to assure adequate voltage for calibration. The battery is charged if necessary.

(2) The tips of a charcoal tube are broken to produce openings of at least 2 mm in diameter.

(3) The sampling train is assembled as shown in Figure X-1.

(4) The pump is turned on and the inside of the soapbubble meter is moistened by immersing the buret in the soap solution and drawing bubbles up the inside until they are able to travel the entire buret length without bursting.

(5) The pump rotameter is adjusted to provide the desired flowrate.

(6) The water manometer is checked to insure that the pressure drop across the sampling train does not exceed 13 inches of water at 1 liter/min or 2.5 inches of water at 200 ml/min.

(7) A soapbubble is started up the buret and the time it takes the bubble to move from one calibration mark to another is measured with a stopwatch.

(8) The procedure in (7) above is repeated at least twice, the results averaged, and the flowrate calculated by dividing the volume between the preselected marks by the time required for the soapbubble to

traverse the distance. If, for the pump being calibrated, the volume of air sampled is calculated as the product of the number of strokes times a stroke factor (given in units of volume/stroke), the stroke factor is the quotient of the volume between the 2 preselected marks divided by the number of strokes.

(9) Data for the calibration include the volume measured, elapsed time or number of strokes, pressure drop, air temperature, atmospheric pressure, serial number of the pump, date, and name of the person performing the calibration.

(c) Sampling Procedure

(1) Both ends of the charcoal tube are broken to provide openings of at least 2 mm, which is half the ID of the tube. A smaller opening causes a limiting orifice effect which reduces the flow through the tube. The smaller section of charcoal in the tube is used as a backup section and therefore is placed nearest the sampling pump. Tubing is used to connect the back of the tube to the pump, but tubing must never be put in front of the charcoal tube. The tube is supported in a vertical position in the worker's breathing zone.

(2) A maximum of 15 liters of air is sampled at a flowrate of 50-1,000 ml/min. For the determination of ceiling concentrations the sampling time is 10 minutes. For the determination of 8-hour time-weighted average concentrations 2 4-hour or 4 2-hour samples are suggested.

(3) The temperature and pressure of the atmosphere being sampled is measured and recorded.

(4) One charcoal tube is treated in the same manner as the sample tubes (break, seal, ship) with the exception that no air is drawn

through it. This tube serves as a blank.

(5) Immediately after sampling, charcoal tubes are capped with plastic caps. Under no circumstances should rubber caps be used. To minimize breakage during transport, capped tubes should be tightly packed in a shipping container. Bulk samples and charcoal tubes should be shipped separately.

VIII. APPENDIX II

ANALYTICAL METHOD FOR XYLENE

Principle of the Method

Xylene vapor trapped on charcoal from a known volume of air is desorbed with carbon disulfide. An aliquot of the desorbed sample is injected into a gas chromatograph. The area of the resulting peak is determined and compared with areas obtained from injection of standards.

Range and Sensitivity

The lower limit of detection of the analytical procedure was found to be less than 12 μg/sample.

Interferences

When the amount of water in the air is so great that condensation actually occurs in the tube, organic vapors will not be trapped. Preliminary experiments indicate that high humidity severely decreases the amount of organic vapor which can be collected before breakthrough of the primary adsorbing section occurs. The capacity of the charcoal tube for xylene may also be reduced by the presence of another organic vapor in high concentration.

Any compound which has about the same retention time as one of the xylene isomers at the gas chromatographic conditions described in this method will interfere with the analysis. This type of interference can be

overcome by changing the operating conditions of the instrument, usually the column and/or the column temperature.

Precision and Accuracy

In a collaborative test, [101] the total relative error in the range of 60-200 ppm (260-870 mg/cu m) was 9.5%. At approximately 5 ppm (20 mg/cu m) this error was 13%.

Advantages and Disadvantages of the Method

The sampling device is small, portable, and involves no liquids. Interferences are minimal and most can be eliminated by altering the chromatographic conditions. The analysis is accomplished using a rapid instrumental method, which can also be used for the simultaneous analysis of 2 or more solvents present in the same sample by changing gas chromatographic conditions from isothermal to a temperature-programmed mode of operation.

One disadvantage of the method is that the amount of sample which can be taken is limited by the number of milligrams that the tube will hold before overloading. When the sample value obtained for the backup section of the charcoal trap exceeds 20% of that found on the front section, the possibility of sample loss exists.

The precision of the method is limited by the reproducibility of the pressure drop across the tubes. This drop will affect the flow rate and cause the volume to be imprecise, because the pump is usually calibrated for one tube only.

Apparatus

(a) Gas chromatograph equipped with a flame ionization detector.

(b) Column (20 ft x 1/8 in) with 10% FFAP stationary phase on 80/100 mesh acid washed DMCS Chromosorb W solid support. Other columns which achieve the desired separation may be used.

(c) A mechanical or electronic integrator or a recorder and some method for determining peak area.

(d) Small glass-stoppered test tubes or equivalent.

(e) Syringes: 10-μl, and convenient sizes for preparation of standards.

Reagents

(a) Carbon disulfide, chromatographic quality.

(b) Xylene, preferably having an isomer distribution close to that of the sample.

(c) Bureau of Mines Grade A helium.

(d) Prepurified hydrogen.

(e) Filtered compressed air.

Analysis of Samples

All glassware used for the laboratory analysis should be washed in detergent followed by tap and distilled water rinses.

(a) Preparation: Each charcoal tube, including the blank from field samples, is scored with a file and broken open in front of the first section of charcoal. The glass wool is removed and discarded. The

charcoal in the first (larger) section is transferred to a small stoppered test tube. The foam separating section is removed and discarded, and the second section of charcoal is transferred to another test tube. The 2 charcoal sections are then analyzed separately.

(b) Desorption: Prior to analysis, 0.5 ml of carbon disulfide is pipetted into each test tube to desorb the xylene from the charcoal. Desorption is complete in 30 minutes if the sample is stirred occasionally.

EXTREME CAUTION MUST BE EXERCISED AT ALL TIMES WHEN USING CARBON DISULFIDE BECAUSE OF ITS HIGH TOXICITY AND FIRE AND EXPLOSION HAZARDS. IT CAN BE IGNITED BY HOT STEAM PIPES. ALL WORK WITH CARBON DISULFIDE MUST BE PERFORMED UNDER AN EXHAUST HOOD.

(c) Typical gas chromatographic operating conditions:

(1) 40 cc/min (70 psig) helium carrier gas flow.

(2) 65 cc/min (24 psig) hydrogen gas flow to detector.

(3) 500 cc/min (50 psig) airflow to detector.

(4) 200 C injector temperature.

(5) 200 C manifold temperature (detector).

(6) 110 C isothermal oven or column temperature.

(d) Injection: The first step in the analysis is the injection of the sample into the gas chromatograph. The solvent flush injection technique is employed. This eliminates difficulties arising from blowback or distillation within the syringe needle, thus increasing the accuracy and reproducibility of the injected sample volume. The 10.0-µl syringe is first flushed with solvent several times to wet the barrel and plunger, then 3.0 µl of solvent are drawn into the syringe. Next, the needle is removed from the solvent and the plunger is pulled back about 0.2 µl to

separate the solvent flush from the sample with an air pocket to be used as a marker. The needle is then immersed in the sample and a 5.0-μl aliquot is withdrawn, taking into consideration the volume of the needle, since the sample in the needle will be completely injected. After the needle is removed from the sample and prior to injection in the gas chromatograph, the plunger is pulled back a short distance to minimize sample evaporation from the needle tip. Duplicate injections should be made of each sample and of the standard. No more than a 3% difference should result in the peak areas that are recorded.

(e) Measurement of area: The areas of the sample peaks are measured by an electronic integrator or some other suitable form of area measurement and preliminary sample results are read from a standard curve prepared as outlined below. The integration of the signals from all three xylene isomers is recommended.

Determination of Desorption Efficiency

The desorption efficiency of a particular compound can vary from one laboratory to another and also from one batch of charcoal to another. Thus it is necessary to determine at least once the percentage of xylene that is removed in the desorption process. This procedure should be repeated for each new batch of charcoal used. The Physical and Chemical Analysis Branch of NIOSH has found desorption efficiencies for xylene varying from 92-99% between batches.

Activated charcoal equivalent to the amount in the first section of the sampling tube (100 mg) is measured into a 5-cm, 4-mm ID glass tube, flame-sealed at one end. This charcoal must be from the same batch as that

used in obtaining the samples and can be obtained from unused charcoal tubes. The open end is capped with Parafilm or equivalent. A known amount of xylene is injected directly into the activated charcoal with a microliter syringe, and the tube is capped with more Parafilm or equivalent. A known amount injected is usually equivalent to that present in a 10-liter sample at a concentration equal to the federal limit of 100 ppm, or about 4.3 mg.

At least 5 tubes are prepared in this manner and allowed to stand overnight or longer to assure complete adsorption of the xylene onto the charcoal. These 5 tubes are referred to as the samples. A parallel blank tube should be treated in the same manner except that no xylene is added to it. The sample and blank tubes are desorbed and analyzed in exactly the same manner as the sampling tube described for unknown air samples.

Two or 3 standards are prepared by injecting the same volume of xylene into 0.5 ml of carbon disulfide with the same syringe used in the preparation of the sample. These are analyzed with the samples.

The desorption efficiency equals the difference between the average peak area of the samples and the peak area of the blank divided by the average peak area of the standards, or:

$$\text{desorption efficiency} = \frac{\text{area sample} - \text{area blank}}{\text{area standard}}$$

Calibration and Standards

It is convenient to express the concentration of standards in terms of mg/0.5 ml carbon disulfide, because samples are desorbed in this amount of carbon disulfide.. The density of the xylene is used to convert

milligrams into microliters for easy measurement with a microliter syringe. A series of standards, varying in concentration over the range of interest, is prepared and analyzed under the same gas chromatographic conditions and during the same time period as the unknown samples. Curves are established by plotting concentration in mg/0.5 ml versus peak area.

Calculations

The weight in mg, corresponding to the total peak area, is read from the standard curve. No volume corrections are needed, because the standard curve is based on mg/0.5 ml carbon disulfide and the volume of sample injected is identical to the volume of the standards injected.

Corrections for the blank from the field sampling are made for each sample by subtracting the amounts of xylene found on the front and back sections of the blank from the amounts found in the respective sections of the sample:

Corrected amount = amount on sample - amount on blank

The corrected amounts present in the front and backup sections of the same sample tube are added to determine the total amount of xylene in the sample. This total amount is divided by the desorption efficiency to obtain the adjusted total amount of xylene in the sample.

The volume of air sampled is converted to standard conditions of 25 C and 760 mm Hg:

$$\text{Adjusted total amount} = \frac{\text{total amount}}{\text{desorption efficiency}}$$

The concentration of xylene in the air sampled, expressed in mg/cu m (which is numerically equal to μg/liter of air) is given by the quotient of the adjusted amount in μg divided by the volume of air sampled in liters:

$$\text{concentration } (\mu g/\text{liter}) = \frac{\text{adjusted amount } (\mu g)}{\text{volume (liters)}}$$

Another method of expressing concentration is ppm:

$$\text{concentration (ppm)} = \text{concentration } (\mu g) \times \frac{24.45}{106} \times \frac{760}{P} \times \frac{(T + 273)}{298}$$

where:

24.45 = molar volume (liter/mole) at 25 C and 760 Torr

106 = molecular weight of xylene (g/mole)

760 = standard pressure

P = pressure (Torr) of air sampled

T = temperature (degrees C) of air sampled

298 = standard temperature (degrees K)

or

$$\text{concentration (ppm)} = \text{concentration } (\mu g) \times \frac{0.588\ (T + 273)}{P}$$