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IX. APPENDIX I

METHOD FOR SAMPLING EPICHLOROHYDRIN IN AIR

The following sampling method is adapted from those described by White et al [89] and in Method No. S118 of the Physical and Chemical Analysis Branch of NIOSH. [90]

Atmospheric Sampling

Collect breathing zone or personal samples representative of the individual employee's exposure. At the time of sample collection, record a description of sampling location and conditions, equipment used, time and rate of sampling, and any other pertinent information. Collect enough samples to permit calculation of a TWA exposure for every operation or location in which there is exposure to epichlorohydrin.

(a) Equipment

The sampling train consists of a charcoal tube and a 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 two 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 the backup section. A plug of glass wool is placed in front of the primary section. Tubes with the above specifications are commercially available.

(2) Pump: A battery-operated pump, complete with clip for

attachment to the employee's belt, capable of operating at 200 ml/minute or less.

(b) Calibration

The accurate calibration of a sampling pump is essential to the correct interpretation of the volume sampled. 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. Maintenance and calibration should be performed on a regular schedule and records of these should be 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, a soapbubble meter is recommended, although other standard calibrating instruments 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 XIII-2. Since the flowrate given by a pump is dependent on the pressure drop across the sampling device, in this case a charcoal tube, the pump must be calibrated while operating with a representative charcoal tube in line.

(1) Check the voltage of the pump battery with a voltmeter to assure adequate voltage for calibration. Charge the battery if necessary.

(2) Break the tips of a charcoal tube to produce openings of at least 2 mm in diameter.

(3) Assemble the sampling train as shown in Figure XIII-2.

(4) Turn on the pump and moisten the inside of the soapbubble meter by immersing the buret in the soap solution. Draw bubbles up the inside until they are able to travel the entire buret length without bursting.

(5) Adjust the pump flowmeter to provide the desired flowrate.

(6) Check the water manometer to ensure that the pressure drop across the sampling train does not exceed 2.5 inches of water at 200 ml/minute.

(7) Start a soapbubble up the buret and measure with a stopwatch the time it takes the bubble to move from one calibration mark to another.

(8) Repeat the procedure in (7) above at least three times, average the results and calculate the flowrate 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 two preselected marks divided by the number of strokes.

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

(c) Sampling Procedure

(1) Break both ends of the charcoal tube 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. Use tubing 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 employee's breathing zone.

(2) Sample a maximum of 20 liters of air at a flowrate of 200 ml/minute. For the determination of ceiling concentrations the sampling time is 15 minutes.

(3) Measure and record the temperature and pressure of the atmosphere being sampled.

(4) Treat at least one charcoal tube in the same manner as the sample tubes (break, seal, and ship), except draw no air through it. This tube serves as a blank.

(5) Immediately after samples are collected, cap the charcoal tubes with plastic caps. Do not use rubber caps. To minimize breakage during transport, pack capped tubes tightly in a shipping container.

X. APPENDIX II

ANALYTICAL METHOD FOR EPICHLOROHYDRIN

The following analytical method is adapted from those described by White et al, [89] by R Hill (written communication, June 1976), and in Method No. S118 of the Physical and Chemical Analysis Branch of NIOSH. [90]

Principle of the Method

Epichlorohydrin 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 those obtained from injection of standards.

Range and Sensitivity

This method was developed to analyze epichlorohydrin over the range of 11.7-43.1 mg/cu m at an atmospheric temperature and pressure of 23 C and 765 mmHg. [90] For a 20-liter sample, the useful range of this method was 2-60 mg/cu m at a detector sensitivity that gives nearly full deflection on the strip chart recorder for a 1-mg sample. The method is capable of measuring smaller amounts down to 50 ppb, as demonstrated by Hill (written communication, June 1976). Desorption efficiency must be determined over the range used.

The upper limit of the range of the method is dependent on the absorptive capacity of the charcoal tube. This capacity varies with the

concentrations of epichlorohydrin and other substances in the air. The first section of the charcoal tube held at least 2 mg of epichlorohydrin when a test atmosphere containing 43.1 mg/cu m of epichlorohydrin in air was sampled at 0.185 liter/minute for 240 minutes; at that time, the concentration of epichlorohydrin in the effluent was less than 5% of that in the influent.

Interferences

Any compound which has about the same retention time as epichlorohydrin under 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, the column temperature, or both. Epichlorohydrin appears to be stable under 80% humidity conditions.

Precision and Accuracy

In a collaborative test, the total relative error at 5 ppm epichlorohydrin (18.9 mg/cu m) was 0.7%. However, definition of total relative error was not given. Hill (written communication, June 1974) has determined that it is possible to analyze down to 0.25 $\mu\text{g/liter}$. Samples were collected from an 80% relative humidity atmosphere containing epichlorohydrin. Following a storage period of 14 days, the charcoal tubes were analyzed. The results of the analyses of two sets were 8.07 ± 0.25 and $11.64 \pm 0.32 \mu\text{g/tube}$. Hill found that the percent relative standard deviation of each set, which reflects the precision of the total sampling

and analytical procedure, was 7.21 and 5.33%, respectively. Under these conditions, the desorption efficiency was estimated to be 82.7%.

Apparatus

- (a) Gas chromatograph equipped with a flame ionization detector.
- (b) Stainless steel column (12 feet x 1/8 inch) with 10% carbowax 20 M stationary phase on 80/100 mesh acid-washed DMCS Chromosorb W solid support. Another column that can be used is stainless steel (6 feet x 2-mm ID) with 80/100 mesh Chromosorb 101. Other columns which achieve the desired separation may be used.
- (c) A mechanical or electronic integrator or a recorder for determining peak area.
- (d) Small glass-stoppered test tubes or equivalent.
- (e) A 10- μ l syringe and other conveniently sized syringes for preparation of the standards.
- (f) Delivery pipets, 1.0-ml pipets.

Reagents

- (a) Carbon disulfide, chromatographic quality.
- (b) Epichlorohydrin, reagent grade.
- (c) Helium, Bureau of Mines Grade.
- (d) Hydrogen, purified.
- (e) Compressed air, filtered.

Analysis of Samples

All glassware used for the laboratory analysis should be washed in detergent and rinsed with tap and distilled water.

(a) Preparation: Score each charcoal tube, including the blank from field samples, with a file and break open in front of the first section of charcoal. Remove and discard the glass wool. Transfer the charcoal in the first (larger) section to a small stoppered test tube. Remove and discard the foam separating sections and transfer the second section of charcoal to another test tube. Analyze the two charcoal sections separately.

(b) Desorption: Prior to analysis, pipet 1.0 ml of carbon disulfide into each test tube to desorb the epichlorohydrin 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 for Chromosorb 101 column:

- (1) 20 ml/minute helium flowrate.
- (3) 200 C injector temperature.
- (3) 230 C manifold temperature (detector).
- (4) 135 C isothermal oven or column temperature.

(d) Injection: The first step in the analysis is the injection of the sample into the gas chromatograph. Employ the solvent flush injection technique. This eliminates difficulties arising from blowback or distillation within the syringe needle, thus increasing the accuracy and reproducibility of the injected sample volume. First, flush the 10.0- μ l syringe with solvent several times to wet the barrel and plunger, then draw 3.0 μ l of solvent into the syringe. Next, remove the needle from the carbon disulfide and pull the plunger back about 0.2 μ l to separate the solvent flush from the sample with an air pocket to be used as a marker. Immerse the needle in the sample and withdraw a 5.0- μ l portion, 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, pull the plunger back a short distance to minimize sample evaporation from the tip. Make duplicate injections of each sample and of the standard. No more than a 3% difference between the peak areas of the similar samples should be accepted as a valid result.

(e) Measurement of area: The areas of the sample peaks are measured by electronic integration or some other suitable method of area measurement. Preliminary sample results are read from a standard curve prepared as outlined below.

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

epichlorohydrin that is removed in the desorption process. Repeat this procedure for each new batch of charcoal used.

Place the same amount of activated charcoal as in the first section of the sampling tube (100 mg) into a 5-cm, 4-mm ID glass tube; flame seal at one end. This charcoal must be from the same batch as that used in sampling and can be obtained from unused charcoal tubes. Cap the open end with Parafilm or equivalent. Inject a known amount of hexane solution containing 94.5 mg/ml of epichlorohydrin directly into the activated charcoal with a microliter syringe and cap the tube with more Parafilm or equivalent.

Prepare at least five tubes in this manner and allow to stand overnight or longer to ensure complete adsorption of the epichlorohydrin on the charcoal. These five tubes are referred to as the samples. Treat a parallel blank tube in the same manner, except add no epichlorohydrin to it. Desorb and analyze the sample and blank tubes in exactly the same manner as the sampling tube described for unknown air samples.

Prepare two or three standards by injecting the same volume of epichlorohydrin into 1.0 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 that of the blank divided by the average peak area of the standards, or:

$$\text{desorption efficiency} = \frac{\text{average weight recovered (mg)}}{\text{weight added (mg)}}$$

Calibration and Standards

It is convenient to express the concentration of standards in terms of mg/ml of carbon disulfide because samples are desorbed in 1 ml of carbon disulfide. Use the density of epichlorohydrin to convert milligrams into microliters for easy measurement with a microliter syringe. Prepare a series of standards varying in concentration over the range of interest and then analyze under the same gas-liquid chromatographic conditions and during the same time period as the unknown samples. Prepare standard curves by plotting concentration in mg/ml versus peak area.

Calculations

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

Make corrections for the blank from the field sampling for each sample by subtracting the amounts of epichlorohydrin found on the front and back sections of the blank from the amounts found in the respective sections of the sample:

$$\text{corrected amount} = \text{amount on sample} - \text{amount on blank}$$

Add the corrected amounts present in the front and in the backup sections of the same sample tube to determine the total amount of epichlorohydrin in the sample. Divide this total amount by the desorption

efficiency to obtain the adjusted total amount of epichlorohydrin in the sample:

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

The concentration of epichlorohydrin in the air sampled, expressed in mg/cu m, is given by the quotient of the adjusted amount in mg divided by the volume of air sampled in cu m:

$$\text{concentration (mg/cu m)} = \frac{\text{adjusted amount (mg)}}{\text{volume (cu m)}}$$

Another method of expressing concentration is ppm:

$$\text{concentration (ppm)} = \text{concentration (mg/cu m)} \times \frac{24.45}{\text{MW}} \times \frac{760}{\text{P}} \times \frac{(\text{T} + 273)}{298}$$

where:

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

760 = standard pressure

P = pressure (mmHg) of air sampled

T = temperature (degrees C) of air sampled

MW = molecular weight of epichlorohydrin (g/mole)

298 = standard temperature (degrees K)