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IX. APPENDIX I

AIR SAMPLING METHOD

The sampling method for airborne carbaryl presented in Appendix I is adapted from a validated NIOSH method. [97]

General Requirements

To evaluate conformance with the standard, collect breathing-zone samples representative of the individual worker's exposure. Record the following on all sampling data sheets:

(a) The date and time of sample collection.

(b) Sampling duration.

(c) Volumetric flowrate of sampling.

(d) Worker's name and job title and description of work station.

(e) Temperature and atmospheric pressure.

(f) Other pertinent information.

Air Sampling

(a) Collect breathing-zone samples as close as practicable to the employee's face, without interfering with the employee's freedom of movement, to characterize the exposure from each job or specific operation dealing with manufacture, formulation, or application of carbaryl.

Collect eight 1-hour breathing-zone samples representative of worker exposure in each operation. Sampling flowrates should be checked frequently. When filters become clogged so that airflow is too restricted, change the filters and initiate the collection of new samples.

(b) Collect samples using a portable sampling pump whose flow can be determined to an accuracy of ± 5% at 1.5 liters/minute. Connect the pump to the filter unit, which consists of a glass-fiber filter (Type A, 37 mm in diameter) mounted in a polystyrene 37-mm, two-piece cassette holder and supported by a backup pad. Use only membrane filters which are free of organic binders. Do not use Tenite filter holders.

(c) Operate the sampler at a known flowrate of 1.5 liters/minute, and record the total sampling time. A sample size of 90 liters is recommended.

(d) Remove the glass-fiber filter from the cassette filter holder within 1 hour of sampling and place it in a clean screwcap bottle. Handle the filter only with clean tweezers. The bottle caps should be lined with Teflon for proper seal. These bottles (a 45-mm tissue-sample holder is satisfactory for shipping) are also used to contain the solution during analysis.

(e) With each batch of 10 samples, submit 1 filter from the same lot used for sample collection, subjecting it to exactly the same handling as the samples except that no air is drawn through it. Label this as a blank.

(f) Take the screwcap bottles which contain the samples and ship them in suitable containers designed to prevent damage while in transit.

Calibration of Sampling Trains

The accurate calibration of a sampling pump is essential for 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. In addition, pumps should be recalibrated if they have been subjected to misuse 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, a 1- or 2-liter buret or wet-test meter is recommended, although other standard calibrating instruments, such as spirometer, Marriott's bottle, or dry-gas meter, can be used.

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 glass-fiber membrane filter is shown in Figure XIII-1. Since the flowrate given by a pump is dependent on the pressure drop of the sampling device, in this case a membrane filter, the pump must be calibrated while operating with a representative filter and backup pad in line.

(a) While the pump is running, check the voltage of the pump battery with a voltmeter to assure adequate voltage for calibration.

Charge the battery if necessary.

(b) Place the glass-fiber membrane filter with backup pad in the filter cassette.

(c) Assemble the sampling train as shown in Figure XIII-1.

(d) Turn the pump on and moisten the inside of the soapbubble meter 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.

(e) Adjust the pump rotameter to provide a flowrate of 1.5 liters/minute.

(f) Start a soapbubble up the buret and measure with a stopwatch the time it takes the bubble to pass through a minimum of 1.0 liter.

(g) Repeat the procedure in (f) 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.

(h) Record the following calibration data: volume measured, elapsed time, air temperature, atmospheric pressure, serial number of the pump, date, and name of the person performing the calibration.

(i) Corrections to the flowrate may be necessary if the atmospheric pressure or temperature at the time of sample collection differs significantly from those conditions under which calibration was performed. Corrected flowrates may be calibrated using the following formula:

$$q\ (\text{sampling}) = q\ (\text{indicated}) \times \sqrt{\frac{P\ (\text{sampling})}{P\ (\text{calibration})} \times \frac{T\ (\text{calibration})}{T\ (\text{sampling})}}$$

where:

q = volumetric flow rate (liters/minute)
P = atmospheric pressure (torr or pounds/square inch)
T = temperature (degrees Kelvin or Rankine)

X. APPENDIX II

ANALYTICAL METHOD FOR CARBARYL

The analytical method for carbaryl presented in Appendix II is a validated NIOSH method. [97]

Principle of the Method

A known volume of air is drawn through a glass-fiber membrane filter to trap the carbaryl particles present. Alcoholic potassium hydroxide is used to extract and hydrolyze the carbaryl trapped in the filter. An aliquot is then reacted with p-nitrobenzenediazonium fluoroborate to form a colored complex. The amount of complex formed is determined with a spectrophotometer to give a quantitative measurement of carbaryl. [97]

Range and Sensitivity

This method has been validated over the range of 1.96-13.43 mg/cu m, at 24 C and 763 mmHg. The probable range of the method is 0.1-18 mg/cu m, based on the range of standards used to prepare the standard curve. For samples of higher concentration where the absorbance is greater than the limits of the standard curve, the samples may be diluted with methanolic potassium hydroxide (prior to removal of an aliquot to use for color development) to extend the upper limit of the range. A concentration of 0.5 mg/cu m of carbaryl in a 90-liter air sample gives a 0.05 absorbance in a 1-cm cell.

Interferences

The presence of any background 1-naphthol will exaggerate the analytical reading. Other substances such as phenols and aromatic amines which form derivatives with p-nitrobenzenediazonium fluoroborate or those which have significant absorbance around 475 nm will interfere if present in the air sample.

Precision and Accuracy

The coefficient of variation (Cv) for the total analytical and sampling method in the range of 1.96-13.43 mg/cu m was 0.057. This value corresponds to a 0.28 mg/cu m standard deviation at the recommended environmental limit of 5 mg/cu m.

A collection efficiency of 1.00 was determined for the collection medium. Thus, no correction for collection efficiency is necessary, and it is assumed that no bias is introduced in the sample collection step. There is also no apparent bias in the sampling and analytical method. Thus, Cv is a satisfactory measure of both accuracy and precision of the sampling and analytical method.

Apparatus

(a) 25-ml volumetric flasks.

(b) Screwcap bottles (45-mm tissue-sample holders with Teflon-lined caps).

(c) Assorted micropipets to deliver between 3 and 35 microliters.

(d) 10-ml glass syringe attached to a stainless steel Luer-Lok fitted filter folder for 13-mm Teflon filters.

(e) 10- and 25-ml glass-stoppered graduated cylinders.

(f) Mechanical wrist-action shaker.

(g) Spectrophotometer cell with 1-cm path length.

(h) Spectrophotometer.

Reagents

(a) Carbaryl, analytical grade.

(b) Potassium hydroxide, 0.1 M in absolute methanol.

(c) p-Nitrobenzenediazonium fluoroborate: Dissolve 25 mg in 5 ml of absolute methanol. Add 20 ml of glacial acetic acid to this solution. Prepare fresh daily.

(d) Glacial acetic acid.

(e) Carbaryl Standard Solution: Dissolve 460 mg of carbaryl in 10 ml of methylene chloride.

Analysis of Samples

(a) Analyze each filter separately.

(b) Add 20 ml of 0.1 M methanolic potassium hydroxide to the screwcap bottle containing the filter.

(c) Place the bottle on a mechanical wrist-action shaker for 5 minutes.

(d) Transfer 2 ml of the solution to a clean screwcap bottle.

(e) Add 17 ml of glacial acetic acid and immediately cover the bottle with a Teflon-lined screwcap. Mix by swirling.

(f) Add 1 ml of p-nitrobenzenediazonium fluoroborate solution. The color will develop in 3-5 minutes. The sample should be analyzed within 20 minutes.

(g) The solution will contain glass fibers from the filter. Use the 10-ml syringe fitted with a filter holder containing a Teflon filter to transfer the solution from the bottle to the 1-cm cell. The solution is poured into the barrel of the syringe and forced through the filter into the cell by moving the plunger down the barrel.

(h) Adjust the baseline of the spectrophotometer to zero with distilled water in both cells.

(i) Read the absorbance of the sample at 475 nm against a blank prepared in the same fashion as the samples.

Calibration and Standards

(a) Pipet 20 ml of 0.1 M methanolic potassium hydroxide into each of six 25-ml volumetric flasks.

(b) Carefully pipet 3, 8, 15, 25, and 35 microliters of the standard solution into the flasks. Process one flask as a blank.

(c) After 5 minutes, pipet 2 ml of each solution into a clean 25-ml volumetric flask.

(d) Add 17 ml of glacial acetic acid and mix.

(e) Add 1 ml of p-nitrobenzenediazonium fluoroborate solution and mix.

(f) Allow the solution to react for 20 minutes.

(g) Adjust the baseline of the spectrophotometer to zero by reading distilled water in both cells.

(h) With the wavelength set at 475 nm, read the absorbance of the sample against the absorbance of the blank.

(i) Construct a calibration curve by plotting absorbance against micrograms of carbaryl in the standard.

Calculations

(a) Determine from the calibration curve the micrograms of carbaryl present in each sample.

(b) The concentration of carbaryl in the air sampled can be expressed in mg/cu m (mg/cu m = μg/liter):

$$\text{Carbaryl, mg/cu m} = \frac{\mu\text{g in sample}}{\text{air volume sampled (liters)}}$$

XI. APPENDIX III

SUGGESTED MEDICAL MANAGEMENT OF SYMPTOMATIC CARBARYL INTOXICATION

Carbaryl intoxication can generally be treated successfully with measures directed toward alleviation of symptoms.

If more aggressive treatment should be considered necessary, atropine sulfate can be administered. Pralidoxime chloride (PAM) and other oximes should not be used in carbaryl poisoning. In his Clinical Handbook on Economic Poisons, [21] Hayes pointed out that, depending on the severity of the case, all methods used for treating organophosphorus compounds may be useful in the management of carbaryl intoxication, with the specific exception of pralidoxime (PAM) which should not be used.