

## VII. RESEARCH NEEDS

There is a need for data concerning the long-range effects of chronic exposures to tetrachloroethylene at or below the recommended environmental limit. Of particular concern is the area of behavioral and neurological effects. A preliminary study showed that certain behavioral and neurological tests will reflect tetrachloroethylene exposure. [50]

While the results of the study are inconclusive due to small numbers of subjects and differences between exposed and unexposed groups, the approach is valid and should be studied further. Additional research could be conclusive if the number of subjects is increased and the experiment is more rigorously designed.

Another area of needed research concerns the fetotoxic and teratogenic effects of tetrachloroethylene. The study by Schwetz et al [84] is the only study that provides information on teratogenesis. The design of this study did not take into consideration the effects of chronic exposure to tetrachloroethylene prior to breeding. Therefore the effects on organogenesis of the fetus of chronic exposure of the mother to tetrachloroethylene or its metabolites are not known. Studies should be designed to provide this information. The findings reported in the study by Schwetz et al [84] should be confirmed by other investigators and extended to other species including primates. Studies of whether tetrachloroethylene has mutagenic potentials were not found in the literature. Such studies should be performed.

The metabolic pathways for tetrachloroethylene are subject to question. Various metabolites have been found after tetrachloroethylene exposure. The process, products, and disposition of inhaled tetrachloroethylene should be determined.

## VIII. REFERENCES

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## IX. APPENDIX I

### SAMPLING PROCEDURE FOR COLLECTION OF TETRACHLOROETHYLENE

#### General Requirements

(a) Air samples representative of the breathing zones of workers must be collected to characterize the exposure from each job or specific operation in each work area.

(b) Samples collected must be representative of exposure of individual workers.

(c) Suggested records:

- (1) The date and time of sample collection.
- (2) Sampling duration.
- (3) Total sample volume.
- (4) Location of sampling.
- (5) Temperature, pressure, and relative humidity at time of sampling.
- (6) Other pertinent information.

#### Sampling

(a) Samples should be collected as near as practicable to the faces of workers without interfering with freedom of movement.

(b) Samples should be collected to permit determination of TWA workday and ceiling exposures for every job involving exposure to tetrachloroethylene in sufficient numbers to express the variability of the exposures in the work situation.

(c) Apparatus for Charcoal Tube Sampling

(1) Pump, battery-operated, complete with clip for attachment to the worker. Airflow through the pump shall be within 5% of the desired rate.

(2) Charcoal tubes: glass tube with both ends flame-sealed, 7 cm long, 6-mm O.D. and 4-mm I.D., containing two sections of 20/40 mesh activated coconut shell charcoal separated by a 2-mm portion of urethane foam. The first is the adsorbing section and contains 100 mg of charcoal from coconut shells. The second, or reserve section, contains 50 mg. A 3-mm portion of urethane foam is placed between the outlet of the tube and the reserve section. A plug of glass wool is placed in front of the adsorbing section. The pressure drop across the tube when in use must be less than 1 inch of mercury at a flowrate of 1 liter/min.

(d) Calibration of Sampling Instruments

(1) Air sampling instruments shall be calibrated with a representative charcoal tube in line, over a normal range of flowrates (25-1,000 ml/min). Calibration curves must be established for each sampling pump and shall be used in adjusting the pump prior to and during each field use. New calibration curves should be established for each sampling pump after making any repairs or modifications to the sampling system.

(2) The flowrate through the sampling system should be spot checked and the proper adjustments made before and during each study to ensure obtaining accurate airflow data.

(e) Collection and Handling of Samples

(1) Immediately before sampling, break both ends of the tube to provide openings at least one-half the internal diameter of the tube (2 mm).

(2) The smaller section of charcoal is used as a reserve and should be positioned nearest the sampling pump.

(3) The charcoal tube should be placed in a vertical position during sampling.

(4) Tubing may be used to connect the back of the tube to the pump, but air being sampled should not be passed through any hose or tubing before entering the charcoal tube.

(5) The sample can be taken at flowrates of 25-1,000 ml/min, depending on the pump. Total sample volumes of 1-12 liters are recommended, eg, a sample could be collected at 200 ml/min for 15 minutes to give a total sample of 3 liters, or at 25 ml/min for 8 hours to give a total sample volume of 12 liters. However, it is also recommended that each sample be collected in 4 hours or less.

(6) Samples should be collected over 15-minute periods at times when the highest exposure is expected. The TWA determination can be made from collecting a series of 15-minute samples.

(7) The charcoal tubes should be capped with inert plastic caps immediately after sampling. Under no circumstances should rubber caps be used.

(8) One charcoal tube, to serve as an analytical blank, should be handled in the same manner as the sample tube (break, seal, and transport) except that no air is sampled through this tube.



## X. APPENDIX II

### ANALYTICAL PROCEDURE FOR DETERMINATION OF TETRACHLOROETHYLENE

#### Principle of the Method

(a) A known volume of air is drawn through a charcoal tube to trap the tetrachloroethylene vapor.

(b) The tetrachloroethylene is desorbed from the charcoal with carbon disulfide.

(c) An aliquot of the desorbed sample is injected into a gas chromatograph.

(d) The area of the resulting peak is determined and compared with areas obtained from the injection of standards.

#### Range and Sensitivity

(a) The lower limit for detection of tetrachloroethylene on a gas chromatograph with a flame ionization detector is 2  $\mu\text{g}/\text{sample}$ .

(b) The upper limit value for tetrachloroethylene is 4.0 mg/sample. This is the estimated amount of tetrachloroethylene which the front section will hold before this compound breaks through to the reserve section of charcoal. If a particular atmosphere is suspected of containing a high concentration of tetrachloroethylene, it is recommended that a smaller volume of air be sampled.

### Interferences

(a) Tetrachloroethylene will not be trapped when the amount of water in the air is so great that condensation occurs in the charcoal sampling tube.

(b) Any compound which has the same retention time as tetrachloroethylene with the chromatographic conditions described in this method could interfere. These may be eliminated by altering operating conditions of the gas chromatograph using a different column packing or using a selective detector, ie, electron capture.

### Advantages of the Method

(a) This method provides one basic method for determining many different organic compounds.

(b) The sampling device is small, portable, and involves no liquids.

(c) The analysis of the tubes can be accomplished rapidly.

### Disadvantages of the Method

(a) The amount of sample which can be taken is limited by the weight of tetrachloroethylene which the tube will hold before overloading.

(b) When the sample value obtained for the reserve section of charcoal exceeds 25% of that found on the front section, the possibility of appreciable sample loss exists.

(c) Other organic compounds in high concentrations may displace tetrachloroethylene from the charcoal.

### Apparatus

- (a) Gas chromatograph equipped with a flame ionization detector.
- (b) Stainless steel column (20 ft x 1/8 in) with 10% free fatty acid polymer (FFAP) stationary phase on 80/100 mesh Chromosorb W (or equivalent), acid-washed and treated with dimethyldichlorosilane.
- (c) A recorder and some method for determining peak area.
- (d) Glass stoppered microtubes of 2.5-ml capacity or 2-ml vials that can be sealed with inert caps.
- (e) Microsyringe of 10- $\mu$ l capacity, and convenient sizes for making standards.
- (f) Pipets 0.5-ml delivery pipets or 1.0-ml pipets graduated in 0.1-ml increments.
- (g) Volumetric flasks of 10-ml capacity or convenient sizes for making standard solutions.

### Reagents

- (a) "Spectroquality" carbon disulfide.
- (b) Tetrachloroethylene, preferably "chromatoquality" grade.
- (c) Bureau of Mines Grade A helium.
- (d) Prepurified hydrogen.
- (e) Filtered compressed air.

### Analysis of Samples

- (a) All equipment used in the analysis should be washed in detergent followed by appropriate tap and distilled water rinses.

(b) Preparation: Each charcoal tube is scored with a file in front of the first section of charcoal and broken open. The glass wool is removed and discarded. The charcoal in the first (larger) section is transferred to a small stoppered test tube. The separating foam is removed and discarded; the second section is transferred to another similar tube or vial. These two sections are analyzed separately. Prior to analysis, 0.5 ml of carbon disulfide is pipetted into each test tube to desorb tetrachloroethylene from the charcoal. Do not pipette by mouth.

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 chromatographic operating conditions:

- (1) 40 ml/min (70 psig) helium carrier gas flow.
- (2) 65 ml/min (24 psig) hydrogen gas flow to detector.
- (3) 500 ml/min (50 psig) airflow to detector.
- (4) 200 C injector temperature.
- (5) 200 C manifold temperature (detector).
- (6) 80 C isothermal oven or column temperature.

(d) Injection: The first step in the analysis is the injection of the sample into the gas chromatograph. To eliminate difficulties arising from blowback or distillation within the syringe needle, the solvent flush injection technique is employed. The 10- $\mu$ l syringe is first flushed with

carbon disulfide several times to wet the barrel and plunger. Three  $\mu\text{l}$  of carbon disulfide are drawn into the syringe to increase the accuracy and reproducibility of the injected sample volume. The needle is removed from the carbon disulfide solvent, and the plunger is pulled back about 0.2  $\mu\text{l}$  to separate the solvent flush from the sample, with a pocket of air to be used as a marker. The needle is then immersed in the sample,, and a 5- $\mu\text{l}$  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, the plunger is pulled back a short distance to minimize evaporation of the sample from the tip of the needle. Duplicate injections of each sample and standard should be made. No more than a 3% difference in area is to be expected.

(e) Measurement of area: The area of the sample peak is determined and preliminary sample results are read from a standard curve prepared as discussed below.

#### Determination of Desorption Efficiency

It is necessary to determine the percentage of tetrachloroethylene on the charcoal that is removed in the desorption process. This desorption efficiency is determined once for a given compound provided the same batch of charcoal is always used.

Activated charcoal, equivalent to the amount in the first section of the sampling tube (100 mg), is measured into a 2-inch long tube, with an inside diameter of 4 mm, flame-sealed at one end. This charcoal must be from the same batch used in obtaining the samples and can be obtained from unused charcoal tubes. The open end is capped with inert plastic. A known

amount of the compound is injected directly into the activated charcoal with a microliter syringe, and the tube is capped with inert plastic.

A minimum of six tubes are prepared in this manner and allowed to stand at least overnight to ensure complete adsorption of tetrachloroethylene onto the charcoal. These six tubes will be referred to as the "desorption samples." A parallel blank tube should be treated in the same manner except that no tetrachloroethylene is added to it. The desorption samples and blanks are desorbed and analyzed in exactly the same manner as previously described.

Two or three desorption standards are prepared for analysis by injecting the same volume of tetrachloroethylene into 0.5 ml of carbon disulfide with the same syringe used in the preparation of the desorption samples. These are analyzed with the desorption samples.

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

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

#### Calibration and Standards

It is convenient to prepare standards in terms of mg tetrachloroethylene/0.5 ml of carbon disulfide because samples are desorbed in this amount of carbon disulfide. To minimize error due to the volatility of carbon disulfide, 20 times the weight can be injected into 10 ml of carbon disulfide. For example, to prepare a 0.3 mg/0.5 ml standard,

6.0 mg of tetrachloroethylene is injected into exactly 10 ml of carbon disulfide in a glass stoppered flask. The density of tetrachloroethylene (1.623 g/ml) is used to convert 6.0 mg into microliters for easy measurement with a microliter syringe. A series of standards is prepared, varying in concentration over the range of interest and analyzed under the same gas chromatographic conditions and during the same time period as the unknown samples. Curves are established by plotting concentration versus average peak area.

#### Calculations

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

(b) Separately determine the weights of tetrachloroethylene on the front and reserve sections of the charcoal tube.

(c) Corrections must be made to the tetrachloroethylene weights determined on both the front and reserve sections for the weights of the respective sections of the blank charcoal tube.

(1) Subtract the weight of tetrachloroethylene found on the front section of the blank charcoal tube from the weight of tetrachloroethylene found on the front section of the sample charcoal tube to give a corrected front section weight.

(2) Subtract the weight of tetrachloroethylene found on the reserve section of the blank charcoal tube from the weight of

tetrachloroethylene found on the reserve section of the sample charcoal tube to give a corrected reserve section weight.

(3) Add the corrected amounts of tetrachloroethylene present on the front and reserve sections of the sample tube to determine the total measured tetrachloroethylene in the sample.

(4) Divide this total weight by the determined desorption efficiency to obtain M, the total mg per sample.

(d) Convert the liters of air sampled (V) to volume (V') at standard conditions of 25 C and 760 mm Hg, as follows:

$$V' = \frac{298VP}{760(T+273)} = \frac{0.392VP}{(T+273)}$$

Where:

V' = volume of sampled air in liters at 25 C and 760 mm Hg

V = measured volume of sampled air in liters

P = barometric pressure in mm Hg, measured at time of sampling

T = temperature of air in degree celsius, measured at time of sampling

(e) The concentration of tetrachloroethylene in the sampled air can be expressed in various ways using M, the weight of tetrachloroethylene obtained in (c)(4), and V', the standardized sample volume, obtained in (d), as follows:

(1)  $\text{mg/liter} = M/V'$

(2)  $\text{mg/cu m} = \mu\text{g/liter} = 1,000 M/V'$

(3)  $\text{ppm} = 147.4 M/V'$