CHARCOAL TUBE METHOD

This practice was developed in order to comply with Part 2, Volume 39, "194 Section 1910.93 OSHA Standard to monitor the exposure levels of personnel to Vinyl Chloride Monomer, a suspected carcinogen.

The program consists of two procedures to establish the exposure level for any employee for any given working shift.

I. Sampling of the breathing zone of the employee & absorption of any Vinyl Chloride Monomer on activated charcoal.

II. Analyses for Vinyl Chloride Monomer concentration desorbed from activated charcoal.

The program developed and followed by Shintech, Inc. follows closely both the "Vinyl Chloride in Air" method published in the NIOSH manual of Analytical Methods, Second Edition, Volume 1, and the ANSI/ASTM D 3686-78 & D 3687-78 general methods to collect and analyze organic compounds.

I. SAMPLING PROCEDURE FOR PERSONNEL MONITORING OF VINYL CHLORIDE MONOMER EXPOSURE.

The following apparatus is employed for the monitoring process.

a) Accuhaler 808 Personal Sampling Pump.

b) 20/40 mesh activated charcoal separated by a 2mm urethan foam section in 11cm glass tube flamed sealed at each end with 360mg of charcoal in front section. These tubes are purchased prepared and lot numbers are recorded with use. A 3mm foam section is between the back charcoal portion and the flame sealed glass end. A siliconized glass wool plug and wire "spring" protects the front section. These are sequentially numbered for any 1 monitor sample period.

c) A tygon tube, approximately 50cm long, links the sorbent tube to the inlet point on the sipin pump.

d) Protective sorbent tubing case.

SAMPLING PROCEDURE AS FOLLOWS:

a) Pumps are charged for a minimum of 24 hrs. prior to distribution.

b) Volume of air pumped is regulated by a restricted orifice to 6ml/ stroke.

c) Pumps are identified and assigned to each individual to be monitored for any given period.

d) Tubes are numbered and placed in individual bags with a pump, tube holder and tubing and data sheet.

e) Each bag is distributed to the appropriate individual. Each tube was broken to allow entrance and exit openings atleast 1/2 the diameter of the main body of the sampling tube. One tube is employed to be worn all of the work schedule. Time and pump counts are recorded for each tube, start and stop on the data sheet. Work area, tube and pump numbers as well as employee signature are also required. (See attached)

f) Tubes are capped with supplied caps when changed and returned to lab freezer. Completely filled paperwork is turned in and pumps collected by Safety Personnel to be recharged.

II. ANALYSIS FOR VINYL CHLORIDE MONOMER CONCENTRATION

Principle: A charcoal tube, through which a known volume of air has been pased, is desorbed with carbon disulfide, and the Vinyl Chloride content analyzed by traditional gas chromatography with a flame ionization detector.

GC Conditions:	Column:
PE 3920 GC	20% OV-101, 80/100 mesh
Oven - 47°C	WAW-DMCS, 2' - 3' attached
Injector - 150°C	to 10% C-20M, 80/100 mesh.
Interface - 200°C	WAW-DMCS, 6' - 8', Columns
Carrier Gas - Nitrogen	may be heated to 140°C to clean.
Carrier Flow - 20 - 30cc/min.	

DESORPTION PROCESS: Charcoal tubes, as received by the Lab are scored just above the front section to facilitate breaking and then stored in a 4°C freezer. 5ml vials are labeled to correspond to tube numbers and filled with 2ml carbon disulfide. This is then placed with tubes in freezer until time to analyze. They are then removed to a well ventilated hood. Each tube is broken at the scored area and the glass wool plug removed by a hooked rod. As described in the NIOSH method 178 for Vinyl Chloride in Air Method, each section (front & back) is added to individual vials of CS_2 so that front and back are analyzed separately to compensate for the use of only one tube. It is important to add the charcoal to the CS_2 and not the other way around as defined in 178-6 of NIOSH. The septa capped vials are then placed on a charcoal developer to be desorbed for 30 minutes. At this time they are ready to be injected into the GC for analysis.

GC CALIBRATION TUBE PREPARATION: A gas sampling bag is filled with a small volume of an appropriate concentration of Vinyl Chloride Monomer. (The present VCM std. of 1 ppm has been found satisfactory). The bag is then evacuated and refilled so as to be nearly full but with no pressure build up in the bag. It is then linked by quick connect through a charcoal tube (with front section of the tube closest to the bag) and attached to an Accuhaler 808 Personal Sampling Pump from MDA Scientific which has been previously battery charged. The pump count is recorded from the pump and it is cut on and left to run for 11 to 12 hours. The pump is then cut off, the pump count recorded and the tube stored in the freezer. The tube is then treated to the desorption process as above described when ready to analyze.

GC ANALYSIS: A 1 ul Hamilton Syringe is employed with a Chaney adapter set at 0.5 ul for injection of any sample into the GC. It has been found that repetitive injections have adequate precision using this method. Twenty minutes is required between injections. The syringe is introduced into the GC Port and injected and the Interface Ready/Run button immediately depressed. (Central Processor/Interface/Printer-Plotter logic is discussed later). Area counts for VCM are then available for use in establishing GC calibration and sample concentration values as follows.

Central Processor Methodology: The Spectra-Physics 4000 Series System is used to integrate and process data received from the GC injections. There are two GC's available for analysis to expedite analysis and also provide a back up. The central processor, interfaces and printer-plotter are linked as described in the SP 4000 manual. Interfaces have a jump between the run reset and T5 screws to provide ability of the system to automatically stop a run at a time set by the program when initiated by the Ready/Run button on the interfaces. A routine external standard method is used as discussed under Method 5 in the SP Manual. The programs for each GC are attached and mathematical manipulations will be described. Further information can be found in the SP manual.

Pump Calibration: The Accuhaler 808 Personal Sampling Pump from MDA Scientific has a restricted orifice through which 6 ml per pump count of air is drawn. Since volumes of gas pumped in calibration prepraration vary with length of time pumped, a correction factor must be determined to correlate samples to the standard.

$$XF = \frac{\text{Multiplying correction factor}}{1}$$

$$XF = \frac{(\text{Total pump count X ml. per pump count})}{1000 \text{ ml.}}$$

GC CALIBRATION: The standard, described under GC Calibration tube preparation, is injected a minimum of three times for each GC. The VCM peak area count is averaged for each GC. The calibration factor is determined by peak area per unit of concentration

$$KF = \frac{\text{Average area count of standard}}{\text{VCM standard concentration used}} \times XF$$

This KF value is entered into the SP 4000 Central processor and can be checked by injecting any sample prepared as was the standard to see that it reports a value close to the standard gas used.

SAMPLE ANALYSIS: Charcoal tubes received by the Lab are subjected to the desorption process previously explained. An XF for each tube is determined using the pump count values reported on the accompanying paperwork for each. In the sample file of the SP 4000 CPU, each person's name and tube number is typed in as well as the XF for that sample.

A blank charcoal tube and a std. sample (prepared by the GC Calibration tube preparation method) are always analyzed along with the personnel sample to verify lack of interference, VCM peak time, and calibration factor.