### VII. RESEARCH NEEDS

For proper assessment of the toxicity of carbon black and evaluation of its potential hazard to the working population, further animal and human studies are needed. The following types of research are especially important.

## **Epidemiologic Studies**

Further research is desirable to assess the effects of long-term occupational exposure to carbon black. Therefore, detailed long-term epidemiologic studies, retrospective and prospective, of worker populations exposed to carbon black should be conducted. As a minimum, epidemiologic studies should include detailed industrial hygiene surveys, separate environmental air measurements of carbon black and PAH's, comprehensive medical and work histories, including history of smoking and other tobacco usage, pulmonary function studies, and physical examinations, with particular attention to the respiratory tract, oral mucosa, heart, and skin. Comparison of morbidity and mortality data from populations exposed to carbon black with that of properly selected control populations, such as those populations exposed to nuisance dusts, should also be performed to develop a more sensitive index of carbon black toxicity.

#### **Animal Studies**

Short- and long-term inhalation and intratracheal insufflation studies have described the respiratory effects of carbon black [18,28,30,31]. In some of the inhalation studies [28,30] carbon black particles also caused effects on the skin, heart, kidneys, liver, and spleen. Studies are needed to further delineate these changes and to distinguish the effects on the lung from such effects, possibly secondary, as those on the heart. Long term studies concerning the tissue distribution of carbon black should be conducted. Dermal effects were found in workers exposed to carbon black [13,15,19]. However, animal experiments did not adequately confirm that these effects resulted from direct application of carbon black [30,39]. There is a paucity of information regarding the ocular effects of carbon black exposure. Additional dermal and ocular irritation studies which simulate the work environment should be undertaken.

## Studies on Carcinogenicity, Mutagenicity, Teratogenicity, and Effects on Reproduction

In a number of animal experiments benzene extracts of carbon black were shown to be carcinogenic [10,39,52]. There are conflicting views on the ability of human plasma to elute PAH's adsorbed on carbon black. Further investigations are needed to clarify whether the extent to which desorption of PAH's from carbon black occurs is of practical concern in the evaluation of the risk of cancer in occupational exposure to carbon black. The interplay of carbon black with other substances in the work environment which might act as initiators and promoters of carcinogenesis should also be investigated. Further research, including extensive chronic and multigeneration reproduction experiments, should be conducted to determine whether mutagenic, teratogenic, or other reproductive effects are caused by carbon black.

## Sampling and Analytical Studies

Studies are needed to improve the accuracy, sensitivity, and precision of the recommended sampling and analytical methods for carbon black. These studies should concentrate on techniques for separating the carbon black from other airborne particulates. Also, studies are needed to elucidate the adsorptive and binding capabilities of carbon black particles in relation to PAH's. The desorption of these PAH's from the carbon black particles by various organic solvent vapors that might be found in workplace environments and the elutability of PAH's by biologic fluids should also be investigated.

## Personal Hygiene Studies

Personal hygiene studies should be conducted to determine the best methods of cleaning skin areas contaminated with carbon black. For example, it may be that acid cleansers will be more effective than the more conventional cleansers, but this has not been demonstrated. These studies should take into account possible skin irritation after a repeated number of washings.

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### IX. APPENDIX I

### SAMPLING AND ANALYTICAL METHOD FOR CARBON BLACK

### Principle of the Method

- (a) A known volume of air is drawn through a glass-fiber filter followed by a  $0.8-\mu m$  pore size silver membrane filter to collect carbon black particles and associated PAH's. The original method recommended a polyvinyl chloride filter but the glass-fiber and silver membrane filters have been substituted to allow for greater accuracy in the subsequent determination of the cyclohexane extractable fraction.
- (b) There is some reason to believe that samples containing carbon black will pick up moisture with sufficient rapidity to make sample weights unstable. Thus, while desiccation of samples prior to weighing should give the most accurate results (if weighings can be made with enough speed), it may be better to equilibrate samples at some higher constant humidity. The directions in the ensuing discussion follow this latter approach. However, in a specific situation, there may be better solutions to the problem of getting stable weighings. Ideally, a constant temperature and humidity balance room should be used for all weighings.

The humidity chosen will be that most often found in the balance room. A relative humidity of 50% is recommended. To achieve any desired relative humidity in the equilibrating chamber (usually a desiccator) aqueous solutions of sulfuric acid can be used. Consult a convenient reference such as the Handbook of Chemistry and Physics [85] table on Constant Humidity with Sulfuric Acid Solutions for the desired sulfuric acid solutions.

### Range and Sensitivity

(a) This method for carbon black was validated over the range of 1.86-7.7 mg/cu m at an atmospheric temperature and pressure range of 18-25 C and 749-761 mmHg, using a 200-liter sample and a polyvinyl chloride filter. Under the conditions of sample size (200 liters), the working range of the method is estimated to be 1.5-10 mg/cu m or 0.3-2 mg total weight of material collected on the filter.

It was also validated for a 100-liter sample over a range of 7.8-27.7 mg/cu m at atmospheric temperature and pressure conditions as above.

- (b) The method may be extended to higher sample concentrations by collecting a smaller volume; this will prevent collection of too much sample particulate so as to cause sample loss due to flaking.
- (c) The range and sensitivity of this method with the glass fiber and silver membrane filters has not been determined but is assumed similar to those stated above.

### Interferences

- (a) The presence of any other particulate material in the air being sampled will be a positive interference since this is a measurement of total dust.
- (b) Information on any other particulate material present should be solicited. If the concentration of other particles is known, then the carbon black concentration can be determined by difference. If other particulate matter is known to be present and its concentration cannot be determined, then this method will not provide an accurate measure of carbon black concentration.

# Precision and Accuracy

- (a) The Coefficient of Variation (CVT) for the total analytical and sampling method in the range of 1.86-7.7 mg/cu m was 0.056. This value corresponds to a 0.20 mg/cu m standard deviation at the OSHA standard level (3.5 mg/cu m).
- (b) A collection efficiency of 98.7% was determined for the collection medium at 7.0 mg/cu m; thus, no bias was introduced in the sample collection step. Likewise, no significant bias in the analytical method is expected other than normal gravimetric precision of the sampling and analytical method.
- (c) The above data on precision and accuracy were determined for a polyvinyl chloride filter but the recommended filters are thought to result in similar precision and accuracy.

# Advantages and Disadvantages

The analysis is simple but the method is non-specific and subject to interference if there are other particles in the air being sampled.

## **Apparatus**

- (a) Sampling Equipment. The sampling unit for the collection of personal air samples for the determination of carbon black has the following components:
- (1) The filter unit, consisting of the glass fiber and silver membrane filters, stainless steel support screen and 37-mm three-piece cassette filter holder.
- (2) A calibrated personal sampling pump whose flow can be determined to an accuracy of 5% at the recommended flow rate. The pump must be calibrated with a filter holder and filters in the line as outlined in Figure XII-1.
  - (3) Thermometer.
  - (4) Manometer.
  - (5) Stopwatch.

- (b) A 37-mm diameter glass-fiber filter.
- (c) A 37-mm diameter,  $0.8-\mu m$  pore size silver membrane filter.
- (d) A plastic petri dish used as filter holder for storage and weighing.
- (e) Desiccator.
- (f) Microbalance capable of weighing to 10  $\mu$ g. Particular care must be given to proper zeroing of the balance. The same balance should be used for weighing filters before and after sample collection.

## Reagents

Aqueous sulfuric acid solution or any other suitable humidity source.

#### Procedure

- (a) Preparation of Filters. All filters must be placed in a chamber over an aqueous sulfuric acid solution for 24 hours to bring the filter to a constant weight at relative humidity prior to use.
  - (b) Sampling Requirements and Shipping of Samples
- (1) To collect carbon black, a personal sampler pump is used to pull air through a silver membrane filter preceded by a glass fiber filter. The filter holder is held together by tape or a shrinkable band. If the filter holder is not tightened snugly, the contaminant will leak around the filter. A piece of flexible tubing is used to connect the filter holder to the pump. Sample at a flowrate of 1-2 liters/minute. After sampling, replace small plugs to seal filter cassettes.
- (2) Blank. With each batch of 10 samples submit one filter from each of the lots of glass fiber and membrane filters which were used for sample collection and which are subjected to exactly the same handling as for the samples except that no air is drawn through them. Label this as a blank.
- (3) Shipping. The filter cassettes should be shipped in a suitable container, designed to prevent damage in transit.

## (c) Analysis of Samples

- (1) If the outer surface of the cassette filter holder is heavily coated with dust, carefully swab the outer surface with a moist paper towel before opening the cassette so as to minimize sample contamination. Discard paper towel.
- (2) Open the cassette filter holder and carefully remove the filters from the holder and stainless steel screen with the aid of filter tweezers. Transfer filters to a petri dish.
  - (3) Bring the filter to constant relative humidity.
  - (4) Weigh the filters in a microbalance.
- (5) If other particulate matter is suspected to be present, appropriate analyses should be made to determine its composition (if necessary) and quantity. This value should be subtracted from the total particulate weight.

#### Calibration and Standards

The only standardization of the analytical method required is that the microbalance be properly zeroed for all weighings and prefereably the same microbalance should be used for weighing filters before and after sample collection.

# Calibration of Sampling Trains

The accurate calibration of a sampling pump is essential for the correct interpretation of the volume indicated. The proper frequency of calibration is dependent on the use, care, and handling to which the pump is subjected. 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. 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, during field procedures, 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 on where the calibration is to be performed. For laboratory testing, a soapbubble meter or spirometer is recommended, although other standard calibrating instruments, such as a wet-test meter 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. Since the flowrate given by a pump is dependent on the pressure drop of the sampling device, in this case a glass fiber filter and a membrane filter, the pump must be calibrated while operating with the representative filters in line. Calibration of the sampling train should be performed at pressure of 1 atmosphere.

- (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 preweighed filters in the filter cassette.
- (c) Assemble the sampling train according to the manufacturer's specifications or as shown in Figure XII-1.
- (d) 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.
  - (e) Adjust the pump rotameter to provide a flowrate of 1.7 liters/minute.
- (f) Start a soapbubble up the buret and measure with stopwatch the time it takes the bubble to pass through a minimum of 1.0 liter.
- (g) Repeat the procedure in (f) at least three times, average the results, and calculate the flowrate by dividing the volume of air 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.

### **Calculations**

- (a) Record the tare weight, in  $\mu$ g, of the dry filters before sampling.
- (b) Record the weight, in  $\mu g$ , of the dried, sample-containing filters.
- (c) The difference between these two weights represents the weight of the material collected on the filters.
- (d) Corrections for the blank must be made for each sample (if found to be necessary, corrections should also be made for other particulate matter:

 $\mu$ g sample =  $\mu$ g found in sampling filter  $\mu$ g blank =  $\mu$ g found in blank filter

(e) Convert volume of air sampled to standard conditions of 25 C and 760 mmHg.

, 
$$Vs = V \times \frac{P}{760} \times \frac{298}{(T + 273)}$$

where:

Vs = volume of air in liters at 25 C and 760 mmHg

V = volume of air in liters as measured

P = barometric pressure in mmHg T = ambient air temperature in degrees Centrigrade

The concentration of the analyte in the air sampled can be expressed in mg/cu m (µg/liter = mg/cu m).

$$mg/cu m = \frac{\mu g \ found}{Vs}$$