INTERNAL CORRESPONDENCE

GA 107a

FROM R. T. Cheng

IN REPLY REFER TO RTC 74:213

TO Ed Lu

DATE 12/9/74

SUBJECT Industrial Hygiene Instruments

Instead of purchasing miniature personal sampling pumps for taking 8-hour VCM samples with small charcoal tubes, an alternative is to sample the air with standard size personal sampling pump with large size charcoal absorption tubes which your laboratory can make. The advantages of the standard size personal sampling pumps are that you can use those pumps for dust sampling, midget impinger samplings, and many other types of operations which a miniature sampling pump is incapable of. Attached is an operation manual from General Tire & Rubber Company and Dow Chemical Company on how to prepare the large size charcoal absorption tubes, and how to run the vinyl chloride samples.

Dr. J. O. Jackson will send you a letter recommending the type of gas chromatograph and other accessories to be purchased for VCM sampling and analysis.

Bob

R. T. Cheng

RTC:mm
Attachment
cc: R. L. Gibson, M. D., w/o attach.
J. O. Jackson, with attach.
H. E. Runion, w/o attach.
W. T. C. Shyeh, w/o attach.

[illegible] Chemical Co.
Jan. 197[illegible])

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Personnel Monitoring
Exposure to VCl

SCOPE:

The VCl in 1 mole of air will be collected on 1 gram of charcoal contained in a filtering device worn by a worker. The VCl will be washed from charcoal into one mole of carbon disulfide and analyzed on a chromatograph.

MATERIAL:

1) Carbon disulfide
2) Charcoal
3) Serum bottles w/stoppers
4) Graduate cylinder (100 ml)
5) Lab balance ± .01 grams
6) Wrist action shaker
7) Micro syringe
8) Cooler
9) Glass wool
10) Stop watch
11) Portable battery operated pump
12) 10 MM OD x 120 MM long glass tubes charcoal absorption units
13) Gas chromatograph

Activated charcoal by Pittsburg Act Carbon Co Type PCB 12X30 also recommend by Dow Chemical

Additional Materials for Calibration

1) Wet test meter
2) Analytical balance
3) Liquid VCl
4) Standard (VCl in air) gas (500 ppm level)

PROCEDURE:

1) Prepare absorption units (glass tubing 4½ inches long) with glass wool, 0.10 grams of charcoal, more glass wool, [illegible] grams of charcoal, then a final pad of glass wool. Charcoal should be held in place by the glass wool so that there are no voids.

2) Attach the absorption unit to the pump [illegible] that the [illegible] the large portion of charcoal first

3) Turn on air pump and adjust flow meter [illegible] the proper setting (See Appendix 1 for pump calibration, flow meter setting, and time requirement for a one mole sample)

4) Attach pump to the belt of the [illegible] monitored and attach the [illegible] to his shirt near the breathing zone, in such a way that it will not restrict his work.

5) Start the pump and the stop watch. Fill out ALL the necessary data [illegible] the test is being conducted ([illegible] form) At the end of the required time stop the pump, remove and seal the ends of the absorption unit.

6) To analyze the absorption unit prepare and cool two serum bottles [illegible] each (60 ± 1 cc) of carbon disulfide. Transfer the small portion of carbon black plus one pad of glass wool to the first serum bottle. [illegible] of CS_2 from the second serum bottle with a syringe, then transfer [illegible] contents of the absorption unit [illegible] the second serum bottle and wash with the contents of the syringe.

7) Stopper the serum bottles and place on wrist action shaker for 30 minutes.

8) Inject sample of the second serum bottle into a chromatograph in duplicate and multiply the average peak height of the MVC peak times the factor for [illegible] ppm (See Appendix #2 for [illegible] establishing curve and factor.)

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9) Determine the MVC in the first serum bottle in the same manner. Multiply it by 1.1, then add it to the result of the second bottle for the total mole ppm.

REPORT:

Report mole parts per million.

APPROVAL: [signature] [signature]

DEPLOYMENT: [signature] [signature]

APPENDIX #1

To calibrate a monitoring pump with a wet test meter.

The procedure requires one mole of air pumped through absorption tube as it is being carried by the worker being monitored.

One mole of air = 22.4 L at standard conditions
25.45 L at ambient conditions
0.90 cu. ft. at ambient conditions

Place absorption tube on the pump filter so that the incoming sample will pass through the large portion of the charcoal first. Attach flexible tubing to exhaust port (top) of a wet test meter and the other end to the absorption tube. Turn on the pump and adjust pump flow so that wet test meter rate is 5.4 cu. ft. an hour at a 1 minute observation. This setting will pump a mole of air in ten minutes.

If the pump being calibrated is not capable of pumping one mole of air in 10 minutes, then simply reduce the volumn of the solvent to the proportion of a mole that the pump can pump.

Example: GT&R pump #1 can only pump 72.3% of a mole of air in 10 minutes so instead of using 1 mole of CS_2 (60.1 cc) only 43.5 cc is used. The ratio remains the same so no further calculation is necessary.

If the pump being used can only pump a small portion of a mole the above procedure cannot be used because there will not be enough solvent to desorbe the TCL from the carbon. This being the case take the sample at the capacity of the pump then multiply the final answer by a dilution factor based on the following formula.

$$\frac{\text{L in a Mole of Air X Moles of Solvent}}{\text{L in Sample}} = \text{Dilution Factor}$$

Example: GT&R Pump #2 (Sipin)pumps at the rate of 2.77 L in 10 minutes. (The solvent is reduced to one half a mole)

$$\frac{25.45 \times .5}{2.77} = 4.59 \text{ Dilution Factor}$$

APPENDIX #2

To establish a curve or peak height factor for chromatograph analysis.

Prepare three serum bottles with 1 mole each of carbon disulfide (76.14 grams or 60.1 ml). Obtain the gross and net weight to the 4th place.

Cool and add through the stoppers approximately 0.05, 0.10 and 0.15 grams of VCl, then reweigh.

Obtain the peak height for each standard by injecting 1.0 ul of each standard in duplicate.

Calculation

$$\text{Mole ppm of Standard} = \frac{A \times C \times 1,000,000}{B \times D}$$

A = Weight of VCl

B = Mole weight VCl (62.5)

C = Mole weight of CS_2 (76.14)

D = Weight CS_2

$$\text{Mole ppm per chart division or factor} = \frac{\text{Mole ppm of standard} \times 100}{\text{Average peak height} \times \% \text{ efficiency of charcoal}}$$

To obtain % efficiency of absorption tube pump one mole of (VCl in air) standard gas, thru an absorption tube as explained in procedure and compare its peak height to those of the standard.

Example: Pittsburgh Activated Carbon P.C.B. 12 x 30 is 100% efficient at 100 ppm level.

APPENDIX #3

Graph #1 is for Pump #1 Columns A & B on GT&R flame ionization chromatograph.

Graph #2 is for Pump #2 Columns A & B on GT&R flame ionization chromatograph. This graph includes the 4.59 dilution factor.

The following conditions are established on GT&R Perkin-Elmer 3920 Chromatograph.

Helium Pressure - 50 Lbs. per sq. inch
Flow - 22 ml per min Columns A & B
Column Packing - 15% Apizon 'L' on Chromozorb 'W'
Columns A & B 15% SE 30 on 80/100 mesh Chromosorb W
Hydrogen Pressure - 30 lbs. per sq. inch Columns A & B
Air - 45 lbs. per sq. inch
Column Temp. - 140°C
Detector Temp. - 200°C
Recorder Chart Speed - 40 mm/min

Note: Because of the high sensitivity of this chromatograph there is a small contamination peak that eludes with the VCL so it is necessary to subtract 6 chart divisions from the VCL peak before applying it to the graph.