NBSIR 74-530

Preparation of Charcoal Sampling Tubes Containing Known Quantities of Adsorbed Solvents

B. C. Cadoff, E. E. Hughes, R. Alvarez and J. K. Taylor

National Bureau of Standards
Department of Commerce
Washington, D. C. 20234

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Final Report

Prepared for

National Institute for Occupational Safety and Health
Division of Laboratories and Criteria Development
Cincinnati, Ohio 45202

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U. S. DEPARTMENT OF COMMERCE, Frederick B. Dent, Secretary

NATIONAL BUREAU OF STANDARDS, Richard W. Roberts, Director

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PREPARATION OF CHARCOAL SAMPLING TUBES CONTAINING KNOWN QUANTITIES OF ADSORBED SOLVENTS

ABSTRACT

The method in widespread use for the determination of the concentration of organic solvents in the work atmosphere consists in collection of the solvent by adsorption on activated charcoal followed by desorption with carbon disulfide and measurement by gas chromatography. This report describes techniques developed to produce reference standards for this determination and modifications in the analytical procedure to improve its precision and accuracy.

1.0 Introduction

This is the final report on work performed under an Interagency Agreement involving the preparation of charcoal tubes containing known amounts of organic solvents. The objective of this work was to develop techniques for the mass production of charcoal tubes containing known amounts of organic solvents, to serve as reference standards. A further objective was to prepare a limited number of tubes for collaborative test of the NIOSH analytical method for measurement of the concentration of organic solvents in air. The first report, issued December 1, 1972, dealt with the preparation of an initial batch of approximately 1000 tubes each containing known amounts of one of seven solvents, viz., benzene, xylene, p-dioxane, ethylene dichloride, chloroform, trichloroethylene and carbon tetrachloride.

This report describes further activities of this project and also summarizes the earlier work.

2.0 Major Accomplishments

1-Production of over 1000 charcoal tubes for NIOSH collaborative studies, each containing one of seven solvents at one of four or five concentration levels (benzene, xylene, p-dioxane, ethylene dichloride, chloroform, trichloroethylene, and carbon tetrachloride) adsorbed on the charcoal. Each tube was certified to contain a known amount of solvent material (expressed in µg/tube) to within ±5 percent of the assigned value. Replicate tubes did not vary from one another by more than ±2 percent.

2-The development of a new accurate gravimetric procedure for determining both the concentrations and long term stability of solvent-in-air mixtures, that gives promise of general applicability not only to solvents but other materials as well. (This work will be submitted for publication in the technical literature.)

3-The design and fabrication of critical orifice sampling assemblies that maintain unchanging sampling rates after many hours of use. (This work will be submitted for publication in the technical literature.)

4-Suggested improvements in the procedure for the analysis of charcoal tubes, involving a means of preventing evaporation of the carbon disulfide used as a desorber, and in the preparation of more accurate standard solutions for calibration of the gas chromatograph.

5-A suggestion for a design change in the charcoal tubes to permit a simpler evaluation of the capacity or "breakthrough" characteristics of tubes in laboratory and field studies.

3.0 Preparation and Analysis of Charcoal Tubes for NIOSH Collaborative Test

Figure 3.1 outlines the steps employed in the preparation of charcoal tubes. The numbers 3.1 to 3.4 refer to the sections of this report where each step is described.

3.1 Preparation of Tanks Containing Known Solvent-in-Air Mixtures

A major requirement of this project was to prepare quantitative mixtures of each of the solvents in air so that these could be used to deposit known quantities of solvent on the charcoal tubes. Standard gas mixtures were of two types: (a) primary mixtures prepared directly from the liquid solvent, and (b) secondary mixtures prepared by diluting the primary solvent-in-air mixture. For each solvent, at least one primary and one secondary gas mixture were used to cover the range of concentrations required.

3.1.1 Primary Mixtures

Basically, these mixtures were made by vacuum-distilling a solvent into a size 1A cylinder (43 liter capacity) and subsequently pressurizing with air. Before filling these tanks with solvent, the entire system was evacuated; the solvent, contained in a small glass container, was then allowed to distill until the vapor pressure in the system reached a predetermined value which depended on the final desired concentration in the tank and the volatility of the solvent at room temperature. The tank was then pressurized from a cylinder containing compressed air. Next, the tank

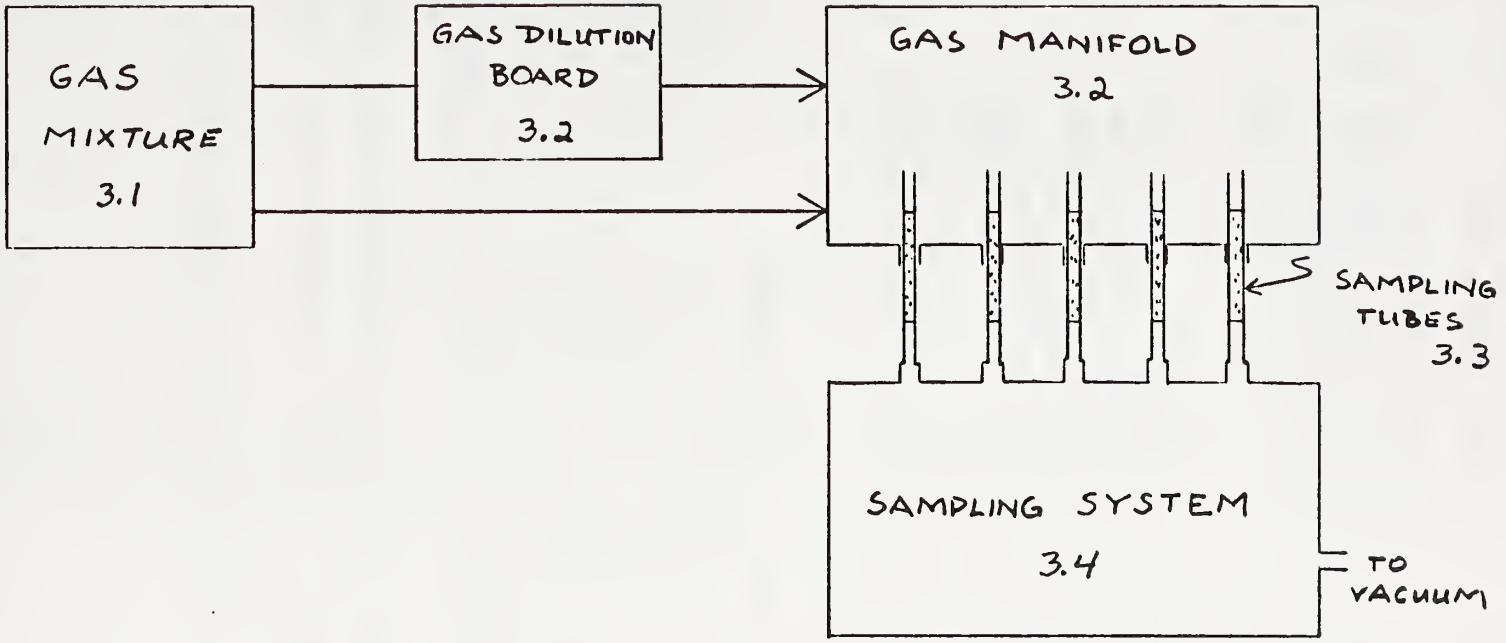


Figure 3.1 Outline of procedure for preparing charcoal tubes.

was heated, using an infrared lamp, to effect complete mixing by convection. A final pressure reading was taken after the tank returned to room temperature.

The concentration of the resulting mixture was determined in either of two ways. One involved the calculation of the concentration based on the ideal gas law (at low concentrations these solvents approach ideality); the second method involved weighing the actual amount of solvent which distilled into the tank [1].

3.1.2 Secondary Mixtures

These mixtures were prepared by transferring a portion of the primary tank mixture to an evacuated second tank and then pressurizing the second tank. These mixtures were usually one tenth as concentrated as the primary ones. By measuring the pressure after transfer of the primary mixture and the final pressure after addition of the diluent gas, the final concentration may be calculated. (This procedure was later found to be uncertain and not suitable for the quantitative preparation of solvent-in-air mixtures. See Section 6.0.)

3.2 Dilution of Tank Mixtures

Further dilution of the tank mixtures was often necessary and for this purpose a gas dilution system was used. This consists of two separately controlled gas flow systems, each containing a flowmeter to monitor the flow, a valve to adjust the flow and a flow controller to keep the flow constant. One stream contains the tank mixture while the other contains the diluent gas (purified air). The two streams are combined in a gas mixer and the resulting mixture then flows to a gas manifold where the charcoal tubes may be filled.

3.2.1 Construction and Calibration of Gas Dilution System

Figure 3.2 shows a diagram of the main components of the gas dilution system.

The following materials were used to construct the system:*

*Mention of a product here and elsewhere in the report does not constitute endorsement by the National Bureau of Standards, nor does it imply that the product is necessarily the best available for the purpose.

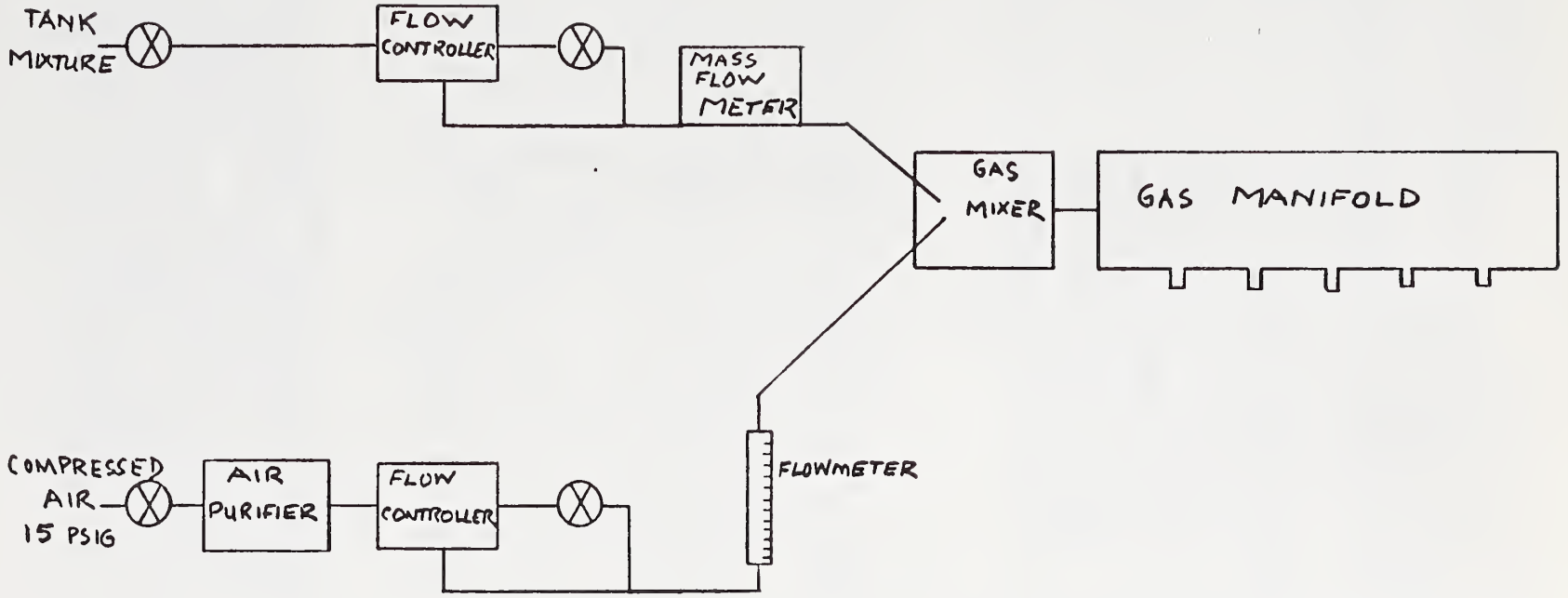


Figure 3.2 Diagram of gas dilution system.

For The Tank Mixture Flow:

Hastings Mass Flow Meter: 3 sizes used, 1000 cc/min, 5000 cc/min, 10000 cc/min

Moore Flow Controller (constant differential type) Model 63 BD

Whitey Valve (brass) 1RS4

For The Compressed Air Flow:

Air Purifier -- Granular charcoal and silica gel

Brooks Rotameter (Sho-Rate) Type: 1355-00C9AAA

Moore Flow Controller (Constant differential type) Model 63 BD-L

Whitey Valve (brass) 4RS4

A 3-liter wet-test meter was used to calibrate all of the flowmeters used. The total system flow was kept at least 1.5 times the combined flow rates of the sampling orifices.

3.2.2 Use of Gas Dilution System to Fill Charcoal Tubes

The filling of tubes was accomplished by raising the apparatus containing the tubes, until the tubes protruded about half-way into the gas manifold. Vacuum was then applied to the sampling system and continued for a total of exactly five minutes. The tubes were constantly bathed by the gas mixture during the sampling period. There are annular spaces between the manifold ports and the sampling tubes that allow excess gas mixture to flow out to atmosphere, thereby making it possible to sample at atmospheric pressure without room air being drawn into the apparatus.

3.3 Charcoal Tubes

The charcoal tubes were manufactured by Mine Safety Appliance Company in accordance with the recommendations of NIOSH [2]. Several of the tubes were selected at random and tested for resistance to air flow. The tubes so tested varied over a range of less than 1 percent; that is the flow through all these tubes, using a critical orifice, did not vary as much as 1 percent. Thus, these sampling tubes were considered to be interchangeable with respect to flow rate.

3.4 The Sampling System

3.4.1 Design and Calibration of Sampling Orifices

The sampling system, shown in Figure 3.3, is constructed of 3/8" pipe with five evenly spaced ports (corresponding to the ports of the gas manifold). In each of these ports, a fitting is attached. Each fitting contains a 20 gauge hypodermic needle, two inches long, which is brazed into the fitting to form an air-tight seal. Atop the fitting is installed a nominal 60 micron filter (Nupro Co.) to protect the hypodermic needle from particulate contamination. Under vacuum, hypodermic needles function as critical orifices and the gage and length of these needles were chosen to provide a flow of approximately 2 liters/minute [3]. These critical orifices, even with the charcoal tubes attached, function to provide a virtually constant flow. The flow data (calibrated with a wet test meter) for these orifices with tubes in place are given in Table 3.1.

3.4.2 Sampling System in Use

Five charcoal tubes are attached to the sampling system with short sections of rubber tubing. The sampling system is then raised and the tubes are inserted halfway into the gas manifold in which the sample stream is flowing. The vacuum is turned on and sampling is continued for exactly five minutes. The vacuum is then shut off and the sampling system is lowered. This procedure produces five sampling tubes containing reproducible quantities of adsorbed solvents.

3.5 Analysis of Charcoal Tubes

3.5.1 Selection of Tubes for Analysis

A total of 35 tubes was initially prepared at each concentration level for each solvent. From these 35 tubes, five were retained for analysis. Of these five, two were selected, analyzed and reported in the December 1, 1972 report.

Figure 3.4 is a representation of the thirty five tubes at each concentration level. The letters A through E refer to the corresponding sampling orifices. The numbers 1 through 7 refer to the run number. Seven runs were necessary in order to prepare 35 tubes at a given concentration level. The shaded boxes represent those tubes selected for retention. In all cases, tube "C" was chosen for analysis, along with another tube from the remaining four shaded boxes. Thus, for the five concentration levels in the benzene series, the following tubes were analyzed and the results were listed in the December 1, 1972 report.

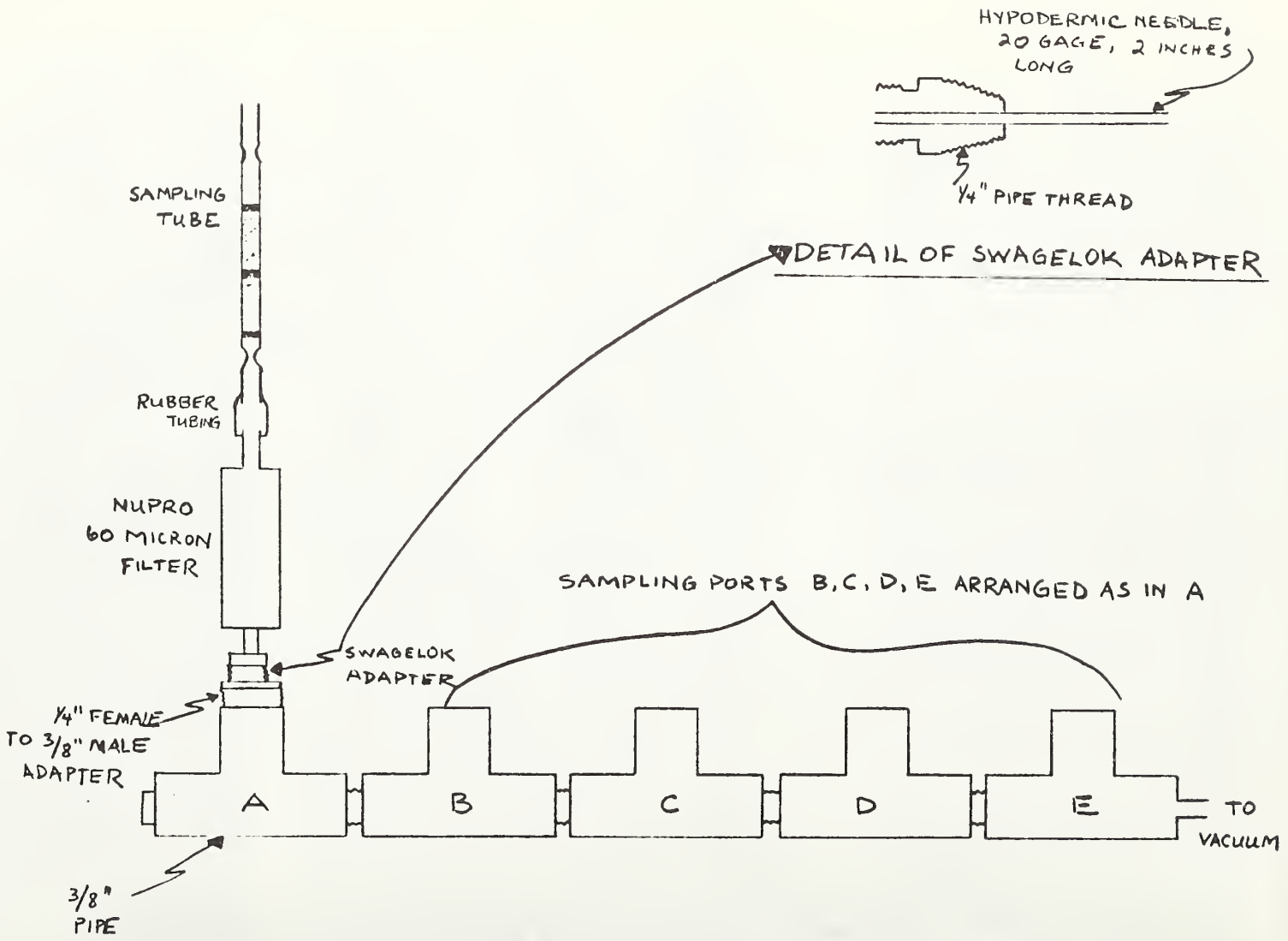


Figure 3.3 Sampling system.

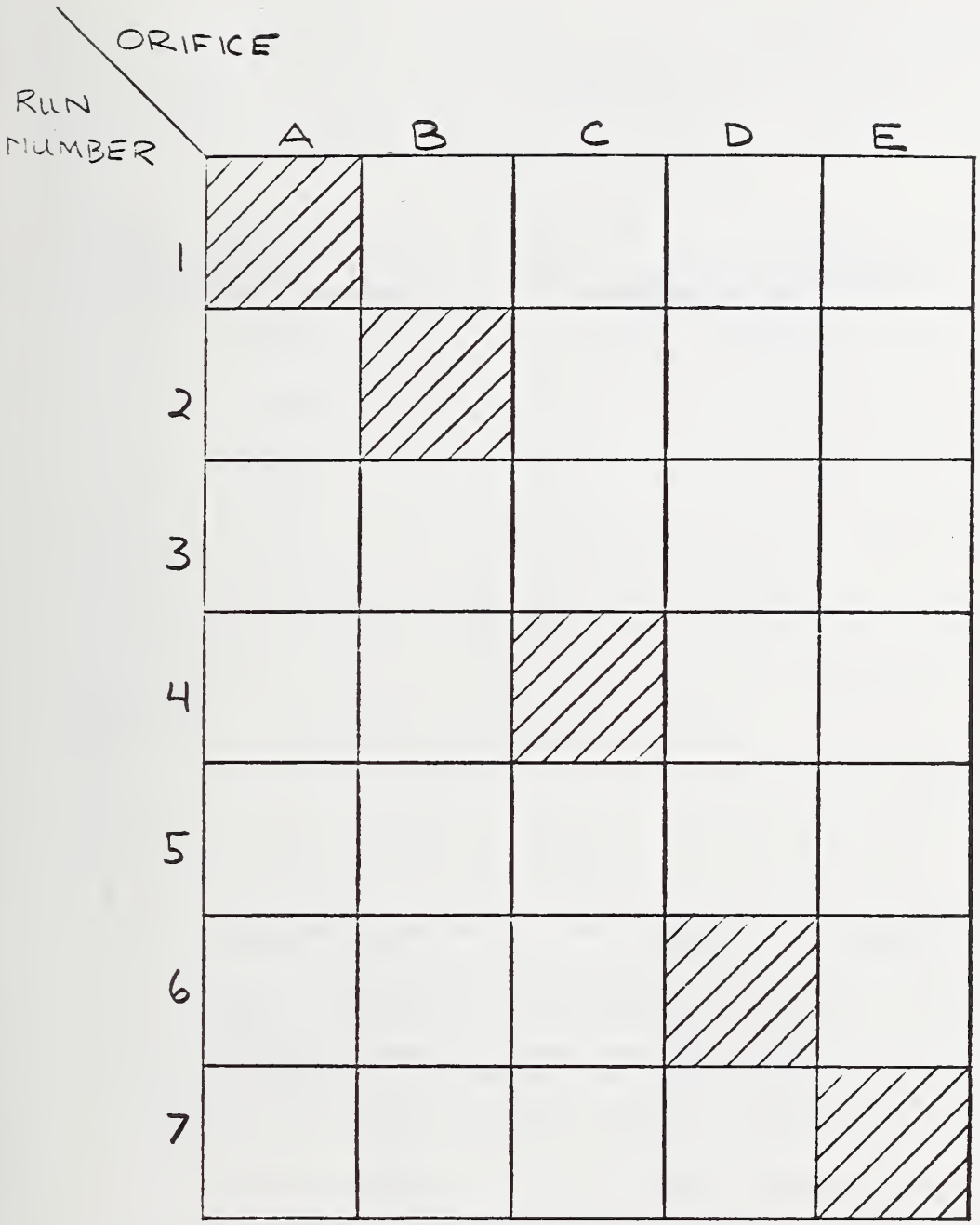


Figure 3.4 A representation of the thirty-five tubes prepared at each concentration level.

Table 3.1

Orifice Flow Rates

Orifice	Flow rate (l/min)	Ratio: $\frac{\text{Orifice x}}{\text{Orifice C}}$
A	1.819	1.085
B	1.624	0.9687
C	1.676	1.000
D	1.741	1.039
E	1.715	1.023

For Concentration Level 1: Tubes A and C

For Concentration Level 2: Tubes B and C

For Concentration Level 3: Tubes D and C

For Concentration Level 4: Tubes E and C

For Concentration Level 5: Tubes A and C

Since that time, some of the tubes originally sent to NIOSH and unused have been returned to NBS for reanalysis.

3.5.2 Revised Values for Charcoal Tubes

The method of preparation of the charcoal tubes used for the NIOSH collaborative tests should be capable of producing tubes with a high degree of reproducibility. Because the tubes were needed before all aspects of their analysis could be verified, they were released with provisional values.

The provisional values were based upon the following data. The tubes were analyzed by a gas chromatographic method and these values were compared with those calculated from gas flow and gas concentration data. This procedure gave greater weight to the latter which was subsequently found to be unjustified. Furthermore, the analytical method, as originally used, introduced more error than was tolerable. The source of this error was found to lie in the manner of preparation of calibration standards. Improvements described below overcame the analytical deficiencies, and an improved gravimetric gas-analysis technique (see section 6.0) will give greater confidence in the determined composition of tank mixtures in the future. However, since the gas mixtures were exhausted during preparation of the tubes, the only feasible means of establishing the concentration levels for the original lot of tubes was analysis of the remaining tubes by the improved chromatographic procedure, so this was done.

The revised analytical procedure uses calibration solutions prepared by using micropipets to deliver the solvent into volumetric flasks containing carbon disulfide. A further modification consists in the use of 2 ml vials with screw-top Teflon sliding-valve tops (Mininert Valves, Precision Sampling Corp., Baton Rouge, Louisiana) for desorption of the solvent from the charcoal adsorbent. This type of vial and closure is also useful for storage of standards, to prevent differential evaporative losses. In fact, standards have been stored in this manner for three weeks without measurable changes in concentration. The modified procedure is described more fully in section 5.0 of this report.

The revised values for the charcoal tubes prepared for collaborative testing are given in Tables A.1 to A.7, respectively, in Appendix A. These values are based on the analysis of at least three tubes at each concentration level.

Six standards were prepared for each solvent and the gas chromatographic response was plotted with respect to concentration, to yield linear calibration curves. Both gravimetric and volumetric standards were prepared and differences between duplicate standards did not exceed 3 percent.

Three tubes were analyzed for each concentration level, using the procedure described in Section 4.0. At least two injections were run for each tube and replication within 2 percent of the G.C. signal was ordinarily observed. For purposes of statistical comparison and convenience, all measurements were normalized to values that would have been collected at the "C" position. The values, so calculated, agreed, on the average, within ±2 percent. From these considerations, it is believed that the values assigned in the tables in this Appendix are precise within ±2 percent. That is to say, all tubes in the collaborative test lot are expected to have a relative agreement within ±2 percent of the tabulated values.

It is conservatively estimated that the tabulated values are within ±5 percent of the "true" values. This reflects the fact that different calibration solutions may vary by 3 percent, the agreement among tubes is 2 percent, and multiple chromatographic injections give signals which have a range of about 2 percent.

4.0 Analytical Procedure

The following is a brief description of the procedure used for analyzing charcoal tubes.

The contents of the charcoal tube are transferred to a 2 ml vial fitted with a screw-cap Teflon Mininert valve. One ml of CS_2 is added and the vial is capped. After desorption is achieved, the valve is opened, which permits sampling with a syringe without allowing evaporation of the solution.

An aliquot is then injected into the gas chromatograph, using the following modified syringe technique:

> Two µl of CS_2 are drawn into the syringe and the plunger drawn back to leave a 2 µl air space. A 5 µl aliquot of sample is then drawn into the syringe, and the plunger is drawn back so that the entire aliquot is

visible in the barrel of the syringe. This enables the analyst to read the exact volume of the aliquot and to verify that no air bubble is present.

The gas chromatographic signal is then converted to μg/tube by reference to a calibration curve. The calibration standards are prepared by using micropipets to deliver a known volume of solvent into a 25 ml or 50 ml volumetric flask. (Micropipets have been reported to be more accurate than microsyringes (4)). These initial standards are then diluted in the usual manner to produce a set of calibration standards.

Duplicate gas chromatographic determinations usually give results that agree within 2 percent. For those cases where the range is greater than 2 percent a third measurement is made.

5.0 Suggested Modifications of the Standard Charcoal Tube

The standard charcoal tube provides two sections of absorber, separated with foam plugs. When both sections are analyzed, the absence of solvent in the back section should serve as an indication of efficiency of sample collection. However, with certain organic solvents the solvent has been observed to migrate from the front to the rear section during storage, hence conclusions based on the two measurements are not possible for these cases [5].

In figure 5.1, a modified charcoal tube is shown which differs from the standard tube in that the two sections of charcoal are separated. In the field, this tube is used to sample atmospheres in the same manner as the standard tube, but upon completion of the sampling, the tube is broken at the construction between the two sections and both sections are sealed with plastic caps. This prevents the apparent migration of solvent vapors, upon storage, from the front to the rear portion of the tube. When both portions of this modified tube are analyzed it is possible to determine whether breakthrough has occurred in the front section of the tube.

6.0 New Gravimetric Procedure for the Analysis of Solvent-in-Air Mixtures

As pointed out earlier in the report, errors were made in determining the concentrations of some of the tanks used for filling the charcoal tubes. The error occurs when the assumption is made that a dilution of a primary tank mixture to produce a secondary tank mixture will yield a concentration of solvent vapor which is directly proportional to the dilution factor. The present work has shown that this error can be as large as 11 percent when, for example, chloroform at

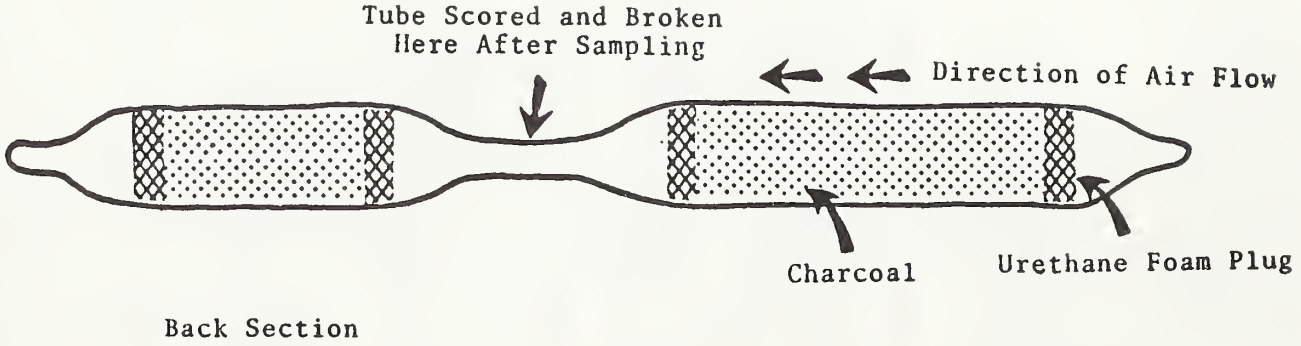


Figure 5.1 Modified charcoal tube

730 ppm is diluted tenfold. This phase of the work on solvents-in-air has concentrated on problems inherent in preparing and analyzing these mixtures.

In the preparation of these mixtures the solvent is introduced in the evacuated size 1-A tanks either by distilling the solvent or by injecting the solvent into the tank. The tank is then pressurized to about 1000 psia. The concentration may be determined from (1) the weight of the solvent admitted, the total volume of the tank and the final pressure; or (2) the pressure of the solvent vapor and the final pressure. In fact, these two measurements agree within about 2 percent when solvent-in-air mixtures in the range of 200-1000 ppm are prepared. The problem, then, occurs upon dilution of these tank mixtures to levels near the TLV range.

The dilution step, say from 600 ppm to 60 ppm may be achieved by withdrawing 10 percent of the original tank mixture and transferring it to a second tank of the same size. This second tank is then diluted with air to a final pressure of 10 times the pressure of the transferred gas. It is at this step that anomalies may present themselves -- i.e., the discrepancy between concentration based on pressure and dilution data and concentration based on gas chromatographic data. This can be seen by comparing the figures given below, in which the gas dilution data are compared with the gas chromatographic data.

Solvent	PPM Gas Method	PPM G.C. Method	Percent Difference $\frac{\text{G.C. - Gas}}{\text{Gas}} \times 100$
Benzene	202 31.7	202 28.8	-9.1%
Ethylene Dichloride	713 71.6	713 66.9	-6.6%
Trichloroethylene	1016 98.1	1016 90.9	-7.3%
Chloroform	728 73.5	728 65.3	-11%

Gas chromatographic analysis of gases is performed by transferring gas at known pressure and volume into the gas chromatograph equipped with a flame ionization detector. Response

of the gas chromatograph measured in terms of area of the resulting peak, is directly proportional to the amount of compound contained in the sample. Therefore, a gas sample which is one tenth as concentrated as a second gas sample should give a gas chromatographic response which is one tenth as large as the second sample. This response is shown to occur, for example, in tank mixtures of propane and methane. Similarly, liquid solutions of each of the seven solvents of interest (benzene, xylene, dioxane, chloroform, carbon tetrachloride, ethylene dichloride, and trichloroethylene) give linear gas chromatographic responses vs. concentration. However, in the gas mixtures of the seven solvents there are significant differences between calculated concentration ratios and ratios obtained by gas chromatography. Although it may be assumed, perhaps quite generally, that the gas chromatographic data are correct, a confirmatory procedure is required to prove this.

A direct method, and one which is now being used, involves collecting and weighing the solvent contained in a known volume of the mixture. In this method, a calibrated orifice is used to obtain a constant flow, the sampling time is carefully controlled, and a collection device -- a charcoal tube -- is carefully weighed before and after the sampling. The concentration can then be easily calculated.

In this procedure the charcoal tube is first connected to a sampling orifice and pure air is drawn through until the tube registers a constant weight after which the solvent-in-air mixture is drawn through the tube for a certain length of time. The weight gain of the tube and the flow through the tube are used to calculate the concentration of the gas mixture. The method provides very reproducible results. An extensive report on this procedure, including results obtained with six solvents, will be published soon [6].

8.0 Charcoal Tube SRM's

The investigations carried out under this Interagency Agreement have demonstrated the feasibility of producing SRM's for the analysis of the organic vapor content of the industrial atmosphere. By the preparation of reference tubes in large lots, and the use of the improved techniques for analysis of the gas mixtures and for verification analysis of the tubes, certificate uncertainties of no more than ± 3 percent would appear to then be possible.

For the preparation of the original 1000 charcoal tubes, a sampling system was used which provided constant flow at

each of five orifices -- but not identical flow. In fact, the range of flow varied by about 10 percent. Thus, five different values were listed for the charcoal tubes at each nominal concentration level. For the SRM's, it was decided to supply tubes, at each concentration level, that could be certified to contain the same amount of solvent, within a more limited range.

Since several thousand tubes are required to be filled for the SRM's, it was necessary to redesign the sampling manifold and to develop a procedure which would provide matched orifices in sufficient number to insure that the tubes could be filled in a reasonable length of time.

The final design, shown in Figure 7.1, has provision for 20 critical orifices. The orifices are mounted on a hollow brass cylinder, 16.5 cm (6.5 in.) in diameter and 1.9 cm (0.75 in.) tall. Each critical orifice is a hypodermic needle (22 gauge) selected so that the range of flow through all orifices vary by no more than 1.5 percent. The selection of the needles necessitated the testing of about 200 needles for flow characteristics. The needles were fitted into Luer-loks (obtained from hypodermic syringes) and were made vacuum-tight with the aid of stopcock grease. The Luer-loks were soldered into the baseplate. This arrangement makes it possible to remove or replace needles when necessary.

Atop the needles are placed Nupro filters to protect the needles from contamination by particulates. The charcoal tubes are attached by rubber tubing to the apparatus. In use, the cylinder baseplate is connected to a vacuum source.

A sketch of the arrangement for filling the tubes is shown in Figure 7.2.

The analytical work to verify the concentration levels of the tubes will be simplified by the fact that matched orifices will be used in their production. The modified procedure described in Section 4.0 of this report will be used to analyze tubes statistically selected from the production lot.

Table 7.1 lists the seven solvents and the ppm levels of interest. The final charcoal tube SRM's will contain quantities of solvent equivalent to that which would be collected in a 10 liter sample, at each of the concentration levels.

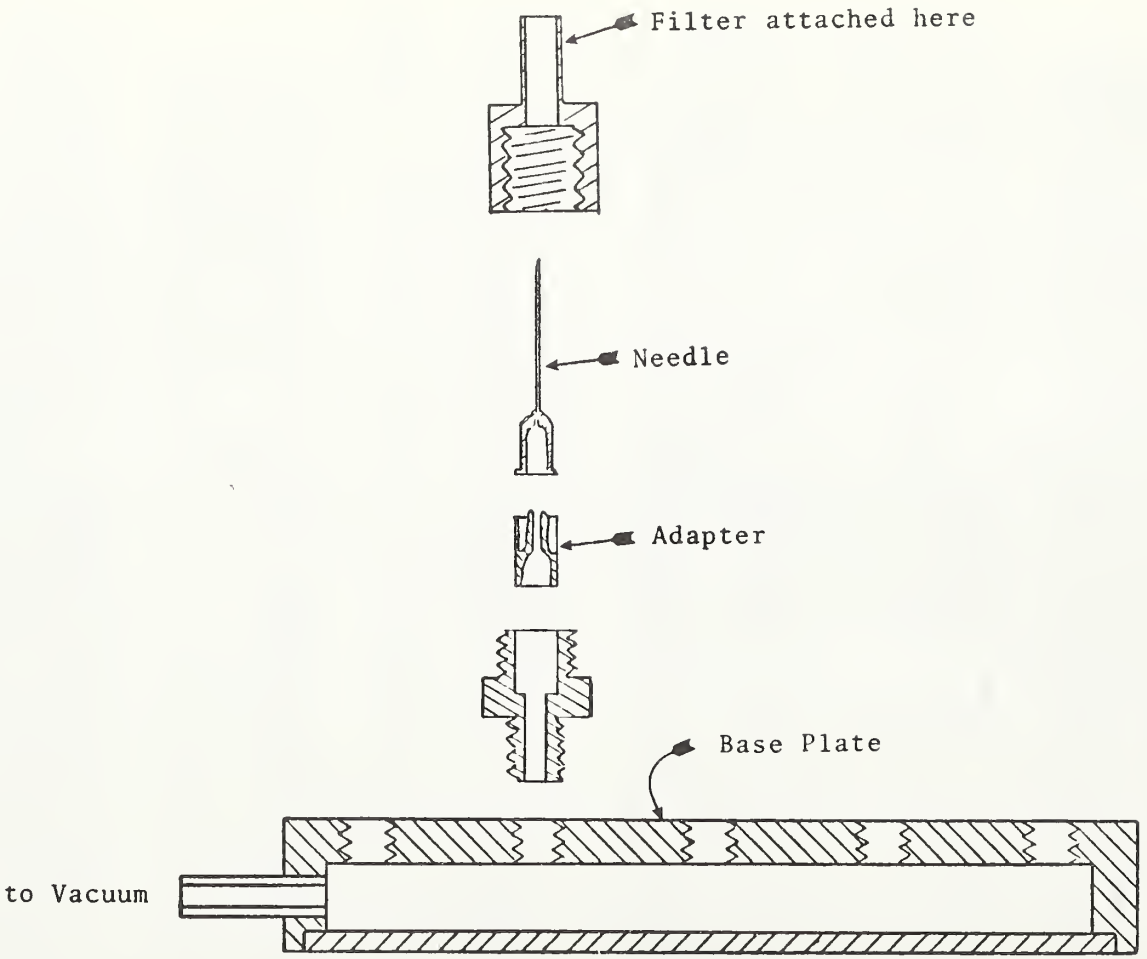


Figure 7.1 Diagram of SRM sample collection system.

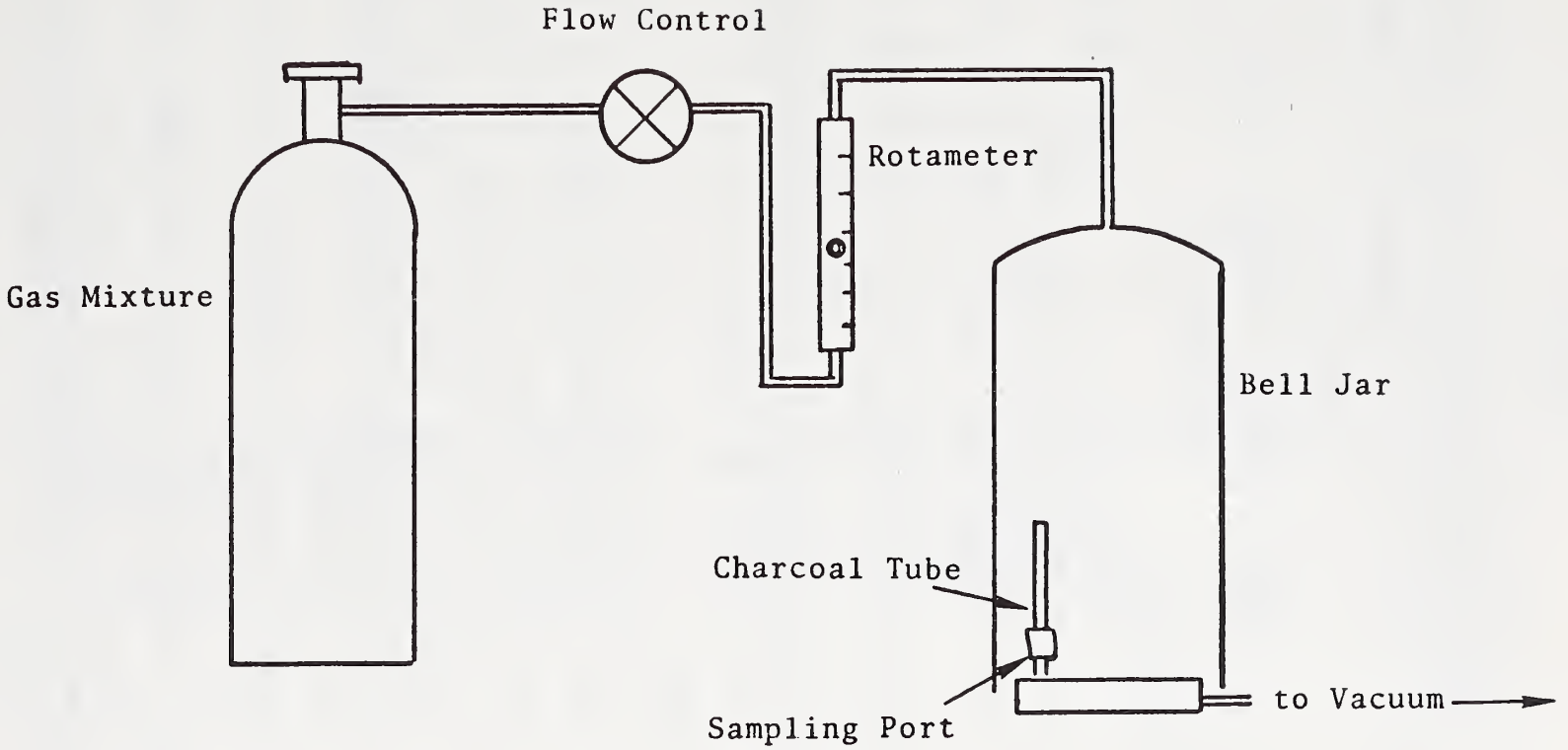


Figure 7.2 Arrangement for filling SRM tubes.

Table 7.1

Concentration Levels for Organic Solvents

Solvent	Concentration (ppm)			
Benzene	0.5	2	8	30
Xylene	1	7	50	300
p-Dioxane	0.5	4	30	200
Ethylene Dichloride	2.5	10	40	150
Chloroform	2.5	10	40	150
Trichloroethylene	5	20	80	300
Carbon Tetrachloride	0.5	2	8	30

8.0 Papers to be Submitted for Publication

It is expected that at least two publications will result from this work. Tentative titles are given:

"A Simple and Precise Critical Orifice Sampling Assembly", B. C. Cadoff, E. E. Hughes and J. K. Taylor

"A Gravimetric Procedure for the Determination of Solvent-in-Air Concentrations", B. C. Cadoff, B. Greifer, P. A. Pella and J. K. Taylor

9.0 References

[1] Some of these gas mixtures were prepared by Prof. L. C. Hall, visiting scientist at NBS.

[2] R. E. Kupel and L. D. White, "Report on a Modified Charcoal Tube", Amer. Ind. Hyg. Assoc. J. 32, 456 (1971).

[3] J. P. Lodge, Jr., J. B. Pate, B. E. Ammons, and G. A. Swanson, "The Use of Hypodermic Needles as Critical Orifices in Air Sampling", J. Air Pollution Control Assoc. 16, 197 (1966).

[4] C. F. Emanuel, "Delivery Precision of Micropipets", Anal. Chem. 45, 1568 (1973).

[5] Personal Communication from Dr. Alexander Teass.

[6] B. C. Cadoff, B. Greifer, P. A. Pella and J. K. Taylor, "Development of Contaminant Generation Systems for Certification of Portable Air Sampling Instruments", NBSIR 74-573.

Appendix A

Tables A.1 to A.7 list the revised values for seven sets of charcoal tubes containing organic solvents which were prepared for NIOSH collaborative tests. These values replace the preliminary values reported December 1, 1972.

Table A.1

REVISED VALUES - CHARCOAL TUBES

BENZENE

Concentration Level	Tube Numbers	µg/Tube
I (Nominal 0.5 ppm)	14A – 20A	15.3
	14B – 20B	13.7
	14C – 20C	14.1
	14D – 20D	14.7
	14E – 20E	14.4
II (Nominal 6 ppm)	7A – 13A	132
	7B – 13B	118
	7C – 13C	122
	7D – 13D	127
	7E – 13E	125
III (Nominal 10 ppm)	21A – 27A	318
	21B – 27B	284
	21C – 27C	293
	21D – 27D	304
	21E – 27E	300
IV (Nominal 18 ppm)	28A – 34A	548
	28B – 34B	489
	28C – 34C	505
	28D – 34D	525
	28E – 34E	517
V (Nominal 50 ppm)	0A – 6A	1535
	0B – 6B	1371
	0C – 6C	1415
	0D – 6D	1470
	0E – 6E	1448

Table A.2

REVISED VALUES - CHARCOAL TUBES

XYLENE

Concentration Level	Tube Numbers	µg/Tube
I (Nominal 5 ppm)	196A – 202A	186
	196B – 202B	166
	196C – 202C	171
	196D – 202D	178
	196E – 202E	175
II (Nominal 60 ppm)	203A – 209A	1911
	203B – 209B	1706
	203C – 209C	1761
	203D – 209D	1830
	203E – 209E	1802
III (Nominal 100 ppm)	217A – 223A	4246
	217B – 223B	3791
	217C – 223C	3913
	217D – 223D	4066
	217E – 223E	4003
IV (Nominal 150 ppm)	210A – 216A	5535
	210B – 216B	4941
	210C – 216C	5101
	210D – 216D	5300
	210E – 216E	5218

Table A.3

REVISED VALUES - CHARCOAL TUBES

p-DIOXANE

Concentration Level	Tube Numbers	µg/Tube
I (Nominal 5 ppm)	189A – 195A	141
	189B – 195B	126
	189C – 195C	130
	189D – 195D	135
	189E – 195E	133
II (Nominal 60 ppm)	126A – 132A	1638
	126B – 132B	1463
	126C – 132C	1510
	126D – 132D	1569
	126E – 132E	1545
III (Nominal 100 ppm)	161A – 167A	2978
	161B – 167B	2659
	161C – 167C	2745
	161D – 167D	2852
	161E – 167E	2808
IV (Nominal 150 ppm)	147A – 153A	4378
	147B – 153B	3909
	147C – 153C	4035
	147D – 153D	4192
	147E – 153E	4128

Table A.4

REVISED VALUES - CHARCOAL TUBES

ETHYLENE DICHLORIDE

Concentration Level	Tube Numbers	µg/Tube
I (Nominal 2.5 ppm)	175A – 181A	92.6
	175B – 181B	82.6
	175C – 181C	85.3
	175D – 181D	88.6
	175E – 181E	87.3
II (Nominal 30 ppm)	133A – 139A	1039
	133B – 139B	928
	133C – 139C	958
	133D – 139D	995
	133E – 139E	980
III (Nominal 50 ppm)	182A – 188A	1958
	182B – 188B	1749
	182C – 188C	1805
	182D – 188D	1875
	182E – 188E	1847
IV (Nominal 90 ppm)	154A – 160A	3280
	154B – 160B	2928
	154C – 160C	3023
	154D – 160D	3141
	154E – 160E	3093
V (Nominal 200 ppm	140A – 146A	7213
	140B – 146B	6440
	140C – 146C	6648
	140D – 146D	6907
	140E – 146D	6801

Table A.5

REVISED VALUES - CHARCOAL TUBES

CHLOROFORM

Concentration Level	Tube Numbers	µg/Tube
I (Nominal 2.5 ppm)	168A – 174A	106
	168B – 174B	94.7
	168C – 174C	97.8
	168D – 174D	102
	168E – 174E	100
II (Nominal 30 ppm)	105A – 111A	1265
	105B – 111B	1130
	105C – 111C	1166
	105D – 111D	1212
	105E – 111E	1193
III (Nominal 50 ppm)	112A – 118A	2027
	112B – 118B	1810
	112C – 118C	1868
	112D – 118D	1941
	112E – 118E	1911
IV (Nominal 90 ppm)	119A – 125A	3992
	119B – 125B	3564
	119C – 125C	3679
	119D – 125D	3823
	119E – 125E	3764

Table A.6

REVISED VALUES - CHARCOAL TUBES

TRICHLOROETHYLENE

Concentration Level	Tube Numbers	µg/Tube
I (Nominal 5 ppm)	42A – 48A	244
	42B – 48B	218
	42C – 48C	225
	42D – 48D	234
	42E – 48E	230
II (Nominal 60 ppm)	77A – 83A	3062
	77B – 83B	2734
	77C – 83C	2822
	77D – 83D	2932
	77E – 83E	2887
III (Nominal 100 ppm)	35A – 41A	5126
	35B – 41B	4576
	35C – 41C	4724
	35D – 41D	4908
	35E – 41E	4833
IV (Nominal 180 ppm)	91A – 97A	9435
	91B – 97B	8424
	91C – 97C	8696
	91D – 97D	9035
	91E – 97E	8896
V (Nominal 300 ppm)	84A – 90A	15369
	84B – 90B	13722
	84C – 90C	14165
	84D – 90D	14717
	84E – 90E	14491

Table A.7

REVISED VALUES - CHARCOAL TUBES

CARBON TETRACHLORIDE

Concentration Level	Tube Numbers	µg/Tube
I (Nominal 0.5 ppm)	63A – 69A	38.1
	63B – 69B	34.0
	63C – 69C	35.1
	63D – 69D	36.5
	63E – 69E	35.9
II (Nominal 6 ppm)	70A – 76A	308
	70B – 76B	275
	70C – 76C	284
	70D – 76D	295
	70E – 76E	291
III (Nominal 10 ppm)	49A – 55A	671
	49B – 55B	599
	49C – 55C	618
	49D – 55D	642
	49E – 55E	632
IV (Nominal 18 ppm)	56A – 62A	1102
	56B – 62B	984
	56C – 62C	1016
	56D – 62D	1056
	56E – 62E	1039
V (Nominal 200 ppm)	98A –104A	11635
	98B –104B	10387
	98C –104C	10723
	98D –104D	11141
	98E –104E	10970

Appendix B

Consensus Values

The initial lot of charcoal tubes described in the report was analyzed by a number of collaborating laboratories. The data reported by them has kindly been made available to us by Dr. Alexander Teass of NIOSH. For convenience of intercomparison, the values reported were first normalized to what would have been expected if the tube used had been prepared at the "C" orifice, by multiplying by the ratio of the flow rates of the respective orifices. Tables B.1 to B.7 summarize the reported values after normalization. The data, so normalized, were used to arrive at consensus values for the charcoal tubes.

If all measurement errors are random, the average of all values reported (consensus value) should be in good agreement with the "true" value. A comparison of the average values with the NBS values indicates large discrepancies, especially for the lowest concentration levels.

A cursory inspection of the data presented in Tables B.1 to B.7 will identify a few values which are obviously outliers and hence unduly influence the consensus values. While no statistical rejection criteria were employed, the values considered to be grossly in error were encircled, as indicated in the tables, and these were eliminated before obtaining the new average or consensus values given in each table, and identified as "Avg (Adj.)". It is obvious that these values are in much better agreement with the NBS values, and lend additional confidence to their validity.

TABLE B.1 Benzene

	CL I	CL II	CL III	CL IV	CL V
LAB 2	20.00	142.20	331.30	488.30	1520.00
LAB 3	65.00	345.40	819.40	1269.20	4089.70
LAB 4A	14.60	124.00	302.40	464.50	1459.10
LAB 4B	13.10	117.00	297.70	510.10	1393.20
LAB 5	17.30	130.50	307.00	482.50	1467.40
LAB 6	12.80	132.80	274.50	474.50	2060.00
LAB 7	17.10	125.90	312.80	469.20	1491.80
LAB 8	15.60	124.90	298.60	450.70	1446.60
LAB 9	21.20	110.80	292.30	451.40	1616.10
LAB 10	15.00	129.00	308.00	519.10	1474.00
LAB 11	17.60	128.00	338.00	541.80	1594.40
LAB 12	14.30	115.20	276.60	447.30	1315.40
LAB 13	14.40	127.90	302.60	496.10	1459.30
Avg. (All)	19.85	142.58	343.17	543.44	1722.08
Avg. (Adj)	16.08	125.68	303.48	482.96	1353.11
NBS Values	14.10	122.00	293.00	505.00	1415.00

TABLE B.2 Xylene

	CL I	CL II	CL III	CL IV
LAB 1	196.30	1796.90	4187.00	5014.40
LAB 2	197.30	1978.00	4513.70	5938.70
LAB 3	128.00	1856.40	4420.60	5633.50
LAB 4A	200.20	2135.50	4767.70	6161.00
LAB 4B	190.90	1749.80	4038.10	5131.90
LAB 5	186.40	1789.30	4007.80	5183.70
LAB 6	253.50	1655.40	4220.00	5611.20
LAB 7	142.70	1645.80	3840.20	5249.30
LAB 8	174.40	1789.20	3940.30	5018.60
LAB 9	562.50	1914.00	3536.00	4827.70
LAB 10	143.00	1767.40	3986.50	5295.80
LAB 12	197.20	1893.60	4098.80	5451.20
Avg. (All)	214.37	1830.94	4129.73	5376.42
Avg. (Adj)	182.72	1830.94	4129.73	5376.42
NBS Values	171.00	1761.00	3913.00	5101.00

TABLE B.3 p-Dioxane

	CL I	CL II	CL III	CL IV
LAB 2	135.30	1570.50	2951.50	4250.30
LAB 3	138.60	1546.70	2676.10	4063.00
LAB 4A	131.80	1510.00	2660.80	4083.00
LAB 4B	141.40	1525.30	2840.00	4031.00
LAB 5	142.40	1493.60	2705.60	4035.00
LAB 8	145.70	1444.00	2763.00	3995.20
LAB 9	258.80	1585.60	2826.20	4193.80
LAB 10	152.00	1664.70	3019.60	4382.20
LAB 12	141.90	1621.40	2887.20	4245.20
Avg. (All)	154.21	1551.31	2814.44	4142.08
Avg. (Adj)	141.14	1551.31	2814.44	4142.08
NBS Values	130.00	1510.00	2745.00	4035.00

TABLE B.4 Ethylene Dichloride

	CL I	CL II	CL III	CL IV	CL V
LAB 2	94.80	975.20	1759.40	2964.20	6430.40
LAB 3	93.60	1009.70	2205.00	3595.80	7173.20
LAB 4A	112.50	1042.40	1927.00	3140.80	6836.80
LAB 5	87.30	950.90	1712.90	2998.70	6299.80
LAB 6	101.10	1193.50	2889.60	3673.90	8390.00
LAB 7	144.00	1045.90	1871.00	3180.00	7050.00
LAB 9	155.00	1037.70	1920.20	3066.60	7252.30
LAB 10	89.00	985.00	1795.70	3050.00	6483.90
LAB 12	85.70	978.20	1780.10	3018.40	6441.70
Avg. (All)	107.00	1024.28	1984.54	3187.60	6928.68
Avg. (Adj)	94.86	1024.28	1871.41	3059.81	6746.01
NBS Values	85.30	958.00	1805.00	3023.00	6648.00

TABLE B.5 Chloroform

	CL I	CL II	CL III	CL IV
LAB 1	88.60	1187.70	1289.40	2546.50
LAB 2	155.00	1270.90	1941.90	3793.90
LAB 3	261.90	2295.10	3480.50	6864.30
LAB 4A	95.80	1111.40	1736.80	3339.20
LAB 4B	95.60	1200.40	1830.90	3579.00
LAB 5	670.10	1668.10	2320.50	4138.20
LAB 6	117.00	1200.00	1960.80	3271.90
LAB 7	101.70	1210.00	1935.50	3792.80
LAB 8	103.00	1228.10	1931.90	3605.00
LAB 9	57.80	1222.30	1677.40	3890.60
LAB 10	94.00	1215.70	1864.00	3563.10
LAB 12	107.30	1220.20	1900.60	3668.50
Avg. (All)	162.32	1335.83	1989.18	3837.75
Avg. (Adj)	101.58	1206.67	1864.42	3664.22
NBS Values	97.80	1166.00	1868.00	3679.00

TABLE B.6 Trichloroethylene

	CL I	CL II	CL III	CL IV	CL V
LAB 2	281.10	2687.80	4594.70	8664.00	14736.50
LAB 3	206.90	1935.50	3420.60	5331.00	10500.00
LAB 4A	257.10	2916.30	4791.70	8839.70	14244.00
LAB 4B	262.10	2872.90	4770.90	8813.20	14122.00
LAB 5	979.30	3655.40	5692.80	9052.30	14626.50
LAB 6	260.80	3281.10	6018.40	10360.00	15789.00
LAB 7	248.00	2900.00	4829.70	8771.90	14300.00
LAB 8	247.00	2883.70	4656.40	8617.50	14151.00
LAB 9	867.20	2580.00	4296.20	7280.30	14994.00
LAB 10	245.00	2885.00	4769.00	8475.50	13817.00
LAB 12	256.00	2982.60	4867.80	8873.20	15270.00
Avg. (All)	373.68	2870.94	4791.65	8461.69	14231.82
Avg. (Adj)	251.56	2887.71	4668.98	8774.76	14605.00
NBS Values	225.00	2822.00	4724.00	8696.00	14165.00

TABLE B.7 Carbon Tetrachloride

	CL I	CL II	CL III	CL IV	CL V
LAB 2	47.30	281.60	568.60	943.30	10972.00
LAB 3	59.30	336.70	694.90	1128.60	11751.50
LAB 4A	32.70	289.40	608.90	1024.80	11136.00
LAB 4B	34.80	263.00	521.00	961.50	10978.00
LAB 5	88.50	239.40	550.30	884.90	10370.50
LAB 7	146.60	329.00	649.80	1016.60	11550.00
LAB 8	32.70	292.30	608.30	994.80	11040.00
LAB 9	18.30	211.70	461.90	710.00	8570.00
LAB 10	35.00	295.20	600.20	981.40	10936.00
LAB 12	65.50	378.80	778.80	1183.50	11034.00
LAB 13	44.00	311.50	603.70	962.50	11730.00
Avg. (All)	54.97	293.51	604.22	981.08	10915.27
Avg. (Adj)	38.01	293.51	604.22	981.08	10915.27
NBS Values	35.10	284.00	618.00	1016.00	10723.00

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