Monitoring Environmental Levels of Phosphine at a Rice Fumigation Site in Sacramento County, November, 1982

by

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SUMMARY

On November 22 and 23, 1982, levels of phosphine were monitored in a computer repair shop located in a large warehouse. Rice in bulk storage was located in a separate room of the warehouse. The rice storage area was tarp-fumigated November 19, 1982, with aluminum phosphide pellets. A few days after fumigation, employees of the computer repair shop detected a "foul odor" in their workspace. The employees were sent home from work after two hours and the computer repair shop was monitored for phosphine. The samples ranged from 0.05 to 0.4 ppm phosphine in the workspace. The Cal/OSHA permissable exposure level (PEL) for phosphine is 0.3 ppm. No reports of pesticide-related illnesses were received pertaining to this incident, but its occurrence stresses the need to consider the proximity of workers not involved in the fumigation process when choosing a site for commodity tarp-fumigation.

INTRODUCTION

Phosphine is a colorless gas with a disagreeable garlic-like odor, that is detectable below the PEL.[1] Phosphine is formed by the action of moisture on metallic phosphides. The chief use of phosphine is as a fumigant. Symptoms noted in workers excessively exposed to phosphine include diarrhea, nausea, vomiting, tightness of chest, coughing, headache and dizziness. These symptoms have been known to occur following intermittent exposures to levels of phosphine gas ranging from 10 to 35 ppm.[2] Exposure to phosphine is not considered to have cumulative effects. From 1975 to 1981 in California, 31 pesticide-related illnesses (systemic illnesses, skin or eye injuries) reported to the California Department of Food and Agriculture, were the result of exposure to phosphine or metallic phosphides.[3]

The American Conference of Governmental Industrial Hygienists (ACGIH) recommends a Threshold Limit Value (TLV) of 0.3 ppm for phosphine exposure on an eight-hour time weighted average (TWA) basis, and a short-term exposure level (STEL) of 1.0 ppm over a period of 15 minutes or less.

Approximately 50,000 tons of rice stored in the investigated rental warehouse space in Sacramento were tarp-fumigated with aluminum phosphide pellets. The warehouse was vacant except for a computer repair firm that was located in the warehouse in a separate room, approximately 100 feet (to the north) from the rice storage area. The commodity was fumigated on November 19, 1982, tarped, then sealed by closing all interior doors and covering the air conditioning ducts with polyethylene sheets. Warning signs were posted and the fumigation was to proceed for one week. On the morning of November 22, 1982, employees of the repair firm complained of a "foul odor" in their workspace. The supervisor sent all employees home after two hours of work and contacted the Sacramento County Agricultural Commissioner's Office to investigate the incident. The Agricultural Commissioner's Office contacted the Worker Health and Safety Unit to collect samples from the workspace for phosphine.

Employees of the pest control operator performing the fumigation were contacted and took steps to ventilate the warehouse. The air conditioning ducts were untarped and an employee donned a self-contained breathing apparatus and entered the rice storage area to open doors in an attempt to purge any gases from the building. The attempt was made to purge the gas to the south, away from the repair shop workspace.

PROCEDURES

Area samples were collected in the repair shop workspace approximately one hour after the workers left. Samples were collected near the back wall of the workspace, closest to the rice storage and fumigation site. Phosphine was trapped on mecuric cyanide impregnated silica gel absorbent tubes (SKC 226-10-05), drawn by MSA C-210 personal sampling pumps. Pumps were calibrated at 100 ml/min air flow using a bubble tube. Samples were drawn in pairs, the pumps located at work stations approximately 30 feet apart. Sampling times were approximately 30 and 40 minutes. One personal sample was drawn from the employee who entered the rice area to open doors. Grab

samples were drawn from locations within and outside the warehouse using instantaneous reading colorimetric detector tubes (Drager tube phosphine 0.1/a, stock no. CH 31101).

After collection, all silica gel tube samples were sealed with the supplied plastic caps. One unused tube was broken open outside the building about 100 feet from the sampling sites then sealed to serve as a storage control. All tubes were sealed in a glass jar with a screw cap and held in a freezer until analysis. Samples were analyzed using National Institute of Occupational Safety and Health (NIOSH) method S332 (see Appendix One).[4] Analysis was completed approximately two weeks after sampling.

RESULTS

Results from the silica gel tube samples are listed in Table One. Results from detector tube samples are listed in Table Two.

DISCUSSION

This study attempted to ascertain if the odor detected by employees in the repair shop workspace was related to off-target movement of phosphine from the rice fumigation. Low levels of phosphine were detected in the air samples collected in the workspace, however, based on this sampling, it is not possible to make any definitive conclusion about the presence of a phosphine exposure hazard.

Colorimetric detector tube samples were collected outside the warehouse towards the south to determine the effectiveness of ventilation. Three days after the fumigation commenced (November 22), measurements were collected in a room adjacent to the rice storage facility (not the repair shop) which showed levels of 2.5 and 3.5 ppm of phosphine. One measurement collected 30 feet south of the warehouse at 1100 hours, showed 2.0 ppm of phosphine. Another measurement, collected one hour later at 75 feet south of the warehouse showed no detectable level of phosphine. Two detector tube samples collected from within the repair shop workspace on the morning of November 23 each showed 0.1 ppm of phosphine, which correlates well with the silica gel tube samples collected later that morning.

Silica gel tube samples collected from the repair shop workspace on November 22 showed levels of phosphine ranging from 0.05 to 0.4 ppm. Samples colted from the workspace on November 23 showed 0.07 and 0.09 ppm phosphine.

One sample collected from the employee opening doors to the rice storage area showed 55 ppm, outside that individual's self-contained breathing apparatus. Breakthrough occurred in three silica gel tube samples, indicating that phosphine levels detected may not be precisely quantitative. Breakthrough was indicated by greater than 25 percent of the total amount of phosphine collected being detected in the back-up section. Significant losses of phosphine may have occurred, and the results from samples with breakthrough are reported as minimum amount present.[5] Speculation as to the cause of this occurence is that channeling was present in the silica gel absorbent bed which could permit incomplete trapping of phosphine from

the sampled air. Another hypothesis considered was that of cross contamination from the high level personal sample to the low level area samples during storage. This hypothesis initially came about when 0.62 micrograms were detected in the back-up section of the shipping control tube possibly indicating the migration of phosphine from tube to tube. However, when background interferences detected in the reagent blanks used in the analysis were subtracted from the analytical values, the level present in the shipping control becomes negligible. It is not known why breakthrough occurred in the low level samples, though breakthrough in the high level sample can be explained by overloading the capacity of the absorbent. This occurrence can be prevented when highly contaminated atmospheres are anticipated, by sampling with two absorbent tubes connected in series.

Since two of six samples collected from the repair shop workspace are of questionable validity, determining if hazardous levels of phosphine were present in the repair shop is not possible. No reports of possible pesticide related illnesses as related to this occurrence were received by the California Department of Food and Agriculture's Worker Health and Safety Unit. With two exceptions, both of these being samples with breakthrough, the measurements collected in the workspace were below the ACGIH recommended TLV of 0.3 ppm.

CONCLUSIONS

Low levels of phosphine were detected in a workspace located in a warehouse, part of which was used to store rice and to fumigate the rice with aluminum phosphide. However, due to breakthrough of phosphine in the sampling tubes, it is not possible to determine if hazardous levels of phosphine were present in the workspace. This occurrence stresses the need to consider the proximity of other work areas when siting bulk commodities scheduled for fumigation.

REFERENCES

1. Anonymous. Odor Thresholds for 53 Commercial Chemicals. Manufacturing Chemicals Association (1978).

2. ACGIH. Documentation of the Threshold Limit Values, Fourth Edition. (1980).

3. Anonymous. Pesticides Reported by Physicians in California as Causes of Illness in Employed Persons, 1975 to 1981. California Department of Food and Agriculture Reports HS No's. 256, 494 498, 644, 709, 857 and 981, available from the Worker Health and Safety Unit.

4. NIOSH. Manual of Analytical Methods, Volume 6. Method No. S332 (1978).

5. Linch, A.L. Evaluation of Ambient Air Quality by Personnel Monitoring, Vol. I. CRC Press (1981).

TABLE ONE

Silica Gel Sampling Tube Sampling Results

Date Sampled	Time Sampled	Sampling Time (min.)	Sample Type	Phosphine Detected (ppm)
11/22/82	1000-1040	40	Area[a]	0.07
11/22/82	1000-1040	40	Area	0.40[b]
11/22/82	1105-1132	27	Area	0.05
11/22/82	1105-1132	27	Area	0.06[b]
11/22/82	1030-1045	15	Personal	55.40[b,c]
11/23/82	1000-1040	40	Area	0.09
11/23/82	1000-1040	40	Area	0.07

[a] All area samples were collected from within the computer repair firm workspace.

[b] Breakthrough occurred in these sampling tubes, phosphine detected is the minimum amount present.

[c] Sample was collected outside the employee's respiratory protection device.

Table Two

Colorimetric Detector Tube Samples

Date Sampled	Time Sampled	Location Sampled	Phosphine Detected (ppm)
11/22/82	1030	Unused room adjacent to the rice storage area	2.5 3.5
11/22/82	1100	30 feet south of the warehouse	2.0
11/22/82	1200	75 feet south of the warehouse	None detected
11/23/82	0640	Within the repair shop workspace	0.1
11/23/82	0715	Within the repair shop workspace	0.1
11/23/82	0930	Inside rice storage area	50.0
11/23/82	1030	Warehouse exterior, south side	None detected

Phosphine

Analyte:	Phosphate	Method No.:	S332
Matrix:	Air	Range:	0.195-0.877 mg/cu m
OSHA Standard:	0.3 ppm (0.4 mg/cu m)	Precision ($\overline{CV}_T$):	0.091
Procedure:	Collection on coated silica gel, permanganate, extraction, colorimetry	Validation Date:	3/17/78

1. Synopsis

1.1 A known volume of air is drawn through a tube containing mercuric cyanide impregnated silica gel to trap the phosphine. The phosphorus is extracted and oxidized to phosphate using a hot, acidic permanganate solution.

1.2 The extracted sample is analyzed for phosphate by formation of the phosphomolybdate complex, extraction into a mixture of isobutanol and toluene and reduction using stannous chloride. The absorbance of the reduced phosphomolybdate complex is measured at 625 nm.

2. Working Range, Sensitivity and Detection Limit

2.1 This method was validated over the range of 0.195-0.877 mg/cu m at an atmospheric temperature and pressure of 19.0°C and 765.3 mm Hg, respectively, using a 16-liter sample. The method may be capable of measuring smaller amounts if the desorption efficiency is adequate. Desorption efficiency must be determined over the range used. The upper limit of the range of the method depends on the adsorptive capacity of the mercuric cyanide treated silica gel. This capacity may vary with the concentration of phosphine and other substances in the air. When an atmosphere at 90% relative humidity containing 0.957 mg/cu m of phosphine was sampled at a flow rate of 0.2 liter per minute, breakthrough was determined to occur at a sampling volume of 20.75 liters (capacity = 19.86 µg PH_3). To minimize the probability of overloading the sampling tube, the sample size recommended is less than two-thirds the 5% breakthrough capacity at >80% RH at twice the OSHA standard.

2.2 Sampling at 0.2 liter per minute for 80 minutes will yield a 16-liter sample with 6.4 µg of PH_3 collected at the OSHA standard level. The sensitivity of the method obtained from the slope of the absorbance vs µg of phosphine calibration curve is 0.0524 absorbance units per µg of PH_3.

2.3 The detection limit of the method determined from twice the standard deviation for the absorbance of six blank treated silica gel tubes corresponds to 0.19 μg PH_3 (or 0.0119 mg/cu m for a 16-liter sample).

3. Interferences

3.1 When two or more compounds are known or suspected to be present in the air, such information, including the suspected identities, should be transmitted with the sample.

3.2 The colorimetric determination of phosphate is subject to interference by any species which also forms a molybdate complex which is extractable into the isobutanol-toluene mix and absorbs at similar wavelengths.

3.3 Any phosphorus compound which is retained by the mercuric cyanide-coated silica gel tube and oxidized to phosphate by hot aqueous permanganate solution will be a major interference. Possible interfering species include PCl_3 and PCl_5 vapors and organic phosphorus compounds. Particulate H_3PO_4, P_4O_{10} and P_4S_{10} are also possible interferents unless a prefilter is used in conjunction with the sorbent tube. Although a prefilter has not been tested with the method, its use is recommended.

3.4 If the possibility of interferences do exist, modification of the analytical procedure must be made to circumvent the problems or an alternative procedure should be used.

4. Precision and Accuracy

4.1 The Coefficient of Variation ($\overline{CV}_T$) for the total sampling method in the range 0.195-0.877 mg/cu m was 0.0908. This value corresponds to a standard deviation of 0.0363 mg/cu m at the OSHA standard level. Statistical information and details of the validation and experimental test procedures can be found in References 11.1 and 11.2.

4.2 On the average, the concentration obtained at the OSHA standard level using the overall sampling and analytical procedure was 0.2% higher than the average taken concentration for a limited number of laboratory experiments. Any difference between the found and taken concentrations may not represent a bias in the sampling and analytical method but rather a random variation from the experimentally determined taken ("true") concentration. Also, collected samples, stored for at least seven days, are stable, thus no recovery corrections should be applied to the final result.

5. Advantages and Disadvantages

5.1 The sampling device is small, portable and involves no liquids.

5.2 The precision of the method is affected by the reproducibility of the pressure drop across the tubes. This drop will affect the flow rate and cause the volume to be imprecise, because the pump is usually calibrated for only one tube.

5.3 The analytical method requires measurement of the absorbance of the phosphomolybdate complex one minute after reduction with stannous chloride. This may present an inconvenience since the samples need to be handled individually.

5.4 A disadvantage of the method is that the amount of sample which can be collected is limited by the number of micrograms of phosphine that the tube will hold before overloading. When the amount of phosphine found on the backup section exceeds 25% of that on the front, the probability of sample loss exists.

6. Apparatus

6.1 Personal Sampling Pump. A calibrated personal pump whose flow rate can be determined within ±5% of the recommended flow rate. The pump must be calibrated with a representative tube in the line.

6.2 Treated Silica Gel Tube. Glass tube with both ends flame sealed 12-cm long with a 6-mm O.D. and a 4-mm I.D. containing two sections of treated silica gel (45/60 mesh, SKC, Inc.). The absorbing section contains 300-mg of the treated silica gel and the backup 150-mg. A small wad of silylated glass wool is also placed between the front adsorbing section and the backup section; a plug of silylated glass wool is also placed in the front of the adsorbing section and at the end of the backup section. The pressure drop across the tube must be less than 2 inches of mercury at a flow rate of 0.2 liter per minute.

The silica gel is coated according to the following procedure:

6.2.1 Dry one hundred grams of silica gel at 90°C for 2 hours.

6.2.2 Prepare a 2% w/v mercuric cyanide solution in water (2 g $Hg(CN)_2$ in 100 mL H_2O).

6.2.3 Add the dried silica gel to the mercuric cyanide solution and let set for 15 minutes with occasional stirring.

6.2.4 Drain the excess mercuric cyanide solution and dry the remaining silica gel at 90°C for 3 hours.

6.2.5 Cool the silica gel to room temperature in a covered beaker.

6.2.6 Expose the silica gel to a humid atmosphere (>80% RH) for 24 hours.

6.3 A spectrophotometer capable of measuring absorbance or transmittance at 625 nm.

6.4 Two matched 5-cm silica cells with tight fitting caps.

6.5 Separatory funnel, 125-mL.

6.6 Beakers, 50-mL.

6.7 Volumetric flasks, 10, 25, 100 and 1000-mL.

6.8 Pipets, 0.2, 10 and 25-mL and other convenient sizes to make standard dilutions.

6.9 Graduated cylinders, 10-mL.

6.10 Water bath (maintained at 65-70°C).

6.11 Syringes, 0.5 and 1.0-mL.

6.12 Balance.

6.13 Barometer.

6.14 Thermometer.

6.15 Stopwatch.

7. Reagents

All reagents should be ACS reagent grade or better.

7.1 Water, distilled or deionized.

7.2 Potassium dihydrogen phosphate, anhydrous, KH_2PO_4.

7.3 Sulfuric acid, concentrated.

7.4 Ammonium molybdate, $(NH_4)_6 Mo_7O_{24} \cdot 4H_2O$.

7.5 Ferrous ammonium sulfate, $Fe(NH_4)_2(SO_4)_2$.

7.6 Potassium permanganate, $KMnO_4$.

7.7 Stannous chloride, $SnCl_2$.

7.8 Glycerol.

7.9 Toluene.

7.10 Isobutanol.

7.11 Methanol.

7.12 Standard phosphate solution. Prepare by dissolving 200 mg of KH_2PO_4 in 1 L of distilled water. (1.00 mL = 49.94 μg PH_3).

7.13 Molybdate solution. Prepare by dissolving 49.4 g of $(NH_4)_6 Mo_7O_{24}.4H_2O$ and 112 mL of concentrated H_2SO_4 in water to a total volume of 1 L.

7.14 Toluene-isobutanol solvent. Mix equal volumes of toluene and isobutyl alcohol.

7.15 Alcoholic sulfuric acid solution. Add 50 mL of concentrated H_2SO_4 to 950 mL of methyl alcohol.

7.16 Ferrous solution. Prepare by dissolving 7.9 g of $Fe(NH_4)_2(SO_4)_2$ and 1 mL of concentrated H_2SO_4 in water with a total volume of 100 mL.

7.17 Stannous chloride reagent. Prepare by dissolving 0.4 g of $SnCl_2$ in 50 mL of glycerol (heat to dissolve).

7.18 Acidic permanganate reagent. Prepare by dissolving 0.316 g of $KMnO_4$ and 6 mL of concentrated H_2SO_4 in a total volume of 1 L H_2O.

8. Procedure

8.1 Cleaning of Equipment

8.1.1 Before use, all glassware should be initially soaked in a mild detergent solution to remove any residual grease or chemicals.

8.1.2 After initial cleaning, the glassware should be thoroughly rinsed with warm tap water, 6 M nitric acid, tap water, distilled water in that order and then dried.

8.2 Calibration of Personal Sampling Pumps. Each personal sampling pump must be calibrated with a representative sampling tube in the line. The tube is described in Section 6.2. This will minimize errors associated with uncertainties in the sample volume collected.

8.3 Collection and Shipping of Samples

8.3.1 Immediately before sampling, break the two ends of the silica gel tubes to provide an opening of at least one half of the internal diameter of the tube (2-mm).

8.3.2 The treated silica gel tubes should be placed in a vertical direction during sampling to minimize channeling through the sorbent bed.

8.3.3 Air being sampled should not be passed through any hose or tubing before entering the tube.

8.3.4 A sample size of 16 liters is recommended. Sample at a known flow rate between 0.2 and 0.01 liter per minute. The flow rate should be known with an accuracy of at least ±5%.

8.3.5 The temperature and pressure of the atmosphere being sampled should be recorded. If the pressure reading is not available, record the elevation.

8.3.6 The treated silica gel tubes should be labeled appropriately and capped with supplied plastic caps. Under no circumstances should rubber caps be used.

8.3.7 With each batch or partial batch of 10 samples, submit one treated silica gel tube which had been handled in the same manner as the sample tubes (break, seal, transport) except that no air is sampled through this tube. This tube should be labeled as blank.

8.3.8 Capped treated silica gel tubes should be packed tightly and padded before they are shipped to minimize tube breakage during shipping.

8.4 Analysis of Samples

8.4.1 Preparation of Samples. In preparation for analysis each treated silica gel tube is scored with a file and broken open. The glass wool is removed with care and discarded making sure that no silica gel is lost in the process. The silica gel in the front section is transferred to a 50-mL beaker. The separating section of glass wool is removed and discarded. The backup section of silica gel is transferred to another container. These two sections are analyzed separately.

8.4.2 Extraction of the Samples. Prior to analysis, 10 mL of the acidic permanganate reagent is pipetted into each

beaker containing the silica gel. The extraction is carried out for 90 minutes at 65-70°C in a water bath. After extraction the acidic permanganate solution is drained into a 10-mL volumetric leaving the silica gel. The volumetric is made to volume with distilled water. The silica gel is washed twice with 3 mL portions of distilled water and the contents drained into another 10 mL volumetric containing 1 mL of ferrous solution. The flask is made up to volume with distilled water and mixed thoroughly.

8.4.3 Spectrophotometer Operation. Turn on the spectrophotometer and allow sufficient time for warmup. Follow the instrument manufacturer's recommendations for specific operating parameters. Adjust the wavelength to 625 nm and set the zero and 100% transmittance scale using 5-cm cells filled with distilled water. Check these settings prior to making any measurement to check on instrument drift.

8.4.4 Analytical Procedure

1. Add the contents of both 10-mL volumetric flasks (extract and washings) to a 125-mL separatory funnel.

2. Add 7.5 mL of molybdate reagent and 25 mL of toluene-isobutanol solvent. Shake for 60 seconds. Allow 60 seconds for the aqueous and nonaqueous layers to separate and discard the lower (aqueous layer).

3. Pipet 10 mL of the nonaqueous layer into a 25-mL volumetric containing 10 mL of the alcoholic sulfuric acid solution.

At this stage steps 4 through 6 must be performed within 1 minute.

4. Add 0.5 mL (25 drops) of stannous chloride reagent and make to volume using alcoholic sulfuric acid solution. Mix thoroughly.

5. Transfer the sample into 5-cm cells and stopper immediately.

6. Measure the absorbance or transmittance at 625 nm using water as a blank.

9. Calibration and Standardization

9.1 Add 10 mL of acidic permanganate solution, 1 mL of ferrous reagent to the separatory funnel.

9.2 Add 20 to 400 µL of the standard phosphorus solution to cover the range of 1 to 10 µg of PH_3. Add 8 to 9 mL of H_2O to make the total volume of the permanganate solution, ferrous solution, phosphorus solution and water to 20 mL. Prepare at least six calibration standards. A blank containing no phosphorus should also be analyzed.

9.3 Proceed as in Sections 8.4.4.2 to 8.4.4.6.

9.4 Prepare a calibration curve by plotting the absorbance of the standards after subtraction of the blank versus the amount of each standard in µg of PH_3 added on linear graph paper.

10. Calculations

10.1 Correction for the blank (obtained by extraction and analysis of the treated silica gel tube marked "blank") must be made for each sample

$$A_C = A_S - A_B$$

A similar procedure is followed for the backup tube.

10.2 The amount of phosphine present in the front tube, corrected for the blank, is found by reading the amount corresponding to A_C from the standard curve. The amount of phosphine found in the backup tube is similarly determined.

10.3 Add the amounts present in the front and backup tubes for the same sample to determine the total weight in the sample.

10.4 Determine the volume of air sampled at ambient conditions based on the appropriate information, such as flow rate (L/min) multiplied by sampling time (min). If a pump using a rotameter for flow rate control was used for sample collection, a pressure and temperature correction must be made for the indicated flow rate. The expression for this correction is:

$$\text{Corrected Volume} = f \times t \left(\sqrt{\frac{P_1}{P_2} \times \frac{T_2}{T_1}} \right)$$

where:

f = sampling flow rate

S332-8

t = sampling time

P_1 = pressure during calibration of sampling pump (mm Hg)

P_2 = pressure of air sampled (mm Hg)

T_1 = temperature during calibration of sampling pump (°K)

T_2 = temperature of air samples (°K)

10.5 The concentration of the analyte in the air sampled can be expressed in mg/cu m, which is numerically equal to µg/L, by

$$\text{mg/cu m} = \frac{\text{Total mg (Section 10.3)}}{\text{Air Volume Sampled (L)}}$$

Another method of expressing concentration is ppm (corrected to standard conditions of 25°C and 760 mm Hg).

$$\text{ppm} = \text{mg/cu m} \times \frac{24.45}{34.00} \times \frac{760}{P} \times \frac{(T + 273)}{298}$$

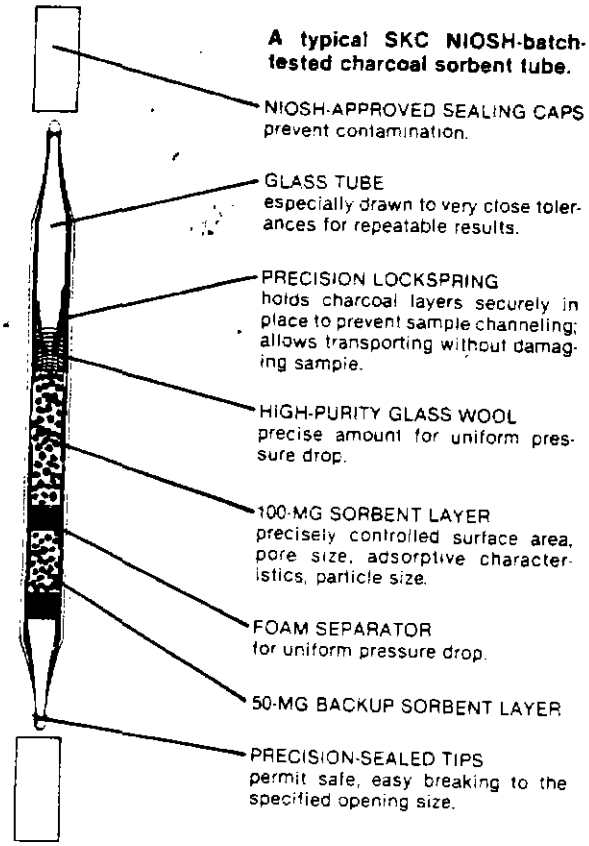
where:

P = pressure (mm Hg) of air sampled
T = temperature (°C) of air sampled
24.45 = molar volume (liter/mole) at 25°C and 760 mm Hg
34.00 = molecular weight of phosphine
760 = standard pressure (mm Hg)
298 = standard temperature (°K)

11. References

11.1 Documentation of NIOSH Validation Tests, National Institute for Occupational Safety and Health, Cincinnati, Ohio (DHEW-NIOSH-Publication No. 77-185), 1977. Available from Superintendent of Documents, Washington, D.C., Order No. 017-33-00231-2.

11.2 Backup Data Report for Phosphine, S332, prepared under NIOSH Contract No. 210-76-0123, 3/17/78.

Figure One

Schematic Diagram of an SKC Adsorbent Sampling Tube