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MONITORING STIBINE AND ARSINE IN LEAD-ACID BATTERY CHARGE GAS AND IN AMBIENT AIR: DEVELOPMENT AND TESTING OF A FIELD KIT

by

R. Varma, G. M. Cook, and N. P. Yao

MASTER

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Chemical Engineering Division

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ABSTRACT

In previous work, an industrial lead-acid battery (5 kW-h) was found to generate a significant amount of stibine and arsine during the overcharge period of a charge cycle and during equalization charging. Therefore, a stibine/arsine detection kit was developed for use in field collection and evaluation of toxic gas release. The equipment includes a metering pump to provide a constant gas flow rate, an absorption tube to collect the SbH_3 and AsH_3, and the necessary controls, filters, etc. A portable spectrophotometer is used to analyze for antimony. The absorbed solution is analyzed for arsenic at the laboratory. This kit was used to collect and measure the stibine and arsine generated from an industrial lead-acid battery and the ambient air around lead-acid modules during cycling.

I. INTRODUCTION

Improved lead-acid batteries are currently being developed by the Office for Electrochemical Project Management of Argonne National Laboratory (ANL) for near-term applications in electric-vehicle propulsion and utility load-leveling. The positive grids of lead-acid cells for the electric-vehicle batteries are expected to be fabricated from low-antimonial lead-alloys (∿1.5 to 4.5% Sb and 0 to 0.3% As). For load-leveling applications, a significant effort has been directed towards the development of 5-10 MW-h batteries consisting of 5-20 kW-h cells, which will also have positive grids of low-antimonial lead alloys (∿2.0 to 4.5% Sb and 0 to 0.3% As). Previous studies by Varma and Yao[1] as well as Holland[2] have shown that stibine and arsine, both of which are toxic gases, may be generated in significant amounts from industrial lead-acid cells containing positive and negative grids of antimonial lead during the overcharge and equalization steps of the charge cycle. It is anticipated that positive electrodes of most lead-acid batteries intended for electric-vehicle and load-leveling applications will continue to be fabricated largely from lead-antimonial grids. Therefore, stibine and arsine must be considered possible effluents of such batteries. The hydrides are also generated by electroreduction of ionic antimony and arsenic species at cathodes during electrowinning of copper, particularly when antimony and arsenic species are contained in the electrolyte as impurities. Indeed, the possibility that stibine and arsine may inadvertently be generated in areas occupied by workers in copper refineries has already been recognized by the copper-zinc electrolytic industries.

In 1976, the National Institute for Occupational Safety and Health (NIOSH) set limits on occupational exposure to these gases--0.05 ppmv or 0.2 mg/m^3 for arsine and 0.1 ppmv or 0.5 mg/m^3 for stibine. Such low exposure limits indicate the need for a device for monitoring stibine and arsine in electrolysis gases generated from charging of lead-acid cells or from copper electrowinning processes. In particular, occupational safety requires that ambient air be monitored for stibine and arsine generated in enclosed or poorly ventilated spaces in battery testing or manufacturing facilities as well as in copper refining plants. Such a kit must have equipment for collecting and, if possible, measuring stibine and arsine from either a battery charge gas or ambient air, must contain a minimum of accessories, and must be cost effective.

II. PAST ANALYTICAL METHODS FOR STIBINE AND ARSINE

The few methods that have been described for direct determination of stibine and arsine in air have employed impregnated paper, indicator tubes, and absorption in chemically reacting absorber solutions with subsequent colorometric determination (silver nitrate, mercuric chloride, and mercuric bromide react with arsine and stibine to produce colored stains). The following discussion outlines the major analytical procedures for determining stibine and arsine contents in ambient air or battery charge gas.

A. Solid-State Colorimetry

The basis of commercially available detector-tube kits or paper-tape based electronic monitors for arsine determination is the reaction of mercuric halides, HgX_2 (where X = Cl, Br), with arsine in the presence of moisture leading to the formation of mercury. This reaction occurs as follows: $3HgX_2 + 3H_2O + AsH_3 = 3Hg + H_3AsO_3 + 6HX$. The reaction product will be a yellow or brown stain, and may be compared visually or photometrically with standards to obtain a measure of arsine concentration in gas or air samples. It should be noted, however, that the presence of PH_3, H_2Se, or H_2S may interfere with the arsene determinations. For example, with the presence of PH_3, the reaction $3HgBr_2 + 2PH_3 \rightarrow Hg_3P_2 + 6HBr$ may take place. Stibine is far less sensitive than arsine in producing colored stains with mercuric halides; therefore, it would be expensive to develop solid-state colorimetry procedures for monitoring stibine.

B. Solution Colorimetry

Small amounts of stibine in air can be absorbed by an aqueous solution of methylfluorone (2, 6, 7-trihydroxy-9 methyl-3H-xanthene-3-one), silver sulfate, and starch; this solution produces colors ranging from lemon yellow through light amber and amber to brown. Short and Wheatley[3] developed a procedure for the determination of stibine in ambient air by the measurement of the absorbance of this colored solution by a spectrophotometer at a wavelength of 530 nm. In the 1960's, this method was used on board ships by the British Admiralty to determine stibine in the range of 0.01 to 0.1 ppmv in air; any arsine present does not interfere with this determination. The disadvantages of the procedure are chiefly the chemical instability as well as the carcinogenic properties of methylfluorone.

Arsine is readily absorbed in a pyridine solution of silver diethylcarbamate; the arsine complex in pyridine solution absorbs at 525 nm, and this provides the basis for the determination of arsine[4] in a gas mixture. However, the corresponding pyridine solution containing antimony absorbs at 510 nm, and may interfere with the determination of the AsH_3 content. Among other methods available, the "molybdenum blue" technique for determining arsenic[5] and the "Rhodamine B"[6] technique for determining antimony are also considered acceptable.

A solution of NaI_3 (NaI plus I_2 in $HClO_4$) was used successfully by Gann[7] to collect stibine and arsine from the charge gas of lead-acid batteries. Antimony was determined by the spectrophotometric measurement of the SbI_4^- species at 425 nm. The arsenic species in the solution was converted into a colloidal dispersion of elemental arsenic and determined photometrically at 420 nm. Gann's method is a reliable procedure for the collection and determination of stibine and arsine in any stream of battery charge gas, or even ambient air. However, extreme caution must be exercised because perchlorates in the dry state may explode.

A 3$\underline{N}$ H_2SO_4 solution containing 8% KI and 1% I_2 by weight has been used to absorb stibine and arsine from a gas stream, trivalent SbI_4^- and H_3AsO_3 species being formed in the solution. In a previous report,[1] the effectiveness of this absorber solution was demonstrated for the collection of stibine and arsine from a lead-acid cell during charging modes under a utility load-leveling duty cycle; a spectrophotometric method was used to measure the SbI_4^- species in solution by measuring its absorbance at 330 or 425 nm ($\varepsilon_{330} = 3.23 \times 10^4$; $\varepsilon_{425} = 4.62 \times 10^3$). By appropriate choice of wavelength, the antimony concentration may be determined over a wide range of concentrations. Arsenic can be determined by reconverting the arsenic species into AsH_3 and measuring the atomic absorption of the atomized arsenic species in an argon-hydrogen air-entrained flame by using atomic absorption spectroscopy (AAS).

C. Gas-Phase Direct Determination

Stibine and arsine in a gas mixture can be determined by AAS. For the gas-phase determination, the water vapor and the hydrides contained in battery charge gas or ambient air are collected in a liquid N_2 ($\sim$-196°C) trap, and then the stibine and arsine are volatilized selectively (the boiling point of liquid AsH_3 is $\sim$-60°C and that of liquid SbH_3 is $\sim$-17°C) into either an argon-hydrogen air-entrained flame or a flameless furnace for atomization. Flameless atomization[8] has a tenfold higher detection sensitivity than that obtained with a flame. The smallest concentrations detected by the flameless-atomization methods are 0.025 ppb for SbH_3 and 0.005 ppb for AsH_3.

III. COMMERCIALLY AVAILABLE FIELD KITS

The following two devices are commercially available for determination of arsine: (1) an inexpensive field kit consisting of glass detector-tubes packed with active material which stains when contacted with arsine, and (2) an electronic monitor which compares the color of an arsine standard with the color developed by a chemical tape on exposure to a gas mixture containing arsine.

The Matheson Model AD-1 Arsine Monitor* is designed to determine arsine at levels of parts per million in ambient air; a chemical tape impregnated with $HgBr_2$ used with this monitor develops color in contact with AsH_3 ($3HgBr_2 + 3H_2O + AsH_3 \rightarrow 3Hg + H_3AsO_3 + 6HBr$). From the display of the cumulative amount of arsine on the monitor, one can calibrate the arsine released (in ppmv) during a short period (*e.g.*, 10.0 L of air-sampled at a rate of 1 L/min). The range of AsH_3 that can be detected is 0.005 to 0.2 ppmv in air. The procedure seems suitable for monitoring arsine in ambient air; however, certain other contaminants of ambient air originating from the same source as the arsine--such as PH_3, H_2Se and H_2S--may interfere with the arsine determination because the contaminant will react with $HgBr_2$ to produce colored products. Costs of arsine monitoring portable units, or units appropriate for in-plant installation, range from $4000 to $6000. For equal amounts of AsH_3 and SbH_3, the meter's response[9] to SbH_3 is about 20% weaker than its response to arsine. Because greater sensitivity to stibine was desired, this monitor was not adopted.

The Kitagawa arsine detector kit* provides for on-site measurement of arsine concentration in the range of 5 to 160 ppm in air (minimum detectable level, 1 ppm). A precision hand pump is used to sample aliquots of air volumes through detector tubes (2- to 3-mm ID, and 10 to 15 cm long) packed with mercuric chloride dispersed in silica gel, and then the stained portion in the tube is measured and evaluated with calibration charts. An arsine kit from the National Draeger Company provides for measurement of arsine at levels of 0.05 to 60 ppmv in air (minimum detection capability, 0.05 ppmv). The arsine detectors based on mercuric chloride, which are similar to those based on mercuric bromide, also have a significantly lower sensitivity for stibine than for arsine. Even though the concentration of SbH_3 is generally many-fold greater than that of AsH_3 in the charge gas from a typical lead-acid battery, the lower sensitivity of SbH_3 towards mercury halide-based detectors places sever limitations on the detector to be chosen for monitoring stibine. Thus, the commercially available arsine monitors were again found to be inappropriate for stibine monitoring, and no attempt was made to modify or upgrade these instruments for use in the Stibine/Arsine Monitoring Field Kit (SAMFK) developed by ANL.

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*Distributed by Matheson Gas Products Co., East Rutherford, NJ.

IV. STIBINE/ARSINE MONITORING FIELD KIT

The SAMFK was designed to collect SbH_3 and AsH_3 in an absorber solution from battery charge gas or ambient air, and to permit on-site spectrophotometric determination of antimony species in solution; this absorber solution can be also analyzed for arsenic at a laboratory site. The SAMFK can be used for monitoring stibine/arsine directly in (1) the charge gas from load-leveling batteries at a utility facility or from electric-vehicle batteries in a charging facility as well as (2) work-space air in occupied areas in battery test facilities or copper-zinc refineries.

A. Kit Description

Appendix A presents a description of the components in SAMFK, which is shown photographically in Fig. 1 and schematically in Fig. 2. The kit can be operated to collect stibine and arsine in three modes. In modes I and II, charge gas is transported out of a battery and into SAMFK with a carrier gas of nitrogen and ambient air, respectively. In mode III operation, SbH_3 in ambient air surrounding a battery is collected with SAMFK.

In mode I operation, the gas lines shown in Fig. 2 are arranged in the order 1→2→3. The source of the N_2 carrier gas is a portable cylinder (see Fig. 3a) containing 0.9 m^3 (32 ft^3) of N_2 at 14 MPa (2000 psig); this cylinder has a double-stage regulator [0-21 MPa (0-3000 psig) and 0-0.7 MPa (0-100 psig)], a flowmeter, and a gas outlet with an OD of $\sim$1 cm (3/8 in.). The battery cap connector shown in Fig. 3b is screwed into the top of a lead-acid battery. This cap has two outlets: one for the introduction of N_2 above the electrolyte (tube G002 and P002) and the other for the removal of the carrier gas and charge gas from the battery and into the kit (tube G001). Two caps, 6.2 cm (5/2 in.) and 3.7 cm (3/2 in.), are provided with the kit for use with lead-acid batteries of different sizes. The device used to collect the charge gas in the SAMFK consists of two gas wash bottles (125 mL), shown in Fig. 4. Bottle B001 is fitted with a frit disc element at the end of the gas inlet tube for gas dispersion. During collection, the charge gas is bubbled through 50 to 100 mL of absorber solution (described later) at the rate of 0 to 1 L/min. Bottle B002 serves as a blank and prevents the corrosive absorber solution from accidentally entering the battery.

In mode II operation, the gas lines shown in Fig. 2 are connected in the order 1→2→4. For this mode, the battery cap connector is screwed into a porthole of a lead-acid battery and the outlet tube of the cap connected to the gas inlet port of the kit. The inlet port of the cap connector is left open to work space air. As shown in Fig. 5, a Bendix pump (Model BDX-56) is used to force the charge gas out of the battery and into bottle B001, which contains 50 to 100 mL of absorber solution. Bottle B002 prevents absorber solution from accidentlly entering the pump. According to the manufacturer, the rechargeable nickel-cadmium battery in the pump provides eight hours of continuous sampling at about 2 L/min, with resistance to flow equivalent to a nominal 20-in. head of water; the flow rates are easily adjustable to lower values. In mode III operation, the gas lines are connected in the same order as used for mode II (1→2→4). For this mode, the Bendix pump sucks the ambient air surrounding a charging battery directly into the kit inlet port; the gas collection bottles are arranged as shown in Fig. 5. Appendix C provides some additional information on the operation of the kit in the above three modes.

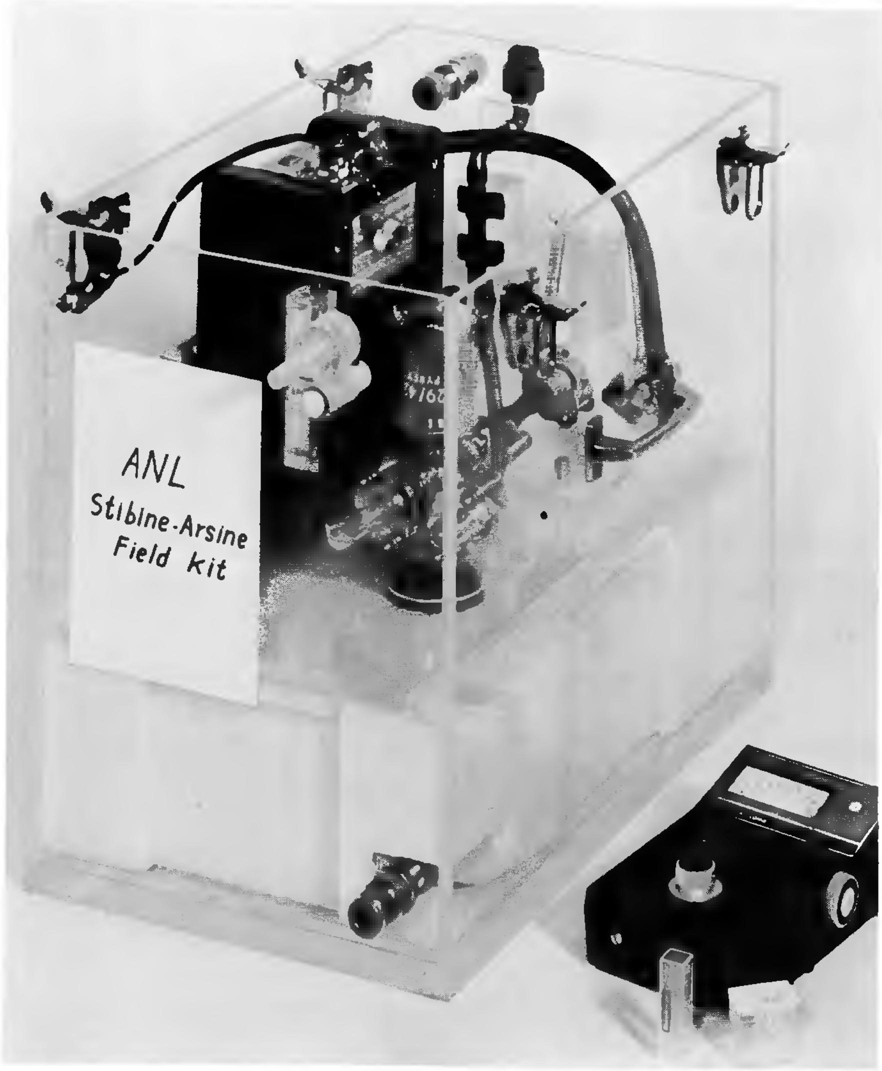


Fig. 1. Photograph of SAMFK. ANL Neg. No. 308-79-52.

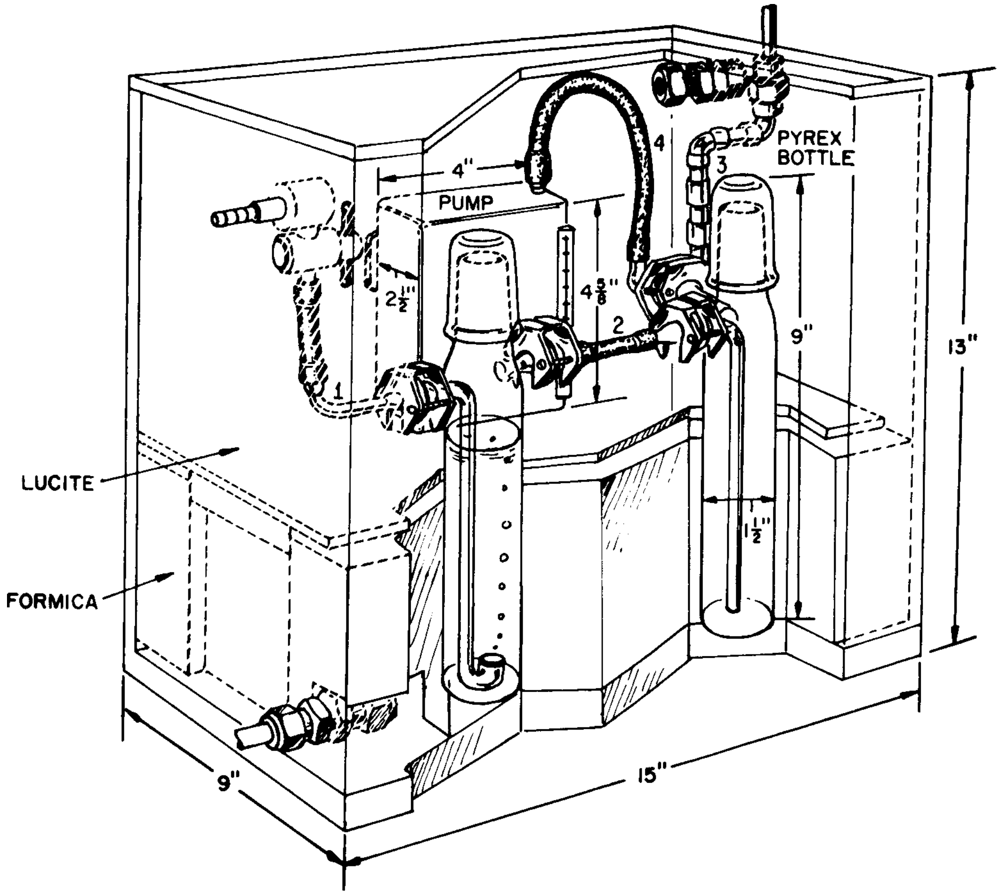
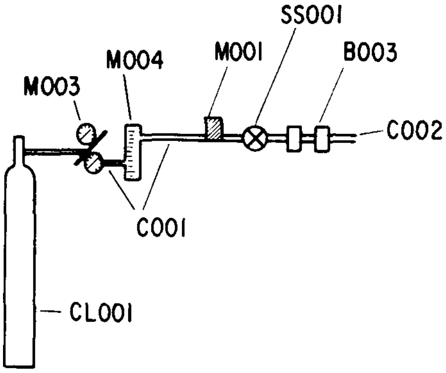


Fig. 2. Schematic Drawing of SAMFK

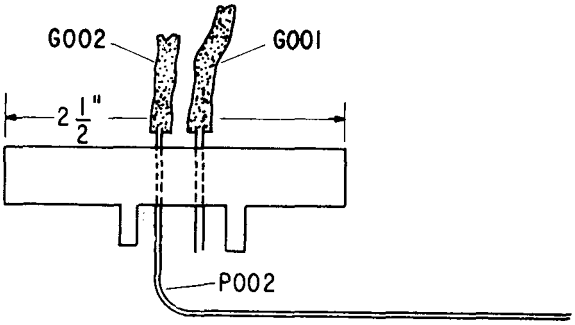
The kit has been designed for use with any suitable absorber solution. However, solid absorbers such as mercuric salts were not considered suitable because of their poor sensitivity to stibine. Of the aqueous absorber solutions, a solution[1] of 80 g of KI and 10 g of I_2 dissolved in one liter of $3\underline{N}$ H_2SO_4 was selected because it is thermally stable, has a long shelf life, can be easily prepared, and can be easily analyzed for antimony on a spectrophotometer. The absorber solution described by Short and Wheatley[3]--methylfuorone, silver sulfate, tartaric acid, and starch dissolved in dilute H_2SO_4--is also effective for stibine and arsine determination, but it is chemically unstable (shelf-life, ∿1 day) and methylfuorone is carcinogenic.

B. Stibine Determination

In the field, the absorbance of the stibine in the absorber solution can be determined with a Bausch and Lomb Spectronic mini-20 Spectrophotometer (see right side of Fig. 1). This spectrophotometer is compact, light in weight (2.2 kg), and rugged enough to be used in adverse field conditions. It features a built in rechargeable power supply (nickel-cadmium 4 AA-size cells with a nominal DC voltage of 5 V), a wavelength range of 400-800 nm, direct reading in either absorbance or transmittance, a field carrying case,



(a) NITROGEN GAS SOURCE



(b) LEAD-ACID BATTERY GAS SAMPLER CAP

Fig. 3.

The Nitrogen Gas Source and Battery Gas Sampler Cap of the SAMFK. (The components corresponding to the numbers in this figure are listed in Appendix B.)

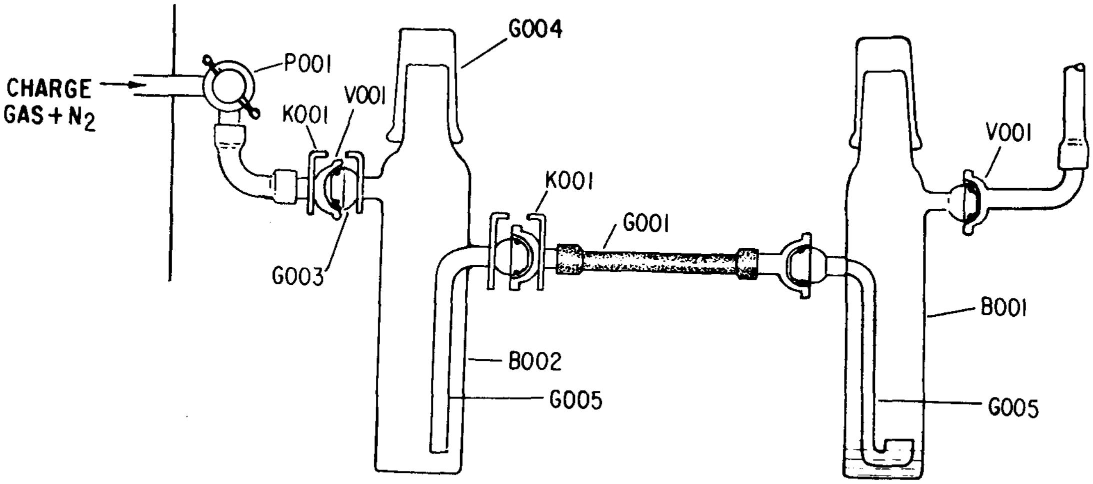


Fig. 4. Mode I Operation. (The components corresponding to the numbers in this figure are given in Appendix B.)

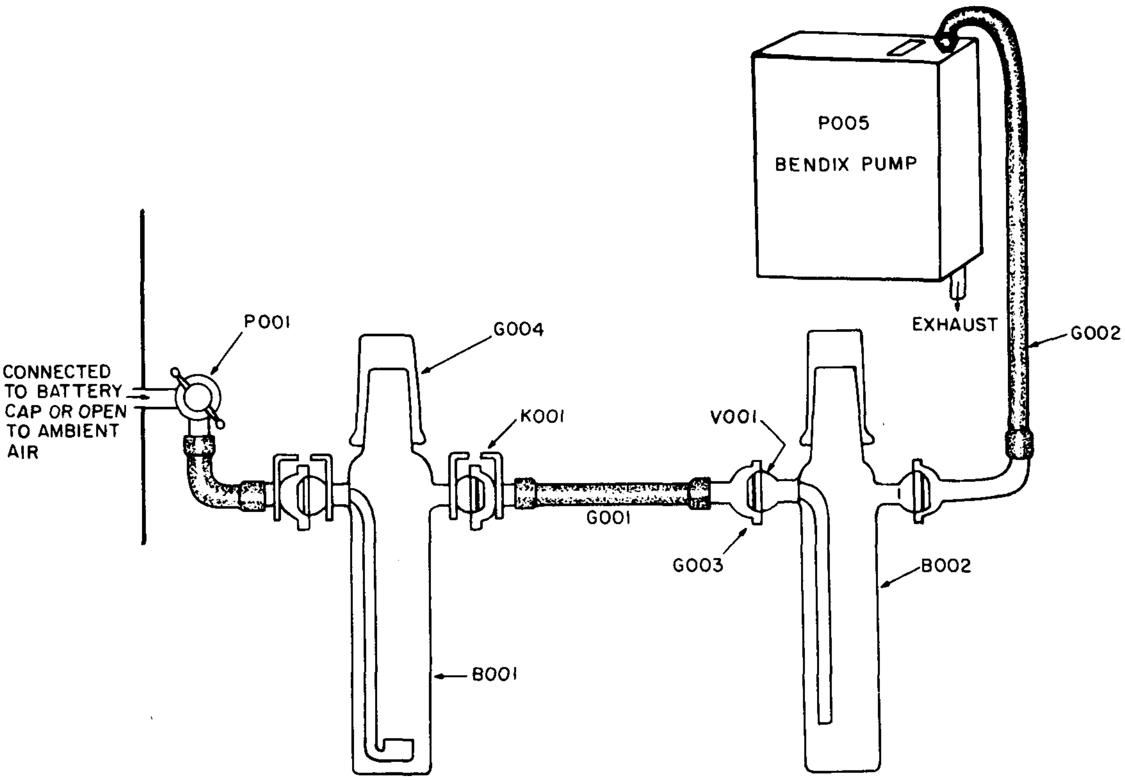


Fig. 5. Mode II and III Operation. (The components corresponding to the numbers in this figure are shown in Appendix B.)

and quartz absorption cells with 2.5-cm and 1-cm optical path lengths. Experience has shown that, at an absorbance wavelength of 425 nm (the point at which SbI_4^- species absorb), the 1-cm cell is optimal for determining stibine content in the range of 8 to 25 μg Sb per milliliter of absorber solution, and the 2.5-cm cell is optimal for determining 0.8 to 8 μg Sb per milliliter. General directions for proper operation of this instrument are provided by the manufacturer.

The mini-20 spectrophotometer has a device for checking the reproducibility of the absorbance measurements. However, the following procedure was used to further ensure reproducibility of measurements. The spectrophotometer was set for measurement at a wavelength of 425 nm, allowed to warm for 10 min, and blanked to 0% transmittance. Before each reading, after a stabilization period of 5 min, the instrument was zeroed to 100% transmittance or 0% absorbance with H_2O in either the 1-cm or 2.5-cm cell.

Although pre-calibrated curves can be used to calculate the stibine content from the spectrophotometrically determined absorbances, more accurate stibine determinations were obtained in the field by generating new calibration curves. The following steps should be followed to determine the stibine content of an absorber solution from a field-derived calibration curve. A primary standard solution, for example one containing ∿200 μg Sb/mL, is prepared by dissolving an appropriate amount of pure potassium antimony tartrate in water acidified with H_2SO_4. Aliquots of the following are then prepared: 2mL of standard solution with no absorber solution, 6 mL of absorber solution through which stibine and arsine have been bubbled, and 10 mL of absorber solution without any antimony. Each of these solutions is placed in separate 25 mL volumetric flasks and labeled Sample (a), (b), and (c), respectively. To provide these aliquots with the same volume of absorber solution, 10 mL and 4 mL of absorber solution are added to Samples (a) and (b), respectively. Next, 1 mL of 50% hypophosphorous acid (H_3PO_3) is added to each of the samples, which are then diluted up to 25 mL with $3\underline{N}$ H_2SO_4. Finally, the solutions are swirled and allowed to stand for twenty minutes, and the absorbances of the resulting pale-yellow or colorless solutions are measured spectrophotometrically. The differences between the absorbances of Samples (a) and (b) and that of Sample (c) are determined and recorded. By incrementally diluting Sample (a) and measuring its net absorbance, one can easily derive a calibration curve. From this curve and the net absorbance of Sample (b), the stibine content of Sample (b) can be computed.

Typically, calibration curves are plotted for two ranges of antimony content: 0.8 to 8.0 µg Sb and 8.0 to 24 µg Sb per milliliter of absorber solution. Figure 6 shows a calibration curve for the range of 8.9 to 24 µg Sb/mL. In this figure, curve I and II were derived from absorbance measurements

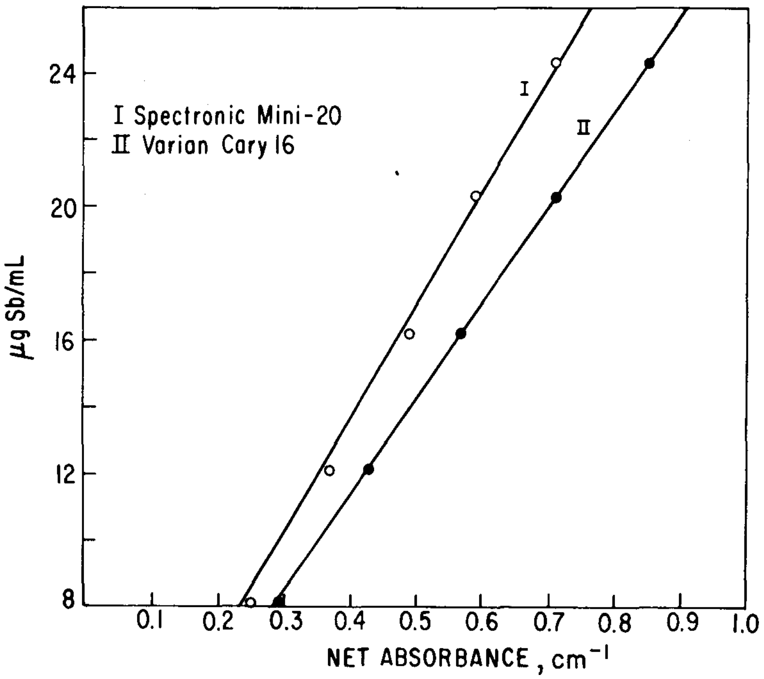


Fig. 6. Typical Calibration Curves for Determining Antimony Content in Absorber Solutions Containing 8 to 24 µg Sb/mL. (Measured on two different spectrophotometers at 425 nm in 1-cm cell.)

by using the Spectronic mini-20 and the Varian Model Cary-16* spectrophotometers, respectively, at a wavelength of 425 nm (1-cm cell). The results of the absorbance measurements made with both of these spectrophotometers are shown in Table 1. A similar type of calibration curve for determining antimony content in the range of 0.8 to 8.0 µg Sb/mL is shown in Fig. 7.

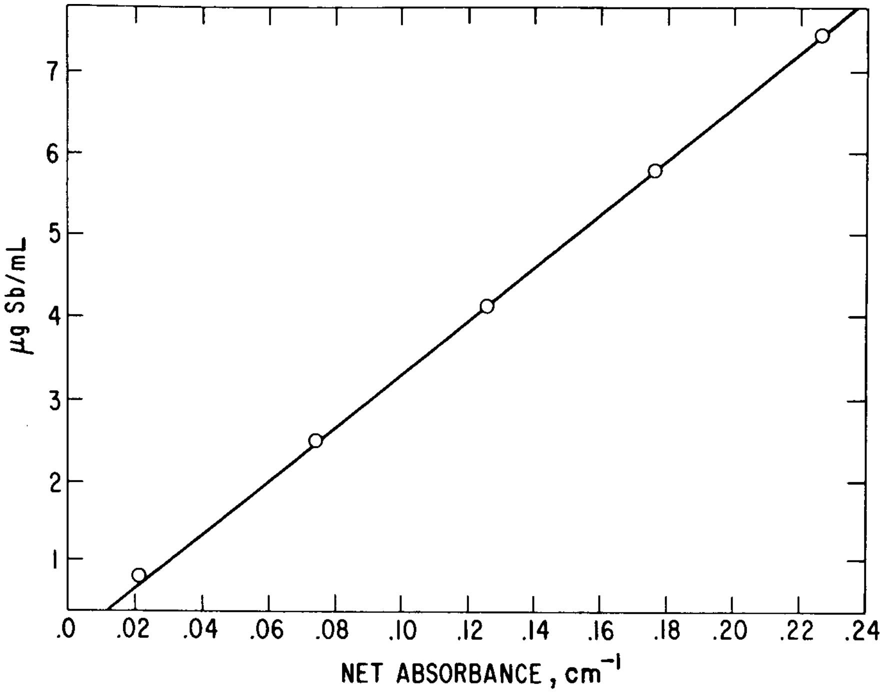


Fig. 7. Typical Calibration Curve for Determining Antimony Content in Absorber Solutions Containing 0.8 to 8.0 µg Sb/mL. (Measured on the mini-20 spectrophotometer at 425 nm in 1-cm cell.)

C. Arsenic Determination

The arsenic content of the absorber solution must be determined in the laboratory. The method of atomic absorption spectroscopy† has been found to be effective for this measurement.

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*An instrument used in the laboratory by the Analytical Chemistry Laboratory at ANL.

†Described in the Appendix of Ref. 1.

Table 1. Absorbances of Standard Solutions Used to Generate a Calibration Curve

Standard Solution, μg Sb/mL	Absorbance, cm^{-1}	
	mini-20	Cary-16[a]
8.1	0.206	0.2948
12.2	0.380	0.4337
16.2	0.500	0.5722
20.3	0.60	0.7161
24.3	0.72	0.8547
Blank	0.01	0.0042

[a] Determined by the Analytical Chemistry Laboratory at ANL.

V. TESTING OF SAMFK

A. Reliability of the Mini-20 Spectrophotometer

Two experiments were performed to assess the reliability of the mini-20 spectrophotometer for field measurements of stibine absorbance. In the first experiment, the absorbances of three absorber solutions containing known amounts of antimony were determined with the mini-20. The calibration curve shown in Fig. 8 was then used to redetermine the antimony content of these three solutions. The known values and the calculated ones are shown in Table 2. As shown in this table, there was fairly good agreement between the known values and those determined with the mini-20 spectrophotometer. In another experiment, the antimony contents of ten antimony-containing absorber solutions were determined by using the mini-20 and the Cary-16 spectrophotometers; these data are presented in Table 3. These data further confirm that the mini-20 spectrophotometer is a reliable instrument for field determinations of antimony absorbance in absorber solution.

B. Cell Tests of Kit

For testing purposes, a 4.2 kW-h industrial lead-acid cell (type C-160-31 manufactured by C&D Batteries) was initially selected. This battery has 15 positive grid plates containing 4.6% Sb and 0.1% As, and 16 negative grid plates containing 4.3% Sb and 0.1% As. A Robicon cell cycler (1000 A) under computer control was utilized for charging and discharging the cell in a continuous manner. To simulate the practice in load-leveling applications, the battery was given four consecutive daily charge and discharge cycles and one weekly equalization charge. The cell was discharged at a constant current of 360 A for 5 h. After each such discharge, the cell was charged at a constant current of 290 A for 5 to 6 h to a maximum voltage of 2.45 V. This was followed by a current-limited, constant-voltage (tapering) charge at 2.45 V for 3 h of overcharge. The weekly equalization was conducted for 4 h at a constant voltage of 2.55 V.

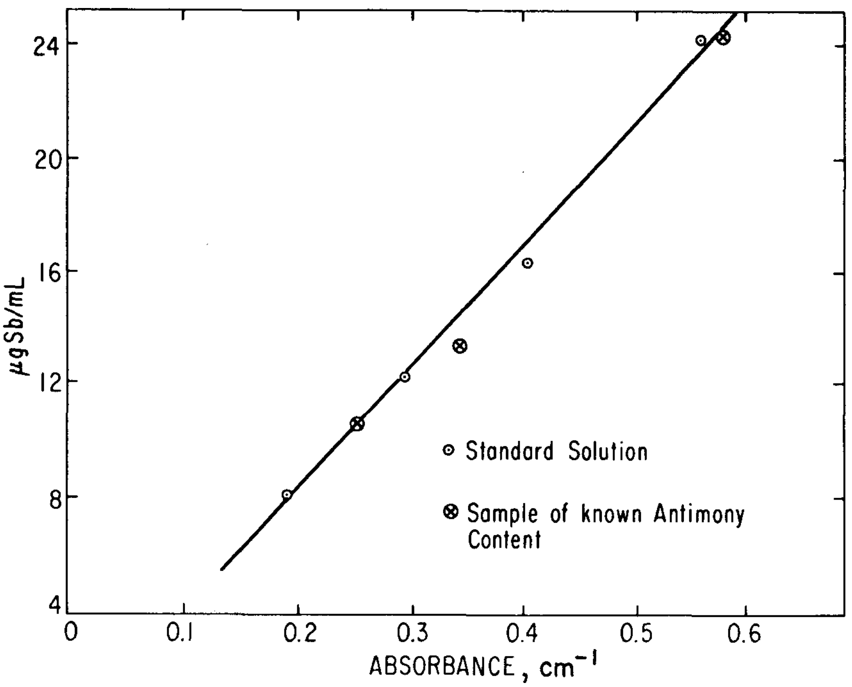

Fig. 8. Calibration Curve for Antimony Content in Standard Solutions. (Determined on the mini-20 Spectrophotometer at 425 nm in 1-cm cell.)

Table 2. Antimony Content of Three Known Solutions

Sample Number	Antimony Content, μg/mL Known Value	Determined using mini-20 Spectrophotometer	% Difference
1	10.6	10.0	6.4
2	13.5	13.6	∿1
3	22.8	24.1	5.8

The stibine and arsine generated by the battery during the charge cycles were bubbled through 100.0 mL aliquot of absorber solution in B001 (mode I operation) of SAMFK; the exit gases were discharged into the exhaust duct of a fumehood. The antimony and arsenic content of the absorber solution was determined by personnel from the ANL Analytical Chemistry Laboratory; a Varian Cary-16 spectrophotometer with a 1-cm cell was used to determine antimony content. The total antimony collected with the kit for a given charge cycle is reported in Table 4; a calibration curve similar to that shown in Fig. 6 (curve II) was used for such determinations. Small variations in cell output from cycle to cycle (∿2.3 μg/mL) are attributed to variations in cell characteristics and/or experimental conditions.

Table 3. Antimony Content of Ten Absorber Solutions Containing Stibine

Sample No.	Total Antimony, μg/mL		Ratio of Two Determinations[b]
	mini-20	Cary-16[a]	
1	11.2	11.2	1.00
2	4.4	7.6	0.59
3	11.7	11.7	1.00
4	11.4	11.4	1.00
5	8.1	8.3	0.98
6	18.6	18.6	1.00
7	10.7	10.8	0.99
8	14.8	14.8	1.00
9	9.2	9.4	0.98
10	11.6	11.5	0.99

[a] Absorbance measurements made by Analytical Chemistry Laboratory at ANL.

[b] Ratio of mini-20 to Cary-16 measurements.

Table 4. Stibine Collected with SAMFK During Charge Cycles of a 4.2 kW-h Lead-Acid Cell

Charge Cycle	Stibine Generation	
	μg Sb/mL[a]	mg SbH_3/100 mL
1	11.2	28.8
2	11.7	29.9
3	11.4	29.3
4	10.9	27.7
5	9.4	24.2
6	11.6	29.9

[a] Standard deviation, ±2.3 μg/100 mL.

The National Battery Test Laboratory (NBTL) was constructed at ANL to test electric-vehicle batteries as they are developed. The laboratory has three test stands where the cells and batteries under test are mounted. Hoods over the test stands are connected to a ventilation system (total capacity, 170 m^3/min) to exhaust any gases that may be generated during charging. During a typical test at NBTL, the cells or battery modules under test are charged and discharged by computer-controlled cyclers. At the suggestion of personnel from the NBTL, stibine/arsine accummulation was monitored in the work-space air near two lead-acid battery modules (three cells* each)

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[*] Type EV-ISOA-FP-1, manufactured by ESB Technology Co.

and in the charge gas of a cell from one of these modules. Both of these modules had a capacity of 180 A-h at the 3-h rate. The cell positive grids were fabricated from lead-antimony alloy (6% Sb and 0.3% As) and the negative plates from lead-calcium grids. The work-space air and charge gas were bubbled through a 50-mL aliquot of absorber solution in B001 of the SAMFK during the charge of the test cells. The SAMFK was operated in mode II to collect the charge gas and mode III to collect the ambient air. Typically, the modules in the discharged (80% depth discharge) condition were charged at a constant current of 40 A for 3-4 h to an average voltage of 2.55 V per cell. This was followed by a current-limited, constant-voltage (tapering) charge at 2.55 V per cell for 4 h. During equalization, the cells in the charged condition were given a constant current of 5 A for 16 h, the average cell voltage during this period rising to 2.8 V. The details of the battery module charge regimen and sample collection are reported in Table 5. The absorber solutions were analyzed in each case for antimony and arsenic by the ANL Analytical Chemistry Laboratory; in addition, antimony was anlayzed on the Bausch and Lomb Spectronic mini-20 Spectrophotometer provided with the kit. The experimental results are given in Table 6, and indicate that the concentrations of SbH_3 and AsH_3 generated from these two battery modules are, in most cases, below the threshold limits of human tolerance prescribed by the Occupational, Safety and Health Administration (OSHA).

VI. CONCLUSION

In previous work,[1] an industrial lead-acid cell (5 kW-h) was found to generate a significant amount of stibine and arsine during the overcharge period of a charge cycle and during equalization charging. Therefore, the SAMFK was developed for collection and evaluation of toxic gas release in the field. The minimum detection level[1] was estimated to be 0.25 μg SbH_3 and 0.01 μg AsH_3 per milliliter of absorber solution with use of standard analytical techniques in the laboratory; the precision is within 5%. When the spectrophotometer provided with SAMFK is used, the minimum detection level is 0.8 μg SbH_3 per milliliter of absorber solution (10% precision).

In mode I and II operation, SAMFK has been found to be effective in collecting SbH_3 and AsH_3 from the gas generated during charging lead-acid batteries as well as from the ambient air near by charging lead-acid modules. In addition, the spectrophotometer provided with the kit has been found to accurately determine the stibine content of this charge gas and ambient air.

For determinations of SbH_3/AsH_3 in ambient air (mode III), the SAMFK can sample air at the rate of 1.0 L/min for an 8-h period. If one assumes that SbH_3 and AsH_3 are present in this ambient air in concentrations of 0.1 and 0.05 ppmv, respectively (the minimum tolerance level for an 8-h exposure permitted by OSHA), then the kit would collect 446 μg of SbH_3 and 139 μg of AsH_3 during an 8-h period. If a 50 mL absorber solution is used in this case, then 8.9 μg Sb and 2.8 μg As per milliliter of absorber

Table 5. Specifications for the Two Modules on Charge at NBTL

	Cell/Battery Module Specifications				
Experiment	Cells on Charge	Individual Cells Under Test	Sample Environment	Time Interval of Sample Collection	Sample Designation
12/5/78	Modules A and B	--	Air	3 h during 40 A charge.	C1
12/5/78	Modules A and B	--	Air	4 h taper charge.	C2
12/6/78	--	Cell #3 of Module A	Charge Gas	4 h after a cell voltage of 2.65 V had been reached.	EC1
12/6/78	Modules A and B	--	Air	4 h after a cell voltage of 2.65 V had been reached.	EC2
12/27/78	Module A		Air	First 4 h of equalization.	EC3
12/27/78	--	Cell #3 of Module A	Charge Gas	Second 4 h of equalization.	EC4
12/27/78	Module A	--	Air	First 4 h of equalization.	EC5

Table 6. Stibine/Arsine in Ambient Air and Battery-Charge Gas from Two Modules on Test at NBTL

	Ambient Air Sample Collection			Toxic Gas in Absorber Solution, μg/mL		Concentration in Ambient Air ppmv	
Sample Designation	Time Period, h	Volume of Air, L	Charge Gas Sample Time from Cell #3, h	Sb[a]	As	SbH_3[a]	AsH_3
C1	3	80	--	<0.1	<0.01	Negligible	
C2	4	60	--	0.24	<0.01	0.04	0
EC1	--	--	4	32.1	1.1	--	--
EC2	4	50	--	0.26	<0.01	0.05	0
EC3	4	130	--	<0.1	<0.01	Negligible	
EC4	--	--	4	28.4	0.7	--	--
EC5	4	130	--	<0.1	<0.01	Negligible	

[a] Average values of determinations using the mini-20 and Cary-16 spectrophotometers.

solution would be collected. This concentration of stibine is significantly above the minimum detection limit of the spectrophotometer in the SAMFK (∿1 μg Sb/mL). Therefore, with the SAMFK, in an 8-h period, ambient air can be monitored for stibine and arsine in concentrations indicated to be dangerous by OSHA. Indeed, the observation has also been found to be true for 4-h collection periods with the SAMFK.

The SAMFK can be used in a safe and easy manner to monitor SbH_3/AsH_3 in battery off-gas in the field. It can also be used for air sampling in any work space of a plant, a battery manufacturing facility, a battery test facility, or a battery charging facility. The cost of the entire package when mass produced should not exceed $2000. The ease and convenience in handling make it possible for an unskilled operator to use this kit for determinations of SbH_3 and AsH_3 in battery charge gas or in ambient air.

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APPENDIX A

SPECIFICATIONS OF THE SAMFK

1. Kit-Box

Dimensions: 37.5 x 32.5 x 17.5 cm.

Material: typically plastic, acrylic, melamine, polyethylene.

Accomodates two gas bottles, a pump, and accessories.

2. Components

Gas bubbler of 125 mL capacity, with fritted inlet tube disc.

An empty blank bottle of 125 mL capacity.

Bendix Air Sampler, Pump Model BDX-56 for flow rates of 0.5-2.5 L/min.

3. Accessories

Universal Gas Sampler consisting of the following parts:

- Gas cylinder containing 32 ft^3 (0.9 m^3) of N_2 at 2000 psig (14 MPa) available from Matheson Company.
- Nitrogen gas regulator (scales 0-2000 and 0-100 psig).
- Flowmeter reading 0-40 L/min (or lower range) from Matheson or any other manufacturer.

Battery Cap Connectors consisting of an air or N_2 inlet port, and a battery gas outlet.

Gas line connectors, valves, tubings, etc.

Bausch and Lomb Spectronic mini-20 Spectrophotometer for on-site determination of antimony.

4. Reagents

Absorber Solution: 80 g KI and 10 g I_2 diluted to 1000 mL with $3\underline{N}$ H_2SO_4.

Analytic Reagent: 50% H_3PO_3 aqueous solution.

5. Chemical Wares

Pippets, 25 mL volumetric flasks, etc.

APPENDIX B

PART LIST FOR THE COMPONENTS OF SAMFK

Part Number		Figure No.
B001	125 mL gas bubbler with inlet tube which ends in fritted disc	4, 5
B002	125 mL empty bottle	4, 5
B003	Brass-tube-union copper tubing (3/8 in. OD)	3a
B004	Bausch and Lomb Spectronic mini-20 Spectrophotometer	1
C001	Copper hard tube (3/8 in. OD)	3a
CL001	Nitrogen gas (at 2000 psig) cylinder from Matheson Company with capacity of 32 ft^3 (1000 L)	3a
G001	Fluorinated rubber tubing (5/16-in. ID, 3/8-in. OD) obtained from Arthur Thomas Company	4, 5
G002	Fluorinated rubber tubing (1/4-in. ID, 5/16-in. OD) obtained from Arthur Thomas Comany	5
G003	Pyrex ball (18/7 in.) and socket joints	4, 5
G004	Ground glass joint with Teflon Sleeve	4, 5
G005	Pyrex Tubing (12-mm OD)	4, 5
K001	Miniature Clamps	5
M001	3-50 psig adjustable relief valve	3a
M003	0-2000 psig N_2 gas regulator (Model 80580)	3a
M004	0-40 L/min flowmeter	3a
P001	Polyethelene two-way valve	4, 5
P002	Polyethylene tubing (1/8-in. OD)	2
P005	Bendix Model BDX-56 pump 0.5-2/min pumping rates	5
SS001	Stainless steel shutoff valve	3a
V001	Viton O-rings	4, 5

APPENDIX C

NOTES ON OPERATION OF SAMFK

Checklist

1. Charge the batteries of the Bendix Model BDX-56 pump and Spectronic mini-20.
2. Set up the kit for the particular mode of operation chosen (see below).
3. Check for leaks in gas lines and repair the leaks.
4. Take strict precautions to ensure discharge or venting of noxious gases and potentially explosive ($H_2 + O_2$) gas mixture in a safe manner.
5. After use, the kit bottles should be washed with pure absorber solution and water in all cases.
6. After continuous use of 1 week, the gas line tubings should be washed or replaced in all cases.

Mode I Operation (Specifically recommended in case of measurement of charge gas from large capacity lead-acid cells with extremely high gassing rates.)

1. Bubble N_2 carrier gas through $3\underline{N}$ H_2SO_4 prior to its introduction into the cell in order to prevent evaporation of water from the cell.
2. Connect the vent to an exhaust capable of air intake at high rate. (This will ensure safety for the working personnel).
3. Adjust relief valve (M001) of the N_2 in gas line to 10 psig.
4. Ensure that gas bubling through the absorber solution is visible and that the nitrogen flowrate is 0-1500 mL/min.
5. Continue collection of toxic hydrides from the battery for the duration of charge or any portion thereof.

Mode II Operation (Uses air as the carrier of the battery charge gas at flow rates of 0.5 to 1.5 L/min.)

1. Bubble air stream through a $3\underline{N}$ H_2SO_4 in a wash bottle prior to introduction at the gas inlet port of the battery cap connector.
2. Connect the pump vent (see Instruction Manual of Bendix Model BDX-56 pump) to an appropriate exhaust or discharge the gas in a safe manner.

Mode III Operation (Useful for sampling ambient air.)

1. Sample air for a long duration, preferably 4 to 8 hours, to get significant accumulation.

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