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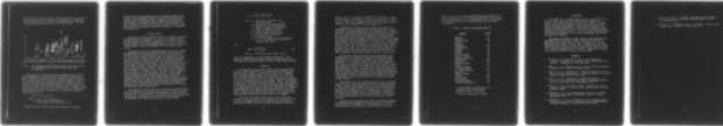
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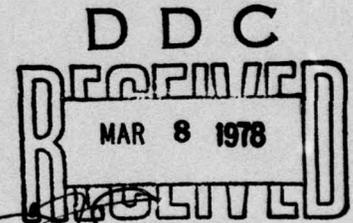
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Report SAM-TR-77-28

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ANALYSIS OF TRACE VOLATILES BY GAS CHROMATOGRAPH/MASS SPECTROMETER DATA SYSTEM

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December 1977

Interim Report for Period January 1976-October 1977

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USAF SCHOOL OF AEROSPACE MEDICINE
Aerospace Medical Division (AFSC)
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This report has been reviewed by the Information Office (OI) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

This technical report has been reviewed and is approved for publication.

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER SAM-TR-77-28	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) ANALYSIS OF TRACE VOLATILES BY GAS CHROMATOGRAPH/ MASS SPECTROMETER DATA SYSTEM.		5. TYPE OF REPORT & PERIOD COVERED Interim Rept Jan 1976-Oct 1977
7. AUTHOR(s) Charles L. Martin, William W. Lackey, James P. Conkle, Richard L. Miller		6. PERFORMING ORG. REPORT NUMBER
9. PERFORMING ORGANIZATION NAME AND ADDRESS USAF School of Aerospace Medicine (VNL) Aerospace Medical Division (AFSC) Brooks Air Force Base, Texas 78235		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 62202F 7930-11-35
11. CONTROLLING OFFICE NAME AND ADDRESS USAF School of Aerospace Medicine (VNL) Aerospace Medical Division (AFSC) Brooks Air Force Base, Texas 78235		12. REPORT DATE Dec 1977
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		13. NUMBER OF PAGES 15
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited.		15. SECURITY CLASS. (of this report) Unclassified
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Sorbent tube sampling Gas chromatograph/mass spectrometer data system Trace contaminant analysis Environmental sampling and analysis		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Methods and procedures are described for the analysis of trapped volatile samples by gas chromatography-mass spectrometry. The Tenax-GC sorbent tube sampler (stainless steel) and the desorption heater block described were developed in this laboratory to satisfy a USAF need for a field sampling method to complement the SAM cryogenic trapping system. The treatment of both sorbent-trapped and cryogenically-trapped samples is discussed showing that analysis and data reduction for sorbent tube samples are considerably less		

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20. ABSTRACT (CONTINUED)

than that required for cryogenic samples. While the sorbent tube sampling method has advantages of lower cost, ease of portability, simplicity, and reduced analysis time, the cryogenic sampling system is the preferred method for trapping light hydrocarbons of less than five carbon atoms.



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ANALYSIS OF TRACE VOLATILES BY
GAS CHROMATOGRAPH/MASS SPECTROMETER DATA SYSTEM

INTRODUCTION

Many research laboratories develop their own methods of sample collection and procedures for sample analysis. Recent developments have been in the area of environmental sampling (1-4, 6-8). This paper describes a sorption tube (USAF Invention No. 12,052) developed for trapping trace level volatiles and the methods and procedures used by USAF School of Aerospace Medicine (USAFSAM) personnel of the Crew Environments Branch in the analysis of samples by gas chromatograph/mass spectrometer data systems (GC/MS/DS). The sorption sample tubes have proved useful in sample analysis by other instruments; however, this report is confined to analysis by GC/MS/DS. Further, with some modifications, the sorbent tube analysis procedures are applicable to the analysis of sample cylinders from the USAFSAM cryogenic trapping system (2).

MATERIALS

The sorption sample tube (Figure 1) is of stainless steel (316), 1.27 cm x 15.24 cm, with a wall thickness of 0.89 mm. The interior wall of the stainless steel tube is electropolished to reduce the surface effects of the metal. About 2.5 g of 35-60 mesh Tenax-GC (a porous polymer of 2,4-diphenyl-p-phenylene oxide) is contained by double stainless steel screens at each end of the tube. The tube ends are connected to a modified Swagelok SS-810-C cap. The cap is centrally drilled and a Cajon VCR vacuum coupling (4VCR-3-A-SS gland and 4VCR-1SS female unit) is welded into the center. A special nylon plug and a 1/8-inch-thick (31.75 mm) Teflon washer seal the tube when not connected for sampling or desorption. The VCR fittings were adopted for this use because they offered a quick and easy method of connection for repeated use of tubes.

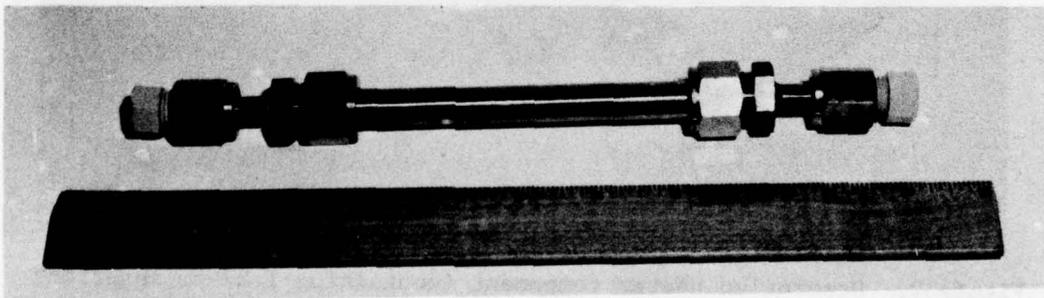


Figure 1. Sorption tube (1.27 cm) component of USAFSAM Sorption Tube Atmospheric Sample System (AF Invention No. 12,052).

The clamshell heater block used for sample tube desorption is shown in Figure 2 with a sample tube in position. The apparatus has two 300-watt heaters, a platinum resistance thermal controller, and a thermocouple to monitor block temperature. The block is constructed of aluminum and is designed with a minimum of mass to enhance the rapid heating and cooling of the sorbent tubes.

The complexity of the instrumentation of an integrated GC/MS/DS discourages a detailed description of all the various components and their functions. An overview of the GC/MS/DS (Figure 3) is a DuPont (21-491, Lot 5) mass spectrometer, a Hewlett Packard (HP) 2100A mini-computer with two 7970B digital tape units, a teletype with a Calcomp plotter, and a Varian gas chromatograph (model 1400, single column) with a hydrogen flame detector. A special feature of the mass spectrometer is the large 4-inch (10.16 cm) stainless steel elbow with an isolation valve attached to the ion source. They are connected to a 4-inch (10.16 cm) oil diffusion pump with a cryo-cooled baffle. This pumping system reduces the MS background; thereby enhancing the signal-to-noise ratio so that the sensitivity is increased. Further, the pumping system permits the use of GC capillary columns where all of the column effluent goes to the MS source, and source pressure is still maintained in the range of 10^{-6} torr.

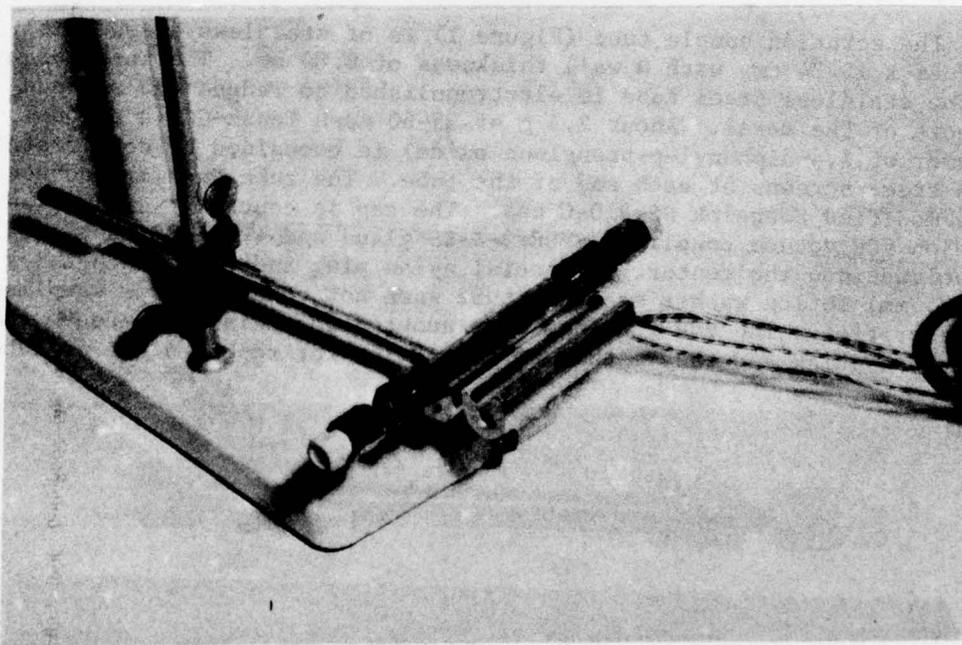


Figure 2. Desorption heater component (containing 1.27-cm sorption tube) of USAFSAM Sorption Tube Atmospheric Sample System (AF Invention No. 12,052).



Figure 3. View of the analytical system: Gas chromatograph (Varian 1400)-mass spectrometer (DuPont 21-491)-data acquisition (DuPont 21-094).

For analysis of most of the sorbent tube samples, the GC is equipped with a microbore (0.7 mm ID x 1.6 mm OD x 3 m) column packed with Porapak Q (120-150 mesh). The column effluent is split one part to the GC detector and two parts to the MS source via a heated stainless steel microbore 1.6 mm OD line and a stainless steel jet separator. The 2-to-1 effluent splitter in the GC oven was fabricated in this laboratory.

The spectra produced by the MS are accumulated by the DuPont 21-094 data system already mentioned. The software permits various manipulations of the data for output and presentation and a library search capability. Our spectra library consists of more than 23,920 spectra (9). Quantitation of the samples is accomplished by use of a HP 3352B laboratory data system which estimates peak areas from the GC flame detector response.

Figure 4 depicts the schematic of the analysis system and Figure 5 shows the actual sample introduction system with the sample tube in place for heating by the clamshell heating block. The pre-column sample loop of 3 cm³ volume is covered with a heating blanket. The two Whitey valves (SS-43YF2) to the left of the flowmeter are in the sample

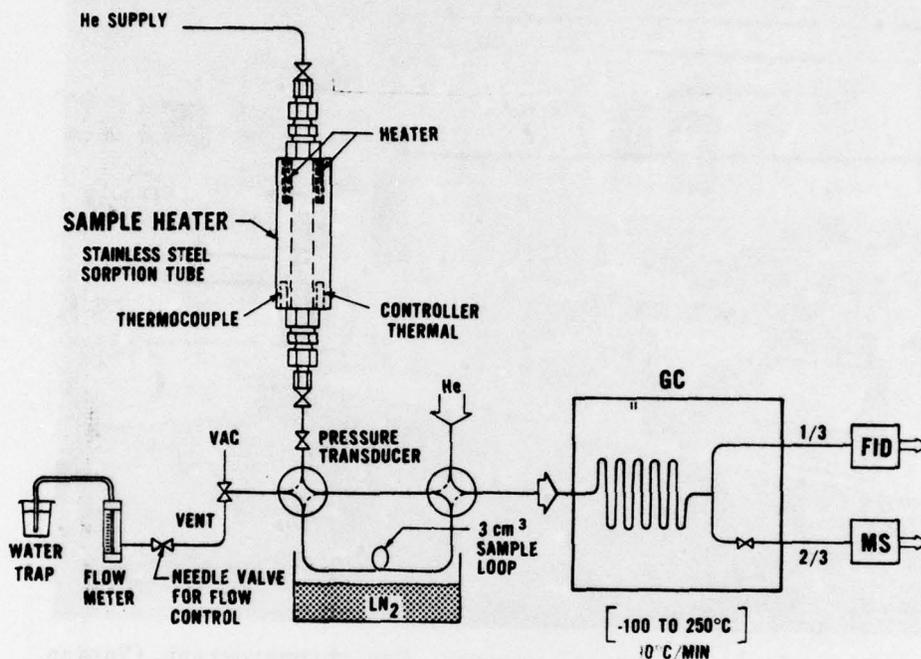


Figure 4. Schematic of analysis system.

injection position. The two valves attached to the sample tube ends are Nupro SS-4-JBA-1 valves modified with Cajon VCR male fittings for rapid connection of sorption tubes. Just to the right of the flowmeter, the handle of a three-way Whitey valve can be seen; this valve is connected to the flowmeter in one position and a vacuum line in the other. This arrangement allows evacuation of the 3-cm³ sample loop and valves up to the valve connecting the sample tube, plus the flow-through system for sample collection in the loop.

METHODS

Sorption Tubes

The sorption tubes are prepared for sampling by connecting them to a GC with a sample loop identical to that shown in Figure 5. The heater block is raised to 240°C while helium (special zero grade) flows through the sorbent bed for 1 hour at 30 cm³/minute. While the sample tube is still at 240°C, a sample is collected in the 3 cm³ sample loop (see the detailed procedure below); and a GC run is made to determine the

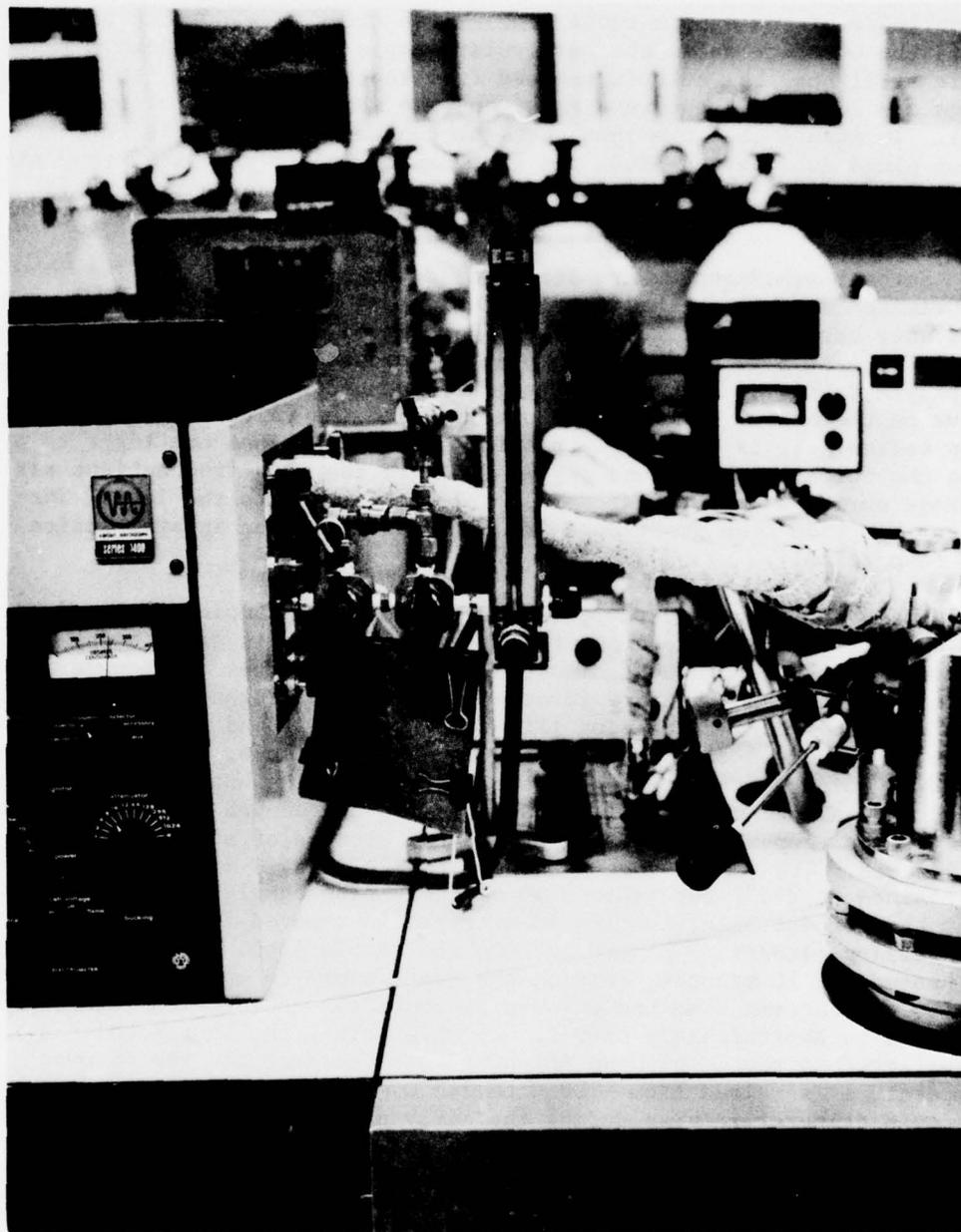


Figure 5. Gas sample introduction system of GC/MS/data system depicting sample injection.

cleanliness of the sample tube. This clean check procedure is repeated until the GC trace shows the particular sample tube to be clean. Then, while still hot, the tube is removed from the heating block, the nylon plugs are secured in the tube ends, and the sample tube is allowed to cool. To insure that the tubes are not contaminated prior to use, the nylon plugs are checked after the tube has cooled to make sure they are firmly set against the 1/8-inch-thick (31.75 mm) Teflon gasket inside the Cajon nut.

Sorbent tube samples are analyzed as shown in Figure 5. The tube is oriented so that the helium flow during desorption is opposite to the flow used during sample collection. Working on only one end of the sample tube at a time, the nylon plug is removed and the tube end is quickly attached to the appropriate valve connection--one end to the valve on the helium line and the other end to the valve above the sample loop valves. It is important that the tube connections are tight to prevent the formation of liquid oxygen (LOX) in the loop from ambient air leakage during sample transfer from the sample tube to the loop. The heater block is then positioned around the sample tube in preparation for desorption.

While the above procedure is carried out, the sample loop, valves, and connecting lines are evacuated (the sample loop is still being heated to 135°C by the heat blanket). The heat blanket is removed from the sample loop and the loop immersed in liquid nitrogen to maintain the loop at -196°C during trapping (Figure 6). To minimize any chemical conversions that might occur during desorption of the sample, the helium flow of 30 cm³/minute is initiated just prior to applying heat to the sample tube, thus allowing the various compounds to be removed from the sample tube as the temperature appropriate to the elution of a given compound is reached. The sample tube is heated to 240°C in 10 minutes and then maintained at 240°C for another 10 minutes. The sample loop is then sealed from the sorbent tube, the liquid nitrogen is removed from the loop, and the heating blanket is placed back on the sample loop. The sample loop is heated for 10 minutes, raising the temperature to approximately 115°C; at the same time the GC oven is routinely cooled with liquid nitrogen to approximately -100°C. At this point, the loop sample is transferred to the GC with an injection of 1 minute, and the GC oven temperature is raised from -100°C to 240°C at approximately 10°C/minute. The oven temperature rise during the early part of the run is controlled by the ambient air admitted to the oven and at 0°C the GC linear programmer controls the temperature at 10°C/min to 240°C. Starting the GC at -100°C allows the low molecular weight gases (Ne, N₂, O₂, Ar, CO, CH₄, CO₂, and N₂O), usually of little interest in the MS analysis, to clear the column prior to the elution of the hydrocarbons. This enhances the library search identification of the various GC peaks because the mass spectra are not masked by extraneous masses contributed by the low molecular weight gases. Further, even though there is little or no GC flame ionization response to these light gases, their presence or absence may be monitored by observing the display register of the data system computer during the run. When the computer is in the SCAN 1 mode (ready

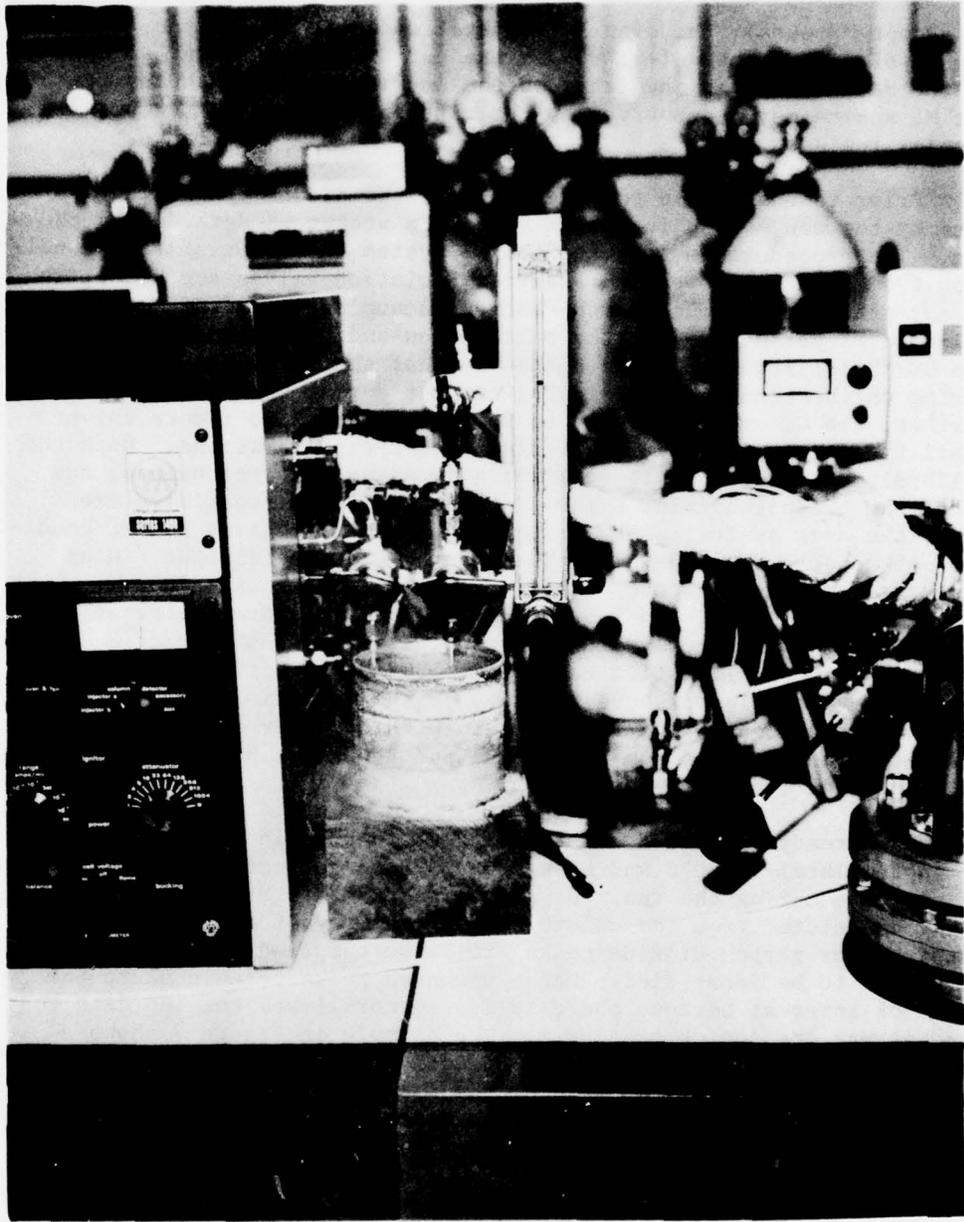


Figure 6. Gas sample introduction system of GC/MS/data system depicting sample transfer from sorption tube to sample loop.

to accept MS data), the display register shows in binary the total number of ions counted (± 1000) at the end of each MS scan, so that even if the data are not being recorded the occurrence of compounds reaching the MS source may be observed by the rise and fall of the computer display register.

Prior to the sample tube desorption, the MS was calibrated and the data system was set up in a SCAN 1 mode to accept MS data during the GC run. The procedure is to bring the MS system to an operational condition from standby: open the analyzer isolation valves and establish steady-state conditions for the source vacuum and temperature with selected MS parameters for the calibration and subsequent GC/MS runs. The GC/MS isolation valve is opened so that the MS source pressure during calibration will be similar to that during the GC/MS runs. Further, the GC oven is cooled to room temperature to reduce the possibility of column bleed interfering with the calibrations. With the calibration mixture (perfluorokerosine¹, methanol, cyclohexane, and water) in the batch inlet system and the MS in the repetitive scan mode, the data system is calibrated to the MS at a given set of conditions through a dialogue with the computer via the teletype. Most analyses are done with the following set of conditions: 70 eV ionization potential, low ion current (~150 ma), source temperature 250°C, source pressure 1×10^{-6} torr, repetitive scan rate of 2 seconds/decade, calibration mass range 12-219, sensitivity (multiplier tube high voltage) set above the knee of the operating curve, and the computer data acquisition rate set at 10 kHz. Other adjustable parameters such as the ion beam focus and repellers had previously been optimized for the above conditions.

As already mentioned, most of the GC runs begin at an oven temperature of approximately -100°C and mass spectra data collection may be initiated at any time during the run. Usually, spectra data are collected after water has eluted from the column because in many of the samples the water and/or carbon dioxide peaks are large compared to the hydrocarbons which are to be identified. Large unwanted peaks often obscure the peaks of interest because the data system normalizes the ion data to reconstruct the ion chromatogram. The example in Figure 7 shows a reconstructed ion chromatogram of an analysis of laboratory room air concentrated on a Tenax-GC sorbent tube. The x-axis is the MS scans recorded during the run, and the coincidence hash marks in the ion plot each represent the total number of ions accumulated for all masses during a given MS scan. Appropriate scans (fracture patterns) are selected to represent the various peaks; these are indicated to the computer via the teletype to be compared to the library spectrum patterns through a library subroutine similar to that described by Hertz et al. (5). The output via the teletype is the five best choices with a goodness of fit (similarity index of spectra), ID number (origin of the spectrum),

¹ PFK-225-Universal Standard #65417, Pierce Chemical Co., Rockford, Ill. 61105.

and the first two choices are named. The choice selected by the computer together with the value of goodness of fit weigh heavily in labeling a given peak; however, the GC retention time and the origin of the sample are important considerations in the identification process.

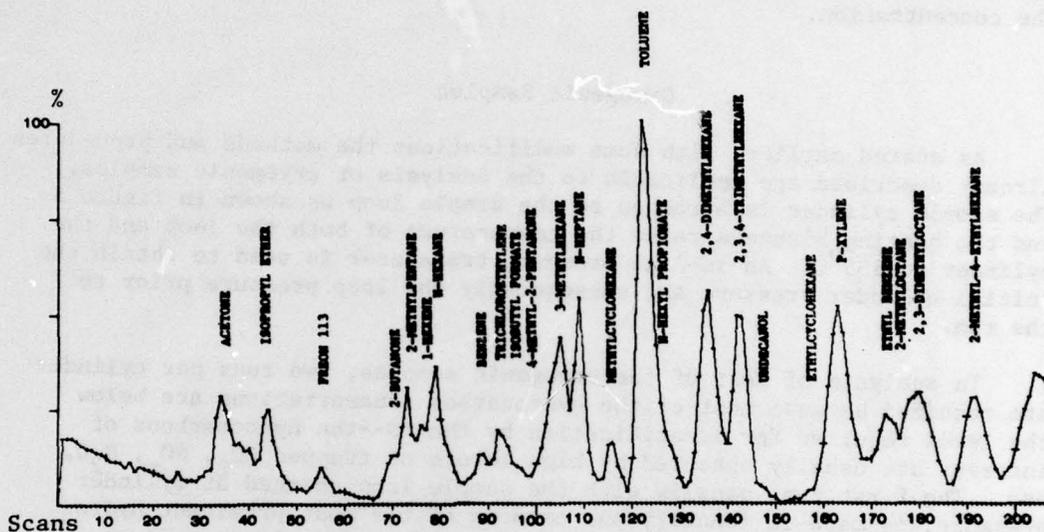


Figure 7. Ion chromatogram from GC/MS/data system: 480 liters of room air (Laboratory Room 36, Bldg 170, Brooks AFB, Texas, 29 June 1977).

During the run, the GC peak areas were accumulated by the HP 3352B Lab Data System to be used in estimating concentration levels of the sample components. This requires that a standard of known concentration must be run under the same conditions as the unknown samples. In most of the sample analyses, the concentrations of the compounds are given as N-hexane. The 3-cm³ sample loop was pressurized to 760 mm Hg at 135°C with a 95 ppm (0.3344 mg/l) N-hexane standard.² After correcting for the heated loop, 0.691 µg of N-hexane were injected on the GC column to obtain the flame detector response, in area units of the peak, which results in a nominal value of 2.42×10^{-7} µg/unit area as a multiplier to calculate concentrations; that is,

$$\mu\text{g}/\text{m}^3 = (k \times A) / (V \times 10^{-3}) \quad (1)$$

where $k = 2.42 \times 10^{-7}$ µg/unit area
 A = units of area of any given peak
 V = total sample volume trapped (in liters).

²Hexane Standard, Lot #020171R prepared by Matheson Gas Products.

In some special cases a standard curve is established because the main interest is in the presence or absence of a particular compound, for instance, dimethylnitrosamine (NDMA). Standards of various concentrations are made up and injected so that a least-squares curve may be established to more precisely state the levels of concentrations. The amount of NDMA is calculated directly from the equation of the standard curve and the result becomes the numerator in equation 1 in estimating the concentration.

Cryogenic Samples

As stated earlier, with some modifications the methods and procedures already described are applicable to the analysis of cryogenic samples. The sample cylinder is attached to the sample loop as shown in Figure 5 and the heating blankets raise the temperature of both the loop and the cylinder to 135°C. An in-line pressure transducer is used to obtain the initial cylinder pressure and subsequently the loop pressure prior to the run.

In analysis of most of the cryogenic samples, two runs per cylinder are required because most of the hydrocarbon concentrations are below the level required for identification by the MS--the hydrocarbons of interest are usually obscured by high levels of trapped CO₂, NO_x, H₂O, etc. The first run, usually with the sample loop charged at cylinder pressure, is made to identify one or more of the most prominent hydrocarbons and estimates of concentrations. The second run is made on the entire cylinder contents in order to obtain maximum MS identification of the sample components. The cylinder contents are collected in the sample loop by a gradual evacuation of the cylinder via the sample loop which is immersed in liquid nitrogen. Frequently the flow through the loop is stopped by a freeze-up of H₂O in the case of the 0°C cylinder or CO₂ in the case of the -175°C cylinder. The -78°C cylinder seldom causes any problems in the concentrating procedure. In the case of the -175°C cylinder the liquid nitrogen is removed from the loop and replaced by a mixture of dry ice and ethanol, which allows the CO₂ to be pumped off while the ice plug, resulting from the 0°C cylinder evacuation, may be removed simply by removing the liquid nitrogen allowing the loop to warm up and then replacing the liquid nitrogen for continuation of trapping. For running the trapped samples the procedure is the same as that described for the sorption tubes.

The treatment of the area data and the calculations are more involved than that described for the sorption tubes because the sample is divided among the three trapping cylinders and the analysis data are spread over six GC/MS runs. For each cylinder a concentration factor (K) is determined from the ratio of compound(s) identified in both the concentrated and unconcentrated runs. Then for any compound in a given cylinder

$$X_n (\mu\text{g}) = \left(\frac{P_c \times V_c}{P_1 \times V_1} \right) + \left(\frac{k A_c}{K} \right) \quad (2)$$

where A_c = area units of compound X in the concentrated run
 k = $\mu\text{g}/\text{unit area}$ for N-hexane standard
 K = concentration factor defined above
 V_c = volume of cylinder and connection to pressure transducer (153.6 cm^3)
 V_1 = volume of sample loop (3 cm^3)
 P_1 = initial pressure of the cylinder
 P_c = pressure of the loop for the unconcentrated run
 X_n = μg of a given compound in a cylinder where $n = 1, 2, 3$ are 0°C , -78°C , and -175°C cylinders respectively.

Then

$$\mu\text{g}/\text{m}^3 = \frac{X_1 + X_2 + X_3}{V_t \times 10^{-3}} \quad (3)$$

where V_t = total sample volume trapped (liters)

gives the concentration of a given compound in the sample. These repetitive calculations are done by a computer program because of the large number of calculations required for even a single sample analysis.

DISCUSSION

The design chosen for the sorbent sample tube was generated from a need for a rugged sampling device to withstand field use and freight shipment and for a method of sampling which would be less cumbersome than a cryogenic sampling system. Our first effort was to use glass tubes of sorbent following the design of Pellizzari et al. (7). The first field trip resulted in half of the tubes being broken in shipment to the site even though they were well packaged. Further, the desorption apparatus was unsatisfactory in several aspects: First, it was difficult to insure that a leak-tight system existed prior to sample tube desorption so that LOX was not formed in the sample loop from ambient air leakage during sample transfer. Secondly, the sorbent tube holder for desorption was contaminated by each sample desorbed which required that the empty holder be checked for cleanliness before each use (a GC run had to be made on a sample trapped from the empty holder). Also, the mass of the holder caused the heating and cooling process to be prolonged.

The sorbent tube sampling system described in this report was designed to avoid these faults. The stainless steel construction gives the tubes the ruggedness required for field use, and the method of connecting the sample tube to the GC sample loop insures a leak-tight

system. Further, our design of a heater block in the form of a clam-shell allows it to be removed at any time after desorption of a sample so that preparation may be made to run the next sample while a run is in progress. Removal of a desorbed tube leaves only the sample loop and connectors to be purged prior to the next sample run.

Stainless steel screens are used to contain the sorbent in the sample tubes because it was found that the glass wool used to contain the sorbent in the glass sample tubes was contaminated with a variety of chemicals. Presumably the contamination was from adsorption of chemicals from the laboratory room air since the glass wool had not previously been used and had been stored in the manufacturer's shipping carton. The variety and quantity of chemicals desorbed from a sample tube packed with glass wool were the deciding factor in the decision to use stainless steel screens to contain the Tenax-GC in the sample tubes. Sickels and Stafford (8) used two glass wool traps to collect water vapor ahead of a Porapak Q sorbent tube. Undoubtedly, the glass wool trapped a large fraction of the organics that would have otherwise been trapped by the Porapak Q sorbent bed. Their results may have been improved with a Tenax-GC sorbent tube which eliminates the need for water vapor traps ahead of the sample tube--Tenax-GC retains water vapor rather poorly.

The supplier of Tenax-GC (Applied Sciences Laboratories, Inc.) indicates the sorbent material is stable up to 350°C; while other investigators (1, 7) use desorption temperatures of 290°C and 270°C respectively, we use 240°C for sample tube conditioning and sample desorption. In this laboratory we have determined that benzene is a major breakdown product due to overheating Tenax-GC and that a significant amount may be trapped in the sample loop (see Methods section) at temperatures above 250°C. A previously used and preconditioned sample tube was maintained at 275°C, with 30 cm³/min helium flow through, for 18 hours; a 10-minute sample trapping in the GC sample loop (immersed in liquid nitrogen) produced a benzene peak which would interfere with any trace analysis. Further, we determined that the quantity of benzene trapped above 250°C was directly related to the desorption or conditioning temperature.

The microbore (0.7 mm ID) columns have been in use in our laboratory for the past 5 years and might be classified as packed capillary columns. An idea of the column resolution may be obtained from Figure 7. Using the base peak (toluene) in the ion plot, the peak width at one-half the peak height is 23 seconds which is about as sharp as the peaks can be at a mass scan rate of 2 seconds/decade. The main disadvantage of these columns is that a liquid injection of more than 4 µl overloads the column so that separation is poor. This limitation is of little consequence in most of the GC/MS/DS analysis in this laboratory.

Figure 7 depicts the results that would be obtained from sampling the environment almost anywhere in the United States. The concentrations of the various compounds would vary depending on where the sampling was done and the degree of industrialization of the area;

however, the array of chemicals identified would be essentially the same. The data in Table 1 show that the concentration levels of all the chemicals identified in this laboratory's room air are well below the current safe limit set by the Occupational Safety and Health Administration.

TABLE 1. ANALYSIS OF LABORATORY ROOM AIR^a

<u>Compound</u>	<u>ng/m³</u> ^b
Acetone	64
Unknown #1	35
Isopropyl ether	68
Freon 113	9
Unknown #2	6
2-Butanone	20
2-Methylpentane	50
1-Hexene	31
n-Hexane	95
Benzene	42
Trichloroethylene	18
Isobutyl formate	13
4-Methyl-2-pentanone	9
3-Methylhexane	148
n-Heptane	113
Methylcyclohexane	43
Toluene	425
n-Hexyl propionate	T
2,4-Dimethylhexane	187
2,3,4-Trimethylhexane	174
Undecanol	T
Ethylcyclohexane	T
p-Xylene	322
Ethyl benzene	114
2-Methyloctane	63
2,3-Dimethyloctane	75
2-Methyl-4-ethylhexane	94

^aTenax-GC sample of 480 liters of air (Laboratory Room 36, Building 170, Brooks AFB, Texas, 29 June 1977).

^bValues are expressed as N-hexane.

CONCLUSIONS

The combination of a GC/MS/DS and the sorbent tube sampler offers a method of rapid sample analysis for multicomponent samples. The analysis of the example run shown in Figure 7 was completed in 2 hours. Further, the sorbent tube sampler is less cumbersome than a cryogenic sampler system and less equipment is shipped for field samples. The sorbent tube sampler requires only a small pump and flowmeter; whereas, a cryogenic system requires a source of ice, dry ice, and liquid nitrogen which may be a problem in the field. Also, the cryogenic system requires considerably more attention in its operation, maintenance, and cylinder preparation for sampling.

Even though the sorbent tube sampler has the above advantages over the cryogenic trapping system, cryogenic trapping is the choice of sampling if light hydrocarbons ($\leq C_5$) are of interest (1). Tenax-GC shows selectivity towards certain classes of compounds and traps light hydrocarbons inefficiently. Further, while this report describes sorbent tubes of Tenax-GC, the sorbent material used should be chosen according to the class of compounds that are of interest. Sample tubes of various other sorbent materials are currently being evaluated.

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