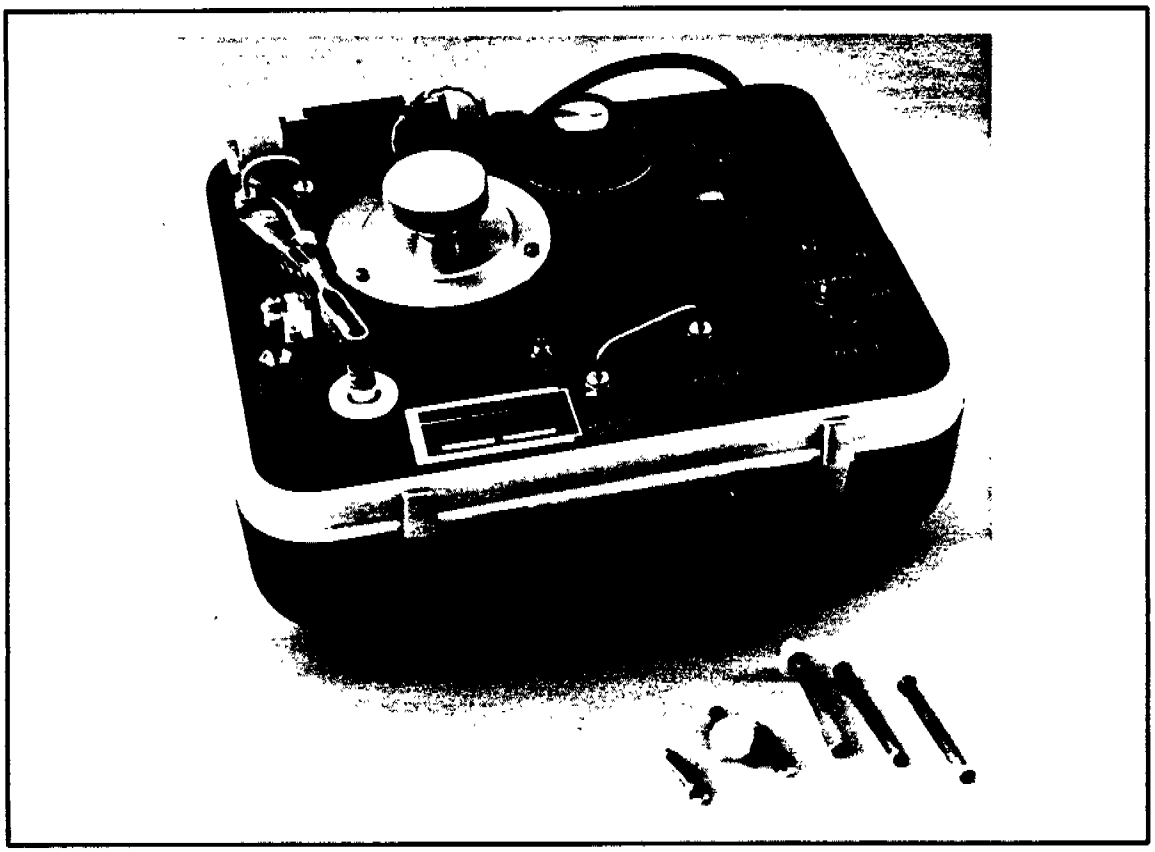

DETERMINING ENVIRONMENTAL CONTAMINATION

With the CENTURY Programmed Thermal Desorber and Organic Vapor Analyzer

Time weighted averages of environmental contamination can be determined using the CENTURY Programmed Thermal Desorber PTD-132A and Organic Vapor Analyer OVA-128 more conveniently and accurately and in less time than with chemical desorption methods.

SORBENT COLLECTOR TUBES—BACKGROUND

The use of sorbent collector tubes is a well accepted technique for evaluating the amount of environmental contamination as a time weighted average (TWA). These tubes are generally packed with a compatible sorbent medium such as activated charcoal, Tenax, or carbon spheres. In use, the tube is opened on both ends either by breaking off the tips, as is the case with glass tubes, or by removing polyethylene caps as in the case of metal tubes. See Figure 1. The tube is then connected with plastic tubing to a portable air pump that draws a known volume of environmental air through the tube during the monitoring period.

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Figure 1

After such exposure, the collector tube is capped to prevent the loss of sample by diffusion. The sample is then analyzed in one of several ways to determine the TWA. The most often used method for tube desorption is the use of chemicals (such as carbon disulphide) to physically extract adsorbed contaminent chemicals for later analysis by gas chromatography.

THE PROBLEM

Though widely utilized, chemical desorption has several disadvantages that hinder rapid and effective time-weighted-average determination. These are:

1. Long, sample analysis time.
2. Expensive, individual sample handling.
3. Careful laboratory procedures required.
4. The use of toxic chemicals.
5. Elaborate calculations required.

THE SOLUTION—THE CENTURY PROGRAMMED THERMAL DESORBER (PTD)

The PTD represents an attractive method for sample handling in preparation for analysis by an analytical instrument. See Figure 2. This device thermally desorbs the adsorbed chemicals from the adsorbent at a temperature preselected by the user, stores the desorbed sample in a small chamber of known volume, and automatically injects aliquots of the sample into a gas chromatograph or other analytical instrument.

At each stage in this process, the PTD method proceeds under user preselected conditions that have been determined by experiment to yield optimum results.

PROCEDURE

The contaminated collector tube is placed in the PTD desorption oven which is then sealed. Upon pressing the "start" button, a small pump begins to withdraw air from a sealed chamber over a piston. As the piston begins to rise, a partial vacuum is formed that draws air from the environment through a charcoal scrubber, through the heated tube, and into the expanding volume under the piston in the storage chamber As the piston reaches the top of the chamber, the pump stops automatically, and the chamber is sealed. The volume of the storage chamber is 300 mL in the fully desorbed position. See Figure 3

After desorption is complete, the user may choose to remove aliquots of sample from the chamber for analysis either manually or automatically.

- If the automatic method is chosen, a pump integral to the PTD may be used to draw sample from the storage chamber through a sample loop of 2.5 mL volume, which is then made part of the carrier gas line of a laboratory gas chromatograph. This is accomplished by means of a solenoid-operated sample valve programmed to inject the sample after a given period of time.
- For the manual mode, a pump external to the PTD may be used to extract sample from the storage chamber. The sample is then injected into a portable gas chromatograph, such as the OVA-128, or other analytical instrument.

In the first case, the PTD contains an integral sample loop that is automatically flushed utilizing a part of the 300 mL sample in the chamber. Since the volume between the sample storage chamber and the sample loop is only a few mL, multiple sample injections are possible before the storage chamber is completely emptied.

For manual injection, as in the case where the PTD is utilized with the Century Portable Organic Vapor Analyzer (OVA), a pump in the OVA draws sample out of the chamber for analysis.

Figure 2

THE BENEFITS OF USING THE PTD SYSTEM

The PTD has several advantages, over currently used chemical desorption techniques, that benefit the user with higher accuracy and savings in cost, time, and manpower. It also relieves laboratory personnel from repetitious and exacting determinations:

1. Desorption and storage of samples is automatic.
2. Sample tubes are reusable.
3. Time-weighted-average calculations are simple.
4. Exacting laboratory technique is unnecessary.
5. No chemicals are necessary.
6. Sample injection is automatic.
7. Replicate sample injections are possible for increasing accuracy.

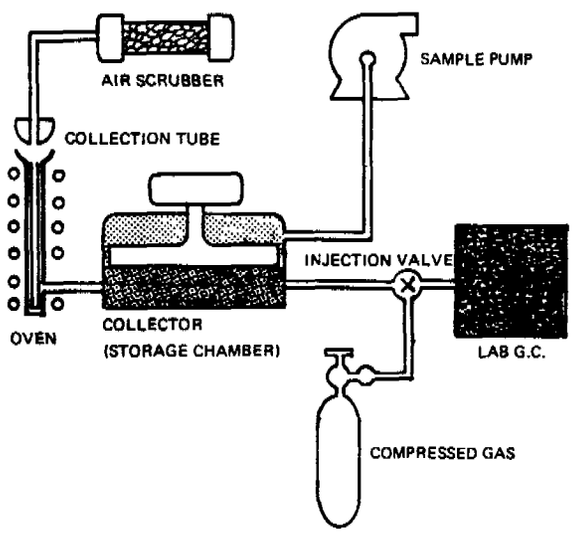


Figure 3

THE CENTURY ORGANIC VAPOR ANALYZER (OVA)

The OVA is a portable gas chromatograph that uses a flame ionization detector that responds to organic vapors. It may be used either as a survey tool or to quantify specific compounds by means of a chromatographic column and sample-injection system. See Figure 4.

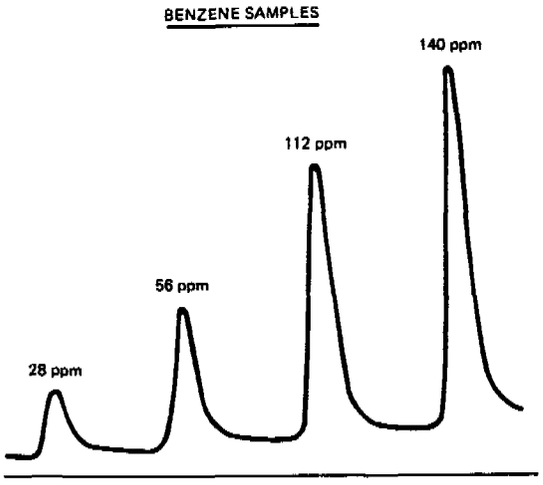
As a gas chromatograph, the OVA incorporates a voltage output sufficient for use with a variety of chart recorders. In this way, chromatograms are produced that serve to both identify a compound (retention time) and quantify it (peak height measurement).

As shown in Figure 5, peak height is proportional to concentration of sample. This relationship can be expressed as a calibration curve as shown in Figure 6.

When expressed as a calibration curve, the operator can rapidly determine concentration of sample thereby speeding up analysis.

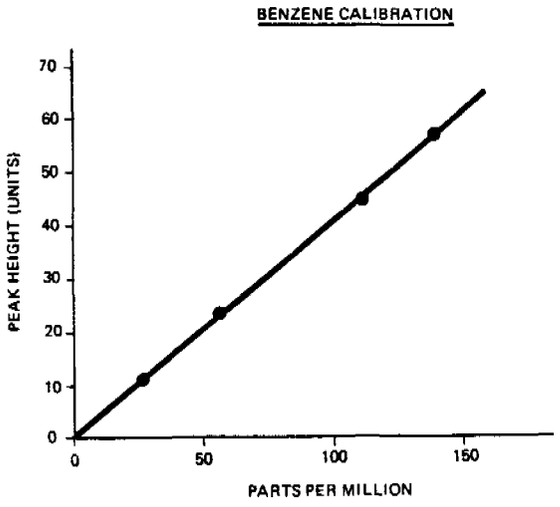
When used with the PTD, the OVA-128 GC is attached directly to the desorber by a teflon tube. (See Figure 7.) When the "read" button on the PTD is depressed, a solenoid valve opens the storage chamber allowing the OVA sample pump to withdraw sample to be used in flushing its sample loop. A steady reading on the OVA meter indicates that the sample loop has been thoroughly flushed and that the manual sample valve may be actuated to begin chromatography.

Figure 4



G-8 COLUMN
0°C
RETENTION TIME = 80 s

Figure 5



CHROMATOGRAPHIC PEAK HEIGHT VS SAMPLE CONCENTRATION
0°C PIP G-8 COLUMN

Figure 6

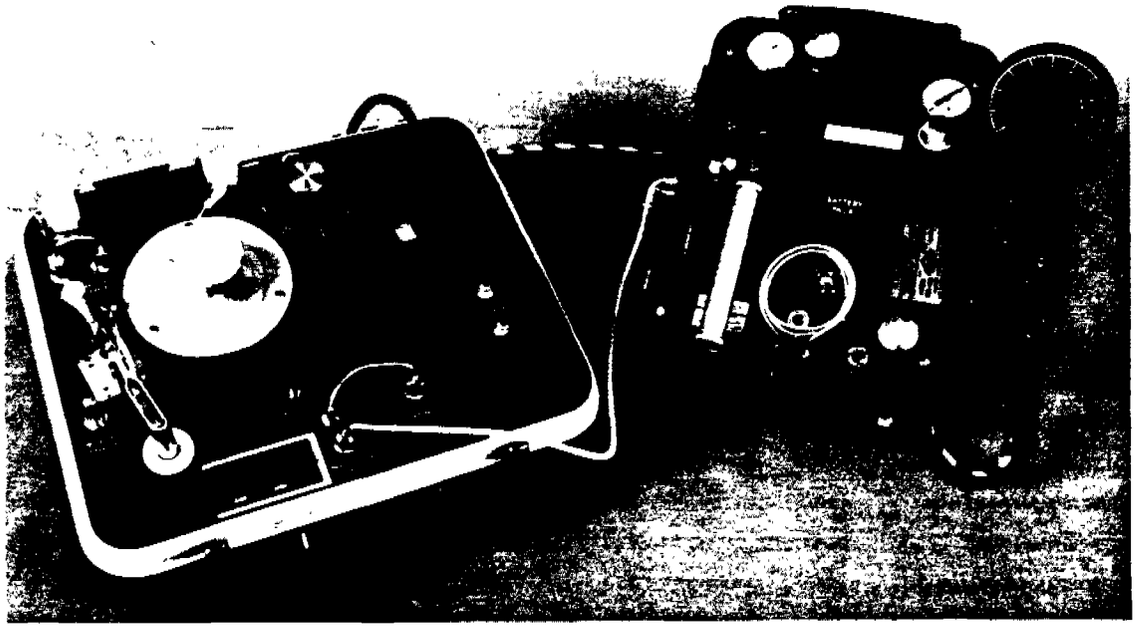


Figure 7

EXAMPLES

The chromatograms shown in Figures 8 through 13 illustrate the use of the PTD with the OVA-128 GC to produce chromatograms that serve both to identify and quantify environmental contaminants.

In each example, a sample of vapor of known concentration was prepared in a sampling jar and a chromatogram was generated using the OVA. In Figure 8, the chromatogram was run on 223 ppm of isopropyl alcohol on an 8-inch tris column at a temperature of 0°C. Under these conditions, the retention time was 214 seconds.

After the initial chromatogram was run, 300 mL of sample was drawn through a collector tube. The sample was then desorbed in the PTD under a known set of conditions. Since the storage chamber of the PTD is 300 mL, the concentration in the chamber should be identical to that in the original sample, assuming 100% desorption efficiency.

In Figure 8, the collector tube was packed with CS-5 (carbon spheres) and was desorbed at 200°C at the rate of 1 mL/s.

A comparison of peak heights (peak height is proportional to concentration) for the chromatograms of sample and desorbed sample gives a direct measure of desorption efficiency. In the example given in Figure 8, desorption efficiency is 100% since the peak heights of the two chromatograms are identical.

TIME WEIGHTED AVERAGE CALCULATION

Calculation of the time weighted average (TWA) for a compound of interest is greatly simplified by use of the PTD. The formula can be expressed as:

$$\text{TWA} = \frac{\text{PTD Chamber Volume}}{\text{Volume of Air Sampled}} \times \text{Concentration of Desorbed Sample}$$

As an example, an air sampling pump was utilized over an eight-hour period to draw environmental air through a metal sample tube at the rate of 20 mL per minute. The tube was later desorbed on the PTD, and the desorbed sample was found to contain 120 ppm of benzene.

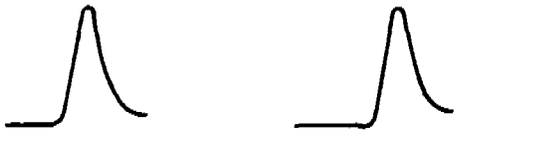
The time weighted average (8 hours) is found using the formula:

$$\text{TWA} = \frac{0.300}{0.02 \times 60 \times 8} \times 120 \text{ ppm} = 3.75 \text{ ppm}$$

The analytical instrument utilized to determine the concentration of desorbed sample can be a gas chromatograph, an infrared analyzer, or another instrument capable of the desired degree of accuracy for the measurement at hand.

SAMPLE	SAMPLE AFTER DESORPTION
COMPOUND	ISOPROPANOL
COLUMN	10% TRIS PROPANE ON SUPELCOPORT
TEMPERATURE	0°C
RETENTION TIME	214 s
SAMPLE CONCENTRATION	223 ppm
DESORPTION TEMPERATURE	200°C
DESORPTION RATE	1 mL/s
DESORPTION TUBE	CS-5
DESORPTION EFFICIENCY	100%

Figure 8



SAMPLE	SAMPLE AFTER DESORPTION
COMPOUND	ACETONE
COLUMN	10% TRIS PROPANE ON SUPELCOPORT
TEMPERATURE	0°C
RETENTION TIME	117 s
SAMPLE CONCENTRATION	223 ppm
DESORPTION TEMPERATURE	200°C
DESORPTION RATE	1 mL/s
DESORPTION TUBE	CS-5
DESORPTION EFFICIENCY	100%

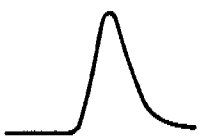
Figure 9

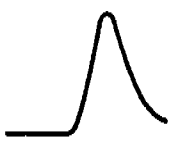
SAMPLE	SAMPLE AFTER DESORPTION
COMPOUND	METHANOL
COLUMN	10% TRIS PROPANE ON SUPELCOPORT
TEMPERATURE	0°C
RETENTION TIME	150 s
SAMPLE CONCENTRATION	668 ppm
DESORPTION TEMPERATURE	200°C
DESORPTION RATE	1 mL/s
DESORPTION TUBE	CS-5
DESORPTION EFFICIENCY	100%

Figure 10

SAMPLE	SAMPLE AFTER DESORPTION
COMPOUND	CHLOROFORM
COLUMN	10% TRIS PROPANE ON SUPELCOPORT
TEMPERATURE	0°C
RETENTION TIME	115 s
SAMPLE CONCENTRATION	223 ppm
DESORPTION TEMPERATURE	200°C
DESORPTION RATE	1 mL/s
DESORPTION TUBE	CS-5
DESORPTION EFFICIENCY	98%

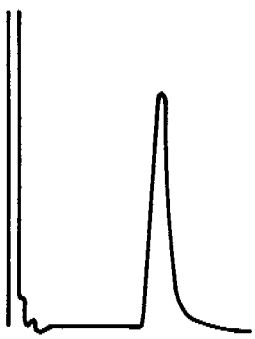
Figure 12

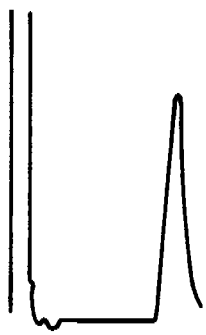




SAMPLE	SAMPLE AFTER DESORPTION
COMPOUND	ETHYL ACETATE
COLUMN	10% TRIS PROPANE ON SUPELCOPORT
TEMPERATURE	0°C
RETENTION TIME	140 s
SAMPLE CONCENTRATION	223 ppm
DESORPTION TEMPERATURE	200°C
DESORPTION RATE	1 mL/s
DESORPTION TUBE	CS-5
DESORPTION EFFICIENCY	95%

Figure 11





SAMPLE	SAMPLE AFTER DESORPTION
COMPOUND	BENZENE
COLUMN	10% SP-21 ON SUPELCOPORT
TEMPERATURE	0°C
RETENTION TIME	80 s
SAMPLE CONCENTRATION	111 ppm
DESORPTION TEMPERATURE	250°C
DESORPTION RATE	1 mL/s
DESORPTION TUBE	CS-5
DESORPTION EFFICIENCY	95%

Figure 13

CONCLUSION

The Organic Vapor Analyzer, and the Programmed Thermal Desorber are powerful analytical tools for collection and analysis of environmental samples, on-site, by relatively inexperienced personnel.

The combined use of the two instruments provides a system that yields real-time answers to questions of worker exposure to hazardous gases and vapors.

As applications are broadened, these instruments will assume a dominant position for environmental monitoring where accurate data, rapidly obtained, is required for flexible response.

REFERENCE LITERATURE

PSS 6-12Z1 B CENTURY Programmed Thermal Desorber (PTD-132 A)
PSS 6-12Z1 A CENTURY Dual-mode Organic Vapor Analyzer (OVA-108 and 128)
PSS 6-12Z1 D CENTURY Organic Vapor Analyzer (OVA-88)

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