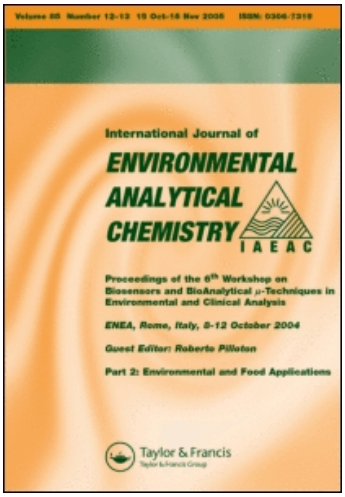

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Automated Monitor Systems for the Continuous Surveillance of Environmental Samples†

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Todays progress in chemical and allied industries as well as the product development leading to such progress depend upon the successful mastering of environmental and safety and health problems. There is a challenging need for the identification and quantification of hazardous substances in the air and water at concentrations in the ppm, ppb and often ppt range or below that value. A continuous surveillance is best achieved by automated monitor devices, responding to specific chemicals of choice.

In our Department of Analytical Research we have been developing surveillance monitor systems for several years and we accumulated experience in the field of ambient air and waste water control.

Our instrumental concept was to construct an easily controlled mobile instrument, distinguished by its high sensitivity and specificity for trace and ultratrace substances, and its versatility and sturdiness in field measurements.

The monitor systems represent integrated analysis systems in which standardization and calibration procedures, trace enrichment steps and gas chromatographic separations are linked together on-line thus permitting a continuous, automated operation. Conventional as well as unconventional detectors, such as a mass spectrometer may be coupled with the separation step. The monitor systems are designed to perform survey analyses at a rate of one to ten analyses per hour on the hour and may be equipped with an acoustic or optical warning device, indicating the moment when a specific substance surpasses a certain critical concentration limit.

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†Presented at the International Workshop on Handling of Environmental and Biological Samples in Chromatography, Lausanne, November 24–25, 1983.

The paper describes three types of instrument designs, distinguished by their degree of sophistication, i.e. the detection limit on one side and the different media in which it operates on the other.

KEY WORDS: Automated Monitor Systems, continuous surveillance, environmental sample.

INTRODUCTION

Environmental considerations and occupational safety and health aspects play an ever increasing role in chemical and allied industry. Today they require a considerable amount of analytical effort.

There exists a challenging need to identify and quantify hazardous substances in ambient air and water at concentrations in the trace and ultratrace region. In many cases a continuous surveillance is requested by corporate demands or governmental requirements. There is a definite need for automated monitor systems, operating in different media and responding to specified chemicals.

During the last five years considerable efforts were launched by the Department of Analytical Research at Ciba-Geigy Ltd. in Basel to develop appropriate surveillance monitors. Much experience has been accumulated in the field of ambient air and waste water control. Some of the systems have been described before.[1-3]

According to the original instrumental concept the monitors consist of different modular units trimmed for specific applications, combined into integrated total analysis systems with a wide versatility, diversification and specificity. Since they were designed for internal corporate use only, they are free of commercial compromises.

OPERATIONAL PRINCIPLES

The operational principle is extremely simple. The air sample is pumped through a tubing system of up to 100 m length and passed through a capillary adsorption tube, packed with a suitable adsorbent. After a certain collection period which may depend upon the desired sensitivity, the adsorbed material is instantly desorbed by a flash evaporator and fed into a packed gas chromatography

column, were it is separated into its components and detected by an appropriate detector system.

The operational modes include a series of analytical steps such as sample taking, sample preparations, separation and detection, followed by data handling and conversion into steering and feedback signals.

All these steps are linked together on-line, resulting in a continuous and automated instrumental unit. Figure 1 represents a diagram of the operational mode.

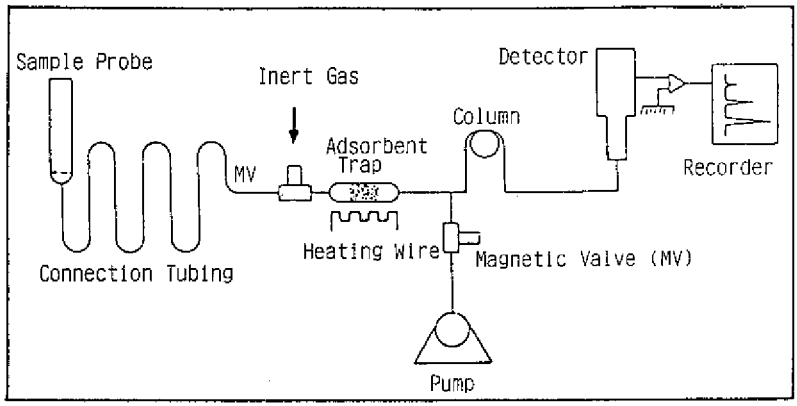

FIGURE 1 Flow Diagram of the Surveillance Monitor.

THE MODULAR UNITS

The integral monitor system represents a combination of two types of modular units, the analytical modules on one side and the electromechanical devices on the other.

Among the analytical units are the miniature version of a gas chromatograph, the adsorption/desorption capillary unit and the detector system. The proper choice of adsorbent material (enrichment trap), separation column and detector is essential for the specificity and sensitivity of the monitor system.

The electromechanical parts include the programmer as the center piece of the entire instrument, a gas unit for the supply of carrier and inert gas, timer and temperature regulator.

In addition the system is continuously calibrated and standardized by a reference source (permeation cell).

OPERATIONAL MODES

The fully automated instrument operates in different steps or functional phases, which may be described as follows:

—Phase I (Starting mode): Rinsing of all analytical parts and connection lines with clean inert gas and sample gas, respectively.

—Phase II (Sampling): Sampling and subsequent trapping of the sample substances on the adsorbent (enrichment).

—Phase III (Rinsing): Rinsing of the adsorption/desorption tube and chromatographic column with inert gas and pressure adjustment.

—Phase IV (Analysis): Thermal desorption, chromatographic separation and detection of the sample, including signal recording and processing.

—Return to the original state (Phase I).

APPLICATIONS

The monitor systems are designed to perform survey analyses in a repetitive, continuous way at a rate of one to ten analyses per hour on the hour. Due to the mechanical and operational sturdiness and the modular versatility the instruments are meant to be used in field investigations. There are three different types of monitor systems we shall describe in more details. They distinguish themselves only by the degree of sophistication.

Surveillance monitor [Dimethyl sulfate monitor]

The surveillance monitor is described in Figure 2. The sampling part is designed to separate dust and other solid particles from the ambient air by a filtering probe. Immediately following this probe

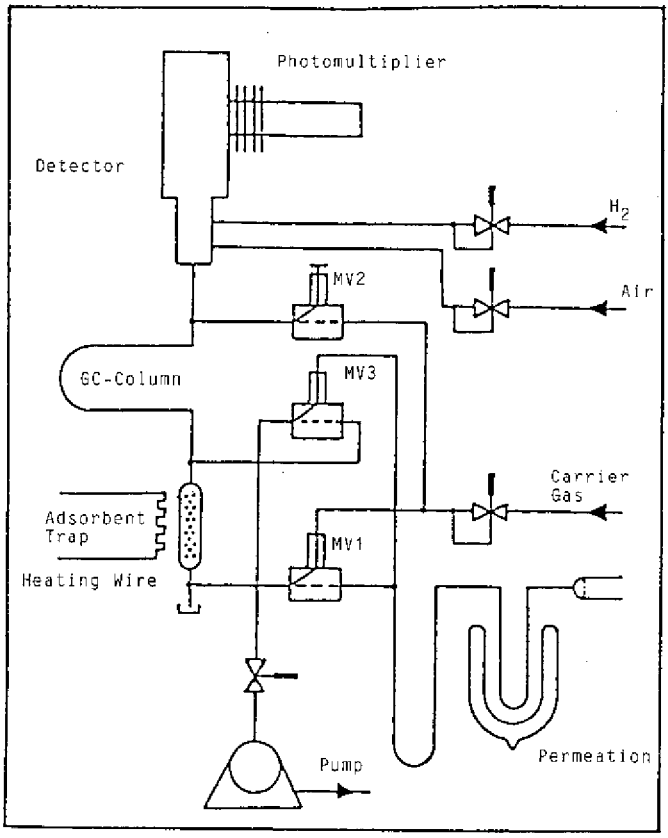

FIGURE 2 Operating Scheme of DMS Monitor.

unit the sample gas is spiked with a standard addition of the chemical to be analyzed, e.g. dimethyl sulfate (DMS), delivered from a permeation cell. The added analyte substance serves three different purposes:

—due to the standard addition the instrument is constantly checked for proper functioning.

—the added material occupies the active sites on the surfaces of the system and prevents losses due to adsorption.

—most gas chromatography detectors exhibit a non-linear response

at low trace concentrations; with the standard addition the operational range of the detector is shifted towards a more sensitive and possibly linear response range.

After a sampling period of about one minute the trapped DMS is desorbed by rapid heating of the adsorption trap to about 140°C. After a gas chromatographic separation DMS is detected by a sulfur-sensitive flame photometric detector, operating at 340 nm. The detection limit is about 10 ppb DMS in ambient air.

Figure 3 represents a typical recording of results obtained from a monitor linked to four different sampling units. These may be separated from each other by distances up to 100 m. The reproducibility of the analytical results are usually within 2% standard deviation.

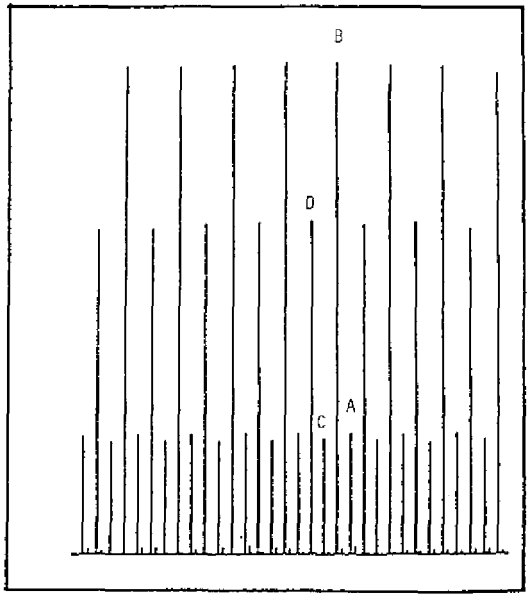

FIGURE 3 Recording Signals from DMS Monitor Connected with 4 Different Probes (A, B, C, D).

Ultratrace monitor [Bis(chloromethyl) ether monitor]

A significant drive for the need of ultratrace analytical techniques stems from occupational safety and health considerations and

requirements. However ultratrace problems are interrelated with problems of ultrahigh resolution separation techniques, and one cannot solve one problem without touching the other. The lower the concentration of a component in a sample the larger is the interference from other substances present in this sample.[3]

We have therefore developed an ultratrace monitor based on two-dimensional chromatography enabling us to penetrate into the ppb range of detection limit and beyond.

The general principle is still the same as in the simple surveillance monitor. However, we included a precolumn to trap larger samples of air. The subsequent analysis includes a fractional desorption and trapping on an adsorption/desorption trap within a first monitor system from which a narrow fraction of the separated substances is transferred to a second monitor system with a new and complete set of adsorption/desorption trap and gas chromatograph (Figure 4).

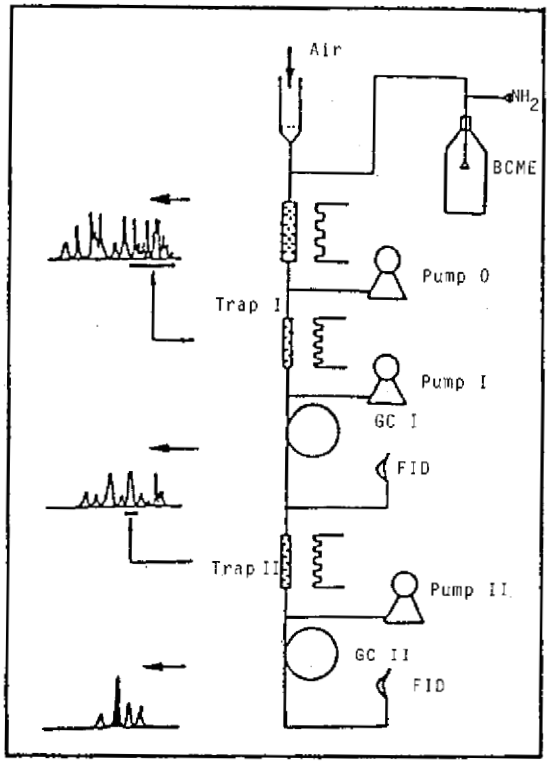

FIGURE 4 Bis(chloromethyl) Ether Monitor.

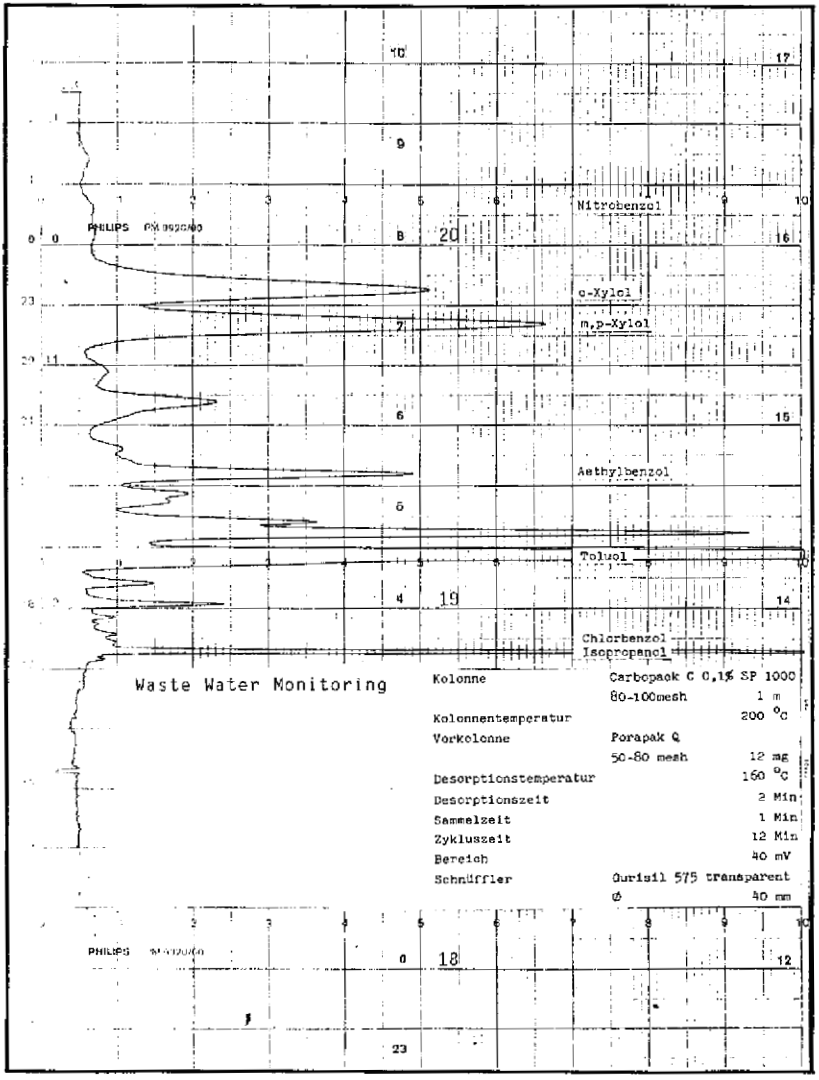

FIGURE 5 Results from a Waste Water Investigations.

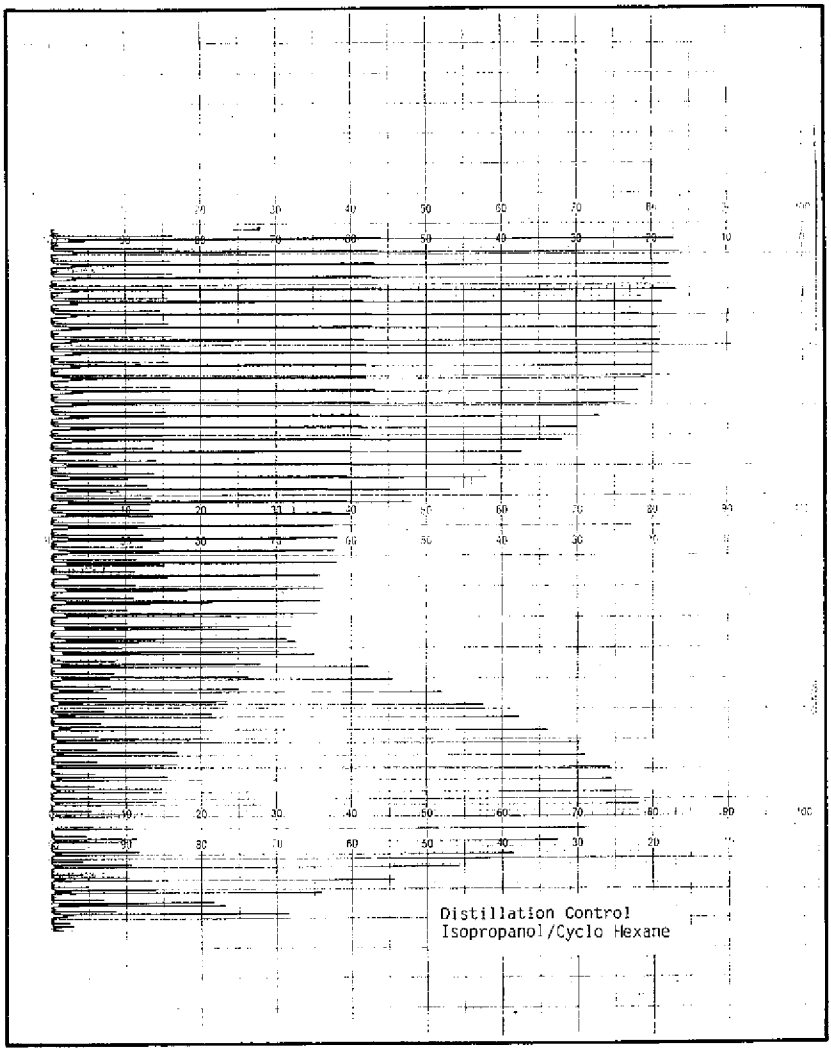

FIGURE 6 Monitor Surveillance of a Distillation Process (Isopropanol/Cyclo Hexane).

Waste water monitor

Due to their volatility most organic solvents and other substances migrate through appropriate membranes. Taking advantage of these properties we have introduced a sniffer device based on a semi-permeable membrane separator, which allows us to adapt and extend the monitor measuring principle to investigations in liquid media and vacuum systems (Figure 5).

This is an interesting aspect in the analytical approach to environmental problem solving in chemical industry and opens a wide field of analytical application.

The waste water monitor, introduced in 1982, has been designed to control and analyze more than a dozen of different volatile solvents and contaminants in water, covering a concentration range between 1 μg/g (ppm) and 100 (mg/g) (10%). It has a cycle period of about 15 minutes and is therefore an ideal instrument to permanently control and survey waste-water effluents.

The same monitor principle has been used to measure components in reaction vessels, distillation columns (Figure 6) and vacuum systems (dryers). These applications demonstrate the versatility of the monitor and its use as a true chemical sensor.

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