DOE/MC/31177-96/C0623

CONF-9510108--47

Field Portable Detection of VOCs Using a SAW/GC System

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Contract Number:

DE-AR21-94MC31177

Conference Title:

Environmental Technology Development Through Industry Partnership

Conference Location:

Morgantown, West Virginia

Conference Dates:

October 3-5, 1995

Conference Sponsor:

U.S. Department of Energy, Office of Environmental Management,
Morgantown Energy Technology Center

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Introduction

This paper describes research on a fast GC vapor analysis system which uses a new type of Surface Acoustic Wave detector technology to characterize organic contamination in soil and groundwater. The project was sponsored by the Department of Energy, Morgantown Energy Technology Center.

Project Objectives

The research objectives were to demonstrate detectability and specificity of a Surface Acoustic Wave Gas Chromatograph (SAW/GC) to a representative number of VOC materials followed by field demonstrations of the new technology at a DOE site. Field testing of the SAW/GC was performed at the DOE Savannah River Site. The performance of the SAW/GC analyzer was validated by comparing results taken with an on-site HP chromatograph. Tests were performed with water, soil and gas samples. By these tests, the SAW based analyzer's ability to identify and quantify the presence of VOCs was to be demonstrated.

Technology Description

The basic structure of a SAW/GC is shown in Figure 1. The system utilizes a two position, 6 port GC valve to switch between sampling and injection modes. In the sample position environmental air is passed through an inlet preconcentrator or water trap and then through a sample loop trap. The function of the loop trap is to concentrate VOC materials when into the sample position. During sampling helium carrier gas flows down a capillary column and impinges onto the surface of a temperature controlled SAW resonator crystal[1] as shown in Figure 2.

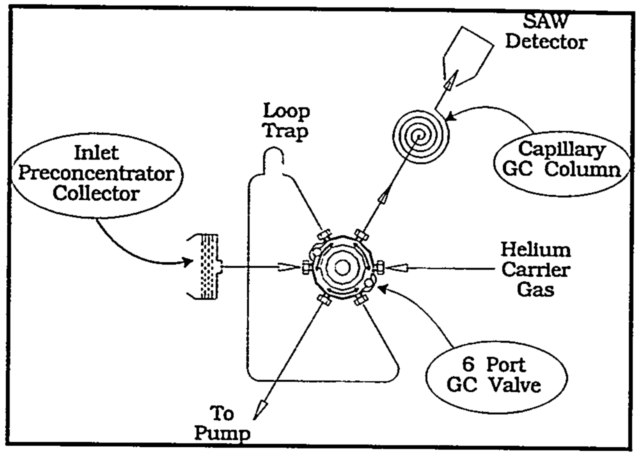

Figure 1. SAW/GC System Description

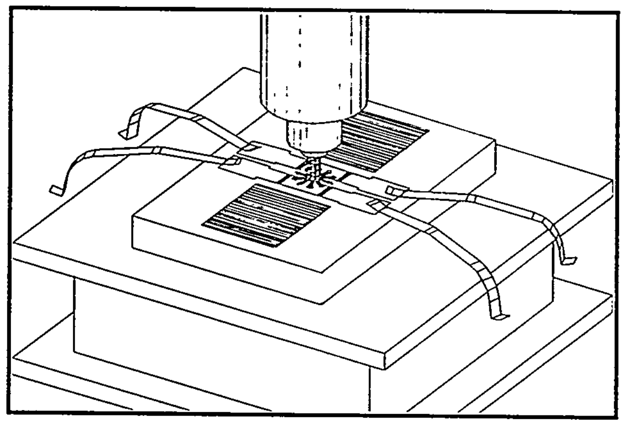

Figure 2. Surface Acoustic Wave GC Detector

[1] United States Patent No. 5,289,715, Vapor Detection Apparatus and Method Using an Acoustic Interferometer.

Research sponsored by the U.S. Dept. of Energy's Morgantown Energy Technology Center, under contract DE-AR21-94MC31177 with Amerasia Technology, Inc., 2301 Townsgate Rd., Westlake Village, CA 91361; telefax: 805-495-1550.

Switching the valve to the inject position causes helium carrier gas to flow backwards through the loop trap and onto the column. After the valve is switched into the inject position the loop trap is rapidly heated to 200°C causing the trapped VOC materials to be released into the GC column. The temperature of the GC column is linearly raised to approximately 125°C over a 5-10 second time and this causes the VOC materials to travel down the column and exit at a time characteristic of the VOC material.

The SAW resonator is a unique type of GC detector. VOC materials as they exit the GC column are trapped on the surface of the resonator and causes a change in the characteristic frequency of the crystal. The adsorption efficiency of each VOC material is a function of the crystal temperature and by operating the crystal at different temperatures the crystal can be made specific to materials based upon the materials vapor pressure. Also, since the crystal acts as a micro-balance it integrates the total amount of material present and to obtain a conventional chromatogram plot of retention time, the derivative of frequency Vs time is calculated. This is in contrast to a conventional GC detector which detects the flux and peak integral calculations are required to obtain the amount of each material present.

Results and Accomplishments

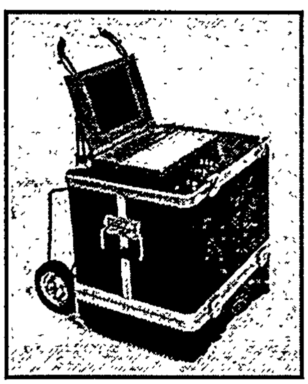

Figure 3. Portable Laboratory SAW/GC

To demonstrate the technology a portable laboratory scale instrument shown in Figure 3 was constructed and tested with the representative VOC materials listed in Table 1.

Table 1. VOC Materials Tested

Material Name	Formula
Trichloroethylene	C_2HCl_3
Tetrachloroethylene	C_2Cl_4
Carbon Tetrachloride	CCl_4
Chloroform	$ChCl_3$
Dichloromethane	CH_2Cl_2
1,2-Dichloroethane	$C_2H_4Cl_2$
1,1,1-Trichloroethane	CH_3CCl_3
1,1-Dichloroethylene	C_2H2Cl_2
1,1,2,2-Tetrachloroethane	$C_2H_2Cl_4$
Trichlorofluoromethane	CCl_3F
Benzene	C_6H_6
Toluene	C_7H_8
Gasoline	--
Diesel Fuel	--

Each material was tested with a calibrated vapor source either purchased as bottled gas or created by injection into a known volume (tedlar bag). Calibration results based upon a 10 second sample are listed in Table 2. In general the sensitivity of the instrument for all materials

Table 2. Representative Calibration Results

VOC Material	Test Concentration (ppm)	Detected Amplitude (Hz)	Detection Limit (ppm)	Scale Factor (Hz/ppm/cc)
Dichloromethane	133	678	5.88	0.45
Chloroform	37	63	17.62	0.15
1,2-Dichloroethane	45	144	9.38	0.28
Trichloroethylene	10	383	0.78	3.4
Toluene	2.4	272	0.26	10.1
Tetrachloroethylen e	1.6	517	0.09	28.7

was 1 ppm or better. For materials with lower vapor pressure, such as toluene and tetrachloroethylene, sensitivity extends well into the ppb range. To achieve ppt sensitivity it is only necessary to extended sample time. However, the advantage of a short sample times is near real time operation.

Field Test Results

To field test the laboratory prototype the instrument was transported to Savannah River where it was used to obtain real time measurements of well head gases. Although the instrument was capable of battery operation for limited periods of time, uninterrupted power was most reliably obtained from the automobile which was used to transport the system to each well head as shown in Figure 5.

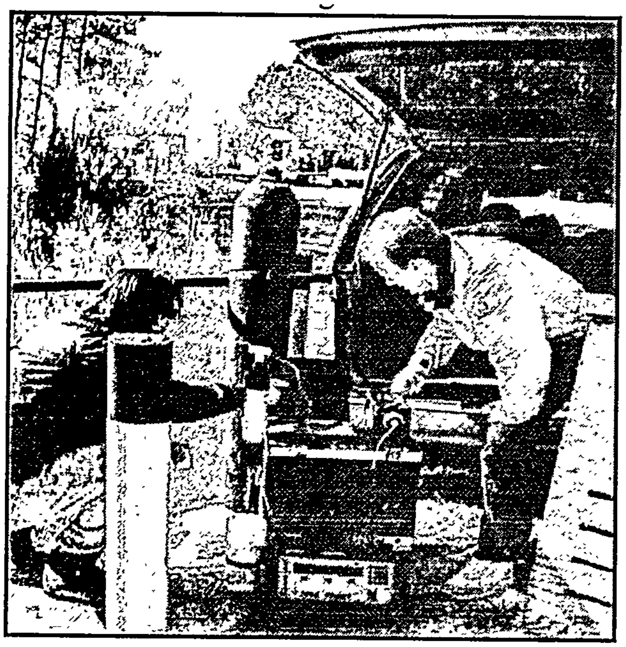

Figure 5. Field Testing SAW/GC at DOE Savannah River Site

To verify the accuracy of the instrument, calibrated tedlar bag samples were used to calibrate the SAW/GC. A typical output screen for one such bag containing approximately 100 ppm TCE and PCE is shown in Figure 4. The user interface shows two chromatograms, one is the derivative of SAW frequency and the other is SAW frequency vs time. The duration of the chromatogram is 10 seconds and retention times for TCE and PCE is 3.54 and 5.54 respectively. The operator can display quatitative information as ppm/ppb, in mass units of picograms or nanograms, or alternately in SAW units of frequency.

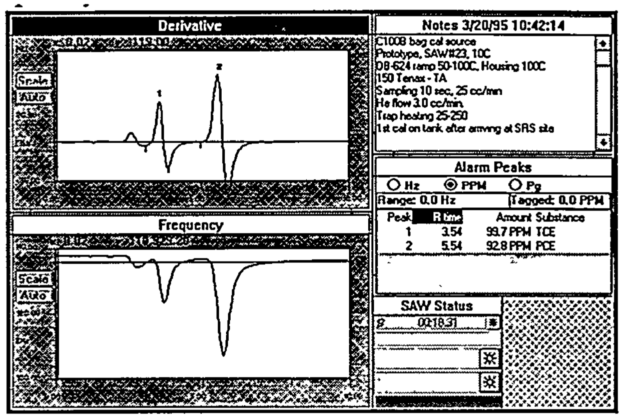

Figure 4. Typical Screen Display Showing PCE (92.8 ppm) and TCE (99.7 ppm) Tedlar Bag Calibration Results

Many different measurements were taken and compared with an on-site HP GC as shown in Figure 6. The results of this relative comparison indicate that the SAW/GC and the HP GC agree within approximately 20%. Much of the variation is attributed to variations in sampling and preconcentration within each instrument.

Application or Benefits

There are many related applications for SAW/GC technology. While at Savannah River the instrument was also used to measure catalytic converter performance, DNAPL probe experiments, and to characterize VOC break through in carbon scrubbers.

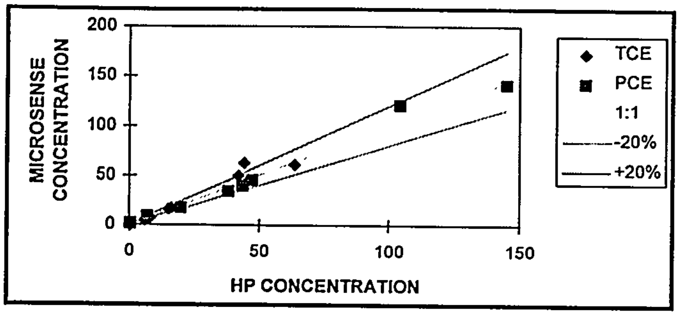

Figure 6. Cross Check of System Accuracy Using HP Laboratory Style GC

The advantages of the SAW/GC are portability, accuracy, and speed. The new SAW sensor demonstrated sufficient specificity and sensitivity to be used as a fast trace analyzer or screening tool at DOE remediation sites. Using the SAW/GC analyzer as a field screening tool, cost savings over current techniques, which require expensive laboratory testing, are estimated to be more than $50,000 per month. We conclude that the cost of the SAW/GC screening instrument will be recovered within less than one month of operation.

Future Plans

Based upon the current results the goals are to begin development of SAW/GC screening instruments for use at DOE remediation sites.

The commercialization effort is being carried out by Electronic Sensor Technology, Inc., a limited partnership company managed by Amerasia Technology and tasked with the development of SAW/GC instruments.

The commercialization effort is being aided by a partnership between Amerasia Technology, Inc., and the Morgantown Energy Technology Center. This new program will involve continued field testing at DOE sites, EPA certification and verification, and the development of new SAW/GC instruments to detect and quantify Dioxins, Furans, and PCBs at DOE sites.

Acknowledgements

We wish to thank Mr. Eddie Christy, our METC Contracting Officer, for excellent support and supervision. Work in this project was supported by the U.S. Department of Energy's Morgantown Energy Technology Center, under contract DE-AR21-94MC31177, which covered the period June 1994 to March 1995.

Also we wish to express our appreciation to Dr. Joe Rosabbi, Westinghouse Savannah River Co., for his support and encouragement during field testing at the Savannah River Site.

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