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Environmental Protection
Agency

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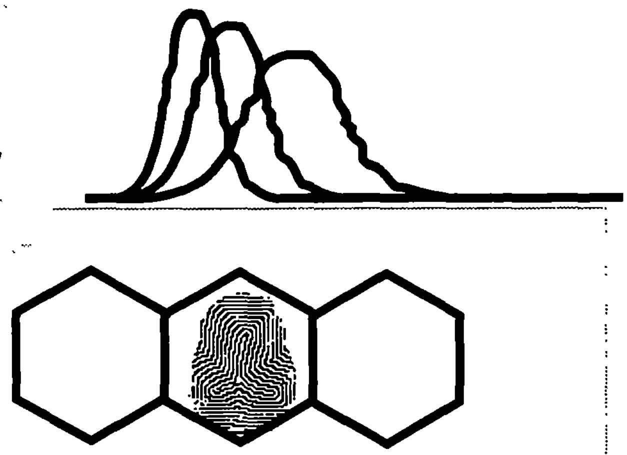
OFFICE OF RESEARCH AND DEVELOPMENT

TECHNOLOGY SUPPORT PROJECT

EPA

Field-Portable Scanning Spectrofluorometer

An EMSL-LV Innovative Technology



The Need:

Field-portable instruments are available for the qualitative and quantitative evaluation of volatile organic compounds and nonvolatile inorganic elements. Compounds that fall between these volatility extremes have received less attention in recent years. And yet these compounds comprise a surprising number of important contamination categories at Superfund and RCRA sites.

Polyaromatic hydrocarbons (PAHs) in complex mixtures such as oils, creosotes, and tars are found on numerous hazardous waste sites and, because of their high molecular weight, present special challenges to analytical chemists and instrument developers. These compounds have relatively high luminescence yields and, therefore, can be readily measured by spectrofluorometry.

A recent technology that is in the production prototype stage is the Field-Portable Scanning Spectrofluorometer (FPSS). It is a lightweight battery-operated instrument that has shown early promise as a screening device for petroleum oils, PAHs and, especially, creosotes.

Creosote (wood preservation) and coal gasification sites are widespread, especially in the southeastern United States. These are complex sites that usually have various PAHs in addition to the creosotes. These compounds are currently quantified by gas chromatography but their tarlike composition makes them difficult to detect and destructive to columns and detectors. The development of a field-portable instrument to rapidly identify and quantify PAH mixtures, such as creosotes, oils, asphalts, or coal tars is an important step in filling a field analytical niche.

The FPSS prototype is ready for field demonstration and comparative studies. It is anticipated that the FPSS will provide a more rugged and less expensive alternative to traditional methods for screening PAHs.

The Use:

Scientists working at the Environmental Monitoring Systems Laboratory-Las Vegas have performed laboratory evaluations of the battery-operated FPSS developed by T. Vo-Dinh and his co-workers at Oak Ridge National Laboratory.[1,2,3] Table 1 shows the physical characteristics of the instrument.

Table 1. Physical Characteristics of the Field-Portable Scanning Spectrofluorometer

	SIZE	WEIGHT
Instrument	48 x 40 x 21 cm (18.5 x 11.5 x 8")	11.5 kg
Battery Pack	31 x 18 x 15 cm (12 x 7 x 6")	11.0 kg

The FPSS can perform emission and synchronous wavelength scans. In the emission mode, relatively low detection limits are achieved (Table 2). The emission mode is useful for the determination of total PAHs or in identifying and classifying oils. In the synchronous mode both the excitation and emission monochromators are scanned simultaneously with a constant wavelength offset. The advantage to synchronous mode is that it separates spectra of compounds with a different number of fused rings, sharpens spectra, and allows the relative amount of various PAH classes to be quantified.

The FPSS consists of three parts: a small suitcase-sized instrument that houses the optics and electronics, a battery pack, and a lap top computer used for instrument control, data storage and analysis. The spectral coverage of the instrument is 210-650 nm. The instrument parameters are chosen by the operator who uses the computer to control the instrument.

The FPSS can be operated two ways: using a standard fluorescence cuvette cell or a bifurcated optical fiber. The optical fiber attachment is 2-meter long and allows direct screening of water samples. The cuvette can be used with liquid samples or extracts of soils. When the optical fiber attachment is used, care must be taken to avoid interference from light. This can be done by covering the sampling area with a black cloth.

Table 2. Limit of Detection (S/N = 3)

	SYNCHRONOUS (cuvette)	EMISSION (cuvette)	SYNCHRONOUS (fiber)
Perkin Elmer LS50 (laboratory instrument)	0.17*	0.02	24
FPSS prototype	3.5	0.55	1

** All concentrations ng/mL of anthracene*

The Limits:

Some areas of concern exist relative to the successful operation of the FPSS in a field situation. The ruggedness of the optical components is crucial to the in situ applicability of the system. The unit was shipped from Oak Ridge National Laboratory to the EMSL-LV without affecting the optical alignment or electronics. The instrument has been demonstrated to withstand normal handling in the laboratory. The instrument is

(continued on next page)

The Limits: (continued)

ready to be demonstrated at a hazardous waste site.

The FPSS is particularly suited to the classification or identification of oils or PAH compounds. It can also be used with site-specific standards to quantify total oils or PAHs. It can be used to determine relative amounts of the PAH classes present. In rare instances, like spills of solvents or PAHs with very high fluorescent yields and sharp structures such as benzo-(a)pyrene, it can be used to detect and quantify identified PAHs. There is greater spectral separation capability when the instrument is operated in synchronous mode but lower detection limits can be achieved using the emission mode.

The Status:

Laboratory evaluations and research efforts have resulted in a draft fluorescence method for the analysis of PAHs which is in the final stages of acceptance by the American Society for Testing and Materials. A comparison of the optical fiber mode and the standard cuvette mode was performed on samples of anthracene in methanol. This study showed the cuvette mode to be 2-3 times more sensitive than the optical fiber mode.

Synchronous luminescence has been demonstrated to be useful in characterizing crude and fuel oils.[4] The technique can be used to produce spectral fingerprints for the identification of oil contamination types and sources. The FPSS proved its ability in a study comparing samples from an oil spill with samples of the source oil which were provided by the U.S. Coast Guard.

The FPSS has shown considerable promise for the classification and quantitation of PAH compounds and oily mixtures. The next step is to take the portable instrument to a hazardous waste site where it can be evaluated against standard methods in a well-planned experimental design. The development of the FPSS was sponsored by the EMSL-LV and commercialization is being planned.

References:

[1] T.Vo-Dinh, "Synchronous Excitation Spectroscopy" in Modern Fluorescence Spectroscopy, Vol. 4, Edited E.L. Wehry, Plenum Press, New York, 1981, pp. 167-192.

[2] T. Vo-Dinh, "Synchronous Luminescence Spectroscopy: Methodology and Applicability", Applied Spectroscopy, Vol. 36, 576, 1982.

[3] J.P. Alarie, Vo-Dinh, T., Miller, G., M.N. Ericson, S.R. Maddox, W. Watts, D. Eastwood, R.L. Lidberg, and M. Dominguez, "Development of a Battery-Operated Portable Synchronous Luminescence Spectrofluorometer", in press, (Review of Scientific Instruments).

[4] K.J. Siddiqui, Lidberg, R.L., Eastwood, D., and Gibson, G., "Expert Systems for Classification and Identification of Waterborne Petroleum Oils", *Monitoring Water in the 1990s, Meeting New Challenges, ASTM STP 1102,* J.R. Hall and G.D. Glysson, Editors, American Society for Testing and Materials, Philadelphia, 1991.

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