



Project Summary

Multispectral Identification and Confirmation of Organic Compounds in Wastewater Extracts

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Application of multispectral identification techniques to samples from industrial and POTW wastewaters revealed identities of 63 compounds that had not been identified by empirical matching of mass spectra with spectral libraries. Twenty-five of the compounds have not been found in wastewaters previously. Multispectral techniques used in the identifications were GC/MS (low resolution), high resolution mass spectrometry (HRMS), positive methane chemical ionization mass spectrometry (CIMS), and gas chromatography combined with Fourier transform infrared spectroscopy (GC/FT-IR).

Some of the same techniques were used to confirm (without obtaining a pure sample of the tentatively identified compound) identifications made by reexamination of GC/MS data tapes generated by contractor laboratories. Eighty percent of the identifications were confirmed. Some of the wrong identifications made by reexamination of tapes were attributed to poor chromatography during the initial analysis.

This Project Summary was developed by EPA's Environmental Research Laboratory, Athens, GA, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Background

Gas chromatography/mass spectrometry (GC/MS) was determined in the

early 1970s to be the method of choice for analysis of organic pollutants in water. In support of the 1976 Consent Decree between the U.S. Environmental Protection Agency (EPA) and the National Resources Defense Council, magnetic tapes of GC/MS analyses of samples acquired by contract laboratories for the Industrial Technology Division (ITD) of EPA's Office of Water were processed through a suite of computer programs designed to identify both priority and non-priority pollutants. The programs originally had been developed for minicomputers and had been applied in the late 1970s and early 1980s to data acquired in the late 1970s. These programs were converted in 1986 and 1987 to operate on the Athens Environmental Research Laboratory's VAX-785 computer and were applied to data acquired by ITD contract laboratories during the early 1980s. Tentative identifications were made by computerized spectra matching from the input data, using historical GC relative retention times and a collection of 110,000 reference mass spectra.

Few identifications of non-priority pollutants or confirmations of tentative computer identifications had been made from the 1970s data. This was attributed both to the use of packed GC columns and to the limited size of the reference database (34,000 mass spectra). In the most recent tape study, the database was nearly three times the size of the one used earlier, and the GC/MS data had been collected using capillary GC column and protocols similar to those of EPA Methods 625 and 1625. As had been anticipated, the number of sample components recognized by the computer

increased as a result of the use of capillary columns. Not anticipated was the fact that the programs designed to select the best spectrum for packed GC columns were less effective in selecting the best spectrum from a capillary GC column. In particular, it was determined that when the peak recognition algorithm was tuned to recognize trace level chromatographic peaks, it was quite apt to ignore high level peaks due to the width of the GC peak.

Sample and Compound Selection

To check the accuracy of the tentative identifications, as well as to determine the identity of compounds not identified by the tape programs, compounds from GC/MS runs of 19 publicly owned treatment works and of 13 organic

chemical plants were selected as "targets" for our reanalysis.

The analytical targets were organized by sample source into five groups. As a result, it is possible that a given target might occur in a publicly owned treatment works sample, but be a confirmation or identification target only in a specific organic chemical plant study. The majority of the peaks in most chromatograms are due to the isotopically enriched compounds that were used for mass spectrometric quantitation of specific analytes by isotope dilution.

New GC/MS chromatograms and, where appropriate, GC/Fourier Transform Infrared Spectroscopy (GC/FT-IR) chromatograms were obtained for retained extracts, and were compared with one another and with the corresponding chromatograms from the

contract laboratory data. Based on these comparisons, sample similarities were recognized that permitted choosing 11 representative sample extracts for the confirmations and identifications. Multispectral analysis of these extracts, consisting of GC separation followed by appropriate combinations of low and high resolution electron impact or chemical ionization mass spectrometry and FT-IR, resulted in 48 confirmations, 63 identifications, and recognition of 12 common compounds that should have been identified in the tape study. The number of compounds identified by the tape study could have been increased by the following: a strict adherence to the 1625 GC protocol, a reduction in the number of internal standards added to the samples, and an improvement in the peak recognition algorithm used by the tape study.

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The complete report, entitled "Multispectral Identification and Confirmation of Organic Compounds in Wastewater Extracts," (Order No. PB-90 160 995/AS; Cost: \$31.00, subject to change) will be available only from:

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