



Special Analytical Services

Introduction

The Environmental Sciences Division (ESD) in Las Vegas has an excellent background in the preparation and analysis of non-typical samples that require special care, in-depth knowledge, and high-tech instrumentation. The EPA Regions are welcome to submit special samples to the ESD through the Technology Support Center.

Representative sampling and subsampling present challenges to field and laboratory personnel. The ESD has experience and expertise in the handling of complex and heterogeneous matrices and in the interpretation of results from non-routine samples.

The following examples illustrate the wide range of capabilities and analytical services available through the ESD.

Ion Composition Elucidation (ICE)

ESD scientists have developed a gas chromatography/high resolution mass spectrometric (GC/HRMS) technique to determine the numbers of atoms of each element that comprise the ions in a mass spectrum.

This information provides confirmatory evidence when a single good mass spectral library match is found and often selects the correct library match when there are multiple matches. When no library matches are found, a compound's identity can be limited to a small number of isomers for which procurement of standards for identification based on similar retention times and mass spectra are practical.

Region 3

Site Characterization

A tar-like sample from a Superfund site in West Virginia was dissolved in methylene chloride and injected onto the GC column. The compositions of 51 apparent molecular ions were determined using Ion Composition Elucidation (ICE). The compounds were mostly benzothiazoles and related compounds. Benzothiazoles are used in the dye and rubber industries. The source of the compounds was indeed a nearby dye plant. See <http://www.epa.gov/nerlesd1/chemistry/ice/hazpost.htm>.

Region 2

Compound Identification

An increased incidence of childhood cancer in Toms River, NJ, made identification of compounds from a municipal well crucial. The compositions of the molecular ion and 10 fragment ions were determined using ICE, which limited the number of possible compounds to a manageable number. With this information in hand, the corporation with an interest in our study supplied a standard from a polymerization process that contained 3 of the 5 isomers observed. Toxicology studies on these compounds were then made under Region 2's supervision. See <http://www.epa.gov/nerlesd1/chemistry/ice/nj.htm>.

Region 9

Disinfection Byproduct

A brominated disinfection byproduct provided only the molecular ion and one fragment ion discernable above the chemical noise in its mass spectrum.

Their compositions were determined using ICE. The composition of the non-brominated compound was consistent with Quinbolone, an anabolic steroid possibly used to stimulate cattle growth in a nearby feed lot. Further experiments by the Region would be necessary to confirm this hypothesis. See <http://www.epa.gov/nerlesd1/chemistry/ice/48asms.htm>.

Region 4

Risk Assessment

Malodorous monitoring well samples from the Tower Chemical site near Orlando, FL, contained numerous compounds. ICE was used to determine the compositions of the apparent molecular ions. Confirmation for library matches was provided and semi-quantitative estimates of the relative amounts of the compounds were listed. A group of aromatic compounds containing Cl atoms was consistent with past pesticide manufacture, while a group of sulfur containing compounds was probably produced by anaerobic bacteria. The presence of compounds containing both Cl and S atoms suggested biotransformation of the pesticide-related compounds. Region 4 used this information in their risk assessments for the site.

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Groundwater Tracers - Tennessee Products

Groundwater migration is typically determined using injection and sampling wells with fluorescent dye tracers as the indicator compounds that are injected and then detected along a migration path. These tracers are determined in samples using synchronous scanning spectrofluorimetry. No separation is performed resulting in less specificity, possible false positives, and the presence of limiting matrix background signal. We have developed capillary electrophoresis/laser-induced fluorescence for the determination of tracers in samples using very simple separation conditions. The selectivity of the separation together with the selective detection provides a result that is more specific for regulatory purposes. This approach may be useful when background or other conditions require a more specific determination. Past work addressed fluorescein and tinopal dyes (488 nm and 354 nm excitations) at the Tennessee Products Superfund site in conjunction with spectrofluorimetry. Present work addresses eosin and rhodamine WT (514 nm and 528 nm excitations) using an Ar/Kr mixed gas laser as the excitation source.

Innovative Methods

The ESD maintains the instrumentation and personnel necessary to perform innovative analysis of difficult and unusual samples. Teaming state-of-the-art equipment with highly specialized, multidisciplinary technical staff enables the Laboratory to provide high quality service to the EPA Regions. The staff at ESD is keeping current with the analytical demands of an increasingly complex environment.

References

Brumley, W. C., Ferguson, P. L., Grange, A. H., Donnelly, J. L., and Farley, J. W., Applications of capillary electrophoresis/laser-induced fluorescence to groundwater migration studies, J. Cap. Elec. 003 (6), 295-299.

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