Accuracy in Trace Analysis

During the development of this method at NBS, the factors investigated included: composition of the eluents, interference due to impurities in the delivery liquid and the possible means of eliminating them with a laboratory-packed trap column, and a comparison of suppression efficiencies between two types of suppressors. This method was used for the determination of Mg$^{2+}$ and Ca$^{2+}$ in SRM 2694, Simulated Rainwater, with precisions and accuracies better than 2% relative [5].

The frontier for ion chromatography is transition metal analysis with ongoing research along many fronts. New resins are being introduced and are being coupled to exotic detectors. The era of "hyphenated" techniques is burgeoning with ion chromatography showing up as the front-end separation method of choice. Recent work at NBS has coupled ion chromatography with direct current plasma emission spectrometry for the determination of phosphorus in copper-based alloys [6].

Future challenges for ion chromatography will be to enhance further its capabilities through improved speed, sensitivity, and resolution. Speciation of metals as a function of oxidation state and complexation will be an important goal. Innovative resins and a new generation of detectors will have to be developed. Fundamental research into the retention mechanisms leading to the separation will be essential.

In conclusion, ion chromatography has proven itself an invaluable tool to analytical chemistry. Its ultimate advantages will lie in its versatility and its capacity for ultra-trace analyses with minimal contamination and total automation.

Quantification of Toxic Chemicals in Selected Human Populations

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The evaluation of risk posed by toxic chemicals to human populations requires a knowledge of the toxicity of compounds and the extent of human exposure to the compounds. The best estimate of exposure is obtained by measuring chemical residues or metabolites in biological samples and extrapolating the measurements to body burdens in study populations. Analytical methods developed at the Centers for Disease Control (CDC) typify the laboratory techniques needed to support epidemiologic studies. In these procedures, specific sample preparation techniques are used to isolate the target compounds, which are then measured by capillary gas chromatography combined with mass spectrometry. Many problems must be solved for these techniques to be applied successfully. Quality control materials must be generated that closely mimic actual specimens and that contain target analytes at appropriate concentrations. Sample contamination, both from internal and external sources, can be a major difficulty. The lack of analytical standards is usually a continuing problem for the analytical chemist, and a successful synthesis program can minimize this problem. Interpreting analytical results at or near the system's limit of detection poses more problems. Techniques must be developed to translate sample concentrations into valid estimates of total body-burden values.

A recent study in which chlorinated phenols and phenoxy acid herbicides in urine were measured exemplifies typical laboratory challenges in trace analysis. In this study, we compared children living near a chemical manufacturing site with a control group of unexposed children. An extensive sample preparation included acid hydrolysis, extraction with benzene, derivatization with diazooethane and column chromatography cleanup. The more volatile compounds are quantified by capillary column
Accuracy in Trace Analysis

gas chromatography/positive chemical ionization/mass spectrometry/mass spectrometry. Less volatile compounds are quantified by using electron capture negative chemical ionization in a single stage mass spectrometry mode. This study required a modified approach to quality control material in that target values were obtained during the analysis of actual specimens. In addition, we have used a multivariate quality control procedure to obtain a single quality control chart that is representative of the 12 study analytes. The synthesis of the pure derivatives provided an ideal material in estimating recovery and diagnosing chromatographic problems. With the method's improved detection limits and specificity, we had an increased frequency of detection for several of the study analytes compared with frequencies for previous studies.

Chemists have encountered significant analytical challenges in measuring 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD) in tissue at the parts per trillion (ppt) level and measuring TCDD in serum at the parts per quadrillion (pqq) level. Such low levels of quantification can only be achieved by using high resolution mass spectrometry. The labor-intensive sample preparation activities have led to the application of automated procedures directed toward increasing sample throughput and optimizing use of the chemist's time. Such sample preparation requires a specialized approach to quality control, including the strategic placement of system blanks. Such low levels of quantitation require care in controlling artifacts and laboratory contamination. The reporting of quantitative results corrected for percent lipid in the samples permits the correlation of quantitative results between different sample matrices. The laboratory must be carefully organized to insure sample coordination, monitoring of quality control, and timely reporting of results. Finally, sample collection in the field has been challenging, particularly in the case of adipose tissue.

Interest in toxic chemical exposure will continue to challenge the analytical chemist to provide better laboratory measurements for assessing exposure. We are developing a method for quantifying a number of volatile organic compounds in human whole blood. Concerns about exposure to this class of compounds has led to controls and monitoring programs on drinking water systems, a major source of human exposure. Our method development has defined the analytical approach as a variation on the traditional purge and trap technique. Work includes adapting instrumental hardware specifically for this sample matrix. Major challenges include a short-lived quality control material, quality control for a significant number of "non-detects" and electronic data handling for the large number of samples involved. The overall objective of this work is to estimate the presence of a number of important volatile compounds in whole blood for a specific population of 1200 people. The results of this study should make it possible to estimate the types and magnitude of exposure encountered by the U.S. population.

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An Evaluation of Jansson's Method to Deconvolve Overlapped Gas Chromatographic Peaks

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Introduction

It has been reported [1,2] that Jansson's Method [3] can be used to deconvolve severely overlapped gas chromatographic peaks. Initial testing [1,2] indicates the method will give improved performance over conventional graphical peak resolution techniques such as perpendicular drop and shoulder quantitation [4,5].

Jansson's Method is an iterative non-linear algorithm that uses the prior knowledge of peak non-negativity and maximum peak height for an improved estimate of the true chromatogram [3]. The method only requires a knowledge of the instrument's impulse response function and maximum peak height. Jansson's Method does not require any prior information on how many peaks are overlapped.