C28 Analysis of Volatile Organic Compounds in Soil - Revisited

Carol A. Erikson, MSPH, Trillium, Inc., 356 Farragut Crossing Drive, Knoxville, TN*

The goal of this presentation is to consider, once again, the accuracy of current sample collection and analysis methods for volatile organic compounds. Results for actual site samples recently collected and analyzed by EPA-approved low level and EPA-approved high-level procedures will be compared, and the implications of these comparisons will be discussed.

The fact that volatile organic compounds (VOCs) are rapidly lost from moist soil and sediment during sample handling procedures (collection, transport, preparation, and analysis) has been well established. Early suggestions that all soil and sediment samples be preserved in methanol at the time of collection were not readily accepted due to concerns about the use and transport of a hazardous solvent in and from the field as well as a perceived loss of sensitivity because higher detection limits (DLs) are typically reported from this approach.

Much research has been done in search of a procedure that minimizes target analyte losses and achieves acceptably low DLs. To-date, few would argue that methanol extraction produces the most consistently accurate results of all evaluated approaches.

With the promulgation of Method 5035 in June of 1997, EPA banished the traditional soil sample collection method and replaced it with a closed-system purge and trap method, which includes several presumably equivalent options. Low concentration soils are collected by placing an aliquot into a septum-sealed screw-cap vial that already contains a sodium bisulfate solution and a stir bar and which is subsequently analyzed without ever being opened. High concentration soils may be methanol-preserved in the field by adding a sample aliquot to a similar vial that contains methanol. As an alternative (and only when necessary), both low-level and high-level soil samples may be transferred unpreserved to the laboratory for further processing. In this case, EPA recommends that appropriate sampling devices be used and that analyses be performed within 48 hours.

REDACTED TABLES

Appropriate sampling devices include, among others, Encores™, which are sized to allow collection of 5-gram or 25-gram soil aliquots with minimal sample disturbance. The Encores can then be sealed for transfer to the laboratory for subsequent preparation and analysis.

In a recent site investigation, three Encores were collected from each soil sample location. Within 48 hours of collection, the aliquots from two of the Encores were preserved in sodium bisulfate for low-level analysis, and the third was preserved in methanol. When high target analyte concentrations were found in the low-level analysis, the methanol extract was run as a “high-level” analysis. In effect, the methanol-preserved sample was viewed as a dilution of the low-level analysis.

For all of the soil samples collected at this particular site and analyzed by both methods, results for all analytes that were within the established calibration range (i.e., that were quantitatively valid) in both the low-level and the high-level analyses were compared (see Table 1 and Figures 1 and 2). Forty-seven paired results, including both chlorinated and nonchlorinated analytes, were available for this evaluation. In every case, the concentration detected in the methanol extract was higher than the concentration found in the low-level purge analysis. Ratios of the high-level analysis results to the low-level analysis results ranged from 2.2 to 170; on average, the high-level results were higher than the low-level results by a factor of 23 - more than an order of magnitude.

Based on these data, it is obvious that current low-level volatiles collection and analysis methods still do not accurately represent environmental field conditions, even when they are conscientiously followed and the quality control results are acceptable.

The implications are crucial. Accurate analytical results from a site investigation are necessary in order to allow the design and implementation of a successful remedial action approach. Accurate analytical results are crucial in order to monitor the progress of a remedial action and to determine when a stopping point has been reached. And, accurate analytical data are absolutely essential when cost allocations for cleanup must be determined.

Volatile Organics, Soil

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