THE ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM

ETV

U.S. Environmental Protection Agency

Environmental Security Technology Certification Program

Oak Ridge National Laboratory

Joint Verification Statement

TECHNOLOGY TYPE:	**EXPLOSIVES DETECTION**
APPLICATION:	**MEASUREMENT OF EXPLOSIVES IN CONTAMINATED SOIL AND WATER**
TECHNOLOGY NAME:	**GC-IONSCAN™**
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The U.S. Environmental Protection Agency (EPA) has created the Environmental Technology Verification Program (ETV) to facilitate the deployment of innovative or improved environmental technologies through performance verification and dissemination of information. The goal of the ETV Program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. ETV seeks to achieve this goal by providing high quality, peer reviewed data on technology performance to those involved in the design, distribution, financing, permitting, purchase, and use of environmental technologies.

ETV works in partnership with recognized standards and testing organizations, stakeholder groups consisting of regulators, buyers, and vendor organizations, with the full participation of individual technology developers. The program evaluates the performance of innovative technologies by developing test plans that are responsive to the needs of stakeholders, conducting field or laboratory tests (as appropriate), collecting and analyzing data, and preparing peer-reviewed reports. All evaluations are conducted in accordance with rigorous quality assurance protocols to ensure that data of known and adequate quality are generated and that the results are defensible.

The Department of Defense (DoD) has a similar verification program known as the Environmental Security Technology Certification Program (ESTCP). The purpose of ESTCP is to demonstrate and validate the most promising innovative technologies that target DoD's most urgent environmental needs and are projected to pay back the investment within 5 years through cost savings and improved efficiencies. ESTCP demonstrations are typically conducted under operational field conditions at DoD facilities. The demonstrations are intended to generate supporting cost and performance data for acceptance or validation of the technology. The goal is to transition mature environmental science and technology projects through the demonstration/ validation phase, enabling promising technologies to receive regulatory and end user acceptance in order to be fielded and commercialized more rapidly.

The Oak Ridge National Laboratory (ORNL) is one of the verification organizations operating under the Site Characterization and Monitoring Technologies (SCMT) program. SCMT, which is administered by EPA's National Exposure Research Laboratory, is one of 12 technology areas under ETV. In this demonstration, ORNL evaluated the performance of explosives detection technologies. This verification statement provides a summary of the test results for Barringer Instruments' GC-IONSCAN™. This verification was conducted jointly with the Department of Defense's (DoD's) Environmental Security Technology Certification Program (ESTCP).

DEMONSTRATION DESCRIPTION

This demonstration was designed to evaluate technologies that detect and measure explosives in soil and water. The demonstration was conducted at ORNL in Oak Ridge, Tennessee, from August 23 through September 1, 1999. Spiked samples of known concentration were used to assess the accuracy of the technology. Environmentally contaminated soil samples, collected from DoD sites in California, Louisiana, Iowa, and Tennessee and ranging in concentration from 0 to approximately 90,000 mg/kg, were used to assess several performance characteristics. Explosives-contaminated water samples from Tennessee, Oregon, and Louisiana with concentrations ranging from 0 to 25,000 μg/L also were analyzed. The primary constituents in the samples were 2,4,6-trinitrotoluene (TNT); isomeric dinitrotoluene (DNT), including both 2,4-dinitrotoluene and 2,6-dinitrotoluene; hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX); and octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX). The results of the soil and water analyses conducted under field conditions by the GC-IONSCAN were compared with results from reference laboratory analyses of homogenous replicate samples determined using EPA SW-846 Method 8330. Details of the demonstration, including a data summary and discussion of results, may be found in the report entitled *Environmental Technology Verification Report: Explosives Detection Technology — Barringer Instruments, GC-IONSCAN™*, EPA 600-R-00/046.

TECHNOLOGY DESCRIPTION

The GC-IONSCAN is a fully transportable field-screening instrument that combines the rapid analysis time of ion mobility spectrometry (IMS) with the separation ability of gas chromatography (GC). The instrument can be operated in IONSCAN mode or in GC-IONSCAN mode to detect explosives. The user can switch between the two modes in less than 30 s through the instrument control panel. In the IONSCAN mode, samples are deposited on a Teflon filter and thermally desorbed directly to the IMS, permitting the quick screening analysis of explosives residues in 6 to 8 s. In the GC-IONSCAN mode, extracts are directly injected onto the GC column and analysis occurs within 1 to 3 minutes, depending on the type of explosive. The use of the IONSCAN mode permits rapid prescreening of samples with identification of the major constituents of the sample and semiquantitative analysis, while the GC-IONSCAN mode permits full characterization and quantitative analysis of the sample. This technology is capable of reporting quantitative data for all of the Method 8330 analytes. The performance assessment described here is only for TNT and RDX because a limited amount of data was available for evaluation of the other analytes. Reporting limits for the GC-IONSCAN ranged from 0.3 to 10 mg/kg for soil and 25 to 1950 μg/L for water.

VERIFICATION OF PERFORMANCE

The following performance characteristics of the GC-IONSCAN were observed:

Precision: For the soil samples, the mean relative standard deviations (RSDs) for RDX and TNT were 54% and 51%, respectively. For water samples, the RSDs were significantly lower, at 20% and 26%, respectively.

Accuracy: For the soil samples, the median percent recoveries for RDX and TNT were 55% and 136%, respectively. The results were generally biased low for RDX and biased high for TNT. For water samples, only a few of the RDX and TNT results were reported above the reporting limits in the spiked samples. The recoveries were significantly lower, with the highest recovery at 46%, indicating that the water results were biased low for both analytes.

False positive/false negative results: Of the 20 blank soils, Barringer reported RDX in one sample (5% false positives) and TNT in five samples (25% false positives). No false positives were reported for RDX and TNT in the 20 blank water samples. False positive and false negative results were also determined by comparing the GC-IONSCAN result to the reference laboratory result for the environmental and spiked samples (e.g., whether the GC-IONSCAN reports a result as a nondetect that the reference laboratory reported as a detection, and vice versa). For the soils, 3% of the RDX results and none of the TNT results were reported as false positives relative to the reference laboratory results. Significantly more samples were reported as nondetects by Barringer (i.e., false negatives) when the laboratory reported a detection (2% for RDX and 13% for TNT). Similar results were observed for water, where 2% of the TNT results and none of the RDX results were false positives, and a higher percentage (39% of the RDX results and 21% of the TNT results) were false negatives.

Completeness: The GC-IONSCAN generated results for all 108 soil samples and all 176 water samples, for a completeness of 100%.

Comparability: A one-to-one sample comparison of the GC-IONSCAN results and the reference laboratory results was performed for all samples (spiked and environmental) that were reported as detects. The correlation coefficient (r) for the comparison of the entire soil data set for TNT was 0.88 (slope (m) = 4.82). When comparability was assessed for specific concentration ranges, the r value did not change dramatically for TNT, ranging from 0.71 to 0.85 depending on the concentrations selected. RDX correlation with the reference laboratory for soil was similar (r values near 0.80), except for concentrations greater than 1,000 mg/kg, where the correlation was lower ($r = 0.28$, $m = 0.14$). For the water samples, comparability with the reference laboratory results for TNT was much lower than the soil comparison ($r = 0.53$). For RDX, the correlation was much higher, at 0.95. Although the correlation was high, the slope of the linear regression line was 0.08, indicating that the GC-IONSCAN RDX results were biased low (see Accuracy).

Sample Throughput: Throughput was approximately three samples per hour for soil and eight samples per hour for water. This rate was accomplished by two operators and included sample preparation and analysis.

Ease of Use: Users unfamiliar with ion mobility spectrometry would require approximately two days of training to operate the GC-IONSCAN. Training is provided by Barringer Instruments. No particular level of educational training is required for the operator, but knowledge of chromatographic techniques would be advantageous.

Overall Evaluation: The overall performance of the GC-IONSCAN for the analysis of RDX and TNT was characterized as precise and biased low (both analytes) for water analyses, and imprecise and biased (low for

RDX and high for TNT) for soil analyses. As with any technology selection, the user must determine if this technology is appropriate for the application and the project's data quality objectives. For more information on this and other verified technologies, visit the ETV web site at *http://www.epa.gov/etv*.

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