

US EPA ARCHIVE DOCUMENT

# THE ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM



U.S. Environmental Protection Agency



Environmental Security  
Technology Certification  
Program



Oak Ridge National Laboratory

## Joint Verification Statement

<b>TECHNOLOGY TYPE:</b>	<b>GAS CHROMATOGRAPHY</b>	
<b>APPLICATION:</b>	<b>MEASUREMENT OF EXPLOSIVES IN CONTAMINATED SOIL</b>	
<b>TECHNOLOGY NAME:</b>	<b>Model 8610C Gas Chromatograph/Thermionic Ionization Detection</b>	
<b>COMPANY:</b>	<b>SRI Instruments</b>	
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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 field tested 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 verification test, ORNL evaluated the performance of explosives detection technologies. This verification statement provides a summary of the test results for SRI Instruments' Model 8610C gas chromatograph with thermionic ionization detection (GC/TID). This verification was conducted jointly with DoD's ESTCP.

#### **VERIFICATION TEST DESCRIPTION**

This verification test was designed to evaluate technologies that detect and measure explosives in soil. The test was conducted at ORNL in Oak Ridge, Tennessee, from August 21 through 30, 2000. 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. 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 analyses conducted under field conditions by the GC/TID were compared with results from reference laboratory analyses of homogenous replicate samples analyzed using EPA SW-846 Method 8330. Details of the verification, including a data summary and discussion of results, may be found in the report entitled *Environmental Technology Verification Report: Explosives Detection Technology—SRI Instruments, GC/TID*, EPA/600/R-01/065.

#### **TECHNOLOGY DESCRIPTION**

The SRI Model 8610C gas chromatograph (GC) is a transportable instrument that can provide on-site analysis of soils for explosives. Coupling this transportable gas chromatograph with a thermionic ionization detector (TID) allows for the determination of explosives in soil matrices following simple sample preparation procedures. Samples are extracted in acetone, diluted, and injected directly onto the GC column within a heated injection port. The high temperature of the injection port instantaneously vaporizes the solvent extract and explosives, allowing them to travel as a vapor through the GC column in the presence of the nitrogen carrier gas. The stationary phase of the GC column and the programmable oven temperature separate the components present in sample extracts based on their relative affinities and vapor pressures. Upon elution from the column's end, compounds containing nitro groups are ionized on the surface of the thermionic bead, and the increased conductivity of atmosphere within the heated detector is measured with a collector electrode. In this verification test, the instrument was verified for its ability to detect and quantify 2,4-dinitrotoluene (2,4-DNT), RDX, and TNT. Analytical run times were typically less than 7 min and reporting limits were typically 0.5 mg/kg.

## VERIFICATION OF PERFORMANCE

The following performance characteristics of SRI's GC/TID were observed.

**Precision:** The mean relative standard deviations (RSDs) for 2,4-DNT, RDX, and TNT were 15%, 14% and 23%, respectively, indicating that the determinations of all analytes were precise.

**Accuracy:** Accuracy was assessed using the performance evaluation (PE) soil samples, which were spiked to nominal TNT and RDX concentrations of 0, 10, 50, 100, 250, and 500 mg/kg each by an independent laboratory. The mean percent recoveries for RDX and TNT were 91% and 97%, respectively, indicating that the analyses were unbiased.

**False positive/false negative results:** Of the 20 blank soils, SRI reported TNT in five samples (25% false positives). No false positives were reported for 2,4-DNT and RDX. False positive and false negative results were also estimated by comparing the GC/TID result to the reference laboratory result for the environmental and spiked samples (e.g., whether SRI reported a result as a nondetect that the reference laboratory reported as a detection, and vice versa). For these soils, 3% of the 2,4-DNT results and 7% of the TNT results were reported as false positives relative to the reference laboratory results, but none of the RDX results were reported as false positives. Similarly, a small percentage of the results were reported as nondetects by SRI (i.e., false negatives) when the laboratory reported a detection (2% for RDX, 4% for TNT, none for 2,4-DNT).

**Completeness:** The GC/TID generated results for all 108 soil samples for a completeness of 100%.

**Comparability:** A one-to-one sample comparison of the GC/TID 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 (excluding one suspect measurement for the reference laboratory) was 0.95 (slope ( $m$ ) = 1.32). When comparability was assessed for specific concentration ranges, the  $r$  value did not change dramatically for TNT, ranging from 0.89 to 0.93 depending on the concentrations selected. RDX correlation coefficient with the reference laboratory for all soil results was slightly lower than TNT ( $r = 0.85$ ,  $m = 0.91$ ). The GC/TID's results for RDX correlated better with the reference laboratory for concentrations <500 mg/kg ( $r = 0.96$ ,  $m = 0.83$ ) than for samples where concentrations were >500 mg/kg ( $r = 0.49$ ,  $m = 0.56$ ). For the limited number of data points where both the reference laboratory and SRI reported results for 2,4-DNT ( $n = 14$ ), the correlation was 0.44 ( $m = 0.33$ ).

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

**Ease of Use:** No particular level of educational training is required for the operator, but knowledge of chromatographic techniques and experience in field instrument deployments would be advantageous.

**Overall Evaluation:** The overall performance of the GC/TID for the analysis of 2,4-DNT, RDX, and TNT was characterized as precise and unbiased. 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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