THE ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM

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ETV Joint Verification Statement

TECHNOLOGY TYPE: **Mobile Mass Spectrometers**

APPLICATION: **Monitoring volatile organic compounds, pesticides, and chemical agents in water**

TECHNOLOGY NAME: **CT-1128 Portable Gas Chromatograph–Mass Spectrometer (GC-MS)**

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The U.S. Environmental Protection Agency (EPA) has established the Environmental Technology Verification (ETV) Program 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 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. Information and ETV documents are available at www.epa.gov/etv.

ETV works in partnership with recognized standards and testing organizations, with stakeholder groups (consisting of buyers, vendor organizations, and permitters), and with 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 (QA) protocols to ensure that data of known and adequate quality are generated and that the results are defensible.

The Advanced Monitoring Systems (AMS) Center, one of six technology areas under ETV, is operated by Battelle in cooperation with EPA's National Exposure Research Laboratory. The AMS Center evaluated the performance of the Constellation Technology Corporation's CT-1128 Portable Gas Chromatograph–Mass Spectrometer (GC-MS). This verification statement provides a summary of the test results.

VERIFICATION TEST DESCRIPTION

Many volatile and semivolatile contaminants in water are detected using bench-top mass spectrometers in a traditional laboratory setting. However, the CT-1128 verified in this test was a portable unit designed to be taken outside the laboratory setting for field analysis. This portability offers an advantage to first-responders and other users who need chemical information when time, sampling, and other limitations preclude analysis in a laboratory.

The ability of the CT-1128 to identify and quantify target contaminants was tested in various water matrices. The CT-1128 was evaluated for the following performance parameters:

- Accuracy
- Precision
- Linearity
- Instrument stability
- Potential matrix and interference effects
- Sensitivity
- Field portability
- Operational factors.

Three classes of contaminants were used for testing: volatile organic compounds (benzene, toluene, ethyl benzene, total xylenes [BTEX]), pesticides (2,4-D and dicrotophos), and chemical warfare agents (VX, GB, and GD). The contaminants were selected based on recommendations from the AMS Center stakeholders.

Performance test (PT) samples were prepared in American Society for Testing and Materials (ASTM) Type II water. The target contaminant concentrations were constructed to bracket the concentrations of interest, which were calculated using LD_{50} values assuming a 70-kilogram individual consuming 250 milliliters of the contaminated water. When LD_{50} data were not available or feasible for testing, maximum contaminant levels (MCLs), as defined by EPA National Primary Drinking Water Regulations, were used. Reference measurements were conducted on PT samples only, to confirm the accuracy of sample preparation. EPA methods 524.2 and 515.1 were used for analyzing BTEX and 2,4-D, respectively. Internally developed methods were used for the remainder of the contaminant reference methods since no external methods were available from commercial laboratories at the time of testing.

The PT samples were used to determine the accuracy of the CT-1128; one set of which was used to establish a calibration curve. Subsequent analyses of the PT samples on multiple testing days were then used to calculate the accuracy of the CT-1128 measurements. To measure the potential matrix effects on the CT-1128 in selected real-world applications, it was challenged by analyzing samples fortified with the target contaminant in various matrices including drinking water (DW) samples (which varied in source and treatment), a weakly buffered water sample, a strongly buffered water sample, and a trihalomethanes (THMs)-fortified water sample. The concentration of a mid-level PT sample was used to fortify the matrix samples. This concentration provided a convenient level that was approximate to or below the concentration of interest for the target contaminants.

In addition to the PT, DW, buffered waters, and THMs-fortified water samples, blanks and unfortified matrix samples were analyzed to confirm negative responses in the absence of target contaminants and also to ensure that no sources of contamination were introduced during the analysis.

Experienced GC-MS operators were used for testing since the vendor suggests that a new user obtain training in the use of a GC-MS prior to operating the CT-1128. The vendor identified solid phase microextraction (SPME) as the technique for preparing the water samples for subsequent GC-MS analysis by the CT-1128. It is very important to note that the methodology provided by the vendor was not optimized for any one specific target chemical. The same SPME fiber type and GC column were used throughout the test for all analytes.

QA oversight of verification testing was provided by Battelle and EPA. Battelle QA staff conducted a technical systems audit, a performance evaluation audit, and a data quality audit of 10% of the test data. This verification statement, the full report on which it is based, and the test/QA plan for this verification test are all available at www.epa.gov/etv/centers/center1.html.

TECHNOLOGY DESCRIPTION

The following description of the CT-1128 is based on information provided by the vendor. This technology description was not verified in this test.

The CT-1128 analyzes, on-site, known and unknown chemicals. The CT-1128 is a lightweight, ruggedized, field deployable GC-MS system that can accommodate the applications of traditional laboratory based GC-MS systems. With the appropriate extraction techniques, analysis may be performed on a variety of matrices including DW, which can be prepared using SPME.

The CT-1128 weighs approximately 75 pounds (34 kilograms) and is 15 inches (38.1 cm) by 23 inches (58.4 cm) by 15 inches (38.1 cm). It is contained in a carrying case housing the entire system. The CT-1128 has a range of 1.6 to 800 atomic mass units with unit resolution throughout the mass range. In selected ion mode (SIM), the CT-1128 can scan for 50 groups of masses with 30 masses per group. For identification of chemicals, the CT-1128 is equipped with an automated mass spectral data base searching function that can use a range of commercial mass spectral libraries (e.g., National Institute of Standards and Technology Mass Spectral Library) as well as user-defined libraries. The system is controlled with a laptop computer that uses a program for GC control and MSD Chemstation (Agilent Technologies) for MS control and data analysis.

The CT-1128, which requires ultra-high purity hydrogen or helium (or nitrogen if desired) for the carrier gas, can use either an external gas tank or its on-board hydrogen storage bottle. The metal hydride storage bottle can be charged with hydrogen to provide a source of carrier gas that is convenient for mobile operation. The mass spectrometer can be tuned using an internal calibrant such as perfluorotributylamine (PFTBA) to perform a standard spectra tune or autotune (for maximum sensitivity over the entire scanning range) depending on the user’s needs. At the time of testing, the cost of the CT-1128 GC-MS system, with optional SPME stirrer/heater, was $140,000.

VERIFICATION RESULTS

Summary of Accuracy, Precision, Linearity, and Stability

Contaminant	**Accuracy**	**Precision**	**Linearity**	**Stability**
	Mean Percent Recovery (R)	**Mean Relative Standard Deviation (RSD)**	**Coefficient of Determination of Curve (r^2)**	**Mean Relative Percent Difference (RPD)**
benzene	172%	10%	1.000	27%
toluene	440%	43%	1.000	52%
ethylbenzene	104%	16%	1.000	9%
xylenes (total)	103%	10%	1.000	12%
2,4-D	62%	21%	0.921	35%
dicrotophos	143%	42%	0.999	92%
GB	108%	24%	1.000	48%
GD	75%	14%	1.000	27%
VX	109%	15%	0.959	27%

Benzene ***accuracy*** was considerably higher than ideal (100%) at 172% recovery (R) because of a change in response several days after establishing the calibration curve. Toluene exhibited significant over-recoveries, with an overall mean R of 440%, though accuracy for ethylbenzene and xylenes (total) was close to 100%. The mean R for 2,4-D and dicrotophos was 62% and 143%, respectively. For GB, the accuracy was close to ideal at 108%. For GD, R was acceptable at 75. The mean R for VX was 109%, though the concentrations tested were significantly higher than the LD_{50} for this agent. ***Precision***, as measured by relative standard deviation (RSD) of replicate samples, ranged from 10% for benzene to 43% for toluene.

In regard to ***linearity***, the calibration curves of seven of the nine contaminants had coefficients of determination (r^2) of 0.999 or greater. The exceptions were those contaminants for which the provided method lacked sensitivity—2,4-D and VX (r^2 of 0.921 and 0.959, respectively), though for all contaminants, r^2 values were greater than 0.920. Instrument ***stability*** was evaluated by comparing the results of mid-level PT samples at the beginning and end of the testing day and determining relative percent difference (RPD) of the PT samples (ideal RPD is 0%). Stability results ranged from 9% RPD for ethylbenzene to 92% for dicrotophos. Only two contaminants, ethylbenzene and xylenes (average RPD of 9% and 12%, respectively), had average RPDs less than 20%, while toluene and dicrotophos had RPDs significantly greater than 20% (52% and 92%, respectively).

Summary of Matrix Effects Observed

Contaminant	**Matrix Effect[a] from Potential Interferents** ✓ = observed						
	DW1	**DW2**	**DW3**	**DW4**	**Weakly Buffered Water**	**Strongly Buffered Water**	**THMs Spiked Water**
benzene					✓	✓	
toluene		✓	✓		✓	✓	
ethylbenzene						✓	
xylenes						✓	
2,4-D	✓	✓	✓	✓		✓	
dicrotophos		✓	✓	✓	✓	✓	
GB			✓			✓	✓
GD	✓		✓	✓		✓	✓
VX	ND	ND	ND	ND	ND	ND	ND

[a] matrix effect defined as recovery ± 30% of average response of daily PT samples fortified at the same concentration
ND = no data; VX matrix testing was not performed due to lack of sensitivity for this contaminant using the vendor-provided method

A ***matrix effect*** was present with the strong buffer matrix, which gave R values outside the 70% to 130% range for all eight of the target contaminants tested. DW3 also showed matrix effects for five of the eight contaminants, which may be due to its origin as a groundwater sample.

With the exception of VX and 2,4-D (for which the provided methods lacked sensitivity), the ***sensitivity*** of the CT-1128 was sufficient to detect the target contaminants at the concentrations of interest (i.e., LD_{50} or MCL concentrations).

Results of CT-1128 Sensitivity Testing for Target Contaminants

Contaminant	Concentration of Interest (mg/L)	Sufficient Sensitivity to Detect Conc. of Interest
benzene	0.005	Yes
toluene	1	Yes
ethylbenzene	0.7	Yes
xylenes (total)	10	Yes
2,4-D	0.07	No
dicrotophos	1400	Yes
GB	20	Yes
GD	1.4	Yes
VX	2.1	No

Field portability and operational factors: Because the CT-1128 requires time for thermal equilibration once electrical power and gas have been supplied, it should be kept on standby (under vacuum and thermally equilibrated) as long as possible when time is a critical factor for analyzing field samples. Mobilization in the field is straightforward, and the CT-1128 requires only a source of electrical power for several hours of field deployment when used with its on-board hydrogen canister for a source of carrier gas. Typical extraction and sample run times ranged from 22 minutes to 32 minutes. Average sample throughput during verification testing was 11 samples per 10-hour working day, or approximately one sample per hour. For 100 samples, the total cost for supplies was approximately $914, not including the GC column and standard chemicals.

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