THE ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM

U.S. Environmental Protection Agency

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ETV Joint Verification Statement

TECHNOLOGY TYPE:	**Trace Metals Analysis System**
APPLICATION:	**ANALYSIS OF ARSENIC IN WATER**
TECHNOLOGY NAME:	**SafeGuard Trace Metals Analyzer**
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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 TraceDetect SafeGuard Trace Metals Analyzer in measuring total arsenic in water. This verification statement provides a summary of the test results.

VERIFICATION TEST DESCRIPTION

The SafeGuard was verified by comparing its arsenic measurements to those from a laboratory-based reference method—inductively coupled plasma mass spectrometry (ICP-MS) performed according to EPA Method 200.8. The SafeGuard performance was verified by analyzing laboratory-prepared performance test (PT) samples, quality control (QC) samples, and environmental samples. All samples were tested using both the SafeGuard and the reference method. The SafeGuard was verified by evaluating accuracy, precision, linearity, method detection limit (MDL), matrix interference effects, operator bias, inter-unit reproducibility, and rate of false positives/false negatives.

Samples were prepared and analyzed according to the vendor's recommended procedures and the test/QA plan. All samples were analyzed without pretreatment except the drinking water samples collected from plumbing. These samples were filtered to remove potential copper contaminants. The results from the SafeGuard were compared to those from the reference method to assess accuracy and linearity. Four aliquots of PT samples and environmental samples were analyzed to assess precision. Seven aliquots of a 5 ppb PT sample were analyzed to assess the detection limit of the SafeGuard. Potential matrix interference effects were assessed by challenging the SafeGuard with PT samples of 10 ppb arsenic concentration that contained both low levels and high levels of potentially interfering substances. All samples were analyzed using two identical SafeGuard units (designated Unit #1 and Unit #2). Results of analyses from the two units were statistically compared to evaluate inter-unit reproducibility. Operator bias was assessed by statistically comparing data from two operators (technical and non-technical) analyzing identical sets of samples on both units. The rates of false positive and false negative results were evaluated relative to the 10-parts-per-billion (ppb) maximum contaminant level for arsenic in drinking water. Other factors that were qualitatively assessed during the test included ease of use, time required for sample analysis, and reliability.

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 SafeGuard is based on information provided by the vendor. This technology description was not verified in this test.

TraceDetect's SafeGuard is designed to automatically measure total arsenic concentrations in drinking water samples (including raw water and treated water) over a range from 1 ppb to over 100 ppb. Once the operator has introduced the sample vial and selected "measure" on the control computer, all calibrations, dilutions, reductions, standard additions, and measurements are performed by the SafeGuard with the results displayed and logged in a data file. The SafeGuard consists of three main components: the expert system, the fluidics system, and TraceDetect's patented NanoBand™ sensor and potentiostat. Each of these components has a part in the measurement process—from controlling the pumps, to adding chemicals, to making measurements and interpreting the results. The SafeGuard uses anodic stripping voltammetry (ASV) and the method of standard addition to make metals measurements. ASV is an electro-analytical method that detects ions in a solution by the potential at which they oxidize and strip away from the surface of an electrode. The SafeGuard is able to measure As (III) and reduce As (V) to As (III) to measure total arsenic. It can be configured to analyze copper, lead, zinc, cadmium, and mercury in water.

The SafeGuard stores data for every measurement and operation. The base of the SafeGuard is 15 inches by 28 inches (381 mm by 711 mm). It is 22 inches (559 mm) high and requires a computer, mouse, monitor, and keyboard. The TraceDetect SafeGuard as configured for measuring arsenic during this verification test was priced at $35,000, excluding options that the customer may require for unique sample preparation (e.g., copper removal from samples, filters for high turbidity samples).

VERIFICATION RESULTS

Accuracy was assessed by comparing the results to Method 200.8[2] results from ICP-MS analysis. The quantitative assessment of accuracy indicated that the relative bias for the SafeGuard ranged from -28% to 7% for the technical operator and -28% to 11% for the non-technical operator (excluding residential well water samples at approximately 600%).

Precision was assessed by analyzing four replicates of each sample. For the technical operator, precision expressed as relative standard deviation (RSD) ranged from 3% to 44%, and for the non-technical operator 2% to 38%. The average RSD for PT samples only was 10% for the technical operator and 9% for the non-technical operator. These results exclude samples for which one or more of the replicate results were not detected by the SafeGuard.

The linearity of response was evaluated by plotting the SafeGuard results against the reference analysis results for the PT samples. The table below summarizes the equations for the linear regressions and presents the 95% confidence interval for the slopes as +/- error. All linear regressions against the reference method results had coefficients of determination (r2) greater than 0.99. The 95% confidence intervals for the slopes indicate that only the technical operator data for Unit # 1 were consistent with a slope of 1 and were not significantly different from the reference analysis results. The 95% confidence intervals for the y-axis intercept included zero for both operators on both units indicating no significant difference from the reference analysis results.

Summary of Linear Regression Equations for SafeGuard and Reference Results

Description	Slope (+/- Error)	Intercept (+/- Error)	Coefficient of Determination
Unit #1, technical operator	1.005 (0.044)	-1.618 (2.32)	0.9942
Unit #2, technical operator	0.808 (0.034)	0.060 (1.70)	0.9936
Unit #1, non-technical operator	0.874 (0.027)	0.155 (1.27)	0.9961
Unit #2, non-technical operator	0.796 (0.019)	0.960 (0.96)	0.9979

The MDL was assessed by analyzing seven replicates of a sample spiked at a level approximately five times the manufacturer's estimated detection limit for the SafeGuard (i.e., 1 ppb x 5 = 5 ppb). The MDLs calculated using the precision data from these replicates ranged from 2.0 ppb to 3.8 ppb.

Results for samples containing low and high levels of interfering compounds indicated that neither level of interference appeared to affect the detection of arsenic, with bias ranging from -19% to 7%, consistent with the bias observed in the absence of interferences. The SafeGuard performance was affected by one of the environmental samples, the residential well water. The native (unspiked) replicates of this sample from both operators and both SafeGuard units reported an arsenic concentration from 2.50 ppb to 9.70 ppb, whereas the reference method reported this sample at 0.89 ppb to 1.12 ppb.

The equations for the linear regressions that were performed to evaluate operator bias and inter-unit reproducibility are summarized below. The 95% confidence interval includes a slope of 1 for Unit # 2, but the 95% confidence interval does not include a slope of 1 for Unit # 1, indicating a significant operator bias (technical results > non-technical results) with that unit. Paired t-tests of the two sets of data indicate that the SafeGuard results were not significantly different at a 0.05 level of significance depending on the operator. Overall, these results indicate at most a small operator bias with one of the two SafeGuard units.

Inter-unit reproducibility was evaluated by comparing the data for the two SafeGuard units used by the technical and non-technical operators. Linear regressions of the data for each unit show that Unit # 2 readings were lower than Unit # 1 readings with both operators, but more strongly with the technical operator. Neither 95%

confidence interval includes a slope of 1, indicating a significant inter-unit bias that is more pronounced with the technical operator than with the non-technical operator. A paired t-test of the data indicated that the results from the two units with the technical operator were significantly different at a 0.05 level of significance; however, the results from the two units with the non-technical operator were not significantly different. Overall, these results show an inter-unit bias with the technical operator, but minimal bias with the non-technical operator.

Summary of Linear Regression Equations for Assessing Operator Bias and Inter-unit Reproducibility

Description	Slope (+/- Error)	Intercept (+/- Error)	Coefficient of Determination
Unit #1, Operator bias	0.872 (0.033)	0.957 (1.18)	0.9886
Unit #2, Operator bias	0.983 (0.033)	0.566 (0.98)	0.9909
Technical operator, Inter-unit reproducibility	0.802 (0.029)	1.208 (1.03)	0.9897
Non-technical operator, Inter-unit reproducibility	0.907 (0.023)	0.864 (0.74)	0.9947

A false positive was defined as a SafeGuard result that was greater than 10 ppb and greater than 125% of the reference concentration, when the reference concentration was less than or equal to 10 ppb. The rates of false positives for the SafeGuard were 0% for both units for the technical operator and 2% and 0% for the non-technical operator (Units # 1 and # 2, respectively). A false negative was defined as a SafeGuard result that was less than or equal to 10 ppb and less than 75% of the reference concentration, when the reference concentration was greater than 10 ppb. The rates of false negatives for the SafeGuard units were 4% and 22% for the technical operator and 18% for both units for the non-technical operator. By averaging these rates, the results indicate that the SafeGuard correctly identified water below the federal drinking water standard (<10 ppb) 99.5% of the time (0.995 = sensitivity) and identified water that did not meet the federal standard (>10 ppb) 85.4% of the time (0.854 = specificity).

The SafeGuard system was easy to use, and the manual and software program were clear and easy to follow. All reagent mixing and instrument flushing are automated. No solution or sample preparation was necessary. The analysis time per sample at room temperature was 30 to 50 minutes. The listed price for SafeGuard at the time of the verification test was $35,000, and the cost for a 45-sample reagent kit was $80. Replacement reagents and supplies are available without purchasing entire kits.

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