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THE ENVIRONMENTAL TECHNOLOGY VERIFICATION
PROGRAM



ETV Joint Verification Statement

TECHNOLOGY TYPE: Arsenic Test Kit

APPLICATION: ANALYSIS OF ARSENIC IN WATER

TECHNOLOGY NAME: AS 75

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The U.S. Environmental Protection Agency (EPA) has created 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 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; with stakeholder groups that consist of buyers, vendor organizations, and permittees; and 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 (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 has recently evaluated the performance of four portable analyzers for arsenic in water. This verification statement provides a summary of the test results for the Peters Engineering AS 75 arsenic test kit.

VERIFICATION TEST DESCRIPTION

The AS 75 test kit is an inexpensive, portable, rapid device designed for on-site analysis of arsenic in water. The AS 75 test kit was verified in terms of its performance on the following parameters: accuracy, precision, linearity, method detection limit (MDL), matrix interference effects, operator bias, and rate of false positives/false negatives. All preparation, calibration, and analyses were performed according to the manufacturer's recommended procedures. Results from the AS 75 test kit were compared to those from a reference method to quantitatively assess accuracy, linearity, and detection limit. Multiple aliquots of performance test samples and drinking water samples were analyzed to assess precision. Identical sets of samples were analyzed independently by two separate operators (a technical and a non-technical Battelle staff member) to test for operator bias. Matrix interference effects were assessed by challenging the test kit with performance test samples of known arsenic concentrations containing both low-level and high-level interferences. False positives and negatives were evaluated relative to the recently established 10 ppb maximum contaminant level for arsenic in drinking water. In addition to the analytical results, the time required for sample analysis and operator observations concerning the use of the test kit (e.g., frequency of calibration, ease of use, maintenance) were recorded.

Three types of samples were used in the verification test: quality control (QC) samples, performance test (PT) samples, and environmental water samples. The QC and PT samples were prepared from National Institute of Standards and Technology traceable purchased standards. The environmental water samples were collected from various drinking water and surface water sources. All samples were analyzed using the AS 75 test kits and by a laboratory reference method. Every tenth sample was analyzed twice by the reference method to document the reference method's precision.

QA oversight of verification testing was provided by Battelle. Battelle QA staff conducted a data quality audit of 10% of the test data, a performance evaluation audit, and a technical systems audit of the procedures used in this verification.

TECHNOLOGY DESCRIPTION

The AS 75 consists of the PeCo test kit, which measures the color change of a filter by visual comparison to a color chart, and an AS 75 tester, which measures the color change of the filter electronically. The AS 75 includes a 100-mL reaction bottle (Erlenmeyer flask), two filter holders (grey and blue), a 50-mL volumetric cylinder, a 10- and a 1-mL pipette, tweezers, gloves, two color charts, filters, cotton, and chemicals and reagents (in tablet form) for 20 tests. The AS 75 can be used for 50-, 10-, 5-, or 1-mL samples; in this test all samples were 50 mL. The AS 75 tester is a hand-held, battery-powered electronic device recommended for determining low concentrations of arsenic in the field. To measure arsenic with the test kit, the Erlenmeyer flask is filled with the sample. The filter is placed into the test filter holder, reagent tablets are dropped in the sample, and the filter holder is placed on the flask. The filter is exposed to arsine gas evolved from the sample solution, resulting in a color change in the filter. When the reaction is complete, the filter is compared visually with the color charts provided with the PeCo test kit, or read by the AS 75 tester, to determine the amount of arsenic in the sample. The PeCo test kit color chart shows discrete color spots corresponding to arsenic concentrations of 10, 20, 30, . . . 100 parts per billion (ppb) for the grey filter holder and 2.5, 5, 10, 20, . . . 60 ppb for the blue filter holder. In a few cases, the operators interpolated between PeCo color readings to estimate the arsenic concentration. The AS75 tester has a display resolution of 1 ppb and can provide arsenic readings below 10 ppb. The battery-operated AS 75 tester provides power for 260 hours under normal conditions, allowing for up to 1,200 measurements. The tester (with the nine-volt battery) weighs 160 g (5.6 oz), and its dimensions are 120 mm by 60 mm by 26 mm (4.7 in by 2.4 in by 1 in).

VERIFICATION OF PERFORMANCE

The AS 75 arsenic test kit measures arsenic in water visually using the PeCo test kit, or electronically using the AS 75 tester. Both approaches were tested in this verification.

Accuracy: Considering results from both technical and non-technical operators, the PeCo test kit gave accuracy (in terms of bias relative to reference results) of 2 to 9%, 6 to 8%, and 2 to 17% for samples near 10 ppb arsenic in the PT, drinking water, and freshwater (FW) sample sets, respectively. At higher arsenic concentrations (23.5 to 92.6 ppb), the PeCo test kit gave biases of 1 to 113% and 21 to 77% for samples in the PT and drinking water sets, respectively. For the AS 75 tester, corresponding biases were 2 to 52%, 1 to 157%, and 6 to 33% for samples near 10 ppb in the PT, drinking water, and FW sets, respectively. At higher concentrations, the AS 75 tester showed biases of 6 to more than 310% and 8 to 50% for samples in the PT and drinking water sets, respectively.

An additional criterion for accuracy was the percentage of AS 75 results that were within 25% of the corresponding reference result or within a corresponding “less than” range. By this criterion, the qualitative accuracy of the PeCo test kit for the PT samples was 86% for the non-technical operator and 83% for the technical operator; for the drinking water samples it was 67% for the non-technical operator and 52% for the technical operator; and for the FW samples it was 96% for the non-technical operator and 92% for the technical operator. The qualitative accuracy of the AS 75 tester for the PT samples was 57% for the non-technical operator and 59% for the technical operator; for the drinking water samples it was 62% for the non-technical operator and 57% for the technical operator; and for the FW samples it was 75% for the non-technical and 88% for the technical operator.

Precision: With the PeCo test kit, the relative standard deviation (RSD) ranged from 0 to 40% for the non-technical operator and 0 to 24% for the technical operator in replicate analyses of the PT samples. The corresponding RSD for the drinking water samples was 27 to 41% for the non-technical operator, and was 0% for the technical operator. With the AS 75 tester, the RSD ranged from 11 to 38% for the non-technical operator and 12 to more than 71% for the technical operator on the PT samples. The RSD for the drinking water samples was 10 to 39% for the non-technical operator and 10 to 89% for the technical operator.

Linearity: The linearity of response of the AS 75 relative to the reference results was assessed using the PT samples containing from 1 to 93 ppb arsenic. The linear regression with the PeCo test kit for the non-technical operator was $\text{ppb} = 0.99 (\pm 0.090) \times (\text{reference, ppb}) + 2.41 (\pm 3.55) \text{ ppb}$, with $r = 0.977$. With the PeCo test kit for the technical operator, it was $\text{ppb} = 0.80 (\pm 0.069) \times (\text{reference, ppb}) + 5.12 (\pm 2.70) \text{ ppb}$, with $r = 0.979$. The linear regression with the AS 75 tester for the non-technical operator was $\text{ppb} = 1.27 (\pm 0.074) \times (\text{reference, ppb}) + 2.25 (\pm 2.93) \text{ ppb}$, with $r = 0.990$. With the AS 75 tester for the technical operator, it was $\text{ppb} = 1.28 (\pm 0.22) \times (\text{reference, ppb}) + 5.70 (\pm 8.69) \text{ ppb}$, with $r = 0.923$.

Method detection limit: A total of seven replicate PT samples were analyzed at a concentration of 25 ppb. With the PeCo test kit, the results from both operators were somewhat erratic. The non-technical operator reported arsenic between 15 and 50 ppb, and the technical operator reported arsenic between 20 and 40 ppb. No MDL was calculated for the PeCo kit, since it is a semi-quantitative test kit. For the AS 75 tester, the calculated MDL for the non-technical operator was 32.7 ppb, and for the technical operator was 28 ppb.

Matrix interference effects: The AS 75 results did not show a significant effect of sample matrices containing low or high levels of sodium chloride, iron, sulfate, or acidity using either the PeCo test kit or the AS 75 tester.

Operator bias: Operator technical training level had no major effect on the AS 75 results, with either the PeCo kit or the AS75 tester.

Rate of false positives/false negatives: The rates of false positives and false negatives of the AS 75 were assessed relative to the reference method using 10 ppb of arsenic as the decision level. The rate of false positives for the PeCo test kit was 0% for the non-technical and 3% for the technical operators. For the AS 75 tester, the false positive rate was 2% for the non-technical operator and 13% for the technical operator relative to the 10-ppb

value. For both the PeCo test kit and the AS 75 tester, the rate of false negatives was 0% for both the non-technical and the technical operators.

Other factors: The cost of the basic PeCo test kit capable of analyzing 100 samples is \$220. The cost of the electronic AS 75 tester is \$330. Refill packs for 100 additional tests cost \$60. The reagents are in tablet form and are ready to use, with no preparation required. The vendor was unable to provide newly prepared reagent tablets for this test, so previously prepared tablets from the vendor's stock were used. Operators found that the larger of the two reagent tablets required up to one and a half hours to dissolve. This kit requires no liquids or concentrated acids, making it safe and easy to carry in the field. The tablets contain no toxic materials.

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