

US EPA ARCHIVE DOCUMENT

THE ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM



TECHNOLOGY TYPE: AQUEOUS LEAD TESTING

APPLICATION: LEAD ANALYSIS FOR DRINKING, WASTE, AND ENVIRONMENTAL WATERS

TECHNOLOGY NAME: AND1000 Fluorimeter and Lead100 Test Kit

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ETV Joint Verification Statement

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 permittees), 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 and 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 verification centers under ETV, is operated by Battelle in cooperation with EPA's National Risk Management Research Laboratory. The AMS Center evaluated the performance of an aqueous lead (Pb) testing device for lead analysis in drinking, waste, and environmental waters. This verification statement provides a summary of the test results for the ANDalyze, Inc. AND1000 fluorimeter and Lead100 test kit (Lead100/AND1000) for determining lead concentrations in environmental water.

VERIFICATION TEST DESCRIPTION

The verification testing of the Lead100/AND1000 was conducted in two rounds: the first from July 10 to July 27, 2012, and the second from February 11 to March 13, 2013 at both various field sites and the Environmental Treatability Laboratory (ETL) at Battelle's Main Campus in Columbus, OH. The verification test assessed the performance of the Lead100/AND1000 relative to key verification parameters including accuracy, precision, sample throughput, and ease of use. These performance parameters were evaluated using multiple variables that challenged the Lead100/AND1000's ability to detect Pb in a variety of aqueous matrices. The technology evaluation was organized as four main tests. Each test evaluated the performance of Lead100/AND1000 operating under different laboratory and field conditions. The four tests were:

1. Initial demonstration of capability (IDC) and performance testing (PT) including determination of the limit of detection (DLOD), determination of linear range (DLR), and determination of the effects of interferences (DEI)
2. Testing accuracy and precision of the instrument for the analysis of finished drinking water samples (bottled water, municipal drinking water, and treated groundwater)
3. Testing accuracy and precision of the instrument for the analysis of environmental water samples (surface water and groundwater) and qualitative performance for the analysis of seawater
4. Testing accuracy and precision of the instrument for the analysis of wastewater effluent samples (municipal wastewater effluent and metal finishing wastewater effluent)

The performance parameters were evaluated quantitatively using the statistical methods and qualitatively through recorded observations. All samples analyzed with the Lead100/AND1000 were also collected, stored, transferred, and analyzed in a certified laboratory using industry accepted analytical methods. All tests were performed with the Lead100/AND1000 operating according to the vendor's recommended procedures as described in the user's manuals and during training provided to the operator.

The first set of tests (IDC and PT) was entirely performed within Battelle's ETL. IDC required the detection of a Pb spike of 25 parts per billion (ppb) in reagent grade water. DLOD was performed by measuring seven replicates of Pb spiked at 10 ppb, five times the vendor-reported limit of detection (2 ppb). The DLR was carried out by measuring the Lead100/AND1000's ability to precisely and accurately measure seven samples with Pb concentrations from 0 to 100 ppb. The samples were analyzed in triplicate and the coefficient of determination was used to assess the linearity of the response of the instrument within this range. Finally, DEI was determined using three synthetic water samples with differing characteristics: low total dissolved solids concentration (LTDS), high total dissolved solids concentration (HTDS), and high iron (Fe) concentration and other dissolved solids (HFe). Each of these synthetic water samples was split into required 100 mL subsamples and received a Pb spike of 25 ppb and 50 ppb before Pb was measured in triplicate from each subsample by Lead100/AND1000. Normal sample preparation procedures were followed for the LTDS and HTDS waters, and a special sample preparation procedure for the removal of Fe interference was used to prepare the HFe sample for Pb analysis.

The next set of tests determined the accuracy and precision of the Lead100/AND1000 in recovery of Pb spikes in finished drinking water. Three sets of samples were prepared with Pb spikes of 25 ppb: finished drinking water samples collected from a water fountain (WF), bottled mineral water purchased from a local supermarket (Bottled Water [BW]), and finished drinking water collected from the effluent of a local water treatment facility treating groundwater (Finished Well Water [FWW]). All waters were analyzed without a Pb spike and in triplicate with a Pb spike of 25 ppb.

The third series of tests aimed at determining the accuracy and precision of the Lead100/AND1000 in recovering Pb spikes in environmental water samples. Three freshwater sources were sampled including water collected from the reach of a freshwater river just outside and upstream of Columbus, OH (River Water [RiW]), samples collected from a freshwater reservoir near the same location (Reservoir Water [ReW]), and raw groundwater collected at the source of a drinking water treatment facility just outside Plain City, OH (Raw Well Water [RWW]), which was collected from the source that feeds the facility from which the Finished Well Water was

collected. In addition, one seawater sample was collected off the coast of West Palm Beach, FL (Seawater [SW]) to determine the accuracy and precision of the Lead100/AND1000 in testing natural waters with high salinity. All four environmental samples were analyzed with no Pb spike and in triplicate after the addition of a Pb spike to 25 ppb. Performance of tests on Seawater differ from the performance of tests on freshwater in that seawater was diluted tenfold before being subjected to Lead100/AND1000 testing and results were analyzed qualitatively, not quantitatively as with freshwater samples.

The final series of tests aimed at determining the accuracy and precision of the Lead100/AND1000 in recovering Pb spikes in wastewater effluent samples. Three samples were analyzed during this series of tests: two effluent samples collected from two separate traditional activated sludge treatment facilities treating domestic wastewater in Columbus, OH (Municipal Wastewater Effluent #1 [MWE#1] and Municipal Wastewater Effluent #2 [MWE#1]) and a sample collected from the effluent of a metal finishing works (Metal Finishing Wastewater Effluent [MFWWE]). The Metal Finishing Wastewater Effluent was collected from a facility conforming to 40 Code of Federal Regulations (CFR) 433 and/or 40 CFR 413 after all on-site pretreatment.

QA oversight of verification testing was provided by Battelle and EPA. Battelle QA staff conducted technical systems audits of all laboratory testing and conducted a data quality audit of at least 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 available at <http://www.epa.gov/nrmrl/std/etv/verifiedtechnologies.html#water>.

TECHNOLOGY DESCRIPTION

The following is a description of the technology, based on information provided by the technology representative. The information provided below was not verified in this test.

The ANDalyze Lead100/AND1000 was designed to detect the presence of and measure the concentration of soluble/bioavailable Pb in drinking water and environmental waters. Testing is intended to take place onsite at the source of collection or in a temperature controlled facility without sample preservation. The test makes use of two primary components: a handheld fluorimeter (AND1000) and a consumable test kit (Lead100) specific to each metal or target (in the present case, Pb).

The AND1000 fluorimeter is specifically designed to provide an interactive experience and allow testing, data storage, and signal output without the use of a separate computational device. The fluorimeter has the capability to analyze multiple targets with the appropriate test kit, though the sole target discussed here is aqueous Pb in drinking water, wastewater effluent, and environmental waters. The AND1000 fluorimeter enables field testing to be performed in two steps. ANDalyze's catalytic deoxyribonucleic acid (DNA) sensors use a metal-specific DNzyme reaction that leads to an increase in fluorescence in the presence of a target contaminant substance (i.e., aqueous Pb). The fluorescence of the reaction is measured by a fluorimeter to determine the concentration of the target heavy metal and is reported in parts per billion (ppb). The product is a quantitative test that is intended to detect metals in a linear range of 2 to 100 ppb — at and below EPA standards for drinking water. The test is performed by injecting a buffered 1 mL water sample through the sensor and into the AND1000 fluorimeter. This sample is then automatically analyzed and results are reported in less than 2 minutes. The AND1000 fluorimeter package includes the fluorimeter, USB to MINI-B cable, 100 µL fixed volume pipette and tips, and pH test strips.

The second component is the Lead100 test kit which is specific to Pb. The test kit provides all necessary materials for in-field instrument calibration and sample testing. This kit contains the DNA sensors specific to Pb. The kit is color coded to facilitate use and avoid measurement error. In addition, a product manual is provided with step-by-step instructions including photographs. It should be noted that laboratory evaluation may require additional supplies and standard laboratory glassware. Each kit includes 25 tests and/or calibrations, 25 sensor bags with sensor and cuvette, 25 sample tubes (with buffer), 25 1 mL syringes, 25 disposable transfer pipettes, 8 mL analyte standard solution, instruction manuals, and material safety data sheets.

VERIFICATION RESULTS

The verification of the Lead100/AND1000 is summarized below.

Accuracy

Percent recovery was used for all Lead100/AND1000 observations to verify acceptability of on-site calibration using control charts. Percent recovery was also used for IDC, ICC, DEI, finished drinking water testing, environmental water testing, and wastewater effluent testing as acceptance criteria for observations, for which percent recoveries between 75 and 125% were considered acceptable. Unacceptable recoveries were first retested, then—if the recovery was still unacceptable—tested again after instrument recalibration, and finally targeted in a root cause analysis (RCA).

No quality control (QC) issues were indicated on control charts for the 12 observations on IDC and ICC related samples. However, retesting was performed four times and on-site recalibration was performed once during IDC and ICC testing, and two IDC samples still did not meet criteria for percent recovery when compared to reference concentrations. Several QC issues were indicated on control charts for the 28 observations corresponding to DLOD and DLR. However, on-site recalibration was not required for DLOD and DLR testing and all samples met criteria for percent recovery when compared to reference concentrations. One QC issue was indicated on control charts for the 25 observations corresponding to DEI. Retesting was performed on four samples during DEI, but on-site recalibration was not required for DEI as all samples met criteria for percent recovery when compared to reference concentrations. Generally, recoveries during IDC, ICC, and DEI were considered acceptable.

Two QC issues were indicated on control charts for the 19 observations corresponding to environmental samples. Retesting was performed on four samples during environmental sample testing and on-site recalibration was required and a RCA was performed for raw well water samples suspected of containing high levels of Fe that interfered with the test. Nevertheless, five observations did not meet criteria for percent recovery when compared to reference concentrations. Two QC issues were indicated on control charts for the 17 observations corresponding to drinking water samples. Retesting was performed on three samples during drinking water sample testing, and on-site recalibration was required twice during drinking water testing. During testing, BW did not appear to meet QC criteria and a RCA was performed. It was determined that these observations were in fact within QC criteria once reference data were received. Nevertheless, four observations did not meet criteria for percent recovery when compared to reference concentrations. No QC issues were indicated on control charts for the 15 observations corresponding to wastewater effluent samples. Retesting was performed on two samples during wastewater effluent testing, and on-site recalibration was required once. Nevertheless, five observations did not meet criteria for percent recovery when compared to reference concentrations. More unacceptable recoveries were observed during drinking water, environmental water, and wastewater effluent testing than in the previous IDC and PT testing; however, recoveries were still generally considered acceptable based on the complexity of those matrices.

Precision

Mean observation value, standard deviation (SD), and coefficient of variation (CV) were determined for all 23 samples analyzed in triplicate. Standard deviations ranged from 0.58 to 11.14, corresponding to coefficients of variation ranging from 3% to 19%.

Linearity of Response

Observations from DLR sample testing were plotted against concentrations from reference analysis. Linearity was calculated in terms of slope (0.8841), intercept (-0.8418), and the square of the correlation coefficient (r^2) (0.9927). Results from the Lead100/AND1000 were generally in good agreement with results from the reference analysis, as indicated by a slope close to 1 and a coefficient of determination close to 1.

Limit of Detection

DLOD testing included statistical analyses in accordance with 40 CFR Part 136. The limit of detection of the Lead100/AND1000 was calculated to be 1.534 $\mu\text{g/L}$ Pb, which is below the vendor's estimated detection limit.

Qualitative Results for Seawater Samples

Precision was also calculated for Seawater samples to assist in qualitative analysis. As expected, the coefficients of variation for Seawater observations were significantly higher than those associated with any other test owing to the high salinity of the matrix. However, observations aligned with reference concentrations (i.e., no observations for samples prepared with 20 µg/L Pb were greater than any observations for samples prepared with 40 µg/L Pb). This demonstrates that the Lead100/AND1000 is capable of indicating whether seawater has a high or low concentration of Pb, despite its low precision when analyzing seawater samples.

Operational Factors

In general, the ease of use of the Lead100/AND1000 was high. In several cases, the rechargeable battery provided with the AND1000 lost power after less than 8 hours. In one instance, the instrument displayed a screen that was foreign to the user making the instrument unusable; however, simply turning the instrument off and rebooting returned the AND1000 to normal working order. The Lead100 test kits generate a significant amount of solid waste if many tests are performed. Under remote/austere sampling conditions in which waste minimization is essential, this should be taken into account by the user. The instrument cannot be used to determine particulate Pb. Additionally, samples are intended to be unpreserved for analysis by the technology and the pH of the samples is an important parameter to measure to ensure that the pH is within the specifications for accurate analysis.

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