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Innovative Technology Verification Report

Field Measurement Technology for Mercury in Soil and Sediment

Milestone Inc.'s Direct Mercury Analyzer (DMA)-80

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

Office of Research and Development
Washington, DC 20460

MEASUREMENT AND MONITORING TECHNOLOGY PROGRAM VERIFICATION STATEMENT

TECHNOLOGY TYPE: **Field Measurement Device**

APPLICATION: **Measurement for Mercury**

TECHNOLOGY NAME: **Milestone Inc.'s Direct Mercury Analyzer (DMA)-80**

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VERIFICATION PROGRAM DESCRIPTION

The U.S. Environmental Protection Agency (EPA) created the Superfund Innovative Technology Evaluation (SITE) and Measurement and Monitoring Technology (MMT) Programs to facilitate deployment of innovative technologies through performance verification and information dissemination. The goal of these programs is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. These programs assist and inform those involved in design, distribution, permitting, and purchase of environmental technologies. This document summarizes results of a demonstration of the Direct Mercury Analyzer (DMA)-80 developed by Milestone Inc.

PROGRAM OPERATION

Under the SITE and MMT Programs, with the full participation of the technology developers, the EPA evaluates and documents the performance of innovative technologies by developing demonstration plans, conducting field tests, collecting and analyzing demonstration data, and preparing reports. The technologies are evaluated under rigorous quality assurance(QA) protocols to produce well-documented data of known quality. The EPA National Exposure Research Laboratory, which demonstrates field sampling, monitoring, and measurement technologies, selected Science Applications International Corporation as the verification organization to assist in field testing five field measurement devices for mercury in soil and sediment. This demonstration was funded by the SITE Program.

DEMONSTRATION DESCRIPTION

In May 2003, the EPA conducted a field demonstration of the DMA-80 and four other field measurement devices for mercury in soil and sediment. This verification statement focuses on the DMA-80; a similar statement has been prepared for each of the other four devices. The performance of the DMA-80 was compared to that of an off-site laboratory using the reference method, "Test Methods for Evaluating Solid Waste" (SW -846) Method 7471B (modified). To verify a wide range of performance attributes, the demonstration had both primary and secondary objectives. The primary objectives were:

(1) Determining the instrument sensitivity with respect to the Method Detection Limit (MDL) and Practical Quantitation Limit (PQL);
(2) Determining the analytical accuracy associated with the field measurement technologies;
(3) Evaluating the precision of the field measurement technologies;
(4) Measuring the amount of time required for mobilization and setup, initial calibration, daily calibration, sample analysis, and demobilization; and
(5) Estimating the costs associated with mercury measurements for the following four categories: capital, labor, supplies, and investigation-derived waste (IDW).

Secondary objectives for the demonstration included:

(1) Documenting the ease of use, as well as the skills and training required to properly operate the device;
(2) Documenting potential health and safety concerns associated with operating the device;
(3) Documenting the portability of the device;
(4) Evaluating the device durability based on its materials of construction and engineering design; and
(5) Documenting the availability of the device and associated spare parts.

The DMA-80 analyzed 59 field soil samples, 13 field sediment samples, 42 spiked field samples, and 59 performance evaluation (PE) standard reference material (SRM) samples in the demonstration. The field samples were collected in four areas contaminated with mercury, the spiked samples were from these same locations, and the PE samples were obtained from a commercial provider.

Collectively, the field and PE samples provided the different matrix types and the different concentrations of mercury needed to perform a comprehensive evaluation of the DMA-80. A complete description of the demonstration and a summary of the results are available in the Innovative Technology Verification Report: "Field Measurement Technology for Mercury in Soil and Sediment—Milestone Inc.'s Direct Mercury Analyzer (DMA)-80"(EPA/600/R-04/012).

TECHNOLOGY DESCRIPTION

The DMA-80 is an atomic adsorption spectrophotometer based on mercury vaporization, amalgamation, desorption, and analysis of samples using an adsorbance spectrophotometer. Mercury samples are heated to 750˚ to 800˚C, causing organic materials to be decomposed and mercury to be vaporized in a carrier gas of oxygen. The oxygen flow carries the vaporized mercury to the amalgamator, where it deposits on gold-covered molecular sieves. Potential interferents are carried out of the system with the continuous gas stream . The m ercury deposits are then desorbed as the amalgamator is heated; vaporized mercury is transported to the spectrophotometer for analysis. The spectrophotometer uses a mercury vapor lamp as its light source. Light from the lamp is directed through an excitation filter before it irradiates the vaporized mercury contained in a quartz cuvette. The detector utilizes two sequential cuvettes: one for low concentration samples and the other for high concentration samples. Light which is not absorbed by the mercury vapors, then passes through an emission filter before being measured by the detector. Results are transmitted to the system controller, where concentrations are calculated based on sample mass and the detector response relative to a calibration curve.

During the demonstration, no extraction or sample digestion was required. Individual samples were mixed manually using a stainless steel spatula. (Note that samples were already considered to be homogeneous based upon the standard operating procedure used by SAIC to homogenize and aliquot all samples.) This same spatula was used to transfer the sample to a nickel weigh boat designed to fit the auto sampler. The sample was then weighed on a digital balance and placed on the 40-slot, auto sampler tray. The sample weight was automatically relayed to the DMA-80 controller; sequential sample numbers were automatically entered by the software in the data table in the location corresponding to the auto sampler location (1 - 40). Site-specific sample identification numbers were entered manually. The sample was analyzed, and the device displayed the mercury concentration in parts per million, which is equivalent to a soil concentration in milligrams per kilogram.

ACTION LIMITS

Action limits and concentrations of interest vary, and are project specific. There are, however, action limits which can be considered as potential reference points. The EPA Region IX Preliminary Remedial Goals for mercury are 23 mg/kg in residential soil and 310 m g/k g in industrial soil.

VERIFICATION OF PERFORMANCE

To ensure data usability, data quality indicators for accuracy, precision, representativeness, completeness, comparability, and sensitivity were assessed for the reference method based on project-specific QA objectives. Key demonstration findings are summarized below for the primary objectives.

Sensitivity: The two primary sensitivity evaluations performed for this demonstration were the MDL and PQL. Both will vary dependent upon whether the matrix is a soil, waste, or aqueous solution. Only soils/sediments were tested during this demonstration, and therefore, MDL calculations and PQL determinations for this evaluation are limited to those matrices. By definition, values measured below the PQL should not be considered accurate or precise and those below the MDL are not distinguishable from background noise.

Method Detection Limit - The evaluation of an MDL requires seven different measurements of a low concentration standard or sample. Following the procedures established in the 40 Code of Federal Regulations (CFR) Part 136, the MDL is estimated between 0.049 and 0.068 mg/kg. The equivalent calculated MDL for the referee laboratory is 0.0026 mg/k g. The calculated MDL is only intended as a statistical estimation and not a true test of instrument sensitivity.

Practical Quantitation Limit - The PQL for this instrument is approximately 0.082 mg/kg (the concentration of a SRM used during the demonstration) for soil and sediment materials. It is possible that the PQL may be as low as the MDL but there were no SRMs tested at this lower concentration. The referee laboratory PQL confirmed during the demonstration is 0.005 mg/kg, with a %D < 10%.

Accuracy: The results from the DMA-80 were compared to the 95% prediction interval for the SRM materials and to the referee laboratory results (Method 7471B). DMA-80 results were within SRM 95% prediction intervals 93% of the time, which suggests significant equivalence to certified standards. The number of Milestone average values less than 30% different from the referee laboratory results or SRM reference values; however, was 16 of 30 different sample lots. Only 2 of 30 Milestone average results have relative percent differences greater than 100% for this same group of samples. However, when making the comparison between Milestone and ALSI data, and taking into account the possible bias associated with both sets of data, this comparison may be within reasonable expectations for considering these two separate analyses to be equivalent. With the exception of a slight low bias for the referee laboratory and a slight high bias for the DMA-80 (similar to biases observed during other inter-laboratory studies), the data sets for the DMA-80 compared to the referee laboratory were considered to be similar and within expected statistical variation.

Precision**:** The precision of the Milestone field instrument is very comparable to the referee laboratory precision, and with in expected precision variation for soil and sediment matrices. The overall average relative standard deviation (RSD) was 23.7% for the referee laboratory and 19.4% for Milestone. Both the laboratory and Milestone precision results are within the predicted 25% RSD objective for precision expected from both analytical and sampling variance.

Measurement Time**:** From the time of sample receipt, Milestone required 22 hours and 10 minutes to prepare a draft data package containing mercury results for 173 samples. One technician performed all setup, calibration checks, sample preparation and analysis, and equipment demobilization. Individual analyses took 5 minutes each (from the time the sample was injected until results were displayed), but the total time per analysis averaged nearly 8 minutes when all field activities and data package preparation were included in the calculation.

Measurement Costs**:** The cost per analysis based upon 173 samples, when renting the DMA-80, is $35.90 per sample. The cost per analysis for the 173 samples, excluding rental fee, is $18.55 per sample. Based on the 3-day field demonstration, the total cost for equipment rental and necessary supplies is estimated at $6,210. The cost breakout by category is: capital costs, 48.3%; supplies, 9.5%; support equipment, 4.5%; labor, 14.5%; and IDW, 23.2%.

Key demonstration findings are summarized below for the secondary objectives.

Ease of Use**:** Based on observations made during the demonstration, the DMA-80 is easy to operate, requiring one field technician with a basic knowledge of chemistry acquired on the job or in a university and training on the DMA-80. A 1-day training course on instrument operation is offered at additional cost; this training would likely be necessary for most device operators who have no previous laboratory experience.

Potential Health and Safety Concerns**:** No significant health and safety concerns were noted during the demonstration. The only potential health and safety concerns identified were the generation of mercury vapors and the use of oxygen as the carrier gas. The vendor recommends and can provide a mercury filter; oxygen can be safely handled using standard laboratory procedures.

Portability: The DMA-80 was not easily portable (by hand) due to its size (80 cm by 42 cm by 30 cm high) and weight (56 kg). It was easy to set up and can be taken any place accessible to a small van or SUV. The instrument is better characterized as mobile rather than field portable. It operates on 110 or 220 volt AC current; no battery power supply is available.

Durability: The DMA-80 was well designed and constructed for durability. The auto sampler piston required re-alignment once early in the demonstration, an operation normally required after shipment. In two incidents related to piston alignment, one sample was dropped by the weigh boat injector and the auto-sampler tray later jammed. These problems were easily rectified, requiring less than 5 minutes each to troubleshoot and fix.

Availability of the Device: The DMA-80 is readily available for lease, or purchase. DMA-80 rental is available on a limited basis. Spare parts and consumable supplies can be added to the original DMA-80 order or can be received within 24 to 48 hours of order placement. Supplies and standards not provided by Milestone are readily available from laboratory supply firms.

PERFORMANCE SUMMARY

In summary, during the demonstration, the DMA-80 exhibited the following desirable characteristics of a field mercury measurement device: (1) good accuracy, (2) good precision, (3) high sample throughput, (4) low measurement costs, and (5) ease of use. During the demonstration the DMA-80 was found to have the following limitation: (1) non-portable due to the instrument size and weight. The demonstration findings collectively indicated that the DMA-80 is a reliable field measurement device for mercury in soil and sediment.