# THE ENVIRONMENTAL TECHNOLOGY VERIFICATION







## **ETV Joint Verification Statement**

**TECHNOLOGY TYPE:** Arsenic Test Kit

APPLICATION: ANALYSIS OF ARSENIC IN WATER

TECHNOLOGY NAME: Nano-Band™ Explorer

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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 permitters; 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 TraceDetect Nano-Band<sup>TM</sup> Explorer.

### VERIFICATION TEST DESCRIPTION

The Nano-Band<sup>TM</sup> Explorer is a portable, rapid device designed for on-site analysis of arsenic in water. The Nano-Band<sup>TM</sup> Explorer 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. Two units of the Nano-Band<sup>TM</sup> Explorer were tested independently by challenging them with test samples representative of those likely to be analyzed using the Nano-Band<sup>TM</sup> Explorer. Each unit of the Nano-Band<sup>TM</sup> Explorer was used to analyze the full set of samples for arsenic. The results from the Nano-Band<sup>TM</sup> Explorer tests 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. 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. 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 purchased standards. The environmental water samples were collected from various drinking water and surface water sources. All samples were analyzed using the two Nano-Band<sup>TM</sup> Explorers and by a laboratory reference method. Every tenth sample was analyzed twice by the reference method to document the reference method's precision.

Identical sets of samples were to be analyzed independently by a technical and a non-technical operator, using separate units of the Nano-Band<sup>TM</sup> Explorer. Unfortunately, due to the failure of one Nano-Band<sup>TM</sup> Explorer, none of the well water (treated or otherwise) or fresh water analyses could be performed by the technical operator. At the vendor's request, such samples were stored at 4°C at Battelle until a vendor representative could perform these analyses approximately three weeks after sample collection.

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 Nano-Band<sup>TM</sup> Explorer uses an anodic stripping voltammetry technique in which information about an analyte is derived from the measurement of electric current as a function of applied potential. The measurement is performed in an electrochemical cell under polarizing conditions on a working electrode. Analysis involves reducing the analyte of interest and collecting it at the working electrode. The analyte is then stripped off (i.e., oxidized) and measured. The stripping step is much shorter than the reduction step, and the consequent increase in the signal-to-noise ratio allows low concentration solutions to be measured. The Nano-Band™ Explorer electrode is an array of 100 sub-electrodes, each less than 0.5 microns thick. The increased mass transport rate afforded by this array allows parts per billion (ppb) measurements in seconds. Iridium electrodes are used to measure lead, mercury, copper, zinc, cadmium, thallium, bismuth, tin, antimony, and silver. Gold electrodes are used to measure arsenic. The three-electrode cell combines a Nano-Band<sup>TM</sup> Explorer electrode with a reference and an auxiliary electrode. The auxiliary and reference electrodes manage the current as it is passed through the working electrode. The Nano-Band<sup>TM</sup> Explorer has a detection limit as low as 0.1 ppb for some metals and displays measurement results in real time using software run on a laptop computer. The nominal detection limit for arsenic in this test was 4 ppb. The Nano Band<sup>TM</sup> Explorer is optimized for trace metals analysis. It can perform anodic and cathodic stripping voltammetry; normal square wave voltammetry; amperometry; cyclic voltammetry; temperature and pH measurements; and long-term data logging. The measurement system includes the Nano-Band<sup>TM</sup> Explorer, one reference and one auxiliary electrode, a measurement manual, a reference manual, Explorer software, a three-foot electrode cable, a temperature sensor, and an electrode cleaning kit. The Nano-Band<sup>TM</sup> Explorer is controlled by a laptop computer, which is not included in the cost of the instrument.

### VERIFICATION OF PERFORMANCE

Accuracy: An evaluation of the accuracy of the Nano-Band<sup>TM</sup> Explorer showed that the percent bias for individual samples ranged from 3 to 64% for the non-technical operator and 1 to 64% for the technical operator for the PT samples. For the non-technical operator, the bias for the individual well water samples (well water and treated well water) was 2 to 32% (average bias of 15% on ten samples), and for the freshwater (FW) samples was up to 499%. Due to instrument failure in the field, the technical operator did not analyze the well water (WW), treated well water (TW), or the FW samples. These samples were stored at 4°C for three weeks before analysis in the laboratory by the vendor representative. The bias for the individual samples was 25 to 92% for the WW and TW, samples and up to 68% for the FW samples. Similar ranges of bias were found when only samples containing 10 ppb or more arsenic were considered. An additional criterion for accuracy was the percentage of sample for which the Nano-Band<sup>TM</sup> result was within 25% of the reference result. By this criterion, the quantitative accuracy of the Nano-Band<sup>TM</sup> Explorer for the PT samples was 55% for the non-technical operator and 74% for the technical operator. The qualitative accuracy for the municipal drinking water samples was 71% for both the non-technical operator, and 21% for the vendor representative. The qualitative accuracy for the FW samples was 75% for the non-technical operator, and 83% for the vendor representative.

**Precision:** The precision of the Nano-Band™ Explorer was determined by calculating the percent relative standard deviation (RSD) of replicate analyses. The RSD ranged from 13 to 91% for the non-technical operator and 3 to 37% for the technical operator on the PT samples. The RSD for the drinking water samples was 11 to 13% for the non-technical operator and 4 to 11% for the vendor representative.

**Linearity:** The linearity of response of the Nano-Band<sup>TM</sup> Explorer was assessed using PT samples containing from 1 to 93 ppb arsenic. The linear regression for the Nano-Band<sup>TM</sup> Explorer for the non-technical operator was ppb =  $1.28 \pm 0.16 \times (\text{reference}, \text{ppb}) - 10.73 \pm 0.08 \times (\text{reference}, \text{ppb}) - 5.56 \pm 0.08 \times (\text{reference}, \text{ppb}) - 5.56 \pm 0.09 \times (\text{reference}, \text{ppb}) - 5.00 \times (\text{$ 

**Method detection limit:** The MDL was determined by analyzing seven replicate samples at a concentration of 25 ppb. The calculated MDL for the non-technical operator was 12.1 ppb and 14.2 ppb for the technical operator.

Matrix interference effects: The Nano-Band™ Explorer did not appear to be affected by matrix interferences added to the samples. However, the data from the two operators were quite different, with the non-technical operator reporting no detectable arsenic in any of the 16 samples. The technical operator reported an average value of 10.4 ppb of arsenic compared to the reference value of 9.91 ppb for the samples with low levels of interferants, and an average value of 11.5 ppb compared to the reference value of 9.94 ppb for the samples with high levels of interferants.

Rate of false positives/false negatives: The rates of false positives and false negatives of the Nano-Band<sup>TM</sup> Explorer were assessed relative to the reference method using 10 ppb of arsenic as the decision level. The rate of false positives for the Nano-Band<sup>TM</sup> Explorer was 0% for the non-technical operator, 13% for the technical operator, and 0% for the vendor representative. The rate of false negatives was 22% for the non-technical operator, 7% for the technical operator, and 100% for the vendor representative (who analyzed WW and TW samples stored for three weeks at 4°C).

**Operator bias:** The Nano-Band<sup>TM</sup> Explorer required some technical ability that at times seemed beyond the capabilities of the non-technical operator. However, none of the operators, including a representative of the Nano-Band<sup>TM</sup> Explorer's vendor, consistently achieved the expected results in this test.

Other factors: The Nano-Band<sup>TM</sup> Explorer sells for \$8,000. The samples take approximately one hour to prepare prior to analysis, seven samples can be prepared simultaneously, and the analysis takes less than one minute per sample. The Nano-Band<sup>TM</sup> Explorer used by the Battelle technical operator failed during testing due to an open circuit fault in the electrode cable. After repairs, sample analyses were completed by a representative of the Nano-Band<sup>TM</sup> Explorer's vendor. The Nano-Band<sup>TM</sup> Explorer requires some reagent preparation prior to entering the field. The reagents include acids and air-sensitive compounds that must be handled with care. The user should wear gloves during reagent preparation.

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