THE ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM

ETV

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ETV Joint Verification Statement

TECHNOLOGY TYPE:	**Nitrate Test Kit**
APPLICATION:	**ANALYSIS OF NITRATE AND NITRITE IN WATER**
TECHNOLOGY NAME:	**F-NTK**
COMPANY:	**Nitrate Elimination Co., Inc. (NECi)**

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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 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 nitrate test kits used to analyze for nitrate and nitrite in water. This verification statement provides a summary of the test results for the Nitrate Elimination Co., Inc., (NECi) F-NTK nitrate test kit.

VERIFICATION TEST DESCRIPTION

The F-NTK nitrate test kit was evaluated for accuracy, precision, linearity, method detection limit, matrix interference effects, and operator bias, as well as ease of use, cost, and sample throughput. Two sets of F-NTK test kits were tested independently by challenging them with test samples representative of those likely to be analyzed using the F-NTK. The results from the F-NTK nitrate analyses were compared to those from a reference method to assess accuracy, linearity, and detection limit. Multiple aliquots of performance test samples and drinking water samples were analyzed to assess precision.

For each set of test kits, identical sets of samples were analyzed independently by two separate operators (a technical and a non-technical person) to test for the existence of operator bias on the test kit performance. Interferences and matrix effects were assessed by separately evaluating accuracy on distinctly different sample matrices (i.e., prepared samples, drinking water, fresh water, and salt water samples). Sample throughput was estimated based on the time required to analyze a sample set. Performance parameters, such as ease of use and reliability, were evaluated based on documented observations of the operators.

Round 1 of the verification test took place over a two-week period at Battelle's laboratories in Columbus, Ohio, followed by a one-day period at Battelle's Ocean Sciences Laboratory in Duxbury, Massachusetts. Three types of samples were used in the verification test: quality control samples, performance test samples, and environmental water samples. The environmental water samples consisted of drinking water, well water, surface freshwater from rivers and a reservoir, and salt water from Massachusetts Bay. The surface freshwater sampling portion of the test was repeated, using the same water sources as in Round 1 of testing. In this Round 2 of testing, F-NTK kits were used that incorporated improved packaging for the reagents. The reference nitrate analysis in all testing was performed using a Dionex DX-500 ion chromatograph according to EPA Method 300.1.

Quality assurance (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 NECi F-NTK provides the reagents and equipment necessary for analyzing for nitrate and nitrite in environmental water samples and water-extracts of soil, plant tissue, and some food. The F-NTK uses an enzyme-based (nitrate reductase) nitrate testing method and contains no toxic or hazardous chemicals. With the F-NTK, nitrate can be analyzed in two ranges of 0.5 to 10 ppm nitrate-N (0.5 to 10 mg of nitrate-N per liter) or 0.05 to 1.0 ppm nitrate-N (0.05 to 1.0 mg of nitrate-N per liter). This verification test used the 0.5 to 10 ppm nitrate-N range. The F-NTK nitrate test kit includes three nitrate standards, which are developed by the user, and a precision color chart for estimating nitrate content. All the necessary tools for conducting the nitrate tests are supplied in the F-NTK, including a test tube rack. The test kit provides semi-quantitative estimates of nitrate-N content.

VERIFICATION OF PERFORMANCE

The F-NTK test kits sometimes showed widely different levels of color formation from nitrate standards when different reagent kits were used. The improved packaging for the F-NTK kits used in Round 2 appears to have resolved that problem. Unfortunately, performance of some of the kits was affected by this variability in Round 1, and only qualitative assessments could be made of some of the kit's performance characteristics, including precision, detection limit, effect of operator skill level, and effects of interferences.

Accuracy: Quantitative accuracy could not be assessed in Round 1 testing. In Round 2, the average percent bias of the current F-NTK test kit relative to the reference method was 28.9% with a technically trained Battelle operator, and 31.4% with a vendor operator, at levels of about 2 to 8 ppm nitrate-N in freshwater samples.

The F-NTK kits gave a result within 1 ppm of the reference result on 80 to 100% of laboratory performance samples with a non-technical operator, and 60 to 70% with a technical operator. The corresponding percentages for drinking water samples were 100% with the non-technical operator and 67 to 100% with the technical operator. For surface freshwater samples, the corresponding averages were 57% with a vendor operator and 62% with a Battelle technical operator. For saltwater samples, the F-NTK kits with both technical and non-technical operators correctly indicated that nitrate levels were below 1 ppm in 100% of the samples.

Linearity: In Round 1, no quantitative evaluation of linearity of response could be made. In Round 2, with surface freshwater samples containing about 2 to 8 ppm nitrate-N, the linear regression equation for F-NTK results with the vendor operator was: F-NTK ppm nitrate-N = $0.538 \times$ (Reference ppm) + 1.02 ppm, with a correlation coefficient of (r) of 0.779. The corresponding equation for results with the Battelle technical operator was: F-NTK ppm nitrate-N = $0.545 \times$ (Reference ppm) + 1.00 ppm, with a correlation coefficient of (r) of 0.789.

Precision: The precision of the F-NTK kits could not be evaluated quantitatively in most cases, because of frequent non-detect results. However, when detectable results were obtained the F-NTK kits typically gave the same result for each replicate analysis of a single sample.

Detection limit: The determination of the F-NTK detection limit was similarly hampered by variability in the response of the kits; but, in Round 2, 1-ppm levels were readily measured with the current version of the kits, which incorporates improved packaging of the reagents.

Interference effects: In Round 1 testing, the presence of high levels of iron, NaCl, sulfate, and acid appeared to result in greater overestimation of nitrate levels than did low levels of these interferents. However, no quantitation of interference effects could be made.

False positives/false negatives: Relative to the reference method, using 2 ppm nitrate-N as the decision level, the rate of false positives of the F-NTK test kit was 14% when used by the technical operator, and zero when used by the non-technical operator. The rate of false negatives was 32% with the technical operator and 38% with the non-technical operator.

Operator bias: The test results show no apparent effect of the operator skill level on F-NTK test results.

Other factors: Each F-NTK kit costs about $30.00, and includes five packets of reagents, each of which is capable of analyzing three standards and five samples. The F-NTK test kit was easy to use, easy to transport, and required no maintenance. Data completeness in the test was 100%. The approximate time of sample analysis is 45 minutes.

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