THE ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM

U.S. Environmental Protection Agency

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ETV Joint Verification Statement

TECHNOLOGY TYPE:	**PORTABLE CYANIDE ANALYZER**		
APPLICATION:	**DETECTING CYANIDE IN WATER**		
TECHNOLOGY NAME:	**Mini-Analyst Model 942-032**		
COMPANY:	**Orbeco-Hellige**		
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The U.S. Environmental Protection Agency (EPA) supports 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 (consisting of buyers, vendor organizations, and permitters), 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 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 seven 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 portable cyanide analyzers used to detect cyanide in water. This verification statement provides a summary of the test results for the Orbeco Mini-Analyst Model 942-032.

VERIFICATION TEST DESCRIPTION

The verification was based on comparing the cyanide concentrations of water samples determined by the Orbeco Mini-Analyst Model 942-032 with cyanide concentrations determined by a laboratory-based reference method (EPA Method 335.1, *Cyanides Amenable to Chlorination*). Two Orbeco Mini-Analyst Model 942-032s were tested independently between January 13 and February 4, 2003; and the results were compared to assess inter-unit reproducibility. Samples used in the verification test included quality control samples, performance test (PT) samples, lethal/near-lethal concentration samples, drinking water samples, and surface water samples. The results from the Orbeco Mini-Analyst Model 942-032 were compared with the reference method to quantitatively assess accuracy and linearity. Multiple aliquots of each test sample were analyzed separately to assess the precision of both the Orbeco Mini-Analyst Model 942-032 and the reference method. To determine the detection limit, a solution with a concentration of 0.010 milligram per liter (mg/L) was used. Seven non-consecutive replicate analyses of this solution were made to obtain precision data with which to determine the method detection limit (MDL). The Orbeco Mini-Analyst Model 942-032 was tested by a technical and a non-technical operator to assess operator bias. Sample throughput was estimated based on the time required to analyze a sample. Ease of use was based on documented observations by the operators and the Battelle Verification Test Coordinator. The Orbeco Mini-Analyst Model 942-032 was used in a field environment as well as in a laboratory setting to assess the impact of field conditions on performance.

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

TECHNOLOGY DESCRIPTION

The following description of the Orbeco Mini-Analyst Model 942-032 was provided by the vendor and does not represent verified information.

The Orbeco Mini-Analyst Model 942-032 is a portable colorimeter in which a sample and a reagent are mixed and analyzed photometrically to provide a quantitative determination of cyanide in the sample. The Orbeco Mini-Analyst Model 942-032 uses a photodiode detector, and results are reported on a liquid crystal display. Permanent calibrations for cyanide analysis are stored in the microprocessor memory. The Orbeco Mini-Analyst Model 942-032 comes with a hard cover carrying case, reagents for 50 samples, pH adjustment and dechlorination reagents, and four reaction vials. First, the samples are preserved to exactly 0.020 M sodium hydroxide. Then, 1 milliliter (mL) each of the buffer and 1.75 M hydrochloric acid, provided by Orbeco, are added to 100 mL of preserved sample. The pH is then adjusted to 6 and 7, as necessary. A 10.0-mL aliquot is taken from the pH-adjusted sample, and a capsule of powdered reagent is added before the sample vial containing the 10.0-mL sample and powdered reagent is shaken and set aside for two minutes. In the meantime, a liquid reagent solution is made up in a separate vial. At the appropriate time, this reagent is added to the original sample vial, and the vial is shaken. After a 15-minute color development period, the sample is placed into the Orbeco Mini-Analyst Model 942-032, and a cyanide concentration is displayed in micrograms per liter. However, for consistency with the reference laboratory results, all data within this report have been converted to milligrams per liter (mg/L). The dimensions of the Orbeco Mini-Analyst Model 942-032 are 6 x 4 x 2 inches, and it weighs 340 grams (12 ounces). The Orbeco Mini-Analyst Model 942-032 operates on four AA batteries. The list prices are $299.00 for the colorimeter and $67.50 for reagents adequate for approximately 50 water samples.

VERIFICATION OF PERFORMANCE

Accuracy: The biases for the Orbeco Mini-Analyst Model 942-032 ranged from 3 to 21% for the PT samples with concentrations ranging from 0.030 to 0.800 mg/L; 5 to 12% for the surface water samples; 3 to 100% for the drinking water samples from around the country; and 25 to 94% for the Columbus, OH, drinking water samples. Since the latter three types of water samples contained no detectable cyanide, they were fortified with 0.200 mg/L of cyanide to test the performance of the Orbeco Mini-Analyst Model 942-032 in water matrices.

Precision: The relative standard deviations ranged from 2 to 16% for the PT samples; 1 to 8% for the surface water samples; 1 to 25% for the drinking water samples from around the country; and 2 to 13% for the Columbus, OH, drinking water samples (except for the non-technical operator's results for the well water analyzed in the laboratory, which had RSDs over 100%).

Linearity: The non-technical operator's results from the Orbeco Mini-Analyst Model 942-032 for the PT samples (0.030 to 0.400 mg/L) plotted against the concentrations of the same samples as determined by the reference method gives the following regression equation:

y (non-technical operator results in mg/L)=0.797 (± 0.028) x (reference result in mg/L) + 0.007 (± 0.006) mg/L with r^2=0.991 and N=32.

The data for the technical operator gives the following regression equation:

y (technical operator results in mg/L)=0.820 (± 0.025) x (reference result in mg/L) + 0.010 (± 0.006) mg/L with r^2=0.993 and N=32.

where the values in parentheses represent the 95% confidence interval of the slope and intercept. Only the technical operator's intercept is significantly different from zero, and the r^2 values are both above 0.99. The linearity of the Orbeco Mini-Analyst Model 942-032 was not dependent on which operator was performing the analyses. The slope of the linear regression was significantly less than unity in both instances. This deviation from unity indicates a low bias in the results generated by the Orbeco Mini-Analyst Model 942-032 compared with the results produced by the reference method.

Method Detection Limit: The MDL was determined to be 0.004 mg/L for the Orbeco Mini-Analyst Model 942-032 when used by the non-technical operator and 0.005 mg/L when used by the technical operator.

Inter-Unit Reproducibility: A linear regression of the data for inter-unit reproducibility gives the following regression equation:

y (Unit #1 result in mg/L)=0.976 (± 0.008) x (Unit #2 result in mg/L) - 0.0009 (± 0.0012) mg/L with r^2=0.998 and N=136.

where the values in parentheses represent the 95% confidence interval of the slope and intercept. The slope is just slightly less than unity, and the intercept is not significantly different from zero. These data indicate that the two Orbeco Mini-Analyst Model 942-032s functioned very similarly to one another.

Lethal/Near-Lethal Dose Response: Samples at 50.0-, 100-, and 250-mg/L concentrations (close to what may be lethal if a volume the size of a typical glass of water was ingested) were prepared and analyzed by the Orbeco Mini-Analyst Model 942-032. Upon adding the reagents to the water sample, the color of the sample changed within five seconds to bright red and then progressed to a dark blue after about five minutes. The change was much more rapid than for any of the PT samples. The PT samples took about 30 seconds to produce a small change in the color of the sample and took the full 15-minute reaction time to reach its analysis color of a clear, light blue. When the samples with lethal/near-lethal concentrations were inserted into the Orbeco Mini-Analyst Model 942-032 after the full reaction time, the digital readout read "off scale." Even without using the Orbeco Mini-Analyst Model 942-032, the reagents and vials would be useful for a first responder seeking to find out whether a toxic level of cyanide is present in a drinking water sample. The presence of such concentrations could be confirmed within minutes by visual observation of the color development process.

Operator Bias: A linear regression of the data for the operator bias assessment gives the following regression equation:

y (non-tech result in mg/L)=0.933 (± 0.056) x (tech result in mg/L) - 0.002 (± 0.008) mg/L with r^2=0.890 and N=136.

where the values in parentheses represent the 95% confidence interval of the slope and intercept. The slope of this regression is less than 10% different from unity, indicating a slight difference in the results produced by the operators. The relatively low coefficient of variation was mostly due to the Flagstaff, AZ, samples. The technical operator generated detectable results, while the non-technical operator did not.

Field Portability: From an operational standpoint, the Orbeco Mini-Analyst Model 942-032 was easily transported to the field setting, and the samples were analyzed in the same fashion as they were in the laboratory. No functional aspects of the Orbeco Mini-Analyst Model 942-032 were compromised by performing the analyses in the field setting. However, performing analyses under extremely cold conditions (sample water temperatures between 4 and 6°C) negatively affected the performance of the Orbeco Mini-Analyst Model 942-032 cyanide reagents.

Ease of Use: The manufacturer recommends adjusting the pH of water samples to be analyzed by the Orbeco Mini-Analyst Model 942-032 to between 6.0 and 7.0. Since gaseous hydrogen cyanide can be released at a pH less than 9.00, this adjustment is not desirable from a safety standpoint, especially if lethal/near-lethal concentrations of cyanide are present. The sample preparation instructions were clear, but the liquid pyridine reagent had an offensive odor, and the granular reagent tablets were difficult to open. Also, the operators thought that it was inconvenient to keep track of the mixing and waiting times during the analysis.

Sample Throughput: Sample preparation, including measuring volumes and using reagents, took two to three minutes per sample. After performing the sample preparation, a 15-minute period of color development was required before sample analysis. Therefore, if only one sample is analyzed, it would take approximately 18 minutes. However, both operators were able to stagger the start of the color development period every two minutes for subsequent samples, so a typical sample set of 12 analyses took 45 to 50 minutes. Since the color development reaction takes place in reusable reaction vials, additional vials would have to be purchased to conveniently analyze large sample sets.

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