THE ENVIRONMENTAL TECHNOLOGY VERIFICATION







ETV Joint Verification Statement

TECHNOLOGY TYPE: Enzymatic Test Kit

APPLICATION: Detecting Chemical Warfare Agents, Carbamate

Pesticides, and Organophosphate Pesticides in Drinking

Water

TECHNOLOGY

NAME: EcloxTM-Pesticide Strips

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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 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 six technology areas under ETV, is operated by Battelle in cooperation with EPA's National Exposure Research Laboratory. The AMS Center evaluated the performance of the Severn Trent Services EcloxTM-Pesticide Strips. This verification statement provides a summary of the test results.

VERIFICATION TEST DESCRIPTION

The objective of this verification test was to evaluate the ability of the EcloxTM-Pesticide Strip to detect chemical agents, carbamate pesticides, and organophosphate (OP) pesticides as contaminants in drinking water (DW). This verification test assessed the performance of the EcloxTM-Pesticide Strip relative to accuracy; false positive and negative rates; precision; potential matrix and interference effects; and various operational factors including operator observations, ease of use, and sample throughput from both a technical and non-technical operators' perspective. The EcloxTM-Pesticide Strip was evaluated using VX, sarin (GB), and soman (GD) (chemical agents); aldicarb (carbamate pesticide); and dicrotophos (OP pesticide) in performance test (PT) and DW samples. Quality Control (QC) samples were also included as part of the test matrix to ensure the integrity of the test. PT samples included the individual contaminants spiked into American Society for Testing and Materials (ASTM) Type II deionized (DI) water at five different concentrations: the lethal dose concentration for each contaminant, along with dilutions at approximately 10, 100, 1,000, and 10,000 times less than the lethal dose. PT samples also included potential interferent samples containing a single concentration (10 times less than the lethal dose) of the contaminant of interest in the presence of calcium (Ca) and magnesium (Mg) spiked into ASTM Type II DI water, and humic and fulvic acids spiked into ASTM Type II DI water. Each interferent mixture was prepared at two concentration levels: near the upper limit of what would be expected in drinking water (250 milligrams per liter (mg/L) total concentration for Ca and Mg. 5 mg/L total concentration for humic and fulvic acids) and at a mid-low range of what would be expected (50 mg/L total concentration for Ca and Mg, 1 mg/L total concentration for humic and fulvic acids). Interferent PT samples were also analyzed without the addition of any contaminant. DW samples consisted of chlorinated filtered surface water, chlorinated unfiltered surface water, chlorinated filtered groundwater, and chloraminated filtered surface water collected from four geographically distributed municipal water sources (OH, NY, FL, and CA, respectively). All DW samples were dechlorinated prior to use. DW samples were analyzed before adding any contaminant and after fortification with each individual contaminant at 10 times less than the lethal dose of that contaminant. QC samples included method blank (MB) samples. All samples were tested in triplicate.

The lethal dose of each contaminant was determined by calculating the concentration at which 250 milliliters (mL) of water is likely to cause the death of a 70-kilogram (kg) person based on human oral LD₅₀ (lethal dose for half of the test subjects) data. Human oral LD₅₀ data were not available for aldicarb, so rat oral LD₅₀ data were used instead. Lethal dose values are provided in the contaminant results tables below. Samples were tested blindly by Battelle technical operators who were trained by other Battelle staff who had been trained by the vendor in the use of the EcloxTM-Pesticide Strip. Contaminants were tested individually, and stock solutions of each contaminant were prepared separately in ASTM Type II DI water. To minimize the loss of analytes to hydrolysis, contaminant stock solutions prepared in DI water were made on a daily basis. In some cases, reference solutions were prepared in ASTM Type II DI water using the stock solutions to prepare the test samples. In other cases, the actual stock solutions were submitted for concentration confirmation by the respective reference analysis.

A subset of the samples was also tested by a non-technical operator using the EcloxTM-Pesticide Strip. The non-technical operator was someone with little to no laboratory experience who would be representative of a first responder. For this test, the non-technical operator was a State of Ohio certified firefighter with Hazardous Waste Operations and Emergency Response (HAZWOPER) training. The non-technical operator was trained in the use of the EcloxTM-Pesticide Strip by another Battelle staff person who was trained by the vendor. Only MB samples and non-toxic control samples were analyzed as part of the operational factors assessment. As the EcloxTM-Pesticide Strip may be used by first-responders, its performance was evaluated under simulated first-response conditions by having the operator dressed in a Level B protective suit, neoprene latex gloves, boots, and a self-contained breathing apparatus (SCBA). The operator had prior experience working in personal protective equipment (PPE). One set of MB samples was also tested without the use of PPE. Ease of use from the perspective of the operator was documented both with and without the PPE.

QA oversight of verification testing was provided by Battelle and EPA. Battelle QA staff conducted a technical systems audit, a performance evaluation audit, and a data quality audit of 10% of the test data. Testing was conducted from November 2005 through February 2006. This verification statement, the full report on which it is based, and the test/QA plan for this verification test are all available at www.epa.gov/etv/centers/center1.html.

TECHNOLOGY DESCRIPTION

The following description of the EcloxTM-Pesticide Strips is based on information provided by the vendor. This technology description was not verified in this test.

The EcloxTM-Pesticide Strips are designed to give a qualitative (i.e., "yes/no") indication for the presence of organophosphate (OP), thiophosphate and carbamate pesticides. It is based on the inhibition of the enzyme acetylcholinesterase. The absence of pesticides turns the strip blue. In the presence of pesticides, the strip remains white.

Each EcloxTM-Pesticide Strip consists of a strip containing two disks, a smaller white disk and a larger pink disk covered with foil. After removing the strip from the packaging, the operator exposes the white disk only and dips it into the sample for one minute. In the next step, the operator removes the strip from the sample and removes the foil cover to expose the pink disk. The operator then folds the strip at the perforation and presses the disks together. This step, in which the disks are held together for three minutes, exposes the pink disk to the suspect test water sample. After the three minute holding time, the operator visually reads the color of the smaller disk. Two results are possible: a blue color indicates the absence of a pesticide and the white color indicates the presence of a pesticide.

The EcloxTM-Pesticide Strips are part of the EcloxTM portable field water quality assessment system, which detects intentional or accidental contamination of water. The EcloxTM system uses a luminometer to determine water toxicity and can be used to test for various contaminants in water. A package of 25 EcloxTM-Pesticide Strips may be purchased separately (from the EcloxTM system) for \$510.00.

VERIFICATION RESULTS

Accuracy was assessed by evaluating how often the EcloxTM-Pesticide Strip result was positive in the presence of a concentration above the limit of detection (LOD). Contaminant-only PT samples were used for this analysis. No LODs were provided for any of the contaminants with the exception of aldicarb, for which the vendor-provided LOD was 0.2 mg/L. For the other contaminants, all analyzed contaminant-only PT samples greater than the concentration level where consistent negative results were obtained were used for calculations. This level was defined at 0.021 mg/L for VX, 0.0020 mg/L for GB, and 0.0014 mg/L for GD.

A false positive response was defined as a response indicating the presence of a contaminant when the PT interferent or DW sample was not spiked with contaminant. A false negative response was defined as a response indicating the absence of a contaminant when the sample was spiked with a contaminant at a concentration greater than the EcloxTM-Pesticide Strip's LOD or consistent negative response level, as defined above. Spiked PT (contaminant and interferent) samples and spiked DW samples were included in the analysis.

The precision of three replicates of each sample set was assessed by calculating the overall number of consistent responses for all the sample sets. Operational aspects of the EcloxTM-Pesticide Strip's performance such as ease of use and sample throughput were evaluated through observations made during testing. Also addressed were qualitative observations of the verification staff from both the technical and non-technical operators' perspective.

VX Summary Table

Parameter		Matrix	VX Concentration	Number Detected/Number of Samples
	Contaminant- Only PT Samples	DI Water	2.1 mg/L ^(a)	3/3
			0.21 mg/L	3/3
			0.021 mg/L $^{(b)}$	0/3
Qualitative			0.0021 mg/L	0/3
Results			0.00021 mg/L	0/3
Rosuits	Interferent PT Samples	Humic and Fulvic Acids	0.21 mg/L	6/6
		Ca and Mg	0.21 mg/L	6/6
	DW Samples	DW	0.21 mg/L	12/12
Accuracy		100% (6 out of 6) of the contaminant-only PT samples gave positive results.		
False Positive Rate		No false positive results (0 out of 24) were observed during the testing with VX.		
False Negative Rate		No false negative results (0 out of 30) were observed during the testing with VX.		
Precision 100% (21 out of 21) of the sample sets showed consistent re among the individual replicates within each set during testin VX.				

GB Summary Table

Parameter		Matrix	GB Concentration	Number Detected/Number of Samples
	Contaminant- Only PT Samples	DI Water	$20 \text{ mg/L}^{(a)}$	3/3
			2.0 mg/L	3/3
			0.2 mg/L	3/3
Qualitative			0.02 mg/L	3/3
Results			0.002 mg/L $^{\rm (b)}$	0/3
Results	Interferent PT Samples	Humic and Fulvic Acids	2.0 mg/L	6/6
		Ca and Mg	2.0 mg/L	6/6
	DW Samples	DW	2.0 mg/L	12/12
Accuracy		100% (12 out of 12) of the contaminant-only PT samples gave positive results.		PT samples gave
False Positive Rate		No false positive results (0 out of 24) were observed during the testing with GB.		
False Negative Rate		No false negative results (0 out of 36) were observed during the testing with GB.		
Precision		100% (21 out of 21) of the sample sets showed consistent results among the individual replicates within each set.		

⁽a) Lethal dose
(b) Consistently negative results observed at and below this concentration

⁽a) Lethal dose (b) Consistently negative results observed at this concentration

GD Summary Table

Paran	ieter	Matrix	GD Concentration	Number Detected/Number of Samples
	Contaminant- Only PT Samples	DI Water	$1.4 \text{ mg/L}^{(a)}$	3/3
			0.14 mg/L	3/3
			0.014 mg/L	1/3 ^(c)
			0.0014 mg/L $^{(b)}$	0/3
Qualitative Results			0.00014 mg/L	0/3
	Interferent PT Samples	Humic and Fulvic Acids	0.14 mg/L	6/6
		Ca and Mg	0.14 mg/L	6/6
-	DW Samples	DW	0.14 mg/L	12/12
Accuracy 78% (7 out of 9) of the contaminant-only PT samples garresults.			amples gave positive	
False Positive Rate No false positive results (0 out of 24) were observed dutesting with GD.			served during the	
False Negative Rate		No false negative results (0 out of 33) were observed during the testing with GD; though two inconclusive results were observed for the 0.014 mg/L contaminant-only PT sample.		
Precision 95% (20 out of 21) of the sample sets showed consistent reamong the individual replicates within each set.				

⁽a) Lethal dose

Aldicarb Summary Table

Parameter		Matrix	Aldicarb Concentration	Number Detected/Number of Samples
	Contaminant- Only PT Samples	DI Water	$260 \text{ mg/L}^{(a)}$	3/3
			26 mg/L	3/3
			2.6 mg/L	0/3
Qualitative			0.26 mg/L $^{(b)}$	0/3
Results			0.026 mg/L	0/3
11000110	Interferent PT Samples	Humic and Fulvic	26 mg/L	6/6
		Acids	20 Hig/L	0/0
		Ca and Mg	26 mg/L	6/6
	DW Samples	DW	26 mg/L	12/12
Accuracy		50% (6 out of 12) of the contaminant-only PT samples gave positive results above the vendor-provided limit of detection for aldicarb (0.2 mg/L).		
False Positive Rate		No false positive results (0 out of 24) were observed during the testing with aldicarb.		
False Negative Rate Six out of the 36 samples yielded false negative results dure testing with aldicarb. The 2.6 mg/L and 0.26 mg/L contain PT samples showed negative responses.			C	
Precision		100% (21 out of 21) of the sample sets showed consistent results among the individual replicates within each set.		

⁽c) Two inconclusive results were observed; operator noted smaller disk to be white with blue around edges and could not categorize results as positive or negative result.

⁽a) Lethal dose (b) Vendor-provided limit of detection (LOD) for aldicarb is 0.2 mg/L.

Dicrotophos Summary Table

Parameter		Matrix	Dicrotophos Concentration	Number Detected/Number of Samples
	Contaminant- Only PT Samples	DI Water	1400 mg/L ^(a)	3/3
			140 mg/L	3/3
			14 mg/L (b)	0/3
Qualitative			1.4 mg/L	0/3
Results			0.14 mg/L	0/3
Results	Interferent PT Samples	Humic and Fulvic Acids	140 mg/L	0/6
		Ca and Mg	140 mg/L	1/6
	DW Samples	DW	140 mg/L	3/12
Accuracy		100% (6 out of 6) of the contaminant-only PT samples gave positive results.		
False Positive Rate		No false positive results (0 out of 24) were observed during the testing with dicrotophos.		
False Negative Rate		Twenty out of the 30 samples yielded false negative results during the testing with dicrotophos. These samples were the spiked		
		potential interferent and DW samples except for one 50 mg/L Ca and		
		Mg, three FL DW samples, three 140 mg/L contaminant-only PT samples, and three 1400 mg/L contaminant-only PT sample.		
Precision		95% (20 out of 21) of the sample sets showed consistent results		
		among the individual replicates within each set.		

⁽a) Lethal dose

Operational Factors:

Technical Operators

The EcloxTM-Pesticide Strips were used by one Battelle technician throughout testing with the pesticides and a different Battelle technician throughout testing with chemical agents. These technicians were trained by another technician, who had received training from the vendor by phone for one-half hour in the use of the kit. Both technicians had extensive laboratory experience. The written instructions, which are provided on a small piece of paper with the strips, consist of eight steps prior to reading the results of the test. During testing, the technicians were able to perform a test, which included all steps from opening the foil packet to reading the results, in an average of 5 minutes using the EcloxTM-Pesticide Strips.

Non-Technical Operator

Unspiked MB samples were tested by a non-technical operator, using the EcloxTM-Pesticide Strips, both with and without PPE. During testing with the PPE on, the samples were analyzed while the operator wore full PPE, consisting of a Level B suit, neoprene latex gloves, boots and SCBA. The SCBA was worn throughout the entire testing procedure by the non-technical operator (only during the tests in which PPE was to be donned) to represent the physical burden borne by a similarly outfitted first responder. However, the operator ran the air from the SCBA only part of the time during testing to conserve the tank. The non-technical operator, in or out of PPE, was able to use the EcloxTM-Pesticide Strips without any difficulty. There were no issues with the duration of the test or impact of wearing gloves during operation.

The Severn Trent Services EcloxTM-Pesticide Strips are highly portable since the test coupons are small, lightweight, securely packed in foil, and do not require additional reagents or extensive manipulation of test apparatus beyond a wristwatch or stopwatch to note time. Results are generally obtained within five minutes. These qualities make it suitable for use in a field or non-laboratory setting.

⁽b) Consistently negative results observed at and below this concentration

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