September 2006

# Environmental Technology Verification Report

## SEVERN TRENT SERVICES
## Eclox™-Pesticide Strips

Prepared by
Battelle

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The Business of Innovation

Under a cooperative agreement with

EPA U.S. Environmental Protection Agency

ETV ETV ETV

September 2006

# Environmental Technology Verification Report

## ETV Advanced Monitoring Systems Center

## Severn Trent Services
## Eclox™-Pesticide Strips

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## Notice

The U.S. Environmental Protection Agency (EPA), through its Office of Research and Development, has financially supported and collaborated in the extramural program described here. This document has been peer reviewed by the Agency. Mention of trade names or commercial products does not constitute endorsement or recommendation by the EPA for use.

# Foreword

The U.S. Environmental Protection Agency (EPA) is charged by Congress with protecting the nation's air, water, and land resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. To meet this mandate, the EPA's Office of Research and Development provides data and science support that can be used to solve environmental problems and to build the scientific knowledge base needed to manage our ecological resources wisely, to understand how pollutants affect our health, and to prevent or reduce environmental risks.

The Environmental Technology Verification (ETV) Program has been established by the EPA to verify the performance characteristics of innovative environmental technology across all media and to report this objective information to permitters, buyers, and users of the technology, thus substantially accelerating the entrance of new environmental technologies into the marketplace. Verification organizations oversee and report verification activities based on testing and quality assurance protocols developed with input from major stakeholders and customer groups associated with the technology area. ETV consists of six environmental technology centers. Information about each of these centers can be found on the Internet at http://www.epa.gov/etv/.

Effective verifications of monitoring technologies are needed to assess environmental quality and to supply cost and performance data to select the most appropriate technology for that assessment. Under a cooperative agreement, Battelle has received EPA funding to plan, coordinate, and conduct such verification tests for "Advanced Monitoring Systems for Air, Water, and Soil" and report the results to the community at large. Information concerning this specific environmental technology area can be found on the Internet at http://www.epa.gov/etv/centers/center1.html.

## Acknowledgments

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## List of Abbreviations

| | |
|---|---|
| AMS | Advanced Monitoring Systems |
| ASTM | American Society for Testing and Materials |
| ATEL | Aqua Tech Environmental Laboratories |
| CWA | chemical warfare agent |
| Ca | calcium |
| DI | deionized |
| DPD | diethyl-p-phenylene diamine |
| DW | drinking water |
| ECD | electron capture detection |
| EPA | U.S. Environmental Protection Agency |
| ETV | Environmental Technology Verification |
| GB | sarin |
| GC | gas chromatography |
| GD | soman |
| HAZWOPER | Hazardous Waste Operations and Emergency Response |
| HDPE | high density polyethylene |
| HMRC | Hazardous Materials Research Facility |
| ICP | inductively coupled plasma |
| kg | kilogram |
| L | liter |
| LC | liquid chromatography |
| $LD_{50}$ | lethal dose for half of test subjects |
| LOD | limit of detection |
| LRB | laboratory record book |
| MB | method blank |
| Mg | magnesium |
| mg/L | milligram per liter |
| mL | milliliter |
| MS | mass spectrometry |
| µMHO | micromho |
| NDR | negative differential resistance |
| NTU | nephelometric turbidity unit |
| OP | organophosphate |

| | |
|---|---|
| PE | performance evaluation |
| PPE | personal protective equipment |
| PT | performance test |
| QA | quality assurance |
| QC | quality control |
| QMP | quality management plan |
| SCBA | self-contained breathing apparatus |
| SM | standard method |
| SOP | standard operating procedure |
| TSA | technical systems audit |

# Chapter 1
# Background

The U.S. Environmental Protection Agency (EPA) supports the Environmental Technology Verification (ETV) Program to facilitate the deployment of innovative 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.

ETV works in partnership with recognized testing organizations; with stakeholder groups consisting 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 EPA's National Exposure Research Laboratory and its verification organization partner, Battelle, operate the Advanced Monitoring Systems (AMS) Center under ETV. The AMS Center recently evaluated the performance of the Severn Trent Services Eclox$^{TM}$-Pesticide Strips in detecting chemical agents, carbamate pesticides, and organophosphate (OP) pesticides in drinking water. Enzymatic test kits were identified as a priority technology category for verification through the AMS Center stakeholder process.

# Chapter 2
# Technology Description

The objective of the ETV AMS Center is to verify the performance characteristics of environmental monitoring technologies for air, water, and soil. This verification report provides results for testing the Severn Trent Services Eclox™-Pesticide Strips. Following is a description of the Eclox™-Pesticide Strips based on information provided by the vendor. The information provided below was not verified in this test.

The Eclox™-Pesticide Strips (Figure 2-1) are designed to give a qualitative (i.e., "yes/no") indication for the presence of 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 Eclox™-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 Eclox™-Pesticide Strips are part of the Eclox™ portable field water quality assessment system, which detects intentional or accidental contamination of water. The Eclox™ system uses a luminometer to determine water toxicity and can be used to test for various contaminants in water. A package of 25 Eclox™-Pesticide Strips may be purchased separately (from the Eclox™ system) for $510.00.

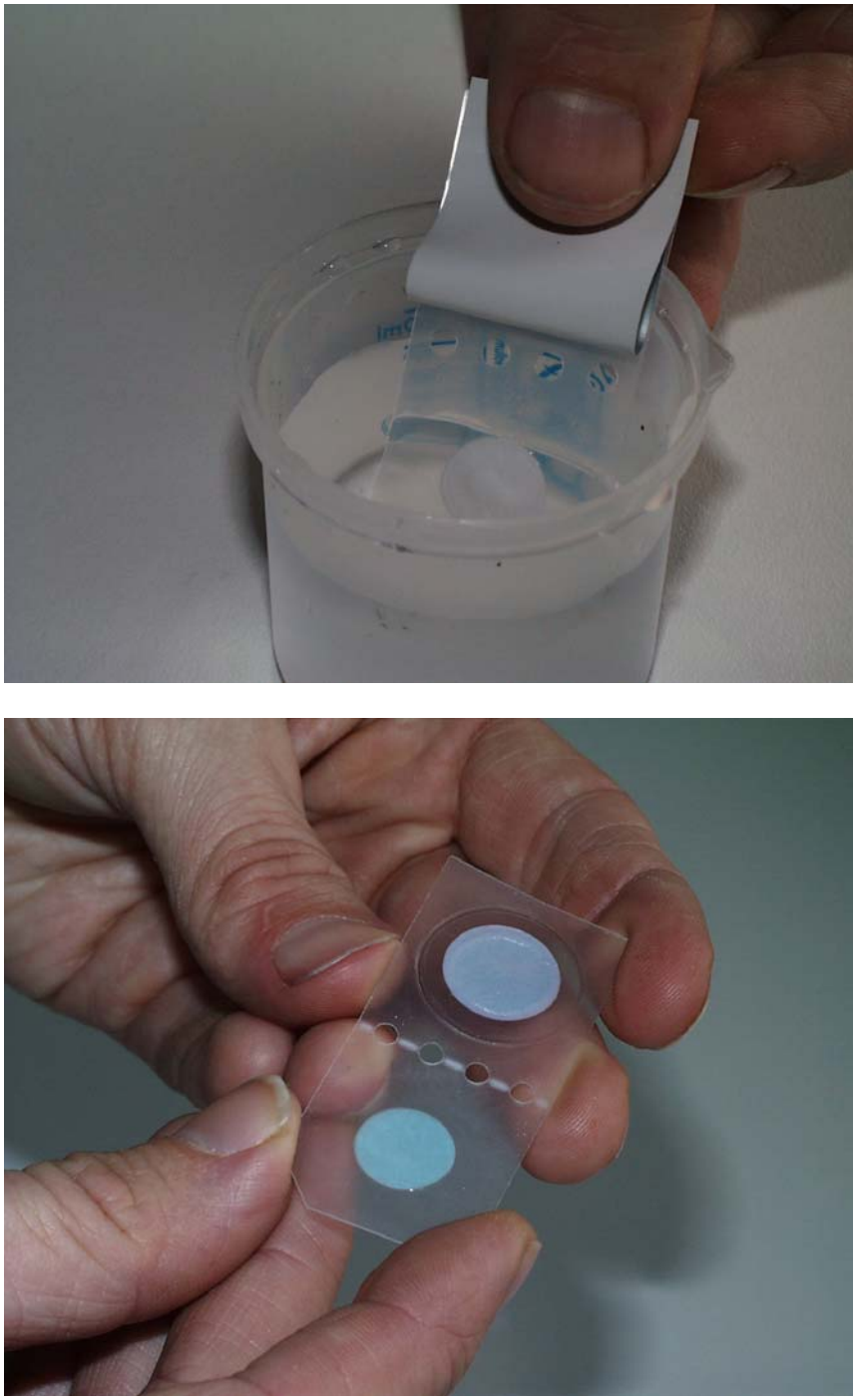

**Figure 2-1. The Eclox™-Pesticide Strip**
An operator introduces the strip into a water sample (top) and views the resulting color change of the smaller disk (bottom). In this case, the blue color of the smaller disk (shown toward the bottom of the lower picture) shows a negative response (indicating the absence of a pesticide in the test water sample).

# Chapter 3
# Test Design

## 3.1 Introduction

Enzymatic test kits, generally designed to be handheld and portable, detect the presence of chemical agents, carbamate pesticides, and/or OP pesticides by relying on the reaction of the cholinesterase enzyme. Under normal conditions, the enzyme reacts as expected with other chemicals present in the test kit. The activity of the enzyme is inhibited, however, by chemical agents, carbamate pesticides, and OP pesticides. The effects of this inhibition will then generally lead to a color change, indicating the presence or absence of these compounds.

The objective of this verification test was to evaluate the ability of the Eclox$^{TM}$-Pesticide Strips to detect chemical agents, carbamate pesticides, and OP pesticides in drinking water. This verification test assessed the performance of the Eclox$^{TM}$-Pesticide Strips relative to

- Accuracy
- False positive and negative rates
- Precision
- Potential matrix and interference effects
- Operational factors (operator observations, ease of use, and sample throughput).

## 3.2 Test Samples

This test evaluated the ability of the Eclox$^{TM}$-Pesticide Strips to detect VX, sarin (GB), soman (GD) (chemical agents); aldicarb (carbamate pesticide); and dicrotophos (OP pesticide) in performance test (PT) and drinking water (DW) samples. Quality Control (QC) samples were also included as part of the test matrix to ensure the integrity of the test. Contaminants were tested individually, and stock solutions of each contaminant were prepared separately in American Society for Testing and Materials (ASTM) Type II deionized (DI) water. Samples were prepared in the appropriate matrix using these stock solutions and analyzed on the same day. To minimize the loss of analytes to hydrolysis, contaminant stock solutions prepared in DI water were made on a daily basis. Chemical agent stock solutions were prepared twice daily,

once in the morning and once in the afternoon. Aliquots of each stock solution were diluted to the appropriate concentration using volumetric glassware and volumetric or calibrated pipettes. In some cases, reference solutions were prepared in ASTM Type II DI water using the stock solutions used to prepare the test samples. In other cases, the actual stock solutions were submitted for concentration confirmation by the respective reference analysis (Table 4-1). Aqua Tech Environmental Laboratories, Inc. (ATEL) of Marion, OH performed the physiochemical characterization for each type of DW sample along with reference analyses of the interferent solutions. All other reference analyses were performed at Battelle.

### *3.2.1 PT Samples*

PT samples were prepared separately in ASTM Type II DI water for each contaminant. The first type of PT samples consisted of ASTM Type II DI water spiked with the contaminant at five different concentrations: the lethal dose concentration given in Table 3-1 for each contaminant, along with dilutions at approximately 10, 100, 1,000, and 10,000 times less than the lethal dose. The contaminants were added individually to each spiked sample. 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 $LD_{50}$ (lethal dose for half of the test subjects) data.[1,2] Human oral $LD_{50}$ data were not available for aldicarb, so rat oral $LD_{50}$ data were used instead.[3] Each concentration level for the PT samples was analyzed in triplicate by the Eclox™-Pesticide Strips.

In addition to the contaminant-only PT samples described above, a second type of PT sample was a potential interferent sample. Three replicates of each interferent PT sample were analyzed by the Eclox™-Pesticide Strips to determine the susceptibility of the strips to these commonly found interferents in DW. One interferent PT sample contained calcium (Ca) and magnesium (Mg) from carbonates spiked into ASTM Type II DI water, and the other contained humic and fulvic acids isolated from the Elliot River (obtained from the International Humic Substances Society) 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 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). These spiked interferent levels were confirmed through analysis of aliquots by ATEL. Also, each contaminant was added to these samples, along with the potential interferent, at a concentration consistent with a 10x dilution of the lethal dose. The resulting samples were analyzed in triplicate by the Eclox™-Pesticide Strips. Table 3-2 lists the PT samples analyzed in this verification test for each contaminant.

**Table 3-1. Lethal Dose of Target Contaminants**

| Contaminant (common name) | Oral Lethal Dose Concentration | Contaminant Class |
|---|---|---|
| VX | 2.1 milligrams/liter (mg/L) | Chemical agent |
| GB (sarin) | 20 mg/L | Chemical agent |
| GD (soman) | 1.4 mg/L | Chemical agent |
| aldicarb | 260 mg/L | Carbamate pesticide |
| dicrotophos | 1400 mg/L | Organophosphate pesticide |

**Table 3-2. Performance Test Samples**

| Type of PT Sample | Sample Characteristics | Concentrations |
|---|---|---|
| Contaminant-only | Contaminants in DI water | VX: 2.1 to 0.00021 mg/L<br>GB: 20 to 0.002 mg/L<br>GD: 1.4 to 0.00014 mg/L<br>aldicarb: 260 to 0.026 mg/L<br>dicrotophos: 1400 to 0.14 mg/L |
| Interferent | Contaminants in 1 mg/L humic and fulvic acids | VX: 0.21 mg/L<br>GB: 2.0 mg/L<br>GD: 0.14 mg/L<br>aldicarb: 26 mg/L<br>dicrotophos: 140 mg/L |
| | Contaminants in 5 mg/L humic and fulvic acids | |
| | Contaminants in 50 mg/L Ca and Mg | |
| | Contaminants in 250 mg/L Ca and Mg | |

### *3.2.2 DW Samples*

Table 3-3 lists the DW samples analyzed for each contaminant in this test. DW samples were collected from four geographically distributed municipal sources (Ohio, New York, California, and Florida) to evaluate the performance of the Eclox™-Pesticide Strips with various DW matrices. These samples varied in their source, treatment, and disinfection process. All samples had undergone either chlorination or chloramination disinfection prior to receipt. Samples were collected from water utility systems with the following treatment and source characteristics:

- Chlorinated filtered surface water source
- Chlorinated unfiltered surface water source
- Chlorinated filtered groundwater source
- Chloraminated filtered surface water source

Approximately 175 liters (L) of each of the DW samples were collected in pre-cleaned, translucent, low-density polyethylene containers. . After sample collection, an aliquot of each DW sample was sent to ATEL to determine the following water quality parameters: concentration of trihalomethanes, haloacetic acids, total organic halides, Ca and Mg, pH, conductivity, alkalinity, turbidity, organic carbon, and hardness. All DW samples were dechlorinated prior to their use with sodium thiosulfate pentahydrate to prevent the degradation of the target contaminants by chlorine. The dechlorination of the DW was qualitatively confirmed by adding a diethyl-p-phenylene diamine (DPD) tablet to an aliquot of DW. If the water did not turn pink, the dechlorination process was successful. If the water did turn pink, additional dechlorinating reagent was added and the dechlorination confirmation procedure repeated. Each DW sample was analyzed before addition of contaminant, as well as after fortification with each individual contaminant at a single concentration level (10x dilution of the lethal dose). Aliquots of each contaminant stock solution were diluted with DW samples to the appropriate concentration. Each sample was tested in triplicate.

**Table 3-3. Drinking Water Samples**

| Drinking Water Sample Description | | | Contaminant Concentrations |
|---|---|---|---|
| **Water Utility** | **Water Treatment** | **Source Type** | |
| Columbus, Ohio (OH DW) | chlorinated filtered | surface | VX: 0.21 mg/L<br>GB: 2.0 mg/L<br>GD: 0.14 mg/L<br>aldicarb: 26 mg/L<br>dicrotophos: 140 mg/L |
| New York City, New York (NY DW) | chlorinated unfiltered | surface | |
| Orlando, Florida (FL DW) | chlorinated filtered | ground | |
| Metropolitan Water District of Southern California (CA DW) | chloraminated filtered | surface | |

### *3.2.3 QC Samples*

QC samples included method blank (MB) samples consisting of ASTM Type II DI water. All MB QC samples were exposed to sample preparation and analysis procedures identical to the test samples. The MB samples were used to ensure that no sources of contamination were introduced in the sample handling and analysis procedures. At least 10% of the test samples (seven samples for each contaminant) were MB samples. All of the test samples and MB samples were analyzed blindly by the operator in that the samples used for analysis were prepared by someone other than the operator and were marked with non-identifying numbers.

### *3.2.4 Operational Factors*

#### *3.2.4.1 Technical Operator*

All of the test samples were analyzed by a technical operator who was trained by other Battelle staff who had been trained by the vendor. Operational factors such as ease of use and sample throughput were evaluated based on observations recorded by the technical operator and the

Verification Test Coordinator. Operational factors were noted during the laboratory portions of the verification test. These observations were summarized to describe the operational performance of the Eclox™-Pesticide Strips in this verification.

*3.2.4.2 Non-Technical Operator*

A subset of the samples was also tested by a non-technical operator using the Eclox™-Pesticide Strips. 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 Eclox™-Pesticide Strips by another Battelle staff person who was trained by the vendor. Since many of the contaminants being tested are highly toxic and unsafe to be handled outside of a special facility, MB samples were analyzed as part of the operational factors assessment. Because no samples spiked with the contaminants of interest were used, only the operational aspects of the Eclox™-Pesticide Strips were evaluated with the non-technical operator. As the Eclox™-Pesticide Strips may be used by first-responders, its performance was evaluated under simulated first-response conditions by having the operator don 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.

## 3.3 Verification Schedule

The verification test of the Eclox™-Pesticide Strips took place from November 2005 through February 2006 at Battelle facilities in Columbus and West Jefferson, Ohio.

## 3.4 Test Procedure

### *3.4.1 Test Sample Preparation and Storage*

All testing for this verification test was conducted within Battelle laboratories. Aldicarb and dicrotophos samples were tested at Battelle's Columbus laboratories, while VX, GB and GD samples were tested at Battelle's Hazardous Materials Research Center (HMRC) facility in West Jefferson, OH. Appropriate safety guidelines associated with each laboratory were followed throughout the verification test. Samples were prepared fresh each day from stock solutions in DI water, an interferent matrix, or a DW matrix. Sample solutions were prepared to the specified concentration based on the concentration of the stock solution, which was confirmed through reference analysis. Test solutions were prepared in 1 L quantities. Appropriate aliquots of this sample preparation were used for each test sample. Triplicate samples of 25 mL were taken from the same sample preparation. Each sample was placed in its own container and labeled only with a sample identification number that was also recorded in a laboratory record book (LRB) along with details of the sample preparation.

### *3.4.2 Test Sample Analysis Procedure*

The Eclox™-Pesticide Strips have no requirements for the level of experience necessary to operate the kit. For each sample, the operator opened the foil packet to remove the contents, keeping the strip while disposing of the packaging (foil and wadding). Though the written instructions direct the operator to keep the foil packet for use later in the test, the vendor provided training did not make any use of the foil packet.

For each test sample, the operator dispensed approximately 25 mL of the test solution into a 50 mL beaker. This amount provided sufficient volume for the next step in which the operator, after folding back the foil covering the white disk (but leaving the larger pink disk covered), completely exposed the white disk (the smaller of the two disks on the Eclox™-Pesticide Strip) to the test solution. The operator submerged the white disk in the test solution for one minute, noting time by use of a wristwatch or stopwatch.

After removing the white disk from the test solution, the operator removed the protective foil cover from the pink disk. Then, the operator folded the strip in half along the perforations and pressed the white disk against the pink disk. The strip was then inserted into a supplied clip and held in the operator's hands for three minutes. In the written instructions, the operator is directed to place the clipped strip into the foil packet for the three minutes, but as noted above this process was not directed to be followed when the operators were trained by the vendor.

### *3.4.3 Drinking Water Characterization*

An aliquot of each DW sample, collected as described in Section 3.2.2, was sent to ATEL to determine the following water quality parameters: turbidity; concentration of dissolved and total organic carbon; conductivity; alkalinity; pH; concentration of Ca and Mg; hardness; and concentration of total organic halides, trihalomethanes, and haloacetic acids. Table 3-4 lists the characterization data from the four water sample types used in this verification test. Water samples were collected and water quality parameters were measured by ATEL in June 2005, while verification testing was tested with the DW between November 2005 and February 2006. The time delay between collection and testing was due to the fact that the water samples were collected for use during a separate ETV test conducted prior to this one. Because of this, an aliquot of each DW was tested by ATEL again in January 2006 to verify some of the parameters with the most potential to change over time. Note that dissolved organic carbon was not retested as this result was verified by the total organic carbon results, additionally the total organic halides and calcium and magnesium were not verified as there was no reason to expect a change in these parameters. The concentrations of most water quality parameters were similar; however, there was a decrease in levels of volatile compounds such as trihalomethanes and haloacetic acids over this time-period.

**Table 3-4. ATEL Water Quality Characterization of Drinking Water Samples**

| | | | Columbus, OH (OH DW) | | New York City, NY (NY DW) | | Orlando, FL (FL DW) | | MWD [b], CA (CA DW) | |
|---|---|---|---|---|---|---|---|---|---|---|
| **Parameter** | **Unit** | **Method** | **2005** | **2006** | **2005** | **2006** | **2005** | **2006** | **2005** | **2006** |
| Turbidity | NTU[a] | EPA 180.1[4] | 0.1 | 0.6 | 1.1 | 1.3 | 0.5 | 0.1 | 0.1 | 0.2 |
| Dissolved Organic Carbon | mg/L | SM 5310[5] | 2.1 | NA | 1.1 | NA | 1.6 | NA | 2.9 | NA |
| Total Organic Carbon | mg/L | SM 5310[5] | 2.1 | 2.3 | 1.6 | 4.1 | 1.7 | 2.1 | 2.5 | 2.7 |
| Specific Conductivity | µMHO[c] | SM 2510[5] | 572 | 602 | 84 | 78 | 322 | 325 | 807 | 812 |
| Alkalinity | mg/L | SM 2320[5] | 40 | 44 | 14 | 12 | 142 | 125 | 71 | 97 |
| pH | | EPA 150.1[6] | 7.6 | 7.4 | 6.9 | 6.8 | 8.5 | 7.6 | 8.0 | 7.9 |
| Calcium | mg/L | EPA 200.8[7] | 33 | NA | 5.6 | NA | 8.8 | NA | 45 | NA |
| Magnesium | mg/L | EPA 200.8[7] | 7.7 | NA | 1.3 | NA | 43 | NA | 20 | NA |
| Hardness | mg/L | EPA 130.2[8] | 118 | 107 | 20 | 26 | 143 | 130 | 192 | 182 |
| Total Organic Halides | µg/L | SM 5320[5] | 220 | NA | 82 | NA | 300 | NA | 170 | NA |
| Trihalomethanes | µg/L/ analyte | EPA 524.2[9] | 74.9 | 16.6 | 39.0 | 23.1 | 56.4 | 41.8 | 39.2 | 24.1 |
| Haloacetic Acids | µg/L/ analyte | EPA 552.2[10] | 32.8 | <6.0 | 39.0 | <6.0 | 34.6 | <6.0 | 17.4 | <6.0 |

[a] NTU = Nephelometric turbidity unit.
[b] MWD = Metropolitan Water District of Southern California
[c] µMHO = micromho

# Chapter 4
# Quality Assurance/Quality Control

QA/QC procedures were performed in accordance with the quality management plan (QMP) for the AMS Center [11] and the test/QA plan [12] for this verification test.

QC procedures as noted in the reference methods or laboratory's operating procedures were followed in confirming analyses of stock or reference solutions of contaminants and interfering compounds and in characterizing the DW. The reference methods for this verification test are listed in Table 4-1. A summary of the QC samples and acceptance criteria associated with each method is presented in Table 7 in the test/QA plan. [12]

## 4.1 Sample Chain-of Custody Procedures

Sample custody was documented throughout collection, shipping, and analysis of the samples. Sample chain-of-custody procedures were in accordance with ASAT. I-009-Draft, *Standard Operating Procedure for Sample Chain-of-Custody*. The chain-of-custody forms summarized the samples collected and analyses requested and were signed by the person relinquishing samples once that person had verified that the custody forms were accurate. The original sample custody forms accompanied the samples; the shipper kept a copy. Upon receipt at the sample destination, sample custody forms were signed by the person receiving the samples once that person had verified that all samples identified on the custody forms were present in the shipping container.

## 4.2 QC Samples

The QC measures for the reference methods included the analysis of a MB sample with the analyses of the reference or stock solution. MB samples were analyzed to ensure that no sources of contamination were present. If the analysis of an MB sample indicated a concentration above the minimum detection limit for the confirmatory instrument, contamination was suspected. Any contamination source(s) were corrected, and proper blank readings were achieved, before proceeding with the analyses. In general, a matrix spike or laboratory fortified spike sample was also analyzed. Average acceptable recoveries for these samples were between 70 and 150%. Samples outside of the acceptable range were generally flagged and rerun once the QC acceptance criteria had been met. QC samples were run with every batch of 1 to 20 samples. Specific QC samples and acceptance criteria associated with each method can be found in the appropriate reference (see Table 4-1). No QC samples were provided with the Eclox$^{TM}$-Pesticide

Strips. MB samples were run as part of the verification test (Section 3.2.3). No contaminants were detected in any of the 32 method blank samples, which were analyzed in triplicate with the Eclox™-Pesticide Strips.

### 4.3 Equipment/Calibration

The instruments used for the reference analyses were calibrated per the standard reference methods being used to make each measurement or the standard operation procedures (SOPs) of the analysis laboratory. Instruments used in the reference analyses for this test included gas chromatography-mass spectrometry (GC-MS), liquid chromatography-mass spectrometry (LC-MS), pH electrodes, inductively coupled plasma-mass spectrometry (ICP-MS), and gas chromatography with electron capture detector (GC-ECD). All calibrations were documented by Battelle in the project LRB. Calibration of mass spectrometers involved a 4- to 8-point calibration curve covering the range of concentrations of the reference solutions to be analyzed. Calibration of each reference instrument was performed as frequently as required by the reference method guidelines.

Pipettes used during solution preparation were maintained and calibrated as required by Battelle SOPs (i.e., minimum of every 6 months). Pipettes were checked and either recalibrated or replaced if they were found out of calibration over the course of testing.

### 4.4 Characterization of Stock Solutions

During testing, aliquots of the stock solutions used for sample preparation were submitted for concentration confirmation via the respective methods. The measured concentration and the reference method used are listed in Table 4-1 for each contaminant and interferent. Average measured recovery and standard deviation are given in cases where more than two samples were tested. Recovery (%R) is calculated by the following equation:

$$\%R = \frac{C}{A} \times 100 \quad (1)$$

where $C$ is the measured concentration (or average measured concentration if more than one sample was tested) and $A$ is the expected concentration of the contaminant or interferent in solution. For aldicarb and dicrotophos, aliquots at two different concentration levels were confirmed through reference analysis. The %R, listed in Table 4-1, represents the average of the %R across both concentration levels for those compounds. Table 4-1 shows that %R values ranged from 85% to 123% across all analytes and interferents.

Contaminant stock solutions were prepared and tested individually. Interferent stock solutions contained multiple analytes in the same solution (e.g., calcium and magnesium or humic and fulvic acids together). Up to four aliquots of each stock solution were analyzed over the course of the verification test. In the case of VX, extra aliquots were analyzed and all were reported in

Table 4-1. Aliquots were preserved or extracted on the day of preparation and stored as prescribed by the standard method.

**Table 4-1. Reference Methods for Target Contaminants and Interferents**

| Target Analyte/Interferent | Reference Method (Instrumentation) | Number of Observations | Expected Concentrations (mg/L) | Average Measured Concentration (mg/L) ± SD | Recovery (%R) ± SD |
|---|---|---|---|---|---|
| VX | Battelle Internally Developed Method (LC-MS) | 10 | 2.1 | 2.1 ± 0.1 | 101 ± 5 |
| GB (sarin) | HMRC-IV-118-05 [13] (GC-MS) | 4 | 20.0 | 17.0 ± 1.4 | 85 ± 7 |
| GD (soman) | HMRC-IV-118-05 [13] (GC-MS) | 4 | 1.4 | 1.7 ± 0.05 | 121 ± 4 |
| aldicarb | SOP for Analysis of Water Sample Extracts for Type 1 Analytes by Liquid Chromatography/Mass Spectrometry [14] (LC-MS) | 2 | 26.0 | 34 | 123 ±7 [a] |
| | | 2 | 260 | 303 | |
| dicrotophos | SOP for Extracting and Preparing Water Samples for Analysis of Dicrotophos, Mevinphos, and Dichlorovos [15] (GC-MS) | 4 | 140 | 157 ± 24 | 108 ± 17 [a] |
| | | 1 | 1400 | 1326 | |
| calcium (Ca) | EPA 200.8 [7] (ICP-MS) | 1 | 125 | 140 | 112 |
| magnesium (Mg) | EPA 200.8 [7] (ICP-MS) | 1 | 125 | 130 | 104 |
| Humic and fulvic acids | Standard Method 5310 [5] Combustion Infrared NDR | 1 | 1.0 | 0.9 | 90 |

[a] Average of two concentration levels

## 4.5 Audits

### *4.5.1 Performance Evaluation Audit*

The concentration of the standards used to prepare the samples fortified with contaminants and potential interfering compounds was confirmed by analyzing standards prepared in ASTM Type II DI water from two separate commercial vendors using the reference methods noted in Table 4-1. The standards from one vendor were used during the verification test, while the standards from the second vendor were used exclusively to confirm the accuracy of the standards from the first vendor.

Given the lack of alternate sources for humic and fulvic acids as well as the security requirements for the chemical agents (VX, GB, and GD) used in this verification test, PE audits were not performed for these contaminants. PE audits were done for all remaining compounds when more than one source of the contaminant or potential interfering compounds was available. PE audits were performed only on compounds used to prepare test samples. Agreement of the standards within 25% (percent difference) was required for the measurements to be considered acceptable. The percent difference (%D) between the measured concentration of the PE sample and the nominal concentration of that sample was calculated using the following equation:

$$\%D = \frac{M}{A} \times 100 \quad (2)$$

where $M$ is the absolute value of the difference between the measured and the expected concentration, and $A$ is the expected concentration. The results of the PE samples are given in Table 4-2. All %D values were within the 25% acceptable tolerance.

**Table 4-2. Performance Evaluation Samples and Percent Difference**

| Contaminant | Expected Concentration (mg/L) | Measured Concentration (mg/L) | Percent Difference (%) |
|---|---|---|---|
| aldicarb | 50 | 57 | 14 |
| dicrotophos | 1000 | 1103 | 10 |
| Ca | 1000 | 890 | 11 |
| Mg | 1000 | 990 | 1 |

### *4.5.2 Technical Systems Audit*

The Battelle Quality Manager conducted technical systems audits (TSAs) in November 2005 (11/01, 11/11, 11/16, 11/18), December 2005 (12/01, 12/29), and January 2006 (01/30) to ensure that the verification test was performed in accordance with the AMS Center QMP, [11] the test/QA plan, [12] published reference methods, and any SOPs used by Battelle. As part of the audit, the Battelle Quality Manager reviewed the reference methods, compared actual test procedures to those specified or referenced in the test/QA plan, and reviewed data acquisition

and handling procedures. The Battelle Quality Manager also observed testing in progress and the reference method sample preparation and analysis, inspected documentation, and reviewed the LRBs used to record testing results. The Battelle Quality Manager also checked calibration certifications and conferred with Battelle staff. Observations and findings from this audit were documented and submitted to the Battelle Verification Test Coordinator for response. No major findings were reported from the audits. The records concerning the TSA are permanently stored with the Battelle Quality Manager.

#### *4.5.3 Audit of Data Quality*

At least 10% of the data acquired during the verification test was audited. The Battelle Quality Manager traced the data from initial acquisition, through reduction and statistical comparisons, to final reporting. All calculations performed on the data undergoing the audit were checked.

### 4.6 QA/QC Reporting

Each assessment and audit was documented in accordance with Section 3.3.4 of the AMS Center QMP. [11] Once the assessment report was prepared, the Battelle Verification Test Coordinator responded to each potential problem and implemented any necessary follow-up corrective action. The Battelle Quality Manager ensured that follow-up corrective action was taken. The results of the TSA were sent to the EPA.

### 4.7 Data Review

Records generated in the verification test were reviewed before they were used to calculate, evaluate, or report verification results. Table 4-3 summarizes the types of data recorded. The review was performed by a technical staff member involved in the verification test but not the staff member who originally generated the record. The person performing the review added his/her initials and the date to a hard copy of the record being reviewed.

**Table 4-3. Summary of Data Recording Process**

| Data to Be Recorded | Responsible Party | Where Recorded | How Often Recorded | Disposition of Data |
|---|---|---|---|---|
| Dates, times, and details of test events | Battelle | ETV laboratory record book or data recording forms | Start/end of test procedure, and at each change of a test parameter | Used to organize and check test results and manually incorporated into data spreadsheets as necessary |
| Sample preparation (dates, concentrations, etc.) | Battelle | ETV laboratory record books | When each solution was prepared | Used to confirm the concentration and integrity of the samples analyzed |
| Enzymatic test kit procedures and sample results | Battelle | ETV data sheets and laboratory record book | Throughout test duration | Manually incorporated into data spreadsheets for statistical analysis and comparisons |
| Reference method sample preparation | Battelle | ETV laboratory record book | Throughout sample preparation | Used to demonstrate validity of samples submitted for reference measurements |
| Reference method procedures, calibrations, QA, etc. | Battelle or subcontract laboratory | Laboratory record book or data recording forms | Throughout sampling and analysis processes | Retained as documentation of reference method performance |
| Reference method analysis results | Battelle or subcontract laboratory | Electronically from reference analytical method | Every sample analysis | Converted to spreadsheets for calculations |

# Chapter 5
# Statistical Methods and Reported Parameters

The Eclox™-Pesticide Strips were evaluated for qualitative results (i.e., positive/negative responses to samples). All data analyses were based on these qualitative results. QC and MB samples were not included in any of the analyses.

## 5.1 Accuracy

Accuracy was assessed by evaluating how often the results of Eclox™-Pesticide Strips were positive in the presence of a concentration above the limit of detection (LOD). Contaminant-only PT samples were used for this analysis. An overall percent agreement was determined by dividing the number of positive responses by the overall number of analyses of contaminant-only PT samples greater than the Eclox™-Pesticide Strip's LOD (see Equation 3). If the LOD was not known or available, then all analyzed contaminant-only PT samples greater than the concentration level where consistent negative results were obtained were used.

$$\textit{Accuracy (\% Agreement)} = \frac{\textit{\# of positive contaminant only PT samples}}{\textit{total \# of contaminant only PT samples}} \times 100 \qquad (3)$$

## 5.2 False Positive/False Negative Rates

A false positive response was defined as a response indicating the presence of a contaminant when the ASTM Type II DI water (including interferent samples) or DW sample was not spiked with contaminant.

A false positive rate was reported as the number of false positive results out of the total number of unspiked samples (Equation 4). 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 LOD for the Eclox™-Pesticide Strips (as defined above). Spiked PT (contaminant and interferent) samples and spiked DW samples were included in the analysis. Contaminant-only PT samples above the LOD (or the level at which consistent negative responses are obtained if the LOD was not known) were included in the analysis. A false negative rate was evaluated as the number of false negative results out of the total number of spiked samples for a particular contaminant (Equation 5). Inconclusive results were not considered positive or negative (so the total number of unspiked or spiked samples was decreased accordingly).

$$\textit{False Positive Rate} = \frac{\textit{\# of positive results}}{\textit{total \# of unspiked samples}} \quad (4)$$

$$\textit{False Negative Rate} = \frac{\textit{\# of negative results}}{\textit{total \# of spiked samples}} \quad (5)$$

### 5.3 Precision

Precision measures the repeatability and reproducibility of the responses of the Eclox™-Pesticide Strips. The precision of three replicates of each sample set was assessed. Responses were considered inconsistent if one or more of the three replicates differed from the response of the other samples in the replicate set. The precision for the Eclox™-Pesticide Strip was assessed by calculating the overall number of consistent responses for all the sample sets. The results are reported as the percentage of consistent responses out of all replicate sets (Equation 6).

$$\textit{Precision (\% Consistent results)} = \frac{\textit{\# of consistent responses of replicate sets}}{\textit{total \# of replicate sets}} \times 100 \quad (6)$$

### 5.4 Potential Matrix and Interferent Effects

The potential effect of the DW matrix on the performance of the Eclox™-Pesticide Strips was evaluated qualitatively by comparing the results for the spiked and unspiked DW samples to those for the PT samples spiked with the contaminant at 10 times less than the lethal dose. Similarly, the potential effect of interferent PT samples was also evaluated. The results indicating the correct or incorrect reporting of the presence of a contaminant were evaluated. The findings are reported and discussed in Section 6.4.

### 5.5 Operational Factors

Operational aspects of the performance of the Eclox™-Pesticide Strips, such as ease of use and sample throughput, were evaluated through observations made during testing. Also addressed are the qualitative observations of the verification staff pertaining to the performance of the Eclox™-Pesticide Strips from both the technical and non-technical operators' perspective.

# Chapter 6
# Test Results

The results for the Severn Trent Services Eclox™-Pesticide Strips are discussed in the following sections.

## 6.1 Accuracy

Accuracy was determined using contaminant-only PT samples with concentrations equal to or above the vendor-provided LOD. No LODs were provided by the vendor for the target contaminants with the exception of aldicarb. If no LOD was provided, only concentrations above which consistent negative results were obtained were used in the calculation. This level was defined at 0.021 mg/L for VX, 0.0020 mg/L for GB, and 0.0014 mg/L for GD. The concentrations used for accuracy testing are denoted in Table 6-1.

Inconclusive results, which occurred when the operator could neither discern the color of the smaller disk as entirely blue (negative for the presence of a contaminant) nor entirely white (positive for the presence of a contaminant), may indicate that the tested concentrations may be around the LOD for the particular contaminant tested. Two inconclusive results occurred during the testing with GD at the concentration of 0.014 mg/L in DI water. In these tests, the operator observed the smaller disk to be white with blue color around the edges.

A LOD of 0.2 mg/L for aldicarb was provided by the vendor. For this reason, accuracy is determined using the PT samples at concentrations of 0.26 mg/L aldicarb and above as shown in Table 6-1.

## 6.2 False Positive/False Negative Rates

Contaminant-only PT samples, interferent PT samples, and DW samples were evaluated to determine false positive and false negative results for the Eclox™-Pesticide Strips. A false positive response was defined as a positive result when the contaminant was not spiked into the sample. A false negative response was defined as a negative result when the sample was spiked with a contaminant at a concentration greater than the level where consistent negative responses were obtained (see Section 6.1). Tables 6-2a through 6-2e present the false positive and false negative responses for VX, GB, GD, aldicarb, and dicrotophos, respectively. The number of positive and negatives samples out of the total replicates analyzed is presented in each table.

**Table 6-1. Contaminant-Only PT Sample Results**

| Contaminant | Concentration (mg/L) | Positive Results Out of Total Replicates | Accuracy |
|---|---|---|---|
| VX | 2.1 [a] | 3/3 | **100% (6/6)** |
| | 0.21 | 3/3 | |
| | 0.021 [b] | 0/3 | |
| | 0.0021 [b] | 0/3 | |
| | 0.00021 [b] | 0/3 | |
| GB | 20 [a] | 3/3 | **100% (12/12)** |
| | 2.0 | 3/3 | |
| | 0.20 | 3/3 | |
| | 0.020 | 3/3 | |
| | 0.0020 [b] | 0/3 | |
| GD | 1.4 [a] | 3/3 | **78% (7/9)** |
| | 0.14 | 3/3 | |
| | 0.014 | 1/3 [c] | |
| | 0.0014 [b] | 0/3 | |
| | 0.00014 [b] | 0/3 | |
| aldicarb | 260 [a] | 3/3 | **50% (6/12)** |
| | 26 | 3/3 | |
| | 2.6 | 0/3 | |
| | 0.26 [d] | 0/3 | |
| | 0.026 [e] | 0/3 | |
| dicrotophos | 1400 [a] | 3/3 | **100% (6/6)** |
| | 140 | 3/3 | |
| | 14 [b] | 0/3 | |
| | 1.4 [b] | 0/3 | |
| | 0.014 [b] | 0/3 | |

[a] Lethal dose
[b] Not used in accuracy calculations because concentrations in these samples are at or below level of consistent negative response
[c] Two inconclusive results were observed
[d] Vendor-provided LOD for aldicarb of 0.2 mg/L
[e] Not used in accuracy calculations because concentration is below vendor-provided LOD

For VX, GB, and GD, only one set of unspiked DW and PT interferent samples were tested and used for the three chemical agents. Thus, the results shown for the unspiked DW and potential interferent samples in Tables 6-2a through 6-2c are the same and from only one set of triplicate samples. For aldicarb and dicrotophos, sets of unspiked DW and PT interferent samples were run separately for each pesticide.

As shown in Tables 6-2a and 6-2b, no false positive or false negative results were observed during testing with VX and GB. For GD (Table 6-2c), neither false positive results nor false negatives were observed; however, two replicates of the 0.014 mg/L GD gave inconclusive results out of 33 total replicates. Testing with aldicarb (Table 6-2d) yielded no false positives yet gave six false negatives out of 36 total replicates. The false negatives were the three replicates each for the 2.6 and 0.26 mg/L aldicarb PT samples (which were included in the calculation since the concentrations are above the vendor-provided LOD). Dicrotophos (Table 6-2e) also yielded no false positives but 20 of 30 samples were false negatives. 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. This may indicate that the Eclox$^{TM}$-Pesticide Strips are prone to matrix effects when used to detect dicrotophos (in matrices/concentrations similar to those tested in this verification test).

### 6.3 Precision

During testing with VX and GB, the Eclox$^{TM}$-Pesticide Strips gave consistent results. That is, all of the 21 sample sets, each consisting of three replicates, had the same results within each replicate set. For GD, 20 of 21 sample sets yielded consistent results. The testing of the 0.014 mg/L GD PT sample yielded two inconclusive results (see Table 6-2c).

Note that only one set of unspiked interferent samples were tested for VX, GB, and GD. These sample sets were shared among the three contaminants. Three of these 8 sample sets had at least one replicate that differed from the other two replicates.

For aldicarb, all of the 21 sample sets had consistent results. For dicrotophos, one spiked interferent sample set (140 mg/L dicrotophos in 50 mg/L total Ca and Mg) gave two non-detect results and one detect result, yielding consistent results among 20 of 21 sample sets.

### 6.4 Potential Matrix and Interferent Effects

The EcloxTM-Pesticide Strips were able to consistently detect VX, GB, GD, aldicarb, and dicrotophos at 10 times less than the respective LD50 concentrations in DI water. The ability of the EcloxTM-Pesticide Strips to detect these contaminants was challenged with potential interferents and DW matrices.

**Table 6-2a. VX False Positive/Negative Results**

| Sample Type | Matrix | Concentration (mg/L) | Positive Results Out of Total Replicates |
|---|---|---|---|
| Contaminant-only PT samples | DI water | 2.1 [(a)] | 3/3 |
| | DI water | 0.21 | 3/3 |
| Interferent PT samples [(b)] | 1 mg/L humic and fulvic acids | Blank | 0/3 |
| | 1 mg/L humic and fulvic acids | 0.21 | 3/3 |
| | 5 mg/L humic and fulvic acids | Blank | 0/3 |
| | 5 mg/L humic and fulvic acids | 0.21 | 3/3 |
| | 50 mg/L Ca and Mg | Blank | 0/3 |
| | 50 mg/L Ca and Mg | 0.21 | 3/3 |
| | 250 mg/L Ca and Mg | Blank | 0/3 |
| | 250 mg/L Ca and Mg | 0.21 | 3/3 |
| DW samples [(b)] | OH DW | Blank | 0/3 |
| | OH DW | 0.21 | 3/3 |
| | CA DW | Blank | 0/3 |
| | CA DW | 0.21 | 3/3 |
| | FL DW | Blank | 0/3 |
| | FL DW | 0.21 | 3/3 |
| | NY DW | Blank | 0/3 |
| | NY DW | 0.21 | 3/3 |
| | **False Positive Rate** | | **0/24** |
| | **False Negative Rate** | | **0/30** |

[(a)] Lethal dose
[(b)] Only one set of unspiked DW and PT interferent samples were run for VX, GB, and GD.

**Table 6-2b. GB False Positive/Negative Results**

| Sample Type | Matrix | Concentration (mg/L) | Positive Results Out of Total Replicates |
|---|---|---|---|
| Contaminant-only PT samples | DI water | 20 [a] | 3/3 |
| | DI water | 2.0 | 3/3 |
| | DI water | 0.20 | 3/3 |
| | DI water | 0.02 | 3/3 |
| Interferent PT samples [b] | 1 mg/L humic and fulvic acids | Blank | 0/3 |
| | 1 mg/L humic and fulvic acids | 2.0 | 3/3 |
| | 5 mg/L humic and fulvic acids | Blank | 0/3 |
| | 5 mg/L humic and fulvic acids | 2.0 | 3/3 |
| | 50 mg/L Ca and Mg | Blank | 0/3 |
| | 50 mg/L Ca and Mg | 2.0 | 3/3 |
| | 250 mg/L Ca and Mg | Blank | 0/3 |
| | 250 mg/L Ca and Mg | 2.0 | 3/3 |
| DW samples [b] | OH DW | Blank | 0/3 |
| | OH DW | 2.0 | 3/3 |
| | CA DW | Blank | 0/3 |
| | CA DW | 2.0 | 3/3 |
| | FL DW | Blank | 0/3 |
| | FL DW | 2.0 | 3/3 |
| | NY DW | Blank | 0/3 |
| | NY DW | 2.0 | 3/3 |
| | **False Positive Rate** | | **0/24** |
| | **False Negative Rate** | | **0/36** |

[a] Lethal dose
[b] Only one set of unspiked DW and PT interferent samples were run for VX, GB, and GD.

**Table 6-2c. GD False Positive/Negative Results**

| Sample Type | Matrix | Concentration (mg/L) | Positive Results Out of Total Replicates |
|---|---|---|---|
| Contaminant-only PT samples | DI water | 1.4 [a] | 3/3 |
| | DI water | 0.14 | 3/3 |
| | DI water | 0.014 | 1/3 [b] |
| Interferent PT samples [c] | 1 mg/L humic and fulvic acids | Blank | 0/3 |
| | 1 mg/L humic and fulvic acids | 0.14 | 3/3 |
| | 5 mg/L humic and fulvic acids | Blank | 0/3 |
| | 5 mg/L humic and fulvic acids | 0.14 | 3/3 |
| | 50 mg/L Ca and Mg | Blank | 0/3 |
| | 50 mg/L Ca and Mg | 0.14 | 3/3 |
| | 250 mg/L Ca and Mg | Blank | 0/3 |
| | 250 mg/L Ca and Mg | 0.14 | 3/3 |
| DW samples [c] | OH DW | Blank | 0/3 |
| | OH DW | 0.14 | 3/3 |
| | CA DW | Blank | 0/3 |
| | CA DW | 0.14 | 3/3 |
| | FL DW | Blank | 0/3 |
| | FL DW | 0.14 | 3/3 |
| | NY DW | Blank | 0/3 |
| | NY DW | 0.14 | 3/3 |
| | **False Positive Rate** | | **0/24** |
| | **False Negative Rate** | | **0/33** |

[a] Lethal dose

[b] Two results were not positive or negative, but inconclusive as interpreted by the operator; these results are excluded from the false negative calculations

[c] Only one set of unspiked DW and PT interferent samples were run for VX, GB, and GD.

**Table 6-2d. Aldicarb False Positive/Negative Results**

| Sample Type | Matrix | Concentration (mg/L) | Positive Results Out of Total Replicates [a] |
|---|---|---|---|
| Contaminant-only PT samples | DI water | 260 [b] | 3/3 |
| | DI water | 26 | 3/3 |
| | DI water | 2.6 | 0/3 [c] |
| | DI water | 0.26 | 0/3 [c] |
| Interferent PT samples | 1 mg/L humic and fulvic acids | Blank | 0/3 |
| | 1 mg/L humic and fulvic acids | 26 | 3/3 |
| | 5 mg/L humic and fulvic acids | Blank | 0/3 |
| | 5 mg/L humic and fulvic acids | 26 | 3/3 |
| | 50 mg/L Ca and Mg | Blank | 0/3 |
| | 50 mg/L Ca and Mg | 26 | 3/3 |
| | 250 mg/L Ca and Mg | Blank | 0/3 |
| | 250 mg/L Ca and Mg | 26 | 3/3 |
| DW samples | OH DW | Blank | 0/3 |
| | OH DW | 26 | 3/3 |
| | CA DW | Blank | 0/3 |
| | CA DW | 26 | 3/3 |
| | FL DW | Blank | 0/3 |
| | FL DW | 26 | 3/3 |
| | NY DW | Blank | 0/3 |
| | NY DW | 26 | 3/3 |
| **False Positive Rate** | | | **0/24** |
| **False Negative Rate** | | | **6/36** |

[a] Shaded results indicate false negative observations
[b] Lethal dose
[c] These test samples produced non-detects above the vendor-provided LOD.

**Table 6-2e. Dicrotophos False Positive/Negative Results**

| Sample Type | Matrix | Concentration (mg/L) | Positive Results Out of Total Replicates [a] |
|---|---|---|---|
| Contaminant-only PT samples | DI water | 1400 [b] | 3/3 |
| | DI water | 140 | 3/3 |
| Interferent PT samples | 1 mg/L humic and fulvic acids | Blank | 0/3 |
| | 1 mg/L humic and fulvic acids | 140 | 0/3 |
| | 5 mg/L humic and fulvic acids | Blank | 0/3 |
| | 5 mg/L humic and fulvic acids | 140 | 0/3 |
| | 50 mg/L Ca and Mg | Blank | 0/3 |
| | 50 mg/L Ca and Mg | 140 | 1/3 |
| | 250 mg/L Ca and Mg | Blank | 0/3 |
| | 250 mg/L Ca and Mg | 140 | 0/3 |
| DW samples | OH DW | Blank | 0/3 |
| | OH DW | 140 | 0/3 |
| | CA DW | Blank | 0/3 |
| | CA DW | 140 | 0/3 |
| | FL DW | Blank | 0/3 |
| | FL DW | 140 | 3/3 |
| | NY DW | Blank | 0/3 |
| | NY DW | 140 | 0/3 |
| **False Positive Rate** | | | **0/24** |
| **False Negative Rate** | | | **20/30** |

[a] Shaded results indicate false negative observations
[b] Lethal dose

### *6.4.1 Interferent PT Samples*

As shown in Tables 6-2a through 6-2d, VX, GB, GD, and aldicarb yielded negative results for all unspiked interferent samples and positive results for all spiked interferent samples for all replicates. This indicates, that for these target analytes, the Eclox$^{TM}$-Pesticide Strips were not affected by any matrix effects from these potential interferent solutions. For dicrotophos (Table 6-2e), however, only one replicate of the 140 mg/L dicrotophos in 50 mg/L Ca and Mg solution gave a positive result (while the other two replicates gave negative results). The other eleven spiked samples for dicrotophos gave negative results, indicating that the ability of the EcloxTM-Pesticide Strips to detect dicrotophos was affected by the interferent matrices.

### *6.4.2 DW Samples*

As shown in Tables 6-2a through 6-2d, VX, GB, GD, and aldicarb yielded negative results for all unspiked DW samples and positive results for all spiked DW samples for all replicates. This indicates that the Eclox$^{TM}$-Pesticide Strips were not affected by any matrix effects from these potential interferent solutions. For dicrotophos (Table 6-2e), however, only the spiked FL DW gave positive results. All of the other spiked DW samples yielded negative results (as did the unspiked DW samples) indicating that the ability of the Eclox$^{TM}$-Pesticide Strips to detect dicrotophos was affected by the OH DW, NY DW, and CA DW matrices.

## 6.5 Operational Factors

### *6.5.1 Technical Operators*

The Eclox$^{TM}$-Pesticide Strips were used by one Battelle technical operator throughout testing with the pesticides, and by a different Battelle technical operator throughout testing with CWA. The technical operators were trained by a Battelle technician, who had received training from the vendor by phone for one-half hour in the use of the kit. Both technical operators had extensive laboratory experience. The written instructions, which are provided on a small piece of paper packaged with the strips, consist of eight steps prior to reading the results of the test. In addition to the straight-forward text, the instructions also provide a small picture of the Eclox$^{TM}$-Pesticide Strip in use as well as a sample figure showing the two outcomes ("no pesticide" and "pesticide"). During testing, the technical operators 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 Eclox$^{TM}$-Pesticide Strips. The operators performing one test at a time averaged 11 tests per hour. Tests can also be conducted in parallel using separate strips for multiple water samples, which may increase sample throughput. The Eclox$^{TM}$-Pesticide Strips do not require any special storage considerations other than storage at room temperature. During the verification test, the strips were kept in a sealed plastic bag that was kept in the laboratory. The Eclox$^{TM}$-Pesticide Strips are reported by the vendor to have a two-year shelf life. Each foil packet is 8.2 cm by 5.1 cm, with notched edges to aid opening.

### *6.5.2 Non-Technical Operator*

Unspiked MB samples were tested by a non-technical operator, using the Eclox™-Pesticide Strips, both with and without PPE (see Section 3.2.4). 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, as shown in Figures 6-1 and 6-2. 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 Eclox™-Pesticide Strips without any difficulty. There were no issues with the duration of the test or impact of wearing gloves during operation. All MB samples yielded negative (i.e., no target contaminants detected) results.

The Severn Trent Services Eclox™-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.

**Figure 6-1. Side View of PPE Worn by Non-Technical Operator**

**Figure 6-2. Testing of Eclox™-Pesticide Strips with the Non-Technical Operator Wearing PPE**

# Chapter 7
# Performance Summary

The results of the Severn Trent Services Eclox™-Pesticide Strip from this verification test for samples containing VX, GB, GD, aldicarb, and dicrotophos are presented in Tables 7-1a-e, respectively. Qualitative responses for each set of sample replicates as well as accuracy, false negative and positive rates, and precision are presented in each table. A summary of the other performance factors associated with the Eclox™-Pesticide Strips is presented at the end of this chapter. These performance factors apply across all contaminants.

**Table 7-1a. VX Summary Table**

| Parameter | | Matrix | VX Concentration | Number Detected/Number of Samples |
|---|---|---|---|---|
| Qualitative Results | 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 |
| | | | 0.0021 mg/L | 0/3 |
| | | | 0.00021 mg/L | 0/3 |
| | 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 during testing with VX. | | |
| False Positive Rate | | No false positive results (0 out of 24) were observed during testing with VX. | | |
| False Negative Rate | | No false negative results (0 out of 30) were observed during testing with VX. | | |
| Precision | | 100% (21 out of 21) of the sample sets showed consistent results among the individual replicates within each set during testing with VX. | | |

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

**Table 7-1b. GB Summary Table**

| Parameter | | Matrix | GB Concentration | Number Detected/Number of Samples |
|---|---|---|---|---|
| Qualitative Results | Contaminant-Only PT Samples | DI Water | 20 mg/L [(a)] | 3/3 |
| | | | 2.0 mg/L | 3/3 |
| | | | 0.2 mg/L | 3/3 |
| | | | 0.02 mg/L | 3/3 |
| | | | 0.002 mg/L [(b)] | 0/3 |
| | 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 during testing with GB. | | |
| False Positive Rate | | No false positive results (0 out of 24) were observed during testing with GB. | | |
| False Negative Rate | | No false negative results (0 out of 36) were observed during testing with GB. | | |
| Precision | | 100% (21 out of 21) of the sample sets showed consistent results among the individual replicates within each set during testing with GB. | | |

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

**Table 7-1c. GD Summary Table**

| Parameter | | Matrix | GD Concentration | Number Detected/Number of Samples |
|---|---|---|---|---|
| Qualitative Results | Contaminant-Only PT Samples | DI Water | 1.4 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 |
| | | | 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 gave positive results during testing with GD. | | |
| False Positive Rate | | No false positive results (0 out of 24) were observed during testing with GD. | | |
| False Negative Rate | | No false negative results (0 out of 33) were observed during 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 results among the individual replicates within each set during testing with GD. | | |

[(a)] Lethal dose

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

[(c)] Two inconclusive results were observed. Inconclusive results occurred when the operator could neither discern he color of the smaller disk as blue (negative for the presence of a contaminant) nor white (positive for the presence of a contaminant).

**Table 7-1d. Aldicarb Summary Table**

| Parameter | | Matrix | Aldicarb Concentration | Number Detected/Number of Samples |
|---|---|---|---|---|
| Qualitative Results | Contaminant-Only PT Samples | DI Water | 260 mg/L [(a)] | 3/3 |
| | | | 26 mg/L | 3/3 |
| | | | 2.6 mg/L | 0/3 |
| | | | 0.26 mg/L [(b)] | 0/3 |
| | | | 0.026 mg/L | 0/3 |
| | Interferent PT Samples | Humic and Fulvic Acids | 26 mg/L | 6/6 |
| | | 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 testing with aldicarb. | | |
| False Negative Rate | | Six out of the 36 samples yielded false negative results during testing with aldicarb. The 2.6 mg/L and 0.26 mg/L contaminant-only PT samples showed negative responses. | | |
| Precision | | 100% (21 out of 21) of the sample sets showed consistent results among the individual replicates within each set during testing with aldicarb. | | |

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

**Table 7-1e. Dicrotophos Summary Table**

| Parameter | | Matrix | Dicrotophos Concentration | Number Detected/Number of Samples |
|---|---|---|---|---|
| Qualitative Results | Contaminant-Only PT Samples | DI Water | 1400 mg/L [(a)] | 3/3 |
| | | | 140 mg/L | 3/3 |
| | | | 14 mg/L [(b)] | 0/3 |
| | | | 1.4 mg/L | 0/3 |
| | | | 0.14 mg/L | 0/3 |
| | 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 during testing with dicrotophos. | | |
| False Positive Rate | | No false positive results (0 out of 24) were observed during testing with dicrotophos. | | |
| False Negative Rate | | Twenty out of the 30 samples yielded false negative results during 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 during testing with dicrotophos. | | |

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

**Operational Factors:**

*Technical Operators*
The Eclox™-Pesticide Strips were used by one Battelle technical operator throughout testing with the pesticides and a different Battelle technical operator throughout testing with CWA. These technical operators were trained by a Battelle technician, who had received training from the vendor by phone for one-half hour in the use of the kit. Both technical operators 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 technical operators 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 Eclox™-Pesticide Strips.

*Non-Technical Operators*
Unspiked MB samples were tested by a non-technical operator, using the Eclox™-Pesticide Strips, both with and without PPE. The SCBA apparatus, including the mask, was worn throughout the entire testing procedure when 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 Eclox™-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 Eclox™-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.

# Chapter 8
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