

FINAL

Pilot Dye Test Work Plan – Tyco Fire Products LP Facility

Prepared for

Tyco Fire Products LP

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ch2m.SM

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Acronyms and Abbreviations

ADCP	Acoustic Doppler Current Profiler
AOC	Administrative Order on Consent
AOR	Agreement on Resolution
BWGMP	<i>Barrier Wall Groundwater Monitoring Plan Update</i>
CH2M	CH2M HILL
EPA	United States Environmental Protection Agency
gpm	gallons per minute
mL/min	milliliters per minute
mm/s	millimeters per second
m/s	meters per second
ppb	parts per billion
ppm	parts per million
QA	quality assurance
QC	quality control
SCUFA	Self-Contained Underwater Fluorescence Apparatus
site	Tyco Fire Products LP facility located at One Stanton Street, Marinette, Wisconsin
Tyco	Tyco Fire Products LP
WDNR	Wisconsin Department of Natural Resources

Introduction

This Pilot Dye Test Work Plan presents the specific approach for a pilot dye test in the Menominee River prior to the proposed full-scale barrier wall dye testing included in the September 2015 *Revised Barrier Wall Groundwater Monitoring Plan Update* (BWGMP) (CH2M HILL [CH2M] 2015) for the Tyco Fire Products LP (Tyco) facility located at One Stanton Street, Marinette, Wisconsin (site). The objective of the BWGMP is to provide the approach to long-term monitoring of the effectiveness of the barrier at containing onsite groundwater. The BWGMP was required by the *Administrative Order on Consent* (AOC) between Tyco and the U.S. Environmental Protection Agency (EPA), dated February 26, 2009, and the April 23, 2014, *Agreement on Resolution of 2013 Five-Year Review Technical Issues* (AOR).

Pending approval of this work plan, the pilot test is planned for the week of September 18, 2017. The full-scale barrier wall dye test is planned for summer 2018. The September 2017 timing for the pilot test also will be beneficial, as it will be under similar conditions as that proposed for the full-scale test when lower river flows are most common and will limit the amount of dye needed during the full-scale test. This work plan includes project background information, a detailed pilot test approach, methodology for field measurements and dilution modeling, quality assurance (QA) and quality control (QC) methods, and the plan for reporting.

1.1 Background

Barrier walls were installed at the site in 1998 to 1999, 2009, and 2010 that effectively separated site groundwater into four containment cells (Figure 1). The containment areas within the vertical barrier wall include:

- Main Plant Area
- Former Salt Vault
- Former 8th Street Slip
- Wetlands Area

The objective of the barrier walls is to contain onsite groundwater affected by arsenic and prevent migration to the adjacent Menominee River. Pursuant to the AOR, EPA requested additional activities to determine the effectiveness of the barrier wall, which included performing a dye test to assess barrier wall effectiveness in the Main Plant Area along the Menominee River. The dye testing results will be *qualitative* in nature: if dye is detected in the river exceeding background concentrations during the full-scale dye test, it will indicate groundwater communication between the contained area and the river, but will not indicate the size or exact location of the communication. Because of the length of the barrier wall, access difficulties to potential dye testing locations, and volume of dye required, the dye test was designed only to measure the effectiveness of representative portions of the barrier wall. However, the information obtained during the dye test will be regarded as being representative of the entire wall containment integrity.

Details of the full-scale dye test are presented in the BWGMP and summarized briefly in this section. The proposed full-scale barrier wall dye testing scheduled for August or September 2018 includes injecting Rhodamine WT dye into groundwater at nine locations near the barrier wall in the Main Plant Area (Figure 2), accompanied by surface water sampling of the Menominee River adjacent to the barrier wall to detect whether dye has seeped through. The barrier wall dye test design required a number of assumptions including dye fate and transport in groundwater, hypothetical size (flow rates) of groundwater seepage rates through the wall, and dilution and dispersion of dye in the Menominee River.

In an April 24, 2017 email to Jeff Danko of CH2M, the Wisconsin Department of Natural Resources (WDNR) requested refinement of calculations of potential Rhodamine WT dye concentrations in the Menominee River under a worst-case barrier wall seepage situation and recommended a pilot dye test. Tyco responded to that request in an email on May 16, 2017 and ultimately agreed to conduct a pilot dye test during a May 23, 2017 conference call with EPA and WDNR.

Tyco submitted a *Dye Pilot Test Scope Technical Memorandum* on July 5, 2017 to EPA and WDNR. EPA provided comments and questions on the *Dye Pilot Test Scope* in an email dated July 12, 2017. These comments were discussed during a teleconference on July 13, 2017 with representatives of Tyco, EPA, WDNR, and CH2M, and it was agreed that a work plan (this document) would be prepared and submitted to EPA and WDNR by August 13, 2017, with comments from the agencies addressed herein.

1.2 Pilot Test Objectives

Conducting a pilot dye test in the Menominee River will enable refinement of required dye injection concentrations for the full-scale test to achieve a balance between ensuring that a smaller seepage rate would be detectable in the river versus the potential impacts (such as visual impacts) from a larger seepage rate. The following objectives have been identified for the pilot dye test:

- Quantitatively and qualitatively assess dispersion and dilution of Rhodamine WT dye released in the Menominee River adjacent to the barrier wall under late-summer conditions:
 - Collect field measurements of Menominee River characteristics and dilutions that will be used to calibrate the dilution model
 - Collect direct measurements of plume mixing at various locations in river that are proximate and distant from barrier walls
- Assess suitability of previously proposed fluorometers for measuring dye concentrations in surface water samples, as well as proposed surface water sampling methods
- Assess river background fluorescence and potential effects from turbid river water
- Assess river flow dynamics along barrier wall at proposed full-scale injection locations and downstream, including in the Main Channel, the Turning Basin, and the South Channel
- Model Rhodamine WT dye dispersion in surface water to demonstrate likely downstream extents of dye at concentrations of potential concern in “worst-case” scenario
- Use data collected and model results to refine full-scale dye test design, if appropriate, including refining groundwater dye addition concentrations to balance detectability of a low-rate wall seepage rate against surface water impacts from a “worst-case” high-seepage rate scenario
- Develop a technical report that summarizes the data collected and modeling and defines, if applicable, proposed changes to the full-scale barrier wall dye test design

To meet these project objectives, experienced personnel will implement this project to develop accurate and defensible field measurements, document the selection and application of an appropriate dilution model, and gain site-specific knowledge and experience prior to full-scale barrier wall dye test in 2018.

It should be noted that the proposed pilot test will not assess dye behavior in groundwater, so assumptions about the fate and transport of Rhodamine WT injected in the groundwater system will remain.

1.3 Pilot Test Approach

The approach for this pilot test involves collecting site-specific field measurements using a dye tracer and then using these field-measured dilutions and ambient conditions to develop model predictions of discharge dilutions for the field-measured conditions. Tracer dye (Rhodamine WT) will be added directly into the river at a known concentration and consistent rate at three locations along the Main Plant barrier wall (Figure 2). River velocities will be measured near the wall along the Main Channel, in the Turning Basin, and in the South Channel prior to dye addition to determine likely dye dispersion characteristics and paths.

Submersible fluorometers will be deployed from a vessel to measure dye concentrations along transects downstream of the dye addition location while the dye is carried past the instruments by ambient currents. Boat-based and shore-based personnel will collect additional surface water grab samples during the pilot test to augment the data collected by the stationary submersible fluorometers.

River velocity data and Rhodamine WT concentration curves will be used to calibrate dispersion coefficients in the mixing model. The calibrated dispersion coefficients will then be used to model dye concentrations adjacent to the barrier wall, expected downstream extent of dye concentrations above 10 parts per billion (ppb; visibility limit and recommended maximum concentration entering drinking water plants), and expected downstream extent of dye concentrations above 0.1 ppb (recommended drinking water concentration) for both worst-case wall seepage rates and low wall seepage rates at following dye concentrations:

- WDNR permitted highest allowable concentration in river (64.9 parts per million [ppm])
- Originally proposed groundwater injection concentrations of 40 ppm and 150 ppm
- Groundwater injection concentration (to be determined) at which a low wall seepage rate (0.2 gallons per minute [gpm]) would be detectable in the river by proposed fluorometers given reasonable but conservative estimates of aquifer dilution, aquifer solids adsorption, river velocities and dispersion, sample distance from seepage location, and river background concentrations

Additionally, water samples will be collected to test the water collection method and proposed fluorometers for the full-scale barrier wall dye test. Measurements of river background fluorescence will be made to assess the potential for background interference and to potentially lower the limits of Rhodamine WT detectability.

Field Methods

Three dye test locations are planned (Figure 2) to assess flow dynamics and dispersion at different locations along the wall by adding a continuous slug of dye for approximately 30 minutes at a known concentration and volume adjacent to the wall. Submersible fluorometers deployed approximately 100 and 300 feet downstream of the release point along two lateral transects (each with a minimum of two fluorometers) will be used to continuously measure dye concentrations through time (to develop response curves). With two instruments deployed within 5 feet of the wall and a second set of two instruments approximately 15 to 20 feet from the wall, measured dye concentrations will provide information on both the longitudinal and lateral dispersion characteristics of the dye plume as it is carried downstream by ambient currents. Boat- and shore-based personnel will collect 10 surface water grab samples per dye test, and the samples will be analyzed using handheld fluorometers to augment data collected by the fixed submersible instruments.

The fieldwork is estimated to take 4 to 5 days and includes instrument preparations and calibrations, installation of field instruments, the dye tracer pilot tests, post-test calibration and downloading of field data. Figure 1 shows the location of the Main Plant barrier wall, and location of the Main Channel, Turning Basin, and South Channel. Figure 2 shows the proposed full-scale dye test injection locations, proposed pilot test dye addition locations and transect locations, and proposed background concentration measurement location.

Details of these field activities and measurements are as follows. Note that due to the fluid nature of dye testing activities and river dynamics, conditions observed prior to the testing will be evaluated and locations, samples, and dye concentrations/additions will be adjusted as needed for each dye test to best assess river conditions.

- Measurements of current velocity (speed and direction) will be recorded in the Menominee River at multiple locations near the barrier wall in the Main Channel, the Turning Basin, and near the South Channel using a current meter. Additional information about the velocity measurements for this test is provided in Section 2.1.
- The field dye test will use Rhodamine WT, a water soluble, fluorescent dye. Additional detailed information about the dye proposed for this test is provided in Section 2.2.
- Preparation of dye to achieve a target concentration of between 200 and 1000 ppb, discussed in Section 2.3.
- Background river fluorescence measurements will be collected before and after the pilot test. The concentration of dye in the samples will be analyzed with both the Turner Designs Self-Contained Underwater Fluorescence Apparatus (SCUFA) and the AquaFluor field fluorometer. The fluorometers will be calibrated with river water collected upstream of the proposed sampling sites. Details are provided in Section 2.4.1.
- Dye will be added to the river for separate tests at the following locations (Section 2.3):
 - Test one—in the Turning Basin (with an additional fluorometer deployed in the South Channel area for this as well as the other two test locations)
 - Test two— near the eastern end of the barrier wall (deploy submersible fluorometers both along Main channel and in Turning Basin)
 - Test three— along the Main Channel

- Dye measurements will be sampled by the submersible fluorometers at a 10-second resolution and will extend for a period of three times the duration of the injection in order to capture the entire dye plume as it is carried over the instruments (Section 2.4.2). At the first location, two dye addition events will occur separately; one dye addition near the surface and a second dye addition near the river bottom.
- Turner Designs SCUFA's will be deployed at two distances downstream of the injection point (approximately 100 and 300 feet), with two deployments at each distance to measure the lateral variation in plume concentration. Additionally, a SCUFA will be deployed near the South Channel to determine whether dye reaches this location. Additional details on SCUFA deployment are provided in Section 2.4.2.
- During each dye test, surface water grab samples will be taken throughout the plume and from land, and a handheld AquaFluor fluorometer will be used to record dye concentrations. Additionally, for testing the sample design prior to the full-scale barrier wall dye test, surface water samples will be collected during the pilot test using similar methods as proposed for the full-scale barrier wall test. Additional details are provided in Section 2.4.3.

Standard operating procedures for the field fluorometers are presented in Appendix A.

2.1 River Velocity Measurements

A RiverRay Acoustic Doppler Current Profiler (ADCP) will be used to measure flow velocities. The RiverRay ADCP measures river velocity profiles and river bottom depths from a small float. A Hach FH950 portable velocity meter, deployed on a wading rod from either an anchored boat or from the barrier wall, will be used to measure spot river velocities. Velocity profile measurements will be collected at the wall, and 10 feet and 20 feet perpendicular to the wall at the following locations:

- The three proposed pilot test dye addition locations and proposed transect locations
- The nine proposed full-scale dye groundwater injection locations
- At select locations in the Turning Basin and near the South Channel to assess flow directions and velocities

River velocity measurements will be conducted prior to the pilot dye tests to inform the location of dye fluorometer transects. Lake seiche effects could alter the steady downstream trajectory of the dye plume, so a handheld current meter will be used by boat-based personnel to conduct spot samples of current velocities during dye tests. Additional river velocity measurements may be taken during or after the pilot dye test if changes in flow patterns are observed (for example, rising or falling water levels).

2.2 Proposed Dye

The pilot test will use Rhodamine WT, a fluorescent, water soluble dye, which is commonly used for surface water tracer studies. At higher concentrations, the red dye is visible to the naked eye. At lower concentrations, the fluorescence of the dye can be measured by fluorometers at concentrations as low as 10 parts per trillion in deionized water; however, in river water the lower range will be 1 to 2 ppb. WDNR approved the use of Keyacid Rhodamine WT Liquid (see Appendix B for the Safety Data Sheet) for the full-scale barrier wall test in a letter dated June 26, 2017, with the provision that the concentration of Rhodamine WT not exceed 64.9 ppm (or milligrams per liter) at any time in the discharge to surface water. The proposed Rhodamine WT injection concentrations (into groundwater) for the full-scale dye test ranged from 40 to 150 ppm, with expected reductions in concentration due to aquifer dilution and absorption prior to reaching the barrier wall. For this pilot test, it has been determined that a Rhodamine WT concentration of 1 ppm (into surface water) will be sufficient.

The following key Rhodamine WT concentration values have been established:

- 64.9 ppm: WDNR approved secondary acute value, which represents the maximum concentration allowable in the river.
- 10 ppb: Dye may be visible in a clear reservoir.
- 10 ppb: EPA recommended maximum concentration entering drinking water plant.
- 0.1 ppb: EPA recommended maximum concentration in drinking water, and National Sanitation Foundation recommended maximum use concentration.

2.3 Dye Additions

Dye will be added to the river within 2 feet laterally of the barrier wall at the three proposed locations shown on Figure 2. At Location 1 in the Turning Basin, separate tests will be conducted with near surface and near bottom discharges to assess if plume dynamics differ based on depth and determine if measurement of the surface dye release is affected by sunlight. A surface release is preferable because visual observations of the dye can complement the fluorometer measurements. At Locations 2 and 3, one test will be conducted, with the discharge depth based on the results from the Location 1 dye test results and river velocity measurements. If time permits and deemed useful, a second test may be conducted at a different depth at Locations 2 and 3.

Dye will be added at a constant concentration and rate (between 100 and 500 milliliters per minute [mL/min]) for approximately 30 to 60 minutes at each location. The rate and length of dye addition may be adjusted based on initial test results to ensure high quality data are obtained. Dye additions will be conducted separately (that is, one test will be conducted at a time). The proposed dye addition concentration is 200 to 1,000 ppb. Rhodamine WT, which comes from the manufacturer in 20 percent solution (200,000 ppm Rhodamine WT) will be diluted with tap or river water to achieve the target concentration using the equation:

$$V_1 = 1000000 * \frac{C_2 * V_2}{C_1}$$

Where V_1 = Volume of 20 percent dye solution required (microliters)

V_2 = Volume of diluted dye needed for dye addition (L) (15 liters in example)

C_1 = Initial concentration of 20 percent dye solution (230,000 ppm)

C_2 = Goal concentration of diluted dye (0.2 to 1 ppm)

To prepare 15 liters of 1 ppm solution (proposed volume at each dye addition location), 75 microliters Rhodamine WT liquid dye would be added to 15 liters of tap or river water in a 10- or 11-gallon container. Dye standards will be made by adding required dye volume/mass using volumetric glassware (pipettes and volumetric flasks) to achieve the desired volume; river water will be used to create the dye standards. Dye standards of 5, 25, 100, and 500 ppb will be made for calibration purposes.

A peristaltic pump with tubing attached to a rod will be used to deliver the diluted dye into the river at the surface and at a near-bottom depth alongside the barrier wall. Dye injection pumps will be set up and will be tested and calibrated prior to use. It is anticipated that the dye injection equipment will be staged on land adjacent to the barrier wall.

2.4 Dye Concentration Measurement

2.4.1 Background Fluorescence and Fluorometer Calibration

Site-specific conditions that can affect fluorescence measurements include background fluorescence. Receiving water may have background fluorescence that must be accounted for and subtracted from the field measurements to calculate actual dilutions. Therefore, background river water will be tested with the fluorometers with no dye concentration before and after the pilot test. Pre-test and post-test calibrations will define the detection limit of dye for the instruments and whether there is background fluorescence and turbidity that could potentially read as dye concentration.

Background river fluorescence will be assessed by collecting three river samples from the western end of the Main Plant barrier wall. Two samples from the groundwater treatment system effluent will also be collected and analyzed. Samples will be collected adjacent to the wall and 15 feet from the wall using either a Kemmerer-style sampler, alpha-type sampler, or a peristaltic pump with tubing attached to a sampling pole. Samples will be analyzed using both the Turner Designs SCUFA and the AquaFluor handheld fluorometer for fluorescence and turbidity. If river conditions change during the pilot test (for example, if the river becomes more turbid or seiche effects), additional background samples will be collected.

The fluorometers will be calibrated with river water collected upstream of the proposed sampling sites. The background fluorescence will be measured using this water; all measurements during calibrations will be corrected based on this background value. The SCUFAs will also require turbidity calibration (according to the manufacturer's specifications), followed by accuracy checks of turbidity concentrations bracketing the expected range observed at the specific deployment sites. High turbidities may affect fluorescence, which will be determined during calibration and accounted for during data processing, if necessary.

2.4.2 Fluorometer Deployment

For the pilot test, Turner Designs SCUFA submersible fluorometers will be deployed to measure Rhodamine WT concentrations in surface water along two transects approximately 100 and 300 feet downstream of the dye addition location. The SCUFAs will be set up to record Rhodamine WT dye concentration and turbidity measurements every 10 seconds and measurements will extend for a period of three times the duration of the injection in order to capture the entire dye plume as it is carried over the instruments. Two SCUFAs will be deployed per transect to measure the lateral variation in plume concentration, with one SCUFA within one to five feet of the barrier wall and the other SCUFA approximately 15 to 20 feet from the wall. SCUFAs on a vertical array at a fixed point are not necessary because of the negligible vertical dispersion over the length scales of the monitoring program. A SCUFA will also be deployed near the South Channel for the duration of the tests. Locations of SCUFAs may be adjusted based on river velocity measurements and initial dye test results and observations, as well as to ensure protection from vessel traffic.

Depending on field conditions, SCUFAs will be deployed by using bottom-anchored moorings and near-surface float or secured from land. Vertical position in the water column will also be determined in the field, with acknowledgement of potential interference from light for near-surface deployments. A two-person crew aboard a boat will deploy equipment, and monitor SCUFAs during the dye additions. The vessel will be equipped with a handheld radio and cell phones to allow communication with the onshore dye addition and sampling personnel.

Field sampling site locations will be recorded using a global positioning system unit with onboard navigation and water depths recorded with a lead-line and compared with depths on the bathymetry

survey chart. A laser ranger-finder may also be used for measurement of distances to the wall or other references and shoreline features.

2.4.3 Surface Water Sampling

To augment the stationary instruments, 10 surface water grab samples per dye test will be taken at points along the plume and from land, and a handheld AquaFluor fluorometer will be used to record dye concentrations.

A different sampling scheme was proposed for the full-scale barrier wall dye test. For that test, a YSI 6820 v2 Sonde equipped with an optical Rhodamine WT sensor was proposed for initial profiling of river fluorescence values at three vertical transects near the groundwater injection location. If no readings exceeding background levels are detected, only one sample per transect would be collected for confirmation analysis using the fluorometer. If there are detections exceeding background levels using the YSI, three samples at different depths would be collected for fluorometer analysis at the depths with the highest readings to confirm the YSI screening readings. Discrete surface water samples were to be collected using either a Kemmerer-style water sampler or a peristaltic pump with tubing attached to a pole. For purposes of testing this sample design prior to the full-scale barrier wall dye test, surface water samples will be collected during the pilot test using similar methods as proposed for the full-scale barrier wall test.

During one of the three proposed pilot tests, initial profiling adjacent to the barrier wall with a YSI sonde will be conducted, followed by collection of discrete surface water samples for analysis by an AquaFluor handheld field fluorometer. Based on the results, the full-scale dye test sampling approach may be appropriately modified to obtain necessary information for evaluation of full-scale dye testing results.

2.5 Field Instruments

Field instruments for the pilot test, including backup units, are listed in Table 1. Standard operating procedures for use of the fluorometers are provided in Appendix A.

Table 1. Field Instruments for Pilot Dye Test

Instrument	Purpose	Accuracy Standard
Turner SCUFA Fluorometer	Fluorescent dye measurements	Detection to 0.5 ppb
MasterFlex Peristaltic Pump	Used for dye addition into river at constant rate; Collect surface water samples for analysis by AquaFluor fluorometer	0.2 mL/min
Hach FH950 Portable Velocity Meter	Measures in-situ current speed	$\pm 2\% \pm 0.015$ m/s for 0 to 3.05 m/s, $\pm 4\%$ for 3.04 to 4.87 m/s
River Ray Acoustic Doppler Current Profiler	Measure in-situ current speed and river bottom depth	$\pm 0.25\%$ of water velocity relative to ADCP, ± 2 mm/s
Kemmerer Water Sampler and/or Alpha Water Sampler	Collect surface water samples	
AquaFluor Fluorometer	Measure background fluorescence and fluorescence of surface water samples	Detection to 0.4 ppb
YSI 6920 v2 sonde or 6820 v2 sonde equipped with Rhodamine WT and Turbidity optical sensors	Measure background fluorescence and fluorescence of surface water samples	Detection to 0.1 ppb
Handheld Global Positioning System	Recording measurement locations	To be determined
Laser range-finder/measuring tape	Recording distances	To be determined

Quality Assurance/Quality Control

The QA/QC objective for the pilot test is to collect measurements of river dilution and river conditions that are of known and acceptable quality. The following requirements will be followed to achieve the objectives:

- Provide verifiable dye addition rates and initial dye concentrations
- Provide verifiable equipment calibration with pre- and post-test calibrations of the fluorometer instruments
- Maintain accurate positioning for measurements
- Provide equipment redundancy (backup equipment)

This work plan has been developed as the basic element of quality assurance and control activities for the field pilot test.

3.1 Equipment Calibration

Equipment will be obtained prior to the beginning of the dye pilot test. Each instrument will be checked and calibrated upon its arrival to confirm it is in working condition. Each instrument will also be calibrated immediately prior to the beginning of the dye pilot test and, when appropriate, following the test. Calibration methods for each instrument are described below.

3.1.1 Current Meters

These instruments are calibrated by the manufacturer according to their specifications prior to deployment. Calibration results will be used during data reduction and the calibration history will be incorporated for the units used. A zero calibration will be conducted in the field prior to deployment (the sensor is placed in a bucket full of still water and zero velocity is selected), if necessary.

3.1.2 Dye Pumps

The dye pumps will be calibrated at the location where it will be used during the dye pilot test. The pumps will be equipped with a micrometer control to accurately determine pumping rate. The flow rate scale will be calibrated with the dye at ambient temperature by repeatedly discharging dye into a graduated cylinder for a fixed period of time at various flow rate scale settings. According to the manufacturer, reproducible metering accuracy of greater than 1 percent can be expected when handling medium-viscosity fluids if fluid differential pressure, fluid viscosity, and electric line voltage remain constant. To verify that none of these factors is affecting expected dye flow rates during dye addition, dye flow rates will be verified and logged prior to the start of dye addition and between dye additions.

3.1.3 Fluorometers

The fluorometers (SCUFAs, AquaFluor, YSI) used will be calibrated using the appropriate dye standards (1, 10, 50, 100, 500 and 1,000 ppb for dye measurements in the receiving waters) according to the manufacturer's specifications such that they measure total dye concentration in the appropriate range for their use. Standards will be prepared with the dye used in the pilot test (background river water). In addition, turbidity sensors will be calibrated using turbidity standards. Fluorometers will be calibrated before the pilot test. Immediately following the dye pilot test, a second set of fluorometer calibration measurements will be recorded using dye and distilled water. This second set of calibration

measurements will be compared to the pre-test calibration data, after correction for temperature. Both calibration curves will be used to correct or adjust the observed dye concentration and dilution.

Dilution Modeling

Following the pilot test, dye tracer data will be summarized and analyzed. Dilution modeling will be conducted using the collected Rhodamine WT measurements and river velocity data. Dilution modeling analyses will first focus on the field tracer pilot test conditions to calibrate river dispersion coefficients. The model effort will then focus on calculating expected Rhodamine WT concentrations adjacent to the barrier wall and downstream under different wall seepage and initial dye concentration scenarios.

4.1 Modeling Objective

The objective of the modeling effort is to develop plausible predictions of Rhodamine WT concentrations in the river adjacent to a seepage point in the barrier wall and downstream extent of Rhodamine WT concentrations above key criteria.

4.2 Model Selection

Initial modeling will be conducted using a Microsoft Excel-based model based on standard river dispersion equations from Fischer et al. (1979). If flow dynamics are deemed too complicated to be assessed by the Microsoft Excel-based model, the U.S. Army Corps of Engineers RMA2 hydraulic model and RMA4 constituent transport model will be used to model flow dynamics and dye concentrations in the area of the Turning Basin and the Main Channel. These models are discussed in more detail below.

4.2.1 Excel-based Fischer Model

The initial dilution model will be based on the classic advection-diffusion equation provided by Fischer et al. (1979), which calculates centerline dilution downstream of a source as a function of current velocity, channel geometry, and a transverse mixing coefficient:

$$C_{\max} = \frac{\dot{M}}{u d} \frac{1}{\sqrt{4\pi\varepsilon_t x/u}}$$

where C_{\max} is the maximum concentration in the center of the plume, u is the steady state ambient current speed, d is the depth of the channel, ε_t is the transverse mixing coefficient, x is the distance downstream from the source, and M is the mass flow rate of concentration into the system.

The transverse dispersion coefficient was calculated according to Fischer via

$$\varepsilon_t = 0.6du^*$$

where the shear velocity u^* is defined as

$$u^* = \sqrt{gdS}$$

with g the gravitational constant and S the river slope.

The lateral variation in concentration from the plume centerline is calculated with a standard exponential decay curve after Yearsley (1989). The spreadsheet model will be set up in matrix form to allow for visualization of spatial concentration contours as well as longitudinal and transverse profiles of constituent concentration through the plume. The generic model assumes steady conditions and uniform shorelines, and thus modifications will have to be made to accommodate locations such as the turning basin or where channel geometry or depth is variable.

Sample outputs from an initial spreadsheet model are shown in Figures 3 through 6. Figure 3 shows a two-dimensional representation of the change in concentration downstream and across the river channel for a point source discharge along a wall. Concentrations are expressed as a percentage of the initial concentration. The spatial axes are not linear. Figure 4 shows a plan view of the concentration plume; again, the spatial axes are not linear. Transects of the predicted concentration plume are presented on Figures 5 and 6, with Figure 5 showing the sensitivity of the longitudinal centerline transect to the lateral mixing coefficient, and Figure 6 showing successive lateral transects showing how the plume spreads with distance downstream of the source.

4.2.2 U.S. Army Corps of Engineers RMA2 and RMA4 Models

The Excel-based approach described above is developed for linear systems, and is most applicable for conditions along the edge of the barrier wall. Additional detail on the expected fate and transport of the dye can be provided with a two-dimensional hydrodynamic and water quality model application, that is more applicable to complex flow patterns, such as the Turning Basin.

The U.S. Army Corps of Engineers RMA2 and RMA4 models can be used to predict hydrodynamics and constituent concentrations in the Menominee River downstream of the project site, including concentrations in the Main River, Turning Basin, and South Channel. The widely used RMA2 hydrodynamic model solves the two-dimensional forms of the continuity equations for mass and momentum to predict current velocities and water levels subject to prescribed river flows at the upstream end of the model domain and river stages at the downstream end of the domain. Equations in the model are depth averaged, meaning it solves for longitudinal and lateral variations in the local hydraulic regime. The RMA4 water quality model solves a standard advection-dispersion equation, using results from the hydrodynamic model for the advective component of mass transfer with time through the model domain, and user-defined dispersion coefficients to account for the dispersive component.

The two-dimensional models require bathymetric survey data to prescribe model geometry, and flow and stage conditions to specify model boundaries. Stage data in the lower Menominee River is available from an active National Oceanic and Atmospheric Administration station (9087088). U.S. Geological Survey gauge 04067500 at McAllister, approximately 22 miles upstream of the river mouth, will provide rough flow estimates, although local dams in Marinette can influence flow. Current meter data and dye pilot test results could be used to calibrate the model parameters (friction, dispersion) to improve predictive results. Model output is a time series of velocity, stage, and concentrations at any point in the model domain. Plan view concentration contour maps of constituent concentration are easily developed with post-processing tools contained in the model package.

Advantages of the two-dimensional model approach are that it can predict concentrations throughout the region of interest, including the turning basin and the south channel. However, the model needs high resolution bathymetric information to correctly prescribe the system geometry in order to provide reasonable results for ambient currents throughout the model domain.

4.2.3 Model Runs

Dilution modeling analyses will first focus on the field tracer pilot test conditions to calibrate river dispersion coefficients. A comparison of model-predicted versus field-measured dilution will be made. The model effort will then focus on calculating expected Rhodamine WT concentrations adjacent to the barrier wall and downstream under different hypothetical wall seepage and initial dye concentration scenarios. All model runs will evaluate dye concentrations adjacent to the barrier wall, expected downstream extent of dye concentrations above 10 ppb (visibility limit and recommended maximum concentration entering drinking water plant), and expected downstream extent of dye concentrations above 0.1 ppb (recommended drinking water concentration). Model runs will be conducted to bracket

both river flows and potential seepage rates (0.2 to 10 gpm) expected during the full-scale dye test at the following concentrations of dye hypothetically seeping through the barrier wall:

- WDNR permitted highest allowable dye concentration in river (64.9 ppm)
- Originally proposed groundwater injection concentrations of 40 ppm and 150 ppm
- Groundwater injection concentration at which a low wall seepage rate (0.2 gpm) would be detectable in the river by proposed fluorometers given reasonable but conservative estimates of aquifer dilution, aquifer solids adsorption, river velocities and dispersion, sample distance from seepage location, and river background concentrations

The dilution model output will be included with the pilot dye test report.

Organization and Schedule

Primary responsibility for the pilot test and field data collections will rest with CH2M's project manager, Heather Ziegelbauer; dye testing subject matter expert, Brad Paulson; and river hydraulics subject matter expert, Kyle Winslow. These personnel are responsible for accomplishing the scope of work, assigning resources, communications, schedules, and reviewing deliverables. The work will be performed by experienced CH2M personnel with help from Tyco staff.

Prior to implementation of the pilot dye test, public notifications will be made by Tyco, including:

- EPA and WDNR (Conor Neal and Kristin DuFresne, at least 14 days prior to the start of work) and WDNR Spill Hotline (1-800-943-0003)
- Abutting and downstream neighbors (Marinette Marine, Dunn Paper, Waupaca Foundry, K&K Integrated Logistics)
- City of Marinette, Wisconsin
- City of Menominee, Michigan

In addition, a public notice will be posted at the six area boat launches (Stephenson Island Launch, Cox Landing, Mystery Ship Launch, 6th Street Launch, Lighthouse Landing, and Red Arrow Launch).

If QA problems or deficiencies requiring special action arise in the field, the CH2M field team leader will identify the appropriate corrective action to be initiated and implemented. Problems are documented in writing, along with the corrective action taken. Table 2 provides the proposed schedule for this pilot test.

Table 2. Schedule for Pilot Dye Test

Date	Task
August 14, 2017	Submit Work Plan to WDNR and EPA
Week of August 21, 2017	Receive Comments from WDNR and EPA; Conference Call to Adjudicate Comments
September 4, 2017	Notify EPA, WDNR, City of Marinette and City of Menominee
Week of September 11, 2017	Public Notifications
Week of September 18, 2017	Implement Pilot Dye Test
November 17, 2017	Submit Pilot Dye Test Report to EPA and WDNR

Data Analysis and Report

The field data collected will be compiled and analyzed to define current velocities and flow patterns near the barrier wall, background river fluorescence, and river dispersion coefficients.

A pilot dye test report will be prepared that summarizes the results of the field data collections, and dilution modeling. Recommendations for adjustments to the full-scale barrier wall dye test approach (such as injection concentrations, sampling techniques/locations) will be made, if necessary. The pilot dye test report will be submitted to the EPA and WDNR by November 17, 2017.

References

CH2M HILL (CH2M). 2015. *Final Revised Barrier Wall Groundwater Monitoring Plan Update*. September.

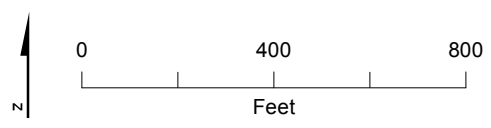
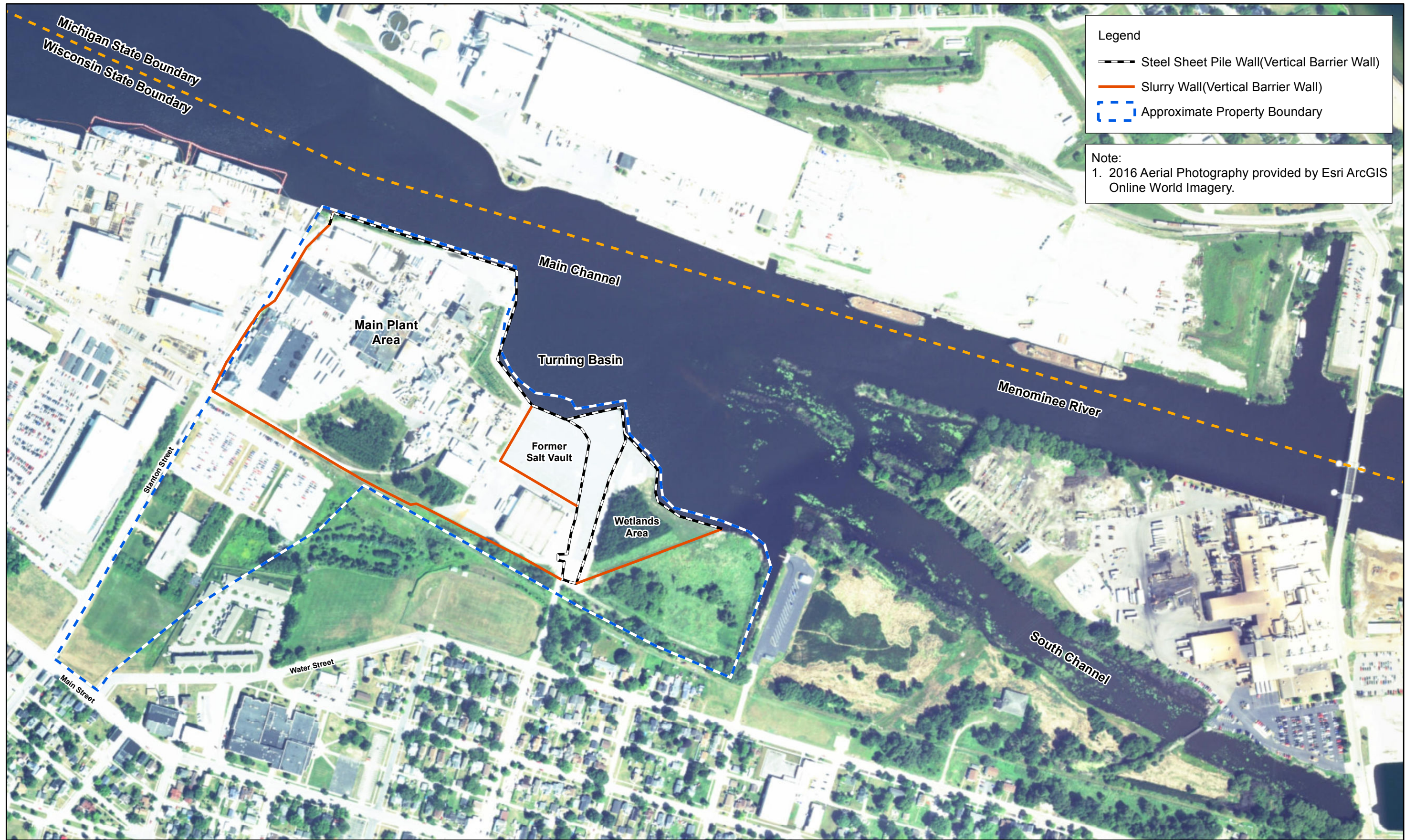
Fischer, H., E. J. List, R. Koh, J. Imberger, and N. Brooks. 1979. *Mixing in Inland and Coastal Waters*. Academic Press, Inc. New York, New York.

U.S. Environmental Protection Agency (EPA). 2009. *Resource Conservation and Recovery Act Administrative Order on Consent, Ansul, Incorporated*. EPA Docket No. RCRA -05-2009-0007542-S-02-001. February 26.

U.S. Environmental Protection Agency (EPA) and Tyco Fire Products LP (Tyco). 2014. *Agreement on Resolution of 2013 Five-Year Review Technical Issues*. April 23.

Yearsley, J., 1989. *Diffusion in Near-shore and Riverine Environments*. U.S. Environmental Protection Agency, Region 10. EPA 910/9-87-168.

Figures



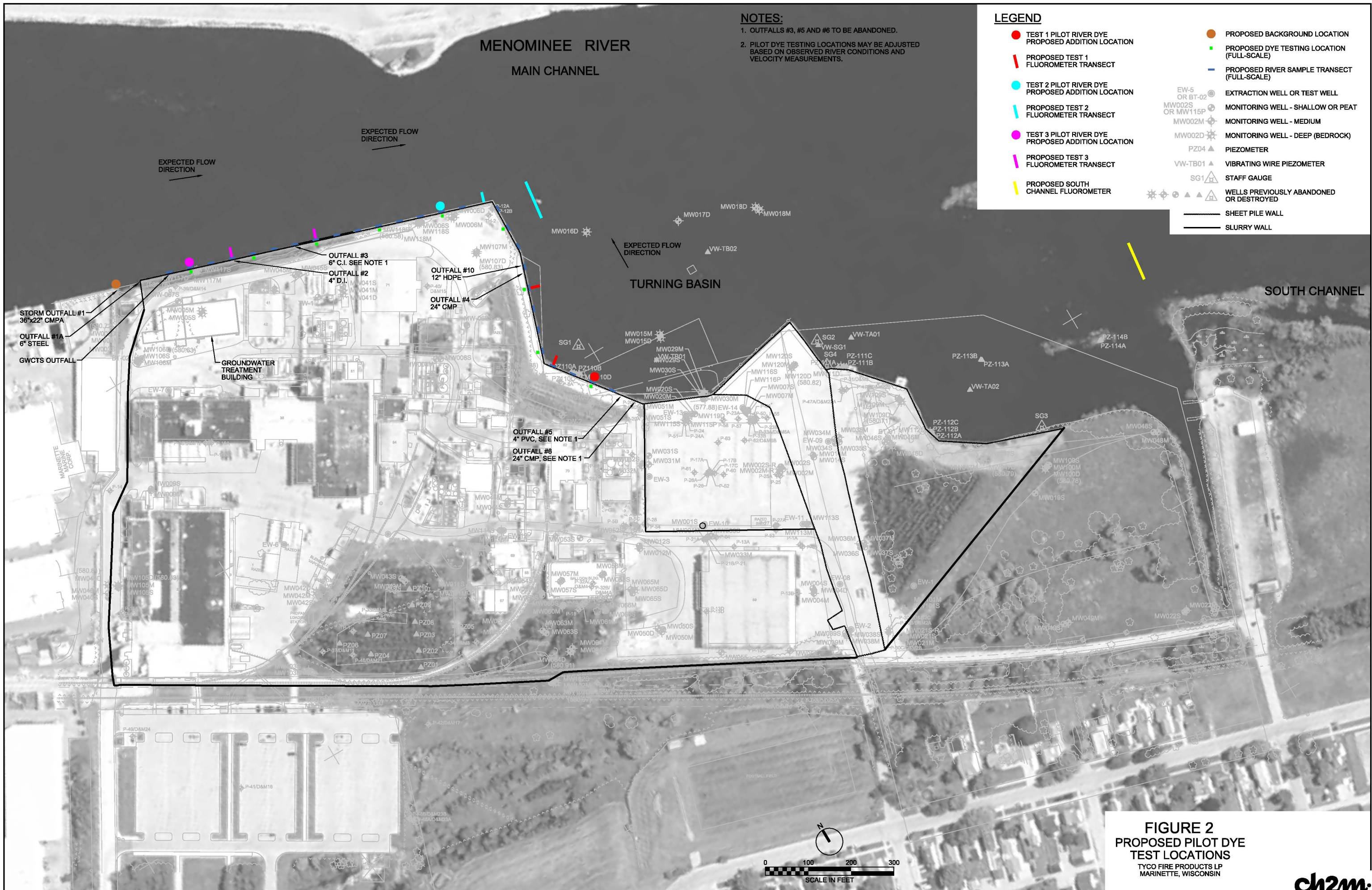
**MENOMINEE RIVER
MAIN CHANNEL**

NOTES:

1. OUTFALLS #3, #5 AND #6 TO BE ABANDONED.
2. PILOT DYE TESTING LOCATIONS MAY BE ADJUSTED BASED ON OBSERVED RIVER CONDITIONS AND VELOCITY MEASUREMENTS.

LEGEND

- TEST 1 PILOT RIVER DYE PROPOSED ADDITION LOCATION
- | PROPOSED TEST 1 FLUOROMETER TRANSECT
- TEST 2 PILOT RIVER DYE PROPOSED ADDITION LOCATION
- | PROPOSED TEST 2 FLUOROMETER TRANSECT
- TEST 3 PILOT RIVER DYE PROPOSED ADDITION LOCATION
- | PROPOSED TEST 3 FLUOROMETER TRANSECT
- | PROPOSED SOUTH CHANNEL FLUOROMETER
- PROPOSED BACKGROUND LOCATION
- PROPOSED DYE TESTING LOCATION (FULL-SCALE)
- | PROPOSED RIVER SAMPLE TRANSECT (FULL-SCALE)
- EW-5 OR BT-02 ● EXTRACTION WELL OR TEST WELL
- MW002S OR MW115P ● MONITORING WELL - SHALLOW OR PEAT
- MW002M ● MONITORING WELL - MEDIUM
- MW002D ● MONITORING WELL - DEEP (BEDROCK)
- PZ04 ▲ PIEZOMETER
- VW-TB01 ▲ VIBRATING WIRE PIEZOMETER
- SG1 ▲ STAFF GAUGE
- ● ● ● ● WELLS PREVIOUSLY ABANDONED OR DESTROYED
- SHEET PILE WALL
- SLURRY WALL



**FIGURE 2
PROPOSED PILOT DYE
TEST LOCATIONS**
TYCO FIRE PRODUCTS LP
MARINETTE, WISCONSIN



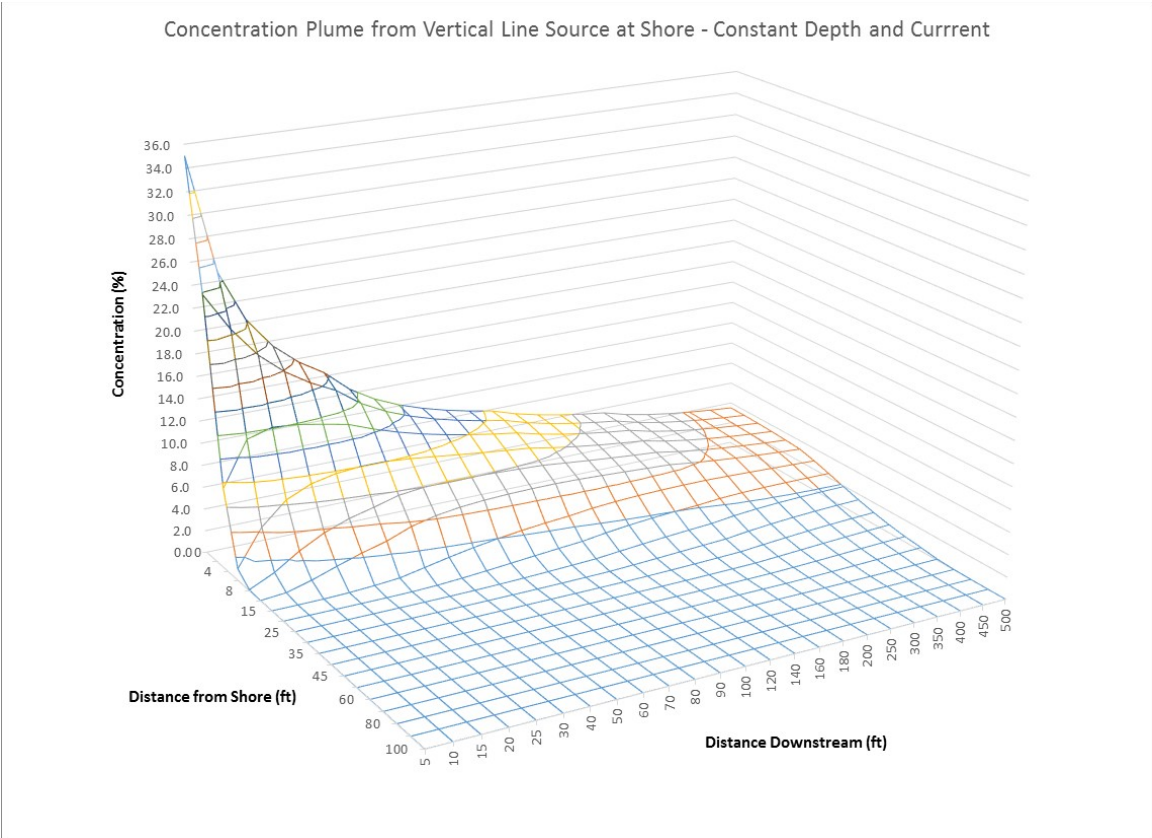


Figure 3. Sample plot of concentration plume for a vertical line source at a wall.

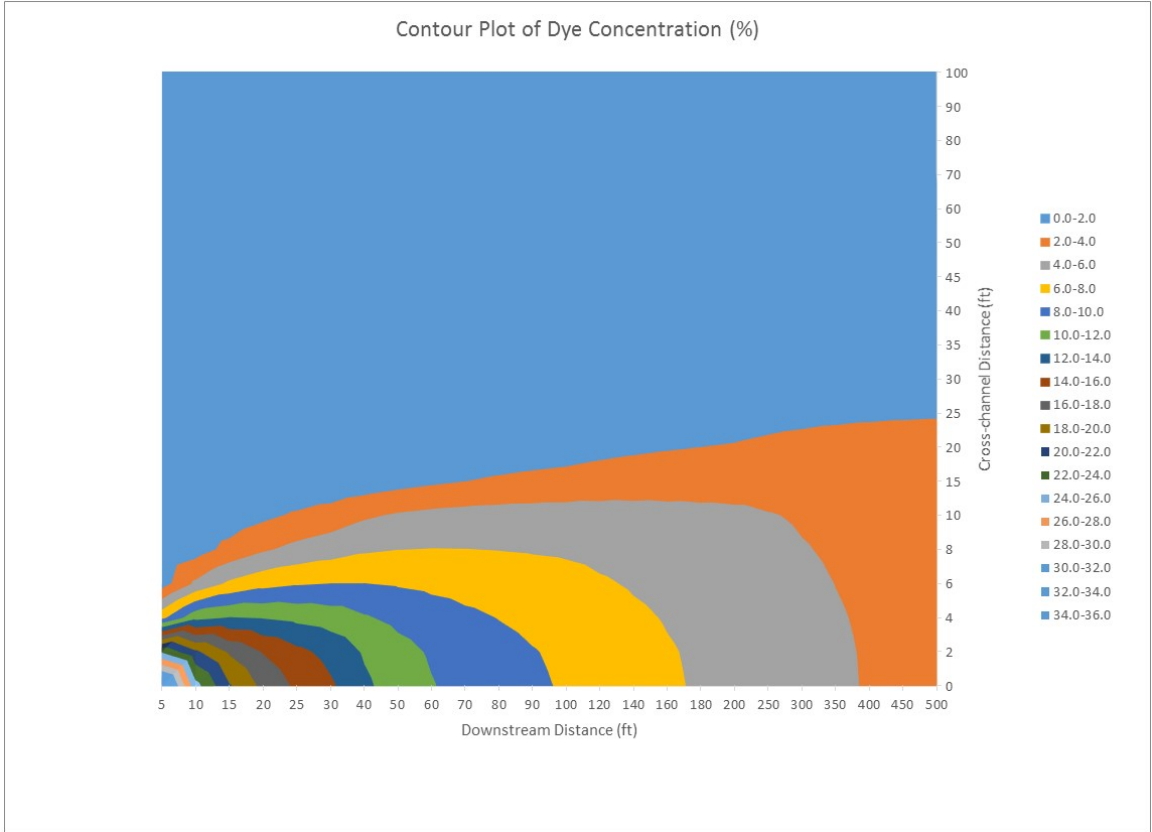


Figure 4. Sample contour plot of dye concentration for vertical line source at wall

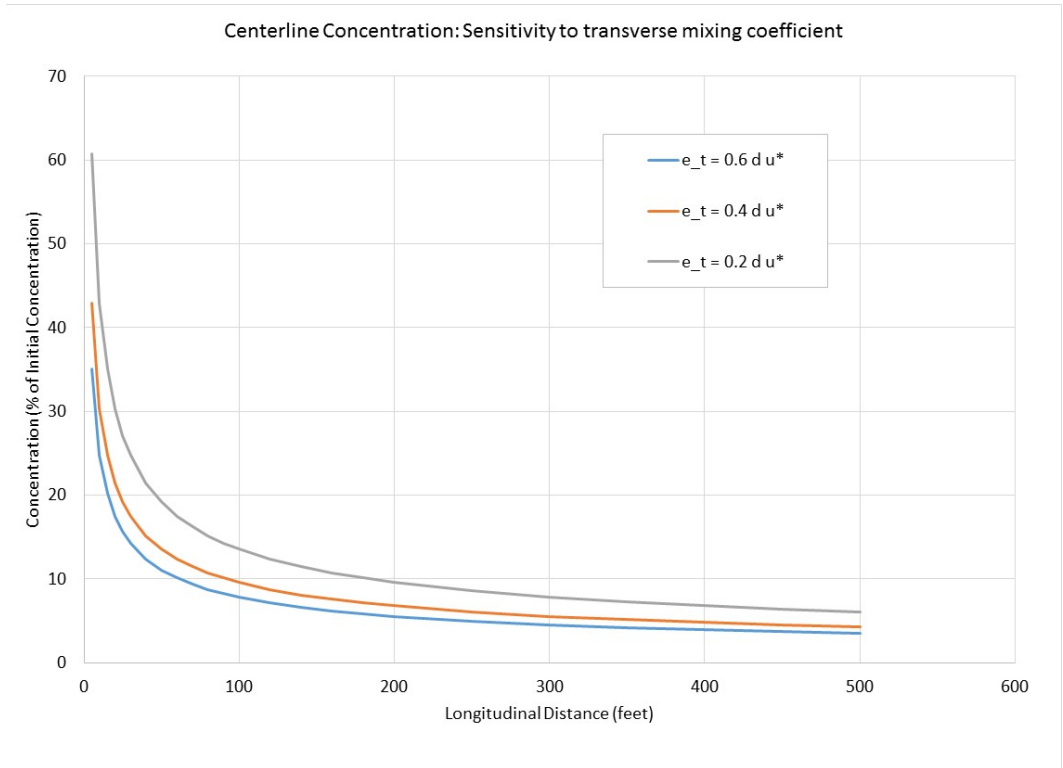


Figure 5. Plume Centerline Concentrations for range in transverse mixing coefficient

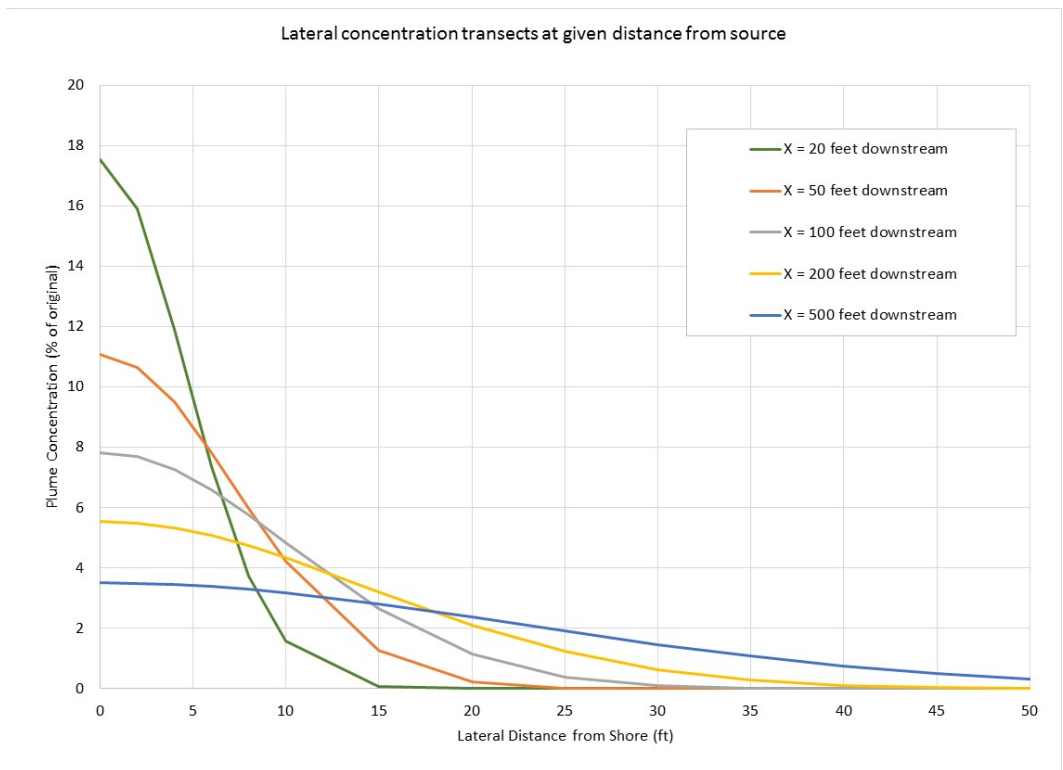


Figure 6. Plume Concentrations along lateral transects between 20 and 500 feet from source

Appendix A
Standard Operating Procedures

Sample Analysis using AquaFluor Handheld Fluorometer

Purpose

SOP-1 provides guidelines for analyzing groundwater and surface water samples using an AquaFluor handheld fluorometer. Accurate measurement of fluorescence is necessary to determine the presence and concentration of Rhodamine WT in water samples collected as part of the dye testing.

Scope

The method described for the analysis is applicable to samples analyzed by an AquaFluor handheld fluorometer. Additional information on using the AquaFluor can be obtained in the *AquaFluor Handheld Fluorometer and Turbidimeter User's Manual* (Turner Designs 2013) and at the Turner Designs website (turnerdesigns.com).

Equipment and Materials

- AquaFluor handheld fluorometer with four AAA batteries
- 10 millimeter (mm) by 10 mm polystyrene cuvettes
- Pipettor and disposable tips or pipettes
- Borosilicate glass sample jars and lids
- Nine 1000-milliliter (mL) volumetric flasks
- Thermometer
- Glassware cleaning supplies
- Clean soft towel or tissue
- Global positioning system
- Field logbook
- Distilled water
- One 10-mL graduated cylinder
- Rhodamine WT

Preparation of Calibration Standards

Additional information on preparing and analyzing calibration standards can be obtained in the *AquaFluor Handheld Fluorometer and Turbidimeter User's Manual* (Turner Designs 2013) and the *Application Note: Preparation of Standards for Dye Studies Using Rhodamine WT* (Turner Designs, undated), which are both available at the Turner Designs website (turnerdesigns.com). Calibration standards of 5, 25, 50, 100, and 500 parts per billion (ppb) will be prepared using background (pre-study) river water and 20% Rhodamine WT solution will be used to prepare the standards.

To prepare calibration standards, follow these steps:

1. Prepare a primary standard of 100 ppb concentration using Rhodamine WT.
2. Use a pipettor or pipette to transfer 10 mL of Rhodamine WT dye solution into a 1000-mL volumetric flask (Flask #1). Fill the flask to the 1000-mL level with river water. This flask contains a Rhodamine WT concentration of 10,000 milliliters per liter (mL/L).

3. Pipette 10 mL of the 10,000 mL/L solution from Flask #1 into a second 1000-mL flask (Flask #2). Fill the flask to the 1000-mL level with river water. Flask #2 contains 100 mL/L Rhodamine WT solution.
4. Pipette 10 mL of the 100 mL/L solution from Flask #2 into a 1000-mL flask (Flask #3). Fill the flask to the 1000-mL level with river water. Flask #3 contains 1 mL/L (1,000 ppb) Rhodamine WT solution.
5. Pipette 5 mL of the 100 mL/L solution from Flask #2 into a 1000-mL flask (Flask #4). Fill the flask to the 1000-mL level with river water. Flask #4 contains 0.5 mL/L (500 ppb) Rhodamine WT solution (500 ppb Standard).
6. Pipette 100 mL of the 1 mL/L solution from Flask #3 into a 1000-mL flask (Flask #5). Fill the flask to the 1000-mL level with river water. Flask #5 contains 0.1 mL/L (100 ppb) Rhodamine WT solution (100 ppb Standard).
7. Pipette 50 mL of the 1 mL/L solution (1,000 ppb) from Flask #3 into a 1000-mL flask (Flask #6). Fill the flask to the 1000-mL level with river water. Flask #6 contains 50 ppb Rhodamine WT solution (50 ppb Standard).
8. Pipette 25 mL of the 1 mL/L solution into a 1000-mL flask (Flask #7). Fill the flask to the 1000-mL level with river water. Flask #7 contains 25 ppb Rhodamine WT (25 ppb Standard).
9. Pipette 10 mL of the 1 mL/L solution into a 1000-mL flask (Flask #8). Fill the flask to the 1000-mL level with river water. Flask #8 contains 10 ppb Rhodamine WT (10 ppb Standard).
10. Pipette 5 mL of the 1 mL/L solution into a 1000-mL flask (Flask #9). Fill the flask to the 1000-mL level with river water. Flask #9 contains 5 ppb Rhodamine WT (5 ppb Standard).
11. Use distilled water as a DI blank sample.
12. Use a sample of background surface water (without any known dye in it) as a standard check of background fluorescence and turbidity.
13. Store standards and standard checks in a cooler or other dark container in clean, non-preserved glass jars.

Calibration Procedure

Calibration should be completed prior to sample analysis. The AquaFluor calibration should be checked daily using the primary standard; if the measured concentration has changed by more than 5 percent of its value, then the AquaFluor should be recalibrated.

To calibrate the AquaFluor, follow these steps:

1. Press the <STD VAL> button, and set the standard value to the concentration of the primary standard (100 ppb), and then press <ENT> or <ESC> to accept the value.
2. Transfer a portion of the blank standard to a 10 mm by 10 mm polystyrene cuvette filled at least $\frac{3}{4}$ full.
3. Measure the temperature of the blank standard.
4. Press the <CAL> button and then press the <ENT> button.
5. Insert the blank sample and then press <ENT>. Wait 10 seconds.
6. Transfer a portion of the primary standard to a clean 10 mm by 10 mm polystyrene cuvette filled at least $\frac{3}{4}$ full, and measure the temperature of the primary standard.
7. Insert the primary standard sample, and press <ENT>. Wait 10 seconds.
8. Press the <ENT> button once the calibration is complete to accept the calibration.

9. Use the standard from Flask #4 (Rhodamine WT concentration of 100 ppb in distilled water) to measure the concentration of Rhodamine WT under ideal conditions (use the Analysis Procedures described herein). Record the concentration and difference between the primary standard and the Flask #4 standard in the field logbook.
10. Analyze a sample of groundwater or surface water without any dye in it to determine if there is any background fluorescence or interference. Record the concentration (if any) in the field logbook.

Sample Handling and Preservation

For sample handling and preservation, follow these steps:

1. Collect the groundwater or surface water sample, and transfer the sample to a clean, unused, nonpreserved, glass jar. When ready to analyze the sample, transfer a portion of the sample to a clean polystyrene cuvette. Make sure the cuvette is at least $\frac{3}{4}$ full.
2. Analyze the sample as soon as possible. The Rhodamine WT dye can degrade in sunlight; thus, samples should be stored in a closed cooler pending analysis.
3. Clean up any spills in the AquaFluor sample chamber quickly by wiping with a clean soft towel or tissue.
4. Verify that the outside of the cuvette is dry during analysis.
5. Confirm that there are no bubbles in the sample.
6. Confirm that the temperature of the samples are similar to the temperature of the calibration standards. If the temperatures are different, record the temperature of the samples in the field logbook or on a sampling sheet.

Analysis Procedure

To analyze the sample, follow these steps:

1. Transfer sample into a 10 mm by 10 mm polystyrene cuvette, and record the sample temperature in the field logbook or on a sampling sheet.
2. Insert sample into the AquaFluor. Verify that the outside of the cuvette is dry.
3. Press the <READ> button. The instrument will measure and average the fluorescence signal for 10 seconds.
4. Record the reading from the top line of the Home Screen in the field logbook or on a sampling sheet.
5. Wait until the "WAIT" message disappears from the display; after which, another sample can be analyzed.
6. If sample result is greater than 400 ppb, dilute sample with known quantity of water, and re-run sample. For a 10x dilution, use a pipettor (with a new, unused disposable tip) to transfer 1 mL of water to a 10-mL graduated cylinder. Fill graduated cylinder with distilled water to the 10-mL line, shake sample, and transfer diluted sample to cuvette for re-analysis.

Calculation

To calculate results, follow these steps:

1. For sample results less than 400 ppb, the displayed value is the actual fluorescence value.
2. For sample results greater than 400 ppb, the sample needs to be diluted so that the diluted sample concentration is less than 400 ppb. In this case, the actual concentration is the displayed value times the dilution amount.

3. Because fluorescence decreases with temperature, a temperature coefficient of 0.0036 degree Celsius ($^{\circ}\text{C}$) will be applied to sample results in the office.

Troubleshooting

Refer to *AquaFluor Handheld Fluorometer and Turbidimeter User's Manual*. Contact Turner Designs at 1-877-316-8049 or the equipment rental provider.

Key Checks and Items

Check batteries daily.

Test calibration daily or more often by periodically analyzing a sample of the primary standard.

Make sure outside of cuvette is dry.

If unsure of result, or if you obtain a result exceeding the background level, re-analyze the sample.

References

Turner Designs. 2013. *AquaFluor Handheld Fluorometer and Turbidimeter User's Manual*. Revision 1.6. February.

Turner Designs. Undated. *Application Note: Preparation of Standards for Dye Studies Using Rhodamine WT, Revision A*.

SCUFA Fluorometer Use

Purpose

SOP-2 provides guidelines for deploying Turner Designs Self-Contained Underwater Fluorescence Apparatus (SCUFA) to measure Rhodamine WT concentrations in surface water. Accurate measurement of fluorescence is necessary to determine the presence and concentration of Rhodamine WT in water samples collected as part of the pilot dye testing.

Scope

The method described for the analysis is applicable to the SCUFA fluorometer. The SCUFAs will be deployed in the river at locations downstream of Rhodamine WT dye releases in the river to measure Rhodamine WT concentrations through time as the dye migrates downstream. Additionally, the SCUFA may be used to measure Rhodamine WT concentrations of collected river samples (background samples, samples collected during dye releases). Additional information on using the SCUFA can be obtained in the *SCUFA User's Manual* (Turner Designs 2004) available at the Turner Designs website (turnerdesigns.com).

Equipment and Materials

- SCUFA with internal data logger and battery pack
- Laptop computer with serial port
- SCUFAsoft interface software and PC interface and power cable
- Two 1-liter glass beaker
- Non-reflective, black felt material for calibrations in beaker
- Borosilicate glass sample jars and lids
- Pipettor and disposable tips or pipettes
- Nine 1000-milliliter (mL) volumetric flasks
- Glassware cleaning supplies
- Global positioning system
- Field logbook
- Clean soft towel or tissue
- One 10-mL graduated cylinder
- Distilled water
- Rhodamine WT

Preparation of Calibration Standards

Additional information on preparing and analyzing calibration standards can be obtained in the *SCUFA User's Manual* (Turner Designs 2004) and the *Application Note: Preparation of Standards for Dye Studies Using Rhodamine WT* (Turner Designs, undated), which are both available at the Turner Designs website (turnerdesigns.com). Calibration standards of 5, 25, 50, 100, and 500 parts per billion (ppb) will be prepared using background (pre-study) river water and 20% Rhodamine WT solution will be used to prepare the standards.

To prepare calibration standards, follow these steps:

1. Prepare a primary standard of 100 ppb concentration using Rhodamine WT.

2. Use a pipettor or pipette to transfer 10 mL of Rhodamine WT dye solution into a 1000-mL volumetric flask (Flask #1). Fill the flask to the 1000-mL level with river water. This flask contains a Rhodamine WT concentration of 10,000 milliliters per liter (mL/L).
3. Pipette 10 mL of the 10,000 mL/L solution from Flask #1 into a second 1000-mL flask (Flask #2). Fill the flask to the 1000-mL level with river water. Flask #2 contains 100 mL/L Rhodamine WT solution.
4. Pipette 10 mL of the 100 mL/L solution from Flask #2 into a 1000-mL flask (Flask #3). Fill the flask to the 1000-mL level with river water. Flask #3 contains 1 mL/L (1,000 ppb) Rhodamine WT solution.
5. Pipette 5 mL of the 100 mL/L solution from Flask #2 into a 1000-mL flask (Flask #4). Fill the flask to the 1000-mL level with river water. Flask #4 contains 0.5 mL/L (500 ppb) Rhodamine WT solution (500 ppb Standard).
6. Pipette 100 mL of the 1 mL/L solution from Flask #3 into a 1000-mL flask (Flask #5). Fill the flask to the 1000-mL level with river water. Flask #5 contains 0.1 mL/L (100 ppb) Rhodamine WT solution (100 ppb Standard).
7. Pipette 50 mL of the 1 mL/L solution (1,000 ppb) from Flask #3 into a 1000-mL flask (Flask #6). Fill the flask to the 1000-mL level with river water. Flask #6 contains 50 ppb Rhodamine WT solution (50 ppb Standard).
8. Pipette 25 mL of the 1 mL/L solution into a 1000-mL flask (Flask #7). Fill the flask to the 1000-mL level with river water. Flask #7 contains 25 ppb Rhodamine WT (25 ppb Standard).
9. Pipette 10 mL of the 1 mL/L solution into a 1000-mL flask (Flask #8). Fill the flask to the 1000-mL level with river water. Flask #8 contains 10 ppb Rhodamine WT (10 ppb Standard).
10. Pipette 5 mL of the 1 mL/L solution into a 1000-mL flask (Flask #9). Fill the flask to the 1000-mL level with river water. Flask #9 contains 5 ppb Rhodamine WT (5 ppb Standard).
11. Use distilled water as a DI blank sample.
12. Use a sample of background surface water (without any known dye in it) as a standard check of background fluorescence and turbidity.
13. Store standards and standard checks in a cooler or other dark container in clean, non-preserved glass jars.

Calibration Procedure

Calibration should be completed prior to deployment and all calibration measurement will be recorded. The SCUFA calibration should be checked daily using the range of standard solutions and these calibration results need to be recorded; if the measured concentration has changed by more than 5 percent of its value, then the SCUFA should be recalibrated.

To calibrate the SCUFA, follow these steps:

1. Connect the SCUFA to the computer and run SCUFAsoft software.
2. Select Temp. Compensation from menu toolbar.
3. Select Rhodamine WT from dropdown menu for Application. Select Apply. Confirm that the Temperature icon on the toolbar is green.
4. Select Calibration from menu toolbar.
5. Choose Turbidity Channel, select Next.
6. Enter calibration standard value (between 50 and 500 ppb) and units (ppb), select Next.
7. Do not select Blank Subtract, select Next.

8. Submerge sensor in calibration standard (See Analysis Procedure section for details). Click calibrate when reading stabilizes, then select Next.
9. Click Next once calibration values are sent to SCUFA. Click Next again.
10. Select Yes for Secondary Standard. Submerge sensor in distilled water. Click record when reading stabilizes, then select Next. Turbidity reading should be 0 Nephelometric Turbidity Units (NTUs).
11. Select Calibration from menu toolbar.
12. Choose Fluorescence Channel, select Next.
13. Enter calibration standard value and units, select Next.
14. Select box for Blank Standard. Submerge sensor in blank solution (river water). Press Blank button. Record Blank value in field logbook. Select Next.
15. Submerge sensor in calibration standard, click on Calibrate button. Select Next.
16. Wait for calibration values to be transferred to SCUFA. Click Next.
17. Click Next button when screen reads that Calibration for this channel is done.
18. Select No for Secondary Standard.
19. Select Diagnostics Screen

Preparing SCUFA for deployment

SCUFAs will be deployed on anchored moorings with surface floats. Prior to deploying, complete the following steps to setup the SCUFA for logging data:

1. Connect SCUFA to laptop and run SCUFAsoft.
2. Set clock on laptop and any other equipment being used (global positioning system, RiverRay, watches). SCUFAsoft updates time on SCUFA using the PC clock.
3. Select Data View from menu toolbar. Select Erase Data to erase all data stored in internal data logger.
4. Select Internal Data Logging Screen from menu toolbar.
5. Set sampling rate to desired rate (10 to 30 seconds). Select either Continuous Sampling or set a time block for recording data. Confirm that there is sufficient memory available for the memory required. Select OK.
6. Select Temp. Compensation from menu toolbar.
7. Select Rhodamine WT from dropdown menu for Application. Select Apply. Confirm that the Temperature icon on the toolbar is green. Unplug the SCUFA from laptop DO NOT EXIT Software before unplugging!
8. Attach battery to SCUFA and inspect window on SCUFA to confirm green light comes on and off at desired sampling intervals.
9. Insert the SCUFA and Battery into the Custom PVC housing for field installations.
10. Deploy SCUFA, recording location and depth where SCUFA is deployed, time dye is released, and time when SCUFA is retrieved. For near surface deployments, make sure sensor is pointing down to avoid effects from surface light.

Retrieving Data

1. Following each dye test, retrieve SCUFA (or if deployed with Turner Data cable, connect to data cable), connect SCUFA to laptop, and start SCUFAsoft software.
2. Select Data View from menu toolbar. Review fluorescence and turbidity plots to determine if data was recorded and shows Rhodamine WT peaks.
3. Select Download button to download data from SCUFA to PC. Assign a unique name to file that identifies the test, the SCUFA that was deployed, and any other identifying information and record in field logbook. File will be in *.tdf format, which is unique to the SCUFAsoft software. To view the data in SCUFAsoft, select Open file button. Confirm that data was recorded. Data can be exported to tab delimited (.txt) or excel file (.xls) by using the Export Data button.
4. If needed to provide space, erase data from internal data logger using the Erase Data button prior to next deployment.

Analysis Procedure

For surface water samples collected for analysis by the SCUFA, follow these steps:

1. Add collected water sample to 1-liter glass beaker.
2. Mount the SCUFA Fluorometer with the optics facing down in a glass beaker, with the SCUFA at least 3 inches above the bottom of the container and at least 1 inch clearance around sides.
3. Place the beaker on a non-reflective, black surface.
4. Verify that the detectors are free of any air bubbles; tilt the SCUFA when submerging to allow air to escape.
5. Record dye and turbidity values and time at which sensor is placed in sample.
6. Rinse the optics with distilled water between sample readings.

Troubleshooting

Refer to *SCUFA User's Manual*. Contact Turner Designs at 1-877-316-8049 or the equipment rental provider.

Key Checks and Items

Check batteries and recharge overnight every day of use.

Check that laptop clock is synched with other clocks used during testing (stopwatches, wristwatches, phone clocks, global positioning system clocks, etc.)

Check memory required against memory available during preparation for each deployment.

Test calibration daily or more often by periodically analyzing a sample of the primary standard.

If unsure of result, re-analyze the sample.

Make sure to clearly record where each SCUFA was deployed during each test and make sure SCUFA data files are saved with names and folders that it is clear which SCUFA and which test that data file refers to.

References

Turner Designs. 2004. *SCUFA User's Manual*. Revision 2.3. September.

Appendix B
Rhodamine WT Safety Data Sheet



Issuing Date 26-Mar-2015

Revision Date 05-May-2014

Version 5

1. Identification of the Substance/Preparation and of the Company/Undertaking

Product Identifier

Product Code 70301027
 Product name KEYACID™ RHODAMINE WT LIQUID

Recommended use of the chemical and restrictions on use

Synonyms N/A

Supplier's details

Recommended use Industrial Use Only.
 Uses advised against No information available

Details of the Supplier of the Safety Data Sheet

Keystone Corporate Office	Keystone Liquid Manufacturing and Technical Facility
2501 W Fulton Street	2165 Highway 292
Chicago, IL 60612	Inman, SC 29349
	Ph 864 473-1601

Ph 312-666-2015
 Ph 1-800-522-4393

Emergency Telephone Number

USA: 1-800-255-3924
 China: (400-1)-400-120-0751
 Other: Collect 1-813-248-0585

2. Hazards Identification

Classification

Not a dangerous substance or mixture according to the Globally Harmonized System (GHS)

EMERGENCY OVERVIEW

signal word

Not Hazardous

The product contains no substances which at their given concentration, are considered to be hazardous to health

appearance Red

Physical State Liquid

Odor No information available

precautionary statements

- P281 - Use personal protective equipment as required
- P262 - Do not get in eyes, on skin, or on clothing
- P261 - Avoid breathing dust/fume/gas/mist/vapors/spray
- P264 - Wash face, hands and any exposed skin thoroughly after handling
- P270 - Do not eat, drink or smoke when using this product
- P501 - Dispose of contents/ container to an approved waste disposal plant

Hazards Not Otherwise Classified (HNOC)

OTHER INFORMATION**Unknown Acute Toxicity**

98.2 percent of the mixture consists of ingredient(s) of unknown acute toxicity

3. Composition/information on Ingredients

This chemical is not considered hazardous by the 2012 OSHA Hazard Communication Standard (29 CFR 1910.1200).

4. First aid measures**First aid measures for different exposure routes**

Eye Contact	Rinse thoroughly with plenty of water for at least 15 minutes, lifting lower and upper eyelids. Consult a physician.
Skin Contact	Wash skin with soap and water.
INHALATION	Move to fresh air.
INGESTION	Rinse mouth.

Most important symptoms/effects, acute and delayed**Main Symptoms** No information available.**Indication of immediate medical attention and special treatment needed, if necessary****Notes to physician** Treat symptomatically.**5. Fire-fighting measures****Suitable extinguishing media**

Use extinguishing measures that are appropriate to local circumstances and the surrounding environment.

Unsuitable extinguishing media Keep away from heat and sources of ignition.**Specific Hazards Arising from the Chemical**

No information available.

Explosion Data**Sensitivity to Mechanical Impact** None.**Sensitivity to Static Discharge** None.**Protective Equipment and Precautions for Firefighters**

As in any fire, wear self-contained breathing apparatus pressure-demand, MSHA/NIOSH (approved or equivalent) and full protective gear.

6. Accidental release measures**Personal precautions, protective equipment and emergency procedures****Personal Precautions** Avoid contact with the skin and the eyes. Use personal protective equipment. For personal protection see section 8. Ensure adequate ventilation.**Environmental Precautions****Environmental Precautions** Prevent further leakage or spillage if safe to do so.**Methods and materials for containment and cleaning up****Methods for containment** Prevent further leakage or spillage if safe to do so.

Methods for Cleaning Up Use personal protective equipment. Dam up. Cover liquid spill with sand, earth or other noncombustible absorbent material. Take up mechanically, placing in appropriate containers for disposal. Clean contaminated surface thoroughly.

7. Handling and Storage

Precautions for Safe Handling

Advice on safe handling Avoid contact with skin, eyes and clothing. Do not breathe vapors/dust. In case of insufficient ventilation, wear suitable respiratory equipment. Wear personal protective equipment. Handle in accordance with good industrial hygiene and safety practice. Remove and wash contaminated clothing before re-use. Provide appropriate exhaust ventilation at places where dust is formed. Do not eat, drink or smoke when using this product. Use personal protection recommended in Section 8.

Conditions for safe storage, including any incompatibilities

Technical measures/Storage conditions Store locked up. Keep containers tightly closed in a dry, cool and well-ventilated place. Keep in properly labeled containers. Store in accordance with the particular national regulations. Store in accordance with local regulations.

Incompatible Products None known based on information supplied.

8. Exposure Controls/Personal Protection

Control Parameters

Exposure guidelines This product, as supplied, does not contain any hazardous materials with occupational exposure limits established by the region specific regulatory bodies

Exposure Controls

Engineering Measures Showers
Eyewash stations
Ventilation systems. Apply technical measures to comply with the occupational exposure limits. Process enclosure and/or ventilation systems.

Individual protection measures, such as personal protective equipment

Eye/Face Protection Face-shield. Chemical resistant goggles must be worn. Eyewash fountains should be provided in areas where there is any possibility that workers could be exposed to the substances; this is irrespective of the recommendation involving the wearing of eye protection. If splashes are likely to occur, wear.. Goggles.

Skin and Body Protection Skin and Body Protection. Wear protective gloves and protective clothing. Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.

Respiratory Protection If exposure limits are exceeded or irritation is experienced, NIOSH/MSHA approved respiratory protection should be worn.

Hygiene Measures Do not eat, drink or smoke when using this product.

9. Physical and Chemical Properties

Physical and Chemical Properties

Physical State	Liquid	Odor	No information available
appearance	Red		
odor threshold	No information available		

<u>Property</u>	<u>VALUES</u>	<u>Remarks • Method</u>
pH	10.00	
melting point		N/A
Boiling point/boiling range	< 110 °C / 230.00 °F °F	
Flash Point	< 121 °C / < 250.00 °F °F	
Evaporation Rate	no data available	
flammability (solid, gas)	no data available	no data available
Flammability Limits in Air		
Upper Flammability Limit	No information available	
Lower Flammability Limit	No information available	
vapor pressure	No information available	
Vapor Density	No information available	
Specific gravity	No information available	N/A
Solubility in Other Solvents	No information available	
Partition coefficient: n-octanol/water	No information available	
Autoignition Temperature	No Data Available	N/A
decomposition temperature	No information available	
Kinematic viscosity	No Data Available	
Dynamic viscosity	No information available	
Explosive Properties	No information available	
Oxidizing properties	No information available	

OTHER INFORMATION

softening point	No information available
Molecular Weight	
VOC Content(%)	No information available.
density	No information available

10. Stability and Reactivity**Reactivity**

no data available

Chemical Stability

Stable under recommended storage conditions.

Hazardous Reactions

None under normal processing

Conditions to avoid

Extremes of temperature and direct sunlight.

Incompatible materials

None known based on information supplied.

Hazardous decomposition products

May emit toxic fumes under fire conditions.

11. Toxicological Information**Information on Likely Routes of Exposure****Product information**

INHALATION	Avoid breathing vapors or mists.
Eye Contact	Avoid contact with eyes.
Skin Contact	Avoid contact with skin.
INGESTION	Not an expected route of exposure.

Information on Toxicological Effects

Symptoms No information available.

Delayed and immediate effects as well as chronic effects from short and long-term exposure

sensitization No information available.
 Germ cell mutagenicity No information available.
 carcinogenicity No information available.
 Reproductive Toxicity No information available.
 Specific target organ systemic toxicity (single exposure) No information available.
 Specific target organ systemic toxicity (repeated exposure) No information available.
 Aspiration Hazard No information available.

Numerical Measures of Toxicity - Product information

Unknown Acute Toxicity 98.2 percent of the mixture consists of ingredient(s) of unknown acute toxicity
 The following values are calculated based on chapter 3.1 of the GHS document .
 ATEmix (oral) 39333 mg/kg

12. Ecological Informationecotoxicity

98.2% of the mixture consists of components(s) of unknown hazards to the aquatic environment

Persistence and Degradability

No information available.

Bioaccumulation

No information available.

Other Adverse Effects

No information available

13. Disposal ConsiderationsWaste treatment

Waste Disposal Methods Should not be released into the environment. Dispose of in accordance with local regulations. This material, as supplied, is not a hazardous waste according to state and federal regulations (40 CFR 261).

Contaminated Packaging Do not re-use empty containers. Dispose of in accordance with local regulations.

14. TRANSPORT INFORMATIONDOT

Description NOT REGULATED

MEX

NOT REGULATED

IATA

Description NOT REGULATED

IMDG

Description NOT REGULATED

RID**ADN****15. Regulatory Information****International Inventories**

TSCA	Listed
EINECS/ELINCS	Listed
DSL	Not Determined
NDSL	Not Determined
PICCS	Not Determined
ENCS	Not Determined
IECSC	Not Determined
AICS	Not Determined
KECL	Not Determined
NZIoC	Not Determined

Legend

TSCA - United States Toxic Substances Control Act Section 8(b) Inventory
 EINECS/ELINCS - European Inventory of Existing Commercial Chemical Substances/EU List of Notified Chemical Substances
 DSL/NDSL - Canadian Domestic Substances List/Non-Domestic Substances List
 PICCS - Philippines Inventory of Chemicals and Chemical Substances
 ENCS - Japan Existing and New Chemical Substances
 IECSC - China Inventory of Existing Chemical Substances
 AICS - Australian Inventory of Chemical Substances
 KECL - Korean Existing and Evaluated Chemical Substances
 NZIoC - New Zealand Inventory of Chemicals

REACH registration number Keystone has taken all relative steps to ensure REACH-Compliance. Please contact us with any REACH-Related questions at REACH@Keystone-Europe.CO.UK

RESTRICTIONS - REACH TITLE VIII No information available

US Federal Regulations**SARA 313**

Section 313 of Title III of the Superfund Amendments and Reauthorization Act of 1986 (SARA). This product does not contain any chemicals which are subject to the reporting requirements of the Act and Title 40 of the Code of Federal Regulations, Part 372

SARA 311/312 Hazard Categories

Acute health hazard	NO.
Chronic health hazard	NO.
Fire hazard	NO.
Sudden Release of Pressure Hazard	NO.
Reactive Hazard	NO.

Clean Water Act

This product does not contain any substances regulated as pollutants pursuant to the Clean Water Act (40 CFR 122.21 and 40 CFR 122.42).

CERCLA

This material, as supplied, does not contain any substances regulated as hazardous substances under the Comprehensive Environmental Response Compensation and Liability Act (CERCLA) (40 CFR 302) or the Superfund Amendments and Reauthorization Act (SARA) (40 CFR 355). There may be specific reporting requirements at the local, regional, or state level pertaining to releases of this material

US State Regulations**California Proposition 65**

This product does not contain any Proposition 65 chemicals

U.S. State Right-to-Know Regulations

This product does not contain any substances regulated by state right-to-know regulations

International regulations

Mexico - Grade

Slight risk, Grade 1.

CANADA**WHMIS Note**

This product has been classified in accordance with the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all the information required by the CPR.

WHMIS Hazard Class

Non-controlled

HMIS	Health Hazard 1	flammability 1	Physical hazard 0	PERSONAL PROTECTION H
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16. Other Information

Revision Date	05-May-2014
Revision note	No information available.
Restrictions on use	No information available.
Contact Information Website	WWW.DYES.COM.

Disclaimer

The information provided on this MSDS is correct to the best of our knowledge, information and belief at the date of its publication. The information given is designed only as a guide for safe handling, use, processing, storage, transportation, disposal and release and is not to be considered as a warranty or quality specification. The information relates only to the specific material designated and may not be valid for such material used in combination with any other material or in any process, unless specified in the text

End of Material Safety Data Sheet

Material Safety Data Sheet

Complies with EC no. 1907/2006
Issue Date: 11/15/2013

Page 1 of 3

Section 1: Chemical Product and Company Identification

Cat#: 19922

Part Name: RHODAMINE WT WATER TRACING DYE

Supplier: Polysciences, Inc.
400 Valley Road
Warrington, PA 18976 USA
MSDS Telephone #215-343-6484 Emergency only #215-378-4526

Identified uses: Laboratory use, manufacture of substances

Section 2: Hazards Identification

Hazard Overview

Causes eye irritation.

GHS Classification

Eye Irritation Category 2A

Signal word: Warning



Hazard and Precautionary Statements

- H319 Causes serious eye irritation.
P280 Wear protective gloves/protective clothing/eye protection/face protection.
P301A IF SWALLOWED do not induce vomiting. Do not give anything to drink. Obtain medical attention without delay.
P301D IF SWALLOWED, induce vomiting as directed by medical personnel.
P302+P352 IF ON SKIN: Wash with plenty of soap and water.
P340 Remove victim to fresh air and keep at rest in a position comfortable for breathing.
P351 Rinse cautiously with water for several minutes.
P361 Remove/Take off immediately all contaminated clothing.
P501 Dispose of contents/container to proper waste area in accordance with institutional practices and local, state or federal regulations.

NFPA Rating

Hazard Ratings:

These ratings are Polysciences' Inc. own assessments of the properties of the material using the ANSI/NFPA 704 Standard. Additional information can be found by consulting in the NFPA published ratings lists (List 325 and List 49).

If no data is listed the information is not available.

Health	Flammability	Reactivity
2	0	1

Section 3: Composition/ Information on Ingredients

Note: Items listed with a CASRN... number have no CAS# available.

Item#	Name	EINECS	CAS#	% in product
1	Rhodamine WT	Unknown	CASRNHX199	6 - 10
2	Water	231-791-2	0007732185	91 - 100

Section 4: First Aid Measures

- Flush eyes with flowing water for at least 15 minutes.
If breathing is difficult, contact emergency personnel.
If swallowed, induce vomiting as directed by medical personnel.
Remove contaminated clothing.
Remove to fresh air.
Wash skin with deluge of water for at least 15 minutes.

Section 5: Fire Fighting Measures

- Flash point, deg F.: no data Method: no data
UEL: no data LEL: no data Autoignition temperature, deg. F.: no data
Flammability Classification: no data Flame Propagation Rate: no data
Hazardous Combustion Products: no data

Section 6: Accidental Release Measures

Any information listed below is to be considered in addition to internal guidelines for isolation of spill, containment of spill, removal of ignition sources from immediate area, and collection for disposal of spill by trained, properly protected clean up personnel.

Absorb liquids on absorbent material.

Contain spilled liquids.

Section 7: Handling and Storage

Store at room temp

Section 8: Exposure Controls/ Personal Protection**OSHA (ACGIH) Exposure Limits**

		TWA		STEL		CEILING	
		ppm	mg/	ppm	mg/m3	ppm	mg/m3
CAS#: 0007732185	IDLH: NE						
OSHA		NE	NE	NE	NE	NE	NE
ACGIH		NE	NE	NE	NE	NE	NE
CAS#: CASRNHX1992	IDLH: NE						
OSHA		NE	NE	NE	NE	NE	NE
ACGIH		NE	NE	NE	NE	NE	NE

The use of eye protection in the form of safety glasses with side shields and the use of skin protection for hands in the form of gloves are considered minimum and non-discretionary in work places and laboratories. Any recommended personal protection equipment or environmental equipment is to be considered as additional to safety glasses and gloves.

Use chemical splash goggles and face shield.

Chemical-resistant gloves should be worn whenever this material is handled. The glove material has to be impermeable and resistant to the product. Gloves should be removed and replaced immediately if there is any indication of degradation or chemical breakthrough.

Rinse and remove gloves immediately after use. Wash hands with soap and water. All glove recommendations presume that the risk of exposure is through splash and not intentional immersion of the hands into the product.

Since glove permeation data does not exist for this material, no recommendation for the glove material can be given for the product.

Permeation data must be obtained from the glove manufacturer to determine if the glove is suitable for the task.

Section 9: Physical and Chemical Properties

Formula:	no data	vapor pressure:	no data
Formula Weight:	no data	vapor density:	heavier than air
boiling point:	no data	Specific gravity:	1.01
melting point:	no data	ph:	10.5 @1.0 %
solubility:	miscible	appearance:	fluorescing red liquid

Section 10: Stability and Reactivity

Chemical Stabilit no data

Conditions to Avoid: no data

Incompatibility with other materials: no data

Hazardous Decomposition Products: no data

Hazardous Polymerization: will not occur

Section 11: Toxicological Information

Acute Data: no data

Subchronic data: no data

Section 12: Ecological Information

LC50: >320 mg/l rainbow trout (96 hour)

LC50: 170 mg/l daphnia magna

No developmental abnormalities or toxicity to oyster larvae at 100 mg/L.

Section 13: Disposal Considerations

The following chart lists the status of the chemical and its components in reference to 40 CFR Part 261.33. If the product is listed by code number the substance may be subject to special federal and state disposal regulations. If no codes are listed the material must be disposed in compliance with all Federal, State and Local Regulations.

CAS#	Waste Code	Regulated Name
0007732185	not listed	not listed
CASRNHX19922	not listed	not listed

Section 14: Transportation Data

Proper Shipping Name NOT REGULATED

Chemical Name

UN

Class

PG

Section 15: Regulatory Information

All components of this product are on the TSCA public inventory.

All components of this product are on the TSCA public inventory.

Prop 65 - Column A identifies those items which are known to the State of California to cause cancer. Column B identified items which are known to the State of California to cause reproductive toxicity.

CAS#	Column A	Column B
0007732185	no	no
CASRNHX19922	no	no

State Regulatory Information :If a CAS# is listed below this material is subject to the listed state right-to-know requirements.

CAS#

0007732185 not listed

CASRNHX199 not listed

SARA Toxic Release Chemicals(as defined in Section 313 of SARA Title III)

This list identifies the toxic chemicals, including their de minimis concentrations for which reporting is required under Section 313 of the Emergency Planning and Community Right-to-Know Act (EPCRA). The list is also referred to as the Toxics Release Inventory (TRI) List.

CAS#	Regulated name	de minimis conc. %	Rep. Thres.
0007732185	not listed	not listed	not listed
CASRNHX19922	not listed	not listed	not listed

SARA Extremely Hazardous Substances and TPQs

This list includes hazardous chemicals as defined in 29 CFR 1910.1200(c); and extremely hazardous substances regulated under Section 302 of SARA Title III with their TPQs (in pounds), as listed in 40 CFR 355, Appendices A and B.

CAS#	Regulated name	TPQ (pounds)	EHS-RQ(pounds)
0007732185	not listed	not listed	not listed
CASRNHX19922	not listed	not listed	not listed

CERCLA

The hazardous substances, and their reportable quantities (RQs) are listed in the federal regulations at 40 CFR Part 302, Table 302.4. Release of a CERCLA hazardous substance in an amount equal to or greater than its RQ, in any 24-hour period, must be reported to the National Response Center at (800) 424-8802.

CAS#	Regulated name	RQ (pounds)
0007732185	Not listed	Not listed
CASRNHX19922	Not listed	Not listed

Section 16: Other Information

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END OF MSDS