## Measurement Quality Objectives for Acute and Chronic Marine Sediment Toxicity Test Methods



The following Measurement Quality Objectives establish recommendations and requirements for acute and chronic marine sediment toxicity testing conducted for the State Water Resources Control Board's Surface Water Ambient Monitoring Program (SWAMP) projects. Non-SWAMP projects should meet the minimum requirements established in the 1994 U.S. EPA guidance document *Methods for Assessing the Toxicity of Sediment-associated Contaminants with Estuarine and Marine Amphipods* (600/R-94/025).

| Negative Control              | Frequency of Analysis  | Measurement Quality Objective   | Data Quality<br>Indicator or<br>Reasoning                         |
|-------------------------------|--|---|---|
| Sediment Control              | A sediment control, consistent with the appropriate U.S. EPA test method, must be used with each analytical batch.   | The sediment control must meet all test acceptability criteria for the species of interest.   | Evaluates the health<br>and sensitivity of the<br>test organisms. |
| Laboratory<br>Overlying Water | Laboratory overlying water, consistent<br>with the appropriate U.S. EPA test<br>method, must be used with each<br>analytical batch.  | Laboratory overlying water must be of<br>uniform quality for the species of<br>interest (refer to U.S. EPA method<br>manual 600/R-94/025).  | Evaluates the health<br>and sensitivity of the<br>test organisms. |
| Positive Control              | Frequency of Analysis  | Measurement Quality Objective   | Data Quality<br>Indicator or<br>Reasoning                         |
| Reference Toxicant<br>Tests   | One reference toxicant test per<br>analytical batch is required when using<br>organisms that are either commercially-<br>supplied or wild-caught. Monthly<br>reference toxicant testing is required for<br>laboratories utilizing in-house cultures. | The last plotted data point (LC50 or<br>EC50) should be within 2 standard<br>deviations of the cumulative mean<br>(n=20). Reference toxicant tests that fall<br>outside of recommended control chart<br>limits are evaluated to determine the<br>validity of associated tests. A reference<br>toxicant test outside of the 2 standard<br>deviations does not invalidate the<br>associated test results. | Used to assess intra-<br>laboratory precision.                    |

#### Table 1. Laboratory Quality Control for Acute and Chronic Marine Sediment Toxicity Test Methods

| Negative Control | Recommended Corrective Action  |
|------------------|--|
| Sediment Control | Laboratories must begin retesting affected samples and the associated control within 7 days of test failure or after resampling. The laboratory should try to determine the source of the control failure, document the investigation, and record the steps taken to prevent a recurrence. |
|                  |  |

# Table 2. Laboratory Quality Control Corrective Actions for Acute and Chronic Marine Sediment Toxicity Test Methods

| Positive Control            | Recommended Corrective Action   |
|-----------------------------|---|
| Reference Toxicant<br>Tests | If the LC50 exceeds ± 2 standard deviations of the running mean of the last 20 reference toxicant tests, the laboratory should investigate sources of variability, take actions to reduce identified sources of variability, and may perform an additional reference toxicant test during the same month. |

### Table 3. Field Quality Control for Acute and Chronic Marine Sediment Toxicity Test Methods

| Quality Control | Frequency of Analysis          | Measurement Quality Objective   | Data Quality Indicator or<br>Reasoning   |
|-----------------|--------------------------------|---|--|
| Field Blanks    | Based on project requirements. | No statistical difference between the laboratory control and the field blank within an analytical batch.  | Used to measure bias<br>introduced during sample<br>collection and handling.         |
| Bottle Blanks   | Based on project requirements. | No statistical difference between the laboratory control and the bottle blank within an analytical batch. | Used to measure bias<br>introduced during washing<br>procedures prior to collection. |

| Quality Control | Recommended Corrective Action   |
|-----------------|---|
| Field Blanks    | If contamination of the field blanks and associated samples is known or suspected, the laboratory should flag the affected data. The project coordinator should be notified so that the sampling team can identify the contamination source(s) and perform corrective actions prior to the next sampling event.         |
| Bottle Blanks   | If contamination of the bottle blanks and associated samples is known or suspected, the laboratory should flag the affected data. The project coordinator should be notified so that the laboratory or vendor can identify the contamination source(s) and perform corrective actions prior to the next sampling event. |

# Table 4. Field Quality Control Corrective Actions for Acute and Chronic Marine Sediment Toxicity Test Methods

#### Table 5. Sample Handling for Acute and Chronic Marine Sediment Toxicity Test Methods

| Container                    | Sample Receipt<br>Temperature | Sample Preservation   | Holding Time  |
|------------------------------|-------------------------------|---|---|
| Amber glass<br>(recommended) | 0 – 6 °C (required)           | Wet or blue ice in field; 0 – 6 °C refrigeration in laboratory<br>(do not freeze); dark at all times (required) | <14 days<br>(recommended) or<br><8 weeks (required) |

| Table 6. 10-Day Acute Marine Sedime  | ent Ampelisca abalta Survival Toxicity Test   |  |
|--|---|--|
| Test Acceptability Criteria  | ≥90% mean survival in the controls (required)   |  |
| Test Type  | Whole sediment, static non-renewal (required)   |  |
| Size at Test Initiation  | 3 – 5 mm (no mature males or females; required)   |  |
| Replication at Test Initiation       4 (required minimum)                                  |   |  |
| Organisms per Replicate  | 20 (required minimum)   |  |
| Food Source  | Do not feed (required)  |  |
|  | 20 °C ± 1°C (recommended); the time-weighted average of daily temperature readings must |  |
| Temperature Range  | be within 1 °C of the desired temperature; the maximum temperature must not deviate     |  |
|  | from the minimum temperature by more than 3 °C (required)                               |  |
| Test Duration  | 10 days (required)  |  |
| Endpoint   | Survival (required)   |  |
| Salinity   | 28 ppt ± 2 ppt (recommended)  |  |
| Light Intensity  | $10 - 20 \mu\text{E/m}^2/\text{s or } 50 - 100 \text{ft-c}$ (recommended)               |  |
| Photoperiod  | Continuous luminance (recommended)  |  |
| Test Chamber Size  | 1 L (recommended)   |  |
| Replicate Volume   | Sediment volume: 175 mL (~2 cm); overlying water volume: 800 mL (recommended)           |  |
| Minimum Sample Volume  | 3 L for one-time grab sample (recommended)  |  |
| Laboratory Control Water Clean natural seawater or reconstituted water (recommended)       |   |  |
| Sediment Control   | Clean sediment from organism collection site (sieved through 500 $\mu$ m screen;        |  |
| Sediment Control   | recommended)  |  |
| Initial Overlying Water Chemistry  | 1 DO, pH, salinity, ammonia, and temperature measurement (required)                     |  |
| Initial Interstitial Water Chemistry 1 pH, salinity, and ammonia measurement (recommended) |   |  |
| Daily Overlying Water Chemistry  | 1 temperature measurement (required)  |  |
| Final Overlying Water Chemistry  | 1 DO, pH, salinity, ammonia, and temperature measurement (required)                     |  |
| Final Interstitial Water Chemistry   | 1 pH, salinity, and ammonia measurement (recommended)                                   |  |
| Initial DO Range   | >90% saturation (recommended)   |  |
|  |   |  |

Table 6. 10-Day Acute Marine Sediment Ampelisca abdita Survival Toxicity Test

| Test Acceptability Criteria           | ≥90% mean survival in the controls (required)  |  |
|---------------------------------------|--|--|
| Test Type                             | Whole sediment, static non-renewal (required)  |  |
| Size at Test Initiation               | 3 – 5 mm (no mature males or females; required)  |  |
| <b>Replication at Test Initiation</b> | 4 (required minimum)   |  |
| Organisms per Replicate               | 20 (required minimum)  |  |
| Food Source                           | Do not feed (required)   |  |
|                                       | 15 °C ± 1 °C (recommended); the time-weighted average of daily temperature readings must |  |
| Temperature Range                     | be within 1 °C of the desired temperature; the maximum temperature must not deviate      |  |
|                                       | from the minimum temperature by more than 3 °C (required)                                |  |
| Test Duration                         | 10 days (required)   |  |
| Endpoint                              | Survival (required)  |  |
| Salinity                              | 20 – 34 ppt ± 2 ppt (recommended)  |  |
| Light Intensity                       | 10 – 20 μE/m²/s or 50 – 100 ft-c (recommended)   |  |
| Photoperiod                           | Continuous luminance (recommended)   |  |
| Test Chamber Size                     | 1 L (recommended)  |  |
| Replicate Volume                      | Sediment volume: 175 mL (~2 cm); overlying water volume: 800 mL (recommended)            |  |
| Minimum Sample Volume                 | 3 L for one-time grab sample (recommended)   |  |
| Laboratory Control Water              | Clean natural seawater or reconstituted water (recommended)                              |  |
| Sediment Control                      | Clean sediment from organism collection site (sieved through 500 µm screen;              |  |
| Sediment Control                      | recommended)   |  |
| Initial Overlying Water Chemistry     | 1 DO, pH, salinity, ammonia, and temperature measurement (required)                      |  |
| Initial Interstitial Water Chemistry  | 1 pH, salinity, and ammonia measurement (recommended)                                    |  |
| Daily Overlying Water Chemistry       | 1 temperature measurement (required)   |  |
| Final Overlying Water Chemistry       | 1 DO, pH, salinity, ammonia, and temperature measurement (required)                      |  |
| Final Interstitial Water Chemistry    | 1 pH, salinity, and ammonia measurement (recommended)                                    |  |
| Initial DO Range                      | >90% saturation (recommended)  |  |
|                                       |  |  |

 Table 7. 10-Day Acute Marine Sediment Echaustorius estuarius Survival Toxicity Test

| annene water interface mythus ganoprovincians Larva bevelopment roxiety rest                               |  |
|--|--|
| est Acceptability Criteria ≥50% mean survival in the controls, and ≥90% mean normal development (required) |  |
| Whole sediment, static non-renewal (required)  |  |
| Within 4 hours of fertilization (required)   |  |
| 6 (required minimum)   |  |
| 250 ± 10% embryos (required minimum)   |  |
| Do not feed (required)   |  |
| 15 °C ± 1 °C (recommended); the maximum temperature must not deviate from the                              |  |
| minimum temperature by more than 3 °C (required)   |  |
| 48 hours (required)  |  |
| Normal development (required)  |  |
| 34 ppt ± 2 ppt (recommended)   |  |
| 10 – 20 μE/m²/s or 50 – 100 ft-c (recommended)   |  |
| 16 hours of ambient laboratory light, 8 hours dark (recommended)   |  |
| Intact sediment cores or 500 mL beaker coupled with 25 $\mu m$ screen tube (recommended)                   |  |
| Sediment volume: 175 mL (~2 cm); overlying water volume: 800 mL (recommended)                              |  |
| 3 L for one-time grab sample (recommended)   |  |
| Clean natural seawater or reconstituted water (recommended)  |  |
| Clean sediment from organism collection site (sieved through 500 $\mu$ m screen; recommended)              |  |
| 1 DO, pH, salinity, ammonia, and temperature measurement (required)  |  |
| 1 temperature measurement (required)   |  |
| 1 DO, pH, salinity, ammonia, and temperature measurement (required)  |  |
| 4.0 mg/L – 100% saturation (recommended)   |  |
|  |  |

 Table 8. 48-Hour Chronic Marine Sediment-Water Interface Mytilus galloprovincialis Larval Development Toxicity Test

| ≥80% normal development in the controls (required)                                       |
|--|
| Whole sediment, static non-renewal (required)  |
| <1 hour, post fertilization (required)   |
| 6 (required minimum)   |
| 250 embryos (required minimum)   |
| Do not feed (required)   |
| 15 °C ± 1 °C (recommended); the maximum temperature must not deviate from the            |
| minimum temperature by more than 3 °C (required)   |
| 72 hours (required)  |
| Normal development (required)  |
| 34 ppt ± 2 ppt (recommended)   |
| 10 – 20 μE/m <sup>2</sup> /s or 50 – 100 ft-c (recommended)                              |
| 16 hours of ambient laboratory light, 8 hours dark (recommended)                         |
| Intact sediment cores or 500 mL beaker coupled with 25 µm screen tube (recommended)      |
| Sediment volume: 175 mL (~2 cm); overlying water volume: 800 mL (recommended)            |
| 3 L for one-time grab sample (recommended)   |
| Clean natural seawater or reconstituted water (recommended)                              |
| Clean sediment from organism collection site (sieved through 500 µm screen; recommended) |
| 1 DO, pH, salinity, ammonia, and temperature measurement (required)                      |
| 1 temperature measurement (required)   |
| 1 DO, pH, salinity, ammonia, and temperature measurement (required)                      |
| 4.0 mg/L – 100% saturation (recommended)   |
|  |

 Table 9. 72-Hour Chronic Marine Sediment-Water Interface Strongylocentrotus purpuratus Larval Development Toxicity Test