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APPLICATION NOTE

Establishing Equilibration Of Chemical Concentration In Pore Water Prior To Conducting Sediment Toxicity Tests

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INTRODUCTION

In sediment toxicity tests, sufficient time is needed for a spiked chemical to equilibrate within the test system prior to exposing the organisms. Shorter equilibration times may be needed for chemicals that degrade rapidly in sediment while longer periods may be needed for those that are more persistent. For organic chemicals, guidelines recommend aging sediment for at least one month before starting a test.[1] For chemicals with a high log KOW (e.g., >6), a period of two months or longer may be necessary, and for metals, a shorter time frame of 1 – 2 weeks may be sufficient1. OECD guideline 2182 recommends a period of 24 – 48 hours of equilibration prior to exposure; however, ASTM E 1706-053 states that this short time period may not be long enough for sediments to equilibrate with spiked chemicals. The EPA recommendation is that periodic monitoring of chemical concentration in the pore water during the sediment aging process should be used as a means to assess the equilibration of the spiked sediments. One technique referenced by the EPA suggested that spiked sediment may be aged under refrigerated conditions in the dark for one month prior to use in the test, and that samples of the aged sediment should be analyzed on a weekly basis to establish equilibrium in the sediment and pore water.[1] While this technique can demonstrate equilibrium under storage conditions, it does not reflect equilibrium under testing conditions. OCSPP 850.1000 suggests that flow-through systems may need to operate for a significant period of time to obtain constant and representative concentrations.[4] Equilibration trials were conducted by EAG Laboratories in flow-through test systems by spiking test chemicals into either natural or formulated sediments and analyzing the pore water concentrations after allowing the spiked sediments to equilibrate in the test system for a set number of days. Results of these trials demonstrate that the appropriate equilibration time for a test chemical can be established within the test system instead of under refrigerated storage conditions.

METHODS

Spiking the Sediment

In each trial represented here the test material was spiked into the sediment.

- **The test material was either incorporated into the test sediment as a neat material (without the use of a solvent carrier) or with the use of a solvent carrier (either reverse-osmosis water, or a volatile solvent such as acetone or methanol).**
- **The spiked sediment was mixed overnight on a rotary mixer or roller mill to achieve homogeneity.**
- **The spiked sediment was distributed among six replicate test compartments, placed in a test system, and held under test conditions for the duration of the trial.**

Analyzing The Pore Water

- **One replicate, at each concentration prepared (typically two), was removed from the test system on each sampling day and the pore water was analyzed to determine concentrations of the test chemical.**
- **Analytical sampling occurred after 2 (or 3), 7, 10, 14, 21 and 28 days of equilibration.**

RESULTS

Determining Equilibration Time In The Test System

The analytical results are examined for signs of equilibrium. In Figure 1, when two intervals do not differ significantly (Chemical Y), the equilibration time might be set for 7 days. When there is a rapid significant loss (Chemical X), the equilibration time would be set for 2 days.

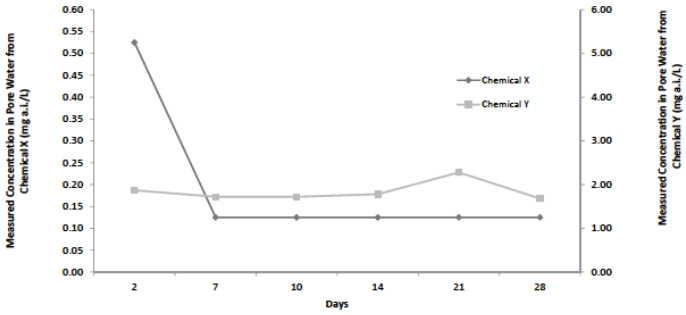

Figure 1: Example of measured concentrations in pure water used to determine time needed to equilibrate the test system prior to adding organisms.

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Examining Trends For Equilibration Time

Based on the results of 25 equilibration trials with different pesticides with a log K_{OW} ranging from 1 to >6, the amount of time needed to equilibrate in a test system ranged from two to 21 days with an overall mean of seven days. The average amount of time for a fungicide to equilibrate in a test system is six days, an herbicide is seven days and an insecticide is nine days (Figure 2).

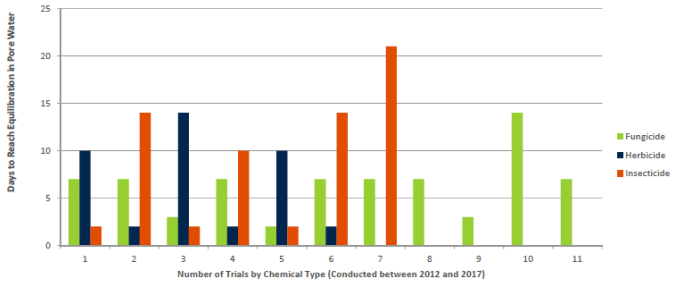

Figure 2: Equilibration time in days for sediment incorporated pesticides.

Equilibration time did not consistently correlate with log K_{OW}, with equilibration times ranging from two to 14 days, and an average of seven days (Figure 3).

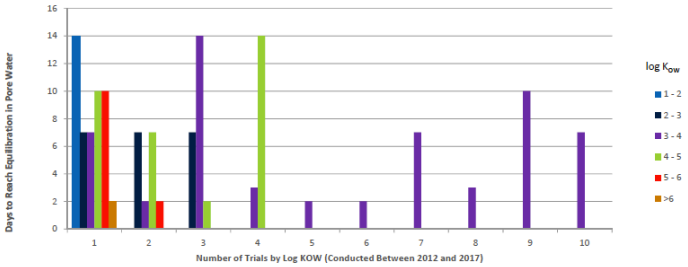

Figure 3: Equilibration time in days for pesticides based on their log K_{ow}

CONCLUSIONS

The results of the 25 trials conducted by EAG Laboratories demonstrate that equilibration of a test chemical in spiked sediment can be effectively determined under conditions of the test. These trials with organic pesticides with a wide range of log K_{OW} values provided equilibration times <21 days. In fact, the overall average time needed to achieve equilibrium in the pore water for spiked sediment equilibrated in the test system was approximately 7 – 9 days.

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REFERENCES

1. U.S. Environmental Protection Agency. 2000. EPA 600/R-99/064: Methods for Measuring the Toxicity and Bioaccumulation of Sediment-Associated Contaminants with Freshwater Invertebrates, second edition.

2. Organization for Economic Cooperation and Development. 2004. OECD Guideline 218: Sediment-Water Chironomid Toxicity Test Using Spiked Sediment. Adopted 13 April 2004.

3. American Society for Testing and Materials. 2005. ASTM Standard E 1706-05: Standard Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Freshwater Invertebrates.

4. U.S. Environmental Protection Agency. 2016 Series 850-Ecological Effects Test Guidelines, OCSPP Number 850.1000: Background and Special Considerations-Tests with Aquatic and Sediment-Dwelling Fauna and Aquatic Microcosms.