



**California Regional Water Quality Control Board
Central Valley Region
Katherine Hart, Chair**



Linda S. Adams
Acting Secretary for
Environmental Protection

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Edmund G. Brown Jr.
Governor

APPROVED
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18 February 2011

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East San Joaquin Water Quality Coalition
1201 L Street
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Mr. Michael Wackman
San Joaquin & Delta Water Quality Coalition
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REVIEW OF PYRETHROIDS ANALYTICAL METHOD VALIDATION DATA PACKAGE

Thank you for the 6 December 2010 submittal of a method validation package from Caltest Analytical Laboratories (Caltest) for the analysis of pyrethroids in sediment using the modified EPA Method 8270 using GC/MS-NCI SIM. The East San Joaquin Water Quality Coalition and the San Joaquin County and Delta Water Quality Coalition (Coalitions) are both requesting approval to use this method (which would modify the Coalitions' QAPPs) because there is currently no EPA-approved method for analyzing pyrethroids in sediment.

Central Valley Regional Water Quality Control Board (Central Valley Water Board) Staff reviewed the validation package and verified that requirements in Monitoring and Reporting Program (MRP) Order No. R5-2008-0005 have been met (see enclosure). Additionally, Staff consulted with the State Water Resources Control Board QA Officer to confirm that all requirements were appropriately met.

All documentation provided in the "Initial Demonstration of Laboratory Capability" for analysis of pyrethroids in sediment is complete and meets the requirements of the MRP Order and the Method Detection Limit and Reporting Limit ELAP criteria. Verification of the method's performance will be determined using recovery rates and other QA/QC data from actual field samples. Therefore, the Coalitions may report results for analyzing pyrethroids in sediment using the modified EPA Method 8270 using GC/MS-NCI SIM. Field performance must be reported.

If you have any questions or comments regarding this letter, or need any further information, please contact Susan Fregien at (916) 464-4813, or by email at sfregien@waterboards.ca.gov.

[Signature]
for Pamela C. Creedon
Executive Officer

Enclosure(s)

cc: Melissa Turner, MLJ-LLC
Patrick Ingram, Caltest Analytical Laboratories
Bill Ray, State Water Resources Control Board

Dania Huggins, CVRWQCB
Chris Jimmerson, CVRWQCB



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Linda S. Adams
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TO: Susan Fregien
Senior Environmental Scientist
Monitoring & Implementation Unit

FROM: Margaret Wong
Water Resources Control Engineer
Monitoring & Implementation Unit

DATE: 17 February 2011

SIGNATURE: _____

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SUBJECT: REVIEW OF PYRETHROIDS ANALYTICAL METHOD VALIDATION DATA

On 6 December 2010, Central Valley Water Board staff received data from Caltest Analytical Laboratories for method validation of a modified EPA Method 8270 using GC/MS-NCI SIM for detection of pyrethroids in sediments. The data included the Caltest Standard Operating procedure (SOP); a copy of the method validation package submitted by the Pyrethroid Working Group; initial calibration data; initial method detection limit study; initial precision and recovery; and other QC data for method blank and surrogate recovery. Presently, there are no EPA-approved methods for analyzing pyrethroids in sediments.

The Pyrethroid Working Group validation package to the United States Environmental Protection Agency (USEPA) also used a GC/MS-NCI SIM method¹. The validation package was submitted under the Federal Insecticide, Fungicide and Rodenticide Act (FIFRA) Environmental Chemistry Methods requirements. It was not submitted to USEPA Office of Water for validation as an alternate or new method for wastewater and drinking water^{2,3}.

I requested guidance from Bill Ray, the State Water Board QA/QC Officer, in determining if the Pyrethroid Working Group method validation package was complete and met the requirements for an alternate test method. EPA Method 8270 is approved for semivolatile organics using GC/MS and is an accepted method for other pesticides in sediments. Other agency laboratories using GC/MS for analyzing pyrethroids in sediment are California Department of Fish and Game (CDFG), California Department of Food and Agriculture, and the US Geological Survey (USGS)⁴. Each of these laboratories use a different extraction method that may impact the recovery of pyrethroids from the sediment.

¹ Reed, R. L., "Validation of the Residue Analytical Method: Residue Analytical Method for the Determination of Residue of Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, Lambda-Cyhalothrin and Permethrin in Sediment – Final Report."
² USEPA, Office of Water, EPA 821-B-98-002. "Protocol for EPA Approval of Alternate Test Procedures for Organic and Inorganic Analytes in Wastewater and Drinking Water." March 1999.
³ USEPA, Office of Water, EPA 821-B-98-003. "Protocol for EPA Approval of New Methods for Organic and Inorganic Analytes in Wastewater and Drinking Water." March 1999.
⁴ Hladik, M. "Methods Development for the analysis of Pyrethroid Pesticides in Environmental Samples – Final Report for CALFED". December 31, 2007.



After examining the documentation provided by Caltest, Bill Ray sent an email stating the package looks complete and the guidelines for alternate method procedures were followed. This method can be considered for analysis of pyrethroids in sediment, but verification of the method's performance will be determined when field samples are taken and analyzed with the required QA/QC samples. Validation data can appear to meet QA/QC requirements in controlled laboratory samples, but matrix interferences can occur when actual samples are analyzed. Recovery rates for the MS/MSD and other QA/QC information will be used to determine if the method can perform as the validation data indicates.

cc: Bill Ray, SWRCB
Dania Huggins
Chris Jimmerson