



Department of Pesticide Regulation



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MEMORANDUM

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SUBJECT: DETERMINATION IF CALIFORNIA DEPARTMENT OF FOOD AND AGRICULTURE'S LINURON, ISOXABEN, MEFENOXAM, METHOMYL AND PROPYZAMIDE METHOD (EMON-SM-05-025) MEETS THE "UNEQUIVOCAL DETECTION" CRITERIA

BACKGROUND

The Pesticide Contamination Prevention Act (Food and Agricultural Code [FAC] sections 13141 et seq.) was passed in 1985 to prevent further pesticide pollution of ground water which may be used for drinking water supplies. FAC section 13149 specifies the conditions under which a pesticide is considered "found" in ground water or soil, and thus subject to formal review as specified. As originally adopted, FAC subsection 13149(d) specified that a finding of a pesticide shall be verified by a second analytical method or a second analytical laboratory approved by the Department of Pesticide Regulation (DPR). However, the law was amended by Senate Bill 810 in 1995 to allow a finding of a pesticide in ground water or soil to be based on a single analytical method conducted by a single analytical laboratory, if the analytical method provides unequivocal identification of a chemical. Following this change, criteria were established to identify methods providing unequivocal identification of a chemical in a February 13, 1996, DPR memo entitled "Definition of unequivocal detection method for the purposes of Senate Bill 810 (Biermann, 1996)."

ISSUE

Does the analytical method for Linuron, Isoxaben, Mefenoxam, Methomyl and Propyzamide used by the California Department of Food and Agriculture (CDFA) meet the definition of an unequivocal detection method?

DISCUSSION AND RECOMMENDATION

CDFA Center for Analytical Chemistry, Environmental Analysis Section (method EMON-SM-05-025) uses an Ultra Performance Liquid Chromatography coupled to triple quadrupole mass spectrometry (UPLC/MS/MS) system for the detection of Linuron, Isoxaben, Mefenoxam, Methomyl and Propyzamide. Prior to injection of sample into the UPLC/MS/MS apparatus, the



well water samples are extracted by passing the sample through a solid phase extraction cartridge, and then concentrated to just dryness. Consequently the well water samples generally contain a minimal amount of background/matrix interference, facilitating the goal of unequivocal detection.

In CDFA method EMON-SM-05-025 for Linuron, Isoxaben, Mefenoxam, Methomyl and Propyzamide analysis, the first mass spectrometer is set to reject all species with mass/charge values that do not correspond to the analyte's molecular ion eluting at that analyte's particular retention time. Each molecular ion is then fragmented in the next stage, and the final mass spectrometer quantifies the pesticides based on either one or two characteristic fragments. Three stepwise factors are used to eliminate possible interferences for Linuron, Isoxaben, Mefenoxam, Methomyl and Propyzamide: chromatographic retention times, molecular ion masses and specific daughter ion masses.

In CDFA method EMON-SM-05-025, the following criteria are used to confirm the presence of Linuron, Isoxaben, Mefenoxam, Methomyl and Propyzamide:

1. Each set of samples will have a matrix blank and a spiked matrix sample.
2. The matrix blank shall be free of target compounds above MDL.
3. The recoveries of the matrix spikes shall be within the control limits.
4. The retention time of unknowns shall be within ± 2 percent of standards for each respective analyte. If there is a known reason of retention time shifting, an explanation memo shall be included.
5. Peak responses of each of the unknown analytes shall be within the calibration range. The sample extract shall be diluted if results fall outside of the calibration curve.
6. Each analyte has a precursor/parent ion and corresponding product/daughter ion that needs to be present in order to quantify that analyte.

Analysis of the Linuron, Isoxaben, Mefenoxam, Methomyl and Propyzamide by method EMON-SM-05-025 is highly specific and qualifies for the **unequivocal detection** designation. Therefore, analysis by a second laboratory or a second method is not necessary for well water samples analyzed for Linuron, Isoxaben, Mefenoxam, Methomyl and Propyzamide by this method.

