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OFFICE OF
PESTICIDES AND TOXIC
SUBSTANCES

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MEMORANDUM

SUBJECT: Review of the Analytical Method for Sulfentrazone in Seawater

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Conclusions:

EFGWB has completed the review of the analytical method for the determination of sulfentrazone in seawater in a toxicity test for Eastern oyster. The method involved a direct injection of the unfiltered seawater sample fortified with sulfentrazone into a high performance liquid chromatography equipped with an UV detector (HPLC/UV). The limit of detection for sulfentrazone was 0.3 ppm. Recoveries for sulfentrazone fortified at 0.6 and 20 ppm levels were 97% and 101%, respectively. The method is valid and can be used to determine sulfentrazone concentrations (i.e., 1.3, 2.2, 3.6, 6, and 10 ppm) in the Eastern oyster study.

It should be noted that another analytical method is available for the determination of sulfentrazone and its major degradate (3-carboxylic acid sulfentrazone) in water at low concentrations. These analytes were quantified by gas chromatography utilizing an electron capture detector (GC/ECD). The limit of detection for sulfentrazone in the GC/ECD method was 0.1 ppb (note: it is 3,000 times lower than that for the HPLC/UV method). Average method recoveries for the GC/ECD method were 102% for sulfentrazone and 92% for 3-carboxylic acid sulfentrazone at fortification levels from 0.5 ppb to 125 ppb. The GC/ECD method can be used to measure sulfentrazone and 3-carboxylic acid sulfentrazone in water at ppb exposure levels.

Background:

Sulfentrazone (also known as methanesulfonamide; F6285) is a herbicide currently being developed by FMC Corporation for control of annual grass, and annual and perennial broadleaf weeds on soybeans. It can be preplant soil-incorporated applied, or preemergence surface applied at a rate of 0.25-0.5 lbs a.i. per acre. If treatments are to be incorporated, incorporate to a depth of 1-3 inches. Only one application per season is allowed.

Discussion:

A Shimadzu high performance liquid chromatography (HPLC) was used in the method validation study. The HPLC was equipped with an UV detector and a Zorbax SB (C-18) column (4.6-nm x 250-nm). Information on the model of the HPLC was not provided in the report. The detector wavelength was set at 220 nm. The mobile phase was acetonitrile:water:phosphoric acid (55:45:0.1) and the flow rate was set at 1 mL per minute. The retention time for sulfentrazone was 5,077 minutes. Triplicate samples of unfiltered seawater were spiked with sulfentrazone at two levels (i.e., 6 and 20 ppm). A matrix blank was also prepared in triplicate from an unfortified aliquot of unfiltered seawater. The limit of detection was 0.3 ppm. Average recoveries were 97% and 101% for 0.6 ppm and 20 ppm, respectively (Table 1). Therefore, the method is valid to determine sulfentrazone in the testing solutions ranging from 1.3 to 10 ppm in the Eastern oyster study. Although the report did not specify the source of the seawater, it appears that the seawater used in the study was reconstituted seawater.

It should be noted that there is another analytical method available for the determination of sulfentrazone in water (Becker. 1996). This method was developed to analyze sulfentrazone and its major degradate, 3-carboxylic acid sulfentrazone, in the groundwater samples. The analytical method started with the addition of acid to acidify the sample, then reflux extraction, which quantitatively decarboxylated the 3-carboxylic acid sulfentrazone to 3-desmethyl sulfentrazone. Further clean-up of the water sample extract included a C_{18} SPE cartridge followed by a silica gel SPE cartridge using

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various hexane:ethyl acetate mixtures. The resulting eluate was concentrated to near dryness and then brought up with acetonitrile. Analytes were quantified by gas chromatography (GC) utilizing an electron capture detector (ECD). The limit of detection was 0.1 ppb for each analyte (note: the limit of detection for sulfentrazone in the GC/ECD method is 3,000 times lower than that for the HPLC/UV method developed for the Eastern oyster study). The method recoveries for the GC/ECD method were 102% and 92% for sulfentrazone and 3-carboxylic acid sulfentrazone, respectively (Table 2). Due to its low detection limits, the GC/ECD method can be used to measure sulfentrazone and 3-carboxylic acid sulfentrazone in water at ppb exposure levels.

Reference:

Becker, J. M. 1996. A combined soil dissipation and small-scale prospective groundwater monitoring study with F6285 4F herbicide. FMC Corporation, Princeton, NJ. MRID 43926814.

Sulfentrazone [illegible]

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