Preparation and Analysis of Opioids in Environmental Samples

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Objective: The purpose of this study was to develop a method for the extraction of Opioids, which included fentanyl, carfentanil, heroin and oxycodone from aqueous, solid and wipe matrices, and the subsequent analysis using Gas Chromatography / Mass Spectrometry – Time-of-Flight (GCMS-TOF).

Significance: Microextraction techniques were utilized to facilitate relatively short sample preparation times and minimal use of solvent, equipment and space to eliminate potential for laboratory contamination. This procedure allows for the detection of opioids, in the sub-nanogram range which equates to reporting limits for aqueous, solid and wipe matrices of 2ug/L – 3.0ug/L, 3.3ug/Kg – 6.0ug/Kg and 0.1ug/wipe – 0.3ug/wipe respectively.

Experimental procedures and equipment used: GCMS-TOF instrumentation utilizing a pulsed splitless injection. The GC run time was 7 minutes which allows for 3.5 injections per hour.

Preparation of aqueous samples: A 50 mL aqueous sample was basified and extracted with 5 mL of methylene chloride – 3x, by separatory funnel. The methylene chloride phase was collected in a 40 mL VOA vial by passing it through a glass funnel containing sodium sulfate and glass wool. The methylene chloride extract was concentrated to 1 mL by TurboVap®.

Preparation of solid samples: 30 grams of sample was extracted with 30 mL of Tris buffer solution (tris-hydroxymethyl aminomethane) for 15 minutes on a shaker table. Following the extraction, the Tris buffer solution was extracted with 5 mL of methylene chloride 3 consecutive times by separatory funnel. The methylene chloride phase was collected in a 40 mL VOA vial by passing it through a glass funnel containing sodium sulfate and glass wool. The methylene chloride extract was concentrated to 1 mL by TurboVap®.

Preparation of wipe samples: A sample wipe was extracted with 30 mL of methylene chloride, by shaker table, for 15 minutes. The methylene chloride extract was concentrated to 1 mL by TurboVap®.

Results: MDL studies (40CFR136, appendix B) were prepared and analyzed for aqueous, solid and wipe matrices, validating the documented method reporting limits.

Conclusions: Data from calibration curve and MDL studies analyzed by the developed method validate the method as a time, resource and laboratory space saving alternative to traditional methods.