CONCURRENT SESSION 3 – CHEMICAL AGENT SAMPLING & ANALYSIS METHODS

Development of Methods and Comparative Analysis of Opiates by UPLC-MSMS and GC-TOF

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Objective: The purpose of this study was to develop methods for the analysis of opiate analogs extracted from various environmental matrices utilizing UPLC-MSMS and GC-TOF. The goal was to develop methods that are amenable to rapid turn-around-times while preserving data quality. In addition, a comparison between GCMS-TOF and UPLC-MSMS preparation times, analysis times, and reporting limits is explored.

Significance: One aspect PHILIS's mission is to provide the EPA with legally defensible data in a rapid turn-around-time environment. These procedures allow for the detection and quantitation of opiate analogs in the low picogram range. Preparation and analysis times are relatively short, and preparation is performed via microextraction.

Experimental Procedures and equipment used: Equipment used: ThermoFishers ALTIS triple quad mass spectrometer (UPLC-MSMS) and LECO's Pegasus BT time of flight (TOF) Extraction procedures for the ALTIS:Water samples were prepared by passing an aliquot through a filter for direct injection on the ALTIS. Soils were extracted using a 50/50 (v/v) mix of methanol/water, shaken and passed through a filter for direct injection on the ALTIS. Wipes were extracted with a 50/50 (v/v) methanol/water, shaken and passed through a filter for direct injection on the ALTIS. Extraction procedures for the TOF:Wipes were prepared by extracting with 100% methanol via a pressurized solvent extractor and concentrating to 1.0 mL

Results Obtained: ALTIS peliminary data supports sub pg/uL recovery levels for aqueous samples. Preliminary data supports sub ug/Kg recovery levels for solid samples. Data supports sub ng/wipe recovery levels for wipe samples TOF: Data supports sub ng/wipe recovery levels for wipe samples. It was also shown that the ALTIS was about 100x more sensitive for fentanyl from wipe matrices. Sample preparation time for wipes to be analyzed by the ALTIS was about 20 mins from sample container to injection, vs the BT-TOF which was about 120 mins from sample container to injection.

Conclusions: Both ALTIS and BT-TOF methods are viable quick turn methods capable of producing legally defensible data. The ALTIS has shown higher sensitivities and quicker sample prep time over the BT-TOF.