Analysis of Chemical Warfare Agents by GC-MS Using Multimode Inlet for Large Volume Injection

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A Multimode inlet (MMI) system was implemented to increase sensitivity through a large volume injection technique (LVI) to achieve lower detection limits for the identification of Chemical Warfare Agents (CWA) using GC with Quadrupole Mass Spec (GC/MS). The Agilent 7890 instrument employed an upgraded injection MMI port to perform LVI in solvent vent mode. The initial inlet temperature of the MMI was set at 20°C, lower than the solvent's boiling point, using liquid CO2. In performing the analysis, sample injection is performed slowly while the inlet vent opens. Once the injection is completed the inlet switches to splitless mode as the inlet temperature rises vaporizing and transferring analytes into the column.

Samples were extracted using a micro-extraction preparation technique. Soils samples required two extraction procedures. For VX, 10g of sample was extracted with 30 mL of tris (hydroxymethyl) aminomethane (Tris buffer) and shaken for 15 minutes on a shaker table at 1500 rpm following a solvent exchange with 5 mL of methylene chloride. Extract was taken to a final volume of 1 mL. For the extraction of GF, HD, GB and GD 10 g of sample were shaken for 15 minutes with 25 mL of Methylene chloride and taken to a final volume of 1mL. 35 mL of water samples were extracted using 2 mL of Methylene chloride and shaking for 2 minutes. Wipe samples were extracted using 10 mL of methylene chloride and shaken for 15 minutes.

The Agilent 7890B with 5977B MSD Instrument was equipped with an MMI inlet with liquid CO_2 coolant system. The inlet was programmed with an initial temperature of 20°C for 0.38 minutes, then a temperature ramp of 600°C/minute until 352°C for 5 minutes. A 25uL injection was performed. The temperature program for the GC started at 40°C for 2.95 minutes followed by a temperature ramp of 25°C/minute until it reaches 320°C, hold it for 1 minute. Replicates for each matrix were analyzed on SCAN mode. The resulting statistical calculations were in accordance with 40 CFR 136, appendix B, revision 2.0. Detection limits were improved over non MMI techniques at a minimum by a factor of 5.

Installation of a guard column from the injector port will protect the analytical column during large volume injections and will provide additional improvement with resolution and detection limits.