

## **FP38 BIOGENERATION OF VOLATILE SELENIUM COMPOUNDS IN BIOSOLIDS/ BIOFUELS AND QUANTIFICATION BY VD/GC/ICPMS. Georges-Marie Momplaisir,**

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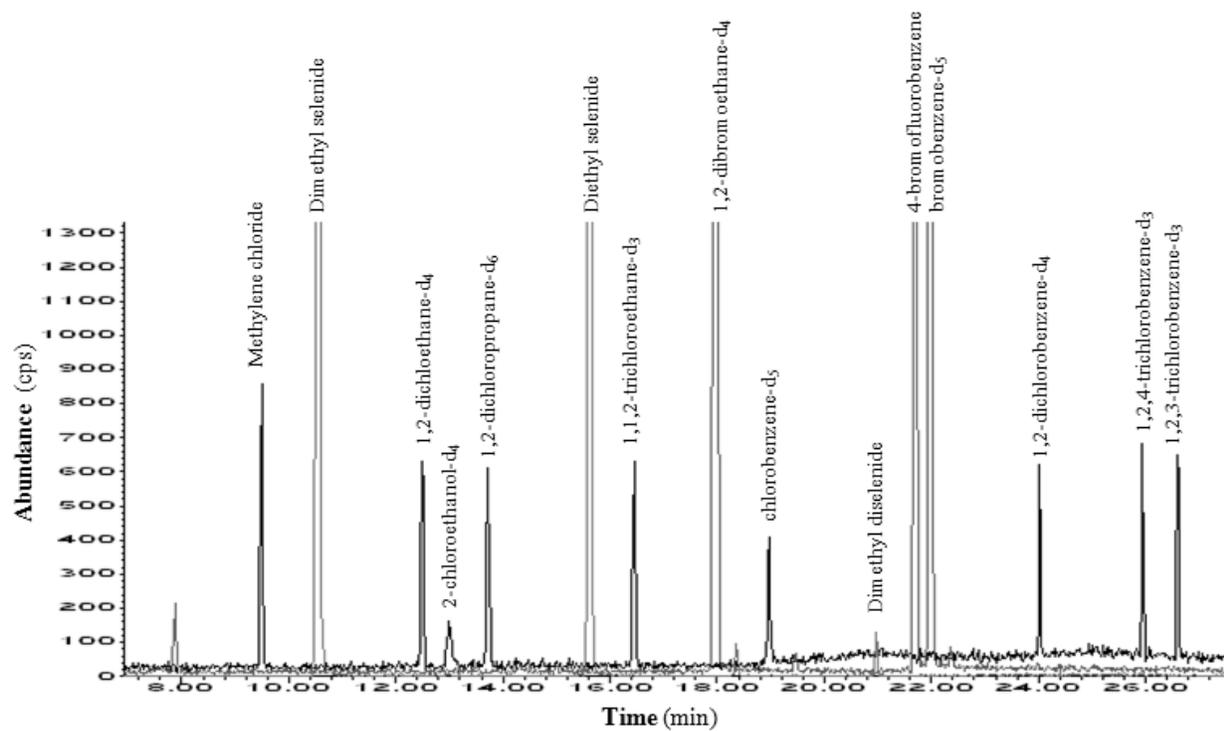
Selenium is known to form a wide range of both volatile and non-volatile compounds in environmental and biological systems. The determination of many of the volatile species of selenium is complicated by the fact that they are either too volatile and/or unstable to survive the isolation and concentration steps required in many analytical procedures. An analytical method based on vacuum distillation-gas chromatography-inductively coupled plasma mass spectrometry (VD/GC/ICPMS) was developed for the on-line extraction and quantification of such selenium compounds within a sample matrix. Several species of arsenic, selenium, antimony, bismuth, tin and tellurium are amenable to this technique, but this presentation focuses only on selenium.

A vacuum distiller/concentrator (Cincinnati Analytical Instruments Model VDC1012 vacuum distiller, Indianapolis, IN) was used to extract the selenium analytes from the sample matrix. A heated transfer line facilitated the transport of analytes contained in vacuum distillates to a GC (Agilent 6980N) equipped with a volatile interface and fitted with a capillary column (30 m x 0.25-mm i.d., 1.5- $\mu$ m film VOCOL, Supelco, Bellefonte, PA). The separated species were entrained in the helium stream that was supplemented downstream with argon gas, via another heated sample transfer line into the ICPMS (Agilent 7500 cx, Agilent Technologies, Palo Alto, CA).

Samples were vacuum distilled for 7.5 min with a 2.5 min transfer to the GC/MS. The VD and the GC transfer lines were held at 200 and 220 °C, respectively. The GC operating conditions were 2.5 min at -20 °C, 40 °C/min ramp to 60 °C, 5 °C/min ramp to 120 °C and held at 120 °C for 1 min, 20 °C/min ramp to 220 °C and held for 12 min resulting in a GC run time of 34 min. The injection was split 20:1 with a constant helium flow rate of 1.0 ml/min. The ICPMS operating conditions included plasma gas flow at 14.7 L/min, auxiliary gas at 0.9 L/min, carrier gas at 0.85 L/min, and forward power at 600 watts. The chromatographic data were acquired using the Agilent ChemStation software in time resolved analysis (TRA) mode with 100-msec dwell time for each element monitored.

The VD-GC-ICPMS method was optimized for dimethyl selenide (DMSe), dimethyl diselenide (DMDS<sub>2</sub>Se) and diethyl selenide (DESe). Analyte quantification was performed following SW-846 Method 8261A (VD/GC/MS) [1] which uses a suite of volatile organic internal standards to measure and adjust recoveries impacted by the matrix effects expected from biosolids (relative to compounds' boiling points and relative volatilities). The internal standards used in this study are volatile compounds that contain Br or Cl and have boiling points or relative volatilities that bracket the range for the selenide compounds. Selenium analytes and internal standards were well resolved in the ion chromatogram (Figure 1). The calibration standard deviations for the compounds fell below 10% (5 to 250 ng for DMSe and DESe and 100 to 250 ng for DMDS<sub>2</sub>Se). The resulting VG/GC/ICPMS method was used to study the biogenesis of selenides in biosolids/biofuels. Preliminary results of this study will be presented.

[1] EPA SW-846 Method 8261A <http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/8061a.pdf>



**Figure 1.** VD/GC/ICPMS chromatogram of a mixture of selenium compounds and internal standards.