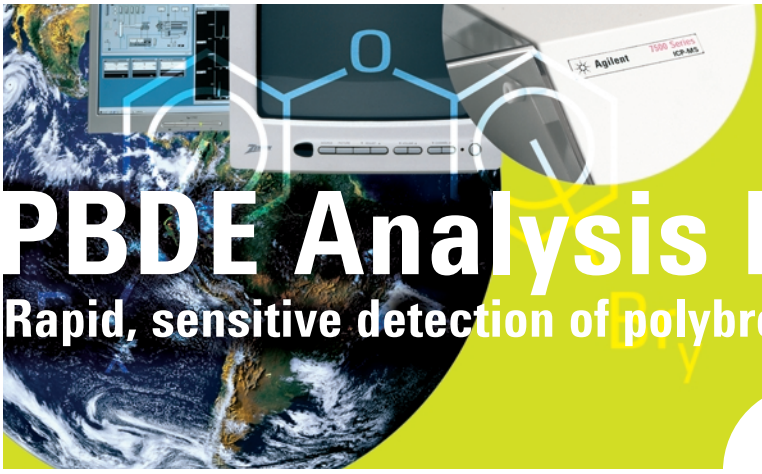

PBDE Analysis by GC-ICP-MS

Rapid, sensitive detection of polybrominated diphenyl ethers

Polybrominated diphenyl ethers (PBDEs) are common flame retardants added to many household products and electronics devices. These chemicals can enter the environment through the dust and residue from such products or into groundwater through landfill seepage. Similar to polychlorinated biphenyls (PCBs) and dioxins, PBDEs have been shown to be persistant, bioaccumulative and toxic[1]. While two of the three commercial PBDE products have been recently banned in Europe and parts of the US, they are ubiquitous in the environment. Levels in breastmilk from North American women are doubling every 2-5 years and are approaching concentrations known to cause thyroid and neurological damage in laboratory animals[1].

There are 209 different PBDE congeners and many are notoriously difficult to detect because they decompose at relatively low temperatures.

Gas chromatography (GC) combined with inductively coupled plasma mass spectrometry (ICP-MS) has been successfully applied to the analysis of PBDEs. ICP-MS possesses several advantages over other halogen-specific GC detectors.

- ICP-MS has very high sensitivity, even for poorly ionized elements such as the halogens
- Absolute selectivity between the halogens, suffering no interference from fluorine, chlorine or iodine
- No suppression of response from co-eluting compounds typical of some elemental detectors
- Tolerant of a range of GC carrier gas options and flows, greatly increasing the chromatographic flexibility
- Compound Independent Calibration (CIC), useful for the quantification of compounds for which standards are expensive or unavailable such as many PBDE congeners

Experimental

The system consisted of an Agilent 6890 GC and Agilent 7500a ICP-MS connected via the Agilent GC-ICP-MS interface. The GC column was a thin-film Agilent DB-5 MS (5M x 0.25mm ID x 0.25 micron). PBDE standards were obtained from AccuStandard Inc., New Haven CT.

Sensitivity and Selectivity

To test the ability of the GC-ICP-MS system to detect low levels of PBDEs in potentially difficult matrices, a PBDE mix was spiked into a mix of commercial PCB standards. The PCB standards Aroclor 1016 and 1260 were used, mixed together at 250 ppm each, making 500 ppm total PCBs. Figure 1 depicts a 50 ppb PBDE mixture spiked into the Aroclor mix. The first seven eluting PBDE congeners co-elute with numerous PCB congeners, present at significantly higher concentrations. The PCB chromatogram (m/z 35) is shown, inverted to simplify viewing. No enhancement or suppression of the bromine signal occurred, and the PBDE congeners are easily separated from the much more abundant PCB congeners.

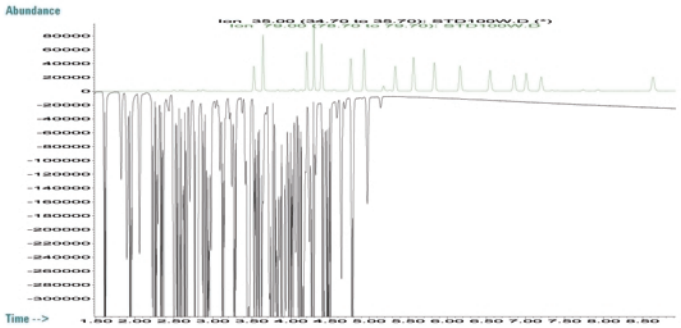

Figure 1. Extracted ion chromatogram, m/z 79 (Br) and 35 (Cl, inverted) of a mixture of PBDEs at 50 ppb each spiked into 500 ppm total PCB as Aroclors 1016 and 1260, total retention time < 10 minutes. Hydrogen carrier gas.

Figure 2 is a calibration graph for PBDE-183, a heptabromo congener, from 10 ppb - 1 ppm. With a dynamic range from low parts per trillion to hundreds of parts per billion in solution, GC-ICP-MS is more sensitive than most other techniques and comparable to or better than high resolution GC-MS. This extreme sensitivity and selectivity mean that complicated sample concentration and cleanup procedures can be minimized.

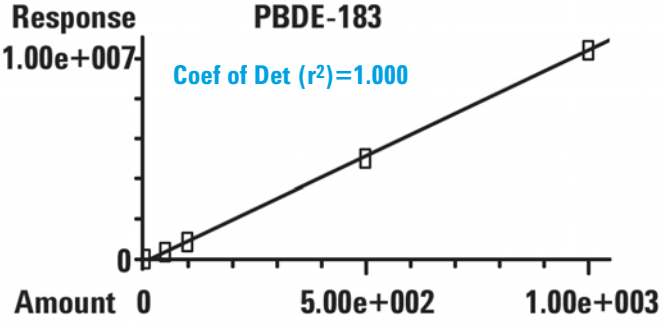

Figure 2. Typical calibration curve for PBDE congener: PBDE-183, 10 ppb - 1 ppm

A New Direction

Combining GC with ICP-MS detection produces a powerful tool that has both the selectivity and sensitivity to expand the troubleshooting capability of any analytical laboratory.

GC-ICP-MS can be used in conjunction with Agilent's Retention Time Locking (RTL) technology to quickly screen for the presence of potential PBDEs in complex matrices without extensive cleanup. Based on the ICP-MS results, confirmatory GC/MS analysis can be performed over narrow retention-time windows, greatly simplifying the task of locating and identifying presumptive hits.

References: 1. S. Lunder and R. Sharp, Tainted Catch, Environmental Working Group, http://www.ewg.org

http://www.agilent.com/chem

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