Detection and Quantitation of Brominated and Chlorinated Hydrocarbons by DART with Linear Ion Trap and Triple Quadrupole Technology

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Overview

Purpose: Halogenated contaminants such as brominated flame retardants (BFRs) and chlorinated pesticides (OCs) have been used for many years. Both BFRs and OCs are present in the environment and pose health hazards. Therefore, detection and quantitation of these compounds is critical. This experiment investigates the use of DART technology to detect six BFRs and five OCs.

Methods: The DART-SVP source (IonSense Inc.) was used to make sample preparation and analysis easier. Both LC-MS and triple quadrupole mass spectrometry (QqQ) technology were used for this study.

Results: Selectivity and quantitation determined on the linear ion trap were confirmed on the TSQ. Further optimisation and troubleshooting can be done for the TSQ method that was achieved using DART ionisation of the BFRs chosen for further study.

Introduction

Halogenated hydrocarbons, also known as BFRs, have been used in various industries for decades. Recently, several classes of BFRs have been detected in the biosphere. One class of BFRs that has gained attention is hexabromobenzene (HBB). While most BFRs have been banned in the United States, many still occur in developing countries. The wide use of BFRs and OCs, as well as their persistence in the environment, makes the detection and monitoring of these compounds important. The proposed DART-SVP as a rapid, easy-to-use technique, along with the need for chromatographic method development, and in situ calibration sample preparation, for detection and quantitation of both BFRs and OCs.

Methods

Sample Preparation

Compounds listed in Table 1 were dissolved in acetonitrile at 1 mg/mL to make stock solutions. Stock solutions were diluted serially to give the following standards: 0.05, 0.1, 0.5, 1, 5, 10, 25, 50, 100 ppm. A stock solution of 0.05 ppm was prepared in an acetonitrile/water solvent. Each compound was spiked into the solvent at a level of 0.05 ppm and this solvent was used as a calibrator blank.

DART Methodology

Preliminary data was acquired on the Thermoscientific LTQ linear ion trap mass spectrometer using the DART-SVP source in 1D transmission mode, with a grid voltage of 800V and temperature of 200°C. Full scan and MS/MS data were acquired for all compounds to determine optimal conditions, including the grid voltage and temperature. The MS/MS data was acquired for each compound in the LTQ using QqQ technology via ESI mode, with a pressure of 0.7 mbar, a sheath gas flow of 5 l/min, and a linear ion trap voltage of 0.7 V.

Conclusions

The linear ion trap with the DART-SVP in 10 mbar helium mode provided excellent stability of BFRs and OCs, providing precise and fragment information. The Quantitative Access MS MS with the DART-SVP in closed infusion mode generated full-scan spectra for BFRs and OCs that were a high-quality signal-to-noise ratio for subsequent isotope correction and detection, providing the best quantitation of BFRs and OCs.

References


Acknowledgements

We would like to thank the Thermo Corporation for providing the DART-SVP source.

FIGURE 2. MS/MS Spectra for tetrabromobisphenol A. Panel A depicts linear ion trap data, Panel B depicts triple quadrupole data. Linear ion trap data was acquired with a corrected collision energy of 40 V. Triple quadrupole data was generated with optimum collision energy for each compound.

FIGURE 3. TSQ MS data for calibration and measurement. Each panel depicts the calibration for the following: Panel A = kepone, 1,2,5,6-tetrabromo-3,3,3-trifluorobenzene, 1,2,5,6-tetrabromo-3,3,3-trifluorophthalic anhydride, 1,2,5,6-tetrabromo-3,3,3-trifluorophenol, 1,2,5,6-tetrabromo-3,3,3-trifluoropropionic acid, 1,2,5,6-tetrabromo-3,3,3-trifluorobutanoic acid, 1,2,5,6-tetrabromo-3,3,3-trifluoropentanoic acid, 1,2,5,6-tetrabromo-3,3,3-trifluorohexanoic acid, 1,2,5,6-tetrabromo-3,3,3-trifluoroundecanoic acid, and 1,2,5,6-tetrabromo-3,3,3-trifluorododecanoic acid.

FIGURE 4. Calibration curves and results for kepone, 1,2,5,6-tetrabromo-3,3,3-trifluorobenzene, 1,2,5,6-tetrabromo-3,3,3-trifluorophthalic anhydride, 1,2,5,6-tetrabromo-3,3,3-trifluorophenol, and 1,2,5,6-tetrabromo-3,3,3-trifluoropropionic acid.

FIGURE 5. Caption.