Utilizing High Efficiency Electron Ionization and MS/MS to Reduce Injection Volumes and Sample Extract Volumes for EPA 8270

Matt Curtis, Fred Feyerherm, Ron Honnold, Dale Walker

The goal of this project is to show the applicability of EI GC/MS/MS measurement for the detection of EPA 8270 analytes using a fast low maintenance interval method. EPA method 8270 is the primary semivolatile organic carbon (SVOC) analysis for multiple types of matrices including soil and water. Due to the high concentrations injected and number of target analytes in this mixture; column degradation, inlet contamination, and ion source contamination may occur causing significant instrument down time. In addition, the sample preparation requires large amounts of solvent for sample extraction and large sample volumes that need to be shipped, which is costly for large labs running this method routinely.

This method shows the advancements in instrumentation and sensitivity to achieve detection limits below those currently being used. The analytical method was developed on the Agilent 7890B GC / 7010 tandem-quadrupole MS in electron ionization mode using the high efficiency ion source. The GC was configured with a MMI (multi-mode inlet), a narrow bore DB-8270D column, and a 7693 A/S. The narrow bore column allowed for shorter run times and lower helium flow rates. Low volume injections were utilized to reduce possible inlet contamination to allow for long runtime before required maintenance. Selected reaction monitoring allowed for low limits of detection and high specificity with closely eluting chromatographic peaks. Data analysis was carried out using Mass Hunter Software.

EPA method 8270D, (2007) outlines the use of full scan EI analyses for the identification and quantitation of the SVOC. If the requested limit of detection is lower than the specified concentration selected ion monitoring is allowed, but specificity can be challenging with complex/dirty matrices. It is recommended in the EPA method to run the sample on a GC/FID prior to GC/MS analysis to reduce the chance of contamination from a highly concentrated contaminate. This doubles the expected analysis time, per sample, and unless the columns of the two instruments are perfectly calibrated the retention times may be different. The ability to run the sample one time with a lower volume injection allows for a single run per sample that will not contaminate the GC/MS system. The narrow bore column reduced runtime by several minutes to 15 minutes while still meeting the chromatographic separation criteria. Compared to data acquired in full scan on a single quadrupole instrument, limits of detection were increased up to 4 orders of magnitude, for the same mass on column. The analysis was performed using electron ionization tandem mass spectrometry (MS/MS) using the Agilent 7010 GC/MS/MS system. The lower volume injected is calculated as mass on column to extrapolate to the reduced sample needed for extraction and still reach the levels of detection and quantitation required.

Ultra low limits of detection, robust for low molecular weight compounds using EI/MS/MS, lower extraction sample size and extraction solvent usage.

Dale R. Walker GCMS Applications Specialist Agilent Technologies 5301 Stevens Creek Blvd Santa Clara Ca 95051