

Rapid Analysis of Semivolatile Organic Compounds by Method 8270D using the Clarus GC/MS

Author

William Goodman
PerkinElmer Life and
Analytical Sciences
710 Bridgeport Avenue
Shelton, CT 06484 USA

Introduction

The purpose of this application is to demonstrate an efficient, robust technique to analyze environmental samples for U.S. Environmental Protection Agency (EPA) Method 8270D. 8270 is a very common GC/MS method to identify and quantify semivolatile organic compounds. The method specifies rigorous quality control (QC) requirements and a wide concentration range.

Experimental conditions

The PerkinElmer® Clarus® GC/MS used in this technique was configured with autosampler, a programmable split/splitless injector port, and TurboMass™ 5.2 GC/MS software with environmental reporting. Inside the injector port, a 2-mm Siltek liner (no wool) interfaced with a 30 m x 0.25 mm x 0.25 µm df Elite-5MS column. A 0.5-µL autosampler syringe was used to make a 0.5-µL injection into a 120 °C injector port that was ballistically ramped to 300 °C. During injection, the column flow rate of helium was increased from 1.2 mL/min to 2 mL/min; this achieved a pressure-pulsed injection to help control the solvent expansion. The injection was splitless for 1.00 minute, after which the flow rate was reduced and the split vent open to 100 mL/min (the split ratio was reduced to 20 mL/min after 5 additional minutes to save gas). The GC oven began at 45 °C and was ramped at

25 °C/min to 260 °C, then ramped at 6 °C/min until the elution of Benzo(b) and Benzo(k)fluoranthene, after which the oven was ramped at the maximum rate to a final temperature of 330 °C. The mass spectrometer method scanned from 35 to 500 u, with a scan time of 0.15 seconds and an inter-scan delay of 0.05 seconds (achieving more than 3 scans a second).

The system sensitivity was such that a 0.5-µL injection of a 1-ppm standard mix resulted in a signal to noise of approximately 500:1 for Pentachlorophenol. If greater sensitivity is needed, the Clarus MS has the capability to run SIFI (single ion/full ion); this is especially useful if a sub-list of compounds requires very low detection limits.

Results and discussion

Two of the many QA/QC requirements in Method 8270, MS tuning and initial calibration, are highlighted here. The tuning of the mass spectrometer used here was slightly modified from system defaults to meet the criteria for Decafluorotriphenylphosphine (dftpp). UltraTune™ automated tuning is included with TurboMass 5.2 software and is designed to meet Method 8270 tune criteria. Method 8270 also requires an initial calibration with at least 5 points, in which all components are below 20% RSD. The Clarus GC/MS system can meet the initial calibration criteria across a range of 1-200 ppm.

Conclusion

The cycle time of the method presented here is approximately 20 minutes, while still achieving 8270 method criteria of 75% baseline resolution for Benzo(b) and Benzo(k)fluoranthene (see Figure 1).

The PerkinElmer Clarus GC/MS provides environmental laboratories a platform for rapid, simple and robust analysis that will meet the QA/QC criteria necessary to routinely run Method 8270.

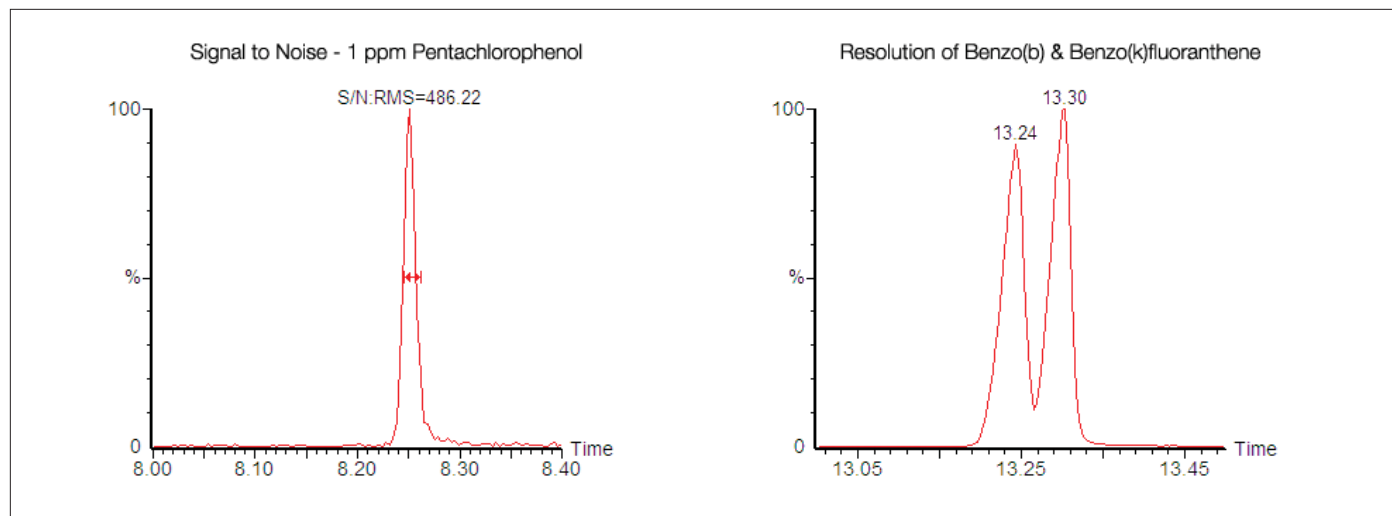


Figure 1. Shown here are the resolution of Benzo(b) and Benzo(k)fluoranthene in a 25 ppm standard and the signal to noise of 1 ppm (0.5 ng on column) of Pentachlorophenol.