PERFORMANCE OF EPA METHOD 8270 USING HYDROGEN CARRIER GAS ON SCION GC-MS

United States Environmental Protection Agency (US-EPA) Method 8270 is an analytical method for the detection of semi-volatile organic compounds in water sources, solid waste matrices, soils and air sampling media, by gas chromatography with mass spectrometry (GC-MS). The method measures a mixture of acids, bases and neutrals in sample extracts. The complexity of these extracts demands a robust instrument that is easy to operate and maintain. Adding to method complexity is the uncertainty in both cost and supply of helium, forcing laboratories to consider hydrogen as a carrier gas. Hydrogen is not an inert gas; it is reactive and can be an explosion hazard if allowed to build up in the GC oven or manifold of the MS.

The SCION helium free analyser will ensure safe routine operation, with no performance change when operating under EPA method 8270 specifications. SCION's unique axial ion source with extended dynamic range detector provides excellent robust operation with minimised unwanted protonation and spectral distortions. In addition, the 456 GC with split/splitless injector and inert pathway prevents compound degradation and reactions with the hydrogen carrier gas.

Experimental

Figure 1 shows the helium free analyser comprised of the SCION 456 GC with single quad (SQ) MS.

Calibration standards containing 76 target compounds were prepared. The calibration ranged from 0.075ppm to 200ppm for the majority of compounds. Internal and surrogate standards were added at a concentration of 40ppm, in dichloromethane.

The tune was performed using decafluorotriphenylphosphine (DFTPP) at a concentration of 50µg/mL with a 1µL injection. A pulsed split injection was used to minimise contact and residence time of compounds in the inlet. This is critical when using hydrogen carrier gas due to its low viscosity and tendency to react with dichloromethane and form hydrochloric acid.

With EPA method 8270 a single goose-neck 4mm open inlet liner is preferred. As the liner does not contain glass wool, compound degradation is eliminated especially when using hydrogen as a carrier gas, due to its reactive properties.

Results

In order for hydrogen to be used as a carrier gas, the specifications of EPA 8270 must be met. These specifications include tuning of the instrument, resolution, calibration, Gaussian Factor and system performance checks. Additionally, dichloromethane must be used as the solvent to minimise degradation in the inlet. The GC-MS system must also produce mass spectra that match the NIST library and demonstrates robust operation when heavy matrices are analysed.

The SCION helium free analyser can be auto-tuned with a tune to target feature for DFTPP, as specified by the EPA method. Figure 2 details the mass spectra

Figure 1. SCION 456 GC with SQ MS

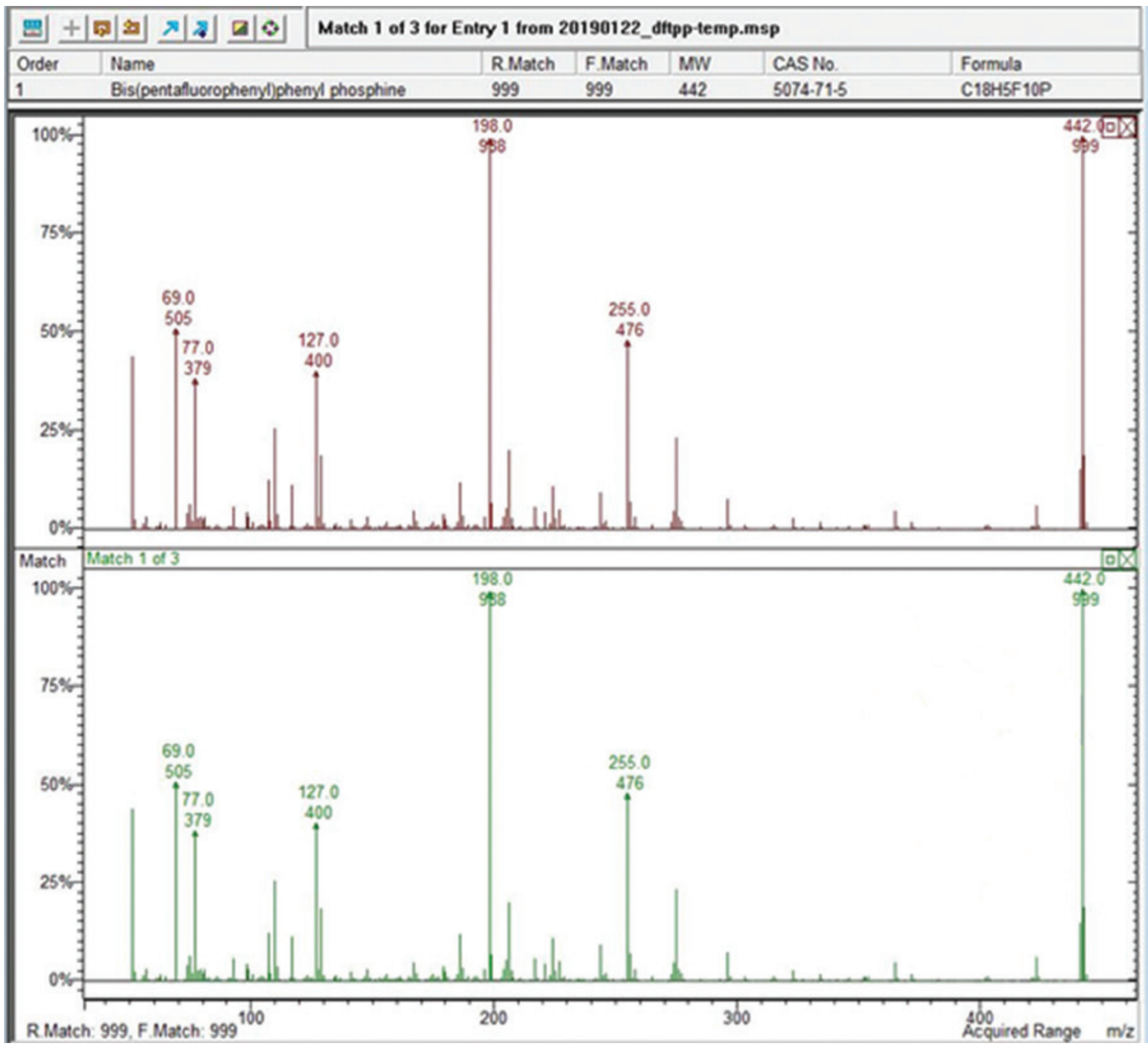


Figure 2. Mass spectra of DFTPP tune sample against NIST library

for DFTPP acquired from the instrument tune and the mass spectra obtained from a NIST search; the match was 99.9%. All requirements listed in the method were met and the tune passed inspection.

When using hydrogen as a carrier gas, spectral quality may be compromised due to unwanted protonation and other reactions in the ion source. However, the SCION axial source and helium free package minimises these reactions allowing high quality library matches to NIST, as shown in Figure 2. During this study, all seventy-six compounds of a sludge test sample were detected using an automated library search against NIST.

Analysing such a wide concentration range, 0.075ppm to 200ppm, demonstrates the excellent capability of the MS through the use of the extended dynamic range detector. The SCION single quadrupole MS uses a feature named 'Compound Based Scanning' (CBS), in which SIM ions for compounds are stored in a library. The scan information, compound retention times and individual dwell times are also stored and are easily selected and loaded directly into a data acquisition method, allowing combined/mixed SIM and full scan modes.

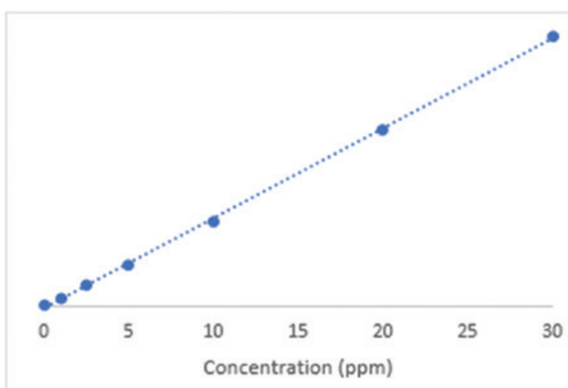
All quality control and system performance checks passed the method specifications. Table 1 details the mean response factors variance for nine typical active compounds analysed. Method EPA 8270 specifies that all target compounds must have a response variance of 15% or less. All variances were below 10%, showing minimal reactivity when hydrogen was used as a carrier gas and dichloromethane used as a solvent.

The calibration curve of hexachlorocyclopentadiene, a system performance check compound, at a low concentration range (0-30ppm) can be found in Figure 3.

Table 1. Mean response factor variance of select active compounds

Active Compound	Response Factor (RSD %)
Hexachloropentadiene	3.9
Dimethyl phthalate	8.1
2,6-dinitrotoluene	5.8
2,4-dinitrotoluene	9.1
Diethyl phthalate	5.8
Dibutyl phthalate	6.0
Butyl benzyl phthalate	8.8
Bis (2-ethylhexyl) phthalate	5.1
Di-n-octyl phthalate	4.5

Figure 3. Calibration curve of hexachlorocyclopentadiene, a system performance check



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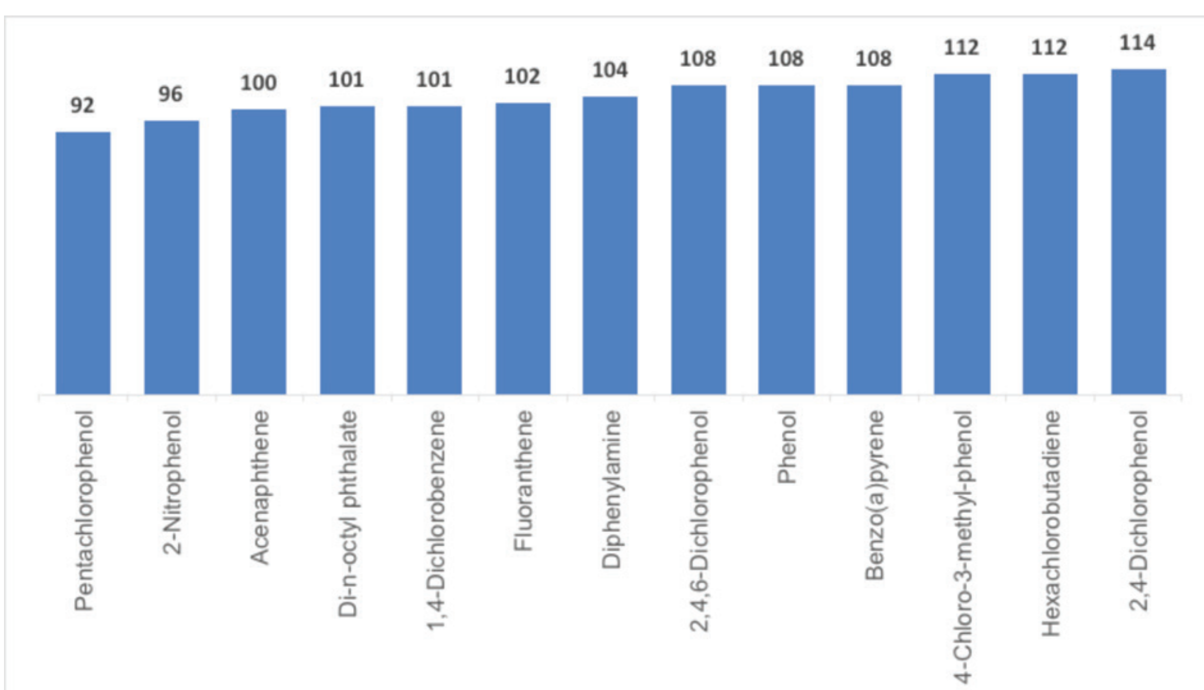

Figure 4. Analyte recovery (%) after repeated sludge injections (n=50)

Average relative response factors for selected system performance check compounds are listed in Table 2. All system performance check compounds passed the criteria. Relative response factors were calculated over a calibration range of 0.075-30ppm.

The longer term robustness of EPA method 8270 using hydrogen as carrier was tested using repeated injections of a sludge extract, spiked at 40 ppm level. A total of fifty injections were made, with the injection of a calibration check after every ten injections. Concentrations and recovery relative to the initial injection were calculated, as shown in Figure 4. Recoveries observed for the calibration check components after running 50 sludge samples were within 92-114%, demonstrating excellent method robustness over time, even with hydrogen as the carrier gas of choice.

Table 2. Average relative response factors of system performance check compounds against EPA 8270 requirements

Compound	Method 8270 Min RRF	RRF on SCION SQ MS
N-nitroso-di-n-propylamine	>0.05	0.109
Hexachlorocyclopentadiene	>0.05	0.138
2,4-dinitrophenol	>0.05	0.070
4-Nitrophenol	>0.05	0.134

Conclusion

As the cost and scarcity of helium rises, laboratories seek to amend methods for the use of hydrogen as carrier gas. EPA method 8270 is one of the most challenging methods covering a wide variety of compound classes, spanning a wide concentration range and various matrices, but it can effectively be converted to method running on Hydrogen carrier gas.

The converted method was proven to pass all key EPA method 8270 criteria for system tuning, inertness, response and linearity. Furthermore, long term method robustness with samples was evaluated and found to be excellent.

The SCION SQ Mass Spectrometer offers excellent performance for EPA method 8270, also on Hydrogen carrier gas. The axial ion source design and EDR ensure quality library comparable mass spectra, excellent sensitivity and great linear dynamic range of the instrument, allowing lower reporting limits in challenging matrices, combatting the typical challenges associated with EPA method 8270.

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