

Poster Reprint

ASMS 2023
Poster number WP 277

Helium to Hydrogen: Explosives & Pesticides & VOAs, Oh My! Successful Transition of GC/MS Analyses

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Introduction

Helium is the best and most used carrier gas in GC/MS. Hydrogen is the second-best alternative to helium. It provides several advantages, including faster analysis times and smaller environmental impact. However, hydrogen is a reactive gas. Hence, every analyte in every method needs to be validated with hydrogen.

This work provides guidance on the GC/MS conditions for the effective transition from helium to hydrogen carrier gas for a variety of applications.

Experimental

HydroInert Source

The HydroInert source is an EI source optimized for use with hydrogen carrier gas. Due to its inert nature, it minimizes undesirable in-source chemical reactions between the analytes and hydrogen. This results in improved library match scores (LMS) vs helium-based libraries and allows using the same target ions in GC/MS and MRM transitions in GC/MS/MS. This makes the transition of methods from helium to hydrogen much easier.

Considerations for GC/MS Method Conversion

The following should be considered when converting from helium to hydrogen.

- Review the document “The EI GC/MS Instrument Helium to Hydrogen Carrier Gas Conversion Guide” [1] for detailed instructions for method conversion from He to H₂ carrier. This covers all aspects, including H₂ safety, you should consider.
- When targeting fragile analytes like pesticides or explosives, use a temperature programmable inlet like the MMI to minimize possible hydrogenation reactions.
- Use Agilent’s Method Translation calculator [1] to pick a column and parameters to obtain the same elution order as with the helium method. Since most helium methods use a 30 m x 0.25 mm id column, the 20 m x 0.18 mm version is a great place to start.
- The increased resolution afforded by hydrogen may allow the development of a faster method.
- For the reasons mentioned above, use the HydroInert source.

Pesticides

Figure 1 compares the chromatograms for 203 pesticides in a spinach QuEChERS extract with He and with the optimized H₂ method. Using Method Translation, the elution order and retention times are the same, greatly simplifying conversion. Note the increased resolution with H₂. This can be further exploited with Method Translation to decrease the run time from 20 min to 10 min [2]. The optimized H₂ method uses the MMI inlet with a temperature programmed Solvent Vent injection of 2 µL. A 2 mm dimpled liner and HP-5MS UI 20m x 20m (0.18mm x 0.18 µm) column set in backflush configuration are also used. With the H₂ optimized method, over 90% of the 203 targets could be quantitated at or below 10 ppb (mg/kg) in spinach extract, which is the default MRL [2].

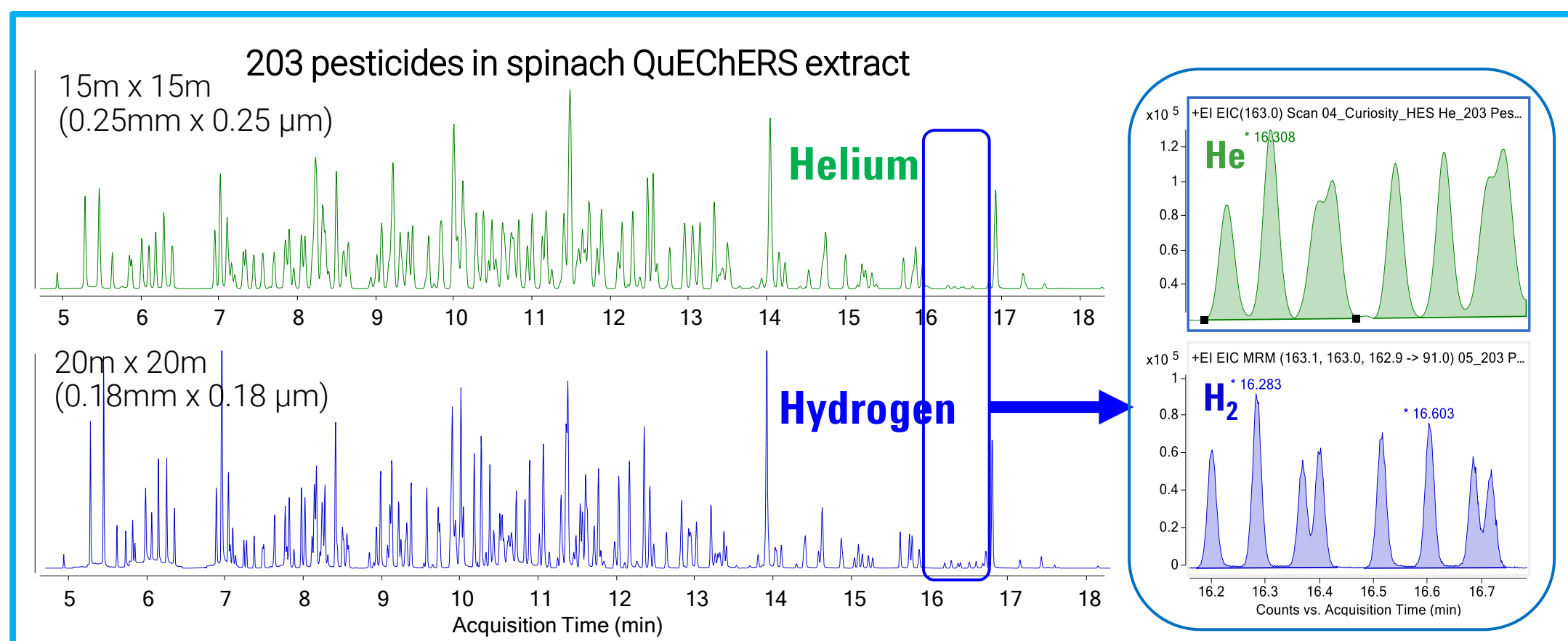


Figure 1. Top: Pesticide method with He carrier. Bottom: Method converted to H₂ carrier using 0.18 mm id column set.

Volatile Organic Compounds in Drinking Water with Headspace-GC/MS

H₂ carrier allowed the separation of 80 volatile organic compounds (VOCs) in 7 minutes. The method used a DB-624 20m (0.18mm x 1 µm) and a pulsed split injection of 20:1. Complete method details and results are provided in reference [3]. Scan mode demonstrated excellent spectral matching against the NIST20 library (average LMS 94), and excellent calibration linearity with an average range of 0.16 to 25 µg/L. In SIM mode, the average range was 0.07 to 25 µg/L, and the average MDL for the 80 compounds was 0.026 µg/L. Fig. 2 shows the chromatogram and highlights the excellent results for nitrobenzene, which is often a problem with H₂ carrier if the HydroInert source is not used.

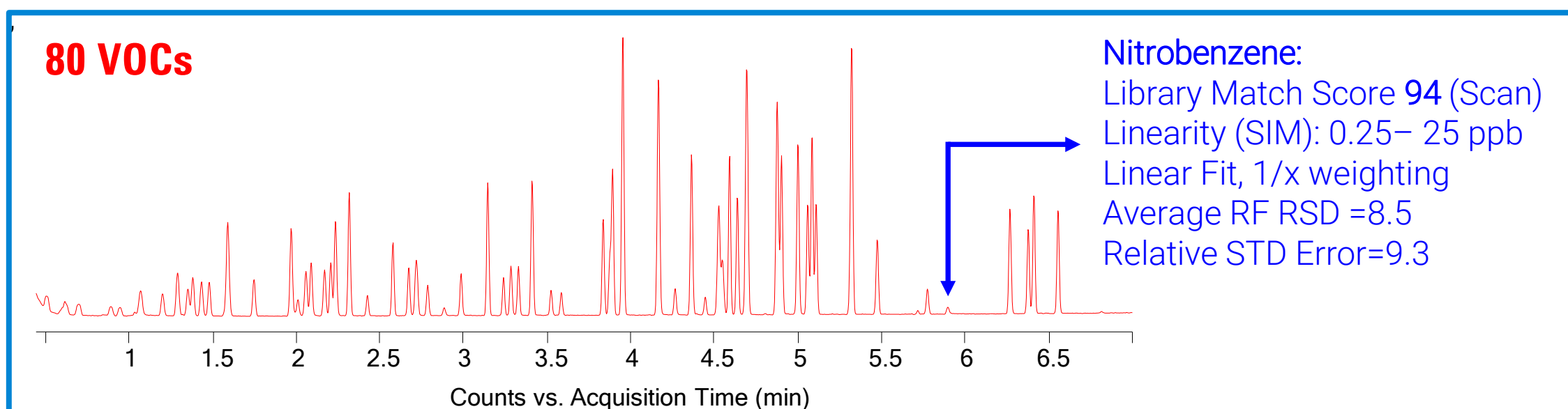


Figure 2. VOCs in water analyzed by headspace/GC/MS.

Semi-volatile Organic Compounds (SVOCs) with EPA 8270E

Fig 3 shows the analysis of 120 target analytes and surrogates using H₂ carrier gas, the HydrolInert source and the 7000E GC/TQ. The use of H₂ carrier and the 0.18 mm id column provided excellent resolution and a run time of only 10.5 min. A 20:1 split injection was used and the MMI inlet was programmed from 250 °C (hold 0.3 min) at 200 °C/min to 350 °C. A calibration range of 0.02-100 µg/mL was obtained for 82 compounds and 0.1-100 µg/mL for 106 compounds. Note the excellent peak shape and resolution in Fig 3. Full details are available in ref [4]. Excellent results were also obtained using the 5977C single quadrupole GC/MSD with H₂ carrier and the HydrolInert source. This is detailed in ref [5].

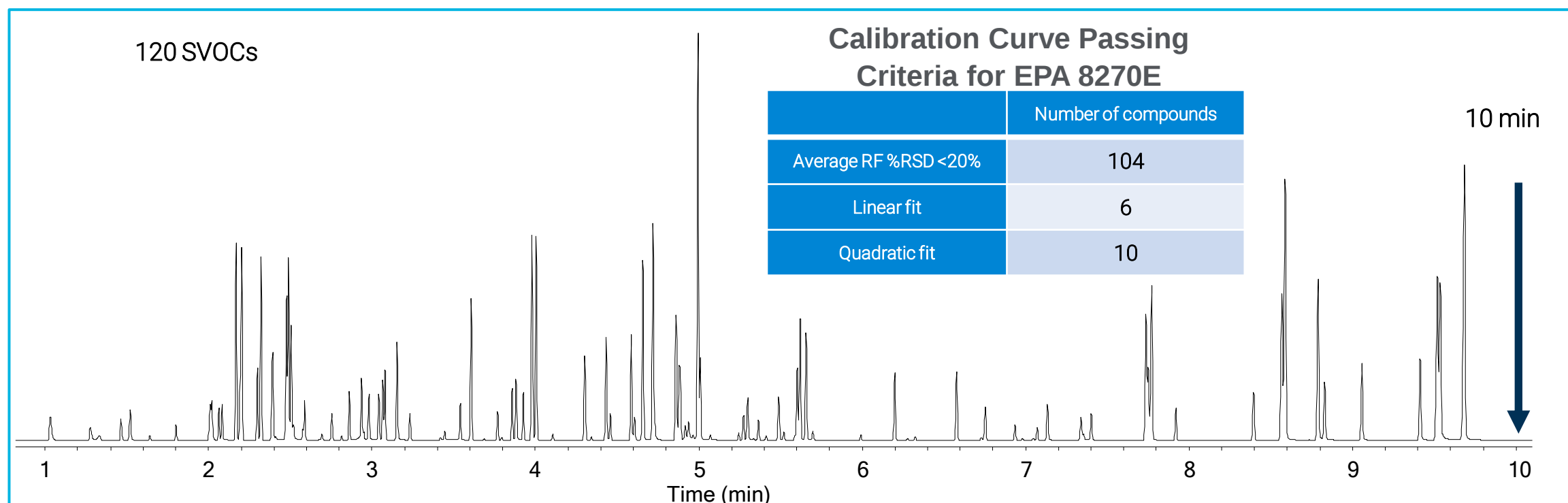


Figure 3. TIC of 120 SVOCs in method converted to H₂ carrier using 20m x 0.18 mm id x 0.18 µm DB-5MSUI column.

Polynuclear Aromatic Hydrocarbons (PAHs)

PAHs are durable compounds that tend to tail with He carrier gas. With H₂ carrier and the HydrolInert source, the peak shape and resolution are significantly improved, as seen in Figs 3. With 5977C GC/MSD, the MDL and linearity are comparable to or better than those with He. Also, the ISTD response was stable across 4 orders of calibration. Excellent linearity was observed over the range of <1 – 1,000 µg/L with an average RSE = 9.5. The average MDL was about 0.1 µg/L. Due in part to the cleaning action of H₂, response stability was shown over 100 injections of a challenging soil extract with GC/MSD. Full details are available in ref [6]. Excellent results were also obtained using the 7000E GC/TQ with H₂ carrier and the HydrolInert source. That system was configured with backflushing and response stability was shown over 500 injections with the challenging soil extract. This is detailed in ref [7].

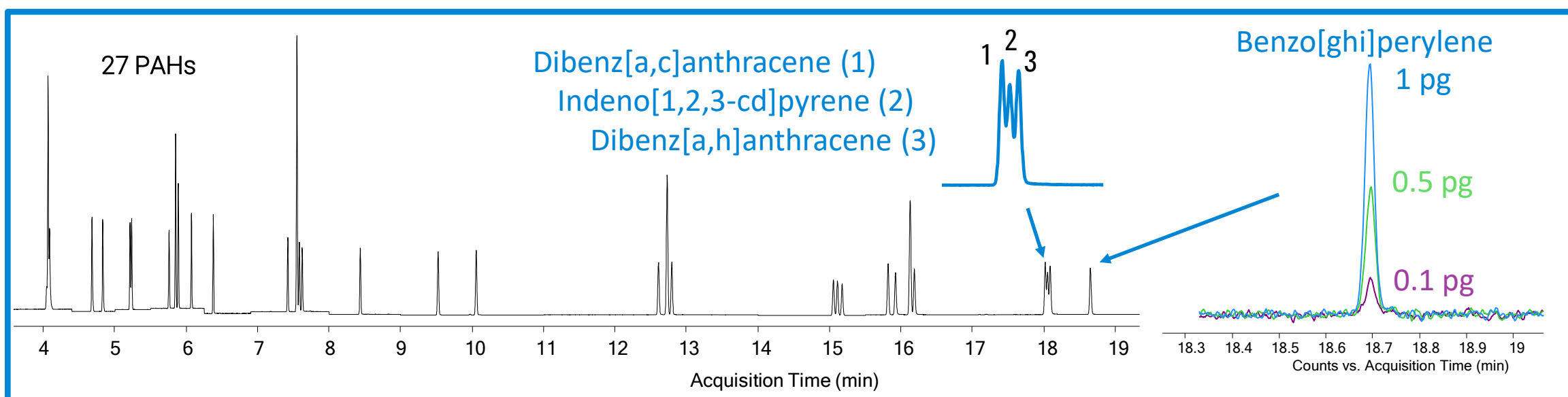


Figure 4. SIM TIC of 27 PAHs in method converted to H₂ carrier using 20m x 0.18 mm id x 0.14 µm DB-EUPAH column.

Results and Discussion

Explosives

Nitro compounds used in explosives are highly prone to hydrogenation, leading to poor library match scores (LMS) with H₂ carrier and traditional EI sources. A group of nitroaromatics commonly encountered in explosives were analyzed using a 50:1 temperature ramped split injection, a 20 m x 180 µm x 0.18 µm DB-5MSUI column, and the HydroInert source with H₂ carrier gas in the 5977C GC/MSD. As seen in Fig 5, excellent LMS values were obtained, indicating minimal hydrogenation.

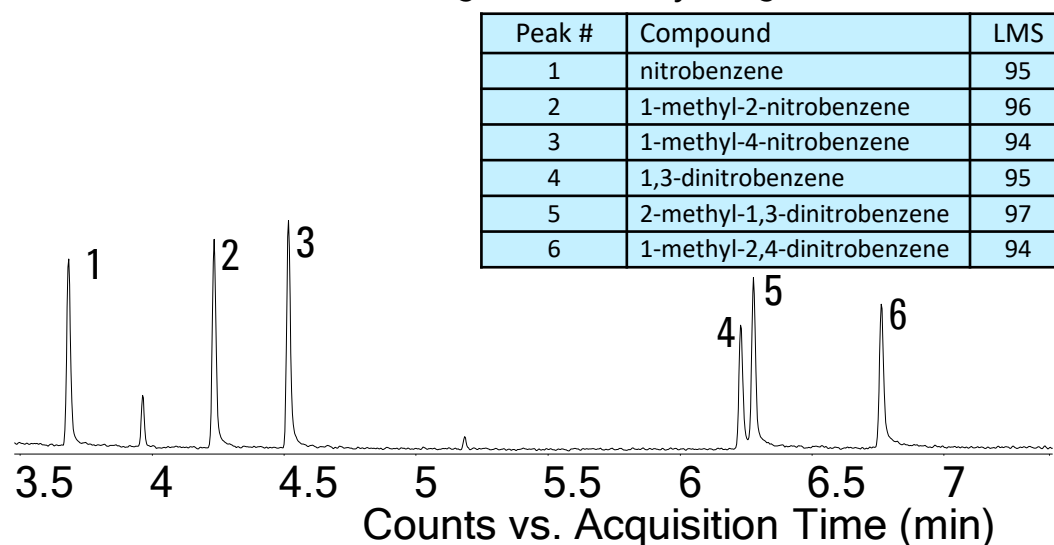


Figure 5. TIC of nitroaromatic compounds.

Conclusions

If He is available at an acceptable price, it is the preferred carrier for GC/MS and should be used. However, as shown in this overview of several converted methods, H₂ can be used if appropriate adjustments are made to accommodate its use.

References

- ¹Agilent EI GC/MS Instrument Helium to Hydrogen Carrier Gas Conversion. User Guide. 5994-2312EN. 2022.
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DE78483406

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Published in USA, May 31, 2023