Poster Reprint

ASMS 2024
Poster number ThP 331

Two Methods to Perform the New US EPA Method 1628 with GC/MSD: Traditional Helium Carrier Gas and Hydrogen Carrier Gas

Jennifer Sanderson and Samuel Haddad

Agilent Technologies, Inc.

Introduction

Polychlorinated Biphenyl (PCBs) and the Environment

Polychlorinated Biphenyls (PCBs) are synthesized compounds that belong to the chlorinated hydrocarbon family of compounds. PCBs were used in a variety of industrial applications such as electrical components, plasticizers and pigments/dyes, until they were banned in 1979 by the Toxic Substances Control Act (TSCA)[1]. PCBs are considered to persistent organic pollutants (POPs) as they do not easily degrade in the environment. PCBs, which are classified as probable human carcinogens, can be found in the air, water, and soil and have been known to bioaccumulate in marine life.

The EPA has recently developed a low-resolution mass spectrometry method that can calibrate 65 PCBs while simultaneously screening for all 209 PCB congeners using 31 labeled PCB compounds[2]. As with most EPA methods, the prescribed methodology utilizes helium as a carrier gas. Helium is the gold standard in GC/MS analysis; however it is an expensive gas to procure, and the availability has proven to be inconsistent over the last decade. In contrast, hydrogen as a carrier gas is relatively cheap and can be used to make methods much faster without sacrificing chromatographic resolution. Presented here are two methods based on the requirements of the EPA 1628 analytical method, one developed with traditional helium carrier gas and second method using hydrogen carrier gas, converted by using Agilent MassHunter Method Translator.

Experimental

Starting Method: Helium Carrier Gas

Separation and detection based on EPA 1628 required criteria using helium as the carrier gas was carried out using the Agilent 8890/5977C GC/MSD. Two Agilent J&W GC Columns with dimensions of 30m x 250µm X0.5µm were configured using a purged union to enable backflush was used to carry out the separation. The MSD had an extractor ion source used for detection using selective ion monitoring (SIM).

Figure 1: Agilent 8890/5977C GC/MSD Instrument

Method Conversion: Hydrogen Carrier Gas

A second 8890/5977C GC/MSD equipped with stainless steel tubing to carry hydrogen gas was outfitted with two Agilent J&W GC columns with dimensions of 20m x180µm X0.36µm connected by a purged union purged union to enable backflush. The HydroInert Ion source was installed in the MSD and the SIM method was adapted for the faster analysis.

Figure 2: Agilent HydroInert Ion Source.

Converting a Helium based method to a Hydrogen Method

The initial method for the 240 labeled and unlabeled PCB compounds was developed using Helium carrier gas. Good separation for the PCB congeners that do not co-elute was achieved under 40 minutes and is show in Figure .

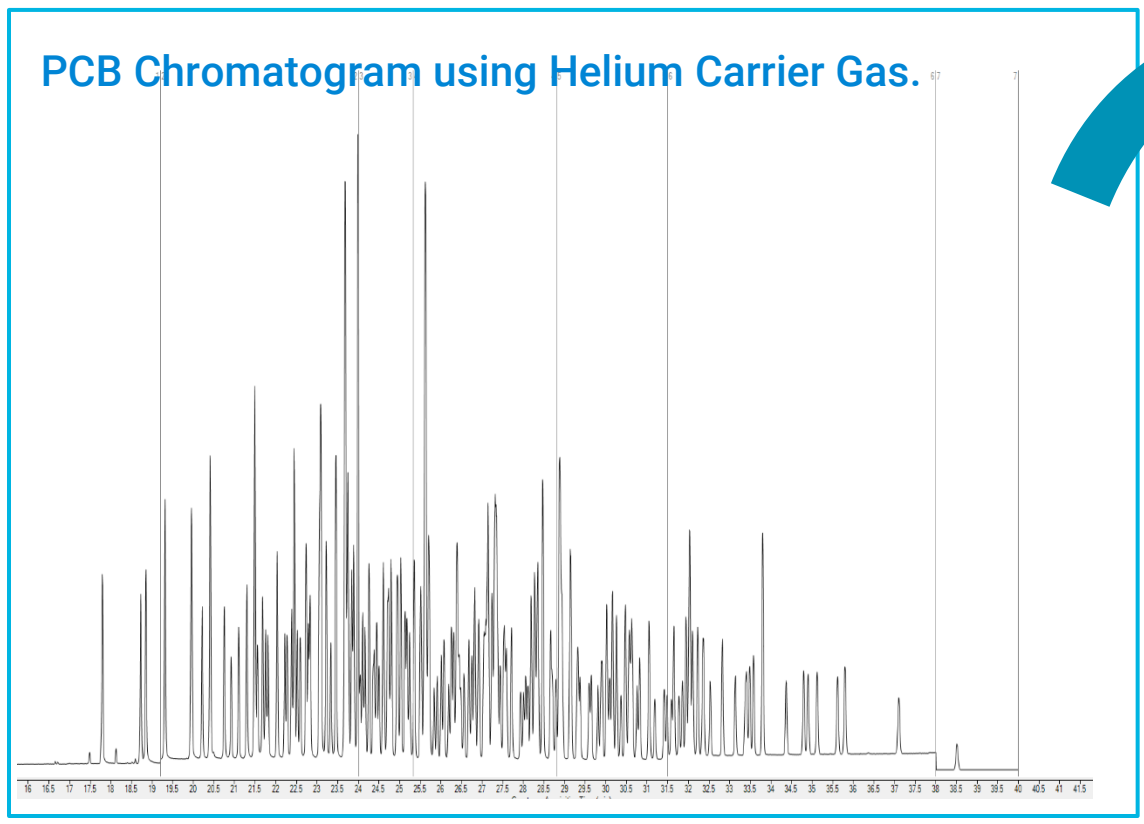

Figure 3: Chromatogram of labeled and unlabeled PCB congeners in helium carrier gas

Agilent MassHunter software feature an embedded method translator calculator (Figure), which was used to approximate a hydrogen method based on the developed helium method. There are three options for the translation: speed, direct translation and efficiency. Translate was used to create the starter hydrogen method.

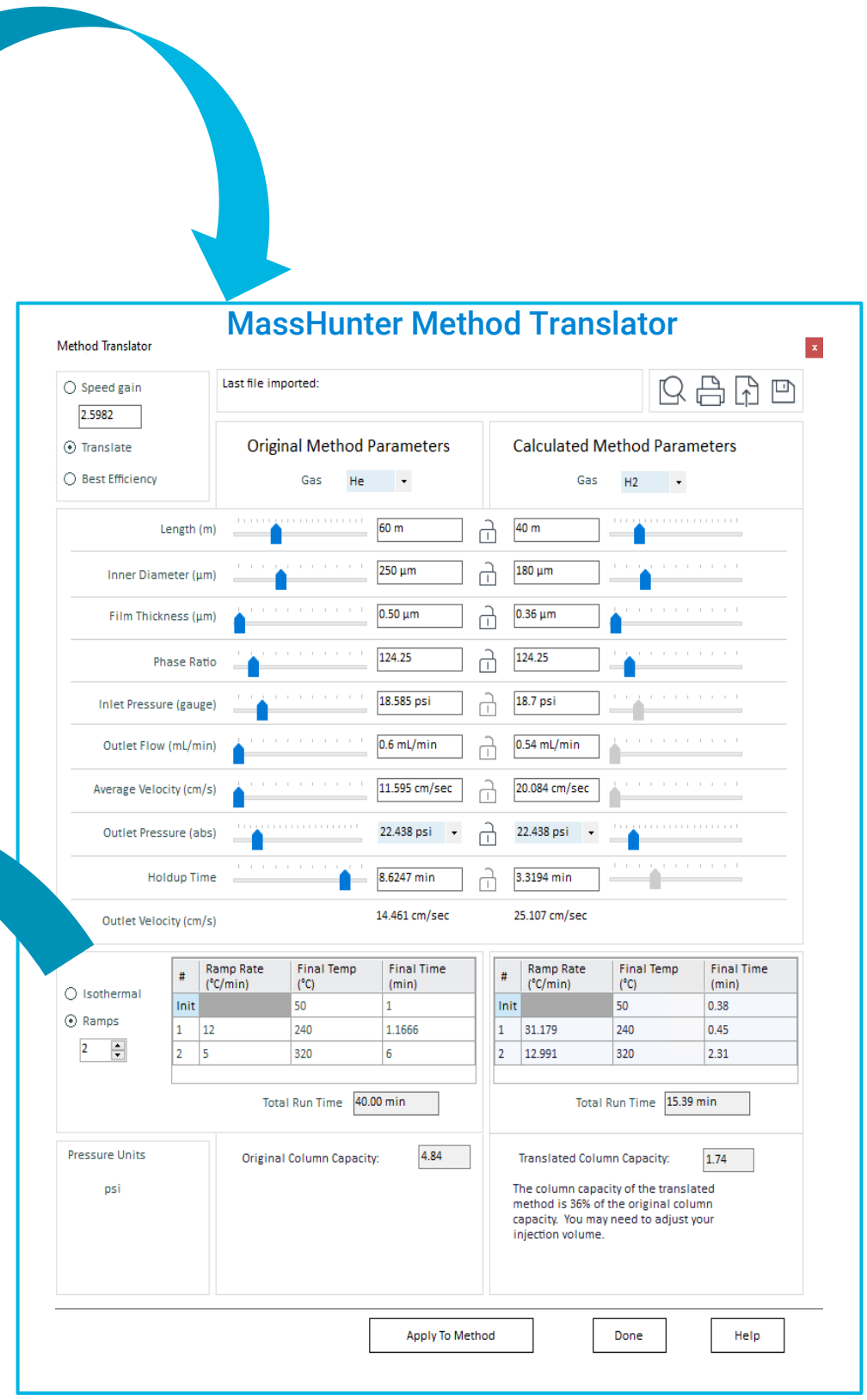

Figure 4: Agilent MassHunter Method Translator

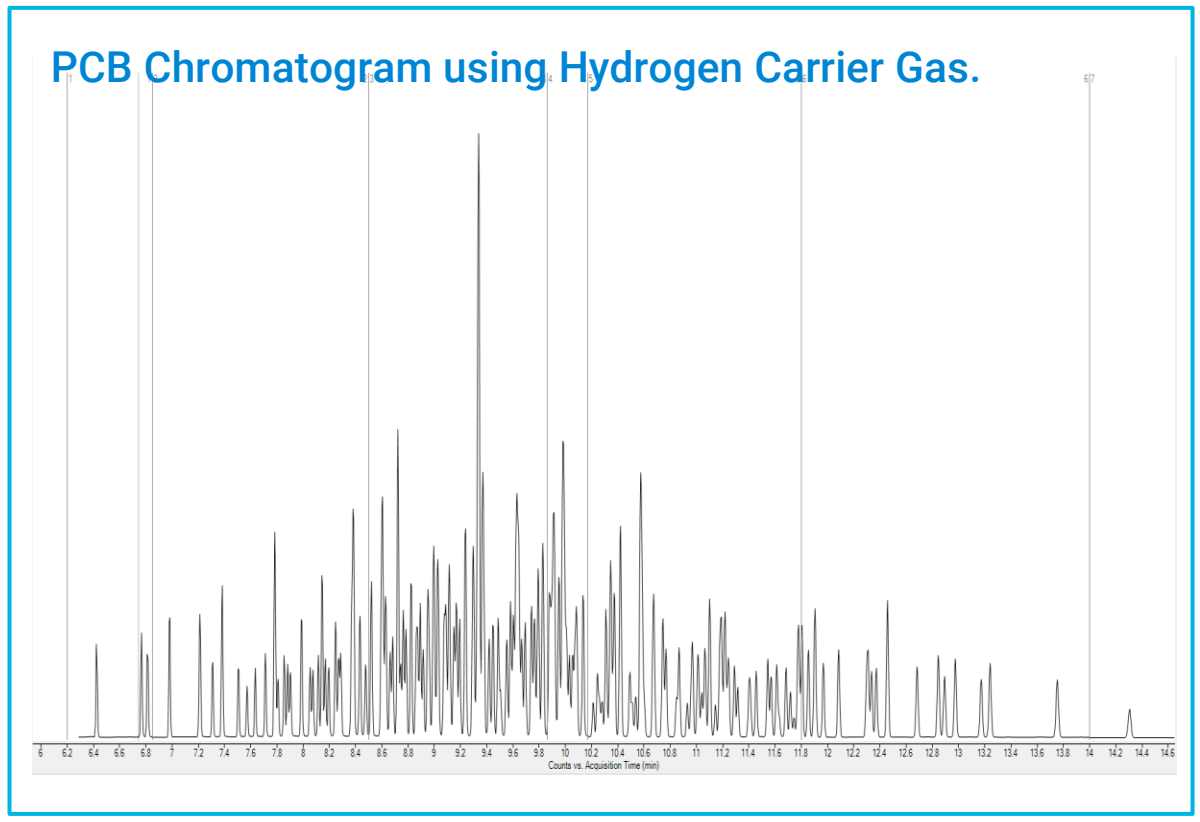

Figure 5: Chromatogram of labeled and unlabeled PCB congeners in hydrogen carrier gas.

The resultant method from the translator was a faster separation method that did not compromise on chromatographic resolution required by EPA 1628 methodology. Adjustments for the SIM mass detection method was needed to accommodate the faster elution, which was done without sacrificing method detection limit.

Calibration Curves from Select PCBs

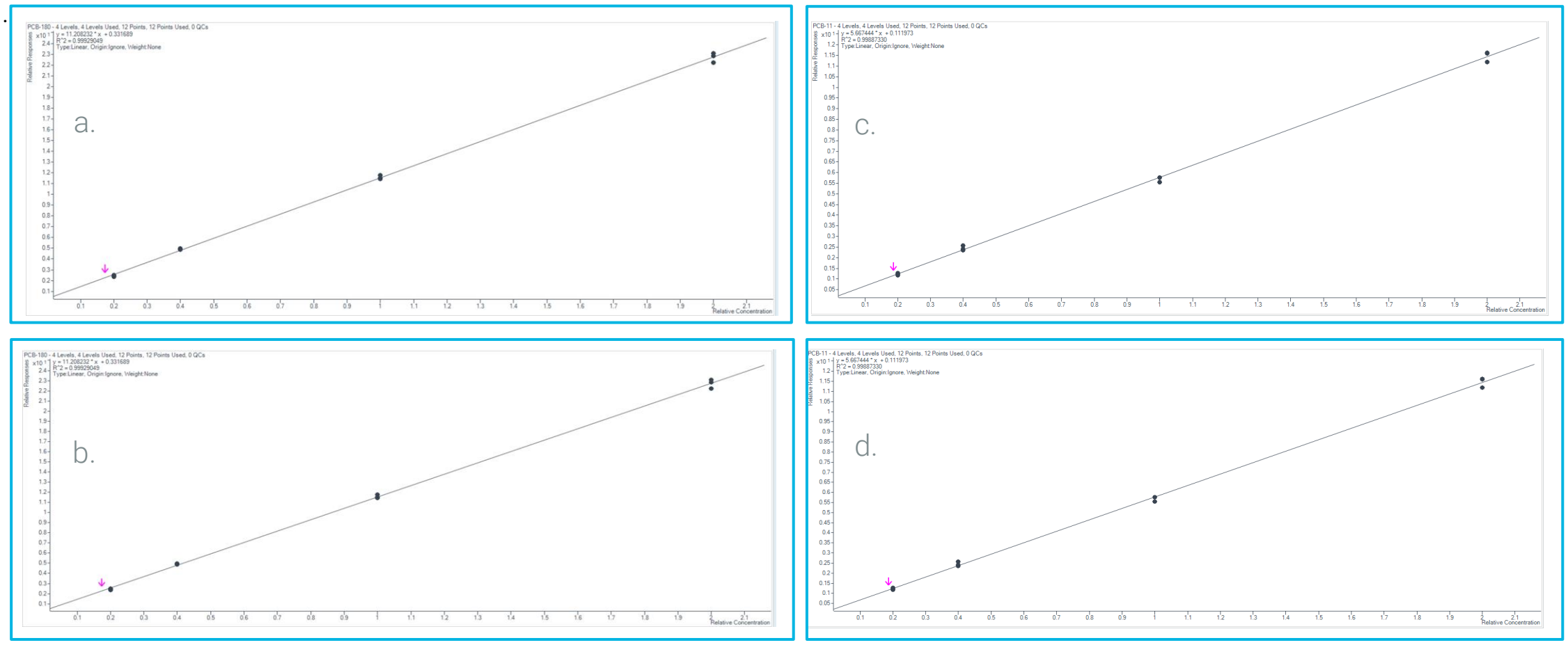

Figure 6: Calibration curves for PCB-180 in (a) helium carrier gas and (b) hydrogen carrier gas. And PCB-11 in (c) helium carrier gas and (d) hydrogen carrier gas.

Calibration Curves from Select PCBs

EPA method 1628 has selected 65 congeners to be quantitated to demonstrate linearity of instrument[2]. There are three methods to carry out this quantitation: true isoptopic dilution (tID), modified isotopic dilution (mID) and extracted internal standard (EIS). Figure a-d above shows four-point calibration curves for PCB congeners 11 and 180, which are calibrated by tID using their respective labeled congeners. The low end of the calibration curve was 2.5 and 5ppb for PCB 11 and 180.

Chromatographic Resolution

EPA 1628 states specific requirement the chromatographic resolution of PCB congeners 28 and 31. The valley of the two peaks must be less than 80% of the height of the smaller peak[2]. This criterion was met with both carrier helium and hydrogen gases.

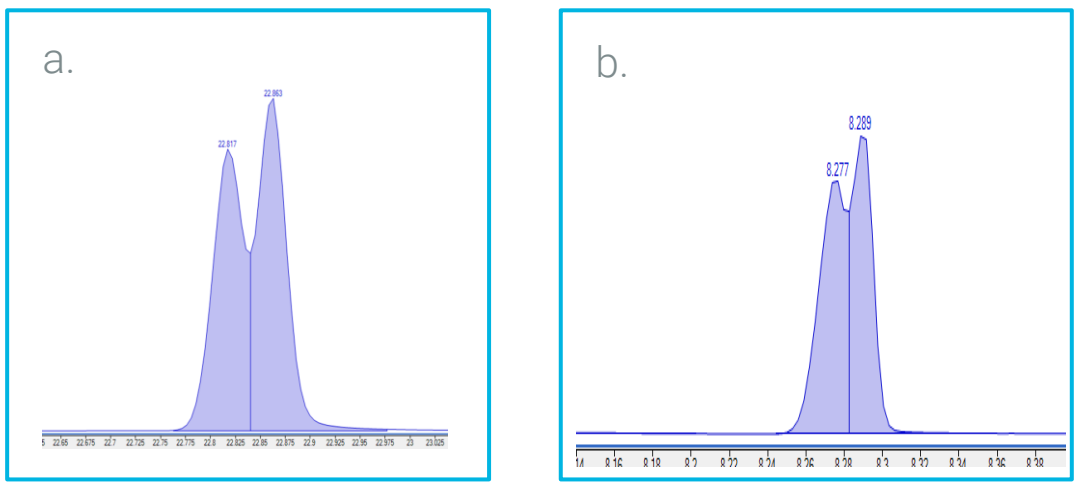

Figure 7: PCB congeners 28 and 31 mixed at roughly 1:1 ratio in helium (a) and hydrogen (b) carrier gases.

Conclusions

Two Robust methods for EPA Method 1628 was developed to analyze 209 native and 35 labeled PCB Congeners

- Using EPA Method 1628 guidelines and requirements, a robust method was developed with helium carrier gas.
- Using MassHunter method translator, a faster method that meets EPA Method 1628 guidelines was developed using Hydrogen carrier gas.
- Both methods can detect and quantitate PCB compound down to the single digit ppb levels.

References

[1]https://www.epa.gov/pcbs/learn-about-polychlorinated-biphenyls.

[2] Method 1628 Polychlorinated Biphenyl (PCB) Congeners in Water, Soil, Sediment, Biosolids, and Tissue by Low-resolution GC/MC using Selected Ion Monitoring (2021) Retrieved May 1, 2024 from https://www.epa.gov/system/files/documents/2021-07/method-1628_pcb-congeners-by-low-resolution-gc-ms_july-2021.pdf.

https://www.agilent.com/en/promotions/asms

DE45116444

Published in USA, May 31,2024

Agilent
Trusted Answers