Featured Application: *Fast PAH Analysis at Low Levels Using the GC Accelerator Kit*

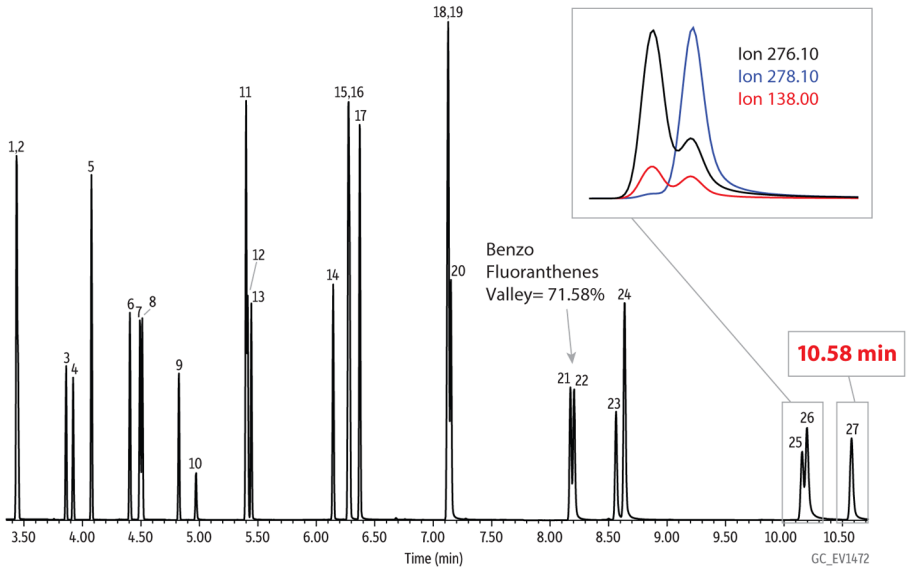
Increase Sample Throughput with New 15-Minute, Low-Level PAH GC-MS Analysis

- Scaled-down column and method open the door to faster PAH analysis; the GC Accelerator kit boosts oven ramp rates to meet the demands of scaled-down methods.
- Use your existing semivolatiles method in SIM mode for low-level PAH analysis.
- Reduce overall cycle time to increase sample throughput.

Fast, low-level analysis of polycyclic aromatic hydrocarbons (PAHs) is of global interest because of the high toxicity and carcinogenic properties of some of these compounds. For GC-MS analyses, the structure of PAHs lends them to analysis in the more sensitive, though selective, selected ion monitoring (SIM) mode because they commonly ionize without significant fragmentation under typical electron ionization conditions, resulting in strong molecular ion signals. This makes it easy to convert an existing semivolatile compound analysis, like U.S. EPA 8270D, that is typically run in full scan mode, to a SIM analysis that is able to detect even lower levels of PAHs.

The PAH GC-MS analysis shown here takes advantage of scaling the traditional column format (30 m, 0.25 mm, 0.25 µm Rxi-5Sil MS) down to a shorter, more efficient column that yields the same separations in less time (20 m, 0.15 mm, 0.15 µm Rxi-5Sil MS). The optimized scaled-down method parameters presented below ensure the elution profile on the new column format will remain the same as is typically observed on the traditional column. For Agilent 6890 and 7890 instruments with 120 V ovens that are not able to meet the ramp rates in the scaled-down analysis, Restek's GC Accelerator kit was used to boost the GC's ramp rate capability without any changes to the hardware or software.

As shown in the chromatogram, this PAH GC-MS analysis resulted in good peak resolution and response even at 0.05 ng on-column with just 15 minutes of analysis time. Anytime low-level PAHs are analyzed, particular care should be taken to mitigate the peak tailing that can result from the MS temperatures being set too low or from not using optimized source parts for the application. For example, in this case using a 9 mm extractor lens is recommended instead of the standard 3 mm version. Note that at particularly low concentrations, calibration may require alternate fit types, but internal testing demonstrated that method calibration requirements could still be met.



Peaks	tR (min)	Conc. (µg/mL)
1. Naphthalene-d8 (IS)	3.44	2.0
2. Naphthalene	3.45	1.0
3. 2-Methylnaphthalene	3.86	1.0
4. 1-Methylnaphthalene	3.92	1.0
5. 2-Fluorobiphenyl (SS)	4.08	2.0
6. Acenaphthylene	4.41	1.0
7. Acenaphthene-d10 (IS)	4.49	2.0
8. Acenaphthene	4.51	1.0
9. Fluorene	4.83	1.0
10. 2,4,6-Tribromophenol (SS)	4.97	2.0
11. Phenanthrene-d10 (IS)	5.40	2.0
12. Phenanthrene	5.41	1.0
13. Anthracene	5.45	1.0
14. Fluoranthene	6.15	1.0
15. Pyrene-d10 (SS)	6.28	2.0
16. Pyrene	6.29	1.0
17. *p*-Terphenyl-D14 (SS)	6.37	2.0
18. Benz[a]anthracene	7.12	1.0
19. Chrysene-d12 (IS)	7.13	2.0
20. Chrysene	7.15	1.0
21. Benzo[b]fluoranthene	8.17	1.0
22. Benzo[k]fluoranthene	8.21	1.0
23. Benzo[a]pyrene	8.56	1.0
24. Perylene-d12 (IS)	8.64	2.0
25. Indeno[1,2,3-cd]pyrene	10.16	1.0
26. Dibenz[a,h]anthracene	10.20	1.0
27. Benzo[ghi]perylene	10.58	1.0

See page 2 for chromatogram conditions.

Chromatogram Conditions

Column	Rxi-5Sil MS, 20 m, 0.15 mm ID, 0.15 µm (cat.# 43816)
Sample	EPA Method 8310 PAH mixture (cat.# 31874) Revised SV internal standard mix (cat.# 31886) Revised B/N surrogate mix (cat.# 31888) Acid surrogate mix (4/89 SOW) (cat.# 31063)
Diluent:	Dichloromethane
Injection	
Inj. Vol.:	1.0 µL split (split ratio 20:1)
Liner:	Topaz 4 mm single taper w/wool (cat.# 23303)
Inj. Temp.:	275 °C
Oven	
Oven Temp.:	60 °C (hold 0.7 min) to 285 °C at 39.8 °C/min to 305 °C at 4.3 °C/min to 320 °C at 28.5 °C/min (hold 3.5 min)
Carrier Gas	He, constant flow
Flow Rate:	1.0 mL/min
Detector	MS
Mode:	SIM

Group	Start Time (min)	Ion(s) (m/z)	Dwell (ms)
1	3.29	102, 108, 128, 136	25
2	3.71	85, 115, 142.1, 172.1	20
3	4.28	76, 82, 152.1, 153.1, 164.1	20
4	4.71	82.40, 142.90, 166.1, 329.8	25
5	5.24	89, 94, 178.1, 188.1	15
6	5.88	101, 106.1, 122.1, 202.1, 212.1, 244.1	20
7	6.83	101, 120.1, 226.1, 228.1, 240.2	10
8	7.77	126, 252.1	25
9	8.44	126, 132.1, 252.1, 264.2	25
10	9.57	138, 139, 276.1, 278.1	25

Transfer Line Temp.:	280 °C
Analyzer Type:	Quadrupole
Source Type:	Extractor
Extractor Lens:	9 mm ID
Source Temp.:	330 °C
Quad Temp.:	180 °C
Solvent Delay Time:	1 min
Tune Type:	DFTPP
Ionization Mode:	EI
Instrument	Agilent 7890B GC & 5977A MSD
Notes	Fast SIM analysis of 16 priority PAHs plus the methylnaphthalenes in a 120 V oven equipped with the GC Accelerator kit (cat.# 23849) (injected 1 µg/mL = 0.05 ng on-column).

GC Accelerator Oven Insert Kit

for Agilent 6890 and 7890 instruments

- Get the same GC separation in less time—use a GC Accelerator kit and the *EZ*GC method translator to accurately convert methods to a scaled-down column format.
- Scaled-down methods let you speed up analysis time and increase sample throughput without capital investment.
- GC Accelerator kit installs easily without damaging the GC column or interfering with the MS interface.

Designed with GC-MS users in mind, the GC Accelerator kit provides a simple way to speed up sample analysis. By reducing oven volume, these inserts allow faster ramp rates to be attained, which reduces oven cycle time and allows for increased sample throughput and more capacity to process rush samples. When faster ramp rates are used, existing methods can be accurately scaled down to smaller, high-efficiency, narrow-bore columns using Restek's *EZ*GC method translator. With a scaled-down column, a properly translated method, and a GC Accelerator kit, you can obtain the same chromatographic separation—often with greater sensitivity—in a fraction of the time without making a capital investment.

Description	qty.	cat.#
GC Accelerator Oven Insert Kit for Agilent 6890 and 7890 instruments	kit	23849

Rxi-5Sil MS Columns (fused silica)

low-polarity phase; Crossbond 1,4-bis(dimethylsiloxy)phenylene dimethyl polysiloxane

- Engineered to be a low-bleed GC-MS column.
- Excellent inertness for active compounds.
- General-purpose columns—ideal for GC-MS analysis of semivolatiles, polycyclic aromatic compounds, chlorinated hydrocarbons, phthalates, phenols, amines, organochlorine pesticides, organophosphorus pesticides, drugs, solvent impurities, and hydrocarbons.
- Temperature range: -60 °C to 350 °C.

Description	temp. limits	qty.	cat.#
20 m, 0.15 mm ID, 0.15 µm	-60 to 320/350 °C	ea.	43816

Topaz 4.0 mm ID Single Taper Inlet Liner w/ Wool

for Agilent GCs equipped with split/splitless inlets

ID x OD x Length	qty.	cat.#
Single Taper, Premium Deactivation, Borosilicate Glass with Quartz Wool		
4.0 mm x 6.5 mm x 78.5 mm	5-pk.	23303

EPA Method 8310 PAH Mixture (18 components)

Acenaphthene (83-32-9)
Acenaphthylene (208-96-8)
Anthracene (120-12-7)
Benz(a)anthracene (56-55-3)
Benzo(a)pyrene (50-32-8)
Benzo(b)fluoranthene (205-99-2)
Benzo(ghi)perylene (191-24-2)
Benzo(k)fluoranthene (207-08-9)
Chrysene (218-01-9)
Dibenz(a,h)anthracene (53-70-3)
Fluoranthene (206-44-0)
Fluorene (86-73-7)
Indeno(1,2,3-cd)pyrene (193-39-5)
1-Methylnaphthalene (90-12-0)
2-Methylnaphthalene (91-57-6)
Naphthalene (91-20-3)
Phenanthrene (85-01-8)
Pyrene (129-00-0)

Description	Conc. in Solvent and Volume	cat.#
EPA Method 8310 PAH Mixture	500 µg/mL each in acetonitrile:toluene (92:8), 1 mL/ampul	31874

Revised SV Internal Standard Mix (7 components)

Acenaphthene-d10 (15067-26-2)
Chrysene-d12 (1719-03-5)
1,4-Dichlorobenzene-d4 (3855-82-1)
1,4-Dioxane-d8 (17647-74-4)
Naphthalene-d8 (1146-65-2)
Perylene-d12 (1520-96-3)
Phenanthrene-d10 (1517-22-2)

Description	Conc. in Solvent and Volume	cat.#
Revised SV Internal Standard Mix	2,000 µg/mL each in methylene chloride, 1 mL/ampul	31885
Revised SV Internal Standard Mix	4,000 µg/mL each in methylene chloride, 1 mL/ampul	31886

Revised B/N Surrogate Mix (4 components)

2-Fluorobiphenyl (321-60-8)
Nitrobenzene-d5 (4165-60-0)
p-Terphenyl-d14 (1718-51-0)
Pyrene-d10 (1718-52-1)

Description	Conc. in Solvent and Volume	cat.#
Revised B/N Surrogate Mix	1,000 µg/mL each in methylene chloride, 1 mL/ampul	31887
Revised B/N Surrogate Mix	5,000 µg/mL each in methylene chloride, 1 mL/ampul	31888
Revised B/N Surrogate Mix	5,000 µg/mL each in methylene chloride, 1 mL/ampul	31888.15
Revised B/N Surrogate Mix, 5 mL	5,000 µg/mL each in methylene chloride, 5 mL/ampul	31889

Certified reference materials (CRMs) manufactured and QC-tested in ISO-accredited labs satisfy your ISO requirements.

Acid Surrogate Mix (4/89 SOW) (3 components)

2-Fluorophenol (367-12-4)
Phenol-d6 (13127-88-3)
2,4,6-Tribromophenol (118-79-6)

Description	Conc. in Solvent and Volume	cat.#
Acid Surrogate Mix (4/89 SOW)	2,000 µg/mL each in methanol, 1 mL/ampul	31025
Acid Surrogate Mix (4/89 SOW)	2,000 µg/mL each in methanol, 1 mL/ampul	31025.15
Acid Surrogate Mix (4/89 SOW)	2,000 µg/mL each in methanol, 1 mL/ampul	31025.25
Acid Surrogate Mix (4/89 SOW)	10,000 µg/mL each in methanol, 1 mL/ampul	31063
Acid Surrogate Mix (4/89 SOW)	10,000 µg/mL each in methanol, 1 mL/ampul	31063.15
Acid Surrogate Mix (4/89 SOW), 5 mL	10,000 µg/mL each in methanol, 5 mL/ampul	31087
Acid Surrogate Mix (4/89 SOW), 10 mL	10,000 µg/mL each in methanol, 10 mL/ampul	33029

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