# **DETERMINATION OF** POLYCYCLIC AROMATIC HYDROCARBONS (HJ 805-2016)

Technology Advantage: Agilent Intuvo 9000 GC with Agilent 5977 MSD



# Introduction

HJ 805-2016 is a method approved by the Chinese Ministry of Environmental Protection for the determination of 16 polycyclic aromatic hydrocarbons (PAHs) in soils and sediments by gas chromatography coupled to mass spectrometry (GC/MS).

The method details procedures for sample extraction, analysis, mass spectrometer performance verification, and quantitation. Prior to each analysis, the mass spectrometer must be tuned to achieved a given set of ion ratios. Also, instrument response must be proven to be consistent across the calibration range.

This application brief demonstrates that the Agilent Intuvo 9000 can easily achieve the performance specification for the analysis of PAHs in soil and sediments, as outlined in method HJ 805-2016.

## Instrumentation

- Agilent Intuvo 9000 GC
- · Agilent 5977 MSD with inert ion source with 6 mm and drawout plate
- Agilent DB-5ms UI column, 30 m × 0.25 mm, 0.5 μm

# **Sample Preparation**

- A standard mixture of 77 semivolatile organic compounds containing the 16 PAHs at a concentration of 200 µg/mL was used to prepare standards for the study (AccuStandard, New Haven, CT).
- Standards were prepared in dichloromethane at the concentrations specified in the method: 2, 5, 10, 20, and 40  $\mu g/mL$  with internal standard concentrations of 40  $\mu g/mL$ .
- A standard solution of decafluorotriphenylphosphine (DFTTP) was diluted to a final concentration of 25 μg/mL.

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### **Results and Discussion**

Figure 1 shows an example chromatogram of the 16 target PAHs, two surrogates, and five internal standards required by the method for the 20  $\mu$ g/mL standard. The chromatogram was constructed by summing the extracted quantitation ions for each compound (the smaller peaks in the chromatogram are from the semivolatiles included in the standard mix). Note that the peak shapes of the PAHs are symmetric, and not tailing. The critical isomers benzo(b)fluoranthene and benzo(k)fluoranthene were greater than 90 % resolved, which is equivalent to the resolution achieved on the Agilent 7890 Series Gas Chromatograph. This indicates an inert and uniformly heated flow path with no unswept or extra-column volume.

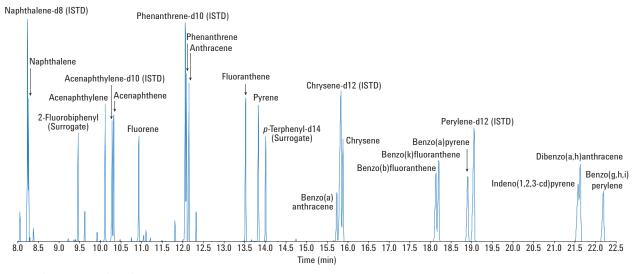


Figure 1. Chromatogram of the 16 PAHs, surrogates, and internal standards.

Before data collection, method HJ 805-2016 requires that the mass spectrometer and ion source are tuned to meet specified DFTPP ion ratios. Table 1 lists the required ion ratios in addition to the measured ion ratios on the Agilent 5977 Series MSD. Results show that the measured ratios are within the ranges required by the method.

Table 1. DFTPP Tuning check	ſ.

Target mass	Rel. to mass	Lower limit %	Upper limit %	Rel. abn %	Pass/Fail	
51	198	30	60	44	Pass	
68	69	0	2	2	Pass	
70	69	0	2	0	Pass	
127	198	40	60	47	Pass	
197	198	0	1	0	Pass	
198	Base peak					
199	198	5	9	7	Pass	
275	198	10	30	26	Pass	
365	198	1	100	7	Pass	
441	443		441<443		Pass	
442	198	40	100	99	Pass	
443	442	17	23	19	Pass	

HJ 805-2016 specifies use of average response factors for calibration. The calibration for each target must demonstrate a relative standard deviation (RSD) in response factors of less than or equal to 20 %. Table 2 lists the average response factor and the RSD across the calibration range. The magnitude and uniformity of response factors indicate good response over the range of the PAHs. Also, the RSDs for response factors are well within the acceptable range required by the method.

#### Table 2. Average response factors and relative deviations.

Compound	Average response factor	RSD (%)
Naphthalene	1.11	2.79
2-Fluorobiphenyl (surrogate)	1.60	1.64
Acenaphthylene	2.18	2.40
Acenaphthene	1.24	1.67
Fluorene	1.40	6.80
Phenanthrene	1.12	2.31
Anthracene	1.13	1.82
Fluoranthene	1.39	5.77
Pyrene	1.48	4.89
4,4'-terphenyl-d14 (surrogate)	0.84	5.52
Benzo(a)anthracene	1.27	2.77
Chrysene	1.24	0.98
Benzo(b)fluoranthene	1.20	9.02
Benzo(k)fluoranthene	1.19	6.66
Benzo(a)pyrene	1.14	5.57
Indeno(1,2,3-cd)pyrene	1.14	9.87
Dibenzo(a,h)anthracene	1.15	10.11
Benzo(g,h,i)perylene	1.16	5.67

# Conclusion

The Agilent Intuvo 9000 GC and Agilent 5977 Series MSD demonstrated outstanding performance for the analysis of PAHs in accordance with method HJ 805-2016. The system was able to achieve tuning ratios as specified in the method in addition to providing quantitation performance within the method guidelines. For more detailed information and methodology, refer to application note [1].

# Reference

 Giardina, M. Analysis of Semivolatile Organic Compounds using the Intuvo 9000 GC, *Agilent Technologies Application Note*, publication number 5991-7180EN, 2016.

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