# Fast analysis of coal tar polycyclic aromatic hydrocarbons on Agilent J&W Select PAH

## Application Note

## Author

John Oostdijk
Agilent Technologies, Inc.

## Introduction

The difficulty in analyzing polycyclic aromatic hydrocarbons (PAHs) is the number of PAHs with the same mass. This makes their separation by GC/MS rather difficult, and so column selectivity and an optimized oven program are necessary for the resolution of PAHs. We describe here the fast analysis of a coal tar sample using an optimized oven program and a 15 m x 0.15 mm x 0.10 µm Select PAH column.

Coal tar is a brown or black liquid of high viscosity that smells of naphthalene and aromatic hydrocarbons. It is obtained from the destructive distillation of coal. In the past, coal tar was sourced as a by-product from the manufacture of coal gas but is now produced during the production of coke for steel making. The crude tar contains many organic compounds, such as benzene, naphthalene, methylbenzene and phenols, which can be obtained by distillation, leaving a residue of pitch. At one time coal tar was the major source of organic chemicals, most of which are now derived from petroleum and natural gas. Coal tar pitch is mainly used as binding agent in the production of carbon electrodes, anodes and Søderberg electrodes, for instance, by the aluminium industry. It is also used as a binding agent for refractories, clay pigeons, active carbon, coal briquetting, road construction and roofing. In addition, small quantities are used for heavy-duty corrosion protection.

The standard reference material for coal tar analysis (SRM 1597a, NIST) is a natural, combustion-related mixture of PAHs from a medium crude coke-oven tar that is dissolved in toluene. It is intended for use in the evaluation and validation of analytical methods for the determination of PAHs. A sample of SRM 1597a was therefore analyzed using an optimized oven program for the Select PAH column, demonstrating the excellent performance of the column with this reference material.

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**Conditions**

| | |
|---|---|
| Technique: | GC/MS, Triple Quad |
| Column: | Select PAH, 15 m x 0.15 mm, df = 0.10 µm (part number CP7461) |
| Sample: | SRM 1597a, concentration approximately 0.2-1030 mg/kg (www.nist.gov) |
| Injection Volume: | 1.0 µL SIM mode, 0.1 µL FS mode |
| Temperature: | 70 °C (0.40 min), 70 °C/min, 180 °C |
| Program: | 7 °C/min, 230 °C (7 min), 50 °C/min, 280 °C (7 min), 30 °C/min, 350 °C (4 min) |
| Carrier Gas: | Helium, constant flow 1.2 mL/min |
| Injection: | 300 °C, Splitless mode, 0.5 min @ 100 mL/min |
| Detection: | Triple Quad, EI in FS or SIM mode, ion source 275 °C, transfer line 300 °C |

# Results and Discussion

The sample was analyzed directly in full scan and SIM mode using the same conditions as for a standard with regulated and interfering PAHs. Chromatograms with peak identifications are shown in Figures 1 to 3. Figure 1 shows chrysene (66 mg/kg) separated from triphenylene (12 mg/kg). Benzo[b,k,j]fluoranthene (66, 37 and 41 mg/kg) was separated from benzo[a]pyrene (94 mg/kg). The MS spectrum from chrysene is also shown. As well as m/z 228, other ions, such as m/z 226 and m/z 229, were formed. PAHs were mostly very stable and only low fragment ions were observed, for example m/z 113 and 114 in the mass spectrum.
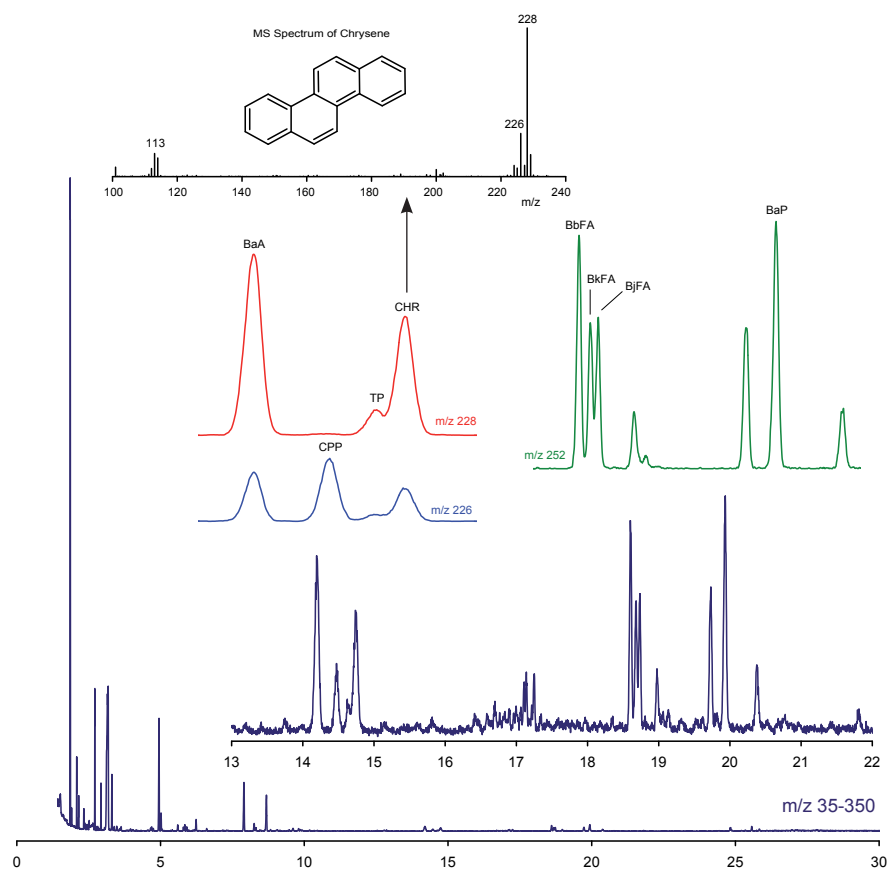
**Table 1. Peak identification for Figure 1**

| Peak | Name |
|---|---|
| BaA | Benzo[a]anthracene |
| BaP | Benzo[a]pyrene |
| BbFA | Benzo[b]fluoranthene |
| BkFa | Benzo[k]fluoranthene |
| BjFA | Benzo[j]fluoranthene |
| CHR | Chrysene |
| CPP | Cyclopenta[c,d]pyrene |
| TP | Triphenylene |



Figure 1. GC/MS analysis of 0.1 µL of SRM 1597a in full scan mode (m/z 35-350) on Select PAH

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*Table 2. Peak identification for Figure 2*

| Peak | Name |
|------|------|
| ACL | Acenaphthylene (263 mg/kg) |
| BaA | Benzo[a]anthracene (98 mg/kg) |
| BaP | Benzo[a]pyrene (94 mg/kg) |
| BghiP | Benzo[g,h,i]perylene (51 mg/kg) |
| DBaeP | Dibenzo[a,e]pyrene (9 mg/kg) |
| FA | Fluoranthene (327 mg/kg) |
| PHE | Phenanthrene (454 mg/kg) |
| PY | Pyrene (240 mg/kg) |
| NA | Naphthalene (1030 mg/kg) |



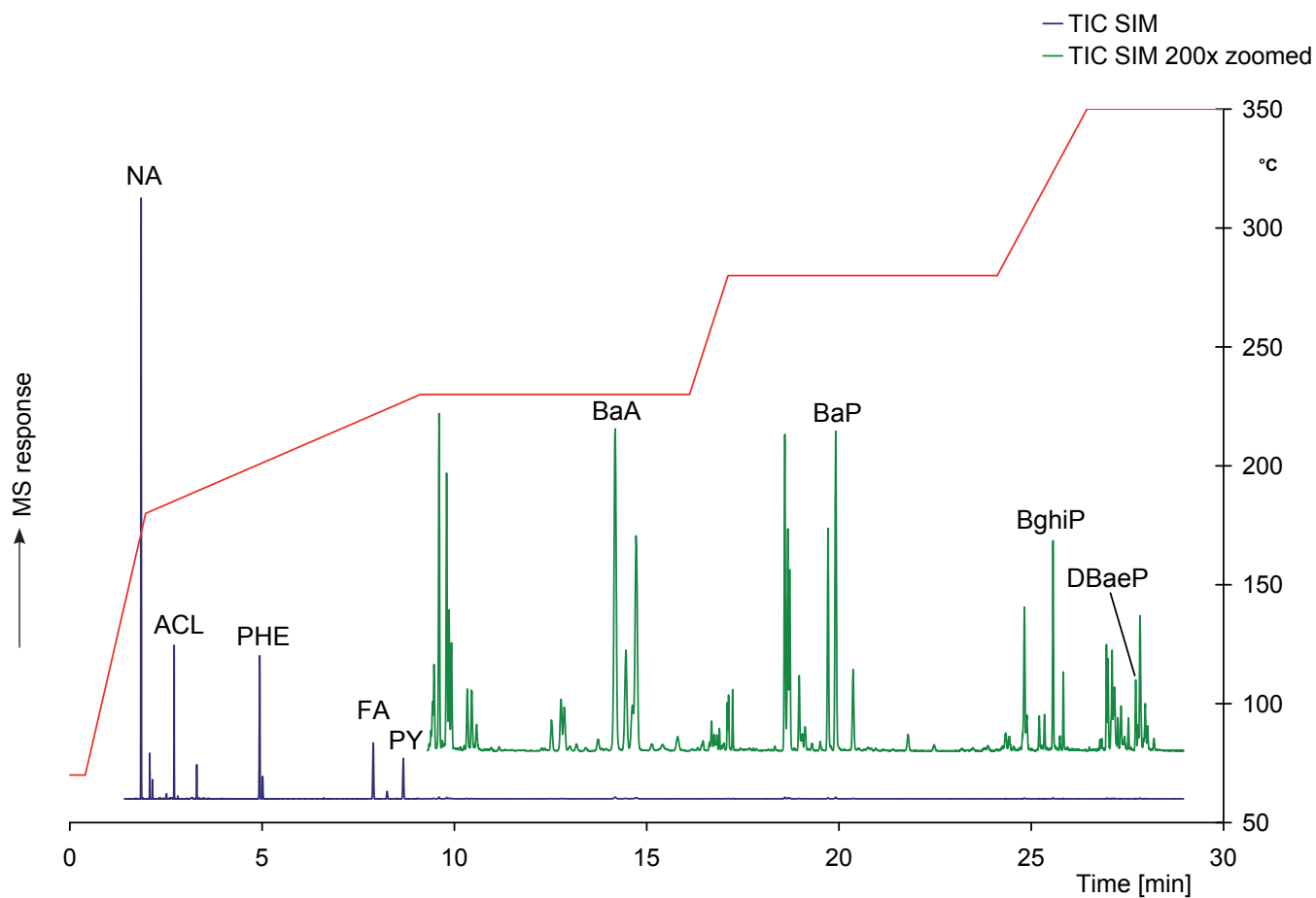*Figure 2. GC/MS analysis of 1 µL of SRM 1597a in SIM mode on Select PAH. The final part of the chromatogram was enlarged because of the large concentration difference in the sample*

**Table 3. Peak identification for Figure 3**

| Peak | Name |
|------|------|
| ATR | Anthanthrene (Dibenzo[d,e,f,m,n,o]chrysene) |
| BaFA | Benzo[a]fluoranthene |
| BaFL | Benzo[a]fluorene |
| BaP | Benzo[a]pyrene |
| BbCHR | Benzo[b]chrysene |
| BbFA | Benzo[b]fluoranthene |
| BbFL | Benzo[b]fluorene |
| BbNTP | Benzo[b]naphto[2,1-d]thiophene |

| Peak | Name |
|------|------|
| BbPer | Benzo[b]peryelene |
| BbTP | Benzo[b]triphenylene |
| BcFL | 7H-benzo[c]fluorene |
| BeP | Benzo[e]pyrene |
| BghiP | Benzo[g,h,i]perylene |
| BjFA | Benzo[j]fluoranthene |
| BkFA | Benzo[k]fluoranthene |
| Cor | Coronene |
| DBaeP | Dibenzo[a,e]pyrene |

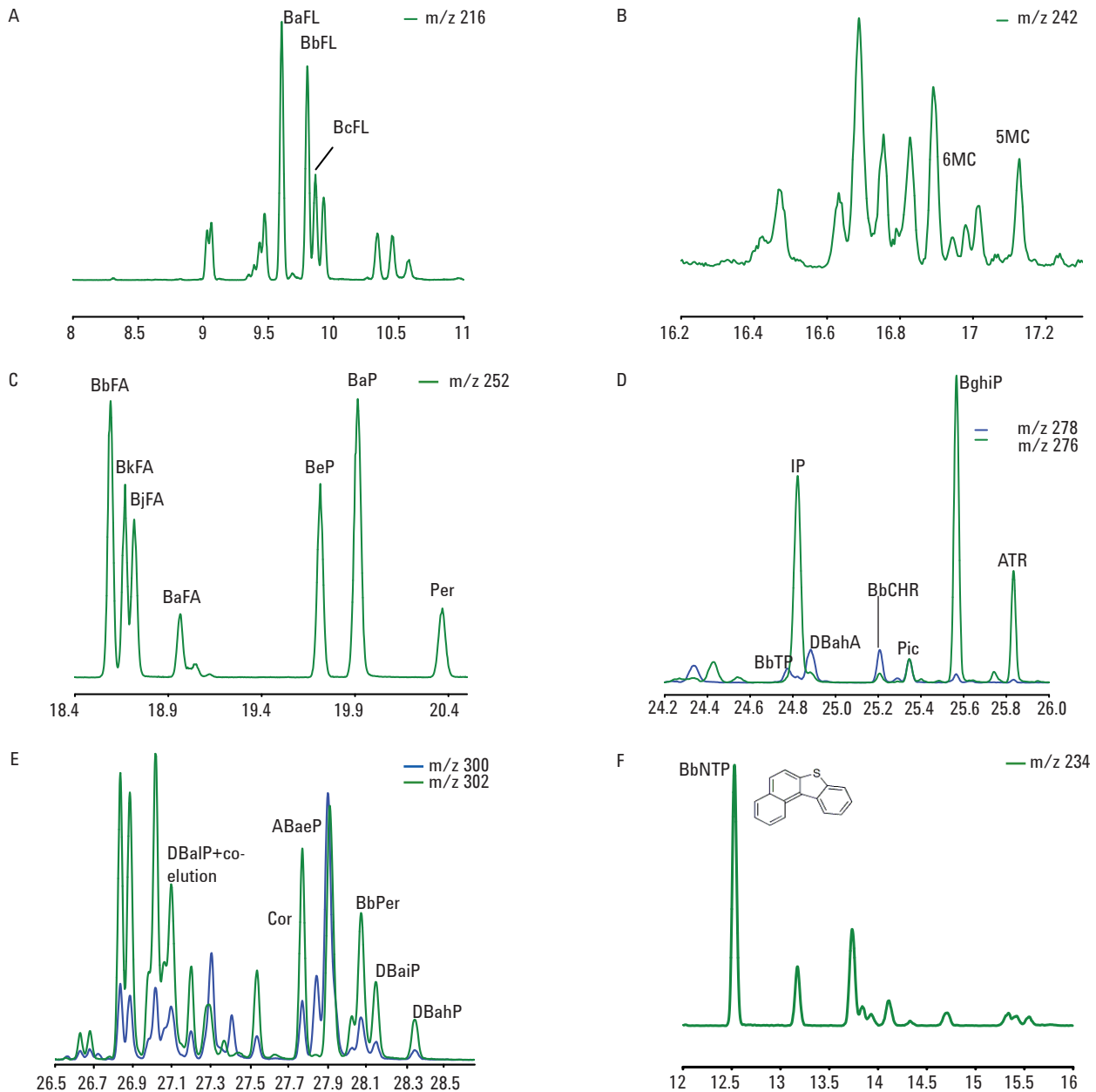| Peak | Name |
|------|------|
| DBahA | Dibenzo[a,h]anthracene |
| DBahP | Dibenzo[a,h]pyrene |
| DBaiP | Dibenzo[a,i]pyrene |
| DBalP | Dibenzo[a,l]pyrene |
| IP | Indeno[1,2,3-cd]pyrene |
| 6MC | 6-Methylchrysene |
| 5MC | 5-Methylchrysene |
| Per | Perylene |
| Pic | Picene |



Figure 3. GC/MS analysis of 1 µL of SRM 1597a in SIM mode on Select PAH . Some examples of critical separations are shown: A. Benzofluorenes, B. Methylchrysenes, C. Benzofluoranthenes and isomers, D. Dibenzoanthracenes and isomers, E. Dibenzopyrenes and isomers, F. Benzo[b]naphtho[ ] thiophene isomers

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# Conclusion

The Select PAH column separated target PAHs in a complex mixture of coal tar in a single run with a run time of 29 min. Together with an optimized oven program, this unique column is the only easy-to-use, single-shot solution that separates all regulated PAH isomers, with no false positives or inaccurate results. With high speed, high resolution and high temperature stability it is the perfect column for accurate PAH analysis.

# References

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