ANALYSIS OF SEMIVOLATILES
SYSTEM SUITABILITY

Technology Advantage: Agilent Intuvo 9000 GC with Agilent 5977 MSD

Introduction

For the analysis of semivolatile organic compounds (SVOCs) by gas chromatography coupled to mass spectrometry (GC/MS), it is of paramount importance to verify that the system is suitable for quantitative analysis before the collection of data. This is of particular importance when data are used for reporting based upon regulatory agency requirements.

For example, the United States Environmental Protection Agency (USEPA) Method 8270D specifies the use of a control standard for verifying the performance of the GC/MS[1]. The method specifies a mixture of decafluorotriphenylphosphine (DFTTP), 4,4′-dichlorodiphenyltrichloroethane (4,4′-DDT), pentachlorophenol, and benzidine to test MS tuning and system inertness.

The DFTPP is used to verify suitable ionization and detection of the the mass spectrometer. The 4,4′-DDT is used to gauge system inertness by determination of the breakdown products 4,4′-DDD and 4,4′-DDE. Benzidine is used as a probe for base activity, and pentachlorophenol is used as a probe for acid activity. If the minimum performance criteria as established by the method cannot be achieved, the system is deemed unsuitable for analysis.

This Application Brief demonstrates that the Agilent Intuvo 9000 GC can easily achieve the system suitability specifications as established in USEPA 8270D for the quantitative analysis of SVOCs in environmental matrices.

Instrumentation

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

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Sample Preparation

- A standard mixture of DFTPP, 4,4′-DDT, benzidine, and pentachlorophenol was purchased from Sigma-Aldrich (St. Louis, MO).
- The mixture was diluted 2:1 in dichloromethane to produce a solution of 25 µg/mL of each component.

Results and Discussion

Figure 1 shows a chromatogram of the control standard. Annotated in Figure 1 are the tailing factors (TFs) for pentachlorophenol and benzidine calculated at 10 % peak height. According to 8270D, the calculated TFs for these compounds cannot exceed 2, or the system is deemed too active with respect to the acid and base probes for analysis.

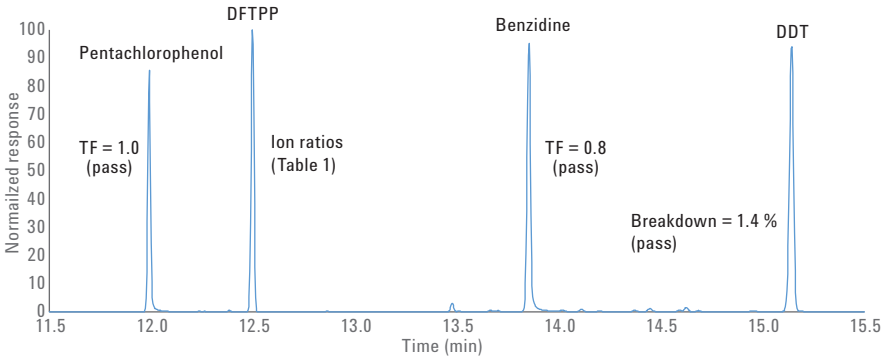

Figure 1. Chromatogram of tuning mix passing 8270D specifications.

Figure 1 also shows the percent breakdown of 4,4′-DDT. The combined breakdown of 4,4′-DDT to 4,4′-DDD and 4,4′-DDE cannot exceed 20 %, or the system is deemed too reactive.

Table 1 lists the relative abundances of ions produced from the electron ionization of DFTPP. To verify the performance of the mass spectrometer, the relative ion abundances must be within the upper and lower bounds, as listed.

Table 1. DFTPP tuning check.

Target mass	Rel to mass	Lower limit %	Upper limit %	Rel. abn %	Pass/Fail
51	442	10	80	31.1	Pass
68	69	0	2	0	Pass
70	69	0	2	0.4	Pass
127	442	10	80	39.4	Pass
197	442	0	2	0	Pass
198	442	50	100	84	Pass
199	198	5	9	6.1	Pass
275	442	10	60	22.4	Pass
365	198	1	100	4.2	Pass
441	442	0	24	15.4	Pass
442	442	100	100	100	Pass
443	442	15	24	18.7	Pass

Conclusion

The control standard specifications required by USEPA Method 8270D were easily achieved using the Agilent Intuvo 9000 GC and an Agilent 5977 MSD and an Intuvo DB 5ms UI column.

For more detailed information and methodology, refer to Application Note 5991-7256EN[2].

References

1. Semivolatile Organic Compounds by GC/MS, U.S. Environmental Protection Agency, Method 8270D, Revision 4, February **2007**.
2. The analysis of semivolatile organic compounds using the Agilent Intuvo 9000 Gas Chromatograph, *Agilent Technologies Application Note*, publication number 5991-7256EN.

Published in USA, September 1, 2016
5991-7183EN

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