TDTS 34

Monitoring trace-level high-boiling compounds (triethyl phosphate and methyl salicylate) in air

Summary

This Application Note demonstrates the long-term stability of the UNITY™ setup with regard to the off-line analysis of two high-boiling analytes, methyl salicyclate (MS) and triethyl phosphate (TEP).

Experimental

A standard solution containing 200 ng of MS and 200 ng of TEP per microlitre of pure methanol was prepared. Various volumes of this standard were introduced to the sampling end of Tenax® TA tubes in a 60 mL/min flow of pure nitrogen using Markes' Calibration Standard Loading Rig (CSLR™).

The tubes were left connected to the CSLR for 2 min, until the bulk of the methanol had been purged to vent.

TD (UNITY):

Prepurge:	1 min at ambient temp. (split on and trap in-line)
Flow path:	190°C
Carrier gas:	24 psi
Cold trap:	Packed with quartz wool and Tenax TA
Trapping temp.:	–10°C
Trap desorb:	300°C for 5 min
Heating rate.	Maximum
Split flow:	31 mL/min
Desorb flow:	27 mL/min
Overall split ratio:	58:1 (split on during primary and secondary desorb)

GC:

Column:	30 m × 0.25 mm × 0.5 µm DB-1 equivalent phase
Temp.:	60°C (2 min), 20°C/min to 250°C (1 min)

FID:

Temp.:	300°C

Results and discussion

Comparison of the data produced (Figures 1 and 2, and Tables 1 and 2) clearly demonstrates the long-term stability of this tube-based thermal desorption system for TEP and MS.

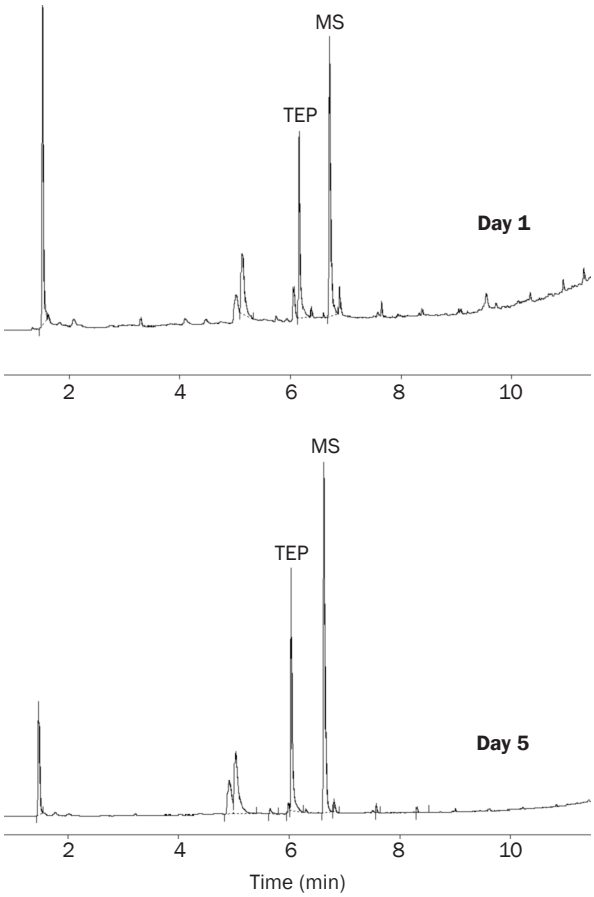

Figure 1: Chromatogram showing performance of FID response to 200 ng of each TEP and MS.

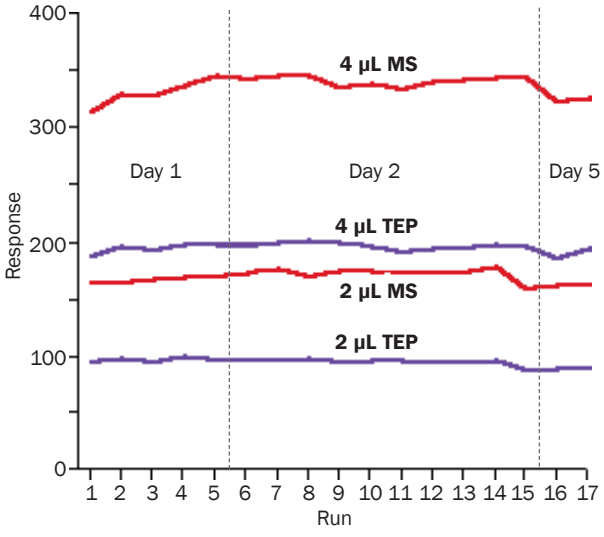

Figure 2: Stability data for TEP and MS over 5 days.

Volume injected (µL)	TEP area counts	MS area counts
2	97.2	154.7
0.5	26.1	40.3
4	190.9	311.2
6	282.3	460.2
1	50.6	84.6
0.5	24.5	42.8
4	197.9	324.6
6	286.4	470.7
1	51.9	85.3
2	94.1	156.3

Table 1: Calibration data for TEP and MS produced on Day 1.

Volume injected (µL)	TEP area counts	MS area counts
2	82.9	160.9
4	184.2	320.3
2	88.2	159.8
4	193.6	323.6

Table 2: Calibration data for TEP and MS produced on Day 5, demonstrating excellent consistency with the data from Day 1.

Although this work describes the off-line monitoring of TEP and MS, round-the-clock on-line analysis is also possible using UNITY combined with an Air Server autosampler. In this case, air and vapours are drawn through a heated, inert link line directly into the electrically-cooled focusing trap of UNITY. Sample flows and times can be adjusted using the electronic mass flow controller built into the Air Server™ accessory. This system allows semi-continuous monitoring of atmospheric concentrations in a given location as they change with time.

Trademarks

Applications were performed under the stated analytical conditions. Operation under different conditions, or with incompatible sample matrices, may impact the performance shown.

Version 2
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