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Further Development of Spiking Thermal Desorption Tubes using GERSTEL Tube Spiking System and TD3.5+

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Introduction

Earlier work performed at Anatune in conjunction with Health and Safety Laboratory (HSL) show the capabilities of using the GERSTEL tube spiking system (TSS) to liquid spike a range of volatile organic compounds (VOCs) in application note AS201. The data produced showed that compounds previously spiked onto thermal desorption tubes using an air-loading system can also be done using liquid spiking. To further develop this work and to continue the collaboration, HSL wanted to further test the capability of the TSS and use semi-volatile organic compounds (SVOCs) as the loading mixture. To be a complete solution, the autosampler and TSS needs to be able to handle a wide range of compounds so that separate systems are no longer required for separate work streams.

The aim of this work was to further demonstrate the feasibility and MPS capabilities of liquid spiking SVOCs onto 3.5 inch TD tubes using the Tube Spiking System (pictured left) for quantitative analysis at HSL on their GC-FID and also analysis at Anatune using GERSTEL's TD3.5+ thermal desorption unit.

Instrumentation

GERSTEL MPS Robotic Dual Head Robotic/Robotic^{Pro}
GERSTEL Tube Spiking System
Agilent 7890GC/7010QQQ
GERSTEL TD3.5+
GERSTEL CCD2
GERSTEL CIS 4
Masshunter B.07

Method

3.5 inch Tenax TD tubes, supplied by Health and Safety Laboratory (HSL) were placed in each of the TSS positions. Each tube was spiked with 5µL of an SVOC standard using the Robotic^{Pro} equipped with Universal Syringe Module and a 10µL syringe. A portion of tubes were sent back to HSL for analysis with the remaining half left for analysis on the GERSTEL TD3.5+. This was done to test the reproducibility of the automated tube spiking.

Transport adapters were applied to each tube and loaded onto TD3.5+ sample tray and analysed using the GERSTEL MPS Robotic to automate the analysis. The TD3.5+ was set to splitless mode with the CIS 4 in solvent vent mode using the inlet purge flow to set a split of 50:1. Samples were analysed using a DB-5 column which was of similar phase to that being used by the customer, with the Mass spec running in SIM mode.
For temperatures, TD transfer temperature was set at 280°C, with a desorption temperature of 30°C up to 260°C. During desorption, the CIS 4 was kept was at -30°C then later ramped up 260°C. A CIS 4 Tenax TDU liner was used to aid trapping of the compounds in the inlet during desorption. To aid in the sorption of analytes form the desorption tubes, a time of ten minutes was used for desorption. For cooling, a GERSTEL CCD2 2-channel cryostatic cooling device was used.

Results

Agilent Masshunter B.07 Quant was used to assess the data, using toluene as internal standard for semi-quantification using a one point calibration. A typical chromatogram of a spiked tube and a blank can be seen below in figure 2.

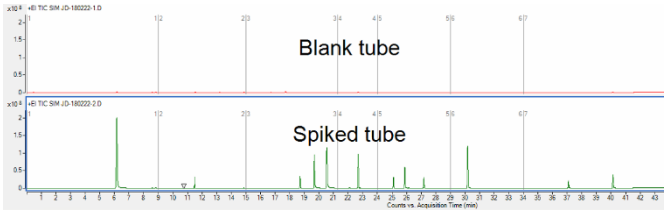

Figure 2. Chromatogram of a spiked tube compared with a blank

The %RSD data for all compounds is displayed below in Table 1 with the data produced by the testing system at HSL in Table 2. Both sets of results show very good data with also the differences between using a GC-FID and GC-MSD and method of quantitation being highlighted.

Compound	Retention Time (min)	%RSD (n=8)
C10	11.4	4.43
C14	18.7	2.60
Dimethyl phthalate	19.7	0.85
Acenaphthene	20.6	1.90
Lauric acid	22.1	42.89
2,2,4-trimethyl-1,3-pentanediol diisobutyrate(TXIB)	22.7	5.99
C17	25.1	4.67
Diallyl phthalate	25.9	4.93
C18	27.2	1.14
Dibutyl phthalate	30.2	1.94
Benzyl butyl phthalate	37.1	5.01
Diethylhexyl phthalate	40.1	6.05

Table 1 –%RSD values for all compounds analysed at Anatune

Compound	Retention Time (min)	%RSD
Toluene	8.4	1.20
C10	17.6	1.21
C14	29.6	1.16
Dimethyl phthalate	30.7	0.94
Acenaphthene	31.8	1.18
Lauric acid	32.7	5.97
2,2,4-trimethyl-1,3-pentanediol diisobutyrate(TXIB)	33.4	1.34
C17	35.2	1.91
Diallyl phthalate	35.8	1.91
C18	36.6	1.30
Dibutyl phthalate	38.8	1.53
Benzyl butyl phthalate	43.6	1.51
Diethylhexyl phthalate	46.3	1.33

Table 2 –%RSD values for all compounds analysed at HSL

It should be noted that the poorer performance of the lauric acid component is more likely to be due to limitations in the chromatographic method than the spiking procedure. In particular, the presence of a polar carboxylic acid group greatly affects chromatography and as a result quantitation.

Whilst formal recovery experiments were not performed, the data produced by HSL was compared to theoretical loading values to produce approximate accuracy values. These values are displayed in Table 3. Further experiments would be needed to formally assess bias and recovery data.

Compound	%Bias
Toluene	1.94
C10	3.27
C14	3.08
Dimethyl phthalate	5.35
acenaphthene	2.89
Lauric acid	5.36
2,2,4-trimethyl-1,3-pentanediol diisobutyrate(TXIB)	5.46
C17	4.66
Diallyl phthalate	4.36
C18	4.18
Dibutyl phthalate	3.82
Benzyl butyl phthalate	3.56
Diethylhexyl phthalate	5.56

Table 2 –%Bias values for all compounds analysed at HSL

Discussion

The tube spiking system has shown to be able to handle a wide range of analytes which lends to the versatility of the MPS rail.
Spiking of thermal desorption tubes can now be automated with analysis also being automated by using GERSTEL thermal desorption unit/TD3.5+.

Acknowledgements

Ian Pengelly, Health and Safety Laboratory, Buxton (UK), Phine Banks, Account Manager, Anatune Ltd, Girton, Cambridgeshire (UK).