Sample Recollection by the CDS 7550S Automated Thermal Desorber

Application Note
General

Author:

Michael Apsokardu
Xiaohui Zhang

Abstract
This application note demonstrates the quantitative recollection of split sample to a clean sample saver tube. This is demonstrated by different split percents on the CDS 7550S automated thermal desorber for a group of VOCs with boiling point up to 218 °C.

Introduction
Gas chromatography (GC) is an analytical technique by separating a mixture of compounds for a downstream detector to identify the chemical composition of each component. Direct liquid injection intot the GC inlet is the most common method of introducing sample onto the GC column. However, in many situations, the analyte identity, analyte concentration, or the matrix are not compatible for GC analysis. To tackle this challenge, various sample introduction techniques, including Purge and Trap, Thermal Desorption, Pyrolysis, and Solid Phase Micro Extraction were developed to introduce analyte onto the GC column to achieve the best separation result.

Among the GC sample introduction techniques, thermal desorption involves heating a thermal desorption sample tube, which is packed with sorbent, to a desired desorption temperature and then purging with inert gas to release volatile organic compounds (VOCs) adsorbed on the sorbent surface. The purging gas, along with mixed VOC analytes, flows through a heated sample pathway in the vapor phase to reach the GC for separation and detection. The sample splitting technology to thermal desorption instrumentation offers the flexibility to reduce the amount of analyte reaching the GC inlet and extend the dynamic range of the detector coupled to the GC. A concern of sample splitting, however, is that it is a single-shot technique whereby sample going to split is lost out of the split vent not allowing for repeat analysis of a given sample. The option to recollect sample going to the split vent on a clean sample tube has many advantages, including repeat sample analysis, method validation, and acquiring experimental results via complementary detection methods.

Here the CDS 7550S automated Thermal Desorber incorporates a sample recollection feature, also referred to as the sample saver, as a function within the sample split option. The sample saver is combined with sample splitting to analyze quantitative performance of this sample saver function. Seven different VOCs with boiling points up to 218 °C are tested for split percents of 50%, 75%, and 95%, which is the recommended range for quantitative splitting for the 7550S.

Experiment Setup
A CDS 7550S automated thermal desorber with the sample split / sample saver option was used for testing. The VOCs desorbed from the thermal desorption sample tube were first split in the 7550S at a user-selected split ratio, which was fulfilled by a mechanism electronically controlled by a Mass Flow Controller (MFC). The portion of the sample split going to the vent was recollected on a clean sample tube. Both the primary sample tube and the sample saver tube were manufactured by Camsco and packed with Carbograph 1/Carbograph 2/ Carboxen 1000 (P/N SU644-4).

7550S Thermal Desorber:

Valve oven:	245 °C
GC transfer line:	250 °C
Tube purge flow:	60 mL/min
Pre-heat time:	15 s
Tube Rest temp.:	37 °C
Tube Dry purge temp.:	37 °C
Tube Dry purge time:	0.1 min
Tube Desorb temp.:	330 °C
Tube Desorb time:	8 min
Primary sample tube:	Camsco P/N SU644-4
Sample saver tube:	Camsco P/N SU644-4
Trap Rest temp.:	-20 °C with Peltier
Trap Desorb temp.:	300 °C
Trap Desorb time:	2 min
Trap Type:	Tenax TA
Peltier transfer line:	250 °C

GCMS QP-2010

GC conditions:

Oven temp.:	35.0 °C
Injection temp.:	240 °C
Injection mode:	Split
Column Flow:	1.01 ml/min
Split Ratio:	40.0
Temp. program:	35.0 °C hold 4 min
	10.0 °C/min to 90.0 °C
	20.0 °C/min to 150.0 °C
	30.0 °C/min to 220.0 °C
	Hold 3.10 min

MS conditions:

Ion Source:	200.00 °C
Interface Temp.:	220.00 °C
Start m/z:	35.00
End m/z:	260.00

For the other portion of the split, VOCs were adsorbed by a secondary focusing trap, which was electronically cooled by a Peltier module. Sample adsorbed inside the focusing trap was then transferred to the GC inlet. The 7550S and GC-MS parameters are listed above:

Benzene, toluene, ethylbenzene, m,p-xylene, o-xylene and naphthalene standards were purchased from Sigma-Aldrich. The standards were mixed and diluted in methanol to a final concentration of 400 mg/L for each component of the stock solution.

1 µL of the stock solution was injected onto a pre-conditioned thermal desorption sample tube. The methanol was removed by purging the sample tube with nitrogen at 110 mL/min for 1 min. This thermal desorption tube was then loaded into the autosampler rack of the 7550S for analysis.

Samples were desorbed from the sample tube to the focusing trap and the clean sample saver tube at 50% split. Half of the sample that was collected on the focusing trap and was then desorbed from the trap to the GC-MS. The other half of the sample collected on the clean sample saver tube was then removed manually from its collection position and then returned to autosampler rack. Sample collected onto the sample saver tube was then desorbed to the focusing trap, without splitting, and then later desorbed to the GC-MS for analysis.

Split percents of 75% and 95% were also tested. When the split percents are 75% and 95%, this is the fraction that is transferred to the split vent where the sample saver tube is located. The remainder of the sample is the fraction transferred to the electronically cooled trap and later desorbed to the GC. The quantitative performance of the sample split / sample saver option was assessed by analyzing accuracy and reproducibility for a total of three different split percents.

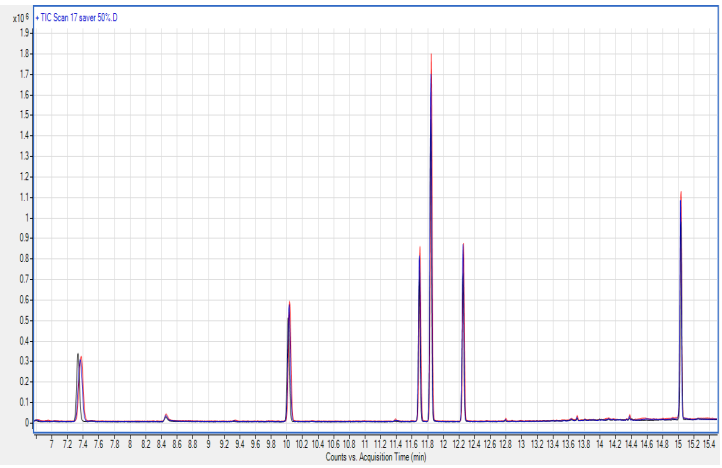

Figure 1: TIC overlay of 3 runs at 50% split ratio.

Table 1: Reproducibility as %RSD from 3 runs at 50%, 75%, and 95% split ratios.

	Split Percent (n=3)					
	50%		75%		95%	
Compound	(% Acc)	(% RSD)	(% Acc)	(% RSD)	(% Acc)	(% RSD)
benzene	103.8	2.2	100.3	0.4	99.3	0.8
toluene	100.1	0.5	99.0	0.1	98.8	0.1
ethylbenzene	100.1	0.5	98.8	0.3	98.7	0.2
m,p-xylene	99.8	0.5	98.6	0.3	98.6	0.2
o-xylene	99.5	0.4	98.5	0.5	98.5	0.2
naphthalene	100.1	0.2	99.2	0.1	99.1	0.1

Results and Discussions

Reproducibility was tested by obtaining % Accuracy (Acc) and %RSD for each of the peaks at a fixed split ratio through multiple runs. Figure 1 is the total ion chromatogram (TIC) overlay from 3 runs at 50% split ratio as an example. Table 1 shows the %Acc and %RSD for each from the three different split percents tested, which were 50%, 75%, and 95%. The %Acc was calculated as the measured fraction of total signal from the sample saver tube compared to the expected fraction of total signal.

From the table above, the data accuracy is within 98.5%-103.8%, as well as precision below 2.2% at each of the different split percents. These results indicate that, not only is sample quantitatively split, but is also quantiatively captured by the sample saver tube.

Conclusions

This application note has showcased a sample saver function in the 7550S automated thermal desorber. The results show that the sample is quantitatively captured by the sample saver tube following sample splitting. This proves that the 7550S is a versatile thermal desorption instrument that offers a sample recollection mechanism to provide users with enhaced flexibility when performing sample analysis.