Application Note No. 063

Testing the Performance of the Focus Direct-TD with Real Samples

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Introduction

The Focus Direct-TD is an automated thermal desorber. It uses the Focus autosampling robot to automatically exchange special packed sample tubes, SepLiners, held in a 98-position DTD tray, into the head of the Optic programmable injector. The head is then closed and sealed pneumatically, and the sample is desorbed onto the head of the column and analysed. The Focus DTD can also be used for the automated desorption of solid and liquid samples using the Difficult Matrix Introduction (DMI) technique.

Two separate organisations suggested different sets of experiments to test the performance of the Focus-DTD in the thermal desorption role.

In Experiment 1, packed SepLiners were spiked with a sample of triethylphosphate (TEP) and analysed to look at:

1) Linearity (to test for sample losses)
2) Relative Standard Deviations (required level <4%)
3) Carryover from the sample tray to the packed liner

No attempt was made in this study to assess the suitability of the adsorbent packing material for the sample.

In Experiment 2, samples of headspace above rosemary leaves, containing high levels of volatiles, and samples from broad bean leaves, containing low levels of volatiles, were used to look at the loss of volatiles to the sample tray.

Also, in Experiment 3, we tested for:

1) Contamination of liners with volatiles from the laboratory
2) Contamination of the injector with volatiles from the lab while exchanging liners

Instrumentation

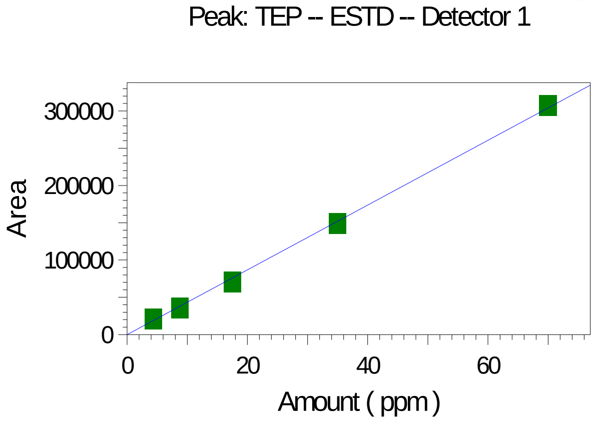
- ATAS Optic 2-200 programmable injector
- ATAS Focus Autosampling Robot with Direct-TD option
- Agilent 5890 GC with FID

Experiment 1: Procedure

Pre-conditioned SepLiners, containing 50 mg Porapak, were spiked with 1 µL TEP at levels of 70, 35, 17.5, 8.75 and 4.375 ppm for the calibration; 70 ppm for the RSD determination; and 1 µL of 70 ppm, the high standard, and 1 µL of the neat TEP to check for cross contamination within the sample tray.

Results & Discussion

A calibration graph was constructed after the analysis of samples containing TEP at levels between 70 and 4.375 ppm. These high concentrations were used due to the use of an FID instead of an NPD - the detector usually used for this sample. The results are shown in Figure 1.



Goodness of fit:	0.998948

Figure 1: *Calibration graph and results for TEP*

The correlation coefficient of 0.999 for the calibration line is good. If there had been any sample loss from the liners in the DTD tray, we would expect the results to deviate from the linearity depending on time spent waiting for analysis.

The concentration of TEP used to determine the repeatability and reproducibility was 70 ppm. Ten spiked SepLiners were placed in the DTD tray and four were analysed on day 1, the remaining six were analysed on day 2, the results are shown in Figure 2.

Area	**1**	**2**	**3**	**4**	**5**	**6**
Day 1	250519	241897	248793	236129		
Day 2	241749	256588	263286	251991	243934	243326

	Mean	**Std Dev**	**RSD**
Day 1	244335	6618	**2.7**
Day 2	250146	8639	**3.5**

Figure 2: *RSDs of 70 ppm TEP run on two consecutive days*

The RSDs were below the required levels of 4% for both day 1, with 2.7%, and day 2, with 3.5%. The mean response was even slightly higher on day 2. This indicates that there was no loss of this sample from the SepLiners while stored in the DTD tray.

To check for carryover from the tray to the packed liner two packed SepLiners, spiked at concentrations of 70 ppm and neat solution, were placed in the DTD tray, analysed an hour later, then immediately replaced with pre-conditioned blank SepLiners. These were analysed one hour later, then a further nineteen hours later, the results are shown in Figure 3.

Sample	**70 ppm**	**Neat**
Sample area on thermal desorption	487722	13495368
Blank SepLiner after 1 hr in tray	0	68214
% Carryover	**0.00**	**0.48**
Blank SepLiner after 19 hours in tray	0	50795
% Carryover	**0.00**	**0.38**

Figure 3: *Carryover from the tray to packed SepLiners*

The results from the high standard, of 70 ppm, showed that there was no carryover between samples via the sample tray. However, the packed SepLiner spiked with the neat solution showed that there was a very small amount, less than 0.5%, carried over between samples, which persisted over nineteen hours.

Experiment 2: Procedure

The samples used consisted of two identical samples from the dual entrainment of rosemary leaves (RM1 & RM2) containing high levels of volatiles; and two identical samples from the dual entrainment of broad bean leaves (BB1 & BB2) containing low levels of volatiles.

The first of each sample (RM1 and BB1) were analysed, and then the second of each sample (RM2 and BB2) were run 16 hours later.

Results & Discussion

Figure 4 shows the two chromatograms of the Rosemary leaves containing high levels of volatiles. The traces are very similar indicating there was very little if any sample loss while the tube was waiting for analysis in the sample tray.

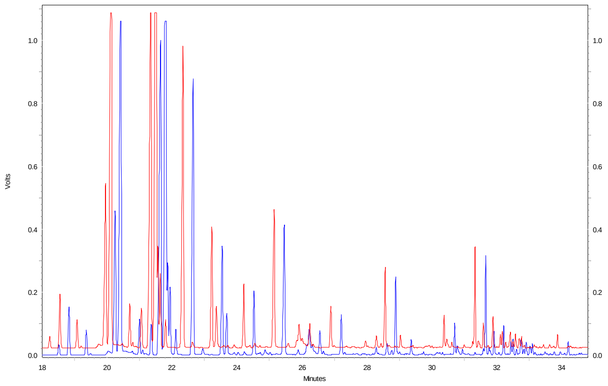


***Figure 4**: Chromatograms showing RM1 (lower curve) and RM2 (upper curve) run 16 hours later, containing high levels of volatiles*

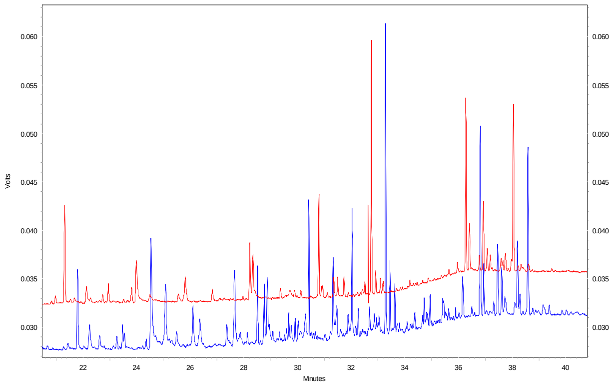


***Figure 5**: Chromatograms showing BB1 (lower curve) and BB2 (upper curve) run 16 hours later, containing low levels of volatiles*

Figure 5 shows the chromatograms from the analysis of the broad bean leaves, 16 hours apart. There is very little sample loss of the low level volatiles.

Experiment 3: Procedure

To test for the contamination of the injector when the head was open, and contamination of the packed liners in the sample tray from volatiles present in the laboratory atmosphere, bottles of hexane, dichloromethane and acetone were left open near the instrument in a normal laboratory environment for two hours before the experiments took place. A SepLiner was conditioned in the injector, the head was opened and the liner removed by the Focus DTD, then immediately replaced, as a normal automated exchange would take place, and re-desorbed to test for exposure of the injector to the laboratory environment. Another SepLiner was conditioned in the injector then left in the sample tray for two hours before re-desorbing, to test for exposure to the lab while in the DTD tray.

Results & Discussion

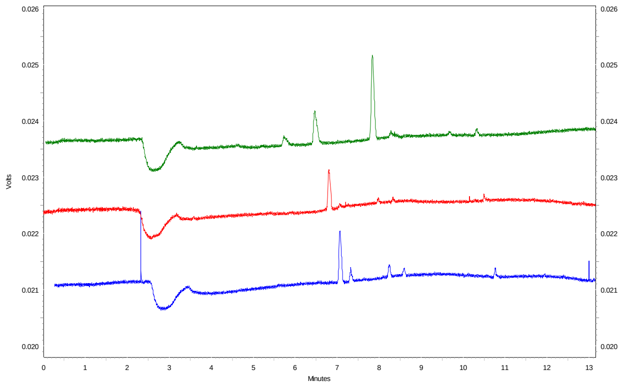


***Figure 6**: Chromatograms showing a re-desorbed blank liner (lower curve) not removing it from the injector, exposure of the injector (middle curve) and exposure of the liner for two hours (upper curve)*

Figure 6 shows that there was no contamination of the injector while the head was open, as there were no solvent peaks present in the trace. Therefore, the back flushing of the injector was efficient in preventing laboratory air from contaminating the injector while the head was open. There is also very little, if any, contamination of the packed liner with volatiles from the laboratory while sealed in the sample tray.

Conclusions

The experiments carried out thoroughly tested the performance of the Focus Direct-TD.

1) They showed that there was very little, if any, cross contamination between samples while in the sample tray, this included even very highly concentrated samples.
2) There was no contamination of the injector and column from highly concentrated volatiles in the laboratory air, and minimal contamination of packed liners in the sample tray from volatiles present.
3) There was very little, if any, losses of high or low concentration volatiles from the sample tube while in the sample tray, therefore good calibration curves were produced and the relative standard deviations were well below the required levels of 4%.

The Focus Direct-TD is therefore suitable and capable of performing well as an automated thermal desorber.

Acknowledgements

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