Application Note No. 086

The On-line VOC Analysis of Industrial Effluents using Ambient Headspace

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Introduction

In a world of ever lowering detection limits and stringent relative standard deviations (RSDs) it is often easy to forget that for some analyses these are not necessary. In the case of monitoring industrial effluents for volatile organic compounds (VOCs) only the ability to observe a sudden upward trend in levels is crucial to the analysis, relatively high RSDs and detection limits of around 0.5 ppm are acceptable.

In this environment, a simple, fully automated, on-line analysis is advantageous, where the instrument is set-up to take, prepare and analyse a sample once an hour, requiring no interaction from the technician, who checks the data system regularly for any changes in results and makes any decisions accordingly. The Focus Robotic Sample Processor is fully programmable and flexible, enabling the set-up of such a system. The Focus flowcell directs a flowing stream of effluent to the instrument for on-line analysis. The syringe can then take a sample from the stream and carry out any required sample preparation. Although the Focus can be fully equipped for headspace analysis using a heated syringe, this is not appropriate in this case, and also not necessary. As relatively high detection limits and RSDs are acceptable, and the samples to be analysed are both volatile and have a low solubility in matrix, the headspace can be equilibrated at room temperature providing satisfactory results. This keeps the method simple, quick and easy to set-up. Calibrations are infrequent, and the only maintenance is the loading of empty vials and the topping up of wash vials.

Instrumentation

- Focus Robotic Sample Processor with Flowcell
- ATAS Optic 2-200 Programmable Injector (a standard Agilent split/splitless injector was used on site) Agilent
- 6890 GC with FID

Analysis Method

1) Remove 250 uL of effluent from flowing stream through the Flowcell
2) Place in empty capped 2 mL vial
3) Aspirate sample and mix
4) Allow sample to equilibrate
5) Take portion of headspace and inject using hot split injection
6) Clean the syringe with water

Results

The calibration curves from the 5 VOCs, toluene, ethyl benzene, o-, m- and p-xylene, are shown below. These were spiked into effluents obtained on different days at concentrations of 0.4 to 4 ppm.

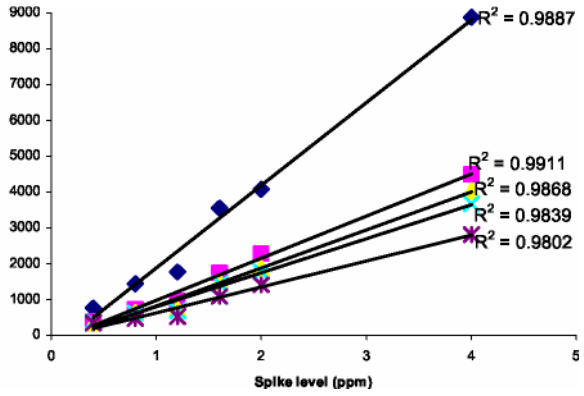

Figure 1: Calibration curves from spiking efluents from diferent days

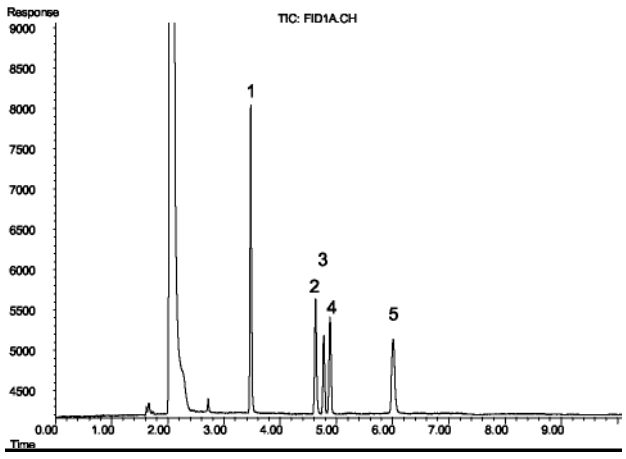

Figure 2: *Chromatogram of a standard of the five VOCs at a concentration of 4 ppm: (1) Toluene, (2) Ethyl benzene, (3) para -Xylene, (4) meta -Xylene and (5) ortho -Xylene*

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The calibration check for the on-line system is shown in Table 1 for two different days. This shows that the ambient headspace is working well, even with slight changes to the ambient temperature.

Table 1: Calibration of the on-line analysis

	Toluene	Ethyl benzene	p-Xylene	m-Xylene	o-Xylene
Concentration (ppm) Day 1	1.68	1.70	1.59	1.69	1.70
Concentration (ppm) Day 2	1.69	1.70	1.59	1.70	1.74

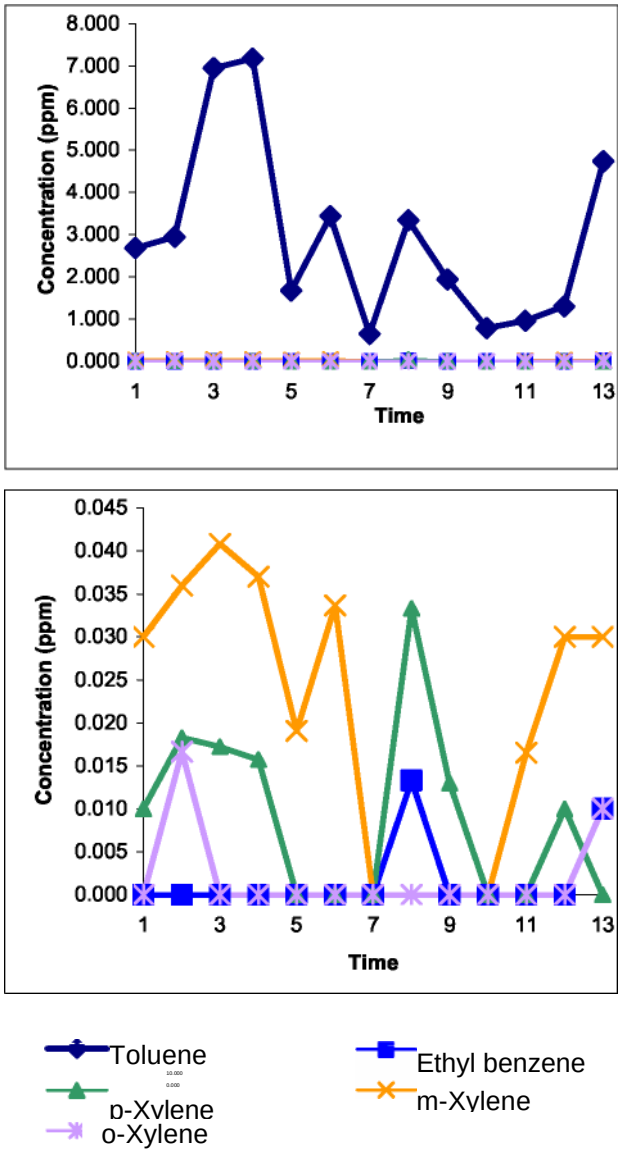

Figure 3: Monitoring the concentration of each of the VOCs over a thirteen hour period

The concentration of VOCs from the hourly monitoring of the effluent is shown for a 13 hour period in Figure 3. From this the trend of concentrations can be monitored for each of the volatile organic compounds and a sharp upward rise can easily be observed.

Conclusions

Ambient headspace for the analysis of VOCs in industrial effluent is a suitable technique. The required limits of detection, calibration and repeatability are met and it is a simple and easy method. The ability to make it a fully functional on-line analysis is advantageous as it requires no user interaction, requiring only the refilling of wash stations and the replacement of vials.

Acknowledgements

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For more information on our applications and techniques please visit our website www.atasgl.com.

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