

Use of the SIFT-MS for a selection of Health and Safety Applications (Visit to HSL)

Kathy Ridgway, Anatune Ltd. Girton, Cambridgeshire (UK).

Introduction

Selected Ion Flow Tube Mass Spectrometry (SIFT-MS) is a real-time volatile organic compounds (VOCs) analyser. At the beginning of June, we spent a week at the Health and Safety Laboratory in Buxton with the voice200 SIFT-MS to evaluate its use for a number of their challenging applications. This note gives a summary of some of the data obtained during that visit.

Selected Ion Flow Tube Mass Spectrometry (SIFT-MS) is a form of direct mass spectrometry that uses precisely controlled soft ionisation to enable real-time, quantitative analysis of volatile organic compounds (VOCs) in air, typically at detection limits of parts-per-trillion level (by volume; pptv). This eliminates the need for sample preparation, pre-concentration and chromatography. Figure 1 shows a schematic of the instrumentation.

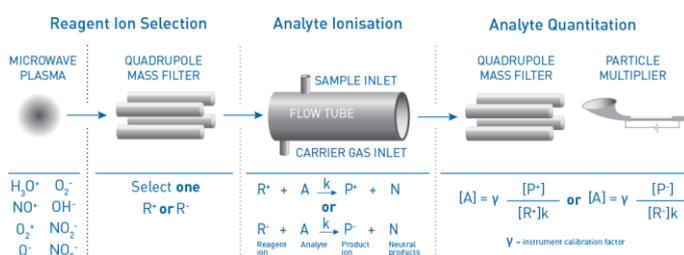


Figure 1: Schematic representation of the SIFT-MS technique.

Reagent ion selection – A microwave discharge through moist air forms the standard SIFT-MS positive and negative ions; H₃O⁺, NO⁺, O₂⁺, OH⁺, O₂⁻, O⁻, NO₂⁺ and NO₃⁻ and these are then selected using a quadrupole mass filter.

Analyte ionization – The selected reagent ion is injected into the flow tube and excess energy is removed through collisions with the carrier gas (either nitrogen or helium). The sample is then introduced and an ion-molecule reaction takes place to form well-characterised product ions.

Analyte quantitation – Product ions and unreacted reagent ions pass into a second quadrupole mass analyser and the analyte concentration is calculated as a ratio of product ions to reagent ions multiplied by a rate constant, *k*, unique to that ion-molecule reaction.

The use of eight, selectable reagent ions, coupled with a library of known reaction products and reaction rates enables SIFT-MS to quantify multiple analytes, in real-time, without the need for prior chromatographic separation.

It has been shown to be suitable for a diverse range of applications, as highlighted in previous application notes (AS146, AS147).

Several applications were demonstrated, some of which had not been tested previously using the SIFT-MS. The Voice200ultra SIFT-MS was set up in the laboratory to enable connection to a number of experiments, in some cases using additional tubing and external pumps. As the SIFT-MS measures concentrations per volume (ppbV, or µgcm⁻¹), where possible vessels/enclosures were used in order to 'contain' the volatiles produced.

Instrumentation

Syft Technologies' Voice200ultra running LabSyft software (version 1.4.9) Helium carrier gas, standard Hex inlet. (Figure 2).



Figure 2: Voice200ultra SIFT-MS with Hex inlet.

Methods and Results

Multicomponent mixture in real time from standards calibration rig.

HSL at Buxton are responsible for producing standards on thermal desorption tubes for proficiency tests. These are prepared using an in-house calibration rig. The SIFT-MS was used to measure the concentration and observe in real time changes to parameters used to dilute the concentration of analytes.

A mixture containing a range of compounds was prepared and concentrations varied by using stepped splits and then adjusting the syringe driver speed and dilution flows. The SIFT-MS was connected to the chamber via PTFE tubing. Compounds monitored were; Diacetyl (2,3-butanedione), benzene, trichloroethylene, toluene, PGME acetate, Styrene, limonene, n-undecane, naphthalene and methyl salicylate. During acquisition splits were changed to reduce the concentration as shown in the table. The real time plot is shown in Figure 3.

Acquisition Time	
0-190s	No split
200-380s	Split 1
400-580s	Split 2
600-780s	Split 2.8
780-1200s	Split 3
1200s-1450s	Vap flow dropped by half
1450-1800s	Vap flow dropped 3.6 (split 3)
1800-2500s	Syringe driver reduced (1/2 to 0.25)
3200s	Tubing disconnected from chamber (lab air)
Last 180s	Tubing re-connected to SIFT-MS, but not chamber (to assess carry over in tubing)

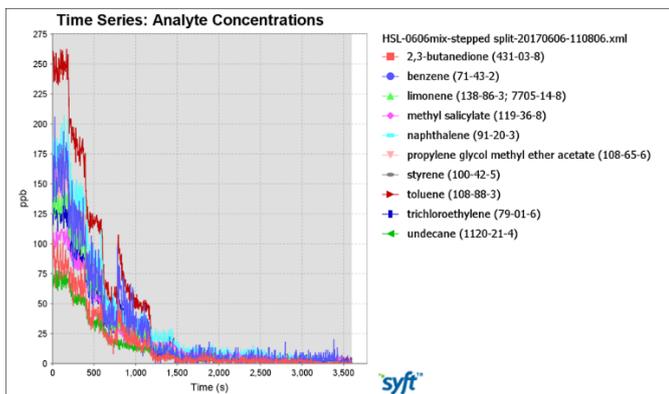


Figure 3: Real time response to concentration changes on calibration rig

Emissions from a 3D printer

The SIFT-MS was set up to monitor emissions from a 3D printer, following an hour program to make a simple cube. The printer was enclosed in a box and tubing inserted into the top of the box above the printer. ABS polymer was used and compounds monitored were styrene, acrylonitrile and 1,3-butadiene (Figure 4). Ethyl benzene was also monitored for the later part of the experiment. After completion a pump was used to flush the chamber and this was also monitored in real time using the SIFT-MS (Figure 5). Subsequently emissions were also monitored from the printer on an open bench. This gave considerably lower concentrations of approximately 4ppb for ethyl benzene and less than 1ppb for the other analytes measured.

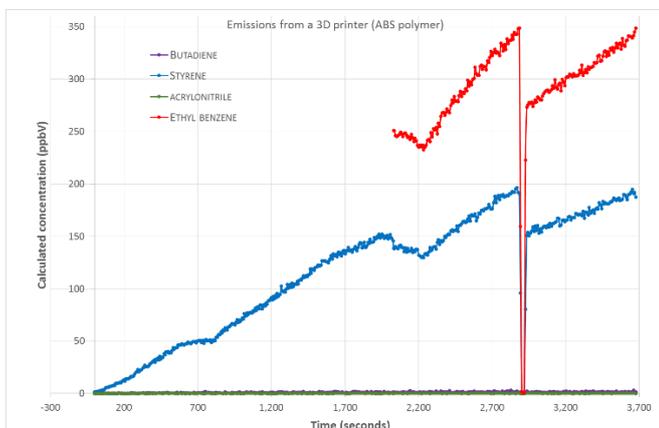


Figure 4: Analytes monitored for enclosed printer

(note at 750s printer nozzle fault noted, printing resumed following adjustment, at 2000s acquisition stopped and re-started, at 2850s, the sampling tube was removed from the box (to check air background).

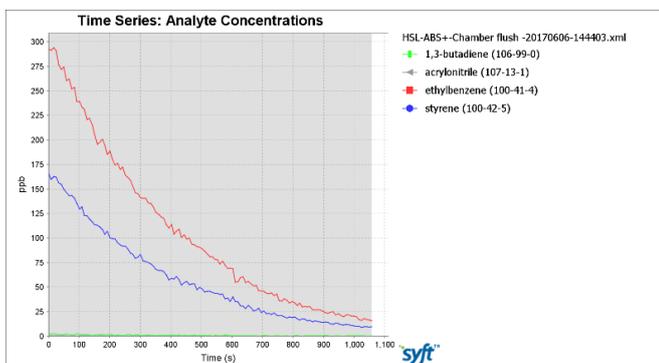


Figure 5: Chamber flushing after printing

Leak testing

This experiment was designed to monitor surrogate compounds that are used to assess the potential of certain devices containing drugs to leak. Phenoxyethanol and tetraethylurea are the proposed surrogates. As these compounds were not already present in the LabSyft library, Full Mass Scan acquisitions were performed to determine the product ions produced. A bell jar was set up for containment of the volatiles as shown in Figure 6. An external pump was attached to the tubing to ensure a stable flow (the SIFT-MS will take in approximately 25ml/min).

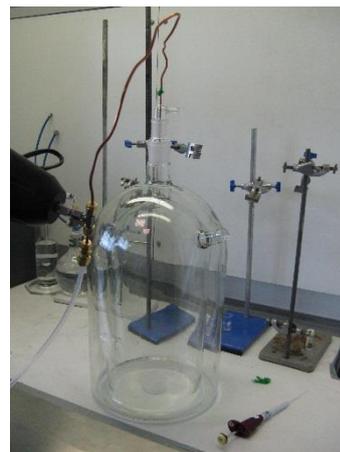


Figure 6: SIFT-MS connected via T-piece to Bell jar.

Full Mass Scan data for tetraethylurea (Figure 7) showed strong product ions at m/z 172 and 171 for NO^+ and m/z 100 and 72 for O_2^+ (only a weak response was observed for m/z 173 (m_w+1) for H_3O^+). Branching ratios and K values were estimated to give approximate concentrations.

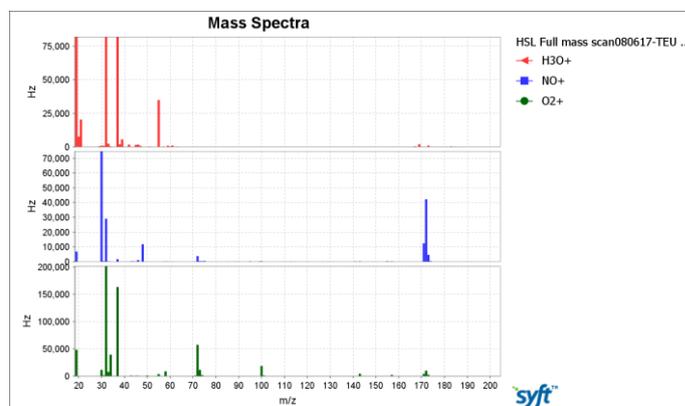


Figure 7: SIFT-MS Full Mass Scan of TetraethylUrea:

For phenoxyethanol, Product ions were observed at m/z 139 (m_w+1) for H_3O^+ , m/z 138 for NO^+ and m/z 94 for O_2^+ .

SIM acquisition methods were set up and used to monitor release of compounds over time when a small amount of solution was placed on a watch glass in a bell jar. For comparison, the same test was performed on a much more volatile analyte (IPA). Results are shown in Figure 8.

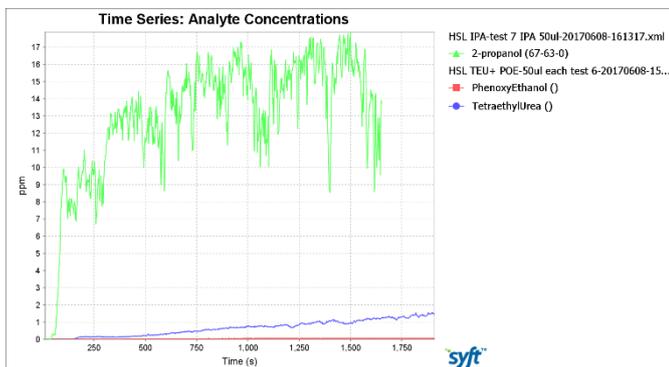


Figure 8: Release of compounds giving different analyte responses (based on volatility)

Analysis of peracetic acid formation

This experiment was designed to investigate the formation of peracetic acid (PAA) in real time in order to compare results with those of a real-time monitor and to compare airborne concentrations of PAA with those of hydrogen peroxide and, in particular, acetic acid, a substance often measured as a surrogate for PAA. A method was set up to measure PAA, hydrogen peroxide and acetic acid generated from a small volume of aqueous PAA solution in the chamber shown in Figure 6.

Figure 9 shows a plot of the three components. PAA and acetic acid show good correlation, so in this instance measurement of acetic acid would be a good indicator of PAA concentration. However, airborne concentrations of hydrogen peroxide were very low and show little correlation with those of PAA. Data from the SIFT-MS showed good correlation with those obtained from the dedicated real time PAA monitor.

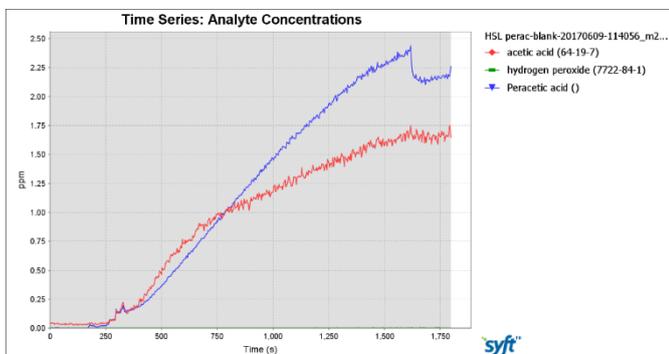


Figure 9: Peracetic acid concentrations measured in real time.

Other experiments

Other experiments performed during the visit, included the monitoring of emissions from a heated micro chamber in real-time (in this case looking at methyl salicylate). The test had been performed by conventional means (trapping on tubes followed by thermal desorption), but further information could be gained through real time analysis.

An investigation was also undertaken to monitor the formation of chloramines through the reaction of bleach and urine (linked to hospital spills, but also thought to be the 'swimming pool' smell).

Conclusions

This application note demonstrates just a selection of health and safety applications that the SIFT-MS can be used for, and the benefits of the additional information that can be obtained from real time monitoring. These examples have demonstrated the range of compounds and the different experimental designs that can be used to enable analysis in seconds, or over a longer period of time. They also show the ease of adding your own compounds of interest to the LabSyft library through full mass scan analysis of the pure compounds.

The team at HSL were able to see the potential of the SIFT-MS first hand and commented... *we found it extremely useful to be able to view concentrations profiles for individual components at ppb levels in real time. This is particularly important in health and safety research where peak concentrations are often of great significance. The results obtained also demonstrated to us how this technology has the potential to significantly reduce the time required to carry out research into health and safety as, with Anatune's assistance, we were able to generate useful data in several topic areas in just a few days. The SIFT-MS equipment was also able to switch between tests with very little set up time required which enabled multiple experiments to be carried out on the same day. Finally, we were impressed at how easily methods could be developed for new and unusual compounds such as tetraethylurea.*

Acknowledgements

I would like to thank all at HSL for their hospitality during our week in Buxton. In particular Ian Pengelly for planning and hosting our visit.

