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High-Throughput VOC and Inorganic Gas Analysis: Automated SIFT-MS

Introduction

Selected ion flow tube mass spectrometry (SIFT-MS) enables direct, real-time monitoring of volatile organic compounds (VOCs) and inorganic gases. Unlike traditional chromatography-based techniques, there is no requirement to separate compounds prior to analysis. Not only can challenging compounds, such as formaldehyde, ammonia and small sulfur compounds, be analyzed without derivatization or preconcentration, but these analyses can be completed in seconds. Automating these analyses realizes the full potential of the fast SIFT-MS-based measurements for diverse applications.

Automation of traditional chromatographic techniques allows instrumentation to be productive 24/7, which is critical in many labs, since sample throughput is typically only 20–60 samples in a 24-hour period. Because the breadth of analysis is constrained by chemical reactivity, multiple columns or several techniques may be needed to comprehensively analyze just one sample. In addition, very dilute samples may require a preconcentration step.

SIFT-MS is a direct mass spectrometry technique¹ that delivers real-time, comprehensive gas and headspace analysis to ultratrace levels with a wide linear range.² It couples ultrasoft, precisely controlled chemical ionization with mass spectrometric detection to rapidly quantify VOCs and permanent gases to low-ppt concentrations by volume (pptv) (see *Figure 1*). Eight chemical ionization agents (reagent ions) are applied in SIFT-MS instruments: H_3O^+ , NO^+ , O_2^+ , O^- , O_2^- , OH^- , NO_2^- and NO_3^- . These react with VOCs and inorganic gases in well-controlled ion–molecule reactions, but do not react—or react only very slowly—with the major components of air (N_2 , O_2 and Ar). This enables SIFT-MS to analyze air at trace and ultratrace levels without preconcentration.

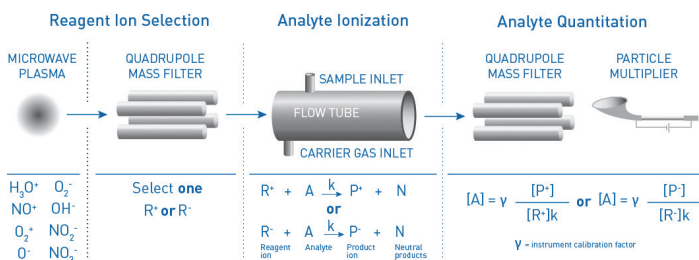


Figure 1 – Schematic representation of the SIFT-MS as applied in the Voice200ultra (Syft Technologies, Christchurch, New Zealand).

Rapid switching of the eight reagent ions provides higher selectivity than other direct mass spectrometry techniques. A comparative study found good correlation between SIFT-MS and GC/MS.³

Automated SIFT-MS

Autosampler integration is the simplest and most cost-effective way to leverage high sample throughput from the rapid, direct gas analysis provided by SIFT-MS. An autosampler improves repeatability and reproducibility compared to manual analysis and eliminates operator error.

Because SIFT-MS is a direct mass spectrometry technique (that is, it has no pre-separation using chromatography), its requirements for an auto-sampler differ from those commonly used with GC. GC-based techniques require rapid injection to achieve good chromatographic separation; chromatography leads to prolonged analysis as individual compounds elute from the column over time. However, SIFT-MS requires steady sample injection for the duration of the analysis, because analysis is carried out simultaneously with injection (*Figure 2*). The GERSTEL (Linthicum, Md.) Multipurpose Sampler (MPS) has proven to be the best-suited off-the-shelf autosampler system for SIFT-MS. Autosampler integration opens diverse applications of the technique for contract and R&D laboratories.

High-throughput static headspace analysis (SHA)

SIFT-MS offers very rapid, highly sensitive quantitation of VOCs and inorganic gases from headspace samples. Because chromatographic separation has been eliminated, SIFT-MS provides simple analysis of very volatile solvents as well as chromatographically challenging compounds, including ammonia, formaldehyde and hydrogen sulfide. *Figure 3* shows an example of rapid screening of three different polymers for residual monomers. Polymer pellet

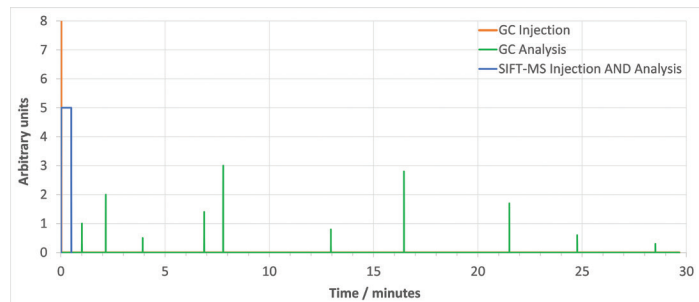


Figure 2 – Sample-injection and analysis requirements of GC-based techniques and SIFT-MS.

samples were analyzed in ground form. The polyoxymethylene (POM) sample is of particular note—formaldehyde is analyzed directly from this sample within seconds without any derivatization, preconcentration or other pretreatment. Analysis speed leads to very high throughput, providing rapid warning for quality issues; it also greatly reduces the cost per analysis. Applications of automated SIFT-MS headspace analysis include residual monomer or solvent analysis in pharmaceuticals and packaging, targeted analysis in foods and beverages and objective sensory screening.

Multiple headspace extraction (MHE)

MHE offers significant advantages over equilibrium SHA when working with solid materials and samples where composition of the matrix varies from sample to sample (e.g., in the analysis of soil samples). With solids, the rate of mass transfer within the material is very low. Equilibrium conditions take so long to achieve that classical static headspace is impractical. Because MHE does not depend on achieving equilibrium between the solid and the headspace, measurements can be made more quickly. Additionally, MHE allows the total VOCs contained within the solid to be calculated, not just the partitioned headspace concentration. This is illustrated using the example of polystyrene analysis for residual styrene monomer (Figure 4). SIFT-MS rapidly makes the required series of multiple headspace extractions, turning MHE into a practical, routine technique—the serious time penalty associated with slow GC or GC/MS analysis is eliminated. Applications of MHE-SIFT-MS include determination of residual solvents in packaging materials, analysis of thermally labile materials and quantitation of VOCs in soil samples.

Rapid analysis of sample bags and canisters

Comprehensive, direct gas analysis capabilities make SIFT-MS ideally suited to high-throughput analysis of industry-standard gas sampling devices, such as sample bags and canisters. No derivatization, drying or preconcentration is required for direct analysis to pptv levels. Further, reactive and labile compounds are readily detected and analyzed before they degrade.

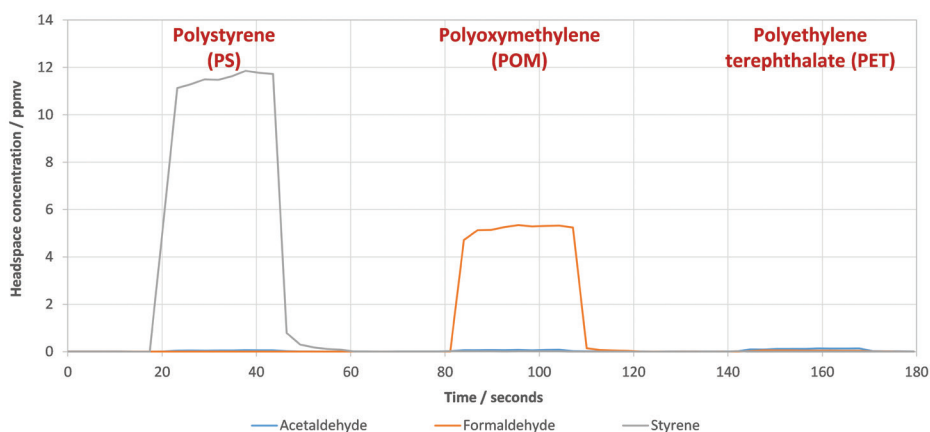


Figure 3 – Residual monomer analysis of different ground polymers, including formaldehyde from POM polymer.

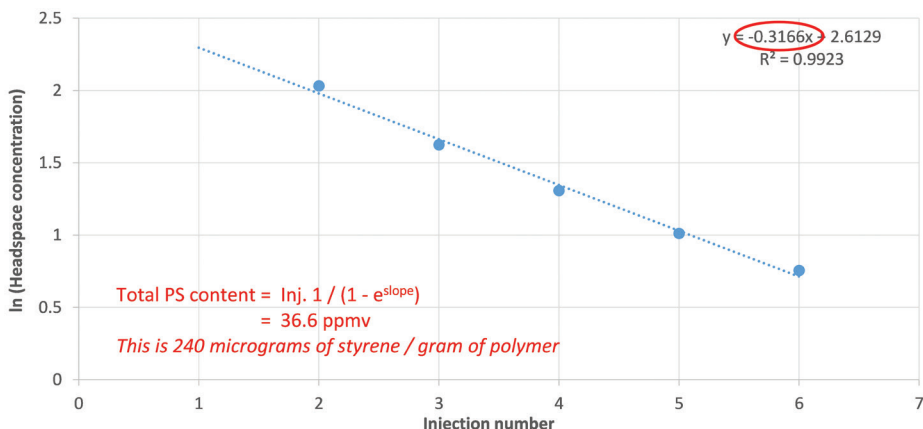


Figure 4 – Automated MHE using SIFT-MS: residual styrene monomer determination in polystyrene (PS) pellets.

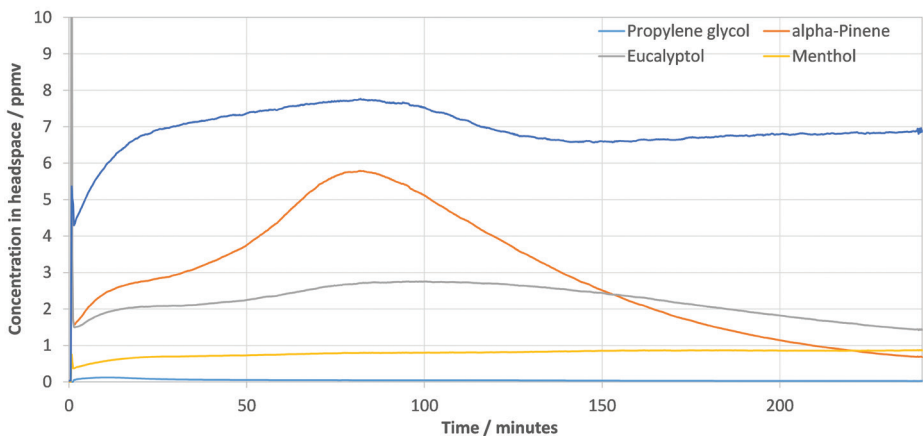


Figure 5 – Continuous analysis of fragrance components as they are slowly released from muscle ointment.

Rapid, automated gas analysis finds numerous applications. High-throughput sample bag analysis is applicable to the occupational safety and stack gas-testing industries. Fast canister analysis provides new opportunities for wide-scale ambient monitoring, indoor air-quality testing, process gas analysis and comprehensive odor analysis (including aldehydes, amines, organosulfurs and volatile fatty acids). Entire laboratory workflows can be restructured, since the fast analysis and wide linear and dynamic ranges of SIFT-MS mean that it can be applied to prescreening of incoming samples and evaluating the cleanliness of canisters.

Continuous headspace analysis (CHA)

Chromatographic techniques are highly impractical probes of dynamic processes, because they require that multiple instantaneous samples be taken, followed by prolonged analysis. As a direct analysis technique, SIFT-MS makes monitoring dynamic processes simple due to the high time resolution, high sensitivity and comprehensive analysis via multiple reagent ions. When coupled with automation, samples

can be run 24 hours/day, speeding up research and development or QA testing. This application is illustrated in *Figure 5*, where release of fragrance compounds from a topical muscle cream is monitored over a four-hour period. In this product, dramatic changes in fragrance composition occur over time. Using these insights, the product development process is enhanced through previously unavailable quantitative data. Applications of CHA include analyzing long-term fragrance or aroma changes, analyzing absorption of VOCs by “scrubbing” materials and monitoring the progress of reactions in chemical processes.

Conclusion

SIFT-MS provides highly sensitive, selective and nondiscriminatory analysis. When coupled with autosampler technology, it delivers unparalleled sample throughput.

References

1. Smith, D.; Spanel, P. Selected ion flow tube mass spectrometry (SIFT-MS) for on-line trace gas analysis. *Mass Spec. Rev.* **2005**, *24*, 661.
2. Prince, B.J.; Milligan, D.B. et al. Applications of selected ion flow tube mass spectrometry to real-time atmospheric monitoring. *Rapid Commun. Mass Spectrom.* **2010**, *24*, 343.
3. Langford, V.S.; Graves, I. et al. Rapid monitoring of volatile organic compounds: a comparison between gas chromatography/mass spectrometry and selected ion flow tube mass spectrometry. *Rapid Commun. Mass Spectrom.* **2014**, *28*, 10.

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