

Hyper-fast Gas Chromatography Coupled with Ion Mobility Spectrometry

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Abstract

Ion mobility spectrometers (IMS) are frequently coupled with gas chromatographs (GC), particularly when analyzing complex samples. However, analysis time remains a critical parameter in gas chromatography. Therefore, a hyper-fast GC represents a promising approach for GC-IMS, allowing for rapid two-dimensional separation in less than 60 s and highest sensitivity. To simultaneously detect both ion polarities, a new IMS with two drift tubes in an axial arrangement has been developed, specifically designed to resolve even hyper-fast GC peaks.

Keywords: Ion mobility spectrometry, hyperfast gas chromatography, process monitoring

Introduction

For several reasons, such as reducing chemical cross sensitivities caused e.g. by competing ionization processes and varying cluster size of reactant ions with humidity or adding a second dimension of separation, ion mobility spectrometers (IMS) are often coupled with gas chromatographs (GC). The combination of GC and IMS is used to tackle complex backgrounds, particularly when analyzing complex sample mixtures. However, this contradicts one major advantage of IMS, which is rapid detection of compounds present at ppbv and even pptv levels in less than one second measurement time, while already offering decent resolving power. Coupling IMS with GC usually extends measurement time to at least several minutes if not tens of minutes. Therefore, a hyper-fast GC is a very promising approach for GC-IMS to combine highest sensitivity of IMS with two dimensional separation while allowing for rapid analysis within the double-digit second range [1].

Instrumentation and Results

This study presents a novel drift tube IMS, specifically designed to resolve even hyper-fast GC peaks [2]. Key is the directed sample gas flow through the ionization region, resulting in a small effective detector volume matching the volume of peaks eluting from the hyper-fast GC, so that the GC peaks do not merge in the IMS reaction region [3]. Figure 1 shows an

exemplary measurement containing a mixture of 4-ketones within a short measurement time of only 30 seconds.

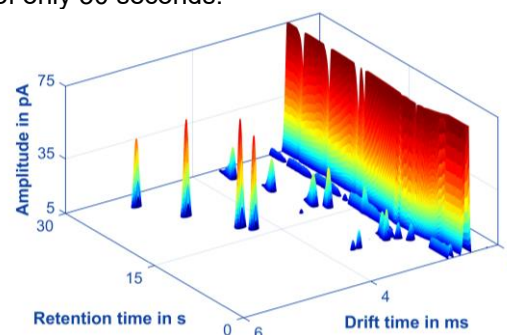


Fig. 1: 3D-GC-IMS chromatogram of an exemplary ketone mix containing 4-heptanone, 2-heptanone, 2-octanone and 2-nonanone.

To minimize condensation of analytes, the IMS is isothermally heated to 120 °C. A drift tube length of only $L_D = 41$ mm with a drift voltage of $U_D = 2.7$ kV leads to high resolving power of $R_p = 60$ and an IMS repetition rate of 100 Hz, even for ions with low reduced ion mobility down to $0.43 \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}$. This setup can resolve very short GC peaks with full width at half maximum of only 140 ms. It is demonstrated that this system enables the rapid analysis of complex mixtures, such as those composed of hops or chemical warfare agents, within a time frame of 60 seconds.

References

- [1] A. Nitschke, M. Hitzemann, J. Winkelholz, A.T. Kirk, M. Lippmann, C. Thoben, J.A. Wittwer, S. Zimmermann, *J. Chromatogr. A* 1736, 465376 (2024); doi: 10.1016/j.chroma.2024.465376.
- [2] A. Nitschke, M. Hitzemann, J. Winkelholz, A.T. Kirk, C. Schaefer, T. Kobelt, C. Thoben, M. Lippmann, J.A. Wittwer, S. Zimmermann, *J. Chromatogr. A* 1750, 465923 (2025); doi: 10.1016/j.chroma.2025.465923.
- [3] V. Maynard, E. Grushka, *Anal. Chem.* 44, 1427–1434 (1972); doi: 10.1021/ac60316a005.