Coupling of Ion Mobility Spectrometry as Detector to a Micro Gas Chromatograph for Enhanced Detection Limits and Applicability

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Introduction

Fast availability of results and low detection limits are key drivers for analysts nowadays. This paper shows a feasibility study for coupling ion mobility spectroscopy (IMS) as detector to a micro gas chromatograph (Micro GC) to achieve both fast run times, and ppb level detection..

Agilent 490 Micro GC.

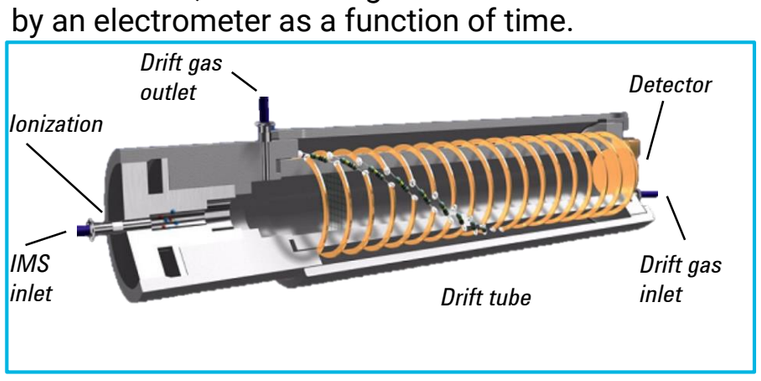
Micro gas chromatographs typically use a microelectro-mechanical (MEMS) version of a thermal conductivity detector (TCD) for compound speciation, achieving detection limit down to low ppm levels. Since most compounds have a thermal conductivity different from the GC carrier gas, the TCD responds to all these compounds and is therefore often entitled as universal detector. .

Ion mobility spectrometry is more sensitive and selective, it can detect gaseous compounds down to 50 – 100 ppb and below. When coupled to a micro gc, the pre separation of compounds significantly improves selectivity.

The advantage of using Micro GC is its fast runtime, typically 30 to 90 seconds, as a result of using MEMS based injector and detector, and isothermally operated narrow bore capillary columns.

Benefits of IMS

The detection principle of Ion Mobility Spectrometry is based on the specific drift times that ions of compounds have in a defined electric field passing a fixed path length drift tube with reversed flow drift gas. Atmospheric ionization of molecules is obtained by soft chemical-ionization initiated by a tritium (3H) source with radiation below exemption limits for all EURATOM countries. For detection, the resulting ion current is measured by an electrometer as a function of time.



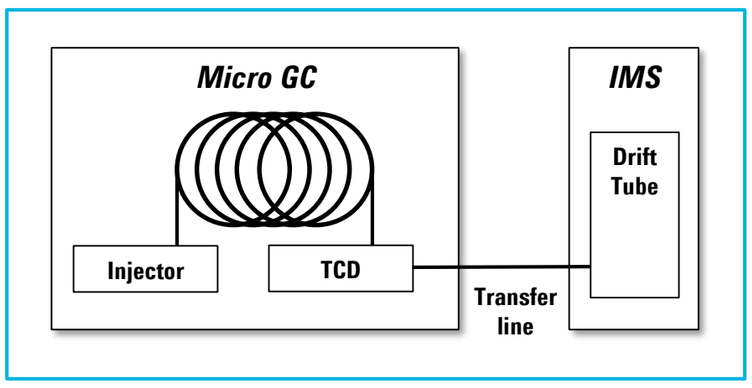
G.A.S. ion mobility spectrometer

The enhanced sensitivity and selectivity by combining Micro GC with IMS allows detection of the smallest impurities of volatile compounds, even in complex mixtures. As an example the analysis of low sulfur in energy rich streams will benefit from a more sensitive and specific detector like the Ion Mobility Spectrometer.

Coupling Micro GC with IMS

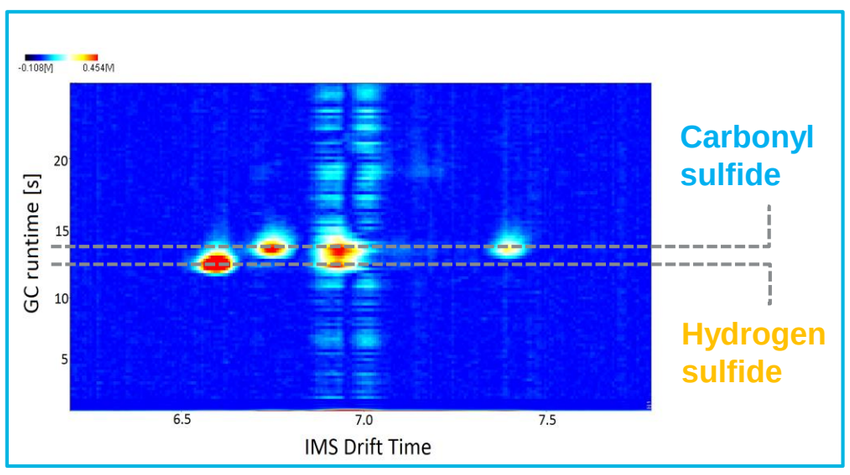
Instrument and sample information

- 490 Micro GC [Agilent Technologies] equipped with 8 meter CP-Sil 5 CB column channel at 40 °C; 200 kpa nitrogen carrier gas; 400 ms injection time; inert and heated transfer line from µ-TCD to IMS at 65 °C.
- Ion Mobility Spectrometer [G.A.S, Dortmund] with 980 mm drift tube; voltage drop 500 V/cm; temperature 45°C; 150 ml/min synthetic air as drift gas.
- Direct connection of the Micro GC to the Ion Mobility Spectrometer is possible as a thermal conductivity detector is non-destructive. The outlet of the TCD is connected to the drift tube using a heated, inert 0.5 mm ID transfer line to prevent active spots and lower dead volume.
- Hydrogen sulfide and carbonyl sulfide in nitrogen, 1 ppm each [Linde Gas]. Futher diluted in nitrogen to required concentrations



Schematics for Micro GC – IMS coupling

Results and Discussion



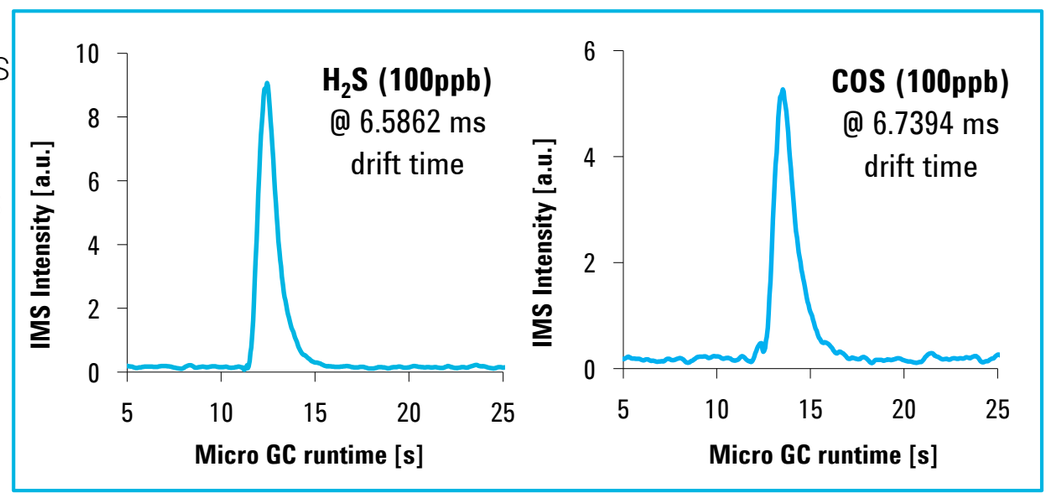
IMS Spectrum of hydrogen sulfide and carbonyl sulfide mixture. (100 ppb each)

Typical limit of detection for hydrogen sulfide and carbonyl sulfide on the TCD (Micro GC) is approximately 2 ppm. The dilutions used for this experiment were below 1 ppm and therefor not seen in the TCD. The IMS, coupled to the Micro GC, can however easily detect these concentrations.

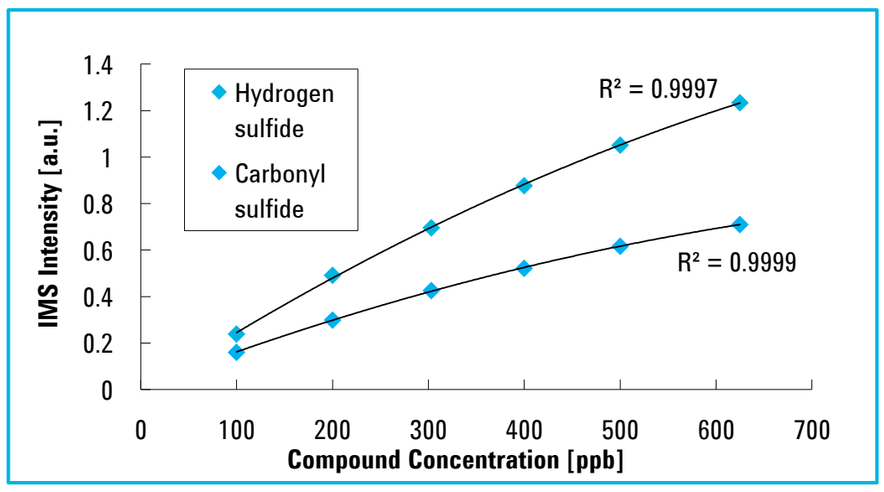
An IMS spectrum for a hydrogen sulfide and carbonyl sulfide mixture (100 ppb each) is given in left picture. This spectrum shows that the compounds of interest can be detected separately, although pre-separation on CP-Sil 5 CB not optimal. Next optimization steps includes testing other column phases such as PLOT U or BOND Q.

A second dimension separation is obtained by filtering at a selected IMS drift time. The chromatograms right are showing the integrated ion current plots. The signal at defined IMS drift time, for each H2S and COS is plotted and integrated indepen-dently. Additional Savitzky-Golay filtering is applied to reduce signal noise.

These chromatograms clearly show the possibility of detecting H2S and COS at < 100 ppb concentrations.



Integrated ion current plot – IMS data filtered on specific drift time.



The calibration curve depicted left shows excellent linearity (polynomic fit) for a 100 – 600 ppb concentration range. IMS typically have 2-3 decades dynamic range, additional experiments should confirm this expectation.

Combining Micro GC with Ion Mobility Spectrometry could ultimately be used for application were both matrix and low level impurities needs to be analyzed in a single run. For example natural gas or biogas analysis were composition is characterized using the thermal conductivity detector and sulfur impurities are analyzed on the IMS simultaneously.

Conclusions

- The results depicted in this poster proofs the concept of coupling an Agilent 490 Micro GC with a G.A.S. IMS coupling.
- Detection limits of lower than 100 ppb can be reached for hydrogen sulfide and carbonyl sulfide.
- Future optimization includes testing of other column types, such as Plot U or Bond Q, for better pre-separation, and confirmation of dynamic range and detection limits.
- Ultimately simultaneous analysis of natural gas composition and low sulfur detection can be obtained by coupling Micro GC and IMS.

References

For more details about the Agilent 490 Micro GC
www.agilent.com/microgc
For more details about the G.A.S. Ion Mobility Spectrometer visit
http://www.gas-dortmund.de.