GAS

SUB-PPM ANALYSIS OF SULFUR COMPOUNDS IN NATURAL GAS USING GAS CHROMATOGRAPHY – ION MOBILITY SPECTROMETRY

SUMMARY

A method for quantification of traces of sulfur compounds in natural gas was developed. H_2S, COS, MeSH and EtSH were detected down to a limit of <10 ppb. Calibration curves from 0 to 1000 ppb were generated for all mentioned compounds.

EXPERIMENTAL

A certified test gas of 1 ppm hydrogen sulphide (H_2S), carbonyl sulphide (COS), methyl mercaptan (MeSH) and ethyl mercaptan (EtSH) in methane was used for method development and calibration. Concentrations between 25 and 1000 ppb were prepared using a set of mass flow controllers and measured using a G.A.S. GC-IMS device.

All compounds are baseline separated. For this purpose two separate GC programs are used, which are running sequentially. One program detects H_2S and COS and the other program is optimized for the detection of MeSH and EtSH. The overall cycle time for detection of all 4 compounds is <10 min and can be lowered depending on the requested concentration range and matrix complexity.

RESULTS

The chromatograms of the baseline separated signals of H_2S and COS (Figure 1) and MeSH and EtSH (Figure 2) at 200 ppb.
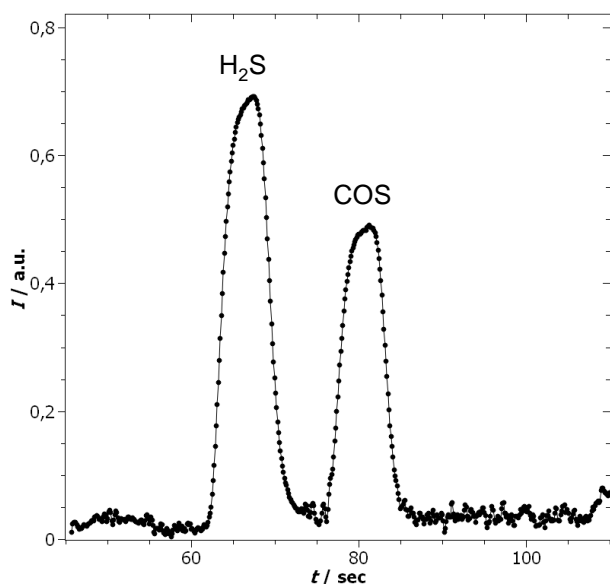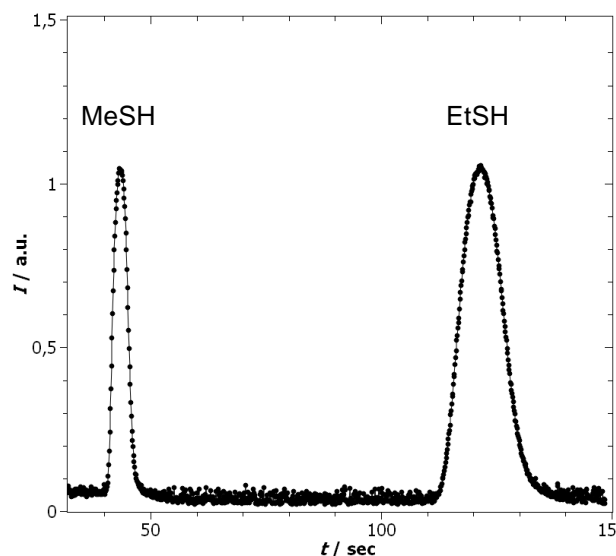

Figure 2: Chromatogram of MeSH and EtSH.

Figure 1: Chromatogram of H_2S and COS.

Figure 3 shows the calibration data of H_2S and COS and figure 4 MeSH and EtSH in the range of 0 to 1000 ppb. Region exhibiting steep slope can be shifted to higher concentration ranges (depending on customers requirements).
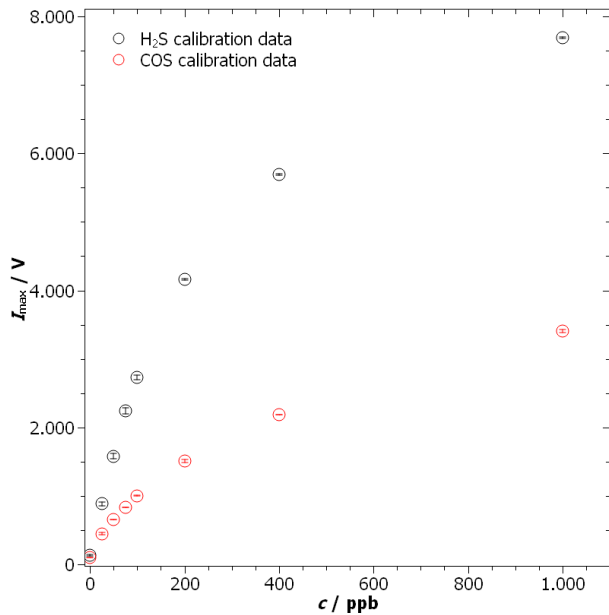

Figure 3: Calibration data of H_2S and COS in the concentration range from 0 to 1000 ppb. Standard deviations are marked (n = 3).
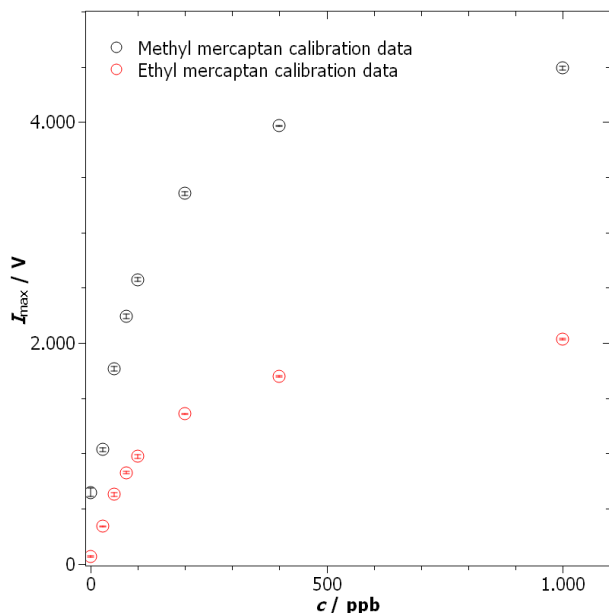

Figure 4: Calibration data of MeSH and EtSH in the concentration range from 0 to 1000 ppb. Standard deviations are marked (n = 3).

REPEATABILITY & LIMIT OF DETECTION

Analyte	Standard deviation*1/ LOD*2
H_2S	3.5 % / 5 ppb
COS	2.1 % / 4 ppb
MeSH	2.5 % / 10 ppb
EtSH	2.8 % / 8 ppb

*1 Standard deviation of signal intensity at a concentration of 50 ppb (n = 50).
*2 Depending on measurement set-up and matrix.
Calculation based on $I_{LOD} = I_{Blank} + 3\sigma_{Blank}$

CONCLUSION

The GC-IMS combines the high selectivity of a gas chromatographic (GC) separation with the extraordinary sensitivity (low ppb_v or µg/L range) of an ion mobility spectrometer (IMS). Therefore it is an excellent analytical tool, even for measurements of low sulfur compounds in complex matrices.

HIGHLIGHTS

› Selective and sensitive quantification of H_2S, COS, MeSH and EtSH
› Limit of detection below 10 ppb for each substance

SPECIFICATIONS

Measuring technique	2-dimensional separation by GC-IMS technology
Run time (Detection H_2S & COS)	90 sec
Run time (Detection MeSH & EtSH)	135 sec
Data transfer	Current loop or Modbus