Development of a flexible GC transfer line for a field-deployable GC-EI/MS

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Introduction

The combination of gas chromatography (GC) and mass spectrometry (MS) is still of great importance in various fields of application. Essential for the overall performance is a careful connection between these two instruments. In most setups this is satisfactorily provided by solid, rigid and well insulated heated metal tubes. However, several applications such as in atmospheric science demand a more flexible and also field deployable transfer design that allows for rapid mounting and provides a reproducible performance in laboratory, smog chamber and field experiments.

This work focuses on the development of a flexible transfer line for a GC-EI/ time-of-flight (TOF) mass spectrometer with the following requirements:

- Rapid assembling/disassembling of the two instruments for shipping without venting of the system or misalignment of the GC-column(s)
- Maintained vacuum with removed transfer line
- High mechanical flexibility to support easy installation
- Performance in terms of resolution and sensitivity comparable to conventional transfer line designs
- Prevention of cold/hot spots and dead volumes

Methods

Instruments

- HTOF (TOFWERK Switzerland)
- Agilent Technologies 7890A GC system
- DB-5 30m x 0.250 mm x 0.25 µm (Agilent Technologies)
- custom capillary feedthrough at 280 °C
- Heated horse for Transfer at 280 °C (Hillesheim GmbH)

Ion source

- Star-Beam EI source (TOFWERK Switzerland)
- 1 mA emission current, 70 eV

Chemicals

- OFN (Supelco), FAMEs (Supelco), Helium (Carbagas), Hexane (Sigma Aldrich)

GC parameters

- 1.31 ml/min (constant flow)
- 35 °C (2min) to 250 °C at 10 °C/min and at 20 °C/min up to 300 °C (5 min)

Experimental Setup

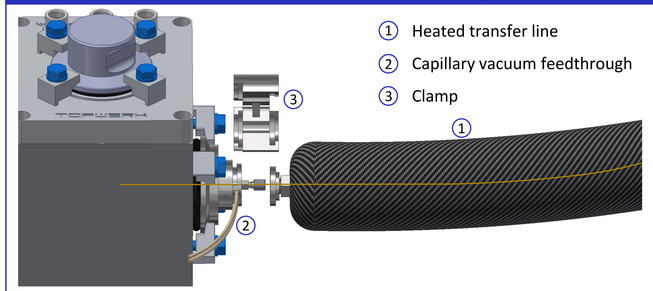

Figure 1: Schematic drawing of the transfer setup consisting of a capillary vacuum feedthrough into the EI-source (left) and a heated transfer line (right)

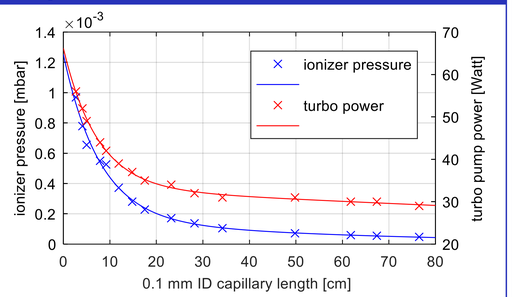

Figure 2: Ionizer pressure and turbo pump power as a function of the flow restricting capillary length (0.1 mm ID)

- Custom built and separately heated capillary feedthrough interfacing the EI source on the left and the heated transfer line on the right.
- MS sided vacuum sealing makes the desired rapid assembling and disassembling of the chromatographic system feasible.
- An integrated zero dead volume GC union (VICI Valco) in combination with a custom designed clamp provide a hand tight connection of the heated transfer line and the capillary feedthrough without cold spots on the column surface.
- A quite tolerable pressure in the EI ion source and MS, preventing the vacuum system from overheating, can be achieved with a surprisingly short piece of 0.1 mm ID capillary.
- In the presented setup an approximately 8 cm long capillary sustains an ionizer pressure of $4x10^{-4}$ mbar with ambient pressure at the inlet. This condition is readily managed by the supplied turbo pump.

Ambient Sampling

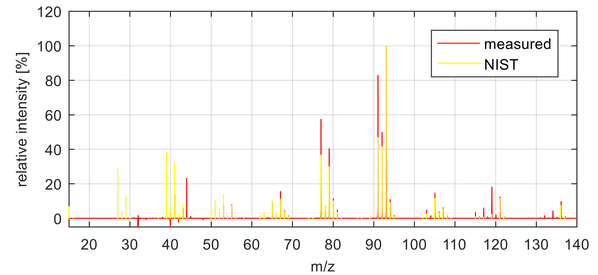

Figure 3: Background subtracted electron ionization mass spectrum of α-Pinene

- The simple and fast disconnection of the transfer line and the entire GC assembly with sustained MS operation readily offers EI spectra acquisition of ambient samples as it is mainly done by atmospheric pressure interface ion sources.
- Vacuum is maintained by the capillary feedthrough.
- Spectrum of pure α-Pinene hold in front of the Inlet.
- An unambiguous and comparable α-pinene spectrum was observed after background subtraction.

Performance

Sensitivity

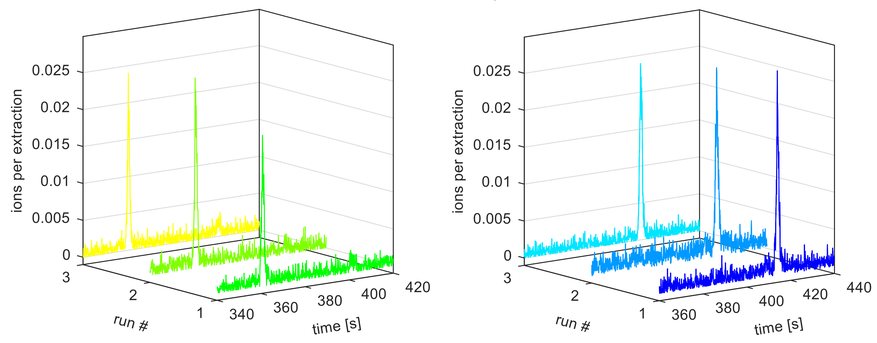

Figure 4: Chromatograms for 1 pg octafluoronaphthalene (OFN) on column. **Left:** conventional setup, **right:** flexible setup

- Evaluation of the performance of the presented field deployable GC transfer line due to comparison with a conventional design.
- A negative impact on separation and sensitivity can occur by e.g. cold spots, wider peak shapes, dead volumes, interaction with the metal surface of the union and a wrong positioning of the capillary inside the ion source.
- As shown in figure 4 “signal to noise ratios” for both setups do not differ significantly for several repetitions of these experiment, resulting in a virtually identical sensitivity.

Chromatographic Performance

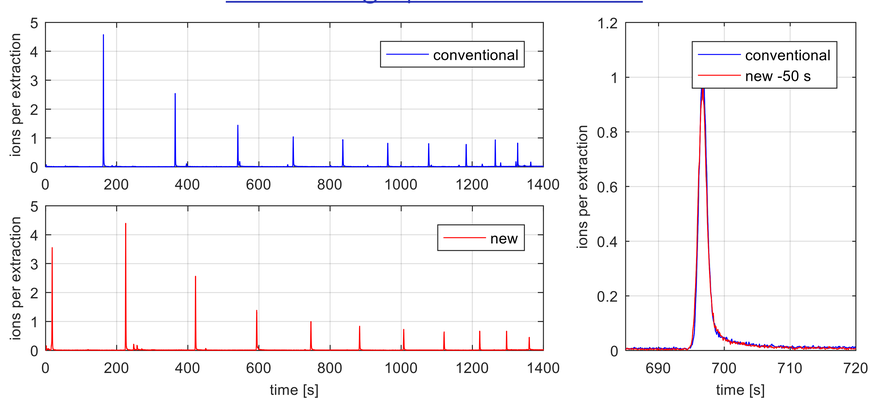

Figure 5: Comparison of chromatographic resolution between both tested setups. **Left:** complete FAMEs chromatogram, **right:** signal of methyl laurate

- Except the obvious shift in the retention time, the chromatographic performance is not affected by the modified transfer line and MS adapter design.
- The chromatogram in figure 5 shows no peak broadening, which indicates an adequate avoidance of cold spots and dead volumes.
- Reproducible chromatogram over the entire temperature range.

Conclusion

The presented flexible transfer line design allows for easy and fast assembling and disassembling of the entire chromatographic stage while maintaining full MS operation. These feature makes this design more field-deployable and adaptable to changing environmental and experimental conditions compared to conventional designs.

- Easy preparation for transport
- Fast and reproducible reinstallation
- Less contamination

Virtually identical performance in terms of sensitivity and chromatographic resolution compared to a conventional setup was obtained.

Outlook

The presented heated tube used as transfer line is still limited in its bending radius to provide the eventual flexibility for our applications and to simplify the handling considerably.

Future work will concentrate on suitable materials with higher flexibility. Those need to withstand up to 350 °C, provide sufficient heat conductions for an even temperature distribution and offer a smaller bending radius.

Literature

[1] NIST Mass Spectrometry Data Center, William E. Wallace, director,“Mass spectra“ in **NIST Chemisty WebBook, NIST Standard Reference Database Number 69**, Eds. P.J. Linstrom and W.G. Mallard (retrieved May 21, 2019).

Acknowledgement

cloud-motion
www.cloud-motion.eu

This Project has received funding from the European Union’s Horizon 2020 research and innovation programme under the Marie Skłodowska-Curie grant agreement No 764991.