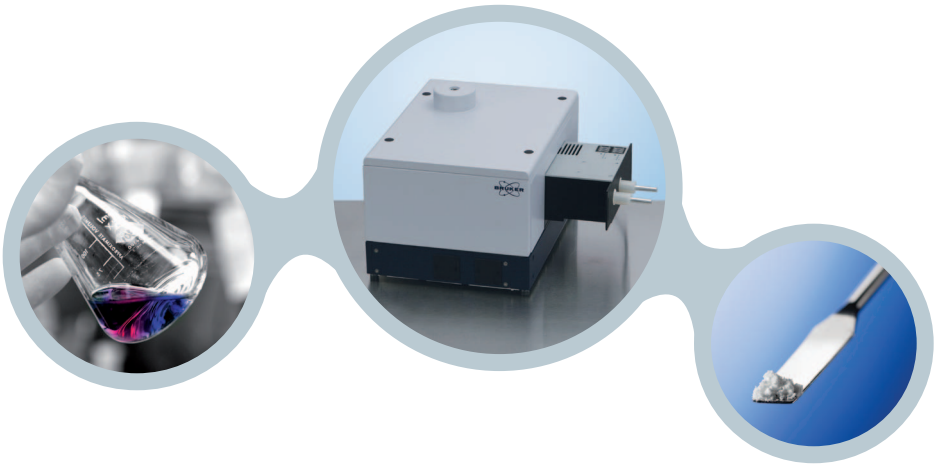


Application Note # AN-76

GC - FT-IR in the ng range

Introduction

Gas chromatography is widely used technique in the field of chemical analysis. It is one of the most powerful separation techniques. The separation in time between the different compounds and the starting point (retention time) is specific to a particular substance. Correlation of the peaks with the constituents of a mixture is not always immediately possible, especially when the constituents are unknown; also, the retention time depends on instrument parameters. What the analyst requires is a method to enable simple and accurate identification of the chromatogram peaks with the constituents of the separated mixture.

Picture 1 GC-IR accessory placed in between the GC and the FT-IR

Measurement conditions

- Capillary Column: 30m, 0.25µm film
- Temperatureramp: 120°C – 160°C
- Injection ratio: 50/1 split
- Injection quantity 0.05µl (2% IBMA in methyl-enechloride)
- Light pipe type: AABSPEC IX 10, 60mm optical path

Experimental

A 2% solution of IBMA in CH_2Cl_2 was prepared and 0.05µl injected manual by syringe. The injection ratio was set to 50/1. Nitrogen was used as carrier gas, flow rate 1ml/min. The injection temperature was set to 200°C, the GC was ramped from 120°C to 160°C by 10°C /min.

Conclusion

The IBMA (Isobutyl-Methacrylate) is generally used as a sensitivity test for GC/FT-IR. The data shown here demonstrates the excellent performance and sensitivity now avail-

able from a high performance FT-IR instrument in combination with the appropriate light pipe. The short (only 60mm) path used in the light pipe here allows operation without diluting eluents with "make-up" gas, commonly done by use of long pathlength light pipes to avoid spectral overflow of separated eluents. The highly concentrated eluent over-compensates for any possible intensity gain given by longer light-pipes. The low volumes light-pipe is therefore better matched to fast eluting capillary GC peaks.

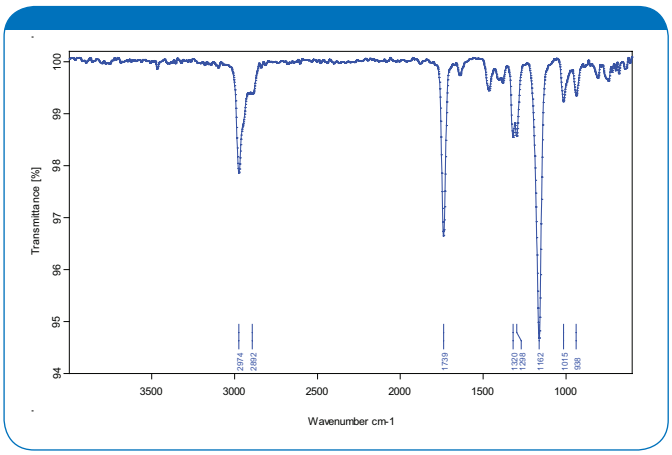
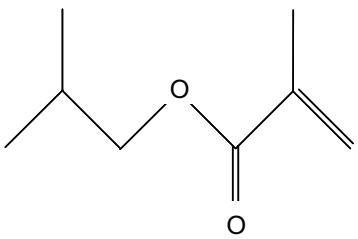


Fig. 1 Spectrum of approximately 20ng of IBMA (Isobutyl-Methacrylate). Signal to noise >50/1.

In combination with powerful FT-IR Instruments low noise level data can be achieved (S/N >1000/1) within sampling times of less then 1 second. Such data rates are needed to follow the eluents separated by GC using high resolution capillary columns. The system provides the user with a choice of capillary column types, all as part of a highly inert, integrated system.

Appendix

Isobutyl methacrylate (IBMA)
CAS No. 97-86-9
Formula: $C_8H_{14}O_2$
Molecular weight: 142.19796
Boiling point: 155°C
Melting point: -37°C
Density 0.89kg/l



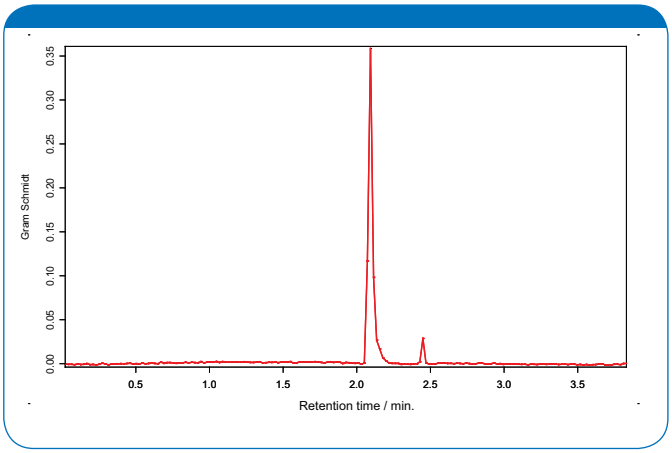


Fig. 2 Gram-Schmidt-Chromatogram for the separation of the IBMA from methylenechlorid solvent

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