

Application Note 120717
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Mass Spectrometric analysis of polar and/or labile compounds by Desorption Electron Impact Ionization (DEI)

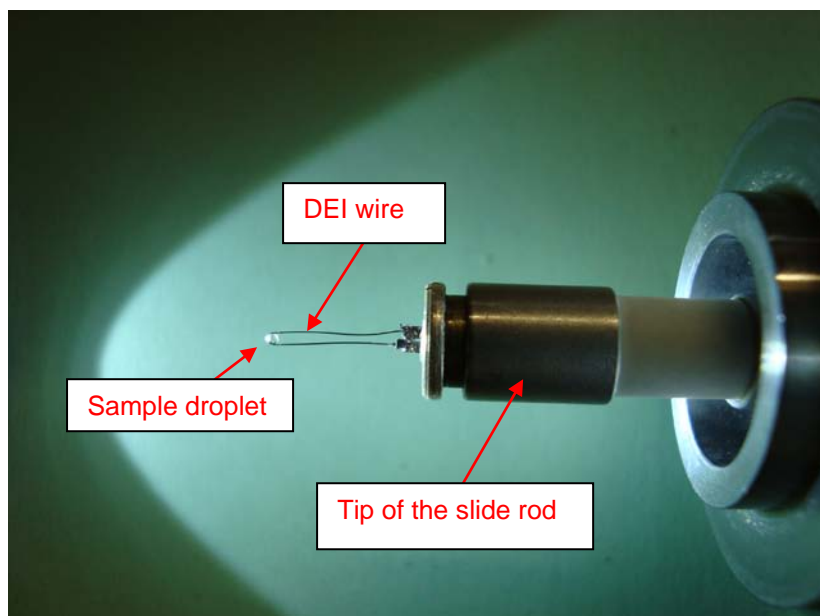
A) Introduction

A number of compounds do not pass a GC column without degradation and can't be analyzed on-line by GC/MS. A supplementary method for fast off-line mass spectrometric analysis of polar and/or thermally labile compounds, especially samples in aqueous solution, offers analytical opportunities. The analysis of such samples by Desorption Electron Impact Ionization (DEI) without chromatographic separation but based on different desorption functions turned out to be a useful tool. In the following an excerpt of a previous publication (Odour Control, Hamburger Berichte 36) describes some orientating experiments and results using odorous model substances using an **AMD QuAS³AR** mass spectrometer.

B) Experiments

B1) The DEI method

A certain amount of the solution to be analyzed is deposited by a syringe on a heatable wire on top of a sample introduction device.



The solvent of the sample droplet is evaporating under atmospheric pressure.

The dried sample is introduced into the EI ion source via a slide rod through a vacuum lock.

The DEI wire is heated by a programmable power supply.

The sample desorption takes place during the application of an increasing current through the wire, resulting in a temperature increase, accordingly.

Fig. 1 Sample solution droplet deposited on the **DEI** wire outside of the **AMD QuAS³AR** mass spectrometer.

Some experiments have been performed, using **Sotolon** considered to be a relevant odorous model substance for demonstration of the **DEI** methodology.

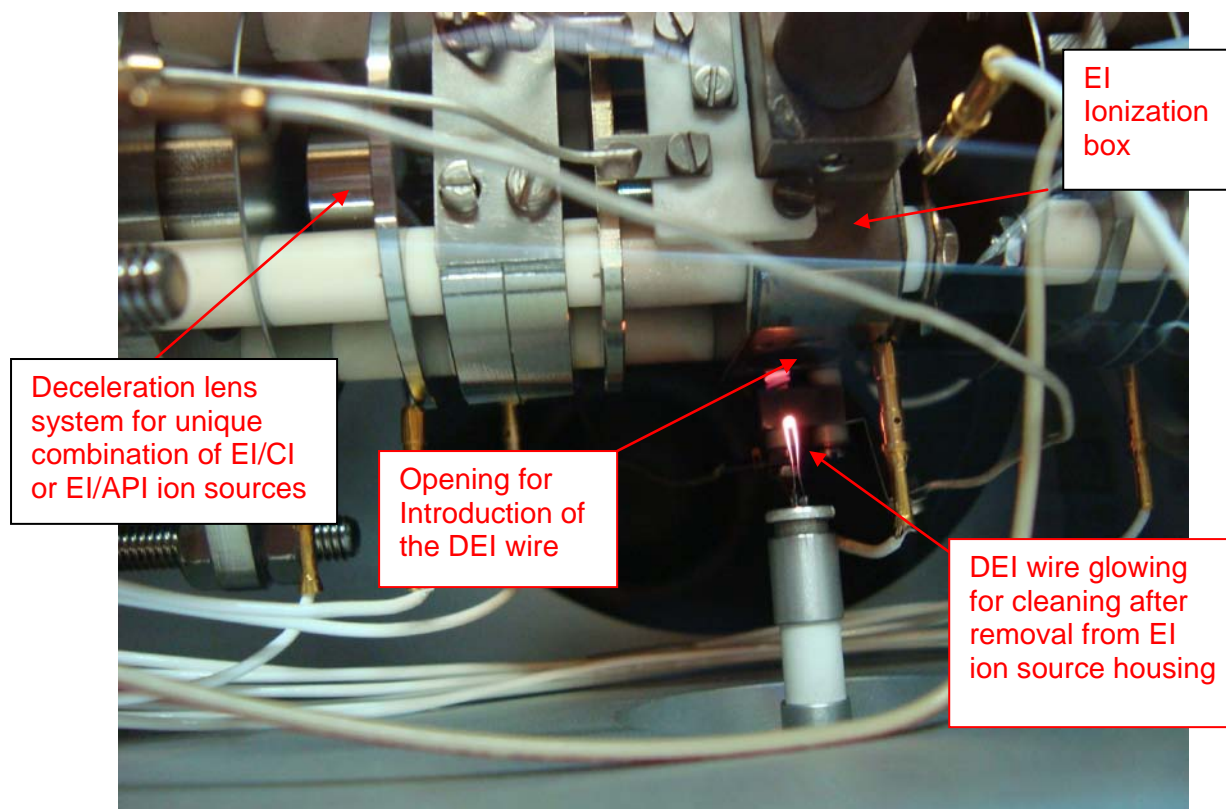


Fig. 2 This figure shows the EI ion source of the **AMD QuAS³AR** mass spectrometer for DEI analyses in a configuration for a combination of EI/CI or EI/API ion sources in series. The DEI tungsten wire has been removed (via slide rod) here from the EI ion source housing after the measurement and is heated up for cleaning at about 1000 °C.



B2) Results

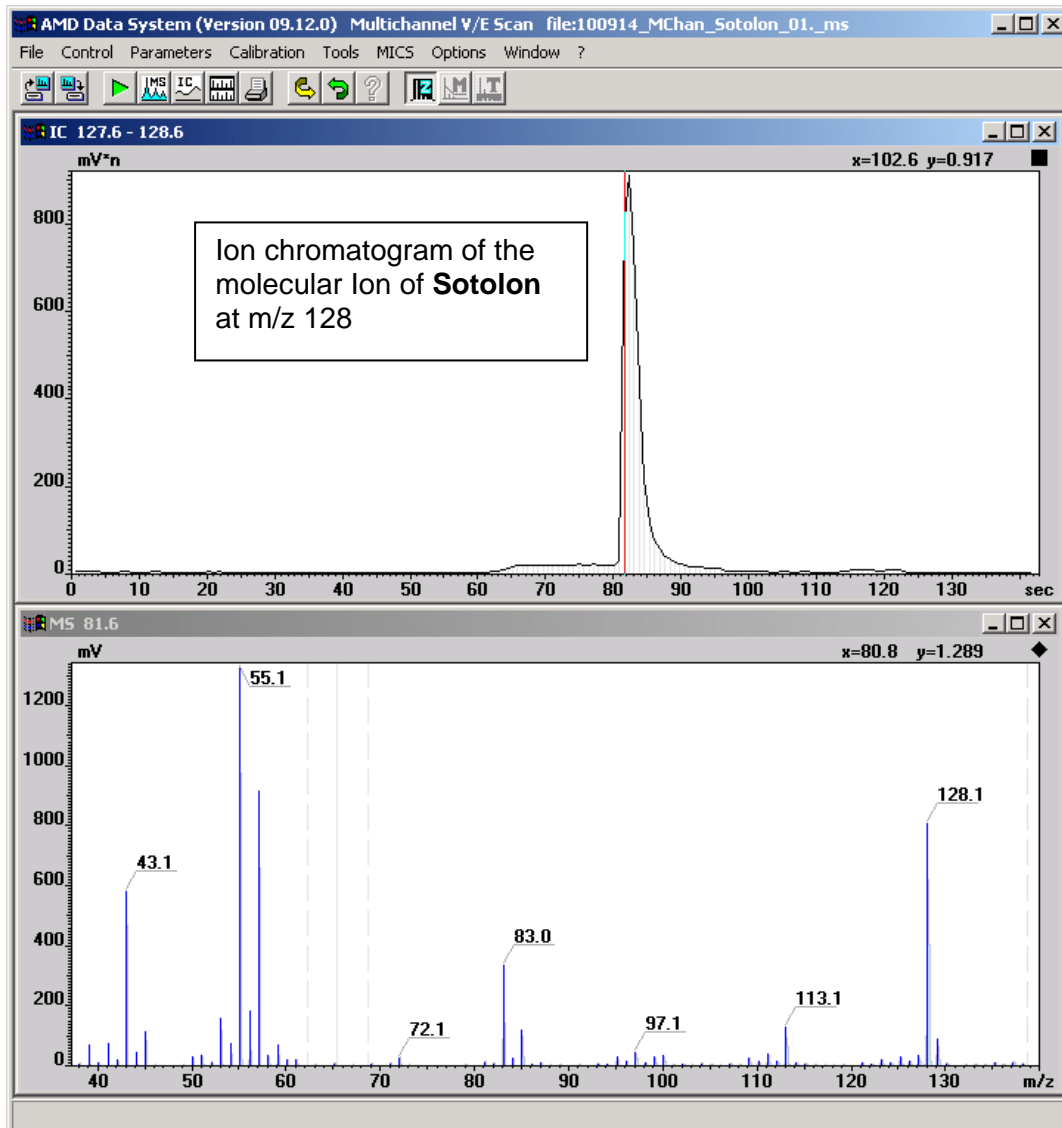


Fig. 3 Ion chromatogram of the molecular ion of **Sotolon** and the corresponding mass spectrum.

The ion chromatogram represents the desorption function which depends on the DEI wire heating program. A rapid current and corresponding temperature increase has been used for a narrow peak in the chromatogram. A sample concentration of **1.0 µg/µL** in MeOH solution has been used. The dynamic range of the mass spectrum indicates that even lower concentrations will yield mass spectra suitable for substance identification

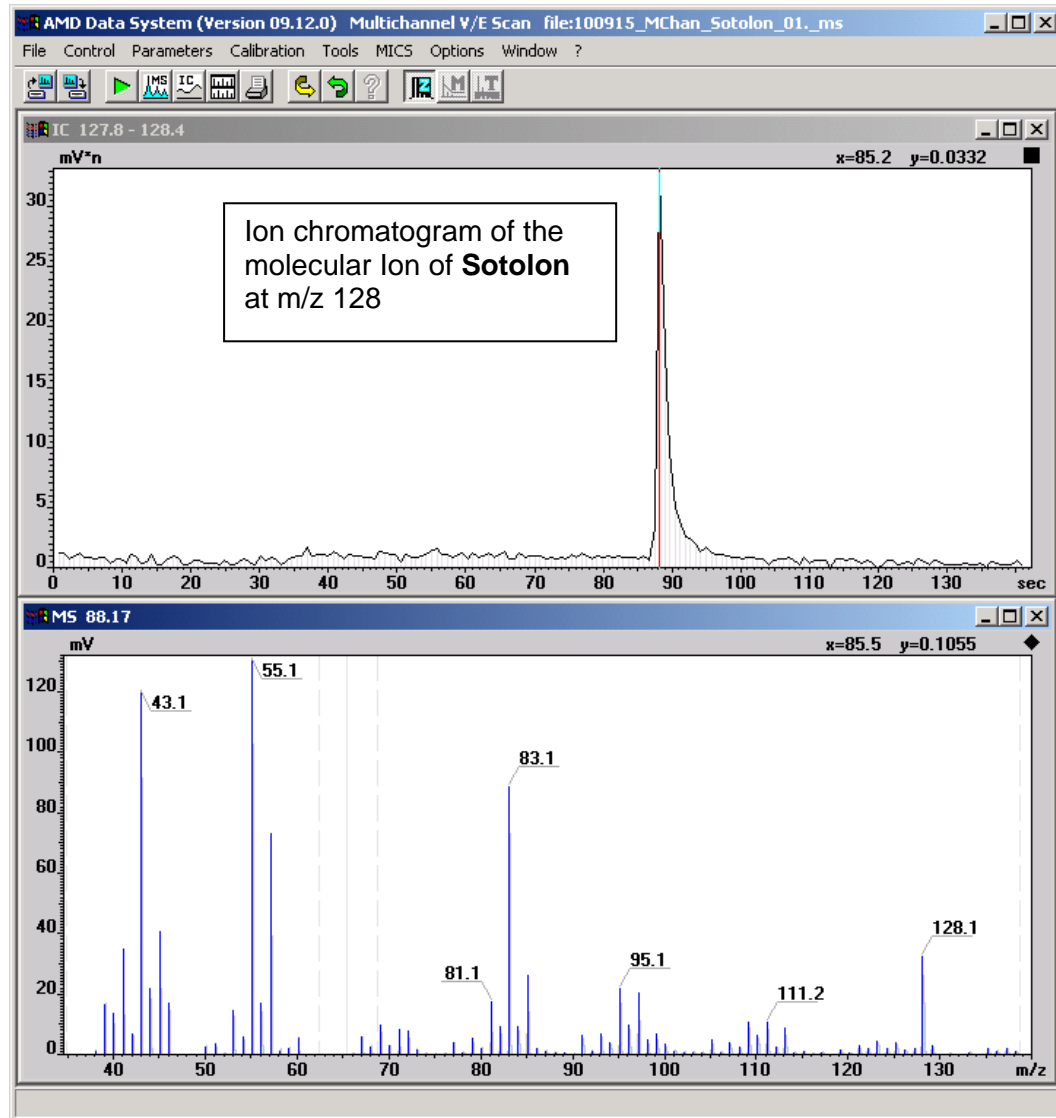


Fig. 4 Ion Chromatogram and mass spectrum at further reduced sample concentration **100 ng/ μ L Sotolon** in an **aqueous solution**. The ion chromatogram shows the same peak shape while the “quasi retention time” is different due to different starting points of the desorption function.

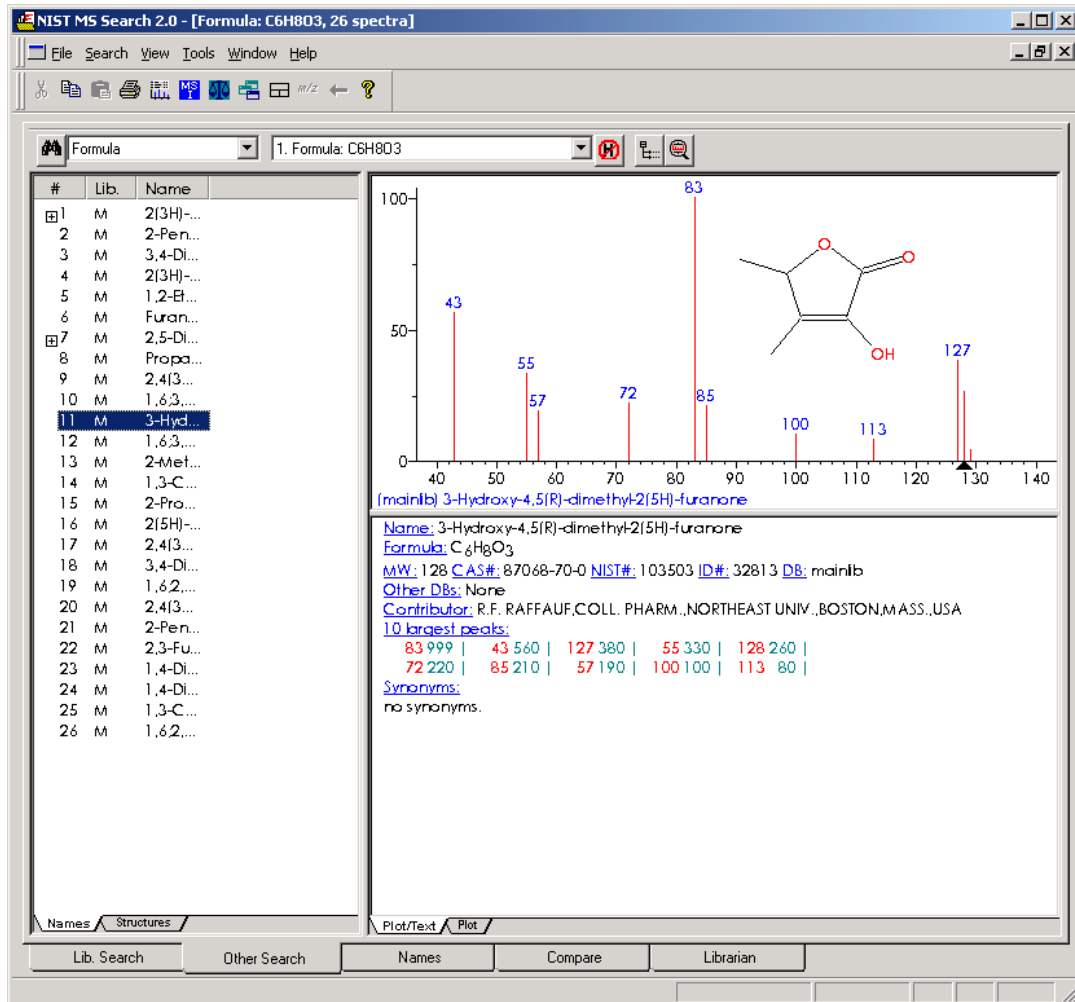


Fig. 5 The **Sotolon** mass spectrum available in the library yields a significant loss of a Hydrogen atom from the molecular ion while the DEI mass spectra above (Fig. 3,4) show a strong molecular ion and all relevant key fragment ions in accord with the library spectrum.

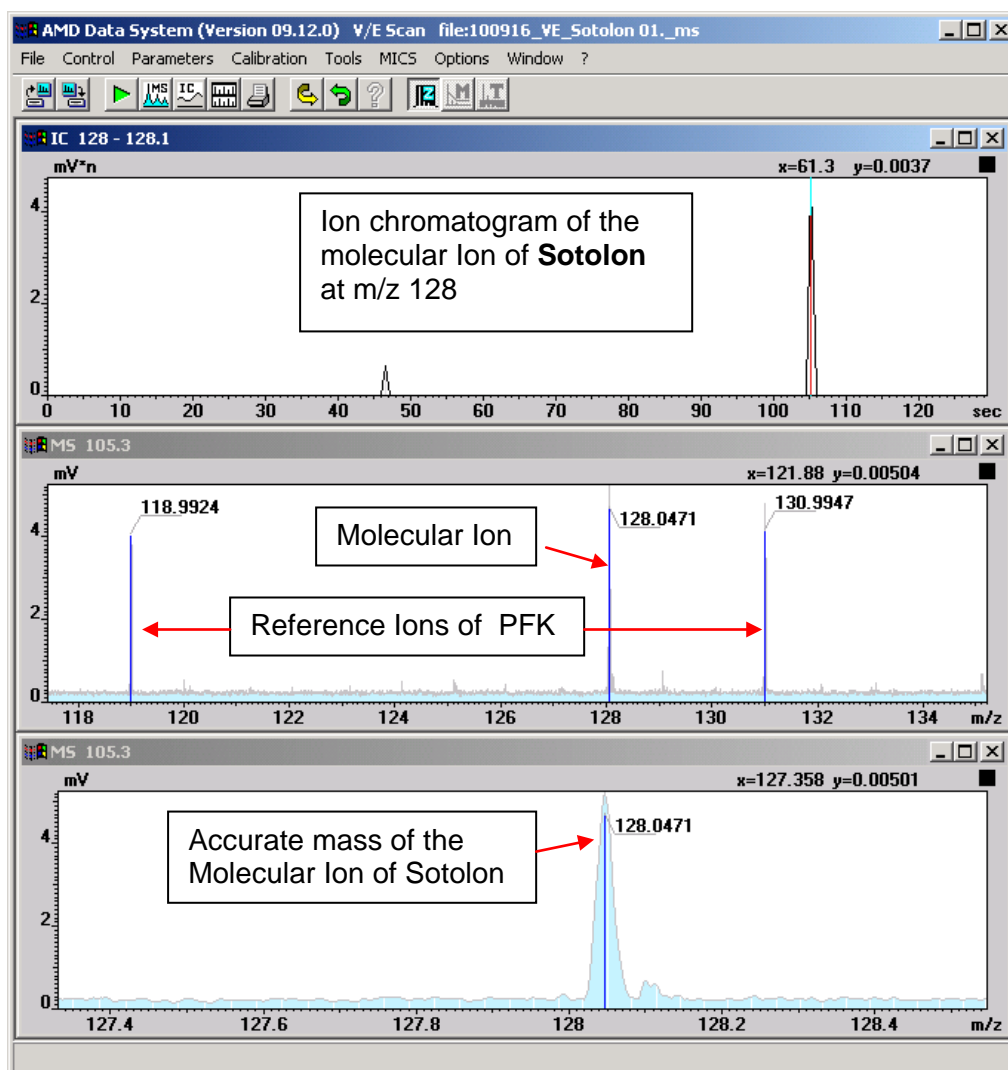


Fig. 6 Ion Chromatogram and mass spectrum at higher mass resolution of 1 $\mu\text{g}/\mu\text{L}$ Sotolon in an aqueous solution. The DEI mass spectra have been recorded at a mass resolution of about 2.500 (10% valley). The accurate mass of the molecular ion has been determined with a deviation of 0.2 mmu or 1.6 ppm (theoretical mass: 128.0473)

Above example of an accurate mass determination demonstrates another opportunity for substance identification using the DEI methodology.

It can be concluded that **DEI methodology** using continuous increasing temperature of the sample carrier (solvent should have been eliminated by evaporation under atmospheric pressure before analysis) and applied to compound analysis results in individual desorption functions, represented by ion chromatograms. The peak maxima on the time scale (quasi retention time) and the peak shapes of the chromatograms may be used for identification of the individual compounds in a mixture

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