Using Solid Phase Microextraction with AccuTOF-DART™ for Fragrance Analysis

Introduction

Solid phase microextraction (SPME) is a well established sampling technique that is often used to isolate volatile organic components in gaseous mixtures. Once the compounds have been collected, the SPME fibers are typically placed into a heated GC inlet which thermally desorbs these components into a GC-MS system for analysis. Normally, this analysis can take between 10 and 30 minutes to complete depending on the complexity of the samples. In this work, the Direct Analysis in Real Time (DART™) heated gas stream is used to desorb and directly introduce a SPME sample into a high-resolution mass spectrometer. This methodology produces comparable information to the traditional GC-MS technique but streamlines the results into only a few seconds of analysis time.

Experimental

A Supelco DVB/Carboxen/PDMS StableFlex SPME fiber was placed in an enclosed plastic bag with a banana for 10 minutes during each analysis. For direct analysis of the SPME fiber, the JEOL AccuTOF-DART™ system was set to the following parameters: needle voltage 3500V, discharge electrode 150V, grid electrode 250V, helium temperature 200 degrees C, and helium flowrate 2.3 L/min. A JEOL GC-Mate II high resolution sector bench top system equipped with a DB5-HT (0.25mm × 30m) was used for the GC-MS portion of the analysis. The GC-Mate II was set to the following parameters: inlet temperature 250 degrees C, split ratio 30, and helium flowrate 1.2 mL/min. The GC oven was set for the following temperature profile: 40 degrees C held for 2 min, ramp from 40 to 260 degrees C at 20 degrees C/min, 260 degrees C held for 2 min.

Results and Conditions

Figure 1 shows a typical AccuTOF-DART™ mass spectrum obtained for a banana headspace sample. At first glance, this spectrum might appear complex, but using the JEOL-provided ChemSW *Search from List Software*, all of the [M+H]+, [M+NH_4]+, and [2M+H]+ for each alcohol, acetate, and butyrate were identified, summed together, and normalized in a matter of seconds. Additionally, these results were directly comparable to the data obtained for the traditional GC-MS analysis done using the GC-Mate II. Figure 2 shows a side-by-side comparison of these data sets. This work clearly demonstrates that the AccuTOF-DART™ can be used with SPME to quickly produce results that are comparable to traditional analysis techniques.

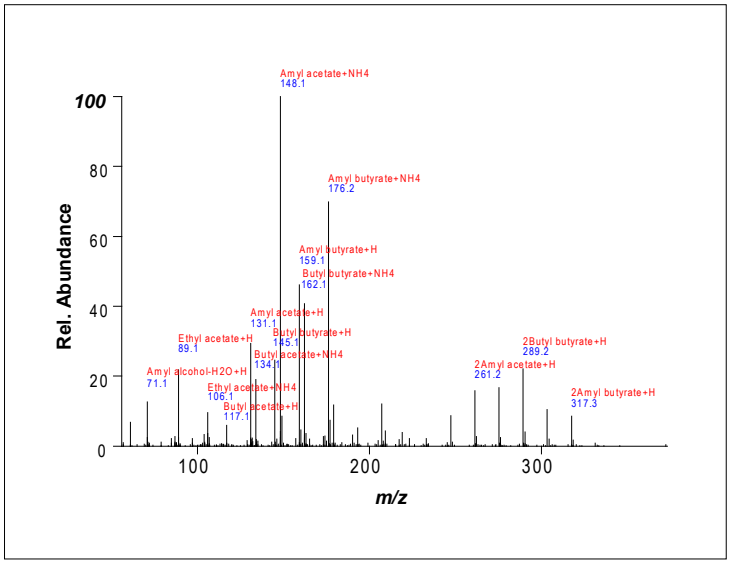

Figure 1. AccuTOF-DART mass spectrum for banana fragrance from SPME fiber.

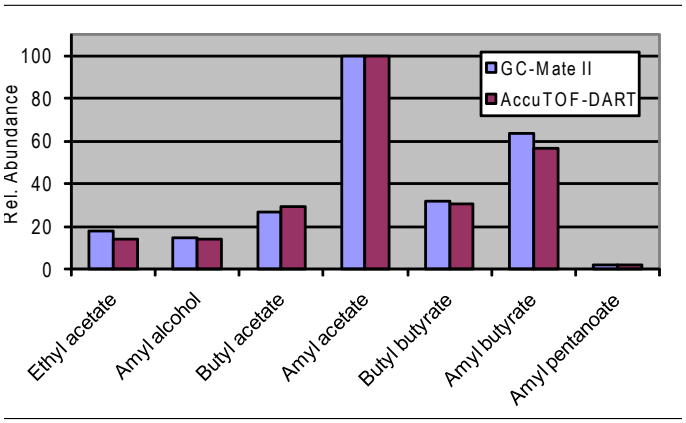

Figure 2. Comparison of relative abundances observed for compound using GC-MS and DART-MS analysis.

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