Chemical Reaction Monitoring with the AccuTOF-DART ™ Mass Spectrometer

Introduction

DART provides a convenient means for monitoring the progress of chemical reactions. Reactants, intermediates, products and byproducts can be detected by simply dipping a glass rod into the reaction pot and then placing the rod in front of the DART ion source. The AccuTOF's ability to measure accurate masses and isotopic abundances makes it possible to confirm or identify the elemental compositions of peaks in the mass spectra. Here we show the use of AccuTOF-DART to monitor the acetylation of 1,2-hexanediol as a function of time.

Experimental

600 µl of 1,5-hexanediol (4.9 mmol) was mixed with 700 µl (12.3 mmol) of glacial acetic acid and one drop of concentrated sulfuric acid in a loosely capped scintillation vial. The reaction was slowly warmed with a heat gun and a fume vent was positioned over the reaction vial and the DART source. Samples were taken periodically for analysis with the AccuTOF-DART by dipping a melting point tube into the reaction mixture and placing the tube in front of the DART ion source for a few seconds. The reaction progress was monitored by plotting the fractional abundances of the protonated molecules (MH^+) as measured for each component in the mass spectra.

Results

Figure 1 shows the time dependence of the fractional abundances of unreacted 1,5-hexanediol (MH^+ at *m/z* 119.1072), the reaction intermediate 1,5-hexanediol monoacetate (MH^+ at *m/z* 161.1178) and the product 1,5-hexanediol diacetate (MH^+ at *m/z* 203.1283). At 20 minutes, roughly equal amounts of the intermediate monoacetate and the diacetate are present. At 90 minutes, the reaction was incomplete and unchanging because an insufficient excess of acetic acid was present. Adding another 100 µl of acetic acid allowed the reaction to go to completion in 120 minutes.

Conclusion

AccuTOF-DART provides a convenient and rapid means for monitoring the progress of chemical reactions. Reactants, intermediates, and products are readily detected.

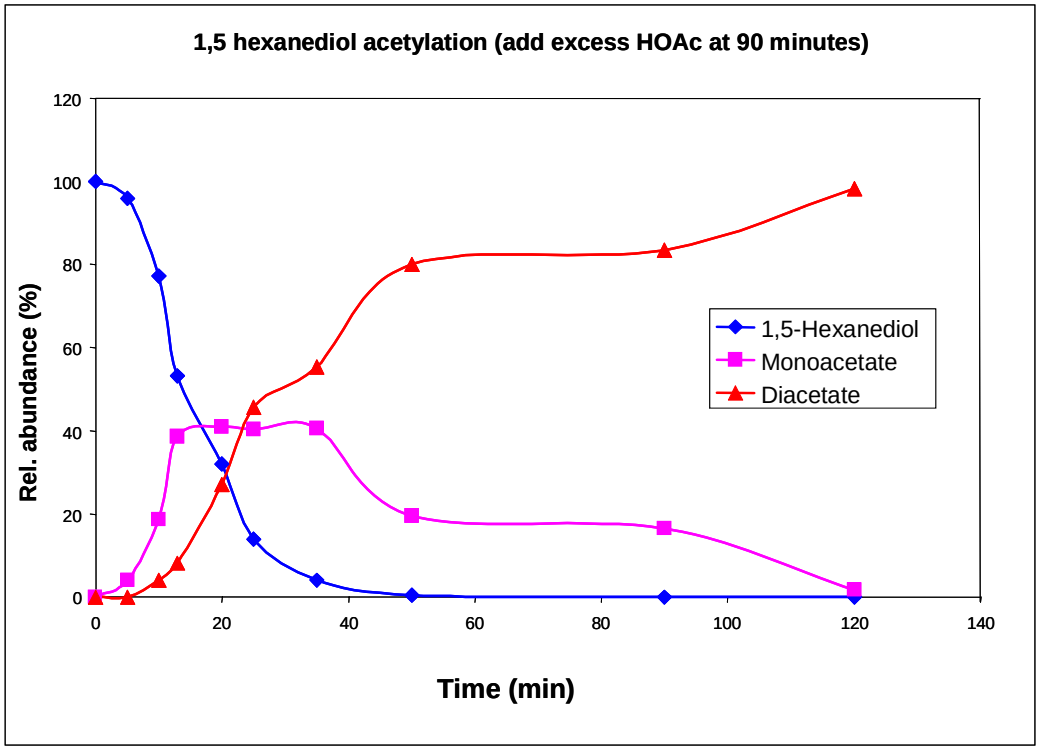

Figure 1. Synthesis of 1,5-hexanediol diacetate monitored by AccuTOF-DART.

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