

Waters Alliance® LC/MS System



Key Words

Electrospray ionization, ESI
Flow injection analysis
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Purity
ZMD mass detector
Z-Spray™ source

Purity Determination of C¹³-Acetate Using Isotope Ratios

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Background

The goal of this study was to demonstrate the applicability of mass spectrometric detection to the analysis of isotope ratios to determine purity. The samples were run previously using the Platform LC Mass Spectrometer and the C¹³-acetate purity result was less than anticipated. The reason for the low purity result was possibly caused by a trace C¹² acetate contamination following the use of ammonium acetate buffers. In this study the acetate sample was run on the ZMD4000 Mass Spectrometer which has a much improved interface facilitating easy cleaning access to all components in the source. This allows the removal of contaminants from previous analyses. Flow injection analysis (FIA) was used because isotopic purity was required for one compound.

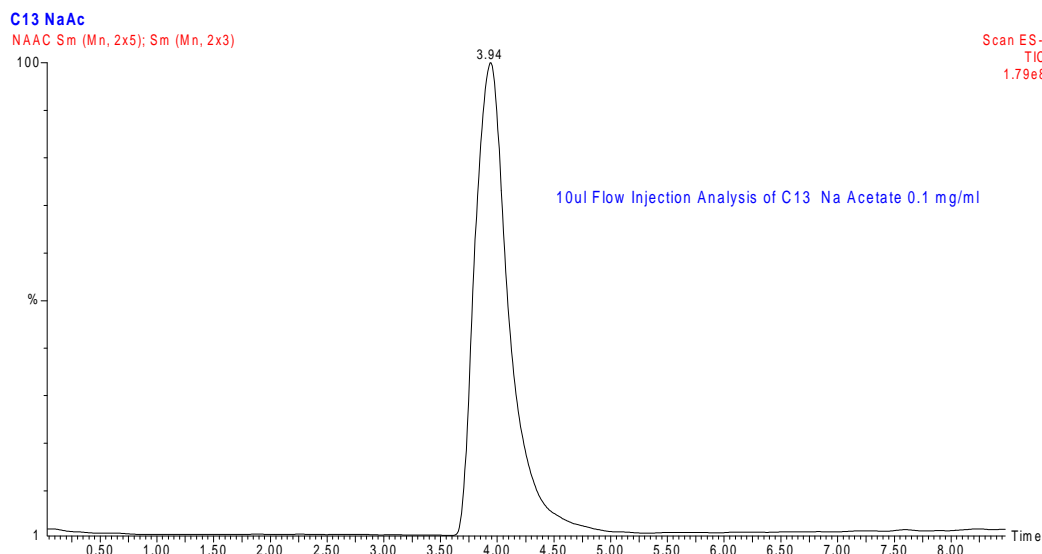
Analytical Conditions

Instrumentation: Waters Alliance® LC/MS System consisting of 2690 Separations Module and ZMD mass detector.

The optimum ionization mode was negative electrospray (ESI) for the best signal-to-noise ratio. However, both positive ESI and atmospheric pressure chemical ionization (APCI) can be used.

Sample: C¹³ sodium acetate was dissolved in 0.1% citric acid. The final concentration was 0.1 mg/mL. Flow injection analysis was done in a mobile phase of 0.1% citric acid at a flow rate of 100 µL/min.

Figure 1: TIC Chromatogram from Flow Injection Analysis of Sodium Acetate



Following optimization of mass spectrometric conditions for negative ion electrospray, samples were injected and the mass spectra were scanned from 50 m/z to 70 m/z. Ions specific to the analyte were extracted from the full scan data.

Figure 1 shows the MassLynx™ total ion current TIC profile from the flow injection analysis of a 10 µL sample. Analysis time to acquire results is about two minutes per sample.

Spectra were extracted from the apex of the sodium acetate peak and isotope ratios were calculated based on the intensity of the [M-H]⁻ ions 59, 60, 61 and 62 (Figure 2). The mass of C¹³-acetate is 60 containing one C¹³ atom.

The purity of the C¹³ sodium acetate was found to be 99.13% by the calculation shown on Figure 2. This was possible because of the wide linear dynamic range of the Dynolite detector.

The Z-Spray™ interface of the ZMD mass detector is easy to clean and can reduce the possibility of errors due to source contamination. It can yield good isotope ratio values for highly purified compounds.

Figure 2: Spectrum of C¹³ Sodium Acetate

