Waters Alliance[®] LC/MS System



Key Words Chloride adducts Dextrose syrup Electrospray ionization, ESI Sensitivity Sugars ZMD mass detector

Sugar Analysis by LC/MS

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Background

Many authors have reported the use of post-column addition of various anions and cations to enhance the electrospray ionization (ESI) sensitivity of sugars. Divalent cations such as Mn^{2+} give quite good sensitivity with positive ESI. The use of NH_4^+ adducts for positive ESI of glucose oligomers has also been reported. The use of anions such as CI⁻ and I⁻ were shown to work well with negative ESI mode.

There are methods developed by others using post-column addition of HCl to improve sensitivity of sugar analyses. However, for simultaneous analysis of sugars and acids it was not satisfactory.

These experiments explore the use of CI salts added to the mobile phase to achieve the same enhaced sensitivity. The salt chosen was LiCI due to its good solubility and only a few millimolar solution was required to generate stable CI adducts.

All work was done on Alliance®HT ZMD system with the Waters High Performance Carbohydrate column using a CH_3CN/H_2O gradient.

Analytical Conditions

The Waters Alliance[®] LC/MS system consisted of a 2790 Separations Module and a ZMD mass detector. The separation was performed on a High-Performance Carbohydrate Analysis column using a gradient at 0.5 mL/min and a column temperature of 40°C. The gradient was 80% acetonitrile-water to 50% acetonitrile-water, both containing 0.001M LiCl. The chloride was used to form the sugar-chloride adducts. Electrospray negative mode was used to generate the negative ions.

Advantage

Sugars are not easily detected by UV and are usually detected with a differential refractometer (RI) which is not a very sensitive technique. This LC/MS method can be a more sensitive alternative.



Figure 1: Ions Formed Between Glucose and Chloride

Figure 2: Peak Assignment from Glucose Syrup DE43 from MS Data

Glucose and glucose oligomers from G2 to G8 were detected.



Figure 3: Electrospray Negative Spectra of Ions from Peaks in DE43 Syrup

The spectra for the chloride adducts of G1 to G5 are shown. Both the Cl³⁵ and Cl³⁷ adducts are present. The molecular weight of glucose is 180.

