CAPSULES
notes and applications from Bioanalytical Systems, Inc.

LCEC Of Sugars At A Copper Electrode

Purpose

Determination of mono- and di-saccharides by D.C. Amperometry at a copper electrode.

LC determination of sugars is of interest to both the food industry and the medical community. Sugars are easily separated by reverse-phase or ion-chromatography techniques, but lack a chromophore that would allow the use of a UV-Visible detector [1].

Several techniques have been developed to detect sugars electrochemically. They can be oxidized at gold or platinum electrodes [2,3], but the product(s) of this oxidation are adsorbed to the surface, and must be removed by alternating pulses of cleaning potentials (Pulsed Amperometric Detection, PAD).

A recent alternative to PAD is the detection of sugars by D.C. Amperometry at a copper electrode [4]. Copper is not prone to the fouling seen with gold and platinum. Therefore, no cleaning pulses are required, and the analysis can be performed using a fixed potential.

Existing Methods

LC with refractive index detection, which suffers from lack of sensitivity and selectivity, and PAD, which requires special equipment to control the waveform.

Conditions

System: BAS 480 Liquid Chromatograph, with the DA-5 Data Analysis and Control system for collecting data.
Detector: BAS LC-4C Amperometric Detector
Electrode: 3 mm copper (MF-1001)
Potential: +550 mV (*vs.* Ag/AgCl)
Column: Hamilton RCX-10 anion exchange, 250 x 4.1 mm, 7 µm particle size.
Mobile Phase: 0.1 M NaOH.
Flow rate: 1.5 mL/min.
Injection Volume: 5 µL
Linear Range: 500 pg to at least 100 ng.

Notes

Chromatograms for various samples and standards are presented in F1 - F3.

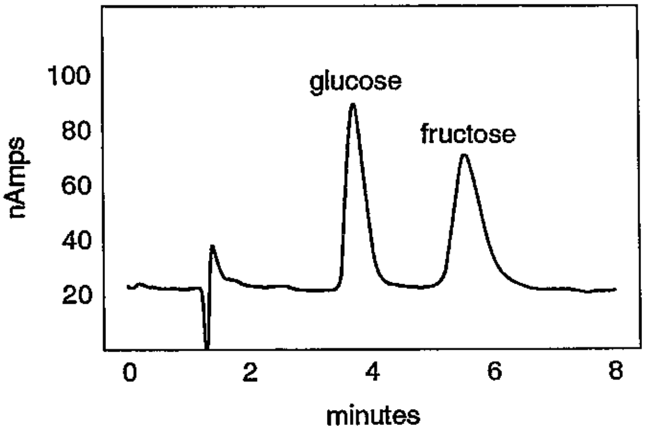

Figure 1. Grape juice, passed through a 25 kdalton membrane filter and diluted 1:10,000 in water.

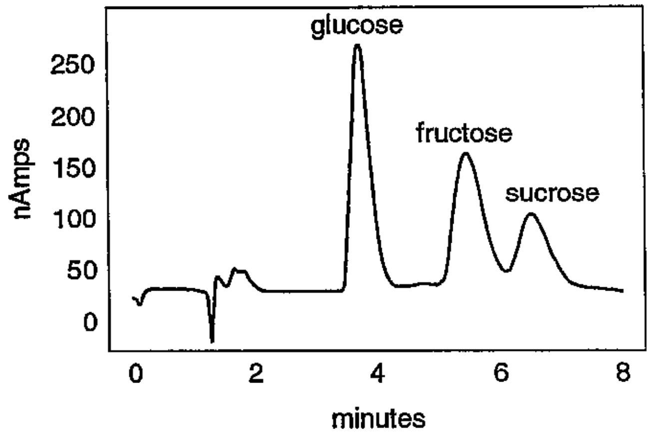

Figure 2. Orange juice, passed through a 25 kdalton membrane filter and diluted 1:1000 in water.

Oxidation of carbohydrates at a copper electrode occurs only at high pH [4]. This allows the use of ion-exchange chromatography, as carbohydrates become anionic at high pH. The mobile phase must be

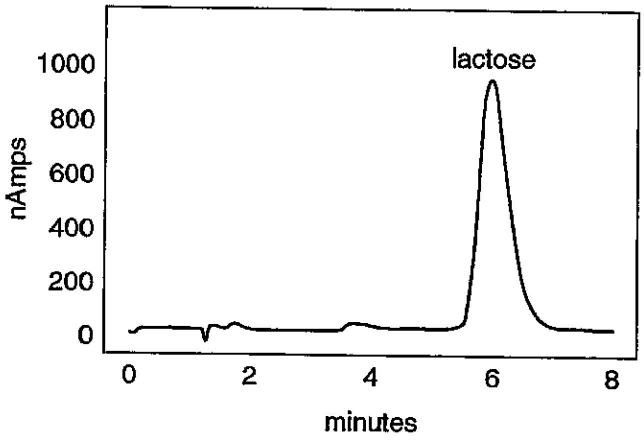

Figure 3. Milk, passed through a 25 kdalton membrane filter and diluted 1:1000 in water.

continuously sparged with inert gas, and all plumbing must be stainless steel, because CO_2 from the air will form carbonates that affect column performance. Because of this, the column manufacturer recommends the use of carbonate-free NaOH.

Since alkaline mobile phases will attack glass, the solvent reservoir must be an inert plastic, such as high-density polyethylene (HDPE).

The high pH mobile phase will affect the appearance of the reference electrode, turning its clear gel filling an opaque green. This did not affect the performance of the reference electrode over several weeks of testing.

The copper electrode will oxidize to a charcoal-gray color. This is normal! Do not polish the electrode merely to remove this coating.

References

1. *P. Luo, M.Z. Luo and R.P. Baldwin, J. Chem. Educ. 70 (1993): 679-681.*
2. *D.C. Johnson and W.R. LaCourse, Anal. Chem. 62 (1990): 589A-597A.*
3. *D.C Johnson, D. Dobberpuhl, R. Robers and P. Vandeberg, J. Chromatogr. 640 (1993): 79-96.*
4. *M.Z Luo, and R.P. Baldwin, J. Electroanal. Chem. 387 (1995): 87-94.*

© COPYRIGHT 1995, Bioanalytical Systems, Inc.

© Copyright Bioanalytical Systems, Inc. ALL RIGHTS RESERVED
No portion may be reproduced without expressed, written permission of Bioanalytical Systems, Inc.