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Fast determination of inorganic cations and low mass amines in a spoiled grape juice sample using IC-MS

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Introduction

Reliable analytical methods for food and beverage samples are required in order to report ingredients for food labeling requirements, to maintain product quality, and to ensure the absence of food contamination and spoilage. Food and beverage samples are almost invariably one of the most complex sample matrices and, therefore, are among the most challenging samples to analyze.

Ion chromatography (IC), using eluent generation and suppressed conductivity, provides chromatographic selectivity, low chemical noise, and high compatibility with a mass spectrometer (MS).[1,2] Additionally, the analytes leave the IC system as ions, further increasing MS compatibility.

Mass spectrometry coupled to IC provides higher selectivity and thus, better detection limits. In addition, MS is one of the most universal detectors, providing powerful screening, structural, and confirmatory information.[2] The heated electrospray ionization (HESI), with applied high temperature aiding voltage, transforms the aqueous IC stream into a fine spray that can enter the MS detector.[1]

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In this application brief, separations of four inorganic cations, one alkyl-, and one alkanolamine in a spoiled grape juice sample were achieved within six minutes. The analytes were detected serially, by suppressed conductivity and single quadrupole mass spectrometry in full scan (FS) and SIM (select ion monitoring) modes without the aid of a desolvation agent during separation or ionization.

This application brief is an update to Application Note 269.[3] Here, the application is performed using a Thermo Scientific™ Dionex™ Integrion™ HPIC™ system coupled to a Thermo Scientific™ ISQ™ EC single quadrupole mass spectrometer.

Experimental

Ion Chromatography

- Dionex Integrion HPIC system, RFIC™ model with a second six-port high-pressure divert valve and Conductivity Detector
- Thermo Scientific™ Dionex™ AS-AP autosampler
- Thermo Scientific™ Dionex™ AXP-MS auxiliary pump to supply water for the suppressor

Mass Spectrometry

- ISQ EC single quadrupole mass spectrometer
- Thermo Scientific syringe pump for method optimization
- HESI II probe

Software

Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) software, 7.2 SR 6

Methods

Columns:	Thermo Scientific™ Dionex™ IonPac™ CG12A-5µm, CS12A-5µm, 3 mm i.d.
Eluent:	33 mM Methanesulfonic acid (MSA)
Eluent Source:	Thermo Scientific™ Dionex™ EGC 500 MSA cartridge, Thermo Scientific™ Dionex™ CR-CTC 600 trap column, high pressure degasser module
Flow Rate:	0.5 mL/min
Injection Vol.:	100 µL
Detection 1:	Suppressed conductivity, Dionex CERS 500e suppressor, external water mode at 0.7 mL/min by the AXP-MS auxiliary pump
Typical Conductance Background:	< 1 µS-min
MS Detection:*	+ESI, +3000 V, Full Scan, 18-250 *m/z* and SIM, HESI II
Temperatures:	Vaporizer: 250 °C; Ion Transfer: 300 °C
Flow (N_2):	Sheath: 60 psi, Aux: 26 psi; Sweep: 0.5 psi
Desolvation agent:	None

SIM mode:	Ion *m/z*	CID (V)
Sodium as $Na_2 \bullet H_2O$	59	10
Ammonium as $NH_4 \bullet H_2O$	36	2
Potassium	39	45
Magnesium as $Mg_2 \bullet H_2O$	66	5
Calcium	40	45
Dimethylamine	46	10
Ethylamine	46	45
Monoethanolamine	62	15
Diethylamine	74	15
Triethanolamine	150	25

*Note: The optimum HESI ion source settings and responses may vary between instruments.

Results

Figure 1 shows the IC-MS results of inorganic cations and amines in a diluted, spoiled grape juice sample with visible mold. The ions were detected in SIM mode from *m/z* 39 to 150 as native ions or water-adducts. The IC and SIM peaks are symmetrical and have strong responses, E3 to E5 ion counts (in SIM mode). Bare, unsolvated calcium *m/z* 39 had the lowest response at 3e2 (300) counts. Notice that the monoethanolamine *m/z* 62 and diethylamine *m/z* 74 were previously undetected by suppressed conductivity but selectively detected by MS. More information can be found in the Thermo Scientific™ AppLabs Library of Analytical Applications.[4]

References

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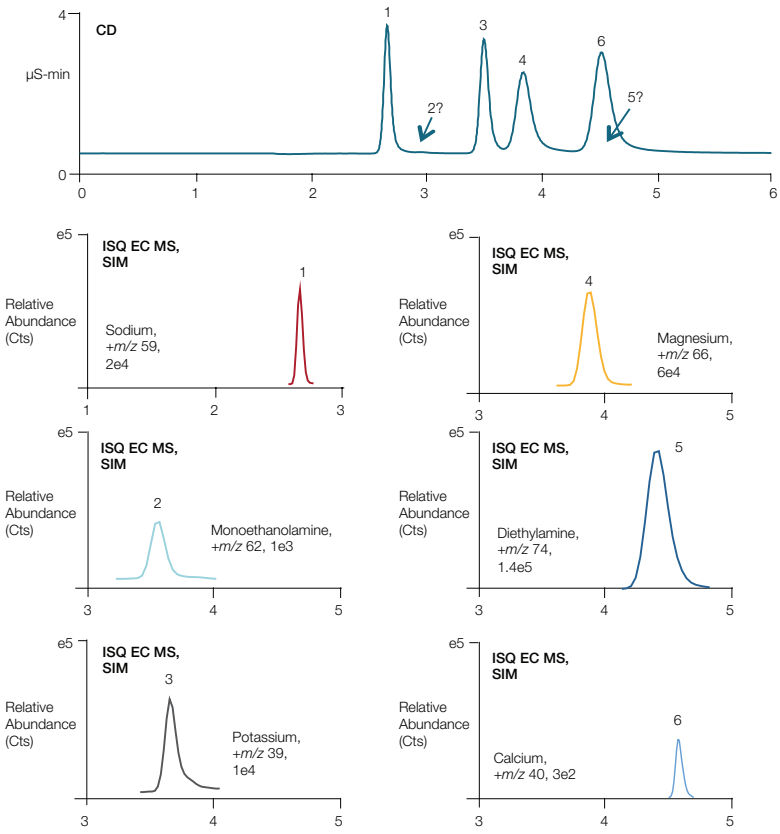

Columns:	Dionex IonPac CG12A, CS12A, 3 mm
Eluent:	33 mM Methanesulfonic Acid (MSA)
Eluent source:	Dionex EGC 500 MSA cartridge, Dionex CR-CTC 600 trap column
Flow rate:	0.5 mL/min
Inject volume:	100 µL
Oven temperature:	30 °C
Detector 1:	Suppressed conductivity, Dionex CERS 500e suppressor, 49 mA, 20 °C, external water mode, 0.7 mL/min by Dionex AXP-MS pump
Detector 2:	ISQ EC, +ESI, +3000 V source, HESI II
Scan mode:	Full scan: 18-250 m/z, SIM
Make-up solvent:	none
Source temp.:	Vaporizer 250 °C , Ion Transfer 300 °C
N2 Gas flow (psi):	Sheath 60, Aux 26, Sweep 0.5
Sample prep.:	1000-fold dilution with DI

Figure 1. Determination of co-eluting amines in a moldy grape juice sample.

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