Detection of common organic acids using a compact ion chromatography system coupled with mass spectrometry

Authors

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Keywords

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Introduction

This application brief demonstrates a method for detecting common organic acids using a Thermo Scientific™ Dionex™ Integrion™ HPIC™ system with a Thermo Scientific™ Dionex™ IonPac™ AS11-HC-4µm column set and coupled to a Thermo Scientific™ ISQ™ EC single quadrupole mass spectrometer. Mass spectrometry (MS), as a detection technique for ion chromatography (IC), has recently gained popularity due to the increasing demand for sensitivity, selectivity, confirmation of identity, and structural interpretation.[1] The ISQ EC single quadrupole mass spectrometer increases analytical confidence by providing sensitive detection and mass confirmation in a product that requires less operator training than a typical mass spectrometer.

Figure 1 shows chromatograms of 0.5 mg/L each of pyruvate, succinate, malate, and citrate standard using a suppressed conductivity detector (CD) and an MS detector in selected ion monitoring (SIM) mode. The CD responds to all charged species regardless of their molecular mass. If there is a coelution of multiple ionic components, the CD cannot discern them and presents them as one peak.

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The MS, on the other hand, is a more universal and selective detector. In SIM mode, it typically yields only one component peak for each target analyte, even in the case of coelution. Under aqueous eluent conditions, succinate and malate coelute, and therefore cannot be identified and quantified with suppressed conductivity detection. However, they can be accurately quantified with mass spectrometric detection because they are shown in different SIM channels, *m/z* 117 for succinate and *m/z* 133 for malate, respectively. Moreover, compared with suppressed conductivity detection, MS provides higher sensitivity for the four common organic acids in Figure 1.
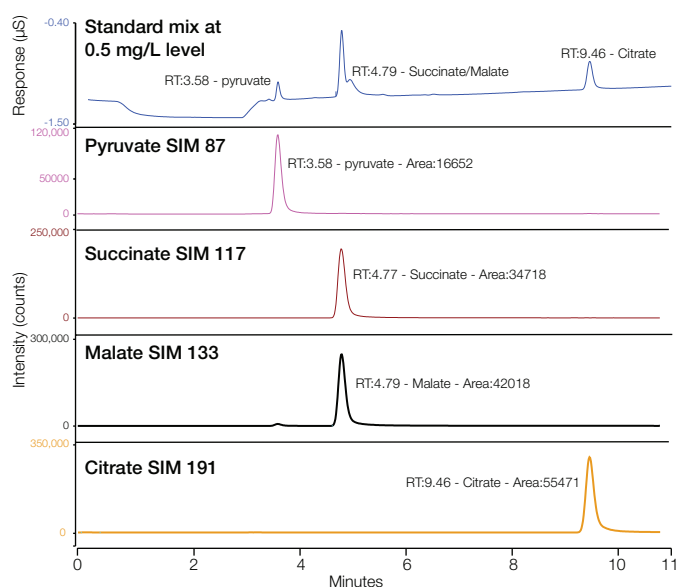
Figure 1. Conductivity and SIM chromatograms of four common organic acids.

Methods

Conditions	
IC system	Dionex Integrion HPIC system
Columns	Dionex IonPac AS11-HC-4 µm Guard, 2 × 50 mm Dionex IonPac AS11-HC-4 µm Analytical, 2 × 250 mm
Eluent source	Thermo Scientific™ Dionex™ EGC 500 KOH Eluent Generator Cartridge with Thermo Scientific™ Dionex™ CR-ATC 600
Gradient	30-60 mM KOH (0-9 min), 60 mM KOH (9-10 min), 30 mM KOH (10.05-11 min)
Flow rate	0.38 mL/min
Injection volume	2.5 µL
Temperature	40 ºC (column compartment), 20 ºC (detector compartment)
System backpressure	~4100 psi
Detection	Suppressed Conductivity, Dionex AERS 500e Electrolytically Regenerated Suppressor (2 mm), AutoSuppression, 57 mA, external water mode via Thermo Scientific™ AXP™ Pump, external water flow rate (at least 2× the eluent flow rate)
Background conductance	~ 0.3 µS
Run time	11 min

Mass Spectrometric Detection	
MS detector	ISQ EC single quadrupole mass spectrometer
Ionization interface	Electrospray ionization (ESI), negative mode
Gas control	Sheath gas pressure: 30 psi Aux gas pressure: 2.0 psi Sweep gas pressure: 0.0 psi
Source voltage	-3000 v
Ion transfer tube temperature	150 ºC
Vaporizer temperature	400 ºC
Scans	SIM mode: 87, 117, 133, 191 Dwell or Scan Times (sec): 0.2 SIM Widths (amu): 0.5 Ion Polarity: Negative SourceCIDVoltage: 0 Chrom. Filter Peak Width (sec): 25.0

This method can also be used for confirmation of organic acid identity in samples. Figure 2 shows that five common organic acids were identified in an animal feed sample: m/z 191 for citrate, m/z 89 for lactate, m/z 133 for malate, m/z 103 for malonate, and m/z 117 for succinate.

References

1. Weiss J. (2016) Handbook of Ion Chromatography, 3 Volume Set, 4th Edition. Wiley-VCH, Weinheim, Germany.
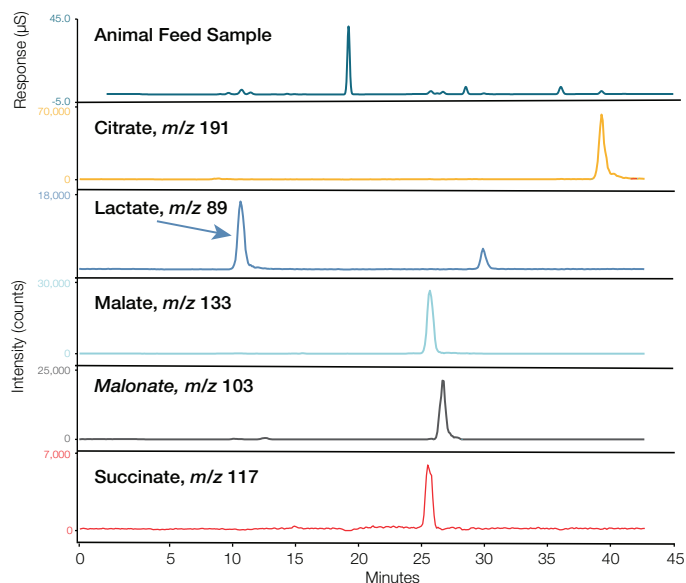
System:	Dionex Integrion HPIC System	
	ISQ EC single quadrupole mass spectrometer	
Column:	Dionex IonPac AS11-HC-4µm Guard, 2 × 50 mm	
	Dionex IonPac AS11-HC-4µm Analytical, 2 × 250 mm	
Eluent source:	Dionex EGC 500 KOH cartridge with CR-ATC 600	
Gradient:	1 mM KOH (0-8 min), 1-30 mM (8-30 min), 30-60 mM (30-44 min), 60 mM (44-45 min)	
Column temperature:	40 °C	
Flow rate:	0.35 mL/min	
Inj. volume:	2.5 µL	
Detection:	Suppressed conductivity, Dionex AERS 500e suppressor, 2 mm, Autosuppression, 52 mA, external water mode (0.7 mL/min) via ASP pump	

MS conditions are the same as shown in "Method: except for as below:
SIM mode: 89, 103, 117, 133, 191

Figure 2. The identification of common organic acids in an animal feed sample by the comparison of retention time and *m/z*.

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