

LC-MS/MS Method for the Rapid Analysis of Five Artificial Sweeteners Using a Core Enhanced Technology Column

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Key Words

Accucore RP-MS, Core Enhanced Technology, solid core, sweeteners, acesulfame potassium, saccharin, sodium cyclamate, aspartame, sucralose

Abstract

This application note demonstrates the use of the Thermo Scientific™ Accucore™ RP-MS HPLC column for the rapid analysis of five artificial sweeteners.

Introduction

Artificial sweeteners are used as a sugar substitute in food and beverage products such as cereal bars and soft drinks. Their use is becoming more popular as consumers are increasingly concerned about obesity and dental decay caused from consuming natural sugars. In addition, artificial sweeteners are ideal for use by people suffering from diabetes.

Acesulfame potassium, saccharin, aspartame, and sucralose are all commonly used artificial sweeteners that have been approved for use by the United States Food and Drug Administration (FDA). Sodium cyclamate has been banned in the United States, although it is approved for use in many other countries, including the UK.

As can be seen in Figures 1 to 5 some of the sweeteners do not have a UV chromophore; therefore, detection at the required limits using HPLC-UV can be a challenge. This application demonstrates an alternative approach using LC-MS/MS with an Accucore RP-MS column.

Accucore HPLC columns use Core Enhanced Technology™ to facilitate fast and high efficiency separations. The 2.6 µm diameter particles are not totally porous, but instead have a solid core and a porous outer layer. The optimized phase bonding creates a series of high coverage, robust phases. Accucore RP-MS column uses an optimized alkyl chain length for more effective coverage of the silica surface. This coverage results in a significant reduction in secondary interactions and thus highly efficient peaks with very low tailing. The tightly controlled 2.6 µm diameter of Accucore particles results in much lower backpressures than typically seen with sub-2 µm materials.



Experimental Details

Consumables	Part Number
Fisher Scientific™ LC-MS grade water	W/0112/17
Fisher Scientific LC-MS grade acetonitrile	A/0638/17
Fisher Scientific LC-MS grade formic acid	A/3295/PB05

Acesulfame potassium, saccharin, sodium cyclamate, aspartame, and sucralose were purchased from Sigma-Aldrich®

Sample Preparation

The sample was prepared to contain 200 ng/mL of acesulfame potassium, saccharin, sodium cyclamate, aspartame, and sucralose in water.

Separation Conditions

Part Number

Instrumentation:	Thermo Scientific Dionex™ UltiMate™ 3000 RS system consisting of an HPG-3200SD high pressure gradient pump, WPS-3000 RS thermostatted split-loop autosampler, and TCC-3000 RS column thermostat	
Column:	Accucore RP-MS 2.6 µm, 50 x 2.1 mm	17626-052130
Mobile phase A:	Water + 0.1% formic acid	
Mobile phase B:	Acetonitrile + 0.1% formic acid	
Gradient:	Time (minutes)	%B
	0.0	5
	0.10	5
	1.00	95
	1.25	95
	1.26	5
	2.00	5
Flow rate:	1 mL/min	
Column temperature:	40 °C	
Injection volume:	10 µL	
Weak injection wash solvent:	Acetonitrile / water (20:80 v/v)	
Strong injection wash solvent:	Acetonitrile / acetone / isopropanol (45:45:10 v/v/v)	

MS Conditions

Instrumentation:	Thermo Scientific TSQ Vantage™ MS
Ionization conditions:	HESI-II
Polarity:	Negative
Spray voltage (V):	4000
Vaporizer temp (°C):	450
Sheath gas pressure (Arbitrary units):	40
Aux gas pressure (Arbitrary units):	20
Capillary temp (°C):	300
Collision pressure (mTorr):	1.5
Cycle time (s):	0.02
Q1 (peak width):	0.7
Q3 (peak width):	0.7

Compound	Sucralose	Sodium cyclamate	Acesulfame potassium	Saccharin	Aspartame
Precursor ion (m/z)	395.1	178.1	162.0	182.1	293.2
Product ion (m/z)	359.1	79.9	82.0	42.0	261.1
Collision energy (eV)	15	28	16	21	14
S-lens (RF voltage)	120	78	56	79	87

Table 1: Transition details

Data Processing

Software: Thermo Scientific LC QUAN™

Results

All five sweeteners were analyzed in less than 1 minute using an Accucore RP-MS column as shown in Figure 6. The retention factor of the first peak was approximately 2.5. Chromatographic baseline resolution was not needed as positive identification of each individual sweetener was achieved using MS/MS detection. Table 2 shows the %RSD of the response for all five compounds with less than 5% RSD illustrating excellent precision.

Compound	Retention Time (min)	Response %RSD
Acesulfame potassium	0.30	3.8
Saccharin	0.55	3.7
Sodium cyclamate	0.61	4.0
Aspartame	0.76	4.6
Sucralose	0.76	3.8

Table 2: Precision data calculated from 6 replicate injections

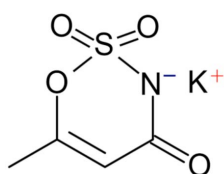


Figure 1: Structure of acesulfame potassium

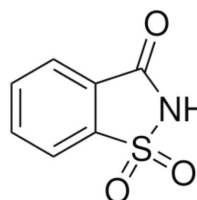


Figure 2: Structure of saccharin

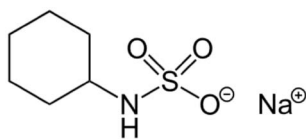


Figure 3: Structure of sodium cyclamate

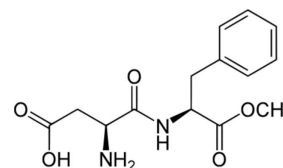


Figure 4: Structure of aspartame

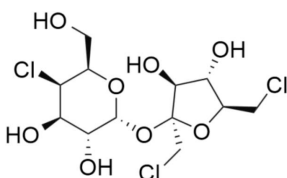


Figure 5: Structure of sucralose

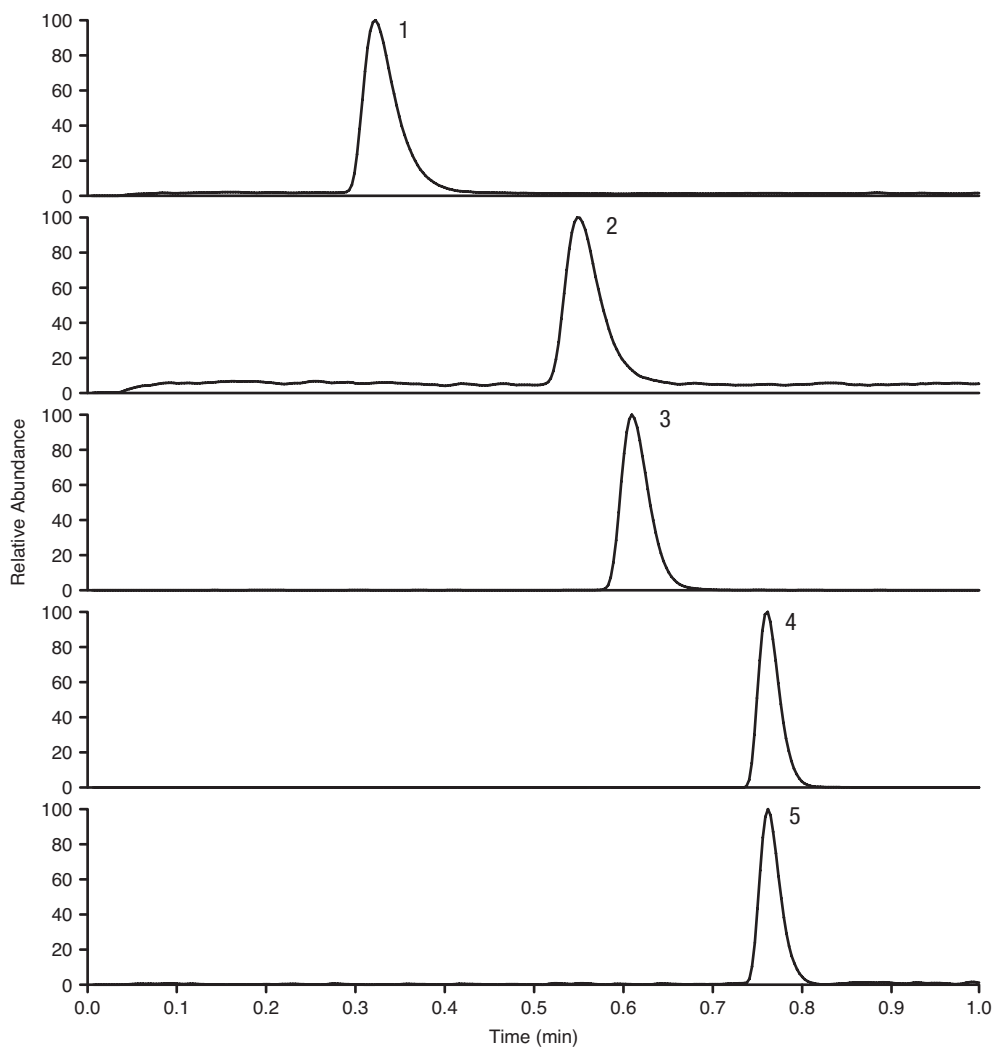


Figure 6: Selected ion chromatograms of acesulfame potassium (1), saccharin (2), sodium cyclamate (3), aspartame (4), and sucralose (5) at 200 ng/mL

Conclusion

This application demonstrates the successful LC-MS/MS analysis of five artificial sweeteners in a run time under 1 minute using an Accucore RP-MS column.

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