Analyzing Synthetic Sweeteners in Waste Water with Robust Sample Preparation

Application Note

Environmental

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Introduction

Synthetic sweeteners are sugar substitutes increasingly used as consumers become concerned about obesity and dental decay resulting from consumption of natural sugars. However, research has shown that some artificial sweeteners can cause tumors in certain animals [1]. To prevent potential danger to human health, it is necessary to control the amount of sweeteners in foods and waters. Sewage treatment plants do not completely remove artificial sweeteners from waste water, and these pollutants can contaminate waters downstream and be present in drinking water. For example, acesulfame, saccharin, cyclamate and sucralose have been detected in all German surface waters analyzed [2].

Agilent solid-phase extraction (SPE) is used to pre-concentrate sweeteners from water prior to LC/MS detection. In this example, we establish a robust SPE method that provides routine detection for four sweeteners (Figure 1), with high recovery values and very good standard deviation.

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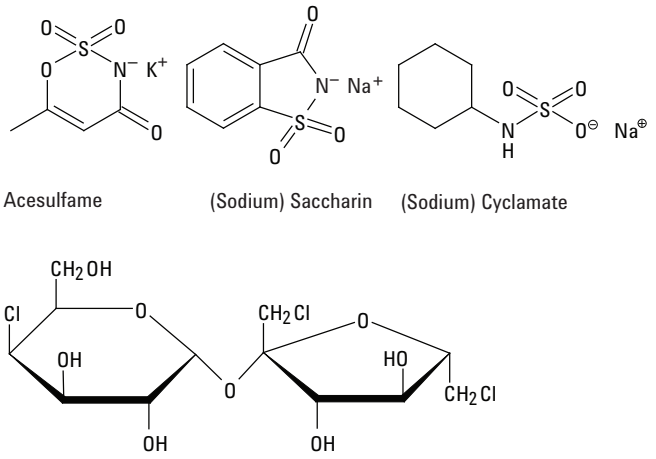

Figure 1. Chemical structure of artificial sweeteners.

Materials and Methods

SPE method

We used Agilent Bond Elut Plexa (200 mg, 6 mL, p/n 12109206) to extract water samples. This polymeric SPE product improves analytical performance and ease-of-use, and is an excellent sorbent for extracting polar and mid-polar species from water. Pre-concentration of the water samples employs SPE with disposable cartridge columns packed with 200 mg of the media. The cartridges are conditioned with 3 mL of methanol followed by 3 mL of acidified HPLC water (sulfuric acid, pH 2) at a flow rate of 5 mL/min. The water samples (100 mL) are acidified with sulfuric acid to pH 2 and loaded at a flow rate of 5 mL/min. Concentration of acesulfame, cyclamate, saccharin and sucralose in the water sample is 1 ppb. Elution of the analytes from the cartridge is done with 5 mL of methanol at a flow rate of 2 mL/min. The solvent is evaporated to near dryness with a stream of nitrogen and re-constituted in 1 mL acetonitrile:water (5:95).

HPLC/MS conditions

Column	Agilent ZORBAX Eclipse XDB-C18, 4.6 × 50 mm, 1.8 µm (p/n 927975-902)	
Mobile phase A	Water, 2 mM ammonium carbonate	
Mobile phase B	Methanol, 2 mM ammonium carbonate	
Flow rate	0.6 mL /min	
Gradient	Time (min)	B (%)
	0.0	2
	7.0	75
	9.0	75
	9.1	2
	15.0	2

Sweetener	RT (min)	Precursor ion (*m/z*)	Product ion (*m/z*)
Acesulfame	2.21	162	82
	2.21	162	78
Cyclamate	3.49	178.2	80
	3.49	178.2	81
Saccharin	2.96	182	42
	2.96	182	106
Sucralose	5.37	395.2	35
	5.37	397	37

Results and Discussion

Table 1 shows the percent recovery and RSD of the four sweeteners with injection volumes of 20 µL and 2 µL. Recovery values greater than 86% were achieved for acesulfame, saccharin and sucralose when 20 µL were injected. Even the very polar analyte cyclamate sodium salt could be detected with a recovery of 74%. However, sucralose was not detected from 2 µL injections.

Table 1. Percent Recovery and RSD Values of Sweeteners in Water Using LC/MS/MS Determination after SPE with Agilent Bond Elut Plexa. Spiked Concentration of Sweeteners was 1 ppb.

	Recovery and RSD (%)			
Injection volume	Acesulfame	Cyclamate	Saccharin	Sucralose
20 µL	86	74	91	86
RSD 20 µL	7	5	2	15
2 µL	92	77	92	nd
RSD 2 µL	7	5	2	–

Conclusions

The Agilent Bond Elut Plexa SPE sorbent was used to evaluate its potential for pre-concentration of four common sweeteners in waste water. The ease-of-use and high recovery values obtained with this sorbent make it an excellent choice for use in routine water control laboratories.

References

1. Takayama *et al*. "Long-Term Toxicity and Carcinogenicity Study of Cyclamate in Nonhuman Primates," *Toxicol. Sci.* (2000) 53: 33-39.
2. Scheurer *et al*. "Analysis and Occurrence of Seven Artificial Sweeteners in German Waste Water and Surface Water and in Soil Aquifer Treatment," *Anal. Bioanal. Chem.* (2009) 394: 1585–1594.

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Printed in the USA
July 25, 2011
5990-8248EN

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