

Analytical Methods

Electronic Supplementary Information

Determination of Eight Artificial Sweeteners in Wastewater by Hydrophilic Interaction Liquid Chromatography - tandem Mass Spectrometry

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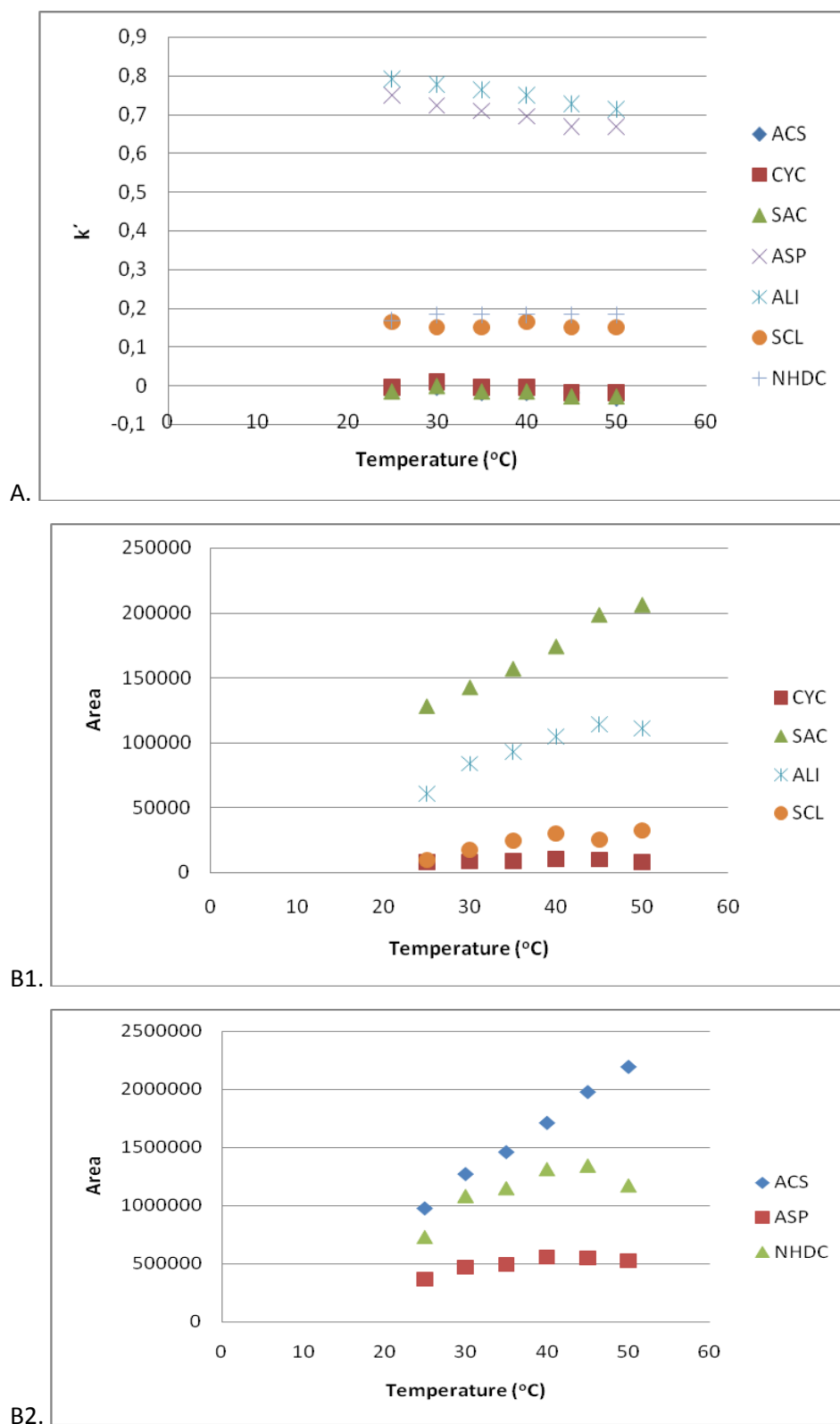


Fig. S1 Effect of the column temperature on: (A) the retention time; (B1) & (B2) the peak area.

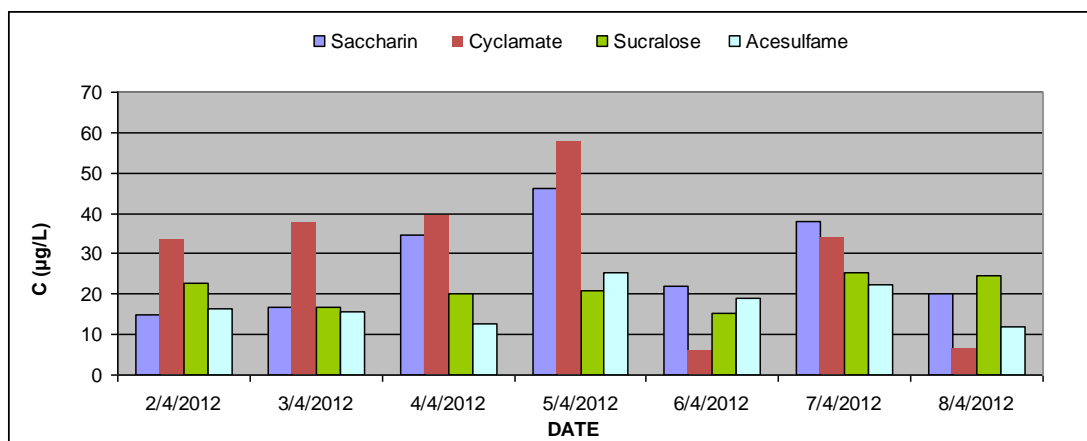


Fig. S2 Daily variation of concentrations of four artificial sweeteners in influent samples.

Table S1 Optimized ESI parameters for the determination of artificial sweeteners by LC-(-ESI)-MS/MS

Optimized Parameters	
Micrometer (in.)	0.45
Probe position	B
Spray voltage (V)	3000
Sheath gas (A.U.)	10
Auxiliary gas (A.U.)	10
Capillary Temperature (°C)	270
Skimmer(A.U.)	0

Table S2 Effect of the buffer on the mass spectrum and the intensity of the precursor ion of artificial sweeteners.

Analyte	Ammonium Formate	Ammonium Acetate
ACS	<p>S# 6010 FULL: PRO: 162 CE: 5 CT: 0.51 #A: 3 3.71e6</p> <p>Relative Abundance vs m/z</p> <p>Peak list (m/z): 48.419, 64.277, 78.164, 82.203, 98.108, 119.029, 144.815, 161.507</p>	<p>S# 2943 FULL: PRO: 162 CE: 5 CT: 0.51 #A: 3 2.83e6</p> <p>Relative Abundance vs m/z</p> <p>Peak list (m/z): 48.424, 64.313, 78.159, 82.207, 102.249, 118.877, 137.237, 161.518</p>
CYC	<p>S# 5188 FULL: PRO: 178 CE: 5 CT: 0.51 #A: 3 3.33e6</p> <p>Relative Abundance vs m/z</p> <p>Peak list (m/z): 79.450, 80.134, 96.070, 113.878, 177.514, 176.728</p>	<p>S# 2230 FULL: PRO: 178 CE: 5 CT: 0.51 #A: 3 1.96e6</p> <p>Relative Abundance vs m/z</p> <p>Peak list (m/z): 79.151, 80.135, 95.187, 177.509, 176.349</p>
SAC	<p>S# 6944 FULL: PRO: 182 CE: 5 CT: 0.51 #A: 3 3.03e6</p> <p>Relative Abundance vs m/z</p> <p>Peak list (m/z): 42.570, 62.407, 79.986, 106.004, 117.655, 135.881, 171.067, 181.425</p>	<p>S# 6057 FULL: PRO: 182 CE: 5 CT: 0.51 #A: 3 1.76e6</p> <p>Relative Abundance vs m/z</p> <p>Peak list (m/z): 42.565, 62.323, 90.199, 105.965, 138.145, 181.428</p>
ASP	<p>S# 7715 FULL: PRO: 293 CE: 5 CT: 0.51 #A: 4 2.66e6</p> <p>Relative Abundance vs m/z</p> <p>Peak list (m/z): 81.106, 97.084, 117.093, 173.099, 199.872, 216.890, 242.897, 260.573, 281.134, 292.375</p>	<p>S# 5335 FULL: PRO: 293 CE: 5 CT: 0.51 #A: 3 1.01e6</p> <p>Relative Abundance vs m/z</p> <p>Peak list (m/z): 42.689, 79.095, 97.034, 118.049, 173.419, 199.974, 216.857, 242.848, 261.107, 280.550, 292.386</p>

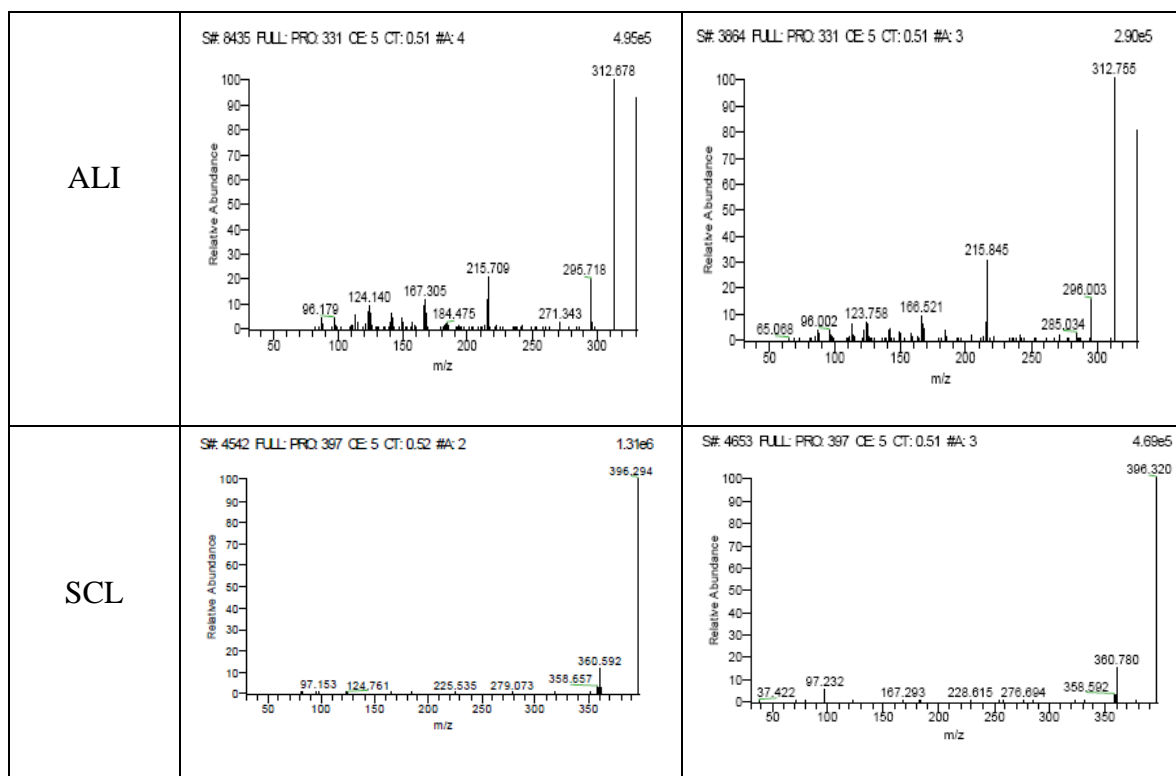


Table S3 Effect of the mobile phase composition on the retention time, capacity factor k' and asymmetry factor on XBridge HILIC column

	Analytes	t_R (min)	k'	T_f
20:80 (ammonium formate pH 5.5: acetonitrile)	ACS	2.72	-0.18	2.0
	CYC	2.75	-0.17	2.0
	SAC	2.73	-0.17	2.0
	ASP	5.64	0.71	2.0
	ALI	6.17	0.87	2.3
	SCL	4.66	0.41	1.4
	NHDC	4.80	0.45	1.2
20:80 (ammonium formate pH=4: acetonitrile)	ACS	3.41	0.03	1.3
	CYC	3.60	0.09	1.3
	SAC	3.44	0.04	1.2
	ASP	6.90	1.09	2.0
	ALI	7.51	1.28	2.1
	SCL	4.69	0.42	0.9
	NHDC	4.89	0.48	2.0
15:85 (ammonium formate pH 5.5: acetonitrile)	ACS	3.16	-0.04	1.2
	CYC	3.41	0.03	1.5
	SAC	3.19	-0.03	1.0
	ASP	9.15	1.77	2.3
	ALI	10.80	2.27	2.0
	SCL	4.72	0.43	1.2
	NHDC	4.86	0.52	1.5
15:5:80 (ammonium formate pH 5.5: methanol: acetonitrile)	ACS	3.13	-0.050	2.0
	CYC	3.31	0.003	2.0
	SAC	3.26	0.01	2.0
	ASP	8.39	1.54	1.5
	ALI	8.36	1.53	2.0
	SCL	4.45	0.35	1.0
	NHDC	4.51	0.37	1.0

Table S4 Effect of the mobile phase composition on the retention time, capacity factor k' and asymmetry factor on Kinetex HILIC column

	Analytes	t_R (min)	k'	T_f
20:80 (ammonium formate: acetonitrile)	ACS	2.19	-0.005	2.0
	CYC	2.22	0.009	1.7
	SAC	2.19	-0.005	2.0
	ASP	3.67	0.668	1.8
	ALI	3.80	0.727	1.5
	SCL	2.56	0.164	1.0
	NHDC	2.62	0.191	1.1
15:85 (ammonium formate: acetonitrile)	ACS	2.19	-0.005	2.0
	CYC	2.19	-0.005	1.7
	SAC	2.20	0.000	1.5
	ASP	4.12	0.873	1.8
	ALI	4.30	0.955	1.8
	SCL	2.62	0.191	1.0
	NHDC	2.68	0.218	1.0
15:5:80 (ammonium formate: methanol: acetonitrile)	ACS	2.19	-0.005	2.0
	CYC	2.22	0.009	1.0
	SAC	2.20	0.000	1.6
	ASP	3.94	0.791	1.6
	ALI	4.09	0.859	1.6
	SCL	2.59	0.177	1.1
	NHDC	2.65	0.205	1.1
15:10: 75 (ammonium formate: methanol: acetonitrile)	ACS	1.98	-0.100	1.6
	CYC	2.02	-0.080	1.1
	SAC	2.02	-0.080	1.5
	ASP	4.35	0.977	1.2
	ALI	4.39	0.995	1.4
	SCL	2.50	0.136	1.0
	NHDC	2.57	0.168	1.0
10:10:80 (ammonium formate: methanol: acetonitrile)	ACS	1.89	-0.141	1.0
	CYC	1.95	-0.114	1.0
	SAC	1.93	-0.123	1.0
	ASP	7.15	2.250	1.1
	ALI	7.30	2.320	0.9
	SCL	2.59	0.177	1.0
	NHDC	2.65	0.205	1.0

Table S5 Recoveries for the eight artificial sweeteners using two different cartridge materials at pH 3 (sample volume 50 mL, spiked amount 5.0 µg/L, n=2)

ABSOLUTE RECOVERIES %								
Cartridge	ASP	ALI	NEO	ACS	CYC	SAC	SCL	NHDC
Isolute® (SDVB) 101	11	38	33	0	3	4	91	18
Strata™X	74	90	97	67	40	79	95	68