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CHEM 3170 Laboratory

Determination of Aspartame in Soft Drinks by Capillary Electrophoresis

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Pages: 10

## Experimental

Sample: Diet Sprite

Theoretical aspartame content: 211 ppm (from label)

Table 1. Instrumental parameters of the capillary electrophoresis instrument used in this experiment.

Instrument	SCIEX P/ACE System MDQ Plus CE system
<b>Capillary</b>	
Material	Fused silica
Internal diameter	50 $\mu\text{m}$
Outer diameter	375 $\mu\text{m}$
Total length	50 cm (40 cm to detector)
Operating Temperature	25 $^{\circ}\text{C}$
Run Time	15 min
Detection	UV, 214 nm (Direct absorbance)
Rinse Pressure	0.1 M NaOH: 20 psi for 3.0 min Water: 20 psi for 1.0 min Rinse buffer: 20 psi for 3.0 min
Injection	Pressure, 1 psi for 5.0 s
Separation Voltage	20 kV

Preparation of standards and samples:

To prepare the standards, an Eppendorf pipet was used to dispense the correct volumes of 1000 ppm aspartame standard solution and 18 M $\Omega$  water into five CE sample vials (see Table 2). Soft drink sample vials were prepared by first degassing the drink using a sonicator, and then dispensing approximately 1 mL into a CE sample vial through a 0.45  $\mu\text{m}$  syringe filter. A triplicate of samples was prepared.

Table 2. The volumes and concentrations of aspartame in each standard.

Standard	Aspartame Conc'n (ppm)	Volume 1000 ppm aspartame stock ( $\mu\text{L}$ )	Volume 18 M $\Omega$ Water ( $\mu\text{L}$ )	Total Volume ( $\mu\text{L}$ )
S1	100	100	900	1000
S2	200	200	800	1000
S3	300	300	700	1000
S4	400	400	600	1000
S5	500	500	500	1000

Each standard solution was run on the CE instrument and the peak area of the peak of interest was obtained. The peak of interest is the aspartame peak at a migration time of approximately 7 mins. The three soft drink samples were similarly run on the CE instrument and the peak areas at approximately 7 min migration time were used to determine concentration.

## Data and Results

Table 3. Capillary electrophoresis results of the aspartame peak for each standard solution.

Standard	Conc'n Aspartame (ppm)	Peak Area	Migration Time (min)
S0	0.00	0	-
S1	100	3596	6.954
S2	200	4360	7.037
S3	300	7909	7.054
S4	400	11724	7.071
S5	500	16302	7.088

Table 4. Capillary electrophoresis results of the aspartame peak for each soft drink sample replicate.

Replicate	Peak Area	Migration Time (min)
1	1216	7.017
2	1170	7.025
3	1135	7.017
<b>Average</b>	<b>1174</b>	

Table 5. Calculated results and uncertainty values from determination of aspartame.

Theoretical Aspartame Concentration	211 ppm
Experimental Aspartame Concentration	53.6 ppm $\pm$ 31.9 ppm
%RSD	59.5%
%Error	74.6%
Slope	31.3 $\pm$ 2.8
Intercept	-502 $\pm$ 850

Use of an internal standard approach could have increased the precision and accuracy of the analysis. Other sources of errors could be inconsistencies in preparation of standards.

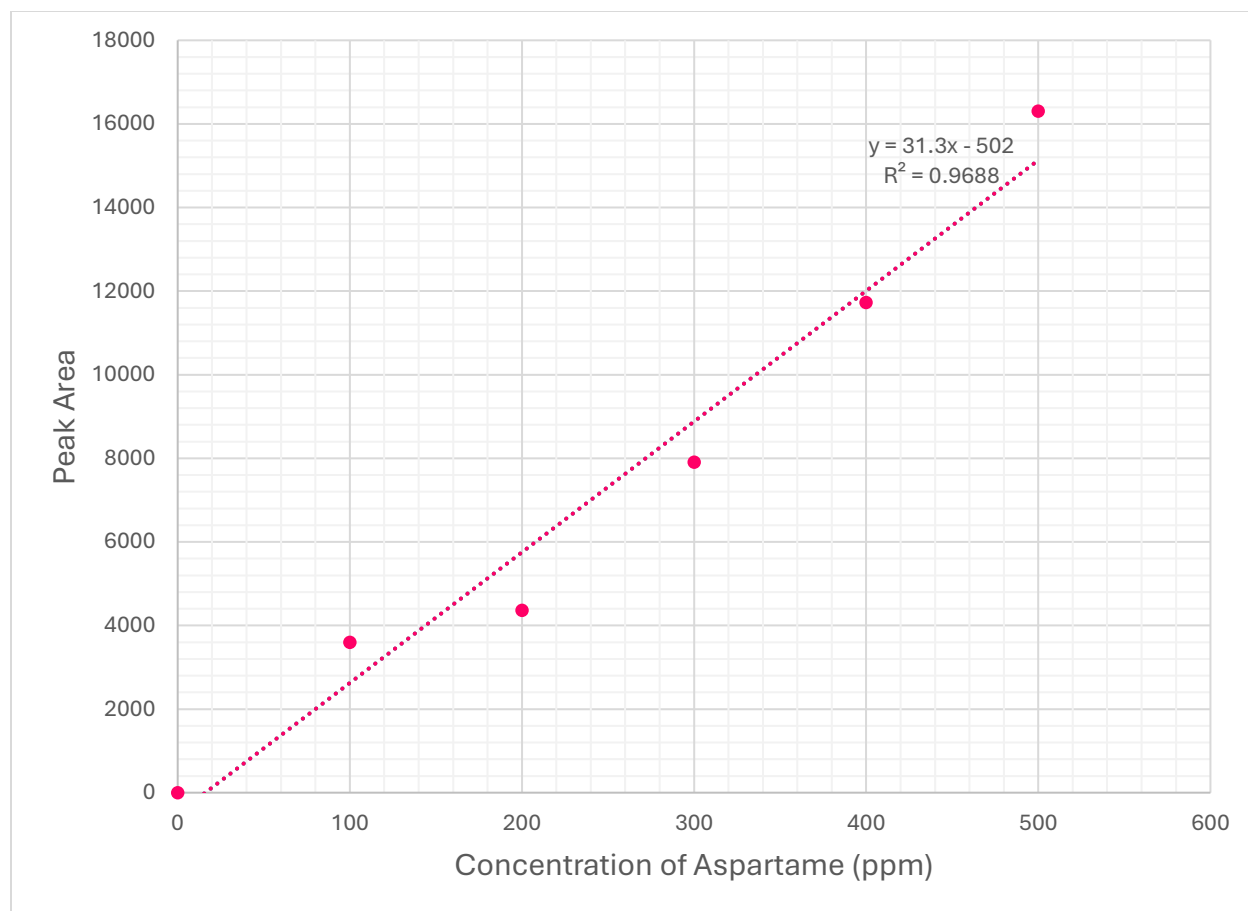


Figure 1. Calibration curve generated from the peak area of the aspartame peak from the standard solutions (see Table 3).

## Calculations

Aspartame concentration in standard 2:

$$C_2 = \frac{C_1 V_1}{V_2} = \frac{(1000 \text{ ppm})(0.200 \text{ mL})}{1.000 \text{ mL}} = 200 \text{ ppm}$$

Aspartame concentration in sample (derived x) using equation of the line:

$$\text{Average Peak Area} = \frac{1216 + 1170 + 1135}{3} = 1174$$

$$\text{Equation of the line: } y = 31.3x - 502$$

$$x = \frac{1174 + 502}{31.3} = 53.6$$

Uncertainty Calculations (see Figure 2):

$$S_y^2 = \frac{\sum d_i}{n - 2} = 1.38 \times 10^6$$

$$D = n \sum x_i^2 - (\sum x_i)^2 = 1.05 \times 10^6$$

$$S_m = \frac{n S_y^2}{D} = \frac{6 \times 1.38 \times 10^6}{1.05 \times 10^6} = 2.81$$

$$S_b = \sqrt{\frac{S_y^2 \times \sum x_i^2}{D}} = \sqrt{\frac{1.38 \times 10^6 \times 5.50 \times 10^5}{1.05 \times 10^6}} = 850.2$$

$$S_x = \frac{s_y}{|m|} \sqrt{\frac{1}{k} + \frac{1}{n} + \frac{y - \bar{y}_i}{m^2 (\sum x_i - \bar{x})^2}} = \frac{1175}{31.3} \times \sqrt{\frac{1}{3} + \frac{1}{6} + \frac{1174 - 7315}{31.3^2 (1500 - 250)^2}}$$

$$= 31.9$$

Percent RSD:

$$\%RSD = \frac{S_x}{x} \times 100\% = \frac{31.9}{53.6} \times 100\% = 59.5\%$$

Percent Error:

$$\% \text{ Error} = \frac{\text{Measured value} - \text{True value}}{\text{True value}} = \frac{211 \text{ ppm} - 53.6 \text{ ppm}}{211 \text{ ppm}} = 74.6\%$$

## Conclusion

The concentration of aspartame in diet sprite was determined by capillary electrophoresis to be 53.6 ppm with a relative standard deviation of 59.5%.

## References

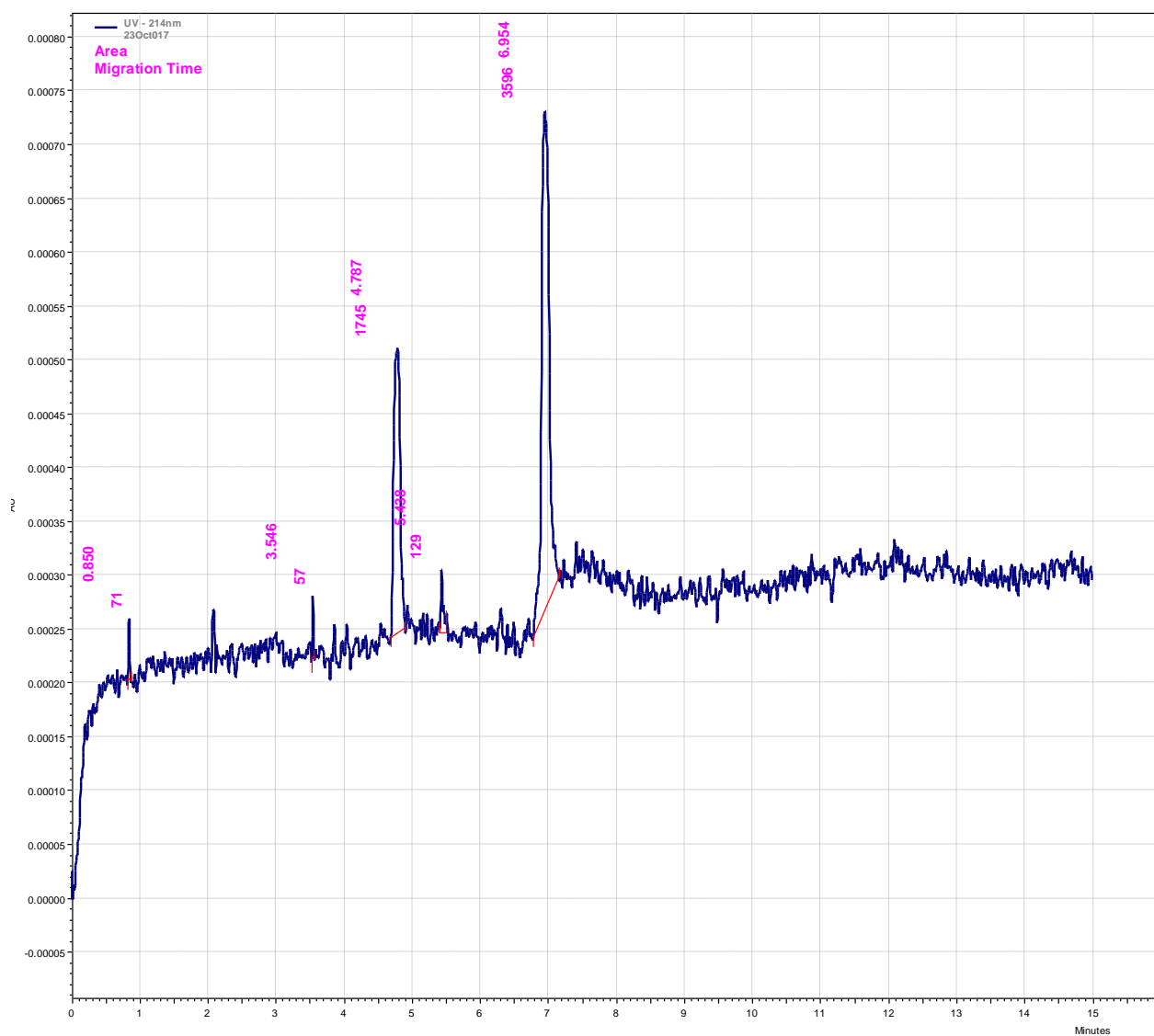
- 1) Donkor, K. Chemistry 3170 Instrumental Analysis Laboratory Manual, Fall 2024
- 2) Baker, D. R. Capillary Electrophoresis; John Wiley & Sons, Inc.: New York, 1995.
- 3) (EFSA Panel on Food Additives and Nutrient Sources added to Food). Scientific Opinion on the re-evaluation of aspartame (E 951) as a food additive. EFSA Journal. 2013 Dec;11(12):3496

## Appendix

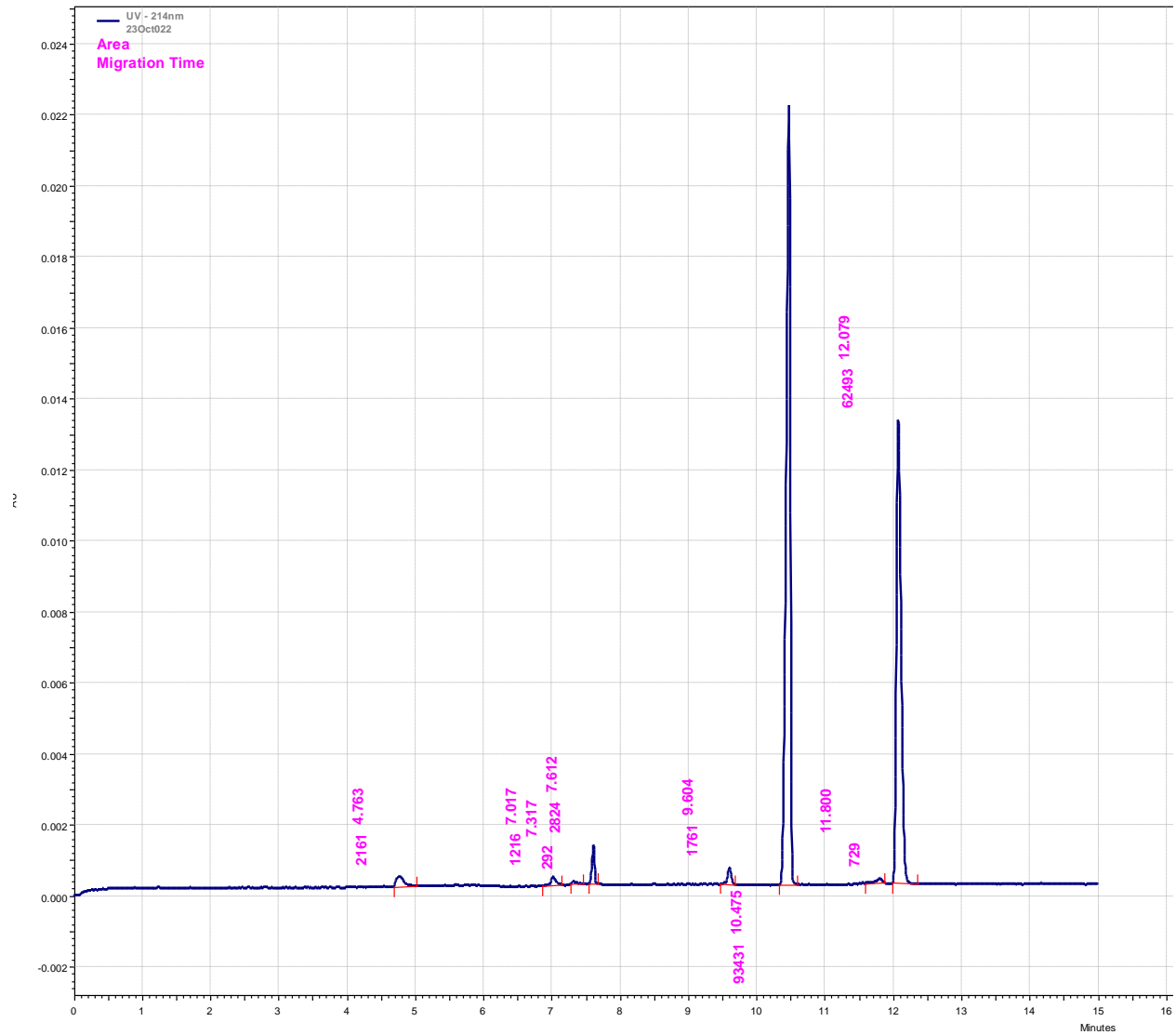
n =	6							
			$x_i$	$y_i$	$x_i y_i$	$x_i^2$	$d_i$	$d_i^2$
			0	0	0	0	502	252195
Unknowns			100	3596	359600	10000	971	943322
1216			200	4360	872000	40000	-1392	1936816
1170			300	7909	2372700	90000	-970	940198
1135			400	11724	4689600	160000	-282	79288
			500	16302	8151000	250000	1169	1367675
		Sums	1500	43891	16444900	550000		5519493.28
			D =	1050000	$S_y =$	1174.68		
		Method of least squares						
			slope =	31.27	intercept =	-502.19		
			$R^2 =$	0.9688	$S_y^2 =$	1379873.32		
			$S_m =$	2.81	$S_b =$	850.17		
		Measured y value =	1173.67		k =	3		
		derived x =	53.6	$s_x =$	31.9			

Appendix 1. Uncertainty table for the method of least squares used to generate the calibration curve (Figure 1).





Appendix 2. Electropherogram obtained from CE analysis of standard 1.



Appendix 3. Electropherogram obtained from CE analysis of the first diet sprite sample.