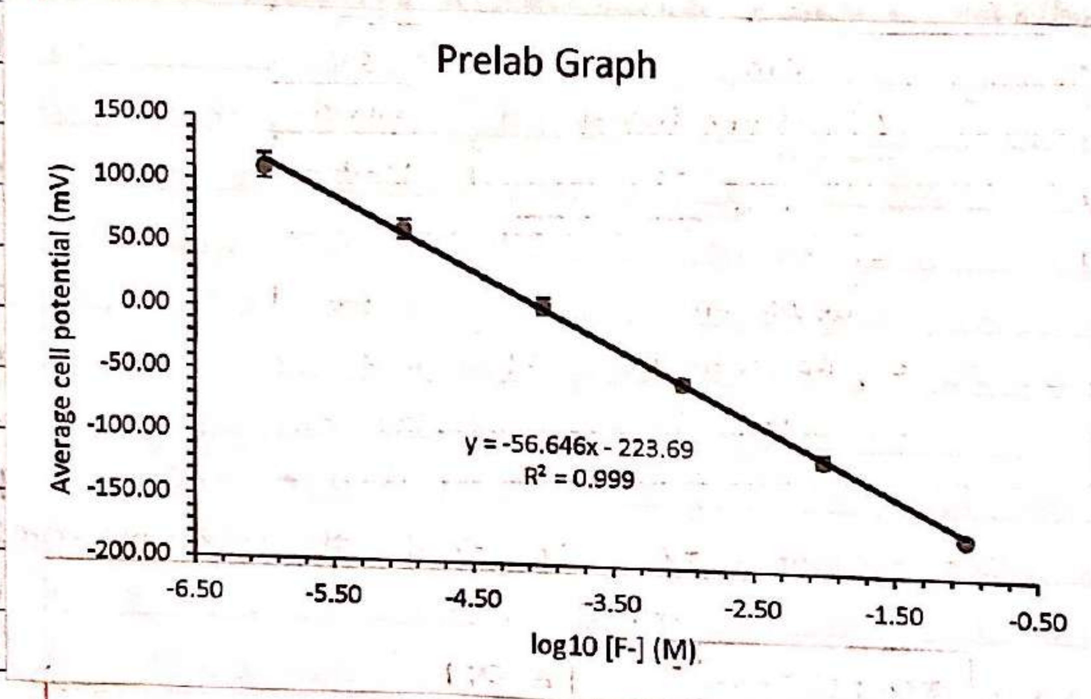
Lab 4
Potentiometric Determination
10/8/2019

Partner: Linh Nguyen

Example Data for Prelab			
Fluoride Standards		Cell Potential (mV)	
Concentration (M)	Log[F-] (M)	Average (n = 5)	Std. Dev
0.000001	-6	111	9.8
0.00001	-5	63.8	7.8
0.0001	-4	5.5	6.2
0.001	-3	-52.9	4.4
0.01	-2	-111.1	5.1
0.1	-1	-168.9	3.5



$\%w\ NaF = \frac{8.5136\times10^{-4}g}{0.2215g}\times100\% = 0.38\%$

Sample weighed (g)	0.2215
Volume dissolution (mL)	100
n	5
Average response (mV)	-14.5
Error in response (mV)	5.2
Concentration of F- (M)	0.000202762
Molar mass of F- (g/mol)	18.998
Error in molar mass (g/mol)	0.0000005
Mass of F- (g)	0.000385208
Weight % of F- (%)	0.17

$y = -56.646x - 223.69$

$56.646x = -223.69 - y$ ← avg. response)

$x = \frac{-223.69 + (-14.5)}{56.646}$

$x = \log_{10}[F^-]$

$[F^-] = 10^{\frac{-223.69+14.5}{56.646}}$

$[F^-] = 0.000202762$ M.

Mass of F^- = $0.000202762 \frac{mol}{L}(0.1L)\times\left(\frac{18.998g}{mol}\right) = 0.000385g$

~~[NaF] = 0.000202762M.~~

~~Mass = 0.000202762~~ $\frac{mol}{L}(0.1L)\left(\frac{41.988g}{mol}\right)$
$= 8.5136\times10^{-4}g$

Weight % F^- = $\frac{0.000385g}{0.2215g}\times100\% = 0.17\%$

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LINEST FUNCTION			
m	-56.6457	-223.6933	b
um	0.8984	3.4987	ub
R^2	0.9990	3.7582	sy
Fisher value	3975.6542	4.0000	df
regression ss	56152.8966	56.4968	residual ss

95% CI	
t table	2.7764
t*um	2.4943
t*ub	9.7140
t* error in average response , U_r	14.4375 ← U_r
numerator value	209.1933
error in numerator e_{num}	17.4012
denominator value	-56.6457
num/denom X	-3.6930
error num/denom e_x	-0.3476
Concentration of F- (M)	0.00020
error in concentration (M)	-0.00016

$$x \pm e_x = \frac{(-223.69 \pm tu_b) - (-14.5 \pm t^* \text{error average response})}{56.646 \pm tu_m}$$

$e_{num} = \sqrt{(tu_b)^2 + (tu_r)^2} = 17.4012$ ✓

$e_{deno} = 2.4943$

$$\frac{e_x}{X} = \sqrt{\left(\frac{17.4012}{(-209.19)}\right)^2 + \left(\frac{2.4943}{-56.6457}\right)^2}$$

$e_x = \pm 0.3476$

$y = 10^x$

$\hookrightarrow \frac{e_y}{y} = (\ln 10) e_x$

$e_y = 0.000202762 (\ln 10)(0.3476)$

$e_y = \pm 0.00016$

$y = 0.000202762$

$e_x = 0.3476$

$[F^-] = 0.00020 \pm 0.00016$ M

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Actual concentration of stock solution

→ 1015 ppm ± 3 ppm

Two-fold serial dilution

Number of dilutions	1	2	3	4	5
Concentration (ppm)	20.3	10.15	5.075	2.538	1.269

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Objective

This experiment is conducted to make the students used to using the LabQuest device in order to determine the ionic strength of certain ions. In this lab, we will determine the fluoride potentiometric in different several samples.

Introduction

Some methods learnt from previous lab will be implemented in this experiment, such as ~~x~~-fold ~~dil~~ serial dilution. In this lab specifically, a calibration curve is created in order to determine the unknown concentration, based on its potentiometric measurements from the LabQuest device.

Procedure

A) Prepare the standard calibration solutions.

1. All dispensing of unknowns and sample are done by TA.

2. Prepare 20-ppm F^- from the stock solution (2.00 mL of 1000-ppm stock solution diluted into 100-mL volumetric flask)

3. The dilution is prepared by using the TISAB solution.

4. From that 20-ppm solution, prepare 10-, 5-, 2.5- and 1.25-ppm calibrating fluoride solutions by 2-fold serial dilutions.

5. Use the actual values of ppm (the nominal concentration is just as a guide).

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First Unknown

Mass of beaker (g)	~~120.8300~~ 120.3800
Mass of beaker + toothpaste (g)	120.6024
Mass of toothpaste (g)	0.2224

Unknown data	
Average cell potential (mV)	11.2
log10 [F-] (ppm)	0.536
[F-] (ppm)	3.437
weight % of F- in toothpaste (%)	0.155
error in weight % of F- in toothpaste, e_w (%)	0.053
Original [F-] in toothpaste (ppm)	1545
error in original [F-] in toothpaste, e_g (ppm)	532

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B) First Unknown: Obtain & Prepare the Toothpaste Sample for Analysis.

1) Obtain a toothpaste sample from the instructor

2) A clean, dry and pre-weighed 250-mL beaker should be used and 0.20 to 0.25g of toothpaste will be added.

3) Weigh the beaker with the toothpaste to determine the net weight of the toothpaste sample.

4) Add 50mL TISAB.

5) Boil the solution gently for 2-3 minutes

6) Filter the suspension by gravity filtration using #1 or #5 filter paper.

7) After cooling, transfer the solution to a 100-mL volumetric flask and make up to volume with TISAB solution.

C) Second Unknown: Obtain the second F⁻ unknown and note its label.

1) Dispensing of unknowns & stock solutions should be done by the TA only.

2) Use the clean, dry vessel to place the second unknown, which will be analyzed as is (no dilution).

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Standard Solution		1	2	3	4	5
concentration (ppm)						
Potential E(mV),	1		-278.2	-264.8	-249.1	-260.5
(replicates)	2		-267.6	-263.3	-280.6	-256.0
	3			-265.3	-257.9	-256.0
	4			-269.9	-257.9	-250.0
	5			-263.5	-253.7	-246.1

copied the values wrongly from partner.

Unknown #		1st	2nd
Potential E(mV),	1	11.0	no 2nd unknown needed.
(replicates)	2	11.4	
	3	10.9	
	4	11.2	
	5	11.6	

Standard solution		1	2	3	4	5
Concentration (ppm)		20.3	10.15	5.075	2.538	1.269
Potential E(mV)	1	-33.0	-15.2	-0.15	15.8	39.0
(replicates)	2	-33.2	-15.1	0.00	15.0	38.9
	3	-33.4	-15.9	-0.30	16.3	39.5
	4	-33.2	-16.0	-0.18	16.1	40.1
	5	-33.3	-15.0	-0.20	16.0	39.7

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D) Making Potentiometric Measurements with ISE

1) Transfer ~20 mL of each standard solution into a clean, dry 50-mL beaker

2) Be sure the solution is well stirred and ~~record the~~ to observe the cell potential E (measured in mV)

3) The reading will drift for about 1 to 3 mins, wait for the signal to stabilize.

4). Make 4 to 5 replicate measurements for all standards, being careful not to cross-contaminate the solutions.

5) Rinse the electrode with small volumes of the next solution to be tested.

6) Repeat these steps for both unknown sample solutions.

7) Make 4-5 replicate measurements for both unknown sample solutions.

8) Make sure the voltage of unknown samples falls within the voltage range of the standards; or else, preparation & measurement of additional calibrating solutions may be needed, as extrapolation is not a good idea.

9) Dispose all solutions as indicated by the instructor. Rinse all glassware and clean the work area. Return all glassware.

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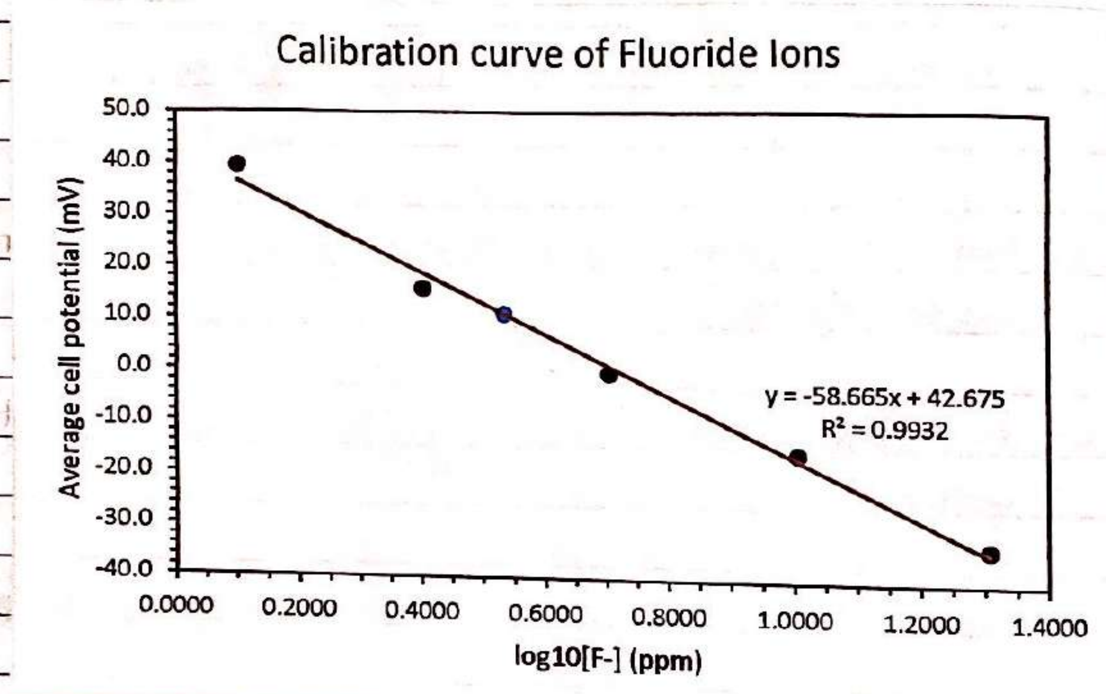
Analysis & Results

The analysis and extra calculations were done in excel file and it was included together with this lab's summary report. The standard calibration curve is as shown below. It follows the normal Nernstian operation, with a slope within -59 ± 4 mV. The blue point is the ~~fluoride content~~ toothpaste sample's result.



Conclusion

The toothpaste sample's cell potential measurement falls within the standard calibration curve, in which the concentration of fluoride ions in the toothpaste can be determined, which was 1545 ± 532 ppm.