**Summary Report**

The objective of this experiment was to determine the concentration of fluoride ions in the toothpaste sample by using the standard calibration curve plotted. The curve plotted was a graph of average cell potential in millivolt (mV) unit against the concentration of fluoride in the solution in logarithmic scale in parts per million (ppm) unit. The standard calibration curve was plotted by preparing 5 different concentration of fluoride solutions. 2 mL of the stock solution was pipetted into a 100-mL volumetric flask and was diluted with TISAB solution up to its mark. From the first dilution, a two-fold serial dilution was made for four times, to have a total of 5 concentrations. 25 mL of the previous solutions were pipetted into a 50-mL volumetric flask and was diluted with TISAB solution up to its mark. Each solution was well stirred and 20 mL of each solution were transferred into a clean and dry 50-mL beaker at a time. A fluoride ion selective combination electrode, which was connected to a LabQuest 2, was placed into each solution to measure the cell potential. The electrode was rinsed with a small volume of the solutions to be tested before actually inserting it into the beaker. 5 replicate measurements were taken for all standard solutions, by making sure to record each measurement after the signal on LabQuest 2 had stabilized, after 1 – 3 minutes of inserting the electrode into the solutions. From there, the replicate measurements of cell potential were averaged for each standard concentration and plotted against their concentrations. The concentrations of each standard solution were as tabulated in **Table 1** and the replicate measurements of cell potential for each concentration were as shown in **Table 2**.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **Concentration of stock solution (ppm)** | 1015 | | | | |
| **Error in concentration of stock solution (ppm)** | 3 | | | | |
| **Number of dilutions** | 1 | 2 | 3 | 4 | 5 |
| **Concentration of the solution (ppm)** | 20.30 | 10.15 | 5.075 | 2.538 | 1.269 |
| **Error in concentration (ppm)** | 0.09 | 0.05 | 0.024 | 0.013 | 0.007 |

Table 1: Concentrations of two-folded serial dilutions

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Concentration (ppm)** | **log10 [F-] (ppm)** | **Replicate measurements of cell potential (mV)** | | | | | | | |
| **1** | **2** | **3** | **4** | **5** | **Average** | **Standard deviation** | **RSD (ppt)** |
| 1.27 | 0.1034 | 39.0 | 38.9 | 39.5 | 40.1 | 39.7 | 39.4 | 0.5 | 12.6 |
| 2.54 | 0.4044 | 15.8 | 15.0 | 16.3 | 16.1 | 16.0 | 15.8 | 0.5 | 31.8 |
| 5.08 | 0.7054 | -0.15 | 0.00 | -0.30 | -0.18 | -0.20 | -0.17 | 0.11 | -653.8 |
| 10.15 | 1.0065 | -15.2 | -15.1 | -15.9 | -16.0 | -15.0 | -15.4 | 0.5 | -30.6 |
| 20.30 | 1.3075 | -33.0 | -33.2 | -33.4 | -33.2 | -33.3 | -33.2 | 0.1 | -4.5 |
| 3.44 | 0.5362 | 11.0 | 11.4 | 10.9 | 11.2 | 11.6 | 11.2 | 0.3 | 25.5 |

Table 2: Replicate measurements of cell potential for each concentration

To prepare for the toothpaste sample, a 0.20 – 0.25 g of sample was obtained from the instructor by weighing the sample in a dry and pre-weighed beaker, as shown in **Table 4,** with its error in mass. 50 mL of TISAB solution was added and gently boiled for several minutes. It took more than 3 minutes as suggested by the lab manual, as it took longer to see some vapor formed on the side of the beaker to indicate the boiling of solution. The suspension was then filtered by gravity filtration using #1 filter paper. The solution was transferred quantitatively into a 100-mL volumetric flask after cooling and diluted by TISAB solution up to its mark. To measure the solution’s cell potential, the same procedure conducted for the standard solutions were applied. 5 replicate measurements were obtained and averaged. The logarithmic concentration of the fluoride ions in the diluted toothpaste sample was determined by using the equation of the best-fit linear line obtained from the standard calibration curve, as shown in **Table 3**. The concentration was tabulated in **Table 2** as highlighted in orange and as blue point in **Figure 1**, the graph of the calibration curve with the best-fit line. The original concentration of fluoride ions in the toothpaste sample in ppm with its error and the weight % of fluoride ions in the toothpaste with its error were then determined, as tabulated in **Table 5**. A second unknown was not examined as instructed by the TA.

Figure 1: Calibration curve of average cell potential versus the logarithmic concentration of fluoride ions in the standard solutions

|  |  |  |  |
| --- | --- | --- | --- |
| **Linest output** | | | |
| **m (mV/ppm)** | -58.6653 | 42.67538 | **b (mV)** |
| **um (mV/ppm)** | 2.800547 | 2.307485 | **ub (mV)** |
| **R2** | 0.99321 | 2.665954 | **sy (mV)** |
| **Fisher value** | 438.8096 | 3 | **d.f** |
| **regression SS** | 3118.756 | 21.32193 | **residual SOS** |

Table 3: LINEST output of the best-fit linear line of the calibration curve

|  |  |
| --- | --- |
| **Mass of beaker (g)** | 120.3800 |
| **Mass of beaker + toothpaste (g)** | 120.6024 |
| **Mass of toothpaste (g)** | 0.2224 |
| **Error in mass, es (g)** | 0.0014 |

Table 4: Mass of toothpaste sample

|  |  |
| --- | --- |
| **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, ew (%)** | 0.053 |
| **Original [F-] in toothpaste (ppm)** | 1545 |
| **error in original [F-] in toothpaste, eg (ppm)** | 532 |

Table 5: The original concentration of fluoride ions in the toothpaste sample

By referring to **Figure 1**, it was a standard calibration curve of concentration of fluoride ions in solutions. A linear trendline was added to the curve with every point’s standard deviation as error bar plotted. However, the error bars were too small that they were not visible in the figure. The slope of the trendline was -58.6653 mV (referring to Table 3), where it laid within the range of -59 ± 4 mV of a normal Nernstian operation. This means that for every concentration, the electrode reads a measurement of cell potential that follows the following equation:

E = k + \* log10[F-], where k is a constant, n is the ionic charge with its sign, and where the temperature is presumed to be 25 degree celcius.

All errors in calculations were determined as 95% confidence interval Student’s t-test. The formula used to calculate each measurement were as shown from **Table 6 – 10**.

|  |  |
| --- | --- |
| **Student's t-value 95% C.I. error analysis** | |
| **uy (mV)** | 0.128 |
| **ub (mV)** | 2.307 |
| **um (mV/ppm)** | 2.801 |
| **t value (C.I. 95%)** | 3.182 |
| **t\*uy (mV)** | 0.408 |
| **t\*ub (mV)** | 7.343 |
| **t\*um (mV/ppm)** | 8.913 |
| **(log10 [F-])\*m (mV)** | -31.455 |
| **ea (mV)** | 7.355 |
| **log10 [F-] (ppm)** | 0.536 |
| **ex (ppm)** | 0.150 |
| **[F-] (ppm)** | 3.437 |
| **ef (ppm)** | 1.183 |
| **Original [F-] in toothpaste (ppm)** | 1545 |
| **eg (ppm)** | 532 |

Table 6: The calculations of each error in measurement that were done in Excel

|  |  |
| --- | --- |
| **Formula used** | |
| **[1st dilution (ppm)]** | (2 mL/100 mL)\*[stock solution (ppm)] |
| **[2nd-5th dilution (ppm)]** | (25 mL/50 mL)\*[previous solution (ppm)] |
| **Error in concentration (ppm)** | |
| **Equation** | **Error calculation formula** |
| **[First dilution ± ey ppm] = (2 ± 0.006 mL / 100 ± 0.08 mL)\*** | ey ppm = [first dilution ppm] \* SQRT((0.006mL/2mL)^2 + |
| **([stock solution] ± 3 ppm)** | (0.08mL/100mL)^2 + (3 ppm / [stock solution ppm])^2) |
| **[Second to fifth solution ± ez ppm] = (25 ± 0.03 mL / 50 ± 0.05 mL) \*** | ez (ppm) = [second to fifth solution(ppm)] \* SQRT((0.03mL/25mL)^2 + (0.05mL/50mL)^2  + |
| **([previous solution] ± previous solutions' error ppm)** | (previous solutions' error (ppm) / [previous solution (ppm)])^2) |
| **[solution (ppm)] means the concentration of the solution in ppm** | |
| **General error propagation formula** | |
| **Equation** | **Error propagation** |
| y = a + b - c | ey = SQRT(ea^2 + eb^2 + ec^2) |
| y = a\*b/c | ey = y\*SQRT[(ea/a)^2 + (eb/b)^2 + (ec/c)^2] |
| y = 10^x | ey = y\*[ln(10)]\*ex |

Table 7: Formula used to calculate values in Table 1

|  |  |
| --- | --- |
| **Type of apparatus** | **Uncertainty (mL)** |
| 2 mL pipette | 0.006 |
| 25 mL pipette | 0.03 |
| 50 mL volumetric flask | 0.05 |
| 100 mL volumetric flask | 0.08 |

Table 8: Tolerances in the apparatus used in this experiment

|  |  |
| --- | --- |
| **Formula** | |
| **log10[F-] (ppm)** | LOG10(concentrations) function in excel |
| **Average cell potential (mV)** | AVERAGE(values) function in excel |
| **Standard deviation of cell potential (mV)** | STDEV(values) function in excel |
| **RSD (ppt)** | Standard deviation/Average \* 10^3 ppt |
| **LINEST Output** | LINEST(y values,x values, 1,1) function in excel |

Table 9: Formula used to determine the values in Table 2 and 3

|  |  |
| --- | --- |
| **Formula** | |
| **uy (mV)** | Standard deviation/SQRT(number of data points) |
| **ub (mV) and um (mV/ppm)** | From LINEST output: LINEST(y values, x values, 1, 1) |
|  | function in excel |
| **t value (C.I. 95%)** | TINV(0.05,df) function in excel, df from LINEST output |
| **log10 [F-] (ppm)** | (Average cell potential - intercept) / slope |
| **[F-] (ppm)** | 10^(log10 [F-]) |
| **1 ppm = 1 g of solute/(10^6) g of solution** | |
| **Original [F-] in toothpaste (ppm)** | [F-] g /(10^6 g)\*(1g/mL)\*(100 mL)/mass of toothpaste (g) \*(10^6) |
| **Weight % of [F-] in toothpaste (%)** | [F-] g /(10^6 g)\*(1g/mL)\*(100 mL)/mass of toothpaste (g) \*(100%) |
| **Error Propagation Formula** | |
| y = mx + b, y = average cell potential (mV), x = log10 [F-] in ppm, | |
| m,b = slope (mV/ppm) and intercept (mV) of calibration curve respectively | |
| **Equation** | **Error propagation** |
| (y ± t\*uy) - (b ± t\*ub) = (m\*x) ± ea | ea = SQRT((t\*uy)^2 + (t\*ub)^2) |
| x ± ex = ((y - b) ± ea) / (m ± t\*um) | ex = x\*SQRT((ea/(y-b))^2 + (t\*um/m)^2) |
| [F-] ± ef = 10^(x ± ex) | ef = [F-]\*(ln 10)\*ex |
| Original [F-] ± eg = ([F-] ± ef)/(10^6)\*(1)\* | eg = Original [F-]\*SQRT((ef/[F-])^2 + (0.08/100)^2 + |
| (100 ± 0.08)/(mass of toothpaste ± es)\*(10^6) | (es/mass of toothpaste)^2) |
| Weight % [F-] ± ew = ([F-] ± ef)/(10^6)\*(1)\* | ew = Weight % [F-]\*SQRT((ef/[F-])^2 + (0.08/100)^2 + |
| (100 ± 0.08)/(mass of toothpaste ± es)\*(100) | (es/mass of toothpaste)^2) |
| **General Propagation Formula** | |
| **Equation** | **Error propagation** |
| y = a + b - c | ey = SQRT(ea^2 + eb^2 + ec^2) |
| y = a\*b/c | ey = y\*SQRT[(ea/a)^2 + (eb/b)^2 + (ec/c)^2] |
| y = 10^x | ey = y\*[ln(10)]\*ex |

Table 10: Formula used to find the values in Table 6