Standardization of a sodium hydroxide (naoh) solution with unknown concentration

Standardization of a Sodium Hydroxide (NaOH) Solution with Unknown Concentration by Titration of Potassium Hydrogen Phthalate (KHP)

Results

Mass KHP (± 0.0001) (g)	KHP (mol)	Initial NaOH (± 0.05) (mL)	Final NaOH (± 0.05) (mL)	NaOH Delivered (± 0. 1) (mL)	NaOH Molarity (± 0. 1) (M)
0.5006	0.00245 1	3. 29	30.49	27. 20	0.9011
0.4894	0.00239 6	2. 97	29.59	26. 62	0.9002
0.4995	0.00244 6	14.69	41.82	27. 13	0.09015
0.4909	0.00240 4	9. 41	36.02	26. 61	0.09032

Table 1. Titration data from Grace Liu

Titration data for four trials for approximately 0. 50 g of KHP titrated with NaOH of unknow concentration. Average NaOH molarity calculated to be 0. 0915 M.

Table 2. Titration data from Yu-Ting Tseng

Mass KHP (± 0. 0001) (g)	KHP (mol)	NaOH Delivered (± 0. 1) (mL)	NaOH Molarity (± 0. 1) (M)
0. 5006	0. 002451	26. 05 mL	0. 09514
0. 4894	0. 002396	25. 81 mL	0. 09510
0. 4995	0. 002446	26. 11 mL	0. 09513
0. 4909	0. 002404	25. 98 mL	0. 09521

Titration data for four trials for approximately 0. 50 g of KHP titrated with NaOH of unknow concentration. Average NaOH molarity calculated to be

0. 09515 M. Titrations performed by Yu-Ting Tseng and taken for comparison

The standard deviation for both data sets was calculated by the method shown below using Equation 1. This sample calculation uses the data show in Table 1 above.

$$s = \sum (x_i - \bar{x})^2 \; n - 1 \quad (1)$$

$$s_1 = (0.09011 - 0.09015)^2 + (0.09002 - 0.09015)^2 + (0.09015 - 0.09015)^2 + (0.09032 - 0.09015)^2 \; 4 - 1$$

$$s_1 = 1.292 \times 10^{-4}$$

$$s_2 = 4.203 \times 10^{-5} \; M$$

As shown, the standard deviation for data set 1 is larger than that of data set 2. Additionally, a confidence interval was calculated for data set 1 to determine the range in which we are 95% sure the “ true” value with fall in. The average for data set 1 was 0. 09015 M with a standard deviation of

$$1.292 \times 10^{-4}$$

M (n= 4). The *t* value at 95% confidence is 3. 182 for three degrees of freedom [(1)] . The confidence interval for this data set was calculated using these values and Equation 2 as shown below.

$$\mu = \bar{x} \pm \frac{ts}{\sqrt{n}} \quad (2)$$

$$\mu = 0 . 09015 \pm (3 . 182) (1 . 292 \times 10 – 4) 4$$

$$\mu = 0 . 09015 \pm 2 . 056 \times 10 – 4 M$$

The confidence interval for data set one is $0 . 09015 \pm 2 . 056 \times 10 – 4 M$

, meaning that we can be 95% sure that the " true" value for the concentration of the NaOH solution used in the titrations is within $2 . 056 \times 10 – 4 M$

of our average value.

Next, an F test was performed to determine if the standard deviations of the two data sets are statistically different. This is done using Equation 3 and the previously calculated standard deviations as shown below.

$$F = s 1 2 s 2 2 \quad (3)$$

$$F calc = (1 . 292 \times 10 – 4) 2 (4 . 203 \times 10 – 5) 2$$

$$F calc = 9 . 454$$

The table value of F for 3 degrees of freedom on both data sets is 9. 28 [1] . Since $F_{calc} > F_{table,}$ the two standard deviations are statistically different at 95% confidence, we reject the null hypothesis that the two sets of measurements are drawn from populations with the same population standard deviation [1] . We then use the proper t test to determine if the results from the two data sets agree with each other within experimental

error. This is done using Equation 4 below and the proper average and standard deviation values calculated above, with n = 4 for both data sets

$$t = \frac{|\bar{x}_1 - \bar{x}_2|}{\sqrt{\frac{s_1^2}{n_1} + \frac{s_2^2}{n_2}}} \quad (4)$$

$$t_{calc} = \frac{|0.09015 - 0.09514|}{\sqrt{\frac{(1.292 \times 10^{-4})^2}{4} + \frac{(4.203 \times 10^{-5})^2}{4}}} = 73.45$$

In order to find the proper table value for *t* we must determine the degrees of freedom. This is done using Equation 5 as shown below.

$$df = \frac{\left(\frac{s_1^2}{n_1} + \frac{s_2^2}{n_2}\right)^2}{\frac{\left(\frac{s_1^2}{n_1}\right)^2}{n_1 - 1} + \frac{\left(\frac{s_2^2}{n_2}\right)^2}{n_2 - 1}} \quad (5)$$

$$df = \frac{\left(\frac{(1.292 \times 10^{-4})^2}{4} + \frac{(4.203 \times 10^{-5})^2}{4}\right)^2}{\frac{\left(\frac{(1.292 \times 10^{-4})^2}{4}\right)^2}{4 - 1} + \frac{\left(\frac{(4.203 \times 10^{-5})^2}{4}\right)^2}{4 - 1}} = 3.627$$

When calculating degrees of freedom, if the result is not a whole number, we always round down to give a more conservative estimate. Thus, there are 3 degrees of freedom involved in our calculation of *t* . With this number calculated, we can determine the table value of *t, t_{table} ,* at 95% confidence, which is 3. 182 [(1)] . We find that $t_{calc} > t_{table}$, therefore we can conclude at 95% confidence that the difference between the two data sets is statistically significant.

Discussion

As stated in the results section above, there is a statistically significant difference between the results of the two data sets, and a rather large one: we know that this difference is not by chance, it is due to experimental error, both systematic and random.

One source of systematic error that is often associated with titrations is incorrect buret readings. Readings differ person to person, so I may always read slightly higher than the exact volume, whereas my partner could read consistently lower than the exact volume. This discrepancy could account for inaccuracy in the measurements and calculations in one data set, as well as differences between two data sets.

Another source of systematic error is reagent purity. If either reagent had aged at all prior to the experiment, it could lead to deviation from the calculated values. For example, if the KHP was not as pure as expected (i. e. absorbed water, etc.), a smaller volume of NaOH would be needed to neutralize it, thus resulting in an experimental molarity that is lower than the molarity calculated beforehand. This is an example of why it is very important to have a good primary standard. A proper primary standard should: be at least 99. 9% pure; not decompose under ordinary storage; be stable when dried by heat or vacuum; have a high molecular weight; and it should be relatively cheap and readily available.

Quite possibly the biggest source of systematic error in titrations is end point determination. Since end point is done visually, there can be a large discrepancy in when a titration is stopped from person to person. Each person has a different ability to see a slight change in color, so a “ faint pink”

will look different to each person and will result in a different amount of titrant delivered based on who is performing the experiment, which will result in a difference in the molarity of the NaOH solution calculated after the experiment. This error, along with the two previously stated, are, more likely than not, present in both mine and my partner's results.

Conclusion

Through a set of four titrations of KHP with a NaOH solution of unknown concentration, we were able to deduce the concentration of this NaOH solution. This NaOH solution will be used in further experiments, thus it is very important to deduce its concentration. As with all laboratory experiments, there are sources of error in these titrations. These errors include the systematic errors of buret reading, primary standard purity, and endpoint determination. Random errors are also present; these include reagent evaporation and contamination, and variations in volumetric glassware and measuring instruments due to variations in temperature. This experiment also promoted the development of important laboratory techniques including titration, weighing by difference, and measurement or reagents, among other important techniques, all of which will be used in future laboratory experiments.

References

(1) Harris, D. C.; Lucy, C. A. *Quantitative Chemical Analysis* ; W. H. Freeman: New York, 2016.