Determination of Percent Chloride in an Unknown Chloride Mixture by Ion Exchange and Back-Titration

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CONTENTS

Appendices

ABSTRACT

Ion exchange is a process in which ions bonded to an exchange media are replaced with different ions that come into contact with the exchange media. This method is useful for replacing interfering ions with non-interfering ones as well as for concentrating ions from dilute solutions, and is commonly used in water softeners. The percent composition of chloride in the unknown chloride mixture 1801 was determined using ion exchange with hydroxide ions, and then measuring the hydroxide ion concentration by over-titration with potassium hydrogen phthalate, $KHC_8H_4O_4$ (aq) (abbreviated to KHP), and back-titrated with sodium hydroxide, NaOH (aq). The average calculated experimental mass percent of chloride ions in the unknown mixture was 34.3%, and a true value to be in between 25% and 43% at the confidence level of 90%. The results and methods were compared with those from an earlier experiment determining the percent chloride of the same chloride using gravimetric analysis. The data gathered in this experiment may affect which methods are chosen to determine the percent compositions of other compounds in future experiments.

EXPERIMENTAL METHODS

A column was filled with Amberlite IRA 910 ion-exchange resin, and saturated with hydroxide ions (from a sodium hydroxide solution). Three samples of a solution of unknown mixture of chloride were drained through the column, each displacing hydroxide ions that were over-titrated with standardized potassium hydrogen phthalate and back-titrated with standardized sodium hydroxide. The specific procedure followed was based on the procedure stated in “Determination Of Percent Chloride By Ion Exchange and Back-Titration,” from *The Official Cooper Union General Chemistry Laboratory Guide* (1). The discussion behind the methods used, such as keeping the solution slightly acidic and rinsing with a dilute acidic solution, are explained in more detail in the discussion section. Below is a summary of the tasks completed.

A 0.1M sodium hydroxide solution was prepared, and four samples were standardized by titration with potassium hydrogen phthalate. A column with an inbuilt filter was filled with approximately 35mL of Amberlite IRA 910 resin and saturated with hydroxide ions by washing with stock sodium hydroxide. Four samples of solution with an exact, known concentration of the unknown chloride mixture were prepared. Each sample unknown solution was drained through the column and eluted with multiple bed volumes of deionized water. The eluent solution was then over-titrated with potassium hydrogen phthalate and back-titrated with the sodium hydroxide solution standardized in the beginning of the experimental procedure.

Four samples of the sodium hydroxide solution were standardized and four samples of the unknown chloride solution were analyzed by forward-titration and back-titration instead of three (from the lab manual procedure). A column with an inbuilt filter was used instead of a buret prepared with a glass wool filter. Due to a misunderstanding of the laboratory guide, rather than preparing one solution of unknown mixture and using 10.00mL aliquots as samples, one solution was prepared for sample 1 and another sample was prepared for samples 2 through 4; this is not, however, a source for error, as the process is identical for the different solutions. A gross error in the experimental procedure from the laboratory guide is that there is no instruction to mix the bottle before use in week three, after two weeks of being left to sit; this may cause an uneven concentration in the bottle and an unpredictable result on the overall calculation of percent chloride. Another potential source of error is that the basic eluent was over-titrated by far more than 3mL of titrant in samples 1 and 2, which may magnify error in reading the buret during the first titration. These errors are discussed in greater detail in the discussion section.

RESULTS

The standardization of the roughly 0.1M sodium hydroxide solution was performed using a solution of potassium hydrogen phthalate of known mass as the analyte and the prepared solution of sodium hydroxide as the titrant. The measured masses of potassium hydrogen phthalate and the volume of sodium hydroxide solution used to titrate the analyte are shown below.

Table 1. Masses of dry KHP and volume of NaOH necessary for titration

	Sample 1	Sample 2	Sample 3	Sample 4
Mass of dry KHP (g)	0.5022	0.4743	0.5009	0.4975
Volume of NaOH to to titrate KHP (mL)	23.11	21.90	23.20	22.50

The values from Table 1 were used to calculate the concentration of each standardized solution (Eq. 5), and mean and standard deviation of these solution concentrations were found (A.II.I). These values are displayed below.

Table 2. Calculated standardized NaOH concentrations and statistical summary

Sample 1	Sample 2	Sample 3	Sample 4
0.1064M	0.1061M	0.1057M	0.1083M
Mean	0.1066M		
Standard Deviation	0.001141M		

The measurements from the forward over-titration and back-titration of the solutions of the unknown chlorides are shown below. The unknown sample is the same as that in the gravimetric analysis (3).

Table 3. Mass of unknown chloride sample and volumes of titrant
for over-titration with KHP and back-titration with NaOH

	Sample 1	Sample 2	Sample 3	Sample 4
Mass of unknown (g)	0.3235*	0.3954	0.3954	0.3954
Volume KHP to overtitrate OH^- ions (mL)	8.11	13.18	5.55	4.85
Volume NaOH to back-titrate over-titrated KHP (mL)	28.1	44.9	17.71	14.34

* The chloride solution in sample 1 was different from the chloride solutions in samples 2, 3, and 4. See experimental section for more details.

The measurements from Table 3 were used to calculate the percent mass composition of chloride in the unknown samples (Eq. 6-8), displayed below. A statistical summary of these percent compositions is also displayed below (see A.II.II).

Table 4. Calculated percent mass composition of chloride and statistical summary

Sample 1	Sample 2	Sample 3	Sample 4
27.2%	43.49%	29.8%	36.9%

Sample mean	34.3% ± 3.68%
Sample standard deviation	7.35%
90% confidence interval for the mean	34% ± 9%

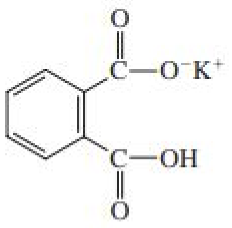
The sample mean of the calculated percent mass composition of chloride in the unknown was 34.3% ± 3.68%, and the sample standard deviation was 7.35%. The Q-test (see A.II.II) did not predict any statistically-unreliable data points, so no data was discarded. The confidence interval indicates that the actual percent composition of chloride in the unknown is between 25% and 43% at a 90% confidence.

DISCUSSION

Much of the insight on the discussion of the experimental method was based off of content from *Fundamentals of Analytical Chemistry* (2).

While the concentration of the prepared sodium hydroxide solution may have been calculated from the mass of the sodium hydroxide pellets used and the volume of the solution, a standardization of the solution was an important first step in the experimental methods to ensure accuracy of the solution's concentration. Sodium hydroxide is highly hygroscopic, and measuring the mass may include the mass of some water on the surface of the pellets.

Potassium hydrogen phthalate, commonly abbreviated KHP, was used to standardize the prepared sodium hydroxide solution and over-titrate the eluent. It has a molecular mass of 204.222g/mol (molar masses calculated from elemental molar masses found in *Fundamentals of Analytical Chemistry* (2)). An image of the structure of KHP is displayed below.



(Image courtesy of *Fundamentals of Analytical Chemistry* (2))

0.5g of potassium hydrogen phthalate is dissolved in 50mL of solution. The pH of this solution is 3.35 (see A.I.I. Eq. 3). Hydrogen phthalate is the weak monoprotic acid formed after the dissociation of phthalic acid. It is amphoteric, able to undergo either of the following hydrolysis reactions:

Reaction 1. $HC_8H_4O_4^-{}_{(aq)} + H_2O_{(aq)} \rightleftharpoons H_2C_8H_4O_{4\,(aq)} + OH^-{}_{(aq)}$

Reaction 2. $HC_8H_4O_4^-{}_{(aq)} + H_2O_{(aq)} \rightleftharpoons C_8H_4O_4^{2-}{}_{(aq)} + H_3O^+{}_{(aq)}$

The net ionic equation of the reaction between hydrogen phthalate and sodium hydroxide is shown below. The pH at the equilibrium point is 9.0 (see A.I.I. Eq. 4).

Reaction 3. $HC_8H_4O_4^-{}_{(aq)} + OH^-{}_{(aq)} \rightleftharpoons C_8H_4O_4^{2-}{}_{(aq)} + H_2O_{(l)}$

An over-titration using potassium hydrogen phthalate titrant followed by a back-titration using sodium hydroxide titrant was used because of the difficulty of determining the endpoint of the forward titration. Using the phenolphthalein indicator, it is easier to notice the endpoint of the titration when the color changes from colorless to pink (acid to base) than from pink to colorless (base to acid). Because the forward titration is of the latter type, a back-titration is used to determine exactly how much excess potassium hydrogen phthalate was reacted in the forward titration. Another reason for over-titration was to keep the stored solution in an acidic solution between weeks 2 and 3 of the experimental procedure; if the collected eluent solution (containing the sodium hydroxide ions that had been exchanged with chloride ions) was left basic, the sodium hydroxide ions would react with the glass of the container and with the carbon dioxide in the air, which would yield inaccurate results for a forward titration conducted the following week.

The likely sources of systematic and random error are, ranked in decreasing order of influence on the results, are: not mixing the sodium hydroxide solution before use in the third week, greatly over-titrating the eluent, and the tolerance of normal equipment operation. These are discussed further below.

The sodium hydroxide solution prepared and standardized in the first week was used in the third week. After sitting in a cabinet for two weeks, it is possible that a vertical gradient of sodium hydroxide concentration formed due to the differences in densities between sodium hydroxide and water. This most likely led to a lower-concentration aliquot near the top of the sodium hydroxide solution to be used during the back-titration. A lower concentration of sodium hydroxide would require more to be used as a titrant, which systematically increases the calculation of percent mass composition of chloride.

A gross error that may have greatly influenced the results is in the over-titration of the basic eluent solution. From the calculation in Eq. 7, only a few millimoles of potassium hydrogen phthalate (corresponding to roughly 1mL of 0.4001M potassium hydrogen phthalate) were necessary to titrate the eluent. The excess titrant in the first two samples greatly exceeded the 3mL volume stated by the laboratory manual: there was roughly 7mL and 12mL of extra titrant used in samples 1 and 2, respectively. In the second titration, 44.9mL of sodium hydroxide were needed to back-titrate this excess, over three times the amount necessary in sample 4 (Table 3). Because of the large ratio of concentrations of potassium hydrogen phthalate to sodium hydroxide solutions used in the titrations (0.4001M potassium hydrogen phthalate and 0.1066M sodium hydroxide; roughly 4:1), small errors in the measurements of the volumes in the forward titration with the more concentrated potassium hydrogen phthalate will be amplified into larger errors in the final calculation. This likely contributed to the result from sample 2 being the farthest from the mean.

Precise measurements were taken with the analytical balance to measure the mass of compounds, which have a tolerance of approximately 0.0002g (0.2mg) for the mass of potassium hydrogen phthalate and mass of unknown solution. Precise measurements were also taken with burets, which have a tolerance of approximately 0.05mL, for the volume of sodium hydroxide during the standardization, potassium hydrogen phthalate during the forward titration, and sodium hydroxide during the back-titration. However, these errors associated with the equipment are small and should not have a major impact on the results.

When compared to the results of the *Gravimetric Determination of Chloride in Unknown Chloride Mixture by Precipitation with Silver Nitrate* (3), the calculated percent mass composition determined doesn't show consistent results. The results are summarized in the table below; see Eq. 6-8 for the calculations of percent mass composition from this lab, and A.II.II. Both analyzed the unknown 1801 chlorine mixture.

Table 6. Summary of percent mass composition of chloride and statistical measures by precipitation gravimetric analysis and ion exchange and titration

	Gravimetric analysis			Ion exchange and titration			
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3	Sample 4
%Cl$^-$	49.83%	52.34%	54.01%	27.2%	43.49%	29.8%	36.9%
Mean %Cl$^-$	52.06% ± 1.22%			34.3% ± 3.68%			
St. dev.	2.11%			7.35%			
Confidence Interval (90% level)	52% ± 4%			34% ± 9%			

The calculated values in the first experiment were much more consistent than those of this experiment. Even though there were fewer samples, the standard deviation of the results from the first experiment is 2.11%, less than one-third of the standard deviation from this experiment (7.352%). The range is more extreme as well— in the gravimetric analysis, the range was less than 5%, while the range of the calculated percent chloride values in this experiment was roughly 16%. The smaller spread of the results of the gravimetric analysis indicates that it is the more reliable experiment.

Because the true percent mass composition of chloride in the unknown is unknown, nothing can be said for certain about the accuracy of these two tests. The 90% confidence intervals do not overlap, so the two experiments' results do not agree on a mean value for the percent chloride (to 90% confidence). However, the more erratic results of this experiment likely cause data obtained from the gravimetric analysis to be more reliable and preferred over the results from this experiment.

In addition to the decreased precision of this experiment's results, the more advanced equipment and more complex experimental procedure in this experiment makes the gravimetric analysis preferrable. The previous experiment only required three precise measurements, all using one piece of equipment (the analytical balance), while this experiment required two precise mass measurements and six precise volume measurements, requiring a buret and column as extra equipment. While this experiment did not have the same source of error of photosensitivity of the chemicals (as did the silver compounds in the gravimetric analysis), there was the potential source of error of leaving bases in glass containers or when exposed to air for long periods of time, and the potential source of error of having a standardized solution of sodium hydroxide settle over two weeks so that its concentration became uneven.

CONCLUSIONS

The calculated mean percent mass composition of chloride in the unknown mixture was 34.3% with a standard deviation of 7.35%. A 90% confidence interval of the true mean percent chloride in the mixture is $34\% \pm 9\%$. The range of the calculated percent masses , as well as the standard deviation, are both high, indicating that this method is not very precise. While accuracy of the experiment cannot be determined absolutely, the large spread of the data indicate poor reliability of this experimental method.

The results and procedure were compared to that of gravimetric analysis (3), which analyzed percent chloride of the same unknown mixture. It was concluded that because of the increased simplicity and precision of the earlier experiment, its method is preferred over the current one. The results from the two experiments did not agree with each other (the 90% confidence interval for the gravimetric analysis was $52\% \pm 4\%$, not overlapping the confidence interval from this procedure); the higher precision of the gravimetric analysis suggest that it is more likely to represent the true value than the ion exchange approach.

There were several flaws with this procedure. An error in the given procedure is that it does not mention to stir the sodium hydroxide before use; this is fixed by a simple addition to the laboratory guide, or if the solution is prepared right before use, both of which ensure homogeneity of the solution. Secondly, to fix the gross error of over-titrating by more than the recommended amount, the amount of potassium hydrogen phthalate necessary to titrate the eluent should be calculated before the titration to have a better idea of the true amount necessary so that the slow color change from pink to colorless is noticed.

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Paul Cucchiara was responsible for authoring the Abstract, Experimental Methods, Discussion, and Conclusion sections of this laboratory report, and served as editor for the sections written by Jonathan Lam.

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APPENDIX I
SAMPLE (REPRESENTATIVE) CALCULATIONS

A.I.I. Calculations for Concentration from Standardization

The estimated mass of sodium hydroxide necessary to create 500mL of 0.1M sodium hydroxide solution, and the estimated mass of potassium phthalate necessary to titrate 25mL of the 0.1M sodium hydroxide, were calculated using equations 1 and 2, respectively. These values were used as approximate values for the mass of sodium hydroxide pellets and potassium phthalate used to make the standardized sodium hydroxide solution.

[Eq. 1] 500mL 0.1M NaOH $\times \frac{1L}{1000mL} \times \frac{0.1 mol\ NaOH}{L} \times \frac{39.997g\ NaOH}{mol\ NaOH} = 2g\ NaOH$

[Eq. 2] 25mL 0.1NaOH $\times \frac{L}{1000mL} \times \frac{0.1 mol\ NaOH}{L} \times \frac{mol\ KHP}{mol\ NaOH} \times \frac{204.222g\ KHP}{mol\ KHP} = 0.5g\ KHP$

(molar masses were calculated from elemental molar masses found in *Fundamentals of Analytical Chemistry* (2))

The pH of the potassium hydrogen phthalate used to standardize the sodium hydroxide is calculated below using an ICE table. The K_a value is given as the second acid dissociation constant of o-phthalic acid in *Fundamentals of Analytical Chemistry* (2).

[Eq. 3] $[HC_8H_4O_4^{-}] = \frac{0.5g \times \frac{mol\ KHC_8H_8O_4}{204.222g}}{0.050L} = 0.049M$

Table 7. ICE table for dissociation of hydrogen phthalate

	$HC_8H_4O_4^{-}$ (aq)	H_2O (l)	$C_8H_4O_4^{2-}$ (aq)	H^+ (aq)
Initial	0.049M	N/A	0	0
Change	-x	N/A	+x	+x
Equilibrium	0.049M-x	N/A	x	x

$$K_a = \frac{[H^+][C_8H_4O_4^{2-}]}{[HC_8H_4O_4^{-}] - x} \approx \frac{x^2}{[HC_8H_4O_4^{-}]} = 3.91 \times 10^{-6}$$

$$pH = -log([H^+])$$

$$x = [H^+] = [C_8H_4O_4^{2-}] = \sqrt{K_a \times [HC_8H_4O_4^{-}]}$$

$$pH = -log(\sqrt{3.91 \times 10^{-6} \times 0.049M}) = 3.4$$

The pH of the potassium hydrogen phthalate at the equilibrium point during the titration with sodium hydroxide is shown calculated below using an ICE table. The direction of the reaction is flipped from how it is shown in reaction 3 so that the value for K_b can easily be used.

[Eq. 4] Volume NaOH = $0.5g\ KC_8H_4O_4 \times \frac{mol\ KHC_8H_4O_4}{204.222g} \times \frac{mol\ NaOH}{mol\ KHC_8H_4O_4} \times \frac{L}{0.1 mol\ NaOH} = 0.0\overline{2}45L$

Total volume solution = $0.0\overline{5}0L\ (KHP) + 0.0\overline{2}45L\ (NaOH) = 0.0\overline{7}45L$

At equivalence point, $[C_8H_4O_4{}^{2-}]$ = moles of titrated $HC_8H_4O_4{}^{-} = [HC_8H_4O_4{}^{-}]$

$$[C_8H_4O_4{}^{2-}] = [HC_8H_4O_4{}^{-}] = \frac{0.5g\ HC_8H_4O_4{}^{-} \times \frac{mol\ HC_8H_4O_4{}^{-}}{204.222g}}{0.0745L} = 0.0\overline{3}287M$$

Table 8. ICE table for reaction between hydrogen phthalate and hydroxide

	$C_8H_4O_4{}^{2-}$ (aq)	H_2O (l)	$HC_8H_4O_4{}^{-}$ (aq)	OH^{-} (aq)
Initial	$0.0\overline{3}287$	N/A	0	0
Change	-x	N/A	+x	+x
Equilibrium	$0.0\overline{3}287 - x$	N/A	x	x

$$K_b = \frac{K_w}{K_a} = \frac{1\times10^{-14}}{3.91\times10^{-6}} = 2.56 \times 10^{-8}$$

$$K_b = \frac{[HC_8H_4O_4{}^{-}][OH^{-}]}{[C_8H_4O_4{}^{2-}] - x} \approx \frac{x^2}{[C_8H_4O_4{}^{2-}]} = 2.56 \times 10^{-8}$$

$$x = [OH^{-}] = [HC_8H_4O_4{}^{-}] = \sqrt{K_b \times [C_8H_4O_4{}^{2-}]}$$

$$pH = 14 - pOH = 14 - (-\,log(\sqrt{2.56 \times 10^{-8} \times 0.0\overline{3}287M})) = 9.0$$

(Only the final result was rounded. Intermediate are displayed rounded arbitrarily for simplicity).

The concentration of the roughly-0.1M sodium hydroxide was calculated from the standardization with KHP using the following calculation.

[Eq. 5] Concentration NaOH (M) = $\frac{mol\ NaOH}{L} = \frac{mass\ KHP \times \frac{mol\ KHP}{204.22g\ KHP} \times \frac{mol\ NaOH}{mol\ KHP}}{(final\ volume\ NaOH - initial\ volume\ NaOH)}$

The calculation for the concentration of the sodium hydroxide solution calculated from the standardization of sample 1 is shown below as a representative calculation. The source of this data can be found in Table 1.

(Sample standardization 1) Concentration of Sample 1 = $\frac{0.5022g \times \frac{mol\ KHP}{204.22g\ KHP} \times \frac{mol\ NaOH}{mol\ KHP}}{0.02931L - 0.00620L} = 0.1064M$

A.I.II. Calculations for Percent Mass Chloride

The amount of excess potassium hydrogen phthalate was calculated from the volume of the standardized sodium hydroxide solution used in the back-titration. The molarity of the standardized solution used in the equation below is the average of the calculated concentrations of the standardized

[Eq. 6] Excess KHP (mol) = (final volume NaOH - initial volume NaOH) $\times \frac{0.1066 mol\ NaOH}{L} \times \frac{excess\ mol\ KHP}{mol\ NOH}$

The excess moles of potassium hydrogen phthalate can be used to calculate the amount of potassium hydrogen phthalate that reacted during the forward titration by subtraction:

[Eq. 7] Reacted KHP (mol) = ((total volume KHP) $\times \frac{0.4001 mol\ KHP}{L}$) - excess mol KHP

The mass percent chloride of the unknown can be calculated via stoichiometry using this value. The value is multiplied by ten at the end because only one-tenth of the volume of the solutions of the unknown chloride (and thus one-tenth of the measured mass of the unknown) was used in each forward titration.

[Eq. 8] $\%Cl^-$ = (reacted KHP) $\times \frac{mol\ OH^-}{mol\ KHP} \times \frac{mol\ Cl^-}{mol\ OH^-} \times \frac{35.45g\ Cl^-}{mol\ Cl^-} \div$ (mass unknown) × 10 × 100%
(molar masses were calculated from elemental molar masses found in *Fundamentals of Analytical Chemistry* (2))

The calculation for the percent mass composition of chloride for sample 1 of the unknown solution is shown below as a representative sample. The source of this data can be found in Table 3. This incorporates Eq. 6 through Eq. 8.

(Sample unknown chloride solution 1)
Excess KHP $= (0.03410L - 0.00600L) \times \frac{0.1066M\ NaOH}{L} \times \frac{mol\ excess\ KHP}{mol\ NaOH} = 0.002995 mol$
Reacted KHP $= ((0.01616L - 0.00805L) \times \frac{0.4001 mol\ KHP}{L}) - 0.002995 mol\ KHP = 0.000249 mol$
$\%Cl^- = 0.000249 mol\ KHP \times \frac{mol\ OH^-}{mol\ KHP} \times \frac{mol\ Cl^-}{mol\ OH^-} \times \frac{35.45g\ Cl^-}{mol\ Cl^-} \div 0.3235g \times 10 \times 100\% = 27.2\%$

APPENDIX II
COMPUTATION OF STATISTICAL MEASURES OF PRECISION

A.II.I. Statistics of Concentrations from Standardization

The calculations for the mean ($\bar{x}$), standard deviation (s), standard error (s_m), variance (s^2), and relative standard deviation of the calculated concentration of the standardized sodium hydroxide solution are shown below. Because the data points are roughly spread uniformly over a small range and not heavily skewed, the mean will be used to represent the center and the standard deviation will be used to estimate the range (as opposed to median and IQR). The data shown below were obtained from table 2.

$n = 4$

$\bar{x} = \frac{1}{4}(0.1064M + 0.1061M + 0.1057M + 0.1083M) = 0.1066M$

$s = \sqrt{\frac{(0.1064M-0.1066M)^2+(0.1061M-0.1066M)^2+(0.1057M-0.1066M)^2+(0.1083M-0.1066M)^2)}{3}} = 0.001141M$

A.II.II. Statistics for Percent Mass Chlorides

The calculations for the mean ($\bar{x}$), standard deviation (s), standard error (s_m), variance (s^2), and relative standard deviation of the calculated percent mass composition of chloride ions in the unknown chloride mixture are shown below. Because the data points are roughly spread uniformly over a small range and not heavily skewed, the mean will be used to represent the center and the standard deviation will be used to estimate the range (as opposed to median and IQR). The data shown below were obtained from table 4.

$n = 4$

$\bar{x} = \frac{1}{4}(27.2\% + 43.49\% + 29.8\% + 36.9\%) = 34.3\%$

$s = \sqrt{\frac{(27.2\%-34.3\%)^2+(43.49\%-34.3\%)^2+(29.8\%-34.3\%)^2+(36.9\%-34.3\%)^2)}{3}} = 7.35\%$

$s_m = \frac{7.35\%}{\sqrt{4}} = 3.68\%$

$s^2 = (7.35\%)^2 = 0.541\%^2$

$rel.\ std.\ dev.\ (ppt) = 1000 \times \frac{7.35\%}{34.3\%} = 214$

The calculation of a 90% confidence interval for the standardized is shown below.

$90\%\ confidence\ level\ t_4 = 2.353$

$uncertainty\ (u) = 2.353 \times \frac{7.35\%}{\sqrt{4}} = 8.65\%$
$90\%\ confidence\ interval = 34\% \pm 9\%$

A Q test is performed below at a 90% confidence level.

$range = 43.49\% - 27.2\% = 16.3\%$
$90\%\ confidence\ Q_{crit_4} = 0.76$
$Q_{43.49\%} = \frac{|43.49\% - 34.3\%|}{16.3\%} = 0.562 < Q_{crit}$
$Q_{27.20\%} = \frac{|27.2\% - 34.3\%|}{16.3\%} = 0.438 < Q_{crit}$

The Q-values for the extrema do not exceed Q_{crit}, so it is not statistically justified to remove any data points. None of the data points are impossible, so no data points will be discarded. The possible sources of error leading to the large standard deviation are discussed in the discussion.