Determination of the content of mg (oh)2 in an indigestion remedy by back titrati...

Science, Chemistry

Milk of Magnesia (Magnesium hydroxide mixture) is a mildly basic mixture, which is commonly used in the treatment of constipation and indigestion, because it neutralises excess acid it is called an antacid. Magnesium hydroxide draws water into the colon by osmosis and induces peristalsis and is therefore also known as an osmotic laxative.

An accurate analysis of Milk of Magnesia must measure the total Mg (OH)2 both dissolved and suspended in solution. This is difficult because Milk of Magnesia is poorly soluble in water and exists as a white opaque solution so each sample taken for titrimetric analysis may not be representative of the whole bottle. To make the solution as homogeneous as possible, the bottle was shaken thoroughly each time an amount was taken.

A direct titration of Milk of Magnesia is difficult due to the cloudy suspension and the fact that some of the solution may stick to the sides of the conical flask, preventing complete titration and altering the measurement of the endpoint. The inherent opaque solution also makes it difficult to detect the color change of the endpoint, because of these reasons a back titration is preferred to measure the %w/w of Mg (OH)2 in Milk of Magnesia.

Excess HCl was reacted with Mg (OH)2 to yield a clear solution, an indicator was added and then the excess acid was back titrated with NaOH. The results obtained from the titration and the subsequent calculations determined whether the Milk of Magnesia mixture that was titrated actually did contain the required amount of Mg (OH)2 (%w/w) as stated in the B. P.

Key Words

Burette: a long vessel with a tap at the bottom which is used to measure accurately the volume of a solution added. The scale can be read to an accuracy of half a division e. g. to 0. 05 cm3.

Bulb Pipette: used to deliver an accurate volume of a solution. This can be 10cm3 or 25 cm3.

Conical flask: used to hold and to make chemicals, substances can be stirred and swirled without the risk of spilling and reduces the loss of the evaporation due to the narrow neck.

Methyl Orange Indicator: a pH indicator frequently used in titrations. It is often chosen to be used in titrations because of its clear colour change.

Titration: The technique of titration is used to find out accurately how much of a chemical substance is dissolved in a given volume of a solution, that is, the concentration of the solution.

British Pharmacopoeia (BP): is the official collection of standards for UK medicinal products and pharmaceutical substances.

Tare: The weight of a container is deducted from gross weight to obtain net weight of the substance inside the container.

%w/w: The weight of a substance contained per 100g.

Introduction

The purpose of this experiment is to determine whether the contents of “ Phillips Milk of Magnesia” actually does contain the required amount of Mg (OH)2 as stated in the B. P.

The B. P. states:

* “ Magnesium Hydroxide Mixture is an aqueous oral suspension of hydrated magnesium oxide.

* The content of Magnesium oxide is calculated as Mg (OH)2 : 7. 45 to 8. 35% w/w. 1

Therefore 100g should contain between 7. 45g and 8. 35g of Magnesium oxide.

The back titration will be repeated on 3 occasions, this is to ensure that a mean average can be calculated and this value will then be used to determine the weight of Magnesium Hydroxide per 100g through mathematical calculation.

Materials

* Burette & Stand

* Bulb pipette (25ml) and filler

* Conical Flask (100ml)

* Beaker (100ml)

* Measuring Cylinder

* White Tile

* Funnel

* Hydrochloric acid (1. 0 mol dm-3)

* Sodium hydroxide (0. 5mol dm-3)

* Magnesium hydroxide suspension B. P

* Distilled water

* Methyl orange indicator

* Electronic scale

Method

Illustration showing how to set up equipment for titration, reading the meniscus and a sample results table. 2

* The equipment was set up as illustrated

* The Magnesium hydroxide mixture was shaken thoroughly to ensure it was as homogeneous as possible.

* 5cm3 of Magnesium hydroxide mixture was measured out using a measuring cylinder.

* A 100cm3 conical flask was “ tared” on the balance, and then the mass of magnesium hydroxide mixture was recorded to 3 decimal places.

* 25cm3 of 1. 0 mol dm-3 of HCl was measured using a pipette and added to the flask with the magnesium hydroxide mixture

* The mixture was swirled until it went clear and 3 drops of Methylene Orange indicator were added, causing the solution to turn red.

* The burette was filled with 0. 5 mol dm-3 of NaOH and the volume mark (bottom of meniscus) was noted to 2 decimal places.

* NaOH was added initially with rapid drops going into the conical flask which was being swirled all the time.

* When the indicator briefly changed colour from yellow back to red the NaOH was added in slow drops and the flask was swirled all the time.

* When the solution turned a permanent yellow, the titration was stopped and the solution was left for a further 2 minutes to see if there was further colour changes.

* The volume mark of NaOH used was noted.

(Volume of acid used = Final volume – Initial volume)

* The titration was repeated on two more occasions (3 occasions in total)

Results

Titration

Initial volume reading (cm3)

Final volume reading (cm3)

Volume of NaOH delivered (cm3)

Mass of

Mg(OH)2 (g)

1

0

3. 75

3. 75

4. 118

2

3. 75

4. 55

0. 80

4. 708

3

4. 55

6. 05

1. 50

4. 399

These are the reactions that occurred during the back titration

1. Mg (OH)2 (aq) + 2HCl (aq) –> MgCl2 (aq) + 2H2O (l)

2. HCl (aq) + NaOH –> NaCl (aq) + H2 O (l)

Calculations

Question 1

25cm3 of HCl was used at 1M concentration.

1. Therefore (25cm3 /1000) x 1. 0 mol dm-3 = 0. 025 moles of HCl used.

Various volumes (3. 75cm3 , 0. 80cm3 and 1. 5 cm3) of NaOH were used at 0. 5 M concentration.

2. (3. 75cm3 /1000) x 0. 5 mol dm-3 = 1. 875 x 10-3 mol (NaOH used)

3. (0. 80cm3/1000) x 0. 5 mol dm-3 = 4 X 10-4 mol (NaOH used)

4. (1. 5 cm3 /1000) x 0. 5 mol dm-3 = 7. 5 x 10-4 mol (NaOH used)

Moles of HCl – Moles of NaOH = Moles of HCl remaining

(N. B mol of NaOH = mol of HCl as ratio 1: 1)

5. 0. 025 mol – 1. 875 x 10-3 mol = 0. 023125 mol

6. 0. 025 mol – 4 X 10-4 mol = 0. 0246 mol

7. 0. 025 mol – 7. 5 x 10-4 mol = 0. 02425 mol

Ratio of Mg (OH)2 and HCl is 1: 2 therefore values for HCl remaining must be divided by 2 to reach the value of moles for Mg(OH)2 .

8. 0. 023125/2 = 0. 0115625 mol

9. 0. 0246/2 = 0. 0123 mol

10. 0. 02425/2 = 0. 012125 mol

Mass of Mg (OH)2 used = amount in moles x Mr

Mr = 24. 305 + (15. 999 x 2) + (1. 0079 x 2) = 58. 3188g

11. 0. 0115625 mol x 58. 319g (3. d. p.) = 0. 674g (3. d. p.)

12. 0. 0123 mol x 58. 319g (3. d. p) = 0. 717g (3. d. p.)

13. 0. 012125 mol x 58. 319g (3. d. p.) = 0. 707g (3. d. p)

Question 2

% w/w content for Mg (OH)2

% w/w = x 100

14. (0. 674g/4. 118g) x 100 = 16. 367% (3. d. p.)

15. (0. 717g/4. 705g) x 100 = 15. 229% (3. d. p.)

16. (0. 707g/4. 399g) x 100 = 16. 072% (3. d. p.)

Question 3

Average of %w/w values

17. (16. 367 + 15. 229 + 16. 072)/3 = 15. 889% (3. d. p.)

The B. P. states that hydrated Magnesium oxide should be between: 7. 45 – 8. 35% w/w.

The average value that was derived (15. 899%) from this titration did not fall in the range specified by the B. P.

Discussion

1. The volume of NaOH delivered varied considerably, the greatest volume delivered was 3. 75 cm3 the least volume delivered was 0. 8cm3, this gives a range of 2. 95cm3. The B. P states that the titration readings should be within 0. 1cm3 of each other, however the mass of magnesium hydroxide mixture also varied considerably between titration, therefore the volume of NaOH delivered would reflect this as smaller quantities of the magnesium hydroxide mixture would have more excess HCl left over and would therefore require more NaOH to turn the solution basic again, this trend is observed in the results table.

2. The three %w/w values which were calculated from the experiment had a range of 1. 138% from the highest value (16. 367%) to the lowest value (15. 229%), the three masses of the magnesium hydroxide mixture had a range of 0. 59g, the highest value was 4. 708g and the lowest value was 4. 118g. As the values were relatively close together there was no reason why an average could not be taken, however if there had been a value that was very far out, then it would have been wise to omit it from the average as it would skew the result.

3. The average % w/w for Mg (OH)2 that was calculated from the experiment results was 15. 889% (3. d. p.). Whereas the B. P. states that the %w/w value should be between 7. 45 – 8. 35 %w/w3. As the value that was derived from the experiment is approximately twice the amount that is stated in the B. P. it is highly likely that there is a second base in the mixture and the accuracy of the actual concentrations of the reagents (NaOH & HCl) can also be questioned as well the fundamental design of the experiment.

On www. medicines. org. uk4 it states that " Philips Milk of Magnesia" also contains NaHCO3 or sodium bicarbonate as it is better known. The presence of sodium bicarbonate, which is also a base, has interfered with the titration and the result is that the presence of sodium bicarbonate has left less excess HCl acid to be titrated against NaOH.

This can be demonstrated through this formula of the actual reaction that occurred.

Mg (OH)2 + NaHCO3 + 3HCl –> NaCl + MgCl2 + 3H2O + CO2

As 3 moles of HCl would have being neutralised instead of 2 moles in the mixture, as calculated earlier there is less excess acid available for the back titration.

If the %w/w is approximately recalculated taking this into account a revised %w/w is formed.

e. g. 15. 889% x 2 = 31. 778%

31. 778% / 3 = 10. 593%

This figure although not within the parameters set by the B. P. is much closer to the figures set than originally calculated.

In hindsight it would have more appropriate to do a complexometric titration instead of the back titration as there is a mixture of metal ions (sodium & magnesium) present in the " Milk of Magnesia" mixture and would have given a more accurate result to calculate if the %w/w of the sample tested complied with values stated in the B. P. The complexometric titration would have allowed one of the metal ions (more likely to be Mg2+) to bind to EDTA to form a complex ion on a 1: 1 ratio, while displacing the indicator, when the indicator was entirely displaced a colour change would occur5.

The quantification of errors due to laboratory equipment.

During any experiment, especially one such as titration where the results obtained depend so entirely on the accuracy of the equipment used, the

errors due to the equipment used during the titration can be quantified to calculate the margin of error of the experiment. 6

% Error of Electronic Balance.

% error = 0. 001g/mass measured x 100

Average weight of mass measured (4. 118g + 4. 708g + 4. 399g/3) = 4. 408g (3. d. p)

0. 001g/4. 408g x 100 = 0. 023%.

% Error of Pipette

% error = 0. 1cm3 /25cm3 x100 = 0. 4%

% Error of Burette

% error = 0. 05cm3 /50cm3 x100 = 0. 1%

% margin of overall error due to lab equipment = 0. 023% + 0. 4% + 0. 1% = ï¿½ 0. 523%

Improvements

There are several ways in which this experiment could be improved, so that the accuracy of the result could be improved.

Firstly, the NaOH and HCl should have been titrated before the titration began to obtain an accurate volumetric standard to determine the exact concentration of NaOH7 and HCl before they were used as they are volatile

solutions and concentration can vary due to evaporation of the solution. This is defined in the B. P. with the specific procedure that must be followed.

Secondly, the measuring cylinder used to measure the Milk of Magnesia mixture could have been rinsed out with distilled water and the contents emptied into the conical flask, more distilled water, which had been measured could have been added to make a larger volume as this would not affect the amount of moles present, or a smaller measuring vessel could have been used so that less mixture would adhere to the sides of the vessel.

Thirdly, the electronic scale had no accompanying document showing a recent calibration, so the accuracy of the instrument could not be guaranteed and therefore the accuracy of the weights measured is under question.

Fourthly, a significant part of any titration is the indicator that is used to measure the end point, if it is not suitable then this can also cause discrepancies. The pH of the Magnesium hydroxide mixture and HCl reaction could have been measured using a pH meter, the resulting pH value could be used to predict the pH of equivalence point using a pH curve, as the Milk of Magnesia reagent was not a pure compound the pH would be too complicated to calculate this pH value would be useful in selecting an appropriate indicator

The Methyl orange indicator was not suitable for this titration. The colour change range for this indicator is pH 2. 9-4. 08. This means that the endpoint occurred while the solution was still acidic. As the titration was between a

strong acid(HCl) which had already reacted with Mg(OH)2 and NaHCO3 resulting in fewer H+ ions available in the solution. Therefore, theoretically a weak acid (fewer H+ ions in solution) reacted with a strong base(NaOH) an indicator such as Methyl Red would have been more appropriate and would given a better indication as to when equivalence(endpoint) was reached (pH 4. 2-6. 3)9.

This would have allowed more NaOH to be added to neutralise as many of the H+ ions before the end point occurred and would have given a more accurate result for %w/w calculations.

Finally, assuming the weighing scale was calibrated, the weight of the Magnesium hydroxide mixture could have been weighed to the same amount on each occasion and this would have helped to ensure the %w/w would be more accurate and values would be closer.

Conclusion

The purpose of this experiment was to determine whether the contents of " Phillips Milk of Magnesia" actually did contain the required amount of Mg (OH)2 as stated in the B. P. (7. 45 – 8. 35% w/w).

This could not be proven in this experiment using a back titration, as the Milk of Magnesia mixture was not a pure compound consisting only of Mg (OH)2 . A second base NaHCO3 was also discovered to be present in the mixture, which interfered with the result significantly. The HCl and NaOH were also not titrated to a volumetric standard, prior to conducting the titration. It is

also highly likely that the indicator used (Methyl orange) was not suitable to indicate the end point, so the results of the experiment are inconclusive. Therefore it can be concluded that the titration method used was not suitable in producing accurate results to determine the %w/w. In retrospect, a complexometric titration would have been more suitable in determining the %w/w more accurately.

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