THE AMMONIA CONTENT OF 'CLOUDY AMMONIA'

INTRODUCTION

Many household products contain a mixture of chemicals, and it is important for quality control to monitor the amount of each chemical. In this experiment we will use a volumetric technique to check the percentage by mass of ammonia that is present in household 'cloudy ammonia'.

Given that ammonia is the only alkali present in the sample of cloudy ammonia, direct titration of the ammonia with a standard acid solution will allow us to find the amount of ammonia present in a measured volume of cloudy ammonia solution. A standard solution of 0.100 M hydrochloric acid will be used.

The reaction that occurs is:

$$NH_3(aq) + H^+(aq) \rightarrow NH_4^+(aq)$$

The solution that forms is slightly acidic, so methyl red is a suitable indicator.

AIM

To determine the percentage of ammonia by mass that is present in household Cloudy Ammonia solution.

MATERIALS

- 4 x 100 ml conical flasks
- 250 ml volumetric flask
- 10 ml measuring cylinder
- White tile
- Methyl red indicator solution
- Small funnel
- 20.0 ml pipette
- Burette and stand
- Wash bottle

METHOD AND SAMPLE RESULTS

1. Record the manufacturer's name, product name and the percentage by mass of ammonia as stated on the label. Check that the product is not out of date according to the 'use by date', if applicable.

   For Example: Assume the label states that the product contains 'not more than 9% ammonia w/w'.

2. Using the percentage by mass of ammonia as stated on the label, calculate the mass of cloudy ammonia required to give 250 ml of 0.10 M solution with respect to ammonia, NH_3.

   For Example:

   i. $n(NH_3) = C \times V$
   $$= 0.10 \times 0.250 = 0.0250 mol$$

   ii. $mass(NH_3) = n \times M$
   $$= 0.0250 mol \times 17.0 = 0.425 g$$

   iii. $Mass\ of\ 9\%\ (w/w)\ solution = mass(NH_3) \times \frac{100}{9}$
   $$= 0.425 \times \frac{100}{9} = 4.72 g$$

3. Weigh a clean, dry 250 ml volumetric flask and stopper. Use a 10 ml measuring cylinder and a small funnel to add approximately the mass of Cloudy Ammonia calculated (assume 1 g=1 ml for cloudy ammonia). Place the stopper in the flask and reweigh the assembly. Calculate the precise mass of Cloudy Ammonia added.

   For Example:

   $$mass(9\% solution) = mass(flask + solution) - mass(flask)$$
   $$= 5.567 g$$

4. Slowly pour about 200 ml of distilled water into the volumetric flask. Insert the stopper and swirl the solution. Add distilled water to bring the volume to the 250 ml mark. Insert the stopper and invert the flask several times so that the solution has a uniform concentration.

5. Use a pipette to transfer 20.00 ml of the ammonia solution into a small conical flask, making sure the stopper is replaced in the standard flask afterwards. Add 2 to 3 drops of methyl red indicator solution to the conical flask.

6. Rinse and fill the burette with the standard solution of HCl. Record the precise concentration of the standard solution of HCl and the initial reading of the burette.

   For Example: Concentration of HCl = 0.107 M

7. Titrate the ammonia solution with the acid. The end point can be identified by the change in colour from yellow to orange-pink. Record the final reading of the burette and calculate the volume of the titre.

8. Repeat steps 5 to 7 until three concordant titres have been obtained. Calculate the average titre from these results.

   For Example: Average titre = 21.45 ml

DISCUSSION QUESTIONS

QUESTION 1
Using the average titre, calculate the amount, in mole, of HCl added to each 20.00 ml aliquot of dilute Cloudy Ammonia.

Solution

QUESTION 2
Calculate the amount, in mole, of NH_3 present in each 20.00 ml aliquot of dilute Cloudy Ammonia. Hence calculate the amount, in mole, of NH_3 that was present in the 250 ml volumetric flask.

Solution

QUESTION 3
Calculate the mass of NH_3 that was present in the 250 ml volumetric flask. Hence calculate the percentage by mass of NH_3 in the original sample. Compare this result with the manufacturer's specifications.

Solution

QUESTION 4
What solutions should the following pieces of equipment be rinsed with? Explain your answers.

(a) Pipette

(b) Conical Flask

QUESTION 5
How might your results have been affected if the product was no longer within its 'use by date'?

Solution

QUESTION 6
How would the final result have been affected if the burette had been rinsed with distilled water rather than standard HCl?

Solution

QUESTION 7
Explain why the Cloudy Ammonia was diluted before titration.

Solution

QUESTION 8
Why is the volumetric flask re-stoppered after each aliquot of ammonia solution is removed? Why is the titration then performed without delay?

Solution

QUESTION 9
Why would phenolphthalein, which changes from pink-violet at pH 10 to colourless at pH 8, be unsuitable for use as an indicator solution in this experiment?

Solution

QUESTION 10
An alternative procedure for determining the amount of ammonia in Cloudy Ammonia is to perform a back titration. Two 20.00 ml aliquots of standard 0.100 M HCl would be put into the titration flask with one 20.00 ml aliquot of dilute (approximately 0.1 M) Cloudy Ammonia solution. The excess HCl would then be titrated against standard 0.1 M sodium carbonate solution using methyl red as an indicator. What advantages are there to this alternative method?

Solution

CONCLUSION

In this experiment, the percentage of ammonia by mass in the sample of Cloudy Ammonia was found to be 8.76 % (w/w). This figure is quite close to the stated value of ‘not more than 9 % ammonia w/w’. However, the slightly lower value could have been due to the high volatility of ammonia and the consequent escape of ammonia during the procedure.

ANSWERS TO DISCUSSION QUESTIONS

QUESTION 1

$$n(HCl) = C \times V$$
$$= 0.107 \times 0.02145 = 2.30 \times 10^{-3}\ mol$$

QUESTION 2

$$NH_3(aq) + H^+(aq) \rightarrow NH_4^+(aq)$$

i. $n(H^+) = n(HCl)$
$$= 2.14 \times 10^{-3}\ mol$$

ii. $n(NH_3) = n(H^+)$
$$= 2.30 \times 10^{-3}\ mol\ in\ each\ 20.00\ ml\ aliquot\ of\ the\ dilute\ solution$$

iii. $2.30 \times 10^{-3}\ mol \quad in \quad 20.00\ ml$
$x\ mol \quad in \quad 250.00\ ml$

$cross\ multiply$: $\quad 20.00 \times x = 2.30 \times 10^{-3} \times 250.00$
$\therefore x = 0.0287\ mol\ in\ 250.00\ ml\ of\ the\ sample$

QUESTION 3

i. $mass(NH_3) = n \times M$
$$= 0.0287\ mol \times 17.0 = 0.488\ g$$

ii. $$\%(NH_3) = \frac{mass\ of\ NH_3\ (g)}{mass\ of\ sample\ (g)} \times 100$$

$$= \frac{0.488}{5.567} \times 100 = 8.76\%(w/w)$$

iii. This value of 8.76 % (w/w) is quite close to the stated value of ‘not more than 9 % ammonia w/w’.

QUESTION 4

(a) Pipette

The final rinse of the pipette should be with the dilute ammonia solution so that the concentration of the sample does not change. If distilled water or the HCl solution were used, the ammonia sample would become less concentrated.

(b) Conical Flask

The conical flask should be rinsed with distilled water so that the amount, in mole, of ammonia does not change. If the conical flask were rinsed with ammonia solution, the amount of ammonia in the flask would increase. If the conical flask were rinsed with HCl solution, the amount of ammonia in the flask would effectively be reduced.

QUESTION 5

Some products deteriorate over time, and the amount of the active ingredient is therefore reduced.

QUESTION 6

- The concentration of HCl would be less.
- A larger volume of HCl would have been required.
- The amount, in mole, of HCl would seem greater.
- Due to mole ratios, the amount, in mole, of NH_3 would seem greater.
- The calculated mass of NH_3 would be greater, and therefore the calculated percentage mass would be higher than the true figure.

QUESTION 7

It is generally accepted that a mean titre should be in the vicinity of 20 ml. A smaller titre leads to a greater percentage error. And a larger titre leads to wasted resources and time, and also greater error if the burette must be refilled. A good compromise between accuracy and effective use of resources is generally considered to be a titre of about 20 ml.

Given that the available HCl had a concentration of about 0.1 M, a good titration would involve about

$$n(HCl) = C \times V$$
$$= 0.1 \times 0.02 = 2 \times 10^{-3}\ mol\ of\ HCl$$

Looking at the mole ratios, a good titration would have

$$n(NH_3) = n(HCl)$$
$$= 2 \times 10^{-3}\ mol\ of\ NH_3\ in\ each\ 20.00\ ml\ aliquot$$

Whereas, if an undiluted sample of ammonia solution had been used, the amount of NH_3 in 20 ml, or 20 g, would have been

$$mass(NH_3) \quad = 9\% \ of \ 20g = 1.8g$$

$$\therefore n(NH_3) \quad = \frac{m}{M}$$
$$= \frac{1.8}{17} = 0.1 mol of \ NH_3 \ in each \ 20 ml \ aliquot$$

According to the following calculations, this would have required an average titre of about 1 L, which is unacceptably high.

$$n(HCl) = n(NH_3)$$
$$= 0.1 mol$$

$$\therefore Vol(HCl) = \frac{n}{C}$$
$$= \frac{0.1}{0.1} = 1L$$

QUESTION 8

Ammonia is volatile and could escape from an unstoppered volumetric flask. Similarly, it could escape from the conical flask before the end point of the titration is reached. The solution would become less concentrated, and the calculated result would be lower than the true figure.

QUESTION 9

Initially, ammonia is a weak base and has pH above 7. HCl is a strong acid and the pH falls as it is added to the ammonia. At the equivalence point of the titration, NH_4^+ has formed and is slightly acidic (pH=5.2). Excess acid then causes the pH to fall rapidly.

Phenolphthalein changes colour in the pH range of 10 to 8. Thus it would change colour before the equivalence point of the titration, and the titration would be stopped significantly early.

QUESTION 10

The advantage of this method is that the ammonia immediately reacts to form the ammonium salt and therefore the concentration does not depend on the time taken to perform the titration.

However, errors are larger in a back titration and it may be difficult to obtain a set of concordant results.