

 ***Analytical Chemistry***

***CHEM234***

***Sec 1***

***Exp5: Title***

***Gravimetric determination of sulfate***

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* ***Abstract:***

***The objective of this experiment is to accurately determine the amount of an analyte by selectively precipitating the analyte (the ion being analyzed) from an aqueous solution using the Gravimetric analysis method. The precipitate is then separated from the remaining aqueous solution by filtration and weighed. The procedure necessitates the weighing of a container, generally a crucible.***

***An unknown sulfate-containing material will be combined with excess precipitating agent (BaCl2) to create a precipitate (BaSO4) in this experiment.***

***So the major reaction in this experiment:***

***MxSO4 (aq) + BaCl2 BaSo4 (s) + MClx (aq)***

**The final percentage of sulfate ion in our unknown sample was:**

* ***Data:***

|  |  |
| --- | --- |
| Sampling for the unknown: M |  |
|  | **Sample 1** |  **Sample 2**  | **Sample 3** |
| Mass of unknown sulfate g  | 0.3176 g | 0.3388 g | 0.300 g |
| Heating of empty crucible (g)  | **Crucible 1** | **Crucible2** | **Crucible3** |
| Mass of empty crucible (g)  | 55.4790 g | 55.4791 g | 55.8989 g |
| Constant mass of crucible (m1) | 55.4758 g | 55.5237 g | 55.9002 g |
| Mass of crucible and precipitate (g) | 55.8929 g | 55.9684 g | 56.3069 g |
| Constant mass of crucible and precipitate, g (m2) | 55.8914 g | 55.9669 g | 56.3060 g |
| Mass of barium sulfate, g (m2-m1) | 0.4156 g | 0.4432 g | 0.4058 g |

* ***Calculated & result:***

*Calculations for sample 1:*

* Gravimetric factor
 = molar mass of sought/ molar mass of ppt
 = molar mass of SO4-2/ molar mass of BaSO4 = 96.06 /233.4
= 0.4116
* Mass of Barium sulfate (g)
 = (Mass of crucible + precipitate) – mass of empty crucible
 = 55.8914 g - 55.4758 g = 0.4156 g
* Percentage of sulfate ion in the sample (%)
= ((W.t of barium sulfate\* gravimetric factor)/ W.t of unknown sample) \* 100%
=( (0.4156 \* 0.4116/ 0.3176)\* 100% = 53.86 %.
* At unknown = 0.3388 g (have a 0.25 M BaCl2 )
Moles of li2So4 = 0.3388/109.49 g/mol = 3.094346 \* 10-3

V = (moles/M) \* 100%

= (3.094346\*10-3/0.25) \* 100

= 12.37738

The excess = (10/100) \* 12.37738

= 1.23773 + 12.37738

= 13.61511 mL

|  |  |  |  |
| --- | --- | --- | --- |
|  | sample1 | sample2 | sample3 |
| Mass of Barium Sulfate (g) | 0.4156 g | 0.4432 g  | 0.4058 g |
| Percentage of sulfate ion in the sample (%) | 53.86 % | 53.84 % | 55.67% |
| The Average of sulfate ion  | 54.46% |

* **Average percentage of sulfate ion in the samples (%):**
= ((53.86+53.84+55.67)/(3))
= 54.46 %

* **Standard deviation of sulfate ion in the samples (%):**
(s) =
* **Grubbs test:**Gcalculated = ((questionable value – mean) / (s))
= ((55.67 – 54.46) / (1.0501))
= 1.152
- The suspension value isn’t outlier because the G table > G test
- **The G table** confidence level of 95% & n = 3 = **1.153
1.152 < 1.153**
* **RSD %:**

**Coefficient of variation = ((s\x) \* 100)**

= (1.0501/54.46) \* 100% = 1.928 %

* **95 % confidence interval(**
= 54.46 ± ((4.303 \* 1.0501) /
= 54.46 ± 2.609 %
* **Discussion & Conclusion:**

The final result of 95% confidence interval for sulfate ion = (54.46 ± 2.609) %, the average 57.069 to 51.851, so we may conclude that we have good accuracy in our experiment because this range is not excessively large. And the Standard deviation of sulfate ion in the samples = 1.0501. And the RSD% = 1.928%

Of the errors that may have occurred: One of these reasons is when the fire fell was placed over the crucible was a little bit of dirt, and this may increase our error rate. Also, the crucible may have been kept outside for a longer period, which is why it either absorbs moisture or hasn't cooled well inside the desiccator. Additionally, one of the reasons for the error is when BaSO4 is quickly poured onto BaCl2. It is possible to dissolve some sediments before they burned and make an error, as well as any errors that occur with heating.

Because excess Cl- reacts with Ag + to create a white silver chloride precipitate, we utilized silver nitrate. As a result, the formation of this precipitate will indicate the need for further washings to get rid of all the excess Cl-.