

***Analytical Chemistry***

***CHEM234***

***Sec 1***

***Unknown: M***

***Exp6: Title***

***Determination of sulfate using an adsorption indicator***

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* **Abstract:**

In comparison to the slow gravimetric method, the titration method utilizing an adsorption indicator, which is a material that indicates an excess of a reactant, is a fast way of estimating the percentage of any chemical in an unknown sample. Coprecipitation of other ions in the solution generated by normal titration addition may, however, result in under consumption of the precipitating reagent. To eliminate this, the ion exchange procedure is utilized to remove all cations that might cause coprecipitation and titration errors. The hydrogen version of cation exchange resin is utilized in a column. In the resin, the cations in the sulfate solution are exchanged for hydrogen ions. In such a titration, the indicator was chosen to be alizarin red S, which is used as an adsorption indicator and also a negatively charged indicator and he's color is yellow so at the end point the color changed to pale pink.

The titration reaction is:

Ba2+ (aq)+ SO4-2(aq) 🡪 BaSO4(s)

The cations in the sulfate solution are exchanged for hydrogen ions of the resin:

2M+ SO42- + 2 Resin−H + → 2 Resin−M+ + 2H + SO42

The sulfuric acid is partially neutralized with magnesium acetate, and the sulfate is titrated with standard barium chloride.

MgAc2 + 2H + SO42- → Mg 2+ SO42- + 2HAc

the final result was obtained as follows from the % sulfate in the (M) unknown: Average % sulfate in the unknown with ion exchange was 56.70% and the average % sulfate in the unknown without ion exchange was 57.43%

* **Data and Calculations:**

**Code for the unknown:** M

**Concentration of BaCl2:** 0.050 M

**Mass of sulfate sample, g:** 1.7640

**Total volume of sulfate solution:** 100.00 ml

**Volume of aliquot used in titration:** 10.00 ml

**Table1:** Adsorptive indicator titration data for both ion exchange and ion without exchange

|  |  |  |
| --- | --- | --- |
| Titration without ion exchange | Sample 1 | Sample 2 |
| Final reading of burette (mL) | 0.00 mL | 0.00 mL |
| Initial reading of burette (mL) | 19.00 mL | 23.20 mL |
| Net volume of BaCl2 (mL) | 19.00 mL | 23.20 mL |
| Titration with ion exchange | **Sample 1** | **Sample 2** |
| Final reading of burette (mL) | 0.00 mL | 0.00 mL |
| Initial reading of burette (mL) | 22.10 mL | 18.90 mL |
| Net volume of BaCl2 (mL) | 22.10 mL | 18.90 mL |

**Table2:** Average percent sulfate in unknown sample "M" as obtained from various analytical two methods:

|  |  |  |
| --- | --- | --- |
|  | **Without ion exchange** | **With ion exchange** |
| **Average Volume (ml)** | 21.10 mL | 20.50 mL |
| **Average moles of barium chloride (mol)** | 1.055 \* 10-3 mol | 1.025 \* 10-3 mole |
| **Average moles of sulfate (mol)** | 1.055 \* 10-3 mole | 1.025 \* 10-3 mole |
| **Average mass of sulfate (g)** | 0.1013 g | 0.1000 g |
| **Average mass of sulfate in the original weighed sample (g)** | 1.0130 g | 1.000 g |
| **Average % sulfate in the unknown** | 57.43% | 56.70% |

**Calculations for the part done without ion exchange:**

* **Average volume (ml)**

= (Net Volume of sample1 + Net volume of sample2)/2   
= (19.00 + 23.20)/2 = 21.10 ml

* **Average moles of BaCl2** = Molar concentration of barium chloride \* average volume   
  = 21.10 \*0.050 \* (10-3) = 1.055\* 10-3 mole of barium chloride
* **Average moles of sulfate** = average moles of barium sulfate  
   = 1.055\* 10-3 mole of sulfate
* **Average mass of sulfate**  
   = molar mass of sulfate \* average moles of sulfate  
   = 96.06 \* 1.055\* 10-3 = 0.1013 g
* **Average mass of sulfate in the original weighed sample**  
  = 0.1013 \* 10  
  = 1.0130 g
* **Average % sulfate in the unknown**   
  = (Average mass of sulfate/ mass of original sample) \*100%  
  = (1.0130 / 1.7640) \*100% = 57.43%
* **% Deviation of sulfate**  
   = ((|%sulfate in the sample with ion exchange - %sulfate in the sample without exchange|) / (% sulfate in the sample with ion exchange))

= ((|56.70 – 57.43|)/ (56.70)) \*100

= 1.288 %

* **Discussion and conclusion:**

**Comparison of results of % of sulfate using the adsorption indicator analysis:**

Without ion exchange and with ion exchange: the percentage obtained by the ion exchange method is lower than the percentage obtained without ion exchange, but it's not correct because coprecipitation of interfering ions causes underconsumption of titrant so the ion exchange we must be higher than percentage without ion exchange. Thus, masking those ions in the ion exchange would increase the consumption of titrant and will give more accurate results but because the ion exchange lower than without ion exchange the titration it's not accurate for me. his is because I used the cylinder in the ion exchange, while I used the pipet with the second value (sample 2) of the ion exchanged the first value was taken in the cylinder, and this led to my low accuracy.

**Comparison of sulfate percentage results using two methods of analysis:**

The average percentage achieved by the gravimetric approach was 54.46 percent, whereas the adsorption indicator analysis method yielded 56.70 percent. Although we utilized back addition in the gravimetric analysis, the result obtained by titration utilizing ion exchange was greater because it masks all the interfering ions, resulting in a more accurate result. because the difference between the two method is just 2.24 percent, so we can conclude that the result was fairly accurate.

Due to barium ions adsorbed on the surface, the precipitate is transformed to positively charged particles when the initial excess of barium is introduced. The negatively charged indicator ion is attracted to the surface and forms a pale pink barium complex as a result (on the surface of precipitate). So this is why we need to keep stirring the solution so that we can observe the pale pink colour appear.

There are many methodological errors that exist, and one of these common errors is that when the solution is poured into buret in the presence of a glass funnel, it isn’t removed during titration after the required solution has been poured causing an increase in the error rate, because it may be contaminated or otherwise, and it is possible that The buret is contaminated and reading through the buret may be inaccurate and cylinder was used instead of pipet, thus not adhering to the required procedures, which caused a percentage of error . One common error is bubbles in the buret. Finally, these errors should be avoided by paying attention that when filling the burette with the solution, we remove the glass funnel in order to take the reading correctly, and that we wash the buret properly and adhere to the given instructions and procedures.