

## CHB-302-P, B.Sc. III sem. (Physical and Volumetric Practical)

**Exp. 1:** Determine the amount (in g/L) of  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  in a given solution with the help of EDTA solution.

Molecular weight of  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O} = 246.5$ . Given EDTA solution strength: M/50.

**Theory:** This determination is based on complexometric titration. The amount of  $\text{Mg}^{2+}$  can be directly determined using EDTA as complexing/chelating agent. A metal ion indicator, (such as Solochrome black or Eriochrome black T (EBT)) is used along with EDTA which helps to understand the end point of the titration. Metal ion solution such as aq.  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  whose concentration is to be determined is made alkaline by using ammonium buffer (aq.  $\text{NH}_3$  and  $\text{NH}_4\text{Cl}$ ) (to maintain pH in range of 7-11) and a pinch of EBT. This solution is then titrated with EDTA solution of known concentration. Initially, metal ions forms 1:1 complex with both EDTA and EBT indicator. While the Mg-EDTA complex is colorless, Mg-EBT complex is of wine-red color (and more stable than the former). (Note: Thus the wine-red color of the solution in conical is due to Mg-EBT complex.) When EDTA solution is added in this solution, the free  $\text{Mg}^{2+}$  reacts with EDTA and makes 1:1 complex. After complete reaction with free  $\text{Mg}^{2+}$  ions, EDTA displaces indicator anion from Mg-EBT complex with the formation of more stable Mg-EDTA complex and the color of the titrating mixture changes from wine red color to blue color at the end point. (Note: At this point, the blue color is just the color of free indicator at that basic pH).

### Procedure:

- 1) 10ml of given  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  solution is taken in a conical flask with a pipette and 2ml of ammonium buffer solution and one pinch EBT indicator were added in it. The color of the solution become wine- red on mixing.
- 2) This solution was then titrated with given known strength M/50 EDTA solution (taken in the burette) until the wine red color turns to blue.
- 3) Repeat the titration to get the concordant readings. (Note: End point is the point where color change occurs. Don't look for depth of the color, look for the change of the color).

### Observation and Calculation:

Table 1: Titration of the 10 ml MgSO<sub>4</sub>.7H<sub>2</sub>O solution with M/50 EDTA solution

No. of observations	Initial burette readings of EDTA (ml)	Final burette readings of EDTA (ml)	Volume of EDTA used (ml)	Concordant volume of EDTA (V <sub>2</sub> , ml)
1				
2				
3				
4				

Since, 1mole of EDTA  $\equiv$  1mole of Mg<sup>2+</sup>

$$M_1 \times V_1 = M_2 \times V_2$$

where,

M<sub>1</sub> = the molar strength of the aq. MgSO<sub>4</sub>.7H<sub>2</sub>O solution

M<sub>2</sub> = the molar strength of the supplied aq. EDTA solution

V<sub>1</sub> = the volume of the standard aq. MgSO<sub>4</sub>.7H<sub>2</sub>O solution = 10 ml

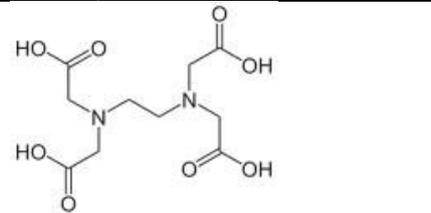
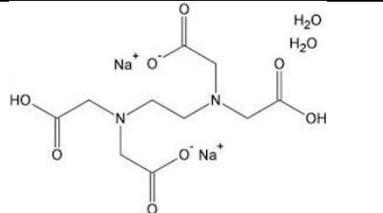
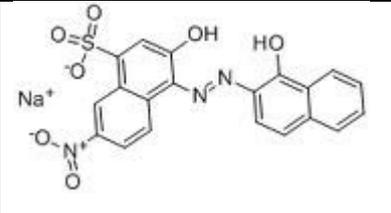
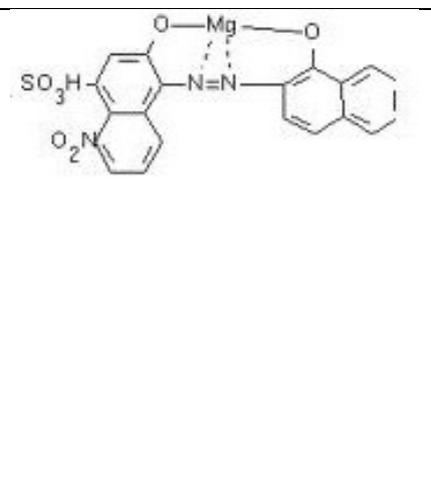
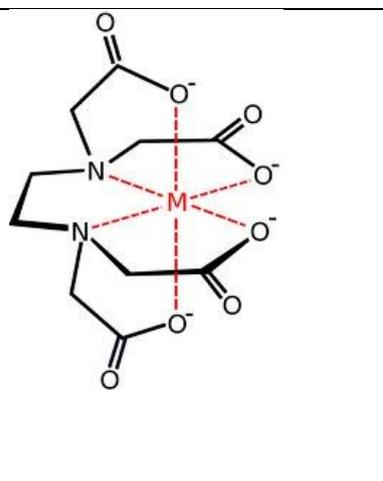
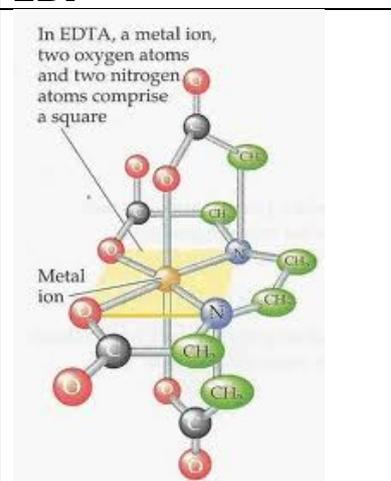
V<sub>2</sub> = the volume of the supplied aq. EDTA solution

So, the strength of the supplied magnesium sulphate solution (M<sub>1</sub>) = M<sub>2</sub> x V<sub>2</sub> / V<sub>1</sub> (M)

Therefore, the amount of MgSO<sub>4</sub>.7H<sub>2</sub>O (in g/L) = M<sub>1</sub> x Molecular weight of MgSO<sub>4</sub>.7H<sub>2</sub>O (i.e., 246.5)

**Results:** The strength of MgSO<sub>4</sub>.7H<sub>2</sub>O in the unknown MgSO<sub>4</sub>.7H<sub>2</sub>O solution is x.xxxx g/L.

**Extra** (learn and draw these structures on white page of your practical note book.):

		
EDTA	Disodium salt of EDTA	EBT
		
EBT-Mg complex	Mg-EDTA complex	Mg-EDTA complex

(Further reading: Vogel's Quantitative Inorganic Chemistry Practical book, pp 309-)

**Exp. 2:** Determine the amount (in g/L) of  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  in a given unknown strength solution with the help of a known strength  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  solution using EDTA solution as an intermediate.

[Molecular weight of  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  = 246.5. Given EDTA solution strength: M/50.]

**Theory:** This determination is based on complexometric titration. The amount of  $\text{Mg}^{2+}$  can be directly determined using EDTA as complexing/chelating agent. A metal ion indicator, (such as Solochrome black or Eriochrome black T (EBT)) is used along with EDTA which helps to understand the end point of the titration. Metal ion solution such as aq.  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  whose concentration is to be determined is made alkaline by using ammonium buffer (aq.  $\text{NH}_3$  and  $\text{NH}_4\text{Cl}$ ) (to maintain pH in range of 7-11) and a pinch of EBT. This solution is then titrated with EDTA solution of known concentration. Initially, metal ions forms 1:1 complex with both EDTA and EBT indicator. While the Mg-EDTA complex is colorless, Mg-EBT complex is of wine-red color (and more stable than the former). (Note: Thus the wine-red color of the solution in conical is due to Mg-EBT complex.) When EDTA solution is added in this solution, the free  $\text{Mg}^{2+}$  reacts with EDTA and makes 1:1 complex. After complete reaction with free  $\text{Mg}^{2+}$  ions, EDTA displaces indicator anion from Mg-EBT complex with the formation of more stable Mg-EDTA complex and the color of the titrating mixture changes from wine red color to blue color at the end point. (Note: At this point, the blue color is just the color of free indicator at that basic pH).

In this experiment, a known strength of  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  solution is prepared and titrated with the given unknown strength EDTA solution to determine its strength. The same EDTA solution is then used to for the determination of the strength of unknown  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  solution.

**Procedure:**

- 1) Exact x.xxxx g (should be close to 0.500 g to prepare approx.. M/50 solution, similar to supplied EDTA solution)  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  was weighed and dissolved with exact 100ml distilled water in a 100 ml volumetric flask. (Note: follow the instruction given in practical class. Remember: Utmost take care is necessary for complete transfer of  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  and subsequent volume makeup. Weight should be recorded carefully).
- 2) 10ml of this prepared  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  solution is taken in a conical flask with a pipette and 2ml of ammonium buffer solution and one pinch EBT indicator were added in it. The color of the solution become wine- red on mixing.

- 3) This solution was then titrated with given unknown strength EDTA solution (taken in the burette) until the wine red color turns to blue.
- 4) Repeat the titration to get the concordant readings.
- 5) The procedure was repeated with the unknown strength (given)  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  solution and concordant readings were obtained.

**Observation and Calculation:**

Weight of the empty weighing glass tube ( $W_1$ ) = .....g

Weight of the weighing glass tube +  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  salt ( $W_2$ ) = .....g

Weight of the  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  salt transferred ( $W_2 - W_1$ ) = x.xxxx g

So, strength of 100ml  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  solution prepared ( $M_1$ ) =  $[(W_2 - W_1) \times 1000] / [246.5 \times 100]$  (M) (Note: should be about 0.02M as EDTA supplied is of similar strength)

Table 1: Titration of the 10 ml known strength  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  solution with unknown strength EDTA solution

No. of observations	Initial burette readings of EDTA (ml)	Final burette readings of EDTA (ml)	Volume of EDTA used (ml)	Concordant volume of EDTA ( $V_2$ , ml)
1				
2				
3				
4				

Table 2: Titration of the 10 ml unknown strength  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  solution with the same EDTA solution

No. of observations	Initial burette readings of EDTA (ml)	Final burette readings of EDTA (ml)	Volume of EDTA used (ml)	Concordant volume of EDTA ( $V_4$ , ml)
1				
2				
3				
4				



- a) For known strength  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  solution (to determine the strength of supplied EDTA):

$$M_2 \times V_2 = M_1 \times V_1$$

Where,

$M_1$  = the molarity of the aq.  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  solution (known)

$M_2$  = the molarity of the supplied aq. EDTA solution

$V_1$  = the volume of the prepared/standard aq.  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  solution = 10 ml

$V_2$  = the volume of the supplied aq. EDTA solution (burette reading)

So, the strength of the supplied aq. EDTA solution ( $M_2$ ) =  $M_1 \times V_1 / V_2$  (M)

b) For unknown strength  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  solution:

$$M_3 \times V_3 = M_2 \times V_4$$

Where,

$M_3$  = the molarity of the unknown strength (supplied) aq.  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  solution

$M_2$  = the molarity of the supplied aq. EDTA solution

$V_3$  = the volume of the supplied aq.  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  solution = 10 ml

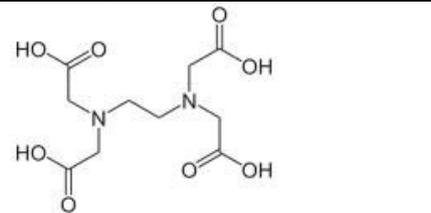
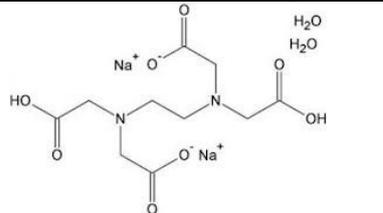
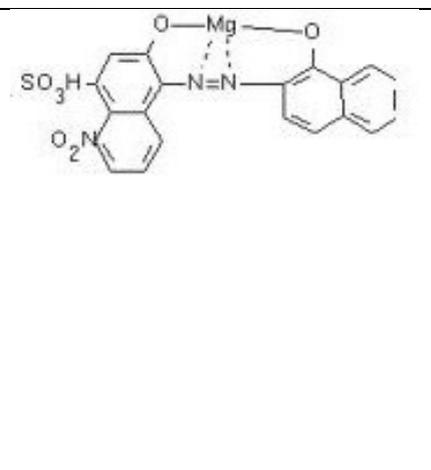
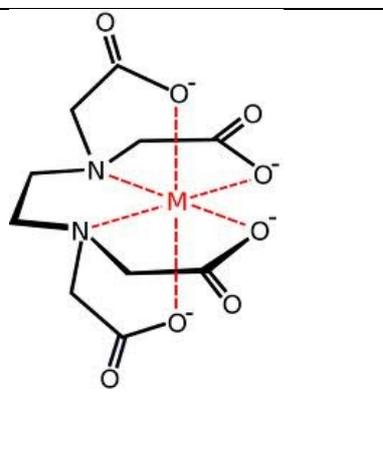
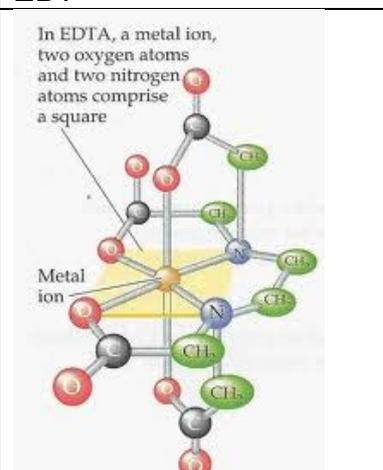
$V_4$  = the volume of the EDTA required (burette reading)

$$\begin{aligned} \text{So, the strength of the supplied } \text{MgSO}_4 \cdot 7\text{H}_2\text{O} \text{ solution, } M_3 &= M_2 \times V_4 / V_3 \text{ (M)} \\ &= (M_1 \times V_1 / V_2) \times V_4 / V_3 \text{ (M)} \end{aligned}$$

Therefore, the amount of  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  (in g/L) =  $M_3 \times 246.5$  (Molecular weight of  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ )

**Results:** The strength of  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  in the unknown  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  solution is x.xxxx g/L.

**Extra** (learn and draw these structures on white side of practical notebook pages):

		
EDTA	Disodium salt of EDTA	EBT
		
EBT-Mg complex	Mg-EDTA complex (or)	Mg-EDTA complex

(Further reading: Vogel's Quantitative Inorganic Chemistry Practical book, pp 309-)