

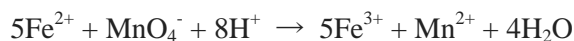
Application Note

Ferrous ion in pickling solution

Industry	:	Iron and steel
Instrument	:	Automatic potentiometric titrator
Measurement method	:	Potentiometric titration / Redox titration
Standards	:	

1. Scope

Ferrous ion (Fe^{2+}) in pickling solution was measured by redox titration. Zimmermann-Reinhardt reagent was added to the sample and then the solution was titrated with 0.02mol/L potassium permanganate (KMnO_4) solution. An inflection point on the titration curve was regarded as the end point, and the Fe^{2+} concentration was calculated from the volume of KMnO_4 solution consumed to titrate sample to the end point. The chemical formula of redox reaction of Fe^{2+} and KMnO_4 is shown below.



When hydrochloric acid is contained in a sample, a portion of KMnO_4 solution is consumed for a reaction for oxidizing chloride ion to chlorine, so that measurement error is caused. To preventing the error, Zimmermann-Reinhardt reagent which contains manganese ion (Mn^{2+}) was added to the sample solution.

2. Precautions

Handle the reagents in a well ventilated room or a draft chamber.

3. Post-measurement procedure

Wash the electrode with pure water and then keep it soaked in pure water so as not to dry the liquid junction of it.

4. Apparatus

Main unit : Automatic potentiometric titrator (preamplifier : STD)
 Electrode : Combined platinum electrode
 (inner solution : 3.3mol/L potassium chloride aqueous solution)

5. Reagents

Titrant : 0.02mol/L KMnO_4 aqueous solution
 Addition reagent : Zimmermann-Reinhardt reagent

6. Procedure

-Preparation of Zimmermann-Reinhardt reagent-

- 1) Add 90g of manganese(II) sulfate pentahydrate (MnSO₄·5H₂O) into a beaker.
- 2) Add 200mL of pure water and dissolve the MnSO₄·5H₂O.
- 3) Add 175mL of phosphoric acid and 350mL of sulfuric acid (1+1).*
- 4) Add pure water until the total amount of the solution becomes 1L.

*When adding phosphoric acid and sulfuric acid (1+1), the solution becomes hot by heat of dissolution, so that add them little by little while cooling. MnSO₄ is also precipitated by adding phosphoric acid. However, the precipitate is re-dissolved by adding pure water.

-Measurement-

- 1) Add 5mL of 10-fold diluted sample into a 200mL tall beaker.
- 2) Add 70mL of pure water and 30mL of Zimmermann-Reinhardt reagent.
- 3) Titrate with 0.02mol/L KMnO₄ aqueous solution to measure Fe²⁺ concentration.

7. Calculation

$$\text{Fe}^{2+} \text{ (g/L)} = (\text{EP1} - \text{BL1}) \times \text{TF} \times \text{C1} \times \text{K1} / (\text{R} \times \text{S})$$

EP1	:	Titer (mL)
BL1	:	Titer for blank titration = 0.0000mL
TF	:	Factor of titrant = 1.0195
C1	:	Concentration conversion coefficient = 5.585mg/mL
K1	:	Unit conversion coefficient = 1
R	:	Dilution factor = 0.1
S	:	Quantity of diluted sample (mL)

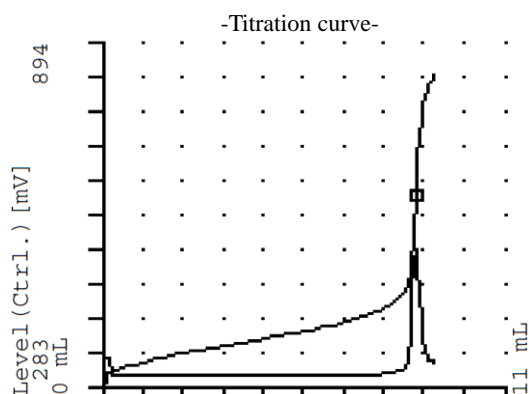
8. Example

-Titration parameter-

<u><Titr. Mode></u>	: Auto Intermit	<u><Ctrl. Para.></u>	
<u><Titr. Form></u>	: EP Stop	Number of EP	: 1
		End Sense	: Auto
<u><Titr. Para.></u>		Gain	: 1
Max. Volume	: 20 (mL)	Data Sampling	: Auto
Channel/Unit(Ctrl.)	: Ch2, mV	Ctrl. Speed	: Standard
Channel/Unit(Ref.)	: Off	Other Ctrl.	: Standard
pH Polarity	: Standard	Auto Int. Mode	: Standard
Titration Type Check	: No Check	Stirrer Speed	: 4
Direction	: Auto		
Wait Time	: 0 (s)		
Dose Mode	: None		

(The measurement parameter and the titration curve are an example of our automatic potentiometric titrator. In some titrators, parameter item may be different or other parameter items may be added.)

-Results-



-Measurement results-

	Quantity of diluted sample	Titer	Fe ²⁺
	(mL)	(mL)	(g/L)
1	5	8.4951	96.74
2	5	8.4745	96.51
3	5	8.4658	96.41
Mean	-	-	96.55
SD	-	-	0.17
RSD (%)	-	-	0.18

9. Summary

In this measurement, the results showed a good repeatability with 0.18% RSD (relative standard deviation).

In some samples, verification of the measurement capability is required. In such case, please contact us.

10. References

- 1) JIS K8137-2018 Iron(II) chloride tetrahydrate (Reagent)