

Application Note

Ferric ion in pickling solution

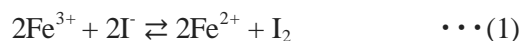
Industry	:	Iron and steel
Instrument	:	Automatic potentiometric titrator
Measurement method	:	Potentiometric titration / Redox titration
Standards	:	

1. Scope

Ferric ion (Fe^{3+}) in pickling solution was measured by redox titration.

Adding potassium iodide (KI) to a sample, iodine (I_2) is released by reaction between Fe^{3+} and iodide ion (I^-). Released free I_2 was titrated with 0.1mol/L sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) solution. An inflection point on the titration curve was regarded as the end point, and the Fe^{3+} concentration was calculated from the volume of $\text{Na}_2\text{S}_2\text{O}_3$ solution consumed to titrate sample to the end point.

The chemical formulas of the I_2 releasing and the titration of Fe^{3+} with $\text{Na}_2\text{S}_2\text{O}_3$ solution are given below.



Though chemical formula (1) is a reversible reaction, the reaction proceeds to the right side quantitatively in a presence of excess I^- and in a strong acid solution (equivalent to 0.1-0.4mol/L hydrochloric acid).

2. Precautions

- 1) Perform redox titration of Fe^{3+} under a strong acidic condition (equivalent to 0.1-0.4mol/L hydrochloric acid).
- 2) 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)

*If adjusting pH of solution, a pH glass electrode is required.

5. Reagents

Titrant	:	0.1mol/L $\text{Na}_2\text{S}_2\text{O}_3$ solution
Addition reagent	:	KI

6. Procedure

-Measurement-

- 1) Add 5mL of a sample into a 200mL tall beaker.
- 2) Add 100mL of pure water. *
- 3) Add 2g of KI and dissolve it, and then cap on the beaker and leave to stand in a dark place for 10 minutes.
- 4) Titrate with 0.1mol/L Na₂S₂O₃ solution to measure Fe³⁺ concentration.

*If acid concentration of the solution is out of a range equivalent to 0.1-0.4mol/L hydrochloric acid after adding pure water, adjust it by concentrated hydrochloric acid, 25w/v% sodium hydroxide solution or others.

7. Calculation

$$\text{Fe}^{3+} (\text{g/L}) = (\text{EP1} - \text{BL1}) \times \text{TF} \times \text{C1} \times \text{K1} / \text{S}$$

EP1	:	Titer (mL)
BL1	:	Titer for blank titration = 0.0000mL
TF	:	Factor of titrant = 1.0146
C1	:	Concentration conversion coefficient = 5.585mg/mL
K1	:	Unit conversion coefficient = 1
S	:	Quantity of sample (mL)

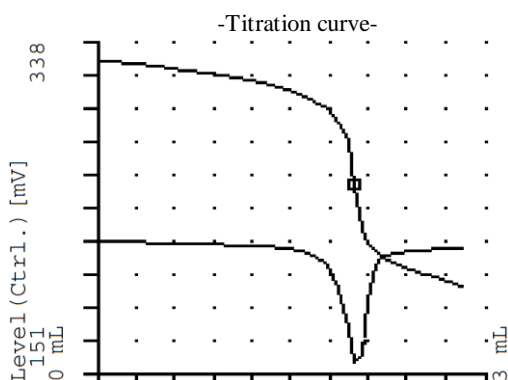
8. Example

-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 sample (mL)	Titer (mL)	Fe ³⁺ (g/L)
1	5	1.9368	2.19
2	5	1.9734	2.24
3	5	1.9670	2.23
Mean	-	-	2.22
SD	-	-	0.03
RSD (%)	-	-	1.19

9. Summary

In this measurement, the results showed a good repeatability with below 1.5% RSD (relative standard deviation).

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

10. References

- 1) S. Takagi, "Experiments and calculations of quantitative analysis", Vol.2, revised ed., KYORITSU SHUPPAN Co., Ltd. (1969)