京都電子工業株式会社
KYOTO ELECTRONICS MANUFACTURING CO.,LTD.

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 ($Na_2S_2O_3$) 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 $Na_2S_2O_3$ solution consumed to titrate sample to the end point.

The chemical formulas of the I_2 releasing and the titration of Fe^{3+} with $Na_2S_2O_3$ solution are given below.

$$2Fe^{3+} + 2I^- \rightleftarrows 2Fe^{2+} + I_2 \quad \cdots(1)$$

$$I_2 + 2Na_2S_2O_3 \rightleftarrows 2NaI + Na_2S_4O_6 \quad \cdots(2)$$

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 $Na_2S_2O_3$ 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 $Na_2S_2O_3$ solution to measure Fe^{3+} 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

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

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-

＜Titr. Mode＞ : Auto Intermit
＜Titr. Form＞ : EP Stop

＜Titr. Para.＞

Max. Volume	: 20 (mL)
Channel/Unit(Ctrl.)	: Ch2, mV
Channel/Unit(Ref.)	: Off
pH Polarity	: Standard
Titr. Type Check	: No Check
Direction	: Auto
Wait Time	: 0 (s)
Dose Mode	: None

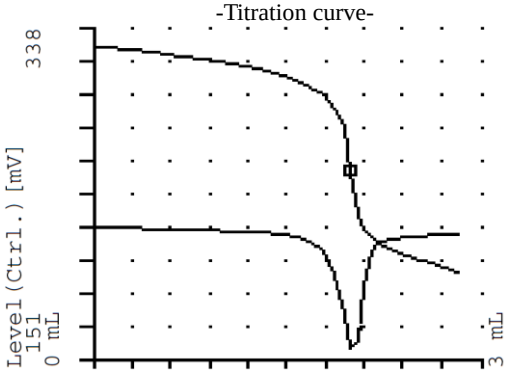
＜Ctrl. Para.＞

Number of EP	: 1
End Sense	: Auto
Gain	: 1
Data Sampling	: Auto
Ctrl. Speed	: Standard
Other Ctrl.	: Standard
Auto Int. Mode	: Standard
Stirrer Speed	: 4

(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-

-Titration curve-



-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)