

Iron and Steel

## Ferric Salt in Etching Solution

Redox titration by  
Automatic Potentiometric Titrator

Standard

JIS K 8142

### 1. Abstract

Here we demonstrate measurement of concentration of ferric salt in etching solution according to the method from technical literatures as referred below. To begin with, add hydrochloric acid, pure water and potassium iodide to precisely weighed sample in a conical flask with stopper, and seal it. Leave the flask for at least 5 minutes in a dark room. Then, titrate thus obtained free iodine (I<sub>2</sub>) with 0.1mol/L sodium thiosulfate to determine concentration of ferric salt.



Eq. (1) shows reversible reaction, but it shifts rightward with coexistence of potassium iodide in strong acid (0.1mol/L~0.4mol/L hydrochloric acid).

### 2. Reference

- 1) Experiment and Calculation in Quantitative Analysis - Vol.2 - Kyoritsu Publication
- 2) JIS K 8142-1994 Hexahydrate of Ferric salt (Reagent)

### 3. Cautions in measurement

- 1) Leaving the sample for 30 minutes in pretreatment of this test is referred to the method according to JIS K 8142-1994 Hexahydrate of Ferric salt (Reagent)
- 2) A convenient combination platinum electrode is used in this application, but the same measurement results can be obtained by using a combination of single platinum electrode and reference electrode.
- 3) For your information, there is another test method different from this application: use a reducer of lower potential than standard redox (+0.77V), thus ferric salt is shifted to ferrous salt followed by titration with potassium permanganate.

## 4. Post-measurement care

Rinse the electrode with water, and keep it dipped in pure water to avoid drying up.

## 5. Equipment

Main unit: Automatic potentiometric titrator (Standard preamplifier STD—)

Electrode: Option Combination platinum electrode

## 6. Reagent

Reagent : 0.1mol/L Sodium thiosulfate

Additive : Hydrochloric acid ( 2+1 ), Potassium iodide

## 7. Analytical procedure

—Preparation of diluted sample —

- 1) Weigh sample precise to 5g in a 100mL measure flask, and add pure water up to the marked line.

—Measurement—

- 1) Deliver diluted sample into a 200mL conical flask with stopper using a 10mL whole pipette.
- 2) Add 80mL water, 10mL hydrochloric acid (2+1) and 3g of potassium iodide.
- 3) Immediately seal it with stopper, and leave for 30 minutes in a dark room.
- 4) Titrate with 0.1mol/L sodium thiosulfate to obtain concentration of ferric salt.
- 5) Likewise, perform a blank test to correct titration volume.

## 8. Formula

Concentration of ferric salt ( % ) = ( EPl - BLl ) × TF × C1 × K1 / ( SIZE × C2 )

EPl : Titration volume ( mL )

BLl : Blank level ( 0.0391mL )

TF : Factor of reagent ( 1.006 )

C1 : Concentration coefficient ( 16.221mg/mL )  
(FeCl<sub>3</sub> equivalent to 1mL of 0.1mol/L Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>)

C2 : Dilution ratio( 0.1 ) (sample 10mL out of total 100mL diluted)

K1 : Unit conversion coefficient ( 0.1 )

SIZE : Sample size at time of diluting sample liquid ( g )

## 9. Example of measurement

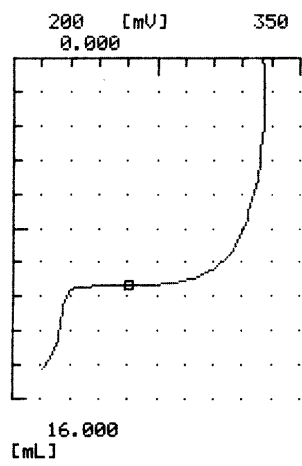
— Ambient condition —

Room temperature : 23 °C	Humidity : 56 %	Weather : Rainy
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### -Titration parameter-

Model : AT-510	
Method No. : 15	
Titr.mode : Auto	
Intermit	
Titr.form : EP Stop	
[Titration parameter]	[Result parameter]
Form : EP Stop	<Calculation>
APB No. : 1	Calc.Type : Sample
Unit No. : 1	Conc.1 : Set
Detector No. : 1	CO1=(EP1-BL1)*TF*C1*K
Unit : mV	1/(
Max. Volume : 20.0mL	SIZE*C2)
Wait Time : 0s	Unit : %
Direction : Auto	EP No. : 1
	Temp.Comp. : Off
[Control parameter]	<Constant>
End Point No. : 1	C1 : 16.221
End Sense : Auto	C2 : 0.1
End Point Area : Off	K1 : 0.1
Separation : Off	<Titr. Constant>
Over Titr.Vol. : 0mL	Factor : 1.006
Gain : 1	<Blank>
Data samp.Pot. : 4.0mV	Blank1 : 0.0391
Data samp.Vol. : 0.5mL	
Stability : 0.5mV/s	
Delay Time : 1s	
Limit Time : 30s	

### -Titration curve-

*** Result ***
Sample No. : 03-01
Date : 2001/05/30 15:00
Sample ID :
Method No. : 15
<Auto Intermit>
Method Name :
Titr.time : 00:07:08
Size : <u>5.0123g</u>
Conc-1 : <u>34.739%</u>
End point-1
Volume : <u>10.7095mL</u>
Potential : 260.5mV


(The above printout data were obtained from titration by AT-510 unit.)

#### «Titration parameter»

Form: of formula / APB No. the burette used in titration / Unit No. [APB Unit File number](#)  
 Detector No. the detector used in titration / Unit: potential unit in EP detection / Max Volume: of titration  
 Wait Time: before titration starts / Direction: of titration

#### «Control parameter»

End Point No.: number of EPs / End sense: of endpoint / End Point Area: detection area / Separation: of potential  
 Over Titr.Vol.: over-titration / Gain: sensitivity of detection signal / Data samp.Pot.: potential changes of sampling signal  
 Data samp.Vol. titration volume for sampling detection signal / Control Speed: of dosing / Stability: of EP sense  
 Delay Time: before stability check / Limit Time: for stability check

#### «Result parameter»

Calc.Type of formula / Conc.1: formula 1 / Unit: of result / EP No. of calculation / Temp.Comp.: temperature compensation  
 C1: concentration coefficient / C2: dilution ratio / K1: unit conversion coefficient / Factor: of reagent / Blank1: blank level 1 / Unit: of result

—Measurement results—

n	Sample (g)	Titration (mL)	FeCl <sub>3</sub> (%)
1	<u>5.0123</u>	<u>10.7095</u>	<u>34.739</u>
2	5.0123	10.7095	34.739
3	5.0123	10.6983	34.703

Concentration of FeCl <sub>3</sub>	
Mean	34.727 %
SD	0.0208 %
RSD	0.0599 %

\* The above results were obtained by 3 tests of the same sample.

\* Red underline shows the data from page 3/4.

## 10. Summary

Etching is a method of metal surface modeling or treatment making use of chemical corrosion, and the solution is used as reducing agent or dye mordant. Ferric salt is one of the most commonly used etching solutions and its quality control is emphasized on concentration level.

The sample measurement shows favorable results of a good repeatability with 0.06% relative standard deviation.

Precise and reliable measurement is assured by the automated potentiometry.

The concentration of ferric salt in etching solution can be perfectly measured by any of the following titration systems manufactured by Kyoto Electronics (KEM).

### 【AT-610】



#### Awarded Product of Supreme Technology from Kyoto City

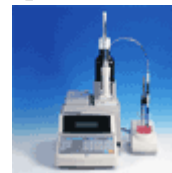
- Easy key entry by touch panel of large color LCD (8-inch wide)
- Simultaneous titration in parallel
- Both potentiometric and Karl Fischer moisture titration (coulometric + volumetric) can be performed at a time.

### 【AT-510】



- Compact and cost performance model
- PC card expands data memory for convenience and versatility.

### 【AT-500N-1】



- Low cost and high performance
- Easy view with back light LCD
- GLP/GMP conformed model

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