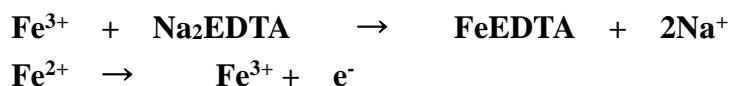


<b>HIRANUMA APPLICATION DATA</b>	Automatic Titrator	Data No.	G6	Apr. 5, 2019
<b>Metals</b>	<b>Determination of ferrous and ferric ion in steel cleaning solution</b>			

## 1. Abstract

A wide variety of the determination methods for each component in acidic solution containing ferrous ion ( $\text{Fe}^{2+}$ ) and ferric ion ( $\text{Fe}^{3+}$ ) has been developed. This report introduces an example of the successive determination for ferric and ferrous ions in the steel cleaning solution.

First,  $\text{Fe}^{3+}$  ions are determined by chelatometric titration with EDTA at acidic pH using salicylic acid as the indicator (purple  $\rightarrow$  yellow). The all  $\text{Fe}^{2+}$  ions are continuously oxidized to  $\text{Fe}^{3+}$  ion with ammonium peroxodisulfate. Finally, the  $\text{Fe}^{3+}$  ion oxidized from  $\text{Fe}^{2+}$  is determined by chelatometric titration with EDTA titrant as well as the above description.



## 2. Configuration of instruments and reagents

### (1) Configuration of instruments

Main unit : Hiranuma Automatic Titrator COM Series  
(M type photometric unit for photometric titration with 530 nm optical filter)

### (2) Reagents

Titration : 0.1 mol/L EDTA standard solution  
Oxidizing solution : Ammonium peroxodisulfate (Ammonium persulfate)  
Indicator reagent : 2 % salicylic acid in ethanol solution

## 3. Measurement procedure

- (1) Dispense 1 mL of sample into a 100 mL tall beaker with volumetric pipette.
- (2) Add 60 mL of DI water.
- (3) Add 0.2 mL of 2 % salicylic acid in ethanol solution.
- (4) Immerse photometric probe and start titration with EDTA standard solution.
- (5) After the endpoint for  $\text{Fe}^{3+}$  ion is detected, add approximately 1 g of ammonium peroxodisulfate during waiting time (S. Timer, 120 sec) to oxidize  $\text{Fe}^{2+}$  to  $\text{Fe}^{3+}$  ion.
- (6) Continuously titrate with EDTA standard solution.

## 4. Measurement conditions and results

### Example of titration condition

#### (1) Titration for Fe<sup>3+</sup> ion

Cnd. No.	1	Constant No.	1	Mode No.	5
Method	Auto	Size	1 mL	Pre Int	0 sec
Buret No.	1	Blank	0 mL	Del K	5
Amp No.	2	Molarity	0.1 mol/L	Del Sens	0 mV
D.Unit	T%	Factor	1.008	Int Time	3 sec
S- Timer	5 sec	K	55.85	Int Sens	3 mV
C.P. mL	0 mL	L	0	BrT Speed	2
T.Timer	0 sec	Unit	g/L	Pulse	40
D.P. mL	0 mL	Formula	(D-B)*K*F*M/S		
End Sens	500	Decimal Places	3		
Over mL	0 mL	Auto input parameter	None		
Max Vol.	20 mL				

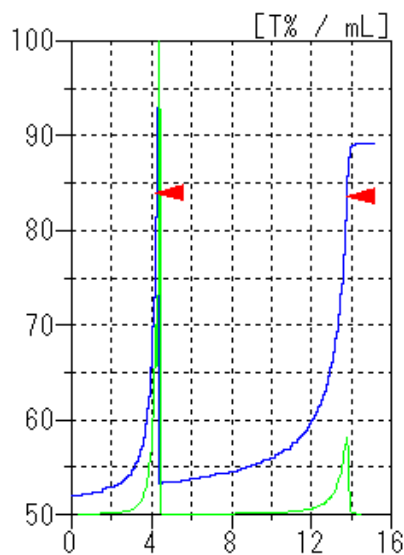
#### (2) Titration for Fe<sup>2+</sup> ion

Cnd. No.	2	Constant No.	2	Mode No.	5
Method	Auto	Size	1 mL	Pre Int	0 sec
Buret No.	1	Blank	0 mL	Del K	5
Amp No.	2	Molarity	0.1 mol/L	Del Sens	0 mV
D.Unit	T%	Factor	1.008	Int Time	3 sec
S- Timer	120 sec	K	55.85	Int Sens	3 mV
C.P. mL	0 mL	L	0	BrT Speed	2
T.Timer	0 sec	Unit	g/L	Pulse	40
D.P. mL	0.3 mL	Formula	(D-B)*K*F*M/S		
End Sens	500	Decimal Places	3		
Over mL	1 mL	Auto input parameter	None		
Max Vol.	20 mL				

\*Some measurement would not be possible depending on optional configuration of system.

## Measurement results

Number of Measurement	Size (mL)	Titrant Volume (mL)	Fe <sup>3+</sup> ion Concentration (g/L)	Titrant Volume (mL)	Fe <sup>2+</sup> ion Concentration (g/L)
1	1	4.178	23.521	9.406	52.953
2	1	4.162	23.431	9.454	53.223
3	1	4.220	23.757	9.537	53.690
Statistic calculation		Avg.	23.6 g/L		53.3 g/L
		SD	0.168 g/L		0.373 g/L
		RSD	0.71 %		0.70 %



Example of titration curve

### 5. Note

The following tips could improve measurement accuracy.

(1) Successive titration of Fe<sup>3+</sup> and Fe<sup>2+</sup> ions

The stability constant of EDTA Chelate for Fe<sup>2+</sup> ion is low at acidic pH, thus this method utilize the property that the coexisting Fe<sup>3+</sup> ion can selectively be reacted with EDTA. The applicable pH region is 2 ~ 3.

(2) Oxidizing agent for Fe<sup>2+</sup> ion

Ammonium peroxodisulfate is used as oxidizing agent for Fe<sup>2+</sup> ion in this report, but hydrogen peroxide is also usable instead. In either case, please note that the excessively added reagents are likely to generate air bubbles which interfere the photometric titration.

Keywords: Ferrous (Fe<sup>2+</sup>) ion, Ferric (Fe<sup>3+</sup>) ion, Chelatometric titration, EDTA, Photometric titration

\*Some measurement would not be possible depending on optional configuration of system.