AQUACOUNTER Application Sheet	COM series	DATA No. G6	1st edition

Metal

Quantification of ferrous ion (Fe^{2+}) and ferric ion (Fe^{3+}) in steel plate washing solution

1. Measurement outline

Various measurement methods have been developed as the methods to quantify each component in acidic solutions containing ferrous ion (Fe^{2+}) and ferric ion (Fe^{3+}). This section introduces an example in which ferric ion was quantified by chelate titration by EDTA in acidic range with salicylic acid as the indicator (purple → yellow) and then ferrous ion was quantified successively by chelate titration with EDTA in a similar fashion to the previous process after oxidizing all ferrous ions into ferric ions with ammonium persulfate.

$$Fe^{2+} - e^{-} \rightarrow Fe^{3+}$$
$$Fe^{3+} + Na_2EDTA \rightarrow FeEDTA + 2Na^{+}$$

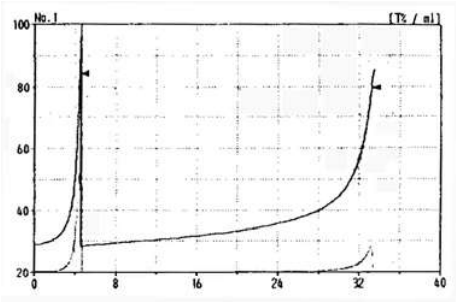
2. Reagents and Electrodes

(1) Reagents	Titrant	0.01mol/L EDTA titrant
	Oxidizing solution	1g ammonium persulfate
	Indicator	0.2mL 2% salicylic acid ethanol solution used
(2) Electrodes	Photometric probe with 530 nm filter	

3. Measurement conditions example (for COM-1600M w/ Photometric unit)

Master file 1 **Condition file: 1 + 2 + 3**					
Parameters for condition file 1 (For Fe^{3+})		**Parameters for condition file 2** (For Fe^{2+})		**Parameters for condition file 3** (For Calculation)	
Method	AUTO	Method	AUTO	Method	Calc
Amp No.	2	Amp No.	2	Size	0
Buret No.	1	Buret No.	1	Blank	0
Meas Unit	T%	Meas Unit	T%	Molarity	0
S Timer	10 sec	S Timer	60 sec	K	0
CP mL	0 mL	CP mL	0 mL	Unit	g/L
DP mL	0 mL	DP mL	0 mL	Formula	CA+CB
End Sens	200	End Sens	200		
Over mL	0 mL	Over mL	0 mL	**Mode No.**	5
Max. Vol.	20 mL	Max. Vol.	20 mL	Pre Int	0 sec
Unit	g/L	Unit	g/L	Del K	9
Blank	0	Blank	0	Del Sens	0 mV
Factor	Titre of the titrant	Factor	Titre of the titrant	Int Time	5 sec
Molarity	0.01	Molarity	0.01	Int Sens	3 mV
K	55.9	K	55.9	Brt Speed	2
Formula	(D-B)×K×F×M/S	Formula	(D-B)×K×F×M/S	Pulse	40
Mode No.	5	Mode No.	5		

4. Measurement example



Measurement results on Fe^{3+}

Sample No.	Sample volume (mL)	Titration value (mL)	Concentration (g/L)
1	1	4.414	24.80
2	1	4.404	24.74
3	1	4.364	24.52
	Avg.		**24.69 g/L**
	Std. Dev.		**0.15 g/L**
	C.V.		**0.60 %**

M

Measurement results on ferrous ion (Fe^{2+})

Sample No.	Sample volume (mL)	Titration value (mL)	Concentration (g/L)
1	1	28.732	161.42
2	1	28.755	161.54
3	1	28.672	161.08
	Avg.		**161.34 g/L**
	Std. Dev.		**0.24 g/L**
	C.V.		**0.15 %**

5. Outline

(1) About successive titration of ferrous ion and ferric ion

This measurement method applies that coexisting Fe^{3+} and EDTA react selectively since Fe^{2+} has small EDTA chelate stability constant in acidic range. pH2 – 3 is favored as the pH range in which measurement is possible.

(2) About Fe^{2+} oxidant

While this section used ammonium persulfate as the oxidant for Fe^{2+}, it is possible to use hydrogen peroxide as an alternative oxidant for this. Since it will exist in excess and tend to generate bubbles during titration when either reagent is used, caution is required that photometric titration may be interfered.

(3) Measurement by potentiometric titration

While this section used photometric titration for measurement, it is also possible to measure by potentiometric titration. Platinum electrode PT-301 (P/N D231244-A) is used as the indicator electrode, and RE-201 is used as the reference electrode. However, wrong end point may be detected when pH varies during the titration process, and it is necessary that titration is conducted by adding buffer so that the pH stays at 2 – 3.

Key words

Ferrous ion, ferric ion, chelate titration, salicylic acid, ammonium persulfate

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