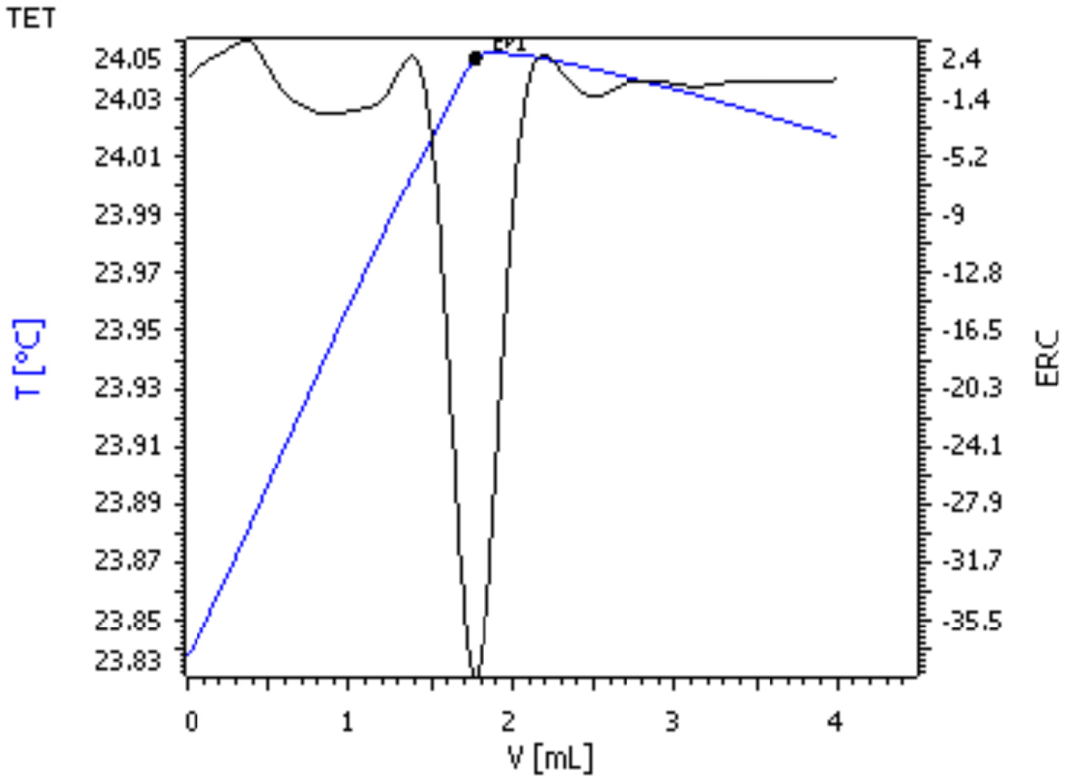
Titration Application Note H–119

Determination of ferric ion in acidic solutions



Determination of ferric ion in acidic and copper-free solutions using thermometric titration. The ferric ion is reduced by iodide. The released iodine reacts exothermically when titrated with thiosulfate solution.

Metrohm

Method description

Principle

Ferric ion is reduced to the ferrous state by iodide in mildly acidic solutions. The iodine released reacts exothermically when titrated by standard sodium thiosulfate solution.

$[Fe^{3+} + e^- \rightarrow Fe^{2+}] \times 2$
$2\ I^- \rightarrow I_2 + 2\ e^-$

$I_2 + 2\ e^- \rightarrow 2\ I^-$
$2\ S_2O_3^{2-} \rightarrow S_4O_6^{2-} + 2\ e^-$

$2\ Fe^{3+} + 2\ S_2O_3^{2-} \rightarrow 2\ Fe^{2+} + S_4O_6^{2-}$

Thus, 1 mol Fe^{3+} corresponds to 1 mol $S_2O_3^{2-}$

Samples

«Sample solutions» were prepared from reagent grade HCl, $Fe(NO_3)_3 \cdot 9H_2O$, and $Al(NO_3)_3 \cdot 9H_2O$ to approximate those that a customer desired to have analyzed. Due to the highly concentrated nature of the customer's own solutions, it was necessary to prepare them in such a manner that they represented a 1:4 dilution.

Nominal concentration of «sample solutions»:

	HCl g/L	Fe^{3+} g/L	Al^{3+} g/L
Sample A	*36.4***	*	56.9***
Sample B	*30.6***	*	0.5***
Sample C	27.1**	*	56.6***

* Results reported below
** Experimental details reported in **AN-H-118**
*** Experimental details reported in **AN-H-120**

Sample preparation

See above

Configuration

Basic equipment list for automated titration

814 USB Sample Processor	2.814.0030
859 Titrotherm	2.859.0010
Sample rack 24 x 75 mL	6.2041.340
Thermoprobe*	6.9011.020
Sample beaker 75 mL	6.1459.400
802 Rod Stirrer	2.802.0010
Stirring propeller 104 mm	6.1909.020
2 × 800 Dosino	2.800.0010
1 × Dosing unit 10 mL	6.3032.210
1 × Dosing unit 5 mL	6.3032.150
tiamo™	6.6056.222

* if determinations are run, where fluoride is present in the solutions, then use the fluoride-resistant HF Thermoprobe for Titrotherm (6.9011.040).

Solutions

Titrant	$c(Na_2S_2O_3)$ = 1 mol/L sodium thiosulfate solution
	glacial acetic acid
	c(KI) = 50% (w/v) potassium iodide
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Standard	potassium iodate, KIO_3, A.R., freshly dried at 110 °C
Standard solution	$c(KIO_3)$ = 0.1 mol/L in freshly-prepared dist. water

Analysis

Based on the anticipated Fe^{3+} content of the solution, employ an aliquot size calculated to give an endpoint volume of at least 1 mL, preferably 3–4 mL. Pipette this aliquot into a titration vessel, add 2 mL glacial acetic acid, and make to approximately 25 mL with dist. water. In this instance, 5 and 10 mL of the 1:4 diluted solutions were used as aliquots. Use a titration program where 5 mL of 50% (w/v) KI solution is added under stirring, and a «wait» period of 20 seconds is allowed before the addition of titrant commences. The titration is suitable for inclusion of an «automatic stop» parameter.

Preparation and standardization of 1 mol/L $Na_2S_2O_3$ solution:

Make an approximately 1 mol/L $Na_2S_2O_3$ solution with freshly prepared deionized water. Store and dispense from a brown glass reagent bottle. Keep in use.
Standardization is most accurately and conveniently performed using an automated *tiamo*™ program. Aliquots ranging from 2 to 10 mL may be automatically dispensed.
The *tiamo*™ program automatically computes the strength of the $Na_2S_2O_3$ solution by regression analysis, as well as the coefficient of determination of the analysis.

Metrohm

Method description

Parameters

Basic experimental parameters

Titrant dose rate (mL/min)	4
ERC EP1 (exothermic)	–10
Data smooting (“filter factor”)	40
Stirring speed (802 Rod Stirrer)	14
Evaluation start (mL)	0.5
Damping until (mL)	0.5

Calculations

$g/L\ Fe^{3+} = ((EP1 - blank) \times C001 \times C002)/C00$

EP1 = endpoint in mL
C00 = sample weight in mL
C001 = concentration of titrant in mol/L
C002 = molecular weight of Fe (55.845 g/mol)

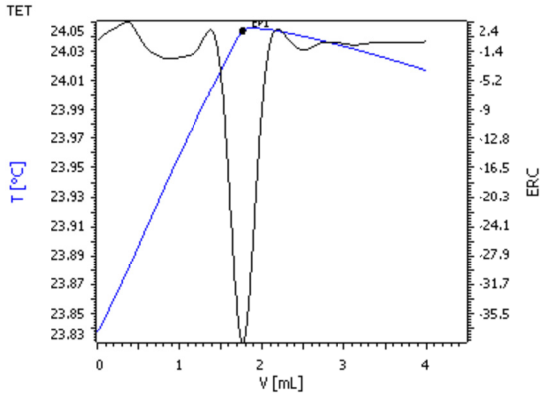
Results

Standardization of 1 mol/L $Na_2S_2O_3$:

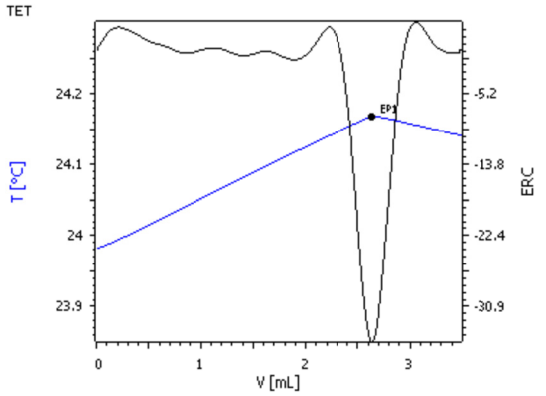
Molarity:	1.0017 mol/L
Blank:	0.069 mL
Coefficient of determination (R^2):	1.0000

Fe^{3+} in g/L	
Sample A	76.0 ± 0.07
Sample B	114.9 ± 0.16
Sample C	78.3 ± 0.15

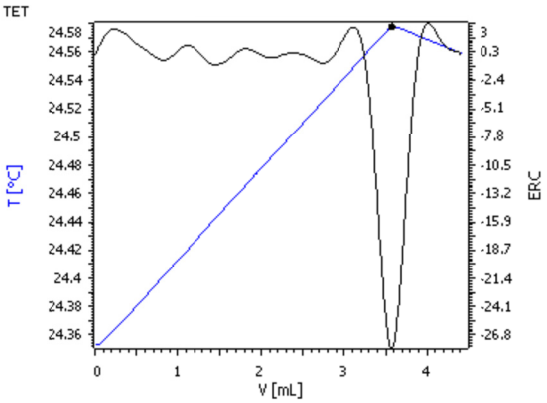
Titration Plots



Titration of sample solution A



Titration of sample solution B



Titration of sample solution C

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