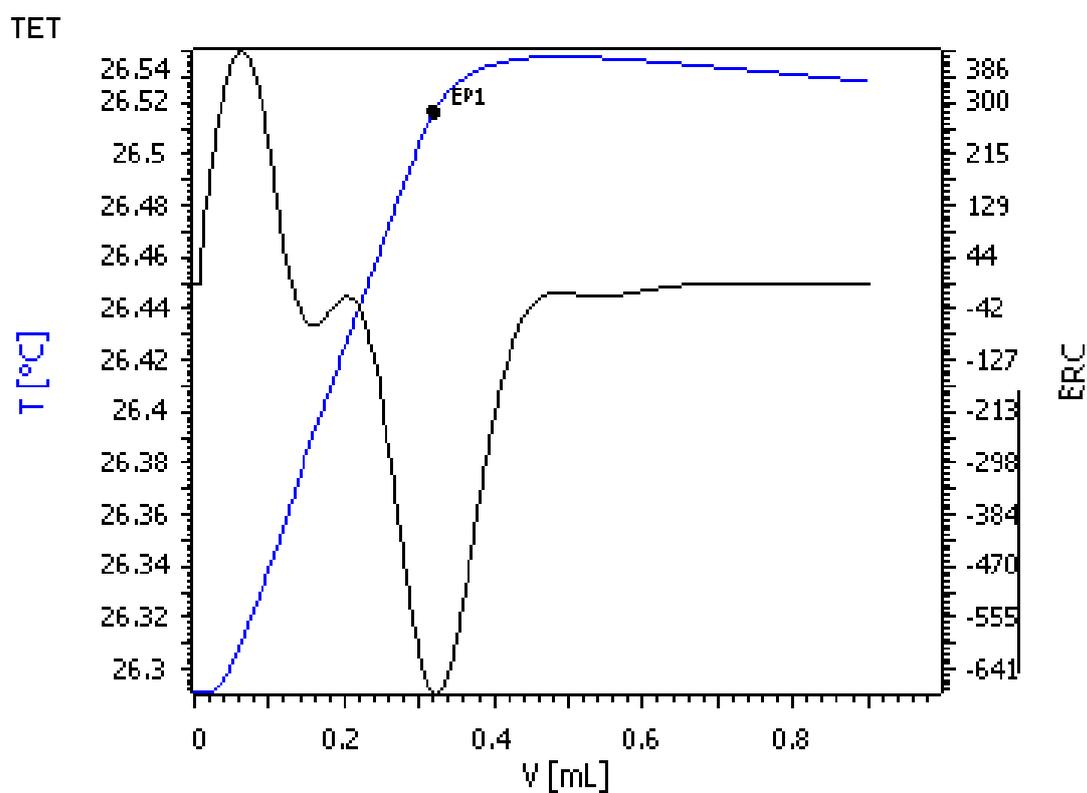


Determination of ferrous ion in acidic solutions with permanganate titrant

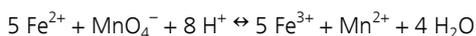
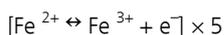
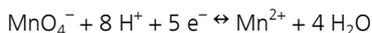


This Application Note looks at the determination of ferrous ion in acidic solutions by redox titration with a permanganate titrant using thermometric titration.

Method description

Principle

Permanganate ion in strongly acidic solution is a suitable titrant for the direct determination of ferrous ion.



Thus, 1 mol $\text{MnO}_4^- \equiv 5$ mol Fe^{2+} . The reaction is strongly exothermic. A disadvantage of permanganate is that solutions have no long-term stability and should be standardized for good accuracy. Solutions should be prepared with freshly produced DI water and stored in brown glass bottles with minimum headspace.

Samples

"Test solution"

Sample preparation

No sample preparation required

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, fluoride resistant | 6.9011.040 |
| Titration Head for Titrotherm | 6.9914.159 |
| Sample beaker/75 mL | 6.1459.400 |
| 802 Stirrer | 2.802.0010 |
| Stirring propeller (104 mm) | 6.1909.020 |
| 1 x 800 Dosino | 2.800.0010 |
| 1 x Dosing unit 10 mL | 6.3032.210 |
| <i>tiamo</i> TM | 6.6056.222 |

Solutions

| | |
|-------------------|--|
| Titrant | $c(\text{KMnO}_4) = 0.25$ mol/L potassium permanganate acid: 10% (w/v) H_2SO_4 solution |
| Acid | 10% (w/v) H_2SO_4 solution |
| Standard solution | $c(\text{Fe}^{2+}) = 0.3$ mol/L ferrous ammonium sulfate (FAS), $(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}$, prepared in 0.1 mol/L H_2SO_4 solution. Prepare |

freshly for each batch of KMnO_4 solution to be standardized. Calculate amount of FAS to be weighed, taking into account the stated purity of the reagent. Note: FAS is not a primary standard, but is suitable for standardizing KMnO_4 solutions used for routine analysis.

"Test solution"

A test solution of ≈ 1 g/L Fe^{2+} was prepared from FAS to simulate a customer solution containing a low level of Fe^{2+} . This was prepared by weighing approximately 1.77 g FAS into a 250 mL volumetric flask containing 10 mL 10% (w/v) H_2SO_4 and making to volume with DI water.

Analysis

Basic method (for ≈ 1 g/L Fe^{2+} solution):

Pipette a 20 mL aliquot of test solution into a titration vessel, add 10 mL 10% (w/v) H_2SO_4 , and titrate with $c(\text{KMnO}_4) = 0.25$ mol/L solution to an exothermic endpoint.

Standardization:

Using a specially constructed *tiamo*TM program, aliquots ranging from 4 to 20 mL in 4 mL increments were dispensed from a Dosino into titration vessels containing 10 mL 10% (w/v) H_2SO_4 and volumes of DI water to give a total volume of approximately 30 mL at the start of the titration. The *tiamo* program performs the regression analysis and calculates the molarity, titration blank and correlation coefficient of the regression line automatically.

Parameters

| | |
|----------------------------------|------|
| Titrant dose rate (mL/min) | 4 |
| ERC EP1 (exothermic) | -100 |
| Data smoothing ("filter factor") | 45 |
| Stirring speed (802 Rod stirrer) | 14 |
| Evaluation start (mL) | 0.5 |
| Damping until (mL) | 0.5 |

Method description

Calculations

$$\text{g/L Fe}^{2+} = ((\text{EP1} - \text{blank}) \times \text{C01} \times \text{C02} \times 5) / \text{C00}$$

EP1 = endpoint in mL

C00 = sample weight in mL

C01 = concentration of permanganate titrant in mol/L

C02 = molecular weight of Fe (55.845 g/mol)

Results

"Test solution" $\text{Fe}^{2+} = 0.99 \pm 0.00 \text{ g/L}$
(theoretical = 1.01 g/L)

Standardization:

$c(\text{KMnO}_4) = 0.2550 \text{ mol/L}$

blank = 0.043 mL

coefficient of determination, $R^2 = 1.0000$

Titration plot

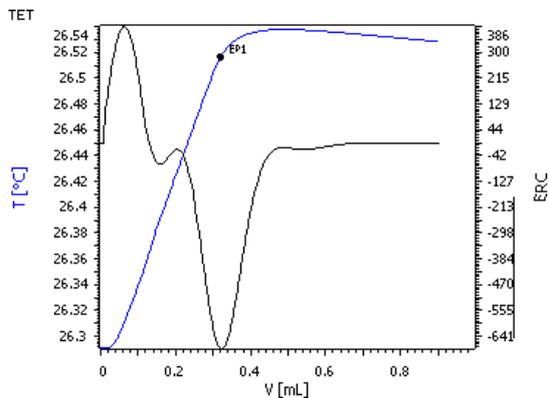


Fig. 1. Titration plot, Fe^{2+} with KMnO_4