Determination of Fe^{2+} next to Fe^{3+} (cerimetric)

Description

In samples that contain both iron (II) and iron (III) ions, the content of Fe^{2+} in addition to Fe^{3+} can be determined by titration with Cerium (IV) sulfate.
The Fe^{2+} is oxidized by Ce (IV) to Fe^{3+}:

$$Fe^{2+} + Ce^{4+} \rightarrow Fe^{3+} + Ce^{3+}$$

Fe^{2+} can already be oxidized by atmospheric oxygen, so work should be done quickly. The sample solution should also be protected from oxidation by CO_2, which e.g. can be generated directly in the titration vessel by adding a spatula tip to Sodium carbonate or Sodium hydrogen carbonate to the (strongly acidic) sample solution.

If the total iron content is to be determined in a sample that contains both iron (II) and iron (III), the Fe^{3+} must be reduced to Fe^{2+}. To reduce the Fe^{3+} e.g. Zinc can be used.

Instruments

Titrator	TL 5000 or higher
Electrode	Pt 62
Cable	L1A
Stirrer	Magnetic stirrer TM 235 or similar
Lab accessory	Erlenmeyer flask 100 ml with stopper
	Magnetic stirrer bar 30 mm

Reagents

1	Cer(IV)sulfate – solution 0,1 mol/L
2	Sulfuric acid 25%
3	Sulfuric acid conc.
4	Elektrolyte solution L3004 (für Pt 62)
5	Sodium carbonate
6	Zinc (powder or granules)
7	Distilled water
All reagents should be of analytical grade or better.	

Titration procedure

Reagents

$Ce(SO_4)_2$ – solution 0,1 mol/L

40,43g $Ce(SO_4)_2$ x $4H_2O$ and 50mL H_2SO_4 are dissolved in about 750 mL dist. Water. After cooling down it is made up to 1.0 L.
$Ce(SO_4)_2$ – solution 0,1 mol/L is also available as ready-to-use solution.

Cleaning and storage of the electrode

The electrode is rinsed with distilled water. The electrolyte solution L300 is suitable for storage of the Pt 62

Sample preparation Iron (II)

The sample is pipetted into a 150 mL beaker and dissolved in dist. Water. 20 mL H_2SO_4 25% and a spatula tip of Sodium carbonate is added and the mixture is made up to 80 mL with dist. Water. The solution is titrated with $Ce(SO_4)_2$ 0,1 mol/L to an equivalence point.

The required sample amount can be estimated according to this rule of thumb:

$$V(mL) = \frac{580 * Titer\ [\frac{mol}{L}]}{expected\ Fe - content\ [g/L]}$$

Sample preparation Iron (III)

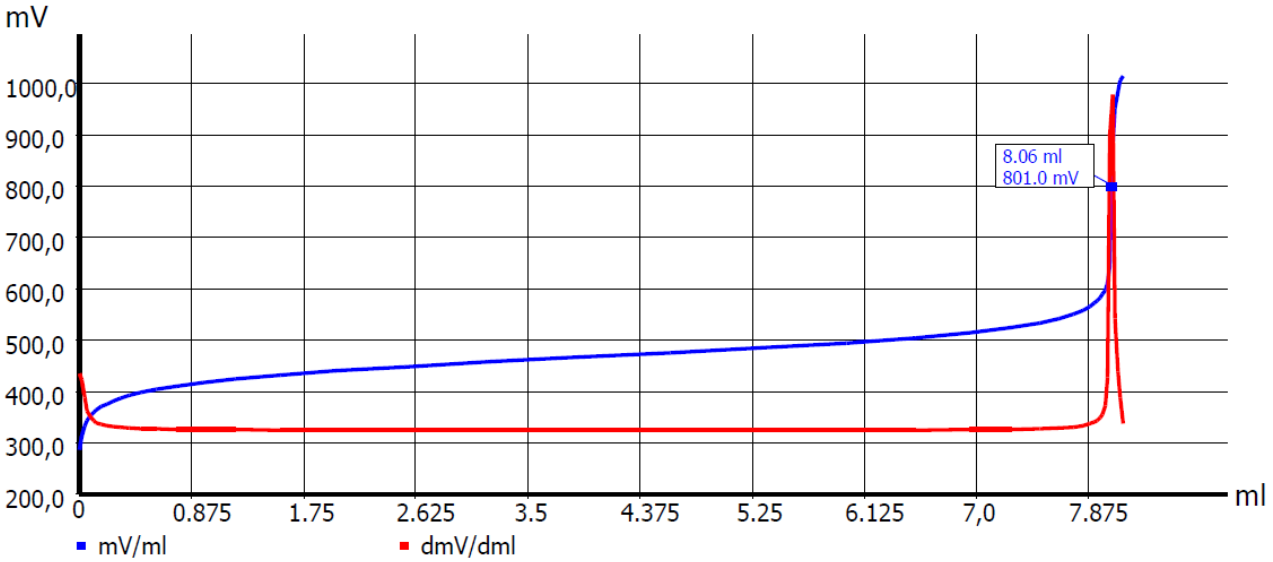
To determine the total iron in the sample which contains also iron (III), any iron (III) present is reduced with zinc.

The sample is pipetted into a 150 mL beaker and made up to 30 mL with dist. Water. 10 mL H_2SO_4 conc. and about 0.5 – 1g Zinc are added. The sample is stirred until the zinc has completely dissolved and the brown color of Fe^{3+} has disappeared. Undissolved zinc must be removed by filtration before the titration.

Then it is made up to 80 mL with dist. water and a spatula tip of Sodium carbonate is added.

The solution is titrated with $Ce(SO_4)_2$ 0,1 mol/L up to an equivalence point.

Titration parameter



Default method	---		
Method type	Automatic titration		
Modus	Dynamic		
Measured value	mV		
Measuring speed / drift	Individual	Minimum holding time	5 s
		Maximum holding time	15 s
		Measuring time	3 s
		Drift	10 mV/min
Initial waiting time	5 s		
Dynamic	steep	Max step size	1.0 ml
		Slope max ml	10
		Min. step size	0.02 ml
		Slope min. ml	230
Damping	none	Titration direction	increase
Pretitration	off	Delay time	0 s
End value	off		
EQ	On (1)	Slope value	1000
Max. titration volume	20 ml		
Dosing speed	100%	Filling speed	30 s

Calculation:

$$Fe\ [g/L] = \frac{(EQ1 - B) * T * M * F1}{V * F2}$$

B	0	Blank value
EQ1		Consumption of titrant at first Equivalence point
T	WA	Actual concentration of the titrant
M	55,845	Molecular weight
V	man	sample volume in mL
F1	1	Conversion factor
F2	1	Conversion factor

Any questions? Please contact the application team:

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