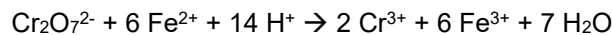


Determination of Chromium in galvanic bath

Description

In electroplating baths for chrome plating, the chromium content must be checked regularly. The titration of Chromium (VI) can be done by direct titration with Ammonium iron (II) sulfate in acidic solution. In the process, the Fe^{2+} is oxidized by the Cr (VI) to Fe^{3+} :



Cr (III) must be oxidized to Cr (VI) before titration. This can be done by boiling with Ammonium peroxodisulfate.

If Cr (III) should be determined in next to Cr (VI), the sample is divided and the Cr (VI) content is determined in one portion, and the total Chromium content is determined in another portion after oxidation with Peroxodisulfate. The Cr (III) content can be calculated from the difference.

The Chromium (VI) content is calculated in g / L CrO_3 . Cr (III) is calculated in g / L Cr_2O_3 .

Instruments

Titration	TL 7000 or higher
Exchangeable head	WA 20
Electrode	Pt 62
Cable	L 1 A
Stirrer	Magnetic stirrer TM 235
Lab accessory	Beaker 150 ml
	Magnetic stirrer bar 30 mm

Reagents

1	Ammonium iron (II) sulfate solution, $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2$ 0.1 mol/L
2	Sulfuric acid conc.
3	Ammonium peroxodisulfate.
4	Elektrolyte solution L3004 (für Pt 62)
7	Distilled water
All reagents should be of analytical grade or better.	

Titration procedure

Reagents

Ammonium iron (II) sulfate – solution 0.1 mol/L

47,1g $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \times 6 \text{ H}_2\text{O}$ are dissolved in about 750 mL dist. Water and 20 mL conc. H_2SO_4 are added. After cooling down it is made up to 1.0 L.

Ammonium iron (II) sulfate – solution 0.1 mol/L is also available as ready-to-use solution.

The titer determination is done as described in the application "Titer determination of ammonium iron (II) sulfate".

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 Chromium (VI)

The sample is pipetted into a 150 mL beaker, 10 mL conc. H_2SO_4 are added and the mixture is made up to 60 - 80 mL with dist. Water. The solution is titrated with Ammonium iron (II) sulfate 0.1 mol/L to an equivalence point.

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

$$V(\text{mL}) = \frac{350 * \text{Titer} \left[\frac{\text{mol}}{\text{L}} \right]}{\text{expected CrO}_3 - \text{content} [\text{g/L}]}$$

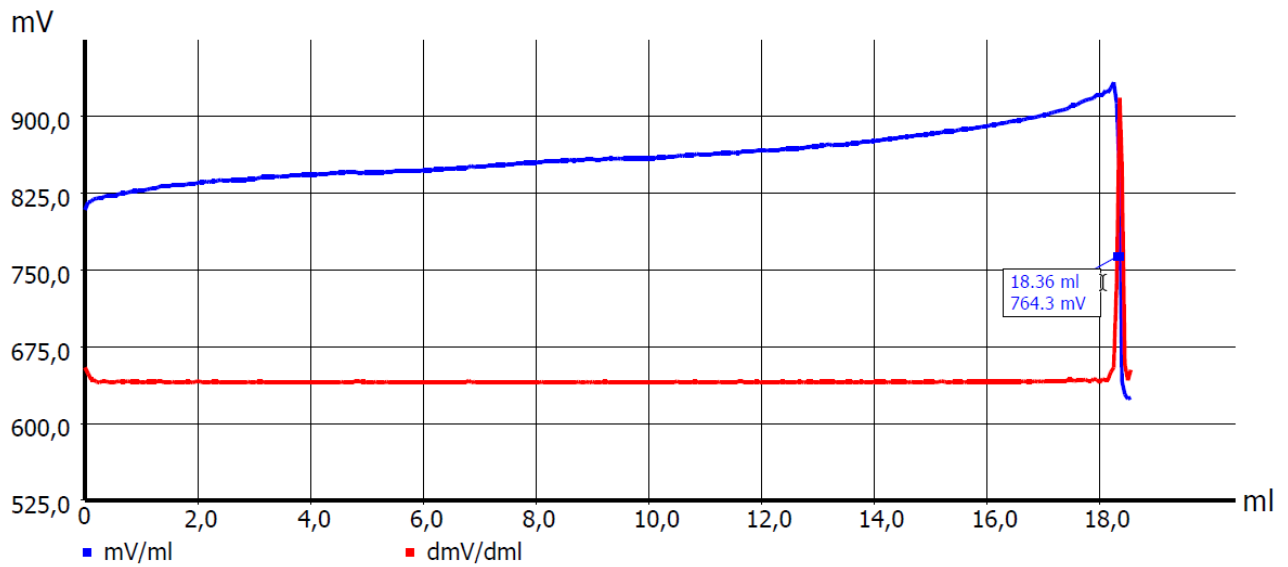
Sample preparation Cr (III) and total Chromium

For the determination of Cr (III) or the total chromium, the Cr (III) contained must first be oxidized to Cr (VI) with ammonium peroxodisulfate.

The same amount of sample as used for the Cr (VI) determination is pipetted into a 250 mL beaker, 10 mL conc. H_2SO_4 are added and made up with dist. water to approx. 150 mL. 2 – 2.5g Ammonium peroxodisulfate are added and the solution is heated to boiling for approx. 20 min. Excess Peroxodisulfate is destroyed due to this heating.

The solution is titrated with Ammonium iron (II) sulfate 0.1 mol/L to an equivalence point.

Titration parameter



Default method	---		
Method type	Automatic titration		
Modus	Linear		
Measured value	mV		
Measuring speed / drift	Individual	Minimum holding time	1 s
		Maximum holding time	10 s
		Measuring time	1 s
		Drift	50 mV/min
Initial waiting time	5 s		
Linear steps	0.05 mL		
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

* If necessary, a pre-titration can be set. The pre-titrated volume should be calculated in such a way that at least 1 ml is titrated linearly before EQ.

Calculation Chromium (VI):

Cr (VI) is calculated as CrO₃.

$$CrO_3 [g/L] = \frac{(EQ1 - B) * T * M * F1}{V * F2}$$

B	0	Blank value
EQ1		Consumption of titrant at first Equivalence point
T	WA	Actual concentration of the titrant
M	99,99	Molecular weight CrO ₃
V	man	sample volume [mL]
F1	1	Conversion factor 1
F2	3	Conversion factor 2

Calculation total Chromium:

Total Chromium is calculated as CrO₃ after oxidation with Peroxodisulfate:

$$CrO_3 [g/L] = \frac{(EQ1 - B) * T * M * F1}{V * F2}$$

B	0	Blank value
EQ1		Consumption of titrant at first Equivalence point
T	WA	Actual concentration of the titrant
M	99,99	Molecular weight CrO ₃
V	man	sample volume [mL]
F1	1	Conversion factor 1
F2	3	Conversion factor 2

Calculation Chromium (III):

Cr (III) is calculated from the difference total Chromium – Chrom (VI) as Cr₂O₃:

$$Cr_2O_3 [g/L] = \frac{(Cr_{total} - Cr(VI)) * M_{Cr2O3}}{M_{CrO3}}$$

Cr _(ges)		Total Chromium in g/L CrO ₃
Cr(IV)		Cr (VI) in g/L CrO ₃
M _(CrO3)	99,99	Molecular weight CrO ₃
M _(Cr2O3)	151,99	Molecular weight Cr ₂ O ₃