

Thermo. Titr. Application Note No. H-014

Title: Determination of Free Acid Content of Solutions Containing Fe(III)

Scope: Determination of free acid in solutions containing metal ions, particularly Fe(III).

Principle: An aliquot of test solution is diluted with propan-2ol and titrated with standard KOH in isopropanol. The effect of titration in an alcoholic environment is considered to be twofold: metal salts are substantially precipitated from solution, and the relationship between pKa's of sulfuric acid and Fe(III) is considerably changed, enabling a clear separation of endpoints

Reagents: 1 mol/L KOH in isopropanol, standardized against potassium hydrogen phthalate
Propan-2-ol

Method: Basic Experimental Parameters:

Data rate (per second)	10
Titrant delivery rate (mL/min.)	2
No. of exothermic endpoints	1
Data smoothing factor	50

Procedure: For the purpose of illustration, synthetic test solutions were made approximately to the composition of actual copper refining solutions according to the table below. Solutions were prepared from concentrated sulfuric acid, ammonium iron (III) sulfate, ammonium iron (II) sulfate and copper (II) sulfate.

Method (continued):	Make-up of synthetic copper ore refining solutions		
	Analyte	“Ore Leach Solution”	“Copper Concentrate Solution”
	Free acid as H ₂ SO ₄ g/L	20	200
	Fe(III) g/L	10	1
	Cu(II) g/L	8	40
	Fe(II) g/L	20	2
	Analysis. Pipette an aliquot of test solution into a titration vessel, and dilute to approximately 25mL with propan-2-ol. Titrate to an exothermic endpoint with standard 1 mol/L KOH in propan-2-ol. For the purpose of obtaining basic analytical precision data reported here, serial dilutions were made. For routine analytical purposes, the use of positive displacement hand („air“) pipettes can yield satisfactory results		

Results:	Synthetic copper ore refining solutions		
	Solution type	Mean Free Acid, as H₂SO₄ g/L	Std. Deviation (n=5)
	“Ore Leach Solution”	22.9	0.05
	“Copper Concentrate Solution”	196.7	0.18

Calculation:

$$\text{Free acid g / L} = \frac{((\text{titre, mL} - \text{blank, mL}) \times M \text{ KOH} \times \text{FW H}_2\text{SO}_4)}{(\text{sample vol., mL} \times 2)}$$

Example:

$$\text{Free acid g / L} = \frac{((2.947 - 0.025) \times 1.000 \times 98.08)}{(6.25 \times 2)} = 22.9$$

Thermometric Titration Plots:

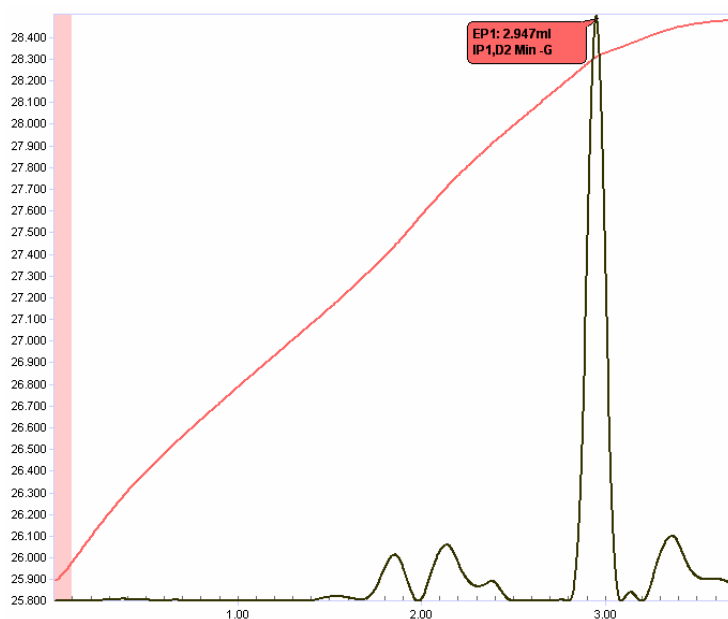


Fig. 1. Free acid in "ore leach solution"

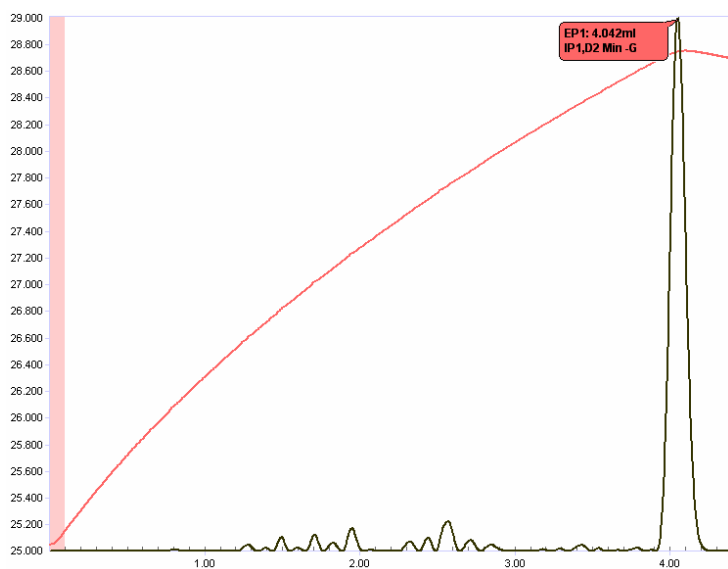


Fig. 2. Free acid in "copper concentrate solution"

Legend:

Red = solution temperature curve

Black = second derivative curve