Thermo. Titr. Application Note No. H-021

Title:	**Determination of Free Acid in Copper Refining Solutions**

***Scope*:**	Determination of free acid in copper refining solutions

***Principle*:**	Thermometric titration of “free acid” values in copper refining solutions with standard NaOH after complexation of Fe(III) with potassium oxalate solution. “Free acid” is expressed as g/L sulfuric acid.

Reagents:	Standard 1mol/L NaOH solution Saturated $K_2C_2O_4$ solution, approximately 300g/L

Method:	Basic Experimental Parameters:	
	Data rate (per second)	10
	Titrant delivery rate (mL/min.)	2
	No. of exothermic endpoints	1
	Data smoothing factor	50
	Basic method. Depending on likely free acid strength, pipette between 1 – 10mL of test solution into a titration beaker. Add 15mL saturated potassium oxalate solution, and dilute to approximately 25mL with D.I. water. Titrate to the first exothermic endpoint with standard 1 mol/L NaOH.	

Results: Analysis of synthetic and actual copper leach and refining solutions

Sample I.D.	Free Acid g/L H_2SO_4
Synth. Soln. ~9 g/L Fe (III)	9.73, 9.80
Synth. Soln. ~4.5 g/L Fe(III)	9.92, 9.94
Synth. Soln. ~2.25 g/L Fe(III)	9.99, 9.99
Synth. Soln. no Fe(III)	10.31, 10.33
Synth. Electrolyte, 0.2g/L Fe(III)	202.6, 203.0
Leach Soln. #1	1.86, 1.88
Leach Soln. #2	2.12, 2.20
Leach Soln #3	1.69, 1.65
“Final” Soln.	6.39, 6.34

Calculation:

$$g/L\ Free\ Acid = \frac{((Titre - blank) \times FW\ H_2SO_4 \times M\ NaOH)}{(sample\ \ vol, mL \times 2)}$$

$$g/L Free\ Acid = \frac{((4.067 - 0.031) \times 98.074 \times 1.0256)}{(1.00 \times 2)} = 203.0\ g/L$$

Thermometric Titration Plots:

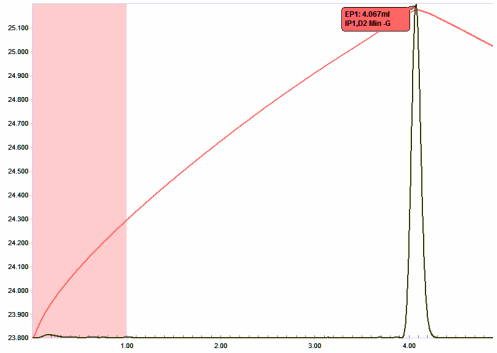

Fig.1. Free acid in electrolyte solutions

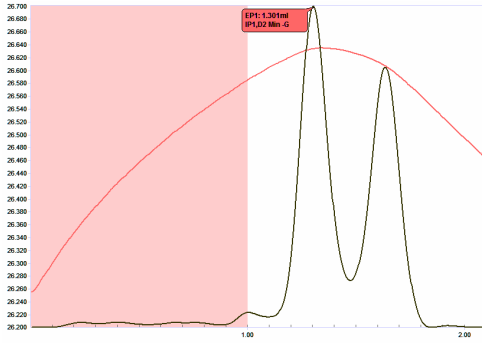

Fig. 2. Free acid in “final” solutions

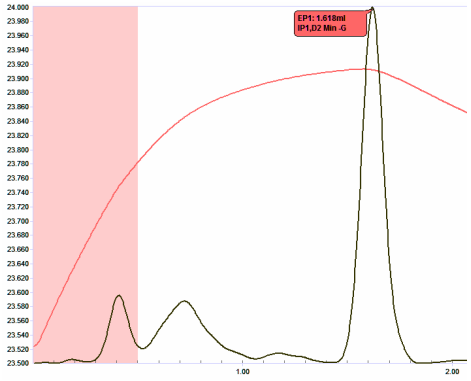

Fig. 3. Free acid in leach solutions

Legend:

Red = solution temperature curve

Black = second derivative curve