

Thermo. Titr. Application Note No. H-104

Title:	Determination of Free Acid in Hydrometallurgical Leach Liquors
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Scope:	Determination of the „free acid“ content of hydrometallurgical leach liquors
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Principle:	A measured amount of acidic hydrometallurgical leach liquor is treated with potassium oxalate solution to mask potential interference from Fe(III) and other metal ions, and then titrated with standard 1 mol/L NaOH solution
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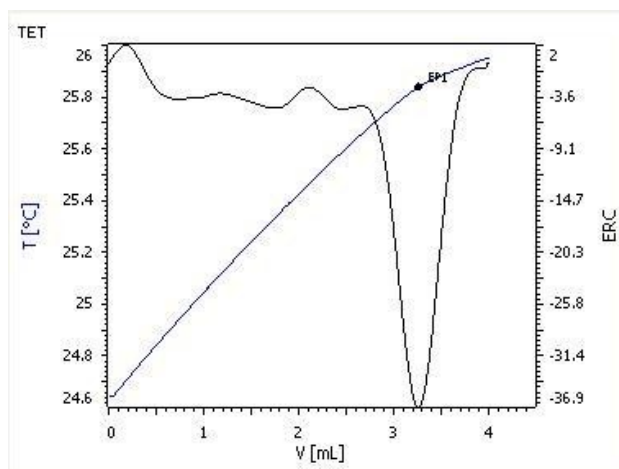
Reagents:	<p>Titrant: standard sodium hydroxide solution, c(NaOH) = 1 mol/L. Prepare from A.R. NaOH and standardize against A.R. potassium hydrogen phthalate, freshly dried at 110°C for 2 hours.</p> <p>Masking solution: 30% w/v potassium oxalate solution</p>
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Method:	<p>Basic Experimental Parameters:</p> <table> <tr> <td>Titrant delivery rate (mL/min.)</td> <td>4</td> </tr> <tr> <td>No. of exothermic endpoints</td> <td>1</td> </tr> <tr> <td>Data smoothing factor (DSF)</td> <td>50</td> </tr> <tr> <td>Stirring speed (802 stirrer)</td> <td>8</td> </tr> <tr> <td>Delay before start of titration (secs.)</td> <td>10</td> </tr> </table> <p>A 10mL aliquot of acidic process liquor is pipetted by volumetric glass pipette into a PP titration tube, and 10mL 30% w/w K₂C₂O₄ solution plus 10mL DI water added. The sample solution is swirled to mix prior to being placed in the sample rack.</p>	Titrant delivery rate (mL/min.)	4	No. of exothermic endpoints	1	Data smoothing factor (DSF)	50	Stirring speed (802 stirrer)	8	Delay before start of titration (secs.)	10
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Example:	<i>Acidic hydrometallurgical leach liquor, containing Fe(II), Fe(III), Mg, Al, Mn, Cr, Cu, Co and Ca.</i>
	15.7±0.06g/L (n=5), expressed as H ₂ SO ₄ equivalent

Calculations:	
Free acid, g/L =	$\frac{((EP \text{ vol.}, \text{ mL} - \text{Blank}, \text{ mL}) \times c(\text{NaOH}) \text{ mol/L} \times \text{FW H}_2\text{SO}_4)}{(\text{Sample vol.}, \text{ mL} \times 2)}$

Titration Plot:



Legend:

Blue curve = solution temperature

Black curve = second derivative (ERC)