Thermo. Titr. Application Note No. H-043

Title:	**Determination of Copper by Iodometric Titration**

***Scope*:**	Determination of copper, principally in copper mining and refining solutions. The method may also be used for determination of purity of copper metal. Optimal results are obtained when aliquots containg copper in the range approximately 3 – 6 mmole Cu are titrated.

***Principle*:**	Thermometric titration of copper with mixed sodium thiosulfate/potassium iodide titrant. Using this mixed titrant, the generation of iodine from iodide only just precedes its consumption by thiosulfate, thus minimizing volatilization losses and unwanted side reactions.

Reagents:	Titrant. 1 mol/L sodium thiosulfate, 1.2 mol/L potassium iodide. Buffer: 25% w/v ammonium bifluoride solution, $NH_4F.HF$

Method:	Basic Experimental Parameters:	
	Titrant delivery rate (mL/min.)	2
	No. of exothermic endpoints	1
	Data smoothing factor	50
	Stirring speed (802 stirrer)	6
	Delay before start (secs.)	15
	For this exercise, the purity of copper wire in electrical cable was determined. Degrease and dry sufficient wire foil to make 500mL of a 0.2 mol/L Cu(II) solution. Place the weighed amount into a 250mL wide mouth erlenmeyer flask, together with a PTFE coated magnetic spin bar. Transfer to a fume hood. Add 30mL concentrated A.R. nitric acid through a funnel which is intended to prevent loss of Cu . After the initial effervescence has subsided, ensure that all copper has dissolved before washing down the sides of the beaker with DI water. Make the volume to approximately 150mL with DI water, and place on a hot plate magnetic stirrer. Boil the solution while stirring vigorously for approximately 30 minutes, to ensure that nitrogen oxides have been expelled.	

	Finally, cautiously add ~1g sulfamic acid in small portions to eliminate the last traces of nitrogen oxides. Cool, and make to volume with DI water in a 500mL volumetric flask. Pipette an aliquot of solution, ideally containing between approximately 3 – 6 mmole Cu. Add 2.5mL $NH_4F.HF$ buffer, and make the titration solution to approximately 30mL with DI water.

Calculation:	
	$\%Cu\ w/w = \frac{((Titre, mL - blank, mL) \times FW\ Cu \times M\ Na_2S_2O_3 \times 100)}{(sample\ mass, g \times 1000)}$

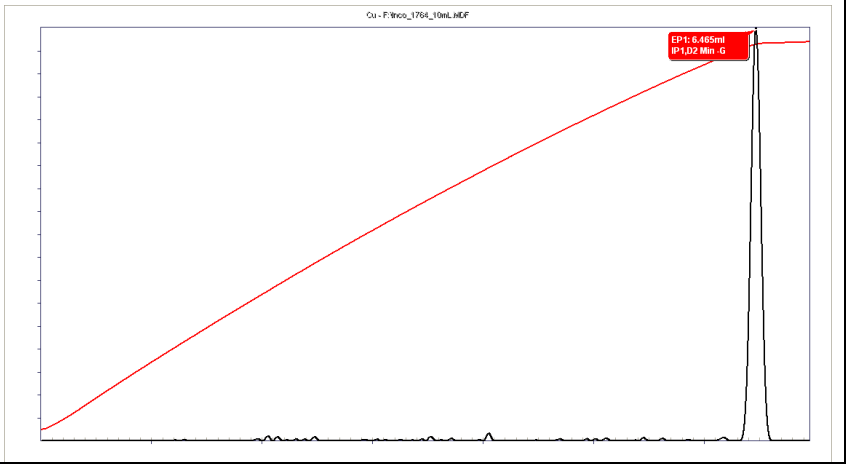
Results:	
Sample: electrical grade copper wire	%Cu = 99.90±0.06, %RSD = 0.06, n=8

Thermometric Titration Plot:

Legend:

Red = solution temperature curve

Black =second derivative curve



Cleaning:	
	The titration assembly (probe, stirrer and Dosino delivery tubes) accumulates an incrustation of CuI cuprous iodide with time. To ensure optimum performance, the Thermoprobe should be removed periodically and gently cleaned with a soft toothbrush moistened with water. If it is desired to remove the incrustion from the rest of the assembly, first soak it in a saturated potassium iodate (KIO_3) solution, followed by soaking in potassium iodide, KI, followed by a thorough rinsing with water.