Thermo. Titr. Application Note No. H-042

Title:	**Standardization of thiosulfate titrant for copper determinations**

***Scope*:**	Standardization of thiosulfate titrant for use in the determination of copper.

***Principle*:**	A mixed sodium thiosulfate/potassium iodide titrant is standardized for use in the determination of copper, employing a standard solution of high purity copper. It is intended that 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 $NH_4F.HF$ (“ammonium bifluoride”) solution, Standard copper solution. Degrease and dry sufficient high purity copper 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.

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

Results:

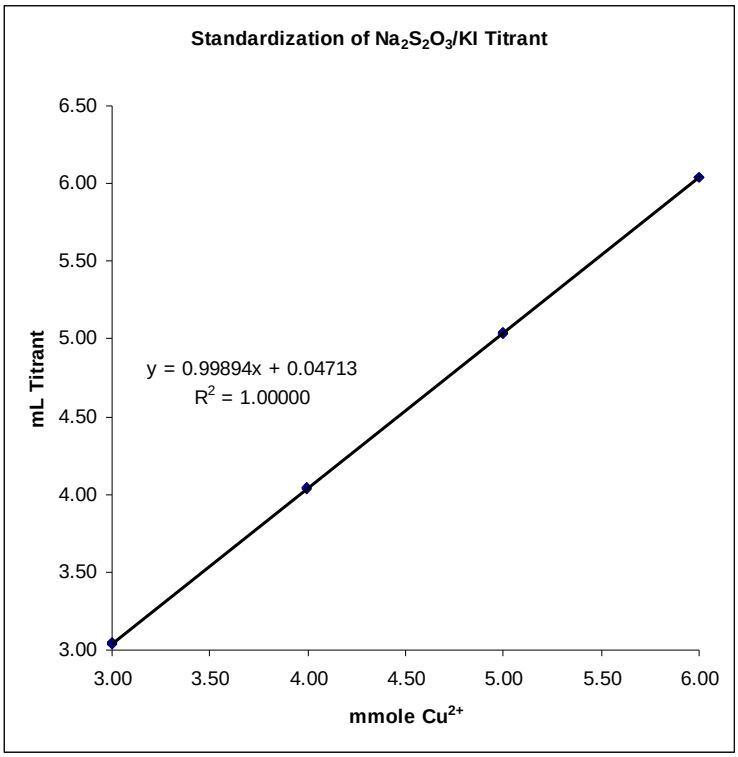
6.3588 Cu (certified min. 99.9%) dissolved and made to 500mL

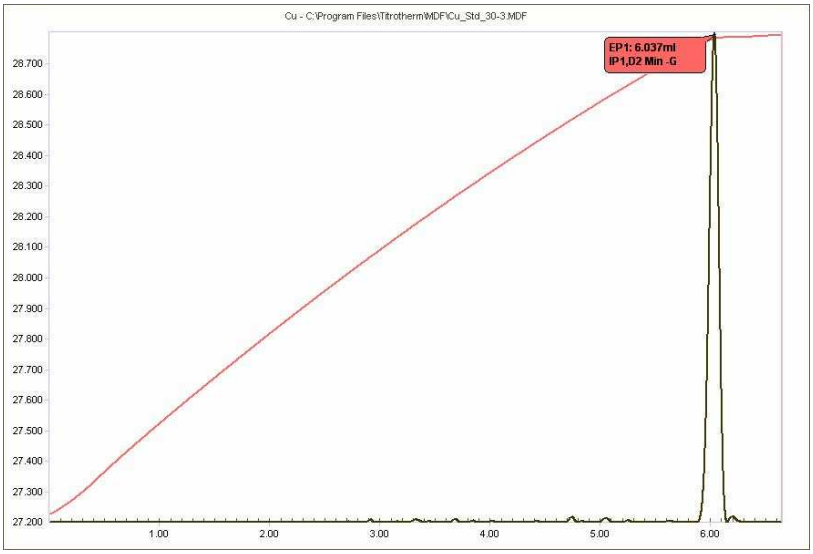
Aliquot, mL	mmole Cu^{2+}	Titre, mL
30	5.9976	6.042, 6.037
25	4.9983	5.037, 5.039
20	3.9986	4.040, 4.046
15	2.9990	3.044, 3.041

Determination of titrant strength and method blank:

Molarity = 1/gradient
= 1/0.99894
= 1.00106 mol/L

Method blank
= y-intercept = 0.0471 mL



Thermometric Titration Plot:	
Legend: *Red = solution temperature curve* *Black =second derivative curve*	

Cleaning:	
	With time, the titration assembly (probe, stirrer and Dosino delivery tubes) accumulates a deposit of cuprous iodide, CuI. 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 deposit 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.