Thermo. Titr. Application Note No. H-020

Title:	**Determination of Chromium in Leather Waste Solutions**

***Scope*:**	Determination of chromium in leather waste solutions in range 1000-30,000ppm

***Principle*:**	Chromium (III) is complexed with an excess of Na_4EDTA, the solution made ammoniacal with an NH_3/NH_4Cl buffer, and back-titrated with a standard copper solution. Although the stability constant of the Cr (III)/EDTA complex is high (pS=24.0), it reacts slowly with EDTA. It is necessary to heat the solution to ensure complete complexation with EDTA. The endpoint is revealed by the difference in the heats of complexation of Cu with EDTA and Cu with ammonia.

Reagents:	~1mol/L $CuSO_4$ solution 1mol/L Na_4EDTA solution. Prepared from Na_2H_2EDTA, neutralized with NaOH NH_3/NH_4Cl buffer. Add 142mL conc. NH_3 solution to 17.5g NH_4Cl, and dilute to 250mL with D.I. water Standard Zn solution, 0.5mol/L, prepared from A.R. Zn and HCl

Method:	Basic Experimental Parameters:	
	Data rate (per second)	10
	Titrant delivery rate (mL/min.)	1.5
	No. of exothermic endpoints	1
	Data smoothing factor	40
	Procedure: Pipette 50.00mL leather waste solution into a 150mL beaker equipped with a spin bar. Add 25.00mL ~1M Na_4EDTA solution. Cover beaker with a watch glass, and heat on a hot plate/magnetic stirrer to ~70°C for ~5 minutes. Allow to cool and transfer quantitatively to a 100mL volumetric flask. Make to volume and mix well.	
	Pipette a 20mL aliquot of this solution (equivalent to 10mL sample solution and 5mL Na_4EDTA solution) into a 140mL polypropylene titration beaker equipped with a spinring. Add 10mL of NH_3/NH_4Cl buffer, and titrate to an inflection	

with standard $CuSO_4$ solution. Record back titre as V_{BT}mL.

$CuSO_4$ and Na_4EDTA Standardisation. Pipette 5.00mL Na_4EDTA solution into a titration beaker containing a spinring. Add 10mL NH_3/NH_4Cl buffer, and 10mL D.I. water. Titrate to an inflection. Record the volume of $CuSO_4$ solution required to titrate it as V_{BL}mL. Repeat this titration at least in triplicate, and average values of V_{BL}

Now pipette 5.00mL Na_4EDTA solution into a titration beaker containing a spinring. Also pipette in 5mL standard Zn solution (molarity = M_{Zn}, volume = V_{Zn}), and add 10mL NH_3/NH_4Cl buffer, and 5mL D.I. water. Back titrate with $CuSO_4$ solution, recording volume as V_{BS}mL. Perform this titration at least in triplicate.

Determine molarity of $CuSO_4$ solution from:

$$M_{CuSO_4} = \frac{M_{Zn} \times V_{Zn}}{(V_{BL} - V_{BS})}$$

As long as the same volume of Na_4EDTA from the same batch is used throughout the determinations, the value of V_{BL} used during the standardisation can be used for the computations.

Results:	Analysis of leather waste solutions	
	Sample	Cr(III), mg/L
	A	8687±23 (n=4)
	B	7698±6 (n=4)

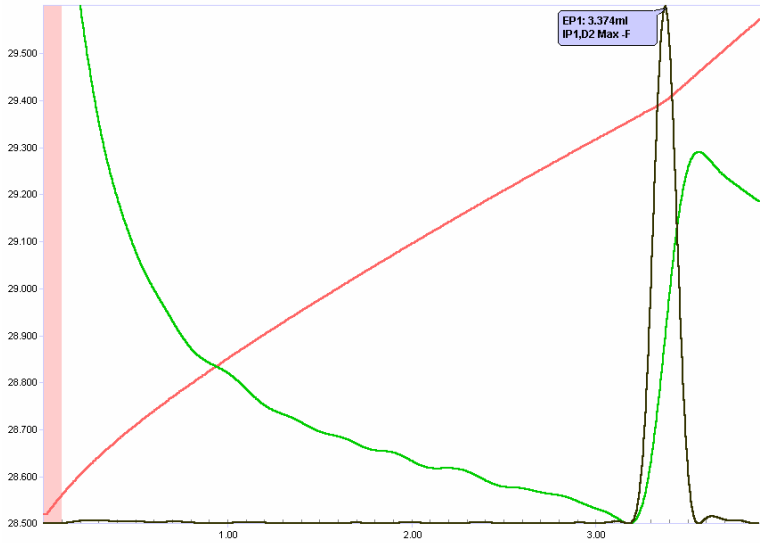
Calculation:

$$ppm\,Cr(III) = \frac{((V_{BL} - V_{BT}) \times M_{CuSO_4} \times FW\,Cr \times 1000}{Sample\ vol.,mL}$$

$$ppm\,Cr(III) = \frac{((5.00 - 3.555) \times 0.9158 \times 58.693 \times 1000)}{10}$$

= 7703 mg/L

Thermometric Titration Plot:



Legend:

Red = solution temperature curve

Green = first derivative curve

Black = second derivative curve