VA Application Note No. V - 128

Title:	Determination of total iron in the ppm range in a chromium electroplating bath

Summary:	The concentration of Fe(total) is determined polarographically in a chromium electroplating bath. The method is suitable for iron in concentrations in the ppm range. Fe(II) and Fe(III) show signals with the same sensitivity.

Sample:	Chromium electroplating bath
Sample preparation:	**Reduction of Cr(VI)** 250 µL Cr bath are diluted to 25 mL with oxalic acid (0.5 mol/L). The solution is slowly heated up while stirring until the color changes.

Analysis of Fe(total)

Oxalate buffer pH 2.0	c(ammonium oxalate) = 0.1 mol/L → adjusted to pH 2 with H_2SO_4
Measuring solution	5 mL ammonium oxalate solution + 5 mL ultrapure water + 250 µL reduced sample
Working electrode (WE)	**MME** (Multi Mode Electrode) 6.1246.020
Auxiliary electrode (AE)	**Pt** 6.0343.000
Reference electrode (RE)	Ag/AgCl/KCl (3 mol/L): 6.0728.020 + 6.1245.010

Parameters

Working electrode	DME
Stirrer speed	2000 rpm
Mode	DP
Purge time	300 s
Deposition potential	
Deposition time	0
Equilibration time	5 s
Pulse amplitude	50 mV
Start potential	+0.1 V
End potential	-0.5 V
Voltage step	6 mV
Voltage step time	0.6 s
Sweep rate	10 mV/s
Peak potential Fe	-150 mV

Results:	Fe
	3.5 g/L

Determination of Fe