

VA Application Note No. V - 191

Title:	Cadmium and lead in metallic materials as part of electrotechnical products
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Summary:	The EU directive on «Restriction of Hazardous Substances» (RoHS) requires the testing of four regulated heavy metals (Pb, Hg, Cd, Cr(VI)) in electrotechnical products. After sample preparation according to IEC 62321 the determination of lead and cadmium in metallic materials can be carried out by anodic stripping voltammetry (ASV) using ammonium oxalate buffer pH 2.
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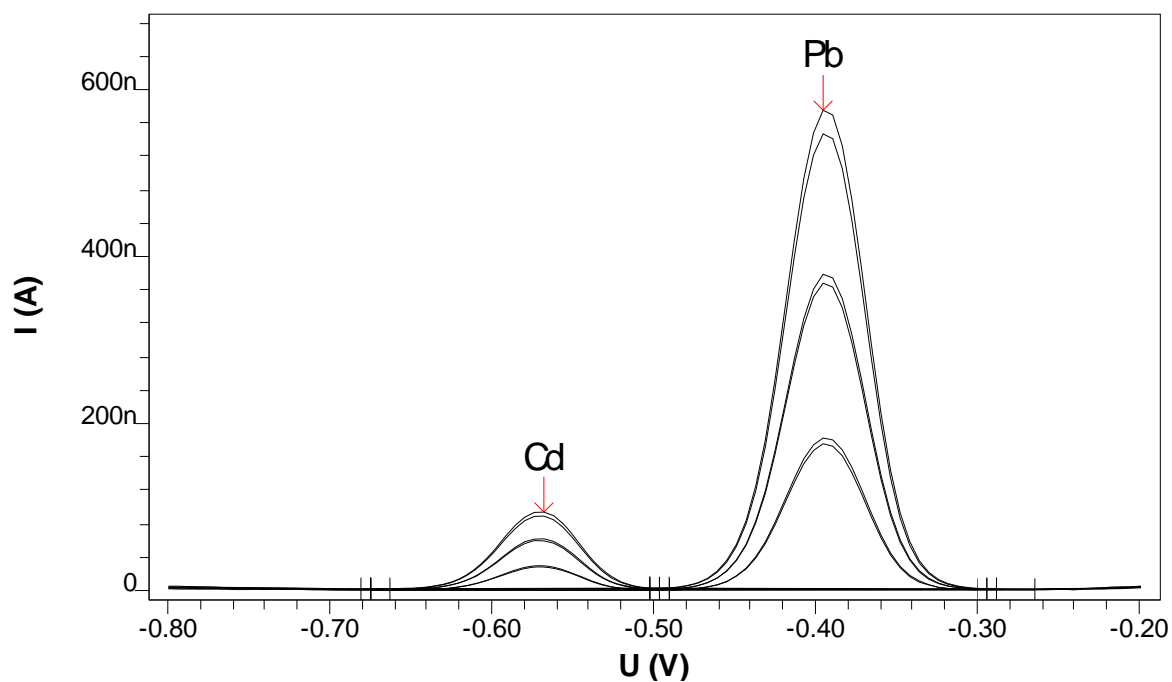
Sample:	Metallic materials
Sample preparation:	Approx. 1 g of sample is dissolved in a mixture of nitric acid and hydrofluoric acid as described in IEC 62321.

Analysis of Cd, Pb																								
Electrolyte	Ammonium oxalate buffer pH 2 c(ammonium oxalate) = 0.1 mol/L																							
Measuring solution	10 mL ultrapure water + 1 mL ammonium oxalate buffer pH 2 + 0.25 mL digested sample solution (equals approx. 2.5 mg of sample)																							
Working electrode (WE)	MME (Multi Mode Electrode) With silanized capillary	6.1246.020 6.1226.050																						
Auxiliary electrode (AE)	Pt	6.0343.000																						
Reference electrode (RE)	Reference system: Ag/AgCl/KCl (3 mol/L) Intermediate electrolyte: c(KCl) = 3 mol/L	6.0728.020 6.1245.010																						
Parameters	<table><tr><td>Working electrode</td><td>HMDE</td></tr><tr><td>Stirrer speed</td><td>2000 rpm</td></tr><tr><td>Mode</td><td>DP</td></tr><tr><td>Purge time</td><td>300 s</td></tr><tr><td>Deposition potential</td><td>-0.85 V</td></tr><tr><td>Deposition time</td><td>30 s</td></tr><tr><td>Equilibration time</td><td>10 s</td></tr><tr><td>Pulse amplitude</td><td>0.05 V</td></tr><tr><td>Start potential</td><td>-0.8 V</td></tr><tr><td>End potential</td><td>-0.2 V</td></tr><tr><td>Voltage step</td><td>0.006 V</td></tr></table>		Working electrode	HMDE	Stirrer speed	2000 rpm	Mode	DP	Purge time	300 s	Deposition potential	-0.85 V	Deposition time	30 s	Equilibration time	10 s	Pulse amplitude	0.05 V	Start potential	-0.8 V	End potential	-0.2 V	Voltage step	0.006 V
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	Voltage step time	0.6 s
	Sweep rate	0.01 V/s
	Peak potential Cd	-0.6 V
	Peak potential Pb	-0.4 V

Results:	Cd	Pb
	92.4 mg/kg	923.5 mg/kg

Determination of Cd and Pb



Cd
c = 92.427 mg/kg
+/- 3.996 mg/kg (4.32%)

Pb
c = 923.545 mg/kg
+/- 36.713 mg/kg (3.98%)

