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The Simultaneous Determination of Cu Pb Cd and Sb by Derivative Adsorption Chronopotentiometry

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abstract: The behaviour of the complexes of copper,lead,cadmium and antimony with salicylfluorone (SAF) adsorbed on HMDE has been investigated by derivative chrono-potentiometry in NH_3/NH_4Cl. The dependence of the peak height on the dt/dE *vs* E curve on preconcentration time, the pH of solution and the constant reducing current are discussed. This method achieved the simultaneous determination of copper, lead, cadmium and antimony in human hair.

Keywords: Simultaneous determination, Cu,Pb,Cd,Sb, complex, chronopotentiometry.

Anodic stripping voltammetry (ASV) is an effective electroanalytical technique for the measurement of trace metal ions. But there are serious interferences due to overlapping stripping peaks and forming of intermetallic complex in complicated system. The peak of copper (ASV) is close to that of mercury. So the result of determination of copper is not satisfactory. Adsorption chronopotentiometry was reported by H.Eskilsson *et al* [1]. In this method, instead of electrolytic accumulation of metal ions with potentiostatic reduction, inorganic complexes can be accumulated by adsorption. However, no adsorption chronopotentionmetric method for simultaneous determination of copper, lead, cadmium and antimony has been reported.

We have determination Cu[2], Pb[3], *etc*. by derivative adsorption chronopotentiometry. In this paper, we report a highly sensitive adsorption chronopotentiometric method for the simultaneous determination of copper,lead,cadmium and antimony.

Experimental

Apparatus: Stripping analyser (Model DPSA-3) was used. Three-electrode system consisted of a hanging mercury drop electrode (HMDE) as the working electrode,a platinum plate as the counter electrode and saturated calomel electrode (SCE) as the reference electrode.

Reagent: Stock solutions of copper, lead, cadmium and antimony were prepared. 1.0×10^{-3}mol/L stock solution of SAF was prepared. All solutions were prepared from analytical reagent grade chemicals and triple distilled water.

Results and discussion

In NH_3/NH_4Cl buffer (pH=9.60), no peaks of Cu(II), Pb(II),Cd(II) and Sb(V) appeared on the derivative adsorption chronopotentiograms (dt/dE *vs* E curve), and one peak of SAF was observed. The peak (P_1) potential was -1.16v. When a micro-amount of Cu(II) was added to the solution containing SAF, a new peak (P_2), appeared at -0.36v. When the same experiment was repeated after the addition of Pb(II), Cd(II), Sb(V), and additional peaks (P_2,P_3,P_4, and P_5) were obsvered . The peaks potentials were -0.58v, -0.70Vand -0.85v, respectively. With increasing concentration of Cu(II), Pb(II), Cd(II), Sb(v), the peak height of P_2, P_3, P_4, and P_5 increased, suggesting that P_2, P_3, P_4 and P_5 resulted from the Cu(II), Pb(II), Cd(II), Sb(v) complex with SAF. The peak heights of P_2, P_3, P_4, and P_5, $(dt/dE)_p$, were proportional to the preconcentration time and were dependent on the preconcentration potential, constant currents and also dependent on the ratio of concentration of NH_3 and concentration of NH_4Cl. When the ratio of NH_3 : NH_4Cl was 3:1(pH=9.60),the peaks height was the largest. The relationship between the peak height of the metal ions complex and the constant current, i_0, accorded with theoretical equaton,$(dt/dE)_p \propto i_0^{-1}$.

Under experimental conditions (0.005mol/L NH_3/NH_4Cl, t_a 30s, i_0 1 μA, 5.0×10^{-6}mol/L SAF), there was linear relationship between $(dt/dE)_p$ and the concentration of Cu(II), Pb(II), Cd(II) or Sb(v), in a range of 8.0×10^{-9}~ 6.0×10^{-7}mol/L for Cu(II), and 6.0×10^{-9}~7.0×10^{-7}mol/L for Pb(II), 1.0×10^{-8}~8.0×10^{-7}mol/L for Cd(II) and 5.0×10^{-9}~7.0×10^{-7}mol/L for Sb(V), respectively. The experimental results showed that ten thousand-fold excess of Na^+, K^+ did not interfere with the determination of Cu(II), Pb(II), Cd(II), or Sb(v), in addition, a five hundred-fold excess of Ca(II), Mg(II), eight-fold excess of Mo(VI),V(V), three-fold excess of Al(III), Sn(IV) or Fe(III) did not interfere with the determination of Cu(II), Pb(II), Cd(II) and Sb(V).

Typical results obtained by derivative adsorption chronopotentiometry with two successive standard additions for samples of human hair and the analysis of samples of hair are summarized in **Table 1**, the recoveries of added metal ions were 91%~98% for Cu, 90%~100% for Pb, 94%~102% for Cd and 94%~104% for Sb.

Table 1 Results of Determination of Cu,Pb,Cd and Sb of hair in samples

Sample	**Determined/ (μg. g^{-1})**				**Recovery/%**			
	Cu	Pb	Cd	Sb	Cu	Pb	Cd	Sb
1#	8.24	1.80	0.60	3.30	98	90	102	94
2#	7.36	0.88	1.26	2.25	90	100	92	95
3#	6.52	1.02	2.10	2.14	91	96	94	104

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