presence of 0.02% gelatin, two solutions with exactly identical composition, but with varying amounts of gelatin (0.02% for Pb and no gelatin for Sb) were used for recording the mixed polarograms. As Pb wave obeys Ilkovič's equation only up to 1.6 mM concentration, Pb is best determined when present in small quantities (< 1.6 mM i.e. corresponding to 6.632 mg of Pb) along with Sb. Presence of Pb, above 1.6 mM, however, did not affect the Sb wave. Calibration curves were recorded with synthetic mixtures. The composition of unknown samples were determined by comparing their wave heights with the calibration curves. With 4.9 mg of Pb^{2+} and 2.9–5.8 mg of Sb^{3+} errors amounted to $\pm 0.1\%$.

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Kurze Mitteilungen

Titrimetric Microdetermination of Nickel and Cobalt, Separately and in Presence of Each Other

Titrimetrische Mikrobestimmung von Nickel und Kobalt, allein und in Mischung

Best. von Nickel neben Kobalt; Volumetrie; Hippursäure als Reagens.

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A new method for the determination of Ni and Co, separately and in mixture is based on titration with hippuric acid. Xylenol orange (and chromazurol red S) are employed as indicators.

Maximum error observed for Ni and Co is 2.0% and 1.9%, resp., when determined separately and 1.9% and 1.9%, resp., when determined from a mixture. Amounts taken were in the range of about 60–400 mg. The following ions cause interferences: Pb^{2+} , Fe^{2+} , Ga^{3+} , Al^{3+} , Nd^{3+} , Gd^{3+} , Zr^{4+} , Te^{4+} , Nb^{4+} , Re^{6+} .

No interferences are caused by Ti^{+} , Mg^{2+} , Cd^{2+} , Be^{2+} , Ca^{2+} , Ba^{2+} , Zn^{2+} , In^{3+} , Pr^{3+} , U^{6+} .

Te^{4+} interferes in the case of Ni only, Sm^{3+} and Au^{3+} in the case of Co. A most important precaution in this method is a thorough cleaning of the beakers used with sodium carbonate or chromic acid, since otherwise the exact colour change may be not obtained.

Procedures

1. *Nickel or Cobalt Separately.* Add 2–3 drops of xylenol orange solution (0.1 g in 100 ml of dist. water) to about 20 ml of the sample solution (pH 4.3 for Ni and 4.9 for Co) containing 60 to 400 mg of Ni or Co. A pink colour develops in the case of Ni and a purple one in the case of Co. Titrate with standard (0.0034 N) aqueous hippuric acid solution to the colour change from pink to yellow or purple to yellow, respectively. Calculate the results on the basis of 1 metal atom to 2 molecules of reagent.

2. *Nickel and Cobalt in Presence of Each Other.* Add 3–5 drops of chromazurol red S solution (0.05 g in 100 ml of dist water) to about 20 ml of the sample solution (pH 4.6) containing 80 to 400 mg of Ni and 60 to 235 mg of Co. Titrate Ni in the first stage with hippuric acid solution to a colour change from orange to red. Now add to the same solution 2–3 drops of xylenol orange indicator and titrate Co also with hippuric acid to a colour change from yellowish to deep red.

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