Simultaneous Gravimetric or Volumetric Determination of Nickel and Cobalt as Isoquinoline Thiocyanate Complexes

Gravimetrische oder volumetrische Bestimmung von Nickel und Kobalt nebeneinander als Isochinolinthiocyanat-Komplexe

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Isoquinoline in combination with thiocyanate has been employed for a gravimetric and a volumetric procedure for the determination of nickel and cobalt in presence of each other. In the gravimetric method the elements are weighed as $\text{Me}(\text{C}_9\text{H}_7\text{N})_4(\text{SCN})_2$, in the volumetric method the elements are precipitated in the presence of isoquinoline and a known excess of thiocyanate, the latter being back-titrated mercurimetrically with diphenyl carbazone as indicator. Nickel is eliminated in both cases by dimethylglyoxime.

In the gravimetric determination, Bi^{3+} , Sb^{3+} , Sn^{2+} , Sn^{4+} , Pd^{2+} , Pt^{2+} , Hg^{2+} and alkaline earths were precipitated with isoquinoline itself, but can be removed by filtration before adding thiocyanate. Zn^{2+} , Cu^{2+} , Cd^{2+} interfere, while copper can be masked with thiosulphate (10 mg for each mg of copper). Mn^{2+} and Pb^{2+} do not interfere. In the volumetric determination Fe^{3+} , Cr^{3+} , Al^{3+} , Bi^{3+} , Sb^{3+} , S^{2-} , Sn^{4+} , Pb^{2+} , Mn^{2+} , alkaline earths and most of the platinum metals do not interfere. Cu^{2+} , Cd^{2+} and Zn^{2+} interfere. The sensitivity and tolerance of this reagent is much higher than that of most other reagents. By gravimetry 9–40 mg of Co and 3–32 mg of Ni were determined with errors between -0.01 to $+0.02$ and -0.02 to $+0.10\%$, respectively. By volumetry the errors were -0.02 to -0.15 and 0 to -0.03% for 4–20 mg of Co and 4–12 mg of Ni, respectively.

Experimental

1. Gravimetric Method. To the sample solution add 15 ml of 0.1 M isoquinoline solution (in 85% formic acid), and 0.2 M thiocyanate solution in amounts about twice than those calculated from the equation $\text{Me}^{2+} + 4(\text{C}_9\text{H}_7\text{N}) + 2\text{SCN}^- \rightarrow \text{Me}(\text{C}_9\text{H}_7\text{N})_4(\text{SCN})_2$. Optimum pH for the precipitation is >4 for Ni and 5.3–9.0 for Co. Make up the volume to 80–100 ml, warm on a water bath for 10–15 min, set aside for 10–20 min, filter over a G4 sintered crucible, wash

twice with water, dry at 90–110°C for 1–2 h and weigh as $\text{Me}(\text{C}_9\text{H}_7\text{N})_4(\text{SCN})_2$. The result corresponds to the sum of Ni + Co. In another aliquot of same size separate nickel by addition of 1% alcoholic solution of dimethylglyoxime, filtrate and determine Co in the same way as above. Ni content is calculated from the difference.

2. Volumetric Method. To the sample solution add 10 ml 0.10 M isoquinoline solution as above and 10 ml of 0.15 M potassium thiocyanate solution, make the volume to 25 ml, warm for 1–2 min on a water bath and filter through Whatman No. 42 filter paper. To 5 ml of the filtrate (the first few ml should be rejected) add 10–15 ml of water, adjust to $\text{pH} \approx 3.0$, add 1–2 drops of alcoholic diphenyl carbazone solution (1%) and titrate with 0.15 M mercuric nitrate solution until the appearance of violet colour. Thiocyanate consumed corresponds to the sum of Ni + Co. To another aliquot of the sample solution add 3–5 ml of dimethylglyoxime (1%), 10 ml of isoquinoline (0.1 M) and 10 ml of thiocyanate solutions (0.15 M), filtrate and titrate as above. The result corresponds to Co. Ni is calculated from the difference.

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Vanadimetric Determination of Isonicotinic Acid Hydrazide Using Redox Indicators and Application to Pharmaceutical Preparations

Vanadimetrische Bestimmung von Isonicotinsäurehydrazid unter Verwendung von Redoxindikatoren und Anwendung auf pharmazeutische Präparate

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A vanadimetric procedure for the determination of isonicotinic acid hydrazide (isoniazid) has already been proposed by Gowda and Rao [2]. It consists in adding an excess of vanadate to the sample solution and back-titrating with Mohr's salt. In that connection it was interesting that Berka, Vulterin and Zyka [1] stated that "vanadate cannot be titrated with isoniazid". We suppose that their difficulty was in the slow disappearance of the red colour of the com-