Intermittent A.C. Arc Method for Spectrographic Analysis of Tantalum Oxide

Spektrographische Analyse von Tantaloxid mit Hilfe eines intermittierenden Wechselstrombogens

Analyse von Tantaloxid; Spektralanalyse; intermitt. Wechselstrombogen

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A spectrographic method has been developed to determine Nb and eleven other trace impurities simultaneously in tantalum oxide. The method does not involve any preparation of sample and is, therefore, simple and straightforward.

Standards are prepared by dry-mixing the spec-pure grade oxides with tantalum oxide supplied by Johnson Matthey and Co. Standards and samples are glued directly on to the flat tops of a pair of graphite electrodes (UCC 107) and excited by a Hilger's low voltage intermittent A.C. arc source (Model FS 110) run at 6 A current. The spectra are

photographed on Ilford N. 30 emulsion employing a JACO 3.4 metre grating spectrograph, in the first order of a 1200 grooves/mm grating blazed at 3300 Å.

Two lines of matrix element Ta at 2808.86 Å and 2943.77 Å, are selected as internal standards by the help of moving plate studies. Nb can be estimated in the range 40–4000 ppm using the line 2950.87 Å. The analysis lines used and minimum amount estimated for other elements are: Al 3092.71 Å 25 ppm; Fe 2966.9 Å 10 ppm; Mn 2801.06 Å 2.5 ppm; Mo 3112.12 Å 30 ppm; Pb 2833.06 Å 5 ppm; Ni 3392.99 Å 5 ppm; Si 2516.19 Å 12.5 ppm; Sn 3034.12 Å 25 ppm; Ti 3372.8 Å 5 ppm; V 3118.38 Å 15 ppm; Zr 3391.97 Å 40 ppm. The sensitivity reported here is lowest for Mo, Ni, Ti and V and is comparable for other impurities with earlier methods.

The coefficient of variation ranges from $\pm 8\%$ for Nb to $\pm 18\%$ for Mo. Experiments showed that the recoveries were better than 85% of the elements added.

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Spectrophotometric Determination of Trace Amounts of Cobalt Using 7-Nitroso-8-hydroxyquinoline-5-sulphonic Acid as Reagent

Spektrophotometrische Bestimmung von Kobaltspuren mit 7-Nitroso-8-hydroxychinolin-5-sulfonsäure

Best. von Kobalt mit 7-Nitroso-8-hydroxychinolin-5-sulfonsäure; Spektralphotometrie

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It has been found that 7-Nitroso-8-hydroxyquinoline-5-sulphonic acid can be well employed for the spectrophotometric determination of traces of cobalt. Physicochemical investigation of the red complex formed showed its composition to be $H[Co(II)L_3]$ (L=ligand) at pH 5.0. The molar absorptivity at 525 nm is $1.15 \times 10^4 \text{ l} \cdot \text{mole}^{-1} \cdot \text{cm}^{-1}$, Beer's law is obeyed within the range of $0.9 - 12.0 \times 10^{-5} \text{ mole of Co/l}$. Absorption of the yellow NaHL or greenish-brown Na_2L did not effect the measurement at 525 nm.

As the monosodium salt of the reagent is stable for only 3 days, it is advisable to use the disodium salt (at least 15 days). The presence of Ca, Sr, Ba, Cd, Zn, Mg, Th, UO_2 , Zr, W in concentrations up to 150 times that of cobalt does not affect the results. Interference was observed for Fe, Mn, Cr, even in about equal concentrations. Chloride, acetate, sulphate and nitrate up to 0.1 M do not interfere. The sensitivity of the method is comparable to that of Nitroso-R-salt. As little as 0.6 ppm of Co can be determined.

Experimental

Reagent. The monosodium salt of the reagent is prepared by nitrosation of 8-hydroxyquinoline-5-sulphonic acid in aqueous solution pH 5 at 0°C. The disodium salt is prepared by addition of the calculated amount of NaOH to the solution of NaHL.

Procedure. Mix 4.0 ml of 0.2 M acetate buffer pH 5 with 2.0 ml of the above reagent solution (0.2 g/100 ml) add an aliquot of the sample solution, make up to 8.0 ml with water and shake. Measure the optical density at 525 nm against a blank solution. Take the results from a calibration curve prepared similarly.

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