

DETERMINATION OF COBALT

Principle of method

Cobalt(III) forms a red complex in acidic solutions of 2-nitroso-1-naphtol, which can be extracted with organic solvents such as benzene, toluene, xylene, carbon tetrachloride, chloroform. Spectrophotometric determination is performed at 367 or 530 nm.

Reagents

Sodium sulfate, anhydrous

Toluene, redistilled

Hydrochloric acid, approx. 2N.: Mix 1 volume of concentrated hydrochloric acid (about 36% w/w; 11N) with 5 volumes of water.

Hydrogen peroxide solution, 3%

Sodium citrate solution.: 40% w/v

Sodium hydroxide .: approx. 2N

2-Nitroso-1-naphtol solution (reagent).: 1 g of 2-nitroso-1-naphtol is dissolved in 100 mL of glacial acetic acid, and 1 g of activated C is added. The solution is shaken before use, and the required amount is filtered.

Standard cobalt solution.: 0.494 g of cobalt(II) nitrate, $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, is dissolved in water and the solution is diluted to 100 mL at 20°C with water; 1 mL \equiv 1 mg Co.

Dilute this solution as required, so that 1 mL \equiv 1 μg Co.

Instrumentation

UV-VIS Spectrophotometer

Hot plate

Determination of cobalt

Slightly acidic solution of cobalt (containing not more than 15 μg Co) is transferred to a 100 mL-beaker. 15 mL of sodium citrate solution is added and the solution is diluted to 50 mL with distilled water. pH of the solution is adjusted to between 3 and 4 by the addition of 2N HCl and 2N NaOH. Precipitate from $\text{Fe}(\text{OH})_3$ is destroyed by heating, and cooling. 10 mL of hydrogen peroxide solution is added. After 5 minutes 1 mL of reagent solution is added. The solution is heated to about 90°C and then let to stand for at least 30 minutes at room temperature.

The solution is transferred to a 125-mL separating funnel, 10 mL toluene is added, and shaken for 2 minutes. The aqueous phase is discarded.

The toluene phase is washed successively by 20 mL of 2N HCl and twice by 20 mL of 2N NaOH. The lower aqueous phases are discarded. The toluene phase is dried over anhydrous sodium sulfate, filtered through a cotton-wool plug into a 25-L volumetric flask. The toluene extract is diluted to the mark with toluene.

Determinations are repeated with a blank sample and check standards containing 5 and 10 μg of Co.

Check standards are used with every batch of the samples to check that the calibration graph still applies.

Absorbances of test and blank solutions as well as the check standards are measured at 367 nm, using a 10 mm cell, against toluene.

Calibration Graph

Measure appropriate amounts of cobalt standard solution, covering the range 0 to 15 μg of Co, into a series of 100-mL beakers and proceed as for the test solution. Measure the absorbances of solutions, and construct a graph relating the absorbances to the microgrammes of Co.

Evaluation of the data

From a previously prepared calibration graph, read the number of milli(micro)grammes of cobalt equivalent to the observed absorbances of the blank and test solutions and calculate the amount of cobalt in the sample.

REFERENCE

Classen, A., and Daamen, A.; Cobalt determination in metals and alloys, *Anal. Chim. Acta* 12 (1955) 547