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SHORT COMMUNICATIONS

SPECTROPHOTOMETRIC DETERMINATION OF NICKEL IN COPPER-BASE ALLOY WITH 2-(2-THIAZOLYLAZO)-*p*-CRESOL

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Summary—A spectrophotometric method for determination of nickel in copper-base alloy with 2-(2-thiazolylazo)-*p*-cresol (TAC) is described. The interferences of foreign ions can be eliminated by masking with a mixture of sodium tartrate and sodium thiosulphate. The nickel–TAC complex has low solubility in water, but is soluble in aqueous ethanol. Beer's law is obeyed for 20–70 μg of nickel in 50 ml of solution, at pH 5.7. The molar absorptivity at 580 nm is 2.6×10^4 l.mole^{-1}.cm^{-1}. The method has been applied successfully to determination of nickel in reference samples.

2-(2-Thiazolylazo)-*p*-cresol (TAC) forms coloured complexes with several metal ions but its use as a colorimetric reagent is limited. It was initially used for determination of copper by Gusev *et al.*[1] and Sommer *et al.*[2] Gusev *et al.* have also proposed use of TAC for determination of nickel in a uranium-base alloy, with chloroform extraction of the complex and absorbance measurement at 610 nm. The present work describes the use of TAC for colorimetric determination of nickel in copper-base alloys, but with use of an aqueous ethanol system.

EXPERIMENTAL

Reagents

TAC solution, 0.1 g in 100 ml of ethanol. Standard nickel solutions: 25 mg/ml and 25 μg/ml. Buffer solution, pH 5.9, prepared by mixing 1.0*M* sodium acetate and 1.0*M* acetic acid in appropriate ratios. Masking solution, consisting of 16.7 g of sodium thiosulphate pentahydrate and 0.15 g of sodium tartrate dissolved in 250 ml of distilled water.

General procedure

Into a 50-ml standard flask transfer a portion of sample solution containing up to 70 μg of nickel. Add 10.0 ml of acetate buffer, 5.0 ml of ethanol, 5.0 ml of masking solution and 2.0 ml of TAC solution, dilute to the mark with water, mix, and after 10 min measure the absorbance at 580 nm in a 1-cm cell against a solution prepared in the same way but without the addition of TAC.

Prepare a calibration graph with appropriate standards.

RESULTS AND DISCUSSION

The absorption maximum of the complex is at 580–600 nm and that of the reagent blank is at 350–380 nm; the spectra do not overlap. Maximal and constant absorbance is obtained for 50 μg of nickel with 1.2 ml of 0.1% TAC solution per 50 ml, so 2.0 ml of TAC solution is selected as optimal. The absorbance is also maximal and constant with 2.0–10.0 ml of ethanol per 50 ml, so use of 5.0 ml is recommended. The maximum allowable amount of masking reagent per 50 ml of solution was found to be 5.0 ml.

Characteristics of the nickel–TAC complex

The absorbance of the complex is pH-dependent, but constant in the range 5.7–6.0. Beer's law is obeyed at 580 nm for 20–70 μg of nickel in 50 ml of solution, at pH 5.7, and the apparent molar absorptivity is 2.6×10^4 l.mole^{-1}.cm^{-1}.

The order of addition of the reactants does not influence formation of the complex. Complete colour development takes 10 min and the colour is then stable for at least 24 hr.

Interferences

The selectivity was investigated by determination of 25 μg of nickel in presence of various amounts of other ions. Iron(II), iron(III) and cobalt(II) interfere.

The interference of copper(II), bismuth, tin(IV), manganese(II), cadmium, molybdenum(VI), lead and aluminium may be reduced somewhat by the masking solution; the tolerance limits are given in Table 1.

Table 1. Effect of foreign ions on determination of 25.1 μg of Ni in presence of mixed masking reagents

Ion	Added, μg	Ion/Ni ratio, *w*/*w*	Ni found, μg	Error, μg
Al^{3+}	376	15	24.5	−0.6
Mg^{2+}	1063	43	25.3	+0.2
Ca^{2+}	1873	75	25.6	+0.5
Cd^{2+}	81	3	24.9	−0.2
Pd^{2+}	307	12	25.7	+0.6
Cr^{3+}	731	29	26.2	+1.1
Bi^{3+}	387	15	25.2	+0.1
Mo(VI)	732	29	25.3	+0.2
Zn^{2+}	101	4	25.4	+0.3
Cu^{2+}	4516	181	25.4	+0.3

Table 2. Analysis of various samples

Sample	Nickel present, %	Nickel found, %
Cast Bronze NBS.52.C	0.76	0.77, 0.78, 0.78
Nickel-Copper Alloy NBS.162a	64.0	63.2, 63.3, 63.5
Copper-Nickel-Zinc-Alloy NBS.157	11.82	11.8, 11.9, 11.8
Bronze 239 CEPED standard	0.30	0.30, 0.30, 0.30

Determination of nickel in bronzes and brasses

Pipette into a 50-ml standard flask an aliquot of the sample solution, containing 20–70 μg of nickel. Add 10.0 ml of pH 5.70 acetate buffer, 5.0 ml of ethanol, 5.0 ml of masking solution and 2.0 ml of TAC (0.1%) solution, dilute to the mark with water, mix, and after 10 min measure the absorbance at 580 nm in a 1-cm cell against a solution prepared in the same way but without the addition of TAC.

Results obtained by applying the proposed method to several standard samples agree well with the certified values (Table 2).

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REFERENCES

1. S. I. Gusev, I. N. Glushkova and L. A. Ketova, *Uch. Zap. Perm. Gos. Univ.*, 1973, **289**, 255.
2. L. Sommer, M. Langová and V. Kubáň, *Collection Czech. Chem. Commun.*, 1976, **41**, 1317.
3. S. I. Gusev, M. V. Zhvakina, I. A. Kozhevnikova and L. S. Mal'tseva, *Zavodsk. Lab.*, 1976, **42**, 19.
4. E. B. Sandell, *Colorimetric Determination of Traces of Metals*, 3rd Ed., p. 83. Interscience, New York, 1959.