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NOTES

Spectrophotometic Determination of Nickel with Di-*o*-Tolylcarbazone Using the Synergistic Effect of Phenanthroline

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In the present note, the spectrophotometric determination of nickel(II) with di-*o*-tolylcarbazone and phenanthroline has been described.

Di-*o*-tolylcarbazone was prepared by the method described by Hubbard and Scott[1], involving presulphate oxidation of di-*o*-tolylcarbazide and purfied by the method suggested by Ghosh and Ray[2]. Di-*o*-tolylcarbazide was synthesized by heating *o*-tolylhydrazine and guaiacol carbonate for 3 h at 160°C as suggested by Noller[3]. The melting point of the di-*o*-tolylcarbazone was 137°C. Diphenylcarbazone has been used as an indicator for the complexometric determination of mercury[4] and molybdenum[5]. But no work has been reported in literature where di-*o*-tolylcarbazone has been used as an analytical reagent. The IR spectral studies and determination of pK value[6] of this reagent has been reported. The present communication deals with spectrophotometric determination of nickel with this reagent.

During the course of investigation of adduct and mixed ligand chelate extraction system[7], we have found that the addition of heterocyclic nitrogen bases such as pyridine and phenanthroline to a chloroform solution of di-*o*-tolylcarbazone gave rise to a reagent which was free from the disadvantages of di-*o*-tolylcarbazone used alone. Nickel was rapidly and quantitatively extracted over a broad pH range (from 6.0 to at least 11.0) to give a very highly coloured mixed ligand complex having an absorption band centred at 520 nm. The mixed ligand complex is sufficiently stable to permit the removal of excess of di-*o*-tolylcarbazone by back extraction with 0.1M sodium hydroxide, so monocolour method is applicable. The molar absorptivity of this complex is 25.1×10^3, which makes a method based on this reaction about four times as senstive as the currently most sensitive method, that using dimethyl glyoxime and an oxidising agent[8].

To 10 ml of a solution containing 1 to 10 g of nickel (upto 25μg may be accommodated), add 5 ml of a phthalate or acetate buffer of pH 6.0 (or dilute ammonia may be used) followed by 15 ml of a chloroform solution 7×10^{-5}M in di-*o*-tolylcarbazone and 3×10^{-5}M in phenanthroline. Shake the phase for 5 min, allow them to settle, and separate them. Back-extract the chloroform layer with 10 ml of 0.1M sodium hydroxide by vigorous shaking for about 1 min. Read the absorbance of the chloroform extract in a 10 mm cell at 520 nm against a similarly treated blank. Construct a calibration curve (a straight line passing through the origin and having a slope of 25.1×10^3 l mole^{-1} cm^{-1}). Typical results are presented in Table 1.

TABLE 1

Ni taken μg	Absorbance at 520 nm	Ni found μg
1.77	0.171	1.79
5.29	0.518	5.44
7.41	0.711	7.23
8.82	0.872	8.97

The extraction of the mixed ligand complex does not in itself provide significant increase in selectivity over the extraction of di-*o*-tolylcarbazone.

An equilibrium study of the reaction of the adduct and mixed ligand complexes formed from various heterocyclic nitrogen bases and di-*o*-tolylcarbazone were published[7] elsewhere.

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