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NOTESolvent Extraction and Spectrophotometric Determination of Nickel(II) with Thiothenoyltriflouroacetone

R. R. Mulye[a]; S. M. Khopkar[a]
[a] DEPARTMENT OF CHEMISTRY, INDIAN INSTITUTE OF TECHNOLOGY, BOMBAY, INDIA

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NOTE

Solvent Extraction and Spectrophotometric Determination of Nickel(II) with Thiothenoyltriflouroacetone

R. R. MULYE and S. M. KHOPKAR

DEPARTMENT OF CHEMISTRY
INDIAN INSTITUTE OF TECHNOLOGY
BOMBAY-400.076, INDIA

Abstract

Nickel can be quantitatively extracted with 0.001 *M* thiothenoyltrifluoroacetone in carbon tetrachloride at pH 6.5. The yellow-orange colored complex is measured spectrophotometrically at 480 nm. The system conforms to Beer's law in the concentration range of 0.5–5 μg/ml of nickel. The complex is stable for 96 hr. Nickel can be separated from several cations and anions.

Thiothenoyltrifluoroacetone (1,1,1-trifluoro-4-(2-thienyl)-4-mercaptobut-3-en-2 one) (i.e., HSTTA) can be used for the solvent extraction of nickel at pH 6.0–6.5. It forms a yellow-orange colored complex which can be measured spectrophotometrically at 480 nm.

Acetylacetone (*1*) was a poor extractant for nickel, while benzoylacetone (*1*) could extract nickel only on vigorous shaking. Dibenzoylmethane (*2*) was effective in the alkaline region. Furoyltrifluoroacetone (*3*) and 2-thenoyltrifluoroacetone (*4*) were used for extraction and colorimetric determination of nickel, but some ions showed strong interferences. The other methods for solvent extraction are reviewed in a recent monograph

(*5*). The method proposed by us here is simple, rapid, sensitive, and applicable at microgram concentration.

EXPERIMENTAL

Apparatus and Reagents

Type SF-4 quartz spectrophotometer. Cambridge pH meter; Wrist action flask shaker.

Thiothenoyltrifluoroacetone was synthesized from 2-thenoyltrifluoroacetone (Fluka A.G.) by the procedure of Berg et al. (*6*). About 0.001 *M* STTA in carbon tetrachloride was used. The reagent was preserved in the refrigerator.

About 5.595 g of nickel sulfate hexahydrate (B.D.H. Anal R) was dissolved in 250 ml of distilled water containing 2.5% sulfuric acid and standardized gravimetrically (*7*). It contained 4.99 mg/ml of nickel. The dilute solution was prepared from stock solution by suitable dilution. Buffer solution of pH 6.5 was prepared by dissolving 72 g of ammonium acetate in a liter of distilled water (*7*).

General Procedure

An aliquot of the solution containing 20 μg of nickel was taken. To this 10 ml of buffer solution of pH 6.5 was added. The volume of the aqueous phase was made up to 25 ml. The solution was then transferred to a separatory funnel and 10 ml of 0.001 *M* STTA in carbon tetrachloride was added. The mixture was then shaken on a wrist action flask shaker for about 10 min. The layers were allowed to settle and separate. The aqueous layer was carefully withdrawn and the yellow-orange colored nickel complex was transferred into a volumetric flask and its absorbance was measured at 480 nm against the reagent blank. The amount of nickel was computed from the calibration curve.

RESULTS AND DISCUSSION

Absorption Spectrum

The absorption spectrum of Ni(II)–STTA complex (Ni = 3.4×10^{-5} *M*) versus the reagent blank is shown in Fig. 1. The spectrum of reagent blank against carbon tetrachloride is also given. It is seen that

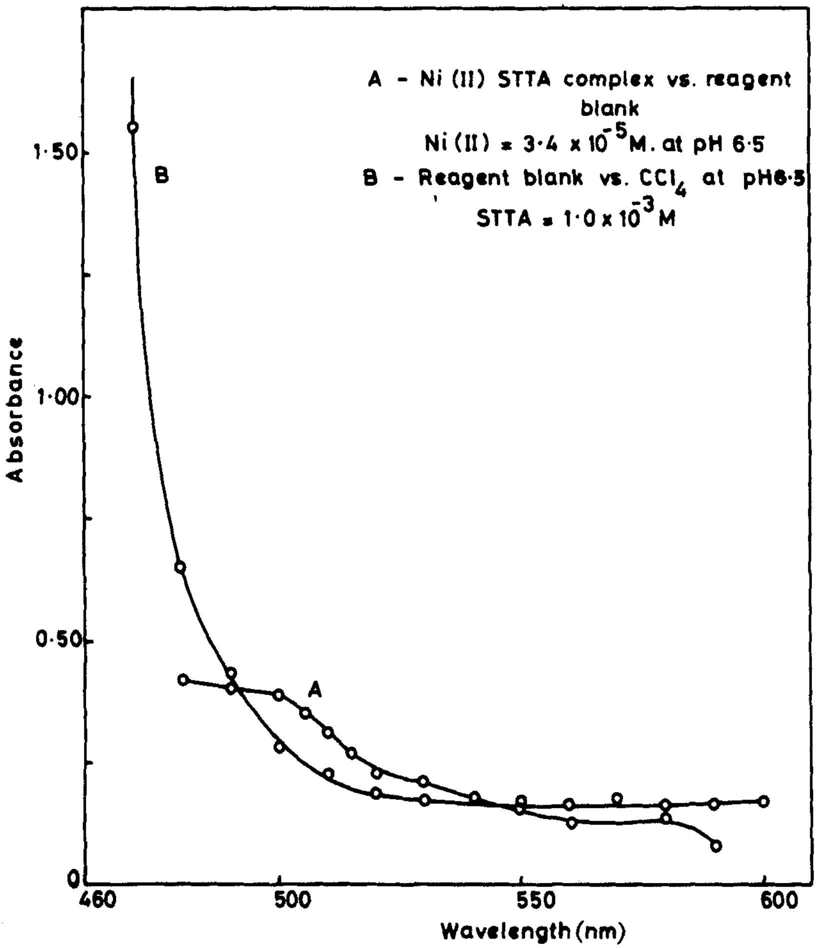

Fig. 1. Absorption spectra of nickel–STTA complex.

the difference in absorbance of the complex and the reagent blank is a maximum at 480 nm; hence, all absorbance measurements should be taken at 480 nm. The molar absorptivity at 480 nm is 1.174 × 10^4 (when nickel is 3.4 × 10^{-5} *M*).

Extraction as a Function of pH

The extraction of nickel(II) was carried out from pH 1.0 to pH 8.5 with 0.001 *M* STTA–CCl_4 (Fig. 2). The results showed that the extrac-

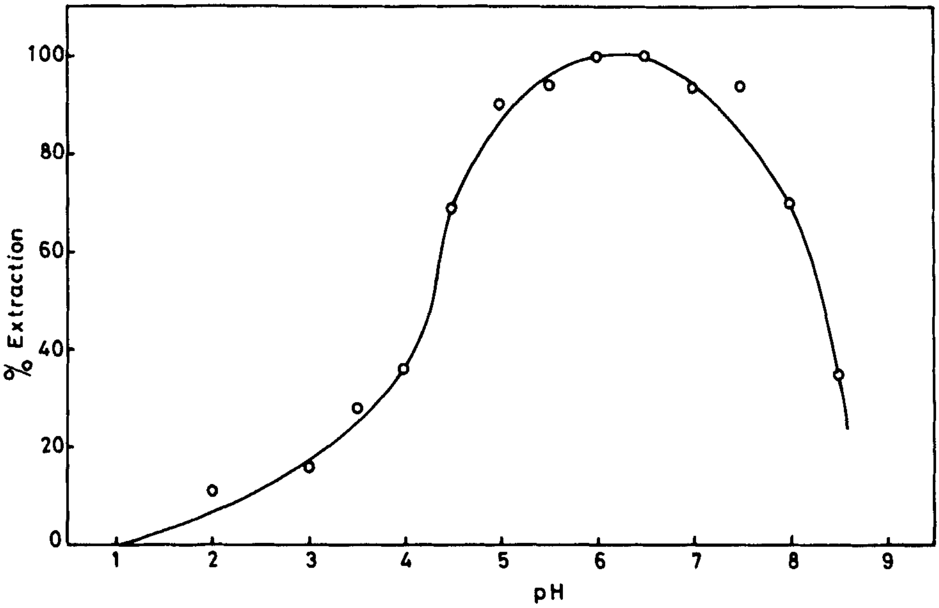

FIG. 2. Extraction of Ni(II)-STTA complex with CCl_4 as a function of pH.

tion commences at pH 2.0 and is quantitative in the pH region of 6.0–6.5. Below and above pH 6.0–6.5, extraction was incomplete.

Beer's Law

Different amounts of nickel(II), ranging from 5–40 μg of nickel per 10 ml of solution were extracted at pH 6.5 with 10 ml of 0.001 *M* STTA-CCl_4. The absorbance of the complex was measured at 480, 490, and 500 nm. It was observed that the system conforms to Beer's law in the concentration range of 0.5–5 μg/ml of nickel at 480 nm.

Effect of Reagent Concentration

The nickel was extracted at pH 6.5 with varying volumes and concentrations of the reagent (Table 1). The results showed that 10 ml of 0.001 *M* STTA was enough for quantitative extraction of nickel. The extraction was incomplete at lower volumes and concentrations of the reagent. At higher concentrations of the reagent there was an insignificant increase in absorbance.

TABLE 1

Effect of Reagent Concentration[a]

Reagent concn $1 \times 10^{-3}\,M$	Volume of reagent (ml)	Absorbance at 480 nm
0.25	10.0	0.450
0.50	10.0	0.440
0.75	10.0	0.430
1.00	10.0	0.410
1.00	5.0[b]	0.390
1.00	7.5[b]	0.400
1.00	10.0	0.410
1.00	12.5	0.375
1.5	10.0	0.420
2.0	10.0	0.430

[a] Ni, 20 μg; pH = 6.5.

[b] Final volume made up to 10 ml.

Stability of the Color of the Complex

The absorbance of the colored complex was measured at elapsed intervals of 0.5, 8, 16, 24, 48, 96, and 128 hr. It was observed that the complex was stable for at least 96 hr.

Period of Equilibration

Varying the shaking period from 3–30 min revealed that the extraction was quantitative in 10 min of equilibration.

Effect of Diversions

The effect of several ions on the extraction behavior of nickel was studied (Table 2). The tolerance limit was set as the amount of ion that could cause a ±2% error in the total recovery of nickel. The results showed that alkali and alkaline earths, thallium(III), beryllium, and common anions were tolerated in a ratio of 1:200. Manganese(II) and thorium showed some interference, but their interference can be eliminated by using suitable masking agents, such as oxalic acid or alkali fluoride, respectively.

TABLE 2

Effect of Other Ions[a]

Tolerance limit (μg)	Other ions present
1.0×10^4	NO_3^-, SO_4^{2-}, Cl^-, K^+, Na^+, NH_4^+
2.5×10^3	Be^{2+}, Ca^{2+}, Sr^{2+}, Ba^{2+}, Mg^{2+}, Rb^+, Li^+
2.0×10^3	Tl^{+3}, Cs^+, SeO_3^{2-}, Br^-, I^-, CN^-, SCN^-, PO_4^{3-}, NO_2^-, CH_3COO^-, $C_2O_4^{2-}$, ascarbate$^-$, tartarate^{3-}
1.5×10^3	F^-, citrate,$^{3-}$
7.5×10^2	Mn^{2+}, Th^{4+}, $S_2O_3^{2-}$
1.0×10^2	WO_4^{2-}, VO_3^-

[a] Ni, 10 μg; pH = 6.5, 0.001 *M* STTA-CCl_4.

The absorbance of the solution from 20 determination was 0.400 ± 0.010. The relative standard deviation was ±1.0%. Sandell's sensitivity was 0.005 μg/cm²/ml. The method is simple, rapid, sensitive, and applicable at tracer concentrations. The overall time of operation is just 30 min.

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