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Synthesis and Characterization of Sulphur Containing Extractant

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ABSTRACT

New sulphur containing extrcatant was synthesized and characterized. The synthesized extractant belongs to the class Schiff's base, 4-(4-dimethylaminobenzylideneimino)-5-methyl-4H-1, 2, 4-triazole-3-thiol. The class of Schiff bases has been reported as chelating agent for many transition metal ions. The efficiency of synthesized extractant was checked by extracting traces of gold (III) from synthetic mixtures and some gold alloys, by using solvent extraction technique. Extractant shows selectivity towards gold (III) and highly efficient.

Keywords : Extractant, Gold (III), Solvent Extraction, Hydrochloric Acid Media.

I. INTRODUCTION

Schiff base was first reported by Hugo Schiff in 1864 [1]. Schiff bases can be prepared by condensing carbonyl compounds and amines in different conditions and in different solvents with the elimination of water molecules. The class of Schiff bases has been reported as chelating agent for many transition metal ions [2-4].

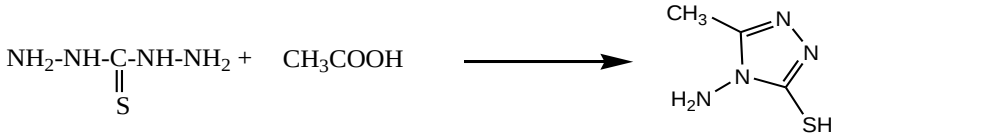
The most important factor in the solvent extraction of metals is high selectivity of the extractant for the recovery of a specified metal. In order to develop or design highly selective reagents for precious metals, it is necessary to take into account the highly favored complexation due to the chelate effect and the HSAB principle. The term "HSAB principle" which was suggested by R.G. Pearson [5] refers to the rule that "hard acids" prefers to associate with "hard bases", and "soft acids" prefers to associate with "soft bases". Since the sulphur atom has a high affinity for so-called "soft metals" such as gold, palladium, and ruthenium, a sulphur containing reagent is expected to have a high selectivity for these metals. Solvent extraction is one of the most efficient separation technique used for extraction of metals. As compare to other separation techniques, solvent extraction is easy and cost effective technique, it require only a simple separatory funnel [6,7]. Gold belongs to precious metal family or platinum group metals which includes ruthenium, rhodium, palladium, platinum, gold, osmium and iridium. These metals are better known for their uses in art, jewelry, coinage and pharmaceuticals (gold and platinum). These metals are also known for their role as investments and a store of value.

II. METHODS AND MATERIAL

Synthesis of 4-(4-dimethylaminobenzylideneimino)-5-methyl-4H-1, 2, 4-triazole-3-thiol (DMABIMTT)

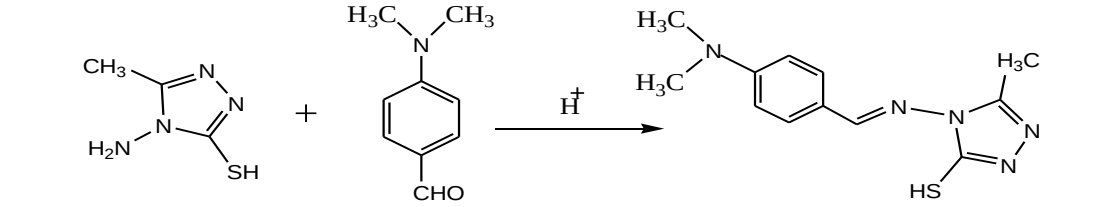
Step I: Formation of 3-methyl-4-amino-5-mercapto-1, 2, 4-triazole [8]

A mixture of thiocarbohydrazide and glacial acetic acid was heated and refluxed for 4h., within an hour of refluxing; a solid started separating from the clear solution. It was then crystallized from water with shining faint yellow crystals (M.P. 203 ^{0}C).



Thiocarbohydrazide Acetic acid 3-methyl- 4-amino-5-mercapto-1, 2, 4-triazole

Step II: The equimolar concentration of 3-ethyl– 4-amino-5-mercapto-1, 2, 4-triazole[2] and 0.02 mole of dimethylaminobenzaldehyde in 50 ml alcohol containing 3 drops of glacial acetic acid was refluxed for 4-5 h. The product obtained was separated and recrystallized from hot ethanol as pale yellow needles (M.P. **178 °C**).



3-methyl- 4-amino-5-mercapto -1, 2, 4-triazole

4-dimethylaminbenzaldehyde

4-(4-dimethylaminobenzylideneamino)-5-methyl-4H-1,2,4-triazole-3-thiol (DMABIMTT)

RESULTS AND DISCUSSION:

DMABIMTT was characterized by NMR, MASS and IR spectra.

PMR spectrum of DMABIMTT in $CDCl_3$ showed delta values (^{1}H NMR ($CDCl_3$) δ) 3.07 (6H,s,-$(CH_3)_2$, 2.43 (3H,s,-CH_3), 6.70 (2H,d,*J* 8 Hz, aromatic protons), 7.76 (2H,d,*J* 8 Hz, aromatic protons), 9.7 (1H,s,-CH), 11.6 (1H, s, -SH).

In the IR spectrum of DMABIMTT the bands has been assigned as (cm^{-1}): 2870.17 (CH), 1595.18 (γC=N) and 756.12 for (γC=S). Mass spectrum of DMABIMTT shows peak at m/e: **260.9** (100.0%) (M^+)

Binary separation of gold(III), from iron(III), cobalt(II), nickel(II) and copper(II):

The method allowed separation and determination of gold (III) from a binary mixture containing either iron (III), cobalt (II), nickel (II) and copper (II). In a typical experiment, solution containing 100µg of gold (III) was taken and known amounts of other metal ions were added. The separation of gold (III) from iron(III), cobalt(II), nickel(II) and copper(II) was accomplished with 0.1M DMABIMTT in chloroform from hydrochloric acid medium. The recovery of gold (III) and those added ions was 99% and results are reported in (Table:1).

Table: 1 Separation of gold(III), from iron(III), cobalt(II), nickel(II) and copper(II)

Composition of metal ions, (µg)	Average % recovery Gold (III)	R.S.D. (%)
Au(III)100, Fe(III)15000	99.1	0.15
Au(III)100, Co(II)10000	98.6	0.19
Au(III)100, Cu(II)5000	98.5	0.17
Au(III)100, Ni(II)5000	97.6	0.25

CONCLUSION

It is concluded that the sulphur containing extractant was synthesized successfully. The results of characterization are in support of synthesized extractant. DMABIMTT is efficiently used for extraction of traces of gold (III) from synthetic mixtures.

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