

Spectrophotometric Determination of Micro Amounts of Gold(III) Using Cyclopentane-spiro-2'-(1-Methyl-2', 4'-Dithio)-s-Triazine

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Gold(III) forms an intense yellow soluble complex with cyclopentane-spiro-2', (1-methyl-2', 4'-dithio)-s triazine (CPSMDTT) in acidic medium. The complex gives maximum absorption at 435 nm in the pH range 3.8 to 4.0. Beer's law is obeyed over the range 1 to 7 ppm of Au(III). The molar absorptivity and Sandell's sensitivity of the complex are $1.28 \times 10^4 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$ and $0.015 \mu\text{g cm}^{-2}$, respectively. Standard deviation is evaluated to be 0.69. The composition of the complex is established by Job's method and mole ratio method as 1 : 3, (Au(III) : CPSMDTT). The effect of various diverse ions is also studied.

INTRODUCTION

Literature reports the use of thio-triazines as chromogenic reagent for the determination of some of the metals^{1,2}. The present work describes a simple and sensitive method for spectrophotometric determination of Au(III) using CPSMDTT as the chromogenic reagent.

EXPERIMENTAL

Cyclopentane-spiro-2'-(1-methyl-2', 4'-dithio)-s-triazine (CPSMDTT) was prepared according to the described method³ and was recrystallised from ethanol to get analytically pure compound (m.pt. 229°C, M.wt. 217). 0.008M solution of the reagent in dimethylformamide (DMF) was used for the studies. The reagent in DMF was found to be stable at least for two months. Standard stock solution of Au(III) was prepared by dissolving AuCl₃ (Johnson-Matthy) in hydrochloric acid (1M) and was standardised⁴. All other chemicals used were of A.R. grade. Spectronic 20, Baush and Lomb Spectrophotometer was used for all spectral measurements, pH measurements were made using Control Dynamics digital pH meter of C.D. Instrumentation Pvt. Ltd.

An aliquot of Au(III) solution containing upto 25 μg to 175 μg of Au(III) was diluted to 20 ml with distilled water, followed by addition of 1 ml acetate buffer⁵ pH 3.9. To the mixture was added 1 ml 0.008M CPSMDTT (in DMF), the mixture was kept at room temperature for 15

minutes and diluted to 25 ml with distilled water. Absorbance of intense yellow coloured complex was measured at 435 nm against distilled water as reference.

RESULTS AND DISCUSSION

The absorption spectrum of the complex formed was recorded over the wavelength range 340 to 600 nm against reagent blank. The complex showed maximum absorbance at 435 nm, reagent blank did not absorb at this wavelength, hence all measurements were made at 435 nm using distilled water as reference.

The study was made using various reagent amounts on 100 μg Au(III). It was found that 0.005M to 0.012M (1 ml) reagent gave constant and maximum absorbance. For subsequent study 1 ml 0.008M CPSMDTT was used.

The system was studied for various pH values using NaOH-HCl of appropriate strengths. It was observed that constant and maximum absorbance was obtained over a very narrow range of pH 3.8-4.0. The system was therefore studied with different buffer solutions. 1 ml of acetate buffer (pH 3.9) was found to be optimum volume and was used for further studies. The complex was found to be stable for 1 hr.

The absorbance of the complex formed with various amounts of Au(III) were measured by suggested procedure. The plot of absorbance against concentration of Au(III) was found to be linear over the range 25 μg to 175 μg of Au(III). The molar absorptivity and Sandell's sensitivity were calculated to be $1.28 \times 10^4 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$ and $0.015 \mu\text{g cm}^{-2}$ respectively. The average of 10 determinations of 100 μg of Au(III) in 25 ml was 99.6 μg with standard deviation of 0.004.

The composition of the complex was established as 1 : 3 (Metal : Ligand) by Job's method. The composition was further confirmed by mole-ratio method.

The effect of diverse ions on Au(III) determination was studied at 2% tolerance limit. In the estimation of 100 μg Au(III) the following ions did not interfere when present in amounts (μg shown in parentheses) Cd^{+2} (10000) Pb^{+2} (10000), Al^{+3} (1500), Ba^{+2} (1600), Mn^{+2} (1700), Fe^{+3} (100), NH_4^+ (2000), As^{+3} (120), Ni^{+2} (150), Zn^{+2} (1500), Mg^{+2} (300), Os^{+8} (120), whereas Pt^{+4} , Fe^{+2} , Rh^{+3} , Pd^{+2} , Cu^{+2} interfered seriously. Among the anions tested, F^- (2000), Br^- (500), I^- (75), NO_2^- (1100), SO_4^{2-} (2000), $\text{S}_2\text{O}_8^{2-}$ (150), EDTA (700) citrate (1500), were tolerated. The presence of more than 100 fold excess of Cl^- , NO_3^- , B_4O_7^- , tartrate, CO_3^{2-} phosphate had no diverse effect, whereas SO_2^{-3} , SCN^- , $\text{S}_2\text{O}_3^{2-}$, thiourea interfered seriously.

The method was thus found to be simple, precise and sensitive one, allowing determination of micro quantities of Au(III).

REFERENCES

1. Nishida and Hiroshi, *Bunseki Kagaku*, **28**, 563 (1979).
2. Maghssoudi, H. Rostam and Ahmad B. Fawzi, *Anal. Chem.*, **47**, 1694 (1975).
3. N. S. Chande and N. P. Shetgiri, Ph.D. Thesis, The Institute of Science, Bombay University, (1989), p. 93-94.
4. F. E. Beamish, J. J. Russell and J. Seath, *Ind. Eng. Chem. Anal. Ed.*, **9**, 174-176 (1937).
5. J. Lurie, Handbook of Analytical Chemistry, Mir Publishers, Moscow, p. 252 (1972).

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