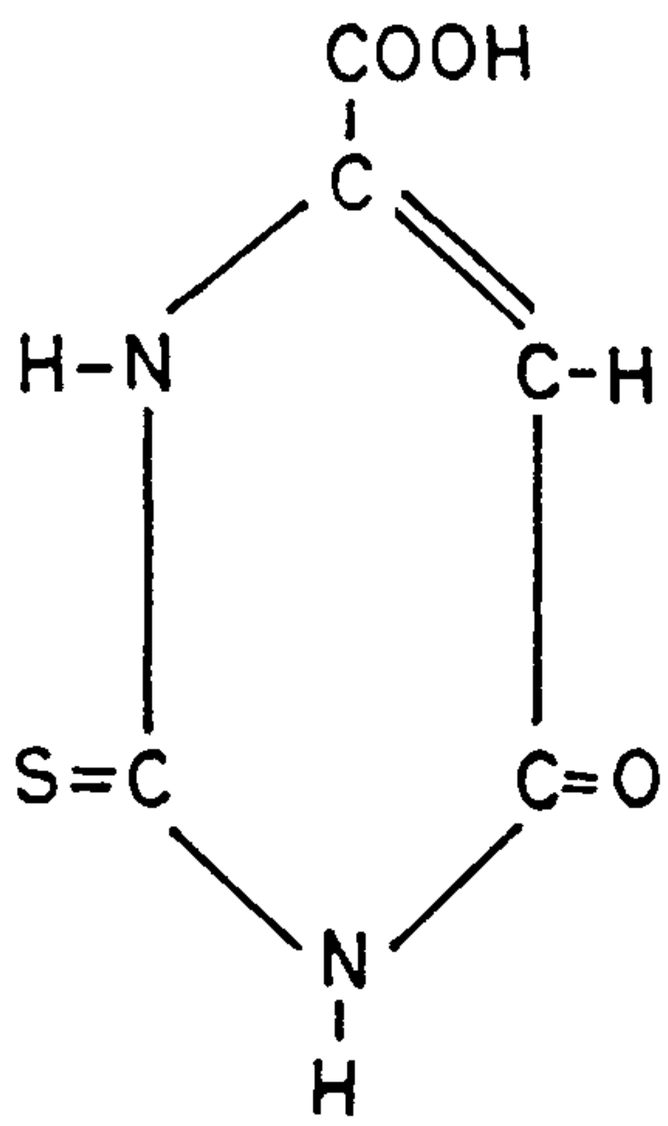
# SHORT COMMUNICATIONS

## GRAVIMETRIC ESTIMATION OF Ag(I) IN THE PRESENCE OF CHLORIDE IMPURITIES USING 2-THIOOROTIC ACID (AMMONIUM SALT)

MADHULIKA SHRIVASTAVA and
G. S. PANDEY

*Department of Chemistry, Government College of Engineering & Technology, Raipur 492 002, India.*

2-THIOOROTIC acid (structure given below) is synthesised by the method of Johnson and Schroeder[1]. The reagent has exhibited its role in many biological activities, and found many applications in the control of diseases[2-4]. It has also found applications in the stabilization of photographic films, and interimage effects in multicolour photographic reproduction[5-7]




The complex forming behaviour of this compound with a number of metal ions has been studied earlier[8-10]. Ag(I) on reaction with 2-thioorotic acid (ammonium salt) forms a stable, yellow coloured sparingly soluble complex $Ag(C_5H_3N_2O_3S)$, the solubility product of which was found to be $2.18 \times 10^{-13}$, compared to $2 \times 10^{-10}$ for AgCl.

*Reagents:*

2-Thioorotic acid (ammonium salt) solution: 5 g of 2-thioorotic acid was dissolved in the minimum volume of liquor ammonia, and was boiled to remove the dissolved ammonia, and filtered. The solution was diluted to 500 ml.

*Procedure:*

The sample solutions of silver nitrate were heated to 60°C, and 2-thioorotic acid solution was then added slowly from a burette with constant stirring till all of the silver precipitated as a yellow coloured silver complex. A few more drops of 2-thioorotic acid solution were added to confirm the quantitative yield. The reaction mixture was digested on a water bath for 30 min, cooled, and then filtered through a sintered crucible (G-4), and washed repeatedly with hot water till the filtrate was free from the 2-thioorotate ions. (The washing tested with $AgNO_3$ solution). The precipitate was dried at 110°C for an hour and weighed. The results obtained for a set of six solutions are shown in table 1.

To study the effect of the presence of chlorides in the estimation, another set of six solutions of Ag-ions was taken, and 1 g of KCl (AnalaR grade) was added in each case. The 2-thioorotic acid solution was then added drop-wise to each beaker containing the precipitate. The mixture was then digested on a water bath for 30 min with occasional stirring. It was then filtered through sintered crucible and washed with hot water as described, and weighed as above. The results have been shown in table 2.

*Effect of the presence of other ions:*

Ni(II), Cu(II), Fe(II), Pd(IV), Pt(IV), Tl(I), Zn(II), Cd(II), Hg(II) and Cr(III) were found to form precipitates with 2-thioorotic acid (ammonium salt) under the conditions of the experiment. These ions

**Table 1** *Estimation of Ag(I) in the absence of chloride ions*

| Wt. of Ag(I) taken (g) | Wt. of Ag(I) found (g) | % deviation |
|---|---|---|
| 0.0539 | 0.0537 | −0.40 |
| 0.1116 | 0.1115 | −0.18 |
| 0.1488 | 0.1482 | −0.40 |
| 0.1674 | 0.1676 | +0.12 |
| 0.2232 | 0.2230 | −0.08 |
| 0.2790 | 0.2798 | +0.29 |

**Table 2** *Estimation of Ag(I) in the presence of chloride ions*

| Wt. of Ag(I) taken as AgCl(g) | Wt. of Ag(I) found (g) | % deviation |
|---|---|---|
| 0 0539 | 0.0537 | −0.40 |
| 0.1116 | 0.1115 | −0.09 |
| 0.1488 | 0.1486 | −0.13 |
| 0.1674 | 0.1676 | +0.12 |
| 0.2232 | 0.2230 | −0.08 |
| 0.2790 | 0.2800 | +0.36 |

must therefore be absent. Bromide, if present even in minute amounts, forms AgBr which is only partially converted to the silver-2-thioorotate complex. Ag(I) undergoes no reaction with the 2-thioorotic acid. Hence bromide and iodide should be absent.

2-Thioorotic acid (amm. salt) has thus been found to be a suitable gravimetric reagent for the estimation of Ag(I) ions. The percentage diviations have been found to be within $\pm 0.4\%$.

An important advantage of the method is that the accuracy of the estimation was not affected adversely by the presence of chloride impurities. The chlorides, on the other hand, facilitated coagulation resulting in easier filtration. Chloride contamination of water and chemical reagents will thus not affect the accuracy of the Ag(I) estimation.

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## STEROLS FROM *RUELLIA PROSTRATA* POIR.

A. K. BANERJEE
*Department of Chemistry, T.D B College, Raniganj, Burdwan 713 347, India.*

THE leaves of *Ruellia prostrata* Poir. is useful for ear diseases and with liquid copal can be used as remedy for gonorrhoea[1]. Earlier investigation revealed only the presence of three flavonoid glycosides *viz.* apigenin 7-glucoside, luteolin 7-glucoside and apigenin 7$\beta$-glucuronide from buds and flowers[2].

In the present communication the presence of sterol mixture obtained from petroleum ether (60–80°) extraction of the dry weed (2 kg) is reported. The gummy matter (16 g), obtained after solvent removal, was chromatographed over silica gel (BDH) and on subsequent elution with petroleum ether-benzene (4:1) furnished TLC pure steroidal fractions ($S_1$ −120 mg.). The sterol fraction on acetylation followed by preparative argentation TLC on a $AgNO_3$-silica gel G (1:4, w/w) plate using $CCl_4$-$CH_2Cl_2$ (5:1, v/v) as developing solvent resulted in the separation of two distinct bands: band 1 ($R_f$ 0.13), band 2 ($R_f$ 0.1) and the most polar faint band ($R_f$ 0.06). All these three bands were subjected to gas chromatographic analysis on a OV-17 scot glass capillary column (30 m × 0.3 mm i.d.) under column temp. 255°C and $N_2$ as carrier gas at the rate of 0.60 ml/min. The constituents were identified through Co-GLC studies with authentic samples. Band 1 gave a mixture of acetates of sitosterol, 24-methyl cholesterol and trace amount of cholesterol. Band 2 afforded stigmasterol acetate and band 3 contains mixture of stigmasterol acetate and trace amount of brassicasterol acetate.

The percentage composition of sterols estimated as their acetates on GLC using identical conditions as previously mentioned, is presented in table 1.

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**Table 1** *Percentage composition of sterols present in steroidal fraction ($S_1$) of R. prostrata*

| Sterol | Composition(%) |
|---|---|
| Stigmasterol | 75.33 |
| Sitosterol | 17 61 |
| 24-methyl cholesterol | 7.04 |
| Cholesterol | traces |
| Brassicasterol | traces |