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NOTE

6-Substituted-1-Hydroxy-1,2,3-Benzotriazoles as Analytical Reagents. Part III : 6-Nitro-1-hydroxy-1,2,3-benzotriazole as Reagent for Silver

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6-Nitro-1-hydroxy-1,2,3-benzotriazole precipitates silver quantitatively as $AgC_6H_4N_4O_3$. The gravimetric estimation of silver by this reagent and the effects of factors like amount of reagent, time of digestion of precipitate and pH on the result have been investigated.

The properties of triazoles and their derivatives to form complexes with metal ions have been utilised by many workers for estimation of metal ions.[1,2] Lorinov *et al.*[3] have reported IR studies of triazoles. IR spectra of some benzo-1,2,3-triazoles have also been reported by O' Sullivan[4]. NMR spectra of 1-(α-aroyloxyarylideneamino)-1,2,3-triazoles have been described by Rodios[5]. Thompson *et al.*[6] have carried out UV studies on triazoles. Several compounds containing the grouping >NOH have been reported to be useful as organic precipitating agents[7]. 1-Hydroxy-1,2,3-benzotriazoles also contain a similar grouping and are, therefore, expected to behave as organic precipiting agents for metal ions. Out of several hydroxy benzotriazoles that have been studied for analytical purpose the use of 6-nitro-1-hydroxy-1,2,3-benzotriazole in gravimetric estimation of silver has been described in the present communication.

6-Nitro-1-hydroxy-1,2,3-benzotriazole was prepared by method given by Macbeth and Price[8]. This was recrystallised before use from hot water and dried in vacuum. Purity of this reagent was established by m.pt., IR and NMR spectra. An aqueous solution (0.7 to 1.0%) of the reagent was used for all estimations. Stock solutions of silver nitrate were prepared by dissolving A.R. grade silver nitrate in distilled water.

The reagent solution react with silver nitrate to form light reddish colour complex. After washing with cold water the complex was dried in drying pistol at about 75°C in vacuum.

An aliquot quantity of silver nitrate solution was diluted to about 100 ml and warmed to 80°C. To it was then added dropwise with constant stirring an excess of hot solution of precipitant. The precipitate was digested on a boiling water-bath for about ½ hr, filtered through

a Gooch Crucible (no. G3) washed with cold water and dried 105–115°C to a constant weight.

In the estimation of silver by the classical method as silver chloride, the solubility of silver chloride causes a rather large error and a correction becomes necessary. Moreover, the method is time consuming, requiring 4–5 hrs for completion. The precipitated silver chloride is highly reactive to light, while silver complex of the 6-nitro-1-hydroxy-1,2,3-benzotriazole is not so. The results are summarised in Table 1 indicate the suitability of this reagent for silver estimations.

TABLE 1

Ag taken (in mg)	Wt. of Complex (in mg)	Ag found (in mg)	Error (in mg)
7.25	22.50	7.156	−0.094
12.72	37.25	12.74	−0.02
20.08	63.23	20.04	−0.04
28.50	90.15	28.48	−0.02
40.08	121.20	40.02	−0.06
45.64	137.23	45.62	−0.02
50.20	155.55	50.18	−0.02
125.60	377.23	125.58	−0.02
200.85	604.00	200.75	−0.05
250.80	754.12	250.60	−0.20
350.40	1056.50	350.14	−0.26

In order to study the effect of pH variation on the precipitation, the estimations were made at various pH values. The results given in Table 2 show that most suitable pH range for such precipitation is 3.5–5.8.

The solution of silver nitrate was kept for different intervals after addition of the precipitant. The results given in Table 3 show that the variation of the interval between the precipitation and filtration does not affect the results.

It is evident from Table 4 that as long as the reagent is not less than $1\frac{1}{2}$ times of stoichiometrically required amount, fairly accurate results are obtained.

TABLE 2

Ag taken = 125.25 mg.

pH Values	Ag found (in mg)	Error (in mg)
2.0	117.21	−8.04
2.5	118.21	−7.04
3.2	123.7	−1.55
3.5	124.4	−0.10
4.0	125.44	−0.06
4.2	125.46	−0.04
4.4	125.23	−0.02
4.6	125.25	±0.00
4.8	125.23	−0.02
5.2	125.2	−0.05
5.5	124.67	−0.58
5.8	124.57	−0.68

TABLE 3

Ag taken = 125.25 mg.

Time (in min.)	Ag found (in mg.)	Error (in mg.)
10	125.25	±0.00
20	125.24	−0.01
45	125.22	−0.03
60	125.21	−0.04
500	125.24	−0.01

TABLE 4

Ag taken = 125.25 mg.

Silver: Reagent (molar ratio)	Wt. of Complex (in mg.)	Ag found (in mg.)	Error (in mg.)
1 : 1.00	360.55	124.90	−0.60
1 : 1.25	362.12	125.10	−0.15
1 : 1.50	362.22	125.14	−0.11
1 : 1.75	362.32	125.24	−0.01
1 : 2.00	361.20	125.16	−0.09
1 : 2.50	361.18	125.25	±0.00
1 : 5.50	360.54	125.10	−0.15

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