Gravimetric and Volumetric Estimation of Camoquine Hydrochloride

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Summary: In the present communication, we report the estimation of camoquine hydrochloride using Reinecke salt (ammonium tetraisothiocyanatodiamminechromate (III), and anilinium tetraisothiocyanatodianilinechromate (III) gravimetrically and volumetrically.

Recently isothiocyanatochromates have been widely used in the analysis of various organic bases of pharmaceutical interest.[1-4] This paper describes that ammonium tetraisothiocyanatodiamminechromate(III) [A] and anilinium tetraisothiocyanatodianiline chromate(III) [B] can be used in the estimation of camoquine hydrochloride in tablets. Aqueous solution of [A] and ethanolic solution of [B] react quantitatively with camoquine hydrochloride giving a yellowish precipitate of the formula camoquine H_2 $[Cr(NCS)_4 L_2]_2$ where L = NH_3 and $C_6 H_5 NH_2$ respectively.

Experimental procedure Reinecke salt (BDH, England) was used in the estimations. Anilinium tetraisothiocyanatodianilianechromate(III) was prepared by method adopted by Ganescu[5]. Camoquine hydrochloride tablets (Parke-Davis)* were used.

Gravimetric Estimation: To an aliquot of camoquine hydrochloride solution, 1.0% aqueous solution of Reinecke salt was added to precipitate out camoquine hydrochloride as camoquine H_2 $[Cr(NCS)_4 (NH_3)_2]_2$ (abbreviated as C.R.). After complete precipitation, the contents were allowed to stand in cold bath for about an hour. The precipitate was collected on a sintered crucible (G_4), washed with cold distilled water and dried at 105-110°C to constant weight (Table-1).

Volumetric Estimation: Comquine H_2 $[Cr(NCS)_4 (C_6 H_5 NH_2)_2]_2$ (abbreviated as C.A.) was precipitated by adding 1% alcoholic solution to sample solution of camoquine hydrochloride and collected on a filter paper (Whatman No. 42). Thereafter about 20 ml of 1.0 N KOH was added to the filter paper plus contents, boiled for about 15 minutes and cooled. The filtrate was acidified with dil HNO_3 and 0.05 N $AgNO_3$ was added to precipitate out all SCN^- ions. The excess of $AgNO_3$ was titrated against 0.05 N KSCN solution by Volhard's method. The precipitate of C.R. has been used for the volumetric estimations (Table 2).

In another method, after decomposition with 1.0 N KOH solution, the precipitate of $Cr(OH)_3$ was dissolved in conc. HCl. To this solution 20 drops of ICl, 20 ml conc. HCl and 10 ml CCl_4 were added. The iodine thus liberated was titrated against 0.1 N KIO_3 with frequent shaking.

Results and Discussion

Camoquine hydrochloride reacts quantitatively with [A] and [B] to form complexes of the general formula, camoquine- $H[Cr(NCS)_4 L_2]_2$ where L=NH_3 and $C_6 H_5 NH_2$ respectively. The structural formulae of the complexes were confirmed by determining the % SCN in the complexes. The complexes formed are insloluble in water hence easy to wash. The complex camoquine $H_2 [Cr(NCS)_4 (NH_3)_2]_2$ is quite stable upto 150°C whereas camoquine $-H_2(Cr(NCS)_4 (C_4 H_5 NH_2)_2]_2$ is thermally unstable and decomposes at about 70°C. Hence gravimetric estimation is applicable only to camoquine $H_2 [Cr(NSC)_4 (NH_3)_2]_2$ while volumetric estimation is is applicable to both. The present methods of assaying camoquine in pharmaceuticals are better and more convenient than the conventional methods described in British Pharamacopoeia[6] and the Pharmacopoeia of India[7]. The gravimetric method is very simple, rapid and convenient. The precipitate obtained is very easy to wash and is stable upto 150°C. Since the molecular weights of the complexes are quite high (i.e. 993.8 and 1207.7 respectively), the method can be applied to micro and semimicro determinations of camoquine in pharmaceutical preparations. The reproducibility of the method lies within ± 2%.

Acknowledgement

*No other commercial brands of camoquine are available.

Table 1 Gravimetric estimation

Expt. No.	Amount of Camoquine reineckate Calcd. (gm)	Amount of Camoquine reineckate Found (gm)	% Error
1.	0.0912	0.0902	− 1.09
2.	0.0912	0.0906	− 0.66
3.	0.1824	0.1816	− 0.44
4.	0.1824	0.1818	− 0.33
5.	0.3648	0.3650	+ 0.05
6.	0.3648	0.3646	− 0.05

Table 2 Volumetric estimation

Expt. No.	Volumetric estimation by Volhard's method			Volumetric estimation by Iodometric method		
	Amount of Camoquine hydrochloride Calcd. (gm)	Amount of Camoquine hydrochloride Found. (gm)	% Error	Amount of Camoquine hydrochloride Calcd. (gm)	Amount of Camoquine hydrochloride Found. (gm)	% Error
	C.R.			C.R.		
1.	0.03378	0.03349	− 0.85	0.03896	0.03876	− 0.51
2.	0.03378	0.03345	− 0.98	0.03896	0.03872	− 0.62
3.	0.06756	0.06725	− 0.45	0.07792	0.07780	− 0.15
4.	0.06756	0.06742	− 0.20	0.07792	0.07774	− 0.23
5.	0.13512	0.13509	− 0.02	0.15584	0.15542	− 0.27
6.	0.13512	0.13504	− 0.05	0.15584	0.15490	− 0.60
	C.A.			C.A.		
1.	0.03378	0.03351	− 0.80	0.03863	0.03850	− 0.34
2.	0.03378	0.03354	− 0.71	0.07726	0.07721	− 0.06
3.	0.06756	0.06730	− 0.38	0.07726	0.07724	− 0.03
4.	0.06756	0.06726	− 0.44	0.15452	0.15440	− 0.08
5.	0.13512	0.13503	− 0.07	0.15452	0.15439	− 0.08
6.	0.13512	0.13498	− 0.10	0.15452	0.15446	− 0.04

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