

solution indicates there is a reaction occurred/not occurred with the base and acid used for titration

5. Furosemide

Aim:-

To perform furosemide assay as per IP

References

1. Delgado J N & Remers W A (Eds), Wilson and Girvold's Text Book of Organic and Medicinal and Pharmaceutical Chemistry, 9th edn (J B Lippincott Co., Philadelphia, PA), 1991, 525.
2. Foye W O (Ed), Principles of Medicinal Chemistry, 3rd edn (Lea & Febiger, Philadelphia, PA), 1989, 408.
3. Gaitonde C D & Jayade P P, Indian Drugs, 28 (1991) 242. 4 Anapure S A, Khanna S & Dighe V S, East Pharm, 32 (1989) 193.
4. Stoberski P, Zakrezevski Z & Szulic A, Farm Pol, 44 (1988) 398.
5. K Basavaiah*, U Chandrashekar & P Nagegowda Indian Journal of Chemical Technology Vol. 12, March 2005, pp. 149-155.

Apparatus and Material

- 6) 50ml burettes (2)
- 7) 600ml beaker
- 8) Burette clamp
- 9) Water bath

Experimental Procedure

Titrimetry

All chemicals used were of analytical reagent grade and double distilled water was used to prepare all solutions. A bromate-bromide mixture (5 mM KBrO_3 - 50 mM KBr) was prepared by dissolving 0.835 g of KBrO_3 and 6 g of KBr in water and diluting to 1 L in a volumetric flask and used for titrimetric work. Methyl orange indicator (0.5 %) was prepared by dissolving 50 mg of dye in 10 mL of water. Hydrochloric acid (2 M) was prepared by diluting 177 mL of concentrated acid (S. d. Fine Chem. India, sp. gr. 1.18) to 1 L with water.

Titrimetric assay

A 10 mL aliquot of pure drug solution containing 2-20 mg of FRU was accurately transferred into a 100 mL titration flask, 10 mL of 2 M HCl was added and titrated with bromate-bromide mixture (5 mM w. r. t. KBrO_3) using 2 drops of methyl orange indicator till the disappearance of the indicator colour. A blank titration was performed and the volume of titrant was subtracted from the volume required for drug solution titration.

The amount of FRU in the measured aliquot was calculated from:

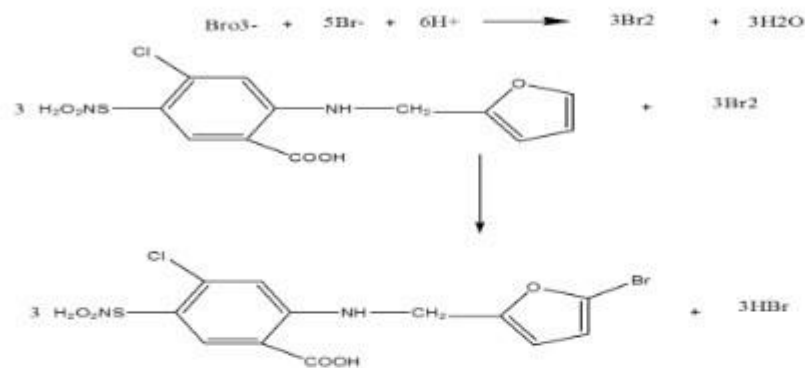
$$\text{Amount (mg)} = VMwR 0.333$$

Where V = volume of bromate-bromide consumed, mL

Mw = relative molecular mass of drug R = molarity of bromate-bromide mixture w. r. t.

KBrO₃.

REACTION



Results and Discussion Optimisation of reaction conditions

Titrimetry

The quantitative nature of the reaction between FRU and in situ generated bromine was checked by titrating 2-20 mg of drug to a methyl orange end point. In the range studied, the reaction stoichiometry was found to be 3:1 (FRU: KBrO₃) which can be represented by scheme 1. The reaction was carried out in HCl medium and the reaction stoichiometry was found to be unaffected when 5-20 mL of 2 M HCl was used in a total volume of 30-40 mL. The linear relationship between the drug amount and the titration end point is apparent from the calculated correlation coefficient of -----obtained by the best fit line via least squares treatment.