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Analysis of drug ofloxacin in bulk and single component pharmaceutical tablets by non-aqueous potentiometric titration method

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Analysis of drug ofloxacin in bulk and single component pharmaceutical tablets has been carried out by non-aqueous potentiometric titration method using isopropyl alcohol as the solvent and KOH in isopropyl alcohol as the titrant. The effect of solvent and concentration on potentiometric analysis of drug ofloxacin as well as its analysis in bulk and single component pharmaceutical tablets has been studied by using a pair of glass and calomel electrode. The method was found to be quite simple, efficient, precise and gave results comparable to those obtained by Indian Pharmacopoeia (I.P.) method.

Keywords: Analysis, non-aqueous, potentiometric titration, ofloxacin.

Introduction

Analysis of various drugs by non-aqueous potentiometric titration method using different electrode pairs has been reported earlier[1]. Numbers of methods for the determination of drug ofloxacin are reported literature[2]. Its estimation by potentiometry and conductometry has been reported earlier by few workers[3]. It has been also determined by spectrophotometric, spectrofluorometric methods[4] and analyzed by high pressure liquid chromatography with column switching[5]. Ofloxacin is distinctly acidic and it could not be titrated directly with aqueous alkali owing to its easy hydrolysis. The basic titrant is also superior to the alkoxide solvents which are more susceptible to the atmospheric moisture and carbondioxide. The purpose of present work is to find out simple analysis method for common pharmaceutical drugs. It will help to analyze raw materials and products for quick check of spurious drugs that are feared to penetrate the markets. In this communication, analysis of drug ofloxacin in bulk and single component pharmaceutical tablets by non-aqueous potentiometric titration method using isopropyl alcohol as the solvent and KOH in isopropyl alcohol as the titrant has been reported. The study of effect of solvent and concentration on potentiometric analysis of drug ofloxacin has also been attempted.

Results and discussion

Study of effect of solvent and concentration on potentiometric analysis of ofloxacin:

In the study of effect of solvent, accuracy of results in analysis of drug ofloxacin by using different solvents was checked by non-aqueous potentiometric titration method. The required volumes of stock solutions of drug ofloxacin in different solvents were diluted to 20 ml and then titrated separately with KOH in isopropyl alcohol. It can be seen from the results that, accuracy of result in analysis of ofloxacin by using solvent isopropyl alcohol is much more with minimum % error as compare to other solvents (Table 1). The potentiometric breaks obtained using the solvents dimethyl formamide and acetone are smoother one as compared to methanol whereas the potentiometric break obtained using isopropyl alcohol is much more pronounced and prominent with maximum potential difference near the equivalence point (Fig. 1). The dielectric constant of isopropyl alcohol is smaller than dimethyl formamide, methanol and acetone. It permitted a large change in the solvated pro-

Table 1. Study of effect of solvent on potentiometric analysis of ofloxacin

Solvent	Weight titrated (mg) (±0.5%)	Weight found (mg)	Error (%)
Acetone	7.2272	7.1128	–1.58
Methanol	7.2272	7.2920	+0.89
Dimethyl formamide	7.2272	7.2608	+0.46
Isopropyl alcohol	7.2272	7.1498	–1.07

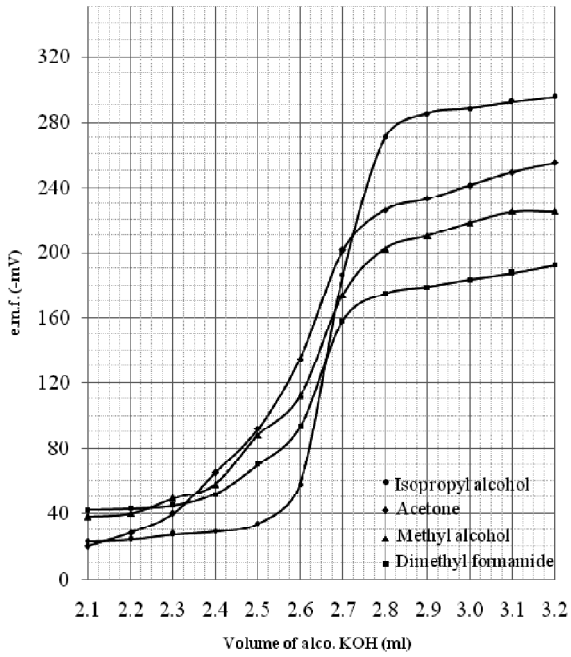

Fig. 1. Study of effect of solvent on potentiometric analysis of ofloxacin.

ton concentration near the end point. As compared to other solvents, isopropyl alcohol can be purified and made anhydrous very easily.

For the study of effect of concentration and to determine the suitable concentration range that gives best results, different volumes of the stock solution of ofloxacin were diluted to 20 ml with isopropyl alcohol and titrated separately with KOH in isopropyl alcohol. It can be seen from the results that, potentiometric method gave an accuracy of ±0.6% for the entire range of 3.610 to 36.100 mg. The results obtained are quite good and much more accurate than other methods with both positive as well as negative errors (Table 2). This method of analysis is found to be better in respect of indicator error than the visual titration method given in pharmacopoeias. The potentiometric breaks obtained are much more pronounced (Fig. 2). The mean, mean deviation and standard deviation values of study of effect of concentration on potentiometric analysis of ofloxacin are 19.855, 9.025, 10.929 (for weight titrated); 19.824, 8.975, 10.863 (for weight found) and 0.016, 0.472, 0.504 (for % error) respectively.

Table 2. Study of effect of concentration on potentiometric analysis of ofloxacin

Weight titrated (mg)	Weight found (mg)	Error (%)
3.610	3.626	+0.44
7.220	7.262	+0.58
10.830	10.889	+0.54
14.440	14.356	–0.58
18.050	18.110	+0.33
21.660	21.780	+0.55
25.270	25.153	–0.46
28.880	28.758	–0.42
32.490	32.328	–0.49
36.100	35.978	–0.33

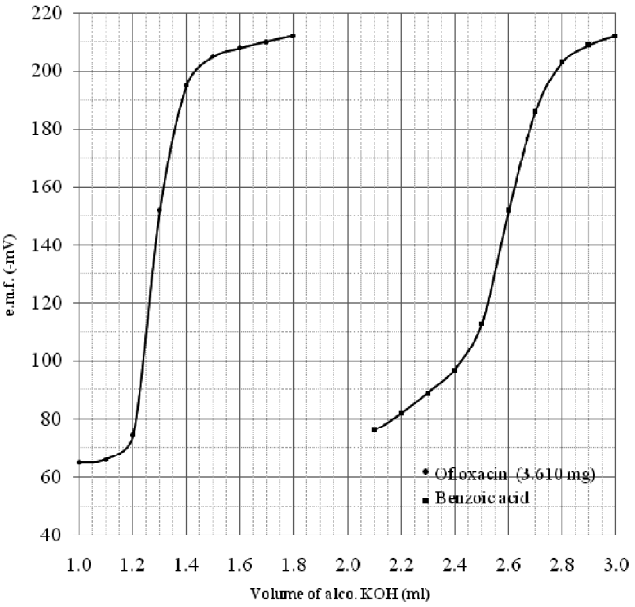

Fig. 2. Study of effect of concentration on potentiometric analysis of ofloxacin.

Analysis of ofloxacin in single component pharmaceutical tablets:

The drug ofloxacin containing ten pharmaceutical tablets of the same batch were accurately weighed and powdered. The required quantity of powder was weighed accurately, it was extracted with isopropyl alcohol and the volume was made to 100 ml. An aliquot of 10 ml of this solution was diluted with isopropyl alcohol to 20 ml and titrated with KOH in isopropyl alcohol using potentiometer. The titrant was standardized by performing potentiometric titration using standard benzoic acid in isopropyl alcohol. The weight of ofloxacin present in one tablet was calculated. The same tablet was analyzed by I.P. method. The results obtained for four different brands of pharmaceutical tablets are tabulated and it is observed that, the present potentiometric method gives fairly accurate and comparable results to those obtained by I.P. method (Table 3). This method is much better, accurate and simple than the methods reported in the literature. It is free from indicator error or interferences. Ofloxacin gets hydrolyzed in presence of aqueous alkali but this is avoided in non-aqueous medium. The most common additives present in the pharmaceutical tablets are calcium carbonate, sugars, gum etc. and as these are insoluble in isopropyl alcohol do not affect the results.

Table 3. Analysis of ofloxacin in single component pharmaceutical tablets

Sample	Label claim	Weight found (mg)	
	(mg)	I.P. method	Present method
A	200.0	201.33	200.84
B	200.0	198.06	199.59
C	200.0	201.88	200.03
D	200.0	198.62	199.74

Experimental

All the titrations were carried out by using digital potentiometer (Equiptronics, EQ-602). Glass and calomel electrodes were used as indicator and reference electrode respectively. Weighing were made on Precisa-310M (±0.001 g) balance. The chemicals and solvents of A.R. grade were used. All solvents were purified and made anhydrous by standard methods[6]. Care was taken to protect the titrant from atmospheric moisture and carbon dioxide. The drug ofloxacin used for present study was obtained from pharmaceutical laboratories. It is of pharmaceutical in nature and is included in pharmacopoeias[7].

Study of effect of solvent and concentration on potentiometric analysis of ofloxacin:

For the study of effect of solvent on potentiometric analysis of ofloxacin, its stock solutions (3.613 mg/ml, ±0.5%) were prepared by dissolving it in the solvents acetone, methanol, dimethyl formamide and isopropyl alcohol. Then 2 ml of these solutions were diluted to 20 ml with same solvents and separately titrated with KOH in isopropyl alcohol using a pair of glass and calomel electrodes. To study the effect of concentration, stock solution of ofloxacin (3.610 mg/ml) was prepared by dissolving it in isopropyl alcohol. Different volumes (1 to 10 ml) of the stock solution were diluted to 20 ml with isopropyl alcohol and separately titrated with KOH in isopropyl alcohol by adding titrant in the lots of 0.1 ml with continuous stirring using magnetic stirrer. The potential developed across two electrodes was measured after each addition. A waiting period of about 1 to 2 min was allowed to get the potential stabilized. The addition was continued till 0.3 to 0.5 ml excess of titrant was added. At the end point readings were recorded for each addition of 0.02 ml of titrant. The end points were found by plotting the graphs of potential developed against volume of the titrant.

Analysis of ofloxacin in single component pharmaceutical tablets:

In this analysis, the drug ofloxacin containing ten tablets of the same batch were accurately weighed and powdered. The powder containing 100 mg of the drug was weighed accurately, treated with 50 ml of isopropyl alcohol and vigorously stirred to dissolve the active component of the tablet. The most common additives present in the pharmaceutical tab-

lets are calcium carbonate, sugars, gum etc. which are mostly insoluble in isopropyl alcohol. The solution was filtered, residue was washed three to four times with small portions of isopropyl alcohol and the volume of solution was made to 100 ml with isopropyl alcohol. An aliquot of 10 ml of this solution was diluted to 20 ml with isopropyl alcohol and titrated with 0.1 *M* of solution of KOH in isopropyl alcohol by potentiometric method using glass and calomel electrodes. The titrant was standardized by potentiometric titration with 0.1 *M* benzoic acid in isopropyl alcohol. The end points were determined by plotting graphs as described earlier; the amount of drug present in titrated weights of tablet powder was calculated. The amount of active component (drug) present in one tablet was calculated from the average weight of the tablet. The same tablets were then analyzed by method of pharmacopoeias and results obtained were compared.

Conclusion

The pharmaceutical drug selected for present study was ofloxacin. Being distinctly acidic it could not be titrated directly with aqueous alkali owing to its easy hydrolysis but the non-aqueous titration of ofloxacin gave better results. The solvent isopropyl alcohol is found to be excellent for all titrations. The basic titrant, potassium hydroxide in isopropyl alcohol was superior to the alkoxide solvents that are more susceptible to atmospheric moisture and carbondioxide. It gave better potentiometric breaks. The calomel and glass electrode pair gave stable potentials which were quickly attained. The potentiometric breaks obtained using these electrode pair systems were quite larger. In present study, method for analysis of acidic drug ofloxacin was developed. It is simple, efficient, precise and can be used even in common laboratories without use of any sophisticated instrument.

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