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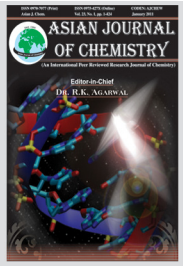

# Spectrophotometric Determination of Buflomedil Hydrochloride with Oxidation of Potassium Permanganate

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A new spectrophotometric method was developed for the determination of buflomedil hydrochloride. The method was based on the oxidation of the drug with potassium permanganate in alkaline medium. Potassium permanganate reacted to buflomedil hydrochloride with the absorbance increasing at 430 and 610 nm and decreasing at 518 nm obviously in the presence of sodium hydroxide. The variation values of absorbance were linear to the concentration of buflomedil hydrochloride within the range of 1-22 μg $mL^{-1}$. The apparent molar absorption coefficients were $1.7 \times 10^4$, $0.7 \times 10^4$ and $1.2 \times 10^4$ L $mol^{-1}$ $cm^{-1}$ at 430, 518 and 610 nm, respectively. By superposing the absorbance at the three wavelengths, the apparent molar absorption coefficients increased to $3.9 \times 10^4$ L $mol^{-1}$ $cm^{-1}$. The proposed method was applied to the determination of buflomedil hydrochloride in pharmaceutical dosage forms with the recovery of 98.2-102.5 %.

**Key Words: Buflomedil hydrochloride, Spectrophotometry, Potassium permanganate, Oxidation.**

## INTRODUCTION

Buflomedil hydrochloride [4-(1-pyrrolidinyl)-1-(2',4',6'-trimethoxyphenyl)-1-butanone hydrochloride, BC] (Fig. 1) is a vasodilator drug, which has the effect of relaxing vascular smooth muscle, dilating blood vessels and reducing vascular resistance. It has been widely used for the treatment of both peripheral and cerebral vascular diseases.

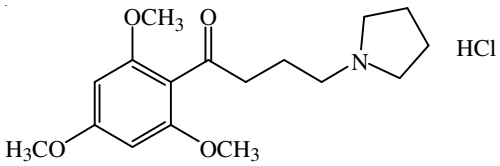


Fig. 1. Structure of buflomedil hydrochloride

Generally, buflomedil hydrochloride are determined mainly by high-performance liquid chromatography[1-3]. Gas chromatography[4] has also been reported for its determination. But the methods of chromatography have the shortcomings of expensive apparatus and complicated operation, *etc*. Spectrophotometry has many advantages such as simplicity of operation and low price, which has been widely used into the determination of compounds of pharmaceutical preparations, *e.g.*, cephalosporin and $H_2$ receptor antagonists[5-7]. In this paper, a spectrophotometric method for the determination of buflomedil hydrochloride based on the oxidation-reduction reaction between potassium permanganate and buflomedil hydrochloride in alkaline medium was carried out. The experimental results indicated that the proposed method was accurate, simple, sensitive and rapid and was successfully used for the determination of buflomedil hydrochloride in pharmaceutical preparations.

## EXPERIMENTAL

Buflomedil hydrochloride (Kanion Pharmaceutical Co. Ltd., China) standard stock solution (1 g $L^{-1}$) was prepared by dissolving 0.1000 g of the drug in water and diluting to the mark in a 100 mL volumetric flask. The standard working solution was obtained by further dilution of this stock solution with water. Potassium permanganate solution (0.5 g $L^{-1}$) and sodium hydroxide solution (0.1 mol $L^{-1}$) were prepared according to routine method. All chemicals used were of analytical grade and double distilled water was used throughout the study.

Buflomedil hydrochloride tablets (Livzon Pharmaceutical Co. Ltd., China) are labeled to contain 150 mg of buflomedil hydrochloride per tablet. buflomedil hydrochloride injections (Kanion Pharmaceutical Co. Ltd., China) are labeled to contain 50 mg of buflomedil hydrochloride per 5 mL.

A 722-N spectrophotometer (Shanghai precision and scientific instrument Co. Ltd., China) with matched 1 cm glass cells was used for all the spectrophotometric determination.

**Preparation of sample solution**

Ten tablets of buflomedil hydrochloride were weighed and finely powdered. An accurately weighed quality of the powder containing about 100 mg of buflomedil hydrochloride was transferred into a 100 mL volumetric flask and diluted to the mark with water. The mixtures was mixed well and filtered. A measured volume (1 mL) of the filtrate was quantitatively diluted to 100 mL with water and the resulting solution was used as sample solution for analysis by the procedure.

**Injection:** An accurately measured volume (1 mL) of buflomedil hydrochloride injection was transferred and diluted to the mark of a 100 mL volumetric flask. 10 mL of the above solution was diluted quantitatively to 100 mL with water. The prepared solution was used as sample solution.

**Procedure:** A suitable volume, accurately measured, of the standard working solution or sample solution was transferred into a 10 mL colorimetric tube. 2.5 mL of potassium permanganate solution and 2 mL of sodium hydroxide solution was added. The solution was diluted to the mark with water and kept aside for 90 min to allow complete reaction. The reagent blank solution which was absence of buflomedil hydrochloride was prepared as above. The absorbance was measured at 430, 518 and 610 nm against reagent blank solution, respectively. The amount of buflomedil hydrochloride present in sample solution was computed from the calibration curves.

## RESULTS AND DISCUSSION

**Absorption spectra:** The absorbance spectra of potassium permanganate in presence and absence of buflomedil hydrochloride were illustrated in Fig. 2. The investigated buflomedil hydrochloride had no absorbance ability at the measuring wavelength. So from Fig. 2, it could be found that the addition of buflomedil hydrochloride to potassium permanganate resulted in the absorbance increasing at 430 and 610 nm while decreasing a 518 nm. For further study, these three wavelengths were selected as the measurement wavelengths.

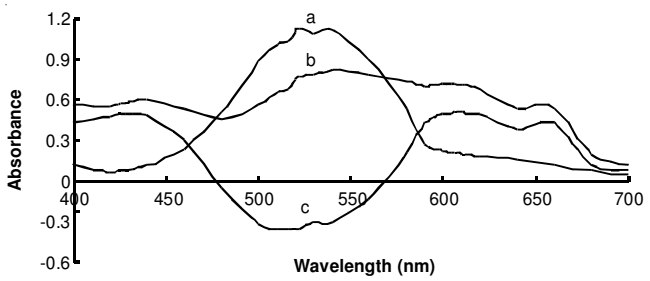


Fig. 2. Absorbance spectra of potassium permanganate against water (a) and the reaction product between potassium permanganate and buflomedil hydrochloride against water (b) and against reagent blank (c) in the medium of NaOH; 0.1 g $L^{-1}$ potassium permanganate; 10 μg $mL^{-1}$ buflomedil hydrochloride; 0.02 mol $L^{-1}$ NaOH; 90 min

**Effect of NaOH concentration:** The effect of the NaOH concentration on the absorbance of the system was studied over the NaOH concentration range of 0.005-0.04 mol $L^{-1}$. The results were shown in Fig. 3. It was found that the absorbance reached maximum and kept constant in the concentration range of 0.02-0.04 mol $L^{-1}$. Therefore, NaOH concentration of 0.02 mol $L^{-1}$ was selected for further study.

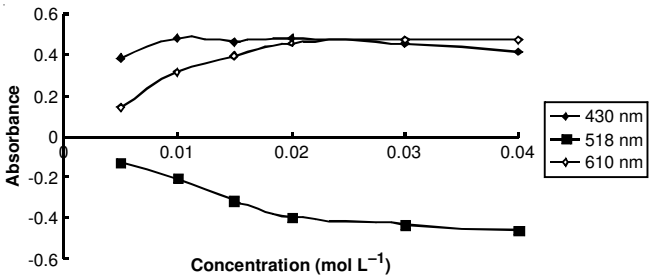


Fig. 3. Effect of NaOH concentration; 0.1 g $L^{-1}$ potassium permanganate; 10 μg $mL^{-1}$ buflomedil hydrochloride; 90 min

**Reaction time and stability:** The absorbance of the system was measured in the time intervals varying from 10-180 min. The results obtained were presented in Fig. 4. The absorbance increased slowly by increasing reaction time and it reached maximum and kept constant at 90-180 min. In this study, the absorbance of the system was measured after keeping the solution aside for 90 min.

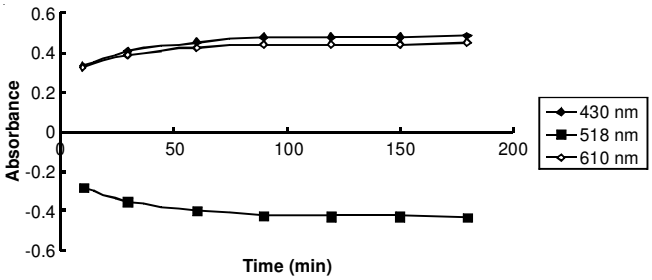


Fig. 4. Effect of reaction time; 0.1 g $L^{-1}$ potassium permanganate; 10 μg $mL^{-1}$ buflomedil hydrochloride; 0.02 mol $L^{-1}$ NaOH

**Effect of potassium permanganate concentration:** The effect of potassium permanganate concentration was investigated in the range of 0.025-0.15 g $L^{-1}$. The results were shown in Fig. 5. As this figure shown, with increase of the potassium permanganate concentration up to 0.1 g $L^{-1}$, the absorbance increased and then it reached maximum and kept constant. For further work, the potassium permanganate concentration of 0.1 g $L^{-1}$ was selected.

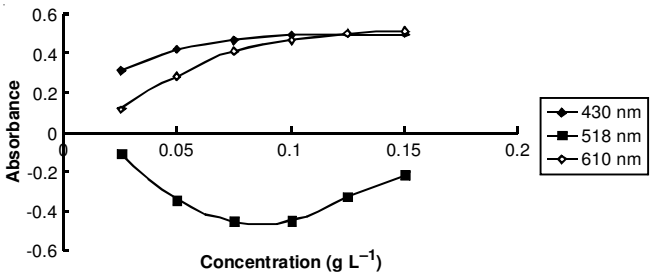


Fig. 5. Effect of potassium permanganate concentration; 10 μg $mL^{-1}$ buflomedil hydrochloride; 0.02 mol $L^{-1}$ NaOH; 90 min

**Calibration curves and detection limits:** Under the above-mentioned optimum conditions, the calibration curves at 430, 518 and 610 nm were constructed (Fig. 6). Regression analysis for the results was carried out using least-square method. The regression equations and related parameters were shown in Table-1. In all cases, Beer's law was obeyed with

TABLE-1
REGRESSION EQUATIONS OF CALIBRATION CURVES AND RELATED PARAMETERS

| Wavelength (nm) | Regression equation | Correlation coefficient | Molar absorption coefficient (L $mol^{-1}$ $cm^{-1}$) | LOD (μg $mL^{-1}$) |
|---|---|---|---|---|
| 430 | $A_{430\ nm} = 0.0506C + 0.0234$ | 0.9990 | $1.7 \times 10^4$ | 0.09 |
| 518 | $A_{518\ nm} = -0.0292C - 0.0844$ | 0.9990 | $0.7 \times 10^4$ | 0.15 |
| 610 | $A_{610\ nm} = 0.0345C + 0.0356$ | 0.9990 | $1.2 \times 10^4$ | 0.13 |
| Superposition | $A_t = 0.1143C + 0.1434$ | 0.9993 | $3.9 \times 10^4$ | 0.04 |

TABLE-2
DETERMINATION OF BC IN PHARMACEUTICAL FORMULATIONS AND THE RESULTS OF RECOVERY

| Samples | Wavelength (nm) | Percentage of labeled value (w/%) | RSD (%) | Added (μg) | Recovered (μg) | Recovery (%) |
|---|---|---|---|---|---|---|
| Tablets | 430 | 102.7 | 1.6 | 10.00 | 9.82 | 98.2 |
| | 518 | 101.6 | 1.6 | 10.00 | 10.12 | 101.3 |
| | 610 | 101.9 | 2.0 | 10.00 | 10.05 | 100.5 |
| | Superposition | 102.2 | 1.8 | 10.00 | 9.97 | 99.7 |
| Injections | 430 | 98.9 | 1.8 | 10.00 | 9.99 | 99.9 |
| | 518 | 99.2 | 2.3 | 10.00 | 10.25 | 102.5 |
| | 610 | 99.5 | 2.0 | 10.00 | 9.98 | 99.8 |
| | Superposition | 99.3 | 2.1 | 10.00 | 10.10 | 101.0 |

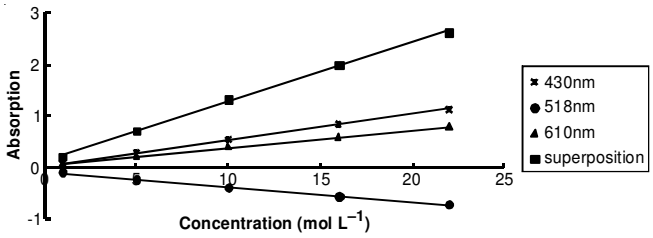


Fig. 6. Calibration curves: 0.1 g $L^{-1}$ potassium permanganate; 0.02 mol $L^{-1}$ NaOH; 90 min

good correlation coefficients in the concentration range of 1-22 μg $mL^{-1}$. By superposing the absorbance of the three wavelength ($A_t = A_{430\ nm} - A_{518\ nm} + A_{610\ nm}$), the apparent molar absorption coefficient increased 2-4 times which means increasing of sensitivity. The limits of detection (LOD) were determined using the formula: LOD = 3σ/b, where σ is the standard deviation of the reagent blank for 11 times determination and b is the slope of the calibration curves and the results were also shown in Table-1.

**Selectivity:** The interference of possible coexisting substances was studied. A sample containing 10 μg $mL^{-1}$ of buflomedil hydrochloride and various concentrations of foreign substances were prepared and the determination of absorbance was carried out as described above in the procedure. When the relative error was within ± 5 %, 100 times concentration of foreign substances such as magnesium stearate, starch, glucose, lactose and sucrose were not interfere.

**Application:** In order to test the utility of this improved method, it was applied to the determination of buflomedil hydrochloride in commercial tablets and injections. Moreover, to check the validity of the method, the standard addition method was applied by adding buflomedil hydrochloride to the previously analyzed tablets and injections. The analytical results were presented in Table-2. It can be concluded that the proposed method provides a good accuracy and precision.

## Conclusion

The acidic potassium permanganate method is widely used in the area of analytical chemistry, while alkaline potassium permanganate method is few used. In alkaline medium, many metal ions will generate precipitation and the interference of them will be eliminated. Therefore, alkaline potassium permanganate method is more selective than acidic method to some extend.

## ACKNOWLEDGEMENTS

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