Spectrophotometric Determination of Aspirin in Pharmaceutical Formulations by Oxidation with Potassium Dichromate

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Summary: A simple and rapid spectrophotometric method for determination of aspirin is described. The method is based on oxidationof aspirin with dichromate ions. Absorbance of Chromium(III) produced was measured at 582 nm. The method is employed for the determination of aspirin in drug samples. The results have been compared with those reported on the samples.

Introduction

Aspirin (acetylsalicylic acid) is well known for its analgestic and antipyretic character. The drug has been usually determined by spectrophotometry [1-3], gas chromatography [4] and high pressure liquid chromatography [5,6]. Though these methods are sensitive enough but require lengthy procedures, sophisticated instrumentation and strict control of conditions.

The present method is base don oxidation of aspirin with potassium dichromate in presence of concentrated sulphuric acid. Addition of aspirin samples to the acidic solution of dichromate produced a green colouration due to the formation of chromium (III) ions. Absorbance of chromium (III) is measured at 582 nm and plotted against aspirin concentration. The method has been employed for determining aspirin in a number of drug samples. Paracetamol and ascorbic acid interfered the determination.

Experimental

Equipment

A Pye Unicam SP 8-400 UV/Vis double beam spectrophotometer with a built-in recorder was used for absorbance measurements.

Reagents

Aspirin sample for stock solution was prepared in author's laboratory by the method described by Vogel [7]. The purity of the sample was determined by the official method [8] and found to be 100.2%. Stock solution containing 1 mg/ml aspirin was prepared by dissolving 0.5g anhydrous aspirin in 500 ml of 9M sulphuric acid. AFter complete dissolution the volume was made to 500 ml with distilled water.

Stock solution of potassium dichromate, 0.1 M, was prepared by dissolving 14.7g of the reagent in 500 ml of (M sulphuric acid.

General Procedure

Place 5 ml of potassium dichromate stock solution in five 25 ml standard flasks. Add 5 ml of 9M sulphuric acid to each flask. Then transfer the aliquots of aspirin solution, containing 0.1-0.5 mg aspirin, into the flasks. Heat the contents at 80-85°C in a water bath for 15 minutes and then dilute to the mark with water. Prepare a compensatory blank without adding any aspirin. Measure the absorbance of each flask solution at 582 nm using compensatory blank as reference.

Drug Samples

For assay of drug samples, 10 tablets of the drug were crushed and ground to fine powder. A quantity of powder equivalent to 200-500 mg aspirin was accurately weighed and dissolved in 100 ml of 9M sulphuric acid. After thorough shaking the solution was filtered through Whatman No.41 filter paper. The undissolved material was washed twice with 100 ml portions of 6M sulphuric acid and finally with 100 ml water. The combined filtrate, and washings were diluted to 500 ml with water.

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An aliquot of prepared sample solution containing appropriate amount of aspirin was treated with dichromate solution and sulphuric acid as described in general procedure. The aspirin content was calculated from calibration graph.

Results and Discussion

In the described procedure, potassium dichromate is used to oxidize aspirin. The absorbance of chromium (III) species produced, measured at 582 nm is taken as a measure of aspirin concentration. To establish the optimum conditions of determination, the effects of various parameters such as acid concentration, time and temperature have been checked. The oxidation reaction is slow but can be accelerated by increasing acid concentration as well as by heating the contents at 80°C.

A linear calibration, shown in Fig.1 was obtained for 0.1-0.5 mg of aspirin. Beer's law was found to be valid upto 2.5 mg/ml aspirin concentration in the final solution. Fig. 2 shows the psectral scans of the reaction mixture. The spectral maxima at 582 nm and 430 nm in Fig.2(a), representing chromium (III) and chromium (VI), respectively, were obtained when water was used as reference. While the spectrum reproduce in Fig. 2(b) showing the maxima for chromium (III) only, was obtained by using compensatory blank as reference. The precision of the method was checked by analysing eight samples of aspirin containing 0.3 mg of the drug. The value of standard deviation obtained was 0.025.

The mechanism of aspirin oxidation with dichromate in the presence of sulphuric acid is probably as follows:

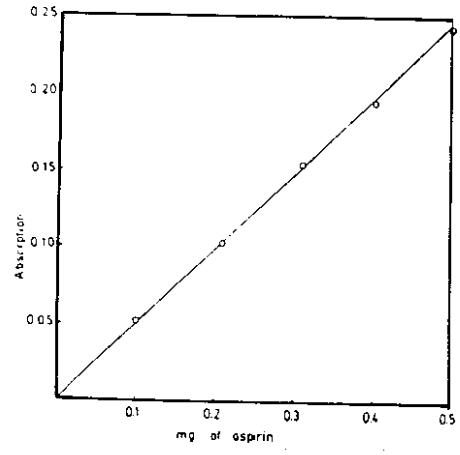


Fig. 1: Calibration of Aspirin determinationin drug.

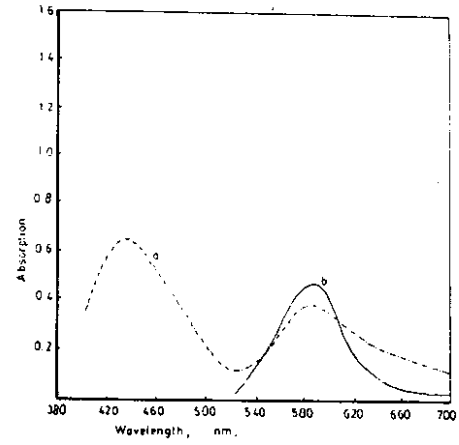
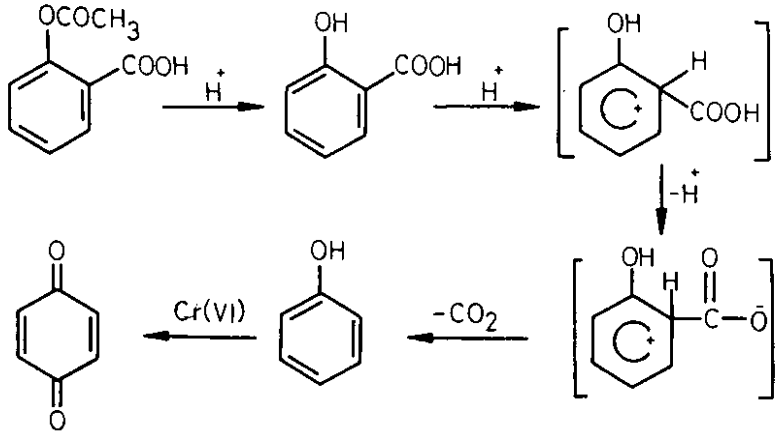


Fig. 2: Absorption spectra of reaction mixture a: (----) using water as reference; b: (_____) using compensatory blank as reference.

Effect of Acid Concentration

Chromium(III) absorbance at 582 nm has been found highly dependent upon sulphuric acid concentration. As shown in Fig. 3, by using 2M sul-



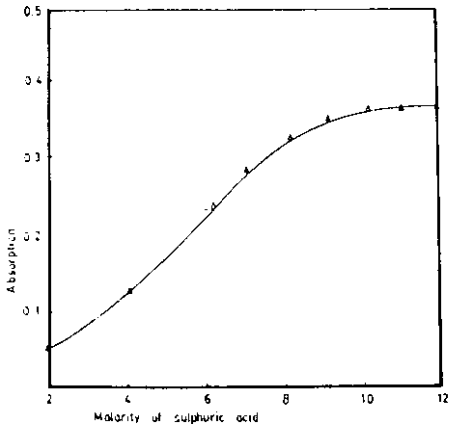


Fig. 3: Effect of sulphuric acid concentration on chromium (III) absorbance

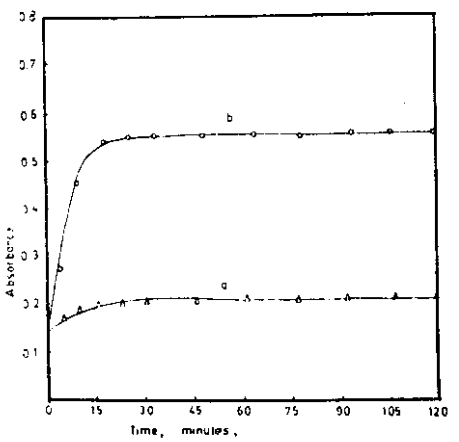


Fig. 4: Effect of time on absorption of chromium (III) a: ___o___ at room temperature; b: ___Δ___ at 80°C.

phuric acid the absorbance was almost negligible. Then it increased significantly with the increase in acid concentration. The maximum absorbance was obtained by using 9M sulphuric acid. Further increase in acid strength did not cause any significant effect on the absorbance.

Effect of Reaction Time

Effect of time has been checked at room temperature as well as at 80°C. As shown in Fig. 4, in both cases the maximum absorbance was obtained in first 15 minutes. At room temperature the absorbance was negligibly small which did not increase to any significant extent even by keeping the reaction mixture for two hours. This reveals that time factor has not any significant role in the oxidation of aspirin.

Effect of Temperature

To check the effect of temperature, the reaction was carried out at different elevated temperatures. It can be seen in Fig. 5 that the absorbance slowly increased with temperature upto 60°C. After that there was an abrupt increase in the absorbance. The maximum absorbance was obtained at 80°C. Further increase in temperature did not show any considerable effect on the absorbance.

Samples

The described method was employed for the determination of aspirin in its pure form as well as in drugs supplied by different companies. Most of results obtained, summarized in Tbale-1, are in well agreement with those reported for these drugs.

Among the other common drugs only paracetamol and ascorbic acid interfered and caused large positive errors. This is because of the fact that paracetamol and ascorbic acid can also be oxidized with potassium dichromate in strong sulphuric acid media resulting in green chromium (III) ions.

Conclusion

The method, described in this work, for aspirin determination is based on oxidation of aspirin with dichromate ion in presence of sulphuric acid. To optimize the conditions for quantitative oxidation, effect of various parameters have been checked. It has been found that oxidation reaction completes at 80°C and in presence of 9M

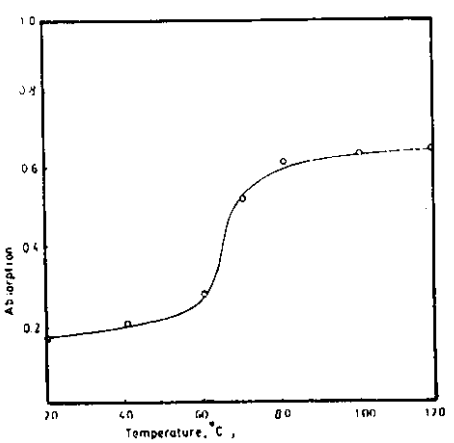


Fig. 5: Effect of temperature

Table 1: Results for Determination of Aspirin in Drug Samples

Drug	Composition indicated of drug, mg		Aspirin found by described method, mg	Error (%)
Aspirin (Bayar Pharm)	300	Aspirin	298	- 0.67
Aspirin (Glaxo Labs.)	100	Aspirin pure	100.2	+ 0.2
Dispirin (Reckit & Colman)	300	Aspirin	303	+ 1.0
Aspro (Welcome)	300	Aspirin	302	+ 0.67
Emprin compound (Welcome)	300	Aspirin	302	+ 0.67
Emprin compound (Welcome)	225 150 120	Aspirin Paracetamol Caffine	350	+ 55
Veganin (Warner, England)	250 250 6.8	Aspirin Paracetamol Codine phosphate	480	+ 92

sulphuric acid. Time factor, does not have any important role in completion of the reaction. However paracetamol, ascorbic acid and caffine interfered the determination. In absence of these interferences, the method has been found simple, rapid and adequately accurate for aspirin determination.

References

1. W.E. Juhl and R.D. Kirchhoefer, *J. Pharm. Sci.* **69**, *544 (1980).*
2. U. Amscheer, *Pharm. Ztg.*, **125**, *555 (1980).*
3. A.G. Fogg, P.J. Savsins and R. Smithson, *Anal. Chim. Acta*, **22**, *215 (1981).*
4. A. Schrocter and M. Zschiesche, *Z. Med. Laboratoriumsdign.*, **22**, *215 (1981).*
5. J.N. Buskin, R.A. Upton and R.L. Williams, *Clin. Chem.* **28**, *1200 (1982).*
6. B. Stuber and K.H. Mueller, *Pharm. Acta Helv.* **57**, *181 (1982).*
7. Vogel, A.I. A Text Book of Practical Organic Chemistry, 3rd Ed. Longmans, London, Page 996 (1957).
8. British Pharamacopia, Vol. I, H.M. Stationary Office, London Page 39 (1980)