

# A RAPID SPECTROPHOTOMETRIC METHOD FOR THE ESTIMATION OF FLUORIDE

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## ABSTRACT

*A rapid spectrophotometric method for the estimation of fluoride has been evolved making use of the bleaching action of the fluoride on yellow cerium (IV) solutions in sulphuric acid. The percentage transmission is recorded at 425 nm. By this method 0.1 mg—25 mg of fluoride present in 25 ml of the sample can be estimated. Small amounts of phosphate, chloride, nitrate and sulphate do not interfere. It is therefore possible to carry out a direct determination of fluoride without prior distillation in presence of the above ions. The interference by other ions like sulphide, sulphite, thiosulphate, carbonate and bicarbonate can be overcome by suitable treatments. This method can be employed for both micro and macro quantities of fluoride ions in solution and offers advantages over other methods currently in use.*

**Key words:** Spectrometric method, estimation of fluoride.

It is well known that fluoride bleaches the yellow colour of cerium (IV) solutions and thus interferes in the estimation if ceric ion [1]. A colorimetric method of estimating fluoride ion in aqueous solution has been standardised in the present investigation making use of this bleaching action, keeping on overall acid concentration of 2.5 N sulphuric acid.

Two instruments (1) Elico Spectrocol Colorimeter (filter) Model CL-23 range 400–700 nm and (2) SP 700 A ultraviolet and visible spectrophotometer range 185 nm–3500 nm have been employed and both give reproducible results.

The experimental details of the procedure adopted are described below. Analytically pure reagents and chemicals have been used for all the experiments.

*Reagents*

(A) *Stock Cerium (IV) Solution* [2]

A standard stock solution (one litre) of ceric sulphate (0.05 M) in 12.5 N sulphuric acid is prepared from 8.5 g of ceric oxide.

(B) *Stock Fluoride Solution*

4.42 g of sodium fluoride is dissolved in a litre of water. Each millilitre of this solution contains 2 mg of fluoride. For lower concentrations of fluoride, the solution is appropriately diluted and aliquots employed. The exact fluoride content of the solution is checked by titration at pH 3.4 against standard thorium nitrate solution using alizarin red-s as indicator [3].

*Procedure*

1. *Preparation of Calibration Chart*

Three calibration curves are plotted, representing the transmission through Ce (IV) solutions (1 ml, 2 ml and 5 ml of solution A) partially bleached by three different fluoride concentrations ranging from 0.1 mg to 1.2 mg, 1 mg to 7 mg and 4 mg to 25 mg respectively. The total volume of the solution is 25 ml and the overall acid concentration is  $2.5 \text{ N} \pm 0.5 \text{ N}$ . The percentage transmission at 425 nm is recorded for these series of samples.

2. *Determination of Fluoride in Test Sample*

In the samples which do not contain any interfering ions, an aliquot is treated with 1 ml/2 ml/5 ml of ceric solution A and the total volume made up to 25 ml. The percentage transmission is recorded and the fluoride content read from the chart.

The results are checked by willard and winter's titrimetric method after distillation. Table I gives both these results. Known amounts of interfering ions have been added to find out their influence on the analytical values.

RESULTS AND DISCUSSION

It is seen from Table I that the analytical results obtained by the two methods are in agreement with each other. It is therefore not necessary to distill the fluoride. Accurate results are obtained even in the presence

TABLE I  
*Estimation of fluoride*

Expr. No.	Fluoride taken 'mg'	Fluoride obtained by titration with Th (NO <sub>3</sub> ) <sub>4</sub> after distillation* 'mg'	Fluoride obtained by spectrophotometric method using Ce (IV) ions 'mg'
1.	3.95	3.92	3.9
2.	6.00	5.98	6.0
3.†	8.00	..	8.00
4.£	10.00	..	10.00
5.Ⓔ	8.00	..	8.0
6.§	15.00	..	15.1

\* Modified method of Willard and Winter.

† Determination carried out in presence of 75 mg of phosphate (Potassium dihydrogen phosphate) present in 25 ml of the solution.

£ Determination carried out in presence of 75 mg of phosphate (Potassium dihydrogen phosphate), 75 mg of chloride (Potassium chloride), 75 mg of nitrate (Potassium nitrate) in 25 ml of the solution.

Ⓔ Determination carried out in presence of 75 mg of carbonate (Sodium carbonate) in 25 ml of the solution.

§ Determination carried out in presence of 30 mg each of sodium sulphite, sodium sulphide and sodium thiosulphate in 25 ml of solution.

of interfering ions. If samples contain sulphite, sulphide, thiosulphate, they should be oxidised to sulphate by sodium peroxide before the determination is carried out. Carbonate or bicarbonate if present, is treated with little sulphuric acid before the addition of cerium solution. Phosphite and phosphide should be oxidised to phosphate by alkaline hypobromite and then suitably acidified. In all cases however, care should be taken to maintain the same acid concentration for the test sample and standard samples employed for calibration.

Several colorimetric methods for the estimation of fluoride ion are available [4, 5]. These methods involve elaborate procedures of preparation and separation of the sample and maintenance of rigid experimental conditions. The present method is simple, rapid and reliable.

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