

Spectrophotometric Determination of Fluoride

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The bleaching effect of fluoride ions on various coloured solutions or lakes, such as the pink zirconium-purpurin lake¹, the reddish violet zirconium-alizarin lake², or the yellowish red zirconium-quinalizarin lake³, has been made the basis of its colorimetric estimation. Various modifications of these methods have been made to carry out the estimations spectrophotometrically. Spectrophotometric determinations of fluoride ions have been effected by utilising its bleaching action on zirconyl eriochrome cyanine-R lake⁴ as well as aluminium hematoxylin lake⁵.

Fluoride ions have also been determined spectrophotometrically by making use of the fact that it prevents full development of colour of the thorium-chrome azurol-S lake⁶.

The present communication describes a spectrophotometric method for the estimation of fluoride ion, based on its bleaching action on the red solution obtained by the interaction of thorium ions and Schiff's base derived from salicylaldehyde and 1-amino-8-naphthol-3,6-disulphonic acid (monosodium salt). The maximum absorption of the thorium-reagent colour occurs at 450 m μ at pH 3.0 to 5.1, whereas the reagent shows very small absorption at this wave length.

All optical measurements were made with a Unicam SP 600 spectrophotometer. pH values were measured with a Cambridge pH-meter.

A standard fluoride solution was made by dissolving chemically pure sodium fluoride in distilled water (1 ml of the solution contained 0.002015 g. fluoride ions). The standard thorium solution was prepared by dissolving crystals of thorium nitrate (E. Merck, G.R.) in distilled water containing a few drops of nitric acid (A.R.). The thorium content was determined as ThO₂ after precipitating it as oxalate (1 ml of the solution contained 0.0003931 g. of thorium). A 0.5% solution of the reagent was made in distilled water. pH values were adjusted with KOH and HNO₃ solutions.

1. Kolthoff and Stansly, *Ind. Eng. Chem., Anal. Ed.*, 1934, 6, 118.

2. Smith and Dutcher, *ibid.*, 1934, 6, 81.

3. Thomson and Taylor, *ibid.*, 1933, 5, 87.

4. Mergregian, *Anal. Chem.*, 1954, 26, 1161.

5. Price and Walker, *ibid.*, 1952, 24, 1593.

6. Revinson and Harley, *ibid.*, 1952, 24, 794.

Procedure.—Several coloured solutions were prepared by mixing 3 ml of thorium solution and 12 ml of the reagent solution in 25-ml flasks and to these different amounts of

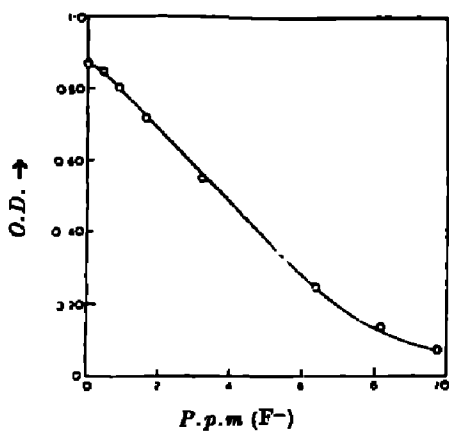


FIG. 1

the standard fluoride solution were added. The pH values were adjusted to 3.0–5.0 and the volumes made up in each case. The optical densities of these solutions were then measured at 450 m μ against the same amount of the reagent solution as blank. The calibration curve (Fig. 1) was prepared by plotting optical densities against the amounts of fluoride ions added. The resultant curve is a straight line of negative slope in the range of 0.4 to 6.4 p.p.m. of fluoride.

Ions like iron (III), vanadate, chromate, phosphate, and sulphate interfere in the above estimations.

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