

Thermo. Titr. Application Note No. H-004

Title:	Determination of Fluoride by Boric Acid Titration
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Scope:	Determination of fluoride in industrial solutions such as acid etching mixtures.
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Principle:	<p>Acidic solutions of fluoride react exothermically with boric acid according to the equations:</p> $\text{H}_3\text{BO}_3 + 3\text{H}^+ + 3\text{F}^- \leftrightarrow \text{HBF}_3(\text{OH}) + 2\text{H}_2\text{O}$ $\text{HBF}_3(\text{OH}) + \text{H}^+ + \text{F}^- \leftrightarrow \text{HBF}_4 + \text{H}_2\text{O} \quad (\text{slow})$ $\text{H}_3\text{BO}_3 + 4\text{H}^+ + 4\text{F}^- \leftrightarrow \text{HBF}_4 + 3\text{H}_2\text{O}$
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Reagents:	0.8 mol/L boric acid solution 10% w/v sulfuric acid solution
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Method:	<p>Basic Experimental Parameters:</p> <p>Titration delivery rate (mL/min.) 2</p> <p>No. of endothermic endpoints 1</p> <p>Data smoothing factor 55</p> <p>Procedure:</p> <p>A 10mL aliquot of sample solution is pipetted into a titration and 10mL of 10% w/v H₂SO₄ is added. Add D.I. water to make the volume to ~25-30mL. The solution is titrated with standard H₃BO₃ solution to a single exothermic endpoint</p>
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Results: Analysis of a sodium fluoride solution:
 Mean (n=5) = 3.75 ± 0.003% w/v

Calculation:
$$\%NaF = \frac{((\text{titre, mL} - \text{blank, mL}) \times M H_3BO_3 \times FW NaF \times 4 \times 100)}{(\text{sample vol, mL} \times 1000)}$$

Note that in this determination, the blank has a negative value, i.e. the blank must be added to rather than subtracted from the titre. This is probably due to the rounding of the endpoint, an artifact of the second reaction with fluoride being non-stoichiometric in the endpoint region due to slow reaction kinetics.

