

Thermo. Titr. Application Note No. H-004

Title:	Determination of Fluoride by Boric Acid Titration
Scope:	Determination of fluoride in industrial solutions such as acid etching mixtures.
Principle:	Acidic solutions of fluoride react exothermically with boric acid according to the equations:
	$H_3BO_3 + 3H^+ + 3F^- \leftrightarrow HBF_3(OH) + 2H_2O$
	$HBF_3(OH) + H^+ + F^- \leftrightarrow HBF_4 + H_2O$ (slow)
	$H_3BO_3 + 4H^+ + 4F^- \leftrightarrow HBF_4 + 3H_2O$
Reagents:	0.8 mol/L boric acid solution 10% w/v sulfuric acid solution
Method:	Basic Experimental Parameters:
	Titrant delivery rate (mL/min.) 2
	No. of endothermic endpoints 1
	Data smoothing factor 55
	Procedure:
	A 10mL aliquot of sample solution is pipetted into a titration and 10mL of 10% w/v H_2SO_4 is added Add D.I. water to make the volume to ~25-30mL. The solution is titrated with standard H_3BO_3 solution to a single exothermic endpoint

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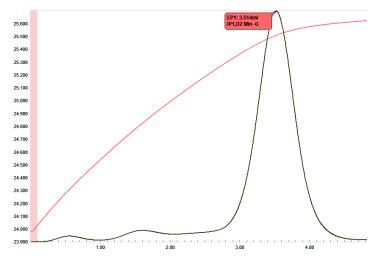
Results: Analysis of a sodium fluoride solution:

Mean $(n=5) = 3.75 \pm 0.003\%$ w/v

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

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.

Thermometric Titration Plot:



Legend:

Red = solution temperature curve

Black = second derivative curve

Note: the endpoint curvature is due to the relatively slow kinetics of the second reaction with fluoride. Nonetheless, good titration precisions can be obtained.