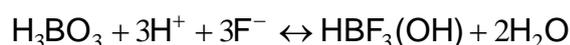


Thermo. Titr. Application Note No. H-093

Title: Determination of Boric Acid by Fluoride Titration

Scope: Determination of boric acid in electroless plating solutions.

Principle: Acidic solutions of fluoride react exothermically with boric acid according to the equations:



To form HBF_4 , boric acid will decompose metal-fluoride complexes, and thus the determination of boric acid by fluoride is not affected by the presence of metals in solution. The titration of boric acid by fluoride also permits determination of boric acid independent of other acidic substances in the solution.

Reagents: 2 mol/L potassium fluoride solution
25% v/v sulfuric acid solution

Method: *Basic Experimental Parameters:*

Titrant delivery rate (mL/min.) 6

No. of endothermic endpoints 1

Data smoothing factor 83

Stirring speed 12

Procedure:

A 5 or 10mL aliquot of electroless plating solution is pipetted into a titration vessel and 1mL of 25% v/v H_2SO_4 is added. D.I. water is added to make the total volume to 30mL (semi-auto) or ~35mL (automated operation using an 814 Sample Processor). The solution is titrated with standard KF solution to a single exothermic endpoint.

Blank determination:

A titration blank for the type of sample under examination is determined by titrating a range of aliquot sizes, and calculating the y-intercept (in mL) of a regression curve formed by plotting aliquot size (x-axis) against mL of titrant delivered (y-axis).

Standardization:

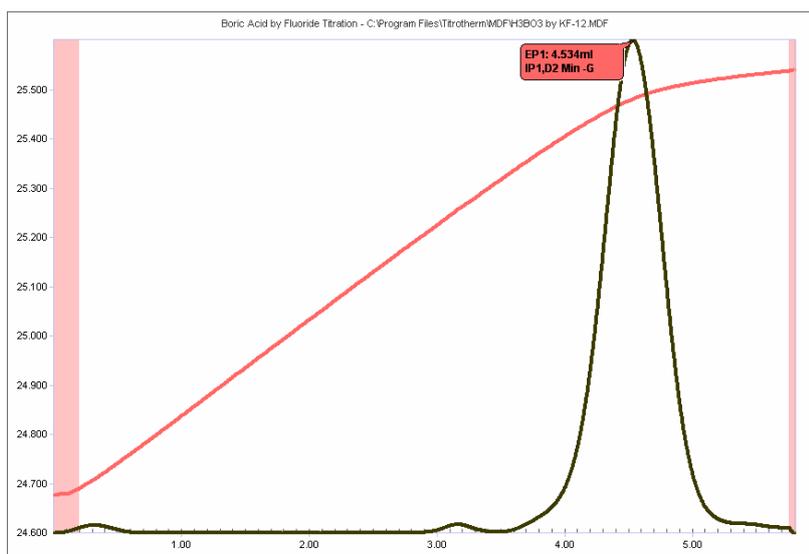
The KF titrant is standardized according to the procedure outlined in AN H-057

Results:	Sample	H₃BO₃% w/v
<i>*as reported in AN H-090</i>	Electroless nickel solution #1	1.32±0.016 (n=5) [Ni = 5.40% w/v]*
	Electroless nickel solution #2	1.34±0.010 (n=5) [Ni = 2.76% w/v]*

Calculation:

$$\%H_3BO_3 = \frac{((\text{titre, mL} - \text{blank, mL}) \times \text{KF mol/L} \times \text{FW } H_3BO_3 \times 100)}{(\text{sample vol, mL} \times 4 \times 1000)}$$

Thermometric Titration Plot:



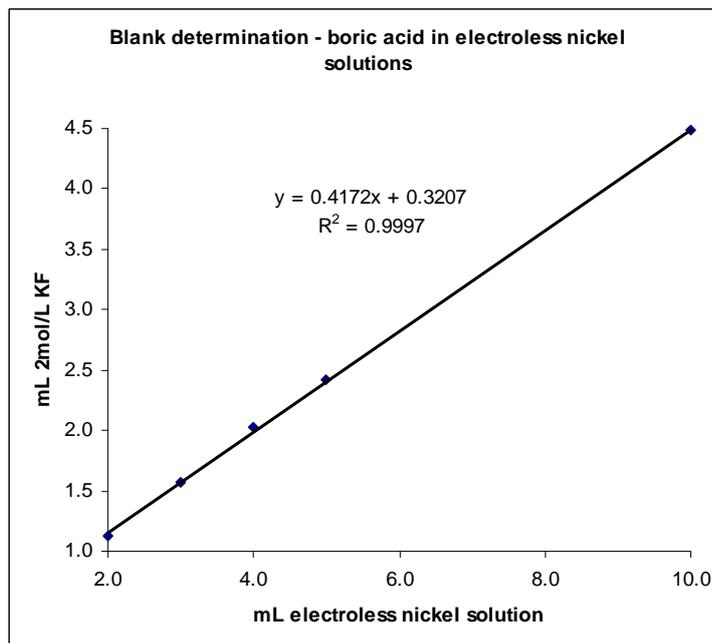
Legend:

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.

Blank determination:



Y-intercept = titration blank = 0.3207mL