

Thermo. Titr. Application Note No. H-100

Title: Determination of total acids in highly acidic etch solutions

Scope: Determination of the total acids concentration in mixtures of nitric-hydrofluoric acid intended for etching silicon substrates.

Principle: Direct thermometric titration of a weighed amount of nitric-hydrofluoric acid etch solution with standard 2 mol/L sodium hydroxide.

Reagents: *Titrant:* Standard 2 mol/L sodium hydroxide solution, standardized against potassium hydrogen phthalate. Protect titrant in Dosino with a soda-lime guard tube, and check the Dosino calibration regularly.

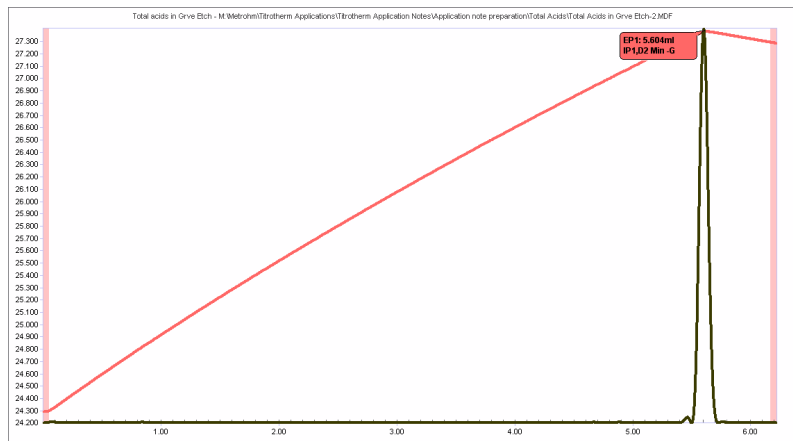
Method: Basic Experimental Parameters:

| | |
|-----------------------------------|-----|
| Titration delivery rate (mL/min.) | 2.5 |
| No. of exothermic endpoints | 1 |
| Data smoothing factor (DSF) | 55 |
| Stirring speed (802 stirrer) | 6 |

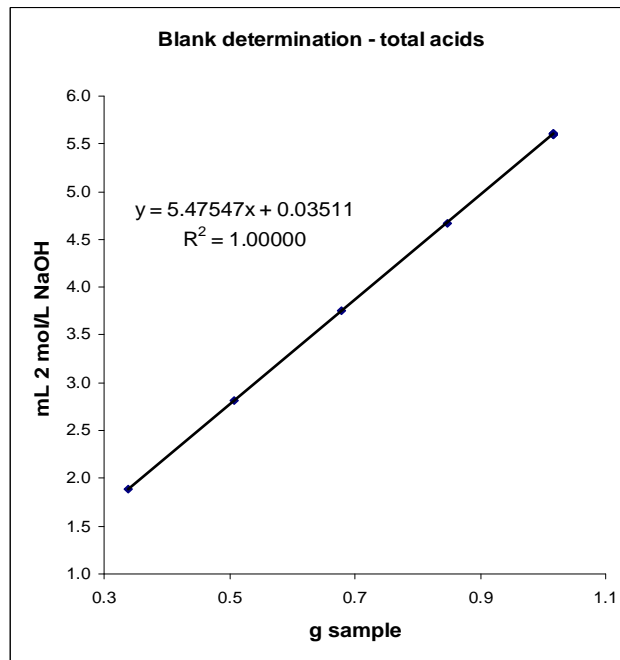
Titration: Weigh accurately ~1g of solution into a clean dry titration vessel (e.g., 6.1450.210 PFA titration vessel, 10-90mL), and add 30mL D.I. water. Titrate with 2 mol/L NaOH to a final endpoint. *Note: in the sample analyzed here. Only one endpoint was observed, probably due to the minute amount of HF present.*

Example:*Etch acid based on undiluted, concentrated 70% HNO₃*Total acids (as % w/w HNO₃) = 69.30±0.04%, n=9**Calculations:**

$$\% \text{HNO}_3 = \frac{((\text{EP, mL} - \text{blank, mL}) \times \text{NaOH mol/L} \times 63.013 \times 100)}{(\text{sample mass, g} \times 1000)}$$

Thermometric Titration Plot:**Legend:***Red = solution**temperature curve**Black =second**derivative curve (for**endpoints)***Blank determinations:**

Titration blank =
y-intercept = 0.0351 mL





Automated Titrotherm system with sample processor