

Ti Application Note No. T- 34

Title: Hydrofluoric and nitric acid in etching baths

Summary: Determination of hydrofluoric and nitric acid in etching baths by potentiometric titration.
a) Determination of the total acid content using the combined Sb electrode and NaOH as titrant.
b) Determination of hydrofluoric acid using the F ISE and $\text{La}(\text{NO}_3)_3$ as titrant.
The concentration of nitric acid is then determined by calculation.

Sample: Two different etching baths

Sample Preparation: none

Instruments and Accessories: 702, 716, 736, 751 or 785 Titrino or 796 Titroprocessor, 6.0421.100 combined Sb electrode, 6.0502.150 F ISE and 6.0726.107 reference electrode

Analysis:

a) Determination of total free acid
Pipet 1.00 mL sample into a plastic beaker and dilute with dist. water to approx. 25 mL, then titrate with $c(\text{NaOH}) = 1 \text{ mol/L}$.

The first equivalence point of the titration curve corresponds to the total free acid, the second EP to the dissolved heavy metal ions contained in the sample.

b) Determination of hydrofluoric acid
Pipet 1.00 mL sample into a plastic beaker and dilute with dist. water to approx. 40 mL. Add approx. 1 g sodium acetate, adjust the pH of the solution to 6.0 by adding NaOH and titrate with $c[\text{La}(\text{NO}_3)_3] = 0.1 \text{ mol/L}$.

Calculation:

a) $\text{mol/L total free acid} = \text{RS } 1 = \text{EP1} * \text{C01} * \text{C02} / \text{C00}$

EP1 = titrant consumption in mL
C00 = 1.00 (sample size in mL)
C01 = 1 (concentration of the titrant in mol/L)
C02 = titer of the titrant

Calculation: b) mol/L HF = RS2 = EP1 * C03 * C04 * C05 / C00

$$\text{mol/L HNO}_3 = \text{RS1} - \text{RS2}$$

EP1 = titrant consumption in mL

C00 = 1.00 (sample size in mL)

C03 = 0.1 (concentration of the titrant in mol/L)

C04 = titer of the titrant

C05 = 3 (stoichiometric factor: 1 La³⁺ reacts with 3 F⁻)

Remarks: In solutions containing an excess of HNO₃ all fluoride is present as HF.

Results:

Sample A: AVG(5) = 0.217 ± 0.005 mol/L HF

AVG(5) = 1.731 ± 0.024 mol/L HNO₃

Sample B: AVG(5) = 3.871 ± 0.029 mol/L HF

AVG(5) = 1.660 ± 0.026 mol/L HNO₃