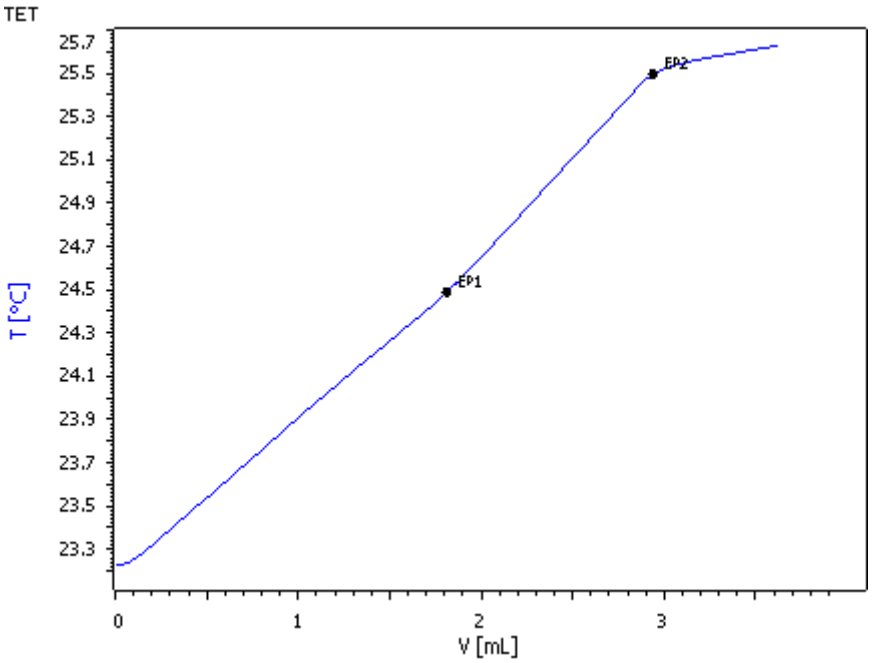
Titration Application Note H–138

Determination of nitric and hydrofluoric acid in etching baths by thermometric titration



Hydrofluoric and nitric acid can be determined in ethanol- and acetonitrile-containing etching baths by thermometric titration. In the titration curve, two different endpoints can be detected; they are used to quantify the two acids.

Metrohm

Method description

Sample

Simulated etching bath

Sample preparation

No sample preparation is required

Configuration

859 Titrotherm	2.859.1010
804 Ti Stand	2.804.0010
800 Dosino, 2x	2.800.0010
20 mL Dosing Unit	6.3032.220
50 mL Dosing Unit	6.3032.250
Thermoprobe HF	6.9011.040

Solutions

Titrant	c(NaOH) = 2 mol/L 80 g sodium hydroxide is weighed into a 1000 mL volumetric flask and filled up to the mark with deionized water
Solvent	1000 mL acetonitrile and 1000 mL ethanol are mixed in a flask.

Analysis

Blank determination

A linear regression of different sample sizes against consumption is performed. 2.0 mL, 3.0 mL, 4.0 mL, 5.0 mL and 6.0 mL sample solution is pipetted into a titration beaker and 30 mL solvent is added, respectively. The solution is titrated with c(NaOH) = 2 mol/L to one endothermic (nitric acid) and one exothermic endpoint (hydrofluoric acid).

Sample determination

The sample analysis is performed in the same way as the blank determination but without the linear regression.

Parameters

Blank / Sample determination

Stirring rate	13
Dosing rate	4 mL/min
Filter factor	65
Damping until	0.5 mL
Stop slope	0.300 °C/mL
Stop slope active after	0.5 mL
Evaluation start	0.2
EP criterion 1	55
EP criterion 2	-100
Reaction type 1	endothermic
Reaction type 2	exothermic

Results

Acid contents (n = 5)

Ratio [HNO_3:HF]	Recovery HF / %	S(rel) / %
80:20	73.0	2.34
60:40	103.6	0.60
40:60	94.7	0.81
20:80	94.6	0.82
10:90	92.4	0.61

Ratio [HNO_3:HF]	Recovery HNO_3 / %	S(rel) / %
80:20	103.6	0.32
60:40	95.4	0.53
40:60	96.1	1.16
20:80	92.4	1.86
10:90	125.2	2.73

Metrohm