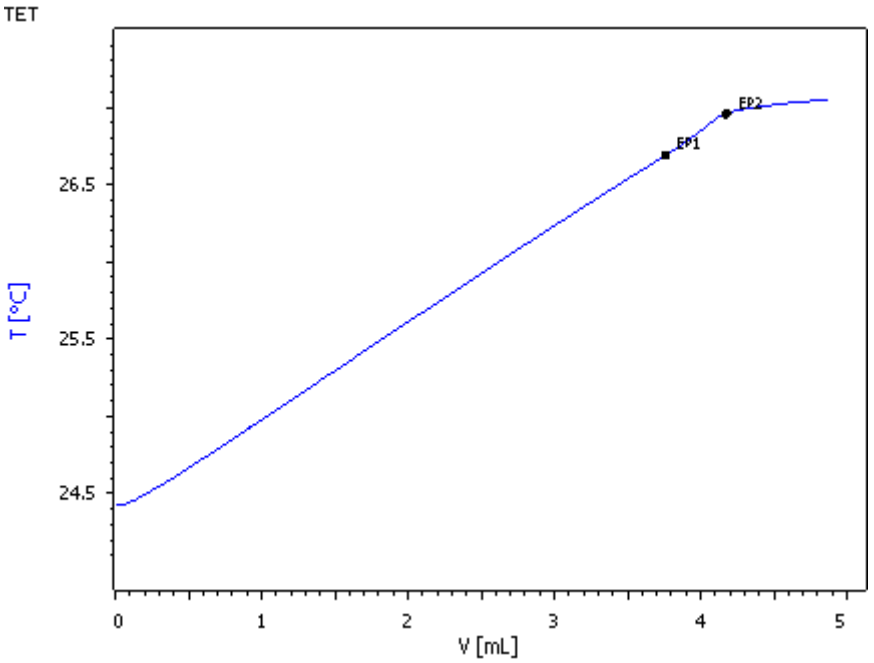
Titration Application Note H–137

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



A mix of hydrochloric and hydrofluoric 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 respective acid.

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

Solutions

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

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 (hydrochloric acid) and one exothermic (hydrofluoric acid) endpoint.

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	0.200 °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 [HCl:HF]	Recovery HCl / %	S(rel) / %	Recovery HF/ %	S(rel) / %
80:20	104.7	0.20	94.8	1.39
60:40	104.3	1.35	104.0	1.69
40:60	104.1	0.87	100.4	2.83
20:80	101.9	1.95	101.5	0.54
10:90	107.2	1.31	101.7	0.21

Metrohm