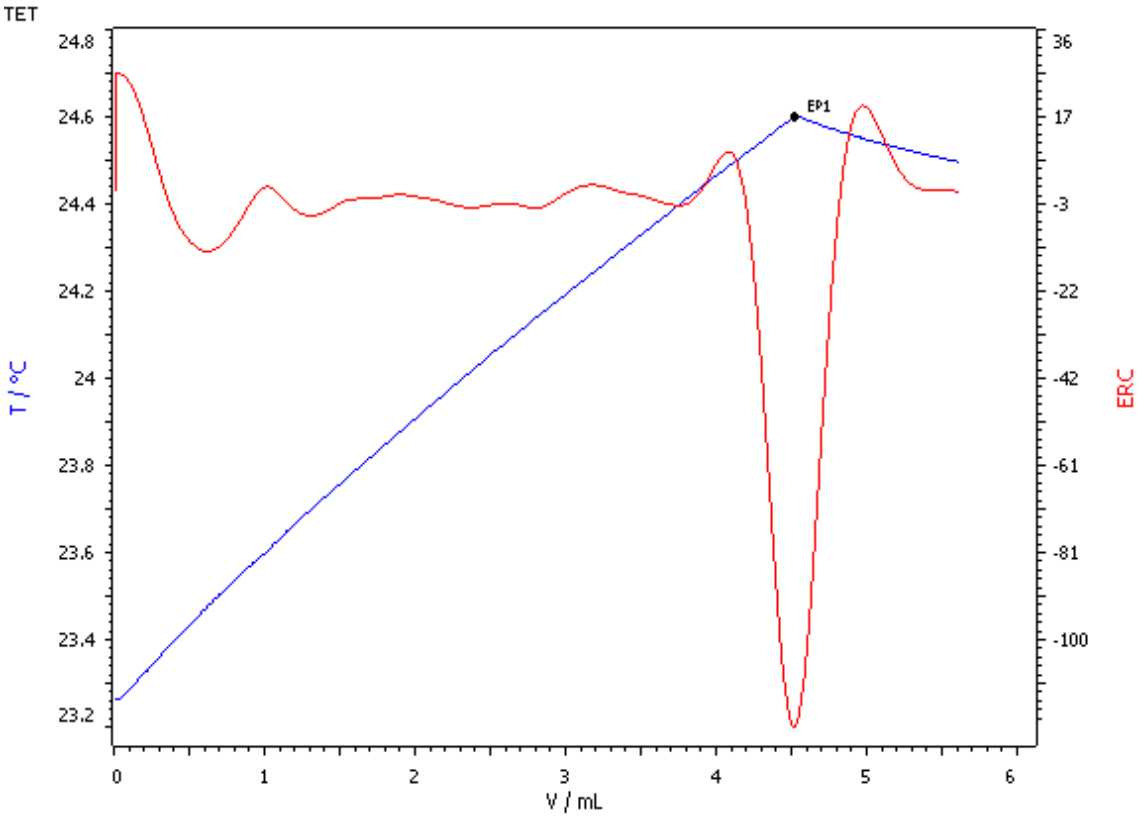
Titration Application Note H–136

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



Hydrochloric and nitric acid can be determined in etching baths by thermometric titration. In a first titration the total acid content is determined by titration with sodium hydroxide. In a second titration the hydrochloric acid content is determined by titration with silver nitrate.

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, 3x	2.800.0010
10 mL Dosing Unit, 2x	6.3032.210
50 mL Dosing Unit	6.3032.250
5 mL Dosing Unit	6.3032.150

Solutions

Titrant 1	$c(AgNO_3)$ = 1 mol/L 84.935 g silver nitrate is weighed into a 500 mL volumetric flask and filled up with to the mark with deionized water.
Titrant 2	c(NaOH) = 2 mol/L 80 g sodium hydroxide is weighed into a volumetric flask and filled up with deionized water to 1000 mL.
Nitric acid, $c(HNO_3)$ = 4 mol/L	387.75 g of concentrated nitric acid is weighed into a volumetric flask and filled up with deionized water to 1000 mL.

Analysis

Blank determination

A linear regression of different sample sizes against consumption is performed. 1.5 mL, 2.0 mL, 2.25 mL sample solution, respectively is pipetted into a titration beaker and 30 mL deion. H_2O is added. At first the solution is titrated with c(NaOH) = 2 mol/L to a single exothermic endpoint (determination of total acid content). To acidify the sample the same volume of $c(HNO_3)$ = 4 mol/L like the total added volume of c(NaOH) = 2 mol/L is added to the mixture. The content of hydrochloric acid is then determined by titration with $c(AgNO_3)$ = 1 mol/L to a single exothermic 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 hydrochloric acid

Stirring rate	10
Dosing rate	4 mL/min
Filter factor	60
Damping until	0.0 mL
Stop slope	-0.060 °C/mL
Stop slope active after	0.5 mL
Evaluation start	0.0 mL
EP criterion	-50
Reaction type	Exothermic

Blank / Sample determination nitric acid

Stirring rate	10
Dosing rate	4 mL/min
Filter factor	60
Damping until	1.5 mL
Stop slope	-0.060 °C/mL
Stop slope active after	0.5 mL
Evaluation start	1.5 mL
EP criterion	-160
Reaction type	Exothermic

Results

Acid contents (n = 5)

Ratio [HCl:HNO_3]	Recovery HCl / %	S(rel) / %
90:10	102.7	0.22
80:20	102.7	0.17
60:40	102.2	0.16
40:60	103.0	0.23
20:80	104.6	2.56
10:90	106.3	0.91

Ratio [HCl:HNO_3]	Recovery HNO_3 / %	S(rel) / %
90:10	97.4	2.46
80:20	96.3	1.43

Method description

60:40	104.8	2.40
40:60	104.1	0.38
20:80	103.1	0.75
10:90	102.9	0.15

Metrohm