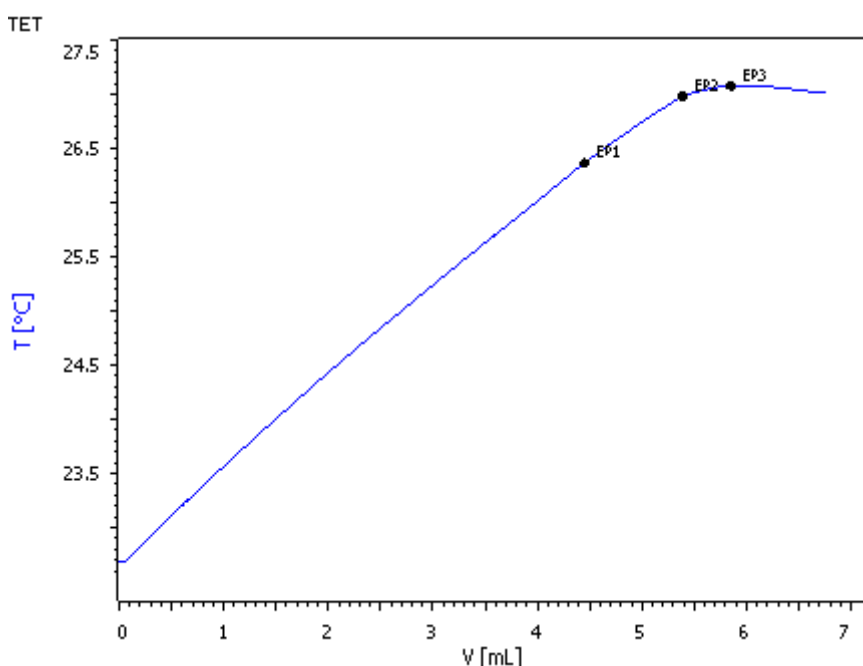


Determination of nitric, phosphoric, and acetic acid in etching baths by thermometric titration



Nitric, phosphoric, and acetic acid can be determined by thermometric titration. In the titration curve, three endpoints can be detected: each one corresponds to one of the acids and is used for quantification.

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) = 3 mol/L 240 g sodium hydroxide is weighed into a 2000 mL volumetric flask and filled up to the mark with deionized water
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Analysis

Blank determination

A linear regression of different sample sizes against consumption is performed. 4.0 mL, 5.0 mL, 6.0 mL, 6.5 mL and 7.0 mL sample solution is pipetted into a titration beaker and 30 mL deion. H₂O is added, respectively. The solution is titrated with c(NaOH) = 3 mol/L to the third 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

Stirring rate	14
Dosing rate	5 mL/min
Filter factor	60
Damping until	0.5 mL for blank
Stop slope	0.050 °C/mL
Stop slope active after	0.5 mL
Evaluation start	0.5 mL for blank

EP criterion 1	-12
EP criterion 2	-30
EP criterion 3	-18
Reaction type	exothermic

Results

Acid contents (n = 5)

Ratio HNO ₃ :H ₃ PO ₄ :HAc	Recovery HNO ₃ / %	S(rel) / %
80:10:10	98.00	0.20
70:15:15	91.79	0.68
60:20:20	96.42	0.87
50:25:25	104.69	0.78
40:30:30	104.72	1.83
30:35:35	95.75	5.45
20:40:40	104.12	8.43

Ratio HNO ₃ :H ₃ PO ₄ :HAc	Recovery H ₃ PO ₄ / %	S(rel) / %
80:10:10	78.22	3.56
70:15:15	100.14	0.97
60:20:20	106.47	0.51
50:25:25	96.13	0.64
40:30:30	94.94	0.51
30:35:35	94.06	0.79
20:40:40	85.20	0.67

Ratio HNO ₃ :H ₃ PO ₄ :HAc	Recovery HAc / %	S(rel) / %
80:10:10	75.46	3.80
70:15:15	98.37	1.02
60:20:20	102.07	0.51
50:25:25	91.40	0.71
40:30:30	95.06	0.53
30:35:35	92.89	0.83
20:40:40	85.02	0.69