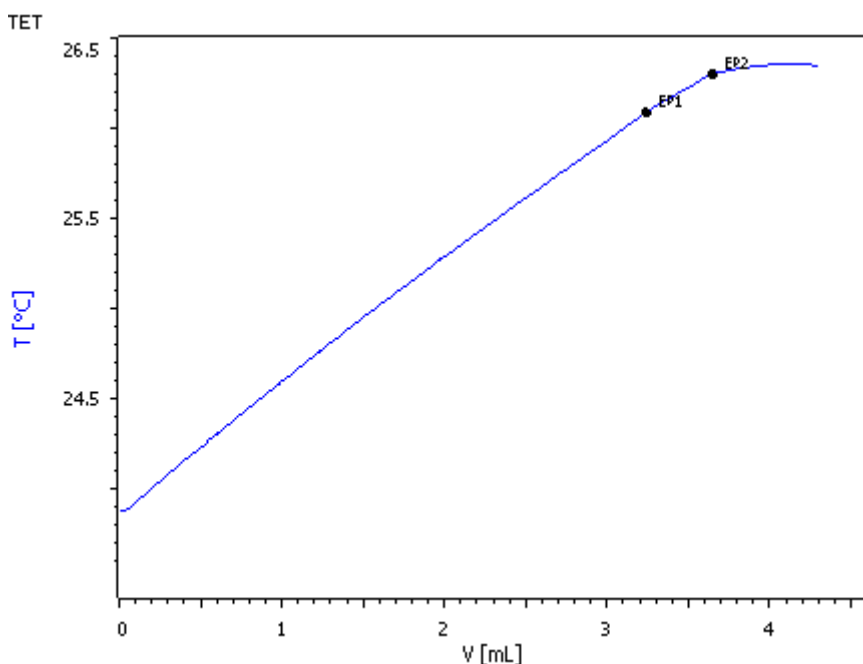


Determination of hydrochloric and phosphoric acid in etching baths by thermometric titration



Varying concentrations of hydrochloric and phosphoric acid can be analyzed by thermometric titration. In the titration curve, two different endpoints can be detected; they are used to determine each acid.

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
10 mL Dosing Unit, 2x	6.3032.210
50 mL Dosing Unit	6.3032.250
Thermoprobe	6.9011.020

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
---------	--

Analysis

Blank determination

A linear regression of different sample sizes against titrant-consumption is performed. 1.5 mL, 2.0 mL, 2.5 mL, 3.0 mL and 3.5 mL sample solution is pipetted into a titration beaker and 30 mL deion. H₂O is added, respectively. The solution is titrated with c(NaOH) = 2 mol/L to the second 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	13
Dosing rate	4 mL/min
Filter factor	40
Damping until	1.0 mL for blank 2.0 mL for sample
Stop slope	0.200 °C/mL
Stop slope active after	0.5 mL

Evaluation start	1.0 mL for blank 2.0 mL for sample
EP criterion 1	-40
EP criterion 2	-60
Reaction type	exothermic

Results

Acid contents (n = 5)

Ratio [HCl:H ₃ PO ₄]	Recovery HCl / %	S(rel) / %
80:20	96.4	1.54
60:40	96.6	2.07
40:60	99.4	2.92
20:80	101.6	5.68

Ratio [HCl:H ₃ PO ₄]	Recovery H ₃ PO ₄ / %	S(rel) / %
80:20	106.4	2.73
60:40	101.6	1.20
40:60	102.5	0.87
20:80	103.4	0.56