Titration Application Note H–134

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



Sulfuric and phosphoric acid can be determined in acid mixtures by thermometric titration. Two different endpoints are detected and they can be used to quantify each present acid.



Method description

Sample

Simulated etching bath

Sample preparation

No sample preparation is required

Evaluation start	1.5 mL
EP criterion 1	-15
EP criterion 2	-15
Reaction type	exothermic

Results

Acid contents (n = 5)

Configuration

•	
859 Titrotherm	2.859.1010
804 Ti Stand	2.804.0010
800 Dosino, 2x	2.800.0010
50 mL Dosing Unit	6.3032.250
20 mL Dosing Unit	6.3022.220
Thermoprobe	6.9011.020

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.

80:20 103.8 0.37
60:40 103.7 1.84
40:60 106.2 3.11
20:80 95.3 4.92
10:90 104.2 9.7

Ratio $[H_2SO_4:H_3PO_4]$	Recovery H ₃ PO ₄ / %	S(rel) / %
80:20	120.2	0.58
60:40	103.1	1.98
40:60	103.6	1.23
20:80	102.2	0.65
10:90	101.9	0.68

Analysis

Blank determination

A linear regression of different sample sizes against consumption is performed. 1.0, 2.0, 3.0, 4.0 and 5.0 mL sample solution is pipetted into a titration beaker and 30 mL deion. H_2O 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 like the blank determination but without the linear regression.

Parameters

Blank / Sample determination

Stirring rate	13
Dosing rate	2 mL/min
Filter factor	65
Damping until	0.5 mL
Stop slope	0.080 °C/mL
Stop slope active after	0.5 mL

