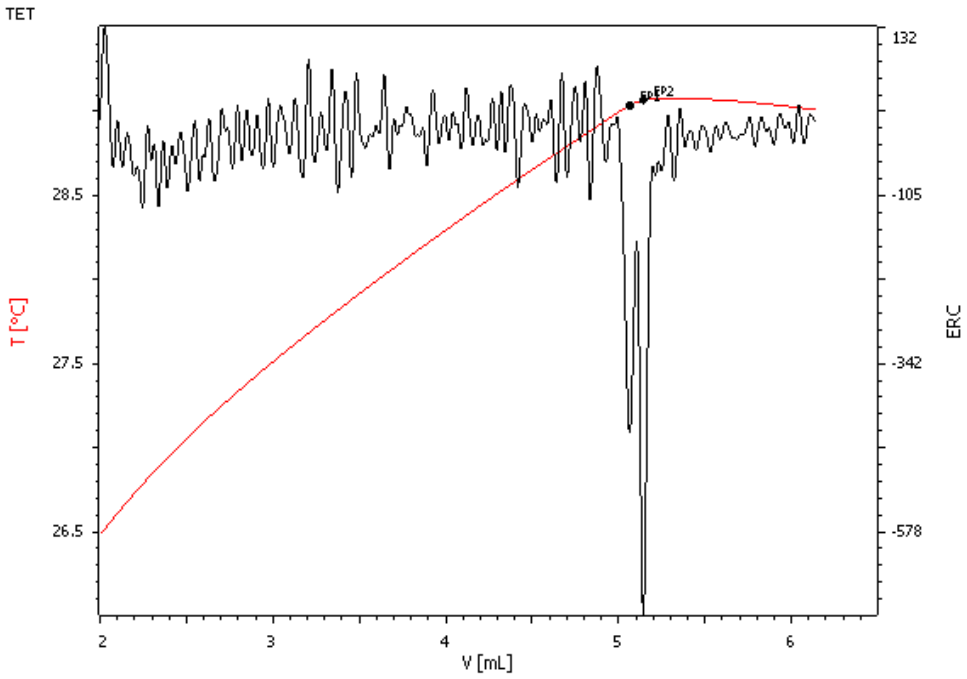
Titration Application Note H-099

# Determination of potassium hydroxide and silica in alkaline etch solution




This Application Note describes a direct thermometric endpoint titration (TET) for the determination of concentrated potassium hydroxide solutions which had been used for the etching of substrates containing silicon.

Metrohm

# Method description

### Sample

High concentrated potassium hydroxide solution which contains a small amounts of $SiO_2$ generated during the etch process of silicon.

### Sample preparation

No sample preparation is necessary.

### Configuration

| | |
|---|---|
| 859 Titrotherm | 2.859.1010 |
| HF Thermoprobe | 6.9011.040 |
| 804 Ti Stand | 2.804.0040 |
| 802 Propeller stirrer | 2.802.0040 |
| Dosing unit 10 mL | 6.3032.210 |
| 800 Dosino | 2.800.0010 |
| PFA titration vessel | 6.1450.210 |
| Mounting ring | 6.2036.000 |
| Titration vessel lid | 6.1414.010 |

### Solutions

| | |
|---|---|
| Titrant | c(HCl) = 2 mol/L |

### Analysis of samples

Approx. 2–2.5 g sample is weighed accurately into a clean dry PFA titration vessel and 30 mL deionized water is added. The solution is titrated with c(HCl)=2 mol/L to a second exothermic endpoint.

### Determination of the method blank

For both KOH and $SiO_2$ a linear regression of different sample sizes against titrant consumption is performed. Therefore approx. 1–3 g sample is weighed accurately into a PFA titration vessel, diluted with 30 mL deionized water and titrated to a second exothermic endpoint. For the linear regression of KOH the consumption to the first endpoint is used. For the linear regression of $SiO_2$ the consumption corresponds to the volume difference between the second and the first endpoint.

### Parameters

| | |
|---|---|
| Titrant dose rate | 1 mL/min |
| No. of exothermic endpoints | 2 |
| ERC EP1 (exothermic) | -350 |
| ERC EP2 (exothermic) | -350 |
| Damping until | 0.2 mL |
| Evaluation start | 2 mL |
| Filter factor | 15 |
| Stirring speed | 8 |

### Result

| %KOH (n = 5) | $s_{rel}$ (%) |
|---|---|
| 30.00 | 0.79 |
| %$SiO_2$ (n = 5) | $s_{rel}$ (%) |
| 0.12 | 2.32 |