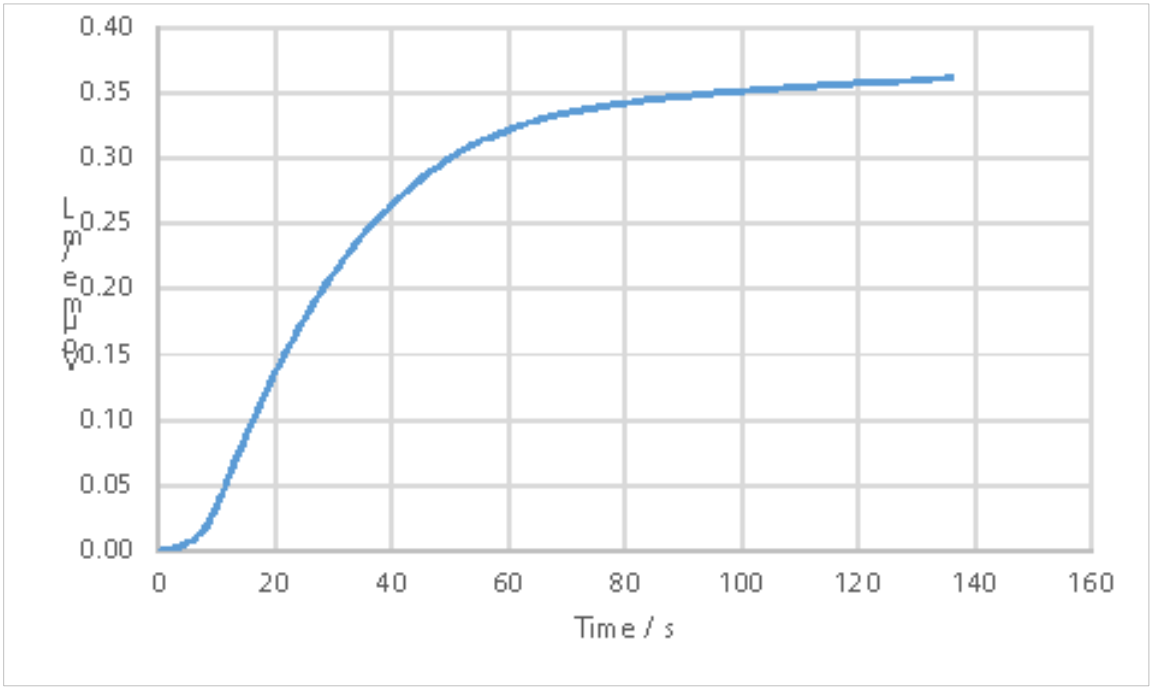
Titration Application Note T-197

Surface glass test according to USP<660>



The surface glass test for hydrolytic resistance combined with the glass grains test determines the glass type. The hydrolytic resistance is determined by the quantity of alkali released from the glass under specified conditions. The released alkali is then titrated with hydrochloric acid to a pH value of 5.5.

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Method description

Sample

Vials, glass type I

Sample preparation

The cleaned containers are filled with carbon dioxide-free water up to the filling volume and placed on the tray of the autoclave. The tray is placed in an autoclave containing a quantity of water such that the tray remains clear of the water. The autoclave with open vent-cock is heated up at a regular rate such that steam is issued vigorously from the vent-cock after 20–30 min, and a vigorous evolution of steam is maintained for a further 10 min. The vent-cock is then closed and the temperature is raised from 100 °C to 121 °C at a rate of 1 °C/min within 20–22 min. The temperature is maintained at 121 ± 1° for 60 ± 1 min from the time when the holding temperature is reached. Afterwards, the autoclave is cooled down to 100 °C at a rate of 0.5 °C/min within 40–44 min, to prevent formation of a vacuum the autoclave is vented during this time. After the temperature in the autoclave reached 95 °C the hot samples are removed from the autoclave and continuously cooled down to room temperature within 30 min, while avoiding thermal shock.

Configuration

905 Titrando	2.905.0010
801 Magnetic stirrer	2.801.0040
800 Dosino	2.800.0010
Dosing unit 5 mL	6.3032.150
Electrode Cable 1 m / U	6.2104.600
Aquatrode plus with Pt1000	6.0257.600

Solutions

Titrant	c(HCl) = 0.01 mol/L 11.901 g dried KBr is weighed in a 1 L volumetric flask and dissolved in dist. water and the flask is filled up to the mark with dist. water.

Analysis

The titration is carried out within 1 h of the removal of the containers from the autoclave.

The liquids obtained from the containers are combined, and mixed. An aliquot (see table below) of the sample is transferred into a conical flask. The solution is then titrated with c(HCl) = 0.01 mol/L to a pH value of 5.5.

For the blank is determined the same volume of carbon dioxide-free water is transferred into a second conical flask and then titrated with c(HCl) = 0.01 mol/L to a pH value of 5.5 as well.

Aliquot volume in dependence on the filling volume of the vials tested

Filling volume vial / mL	Aliquot volume / mL
<3	25.0
3–30	50.0
30–100	100.0
>100	100.0

Parameters

Titration mode	SET pH
Stirring rate	8
Pause	0 s
End point 1 at pH	5.5
Dynamics	2
Max. dosing rate	1 mL/min
Min. dosing rate	1 µL/min
Stop criterion	Drift
Stop drift	20 µL/min
Titration direction	Negative
Extraction time	0 s

Results

Consumed mL c(HCl)) 0.01 mol/L per 100 mL test solution, (n = 2)	s(abs)	s(rel) / %
0.71 mL	0.02 mL	2.69

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