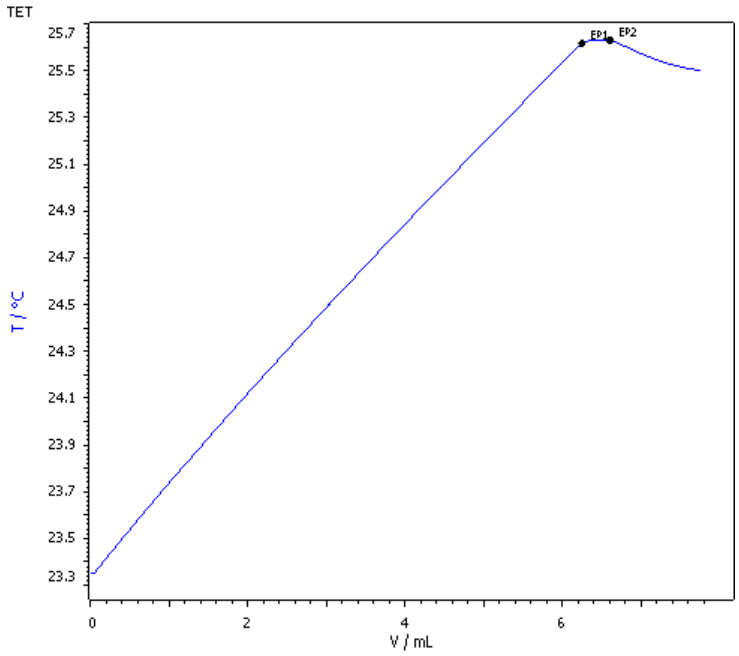
Titration Application Note H-026

Determination of caustic, soda, carbonate and alumina in a spent Bayer Process liquor



This Application Note describes a method based on procedures developed by Watts-Utley and VanDalen-Ward for the analysis of caustic, soda, carbonate and alumina in Bayer Process liquors.

Metrohm

Method description

Sample

"Spent" Bayer Process liquor

Sample preparation

25 mL of the spent liquor is pipetted into a 500 mL volumetric flask, and made to volume with deionized water. The pipette was allowed to drain for 10 minutes due to the viscosity of process liquors.

Configuration

859 Titrotherm incl. 804 Titration stand 802 Rod stirrer 800 Dosing, 1x 10 mL Dosing unit, 1x Thermoprobe	2.859.1010
800 Dosino, 3x	2.800.0010
10 mL Dosing unit	6.3032.210
50 mL Dosing unit	6.3032.250

Solutions

Titrant	c(HCl) = 1.5 mol/L 147.8 g conc. HCl is weighed into a 1 L volumetric flask containing 500 mL deion. H_2O. After cooling down to room temperature, the flask is filled up to the mark with deion. H_2O.
Complexing solution 1	Nearly saturated solution of potassium sodium tartrate, $\beta(KNaC_4H_4O_6)$ = approx. 614 g/L. This highly concentrated complexing solution has been found to be quite stable when stored in conditions above 22 °C, however, if stored at lower temperatures it should be periodically checked for signs of crystallization.
Complexing solution 2	Nearly saturated solution of potassium fluoride, $\beta(KF)$ = approx. 620 g/L. This highly concentrated complexing solution has been found to be quite stable when stored in conditions above 22 °C, however, if stored at lower temperatures it should be periodically checked for signs of crystallization.

Analysis

Blank determination

A linear regression of different sample sizes against consumption is performed. For this purpose aliquots of 30, 25, 20 and 15 mL of the diluted solution are pipetted into titration vessels and made up to approximately 30 mL with deionized water. 10 mL complexing solution 1 (potassium potassium sodium tartrate) is added. After a pause of 10 s the solution is titrated with c(HCl) = 1 mol/L until after the second exothermic endpoint.

To the titrated solution 10 mL of complexing solution 2 (KF) is added and after another pause of 20 s the solution is titrated again with c(HCl) = 1 mol/L until after the first exothermic endpoint.

Each aliquot should be prepared only a few minutes prior to titration, due to risk of absorption of atmospheric CO_2.

Sample determination

The sample analysis is performed in the same way as the blank determination but omitting the linear regression.

Parameters

Caustic and soda determination

Stirring rate	12
Dosing rate	4 mL/min
Filter factor	40
Damping until	1 mL
Stop slope	<-0.12 °C/mL
Stop slope active after	1.0 mL
Additional volume after stop	1.0 mL
Evaluation start	1 mL
Reaction type 1	Exothermic
EP criterion 1	-70
Reaction type 2	Exothermic
EP criterion 2	-50

Method description

Alumina determination

Stirring rate	15
Dosing rate	4 mL/min
Filter factor	35
Damping until	1 mL
Stop slope	<-0.05 °C/mL
Stop slope active after	1.0 mL
Additional volume after stop	0.5 mL
Evaluation start	1 mL
Reaction type 1	Exothermic
EP criterion 1	-20

Results

Mean contents per American convention (n = 10)

	Mean	s(rel) / %
Caustic / (g/L)	340.2	0.06
Soda / (g/L)	353.0	0.10
Carbonate / (g/L)	12.6	2.80
Alumina / (g/L)	97.8	0.16