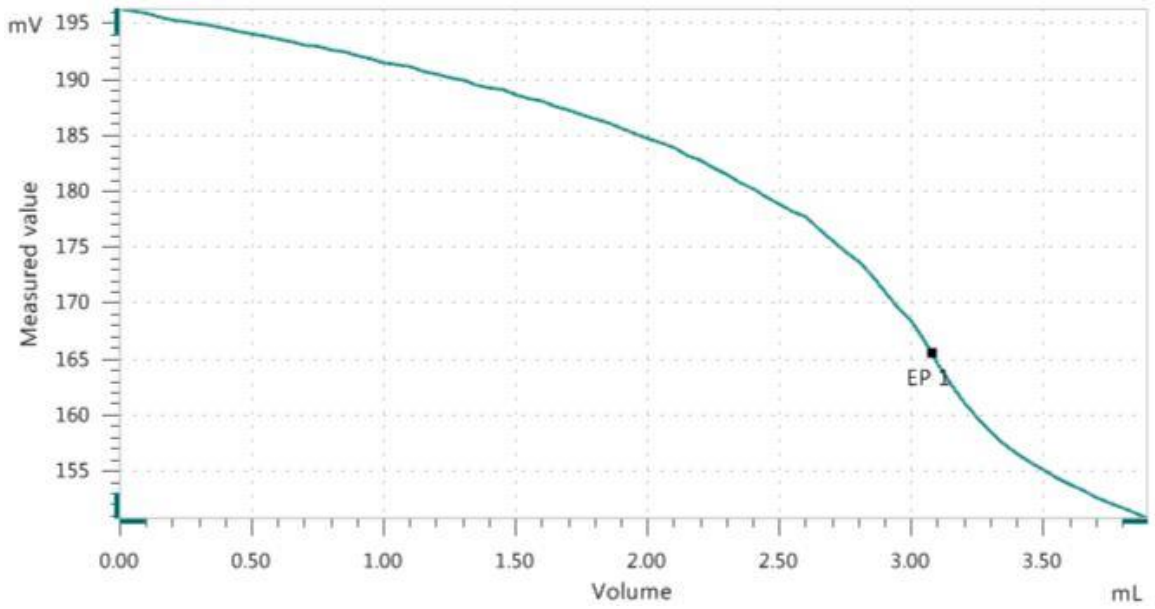
Titration Application Note T-170

Determination of nitrilotriacetates in detergents according to ASTM D4954

Nitrilotriacetate (NTA) is a chelating agent and is used in detergents as water



softener. It forms complexes with metal ions such as Ca^{2+}, Cu^{2+}, and Fe^{3+} and prevents, among others, the formation of limestone and calcification. The NTA content is therefore an important parameter for the quality of detergents and is determined by back-titration of an excess of cupric nitrate.

Metrohm

Method description

Sample

Detergent powder spiked with 6 % NTA.

Sample preparation

The detergent is dissolved in deionized water and the pH is adjusted 7.0 with conc. HNO_3.

Configuration

Main module Pick&Place S	2.1010.0010
Pick&Place module	2.1014.0010
"Peristaltic" (2-channel) pump module	2.1016.0010
Gripper fingers 42.8 - 65 mm	6.02601.010
Dummy panel for module plate	6.02600.000
OMNIS Rod Stirrer "Sample Robot"	2.1006.0010
Titration head 6xNS14 / 3xNS9 (P&P)	6.01403.000
Stirring propeller 30 mm ETFE	6.01900.010
OMNIS sample rack 9 x 250 mL, 2x	6.02041.010
Sample beaker (10x) PP 250 mL (P&P), 2x	6.01400.100
OMNIS Titrator (Advanced)	2.1001.0210
Cable MDL PL/SO 1 m, 4x	6.02102.020
OMNIS Dosing Module, 4x	2.1003.0010
OMNIS 5 mL cylinder unit	6.03001.150
OMNIS 10 mL cylinder unit	6.03001.210
OMNIS 20 mL cylinder unit	6.03001.220
OMNIS 50 mL cylinder unit, 3x	6.03001.250
Analog measuring module	6.02101.010
Cu-ISE	6.0502.140
Unitrode with Pt1000 (Head U)	6.0258.600
Electrode cable plug-in head G / plug P, 1.5 m for Cu-ISE	6.02104.010
Electrode cable plug-in head U / plug P, 1.5 m for Unitrode	6.02104.610
OMNIS Stand-alone license (including one instrument license)	6.06003.010
OMNIS instrument license, 1x	6.06002.010

Solutions

Titrant	c(NTA) = 0.3 mol/L
Cupric nitrate solution	$c(Cu(NO_3)_2)$ = 0.1 mol/L
Acetate buffer	Acetate buffer with pH = 4.65
EDTA solution	$c(Na_2EDTA)$ = 0.1 mol/L
HNO_3	$w(HNO_3)$ = 65 %
NaOH	w(NaOH) = 20 %

Analysis

100 mL prepared sample is pipetted into the sample beaker and placed on the rack. While stirring 3 mL conc. HNO_3 is added. The sample is then left to settle for 5 min. The addition of conc. HNO_3 is continued until a pH of 0 is reached. After the addition of 25 mL $c(Cu(NO_3)_2)$ = 0.1 mol/L, the pH is adjusted to approximately 1.05 with w(NaOH) = 20%. Afterwards 25 mL acetate buffer is added and the pH is adjusted to approximately 4.65 with w(NaOH) = 20%. Then the solution is titrated with c(NTA) = 0.3 mol/L until after the equivalence point.

Parameters

Mode	MET U
Pause	30 s
Volume increment	0.05 mL
Stirring rate	8
Signal drift	30 mV/min
Max. waiting time	32 s
Min. waiting time	5 s
Dosing rate	Maximum
Stop volume	10 mL
Stop EP	1
Volume after EP	1.0 mL
EP criterion	5 mV
EP recognition	Greatest

Results

Content w_{NTA} / (%) (n = 5)	s(rel) / %
8.41	0.49