
Application Bulletin

Of interest to: General analytical laboratories; Detergents

A 3, 12

Potentiometric determination of nitrilotriacetic acid (NTA) and/or ethylenediaminetetraacetic acid (EDTA) in washing agents

Summary

Application Bulletin No. 76 describes the polarographic determination of small concentrations (1 ... 100 mg/L) of NTA and EDTA in water. Since in certain countries legislation has been introduced requiring that phosphates in washing agents should be replaced NTA and EDTA have become more and more important as complexing agents and builders.

This bulletin describes the determination of higher concentrations of NTA and/or EDTA in washing agents by means of potentiometric titration. The ion-selective copper electrode is used as indicator electrode. Other components normally found in washing agents do not interfere with this determination.

Instruments and accessories

- 702 SET/MET Titrino, 716 DMS Titrino, 736 GP Titrino, 751 GPD Titrino or 785 DMP Titrino or 726 or 796 Titroprocessor with 700 Dosino or 685 Dosimat
 - 2.728.0040 Magnetic Stirrer
 - 6.3014.213 or 6.3014.223 Exchange Unit
 - 6.0502.140 ion-selective copper electrode (Cu ISE) with 6.2104.020 electrode cable
 - 6.0726.100 double-junction Ag/AgCl reference electrode (bridge electrolyte KNO₃ sat.) with 6.2106.020 electrode cable
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Reagents

- Titrant: $c(\text{Cu}^{2+}) = 0.01 \text{ mol/L}$:
Dissolve 2.416 g $\text{Cu}(\text{NO}_3)_2 \cdot 3 \text{H}_2\text{O}$ in dist. water, add 0.5 mL conc. HNO_3 and make up to 1 L with dist. water.
- NTA standard solution, $c(\text{NTA}) = 0.01 \text{ mol/L}$:
Make a slurry with 1.9114 g NTA and dist. water. While stirring, add $w(\text{NaOH}) = 32\%$ drop by drop until the solution is clear, then make up to 1 L with dist. water.

- EDTA standard solution, $c(\text{EDTA}) = 0.01 \text{ mol/L}$:
Make a slurry with 2.9225 g EDTA and dist. water. While stirring, add $w(\text{NaOH}) = 32\%$ drop by drop until the solution is clear, then make up to 1 L with dist. water.
- Buffer solution $\text{pH} = 9.6$:
Dissolve 80 g NH_4NO_3 in dist. water, add 70 mL $w(\text{NH}_3) = 25\%$ and make up to 1 L with dist. water.

Analysis

- Weigh 0.5 ... 1 g sample into a 100 mL volumetric flask, add 50 mL dist. water and dissolve as well as possible by heating lightly up to approx. 40 °C. After cooling down, carefully fill to the mark with dist. water and mix.
- Pipet 10.0 mL of the mixture (corresponding to 50 ... 100 mg of the original sample) into a glass beaker and add 2.0 mL $c(\text{NTA}) = 0.01 \text{ mol/L}$ or $c(\text{EDTA}) = 0.01 \text{ mol/L}$. After addition of 10 mL buffer solution $\text{pH} = 9.6$ titrate with $c(\text{Cu}^{2+}) = 0.01 \text{ mol/L}$.

Calculation

1 mL $c(\text{Cu}^{2+}) = 0.01 \text{ mol/L}$ corresponds to 1.9114 mg NTA or 2.9225 mg EDTA

$$\% \text{ NTA} = (\text{EP1} - \text{C31}) * \text{C01} * \text{C03} / \text{C00}$$

$$\% \text{ EDTA} = (\text{EP1} - \text{C31}) * \text{C02} * \text{C03} / \text{C00}$$

EP1 = titrant consumption in mL

C00 = 50 ... 100 (sample mass used in mg original sample)

C01 = 1.9114

C02 = 2.9225

C03 = 100 (conversion factor for %)

C31 = 2.0 [added volume of $c(\text{NTA}) = 0.01 \text{ mol/L}$ or $c(\text{EDTA}) = 0.01 \text{ mol/L}$ in mL]

Remarks

- Mixtures of NTA and EDTA are usually determined as a whole. Separation of the two components is only possible if the mixture ratio is most favorable. In that case EDTA is determined first.
- If separate determination of the individual components is required and the mixture ratio is unfavorable, please refer to Application Bulletin No. 76.

```

'pa
785 DMP Titrino      02287  785.0010
user
date 1999-05-11    time 14:02      9
DET U
parameters
>titration parameters
  meas.pt.density      4
  min.incr.            10.0 µl
  dos.rate             max. ml/min
  signal drift         50 mV/min
  equilibr.time        26 s
  start V:             OFF
  pause                0 s
  meas.input:          1
  temperature          25.0 °C
>stop conditions
  stop V:              abs.
  stop V               20 ml
  stop U               OFF mV
  stop EP              9
  filling rate         max. ml/min
>statistics
  status:              OFF
>evaluation
  EPC                  5
  EP recognition:      all
  fix EP1 at U        OFF mV
  pK/HNP:              OFF
>preselections
  req.ident:           OFF
  req.smpl size:       OFF
  limit smpl size:    OFF
  activate pulse:     OFF
=====
    
```

Fig. 1: Parameter settings on the 785 DMP Titrino.

```

'fr
785 DMP Titrino      02287  785.0010
user
date 1999-05-11    time 13:59      9
U(init)            -239 mV DET U
smpl size           88.3 mg
EP1                 5.582 ml          -165 mV
NTA                  1.26 %
stop V reached
=====
    
```

```

'cu
785 DMP Titrino      02287  785.0010
user
date 1999-05-11    time 14:00      9
start V             0.000 ml DET U
5.0 ml/div          dU=50.0 mV/div
    
```

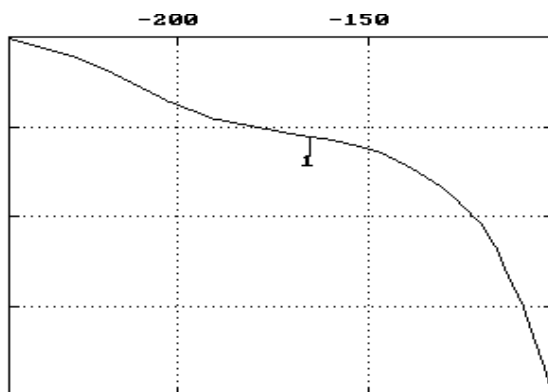


Fig. 2: Result block and titration curve for the determination of NTA in a washing powder.

```
'fr
785 DMP Titrino      02287  785.0010
user
date 1999-05-11    time 13:38      5
U(init)           -240 mV DET U
EP1                4.902 ml      -141 mV
EDTA               2.98 %
manual stop
=====
```

```
'cu
785 DMP Titrino      02287  785.0010
user
date 1999-05-11    time 13:38      5
start V           0.000 ml DET U
5.0 ml/div        dU=50.0 mV/div
```

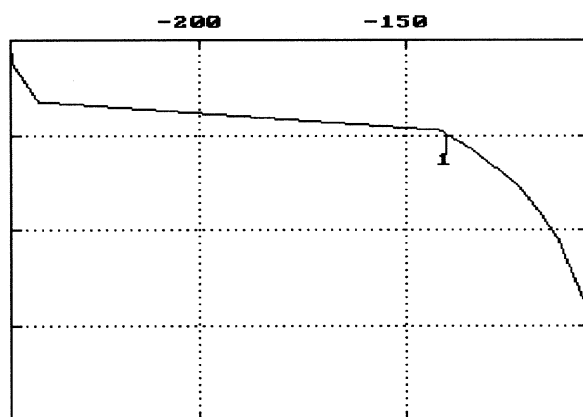


Fig. 3: Result block and titration curve for the determination of EDTA in a washing powder.

Literature

- G. A. Rechnitz, N. C. Kenny
Determination of nitrilotriacetic acid (NTA) by using ion-selective membrane electrodes
Anal. Lett. 3 (1970) 509–514
Ref.: Fresenius Z. Anal. Chem. 255 (1971) 144.
- M. Taddia, M. T. Lippolis, L. Patorelli
Potentiometric determination of EDTA and NTA in detergents
Microchem. J. 24 (1979) 102–106
Ref.: Fresenius Z. Anal. Chem. 300 (1980) 80.
- M. M. Shoukry
Potentiometric and conductometric studies of the mixed ligand complexes of cadmium(II) involving NTA
Fresenius Z. Anal. Chem. 334 (1989) 639.