

Application Bulletin



Of interest for:
Textile and plastic laboratories

No. 68/1 e

Potentiometric determination of carboxyl and amino end groups in polyamide fibres

Summary	<ul style="list-style-type: none">▶ The indication of titration endpoints of the end groups reacting weakly alkaline or acid in a non-aqueous solution is often difficult. A considerable improvement is possible by the use of convenient titration reagents (TBAOH = tetrabutyl ammonium hydroxide for carboxyl end groups; perchloric acid for amino end groups).▶ With the choice of benzyl alcohol as a solvent there has been reached an improvement in evaluation.▶ Also of importance is the choice of the electrode combination and the measuring setup. With the differential potentiometry with the three electrode technique there has been reached a strong improvement for titrations in solutions with low conductivity. Interference signals are eliminated.
Apparatus	<ul style="list-style-type: none">▶ 2.536.0110* Potentiograph or 2.686.0100 or 2.682.0010 or 2.670.0020* Titroprocessor with Titration Stand 2.665.0030 (* in addition 6.5104.000 Differential Amplifier)▶ 6.3006.213 Exchange Units▶ 6.0102.102 Glass electrode (electrode entry 1)▶ Shielded Reference electrode 6.0729.100 (electrolyte LiCl sat. in benzyl alcohol) (electrode entry 2)▶ Earthed burette tip 6.1540.010 or Platinum rod electrode (electrode entry 3)
Reagents	<ul style="list-style-type: none">▶ $c(\text{TBAOH}) = 0.1 \text{ mol/L}$ in isopropanol▶ $c(\text{HClO}_4) = 0.1 \text{ mol/L}$ in glacial acetic acid▶ Benzyl alcohol AR grade▶ <i>m</i>-anthranilic acid AR grade (for titer determination)
Method	<ul style="list-style-type: none">▶ Determination of carboxyl end groups 0.5 ... 1.5 g sample (according to content of R-COOH) are weighed into a beaker, mixed with 100 mL benzyl alcohol and diluted by heating up to boiling point. After cooling down to 100° ... 80°C titrate with TBAOH. The burette tip should be immersed only a little bit into the solution. A blank value is determined under the same conditions.▶ Determination of amino end groups 0.5 ... 1.0 g sample (according to content of R-NH₂) are weighed into a beaker, mixed with 100 mL benzyl alcohol and diluted by heating up to boiling point. After cooling down to 100° ... 80°C titrate with perchloric acid. The burette tip should be immersed only a little bit into the solution. A blank value is determined under the same conditions.

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Calculations

$$\mu\text{Eq./kg (COOH/NH}_2) = \frac{(A - B) \times t \times 100}{E}$$

whereby:

A = consumption in mL titrating reagent for the sample

B = consumption in mL titrating reagent for the blank value

t = factor of the titrating reagent

E = weight of sample in grammes

Remarks

- ▶ For determining the titre of the titrating reagent, m-anthranilic acid can be used.
- ▶ It is advisable to soak the glass electrode in distilled water after carrying out a few titrations. This assists in retaining its response sensitivity.
- ▶ The burette tip should be immersed only a little into the hot solution to be titrated, because otherwise the titrating reagent in the burette tip would start boiling.
- ▶ The titration should be performed with a mV range of the apparatus, and at slow speed.
- ▶ It is also possible to use other solvents. For
 - R-COOH: benzyl alcohol/nitrobenzene 1:1
 - o-Cresol/chloroform 1:2
 - R-NH₂: phenol/chloroform 2.5:1 (chloroform should only be added before the titration)

Literature

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Fig. 1 Carboxyl end groups in Polyester, 682 Titroprocessor

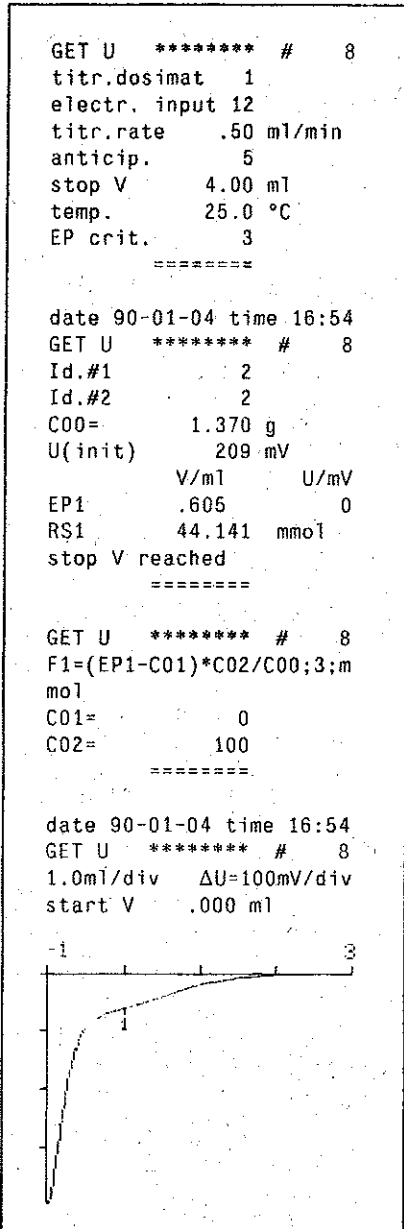


Fig. 2 Amino end groups in Polyester, 686 Titroprocessor

