International Standard ISO 6384

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Surface active agents — Technical ethoxylated fatty amines — Methods of analysis

Agents de surface — Amines grasses éthoxylées techniques — Méthode d'analyse

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 6384 was developed by Technical Committee ISO/TC 91, *Surface active agents*, and was circulated to the member bodies in October 1979.

It has been approved by the member bodies of the following countries :

Australia
Austria
Belgium
Chile
Egypt, Arab Rep. of
France
Germany, F.R.
Hungary
India
Italy
Japan
Korea, Rep. of
Netherlands
Poland
Romania
South Africa, Rep. of
Spain
Switzerland
USA
USSR

No member body expressed disapproval of the document.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

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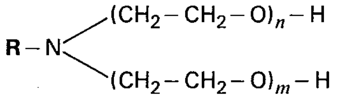
INTERNATIONAL STANDARD 6384-81 4851903 0047589 2 **ISO 6384-1981 (E)**

Surface active agents — Technical ethoxylated fatty amines — Methods of analysis

0 Introduction

The products which are the subject of this International Standard are ethers of fatty amines, corresponding to the general formula :



$$R-N\begin{cases}(CH_2-CH_2-O)_n-H\\(CH_2-CH_2-O)_m-H\end{cases}$$

where

R is a saturated or unsaturated aliphatic radical which may have a mean chain length of 8 to 22 carbon atoms;

the sum $(m + n)$ may take all values according to the uses of the product, but varies generally between 1 and 50.

1 Scope

This International Standard specifies methods of analysis for technical ethoxylated fatty amines. It covers the following determinations :

- Measurement of apparent pH in methanolic medium.
- Determination of water content.
- Determination of the distribution of primary, secondary and tertiary amines contents.
- Determination of oxyethylene groups content.
- Determination of sulphated ash.
- Determination of free polyethylene glycols content.
- Examination of polyethylene glycols.

2 Field of application

This International Standard is applicable to technical ethoxylated fatty amines, in the form of flakes, in lumps, paste or liquid, and free from any products extraneous to their manufacture.

It is not applicable to ethoxylated derivates of amido-amines and imidazolines.

3 References

ISO 607, *Surface active agents and detergents — Methods of sample division.*

ISO 2270, *Surface active agents — Ethylene oxide adducts — Iodometric determination of oxyethylene groups.*

ISO 4317, *Surface active agents — Determination of water content — Karl Fischer method.*

ISO 4322, *Non-ionic surface active agents — Determination of sulphated ash — Gravimetric method.*

ISO 4326, *Non-ionic surface active agents — Polyethoxylated derivatives — Determination of hydroxyl value — Acetic anhydride method.*

ISO 4327, *Non-ionic surface active agents — Polyalkoxylated derivatives — Determination of hydroxyl value — Phthalic anhydride method.*

4 General principle

On separate test portions :

- measurement of the apparent pH in methanolic medium;
- determination of the water content;
- determination of the distribution of primary, secondary and tertiary amines contents;
- determination of oxyethylene groups content;
- determination of sulphated ash.

Preparation of an aqueous or methanolic solution of a test portion, and isolation of the free polyethylene glycols; on the separated polyethylene glycols, verification of the absence of nitrogen and determination of their relative molecular mass.

5 Sampling

Prepare and store a laboratory sample of approximately 300 g of the crude product according to the instructions given in ISO 607.

6 Methods of analysis

6.1 Measurement of apparent pH in methanolic medium

6.1.1 Introduction

The pH value is an important characteristic of these products and it is used for assessment of their application, their activity and their stability.

As some ethoxylated fatty amines are not very soluble in water or dissolve with difficulty, the measurement of pH is made on the amine in a methanolic medium instead of as an aqueous solution. The result of the measurement in a methanolic medium is conventionally called the apparent pH.

NOTE — If it is wished to determine the actual pH of these products in methanolic medium, it is necessary to use a reference electrode of calomel — potassium chloride, saturated 68 % (*V*/*V*) methanolic solution, and the following two standard 50 % (*V*/*V*) methanolic solutions :

- methanolic solution of acetates, pH 5,54 at 20 °C : solution containing 0,02 mol/l of acetic acid (CH_3COOH), 0,02 mol/l of sodium acetate (CH_3COONa), and 0,02 mol/l of sodium chloride ($NaCl$);
- methanolic solution of hydrogenophosphates, pH 7,90 at 20 °C : solution containing 0,02 mol/l of potassium dihydrogenorthophosphate (KH_2PO_4), 0,02 mol/l of disodium hydrogenorthophosphate (Na_2HPO_4) and 0,02 mol/l of sodium chloride ($NaCl$).

6.1.2 Principle

Potentiometric measurement of the potential difference between a glass electrode and a reference electrode dipping into a methanolic medium containing the ethoxylated fatty amines.

6.1.3 Reagents

During the analysis, use only reagents of recognized analytical grade.

6.1.3.1 Distilled water, free from and protected from absorption of carbon dioxide.

6.1.3.2 Methanol.

6.1.3.3 Standard solutions.

Two standard solutions, chosen from those commonly used, are necessary to calibrate the pH meter. Their pH values should, if possible, be on either side of the expected pH of the test solution, but, if not, one should not differ from the expected pH by more than 1 pH unit.

Examples of standard solutions :

a) **Potassium hydrogen phthalate** ($KOOC-C_6H_4-COOH$), approximately 10 g/l solution, apparent pH = 4,00 at 20 °C.

Dissolve 10,12 g of potassium hydrogen phthalate, previously dried for 2 h at 100 to 130 °C, in a 50/50 (*V*/*V*) water-methanol mixture and dilute to 1 litre with the same mixture.

b) **Sodium tetraborate decahydrate** ($Na_2B_4O_7.10H_2O$), approximately 3,8 g/l solution, apparent pH = 9,22 at 20 °C.

Dissolve 3,80 g of sodium tetraborate in a 50/50 (*V*/*V*) water-methanol mixture and dilute to 1 litre with the same mixture.

6.1.4 Apparatus

6.1.4.1 pH meter, including a temperature compensation system, and graduated in 0,1 pH unit.

6.1.4.2 Glass electrode.

6.1.4.3 Calomel-saturated potassium chloride electrode.

6.1.4.4 Combined electrode, as an alternative to the glass (6.1.4.2) and reference (6.1.4.3) electrodes.

6.1.5 Procedure

6.1.5.1 Test portion

Weigh, to the nearest 0,01 g, about 10 g of the laboratory sample.

6.1.5.2 Determination

6.1.5.2.1 Preparation of the test solution

Prepare a 5 % (*m*/*m*) solution of the laboratory sample in a 50/50 (*V*/*V*) water-methanol mixture.

6.1.5.2.2 Calibration of pH meter

Calibrate the pH meter (6.1.4.1) according to the manufacturer's instructions, using the two standard solutions (6.1.3.3) chosen, at the temperature specified in 6.1.5.2.3.

6.1.5.2.3 Measurement of pH

For all the measurements, the temperatures of the electrodes, of the water used for washing and of the standard solutions (6.1.3.3) or the test solutions, shall be adjusted to 20 ± 1 °C and should be as close to each other as possible. These temperatures shall not differ by more than 1 °C.

Once the apparatus has been calibrated, wash the electrodes first with the water (6.1.3.1), then with the test solution (6.1.5.2.1). Mix the test solution, place a sufficient volume in the measuring vessel and dip the electrodes into it.

Check that the reading of the pH meter is stable for 1 min and then record the value.

Repeat the same operations with a fresh portion of the test solution. If this second measurement differs from the first by 0,1 or more pH unit, carry out a third measurement. If this value is inconclusive, repeat the test, including the calibration.

NOTE — Ethoxylated fatty amines have a tendency to adsorb onto the glass : check the electrodes frequently, therefore, and, if necessary, clean them with a sulphuric-nitric acid or sulphuric-chromic acid mixture. Also use a series of new electrodes for checking the cleanliness of the electrodes.

6.1.6 Expression of results

Express the result of apparent pH in methanolic medium in pH units, at 20 °C, and round the mean value to the nearest 0,1 pH unit.

6.2 Determination of water content

Carry out the determination of water content by the Karl Fischer method specified in ISO 4317.

6.3 Determination of distribution of primary, secondary and tertiary amines contents

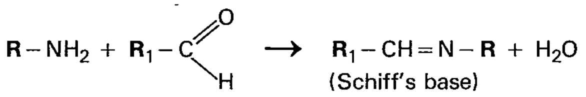
6.3.1 Simultaneous determination of total alkalinity, secondary + tertiary alkalinity and primary alkalinity

6.3.1.1 Principle

Transformation of primary amines to Schiff's bases by reaction with salicylaldehyde. Potentiometric titration of the Schiff's bases less basic than the original primary amines, by acidimetric titration in non-aqueous ternary solvent in the presence of secondary and tertiary amines.

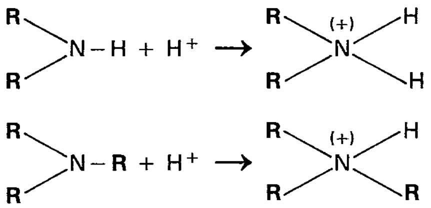
From the curve from the potentiometric titration, evaluation of the overall secondary and tertiary alkalinity and of the total alkalinity; then deduction of the primary alkalinity.

6.3.1.2 Reactions

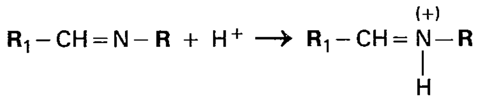


$$\mathbf{R}-NH_2 + \mathbf{R}_1-CHO \longrightarrow \underset{\text{(Schiff's base)}}{\mathbf{R}_1-CH=N-\mathbf{R}} + H_2O$$

Strong basicity



Weak basicity



$$\mathbf{R}_1-CH=N-\mathbf{R} + H^+ \longrightarrow \mathbf{R}_1-CH=\overset{(+)}{\underset{H}{N}}-\mathbf{R}$$

6.3.1.3 Reagents

During the analysis, use only reagents of recognized analytical grade.

6.3.1.3.1 1,4-Dioxane.

6.3.1.3.2 Acetic acid.

6.3.1.3.3 Salicylaldehyde.

6.3.1.3.4 Nitroethane.

6.3.1.3.5 Perchloric acid, standard volumetric solution, $c(HClO_4) \approx 0{,}1$ mol/l[1], in 1,4-dioxane.

6.3.1.3.5.1 Preparation

Dissolve 14,35 g of 70 % (*m*/*m*) perchloric acid (ϱ_{20} = 1,67 g/ml) in 1 litre of the 1,4-dioxane (6.3.1.3.1).

6.3.1.3.5.2 Standardization

In a 250 ml beaker, weigh, to the nearest 0,01 g, 0,300 g of potassium hydrogen phthalate. Add 150 ml of the acetic acid (6.3.1.3.2) to ensure dissolution. Titrate potentiometrically with the perchloric acid solution (6.3.1.3.5.1).

6.3.1.3.5.3 Calculation of concentration

The concentration, *c*, of the perchloric acid solution, expressed in moles per litre, is given by the formula

$$\frac{m_0 \times 1\,000}{204{,}2 \times V_0}$$

where

m_0 is the mass, in grams, of the potassium hydrogen phthalate used for standardization (6.3.1.3.5.2);

V_0 is the volume, in millilitres, of the perchloric acid solution (6.3.1.3.5.1) used to reach the equivalence point;

204,2 is the relative molecular mass of potassium hydrogen phthalate.

6.3.1.4 Apparatus

Ordinary laboratory apparatus and, in particular :

6.3.1.4.1 Pipettes, of capacity 5 ml, complying with the requirements of ISO 648.

6.3.1.4.2 Burettes, of capacity 50 ml, complying with the requirements of ISO/R 385, class A.

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[1] Formerly expressed as "approximately 0,1 N standard volumetric solution".

6.3.1.4.3 Magnetic stirrer.

6.3.1.4.4 pH meter, if possible with a derived function recorder.

6.3.1.4.5 Glass electrode.

6.3.1.4.6 Calomel-saturated potassium chloride electrode.

6.3.1.4.7 Combined electrode, as an alternative to the glass (6.3.1.4.5) and reference (6.3.1.4.6) electrodes. (Choose one especially conceived for non-aqueous solutions, i.e. with a filling of organic electrolyte.)

6.3.1.5 Procedure

CAUTION — Take the usual precautions and comply with relevant safety instructions for the handling of poisonous substances, in particular for the 1,4-dioxane which may be carcinogenic, and for the handling of explosive substances, in particular for perchloric acid.

6.3.1.5.1 Test portion

In a 250 ml beaker, weigh, to the nearest 0,001 g, a mass of the laboratory sample according to the oxyethylene content as follows :

- 0,5 to 1 g for low contents;
- 1,5 to 2 g for high contents.

6.3.1.5.2 Determination

Dissolve the test portion (6.3.1.5.1) in 20 ml of the 1,4-dioxane (6.3.1.3.1) and heat, if necessary, on a boiling water bath. Add 5 ml of the acetic acid (6.3.1.3.2) and 5 ml of the salicylaldehyde (6.3.1.3.3). Mix by shaking and leave for 30 min at ambient temperature. Add 50 ml of the 1,4-dioxane (6.3.1.3.1) and 20 ml of the nitroethane (6.3.1.3.4) and then titrate potentiometrically with the perchloric acid solution (6.3.1.3.5).

6.3.1.6 Expression of results

The total alkalinity or the neutralization equivalent, expressed in milliequivalents per gram, is given by the formula

$$\frac{c \times V_2}{m_1}$$

The secondary + tertiary alkalinity, expressed in milliequivalents per gram, is given by the formula

$$\frac{c \times V_1}{m_1}$$

The primary alkalinity, expressed in milliequivalents per gram, is given by the formula

$$\frac{c\,(V_2 - V_1)}{m_1}$$

where

c is the concentration, in moles per litre, of the perchloric acid solution calculated in accordance with 6.3.1.3.5.3;

V_1 is the volume, in millilitres, of the perchloric acid solution (6.3.1.3.5) used to reach the first equivalence point;

V_2 is the volume, in millilitres, of the perchloric acid solution (6.3.1.3.5) used to reach the second equivalence point;

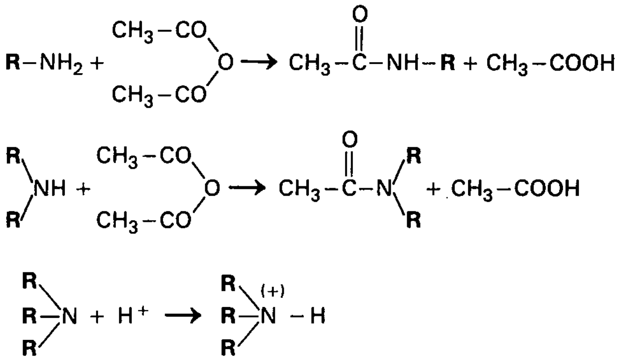
m_1 is the mass, in grams, of the test portion (6.3.1.5.1).

6.3.2 Determination of tertiary alkalinity

6.3.2.1 Principle

After acetylation of primary and secondary amines, determination of the tertiary alkalinity by selective potentiometric titration by acidimetry in acetic acid solution.

6.3.2.2 Reactions



$$\mathbf{R}{-}NH_2 + \begin{matrix}CH_3{-}CO\\ O\\ CH_3{-}CO\end{matrix} \longrightarrow CH_3{-}\overset{\overset{O}{\|}}{C}{-}NH{-}\mathbf{R} + CH_3{-}COOH$$

$$\begin{matrix}\mathbf{R}\\ NH\\ \mathbf{R}\end{matrix} + \begin{matrix}CH_3{-}CO\\ O\\ CH_3{-}CO\end{matrix} \longrightarrow CH_3{-}\overset{\overset{O}{\|}}{C}{-}N\begin{matrix}\mathbf{R}\\ \mathbf{R}\end{matrix} + CH_3{-}COOH$$

$$\begin{matrix}\mathbf{R}\\ \mathbf{R}{-}N\\ \mathbf{R}\end{matrix} + H^+ \longrightarrow \begin{matrix}\mathbf{R}\;{}^{(+)}\\ \mathbf{R}{-}N{-}H\\ \mathbf{R}\end{matrix}$$

6.3.2.3 Reagents

During the analysis, use only reagents of recognized analytical grade.

6.3.2.3.1 Acetic acid.

6.3.2.3.2 Acetic anhydride.

6.3.2.3.3 Perchloric acid, standard volumetric solution, $c(HClO_4) \approx 0{,}1$ mol/l[1], in 1,4-dioxane.

For the preparation and standardization of this solution, see 6.3.1.3.5.

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[1] Formerly expressed as "approximately 0,1 N standard volumetric solution".

6.3.2.4 Apparatus

Ordinary laboratory apparatus and in addition to that specified in 6.3.1.4 :

6.3.2.4.1 Flask, of capacity 50 ml, having a ground glass neck.

6.3.2.4.2 Condenser, having a ground glass joint to fit the flask (6.3.2.4.1), and complying with the requirements of ISO 4799.

6.3.2.5 Procedure

6.3.2.5.1 Test portion

In the flask (6.3.2.4.1), weigh, to the nearest 0,001 g, 1 to 1,5 g of the laboratory sample.

6.3.2.5.2 Determination

Add 10 ml of the acetic anhydride (6.3.2.3.2) to the test portion (6.3.2.5.1) in the flask, and, after fitting the condenser (6.3.2.4.2), heat and reflux for 1 h.

Allow to cool to ambient temperature and transfer the contents of the flask quantitatively to a 250 ml beaker with 100 ml of the acetic acid (6.3.2.3.1). Titrate potentiometrically with the perchloric acid solution (6.3.2.3.3).

6.3.2.6 Expression of results

The tertiary alkalinity, expressed in milliequivalents per gram, is given by the formula

$$\frac{c \times V_3}{m_2}$$

where

c is the concentration, in moles per litre, of the perchloric acid solution calculated in accordance with 6.3.1.3.5.3;

V_3 is the volume, in millilitres, of the perchloric acid solution (6.3.2.3.3) used to reach the equivalence point;

m_2 is the mass, in grams, of the test portion (6.3.2.5.1).

6.3.3 General expression of results

6.3.3.1 Method of calculation

The total alkalinity, expressed in milliequivalents per gram, is given by the formula

$$\frac{c \times V_2}{m_1}$$

The primary alkalinity, expressed in milliequivalents per gram, is given by the formula

$$\frac{c\,(V_2 - V_1)}{m_1}$$

The secondary alkalinity, expressed in milliequivalents per gram, is given by the formula

$$c\left(\frac{V_1}{m_1} - \frac{V_3}{m_2}\right)$$

The tertiary alkalinity, expressed in milliequivalents per gram, is given by the formula

$$\frac{c \times V_3}{m_2}$$

6.3.3.2 Precision

Comparative analyses on a sample of ethoxylated fatty amine C_{12}, comprising 24 % (molar) of primary amines C_{12}, 31 % (molar) of amines with one oxyethylene group, and 45 % (molar) of amines with two oxyethylene groups; carried out in 9 laboratories, have given the following statistical results :

a) **total alkalinity** (6.3.3)

- mean (milliequivalents per gram) : 4,18
- standard deviation of repeatability : 0,014
- standard deviation of reproducibility : 0,19

b) **secondary + tertiary alkalinity** (6.3.1)

- mean (milliequivalents per gram) : 3,19
- standard deviation of repeatability : 0,026
- standard deviation of reproducibility : 0,339

c) **tertiary alkalinity** (6.3.2)

- mean (milliequivalents per gram) : 1,88
- standard deviation of repeatability : 0,06
- standard deviation of reproducibility : 0,06

6.4 Determination of oxyethylene groups content

Carry out the determination of oxyethylene groups by the method specified in ISO 2270 taking into account the following remarks.

6.4.1 With regard to the procedure

When there is formation of tars sticking to the bottom of the flask, take care to transfer quantitatively the whole residue to the titration flask after dissolving it completely in methanol.

6.4.2 With regard to the titration

In case of formation of tars, carry out the titration potentiometrically to overcome the difficulty in observing the end of the titration due to the dark colour of the solution.

6.4.3 With regard to the calculation of ethylene oxide content

The formula given in the ISO 2270 does not allow calculation, for products containing nitrogen, of the actual ethylene oxide content because the decomposition reaction during the determination stops either at the stage of one oxyethylene group for secondary amines, or at the stage of two oxyethylene groups for tertiary amines; consequently, take into account the secondary and tertiary alkalinities of the products.

The actual ethylene oxide content, expressed as a percentage by mass, is given by the formula

$$\mathrm{OE} = \mathrm{OE}_0 + 4{,}4\,(2\,x_3 + x_2)$$

where

OE_0 is the ethylene oxide content, expressed as a percentage by mass, determined by the method specified in ISO 2270;

x_2 is the secondary alkalinity, in milliequivalents per gram, calculated in accordance with 6.3.3;

x_3 is the tertiary alkalinity, in milliequivalents per gram, calculated in accordance with 6.3.2.

NOTE — In the case of mixtures of weakly ethoxylated fatty amines and free secondary fatty amines, the calculated ethylene oxide content may exceed the actual content; in this case, verify the presence of secondary fatty amines.

6.5 Determination of sulphated ash

Carry out the determination of sulphated ash by the method specified in ISO 4322.

6.6 Determination of free polyethylene glycols content

6.6.1 Principle

Treating or passing an ethoxylated amine solution with or over an ion-exchange resin, chemisorption of the ethoxylated amine, and elution of the neutral products (polyethylene glycols).

6.6.2 Reagents

During the analysis, use only reagents of recognized analytical grade, and distilled water or water of equivalent purity.

6.6.2.1 Hydrochloric acid, approximately 100 g/l solution.

6.6.2.2 Ion-exchange resin, cationic type, highly acid (sulphonic group) with a rate of reticulation of 2 % of divinylbenzene, of particle size 150 to 300 µm.[1]

6.6.2.3 Methanol.

6.6.3 Apparatus

Ordinary laboratory apparatus and in particular :

6.6.3.1 Chromatography column, of height 25 to 30 cm and of internal diameter approximately 2 cm.

6.6.3.2 Distillation apparatus to which the flask of the rotary evaporator (6.6.3.3) can be fitted.

6.6.3.3 Rotary vacuum evaporator, fitted with a 250 ml flat-bottomed flask having a ground glass neck.

6.6.4 Procedure

6.6.4.1 Test portion

Weigh, to the nearest 0,001 g, a mass of the laboratory sample corresponding to approximately 5 milliequivalents of ethoxylated amine.

6.6.4.2 Passing over a column

a) Preparation of the ion-exchange column

Prepare a suspension of the ion-exchange resin (6.6.2.2) in water and use it to fill the column (6.6.3.1) to a height of about 15 cm.

Wash the resin with 200 ml of the hydrochloric acid solution (6.6.2.1) passed at a rate of 3 ml/min.

Then wash the resin with water until the eluate has the same pH as water.

b) Determination

Dissolve the test portion (6.6.4.1) in 40 ml of water or methanol (6.6.2.3). Pass this solution over the resin at a rate of 1 ml/min. Then wash with 500 ml of water at a rate of 3 ml/min. Introduce the whole of the eluate into the previously weighed flask of the rotary vacuum evaporator (6.6.3.3) and remove most of the water in the eluate using the distillation apparatus (6.6.3.2); finish the drying on the rotary vacuum evaporator (6.6.3.3) controlled at approximately 30 °C, in three periods of 10 min, making intermediate weighings.

c) Regeneration of the resin

Regenerate the resin with hydrochloric acid and water as described in 6.6.4.2.

6.6.4.3 Treatment in beaker (case of weakly ethoxylated amines)

a) Preparation of resin

In a 250 ml beaker, weigh about 25 g of the ion-exchange resin (6.6.2.2), add 200 ml of the hydrochloric acid solution

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[1] A suitable resin is available commercially. Details can be obtained from the Secretariat of ISO/TC 91 (AFNOR) or ISO Central Secretariat.

(6.6.2.1) and stir slowly with a magnetic stirrer for 30 min at 20 °C. Filter the resin on a sintered glass filter funnel of porosity P 16. Wash abundantly with water until the filtrate has the same pH as the water.

b) Determination

In a 250 ml beaker, dissolve the test portion (6.6.4.1) in 40 ml of water or the methanol (6.6.2.3). Introduce 25 g of the prepared resin [6.6.4.3 a)] and stir for 30 min at ambient temperature. Filter the resin and collect all the filtrate and washings in the previously weighed flask of the rotary vacuum evaporator (6.6.3.3) and remove most of the water in the filtrate using the distillation apparatus (6.6.3.2); finish the drying on the rotary vacuum evaporator (6.6.3.3) controlled at approximately 30 °C, in three periods of 10 min, making intermediate weighings.

6.6.5 Expression of results

6.6.5.1 Method of calculation

The polyethylene glycol content, expressed as a percentage by mass, is given by the formula

$$\frac{m_4 \times 100}{m_3}$$

where

m_3 is the mass, in grams, of the test portion (6.6.4.1);

m_4 is the mass, in grams, of the polyethylene glycol obtained in 6.6.4.2 b) or 6.6.4.3 b).

6.6.5.2 Precision

Comparative analyses on a sample of ethoxylated amine C_{12} having 5 oxyethylene groups, carried out in 12 laboratories, have given the following statistical results :

- mean [% (m/m)] : 2,29
- standard deviation of repeatability : 0,16
- standard deviation of reproducibility : 0,29

6.7 Examination of polyethylene glycols

6.7.1 Check for absence of nitrogen

On the polyethylene glycol separated in 6.6, determine the neutralization equivalent by the method specified in 6.3; any ethoxylated amines present in the extracted polyethylene glycol can thus be detected.

6.7.2 Determination of the relative molecular mass of the polyethylene glycol

Carry out the determination of the relative molecular mass of the polyethylene glycol using the method for determination of hydroxyl value specified in ISO 4326 or ISO 4327.

6.8 Test report

The test report shall include the following information :

a) all information necessary for the complete identification of the sample;

b) the reference of the method used (the reference of this International Standard or those of the International Standards to which reference is made);

c) the results and the method of expression used;

d) the test conditions;

e) any details not specified in this International Standard or in the International Standards to which reference is made, or regarded as optional, as well as any incidents likely to have affected the results.