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Plastics — Polyamides — Determination of ε-caprolactam and ω-laurolactam by gas chromatography

Plastiques — Polyamides — Détermination du ε-caprolactame et du ω-laurolactame par chromatographie en phase gazeuse

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 11337 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This second edition cancels and replaces the first edition (ISO 11337:2004), which has been technically revised. It also incorporates the Technical Corrigendum ISO 11337:2004/Cor.1:2007.

Plastics — Polyamides — Determination of ε-caprolactam and ω-laurolactam by gas chromatography

SAFETY STATEMENT — Persons using this document should be familiar with normal laboratory practice, if applicable. This document does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any regulatory requirements.

1 Scope

This International Standard specifies a method for determining ε-caprolactam and ω-laurolactam in polyamides by gas chromatography. It is suitable particularly for the determination of ε-caprolactam in polyamide 6 and ω-laurolactam in polyamide 12. Bearing in mind that gas chromatography offers a wide range of possible conditions, the method specified is that shown to have been suitable in practice.

Two variants of the basic method are specified:

— Method A is an extraction method with boiling methanol, and the extract is injected into a gas chromatograph.

— Method B is a method using a solvent, and the solution is injected into a gas chromatograph.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 472, *Plastics — Vocabulary*

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

3 Terms and definitions

For the purposes for this document, the terms and definitions given in ISO 472 apply.

4 Method A: Extraction method

4.1 Principle

A test portion is extracted with boiling methanol and a small volume of the extract injected into a gas chromatograph equipped with a flame-ionization detector to separate and detect the volatile components. The extract contains 1-dodecanol as an internal standard.

4.2 Reagents

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

4.2.1 Methanol.

4.2.2 1-Dodecanol.

4.2.3 ε-Caprolactam.

4.3 Apparatus and materials

Ordinary laboratory apparatus, plus the following:

4.3.1 Mill, for reducing the sample to the required grain size.

A mill in which the sample is ground at a low temperature is preferred. Large pieces can be reduced in size with a pair of scissors before they are fed to the mill.

4.3.2 Two sieves, with aperture sizes of 710 µm and 500 µm respectively, complying with the requirements of ISO 565.

4.3.3 Extraction apparatus, that will accommodate an extraction crucible or porous ceramic thimble containing the test portion.

The apparatus shall be of such a design that the crucible or thimble is heated by the rising methanol vapour or the apparatus shall be constructed of an extraction flask with a Soxhlet-type reflux condenser.

Examples of suitable extraction apparatus designed along these lines are

EXAMPLE 1

— 250 ml extraction flask;

— extraction chamber to accommodate the extraction crucible so that it is enveloped on all sides by the rising methanol vapour and the condensed methanol drips through it continuously;

— glass triangle to support the crucible;

— reflux condenser;

— sintered-glass filter crucible, pore size 40 µm to 50 µm, capacity 30 ml;

— porcelain filter-plate of slightly smaller diameter than the crucible, with holes of diameter 0,4 mm.

EXAMPLE 2

— 250 ml extraction flask;

— jacketed Soxhlet extractor;

— reflux condenser;

— sintered-glass filter crucible, pore size 40 µm to 50 µm, capacity 30 ml, or a porous ceramic thimble of similar capacity (the dimensions shall be such that the crucible or thimble can be satisfactorily accommodated in the Soxhlet apparatus);

— porcelain filter-plate of slightly smaller diameter than the crucible or thimble, as appropriate, with holes of diameter 0,4 mm.

4.3.4 Suitable heating device for extraction apparatus.

4.3.5 Analytical balance, accurate to 0,000 2 g.

4.3.6 Liquid nitrogen or **solid carbon dioxide**, if necessary.

4.3.7 Gas chromatograph, with flame-ionization detector.

a) Column

The following columns are suitable:

— a glass column (3 mm ∅ × 1,6 m), packed with acid-washed Chromosorb W[1)] of particle diameter 0,149 mm to 0,177 mm (80 mesh to 100 mesh) coated with 10 % (by mass) poly(ethylene glycol) 20M;

— a megabore Carbowax[1)] column (0,53 mm ∅ × 15 m) of corresponding separation efficiency.

The method of packing is not specified but shall be such as to obtain satisfactory separation efficiency.

Other column dimensions are permissible, but only if they have been proved to give the same results.

A capillary column may also be used.

Suggested operating conditions are shown in Table 1.

Table 1 — Operating conditions for gas chromatograph

Item	Value
Column temperature	200 °C
Injector temperature	250 °C
Detector temperature	250 °C
Carrier gas	Helium or nitrogen
Carrier gas flow rate	20 ml/min[a]

[a] This value is for the glass column. For any other type of column, a suitable flow rate will have to be chosen.

1) Examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

b) Detector

Use a flame-ionization detector in which the hydrogen and air flow rates can be adjusted so that:

— sensitivity is high;

— the relationship between response and concentration is linear over the whole measurement range;

— small changes in flow rate produce only insignificant effects on response and sensitivity.

4.3.8 Microsyringes, with capacities from 1 µl to 10 µl.

4.4 Preparation of test sample

Take a representative sample of the polymer and grind it in the mill (4.3.1). Grind the material in small portions to prevent undue heat development (i.e. to avoid the temperature rising above about 40 °C), letting the mill cool down in between portions. Solid carbon dioxide or liquid nitrogen (4.3.6) may be ground together with the polymer to prevent heat build-up. With a large mill having a greater heat capacity, these precautions may not be required. Collect the fraction that passes through a sieve with mesh aperture 710 µm (4.3.2), but not through the one with mesh aperture 500 µm.

4.5 Procedure

4.5.1 Test portion

Weigh, to the nearest 0,001 g, (5 ± 0,5) g (mass m_0) of the test sample into the filter crucible or porous thimble (4.3.3). With low-concentration samples, it is preferable to increase the mass of the test portion so that it contains approximately 0,01 g to 0,05 g of ε-caprolactam.

NOTE Polyamides can contain a small amount of water, forming part of the mass of the test portion (m_0). This water is not allowed for in the calculation of the methanol-extractable matter content since its effect is small compared the variance of the determination.

4.5.2 Extraction

Cover the test portion (see 4.5.1) with the filter-plate, pour about 50 ml of methanol (4.2.1) into the extraction flask, place the crucible or thimble containing the test portion in the extraction chamber and fit the condenser to the chamber. Heat the solvent in the flask to boiling. When the apparatus described in 4.3.3, Example 1, is used, adjust the rate of reflux to 1 to 2 drops per second and ensure that the drops fall into the crucible. When a Soxhlet extractor as described in 4.3.3, Example 2, is used, adjust the heating so that there are five to eight siphonings per hour.

Extract for a period of 3 h ± 5 min and then allow the extractor to cool to ambient temperature, overnight if necessary.

Detach the extraction flask with its contents and analyse by gas chromatography, using the procedure specified in 4.5.3 to 4.5.7.

4.5.3 Preparation of internal-standard solution

Weigh out, to the nearest 0,000 2 g, (2 ± 0,2) g of 1-dodecanol (4.2.2) and transfer it to a 1 l volumetric flask. Dissolve in methanol and make up to the mark with the same solvent.

While 1-dodecanol is the preferred internal standard, it is also possible to use isopropanol.

4.5.4 Preparation of sample solution

Transfer the extract obtained in 4.5.2 to a 100 ml volumetric flask and add 10 ml of the internal-standard solution prepared in 4.5.3. Rinse the extraction flask with small amounts of methanol, add the rinsings to the volumetric flask and make up to the mark with methanol.

4.5.5 Preparation of calibration solution

Weigh, to the nearest 0,000 2 g, (0,05 ± 0,005) g of ε-caprolactam (4.2.3) and transfer to a 100 ml volumetric flask. Add 10 ml of the internal-standard solution prepared in 4.5.3. Dissolve in methanol and make up to the mark with the same solvent.

4.5.6 Gas-chromatographic analysis of sample and calibration solutions

Inject a suitable volume between 1 µl and 10 µl (depending on the sensitivity of the detector used) of the sample solution prepared in 4.5.4 or the calibration solution prepared in 4.5.5.

NOTE When using a capillary column, it is advisable to limit the injection volume to 5 µl to avoid overloading the column.

The volume injected is not critical for the results, but shall be identical for corresponding sample and calibration solutions. Always record the calibration chromatogram at the same sensitivity setting as used for the corresponding sample chromatogram.

Multi-point calibration is recommended. For this, prepare a series of three calibration solutions with increasing concentrations in the range of the expected ε-caprolactam concentration in the sample solution. Express the result as the mean of the three calibration factors obtained.

Continue to record the chromatogram until the ε-caprolactam and internal standard have been completely eluted, then flush the column with carrier gas until the normal baseline is restored.

4.5.7 Evaluation of gas chromatographic peaks

The retention times of ε-caprolactam, methanol and 1-dodecanol shall be known, at least relative to each other. The values are dependent on the column length, the column temperature and other parameters, and they vary according to the density of the column packing and the age of the column. Typical values of retention times are shown in Table 2.

Table 2 — Typical values of retention times

Substance	Retention time in minutes	Retention time relative to 1-dodecanol
Methanol	2,80	0,21
ε-Caprolactam	5,14	0,39
1-Dodecanol	13,17	1,0

Determine the areas of the ε-caprolactam and 1-dodecanol peaks by:

a) electronic integration;

or

b) estimation using the following equation: peak area = peak height × width at half-height.

Use of method b) is recommended only for peaks on a horizontal baseline and having a shape close to that of an isosceles triangle, in order to minimize the inaccuracy of measurement. The method of peak evaluation chosen shall be identical for corresponding peaks of sample and calibration solutions.

4.6 Expression of results

The ε-caprolactam content, w, in the sample analysed is calculated, as a percentage by mass, from the equation:

$$w = \frac{A_{s'} \times A_a \times m_{a'}}{A_s \times A_{a'} \times m_0} \times 100 = \frac{A_a \times f_{s'} \times m_{s'}}{A_s \times f_{a'} \times m_0} \times 100$$

where

A_s is the area of the 1-dodecanol peak from the test solution;

$A_{s'}$ is the area of the 1-dodecanol peak from the calibration solution;

A_a is the area of the ε-caprolactam peak from the test solution;

$A_{a'}$ is the area of the ε-caprolactam peak from the calibration solution;

$m_{a'}$ is the amount of ε-caprolactam, in grams, weighed into the calibration solution in 4.5.5;

$m_{s'}$ is the amount of 1-dodecanol, in grams, weighed into the calibration solution in 4.5.5;

m_0 is the mass, in grams, of the test portion;

$f_{s'}$ is the calibration factor for ε-caprolactam:

$$f_{s'} = A_{s'}/m_{s'}$$

$f_{a'}$ is the calibration factor for 1-dodecanol:

$$f_{a'} = A_{a'}/m_{a'}$$

4.7 Precision

The precision of this method is not known because inter-laboratory data are not available. Inter-laboratory data are being obtained and will be added at a subsequent revision.

4.8 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) all details necessary for complete identification of the polyamide tested;

c) any deviation from the specifications for the gas-chromatographic equipment or from the procedure given in this International Standard;

d) the ε-caprolactam content, expressed as a percentage by mass;

e) the date of the determination.

5 Method B: Dissolution method

5.1 Principle

A small quantity of the sample to be analysed (about 0,5 g) is dissolved in an appropriate quantity of a suitable solvent containing an adequate quantity of internal standard.

A suitable volume of the solution thus obtained is then injected into a gas chromatograph to separate the ε-caprolactam or ω-laurolactam from the internal standard and allow the peak areas to be determined.

This method uses ε-caprolactam or ω-laurolactam as an internal standard, so it is important to be sure before the determination that the sample does not itself contain the internal standard used.

When analysing blends or copolyamides containing both ε-caprolactam and ω-laurolactam, 1-dodecanol, 2-azacyclononane or 2-azacyclooctanone may be used as the internal standard instead of ε-caprolactam or ω-laurolactam.

5.2 Reagents

During the analysis, use only reagents of analytical grade or the grade specified.

5.2.1 2,2,2-Trifluoroethanol (TFE).

5.2.2 Trichloromethane (chloroform).

5.2.3 ε-Caprolactam, minimum purity 99,5 %.

5.2.4 ω-Laurolactam, minimum purity 99,5 %.

5.2.5 Anhydrous ethanol.

5.2.6 Anhydrous methanol.

5.3 Apparatus

Ordinary laboratory apparatus, plus the following:

5.3.1 Gas chromatograph, equipped with an injector for liquid samples and with a ground-glass liner (removable for periodic cleaning) that can eliminate non-volatile polymeric residues; a flame-ionization detector and a recorder (or, better, a computer-integrator).

a) Column

A glass column (2 mm ∅ × 1 m) packed with Chromosorb W[2)] (80 mesh to 100 mesh) coated with 10 % (by mass) poly(ethylene glycol) 20M is suitable.

Other, similar, columns of corresponding separation efficiency may also used.

The use of a capillary column should be avoided as the polyamide remaining in the glass liner generates a large number of volatile impurities at the high temperatures used, dramatically reducing the lifetime of the column.

2) Example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

The method of packing is not specified but shall be such as to obtain satisfactory separation efficiency.

Other column dimensions are permissible, but only if they have been proved to give the same results.

Suggested operating conditions are shown in Table 3.

The temperatures and temperature-increase rate suggested are not the only possible ones. Any other temperature and temperature-increase rate that will give good separation of the solvent, ε-caprolactam and ω-laurolactam, and at the same time good peak shapes, is acceptable.

Table 3 — Operating conditions for gas chromatograph

Item	Value
Oven temperature	Hold at 175 °C for 5 min. Then increase at 10 °C/min. Hold at 205 °C for 7 min.
Injector temperature	300 °C
Detector temperature	300 °C
Carrier gas	Nitrogen
Carrier gas flow rate	35 ml/min to 60 ml/min
Injection volume	2 µl
Detector sensitivity	Has to be chosen as a function of the instrument and as a function of the lactam concentration in the sample or in the calibration solution.

b) Detector

Use a flame-ionization detector in which the hydrogen and air flow rates can be adjusted so that:

— sensitivity is high;

— the relationship between response and concentration is linear over the whole measurement range;

— small changes in flow rate produce only insignificant effects on response and sensitivity.

Every 10 injections, it is recommended that the column be cleaned with an injection of 2 µl of anhydrous ethanol.

5.3.2 Microsyringes, with capacities from 5 µl to 10 µl and with a needle without an internal wire (in order to avoid polymer blocking in the needle itself).

5.3.3 Analytical balance, accurate to ±0,000 2 g.

5.3.4 Distillation apparatus.

It is possible to use a simple Claisen distillation flask with a short column (of, for instance, the Vigreux type) 400 mm to 600 mm in length.

5.3.5 Stirring device, capable of being heated to at least 70 °C.

This may be a shaking system immersed in a water bath or mounted on a hotplate heated to the required temperature, equipped with a series of magnetic stirrers.

5.4 Preparation of internal-standard solutions

5.4.1 General

For calibration solutions, methanol (5.2.6) may be used as the solvent instead of 2,2,2-trifluoroethanol (5.2.1). The internal standards (see 5.4.2, 5.4.3 and 5.4.4) shall be prepared using 2,2,2-trifluoroethanol, however.

5.4.2 Internal standard for unextracted polyamide 6

In a small weighing bottle, weigh, to the nearest 0,000 2 g, 5,0 g of ω-laurolactam and transfer it to a 1 l volumetric flask. Dissolve in 2,2,2-trifluoroethanol and make up to the mark with the same solvent. This solution (solution S_A) is the internal standard to be used with polyamide 6, or its copolymers not containing polyamide 12, which have not yet been subjected to the extraction process for the elimination of the residual ε-caprolactam (ε-caprolactam content >5 %).

5.4.3 Internal standard for extracted polyamide 6

Weigh, to the nearest 0,000 2 g, 0,25 g of ω-laurolactam (5.2.4) and transfer it to a 1 l volumetric flask. Dissolve in 2,2,2-trifluoroethanol and make up to the mark with the same solvent. This solution (solution S_B) is the internal standard to be used with polyamide 6, or its copolymers not containing polyamide 12, which have been subjected to the extraction process for the elimination of the residual ε-caprolactam (ε-caprolactam content <1 %).

5.4.4 Internal standard for polyamide 12

Weigh, to the nearest 0,000 2 g, 0,25 g of ε-caprolactam (5.2.3) and transfer it to a 1 l volumetric flask. Dissolve in a solvent prepared by mixing together 60 parts by volume of 2,2,2-trifluoroethanol and 40 parts by volume of chloroform and make up to the mark with the same solvent. This solution (solution S_C) is the internal standard to be used with polyamide 12, or its copolymers not containing polyamide 6.

5.4.5 Preparation of calibration solutions

5.4.5.1 General

Depending on the type of material, three different sets of calibration solutions might have to be prepared:

a) a set of calibration solutions (A_1, A_2, A_3) for unextracted polyamide 6 and its copolymers;

b) a set of calibration solutions (B_1, B_2, B_3) for extracted polyamide 6 and its copolymers;

c) a set of calibration solutions (C_1, C_2, C_3) for polyamide 12 and its copolymers.

The calibration solution concentrations given below should be considered as suggestions. Other concentrations may be used if the lactam content of the sample is in a different range.

For the preparation of solutions B_1, B_2 and B_3 and C_1, C_2 and C_3, it is suggested that stock solutions be prepared which can then be diluted as and when required, as indicated below.

5.4.5.2 Calibration solutions A_1, A_2 and A_3 for use with unextracted polyamide 6

Calibration solution A_1: In a small weighing bottle, weigh, to the nearest 0,000 2 g, 0,250 0 g of ε-caprolactam and transfer it to a 100 ml volumetric flask. Dissolve in internal-standard solution S_A (see 5.4.2) and make up to the mark with the same solution.

Calibration solution A_2: Repeat the operations with 0,500 0 g of ε-caprolactam.

Calibration solution A_3: Repeat the operations with 0,750 0 g of ε-caprolactam.

5.4.5.3 Calibration solutions B_1, B_2 and B_3 for use with extracted polyamide 6

Stock solution B: In a small weighing bottle, weigh, to the nearest 0,000 2 g, 0,050 0 g of ε-caprolactam and transfer it to a 100 ml volumetric flask. Dissolve in internal-standard solution S_B (see 5.4.3) and make up to the mark with the same solution.

Calibration solution B_1: Pipette 10 ml of stock solution B into a 50 ml volumetric flask and make up to the mark with internal-standard solution S_B.

Calibration solution B_2: Pipette 20 ml of stock solution B into a 50 ml volumetric flask and make up to the mark with internal-standard solution S_B.

Calibration solution B_3: Pipette 30 ml of stock solution B into a 50 ml volumetric flask and make up to the mark with internal-standard solution S_B.

5.4.5.4 Calibration solutions C_1, C_2 and C_3 for use with polyamide 12

Stock solution C: In a small weighing bottle, weigh, to the nearest 0,000 2 g, 0,100 0 g of ω-laurolactam and transfer it to a 100 ml volumetric flask. Dissolve in internal-standard solution S_C (see 5.4.4) and make up to the mark with the same solution.

Calibration solution C_1: Pipette 10 ml of stock solution C into a 50 ml volumetric flask and make up to the mark with internal-standard solution S_C.

Calibration solution C_2: Pipette 20 ml of stock solution C into a 50 ml volumetric flask and make up to the mark with internal-standard solution S_C.

Calibration solution C_3: Pipette 30 ml of stock solution C into a 50 ml volumetric flask and make up to the mark with internal-standard solution S_C.

Calibration solutions may be used for many analyses. However, they shall be kept in a closed container because the volatility of 2,2,2-trifluoroethanol and chloroform would otherwise cause the concentration to increase with time and, in the case of the TFE/chloroform mixture, also a variation in the ratio of the two components.

5.4.6 Calibration

After preparation of the three calibration solutions of different ε-caprolactam or ω-laurolactam concentration, inject e.g. 2 µl of each into the gas chromatograph under the conditions specified for the analysis. Make three injections of each solution and take the average of the three values as the result.

It is possible to determine the relative response factor of each component for any of the three solutions. Taking the average of the three values obtained gives the relative response factor of each component or, better, the relative response factor of the lactam to be determined, taking the response factor of the calibration lactam as 1,000.

It is also possible to prepare a calibration curve by plotting the quantity of the lactam to be determined against the ratio of the peak area of the lactam to be determined to the peak area of the calibration lactam.

5.5 Procedure

5.5.1 Preparation and injection of sample solution

Into a 25 ml flask (a volumetric flask or any other suitable flask having, preferably, a volume close to this value), weigh, to the nearest 0,001 g, (0,5 ± 0,1) g of the sample to be analysed (mass m_0) and add 10,00 ml of the selected internal-standard solution (prepared in accordance with 5.4.2, 5.4.3 or 5.4.4). Then dissolve at 40 °C to 60 °C with stirring or vigorous shaking in a water bath. Dissolution takes from 30 min to 60 min,

depending on the polymer viscosity. After cooling to room temperature, the sample solution is ready to be injected.

NOTE Polyamides may contain a small amount of water, forming part of the mass of the test portion (m_0). This water is not allowed for in the calculation of the result since its effect is small compared with the variance of the determination.

The volume of solution that is usually injected is 2 µl. The actual volume injected is not critical for the results, but shall be the same for both the sample and the calibration solutions (see 5.4.5).

Make three injections for each solution and take the average of the three values obtained.

5.5.2 Evaluation of gas-chromatographic peaks

The retention times of the two lactams shall be known, at least relative to each other. The values are dependent on the column length, the column temperature and other parameters, such as the density of the column packing and the age of the column.

Typical values of retention times for lactams are shown in Table 4.

Table 4 — Typical values of retention times of lactams

Lactam	Retention time in minutes	Relative retention time (with respect to internal standard)	
		ε-Caprolactam as standard	ω-Laurolactam as standard
ε-Caprolactam	4,35	1,000	0,362
ω-Laurolactam	12,01	2,761	1,000

Determine the areas of the ε-caprolactam and ω-laurolactam peaks by:

a) electronic integration;

or

b) estimation using the following equation: peak area = peak height × width at half-height.

Use of method b) is recommended only for peaks on a horizontal baseline and having a shape close to that of an isosceles triangle, in order to minimize the inaccuracy of measurement. The method of peak evaluation chosen shall be identical for corresponding peaks of the sample and calibration solutions.

5.5.3 Solvent recovery

In view of the high price of 2,2,2-trifluoroethanol, it is essential to recover it. This can be done by distilling the sample solutions in a normal laboratory apparatus specified in 5.3.4. In view of the low boiling point of 2,2,2-trifluoroethanol, it is not necessary to work under vacuum.

Analyse the distilled solvent by gas chromatography, using the same conditions as used to determine the lactam content of a sample. If no ε-caprolactam or ω-laurolactam peaks are found, the recovered 2,2,2-trifluoroethanol is suitable for use in further analyses. If measurable quantities of ε-caprolactam or ω-laurolactam are found, carry out a second distillation.

When the TFE/chloroform (60/40) mixture is used, it is necessary to check the ratio of the two components and readjust it, if necessary, by adding more of the component whose concentration is too low.

5.6 Expression of results

Calculate the relevant relative response factor f_a (see 5.4.6).

The content w of the lactam being determined (ε-caprolactam or ω-laurolactam) in the sample analysed is calculated, as a percentage by mass, from the equation:

$$w = \frac{A_a \times f_a \times m_s}{A_s \times f_s \times m_0} \times 100$$

where

A_s is the area of the internal-standard peak;

A_a is the area of the peak of the lactam being determined;

f_s is the relative response factor of the internal standard (= 1);

f_a is the relative response factor of the lactam being determined;

m_s is the mass, in grams, of internal standard in 10 ml of the internal-standard solution;

m_0 is the mass, in grams, of the test portion.

Any other method of calculation carried out by a computer linked to the gas chromatograph is acceptable provided the final result is the same.

5.7 Precision

The precision of this method is not known because inter-laboratory data are not available. Inter-laboratory data are being obtained and will be added at a subsequent revision.

5.8 Test report

The test report shall include the following particulars:

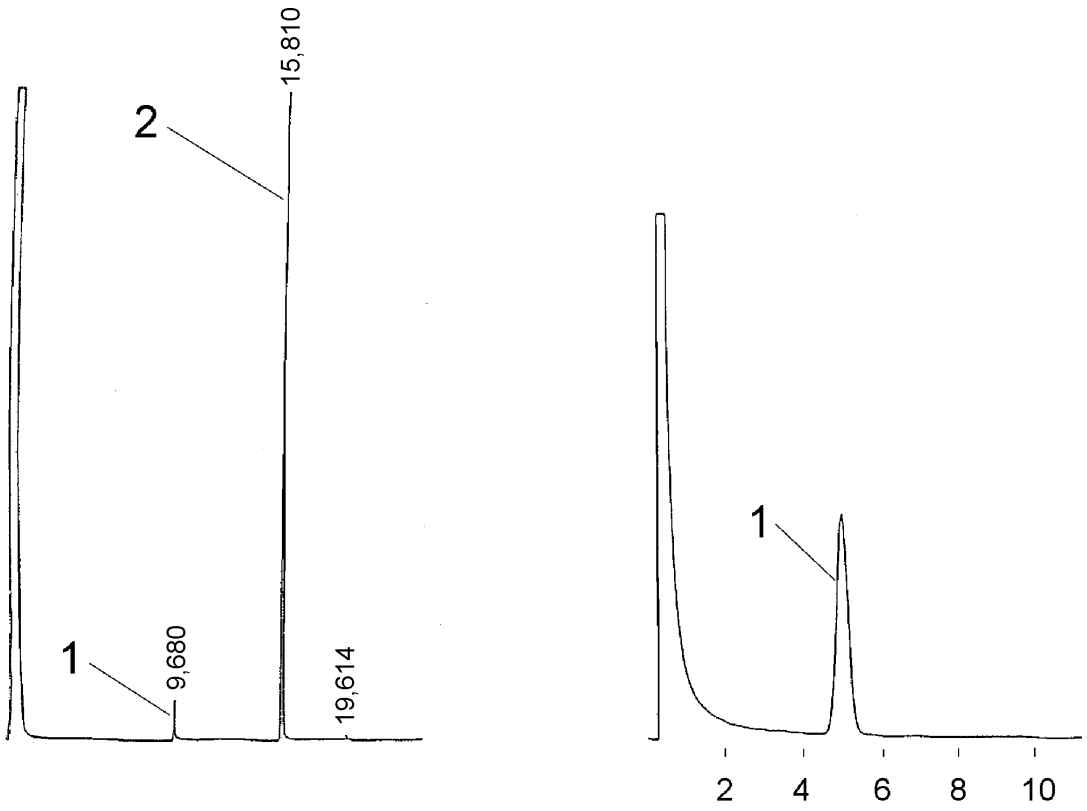
a) a reference to this International Standard;

b) all details necessary for complete identification of the polyamide tested;

c) any deviation from the specifications for the gas-chromatographic equipment or from the procedure given in this International Standard;

d) the lactam content, expressed as a percentage by mass;

e) the date of the determination.



a) Capillary column **b) Megabore column**

Key

1 ε-caprolactam 2 1-dodecanol

Figure 1 — Gas chromatograms obtained with method A

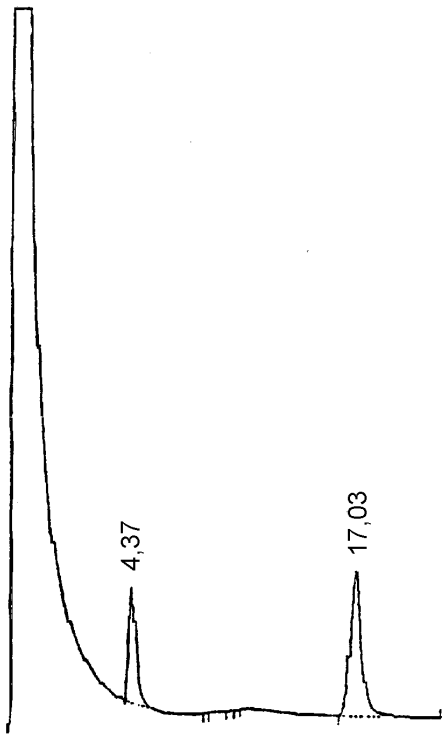
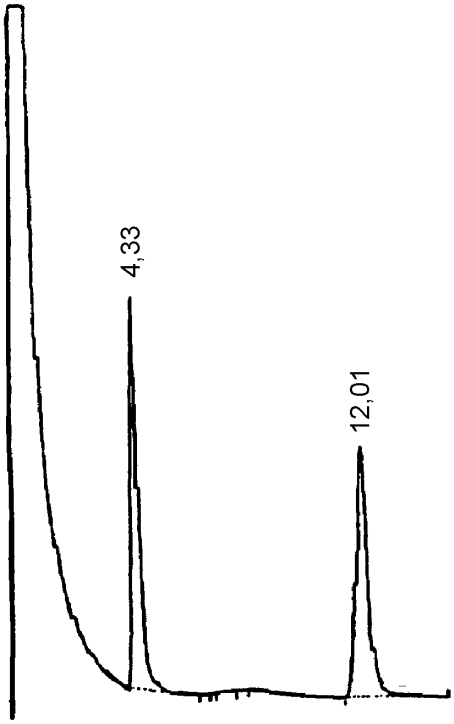
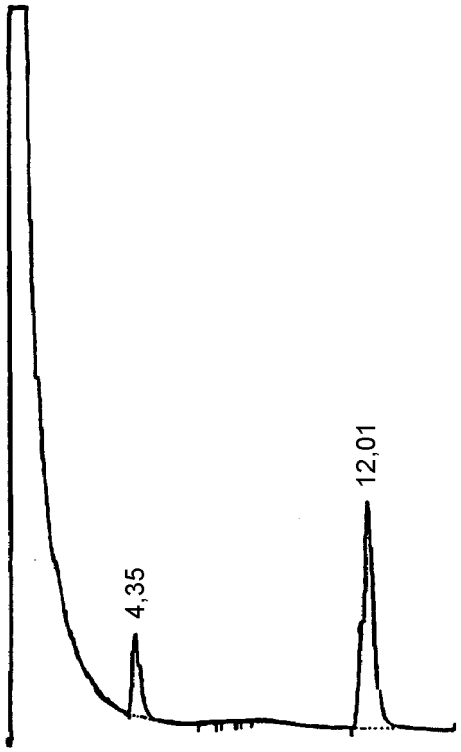


Figure 2 — Chromatogram for 0,65 % of ω-laurolactam in polyamide 12 (method B)



a) 0,44 % ε-caprolactam

b) 0,34 % ε-caprolactam



c) 0,19 % ε-caprolactam

Figure 3 — Chromatograms for three different contents of ε-caprolactam in polyamide 6 (method B)

ICS 83.080.20

Price based on 14 pages