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Caprolactam for industrial use — Determination of volatile bases content — Titrimetric method after distillation

Caprolactame à usage industriel — Dosage des bases volatiles — Méthode titrimétrique après distillation

Reference number
ISO 8661 : 1988 (E)

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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INTERNATIONAL STANDARD ISO 8661 : 1988 (E)

Caprolactam for industrial use — Determination of volatile bases content — Titrimetric method after distillation

1 Scope

This International Standard specifies a titrimetric method for the determination of the volatile bases content of caprolactam for industrial use.

2 Principle

Distillation of the volatile bases in alkaline medium under specified conditions and collection in a known volume of a standard volumetric hydrochloric acid solution in the presence of an indicator. Titration of the excess of the acid with a standard volumetric sodium hydroxide solution.

3 Reagents and material

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

3.1 Sodium hydroxide, 160 g/l solution.

3.2 Sodium hydroxide, standard volumetric solution, c(NaOH) = 0,01 mol/l.

3.3 Hydrochloric acid, standard volumetric solution, c(HCl) = 0,01 mol/l.

3.4 Mixed indicator solution.

Dissolve 0,3 g of methyl red in 100 ml of methanol and mix with 0,3 g of methylene blue dissolved in 300 ml of methanol.

3.5 Pumice grains.

4 Apparatus

Ordinary laboratory apparatus and

4.1 Distillation apparatus (see figure 1).

4.2 Heater, electric or gas.

5 Procedure

5.1 Test portion

Weigh, to the nearest 0,01 g, 20 g of the laboratory sample.

5.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of all the reagents, but omitting the test portion. The blank test value shall be the average of three parallel measurements.

5.3 Determination

Dissolve the test portion (5.1) in water in the conical flask (A) (see figure 1), dilute to 150 ml and add a few grains of the pumice (3.5) as boiling aids.

Place 10,0 ml of the standard volumetric hydrochloric acid solution (3.3) in the receiver (D). Add 30 ml of water and 5 drops of the mixed indicator solution (3.4). The outlet of the condenser shall be immersed in the liquid contained in the receiver (D).

Add 50 ml of the sodium hydroxide solution (3.1) to the contents of the conical flask (A) and immediately connect the distillation apparatus.

Distil at a constant rate such that 100 ml of distillate are collected in 30 min.

Disconnect the receiver from the apparatus. Rinse the delivery tube (E) with water, collecting the washings in the receiver.

Titrate the excess hydrochloric acid with the standard volumetric sodium hydroxide solution (3.2).

6 Expression of results

6.1 Method of calculation

The volatile bases content, expressed in millimoles of NaOH per kilogram, is given by the formula

$$\frac{(V_0 - V_1)c}{20} \times 1\,000$$

$$= 50\,(V_0 - V_1)c$$

where

V_0 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (3.2) used in the blank test (5.2);

V_1 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (3.2) used in the determination (5.3);

c is the actual concentration, in moles of NaOH per litre, of the standard volumetric sodium hydroxide solution (3.2).

Round the results to the first decimal place.

6.2 Precision

When calculated in accordance with ISO 5725*), the precision shall be as follows:

r (repeatability) = 0,03 mmol/kg

R (reproducibility) = 0,05 mmol/kg

7 Test report

The test report shall include the following particulars:

a) identification of the sample;

b) the method used;

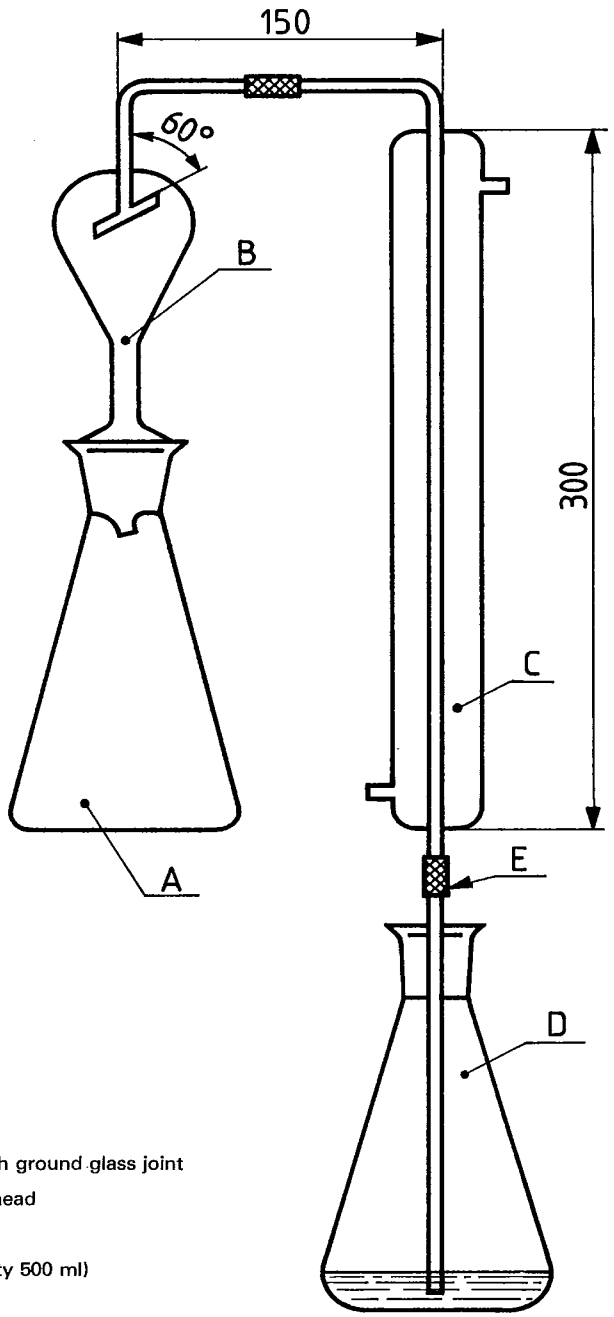
c) the results and the way in which they have been expressed;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard, or any operation regarded as optional.

*) ISO 5725 : 1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

Dimensions in millimetres



A Conical flask with ground glass joint
B Reitmeir splash head
C Condenser
D Receiver (capacity 500 ml)
E Delivery tube

Figure 1 — Distillation apparatus

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