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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*

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

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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, $c(\text{NaOH}) = 0,01 \text{ mol/l}$.

3.3 Hydrochloric acid, standard volumetric solution, $c(\text{HCl}) = 0,01 \text{ mol/l}$.

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.