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Caprolactam for industrial use — Determination of absorbance at a wavelength of 290 nm

Caprolactame à usage industriel — Détermination de l'absorbance à la longueur d'onde de 290 nm

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Foreword

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This document 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 7059:1982), which has been technically revised.

The main changes are as follows:

— the particular reference to hydrogen or deuterium lamps has been removed due to the existence of other equivalent UV light sources;

— the option to use a flow-through cell for the absorption measurement has been added;

— the temperature of measured solution has been taken into account.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Caprolactam for industrial use — Determination of absorbance at a wavelength of 290 nm

1 Scope

This document specifies a spectrometric method for the determination of the absorbance at a wavelength of 290 nm of caprolactam for industrial use.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

The absorbance of a solution of 50 % caprolactam in water is measured in a 1 cm path length cell at a wavelength of 290 nm using an ultraviolet spectrometer.

5 Reagents

During the analysis, use only distilled water or water of equivalent purity.

6 Apparatus

Ordinary laboratory apparatus and the following.

6.1 Ultraviolet spectrometer, capable of measuring the absorbance at a wavelength of 290 nm.

6.2 Two quartz cells, with optical path length 1 cm,

Or alternatively

6.3 One flow-through cell (quartz –1 cm) in combination with a pump system.

7 Procedure

7.1 Test portion and preparation of the test solution

Weigh (50,0 ± 0,1) g of the test sample, dissolve it in (50,0 ± 0,1) ml of water, and mix. Allow the solution to cool down to room temperature in case of preparing the solution with liquid (melted) caprolactam or let it warm to room temperature in case of preparing the solution with solid caprolactam.

7.2 Determination

7.2.1 Spectrometric measurements using two quartz cells

Fill one of the cells (6.2) with the test solution (see 7.1) and fill the other cell with water. Carry out the spectrometric measurements, using the spectrometer (6.1) set at a wavelength of 290 nm, after having adjusted the instrument to zero absorbance against water.

To determine the correction for the difference in absorbances at 290 nm of the cells, fill the two cells used for the measurements with water and measure the absorbance of each cell at a wavelength of 290 nm. One of the cells will read "zero" since it has been used to adjust the instrument to zero absorbance.

7.2.2 Spectrometric measurements using one flow-through cell

Set the instrument for measurements (6.3) at 290 nm. Adjust the equipment to zero absorbance with water and measure the absorbance of the test solution (see 7.1). Rinse cell with water and check the effectiveness of the rinsing with the measurement of another water sample.

NOTE Typically, rinsing with 20 to 30 times the volume of the cell plus connecting tubing will result in sufficient exchange of sample or water.

The difference between parallel measurements should not exceed 0,001 · l of absorbance where l is the path length in cm.

8 Expression of results

For spectrometric measurements using two quartz cells (see 7.2.1) the absorbance at a wavelength of 290 nm, expressed in relation to an optical path length l, is given by Formula (1):

$$A_n = \frac{(A_1 - A_0)}{l} \tag{1}$$

where

A_n is the absorbance at 290 nm normalised to the optical path length;

A_0 is the correction for the difference in absorbances at 290 nm of the cells;

A_1 is the absorbance at 290 nm of the test solution (see 7.1);

l is the optical path length, in centimetres, of the cell(s).

For spectrometric measurements using one flow-through cell (see 7.2.2) the absorbance at a wavelength of 290 nm, expressed in relation to an optical path length l, is given by Formula (2):

$$A_n = \frac{A}{l} \tag{2}$$

where

A is the absorbance at 290 nm of the test solution (see 7.1) after zeroing with water;

l is the optical path length, in centimetres, of the cell.