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Caprolactam for industrial use — Determination of permanganate index — Spectrometric method

*Caprolactame à usage industriel — Détermination de l'indice de permanganate — Méthode
spectrométrique*

Reference number
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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.

International Standard ISO 8660 was prepared by Technical Committee ISO/TC 47, *Chemistry*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Caprolactam for industrial use — Determination of permanganate index — Spectrometric method

1 Scope

This International Standard specifies a spectrometric method for the determination of the permanganate index of caprolactam for industrial use. The permanganate index defines the stability of caprolactam to the action of dilute potassium permanganate in a neutral aqueous medium and is a conventional measure of the content of oxidizable impurities.

2 Definition

For the purposes of this International Standard, the following definition applies:

permanganate index: Index based on the measurement of the absorbance at 420 nm of a 3 % (*m/m*) aqueous caprolactam solution compared with a blank (water), after addition of a standard solution of potassium permanganate, $c(\text{KMnO}_4) = 0,002 \text{ mol/l}$, to each and allowing each to stand for 10 min.

The permanganate index is the difference between the absorbance of the test sample and that of the blank, multiplied by 100/3.

3 Principle

Addition of equal volumes of potassium permanganate solution to a caprolactam test solution and to a blank (water).

After standing for a specified time, comparison of the absorbance, at a wavelength of 420 nm, of the test solution and of the blank in cells of 5 cm thickness.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only water free from oxidizable matter (4.1).

4.1 Water, free from oxidizable matter.

Use distilled water which meets the following requirements, or water of equivalent purity:

- absorbance of a blank (6.4) not more than 0,02;
- pH 6,2 to 6,5; if necessary, adjust the pH to the required value using either a 0,40 g/l sodium hydroxide solution or a 0,50 g/l sulfuric acid solution.

If the water does not meet the specified absorbance value, add 1 g of potassium permanganate per litre of water and store this solution for 24 h. Then distil using a 600 mm Vigreux, or equivalent, fractionating column until 750 ml of distillate has been collected and use this distillate for the analysis, after verifying that it complies with the above-mentioned requirements.

4.2 Potassium permanganate, standard solution $c(\text{KMnO}_4) = 0,002 \text{ mol/l}$.

Weigh, to the nearest 0,001 g, 0,316 g of potassium permanganate. Transfer to a 1 000 ml one-mark volumetric flask, dissolve and dilute to the mark with the water free from oxidizable matter (4.1).

Store, for not longer than 1 week, in a brown bottle at a temperature not exceeding 25 °C.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Spectrometer with selector for continuous variation of wavelength (single-beam or double-beam), or

5.2 Spectrometer with selector for discontinuous variation of wavelength, fitted with filters having a maximum transmission at about 420 nm.

5.3 Two cells, of thickness 5 cm.

5.4 Thermostated water bath, capable of being controlled at $25 \pm 0,5 \text{ °C}$.

5.5 Stop-watch.

6 Procedure

6.1 Cleaning of the equipment

Clean all glassware first with sulfuric acid solution, $\rho 1,84 \text{ g/ml}$, then with distilled water; complete cleaning with hydrochloric acid solution, $\rho 1,19 \text{ g/ml}$, and with distilled water and finally wash with the water free from oxidizable matter (4.1).