INTERNATIONAL STANDARD

ISO 8660

First edition
1988-04-15

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION
ORGANISATION INTERNATIONALE DE NORMALISATION
МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

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

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

Reference number
ISO 8660 : 1988 (E)

Foreword

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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.

Printed in Switzerland

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, $c(KMnO_4)$ = 0,002 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 $c(KMnO_4)$ = 0,002 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 ± 0,5 °C.

5.5 Stop-watch.

6 Procedure

6.1 Cleaning of the equipment

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

6.2 Preparation of test solution

Weigh, to the nearest 0,01 g, 50,0 g of well homogenized caprolactam in a 250 ml conical flask. Add 50,0 g of water (4.1) and dissolve the caprolactam to obtain a 50 % (m/m) solution.

Transfer 6,00 g of the caprolactam test solution into a 100 ml one-mark volumetric flask, dilute to the mark with water (4.1), mix and place the flask in the water bath (5.4) controlled at 25 ± 0,5 °C.

Maintain the flask at this temperature for not less than 15 min.

6.3 Determination

Add to the test solution (6.2) 2,00 ml of the potassium permanganate solution (4.2), immediately switch on the stopwatch (5.5), thoroughly mix the contents of the flask and place it again in the water bath. After 9 min, fill one of the cells (5.3) with the solution and 10 min ± 10 s after adding the potassium permanganate solution measure the absorbance of the solution against water at 420 nm.

6.4 Blank test

Fill a 100 ml one-mark volumetric flask to the mark with water (4.1) and proceed as specified in 6.3.

7 Expression of results

7.1 Method of calculation

The permanganate index is given by the formula

$$(A_1 - A_0) \times \frac{100}{3}$$

where

A_0 is the absorbance of the blank solution (6.4);

A_1 is the absorbance of the test solution (6.2).

7.2 Precision

Under test.

8 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.

UDC 661.717 : 543.42

Descriptors : industrial products, organic compounds, nitrogen organic compounds, caprolactams, chemical analysis, determination of content, permanganate number, spectrophotometric analysis.

Price based on 2 pages