INTERNATIONAL STANDARD

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Plastics — Determination of permanganate absorption number of caprolactam — Spectrometric method

Plastiques — Détermination de l'indice d'absorption de permanganate du caprolactame — Méthode spectrométrique

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 8660 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 8660:1988), which has been technically revised.

Introduction

The permanganate index, expressed as the permanganate absorption number (PAN), defines the stability of a caprolactam sample to potassium permanganate in a buffered neutral aqueous solution and is a measure of the purity of the caprolactam in relation to the presence of oxidizable impurities, e.g. unsaturated caprolactams.

Permanganate in a neutral aqueous solution is a strong oxidizing agent, capable of oxidizing the impurities in caprolactam. The determination of the permanganate absorption number is based on measurement of the absorbance of a 3 % (by mass) aqueous caprolactam solution at a wavelength of 420 nm. The measurement is carried out 10 min after adding a potassium permanganate solution of 0,002 mol/l. To correct for the oxidation of water, a blank determination is carried out.

In general, the oxidation reaction with unsaturated organic compounds is faster than with saturated organic compounds. Unsaturated caprolactam is oxidized at a faster rate than saturated caprolactam. The reaction speed depends upon the reducing agent and, in addition to other experimental conditions, strongly on the pH of the solution. The majority of the unsaturated impurities in caprolactam are considered to react within a period of 10 min. The reaction does not end there, however, as the oxidation of slowly oxidizing compounds, e.g. caprolactam, will continue.

The amount of manganese dioxide generated during the reaction is determined at a wavelength of 420 nm. The contribution of caprolactam at this wavelength in the method described here is less than 0,2 % of the total absorbance.

The method is sensitive to external factors and therefore needs to be followed closely. The results obtained with the method in this edition of ISO 8660 are approximately 11 % lower than those obtained with ISO 8660:1988. The difference is due to buffering of the test solution and the blank at pH 7,0. Buffering the solution at a pH of 7,0 results in a significantly higher precision of the method, as fluctuations caused by the acidity/alkalinity of the sample are eliminated.

Plastics — Determination of permanganate absorption number of caprolactam — Spectrometric method

WARNING — This International Standard may involve hazardous chemicals, materials or operations. This standard does not purport to address the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish proper safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a spectrometric method for the determination of the permanganate absorption number of caprolactam for industrial use. The permanganate absorption number is a measure of the oxidizable impurities in caprolactam, determined in a buffered neutral aqueous medium under fixed conditions.

The method is applicable to purified caprolactam (and intermediates in the caprolactam purification process) with an alkalinity or acidity lower than 0,50 mmol/kg and a PAN value of up to 35.

2 Term and definition

For the purposes of this International Standard, the following term and definition apply.

2.1
permanganate absorption number
PAN
index based on measurement of the absorbance at 420 nm of a 3 % (by mass) buffered neutral aqueous caprolactam solution in comparison with a blank consisting of buffered water, after addition of a standard solution of potassium permanganate, $c = 0{,}002$ mol/l, to each and allowing each to stand for 10 min, the absorbance being measured in a cell of pathlength 5 cm

NOTE The PAN is the difference between the absorbance of the test solution and that of the blank, multiplied by 100/3 for a glass cell of pathlength 5 cm.

3 Principle

Equal amounts of potassium permanganate are added to a buffered caprolactam test solution and to a blank consisting of buffered water. After a reaction time of 10 min, the absorbance at a wavelength of 420 nm of the caprolactam test solution and the blank are compared in glass cells with a pathlength of 5 cm.

4 Reagents

During the analysis, use only reagents of recognized analytical grade or of the grade specified.

4.1 **Sulfuric acid**, p.a., 95 % (by mass) to 97 % (by mass).

4.2 **Hydrochloric acid**, p.a., 37 % (by mass).

4.3 **Oxalic acid dihydrate**, $c = 0{,}1$ mol/l.