INTERNATIONAL STANDARD

ISO 1656

Fifth edition
2019-07

Rubber, raw natural, and rubber latex, natural — Determination of nitrogen content

Caoutchouc brut naturel et latex de caoutchouc naturel — Dosage de l'azote

Reference number
ISO 1656:2019(E)

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Published in Switzerland

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Contents

Page

Foreword ..... **iv**
Introduction ..... **vi**
1 Scope **1**
2 Normative references **1**
3 Terms and definitions **1**
4 Principle **1**
5 Macro-method **2**
5.1 Reagents 2
5.2 Apparatus 3
5.3 Sampling and preparation of test portion 3
5.4 Procedure 3
5.5 Blank test 4
5.6 Expression of results 4
6 Semi-micro method **5**
6.1 Reagents 5
6.2 Apparatus 7
6.3 Sampling and preparation of test portion 15
6.4 Procedure 15
6.5 Blank test 16
6.6 Expression of results 17
7 Precision **17**
8 Test report **17**
Annex A (informative) **Precision** **19**
Bibliography **21**

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This fifth edition cancels and replaces the fourth edition (ISO 1656:2014), which has been technically revised.

The main changes compared to the previous edition are as follows:

— an introduction has been added to explain the purpose of this revision;

— in Clause 4, a known mass of the sample is now digested with a mixture of sulfuric acid and catalytic amounts;

— the list of reagents in 5.1 and 6.1 has been updated;

— in the formulae, the exact concentration of the standard volumetric solutions are expressed in eq/dm^3 with three significant decimal figures;

— the content of total solids in latex has been changed from 2 g to 10 g in 5.3, and from 0,1 g of total solids to 5 g in 6.3;

— in 5.4.1, the tolerance on weighting of sample has been changed from 0,5 mg to 0,1 mg and the amount of catalyst mixture has been added;

— in 5.5, a note has been added to warn of the non-conformance of the blank test;

— In Figures 8 and 10, the length of the condenser has been changed from 250 mm to 300 mm; and the length of the condenser tube has been changed from 500 mm to 600 mm;

— in Clause 8, the note has been deleted since the concentrations of the standard volumetric solutions used have been standardization;

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— the precision data have been updated according to the result of the ITP and former Annex A has been deleted.

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.

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Introduction

The purpose of this document is to develop a method to determination of the nitrogen in natural rubber by Kjeldahl process using non-toxic catalyst (no selenium in catalyst mixture). The method is easy to operate, safe and environment friendly. And it does not need alternative analyser.

The previous edition of this document provided a method which had the advantage of being simple and accurate, using ordinary equipment at low cost of analysis. However, it used selenium or sodium selenate in the catalysts which is harmful to environment and human health.

Within Rubber Based Products Working Group of the ASEAN Consultative Committee on Standards and Quality activities, (RPBWG/ACCSQ), Vietnam conducted studies on this matter and finally found some suitable mixtures of catalyst to replace selenium. The mixture of $TiO_2/CuSO_4/K_2SO_4$ is the best catalyst mixture to replace the previous one $Se/CuSO_4/K_2SO_4$. It gives testing results of high accuracy, and compared to the previous one, it is safe to the technicians and the environment and easy to operate.

In addition, the total cost of the new catalyst is much cheaper than the old one (about 50 % compared to the selenium mixture catalyst).

Statistical data are available to prove the reliability of this method and its good repeatability. These data come from an ITP carried out among ASEAN member's laboratories. This ITP was permitted by the ASEAN Secretariat and was organized by the Malaysian Rubber Board (MRB). The ITP was conducted both to compare the results using selenium catalyst with the alternative catalyst, and also to demonstrate the stability of the method.

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Rubber, raw natural, and rubber latex, natural — Determination of nitrogen content

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices.

1 Scope

This document specifies a macro-method and a semi-micro method for the determination of nitrogen in raw natural rubber and in natural rubber latex using variants of the Kjeldahl process.

NOTE The determination of nitrogen in natural rubber is usually carried out in order to arrive at an estimate of the protein content. Minor amounts of non-proteinous nitrogen containing constituents are also present. However, in the dry solids prepared from natural rubber latex, these materials can make a substantial contribution to the total nitrogen content.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 123, *Rubber latex — Sampling*

ISO 124, *Latex, rubber — Determination of total solids content*

ISO 1795, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedures*

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

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

4 Principle

A known mass of the sample is digested with a mixture of sulfuric acid and catalytic amount converting nitrogen compounds into ammonium hydrogen sulfate from which the ammonia is distilled after making the mixture alkaline.

The distilled ammonia is absorbed either in standard volumetric sulfuric acid solution followed by titration of the excess acid with a standard volumetric base solution or in boric acid solution followed by titration with standard volumetric acid solution (as boric acid is a weak acid, it does not affect the indicator used for this titration).

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