

BS ISO 1656:2014



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

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National foreword

This British Standard is the UK implementation of ISO 1656:2014. It supersedes BS 7164-21:1996 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/23, Test methods for rubber and non-black compounding ingredients.

A list of organizations represented on this committee can be obtained on request to its secretary.

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

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



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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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This fourth edition cancels and replaces the third edition (ISO 1656:1996), which has been technically revised.

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

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International Standard 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 and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard 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, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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*

ISO/TR 9272, *Rubber and rubber products — Determination of precision for test method standards*

3 Principle

A known mass of the sample is digested with a mixture of sulfuric acid, potassium sulfate, and catalytic amounts of copper sulfate and selenium or sodium selenate, thereby 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).

4 Macro-method

4.1 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1.1 Catalyst mixture or catalyst solution

CAUTION — When working with selenium, avoid breathing vapours and/or contact with skin or clothing. Work only with adequate ventilation.

4.1.1.1 Catalyst mixture

Prepare a finely divided intimate mixture of the following:

- 30 parts by mass of anhydrous potassium sulfate (K_2SO_4);
- four parts by mass of copper sulfate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$);
- one part by mass of selenium powder or two parts by mass of sodium selenate decahydrate ($\text{Na}_2\text{SeO}_4 \cdot 10\text{H}_2\text{O}$).

4.1.1.2 Catalyst solution

Dissolve, with heating, the following:

- 110 g of anhydrous potassium sulfate;
- 14,7 g of copper sulfate pentahydrate;
- 3,7 g of selenium or 7,49 g of sodium selenate, in 600 cm³ of sulfuric acid (4.1.2).

4.1.2 Sulfuric acid, $\rho = 1,84 \text{ g/cm}^3$.

4.1.3 Sulfuric acid standard volumetric solution, $c(\text{H}_2\text{SO}_4) = 0,05 \text{ mol/dm}^3$.

4.1.4 Sodium hydroxide standard volumetric solution, $c(\text{NaOH}) = 0,1 \text{ mol/dm}^3$.

4.1.5 Sodium hydroxide solution, $c(\text{NaOH})$ approximately 10 mol/dm³.

Dissolve 400 g of solid sodium hydroxide in 600 cm³ of water and dilute to 1 000 cm³.

4.1.6 Boric acid solution, $c(\text{H}_3\text{BO}_3)$ approximately 0,17 mol/dm³.

Dissolve 10,5 g of solid boric acid in water, warming if necessary, and dilute to 1 000 cm³ then cool the solution to room temperature.

4.1.7 Mixed indicator solution.

Dissolve 0,1 g of methyl red and 0,05 g of methylene blue in 100 cm³ of at least 95 % (V/V) ethanol.

This indicator might deteriorate during storage and shall therefore be freshly prepared.

4.2 Apparatus

Ordinary laboratory apparatus and Kjeldahl apparatus, with a digestion flask of capacity 800 cm³.

4.3 Sampling and preparation of test portion

For the determination of nitrogen in raw solid rubber, a test portion shall be taken from the homogenized piece, sampled and prepared in accordance with ISO 1795.

For the determination of nitrogen in latex, a representative portion (as specified in ISO 123) of thoroughly mixed latex containing about 2 g of total solids shall be dried to constant mass, as specified in ISO 124.

4.4 Procedure

4.4.1 Cut about 2 g of the rubber or dried latex, weighed to the nearest 0,5 mg, into small pieces and place in the digestion flask (see [4.2](#)). Add about 13 g of the catalyst mixture ([4.1.1.1](#)) and 60 cm³ of the sulfuric acid ([4.1.2](#)) or, alternatively, 65 cm³ of the catalyst solution ([4.1.1.2](#)). Mix the contents of the flask by swirling and then boil gently until the solution is clear. Continue boiling for 1 h.

NOTE Acidic fumes evolved during digestion will be trapped in an alkaline solution and will be neutralized before being discharged.

Allow the digestion flask and its contents to cool to room temperature then cautiously add 200 cm³ of water and mix by swirling.

Place the receiving flask containing the absorbing solution in position, connect the distillation unit, and then slowly add 150 cm³ of the sodium hydroxide solution ([4.1.5](#)) to the digestion flask from a dropping funnel.

4.4.2 Carry out the absorption and titration of the liberated ammonia by the procedure described in [4.4.2.1](#) or [4.4.2.2](#). The temperature of the receiving flask shall be maintained below 30 °C to prevent any loss of ammonia.

NOTE Ensure proper disposal of the selenium-containing waste in the distillation flask.

4.4.2.1 Place 75 cm³ of water and, by means of a pipette, 25 cm³ of the standard volumetric sulfuric acid solution ([4.1.3](#)) in the receiving flask of the distillation unit together with two drops of mixed indicator solution ([4.1.7](#)). Position the receiving flask so that the end of the delivery tube from the condenser dips below the surface of the absorbing solution. While holding the stopper of the digestion flask in place, thoroughly mix the contents by swirling. Immediately commence distillation and continue at a steady rate until 200 cm³ of distillate have been collected. If the colour of the indicator changes, it indicates alkalinity of the absorbing solution. Discontinue the determination and repeat the procedure using more sulfuric acid or a smaller test portion.

When the distillation is complete (normally, when the volume in the flask reaches about 300 cm³), titrate the contents with the sodium hydroxide solution ([4.1.4](#)), reading the burette to the nearest 0,02 cm³.

4.4.2.2 Place 100 cm³ of the boric acid solution ([4.1.6](#)) in the receiving flask of the distillation unit with two drops of the mixed indicator solution ([4.1.7](#)). Carry out the distillation as described in [4.4.2.1](#) and titrate the distillate with the sulfuric acid solution ([4.1.3](#)), reading the burette to the nearest 0,02 cm³.

NOTE If the concentrations of the standard volumetric solution used are not exactly as specified in the list of reagents, appropriate corrections are to be made.

4.5 Blank test

In parallel with the determination, carry out a blank test using the same quantities of reagents under the same operating conditions but omitting the test portion.

4.6 Expression of results

4.6.1 When sulfuric acid is used as the absorbing solution as specified in [4.4.2.1](#), the nitrogen content of the rubber, expressed as a percentage by mass, is given by Formula (1):