SRI LANKA STANDARD 484 PART 3: 2022
(ISO 1656:2019)
UDC 678

METHODS OF TESTING VULCANIZED RUBBER
PART 3: DETERMINATION OF NITROGEN CONTENT
(*Third Revision*)

SRI LANKA STANDARDS INSTITUTION

Sri Lanka Standard
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SLS 484 PART 3: 2022
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Sri Lanka Standard
METHODS OF TESTING VULCANIZED RUBBER
PART 3: DETERMINATION OF NITROGEN CONTENT
(*Third Revision*)

NATIONAL FOREWORD

This Standard was approved by the Sectoral Committee on Chemical and Polymer Technology and authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 2022-10-25

SLS 484: Part 3 was first published in 1980 and revised in 2006 and 2018 as an adoption of **ISO 1656**. The text of the above International Standard has been technically revised as **ISO 1656: 2019** Rubber, raw natural, and rubber latex, natural — Determination of nitrogen content. The International Standard **ISO 1656: 2019** has been accepted for adoption as the Third Revision of **SLS 484: Part 3**.

This Sri Lanka Standard is identical with **ISO 1656: 2019**, published by the International Organization for Standardization (ISO).

ISO 1656: 2019 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.

TERMINOLOGY AND CONVENTIONS

The text of the International Standard has been accepted as suitable for publication, without deviation, as a Sri Lanka Standard. However, certain terminology and conventions are not identical with those used in Sri Lanka Standards. Attention is therefore drawn to the following:

a) Wherever the words “International Standard” appear referring to a particular Standard they should be interpreted as “Sri Lanka Standard”.

b) The comma has been used throughout as a decimal marker. In Sri Lanka Standards it is the current practice to use the full point at the base as the decimal marker.

c) Wherever page numbers are quoted, they are ISO page numbers.

Cross References

International Standard	Corresponding Sri Lanka Standard
ISO 123, Rubber latex — Sampling	SLS 1304-1 Methods of testing of natural rubber latex - Sampling of latex rubber
ISO 124, Latex, rubber — Determination of total solids content	SLS 1304-2 Methods of testing of natural rubber lattices - Determination of total solid content
ISO 1795, Rubber, raw natural and raw synthetic — Sampling and further preparative procedures	SLS 1297 Method of sampling and further preparative procedures for rubber, raw natural and raw synthetic

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SLS 484 PART 3: 2022

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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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 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;

- 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.

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.

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).

5 Macro-method

5.1 Reagents

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

5.1.1 Catalyst mixture.

5.1.1.1 Titanium dioxide catalyst mixture.

Mix well these chemicals as follows (it is recommended to use mortar and pestle):

- 100 g of anhydrous potassium sulfate (K_2SO_4);
- 3 g of copper sulfate pentahydrate ($CuSO_4{\cdot}5H_2O$);
- 3 g of titanium dioxide (TiO_2).

5.1.1.2 Selenium catalyst mixture.

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

Mix well these chemicals as follow (using mortar and pestle are recommended):

- 30 parts by mass of anhydrous potassium sulfate (K_2SO_4);
- 4 parts by mass of copper sulfate pentahydrate ($CuSO_4{\cdot}5H_2O$);
- 1 part of selenium powder or 2 parts by mass of sodium selenate decahydrate ($Na_2SeO_4.10H_2O$).

5.1.2 Sulfuric acid, ρ 1,84 g/cm^3.

5.1.3 Disodium tetraborate solution (reference standard solution), $c(Na_2B_4O_7)$ = 0,10 eq/dm^3 (0,050 mol/dm^3).

Weigh exactly 19,261 6 g of $Na_2B_4O_7{\cdot}10H_2O$ (M = 381,38 g/mol; N = 190,69 g/eq; assay = 99 %) in a beaker, dissolve with some water. Using a glass rod, careful pour them into a 1 000 cm^3 volumetric flask, rinse the beaker and glass rod for a few times, and add the rinsing solution into the volumetric flask, make up to mark with water and shake well.

5.1.4 Sulfuric acid standard volumetric solution, $c(H_2SO_4)$ = 0,1 eq/dm^3 (0,05 mol/dm^3).

Standardization of this solution by a reference standard solution is done as follows.

- Take 10,0 cm^3 of $Na_2B_4O_7$, $c(Na_2B_4O_7)$ = 0,10 eq/dm^3 (0,050 mol/dm^3) (5.1.3) into a 100 cm^3 flask.
- Add 2 drops of indicator (5.1.8), titrate this solution with H_2SO_4 (5.1.4). The end of the titration is the change of solution from green colour to pink.
- Express normal concentration of H_2SO_4, as given by Formula (1).

$$c_2 = \frac{c_1 \times V_1}{V_2} \qquad (1)$$

where

V_1 is the volume of $Na_2B_4O_7$ solution (5.1.3), expressed in cm^3;

V_2 is the volume of H_2SO_4 solution (5.1.4) required for the titration, expressed in cm^3;

c_1 is the concentration of the reference solution, $Na_2B_4O_7$ solution (5.1.3) expressed in eq/dm^3;

c_2 is the exact concentration of H_2SO_4 solution (5.1.4), expressed in eq/dm^3.

Calculate and take the correct concentration of the sulfuric acid standard solution with three significant decimal figures.

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

Standardization of this solution is carried out by the same way as demonstrated in 5.1.4, using the 0,1 eq/dm^3 sulphuric acid solution with the exact concentrate as a reference standard solution.

5.1.6 Sodium hydroxide solution, $c(NaOH)$ approximately 10 mol/dm^3 (density of 40 %).

Dissolve 400 g of solid sodium hydroxide in about 600 cm^3 of water and dilute to 1 000 cm^3.

5.1.7 Boric acid solution, $c(H_3BO_3)$ approximately 0,17 mol/dm^3.

Dissolve 10,5 g of solid boric acid in water, warming if necessary, and dilute to 1 000 cm^3, then cool the solution to room temperature.

5.1.8 Mixed indicator solution.

Dissolve 0,1 g of methyl red and 0,05 g of methylene blue in 100 cm^3 of at least 95 % (volume fraction) ethanol.

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

5.2 Apparatus

Ordinary laboratory apparatus and Kjeldahl apparatus with a digestion flask of capacity 800 cm^3.

5.3 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 10 g of total solids shall be dried to constant mass, as specified in ISO 124.

5.4 Procedure

5.4.1 Cut about 2 g of the rubber or dried latex, weighed to the nearest 0,1 mg, into small pieces and place in the digestion flask (see 5.2). Add about 18 g of the titanium dioxide catalyst mixture (5.1.1.1), or 13 g of the selenium catalyst mixture (5.1.1.2), and 60 cm^3 of the sulfuric acid (5.1.2). 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 are trapped in an alkaline solution and are neutralized before being discharged.

Allow the digestion flask and its contents to cool to room temperature then cautiously add 200 cm^3 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 cm^3 of the sodium hydroxide solution (5.1.6) to the digestion flask from a dropping funnel.

5.4.2 Carry out the absorption and titration of the liberated ammonia by the procedure described in a) or b). The temperature of the receiving flask shall be maintained below 30 °C to prevent any loss of ammonia. Ensure proper disposal of the selenium-containing waste in the distillation flask.

a) Place 75 cm^3 of water and, by means of a pipette, 25,0 cm^3 of the standard volumetric sulfuric acid solution (5.1.4) in the receiving flask of the distillation unit together with two drops of mixed indicator solution (5.1.8). 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 begin distillation and continue at a steady rate until 200 cm^3 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 cm^3), titrate the contents with the sodium hydroxide solution (5.1.5), reading the burette to the nearest 0,02 cm^3.

b) Place 100 cm^3 of the boric acid solution (5.1.7) in the receiving flask of the distillation unit with two drops of the mixed indicator solution (5.1.8). Carry out the distillation as described in a) and titrate the distillate with the sulfuric acid solution (5.1.4), reading the burette to the nearest 0,02 cm^3.

5.5 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.

NOTE Blank tests within the same laboratory are almost the same across time. Normally, blank test just shows trace of ammonia of almost equal to nil, if the titrant of the blank test is abnormal. It means that this batch of testing is contaminated. An audit of all the processes, including the equipment and the environment of the laboratory is recommended to eliminate the source of the influencing factors.

5.6 Expression of results

5.6.1 When sulfuric acid is used as the absorbing solution as specified in 5.4.2 a), the nitrogen content of the rubber, c_N, expressed as a percentage by mass, is given by Formula (2):

$$c_N = \frac{(V_2 - V_1) \times c \times 1{,}4}{m} \tag{2}$$

where

V_1 is the volume of sodium hydroxide solution (5.1.5) required for the titration, expressed in cm^3;

V_2 is the volume of sodium hydroxide solution (5.1.5) required for the titration in the blank test, expressed in cm^3;

c is the exact concentration of sodium hydroxide (5.1.5), expressed in eq/dm^3, with three significant decimal figures;

m is the mass of the test portion, expressed in g.

Express the results to the nearest 0,01 %.

5.6.2 When boric acid is used as the absorbing solution as specified in 5.4.2 b), the nitrogen content of the rubber, c_N, expressed as a percentage by mass, is given by Formula (3):

$$c_N = \frac{(V_3 - V_4) \times c \times 1,4}{m} \qquad (3)$$

where

V_3 is the volume of sulfuric acid solution (5.1.4) required for the titration of the sample, expressed in cm^3;

V_4 is the volume of sulfuric acid solution (5.1.4) required for the titration in the blank test, expressed in cm^3;

c is the exact concentration of sulfuric acid (5.1.4), expressed in eq/dm^3, with three significant decimal figures;

m is the mass of the test portion, expressed in g.

Express the results to the nearest 0,01 %.

6 Semi-micro method

6.1 Reagents

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

6.1.1 Catalyst mixture

6.1.1.1 Titanium dioxide catalyst mixture.

Mix well these chemicals as follows (it is recommended to use mortar and pestle):

— 100 g of anhydrous potassium sulfate (K_2SO_4);

— 3 g of copper sulfate pentahydrate ($CuSO_4{\cdot}5H_2O$);

— 3 g of titanium dioxide (TiO_2).

6.1.1.2 Selenium catalyst mixture.

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

Mix well these chemicals as follow (it is recommended to use mortar and pestle):

— 30 parts by mass of anhydrous potassium sulfate (K_2SO_4);

— 4 parts by mass of copper sulfate pentahydrate ($CuSO_4.5H_2O$);

— 1 part of selenium powder or 2 parts by mass of sodium selenate decahydrate ($Na_2SeO_4{\cdot}10H_2O$).

6.1.2 Sulfuric acid, ρ 1,84 g/cm^3.

6.1.3 Disodium tetra borate solution (reference standard solution), $c(Na_2B_4O_7)$ = 0,020 eq/dm^3 (0,010 mol/dm^3).

Weigh exactly 3,852 3 g of $Na_2B_4O_7.10H_2O$ (M = 381,38 g/mol; *N* = 190,69 g/eq; assay = 99 %) in a beaker, dissolve with some water. Using a glass rod, careful pour them into a 1 000 cm^3 volumetric flask, rinse the beaker and glass rod for a few times, and add the rinsing solution into the volumetric flask, make up to mark with water and shake well.

6.1.4 Sulfuric acid standard volumetric solution, $c(H_2SO_4)$ = 0,02 eq/dm^3 (0,01 mol/dm^3).

Standardization of this solution by a reference standard solution is done as follows.

- Take 10,0 cm^3 of $Na_2B_4O_7$, $c(Na_2B_4O_7)$ = 0,020 eq/dm^3 (0,010 mol/dm^3) (6.1.3) into a 100 cm^3 flask.
- Add 2 drops of indicator (6.1.8), titrate this solution with H_2SO_4 (6.1.4). The end of the titration is the change of solution from green colour to pink.
- Express as normal concentration of H_2SO_4, as given by Formula (4).

$$c_2 = \frac{c_1 \times V_1}{V_2} \tag{4}$$

where

V_1 is the volume of $Na_2B_4O_7$ solution (6.1.3), expressed in cm^3;

V_2 is the volume of H_2SO_4 solution (6.1.4) required for the titration, expressed in cm^3;

c_1 is the concentration of the reference solution, $Na_2B_4O_7$ solution (6.1.3) expressed in eq/dm^3;

c_2 is the exact concentration of H_2SO_4 solution (6.1.4), expressed in eq/dm^3.

Calculate and take the correct concentration of the sulfuric acid standard solution with three significant decimal figures.

6.1.5 Sodium hydroxide standard volumetric solution, $c(NaOH)$ = 0,02 eq/dm^3 (0,02 mol/dm^3), carbonate-free.

Standardization of this solution by the same way as demonstrated in 6.1.4, using the 0,02 eq/dm^3 sulphuric acid solution with the exactly concentrate as a reference standard solution.

6.1.6 Sodium hydroxide solution, $c(NaOH)$ approximately 10 mol/dm^3 (density of 40 %).

Dissolve 400 g of solid sodium hydroxide in about 600 cm^3 of water and dilute to 1 000 cm^3.

6.1.7 Boric acid solution, $c(H_3B0_3)$ 0,17 mol/dm^3.

Dissolve 10,5 g of solid boric acid in 200 cm^3 of water, warming if necessary, and dilute to 1 000 cm^3, then cool the solution to room temperature.

6.1.8 Mixed indicator solution.

Dissolve 0,1 g of methyl red and 0,05 g of methylene blue in 100 cm^3 of at least 95 % (volume fraction) ethanol.

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

6.2 Apparatus

Ordinary laboratory apparatus and the following are to be used.

6.2.1 Semi-micro Kjeldahl digestion apparatus.

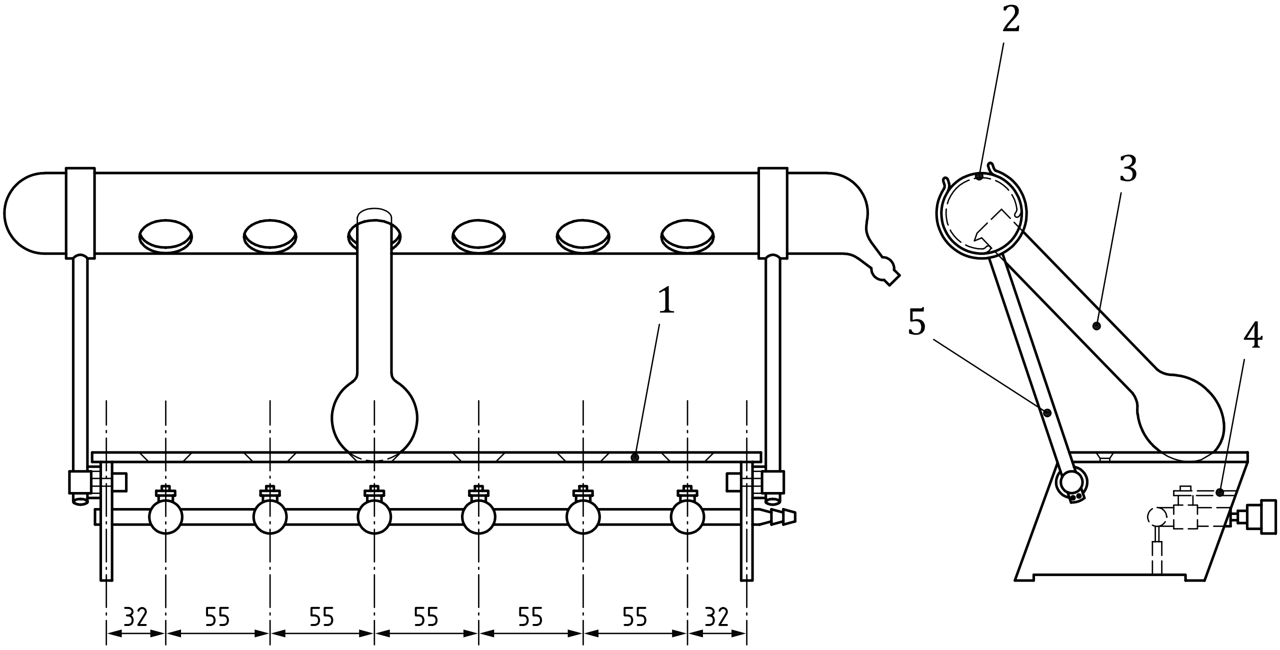
6.2.1.1 Digestion flasks of capacity 30 cm^3 and 10 cm^3 (for an example of a typical apparatus, see Figures 1, 2 and 3).

6.2.1.2 Automated digestion block (for an example of a typical apparatus, see Figure 4).

6.2.2 Semi-micro Kjeldahl distillation unit, with a condenser tube of silver, borosilicate glass, or tin (for an example, see Figures 5 to 10).

6.2.3 Semi-micro burette of capacity 5 cm^3 or 10 cm^3, graduated in 0,02 cm^3 divisions.

Dimensions in millimetres



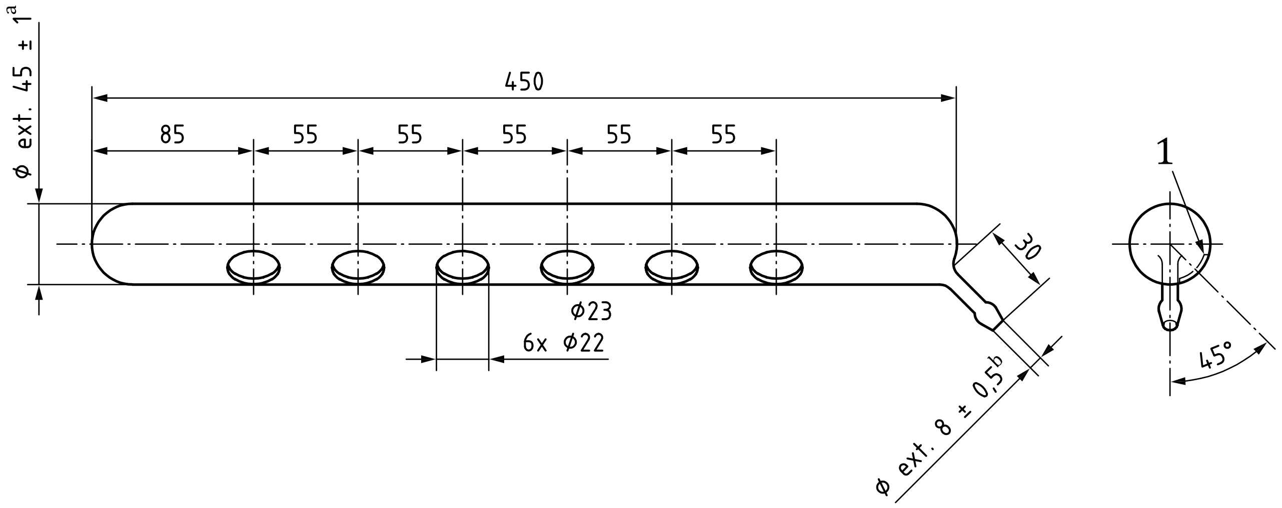
Key

1	shelf made of heat-resistant, thermally insulating material
2	exhaust tube
3	digestion flask
4	micro-burner
5	support rod with adjustment for angle and length

NOTE Where no tolerances are shown, normal working tolerances are allowed.

Figure 1 — Assembly of digestion apparatus for the semi-micro method

Dimensions in millimetres



Key

1 internal flange

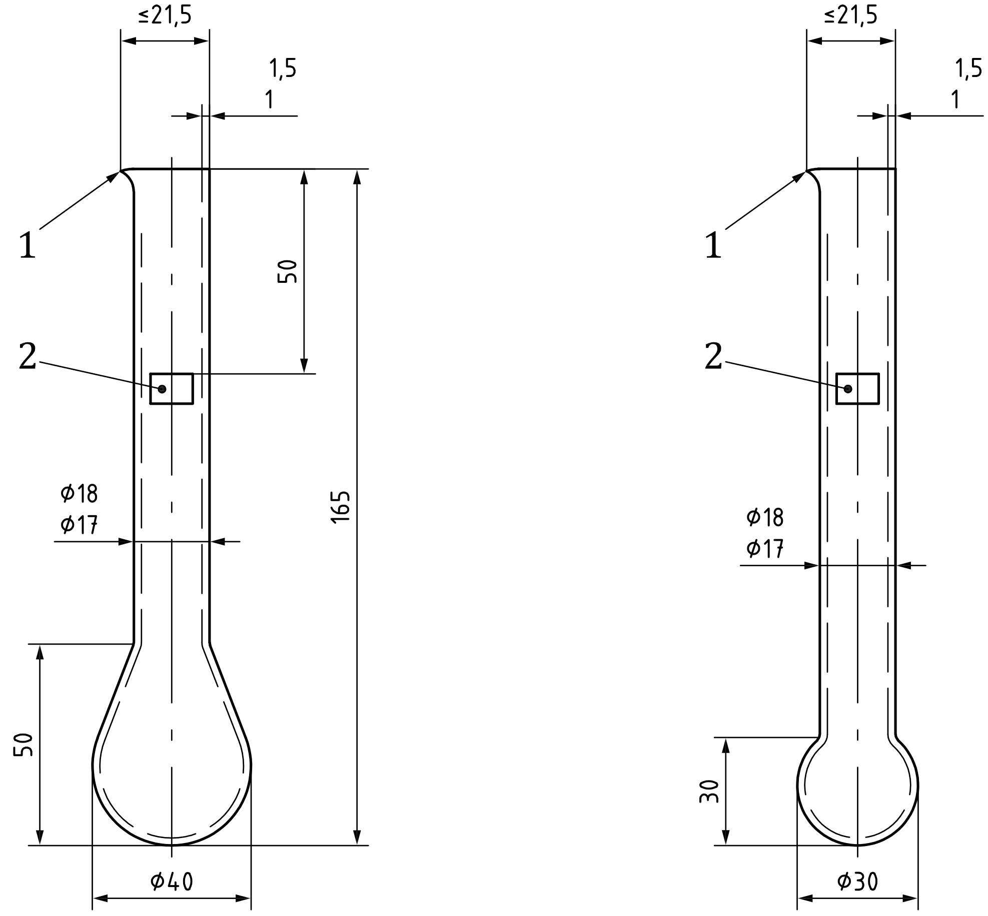
[a] Wall 1,5 mm to 2,25 mm.

[b] Wall 1,25 mm to 1,75 mm.

NOTE Where no tolerances are shown, normal working tolerances are allowed.

Figure 2 — Exhaust tube for the semi-micro method

Dimensions in millimetres



a) 30 cm^3 flask **b) 10 cm^3 flask**

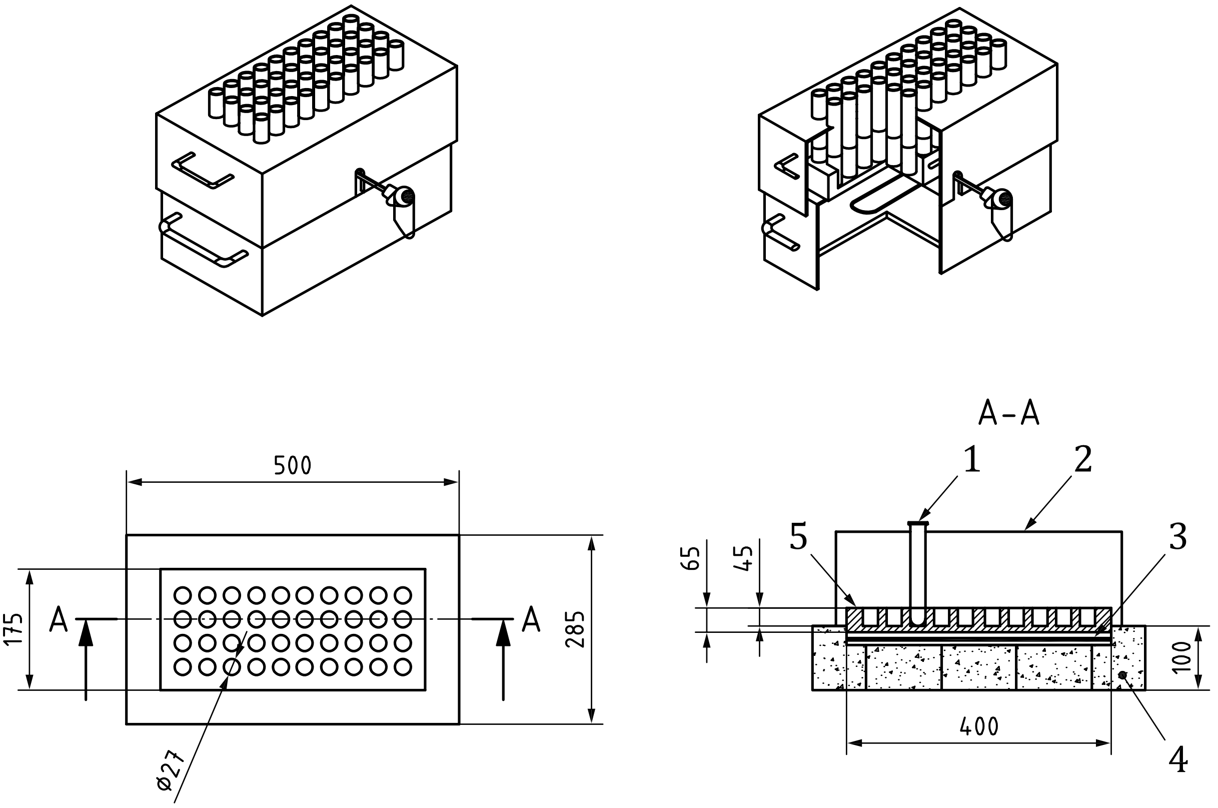
Key

1 spout

2 labelling badges

NOTE Where no tolerances are shown, normal working tolerances are allowed.

Figure 3 — Digestion flasks for the semi-micro method

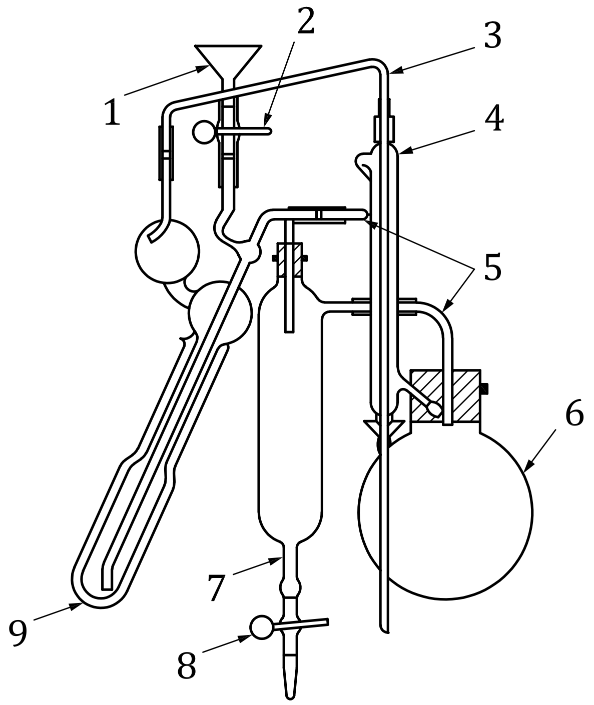


Key

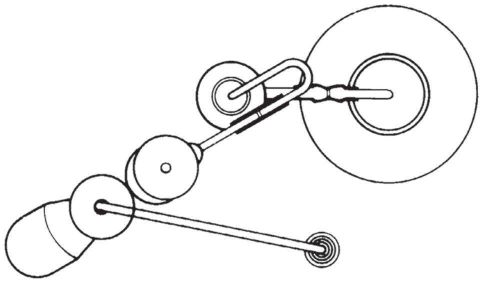
1	tube
2	tube support
3	heater
4	insulator
5	aluminium block

NOTE Where no tolerances are shown, normal working tolerances are allowed.

Figure 4 — Digestion block



a) Elevation (three-quarter view as indicated by plan below)



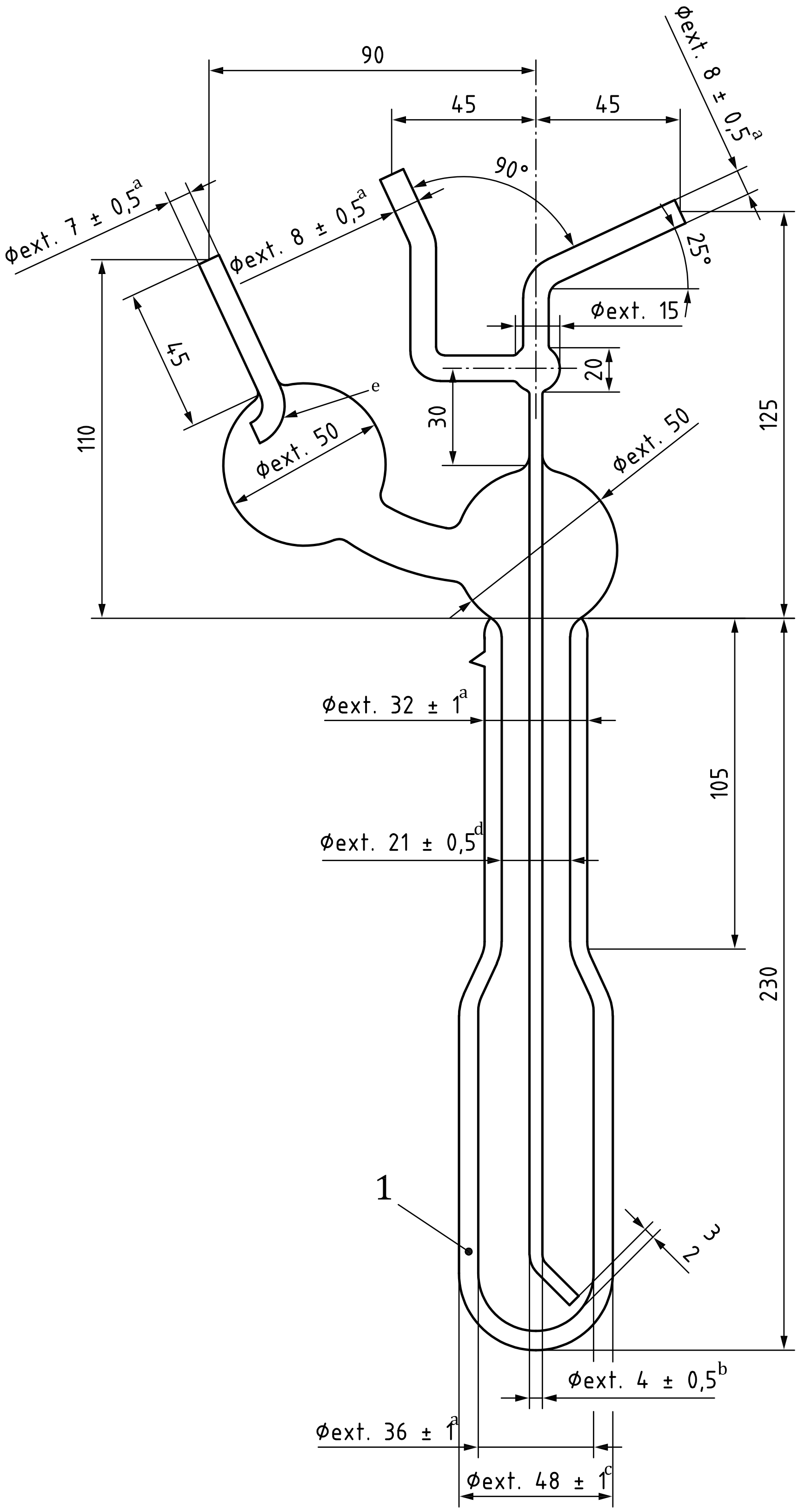
b) Plan

Key

1	funnel, ⌀ 50 mm
2	spring clip
3	condenser tube
4	condenser
5	connecting tubes bent to fit after flasks are in position
6	steam-generating flask (1 dm^3 flask with bolt head)
7	trap
8	spring clip
9	distillation flask

NOTE Where no tolerances are shown, normal working tolerances are allowed.

Figure 5 — Assembly of distillation apparatus for the semi-micro method



Key

1 jacket highly evacuated

[a] Wall 1,25 mm to 1,75 mm.

[b] Wall 0,5 mm to 1,0 mm.

[c] Wall 1,5 mm to 2,25 mm.

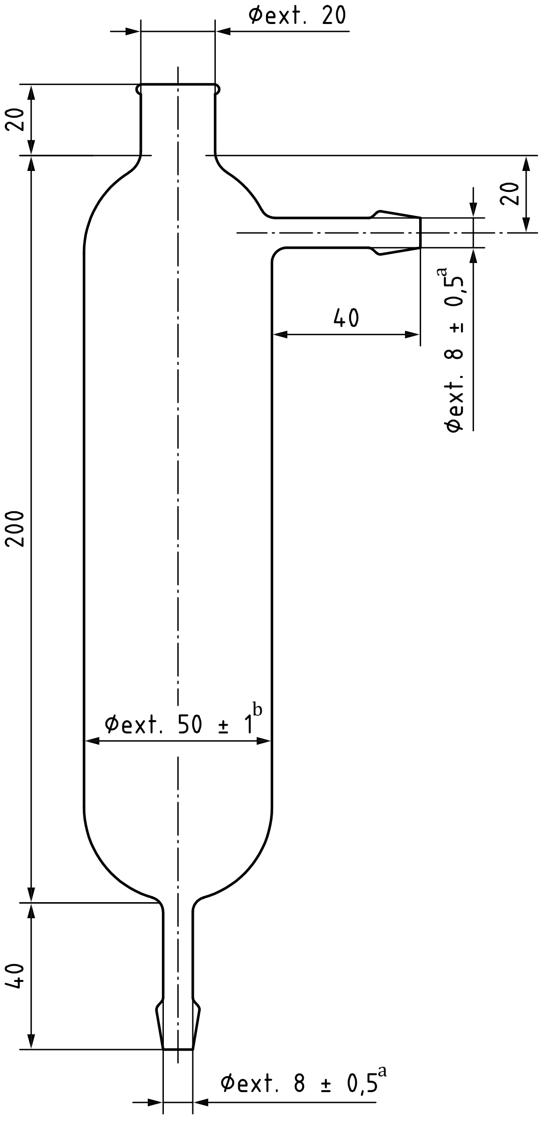
[d] Wall 1,0 mm to 1,5 mm.

[e] Hole ø 3 mm to 4 mm.

NOTE Where no tolerances are shown, normal working tolerances are allowed.

Figure 6 — Distillation flask for the semi-micro method

Dimensions in millimetres



Key

[a] Wall 1,25 mm to 1,75 mm.

[b] Wall 1,25 mm to 2,25 mm.

NOTE Where no tolerances are shown, normal working tolerances are allowed.

Figure 7 — Trap for the semi-micro method

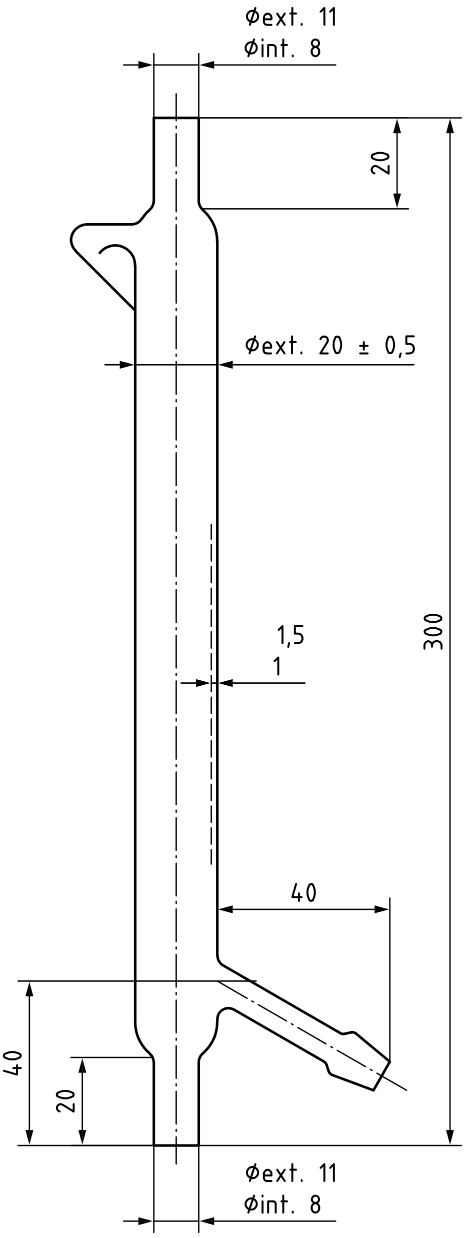
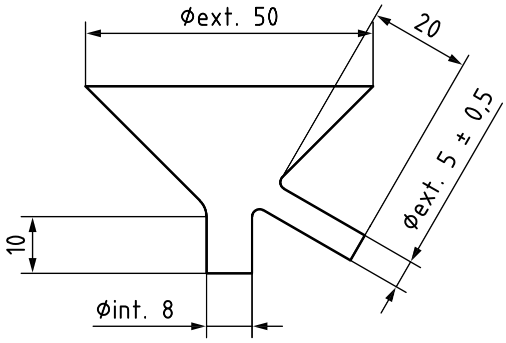


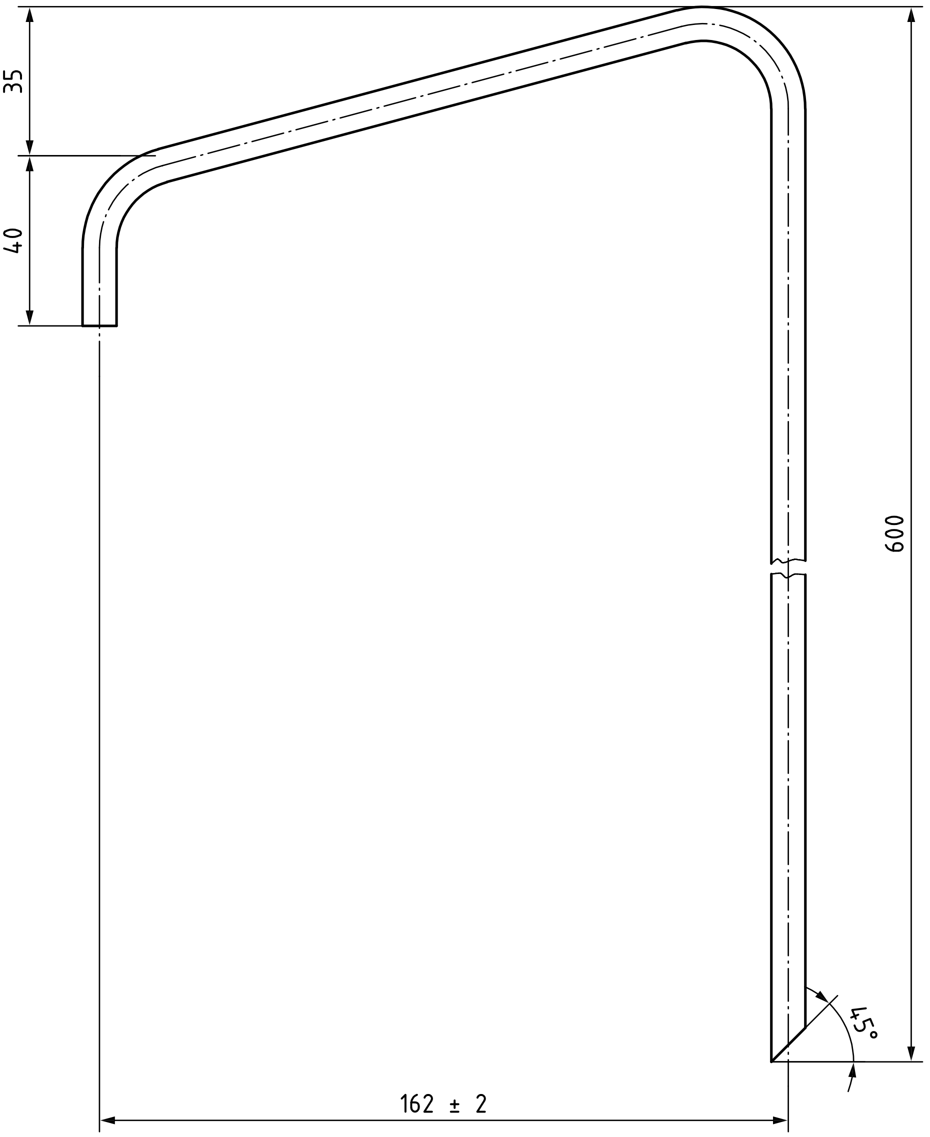
Figure 8 — Condenser jacket for the semi-micro method



NOTE Where no tolerances are shown, normal working tolerances are allowed.

Figure 9 — Drip funnel for the semi-micro method

Dimensions in millimetres



NOTE Where no tolerances are shown, normal working tolerances are allowed.

Figure 10 — Condenser tube for the semi-micro method

6.3 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 5 g of total solids shall be dried to constant mass, as specified in ISO 124.

6.4 Procedure

6.4.1 Cut about 0,1 g to 0,2 g of the rubber or dried latex, weight to the nearest 0,1 mg and place in a digestion flask (6.2.1.1). Add about 0,9 g of the titanium dioxide catalyst mixture (6.1.1.1), or 0,65 g of the selenium catalyst mixture (6.1.1.2) and 3,0 cm^3 of the sulfuric acid (6.1.2) and heat the contents carefully to the boiling point. Continue boiling for a further 30 min after the digest has become clear and green with no yellow tint.

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

Avoid excess boiling, as indicated by a tendency for the digest to solidify on cooling, since this can lead to loss of nitrogen.

Bring the water in the steam generator of the distillation unit to boil and pass steam through the semi-micro Kjeldahl distillation unit (6.2.2), including the receiving flask, for at least 2 min. The water jacket of the condenser shall not be empty of water during the steaming-out operation.

Cooling water pipe shall not be empty and let the water run continuously in the direction from bottom to top to steam distillation ammonia is condensed and is completely absorbed in the absorption solution

Meanwhile, cool the digestion flask to room temperature or below, add about 10 cm^3 of water, and immediately transfer the contents to the distillation flask at the conclusion of the steaming-out process. Complete the transfer by rinsing three times with about 3 cm^3 portions of water and draining the flask thoroughly after each transfer.

6.4.2 Discard any condensate which has been collected in the receiver and complete the distillation and titration of ammonia by the procedure described in a) or b). The temperature of the receiving flask shall be maintained below 30 °C to prevent loss of ammonia.

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

a) Add from the semi-micro burette (6.2.3) to the steamed-out receiver of the distillation apparatus a measured volume of sulfuric acid solution (6.1.4), using at least 5,0 cm^3 (the exact volume depending on the amount of nitrogen expected), together with two drops of the mixed indicator solution (6.1.8) and about 5 cm^3 of water. Position the receiver so that the end of the delivery tube from the condenser dips below the surface of the acid. It is an advantage to tilt the receiver slightly to gain a greater depth of liquid.

   Add approximately 10 cm^3 of the sodium hydroxide solution (6.1.6) to the distillation flask by means of a measuring cylinder and pass steam from the generator through the distillation flask for 10 min to 12 min at such a rate that the final volume of liquid in the receiver is about 70 cm^3. If the colour of the indicator changes, indicating 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, lower the receiving flask until the tip of the condenser is above the level of the acid, continue the distillation for another 1 min and then rinse the tip of the condenser tube with a few cubic centimetres of water which shall be collected in the distillate. Immediately titrate the contents of the receiving flask with the sodium hydroxide solution (6.1.5), reading the burette to the nearest 0,02 cm^3.

b) Place about 10 cm^3 of the boric acid solution (6.1.7) in the steamed-out receiving flask with two drops of the mixed indicator solution (6.1.8). Carry out the distillation as described in a), but note that, in the presence of boric acid, the indicator colour should change immediately distillation of ammonia commences. Titrate the distillate with sulfuric acid solution (6.1.4), reading the burette to the nearest 0,02 cm^3.

6.5 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.

6.6 Expression of results

6.6.1 When sulfuric acid is used as the absorbing solution as specified in 6.4.2 a), the nitrogen content of the rubber, c_N, expressed as a percentage by mass, is given by Formula (5):

$$c_N = \frac{(V_2 - V_1) \times c \times 1{,}4}{m} \tag{5}$$

where

V_1 is the volume of sodium hydroxide solution (6.1.5) required for the titration, expressed in cm^3;

V_2 is the volume of sodium hydroxide solution (6.1.5) required for the titration in the blank test, expressed in cm^3;

c is the exact concentration of sodium hydroxide (6.1.5), with three significant decimal figures, expressed in eq/dm^3;

m is the mass of the test portion, expressed in g;.

Express the results to the nearest 0,01 %.

6.6.2 When boric acid is used as the absorbing solution as specified in 6.4.2 b), the nitrogen content of the rubber, c_N, expressed as a percentage by mass, is given by Formula (6):

$$c_N = \frac{(V_3 - V_4) \times c \times 1{,}4}{m} \tag{6}$$

where

V_3 is the volume of sulfuric acid solution (6.1.4) required for the titration, expressed in cm^3;

V_4 is the volume of sulfuric acid solution (6.1.4) required for the titration in the blank test, expressed in cm^3;

c is the exact concentration of sulfuric acid (6.1.4), with three significant decimal figures, expressed in eq/dm^3;

m is the mass of the test portion, expressed in g.

Express the results to the nearest 0,01 %.

7 Precision

See Annex A.

8 Test report

The test report shall include the following information:

a) a reference to this document (i.e. ISO 1656:2019) and the method used;

b) all details necessary for the identification of the material tested;

c) the results and the units in which they are expressed;

d) any unusual features noted during the determination;

e) any operation not included in this document or in the International Standards to which reference is made and any operation regarded as optional;

f) the date of the test.

Annex A
(informative)

Precision

A.1 General

The precision calculations to express repeatability and reproducibility were performed in accordance with ISO/TR 9272. Annex A gives guidance on the use of repeatability and reproducibility.

A.2 Precision details

With the permission of the ASEAN Secretariat, an interlaboratory test programme (ITP) was organized in 2015 by the Malaysian Rubber Board using semi-micro Kjeldahl digestion apparatus. This ITP was also carried out to compare nitrogen results between the two catalysts selenium and titanium dioxide.

The following three types of materials were sent to each laboratory:

a) SMR CV 60;

b) SMR L;

c) SMR 20.

For all samples, a test result was taken as the mean of three separate determinations.

A “type 1” precision was measured in the ITP. The time period for repeatability and reproducibility was on a scale of days. A total of 14 laboratories from 5 ASEAN countries participated in the ITP for nitrogen determination in raw rubber.

A.3 Precision results

The summary of the precision results for the use of two catalysts method is given in Tables A.1 and A.2.

Table A.1 — Type 1 precision — Semi-micro Kjeldahl apparatus using TiO_2 catalyst

Rubber sample	**Average nitrogen content** % (mass fraction)	**Within-laboratory repeatability**		**Interlaboratory reproducibility**	
		r	(*r*)	*R*	(*R*)
SMR CV 60	0,357 5	0,034 3	9,59	0,074 8	20,92
SMR 20	0,293 7	0,028 2	9,60	0,051 3	17,47
SMR L	0,346 4	0,031 1	8,98	0,070 8	20,44

r repeatability, in percent by mass
(*r*) repeatability, in percent (relative) of the average
R reproducibility, in percent by mass
(*R*) reproducibility, in percent (relative) of the average

Table A.2 — Type 1 precision — Semi-micro Kjeldahl apparatus using Selenium catalyst

Rubber sample	Average nitrogen content % (mass fraction)	Within-laboratory repeatability		Interlaboratory reproducibility	
		r	(*r*)	*R*	(*R*)
SMR CV 60	0,343 6	0,034 1	9,92	0,056 3	16,39
SMR 20	0,290 7	0,028 2	9,77	0,070 3	24,18
SMR L	0,341 9	0,028 5	8,33	0,073 8	21,59

r repeatability, in percent by mass

(*r*) repeatability, in percent (relative) of the average

R reproducibility, in percent by mass

(*R*) reproducibility, in percent (relative) of the average

Bibliography

[1] ISO/TR 9272[1], *Rubber and rubber products — Determination of precision for test method standards*

[2] ACCSQ/RBPWG *Report of the sixteenth meeting of the Rubber Based Product Working Group (RBPWG)*; Agenda item 7.3. ASEAN common position on new standards for rubber products at international fora; 6-7 Mar. 2013, Yangon, Myanmar

[3] ACCSQ/RBPWG *Report of the ninth meeting of the task force on Rubber-based products (TFRB)*; Agenda item 7. ASEAN common position on new standards for rubber products at international for a; 18 March 2014, Halong City, Vietnam

[4] ACCSQ/RBPWG *Report of the seventeenth meeting of the RBPWG*; Agenda item 7.3. ASEAN common position on new standards for rubber products at international fora; 3-5 Sept. 2013, Phnom Penh, Cambodia

[5] ACCSQ/RBPWG *Report of the eleventh meeting of the Rubber Based Task Force on the Rubber Based Product*; Agenda item 7. ASEAN common position in International fora on the development of new ISO standard; 17-18 Mar., Kuala Lumpur, Malaysia

[6] ACCSQ/RBPWG 11th TFRBP - Doc 12 Ag 7 - *RBPWG Nitrogen content Crosscheck*. Malaysian Rubber Board; Mar. 2015

[7] NGUYEN N.T.T. et al. *New mixture catalyst to replace selenium in determination of nitrogen using Kjeldahl method*. Proceedings International Rubber Conference 2015: Productivity And Quality Towards A Sustainable And Profitable Natural Rubber Sector; 2nd – 3rd November 2015, Ho Chi Minh City, Vietnam; Page 429 – 436

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1) Withdrawn.

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