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Nitrile rubber — Determination of residual unsaturation in hydrogenated nitrile rubber (HNBR) by iodine value

Caoutchouc nitrile — Détermination de l'insaturation résiduelle dans le caoutchouc nitrile hydrogéné (HNBR) par l'indice d'iode

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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 17564 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analyses*.

Annex A forms a normative part of this International Standard.

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Nitrile rubber — Determination of residual unsaturation in hydrogenated nitrile rubber (HNBR) by iodine value

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This 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 method using Wijs' solution to determine the iodine value (i.e. the residual unsaturation) of hydrogenated nitrile rubber (HNBR).

2 Normative references

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The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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ISO 1795:2000, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedures*.

ISO/TR 9272:1986, *Rubber and rubber products — Determination of precision for test method standards.*

3 Principle

A sample of raw, unvulcanized HNBR is dissolved in chloroform. A known excess of Wijs' solution is added to the solution and a fixed time is allowed for addition of iodine to the residual unsaturation in the HNBR. Unreacted Wijs' solution is then neutralized with potassium iodide solution, the iodine thus liberated titrated with standard sodium thiosulfate and the iodine value (residual unsaturation) calculated.

4 Reagents

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

4.1 Chloroform.

4.2 Wijs' solution (see Annex A).

4.3 100 g/l aqueous potassium iodide solution.

4.4 10 g/l starch solution.

4.5 0,1 mol/l standard volumetric sodium thiosulfate solution.

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5 Apparatus

5.1 Mechanical shaker.

5.2 Constant-temperature bath, capable of being maintained at (25 ± 1) °C.

5.3 Analytical balance, accurate to 0,1 mg.

5.4 Glass-stoppered conical flask, capacity 300 cm^3.

5.5 Pipettes, capacity 10 cm^3 and 25 cm^3.

5.6 Burette, capacity 50 cm^3, graduated at 0,1 cm^3 intervals.

6 Procedure

6.1 From a sample obtained in accordance with ISO 1795, weigh out, to the nearest 0,1 mg, a test portion of size corresponding to the suspected degree of unsaturation (iodine value) as indicated in Table 1 and place it in a 300 cm^3 glass-stoppered conical flask.

Table 1 — Recommended test portion sizes

Suspected degree of unsaturation (iodine value)	Mass of test portion g
> 30	0,35 to 0,40
15 to 30	0,40 to 0,50
8 to 15	0,50 to 0,70
< 8	0,90 to 1,0

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6.2 Add 50 cm^3 of chloroform (4.1) to the flask, stopper it and place on a mechanical shaker until the test portion has completely dissolved. Then place the flask in a constant-temperature bath at (25 ± 1) °C for 30 min.

6.3 Remove the flask from the bath and accurately pipette 25 cm^3 of Wijs' solution (4.2) into the flask. Immediately stopper the flask and swirl gently to mix. Place the flask in the constant-temperature bath for 2 h ± 5 min to complete the iodine addition reaction.

6.4 Once the iodine addition reaction is complete, remove the flask from the bath and quickly add, by pipette, 10 cm^3 of potassium iodide solution (4.3). Immediately stopper the flask and shake vigorously.

6.5 Loosen the stopper slightly and, using a wash bottle, wash the stopper and mouth of the flask with the minimum amount of distilled water, ensuring the washings run directly into the flask. Replace the stopper, swirl gently and allow the flask to stand for 5 min.

6.6 Within 20 min, titrate with sodium thiosulfate solution (4.5) while swirling the flask gently. When the upper (aqueous) layer becomes slightly yellow, add about 1 cm^3 of starch solution (4.4). Stopper the flask and shake vigorously. Continue the titration, shaking the flask vigorously at intervals, until the purple colour of the iodine/starch complex vanishes. It is important that the titration with sodium thiosulfate be completed within 30 min after the addition of the potassium iodide solution.

NOTE It is important to shake the flask vigorously after addition of the starch solution in order to ensure complete removal of the iodine from the chloroform into the water layer where it becomes available for the reaction with the starch.

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6.7 Let the flask stand for 30 min. Should the colour reappear, add additional titrant with vigorous shaking until no additional colour appears on standing for 30 min.

6.8 Conduct a blank titration, performing steps 6.2 to 6.7.

7 Calculation

Calculate the iodine value from the following equation:

$$A = \frac{(V_1 - V_0) \times c \times 12{,}69}{m}$$

where

A is the iodine value (g iodine/100 g of sample);

V_1 is the volume of sodium thiosulfate solution used to titrate the test portion (cm^3);

V_0 is the volume of sodium thiosulfate solution used for the blank titration (cm^3);

m is the mass of the test portion (g);

c is the concentration of the sodium thiosulfate solution (mol/l);

12,69 is the atomic mass of iodine × 100/1 000.

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8 Precision

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8.1 The precision was determined by means of an interlaboratory trials programme. Three different materials (grades of HNBR) with different degrees of unsaturation were used in the programme. These were analysed in four laboratories on two different days one week apart. Duplicate analyses were run on each day.

8.2 The precision calculations to express repeatability and reproducibility were performed in accordance with ISO/TR 9272. Consult this for precision concepts and nomenclature.

8.3 Type 1/Class II interlaboratory precision was determined. Both the repeatability and the reproducibility determined are short-term, since a period of one week separates test results. For all data, $p = 4$, $q = 3$ and $n = 4$.

The precision analysis followed the general procedure set forth in ISO/TR 9272:1986. Each cell in Figure 1 of ISO/TR 9272:1986 contains four values (two test days, two results each day). The repeatability values contain two undifferentiated sources of variation, replicated within days and between days. The final values of the precision parameters are given in Table 2. These precision values shall not be used for acceptance/rejection of materials without documentation that they are for those materials and that the test protocols include this test method.

8.4 Repeatability: The repeatability r of the iodine value of HNBR has been established as the appropriate value of any parameter tabulated in Table 2. Two single test results obtained in the same laboratory, under normal test method procedures, that differ by more than this tabulated r shall be considered suspect and shall dictate that some appropriate investigative action be taken.

8.5 Reproducibility: The reproducibility R of the iodine value of HNBR has been established as the appropriate value of any parameter tabulated in Table 2. Two single test results obtained in separate laboratories, under normal test method procedures, that differ by more than this tabulated r shall be considered suspect and shall dictate that some appropriate investigative action be taken.

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Table 2 — Precision data

HNBR sample	Iodine value (mean) g/100 g	Within laboratory			Between laboratories		
		s_r	r	(r)	s_R	R	(R)
1	6,39	0,046	0,215	3,36	0,248	0,498	7,79
2	12,57	0,027	0,164	1,30	0,568	0,454	3,61
3	28,75	0,051	0,226	0,786	1,433	1,197	4,16

where

s_r is the within-laboratory standard deviation;

r is the repeatability (in measurement units);

(r) is the repeatability (as percentage of average for material);

s_R is the between-laboratory standard deviation;

R is the reproducibility (in measurement units);

(R) is the reproducibility (as percentage of average for material).

9 Test report

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The test report shall include the following information:

a) a reference to this International Standard;

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b) all details necessary for complete identification of the sample analysed;

c) the iodine value, expressed to the nearest 0,1 iodine value units.

d) the date of the analysis.

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Annex A
(normative)

Preparation of Wijs' solution

A.1 Weigh out, to the nearest 0,1 g, between 4,8 g and 5,2 g of iodine trichloride and place it in a 1 l brown bottle with a PTFE-lined screw cap.

A.2 Weigh out, to the nearest 0,1 g, 5,5 g of iodine and place it in a 1 l glass-stoppered conical flask containing 640 cm^3 of glacial acetic acid. Stopper the flask and swirl carefully to dissolve the iodine.

A.3 Carefully pour the iodine/acetic acid solution into the brown bottle containing the iodine trichloride. Stopper the bottle and swirl carefully to mix.

A.4 Affix a label to the bottle indicating the preparation date. Store the bottle in a dark place. The solution shall not be used more than 30 days after preparation.

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