## FINAL DRAFT

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# Epoxidised natural rubber — Specifications

Caoutchouc naturel époxydé — Spécifications

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<u>ISO/FDIS 24483</u> https://standards.iteh.ai/catalog/standards/sist/177319ed-8585-4ee4-8131-500658ae3a09/iso-fdis-24483

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## Foreword

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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 <a href="https://www.iso.org/directives">www.iso.org/directives</a>).

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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*.

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 <u>www.iso.org/members.html</u>.

## Introduction

Significant developments have taken place in the supply of raw natural rubber, especially in relation to the number of different grades that are technically specified. The constant viscosity (CV), latex (L) and gel content (LoV) grades have been added in addition to the initial grades covered in the first edition of ISO 2000. The development and availability of physical and/or chemically modified grades are now reported in the technical literature with the aim to expand natural rubber (NR) applications in rubber products. Demand and acknowledgment of these modified grades grow from year to year. On this note, it is important to establish a standard reference for the specification related to these modified natural rubber grades. In this document the emphasis is given to the epoxidised natural rubber (ENR) grade produced through a chemical modification route on natural rubber.

This document encompasses natural raw rubber specification for ENR produced through a chemical modification route known as epoxidation reaction. Typically, the most economical epoxidation reaction route is through the in situ peracid method. Through the process the peracid is formed from hydrogen peroxide and formic acid. The epoxidation reaction involves with substitution of the NR double bond structure (C=C) with epoxy ring structure (C-O-C) which later determines the grade of the ENR rubber. In general, any level of epoxidation can be produced according to the specific formulation. However, currently only two grades (25 mol% and 50 mol%) are available commercially. Different level of epoxidation in the ENR rubber specifies its usage in rubber product applications. The ENR acronym identifies and distinguishes the grades from other natural rubber grades. Unlike other technically specified rubber grades, the processing of the ENR material requires meticulous control during the process because every step is critical and might affect the properties of the final product.

This document encompasses some rubbers that are better defined elsewhere. For more precise specifications, reference can be made to such specifications in particular cases which may be found in national standards or in the literature of manufacturers of these ENR grades.

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## **Epoxidised natural rubber — Specifications**

### 1 Scope

This document specifies the physical and chemical requirements of epoxidised natural rubber (ENR) based on the epoxidation level of the natural rubber.

### 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 247-1, Rubber — Determination of ash — Part 1: Combustion method

ISO 289-1, Rubber, unvulcanized — Determinations using a shearing-disc viscometer — Part 1: Determination of Mooney viscosity

ISO 1656, Rubber, raw natural, and rubber latex, natural — Determination of nitrogen content

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

ISO 4660, Rubber, raw natural — Colour index test

ISO/DIS 5260<sup>1</sup>), Epoxidized natural rubber — Determination of epoxidation and ring opening level by NMR spectrometry

<u>ISO/FDIS 24483</u>

ISO 20299-2, Film for wrapping rubber bales — Part 2: Natural rubber

ASTM D3418, Standard Test Method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry

### 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

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

— IEC Electropedia: available at <u>https://www.electropedia.org/</u>

#### 3.1

### epoxidised natural rubber

ENR

natural rubber which has been chemically treated and modified through the addition of oxygen atom onto the double bond structure of the *cis*-1,4-polyisoprene to form three-membered ether (C-O-C) in a cyclic form by a process known as epoxidation reaction

<sup>1)</sup> Under preparation. Stage at the time of publication: ISO/DIS 5260.

# 3.2 whole field latex

**WF** latex material derived from *Hevea brasiliensis* which may be diluted but is not fractionated

[SOURCE: ISO 2000:2020, 3.5]

#### 3.3

### centrifuged latex

CF

latex material derived from Hevea brasiliensis, concentrated to 60 % dry rubber content using methods of centrifugation

### 3.4

### epoxidation level

level and/or degree of the epoxy groups (C-O-C) in the NR structure

#### 3.5

### ring opening level

level and/or degree of ring opening due to the formation of secondary by product from uncontrolled condition of epoxidation reaction in the natural rubber structure

### 4 Material composition

Epoxidised natural rubber (ENR) shall be graded based on the following raw materials:

- whole field latex;
- centrifuged latex.

The ENR is composed of a polyisoprene backbone with different levels of epoxy structure after being chemically modified in the latex stage and subsequently dried to a solid form.

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### 5 Grade structure

The ENR grade is based on the level of epoxidation and the type of material used in its production (see <u>Table 1</u>).

Raw material	Characteristic	Grades			
hole field latex treated with stabilisers	With specification on nitrogen value	ENR 25 FL			
and epoxidation agents	with specification on fift ogen value	ENR 50 FL			
ntrifuged latex treated with stabilisers	With no specification on nitrogen value	ENR 25			
and epoxidation agents	with no specification on introgen value	ENR 50			
OTE For the grades of ENR 25 and ENR 50, the numerals represent the mole percentage of the epoxide level i.e. 25 mold 50 mol% of the unsaturation in the rubber are converted through modification to the epoxide groups.					

#### Table 1 — Grades of ENR

## 6 Specification of requirement

Any specific values for physical and chemical properties shall be based upon the grade and type of raw materials according to <u>Table 2</u>.

Raw material	Latex concentrate		Field latex		Test method
Droportion	Grade		Grade		
Properties	ENR 25	ENR 50	ENR 25 FL	ENR 50 FL	
Ash content maximum	0.25	5 0,25	0,25	0,25	ISO 247-1
% (mass fraction)	0,25				
Nitrogen content maximum	NI / A	NI / A	0.15	0.15	
% (mass fraction)	N/A	N/A	0,15	0,15	ISO 1656
Epoxidation level	25 - 2	50 ± 2 2	25 ± 2	50 ± 2	- ISO 52601
% (mole fraction)	25 ± 2				
Ring opening level maximum	2.0	4,0	2,0	4,0	
% (mole fraction)	2,0				
Mooney viscosity	70 += 100 70 +	$70 \pm 100$	) to 100 70 to 100	70 to 100	ISO 289-1
ML (1+4) at 100 °C	70 to 100	/U to 100			
Glass transition temperature	45 + 2	-45 ± 2 -22 ± 2	-45 ± 2	-22 ± 2	ASTM D3418
°C	-45 ± 2				
Lovibond colour index maxi- mum (optional)	4,0	4,0	4,0	4,0	ISO 4660
NOTE N/A means not available.	TAND		DDFVI		

Table 2 — Specification of ENR

# 7 Sampling (standards.iteh.ai

The ENR shall be sampled in accordance with ISO 1795, unless otherwise agreed between the interested parties. ISO/FDIS 24483

Each sample derived from the lot shall comply with the requirements agreed for that grade of ENR.

## 8 Packaging

The ENR should normally be packaged in bales of nominal mass 33,3 kg or 35 kg (tolerance ±0,5 %).

NOTE 1 Since 36 bales of 33,3 kg make up a 1,2 tonne, it can be the preferred size.

Each bale shall be:

- identified;
- marked;
- wrapped either in polyethylene film as specified in ISO 20299-2 or in some other form of packaging as agreed between the interested parties.

NOTE 2 The thickness of non-strippable polyethylene film specified in ISO 20299-2:2017, 5.1, is 0,03 mm to 0,05 mm. However, on agreement between the interested parties, a maximum thickness of 0,065 mm can be used especially if the removal of the packaging film is desired.

## **Bibliography**

- [1] ISO 2000:2020, Rubber, raw natural Guidelines for the specification of technically specified rubber (TSR)
- [2] *Epoxidised natural rubber*, Technical Bulletin, Rubber Research Institute of Malaysia 1983
- [3] *Epoxyprene Epoxidised Natural Rubber*, Kumpulan Guthrie Berhad, Material information document No ENR01-07
- [4] *Ekoprena*, Publication Division, Malaysian Rubber Board, Bangunan Getah Asli, 148 Jalan Ampang, Kuala Lumpur 54050

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