INTERNATIONAL STANDARD

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Rubber, raw natural — Guidelines and requirements for technically specified low-protein natural rubber



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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).

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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 www.iso.org/members.html.

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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 rubber (CV), latex (l) and gel content (LoV) grades have been added in addition to the initial grades covered in ISO 2000. The development and availability of physical and/or chemically modified grades are now reported in the technical literature (see Annex A).

This document encompasses raw natural rubber, which has been subjected to a process of removal of naturally occurring proteins that is commonly and commercially referred to as deproteinised natural rubber (DPNR).

However, the processes employed for removal of the proteins from natural rubber are not complete. The measurement of these residual proteins and specifying limits for these proteins is necessary to specify these dry rubber grades of low protein rubber. Low protein natural rubber (LPNR) would identify and distinguish these grades from the latex grades and from a completely protein free natural rubber should it become available (see Annex B).

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

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Rubber, raw natural — Guidelines and requirements for technically specified low-protein natural rubber

1 Scope

This document provides guidelines and gives requirements on the specification of low protein natural rubber (LPNR). A grading system is proposed, based on the origin of the natural rubber content and differentiated by the two processes, enzymatic and non-enzymatic, applied for removal of the proteins.

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 248-1, Rubber, raw — Determination of volatile-matter content — Part 1: Hot-mill method and oven method

ISO 249, Rubber, raw natural — Determination of dirt content

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 2007, Rubber, unvulcanized — Determination of plasticity — Rapid-plastimeter method

ISO 2930, Rubber, raw natural Determination of plasticity retention index (PRI)

ASTM D5712, Standard Test Method for Analysis of Aqueous Extractable Protein in Latex, Natural Rubber, and Elastomeric Products Using the Modified Lowry Method

ASTM D6499, Standard Test Method for Immunological Measurement of Antigenic Protein in Hevea Natural Rubber (HNR) and its Products

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 https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

3.1

low protein natural rubber

natural rubber obtained by a deproteinisation process from the latex of *Hevea brasiliensis* and typically processed into block rubber, and having properties complying with the criteria for the grade concerned

3.2

constant viscosity rubber

CV

natural rubber whose viscosity is controlled, typically by treating it with viscosity-stabilizing agents prior to the drying process

3.3

whole field latex

WF

latex material derived from Hevea Brasiliensis which may be diluted but is not fractionated

4 Material composition

Low protein natural rubber (LPNR) shall be divided into the following two principal groups, based on

- a) the treatment process applied to the whole field latex to remove the proteins, and
- b) whose viscosity is controlled, typically by treating it with viscosity stabilizing agents prior to the drying process.

5 Grade structure

The grade structure of LPNR shall be based on the properties of the deproteinized latex natural rubber (DPLNR) and the type of material used in its production. See <u>Table 1</u>.

Table 1 — Grades of LPNR

Raw material	Characteristics	Grades
Whole field latex	With no specified viscosity -S	E-WFS
treated with an enzymatic protein extraction agent	With controlled viscosity	E-CV55 or E-CV65
Whole field latex	With no specified viscosity -S	NE-WFS
treated with a non- enzymatic protein extraction agent	With controlled viscosity	NE- CV55 or NE-CV65

CV: constant viscosity

E: enzymatic

NE: non enzymatic

WFS: whole field latex viscosity not specified

NOTE Several proteolytic enzymes have been reported for the removal of proteins.

Nonenzymatic agents for protein removal include fumed silica, urea and aluminium hydroxide.

6 Specification of requirement

Any specific values for physical and chemical properties shall be based upon the grade (see Table 2).

Table 2 — Typical properties of LPNR

Properties	Grade ^a						
	E-WFS	E-CV55	E-CV65	NE-WFS	NE-CV55	NE-CV65	Test method
Colour coding mark- er ^b	Green	Blue ^b	Blue ^b	Green	Blue ^b	Blue ^b	
Dirt retained on sieve							
maximum	0,03	0,03	0,03	0,03	0,03	0,03	ISO 249
% (mass fraction)							
Ash							
maximum	0,25	0,25	0,25	0,25	0,25	0,25	ISO 247-1
% (mass fraction)						2.D	
Nitrogen content						\$10.	
maximum	0,15	0,15	0,15	0,15	0,15	0,15	ISO 1656
% (mass fraction)					-C		
Volatile matter con-					1/2		
tent	0,5	0,5	0,5	0,5	Z 9,5	0,5	ISO 248-1
maximum	,	,	,		$Q_{\mathbf{x}}$		
% (mass fraction)				11/			
Initial plasticity (Po)	N/A	N/A	N/A	N/A	N/A	N/A	ISO 2007
minimum		11,11					
Plasticity retention index (PRI)	40	40	400 1	40	40	40	ISO 2930
minimum			7,				
Mooney viscosity	oney viscosity N/A		V	NT /A	FF . Fc	(F , Fc	ICO 200 1
ML(1+4) at 100 °C	N/A	55 ± 5°	65 ± 5 ^c	N/A	55 ± 5 ^c	65 ± 5 ^c	ISO 289-1
Extractable protein	100	100	100	100	100	100	ASTM D5712 ^d
maximum (μg/g)							
Antigenic protein	(). ()	10	_	_	_	10	ASTM D6499d
maximum (μg/g)e							

a The raw material is given in Table 1.

CV: constant viscosity

E: enzymatic

NE: non enzymatic

WFS: whole field latex viscosity not specified

N/A: not applicable

7 Sampling

LPNR shall be sampled in accordance with ISO 1795, unless otherwise agreed between interested parties.

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

b Suggested colours.

^c Other viscosity levels may be agreed between interested parties for specific applications, e.g. low viscosity CV40 for adhesives. In lieu of PRI the application of a maximum P of 12 units may also be agreed between interested parties.

d ASTM Standard in the absence of an equivalent ISO Standard.

e Grade suitable for food and medical application.

Packaging

LPNR should normally be packed in bales of nominal mass 33,30 kg \pm 0,17 kg.

36 bales are packaged in 1,2 metric tonne wooden crates.

The nominal dimensions of the bale are 330 mm x 670 mm x 170 mm.

On agreement between the interested parties, bales of other masses and nominal dimensions may be used.

Each bale shall be:

- identified;
- marked;
- wrapped either in polyethylene film compatible with natural rubber, having a preferred thickness of 0,030 mm to 0,050 mm, density of 0,92 g/cm³, melting point 110 °C or a Vicat softening point lower than 95 °C (as set out in ISO 20299-2); or
- in some other form of packaging as agreed between interested parties.

ess of children the full Processing the children the full Processing the children the full Processing to the children the chil 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.

Annex A

(informative)

Low protein natural rubber (LPNR)

A.1 General

This annex is an edited extract from RRIM Technology Bulletin No 11[3].

Low protein natural rubber (LPNR) is a purified form of natural rubber (NR) from which most of the ash and protein components have been removed. The rubber is produced under very closely controlled process conditions. It contains about 96 % rubber hydrocarbons compared to about 93 % for normal natural rubber grades. The removal of these non-rubber components confers special attributes to the rubber which enhance its value in certain specialized applications.

A.2 LPNR CV production process

- **A.2.1** LPNR is prepared by treating field latex either with an enzyme or non-enzymatic materials.
- **A.2.2** The enzyme process uses a proteinase to hydrolyse the proteins in the latex which are then washed away during processing. Ammonia, a nontionic surfactant, a proteinase and hydroxylamine neutral sulfate (HNS) are added into bulked field latex and allowed to react for 72 h in a stainless-steel conical bottom reaction tank. After the completion of the enzymatic hydrolysis reaction, the reacted latex is neutralized with dilute formic acid and coagulated by steam in a specially designed steam column coagulator. The resultant coagulatare then processed in a continuous fashion through a series of crepers and finally chopped into small crumbs in a shredder. The crumbs are pumped through a static screen before entering the dryer boxes. The wet crumbs are then dried at about 85 °C for about 6 h to 8 h in a hot air dryer. After cooling, the crumbs are weighed, pressed, baled and packed according to ISO 2000.
- **A.2.3** For the non-enzymatic process, there are several processes reported in the technical and commercial literature. Preserved whole field latex is treated with a protein extraction agent and allowed to react from periods ranging from 24 h to 72 h depending on the specific process employed. The protein extraction agents include Sodium lauryl sulfate (or other anionic surfactants), urea, fumed silica and metal hydroxides. The latex can be coagulated using either formic or acetic acid. The resultant coagula are then water-washed in a continuous fashion through a series of crepers and finally chopped into small crumbs in a shredder. The crumbs are pumped through a static screen before entering the dryer boxes. The wet crumbs are then dried at about 85 °C for about 6 h to 8 h in a hot air dryer. After cooling, the crumbs are weighed, pressed, baled and packed according to ISO 2000.

A.3 LPNR — Grades

The two following principal grades of LPNR are available:

- LPNR CV Viscosity stabilized between 60 to 70 Mooney units;
- LPNR S No viscosity stabilization feature; the initial viscosity is between 60 to 70 Mooney units.
 Owing to the eradication of non-rubber content, the LPNR S has very much less storage hardening than the normal NR.

Both grades are produced either by the enzyme or non-enzyme treatment processes described in A.2.2 and A.2.3.

A.4 Summary of the characteristics of LPNR

- Very low protein content.
- Very low dirt and ash contents.
- Low volatile matter content.
- Light colour.
- Negligible nitrosamines.
- Very low antigen content.

A.5 Vulcanization and applications

These raw rubber characteristics are reflected in the following valuable attributes of LPVR vulcanisates, particularly when compounded using soluble sulfur-accelerator vulcanising systems.

- Lower creep and stress relaxation.
- Lower water absorption.
- Greater consistency in modulus under conditions of variable humidity.

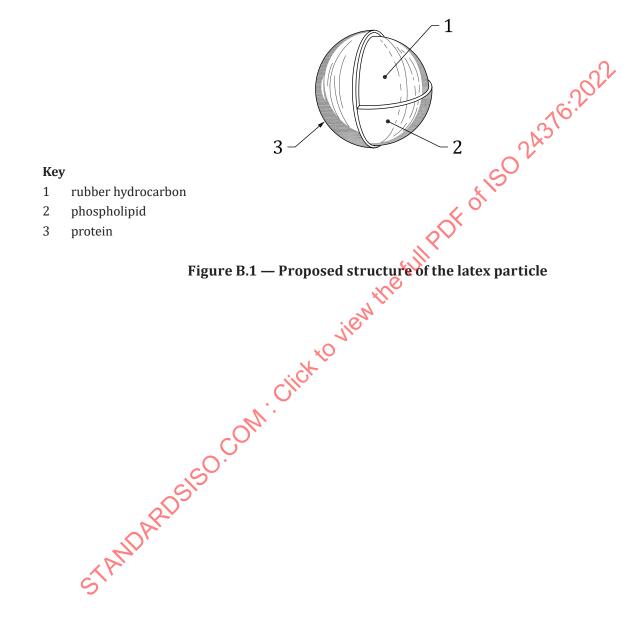
Established and potential areas of application are given in Table A.

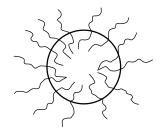
Table A.1 — Typical properties of LPNR

Application	Property
Low stress relaxation and creep	Seals, joint rings, hydro-mounts
Low water absorption	Undersea applications
Good dynamic properties	Anti-vibration mountings
Low protein	Medical and food applications

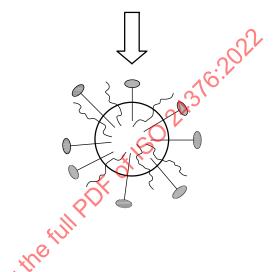
Annex B (informative)

Examples of the removal of proteins from raw natural rubber





+ surfactant + protease



Key



latex particle

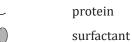


Figure B.2 — Removal of proteins with proteolytic enzymes