

---

---

**Rubber, raw synthetic —  
Determination of the molecular-mass  
distribution of solution polymers by  
gel permeation chromatography**

*Caoutchouc synthétique brut — Détermination de la répartition de la  
masse moléculaire pour les caoutchoucs polymérisés en solution par  
chromatographie par perméation de gel*



**COPYRIGHT PROTECTED DOCUMENT**

© ISO 2016, Published in Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office  
Ch. de Blandonnet 8 • CP 401  
CH-1214 Vernier, Geneva, Switzerland  
Tel. +41 22 749 01 11  
Fax +41 22 749 09 47  
copyright@iso.org  
www.iso.org

<b>Contents</b>		Page
<b>Foreword</b> .....		<b>iv</b>
<b>1 Scope</b> .....		<b>1</b>
<b>2 Principle</b> .....		<b>1</b>
<b>3 General</b> .....		<b>1</b>
<b>4 Reagents and materials</b> .....		<b>2</b>
<b>5 Apparatus</b> .....		<b>3</b>
<b>6 Analytical conditions</b> .....		<b>5</b>
<b>7 Procedure</b> .....		<b>5</b>
7.1 Solvent degassing.....		5
7.2 Calibration.....		5
7.3 Preparation of test solution.....		7
7.4 Analysis.....		8
<b>8 Expression of results</b> .....		<b>8</b>
<b>9 Precision</b> .....		<b>9</b>
<b>10 Test report</b> .....		<b>9</b>
<b>Annex A (informative) Molecular-mass parameters determined by instrumental software</b> .....		<b>10</b>
<b>Annex B (informative) Calculation of molecular-mass parameters by manual procedure</b> .....		<b>14</b>
<b>Annex C (informative) Comparison of results obtained by automatic procedure (software) and manual procedure</b> .....		<b>17</b>
<b>Annex D (informative) Precision (only for instrumental software procedure)</b> .....		<b>18</b>
<b>Bibliography</b> .....		<b>20</b>

## 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. [www.iso.org/directives](http://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. [www.iso.org/patents](http://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 second edition cancels and replaces the first edition (ISO 11344:2004), which has been technically revised by replacing the hazardous *o*-dichlorobenzene with BHT (butylated hydroxy toluene) in the procedure. It also incorporates the Technical Corrigendum ISO 11344:2004/Cor.1:2008.

# Rubber, raw synthetic — Determination of the molecular-mass distribution of solution polymers by gel permeation chromatography

**WARNING 1** — 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.

**WARNING 2** — Certain procedures specified in this International Standard might involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

## 1 Scope

This International Standard describes a method for the determination of the molecular mass, expressed as polystyrene, and the molecular-mass distribution of polymers produced in solution which are completely soluble in tetrahydrofuran (THF) and which have a molecular-mass range from  $5 \times 10^3$  to  $1 \times 10^6$ .

It is not the purpose of this International Standard to explain the theory of gel permeation chromatography.

## 2 Principle

The molecular components of a polymer are separated on the basis of macromolecule size on a gel permeation column. A known quantity of a dilute solution of the polymer is injected into a stream of solvent, which carries it through the column at a constant rate. The concentration of the separated molecular components in the solvent stream is measured by a suitable detector. Through the use of a calibration curve, both the number-average molecular mass ( $M_n$ ) and mass-average molecular mass ( $M_w$ ) of the material analysed can be determined from the retention time and the corresponding concentration.

## 3 General

**3.1** Gel permeation chromatography (GPC), which is also known as size exclusion chromatography (SEC), is a particular type of liquid chromatography which allows the separation of the various components of a polymer based on molecular size.

**3.2** The molecules of a polymer do not all have the same mass, but comprise a range of different masses. For this reason, the usual concept of molecular mass is not applicable to polymeric materials. Instead, different average molecular masses are determined as shown in [Table 1](#).