
**Rubber, raw natural — Determination of
average molecular mass and molecular-
mass distribution by size exclusion
chromatography (SEC)**

*Caoutchouc naturel brut — Détermination de la masse moléculaire
moyenne et de la répartition des masses moléculaires par
chromatographie d'exclusion stérique (SEC)*



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Foreword

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Rubber, raw natural — Determination of average molecular mass and molecular-mass distribution by size exclusion chromatography (SEC)

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1 Scope

This International Standard specifies a method of determining the average molecular mass and the molecular-mass distribution of raw natural rubber dissolved in tetrahydrofuran. A set of polystyrene standards is used for calibration purposes (i.e. the method is a relative one).

An alternative method, using cyclohexane as solvent and polyisoprene standards, is included in an informative annex.

2 Principle

Dried natural rubber is dissolved in tetrahydrofuran at room temperature. The solution is filtered to remove “gel” (slightly crosslinked rubber) and other insoluble materials. The filtrate is used to determine the molecular mass by size exclusion chromatography^[1]. From the chromatogram, the number-average molecular mass (M_n), the mass-average molecular mass (M_w) and a polydispersity value (M_w/M_n) are calculated.

3 Materials

3.1 The recommended solvent is HPLC-grade tetrahydrofuran (THF). If analytical-grade THF is used, 0,5 % of butylated hydroxytoluene (BHT) shall be added to the solvent as an antioxidant. The BHT is strongly retained and acts as a marker for the end of the chromatogram. It also allows minor variations in run time to be corrected, as well as indicating when a serious change in the column conditions has occurred.

3.2 The solvent is filtered through a polytetrafluoroethylene (PTFE) membrane (4.2) before use.

3.3 A set of polystyrene standards with proper traceability shall be used, typically covering an M_w range from $6,5 \times 10^3$ to $1,06 \times 10^7$. An example of a suitable set of polystyrene standards is given in Table 1.

Table 1 — Polystyrene standards

No. of standard	M_p	M_w/M_n
1	$3,55 \times 10^5$	1,08
2	$7,06 \times 10^5$	1,12
3	$2,89 \times 10^6$	1,36
4	$3,84 \times 10^6$	1,30
5	$4,48 \times 10^6$	1,47
6	$5,48 \times 10^6$	1,40
7	$6,77 \times 10^6$	1,37
8	$8,42 \times 10^6$	1,33