INTERNATIONAL STANDARD

ISO 289-4

second edition
2014-01-01

Rubber, unvulcanized — Determinations using a shearing-disc viscometer —

Part 4: Determination of the Mooney stress-relaxation rate

Caoutchouc non vulcanisé — Détermination utilisant un consistomètre à disque de cisaillement —

Partie 4: Détermination du taux de relaxation de contrainte Mooney

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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 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 (see 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-289-4:2003), of which it constitutes a minor revision. The following are the changes:

- Annex A has been updated
- Annex B has been added
- Annex C has been added

ISO 289 consists of the following parts, under the general title *Rubber, unvulcanized — Determinations using a shearing-disc viscometer*:

— *Part 1: Determination of Mooney viscosity*

— *Part 2: Determination of pre-vulcanization characteristics*

— *Part 3: Determination of the Delta Mooney value for non-pigmented, oil-extended emulsion-polymerized SBR*

— *Part 4: Determination of the Mooney stress-relaxation rate*

In this corrected version of ISO 289-4:2003, the following changes have been incorporated:

- page iv: corrected spelling of the TC title;
- pages v, 1, 2, 3, 6, 7: modifications of bibliographic reference numbers;
- page 2: addition of the word "thickness" to NOTE 2;
- page 3: change of symbol for Mooney units — from *M* to *T*;
- page 4: Figure 1, addition of symbol *t*, to the key for the abscissa;
- page 6: subclause A.2.1, new text;
- page 8: Table A.1, deletion of the second line of the table title and also of the word "NOTE" above the table footnotes;
- pages 13 and 14: Bibliography, change in order and numbering of references, deletion of former reference [6], detailed changes in former references

Introduction

Mooney viscosity, as defined in ISO 289-1, is one of the most widely accepted rubber characterization parameters. However, Mooney viscosity alone is usually insufficient to guarantee that other rheological properties are well controlled [1]. It does not give any information about the elasticity of raw and unvulcanized rubbers [2]. Viscosity and elasticity can change independently, therefore it is important to have test procedures available that are able to measure both properties independently.

Mooney viscosity is measured at one specific shear rate and rubbers exhibit shear rate-dependant viscosity. Sophisticated test equipment to measure the viscosity of a rubber as a function of the shear rate is available. Generally speaking, this type of equipment, its operation and the interpretation of the results is too complicated to be used as a standard quality control tool at present.

As described in the literature [3] the Mooney stress-relaxation is related to the elastic effects in the rheology of unvulcanized rubbers. It can be measured relatively easily and only takes a few seconds extra at the end of a standard Mooney viscosity measurement. The MSR parameter is independent from Mooney viscosity.

Mooney stress-relaxation, combined with the conventional Mooney viscosity, gives a better description of the visco-elastic behaviour of uncompounded as well as compounded, unvulcanized rubbers [4]. Mooney stress relaxation measurements have been proposed as quality control tool [5] [6].

The short interval method as described in this part of ISO 289 is a refinement of the evaluation procedures for Mooney stress relaxation measurements. Short interval evaluation leads to higher reproducibility compared to using an extended interval.

Using a short interval, a major parameter relevant to rubber rheology can be obtained from Mooney stress relaxation experiments *viz.* the Mooney stress-relaxation rate (MSR) i.e. the rate of decay of torque versus time [7] [8] [9] [10].

The Mooney stress-relaxation rate also has been referred to as "slope", where the latter is sometimes presented as a positive and sometimes as a negative value. As the method described in this part of ISO 289 uses a specific evaluation interval and the parameter is always referred to as a positive value, a new distinctive name has been chosen [11] [12] [13] [14].

Data are available to show that the described method distinguishes polymers (EPDM) with different high molecular weight fractions despite the short evaluation interval.

Rubber, unvulcanized — Determinations using a shearing-disc viscometer —

Part 4: Determination of the Mooney stress-relaxation rate

1 Scope

This part of ISO 289 specifies a method of use of a shearing-disc viscometer for measuring the Mooney stress-relaxation rate (MSR) of uncompounded or compounded unvulcanized rubbers, characterizing the elastic response of those materials next to the viscous response as measured by the Mooney viscosity. The intended use of ISO 289-4 is on Quality Control measurements.

2 Normative references

The following referenced documents are indispensable for the application 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 289-1:1994, *Rubber, unvulcanized — Determinations using a shearing-disc viscometer — Part 1: Determination of Mooney viscosity*

3 Terms and definitions

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

3.1
Mooney stress-relaxation rate
MSR
absolute value of the slope of the linear regression line of the log(torque) versus log(time) plot over a specified time interval after stopping the rotor at the end of a Mooney viscosity measurement

NOTE The MSR measurement is a stress relaxation measurement which covers a broad spectrum of relaxation times and is sensitive to polymer structure at a specified relaxation time interval.

4 Principle

The test consists of determining the decay of the Mooney torque immediately after the determination of the Mooney viscosity. After abruptly stopping the rotor at the end of the Mooney viscosity measurement, the decrease in torque is recorded as a function of time. The rate of change of the torque is evaluated over a short time interval assuming power law validity, in accordance with theoretical predictions [15].

5 Apparatus

The apparatus specified in, and calibrated in accordance with ISO 289-1 shall be used. Furthermore the apparatus shall be able to stop the rotation of the disc within 0,1 s, reset the zero torque point to the static zero for a stationary rotor and record the torque at least every 0,2 s after stopping the rotor.

NOTE 1 There is a difference in zero torque for a stationary and a rotating rotor. Resetting of the zero torque point for the rotating rotor before every measurement is recommended. This results in a negative torque signal during the preheating time as can be seen in Figure 1.

NOTE 2 The use of a barrier film is recommended. The polymer type and the thickness of such a film might affect ~~have an influence on~~ the test results as described in ISO 289-1. ~~This part of ISO 289 was developed using a polypropylene film of 20 µm thickness, with a DSC peak temperature of 161 °C (second heating curve).~~

6 Preparation of test piece

Run a Mooney viscosity test as described in ISO 289-1.

7 Temperature and duration of the test

Use the test conditions as described in ISO 289-1.

8 Procedure

Conduct the test following the procedure described in Clause 7 of ISO 289-1~~:1994~~.

If the viscosity has not been recorded continuously, plot the observed Mooney viscosity values as specified in ISO 289-1.

NOTE 1 An automatic recorder is strongly recommended. The use of specialized data acquisition software is preferred in order to enable automated calculations.

At the end of the viscosity test, stop the rotation of the disc within 0,1 s, reset the zero torque point to the static zero for a stationary rotor and record the torque at least every 0,2 s.

NOTE 2 Resetting torque to a static zero is necessary because the dynamic zero used for the viscosity test would result in a negative torque value once the material has completely relaxed with a stationary disc. The relaxation of torque for most polymers is so rapid that stopping the rotor, resetting zero and recording the relaxing torque must be controlled automatically.

The relaxation data shall be collected starting 1,6 s after the rotor is stopped, and continuing until 5,0 s after the rotor is stopped. This normally gives a total of 18 data points. A typical chart of a Mooney viscosity test followed by a stress relaxation test is shown in Figure 1.

NOTE 3 The use of different evaluation intervals and or different data sampling schemes result in different Mooney stress-relaxation rate values. Longer evaluation intervals may result in increased errors. This is due to the lower signal to noise ratio at progressing relaxation times. Most of the work done to develop this part of ISO 289 has been based on EPDM. It might be anticipated that for other polymers different evaluation intervals and/or different sampling schemes are more appropriate [16] [17]. Deviations from this document should be agreed upon between the supplier and customer and always mentioned in the test report.

9 Calculation and expression of results

Analysis of Mooney stress-relaxation rate data (torque versus time data) consists of a) developing a plot of torque (Mooney units) versus time in seconds in a log-log plot as shown in Figure 2, and b) calculating the constants of the power law model of material response, as represented by Equation (1).

$$T = k(t)^{\alpha} \tag{1}$$

where

T gives the Mooney units (torque) during the stress relaxation test;

k is a constant equal to the torque in Mooney units 1 s after the rotor is stopped;

t is the time after rotor stop, in seconds;

α is an exponent that determines the rate of stress relaxation.

If Equation (1) is transformed by taking the log of both sides, Equation (2) is obtained:

$$\log T = \alpha(\log t) + \log k \tag{2}$$

This has the form of a linear regression equation where α equals the slope, $\log k$ equals the intercept and $\log T$ and $\log t$ correspond respectively to the dependent and independent variables. In a plot of $\log T$ versus $\log t$, as shown in Figure 2, the slope of the graph, ($\log T/\log t$), is equal to α. The absolute value of the slope, $|\alpha|$, rounded to the nearest third decimal is the Mooney stress-relaxation rate.

Report the results of a typical test in the following format:

MSR = 0,941 ± 0,006

NOTE The number 0,006 in this expression stands for the standard error as calculated as part of the regression analysis. It is an estimation of the random error on the MSR value. It is calculated as:

$$\sqrt{\frac{\frac{1}{n-2}\sum e_i^{2}}{\sum (X_i - \overline{X})^{2}}}$$

where

e_i is the difference between measured value and the estimated value based on the linear regression, the so-called residual;

n is the number of measurements [18].

The MSR should always be reported in combination with the Mooney viscosity.

10 Test report

The test report shall include the following information.

a) all of the information described in Clause 10 of ISO 289-1:1994;

b) reference to this part of ISO 289, i.e. ISO 289-4;

c) manufacturer and model of the viscometer used;

d) relaxation time interval used if it differs from the one stated in Clause 8;

e) Mooney stress-relaxation rate (MSR) rounded to the nearest third decimal;

f) standard error for the MSR as calculated as part of the regression analysis rounded to the nearest third decimal;

g) any operation not included in this part of ISO 289 or regarded as optional.

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Preview:
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with a preview included in it.
Comment:
This EPS picture will print to a
PostScript printer, but not to
other types of printers.

Key

1 preheating time
2 Mooney viscosity part
3 stress relaxation part

X time, t, s
Y torque, T, Mooney units

Figure 1 — Mooney viscosity curve with Mooney stress relaxation part

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with a preview included in it.
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PostScript printer, but not to
other types of printers.

$y = -0{,}941\,4\,x + 1{,}172\,4$; $R^2 = 0{,}999\,4$; standard error = 0,005 9

X log (t)
Y log (T)

Figure 2 — Mooney stress-relaxation rate parameters

Annex A
(informative)

Precision statement

A.1 General ~~The test programme~~

The precision calculations to provide repeatability and reproducibility values were performed in accordance with ISO/TR 9272~~:2005~~[19], the guidance document for ISO/TC 45 test methods. Precision concepts and nomenclature are also given in ISO/TR 9272.

~~The interlaboratory test programme (ITP) for precision evaluation for Mooney stress-relaxation rate was conducted in 2001 using the procedures and guidelines as described in ISO/TR 9272.~~

~~The ITP was conducted with 14 polymers. Polymers 1 to 6 were EPDM grade materials and Polymers 7 to 14 were non-EPDM materials. Some details on these polymers are listed in Table A.1. Six laboratories participated in the ITP and Type 1 precision was evaluated for Mooney stress-relaxation rate, with one measurement on each of three separate test days within the time span of two weeks.~~

~~The precision results as determined by this ITP may not be applied to acceptance or rejection testing for any group of materials or products without documentation that the results of this precision evaluation actually apply to the products or materials tested.~~

A.2 Programme details ~~Results~~

A.2.1 ~~**General**~~ The first interlaboratory test programme (ITP) was organized and conducted in 2001. The Mooney stress-relation rate (MSR, single measurements) were made on three separate test days within the time span of two weeks.

A total of 6 laboratories participated in this ITP. A type1 precision evaluation with outliers replacement according the procedures of ISO/TR 9272 was carried out. Some details of the polymers are listed in Table A.1. An heat-stable polypropylene film of 0,020 mm was used.

~~The precision results are given in Table A.1, for each of the 14 polymers where the precision values are sorted for each category by mean stress-relaxation rate. Repeatability and reproducibility statements are given in A.2.2 and A.2.3.~~

A.2.2 ~~**Repeatability**~~ A second ITP was carried out on 2012. The MSR tests (single measurements) were made on two separate days (one week interval) in May 2012.

This second ITP corresponds to a type 1 precision evaluation (without outlier replacements) with no preparation or processing steps in the participation laboratories. A total of 14 laboratories participated. Some details of the polymers are listed in Table A.2. An heat-stable polyester film of 0,025 mm was used.

~~The repeatability, or local domain precision, of this test method has been established by the values found in Table A.1, for each of the listed polymers. Two single test results (obtained by the proper use of this part of ISO 289) that differ by more than the tabulated values for *r*, in measurement units and (*r*), in percent, shall be considered as suspect, i.e. to have come from different populations. Such a decision suggests that some appropriate investigative action be taken.~~

~~In general the repeatability (*r*), is in the 1 % to 6 % range. As an average, the non-EPDM materials have a slightly better repeatability than the EPDM materials.~~

~~A.2.3 Reproducibility~~

~~The reproducibility, or global domain precision, of this test method has been established by the values found in Table A.1, for each of the listed polymers. Two single test results obtained in different laboratories (by the proper use of this part of ISO 289) that differ by more than the tabulated values for *R*, in measurement units and (*R*), in percent, shall be considered as suspect, i.e. to have come from different populations. Such a decision suggests that some appropriate investigative action be taken.~~

~~The reproducibility (*R*), is in the 3 % to 8 % range with one exception — EPDM 5, which has a value of 16 %. There appears to be no difference in overall reproducibility for the two categories of polymers.~~

A.3 Precision results ~~Inter-laboratory agreement~~

~~Although all laboratories had good agreement for average slope values for all 14 polymers (only one exception among all labs), two of the laboratories generated outlier values (at the 5 % significance level) for within-lab standard deviations for several of the polymers. These outlier values were replaced with special replacement values by the procedures as outlined in ISO/TR 9272 [13]. For an ITP that has a bare minimum of participating laboratories (i.e. 6) the treatment of outliers by deletion, reduces the degrees of freedom for precision evaluation and therefore seriously compromises the final precision. Thus for any ITP with a bare minimum of participating lab oratories, the outlier replacement option is required. This option replaces the outlying value(s) with lower replac ement value(s), for either the averages or standard deviations, as obtained by a special procedure that maintains the distributional character of the databa se as generated by the program.~~

The precision results are listed in Tables A.1 and A.2

The precision evaluation of 2001 showed compared to the ITP 2012 in generally better precision results for all materials, in particularly for the reproducibility (*R*). The reason for the reduced (*R*) could be divers, differences in selected polymers, equipment, way of stopping the motor, calculation of the MSR rate by the equipment software and evaluation method.

Additionally, another precision evaluation was performed based on the original MSR rate values and on the raw interval data obtained from 8 of the 14 participants in the second ITP. Form these raw data the MSR rate values for each participating laboratory were recalculated. The manually recalculated MSR rate values deviated a little bit in nearly all cases from the originally values. The precision calculation was in both cases carried out according a type 1 precision evaluation as described in paragraph A.2.2.The results are listed in Table A3.1 and A3.2. In generally a somewhat better repeatability (*r*) was found for the MSR rate values based on the 8 originally values, but a large repeatability (*r*) for sample EPDM-1 based on the recalculated MSR rate values. In generally, the reproducibility (*R*) based on the recalculated MSR rate values improved in most cases. Care should be taken when comparing results obtained with different equipment.

A.4 Bias

Bias is the difference between a test value and a reference or true value. Reference values do not exist for this test method, therefore bias cannot be determined.

Legend to the Tables

NOTE

Sr is the within-laboratory standard deviation (in measurement units)

r is the repeatability (in measurement units)

(*r*) is the repeatability (in percent of mean level)

SR is the between-laboratory standard deviation (for total between laboratory variation in measurement units)

R is the reproducibility (in measurement units)

(*R*) is the reproducibility (in percent of mean level)

a number of labs in the revised database after option 1 deletion

Table A.1 — Type 1: Precision for Mooney stress-relaxation rate
Precision values sorted for each category — By mean stress-relaxation rate (Evaluation 2001)

Polymer	ML	MSR							
	Mean level	Mean level	Within laboratories			Between laboratories			Number of labs[a]
			Sr	*r*	(*r*)	*SR*	*R*	(*R*)	
EPDM-1	76,9	0,393	0,001 3	0,003 7	0,95	0,004 9	0,013 7	3,49	6
EPDM-2	72,8	0,447	0,001 5	0,004 1	0,91	0,005 1	0,014 2	3,18	6
EPDM-3	80,2	0,538	0,002 4	0,006 8	1,26	0,006 4	0,017 6	3,31	6
EPDM-4	55,5	0,750	0,010 7	0,030 1	4,01	0,016 1	0,045 1	6,01	6
EPDM-5	24,5	0,750	0,012 6	0,035 2	4,69	0,042 5	0,119 1	15,88	6
EPDM-6	46,7	0,931	0,018 8	0,052 7	5,66	0,024 9	0,069 6	7,48	6
IIR-1	78,2	0,234	0,002 2	0,006 2	2,64	0,003 3	0,009 2	3,95	5
IIR-2	51,7	0,623	0,004 7	0,013 2	2,12	0,007 1	0,020 0	3,21	5
SBR 1712	54,2	0,334	0,002 1	0,005 9	1,77	0,004 1	0,011 5	3,45	6
SBR 1500	49,6	0,415	0,002 7	0,007 7	1,86	0,006 9	0,019 2	4,64	6
NBR-1	77,9	0,343	0,002 4	0,006 7	1,95	0,005 1	0,014 2	4,13	6
NBR-2	81,6	0,373	0,001 1	0,003 1	0,84	0,005 2	0,014 5	3,88	6
NBR-3	30,7	0,424	0,004 8	0,013 5	3,18	0,011 9	0,033 2	7,84	6
NBR-4	32,3	0,571	0,003 7	0,010 5	1,83	0,016 0	0,044 9	7,87	6

Table A.2 — Type 1: Precision for Mooney stress-relaxation rate
Precision values sorted for each category — By mean stress-relaxation rate (Evaluation 2012)

Polymer	ML	MSR							
	Mean level	Mean level	Within laboratories			Between laboratories			Number of labs[a]
			Sr	*R*	(*r*)	*SR*	*R*	(*R*)	
EPDM-1	22,1	0,682	0,006	0,017	2,4	0,062	0,174	25,4	11
EPDM-2	33,9	0,580	0,018	0,052	8,9	0,042	0,117	20,2	12
IIR-1	55,5	0,578	0,014	0,039	6,8	0,045	0,126	21,7	13
IIR-2	52,1	0,639	0,012	0,035	5,4	0,040	0,113	11,7	11
EPDM-3	64,5	0,484	0,008	0,022	4,5	0,024	0,069	14,2	11
EPDM-4	62.8	0,687	0,005	0,015	2,1	0,041	0,115	16,8	9
EPDM-5	80,2	0,440	0,005	0,014	3,0	0,024	0,068	15,4	11
EPDM-8	74,8	0,875	0,013	0,037	4,2	0,068	0,192	21,9	10

Table A.3.1 — Type 1: Precision for Mooney stress-relaxation rate
Precision values sorted for each category — By mean stress-relaxation rate (Evaluation 2012)

Polymer	ML	MSR							
	Mean level	Mean level	Within laboratories			Between laboratories			Number of labs[a]
			Sr	*r*	(*r*)	*SR*	*R*	(*R*)	
EPDM-1	22,0	0,676	0,007	0,019	2,8	0,061	0,172	25,4	6
EPDM-2	34,0	0,574	0,007	0,018	3,2	0,041	0,115	20,0	6
IIR-1	55,7	0,560	0,004	0,010	1,9	0,028	0,079	14,0	6
IIR-2	52,0	0,636	0,008	0,023	3,6	0,036	0,100	15,6	6
EPDM-3	64,5	0,484	0,005	0,013	2,8	0,032	0,088	18,3	6
EPDM-4	62,9	0,702	0,003	0,009	1,3	0,038	0,105	15,0	5
EPDM-5	80,3	0,440	0,002	0,007	1,5	0,028	0,078	17,7	6
EPDM-8	74,8	0,877	0,015	0,043	4,9	0,054	0,153	17,4	6
NOTE Precision data calculated on original data from participants									

Table A.3.2 — Type 1: Precision for Mooney stress-relaxation rate
Precision values sorted for each category — By mean stress-relaxation rate (Evaluation 2012)

Polymer	ML	MSR							
	Mean level	Mean level	Within laboratories			Between laboratories			Number of labs[a]
			Sr	*r*	(*r*)	*SR*	*R*	(*R*)	
EPDM-1	22,0	0,680	0,029	0,080	11,8	0,050	0,139	20,5	7
EPDM-2	34,0	0,583	0,013	0,037	6,4	0,030	0,083	14,3	7
IIR-1	55,7	0,556	0,004	0,010	1,8	0,014	0,040	7,2	6
IIR-2	52,0	0,614	0,008	0,021	3,5	0,014	0,038	6,2	6
EPDM-3	64,5	0,491	0,013	0,037	7,5	0,015	0,042	8,6	6
EPDM-4	62,9	0,702	0,010	0,029	4,1	0,033	0,092	13,2	7
EPDM-5	80,3	0,449	0,004	0,010	2,3	0,018	0,051	11,3	6
EPDM-8	74,8	0,887	0,017	0,047	5,3	0,042	0,118	13,2	7
NOTE Precision data based on participants raw data (manually recalculation)									

Annex B
(informative)

Motor influence

B.1 General

The possible influence of different equipment is described in this annex. The Mooney relaxation measurements of 2 test devices from the same manufacturer equipped with different motors were compared.

B.2 Results and discussion

The results are listed in table B.1. The measurements were carried out duple with 2 different polymers and the Mooney stress-relaxation rate values were calculated by the supplied (machine) software. In these examples a decrease of MSR values up to 12% were found.

Table B.1 — Mooney stress-relaxation rate values

	Motor type 1		Motor type 2		Difference	
Polymer	MI(1+4)125	MSR-T1	MI(1+4)125	MSR-T2	MSR-T1 − MSR-T2	
	MU	MSR	MU	MSR	ΔMSR	%
EPDM	77,2	0,516	76,9	0,458	0,062	11,9
	77,2	0,514	77,1	0,449	0,065	12,6
IIR	53,9	0,547	53,5	0,506	0,041	7,5
	54,3	0,540	53,6	0,504	0,035	6,4

In Figure B.1 the decrease of Mooney values in the first 5 seconds after stopping the measurement is given for one EPDM measurement. This figure clearly shows the initial more faster decrease of the Mooney value for rotor type 2 compared with rotor type1, resulting in a lower slope value for motor type 2.

These observations indicates that care should be taken when comparing MSR data measured on different devices. In principle the initial part of the MSR curves should be analysed too.

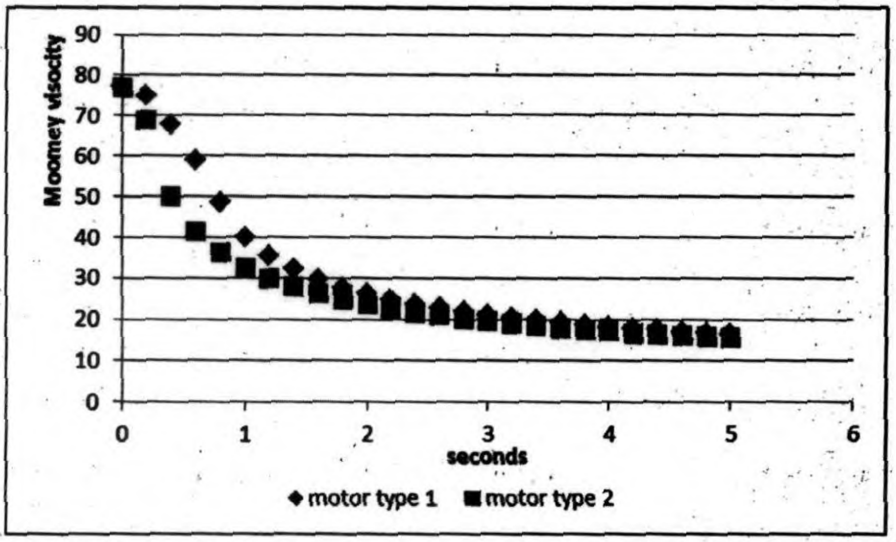

Figure B.エラー! スイッチの指定が正しくありません。.1 — B. The first 5 seconds of the Mooney stress relaxation part of the Mooney Curve

Annex C
(normative)

Calibration schedule

C.1 Inspection

Before any calibration is undertaken, the condition of the items to be calibrated shall be ascertained by inspection and recorded in any calibration report or certificate. It shall be reported whether calibration was carried out in the "as-received" condition or after rectification of any abnormality or fault.

It shall be ascertained that the apparatus is generally fit for the intended purpose, including any parameters specified as approximate and for which the apparatus does not therefore need to be formally calibrated. If such parameters are liable to change, then the need for periodic checks shall be written into the detailed calibration procedures.

C.2 Schedule

Verification/calibration of the test apparatus is a mandatory part of this International Standard. However, the frequency of calibration and procedures used are, unless otherwise stated, at the discretion of the individual laboratory, using ISO 18899 for guidance.

The calibration schedule given in Table C.1 has been compiled by listing all of the parameters specified in the test method, together with the specified requirement. A parameter and requirement can relate to the main test apparatus, to part of that apparatus or to an ancillary apparatus necessary for the test.

For each parameter, a calibration procedure is indicated by reference to ISO 18899, to another publication, or to a procedure particular to the test method which is detailed (whenever a calibration procedure which is more specific or detailed than that in ISO 18899 is available, it shall be used in preference).

The verification frequency for each parameter is given by a code-letter. The code-letters used in the calibration schedule are:

- N initial verification only;
- S standard interval as given in ISO 18899;
- U in use.

Table C.1 – Calibration frequency schedule

Parameter	Requirements(s)	Subclause in ISO 18899:2004	Verification frequency schedule	Notes
Surface hardness of the dies	≥ 60 HRC	15.5	N	
Dimensions of the dies	See 4.1	15.2	S	
Die grooves	See 4.1	15.2	S	
Surface hardness rotor	≥ 60 HRC	15.5	N	
Dimensions rotor	See 4.2	15.2	S	
Rotor grooves	See 4.2	15.2	S	
Angular velocity	0,209 rad/s ± 0,002 rad/s	23.2	S	
Temperature accuracy	± 0,25°C	18	S	
Temperature stability at steady state	± 0,5°C	18	S	
Die closure	11,5kN ± 0,5kN	21.3	S	
Torque	See 4.6	21.4	S	
Test piece	See 5	15.8	U	

In addition to the items listed in the table, use of the following is implied, all of which need calibrating in accordance with ISO 18899:

- instruments for determining dimensions of the dies
- load cell for die closure check
- thermometer for monitoring the condition and test temperatures

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