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Plastics — Determination of dynamic mechanical properties —

Part 2:
Torsion-pendulum method

Plastiques — Détermination des propriétés mécaniques dynamiques —

Partie 2: Méthode au pendule de torsion

Reference number
ISO 6721-2:2008(E)

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Contents

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 6721-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 2, *Mechanical properties*.

This second edition cancels and replaces the first edition (ISO 6721-2:1994), of which it constitutes a minor revision. It also incorporates the Technical Corrigendum ISO 6721-2:1994/Cor.1:1995. Apart from the inclusion of the Corrigendum (which concerns the last sentence in the first paragraph in Annex C), the main changes are the updating of the references and the correction of ISO 6721-3 to ISO 6721-1 in Subclause 5.6.

ISO 6721 consists of the following parts, under the general title *Plastics — Determination of dynamic mechanical properties*:

- *Part 1: General principles*
- *Part 2: Torsion-pendulum method*
- *Part 3: Flexural vibration — Resonance-curve method*
- *Part 4: Tensile vibration — Non-resonance method*
- *Part 5: Flexural vibration — Non-resonance method*
- *Part 6: Shear vibration — Non-resonance method*
- *Part 7: Torsional vibration — Non-resonance method*
- *Part 8: Longitudinal and shear vibration — Wave-propagation method*
- *Part 9: Tensile vibration — Sonic-pulse propagation method*
- *Part 10: Complex shear viscosity using a parallel-plate oscillatory rheometer*

Plastics — Determination of dynamic mechanical properties —

Part 2:
Torsion-pendulum method

1 Scope

This part of ISO 6721 specifies two methods (A and B) for determining the linear dynamic mechanical properties of plastics, i.e. the storage and loss components of the torsional modulus, as a function of temperature, for small deformations within the frequency range from 0,1 Hz to 10 Hz.

The temperature dependence of these properties, measured over a sufficiently broad range of temperatures (for example from −50 °C to +150 °C for the majority of commercially available plastics), gives information on the transition regions (for example the glass transition and the melting transition) of the polymer. It also provides information concerning the onset of plastic flow. The two methods described are not applicable to non-symmetrical laminates (see ISO 6721-3, *Plastics — Determination of dynamic mechanical properties — Part 3: Flexural vibration — Resonance-curve method*). The methods are not suitable for testing rubbers, for which the user is referred to ISO 4664-2, *Rubber, vulcanized or thermoplastic — Determination of dynamic properties — Part 2: Torsion pendulum methods at low frequencies*.

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 6721-1:2001, *Plastics — Determination of dynamic mechanical properties — Part 1: General principles*

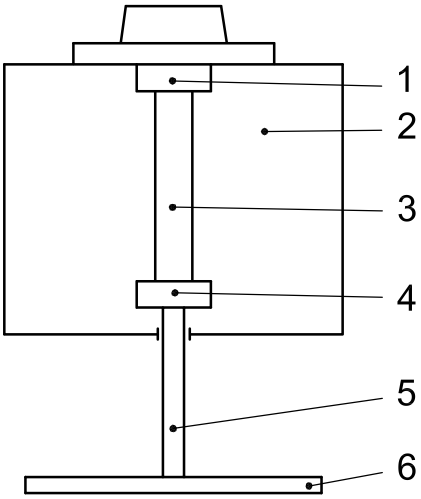
3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 6721-1:2001, Clause 3, apply.

4 Principle

A test specimen of uniform cross-section is gripped by two clamps, one of them fixed and the other connected to a disc, which acts as an inertial member, by a rod. The end of the specimen connected to the disc is excited, together with the disc, to execute freely decaying torsional oscillations. The oscillation mode is that designated IV in ISO 6721-1:2001, Table 2, and the type of modulus is G_{to} as defined in ISO 6721-1:2001, Table 3.

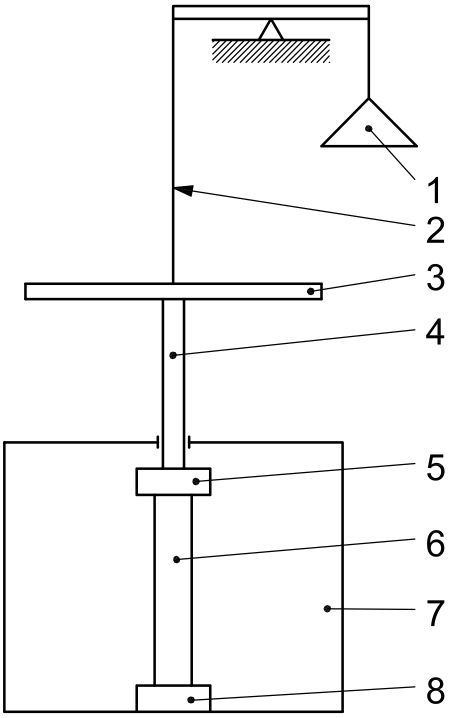
The inertial member is suspended either from the specimen (method A, see Figure 1) or from a wire (method B, see Figure 2). In the latter case, the wire is also part of the elastically oscillating system.



Key

1	upper (fixed) clamp
2	temperature-controlled chamber
3	test specimen
4	lower (movable) clamp
5	rod
6	inertial member

Figure 1 — Apparatus for method A



Key

1	counterweight
2	wire
3	inertial member
4	rod
5	upper (movable) clamp
6	test specimen
7	temperature-controlled chamber
8	lower (fixed) clamp

Figure 2 — Apparatus for method B

During a temperature run, the same inertial member can be used throughout the whole run, which results in a frequency decreasing naturally with increasing temperature, or the inertial member can be replaced at intervals by a member of different moment of inertia in order to keep the frequency approximately constant.

During the test, the frequency and the decaying amplitude are measured. From these quantities, the storage component G'_{to} and loss component G''_{to} of the torsional complex modulus G^*_{to} can be calculated.

5 Test apparatus

5.1 Pendulum

Two types of torsion pendulum are specified for use with this part of ISO 6721:

a) the inertial member is suspended from the test specimen and the lower end of the specimen is excited (method A, Figure 1);

b) the inertial member is suspended from a wire attached to a counterweight and the upper end of the specimen is excited (method B, Figure 2).

Both types of pendulum consists of an inertial member, two clamps for gripping the specimen (one of which is connected to the inertial member by a rod) and a temperature-controlled chamber enclosing the specimen and the clamps. For method B, a counterweight and connecting wire are also required.

5.2 Inertial member

5.2.1 General

The moment of inertia, I, of the inertial member, which may be made of aluminium, for instance, shall be selected as a function of the torsional stiffness of the specimen, so that the temperature-dependent natural frequency of the system lies between approximately 0,1 Hz and 10 Hz.

When testing standard specimens (see 6.2), a moment of inertia, I, of about 3×10^{-5} kg·m^2 is recommended if the same inertial member is to be used throughout a run.

NOTE For certain materials, e.g. filled polymers, a value of I of about 5×10^{-5} kg·m^2 may be necessary.

If a constant frequency is desired over a broad temperature range, interchangeable inertial members with different values of I may be used, thereby permitting the moment of inertia to be varied in steps of less than 20 %, i.e. the frequency to be corrected in steps of less than 10 %. When testing standard specimens (see 6.2) at a frequency of about 1 Hz, a maximum moment of inertia of about 3×10^{-3} kg·m^2 is recommended.

5.2.2 **Method A** (see Figure 1)

The total mass of the inertial member, the lower clamp and the connecting rod shall be such that the weight, W, carried by the specimen is not too high [see Annex A, Equation (A.2)].

5.2.3 **Method B** (see Figure 2)

The total mass of the inertial member, the upper clamp and the rod must be balanced by a suitable counterweight, so that the longitudinal force, W, acting on the specimen is minimized [see Annex A, Equation (A.2)]. The wire supporting these parts is part of the elastically oscillating system.

5.3 Clamps

The clamps shall be designed to prevent movement of the portion of the specimens gripped within them. They shall be self-aligning in order to ensure that the specimen axis remains aligned with the axis of rotation and the test specimen remains adequately secured over the whole temperature range without distortion occurring, thus allowing the free length of the specimen to be accurately determined.

The movable clamp shall be of low mass.

The moment of inertia of the whole system (consisting of the movable clamp, the inertial member and the connecting rod) shall be determined experimentally.

To prevent heat passing from the specimen out of the temperature-controlled chamber and in the opposite direction, the rod connecting the movable clamp and the inertial member shall be thermally non-conducting.

5.4 Oscillation-inducing device

The oscillation-inducing device shall be capable of applying to the pendulum a torsional impulse such that the pendulum oscillates initially through an angle of not more than 1,5° in each direction for normal materials, or not more than 3° in each direction for low-modulus materials (such as elastomers).

5.5 Oscillation-frequency and oscillation-amplitude recording equipment

Optical, electrical or other recording systems may be used provided they have no significant influence on the oscillating system. The entire equipment for measuring frequency and amplitude shall be accurate to ± 1 % (within the transition region ± 5 %).

5.6 Temperature-controlled chamber

See ISO 6721-1:2001, Subclause 5.3.

5.7 Gas supply

See ISO 6721-1:2001, Subclause 5.4.

5.8 Temperature-measurement device

See ISO 6721-1:2001, Subclause 5.5.

5.9 Devices for measuring test-specimen dimensions

See ISO 6721-1:2001, Subclause 5.6.

6 Test specimens

6.1 General

See ISO 6721-1:2001, Clause 6.

6.2 Shape and dimensions

Rectangular test specimens having the following dimensions are recommended:

free length, L:	40 mm to 120 mm, preferably 50 mm
width, b:	5 mm to 11 mm, preferably 10 mm
thickness, h:	0,13 mm to 2 mm, preferably 1 mm

Specimens which are rectangular in cross-section but whose thickness and/or width varies along the main axis of the specimen by more than 3 % of the mean value shall not be used. When comparing different materials, the dimensions of the specimens shall be identical. Specimen dimensions differing from the preferred ones (50 mm × 10 mm × 1 mm) should be chosen to conserve geometric similarity with the preferred specimen shape.

Alternative specimen shapes may be used (e.g. cylindrical or tubular); in such cases, dimensions and tolerances shall be agreed upon by the interested parties.

6.3 Preparation

See ISO 6721-1:2001, Subclause 6.3.

7 Number of specimens

See ISO 6721-1:2001, Clause 7.

8 Conditioning

See ISO 6721-1:2001, Clause 8.

If mechanical conditioning of the specimen is required, the specimen shall be twisted through an angle greater than 5°, but less than 90° in both directions about the torsional-test axis and returned to its normal position.

9 Procedure

9.1 Test atmosphere

See ISO 6721-1:2001, Subclause 9.1.

9.2 Measurement of specimen cross-section

See ISO 6721-1:2001, Subclause 9.2.

9.3 Mounting the test specimens

Clamp the test specimen between the upper and lower clamps. The longitudinal axis of the test specimen shall coincide with the axis of rotation of the oscillating system. Any misalignment of the specimen will cause lateral oscillations that will interfere with the normal oscillation process.

After clamping the test specimen, measure the distance between the clamps (the free length L) to ± 0,5 %. When setting up the oscillating system in the chamber, check to make sure that the test specimen is not stressed.

After assembling the oscillating system complete with test specimen, and checking its alignment, start the heating or cooling (see 9.4).

9.4 Varying the temperature

See ISO 6721-1:2001, Subclause 9.4.

9.5 Performing the test

Start the free oscillations by setting the pendulum (5.1) in motion using the oscillation-inducing device (5.4).

Record the oscillation frequency and the oscillation amplitude as it decays.

Check that no amplitude decay is caused either by friction between moving and fixed parts of the apparatus or non-linear behaviour of the material under test (see ISO 6721-1:2001, Annex B).

If the frequency is kept fixed during a temperature run, ensure that the inertial member is changed as and when necessary.

10 Expression of results

10.1 Symbols and correction factors

b	width, in metres, of a rectangular specimen
h	thickness, in metres, of a rectangular specimen
L	free length, in metres, of specimen
I	moment of inertia, expressed in kilogram metre squared (kg·m^2), of the inertial member (if appropriate, including the movable clamp and the connecting rod)
f_d	frequency, in hertz, of the damped oscillating system
f_0	frequency, in hertz, of the pendulum as used in method B, without the specimen
Λ	logarithmic decrement for damped oscillations of pendulum plus specimen
Λ_0	logarithmic decrement for damped oscillations of a method B pendulum, without the specimen
F_g	so-called dimensional factor for the specimen, expressed in reciprocal cubic metres (m^{-3})

For specimens with a rectangular cross-section:

$$F_\mathrm{g} = 3L/bh^3F_\mathrm{c} \tag{1}$$

where F_c is the so-called dimensional correction factor.

When $0 \leqslant h/b \leqslant 0{,}6$

$$F_\mathrm{c} = 1 - 0{,}63h/b \tag{2}$$

When $0{,}6 \leqslant h/b \leqslant 1$

$$F_\mathrm{c} = 0{,}843/(1 + h^2/b^2) \tag{3}$$

For specimens with a circular cross-section:

$$F_\mathrm{g} = 32L/\pi d^4 \tag{4}$$

where d is the diameter, in metres, of the specimen.

F_d damping correction factor, given by the equation

$$F_\mathrm{d} = 1 - (\Lambda/2\pi)^2 \tag{5}$$

G'_to torsional storage modulus, in pascals, of the specimen

G''_to torsional loss modulus, in pascals, of the specimen

NOTE 1 For reasons given in Annex B, the symbol F_d used for the damping correction factor has a different subscript from that used previously in ISO 537 (now withdrawn).

NOTE 2 Equations (2) and (3) are only approximately valid, the maximum error being 0,9 % (see Annex C).

NOTE 3 The dimensional factor does not include any length corrections to allow for clamping effects. Therefore, only measurements carried out on specimens with the same thickness, width and length ratios will yield accurately comparable results (see ISO 6721-1:2001, Table 1 and Note 6 to Definition 3.1).

10.2 Calculation of logarithmic decrement, Λ

The logarithmic decrement, Λ, may be calculated using the following equation:

$$\Lambda = \ln (X_q/X_{q+1}) \tag{6}$$

where X_q and X_{q+1} are the amplitudes of two successive oscillations in the same direction (see ISO 6721-1:2001, Definition 3.10).

To calculate Λ from the amplitudes of any two oscillations p and q in the same direction, use the equation

$$\Lambda = \frac{1}{p-q} \ln \left(X_q / X_p \right) \tag{7}$$

where

X_p is the amplitude of the pth oscillation;

X_q is the amplitude of the qth oscillation.

The following equation shall be used in the case of amplitudes that cannot be recorded on a damped sinusoidal curve with an accurate baseline (see Figure 3):

$$\Lambda = \ln \left(X_q^* \middle/ X_{q+1}^* \right) = \frac{1}{p-q} \ln \left(X_q^* \middle/ X_p^* \right) \tag{8}$$

where $X_p^*, \ldots, X_q^*, X_{q+1}^*$ are the differences between successive positive and negative amplitudes of the oscillation concerned,

i.e. $X_q^* = X_q^+ - X_q^-$

NOTE Equation (8) only corrects for a constant shift in the baseline, not for a time-dependent baseline drift. A time-dependent baseline drift may be caused by the non-oscillating part of the relaxation process, following application of the single pulse to start the system oscillating. It can be decreased by using double-pulse starting, with each pulse applied in a different direction.

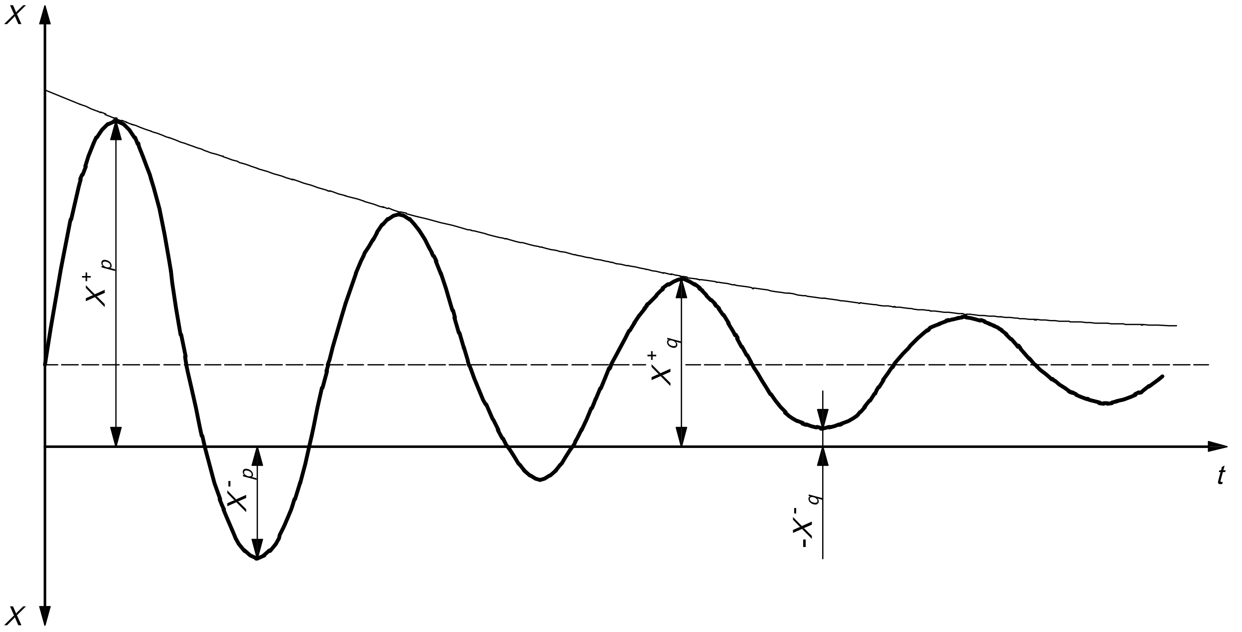


Figure 3 — Amplitude X versus time t for damped vibrations showing a baseline shift

10.3 Calculation of torsional storage modulus, G'_{to}

The torsional storage modulus (see ISO 6721-1:2001, Definition 3.2) of a test specimen with a rectangular cross-section may be calculated from the equation

$$G'_{\text{to}} = 4\pi^2 I \left(f_{\text{d}}^2 F_{\text{d}} - f_0^2 \right) F_{\text{g}} \tag{9}$$

Assuming a rectangular cross-section with a low h/b ratio [see Equations (1) and (2)] and inserting Equation (5), one obtains

$$G'_{\text{to}} = 12\pi^2 I f_{\text{d}}^2 \left[1 - (\Lambda/2\pi)^2 - (f_0/f_{\text{d}})^2 \right] \times L/bh^3\, F_{\text{c}} \tag{10}$$

f_0 being 0 for method A.

For rubber-like materials, high longitudinal forces acting upon the specimen shall be avoided (see Annex A).

10.4 Calculation of torsional loss modulus, G''_{to}

The torsional loss modulus G''_{to} (see ISO 6721-1:2001, Definition 3.3) may be calculated from the equation

$$G''_{\text{to}} = 4\pi I f_{\text{d}}^2 \left(\Lambda - \Lambda_0 \right) F_{\text{g}} \tag{11}$$

Λ_0 being 0 for method A.

For method B, if $\Lambda_0 \ll \Lambda$ and assuming a rectangular cross-section with a low h/b ratio [see Equations (1) and (2)] and inserting Equation (5), one obtains

$$G''_{\text{to}} = 12\pi I f_{\text{d}}^2 \Lambda L / bh^3 F_{\text{c}} \tag{12}$$

11 Precision

The precision of this technique has been determined from the results of round robins in which 15 laboratories participated[7]. Interlaboratory precision was as follows:

for G'_{to} in the glassy region: ± 7 %;

for G'_{to} at the glass-transition temperature: ± 30 %;

for G''_{to} below the glass-transition temperature: ± 10 %.

Utilizing G'_{to} or G''_{to}, the glass-transition temperature could be determined to within ± 3 °C. The values for intralaboratory precision were about half those for interlaboratory precision.

NOTE The glass-transition temperature was determined from the point of inflexion of the log G'_{to} versus temperature curve or from the maximum of the G''_{to} versus temperature curve associated with the glass transition.

12 Test report

The test report shall include the following information:

a) a reference to this part of ISO 6721, plus the method (i.e. the type of pendulum) used (A or B), e.g. ISO 6721-2B;

b) to m) see ISO 6721-1:2001, Clause 12;

n) if a fixed frequency was used: the frequency chosen and the variation in frequency caused by changing the inertial member;

o) if the same inertial member was used: the frequency range between the minimum temperature and the maximum temperature;

p) if method A was used: the mass of the inertial member.

Annex A
(normative)

Influence of longitudinal force, W

As stated in ISO 537, one of the predecessors to this International Standard, a longitudinally superimposed force W acting on the specimen generates an additional torsional stiffness, resulting in an apparent modulus increase ΔG_W (designated S_E in ISO 537). This longitudinal force, W, is the total weight of all the parts that are suspended from the specimen. Therefore, the appropriate correction is necessary only for the pendulum used in method A, in which the disc, the rod and the lower clamp are suspended from the specimen. For the pendulum used in method B, the counterweight balances the force W (see Figures 1 and 2).

The modulus correction is given by

$$\Delta G_W = Wb/4h^3F_c \tag{A.1}$$

and is subtracted from the value of the storage modulus, calculated from Equation (9). A number of points should be noted with regard to this correction, however:

- It is necessary only for measurements in the rubber elastic region.
- Rubbers show a so-called “primary normal stress difference”, however, which increases in proportion to the square of the shear strain. This effect produces a non-linear, i.e. non-harmonic, distortion of the vibrations, which can be avoided by restricting measurements to small-amplitude vibrations.
- It is unclear how this effect should be taken into account for the loss modulus G'', since the basic equations have not been developed for viscoelasticity.

In order to overcome the difficulties listed above, any weight, W, that generates a modulus correction ΔG_W greater than 1 % of the storage modulus G'_{to} shall be avoided, i.e. the following condition shall be satisfied:

$$W \leqslant 0{,}04 G'_{to} h^3 F_c / b \tag{A.2}$$

The moment of inertia of a solid circular inertial member of constant thickness and diameter d and mass m is

$$I = md^2/8 \tag{A.3}$$

The moment of inertia, I, and the mass, m, can therefore be adjusted independently to some extent.

Annex B
(informative)

Damping correction factor, F_d

Several different mathematical treatments for the torsion-pendulum test are given in the literature (see References [1] to [6]).

All authors have arrived at the same result as far as the loss modulus, G''_to, given by Equation (11) is concerned.

With respect to the storage modulus, however, the equations given in References [2] and [3] agree with that which was given in ISO 537 (now withdrawn) in that the correction term $(\Lambda/\pi)^2$ in ISO 537 was positive, rather than negative as in Equation (5) of this part of ISO 6721. According to Struik[4] and Schaefer[6], this results from the use of an over-simplified version of the equation of motion which neglects Boltzmann's superposition principle.

According to Nielsen[1], the positive correction term does not relate to the storage part, but to the magnitude of the complex modulus given by

$$\left|G_\mathrm{to}\right| = 4\pi^2 I f_\mathrm{d}^2 \left[1 + \left(\Lambda/2\pi\right)^2\right] F_\mathrm{g} \tag{B.1}$$

On the other hand, using Equation (10) with the negative correction term and Equation (12), the magnitude of the complex modulus is

$$\left|G_\mathrm{to}\right| = 4\pi^2 I f_\mathrm{d}^2 F_\mathrm{g} \sqrt{1 + 2\left(\Lambda/2\pi\right)^2 + \left(\Lambda/2\pi\right)^4} \tag{B.2}$$

Assuming $\Lambda \leqslant 2{,}4$, this can be approximated (error $\leqslant$ 1 %) to

$$\left|G_\mathrm{to}\right| \approx 4\pi^2 I f_\mathrm{d}^2 F_\mathrm{g} \left[1 + \left(\Lambda/2\pi\right)^2\right] \tag{B.3}$$

This is consistent with the interpretation given by Nielsen [see Equation (B.1)].

Schaefer[6] has shown that Equation (10) is exactly valid for any purely positive relaxation spectrum.

Annex C
(informative)

Dimensional correction factor, F_c

Equations (2) and (3) for the correction factor F_c are only approximately valid. Equation (2) represents a first-order approximation for small thickness-to-width (h/b) ratios as does Equation (3), but only h/b ratios close to 1. Figure C.1 shows the error involved in using Equations (2) and (3) rather than the exact but complicated equation

$$F_c = 1 - \frac{192}{\pi^5}(h/b) \times \sum_{n=0} \tanh\left[(2n+1)\pi b/2h\right](2n+1)^{-5} \tag{C.1}$$

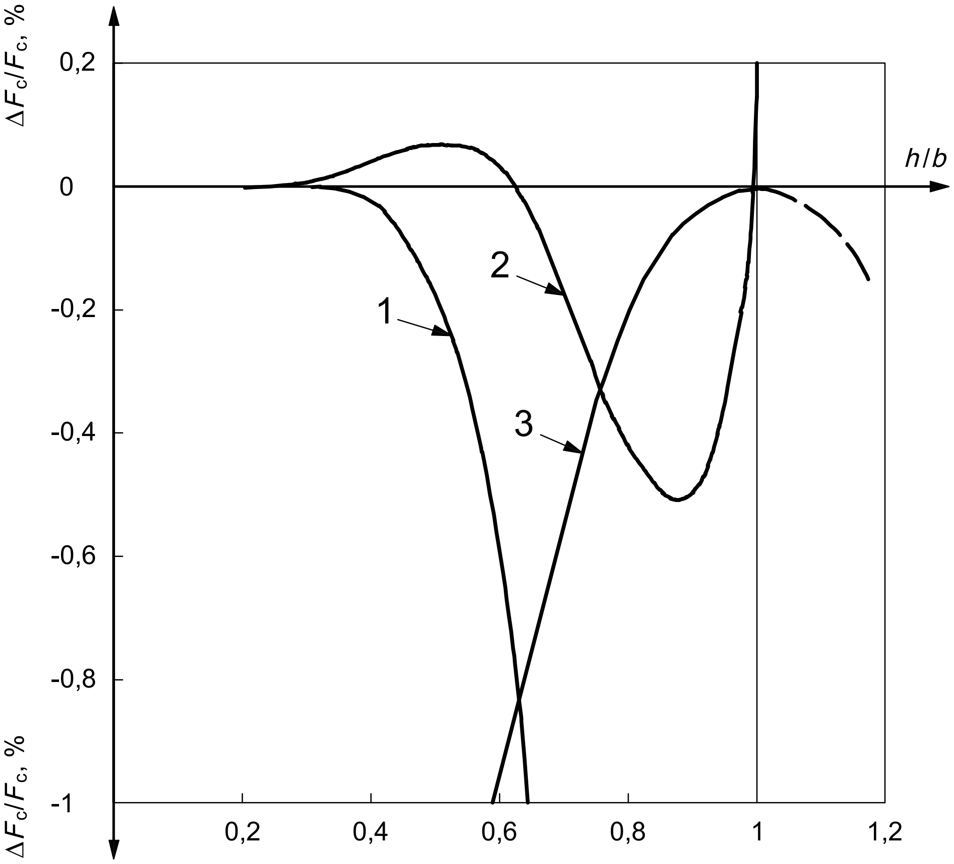
given by Nederveen and Van Der Wal[8], this error being plotted against the ratio h/b. If the range of h/b is limited to a maximum value of 0,6 in Equation (2) and a minimum value of 0,6 in Equation (3), the maximum error is 0,9 %.

The withdrawn standard ISO 537 gave a second-order approximation which, when rearranged into the same form as Equations (2) and (3), gives the following equation:

$$F_c = 1 - 0{,}63(h/b)\left(1 - h^4/12b^4\right) \tag{C.2}$$

The error involved in using this equation rather than Equation (C.1) is also plotted in Figure C.1, which shows that Equation (C.2) is more accurate only over the range 0,5 $< h/b <$ 0,75.

For thin specimens (i.e. those with the recommended h/b ratio), and owing to its simple form, Equation (2) is superior to Equation (C.2) [Equation (3) has been included merely for the sake of completeness].



Key

1 Equation (2)

2 Equation (C.2)

3 Equation (3)

Figure C.1 — Relative error, $\Delta F_c/F_c$, involved in calculating the dimensional correction factor, F_c, using the approximate Equations (2), (3) and (C.2) rather than the exact Equation (C.1)

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