INTERNATIONAL STANDARD

ISO 6721-1

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Plastics — Determination of dynamic mechanical properties —

Part 1: General principles

Plastiques — Détermination des propriétés mécaniques dynamiques —

Partie 1: Principes généraux

Reference number
ISO 6721-1:2001(E)

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 3.

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 part of ISO 6721 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 6721-1 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 2, *Mechanical properties*.

This second edition cancels and replaces the first edition (ISO 6721-1:1994), of which it constitutes a minor revision (two further references have been added to the bibliography).

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*

Additional parts are planned.

Annexes A and B of this part of ISO 6721 are for information only.

Introduction

The methods specified in the first nine parts of ISO 6721 can be used for determining storage and loss moduli of plastics over a range of temperatures or frequencies by varying the temperature of the specimen or the frequency of oscillation. Plots of the storage or loss moduli, or both, are indicative of viscoelastic characteristics of the specimen. Regions of rapid changes in viscoelastic properties at particular temperatures or frequencies are normally referred to as transition regions. Furthermore, from the temperature and frequency dependencies of the loss moduli, the damping of sound and vibration of polymer or metal-polymer systems can be estimated.

Apparent discrepancies may arise in results obtained under different experimental conditions. Without changing the observed data, reporting in full (as described in the various parts of ISO 6721) the conditions under which the data were obtained will enable apparent differences observed in different studies to be reconciled.

The definitions of complex moduli apply exactly only to sinusoidal oscillations with constant amplitude and constant frequency during each measurement. On the other hand, measurements of small phase angles between stress and strain involve some difficulties under these conditions. Because these difficulties are not involved in some methods based on freely decaying vibrations and/or varying frequency near resonance, these methods are used frequently (see ISO 6721-2 and ISO 6721-3). In these cases, some of the equations that define the viscoelastic properties are only approximately valid.

Plastics — Determination of dynamic mechanical properties —

Part 1:
General principles

1 Scope

The various parts of ISO 6721 specify methods for the determination of the dynamic mechanical properties of rigid plastics within the region of linear viscoelastic behaviour. This part of ISO 6721 is an introductory section which includes the definitions and all aspects that are common to the individual test methods described in the subsequent parts.

Different deformation modes may produce results that are not directly comparable. For example, tensile vibration results in a stress which is uniform across the whole thickness of the specimen, whereas flexural measurements are influenced preferentially by the properties of the surface regions of the specimen.

Values derived from flexural-test data will be comparable to those derived from tensile-test data only at strain levels where the stress-strain relationship is linear and for specimens which have a homogeneous structure.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 6721. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 6721 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 291:1997, *Plastics — Standard atmospheres for conditioning and testing*.

ISO 293:1986, *Plastics — Compression moulding test specimens of thermoplastic materials*.

ISO 294 (all parts), *Plastics — Injection moulding of test specimens of thermoplastic materials*.

ISO 295:1991, *Plastics — Compression moulding of test specimens of thermosetting materials*.

ISO 1268 (all parts), *Plastics — Methods of producting test plates*.

ISO 2818:1994, *Plastics — Preparation of test specimens by machining*.

ISO 4593:1993, *Plastics — Film and sheeting — Determination of thickness by mechanical scanning*.

ISO 6721-2:1994, *Plastics — Determination of dynamic mechanical properties — Part 2: Torsion-pendulum method.*

ISO 6721-3:1994, *Plastics — Determination of dynamic mechanical properties — Part 3: Flexural vibration — Resonance-curve method*.

3 Terms and definitions

For the purposes of this part of ISO 6721, the following terms and definitions apply.

NOTE Most of the terms defined here are also defined in ISO 472:1999, *Plastics — Vocabulary*. The definitions given here are not strictly identical with, but are equivalent to, those in ISO 472:1999.

3.1
complex modulus
M^*
the ratio of dynamic stress, given by $\sigma(t) = \sigma_\text{A} \exp(\text{i}2\pi ft)$, and dynamic strain, given by $\varepsilon(t) = \varepsilon_\text{A} \exp[\text{i}(2\pi ft - \delta)]$, of a viscoelastic material that is subjected to a sinusoidal vibration, where σ_A and ε_A are the amplitudes of the stress and strain cycles, f is the frequency, δ is the phase angle between stress and strain (see 3.5 and Figure 1) and t is time

It is expressed in Pascals (Pa).

Depending on the mode of deformation, the complex modulus may be one of several types: E^*, G^*, K^* or L^* (see Table 3).

$$M^* = M' + \text{i}M'' \qquad \text{(see 3.2 and 3.3)} \qquad (1)$$

where

$$\text{i} = (-1)^{1/2} = \sqrt{-1}$$

For the relationships between the different types of complex modulus, see Table 1.

NOTE 1 For isotropic viscoelastic materials, only two of the elastic parameters G^*, E^*, K^*, L^* and μ^* are independent (μ^* is the complex Poisson's ratio, given by $\mu^* = \mu' + \mu''$).

NOTE 2 The most critical term containing Poisson's ratio μ is the "volume term" $1 - 2\mu$, which has values between 0 and 0,4 for μ between 0,5 and 0,3. The relationships in Table 1 containing the "volume term" $1 - 2\mu$ can only be used if this term is known with sufficient accuracy.

It can be seen from Table 1 that the volumetric term $1 - 2\mu$ can only be estimated with any confidence from a knowledge of the bulk modulus K or the uniaxial-strain modulus L and either E or G. This is because K and L measurements involve deformations when the volumetric strain component is relatively large.

NOTE 3 Up to now, no measurement of the dynamic mechanical bulk modulus K, and only a small number of results relating to relaxation experiments measuring $K(t)$, have been described in the literature.

NOTE 4 The uniaxial-strain modulus L is based upon a load with a high hydrostatic-stress component. Therefore values of L compensate for the lack of K values, and the "volume term" $1 - 2\mu$ can be estimated with sufficient accuracy based upon the modulus pairs (G, L) and (E, L). The pair (G, L) is preferred, because G is based upon loads without a hydrostatic component.

NOTE 5 The relationships given in Table 1 are valid for the complex moduli as well as their magnitudes (see 3.4).

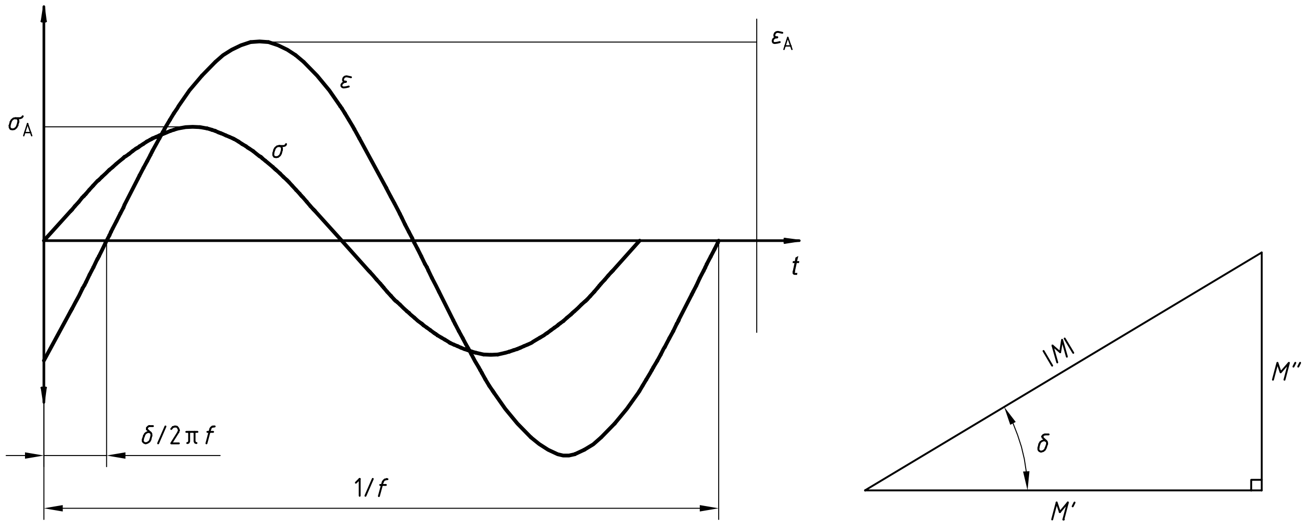
NOTE 6 Most of the relationships for calculating the moduli given in the other parts of this International Standard are, to some extent, approximate. They do not take into account e.g. "end effects" caused by clamping the specimens, and they include other simplifications. Using the relationships given in Table 1 therefore often requires additional corrections to be made. These are given in the literature (see e.g. references [1] and [2] in the Bibliography).

NOTE 7 For linear-viscoelastic behaviour, the complex compliance C^* is the reciprocal of the complex modulus M^*, i.e.

$$M^* = (C^*)^{-1} \qquad (2)$$

Thus

$$M' + \mathrm{i}M'' = \frac{C' - \mathrm{i}C''}{(C')^2 + (C'')^2} \tag{3}$$



a) The phase shift $\delta/2\pi f$ between the stress σ and strain ε in a viscoelastic material subjected to sinusoidal oscillation (σ_A and ε_A are the respective amplitudes, f is the frequency).

b) The relationship between the storage modulus M', the loss modulus M'', the phase angle δ and the magnitude $|M|$ of the complex modulus M^*.

Figure 1 — Phase angle and complex modulus

Table 1 — Relationships between moduli for uniformly isotropic materials

	G and μ	E and μ	K and μ	G and E	G and K	E and K	G and L[a]
Poisson's ratio, μ $1 - 2\mu =$[b]				$3 - \frac{E}{G}$	$\frac{G/K}{1+G/3K}$	$\frac{E}{3K}$	$\frac{1}{L/G - 1}$
Shear modulus, $G =$		$\frac{E}{2(1+\mu)}$	$\frac{3K(1-2\mu)}{2(1+\mu)}$			$\frac{E}{3 - E/3K}$	
Tensile modulus, $E =$	$2G(1+\mu)$		$3K(1-2\mu)$		$\frac{3G}{1+G/3K}$		$\frac{3G(1-4G/3L)}{1-G/L}$
Bulk modulus, $K =$[c]	$\frac{2G(1+\mu)}{3(1-2\mu)}$	$\frac{E}{3(1-2\mu)}$		$\frac{G}{3(3G/E - 1)}$			$L - \frac{4G}{3}$
Unaxial-strain or longitudinal-wave modulus, $L =$	$\frac{2G(1-\mu)}{1-2\mu}$	$\frac{E(1-\mu)}{(1+\mu)(1-2\mu)}$	$\frac{3K(1-\mu)}{1+\mu}$	$\frac{G(4G/E-1)}{3G/E-1}$	$K + \frac{4G}{3}$	$\frac{K(1+E/3K)}{1-E/9K}$	

[a] See note 4 to definition 3.1.
[b] See note 2 to definition 3.1.
[c] See note 3 to definition 3.1.

3.2
storage modulus
M'
the real part of the complex modulus M^* [see Figure 1 b)]

The storage modulus is expressed in pascals (Pa).

It is proportional to the maximum energy stored during a loading cycle and represents the stiffness of a viscoelastic material.

The different types of storage modulus, corresponding to different modes of deformation, are: E'_{t} tensile storage modulus, E'_{f} flexural storage modulus, G'_{s} shear storage modulus, G'_{to} torsional storage modulus, K' bulk storage modulus, L'_{c} uniaxial-strain and L'_{w} longitudinal-wave storage modulus.

3.3
loss modulus
M''
the imaginary part of the complex modulus [see Figure 1 b)]

The loss modulus is expressed in pascals (Pa).

It is proportional to the energy dissipated (lost) during one loading cycle. As with the storage modulus (see 3.2), the mode of deformation is designated as in Table 3, e.g. E''_{t} is the tensile loss modulus.

3.4
magnitude $|M|$ of the complex modulus
the root mean square value of the storage and the loss moduli as given by the equation

$$|M|^2 = (M')^2 + (M'')^2 = (\sigma_{\mathrm{A}}/\varepsilon_{\mathrm{A}})^2 \quad (4)$$

where σ_{A} and ε_{A} are the amplitudes of the stress and the strain cycles, respectively.

The complex modulus is expressed in pascals (Pa).

The relationship between the storage modulus M', the loss modulus M'', the phase angle δ, and the magnitude $|M|$ of the complex modulus is shown in Figure 1 b). As with the storage modulus, the mode of deformation is designated as in Table 3, e.g. $|E_{\mathrm{t}}|$ is the magnitude of the tensile complex modulus.

3.5
phase angle
δ
the phase difference between the dynamic stress and the dynamic strain in a viscoelastic material subjected to a sinusoidal oscillation (see Figure 1)

The phase angle is expressed in radians (rad).

As with the storage modulus (see 3.2), the mode of deformation is designated as in Table 3, e.g. δ_{t} is the tensile phase angle.

3.6
loss factor (tan δ)
the ratio between the loss modulus and the storage modulus, given by the equation

$$\tan\delta = M''/M' \quad (5)$$

where δ is the phase angle (see 3.5) between the stress and the strain

The loss factor is expressed as a dimensionless number.

The loss factor tan δ is commonly used as a measure of the damping in a viscoelastic system. As with the storage modulus (see 3.2), the mode of deformation is designated as in Table 3, e.g. tan δ_{t} is the tensile loss factor.

3.7
stress-strain hysteresis loop
the stress expressed as a function of the strain in a viscoelastic material subject to sinusoidal vibrations

NOTE Provided the viscoelasticity is linear in nature, this curve is an ellipse (see Figure 2).

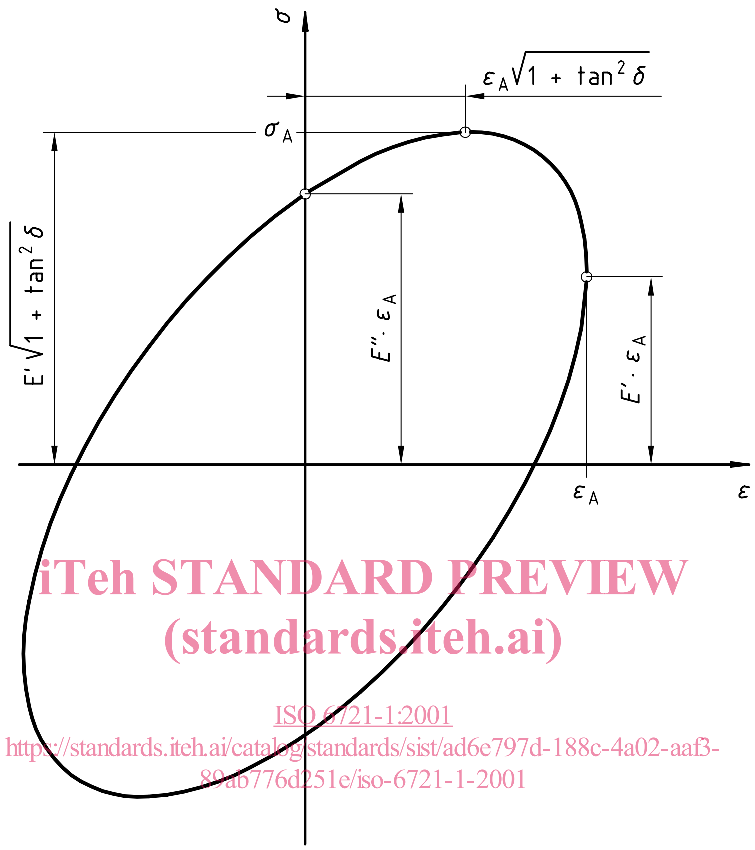


Figure 2 — Dynamic stress-strain hysteresis loop for a linear-viscoelastic material subject to sinusoidal tensile vibrations

3.8
damped vibration
the time-dependent deformation or deformation rate $X(t)$ of a viscoelastic system undergoing freely decaying vibrations (see Figure 3), given by the equation

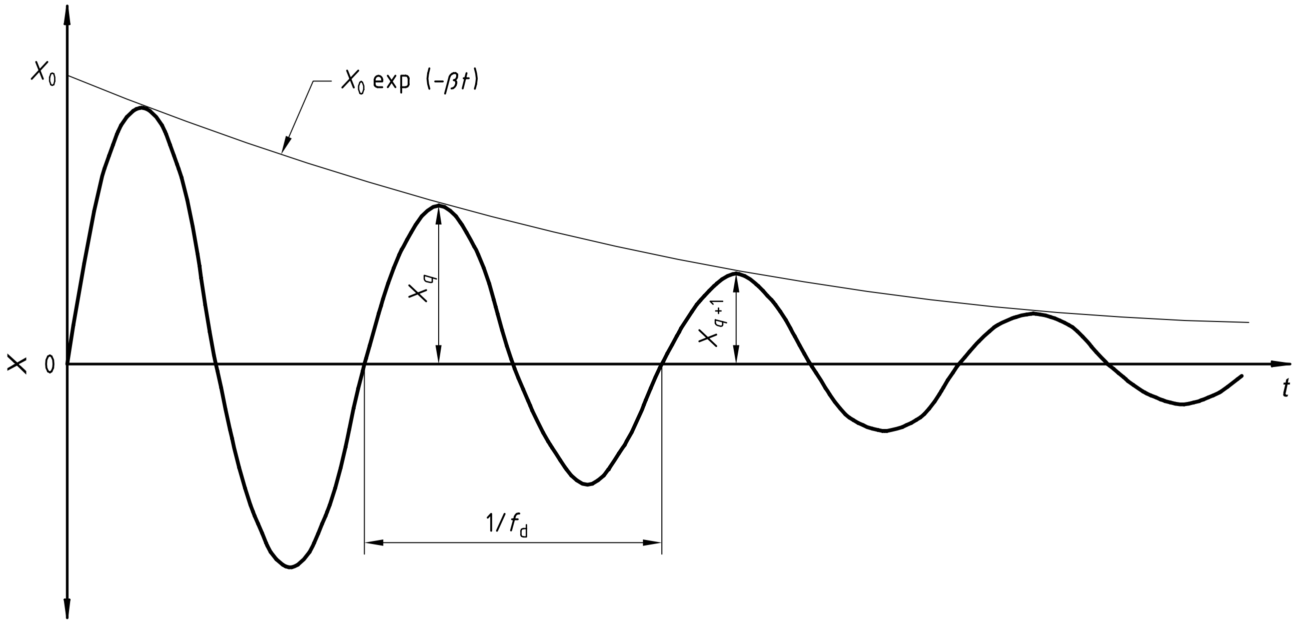
$$X(t) = X_0 \exp(-\beta t) \times \sin 2\pi f_d t \quad (6)$$

where

X_0 is the magnitude, at zero time, of the envelope of the cycle amplitudes;

f_d is the frequency of the damped system;

β is the decay constant (see 3.9).



[X is the time-dependent deformation or deformation rate, X_q is the amplitude of the qth cycle and X_0 and β define the envelope of the exponential decay of the cycle amplitudes — see equation (6).]

Figure 3 — Damped-vibration curve for a viscoelastic system undergoing freely decaying vibrations

3.9
decay constant
β
the coefficient that determines the time-dependent decay of damped free vibrations, i.e. the time dependence of the amplitude X_q of the deformation or deformation rate [see Figure 3 and equation (6)]

The decay constant is expressed in reciprocal seconds (s^{-1}).

3.10
logarithmic decrement
Λ
the natural logarithm of the ratio of two successive amplitudes, in the same direction, of damped free oscillations of a viscoelastic system (see Figure 3), given by the equation

$$\Lambda = \ln (X_q / X_{q+1}) \tag{7}$$

where X_q and X_{q+1} are two successive amplitudes of deformation or deformation rate in the same direction

The logarithmic decrement is expressed as a dimensionless number.

It is used as a measure of the damping in a viscoelastic system.

Expressed in terms of the decay constant β and the frequency f_d, the logarithmic decrement is given by the equation

$$\Lambda = \beta / f_d \tag{8}$$

The loss factor tan δ is related to the logarithmic decrement by the approximate equation

$$\tan \delta \approx \Lambda / \pi \tag{9}$$

NOTE Damped freely decaying vibrations are especially suitable for analysing the type of damping in the material under test (i.e. whether the viscoelastic behaviour is linear or non-linear) and the friction between moving and fixed components of the apparatus (see annex B).

3.11
resonance curve
the curve representing the frequency dependence of the deformation amplitude D_A or deformation-rate amplitude R_A of an inert viscoelastic system subjected to forced vibrations at constant load amplitude L_A and at frequencies close to and including resonance (see Figure 4 and annex A)

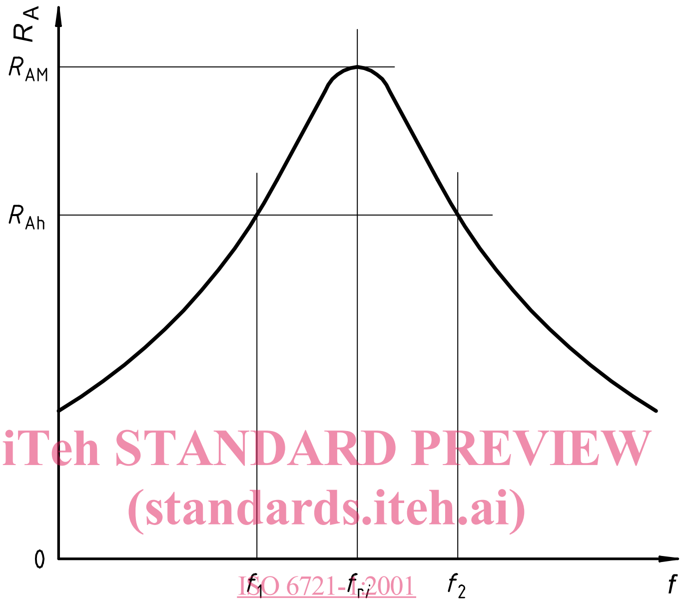


Figure 4 — Resonance curve for a viscoelastic system subjected to forced vibrations
(Deformation-rate amplitude R_A versus frequency f at constant load amplitude; logarithmic frequency scale)

3.12
resonance frequencies
f_{ri}
the frequencies of the peak amplitudes in a resonance curve

The subscript i refers to the order of the resonance vibration.

Resonance frequencies are expressed in hertz (Hz).

NOTE Resonance frequencies for viscoelastic materials derived from measurements of displacement amplitude will be slightly different from those obtained from displacement-rate measurements, the difference being larger the greater the loss in the material (see annex A). Storage and loss moduli are accurately related by simple expressions to resonance frequencies obtained from displacement-rate curves. The use of resonance frequencies based on displacement measurements leads to a small error which is only significant when the specimen exhibits high loss. Under these conditions, resonance tests are not suitable.

3.13
width of a resonance peak
Δf_i
the difference between the frequencies f_1 and f_2 of the ith-order resonance peak, where the height R_{Ah} of the resonance curve at f_1 and f_2 is related to the peak height R_{AMi} of the ith mode by

$$R_{Ah} = 2^{-1/2} R_{AM} = 0{,}707 R_{AM} \tag{10}$$

(see Figure 4)