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**Methods of test for refractory products —**

**Part 1:**

**Determination of dynamic Young's  
modulus (MOE) by impulse excitation of  
vibration**

*Méthodes d'essai pour produits réfractaires —*

*Partie 1: Détermination du module de Young dynamique (MOE) par  
excitation de vibration par impulsion*



Reference number  
ISO 12680-1:2005(E)

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## 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 12680-1 was prepared by Technical Committee ISO/TC 33, *Refractories*.

ISO 12680 consists of the following parts, under the general title *Methods of test for refractory products*:

— *Part 1: Determination of dynamic Young's modulus (MOE) by impulse excitation of vibration*

The following part is under preparation:

— *Part 2: Determination of static modulus of elasticity*

# Methods of test for refractory products —

## Part 1:

# Determination of dynamic Young's modulus (MOE) by impulse excitation of vibration

## 1 Scope

This part of ISO 12680 specifies a method for determining the dynamic Young's modulus of rectangular cross-section bars and circular cross-section specimens of refractories by impulse excitation of vibration. The dynamic Young's modulus is determined using the resonant frequency of the specimen in its flexural mode of vibration.

**NOTE** Although not specifically described in this part of ISO 12680, this method can also be used at high temperatures with suitable equipment modification.

This part of ISO 12680 does not address the safety issues associated with its use. It is the responsibility of the users of this standard to establish appropriate safety and health practices.

## 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 5022:1979, *Shaped refractory products — Sampling and acceptance testing*

ISO 8656-1:1988, *Refractory products — Sampling of raw materials and unshaped products — Part 1: Sampling scheme*

## 3 Terms and definitions

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

### 3.1

#### **modulus of elasticity**

#### **MOE**

ratio of stress to strain below the proportional limit

### 3.2

#### **proportional limit**

greatest stress which a material is capable of sustaining without deviation from proportionality of stress to strain (Hooke's Law)

### 3.3

#### **anti-nodes**

locations, generally two or more, of local maximum displacement in an unconstrained slender bar or rod in resonance

**NOTE** For the fundamental flexural resonance, the anti-nodes are located at the two ends and the centre of the specimen.

### 3.4

#### **flexural vibrations**

displacements in a slender rod or bar in the plane normal to its length

### 3.5

#### **homogeneous**

uniform composition, density and texture

NOTE A result of homogeneity is that any smaller specimen taken from the original is representative of the whole. In refractory practice, as long as the geometrical dimensions of the specimen are large with respect to the size of individual grains, crystals, components, pores and microcracks, the body can be considered homogeneous.

### 3.6

#### **in-plane flexure**, noun

flexural mode for rectangular parallelepiped geometry specimens in which the direction of the displacement is in the major plane of the specimen

### 3.7

#### **isotropic**, adj.

condition of a specimen such that the values of the elastic properties are the same in all directions in the specimen

### 3.8

#### **nodes**

location on a slender rod or bar in resonance having a constant zero displacement

NOTE For the fundamental flexural resonance of such a rod or bar, the nodes are located at  $0,224 L$  from each end, where  $L$  is the length of the specimen.

### 3.9

#### **out-of-plane flexure**

flexural mode for rectangular parallelepiped geometry specimens in which the direction of the displacement is perpendicular to the major plane of the specimen

### 3.10

#### **resonant frequency**

natural frequencies of vibration of a body driven into flexural vibration

NOTE Resonant frequencies are determined by the elastic modulus, mass and dimensions of the specimen. The lowest resonant frequency in a vibrational mode is the fundamental resonant frequency of that mode.

### 3.11

#### **slender rod**

#### **slender bar**

specimen whose ratio of length to minimum cross-section thickness or diameter is at least 5

NOTE This applies to dynamic elastic property testing.

## 4 Principle

A test specimen of suitable geometry is excited mechanically with a single elastic strike of an impulse tool, called a hammer, and its fundamental resonant frequency is determined.

A transducer (e.g. contact accelerometer or non-contacting microphone) senses the mechanical vibrations in the specimen resulting from the excitation and transforms the vibrations into electrical signals. Specimen supports, impulse locations and signal pick-up points are selected to induce and measure a specific mode of transient vibrations, i.e. the flexural mode. The signals are analysed and a signal analyser that provides data about the frequency and/or the period of the specimen's vibration determines the fundamental resonant frequency. The appropriate fundamental resonant frequency, dimensions and mass of the specimen are used to calculate the dynamic Young's modulus.

## 5 Significance and use

This test method may be used for refractory characterization, development and quality control purposes.

This test method is appropriate for determining the modulus of elasticity of refractory bodies that are homogeneous in nature.

This method addresses the determination of the dynamic moduli of elasticity of slender rectangular bars and cylindrical rods.

This test method is non-destructive in use so it may be used on specimens prepared for other tests. The specimens are subjected to only minute strains; hence the moduli are measured at or near the origin of the stress-strain curve with a minimum possibility of specimen fracture.

The test provides options for variations in test specimen sizes and procedure to accommodate most refractory compositions and textures.

The impulse excitation test method utilizes an impact tool (hammer) and simple supports for the test specimen.

This test method is not suitable for specimens with major cracks or voids.

This test method is limited to determining moduli of specimens with regular geometries, such as rectangular parallelepipeds and cylinders, for which analytical equations are available to relate geometry, mass and modulus to the resonant vibration frequency.

The analytical equations assume parallel or concentric dimensions for the geometry of the specimens. Deviations in the dimensions of the specimens will introduce errors in the calculations and in the results of the tests.

Uneven or excessively rough surfaces of as-formed specimens can have a significant effect on the accuracy of the determination. The dynamic modulus value is inversely proportional to the cube of the thickness so the thickness variation is significant.

This test method assumes that the specimen is vibrating freely with no significant restraint or impediment. Specimen supports should be designed and located so the specimen can vibrate freely in the proper mode.

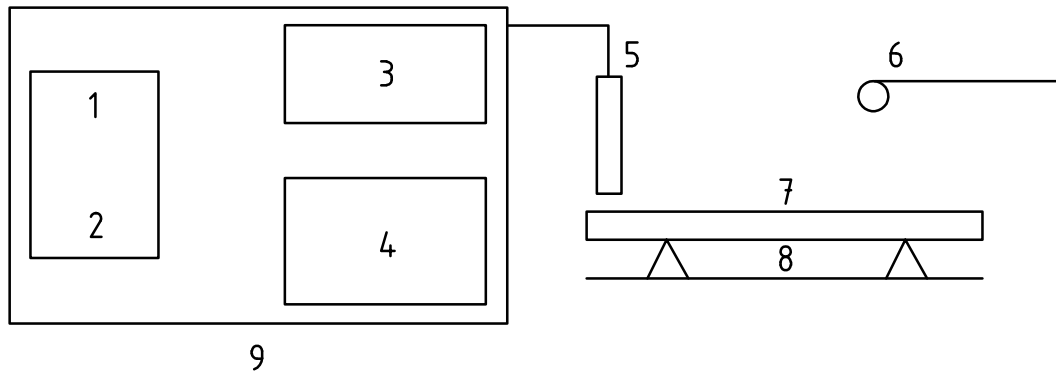
## 6 Apparatus

### 6.1 Excitation apparatus

This apparatus is used to excite vibrations in the test specimens and then accurately detect, analyse and measure the fundamental resonant frequency or period of a vibrating beam. Figure 1 shows a block diagram of such an apparatus. It consists of a small hammer, a suitable pickup transducer to convert the mechanical vibrations into electrical signals, an electronic signal analyser system consisting of a signal conditioner/amplifier, a signal analyser and a frequency read-out device <sup>1)</sup>.

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1) An example of a suitable instrument is the Grindosonic instrument, manufactured by J.W. Lemmens, Inc., 3466 Bridgeland Drive, Suite 230, St. Louis MO, 63044-2602 USA. This information is given for the convenience of users of this part of ISO 12680 and does not constitute an endorsement by ISO of this equipment.



**Key**

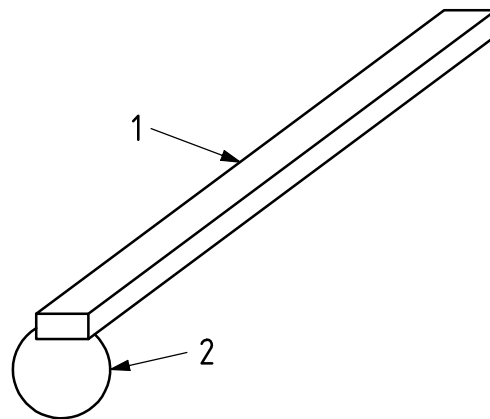
1 numerical display of the measured frequency	4 frequency analyser	7 test specimen
2 read-out device	5 transducer	8 support system
3 signal amplifier	6 impulser	9 electrical system

**Figure 1 — Block diagram of typical test apparatus**

**6.2 Striker hammer**

The hammer shall have a mass sufficient to induce a measurable mechanical vibration in the test specimen but shall be not large enough to physically displace or damage the test specimen. A typical small hammer is shown in Figure 2. Larger specimens may require larger striker hammers.

NOTE The size of the striker hammer depends on the size and physical properties of the specimens to be tested.



**Key**

1 flexible polymer rod
2 steel or other hard metal ball

**Figure 2 — Typical design for striker hammer**

**6.3 Signal pickup**

The excited vibrational signals in the test specimens are detected by transducers in direct contact with the specimen or by non-contact transducers. Common contact transducers are accelerometers using piezoelectric or strain gauge devices to measure vibration. A common non-contact transducer is an acoustic microphone, but laser, magnetic or capacitance methods may be used as well. The frequency range of the transducer shall be sufficient to measure the expected frequencies of the test specimens. A suitable range is 50 Hz to 20 kHz for many refractory test specimens. Smaller and stiffer specimens vibrate at higher frequencies. The frequency response of the transducer across the frequency range of interest shall have a bandwidth of at least 10 % of the maximum measured frequency before -3 dB power loss occurs.



## 6.4 Electronic signal analysis system

The system consists of a signal conditioner/amplifier, signal analyser and frequency read-out device. The system shall have sufficient accuracy and precision to measure the test specimen's frequencies to an accuracy of 0,1 %. The signal conditioner/amplifier shall be suitable to power the transducer and to provide an appropriate amplified signal to the signal analyser. The signal analyser consists of a frequency counting device and a read-out device. Appropriate devices are frequency counter systems with storage capacity or digital storage oscilloscopes with frequency counter modules. With the digital storage oscilloscopes, a Fast Fourier Transform signal analysis system may be useful for analysing more complex waveforms and identifying the fundamental resonant frequency of the test specimen.

## 6.5 Specimen support

The specimen supports serve to isolate the specimens from extraneous vibrations without restricting the desired mode of vibration of the specimens. The support materials shall be stable at the test temperature. For ambient conditions, support materials may be either soft or rigid. Examples of soft materials are compliant elastomerics such as polyurethane foam strips. Specimens should be supported on flat surfaces of these foam strips. Rigid material supports, such as metal or ceramic, shall have sharp knife edges or cylindrical surfaces for the test specimens to rest on. The rigid supports, in turn, shall rest on isolation pads to prevent the pick-up of spurious vibrations by the specimen and then the transducer. Wire suspension specimen support systems may also be used. Support the specimens at the nodes located at 0,224  $L$  of the total length measured from each end of the specimen (see 9.4.1).

## 7 Sampling

The number of specimens to be tested shall be determined in accordance with ISO 5022 for shaped products or ISO 8656-1 for unshaped products, or using a sampling plan agreed upon between the interested parties.

## 8 Test specimens

### 8.1 Specimen geometry

The specimens shall be simple beams or slender rods, either rectangular or circular in cross section. A minimum ratio of 3 for the length to cross-section thickness or diameter shall be used.

### 8.2 Specimen dimensions

Resonant frequencies of specimens are a function of the specimens' dimensions as well as their masses and moduli of elasticity. Using an estimated modulus, specimen size shall be chosen so that the expected resonant frequencies fall within the frequency response range of the transducers and the signal analysis system to be used. The smallest dimension of the specimen shall be at least 4 times the largest grain or particle size.

### 8.3 Surface finishing of specimens

All surfaces of a rectangular specimen shall be flat and sufficiently parallel so that opposite faces across the length, width and thickness dimensions are parallel to within 1 %. The diameter of a cylindrical specimen shall vary by no more than 1 %.

## 9 Procedure

### 9.1 Determination of specimen mass

Determine the mass of specimens to a precision of 0,2 %.

Determine the specimen length, width and thickness to a precision of 0,2 %.

## 9.2 Stabilization of electrical equipment

Activate all electrical equipment and allow it to stabilize.

If proprietary equipment is used, stabilization shall be carried out according to the manufacturer's recommendations.

## 9.3 Accuracy and response of equipment

Verify the response and accuracy of the equipment using a specimen whose modulus of elasticity was determined previously.

## 9.4 Determination of fundamental flexural resonant frequency (out-of-plane flexure)

**9.4.1** Place the specimen on supports located at the fundamental nodal points, i.e.  $0,224 L$  from each end (see Figure 3).

The specimen may be placed on a foam pad as an optional support system.

**9.4.2** Determine the direction of maximum sensitivity for the transducer. Orient the transducer in the direction that will utilize this maximum sensitivity to detect the desired vibrations.

### a) Direct-contact transducers

Place the direct-contact transducer in contact with the test specimen to pick up the desired vibration. If the transducer is placed at an antinode (a location of maximum displacement), its mass may load the test specimen and modify the natural vibration. The transducer should preferably be placed only as far from the nodal points as necessary to obtain a reading (see Figure 3). This location will minimize the damping effect of the contacting transducer. The transducer contact force should be sufficient to obtain a good response, yet only minimally interfere with the free vibration of the specimen.

### b) Non-contact transducers

Place the non-contact transducer over an antinode point, close enough to the test specimen to pick up the desired vibration but not so close as to interfere with the free vibration of the test specimen (see Figure 3).

**9.4.3** Strike the test specimen lightly either at the centre of the specimen or at the opposite end of the specimen from the detecting transducer (see Figure 3).

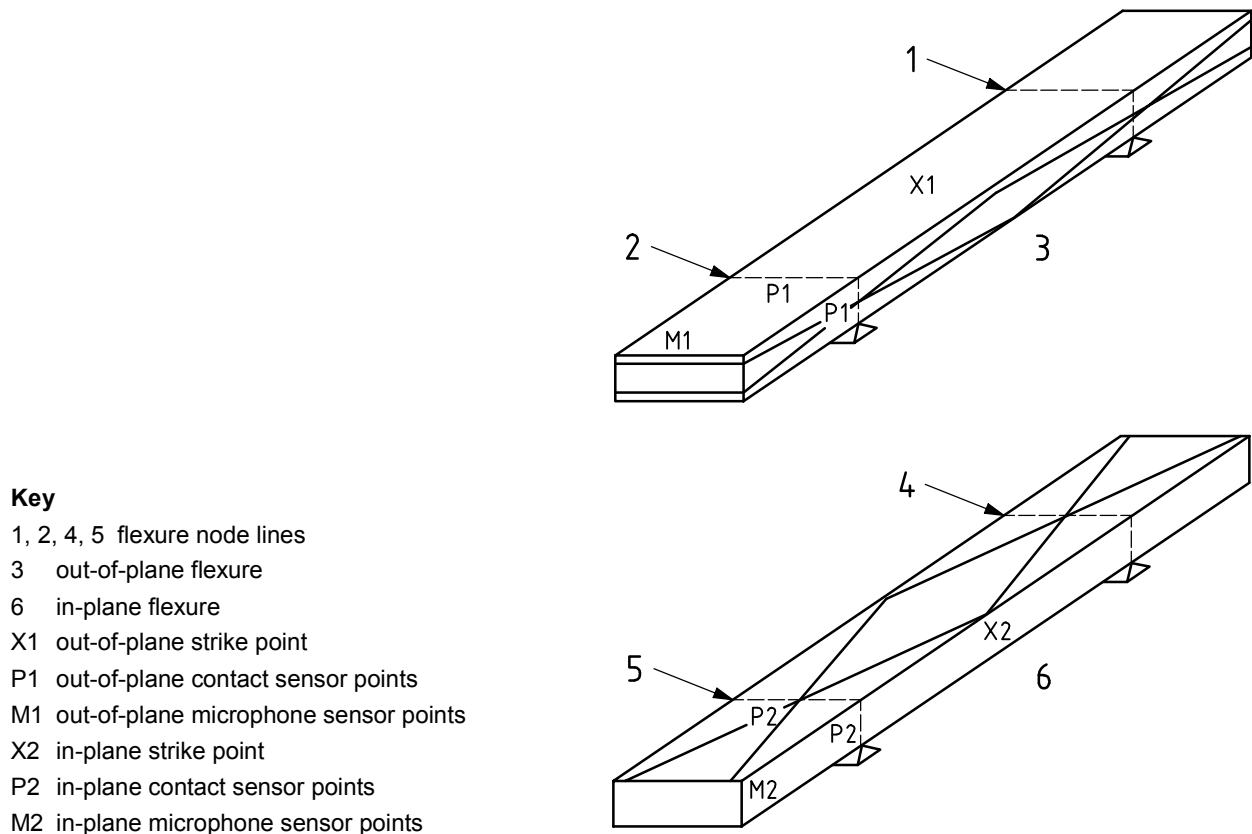
**9.4.4** Record the reading from the electronic frequency analyser system. Repeat this striking and reading sequence until five consecutive readings lie with 1 % of each other. Use the mean average of these five readings to determine the fundamental resonant frequency of the test specimen in flexure.

## 9.5 Determination of fundamental flexural resonant frequency (in-plane flexure)

Carry out the procedure given in 9.4 and measure the vibration of the test specimen in the major plane of the specimen. This measurement can be carried out in either of two ways:

- a) rotate the transducer and striker hammer impact locations around the specimen's long axis  $90^\circ$  from their orientation when measuring the out-of-plane flexure; or
- b) rotate the specimen  $90^\circ$  around its long axis and reposition it on its supports.

When calculating the modulus of elasticity from these in-plane flexure measurements, transpose the width and thickness dimensions from the formula used for the out-of-plane flexure frequency determination of the modulus. For homogeneous, isotropic materials, the moduli calculated from the in-plane flexure tests should be the same as those calculated from the out-of-plane flexure tests. Thus the comparison of the in-plane and the out-of-plane measurements can be used as a check of the experimental methods and calculations.



**Figure 3 — Rectangular specimen tested for out-of-plane flexure and in-plane flexure**

## 10 Calculations

### 10.1 Rectangular specimens

**10.1.1** Calculate the Young's modulus,  $E$ , in pascals, for a rectangular specimen using the basic equation:

$$E = 0,946 5 \left( \frac{mf_1^2}{b} \right) \left( \frac{L^3}{t^3} \right) T_1 \quad (1)$$

where

$m$  is the mass of the test specimen, in grams;

$b$  is the width of the test specimen, in millimetres;

$L$  is the length of the test specimen, in millimetres;

$t$  is the thickness of the test specimen, in millimetres;

$f_1$  is the fundamental resonant frequency of the test specimen in flexure, in hertz;

$T_1$  is the correction factor for the fundamental flexural mode to account for the finite thickness of the bar, Poisson's ratio, etc. calculated from Equation (2):

$$T_1 = 1 + 6,585 (1 + 0,075 2 \mu + 0,810 9 \mu^2) \left( \frac{t}{L} \right)^2 - 0,868 \left( \frac{t}{L} \right)^4 - J \quad (2)$$

where

$$J = \frac{\left[ 8,340 (1 + 0,202 3 \mu + 2,173 \mu^2) \left(\frac{t}{L}\right)^4 \right]}{\left[ 1 + 6,338 (1 + 0,140 8 \mu + 1,536 \mu^2) \left(\frac{t}{L}\right)^2 \right]} \quad (3)$$

and  $\mu$  is Poisson's ratio

**10.1.2** If  $L/t$  is equal to or greater than 20,  $T_1$  can be simplified (see 10.2.1) to

$$T = 1 + 6,585 \left(\frac{t}{L}\right)^2 \quad (4)$$

and  $E$  can be calculated directly.

**10.1.3** If  $L/t$  is less than 20 and Poisson's ratio is known,  $T_1$  can be calculated from Equation (2) and then used to calculate Young's modulus,  $E$ .

NOTE The frequency ( $f_1$ ) is the flexural frequency.

**10.1.4** Young's modulus can be calculated from the flexural resonant frequency alone by assuming a value for Poisson's ratio. Unless the value of Poisson's ratio is known or measured by other means, 0,15 shall be used as it is considered an appropriate value for most refractories (see 10.2.3).

## 10.2 Cylindrical rod specimens

**10.2.1** Calculate Young's modulus,  $E$ , in pascals, for a cylindrical rod specimen using the basic equation:

$$E = 1,606 7 \left(\frac{L^3}{D^4}\right) (mf_1^2) T_1 \quad (5)$$

where

$m$  is the mass of test specimen, in grams;

$L$  is the length of test specimen, in millimetres;

$D$  is the diameter of test specimen, in millimetres;

$f_1$  is the fundamental resonant frequency of test specimen in flexure, in hertz;

$T_1$  is the correction factor for the fundamental flexural mode to account for the finite diameter of the specimen, Poisson's ratio, etc., calculated from the equation

$$T_1 = 1 + 4,939 (1 + 0,075 2 \mu + 0,810 9 \mu^2) \left(\frac{D}{L}\right)^2 - 0,488 3 \left(\frac{D}{L}\right)^4 - J \quad (6)$$

where

$$J = \frac{\left[ 4,691 (1 + 0,202 3 \mu + 2,173 \mu^2) \left(\frac{D}{L}\right)^4 \right]}{\left[ 1 + 4,754 (1 + 0,140 8 \mu + 1,536 \mu^2) \left(\frac{D}{L}\right)^2 \right]} \quad (7)$$

and  $\mu$  is Poisson's ratio.

**10.2.2** If  $L/D$  is equal to or greater than 20, then  $T_1$  can be simplified to

$$T_1 = \left[ 1 + 4,939 \left( \frac{D}{L} \right)^2 \right] \quad (8)$$

and  $E$  can be calculated directly.

**10.2.3** If  $L/D$  is less than 20 and Poisson's ratio is known,  $T_1$  can be calculated from Equation (6) and then used to calculate Young's modulus,  $E$ .

NOTE The frequency ( $f_1$ ) is the flexural frequency.

### 10.3 Alternative calculations

#### 10.3.1 Calculation from flexural resonant frequency

Young's modulus can be calculated from the flexural resonant frequency alone by assuming a value for Poisson's ratio. Unless the value of Poisson's ratio is known or measured by other means, 0,15 is considered an appropriate value for most refractories.

#### 10.3.2 Calculation at temperatures other than room temperature

If measurements are made at temperatures other than room temperature, calculate Young's modulus at elevated temperature,  $E_T$ , in pascals, corrected for the thermal expansion effects, using Equation (9):

$$E_T = E_0 \left( \frac{f_T}{f_0} \right)^2 \left[ \frac{1}{1 + \alpha (T_T - T_0)} \right] \quad (9)$$

where

$E_0$  is the Young's modulus at room temperature ( $T_0$ );

$f_T$  is the resonant frequency of specimen at elevated temperature;

$f_0$  is the resonant frequency of specimen at room temperature;

$\alpha$  is the average linear thermal expansion coefficient of specimen from room temperature to elevated temperature, expressed as change in length per unit length per degree Celsius.

## 11 Test report

The test report shall include the following information:

- a) all information necessary for identification of the sample tested;
- b) a reference to this International Standard (ISO 12680-1:2005);
- c) details of the method used, including
  - 1) the number of items tested,
  - 2) the size and shape of the test specimens,
  - 3) the condition of the specimens, including the method of preparing the specimens from the refractory shapes submitted for test,
  - 4) the temperature of the specimens when being tested, and
  - 5) the Poisson ratio used in the calculations and how it was obtained;
- d) the results of the test, including the results of the individual determinations and their mean, calculated as specified in Clause 10, including numerical values obtained for the measured fundamental resonant frequencies and the calculated dynamic Young's moduli of elasticity;
- e) any observations of the test specimens or the performance of the test, including those made during the test, which may assist the interpretation of the results;
- f) the name of the test establishment;
- g) any deviations from the procedure specified;
- h) any unusual features (anomalies) observed during the test;
- i) the date and time the test was begun and terminated.

## Annex A (informative)

### Factors affecting accuracy of determinations

A propagation of errors analysis of the equations for calculating  $E$ , using the tolerances for dimensions, mass and frequency measurements noted in this method, has shown that a 1 % error in the measurement of the key variables produces a range of errors in the calculated modulus of elasticity, which is based on the variable's exponent in the equation used to calculate the modulus. Table A.1 shows error in the calculated modulus of elasticity resulting from errors in the different key measured variables.

**Table A.1 — Effect of measured variables errors on calculated modulus of elasticity**

Variable	Measurement error %	Variable exponent in modulus equation	Calculated modulus of elasticity error %
Frequency, $f$	1	$f^2$	2
Length, $L$	1	$L^3$	3
Mass, $m$	1	$m$	1
Width, $b$	1	$b^{-1}$	1
Thickness, $t$	1	$t^{-3}$	3
Diameter, $D$	1	$D^{-4}$	4

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