INTERNATIONAL STANDARD

ISO 6721-8

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Plastics — Determination of dynamic mechanical properties —

Part 8:
Longitudinal and shear vibration — Wave-propagation method

Plastiques — Détermination des propriétés mécaniques dynamiques —

Partie 8: Vibrations longitudinale et en cisaillement — Méthode de propagation des ondes

Reference number
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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.

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.

International Standard ISO 6721-8 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 2, *Mechanical properties*.

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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 8:
Longitudinal and shear vibration — Wave-propagation method

1 SCOPE

This part of the International Standard ISO 6721 describes an ultrasonic wave propagation method for determining the storage components of the longitudinal complex modulus L^* and the shear complex modulus G^* of polymers at discrete frequencies typically in the range 0.5 MHz to 5 MHz. The method is suitable for measuring materials with storage moduli in the range 0.01 GPa to 200 GPa and with loss factors below 0.1 at around 1 MHz. With materials that have a higher loss, significant errors in velocity measurement are introduced through waveform distortion and can only be reduced using procedures that are outside the scope of this standard.

The method allows measurements to be made on small specimens, typically 50 mm x 20 mm x 5 mm, or small regions of larger specimens or sheets. It is therefore possible to obtain information on the homogeneity or anisotropy (see clause 10.5) of modulus in a specimen.

2 NORMATIVE REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to use the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1183:1987, Plastics - Methods for determining the density and relative density of non-cellular plastics.

ISO 6721-1:1994, Plastics - Determination of dynamic mechanical properties - Part 1: General principles.

3 DEFINITIONS

See ISO 6721-1, 4.

3.1 LONGITUDINAL MODULUS

The ratio of a uniaxial tensile or compressive stress applied to a specimen to the resulting uniaxial strain when the strain in a plane transverse to the axis of applied stress is zero. See table 5 in ISO 6721-1 for relationships between this and other moduli.

3.2 LONGITUDINAL ACOUSTIC WAVE

A sound wave in which the particle displacement is in the direction of wave propagation.

3.3 TRANSVERSE ACOUSTIC WAVE

A sound wave in which the particle displacement is perpendicular to the direction of wave propagation.

3.4 BULK WAVE

The mode of propagation of an acoustic wave in a material whose boundaries normal to the direction of propagation are infinitely remote. This mode is realised in practice for waves whose wavelength is much less than the dimensions of the specimen transverse to the direction of propagation. In practice, the acoustic wave frequency is then ultrasonic.

4 PRINCIPLE

Measurements are made of the velocity of longitudinal and transverse acoustic waves in a specimen and the specimen density. The frequency of the wave is chosen so that its wavelength in the specimen is significantly less than the specimen dimensions in a plane transverse to the direction of wave propagation. The wave then propagates as a bulk wave. The longitudinal and shear storage moduli are given by the product of the material density and the square of the longitudinal and the shear wave velocities respectively.

Two methods are described in this international standard for measuring wave velocities. In the immersion method, the specimen intercepts a beam of longitudinal acoustic wave pulses passing between a transmitting and receiving transducer in a bath of a suitable liquid. At normal incidence, longitudinal wave pulses are excited in the specimen. As the angle of incidence is increased, the amplitude of the longitudinal refracted wave decreases and a refracted transverse (shear) wave is generated. Longitudinal and transverse wave velocities are deduced from measurements of differences in pulse transit times with and without the specimen in the beam and a knowledge of the velocity of sound in the liquid.

In the transducer contact method, the specimen is sandwiched between two transducers, one launching and the other receiving acoustic wave pulses. For the determinaion of longitudinal and transverse wave velocities, transducer pairs having longitudinal and transverse polarisations, respectively, are used. Wave velocities are again obtained from measurements of differences in pulse transit times with and without the specimen in the beam.

5 TESTING DEVICE

5.1 APPARATUS

The requirements of the apparatus are that it shall enable measurement of the velocities of longitudinal and transverse ultrasonic waves in a specimen. Two methods are described in this International Standard.

5.1.1 Method A: Immersion method

Figure 1a shows, schematically, suitable apparatus for measuring velocity by an immersion method. Two ultrasonic transducers are mounted coaxially in a bath containing a liquid, one acts as a transmitter T of longitudinal ultrasonic wave pulses and the other as a receiver R. The transmitter is driven by a series of high-voltage, short-duration electrical pulses from the transducer drive unit. A pulse repetition interval of about 1 ms is satisfactory. Acoustic pulses launched by the transmitter travel through the liquid and the specimen and are detected by the receiving transducer. The specimen is mounted on a turntable, located between the transducers T and R, such that the angle of incidence of the acoustic beam can be varied and measured to $\pm$ 0.5°. The specimen can be removed from the beam. The receiving transducer is connected to electronic equipment that will enable measurement of the difference in the arrival times of pulses received with and without the specimen in the beam. An oscilloscope, whose timebase is accurately calibrated and triggered by the transducer drive unit, is suitable for this purpose.

The receiving transducer may be replaced by a reflecting surface, such as a metal block, positioned normal to the axis of the transmitter as shown in figure 1b. The transmitting transducer is now used to detect the beam of pulses reflected back through the liquid and the specimen and is connected to the transit-time measuring equipment (see note 1).

(Note 1. This test arrangement may be more appropriate if the specimen is only available as a thin sheet since the transit time in the specimen is twice that obtained using the transmitter and receiver arrangement.)

5.1.2 Method B. Transducer contact method

Figure 2a shows a method for measuring wave velocity by direct contact between the transmitting and receiving transducers and the surfaces of the specimen. For the determination of the longitudinal wave velocity, transducer pairs that launch and receive longitudinal acoustic waves are used whilst, for the determination of the transverse wave velocity, shear (transverse) wave transducers are employed. The transducer separation can be varied to accommodate specimens of different thickness including direct contact between the two transducers. A coupling fluid is necessary to maximise the pulse amplitude transmitted to the specimen and to the receiver.

The receiver may be replaced by a reflecting surface in contact with the specimen as shown in figure 2b. The transmitting transducer is now used to detect the beam of pulses reflected back through the specimen and is connected to the transit time measuring equipment (see note 1).

5.2 TRANSDUCERS

When driven by the transducer drive unit, the transmitter should produce a short pulse at its natural frequency that has a duration of around three or four cycles. A suitable waveform is shown in figure 3. Pulses of longer duration are satisfactory but may not allow measurement of wave velocities by timing the interval between pulses that have been internally reflected by the specimen surfaces owing to an overlap of those pulses.

In either of the test arrangements shown in figure 2, the transmitter should possess a suitable buffer material located between the acoustic resonating device and the surface of the transmitter in order to prevent the contact with the specimen, the receiver or the reflector from influencing the acoustic performance of the transmitter and hence the shape of the pulses generated.

5.3 TRANSIT-TIME MEASURING EQUIPMENT

Data processing equipment shall be capable of measuring the time interval between two received pulses to an accuracy of ± 0.5% of the time interval (see note 2).

(Note 2. The time interval between received pulses will depend upon the thickness of the specimen and the wave velocity in the material. For attenuating materials, such as most polymers, where specimens of only a few millimetres in thickness can be used, time intervals will be in the region of one microsecond.)

The use of a digital storage oscilloscope having a high sampling rate or an oscilloscope whose time base is triggered by the transducer drive unit through an accurate digital delay circuit are suitable for this purpose.

5.4 TEMPERATURE MEASUREMENT AND CONTROL

See ISO 6721-1, sub clause 5.5 and note 3.

(Note 3. The determination of wave velocity using the methods described in this standard involve measuring the time interval between two received pulses. When these pulses are obtained with and without the specimen in the acoustic beam, it is important that the temperature of the apparatus has not changed significantly between the two measurements. As general guidance, any temperature change should be less than 0.5 °K using the transducer contact method and less than 0.2 °K using the immersion method.)

6 TEST SPECIMENS

See ISO 6721-1, clause 6.

6.1 SHAPE AND DIMENSIONS

Test specimens in the shape of a bar or plate are suitable. The surfaces normal to the wave direction must be smooth, plane and parallel over an area comparable with the area of the faces of the transmitter and receiver. The dimension d of the specimen in the wave direction shall not vary by more than ± 0.2% over this area.

In order to ensure that it is the bulk wave velocity that is measured (see clause 4.4), the dimensions transverse to the wave direction shall be greater than 3 x the longitudinal pulse wavelength in the specimen. The wavelength λ(m) can be calculated from a knowledge of the pulse frequency f(Hz) and the longitudinal wave velocity in the specimen v_L(ms^{-1}) using the equation

$$\lambda = \frac{v_L}{f} \qquad (1)$$

6.2 PREPARATION

See ISO 6721-1, sub clause 6.2.

7 NUMBER OF SPECIMENS

See ISO 6721-1, clause 7.

8 CONDITIONING

See ISO 6721-1, clause 8.

9 PROCEDURE

9.1 TEST ATMOSPHERE

See ISO 6721-1, sub clause 9.1.

9.2 MEASURING THE SPECIMEN DIMENSION

Measure the dimension of the specimen in the direction of wave propagation at 3 points within the area through which the ultrasonic beam will travel. If these measurements vary by more than ± 0.5%, identify a different region of the specimen or choose another specimen.

9.3 PERFORMING THE TEST

9.3.1 Method A: Immersion method

With the specimen absent from the ultrasonic beam, identify a reference point on the received pulse that may be used to accurately record an arrival time for the pulse. A point where the pulse amplitude passes through zero volts early in the pulse is recommended for this purpose as shown in figure 3. Record the reference point time (see note 4).

(Note 4. With viscoelastic or multiphase materials, changes in pulse shape after transmission through the specimen can arise because of dispersion or scattering. This leads to an error in transit-time measurement which is difficult to quantify. This error can be minimised by selecting a reference point near the leading edge of the pulse).

Place the specimen on the turntable and ensure that the incidence surface is perpendicular to within ±0.5° to the common axis of the transmitter and the receiver. Record the arrival time of the reference point on the pulse transmitted through the specimen. Rotate the turntable until the refracted transverse wave is near its maximum amplitude. Record the arrival time of the transmitted transverse wave pulse (see note 5) and the angle of incidence.

(Note 5. The amplitude of the refracted longitudinal wave will decrease as the angle of incidence increases. If both the refracted longitudinal and transverse waves are visible in the received waveform, the transverse wave will have the larger arrival time).

Further measurements of the arrival time of the refracted transverse wave pulse can be made at other angles of incidence to increase the accuracy of the transverse wave velocity measurement.

If additional pulses are visible in the received waveform caused by internal reflections at the specimen surfaces, then an alternative or additional measurement of wave velocity can be made by recording the arrival times of consecutive pulses. This alternative measurement is generally unsuitable at angles of incidence above zero since the internally reflected beam will be displaced from the beam axis leading to errors in arrival time measurement.

9.3.2 Method B: Transducer contact method

Bring the transmitting transducer into contact with the receiving transducer (figure 2a) or reflector (figure 2b) using a coupling fluid and sufficient pressure to give a received pulse whose amplitude and arrival time do not change significantly with any increase in the applied pressure. Record the arrival time of a suitable reference point on the received pulse as described in clause 9.3.1.

Place the specimen between the transmitting and receiving transducers or between the transmitter and the reflector. The contact pressure shall not cause a reduction in the specimen thickness or more than 0.5%. Record the arrival time of the reference point on the pulse transmitted through the specimen (see note 4).

Carry out these measurements using both longitudinal and transverse wave transducers.

If additional pulses are visible in the received waveform caused by internal reflections at the specimen surfaces, then an alternative or additional measurement of wave velocity can be made by recording the reference point times for consecutive pulses.

9.3.3 Measurement of the specimen density

Measure the density to an accuracy of ± 0.5% using one of the procedures described in ISO 1183.

9.4 VARYING THE TEMPERATURE

See ISO 6721-1, sub clause 9.4.

10 EXPRESSION OF THE RESULTS

10.1 NOMENCLATURE

d(m) - dimension of the specimen in the direction of wave propagation

t_L, t_T(s) - arrival times of longitudinal and transverse wave pulses, respectively

t_R(s) - pulse arrival time without the specimen in the beam

t_{2L}, t_{2T}(s) - arrival times of longitudinal and transverse wave pulses, respectively, that are produced by internal reflections at the specimen surfaces

I - angle of incidence of the acoustic beam on the specimen in the immersion method

R - angle of refraction of the acoustic beam by the specimen in the immersion method

$v_L(ms^{-1})$ - longitudinal wave velocity in the specimen

$v_T(ms^{-1})$ - transverse wave velocity in the specimen

$v_W(ms^{-1})$ - velocity of sound in the liquid at the temperature of the measurement using the immersion method

$\rho(kgm^{-3})$ - specimen density

10.2 DETERMINATION OF THE LONGITUDINAL WAVE VELOCITY v_L

10.2.1 Method A: The immersion method

Where measurements of longitudinal pulse arrival times have been made using both a transmitting and a receiving transducer (figure 1a), the wave velocity is given by

$$v_L = \frac{dv_W}{d - v_W(t_R - t_L)} \qquad (2)$$

If a single transducer test arrangement is employed (figure 1b), the wave velocity becomes

$$v_L = \frac{2dv_W}{2d - v_W(t_R - t_L)} \qquad (3)$$

If the arrival time of the longitudinal pulse that has been internally reflected at both specimen surfaces is measured, the wave velocity is calculated using

$$v_L = \frac{2d}{t_{2L} - t_L} \qquad (4)$$

for both transducer arrangements.

10.2.2 Method B. The transducer contact method

Where measurements of longitudinal pulse arrival times have been made using both transmitting and receiving transducers (figure 2a), the wave velocity is given by

$$v_L = \frac{d}{t_L - t_R} \tag{5}$$

If a single transducer test arrangement is employed (figure 2b), the wave velocity becomes

$$v_L = \frac{2d}{t_L - t_R} \tag{6}$$

If the arrival time of the pulse that has been internally reflected at both specimen surfaces is measured, the wave velocity is calculated using

$$v_L = \frac{2d}{t_{2L} - t_L} \tag{7}$$

for both transducer arrangements.

10.3 DETERMINATION OF THE TRANSVERSE WAVE VELOCITY v_T

10.3.1 Method A: The immersion method

Where measurements of transverse pulse arrival times have been made using a two transducer test arrangement (figure 1a), the wave velocity is given by

$$v_T = \frac{dv_w}{d\cos(R-I) - v_w(t_R - t_T)\cos R} \tag{8}$$

where $\tan R = \dfrac{d\sin I}{d\cos I - v_w(t_R - t_T)}$ (9)

If a single transducer arrangement is employed (figure 1b), the wave velocity becomes

$$v_T = \frac{2dv_w}{2d\cos(R-I) - v_w(t_R - t_T)\cos R} \tag{10}$$

where $\tan R = \dfrac{2d\sin I}{2d\cos I - v_w(t_R - t_T)}$ (11)

10.3.2 Method B: The transducer contact method

Where measurements of transverse pulse arrival times have been made using both transmitting and receiving transducers (figure 2a), the wave velocity becomes

$$v_T = \frac{d}{t_T - t_R} \tag{12}$$

If a single transducer test arrangement is employed (figure 2b), the wave velocity becomes

$$v_T = \frac{2d}{t_T - t_R} \tag{13}$$

If the arrival time of the transverse pulse that has been internally reflected at both specimen surfaces is measured, the wave velocity is calculated using

$$v_T = \frac{2d}{t_{2T} - t_T} \tag{14}$$

for both transducer arrangements.

10.4 CALCULATION OF DYNAMIC PROPERTIES

The longitudinal storage modulus L′ is calculated using

$$L' = \rho\, v_L^2 \tag{15}$$

The shear storage modulus G′ is calculated using

$$G' = \rho v_T^2 \tag{16}$$

The Youngs storage modulus E′ is given by

$$E' = \frac{G'(3L' - 4G')}{L' - G'} \tag{17}$$

The bulk storage modulus K′ is given by

$$K' = L' - 4G'/3 \tag{18}$$

The real part of the dynamic Poissons ratio ν′ is given by

$$v' = \frac{L'-2G'}{2(L'-G')} \quad (19)$$

$$= \frac{v_L^2-2v_T^2}{2(v_L^2-v_T^2)} \quad (20)$$

10.5 INFLUENCE OF MATERIAL ANISOTROPY

For isotropic materials, values for the longitudinal and transverse wave velocity are independent of the direction in the material along which the measurement is made. For anisotropic materials, measurements of v_L and v_T along different directions in a specimen can be interpreted to reveal directions of preferred fibre or molecular orientation and the extent of the associated anisotropy of properties. Equations (1) to (15) are then valid but equations (16) to (20) are no longer accurate. For these materials, all the moduli appropriate to the symmetry of the material can be determined from measurements of longitudinal and transverse wave velocities along suitable directions in the specimen. The data analysis needed for this is outside the scope of this standard.

For materials that are only weakly anisotropic, a mean value for v_T can be obtained from measurements of transverse wave velocity with direction R using equations (8) or (10) for different angles of incidence I. A mean value for G′ can then be calculated using equation (16) with an uncertainty that can be estimated from the variation of v_T with R. Using an average value for L′ obtained from measurements at normal incidence to different faces of the specimen, approximate values for E′, K′ and v′ can be calculated using equations (17) to (20).

11 PRECISION

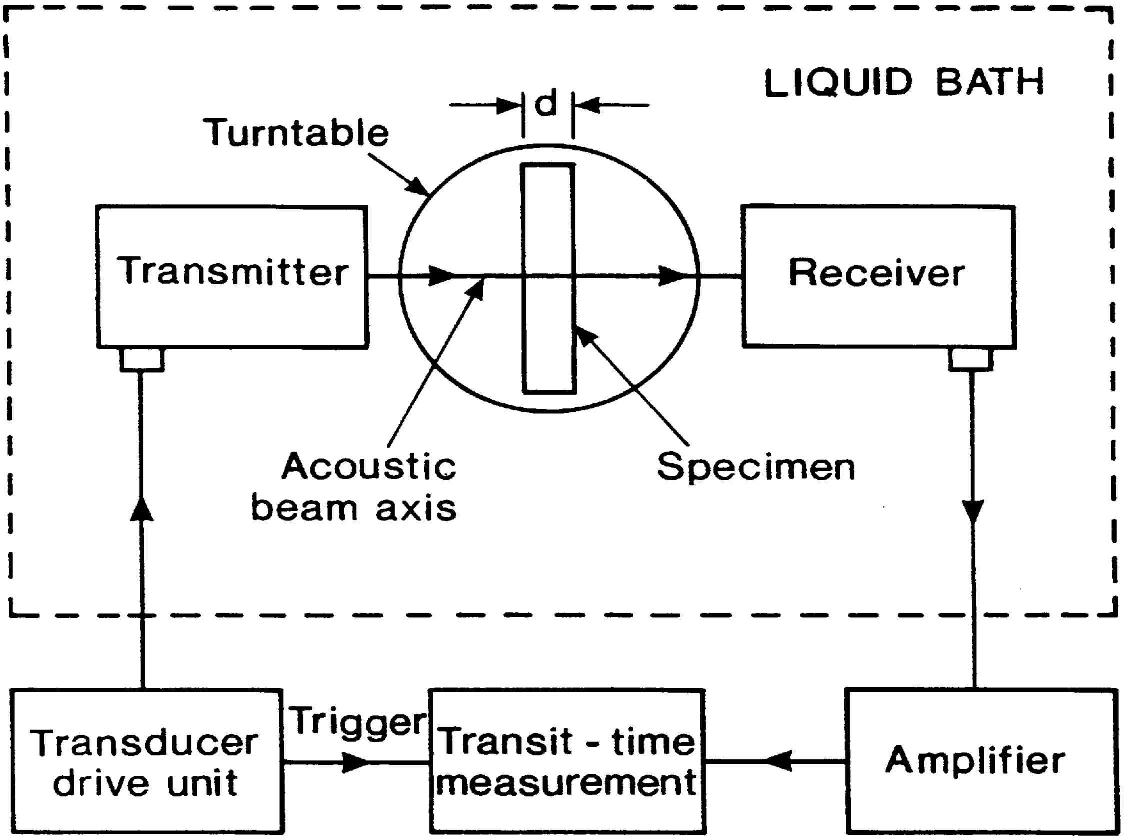
The precision of this test method is not known because interlaboratory data are not available. When interlaboratory data are obtained, a precision statement will be added with the next revision.

12 TEST REPORT

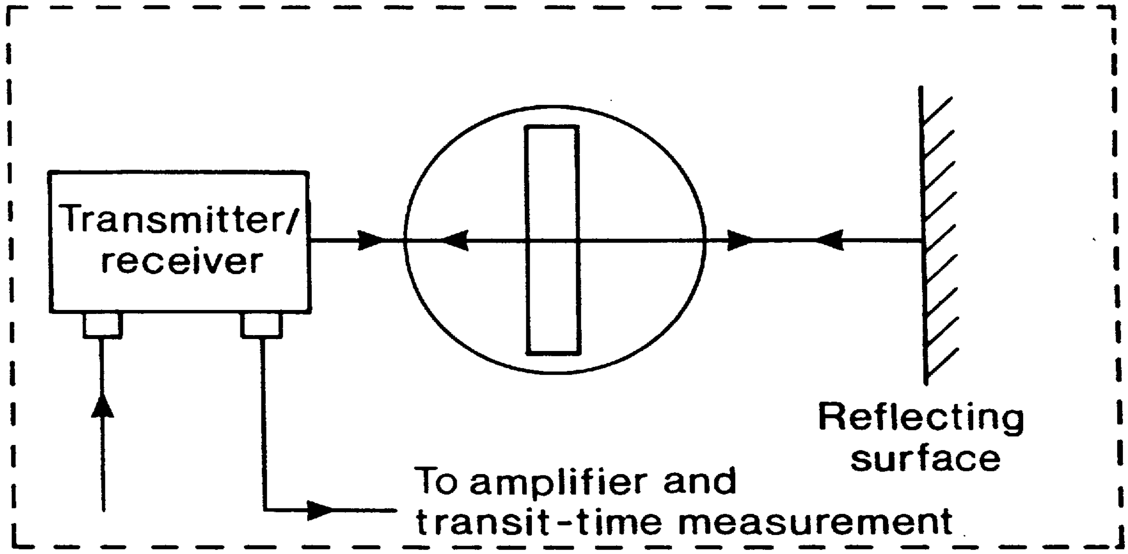
The test report shall contain the following information:

a) reference to this part of ISO 6721.

b) to m) see ISO 6721-1, clause 12.

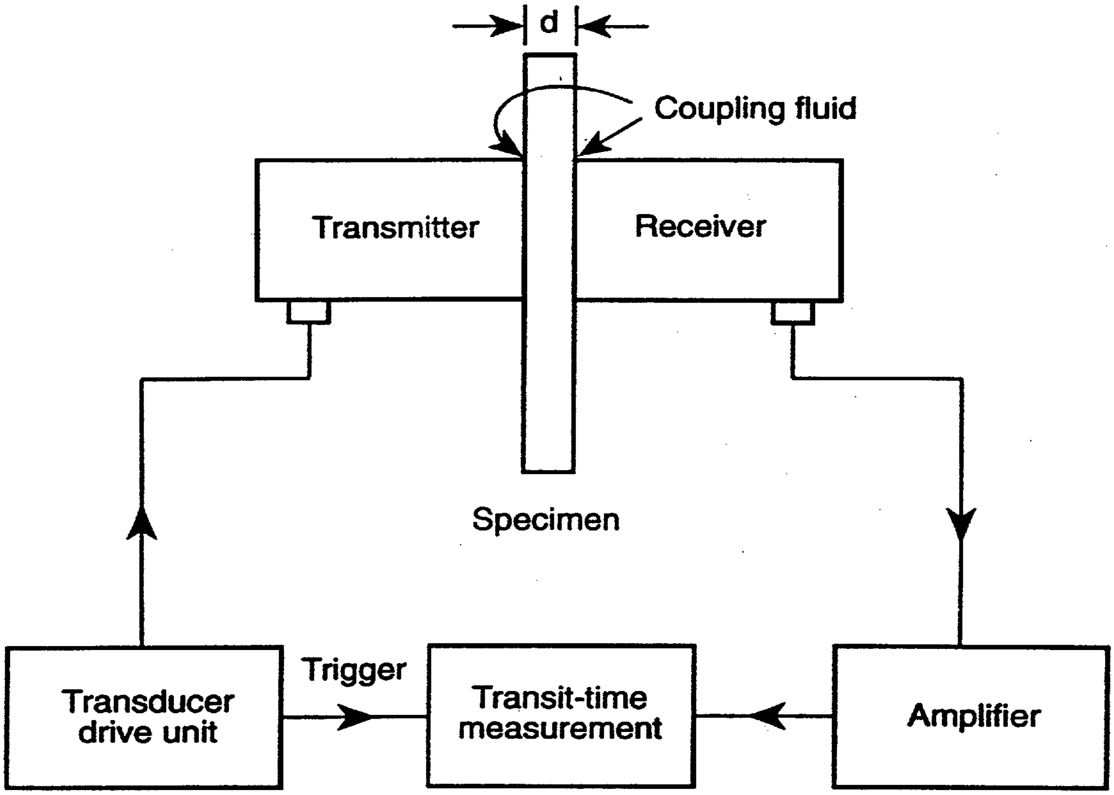


1 (a)

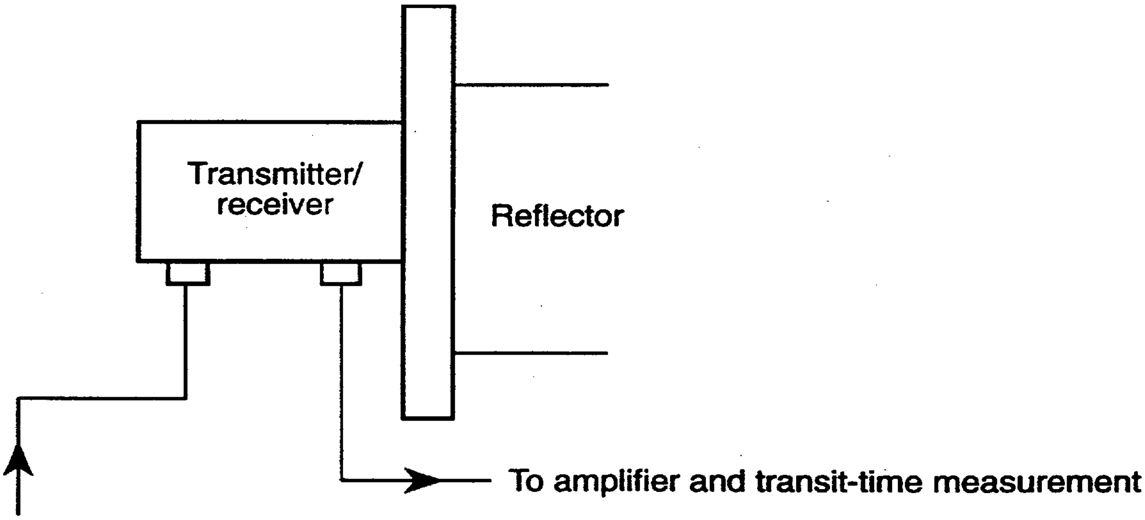


1 (b)

Figure 1 Measurement of ultrasonic wave velocity using the immersion method (a) with both a transmitting and a receiving transducer and (b) with a single transducer acting as a transmitter and a receiver



2 (a)



2 (b)

Figure 2 Measurement of ultrasonic wave velocity using the transducer contact method (a) with both a transmitting and a receiving transducer and (b) with a single transducer acting as a transmitter and a receiver.

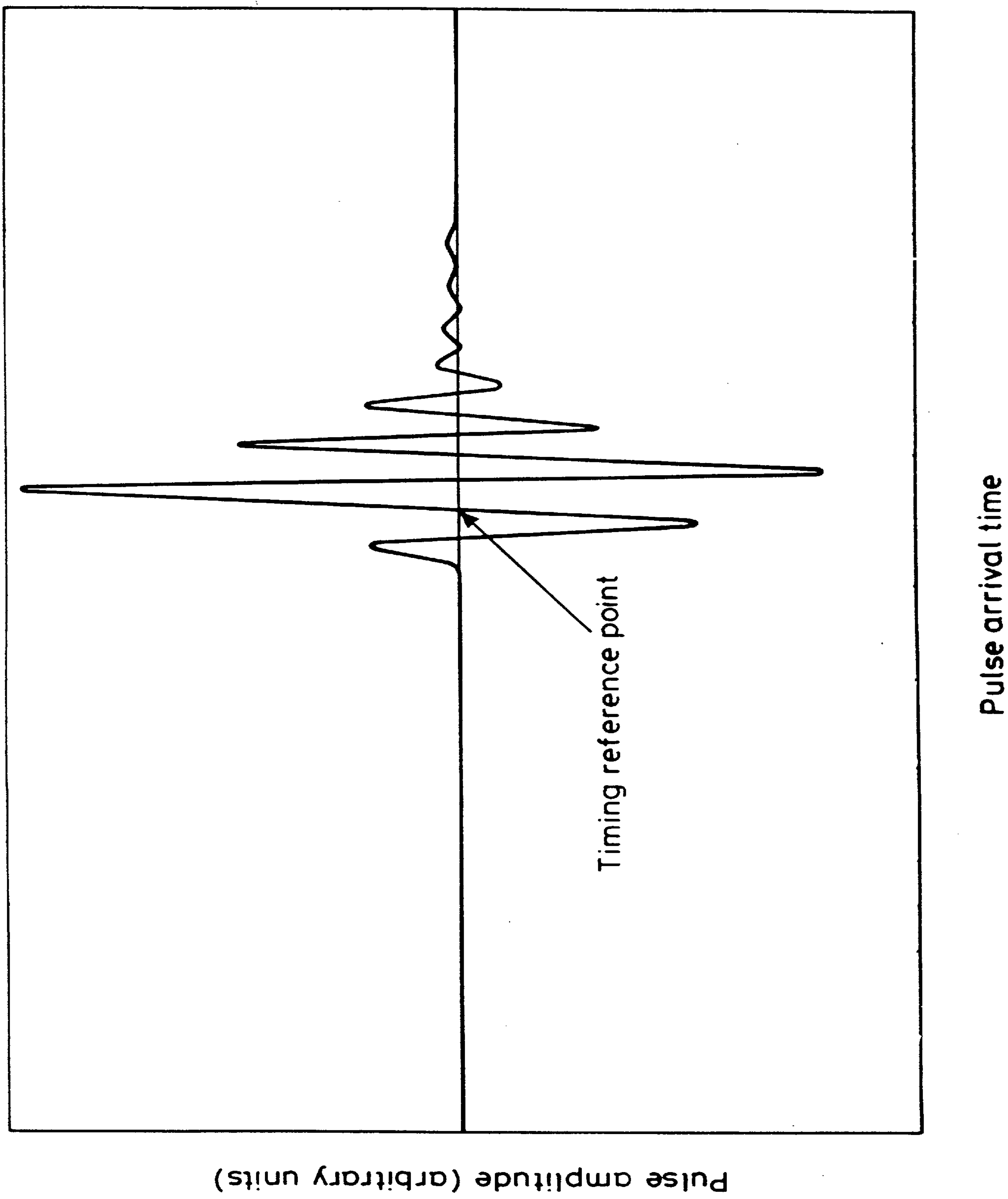


Figure 3 Waveform of a suitable pulse for the measurement of velocity.

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