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Rubber – Measurement of vulcanization characteristics with the oscillating disc curemeter

Caoutchouc – Détermination des caractéristiques de vulcanisation à l'aide du rhéomètre à disque oscillant

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FOREWORD

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3417 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products*, and was circulated to the member bodies in June 1974.

It has been approved by the member bodies of the following countries :

Australia	Italy	Sweden
Austria	Malaysia	Switzerland
Belgium	Mexico	Thailand
Brazil	Netherlands	Turkey
Canada	Poland	United Kingdom
Egypt, Arab Rep. of	Portugal	U.S.A.
Hungary	Romania	Yugoslavia
India	Spain	

The member bodies of the following countries expressed disapproval of the document :

Czechoslovakia
France
Germany

Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for determining selected vulcanization characteristics of a rubber compound by means of the oscillating disc curemeter.

2 PRINCIPLE

2.1 A test piece of rubber is contained in a sealed test cavity under an initial positive pressure and maintained at an elevated temperature. A biconical disc is embedded in the test piece and is oscillated through a small rotary amplitude. This action exerts a shear strain on the test piece and the force (torque) required to oscillate the disc depends on the stiffness (shear modulus) of the rubber. The torque is recorded autographically as a function of time.

NOTE — Direct proportionality between torque and stiffness cannot be expected under all test conditions because — particularly in higher torque ranges — an elastic deformation of disc shaft and driving device must be taken into account. Moreover, in cases of small amplitudes of deformation, the strain can be expected to have a considerable elastic component. For routine control purposes, corrections are not necessary.

2.2 The stiffness of the rubber test piece increases as vulcanization begins. A complete curve is obtained when the recorder torque either rises to an equilibrium value or to a maximum value (see figure 1). The time required to obtain a vulcanization curve is a function of the test temperature and the characteristics of the rubber compound.

2.3 The following measurements may be taken from the curve :

a) minimum torque;

b) time to incipient cure (scorch time);

c) time to a percentage of full cure;

d) maximum, plateau or highest torque attained within a specified period of time.

The minimum torque depends on the stiffness and the viscosity at low shear rate of the unvulcanized compound. The time of incipient cure is a measure of processing safety. The time to optimum cure is measured at some percentage of the highest torque and the highest torque is a measure of the stiffness of the fully cured vulcanizate at the test temperature. The cure rate index is the average slope of the rising cure curve.

3 APPARATUS

3.1 Curemeter, consisting of a biconical disc in a temperature-controlled die cavity. The shaft of the disc is secured in a drive shaft and oscillated through a small rotary amplitude.

The torque applied to the disc represents the resistance of the rubber test piece to deformation and is recorded autographically to yield a curve of torque versus time. The die-disc relationship is shown in figure 2.

3.2 Temperature measurement

3.2.1 The temperature-measuring systems shall enable the temperature of the dies to be measured within ± 0,1 °C. Calibrated thermocouples, or other suitable temperature sensors inserted in the dies, shall be used for periodically checking the die temperatures.

3.2.2 The dies should be mounted in electrically heated (aluminium) platens. Temperature controllers shall be used for controlling the temperature of each platen within ± 0,3 °C at steady state. If the temperature of the dies is set at 150 ± 0,3 °C, the heat conductance shall suffice to cause recovery of the initial temperature within 1,0 °C within 2 min after a test piece at 23 ± 5 °C is placed in the die chamber.

3.3 Die cavity

3.3.1 The dies shall be manufactured from a non-deforming tool steel having a minimum Rockwell hardness of HRC 50.

The geometry of the dies is shown in figures 3 and 4. The dimensions of the dies are given in tables 1 and 2. Suitable means shall be employed by design of dies or otherwise to apply pressure on the test piece throughout the test in order to minimize slippage between the disc and the rubber. Holes shall be drilled in both the upper and lower die member according to the dimensions given in tables 1 and 2 to enable temperature sensors to be inserted. Surfaces of the die cavity shall contain rectangular shaped grooves located at 20° intervals to minimize slippage. The lower die dimensions are given in table 1. The upper die member shall contain identically shaped grooves. The dimensions of the upper die are given in table 2.

3.3.2 The lower die shall have a hole in the centre to allow for insertion of the disc stem. A suitable low constant friction seal shall be provided in this hole to prevent material from leaking from the cavity.

3.4 Disc

The biconical disc shall be fabricated from a non-deforming tool steel having a minimum Rockwell hardness of HRC 50. The disc is shown in figure 5, and the critical dimensions are given in table 3.

3.5 Disc oscillation

The frequency of the rotary oscillation of the disc shall be 1,7 ± 0,1 Hz allowing for particular purposes to use other frequencies in the range 0,05 to 2 Hz. The maximum angular displacement of the disc shall be 1,00 ± 0,02° about its central position (total amplitude 2°) when the die cavity is empty.

Other frequencies and amplitudes may be used when specified for particular purposes. With different frequencies or amplitudes, different results will be obtained.

NOTES

1 With the die chamber empty, the amplitude of the rotor has to be maintained constant at 1,00° within a tolerance range of ± 0,02°. If a torque is acting on the disc, the resulting decrease in the angle of oscillation with increasing torque should be a linear function having a slope within the limits of 0,05 ± 0,002° /N·m. Suitable devices have to be provided to verify both the initial amplitude of oscillation and the decrease in amplitude with applied torque.

2 An initial amplitude of oscillation of 3° can be used in cases where danger of slippage between test piece and die cavity or disc can be excluded (first of all by regular cleaning of the rotor, see note to 7.2.2). A higher sensitivity in testing may be obtained at this amplitude, which may be useful in production control.

3.6 Die closure

The dies shall be closed and held closed during the test by a pneumatic cylinder with a force of 11,0 ± 0,5 kN.

3.7 Torque measurements

A device which produces a signal that is directly proportional to the torque required to turn the disc shall be used to measure the torque on the disc.

3.8 Recordings

A recorder shall be used to record the signal from the torque-measuring device. The recorder shall have a speed of response for full scale deflection on the torque scale of 1 s or less. The torque shall be recorded with an accuracy of ± 0,5 % of the torque range. Three torque ranges from 0 to 2,5, 0 to 5 and 0 to 10 N·m shall be provided.

4 CALIBRATION OF TORQUE TRANSDUCER AND RECORDER

4.1 Provisions shall be made for electronic verification of the recorder and torque transducer. One way is a calibration by means of a resistor incorporated in the torque-measuring circuit and which simulates an applied torque of specified value.

4.2 The torque-measuring system shall be calibrated by means of masses or by a torque standard system such as a calibrated torsion spring.

NOTE — In order to detect differences between curemeters or changes with use in a single curemeter, tests on referee compounds are useful. The referee compound should have a shear modulus equal to or greater than the production compounds being tested, and it should be homogeneous and stable for several weeks. Several tests should be made on calibrated curemeter(s) in good condition and a median curve established for the referee compound. Small changes with use or small differences between curemeters may be compensated by small adjustments in the torque span control to make subsequent tests of the referee compound agree with the established median curve. If large deviations from the median curve are observed, adjustment in the torque span control is not to be used for correction. The cause of the large deviation should be determined and necessary repairs or maintenance performed.

5 TEST PIECE

5.1 A test piece approximately 30 mm in diameter and 12,5 mm in thickness or of the equivalent volume shall be used for each test. Preferably, the test piece shall be cut from a previously sheeted sample, which shall be as free from air as practical. A total volume of 8 cm^3 for the test piece is considered optimum.

NOTE — In practice, the optimum sized test piece will be obtained by taking that mass of compound which will occupy the optimum volume.

5.2 Proper test piece size is assured if a small amount of compound is extruded between all edges of the dies. Oversized test pieces cool the cavity excessively during the early part of the test cycle and invalidate the test.

6 TEST TEMPERATURE

Recommended test temperatures are from 100 to 200 °C. Other temperatures may be used if necessary. Tolerances for these temperatures shall be ± 0,3 °C.

7 PROCEDURE

7.1 Preparation for test

Bring the temperature of both die members to the test temperature with the disc in place and the die cavity in the closed position. With the disc in place and the dies closed, adjust the recorder pen to the zero torque line on the chart. Position the pen to the zero time position on the chart. Calibrate the recorder if needed (see 4.1) and select the correct torque range.

7.2 Loading the curemeter

7.2.1 Open the dies, place the test piece on the top of the disc and close the dies within 5 s.

NOTE — When testing sticky compounds, insert some suitable thin film material below the rotor and above the test piece to keep the compound from sticking to the dies.

7.2.2 Time shall be counted from the instant the dies are closed. The disc may be oscillating at zero time or started not later than 1 min after the dies are closed.

NOTE — A deposit of material from the rubber compounds under test may build up on the disc and dies. This may affect the final torque values. It is suggested that stable compounds be tested daily to detect this occurrence. If such contamination develops, it may be removed by very light blasting with a mild abrasive. Extreme care must be used in this operation to retain sharpness of serrations and not change dimensions. Ultrasonic cleaning or cleaning with hot solvents or non-corrosive cleaning solutions may also remove the deposit. If solvent or solution cleaning is used the first two sets of results after cleaning should be rejected.

8 TEST REPORT

8.1 The following values, where applicable, shall be taken from the cure curve.

8.1.1 M_L = minimum torque, in newton metres.

8.1.2 M_{HF} = plateau torque, in newton metres.

8.1.3 M_{HR} = maximum torque (reverting curve), in newton metres.

8.1.4 M_H = highest torque value attained in a curve where no plateau or maximum value is obtained, in newton metres, after the specified time.

8.1.5 t_{sx} = time, in minutes, to x units of torque increase, above M_L.

8.1.6 $t_c(y)$ = cure time, in minutes, to y % of full torque development (see note below).

8.1.7 $t'_c(y)$ = cure time, in minutes, for torque to increase to $y/100\,(M_H - M_L) + M_L$ (see note below).

8.1.8 Cure rate index $= 100/(t_c(y) - t_{sx})$ parameter, proportional to the average slope of the cure rate curve in the steep region.

NOTE — These parameters are in a generalized format. Unless otherwise specified, it is recommended that the following specific parameters be used :

t_{s1} = time, in minutes, for torque to increase 0,1 N·m above M_L;

t'_c (50) = time, in minutes, for torque to reach $M_L + 0{,}5\,(M_H - M_L)$;

t'_c (90) = time, in minutes, for torque to reach $M_L + 0{,}9\,(M_H - M_L)$.

If an amplitude of 3° is used instead of the standard 1°, t_{s2} should be used in place of t_{s1}; i.e. time, in minutes, for the torque to increase 0,2 N·m above M_L.

8.2 The test report shall also include the following particulars :

a) conditions other than those specified in this International Standard;

b) nominal amplitude of oscillation; report as half of total displacement, i.e. 1° for a total displacement of 2°;

c) temperature of test, in degrees Celsius;

d) torque range selected, in newton metres;

e) frequency of oscillation, in hertz;

f) chart time motor speed, in minutes, for full-scale pen travel on recorder chart;

g) pre-heat time, if any, in minutes.

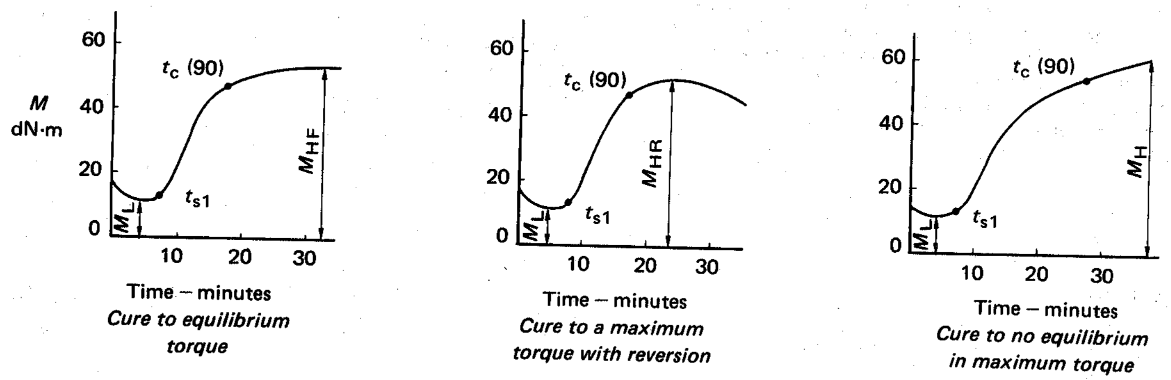


FIGURE 1 – **Types of vulcanization curves**

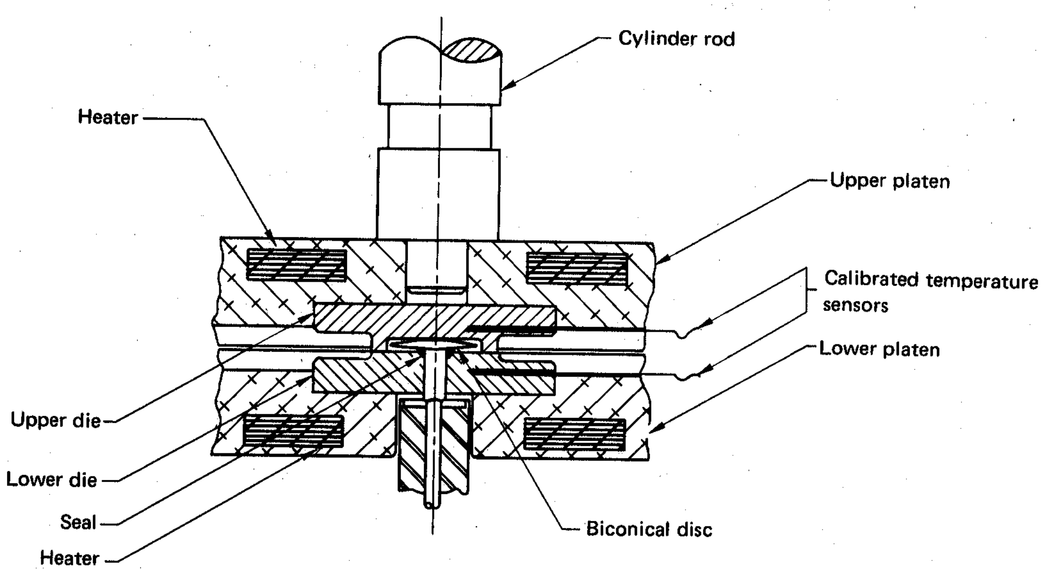


FIGURE 2 – **Curemeter assembly**

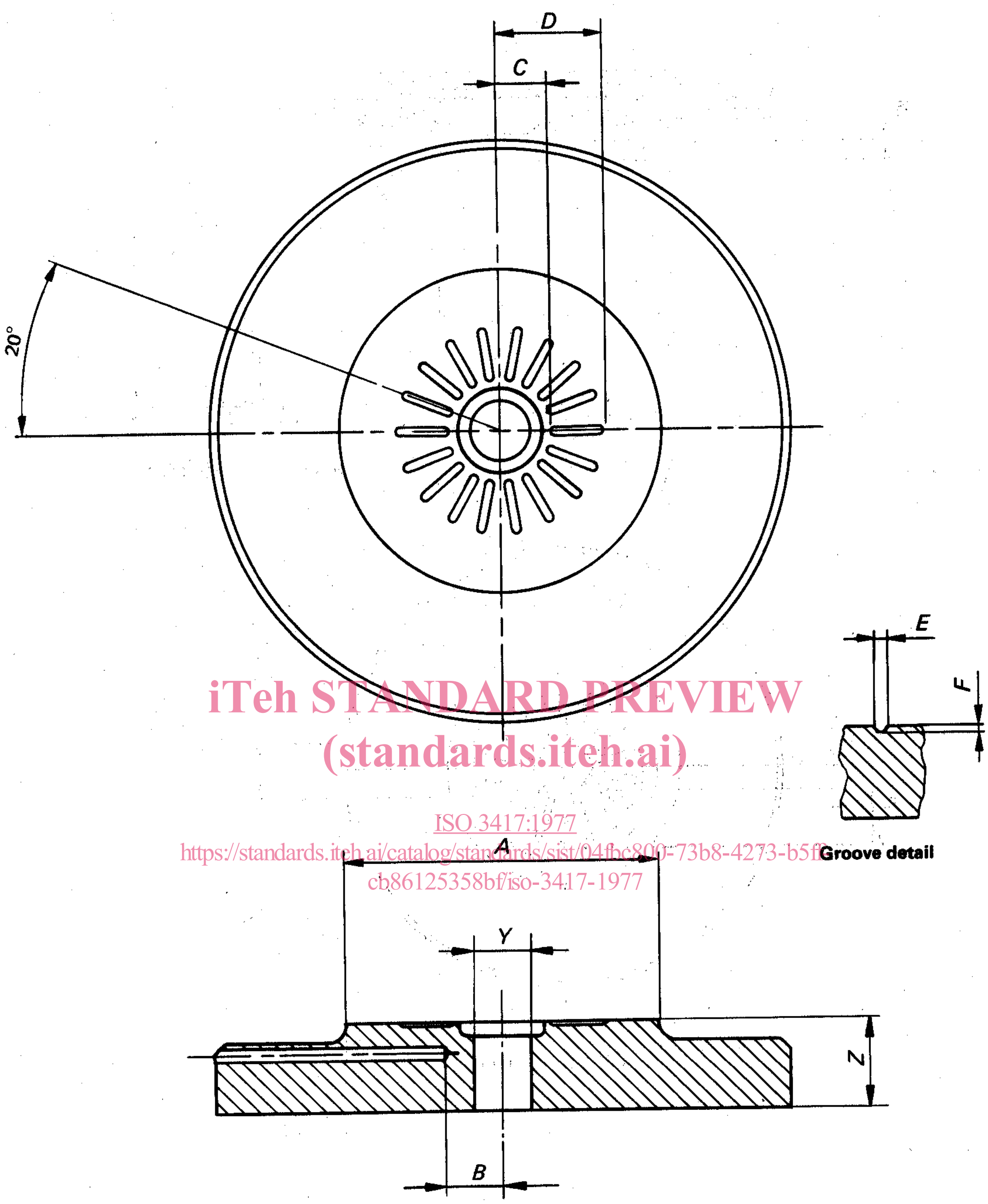
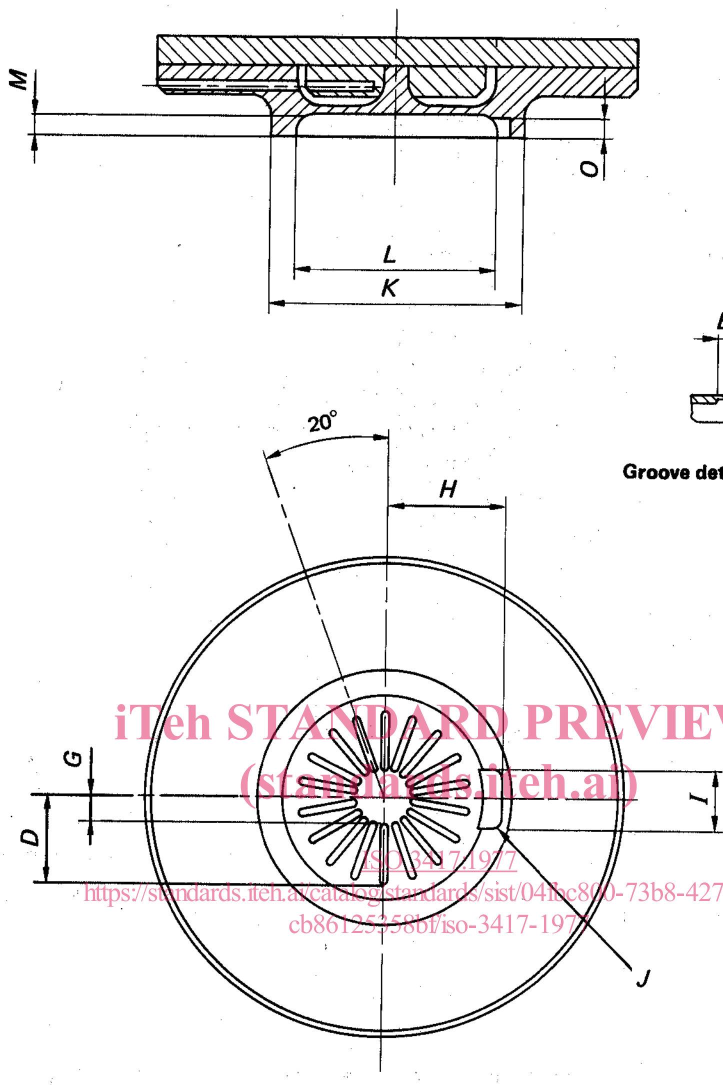
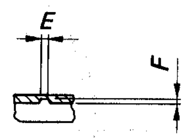


FIGURE 3 — **Lower die**

TABLE 1 — **Lower die dimensions**

Code	**Dimension** mm	**Tolerance** mm
A	55,9	± 0,2
B	10,6	± 0,3
C	8,0	± 0,2
D	18,3	± 0,2
E	1,6	± 0,2
F	0,8	± 0,1
Y	10,03	± 0,03
Z	16,4	± 0,2





Groove detail (see table 1)

FIGURE 4 – **Upper die**

TABLE 2 – **Upper die dimensions**

Code	Dimension mm	Tolerance mm
G	4,8	± 0,2
H	24,1	± 0,1
I	12,7	± 0,4
J	2,4	± 0,4
K	54,6	± 0,2
L	41,9	± 0,1
M	5,35	± 0,01
O	4,6	± 0,2

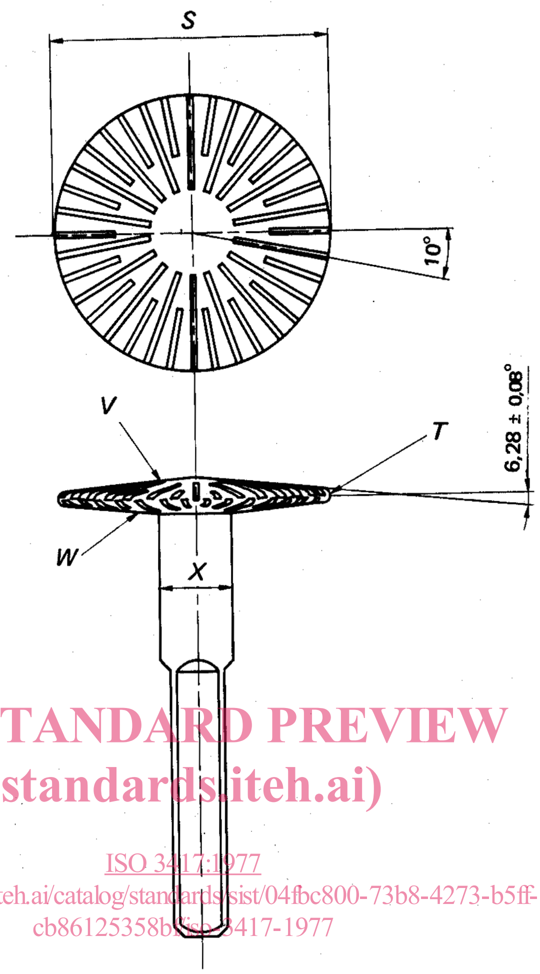


FIGURE 5 – **Biconical disc**

TABLE 3 – **Disc dimensions**

Code	**Dimension** mm		**Tolerance** mm
S	Diameter	35,55	± 0,01
T	Radius	0,80	± 0,03
*V**	Groove width	0,80	± 0,05
	Groove depth	0,8	± 0,1
	Groove lengths	7,5 min. 12,5 min.	
*W**	Groove width	0,80	± 0,05
	Groove depth	0,8	± 0,1
	Groove lengths	7,5 min. 9,5 min.	
X	Diameter	9,51	± 0,01
	Length of the circular part of the disc shaft	20,0	± 0,5
	Length of the square part of the disc shaft	35,0	± 0,5

* Grooves on top and bottom surfaces should be displaced 5°.