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INTERNATIONAL STANDARD ISO 3387

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Rubbers — Determination of crystallization effects by hardness measurements

Caoutchoucs — Détermination des effets de la cristallisation au moyen de mesurages de dureté

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Rubbers — Determination of crystallization effects by hardness measurements

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a test based on hardness measurements for determining the progressive stiffening of rubber with time, caused by crystallization. It is limited to materials having an initial hardness at test temperature of 10 to 85 IRHD.

The method is applicable to raw, unvulcanized (compounded), and vulcanized rubber. It is mainly of interest for rubber with a marked crystallization tendency at temperatures experienced in cold climates, such as, for instance, chloroprene and natural rubber.

The method is not applicable to fast-crystallizing materials which crystallize to a considerable degree within the time-span of 15 min used for conditioning at test temperature.

2 REFERENCES

ISO 48, *Vulcanized rubbers — Determination of hardness (hardness between 30 and 85 IRHD).*

ISO 471, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO 1400, *Vulcanized rubbers of high hardness (85 to 100 IRHD) — Determination of hardness.*

ISO 1818, *Vulcanized rubbers of low hardness (10 to 35 IRHD) — Determination of hardness.*

ISO 1826, *Rubbers — Time-lapse between vulcanization and testing.*

ISO 3383, *Rubber — General directions for achieving elevated or sub-normal temperatures for tests.*

ISO 4661, *Rubber — Preparation of test pieces.*

3 PRINCIPLE

Measurement, in a test piece stored at the desired temperature, of either

a) the increase in hardness after a specified storage time, or

b) the time required for a specified increase in hardness to occur.

NOTES

1 Tests may be carried out on test pieces of different thicknesses. These do not necessarily give the same values of hardness readings. Tests intended to be comparable should be made on test pieces of the same thickness.

2 The different methods of calculating data to be reported may give somewhat different values. Comparisons of values obtained with different methods should be avoided.

4 APPARATUS

4.1 Cold chamber, in accordance with ISO 3383, capable of being maintained within ± 1 °C of the specified temperature and using a gaseous heat-transfer medium.

As all final handling and measurements are to be made within the cold chamber, it shall be possible to perform these operations while the test piece temperature remains within the permissible variations. This may be done by providing suitable equipment which permits manipulation of materials within the chamber from the outside (for example by means of handholes and gloves through the door or wall of the cabinet).

4.2 Hardness gauges, in accordance with ISO 48 (normal apparatus), ISO 1400 and ISO 1818. Lubricants, if used, shall be of a type not causing friction in the instrument at test temperature.

4.3 Tweezers or tongs, for handling of the test pieces.

4.4 Gloves, for handling of the test equipment.

4.5 Heated press, for the preparation of raw and unvulcanized (compounded) test pieces.

5 TEST PIECES

5.1 Dimensions

The test piece shall have its upper and lower surfaces flat, smooth and parallel to one another. The standard

6.4 Hardness increase due to crystallization

Repeat hardness measurements, as specified in 6.1, after the specified times of storage at test temperature.

NOTE — After all measurements have been completed, it is advisable to dry all apparatus by warming it with circulating air to approximately 40 °C.

7 TEMPERATURE AND DURATION OF TEST

7.1 Temperature

The test shall be carried out at one of the following temperatures (see ISO 471) :

23 ± 2 °C*

27 ± 2 °C*

+ 10 ± 1 °C

0 ± 1 °C

− 10 ± 1 °C

− 25 ± 1 °C

− 40 ± 1 °C

− 55 ± 1 °C

− 70 ± 1 °C

If not specified for special reasons, the test shall be carried out at the temperature which is closest to the one where the crystallization rate is at its maximum, whenever this is known.

NOTE — Generally crystallization rates are known to have their maxima at the following approximate temperatures.

Rubber polymer	Temperature of maximum crystallization rate, °C
Chloroprene rubber	− 10
Polyurethane rubber	− 10
Natural rubber (1,4-*cis*-polyisoprene)	− 25
Dimethyl silicone rubber	− 55
1,4-*cis*-polybutadiene	− 55

7.2 Duration

Hardness measurements are generally taken after $24^{\ 0}_{-0,5}$ h and $168^{\ 0}_{-2}$ h of storage at the test temperature. Intermediate times of reading that allow plotting hardness against time shall be used (48 and 96 h are suggested). Longer times of storage may be used if hardness is still increasing at 168 h.

If the hardness increase after $24^{\ 0}_{-0,5}$ h is more than 10 IRHD above the reading of initial hardness, the test shall be repeated using shorter times of storage (1, 2, 4 and 8 h are suggested).

8 EXPRESSION OF RESULTS

8.1 For specification purposes, the hardness increase between the initial hardness reading and the reading taken after $168^{\ 0}_{-2}$ h storage shall be calculated and stated in the test report (see the figure, graph A). If this hardness increase is greater than 10 IRHD, the readings at different times shall be plotted against time (time on logarithmic scale) and a smooth curve fitted to the points. From the curve, the time corresponding to a hardness increase of 10 IRHD shall be obtained by interpolation (see the figure, graph B).

The same procedure is applied, using the shorter time scale, when the hardness increase after $24^{\ 0}_{-0,5}$ h exceeds 10 IRHD.

The increase in hardness after a specified time or the time for a specified increase in hardness may also be used for reporting of data to comply with requirements in certain specifications (see the figure, graph C).

8.2 For scientific purposes, the time for half the hardness increase to occur between initial and final hardness may be given (see the figure, graph D), using the smooth curve of hardness versus time. This assumes that hardness measurements are extended in time to secure the level of final hardness.

9 TEST REPORT

The test report shall include the following information :

a) Sample details :

1) a full description of the sample and its origin;

2) compound preparation details, and cure time and temperature, where appropriate;

3) method of preparation of test pieces from sample;

4) thickness of test piece and whether made up of one or two pieces.

b) Test method and test details :

1) number of this International Standard;

2) number of the International Standard in accordance with which the hardness measurements were carried out;

* Standard laboratory temperatures

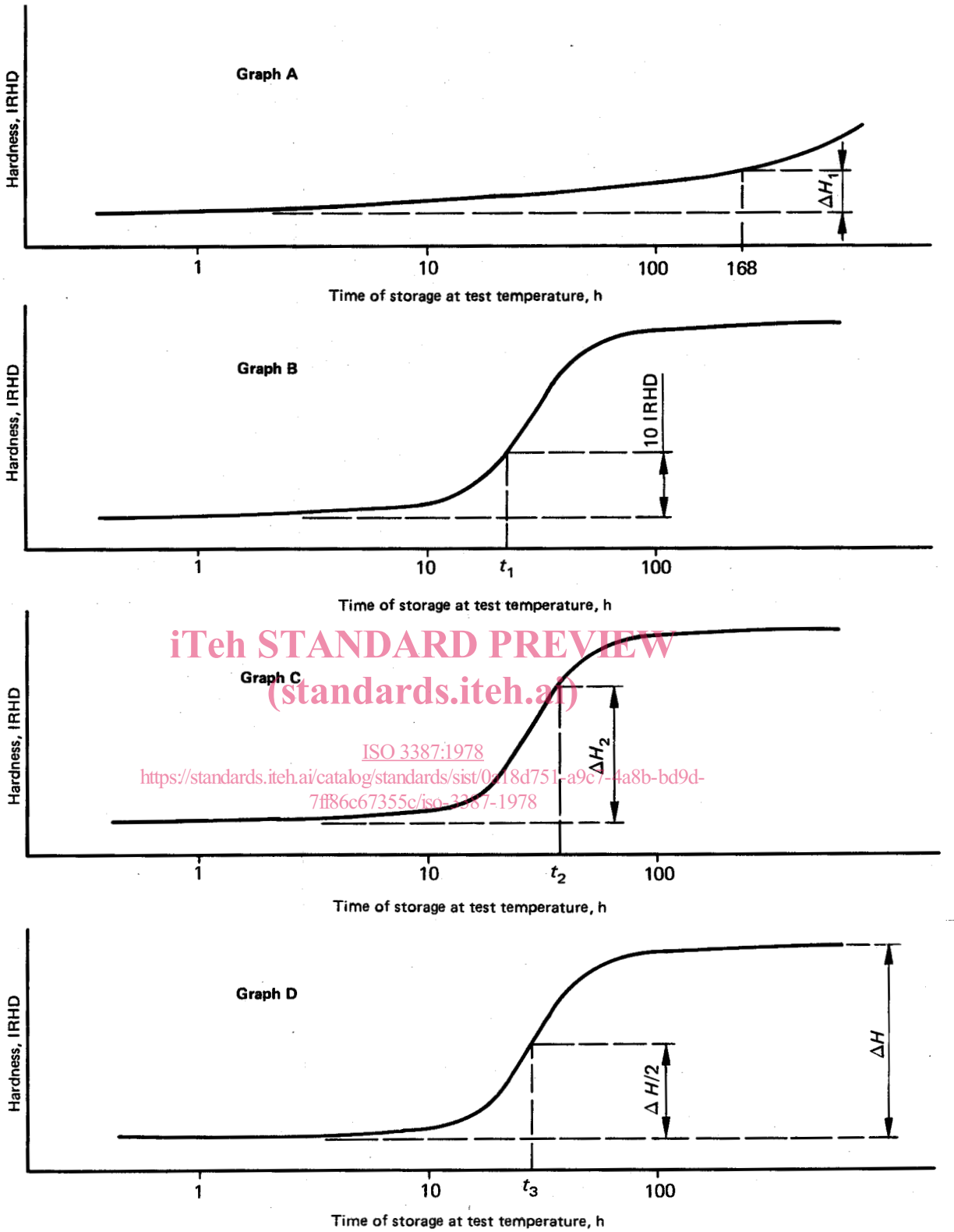

FIGURE – **Different ways of reporting data from the smooth curve obtained by plotting the data of hardness readings against time of storage at test temperature**

Graph A : Hardness increase after 168 $^{0}_{-2}$ h is less than 10 IRHD. Report the actual hardness increase ΔH_1 after 168 $^{0}_{-2}$ h.

Graph B : Report time t_1 for 10 IRHD increase to occur.

Graph C : Report time t_2 for a specified hardness increase ΔH_2 to occur, or report the hardness increase ΔH_2 after a specified time t_2.

Graph D : Report time t_3 for half the hardness increase between initial and final hardness to occur.