INTERNATIONAL STANDARD

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Rubber, vulcanized or thermoplastic — Determination of adhesion to a rigid substrate — 90° peel method

Caoutchouc vulcanisé ou thermoplastique — Détermination de l'adhérence à un substrat rigide — Méthode par pelage à angle droit

INTERNATIONAL ISO

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 813 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Physical and degradation tests*.

This third edition cancels and replaces the second edition (ISO 813:1986), which has been technically revised.

Annexes A and B of this International Standard are for information only.

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Rubber, vulcanized or thermoplastic — Determination of adhesion to a rigid substrate — 90° peel method

1 Scope

This International Standard specifies a method for the determination of the adhesive strength of a vulcanized or thermoplastic rubber bonded to a rigid substrate, using a test piece comprising a strip of rubber bonded to a single plate of rigid material

The method is applicable primarily to test pieces prepared in the laboratory under standard conditions, such as may be used to provide data for the choice of rubber compounds or adhesive systems, the development of such materials and the control of manufacturing processes.

NOTE — This method is not suitable for high-hardness rubbers.

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 investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 471:1995, *Rubber — Temperatures, humidities and times for conditioning and testing.*

ISO 3383:1985, *Rubber — General directions for achieving elevated or subnormal temperatures for test purposes.*

ISO 4648:1991, *Rubber vulcanized or thermoplastic — Determination of dimensions of test pieces and products for test purposes.*

ISO 5893:1993, *Rubber and plastics test equipment — Tensile, flexural and compression types (constant rate of traverse) — Description.*

3 Principle

The force required to cause separation of a strip of rubber bonded to a rigid substrate is measured, the angle of separation being 90° and the width and thickness of the rubber and the rigid material being fixed within specified limits.

4 Apparatus

4.1 Tensile-testing machine, complying with the requirements of ISO 5893, capable of measuring force with an accuracy corresponding to grade B as defined in ISO 5893, and with a rate of traverse of the moving grip of 50 mm/min ± 5 mm/min.

NOTE — Inertia (pendulum) type dynamometers tend to give results which differ because of frictional and inertial effects. An inertialess (for example electronic or optical-transducer type) dynamometer gives result which are free from these effects and is therefore to be preferred.

4.2 Fixture, for holding the test piece to the moving grip of the testing machine (4.1) so that the direction of pull to cause separation is at all times during the test as nearly perpendicular as possible to the plane of the bond between the rubber and the rigid substrate, i.e. making an angle of 90° with the face of the fixture.

The fixture shown in figure 1 complies with this requirement.

Dimensions in millimetres

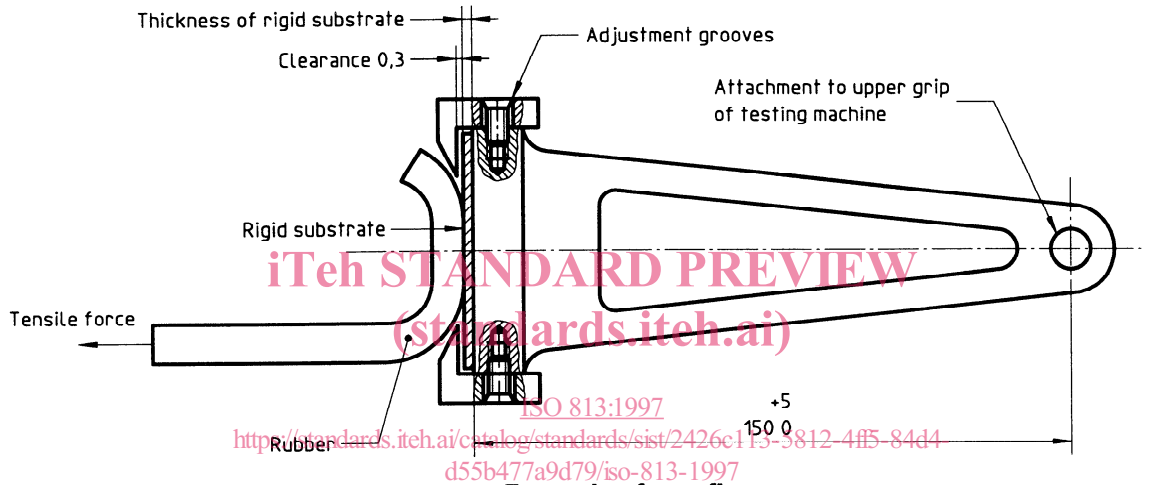

Figure 1 — Example of test fixture

4.3 Grip, so designed that it does not allow the rubber to slip or cause it to rupture.

4.4 Temperature-controlled chamber, in accordance with ISO 3383, for carrying out tests at temperatures between – 70 °C and + 250 °C with tolerances as defined in ISO 471.

5 Test pieces

5.1 Dimensions

The standard test piece shall consist of a strip of rubber 6 mm ± 0,1 mm thick in the test area, 25 mm ± 0,1 mm wide and 125 mm long, bonded to a square area 25 mm long and 25 mm ± 0,1 mm wide on the surface of a strip of rigid substrate, the determination of the dimensions of the test piece being in accordance with ISO 4648.

The substrate shall be sufficiently thick to prevent deformation during the test: a thickness of 1,5 mm ± 0,1 mm is recommended for metal, but greater thicknesses may be necessary when a plastic or other material is used. The width shall be 25 mm ± 0,1 mm and the length 60 mm ± 1 mm.

Each test piece shall be prepared so that the bonded area 25 mm long and 25 mm ± 0,1 mm wide is approximately in the middle of the substrate shown in figure 2.

Dimensions in millimetres

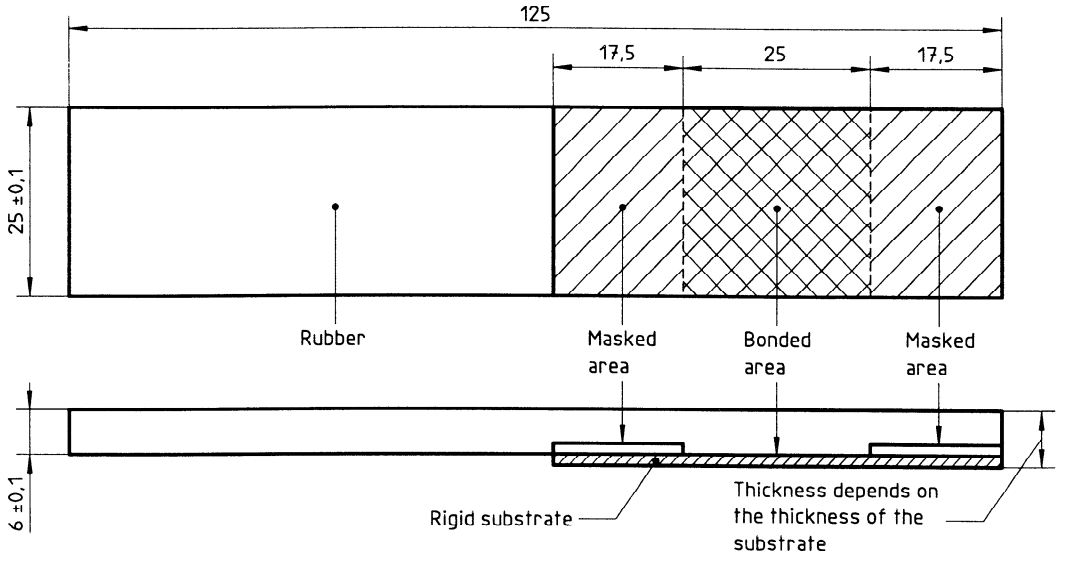

Figure 2 — Standard test piece

5.2 Preparation

5.2.1 Two types of mould may be used for the preparation of test pieces: moulds for several test pieces or moulds for single test pieces.

5.2.1.1 When test pieces are to be made using one type of rubber and one type of adhesive system, a mould for several test pieces may be used. The inside mould dimension parallel to the longitudinal axis of the strip of substrate shall be 125 mm. The dimension corresponding to the transverse axis of the strip of substrate may be altered, depending on the number of test pieces to be prepared at a time. The dimension perpendicular to the longitudinal and transverse axes of the substrate may be altered, for instance using metal plates of various thicknesses, depending on the thickness of the substrate, in order to leave a depth of 6 mm ± 0,05 mm free for the rubber.

5.2.1.2 When only one test piece is to be made from a given rubber, a mould as specified in 5.2.1.1 shall be used, except that the dimension corresponding to the transverse axis shall be restricted to the width of the test piece.

5.2.2 When testing unvulcanized rubber or thermoplastic rubber, cut slabs of sufficient thickness to the required size for the mould (length 125 mm, width corresponding to the number of test pieces to be prepared) so as to ensure maximum pressure between the rubber and the substrate during the test piece preparation process.

5.2.3 During test piece preparation, take great care to keep the surfaces to be bonded clean and free from dust, moisture and other foreign matter.

5.2.3.1 Prepare, in the way required by the adhesive system under investigation, the surface to be bonded of strips of substrate of the dimensions specified in 5.1. Mask both ends with pressure-sensitive tape so that only the area specified in 5.1 is available for bonding.

5.2.3.2 Clean the rubber surface to be bonded, using solvent or another technique as required by the adhesive system under investigation.

5.2.3.3 Apply the adhesive and assemble the strips of substrate and rubber. Place the assembly in the mould, with the substrate at the bottom. When more than one test piece is being prepared at a time, place the strips of substrate approximately 3 mm apart to permit subsequent separation of the test pieces.

5.2.4 In the case of a rubber bonded with a heat-curing adhesive, carry out the vulcanization and/or bonding by heating the mould under pressure in a suitable press for the time and at the temperature required by the system being investigated.

Take great care when removing the test pieces from the mould to avoid subjecting the bonded surfaces to undue stress before the test pieces have cooled.

NOTE — When the substrate used is a thermoplastic material, the strip may in some cases need to be raised to a temperature above its softening point in order to ensure adhesion. When the substrate is a thermosetting polymer, the starting material may in some cases be a low-viscosity prepolymer at the test piece preparation temperature, whose polymerization is completed during the process. The mould and the moulding pressure will have to be adapted to such cases.

5.2.5 When more than one test piece is prepared at a time, separate the test pieces from each other for testing. Do this by cutting with scissors, a knife or another suitable tool. The edges of each test piece may then be buffed on a belt sander to bring the edge of the rubber flush with the edge of the substrate. Take care not to overheat the test piece and not to reduce the width of the test piece beyond the tolerance allowed.

5.3 Number

Four test pieces shall be tested.

5.4 Conditioning before testing

The time interval between the preparation of the test pieces and testing shall be in accordance with the requirements of ISO 471. It shall be at least 16 h after moulding.

Condition the test pieces in accordance with the requirements of ISO 471 for at least 16 h at a standard temperature (23 °C ± 2 °C or 27 °C ± 2 °C) immediately before the test.

When a test at other than standard temperature is required, carry out additional conditioning at this temperature for long enough to bring the material to the required temperature. ISO 3383 gives guidances on the conditioning time necessary to reach temperature equilibrium.

6 Test temperature and humidity

Unless otherwise specified, carry out testing at standard laboratory temperature and humidity, as specified in ISO 471.

When other test temperatures are required, take them as far as possible, from the list of preferred temperatures given in ISO 471.

Use the same temperature throughout any one test, as well as any series of tests intended to be comparable.

7 Procedure

7.1 Place the test piece symmetrically in the fixture (4.2), with the end to be pulled apart facing towards the operator. Before applying the load, strip the rubber from the substrate for a distance of approximately 1,5 mm using a sharp knife. Place the free end of the rubber strip in the grip (4.3). In the case of tests at other than standard temperature, keep the test piece in the temperature-controlled chamber (4.4) for a sufficient length of time after closure of the chamber for the test piece to reach the required temperature. Then move the grip of the tensile-testing machine at 50 mm/min ± 5 mm/min until separation is complete. Record the maximum force required to cause separation.

NOTE — Other speeds of separation may be used to examine the viscoelastic behaviour of the bond (see annex A). In such cases, the use of logarithmic increments in the speed of separation is recommended.

7.2 An autographic recording of the force of adhesion over the full length of the test piece may also be made.

7.3 During the test, cut the rubber back to the substrate with the knife whenever the rubber tends to tear.

8 Expression of results

8.1 Adhesion strength

Calculate the adhesion strength, in newtons per millimetre, by dividing the maximum force, in newtons, recorded in 7.1 by the width, in millimetres, of the test piece.

8.2 Adhesion failure symbols

Assess the type of failure, using the following symbols:

R when the failure is in the rubber.

RC when the failure is at the interface between the rubber and the layer of adhesive.

CP when the failure is at the interface between the layer of adhesive and the primer (if used).

PS when the failure is at the interface between the primer and the substrate.

CS when the failure is at the interface between the adhesive and the substrate (when no primer is used).

D when the failure is at the interface between the rubber and the substrate in the case of direct adhesion, i.e. no adhesive used.

S when the failure is in the substrate.

9 Precision

No precision data are currently available for this method.

10 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for identification of the sample tested;

c) the test results, expressed in accordance with clause 8;

d) a description of the type(s) of failure, expressed in accordance with 8.2, indicating the percentage of each type of failure;

e) a description of the test piece, its constitutive elements and the method used to bond them together;

f) the date of preparation of the test piece and, if applicable, of its constitutive elements;

g) the conditions under which the test pieces were assembled before testing;

h) the test temperature;

i) the test speed;

j) the date of the test;

k) any unusual features noted during the determination.

Annex A
(informative)

Analysis of the viscoelastic behaviour of the bond

If, for a given system, tests are carried out at several speeds and several temperatures in order to achieve a better understanding of the viscoelastic behaviour of the bond, covering the transition from cohesive failure to adhesive failure of the test piece, the adhesion strength (8.1) may be plotted on a graph as a function of speed for each temperature. A semi-logarithmic graph is used, with the adhesion strength along the y-axis on a linear scale and speed along the x-axis on a logarithmic scale. All the isothermal curves may be drawn on the same graph.

NOTE — These data are generally subjected to further processing based on the assumption that there is a degree of equivalence between the time dependence and the temperature dependence of the relaxation mechanisms of rubbers (the Williams, Landel and Ferry transform). Thus it is possible to draw a single master curve representing, for a standard temperature, the adhesion behaviour over a range of speeds much wider than could be obtained experimentally, and showing the transition from one mode of failure to the other. The data-processing technique used is not specific to adhesion behaviour and is therefore not described here. For further information, the reader is referred to the publications given in annex B.

Annex B
(informative)

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