BRITISH STANDARD

BS 903 :
Part A37 : 1997
ISO 6505 : 1997

Physical testing of rubber

Part A37. Determination of tendency to adhere to and to corrode metals

ICS 83.060

Committees responsible for this British Standard

The preparation of this British Standard was entrusted to Technical Committee PRI/22, Physical testing of rubber, upon which the following bodies were represented:

British Rubber Manufacturers' Association Ltd.
GAMBICA (BEAMA Ltd.)
Ministry of Defence
RAPRA Technology Ltd.
SATRA Footwear Technology Centre
Tun Abdul Razak Research Centre
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Contents

National foreword

This Part of BS 903 reproduces verbatim ISO 6505 : 1997 and implements it as the UK national standard. It supersedes BS 903 : Part A37 : 1987 which is withdrawn.

This British Standard is published under the direction of the Sector Board for Materials and Chemicals whose Technical Committee PRI/22 has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on interpretation, or proposals for change, and keep UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

An informative national annex is included for vulcanizates which are not in direct contact with metal. This national annex is reproduced from method B of BS 903 : Part A37 : 1987 and is included because zinc is generally accepted as the metal most susceptible to corrosion of the metals in common use and can also be obtained in an extremely pure form.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 8, an inside back cover and a back cover.

Introduction

In assemblies which include both metallic and rubber components, it is essential to avoid unintentional adhesion of rubber to metal, and corrosion of the metal by the rubber. Adhesion occurs only where there is direct contact between the metal and the rubber, but corrosion may also arise, within a closed system, on metal components remote from the rubber, such corrosion being due to volatile materials emanating from the rubber.

Since some metals corrode more readily than others, it is not possible to specify an optimum test condition for assessing the resistance to corrosion of all metals or alloys. Furthermore, the ranking of a metal's susceptibility to corrosion will depend upon the environment in which it is exposed to the rubber, e.g. in the presence of high humidity the effects on steel in particular can be severe.

Rubber, vulcanized or thermoplastic — Determination of tendency to adhere to and to corrode metals

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies a method for the determination of the tendency of vulcanized or thermoplastic rubbers to adhere to metals and to corrode metals when exposed to a specified test environment.

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 are 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 209-1:1989, *Wrought aluminium and aluminium alloys - Chemical composition and forms of products - Part 1: Chemical composition*

ISO 426-1:1983, *Wrought copper-zinc alloys - Chemical composition and forms of wrought products - Part 1: Non-leaded and special copper-zinc alloys*

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

ISO 630:1995, *Structural steels - Plates, wide flats, bars, sections and profiles*

ISO 1337:1980, *Wrought coppers (having minimum copper contents of 99,85 %) - Chemical composition and forms of wrought products*

ISO 3310-1:1990, *Test sieves - Technical requirements and testing - Part 1: Test sieves of metal wire cloth*

ISO 3383:1985, *Rubber - General directions for achieving elevated or sub-normal temperatures for test purposes*

ISO 4661-1:1993, *Rubber, vulcanized or thermoplastic - Preparation of samples and test pieces - Part 1: Physical tests*

3 Principle

Rubber test pieces are held between metal test strips under specified conditions for a specified period. Subsequent visual examination of the metal surface provides an subjective indication of the degree of adhesion to the metal by the rubber and corrosion of the metal.

4 Materials

4.1 Acetone (for cleaning of metal), of recognized analytical quality.

4.2 Other suitable solvents (for cleaning of rubber), of recognized analytical quality and which do not have any deleterious effects on the rubber under test.

4.3 Pumice powder, passing a test sieve of nominal aperture size 53 µm, complying with the requirements of ISO 3310-1.

4.4 Distilled water or water of equivalent purity.

5 Apparatus

Usual laboratory equipment, and

5.1 Support jig, to align the metal test strips and rubber test pieces, capable of supporting the clamping force, and with a facility for setting clamps to maintain the clamping force on the assembled test piece "sandwich" throughout the test period (see the figure).

5.2 Test chamber, complying with the requirements specified in ISO 3383, with facilities for controlling the temperature within the tolerance given in ISO 471.

For tests other than those in a "dry" atmosphere a suitable means for the control of humidity shall be provided.

Note 1 - The use of saturated salt solutions has been found suitable.

5.3 Polyethylene gloves or other suitable equipment to prevent direct contact with the test surfaces.

5.4 Magnifying glass, of magnification 3x to 5x.

6 Test metals

The test metals to be used shall be those specified in the relevant material specification. If the metals are not specified, they shall be selected from the standard test metals specified in the table.

Table

Standard Test Metal	Description
Aluminium	ISO 209-1: Grade Al Cu4 Si Mg, condition TF
Brass	ISO 426-1: Grade Cu Zn 37, HA or HB temper
Copper	ISO 1337: Grade Cu-ETP, HA or HB temper
Carbon steel	ISO 630: Grade Fe 360A

The test metals shall be in the form of strips 25 mm wide and at least 100 mm long. The thickness of any strip shall be sufficient to withstand the clamping force without bending. If only thin foil is available, it shall be supported by a rigid backing material previously shown to be non-corrosive to the test metals.

7 Rubber test pieces

7.1 Preparation

The rubber test pieces shall be in the form of squares of side 20 mm ±0,5 mm, preferably of thickness 2,0 mm ±0,2 mm. They shall be cut or punched from sheet or from the product under evaluation in accordance with ISO 4661-1.

7.2 Number

At least two rubber test pieces shall be used for each test.

7.3 Time interval between forming the material and testing

The time interval between forming the material and testing shall be in accordance with ISO 471.

7.4 Storage

Samples and test pieces shall be protected from light as completely as possible during the interval between forming and testing.

8 Test conditions

8.1 Temperature

The test temperature shall be selected from the list in ISO 471.

8.2 Test period

The duration of test shall be selected from the following:

24 h +0/-2 h; 72 h +0/-2 h; 168 h ±2 h; or multiples of 7 days.

8.3 Humidity

A dry atmosphere having a relative humidity of less than 10 % shall normally be used. Where tests are required by a particular application in other environments, e.g. in atmospheres having high humidity, only like metals shall be used in the construction of the test piece "sandwich" in order to avoid electrolytic effects.

Note 2 - This test is commonly carried out at low humidities to ensure that corrosion resulting from causes other than that of the rubber is minimized.

9 Procedure

9.1 Precaution

In all operations, it is essential that the rubber test pieces and the metal test strips are handled only by means of the polyethylene gloves or other protective equipment (5.3). This precaution is essential in order to minimize surface contamination of the test piece and metal strips.

9.2 Preparation of rubber test pieces for test

Clean all the surfaces of the rubber test pieces with cotton wool pads moistened with a suitable solvent (4.2) to remove surface contamination (by mould release agents, for example). The solvent to be used will depend on the rubber under test; it shall not have any deleterious effects on the rubber (e.g. acetone should not be used for nitrile rubber; isopropyl alcohol is preferred for this material).

Allow the test piece to dry in air. When dry, store the test pieces, unless otherwise specified, in a clean desiccator over silica gel at standard laboratory temperature (see ISO 471) for at least 24 h immediately prior to testing.

Note 3 - Since cleaning of the test pieces may also remove from the rubber surface such materials as waxes, antiozonants, etc., which would normally be expected to affect the adhesion and corrosion properties of the rubber, sufficient time should be allowed in the desiccator for the re-formation of the "original" surface before testing.

9.3 Number of metal test strips

For each test, use two appropriate metal test strips as specified in the material specification or selected from the metals specified in clause 6. The two strips may be of the same metal or dissimilar metals.

9.4 Preparation of surfaces of metal test strips

Thoroughly scour the test surfaces of the metal test strips using a pumice powder (4.3) water slurry applied with a cotton wool pad until a matt surface is obtained. Thoroughly rinse the metal strips with water (4.4) and then with acetone (4.1) and finally dry in air. If the prepared metal test strips are not to be used immediately after cleaning, they should be stored in a clean desiccator over silica gel for not more than 24 h before testing.

9.5 Determination

Place two rubber test pieces, prepared as specified in 9.2, between the surfaces of two metal strips, prepared as specified in 9.4, so that the rubber test pieces are approximately 40 mm apart and approximately equidistant from the ends of the metal strips (see the figure). Align the metal/rubber/metal sandwich so formed in the support jig and apply a mass of 10 kg ±0,1 kg (equivalent to 122,5 kPa on the rubber) to the test piece sandwich. Tighten the two screw clamps, one at each end of the sandwich, with just sufficient force to maintain the clamping force when the 10 kg mass is removed. Remove the 10 kg mass from the jig, place the sandwich in the test chamber (5.2) and maintain at the test temperature for the test period (see clause 8).

At the end of the test period, remove the sandwich from the test environment, allow to cool, if appropriate, to standard laboratory temperature and maintain at this temperature for at least 1 h, release the screw clamps and carefully separate the metal strips from the rubber test pieces. For tests other than those in the dry atmosphere, dry the metal surfaces with cotton wool pads. Keep the metal in an atmosphere at standard laboratory temperature with a relative humidity of 50 % ±5 % for 16 h to 24 h. At the end of this period, examine the surface of the metal previously in contact with the rubber for signs of adhesion and corrosion. Use the magnifying glass (5.4) in examining for corrosion.

10 Expression of results

10.1 Degree of adhesion

Evaluate the degree of adhesion according to the following criteria:

a. Complete separation from both metal surfaces. No indication of adhesion.

b. Considerable force necessary to separate metal surfaces. Particles of rubber remain adhering to one or both metal surfaces.

10.2 Degree of corrosion

Evaluate the degree of corrosion according to the following criteria:

a. No surface stain or corrosion.

b. Surfaces stain or discolouration present, but no corrosion as defined by pitting or erosion of one or both metal surfaces.

c. Corrosion as evidenced by pitting and erosion on one or both metal surfaces.

11 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) sample details:

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

2) compound details and curing conditions, where appropriate;

3) method of preparation of test pieces from sample;

4) type of metal test strips used;

c) test details:

1) standard laboratory temperature and humidity used;

2) duration, temperature and test environment;

3) any deviations from the standard procedure;

d) test result:

1) details of any adhesion and/or corrosion;

e) the date of test.

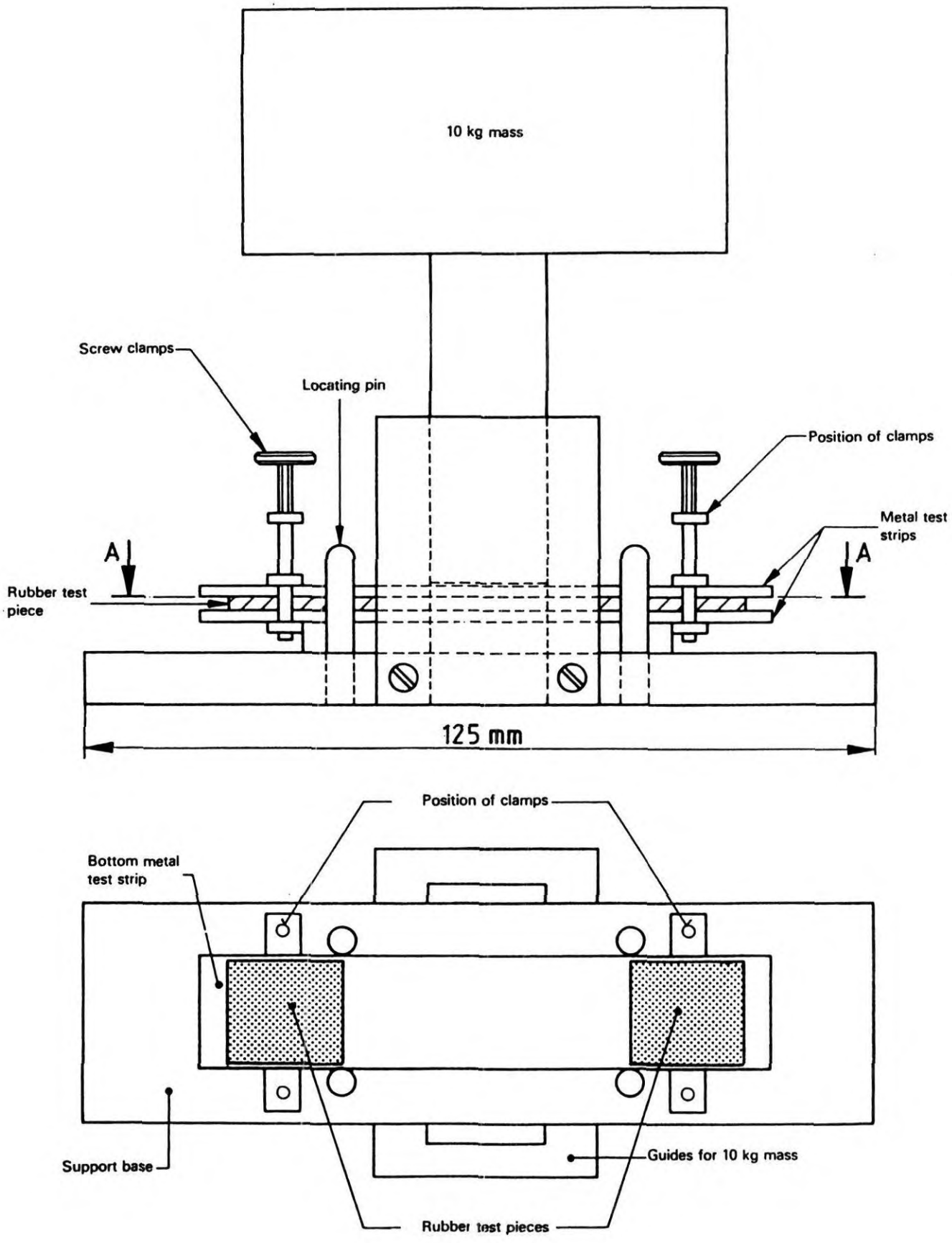


Figure - Typical support jig

National annex NA (informative) Method for vulcanizates not in direct contact with metal

NA.1 Principle

Zinc metal is exposed to volatile substances emanating from vulcanized rubber test pieces in a water saturated atmosphere at an elevated temperature of 50 °C. Corrosion of the metal is measured quantitatively by the subsequent mass loss of the metal test piece after removing, by chemical means, the corrosive products formed during the test.

NOTE. Because corrosion of metals other than the zinc specified does not necessarily follow a fixed pattern, and since the method is extremely sensitive to contamination, considerable expertise is required in the performance of the test and interpretation of the results. Consequently this method is primarily intended to be used in the acquisition of data for design and development of compounds or as a material type test. For this reason zinc is chosen as the test metal since it can be obtained in an extremely pure form, is in common use and is susceptible to corrosion.

NA.2 Reagents

NA.2.1 *General.* All reagents shall be of analytical reagent quality.

NA.2.2 *Distilled water*, conforming to grade 3 of BS EN ISO 3696.

NA.2.3 *Chromium trioxide* (CrO_3), 100 g/l solution in distilled water.

NA.2.4 *Acetone.*

NA.2.5 *Hydrochloric acid*, 5 mol/l solution.

NA.2.6 *Pumice powder*, passing through a test sieve having a mesh size of (63 ± 10) μm that conforms to BS 410.

NA.2.7 *Silica gel.*

NA.3 Apparatus

NA.3.1 *Glass test vessel*, as illustrated in figure NA.1, consisting of a wide mouth glass bottle of approximately (425 ± 25) ml capacity fitted with a ground glass stopper joint size 54/44, with glass hooks for supporting the test pieces. The stopper is also fitted with a breathing tube consisting of a length of glass capillary tubing with an internal diameter of 0.75 mm.

NA.3.2 *Test chamber*, capable of being maintained at (50 ± 1) °C, and conforming to BS 903 : Part A32.

NA.3.3 *Polyethylene gloves* or *polypropylene forceps.*

NA.3.4 *Analytical balance*, accurate to 0.1 mg.

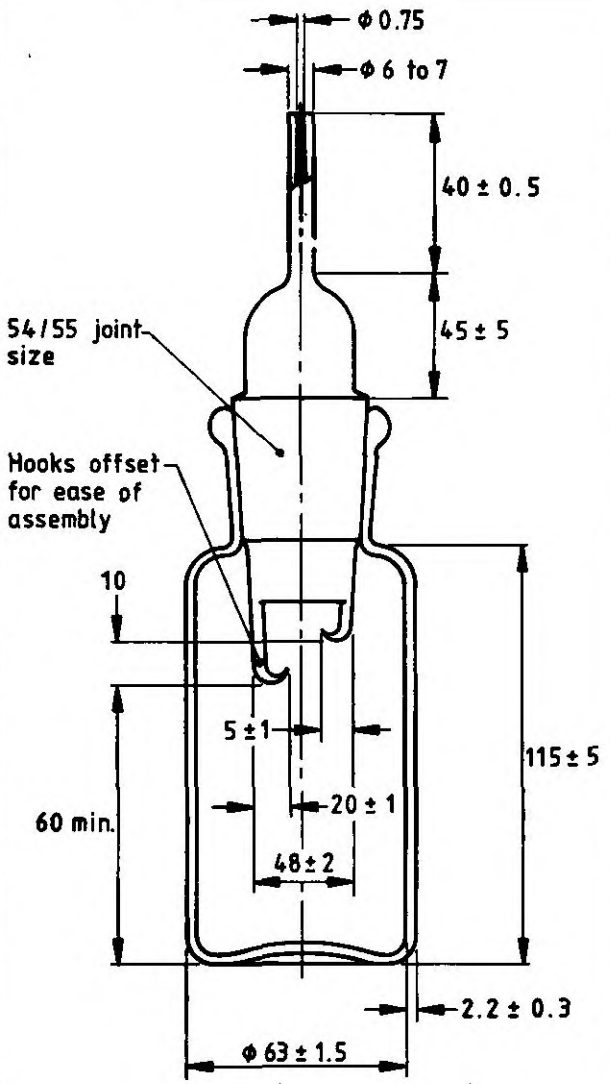
NA.3.5 *Cotton wool pads.*

NA.3.6 *Desiccator.*

NA.3.7 *Fine file.*

NA.4 Zinc test pieces

The zinc test pieces shall be of size 50 mm × 25 mm, cut from high purity (not less than 99.99 % Zn) rolled zinc sheet of approximately 0.9 mm thickness.



All dimensions are in millimetres.

Figure NA.1 Glass test vessel

NA.5 Rubber test pieces

The test pieces shall be in the form of rectangles of (50 ± 1) mm length and (25 ± 1) mm width cut or punched from vulcanized sheet of uniform thickness (2.0 ± 0.2) mm. A hole shall be punched or cut in each test piece to enable it to be suspended from one of the glass hooks of the test vessel (see **NA.3.1**).

NA.6 Time lapse between vulcanization and testing

For all test purposes the minimum time between vulcanization and testing shall be 16 h. For non-product tests, the maximum time between vulcanization and testing shall be 28 days.

NOTE. For evaluations intended to be comparable, the tests as far as possible should be carried out after the same time interval. For product tests, whenever possible, the time between vulcanization and testing should not exceed 90 days.

In other cases tests shall be made within 60 days of the date of receipt of the product by the customer.

Samples and test pieces shall be protected from light as completely as possible during the interval between vulcanization and testing.

NA.7 Preparation of samples

NA.7.1 ***Handling of test pieces***. In all the operations described in **NA.7.2** to **NA.8**, the rubber test pieces and the zinc test pieces shall be handled only by means of the polyethylene gloves or polypropylene forceps (**NA.3.3**).

NOTE. This precaution is necessary in order to minimize surface contamination.

NA.7.2 ***Number of test pieces***. For each test two rubber test pieces and two zinc test pieces are required together with at least two further zinc test pieces as controls.

NA.7.3 ***Preparation of rubber test pieces***. Clean the entire surface of the test pieces, with cotton wool pads (**NA.3.5**) moistened with a suitable solvent to remove any surface contamination, e.g. by mould release agents. The solvent used depends on the rubber under test, and shall not have any deleterious effects on the vulcanizate, e.g. acetone shall not be used for nitrile rubbers. Allow the test pieces to dry in air. When dry, store the test pieces in a clean desiccator (**NA.3.6**) over silica gel (**NA.2.7**) for at least 24 h at (23 ± 2) °C immediately prior to testing.

NOTE 1. Attention is drawn to the information in BS 6716 and BS 903 : Part A16.

NOTE 2. Since the cleaning of the test pieces may also remove such material as waxes, anti-ozonants, etc. from the rubber surface, which would normally be expected to affect the corrosive properties of the vulcanizate, sufficient time in the desiccator should be allowed for the re-formation of the 'original' surface before testing.

NA.7.4 ***Preparation of zinc test pieces***. Drill a hole in the zinc test piece to enable it to be suspended vertically from one of the glass hooks of the glass test vessel (**NA.3.1**).

Remove all burrs and rough edges from the zinc test piece with a fine file (**NA.3.7**). Scour thoroughly both faces and the edges of the test piece using pumice powder (**NA.2.6**) and cotton wool pads wetted with distilled water (**NA.2.2**) until a matt surface is obtained. Rinse with acetone (**NA.2.4**), dry in air and weigh to an accuracy of 0.01 g. Immerse the test piece in the 5 mol/l hydrochloric acid solution (**NA.2.5**) at room temperature for 15 s to 30 s. Rinse well in running tap water followed by distilled water, swabbing the surface with cotton wool. Rinse with acetone, dry in air and reweigh. If the amount of zinc removed is less than 0.5 g repeat the acid treatment. After removal of at least 0.5 g of zinc, again scour both surfaces and the edges of the zinc test piece with pumice powder. Rinse well with water conforming to BS EN ISO 3696, rinse with acetone and allow to dry in air. Store the test piece in a clean desiccator over silica gel for up to 24 h immediately before testing. Before the corrosion test is commenced examine the zinc test pieces. Evidence of corrosion at this stage is indicative of contamination during the cleaning stage and if such evidence is present, repeat the cleaning procedure.

NA.8 Testing procedure

Add 5 ml of distilled water to a thoroughly cleaned glass test vessel, suspend a rubber test piece from one hook on the glass stopper and a prepared zinc test piece, weighed immediately before use to an accuracy of 0.1 mg, from the other hook, taking care that the rubber test piece does not touch the zinc test piece. Place the stopper in the test vessel and maintain the assembly in the test chamber at (50 ± 1) °C for 3 weeks. Carry out two tests on each sample of vulcanized rubber. With each series of tests prepare at least two controls using a zinc test piece suspended over distilled water without a rubber test piece. Ensure that distilled water is present in all test vessels so that a water saturated atmosphere is maintained for the duration of the test. At the end of the 3 week period remove each zinc test piece. A visual assessment of the extent of the corrosion may be made at this stage if required.

Remove any corrosive products by immersing the zinc test piece in a chromium trioxide solution (**NA.2.3**) maintained at 90 °C to 95 °C, until effervescence has ceased or for 3 min, whichever is the longer period. Rinse well with distilled water to remove residues, using a small brush if necessary. Rinse with acetone, dry in air and reweigh the zinc test piece. If the corrosive product has not been completely removed, repeat the chromium trioxide solution immersion.

NOTE. The temperature of 50 °C has been shown to give satisfactory results for most vulcanizates within a reasonable period. Laboratory tests at temperatures of 30 °C or 45 °C may correlate more closely with long term storage trials but times are necessarily longer. At temperatures above 50 °C, reproducible results are difficult to obtain even for the zinc control pieces.

NA.9 Calculation of results

The mass loss per unit area of zinc M (in g/m^2) shall be calculated as follows:

$$M = W_1 \times 400$$

where

W_1 is the average loss in mass of zinc test pieces in the duplicate determinations (in g).

Where the average mass loss of the controls is greater than 4 g/m^2 the series of tests shall be repeated. Values for mass loss of controls greater than 4 g/m^2 are indicative of contamination during the preparation or testing.

NA.10 Test report

The test report shall include the following information:

a) identification of the rubber test pieces;

b) mass loss of zinc in g/m^2;

c) method used;

d) any special features not covered in this method or regarded as optional;

e) date of test.

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