INTERNATIONAL STANDARD 4624

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Paints and varnishes — Pull-off test for adhesion

Peintures et vernis — Essai de traction

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

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 4624 was developed by Technical Committee ISO/TC 35, *Paints and varnishes*, and was circulated to the member bodies in August 1976.

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

Australia	Germany	Poland
Austria	India	Portugal
Belgium	Iran	Romania
Brazil	Israel	South Africa, Rep. of
Bulgaria	Korea, Rep. of	Sweden
Canada	Mexico	Switzerland
Chile	Netherlands	Turkey
Czechoslovakia	New Zealand	United Kingdom
Egypt, Arab Rep. of	Norway	Yugoslavia
France	Peru	

The member body of the following country expressed disapproval of the document on technical grounds :

Italy

Printed in Switzerland

Paints and varnishes — Pull-off test for adhesion

0 INTRODUCTION

This International Standard is one of a series of standards dealing with the sampling and testing of paints, varnishes and related products. It should be read in conjunction with ISO 1512, ISO 1513, ISO 1514 and ISO 2808.

This International Standard specifies a method for assessing the adhesion of a single coating or a multi-coat system of paint, varnish or related product by measuring the minimum tensile stress necessary to detach or to rupture the coating in a direction perpendicular to the substrate.

The test result is influenced not only by the mechanical properties of the system under test, but also by the nature and preparation of the substrate, the method of paint application, the drying conditions of the coating, temperature, humidity and other factors.

The method of test specified below requires to be completed, for any particular application, by the following supplementary information. This information should be derived from the national standard or other document for the product under test or, if appropriate, shall be the subject of agreement between the interested parties.

a) Material and surface preparation of test assembly surface or substrate.

b) Method of application of test coating to the substrate or test cylinder, if appropriate.

c) Duration and conditions of drying of the coating (or conditions of stoving and ageing, if applicable) before testing.

d) Thickness, in micrometres, of the dry coating, including the method of measurement in accordance with ISO 2808, and whether it is a single coating or a multi-coat system.

e) The adhesive (and mixing ratios, if applicable) and curing conditions (see also clause 4).

f) Duration and conditions between assembly and testing.

g) Type of pull-off test assembly used (see 7.3).

h) Type of tensile tester and diameter of test cylinder.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies methods for carrying out a pull-off test on a single coating or a multi-coat system of paint, varnish or related product.

The test may be applied using a wide range of substrates. Different procedures are specified according to whether the substrate is deformable, for example thin metal, plastics and wood, or rigid, for example thick concrete and metal plates. For special purposes, the coating may be applied to a test cylinder and, in this case, a method for determination of the coating thickness shall be agreed between the interested parties.

The test result is the minimum tensile stress necessary to break the weakest interface (adhesive failure) or the weakest component (cohesive failure) of the test assembly. Mixed adhesive/cohesive failures may also occur.

2 REFERENCES

ISO 1512, *Paints and varnishes — Sampling.*

ISO 1513, *Paints and varnishes — Examination and preparation of samples for testing.*

ISO 1514, *Paints and varnishes — Standard panels for testing.*

ISO 2808, *Paints and varnishes — Determination of film thickness.*

3 APPARATUS

3.1 Tensile tester, suitable for carrying out the chosen procedure specified in clause 7. The tensile stress shall be applied in a direction perpendicular to the plane of the coated substrate and shall be increased at a substantially uniform rate, not greater than 1 MPa/s*, such that failure of the test assembly occurs within 90 s. Suitable designs for applying the tensile stress are shown in figures 1 and 2.

3.2 Test cylinders, suitable for use with the tensile tester (3.1), steel faced, of diameter 20 mm (unless otherwise agreed) and of sufficient thickness to ensure freedom from distortion during the test. It is recommended that the length of the test cylinder should be not less than half its diameter. The faces shall be machined perpendicular to the long axis of the cylinder before use.

* 1 MPa/s = 1 $MN/m^2.s$

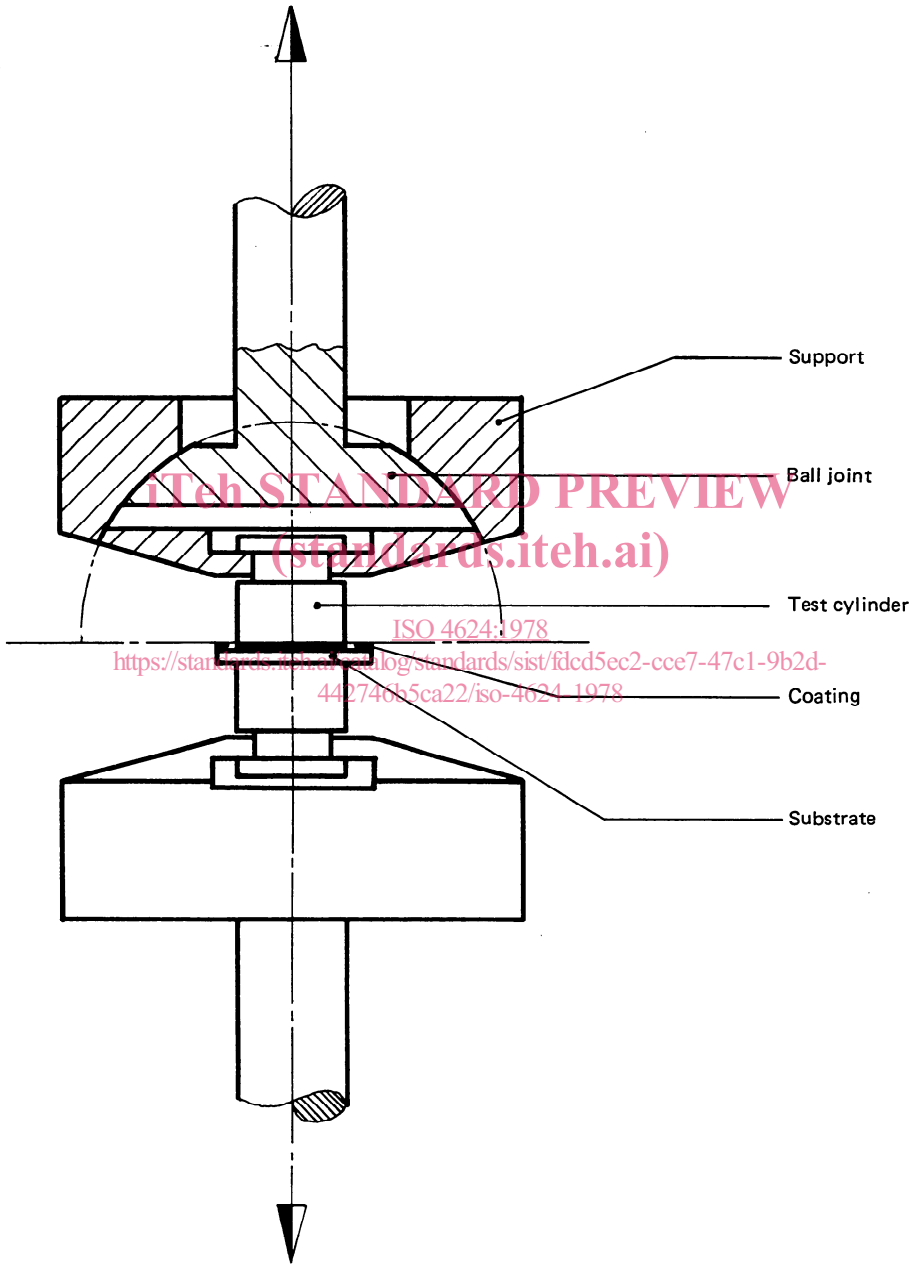

FIGURE 1 — **Example of a suitable test apparatus for methods specified in 7.3.1 and 7.3.3**

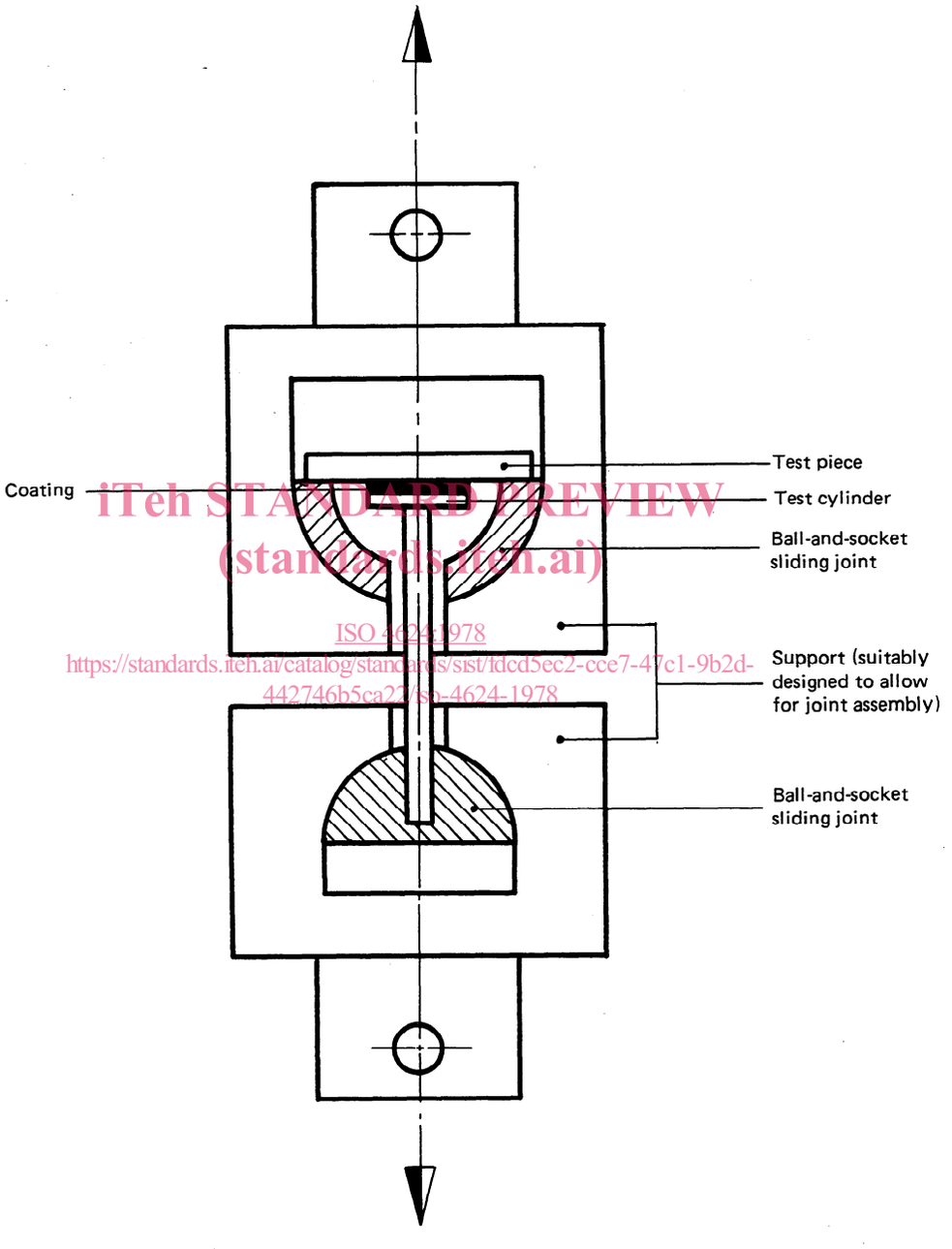

FIGURE 2 — **Example of a suitable test apparatus for method specified in 7.3.2**

3.3 Centring device, for ensuring proper coaxial alignment of the test assembly during the adhesion process used as described in 7.3.1 and 7.3.3. A suitable design is shown in figure 3.

3.4 Cutting device, such as a sharp knife, for cutting through cured adhesive and the paint coating to the substrate, round the circumference of the test cylinder.

4 ADHESIVES

Special attention is required in selecting suitable adhesives to be used in the test.

To produce failure of the coating, it is essential that the cohesive and bonding properties of the adhesive be greater than those of the coating under test.

Preliminary screening of adhesives shall be carried out in order to determine their suitability for use. Suitable adhesives and, if applicable, their unmixed components shall cause little or no visible change in the coating under test when left in contact with the coating for a period equivalent to the curing time of the adhesive.

An adhesive may be considered suitable for a particular coating if it gives the same test result as that produced by using a different class or type of adhesive when similarly tested.

NOTE — In most cases, cyanoacrylate, two-component solventless epoxide and peroxide-catalysed polyester adhesives have been found suitable. Cyanoacrylate and polyester adhesives have a short curing time and are preferred for use with coatings that have been subjected to highly humid conditions.

5 SAMPLING

Take a representative sample of the product to be tested (or of each product in the case of a multi-coat system) as specified in ISO 1512. Then examine and prepare the sample for testing as specified in ISO 1513.

6 TEST SUBSTRATES

6.1 Preparation and coating of test substrate

Prepare the specified test substrate in accordance with ISO 1514 and then coat it by the specified method with the product or system under test.

6.2 Drying the test coating

Dry (or stove and age) the coated test substrate for the specified time and under the specified conditions and condition it at 23 ± 2 °C and (50 ± 5) % relative humidity for a minimum of 24 h. Then carry out the appropriate test procedure as soon as possible.

6.3 Thickness of coating

Determine the thickness, in micrometres, of the dry coating by one of the procedures specified in ISO 2808. See also clause 1.

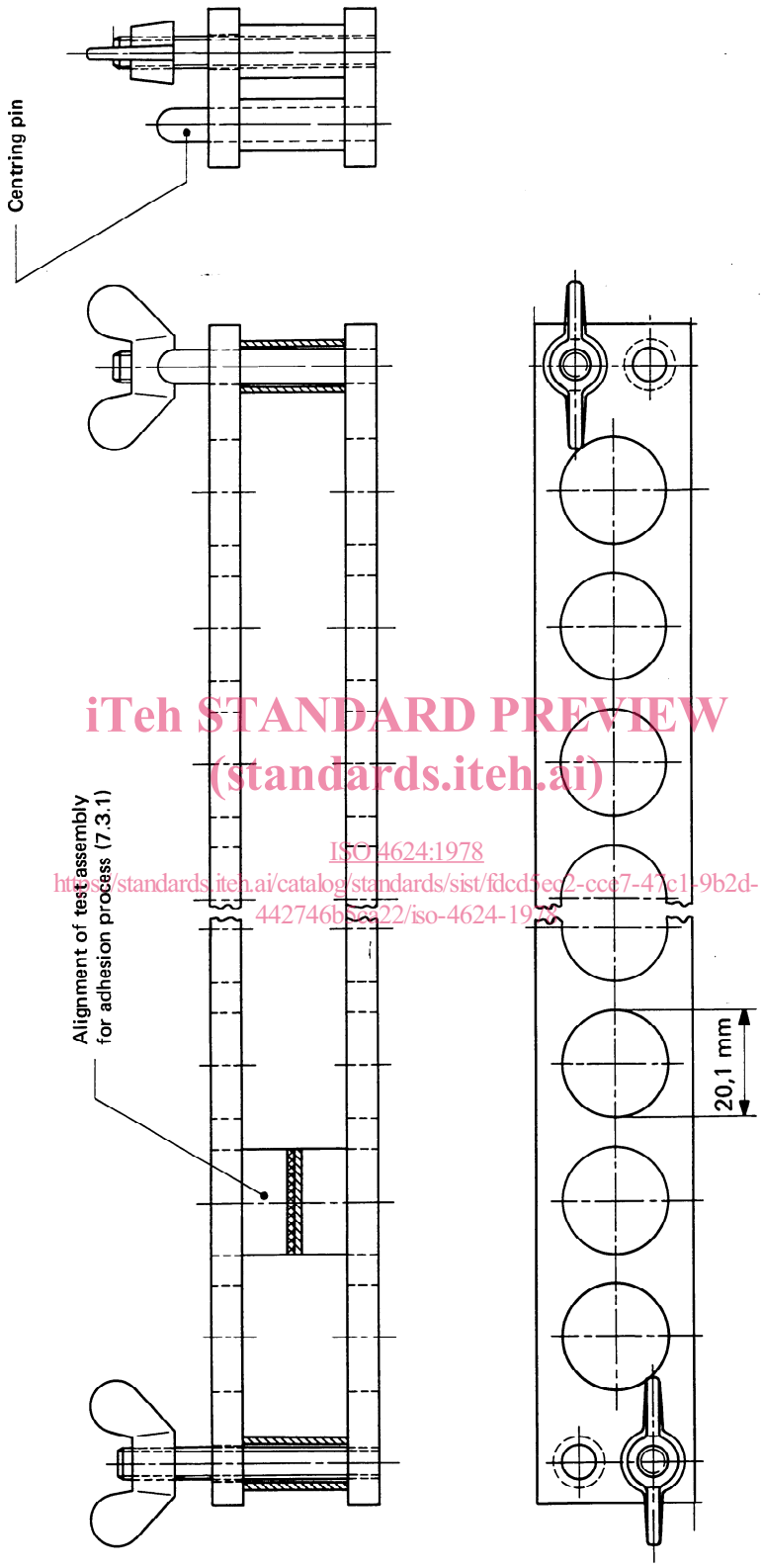

FIGURE 3 – Example of suitable centring device for 20 mm diameter test cylinders

7 PROCEDURE

7.1 Ambient conditions

Carry out the test at 23 ± 2 °C and (50 ± 5) % relative humidity.

7.2 Adhesives

Prepare and apply the adhesive in accordance with the manufacturer's instructions. Use the minimum quantity of adhesive required to produce a firm, continuous and even bond between the components of the test assembly. Remove any excess adhesive immediately if possible.

7.3 Test assemblies

7.3.1 *General method for testing both rigid and deformable substrates*

Use as the test piece an area, cut from the coated substrate (disc of minimum diameter 30 mm or square of minimum side 30 mm). Take care not to distort the test piece. Apply the adhesive evenly to the surfaces of two freshly-cleaned test cylinders (3.2) of equal diameter (see notes 1 and 3). Place the test piece between the adhesive-coated faces of the test cylinders such that the test cylinders are coaxially aligned in the centre of the piece, as shown in figure 4. Align the test assembly in the centring device (3.3) and maintain the alignment for a period equal to the curing time of the adhesive (see note 2). At the end of this period, carefully use the cutting device (3.4) to cut around the circumference of the test cylinders through to the substrate.

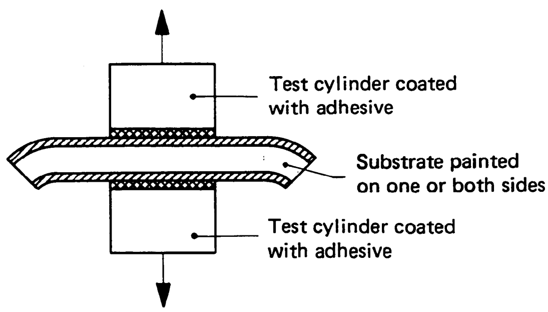

FIGURE 4 — **Test assembly, sandwich method (substrate painted on one or both sides)**

NOTES

1 The adhesion at the adhesive coating interface may be improved by lightly abrading the surface of the dried coating before application of the adhesive-coated face of the test cylinder.

2 In special tests under highly humid conditions, the curing time of the adhesive shall be as short as possible.

3 In the method for deformable substrates, if a poor adhesive bond is expected between the uncoated face of the substrate and the test cylinder, coat both faces of the substrate with the product under test.

7.3.2 *Method for testing from one side only (suitable for rigid substrates only)*

Apply the adhesive evenly to the uncoated, freshly-cleaned surface of a test cylinder (3.2). Place the adhesive-coated face of the test cylinder in contact with the coating, (see note 1 in 7.3.1), for a period equal to the curing time of the adhesive (see note 2 in 7.3.1). At the end of this period, carefully use the cutting device (3.4) to cut around the circumference of the test cylinder through to the substrate.

Place the outer ring in position and test as indicated in figure 5.

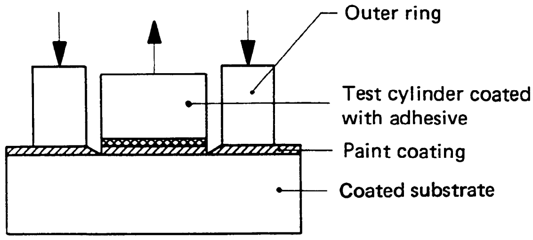

FIGURE 5 — **Test assembly for rigid substrates**

7.3.3 *Method using test cylinders*

Apply the adhesive evenly to the uncoated, freshly-cleaned surface of a test cylinder. Place the adhesive-coated surface of the test cylinder in contact with the surface of the cylinder coated with the product under test, as shown in figure 6, and align the test assembly in the centring device (3.3) for a period equal to the curing time of the adhesive.

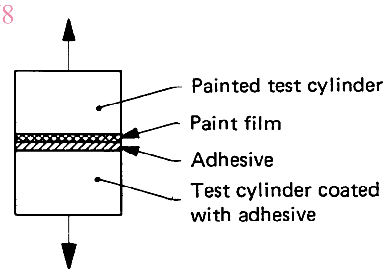

FIGURE 6 — **Test assembly using test cylinders**

7.4 Measurement

Immediately after the period allowed for the curing of the adhesive, place the test assembly in the tensile tester (3.1), taking care to align the test cylinders so that the tensile force is applied uniformly across the test area, without bending moment. Apply a tensile stress, increasing at a rate not greater than 1 MPa/s, perpendicular to the plane of the coated substrate such that failure of the test assembly shall occur within 90 s of initial application of the stress.

Record the tensile stress to break the test assembly and examine the fracture surfaces in accordance with 9.2.

7.5 Number of determinations

Carry out at least three determinations. For referee purposes, carry out a minimum of five determinations. Report the results of all determinations.

8 NOTES ON PROCEDURE

8.1 The results may be influenced by the test assembly used. Furthermore, the results are not reproducible unless coaxial alignment of the tensile forces is ensured.

8.2 Where failure is mainly associated with the adhesives, the use of another type of adhesive may enable more useful results to be obtained.

9 EXPRESSION OF RESULTS

9.1 Breaking strength

The breaking strength, in megapascals, for each test assembly is given by the formula

$$\frac{4F}{\pi d^2}$$

where

F is the breaking force, in newtons;

d is the diameter, in millimetres, of the test cylinder.

In the case of test cylinders of diameter 20 mm, the breaking strength, in megapascals, is given by the formula

$$\frac{4F}{400\pi} = \frac{F}{314}$$

9.2 Nature of failure

Express the result as the percentage area and site of fracture in the system under test in terms of adhesive, cohesive or adhesive/cohesive failure.

For convenience, the following scheme may be used to describe the results observed.

A = Cohesive failure of substrate

A/B = Adhesive failure between substrate and first coat

B = Cohesive failure of first coat

B/C = Adhesive failure between first and second coats

–/Y = Adhesive failure between final coat and adhesive

Y = Cohesive failure of adhesive

Y/Z = Adhesive failure between adhesive and test cylinder

Example : If a paint system tested in the pull-off test breaks at a tensile stress of 20 MPa and examination of the area on each side of the site of separation reveals approximately 30 % of the test cylinder area associated with cohesive break of the first coat and 70 % of the test cylinder area associated with intercoat adhesive break between the first and second coats, the pull-off test result is expressed as

20 MPa, 30 % B, 70 % B/C

10 TEST REPORT

The test report shall contain at least the following information :

a) the type and identification of the product(s) tested;

b) a reference to this International Standard or to a corresponding national standard;

c) the items of supplementary information referred to in the Introduction to this International Standard;

d) a reference to the national standard or other document supplying the information referred to in c) above;

e) the result of the test, reported as required in 9.1 and 9.2 (together with any further details required by the document referred to in c) above);

f) any deviation, by agreement or otherwise, from the procedure specified;

g) the date of the test.