INTERNATIONAL STANDARD

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Rubber — Assessment of carbon black dispersion — Rapid comparative methods

Caoutchouc — Évaluation de la dispersion du noir de carbone — Méthodes comparatives rapides

Reference number
ISO 11345:1997(E)

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 11345 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Physical and degradation tests.*

Annex A of this International Standard is for information only.

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Introduction

The degree of carbon black dispersion in a rubber compound is important because certain physical properties, e.g. tensile strength, hysteresis and abrasion resistance, are influenced by dispersion.

This method makes use of the well known fact that in a compound in which the ingredients are well dispersed, light is reflected from a freshly cut surface revealing a smooth, unblemished texture. The presence of improperly dispersed ingredients is shown by irregularities which usually take the form of circular, convex "bumps" or concave pockmarks on the surface, and their presence indicates a less-than-perfect dispersion of the compounding ingredients. The size and frequency of these irregularities may be used to judge the degree to which the compound falls short of an optimum dispersion. A set of ten standards based on size and frequency of these irregularities has been established to which numerical ratings have been assigned. This scheme provides a means of evaluating dispersion in a rubber compound and assigns numerical designations to the degrees of dispersion.

This International Standard describes test procedures for assessing the degree of macrodispersion of carbon black in rubber. The methods are primarily intended to be used as rapid factory controls during mixing and subsequent processing stages to assure adequate carbon black dispersion. Two test methods are described:

Method A: Visual microscopic or photographic/microscopic inspection

Method B: Split-field microscopic inspection

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Rubber — Assessment of carbon black dispersion — Rapid comparative methods

WARNING - Persons using this International Standard shall 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 qualitative visual test methods for the rapid and comparative assessment of macrodispersion only of carbon black in rubber. Ratings are made relative to a set of standard photographs (ratings 1 - 10), and the results are expressed on a numerical scale. This International Standard, with its standard photographs, is applicable only to compounds which contain carbon black.

2 Principle

The carbon black filled rubber compound is cut to expose a fresh surface for examination at a magnification of 30 x.

Two alternative methods are described:

Method A: Visual microscopic or photographic/microscopic inspection

Method B: Split-field microscopic inspection

The dispersion level of the carbon black is compared with a series of ten photographic or electronically stored standards photographed with oblique illumination of 30° at an effective magnification of 30 × and then rated numerically from 10 (excellent) to 1 (very poor). (Figure 1).

A rating of 10 indicates a state of dispersion having near maximum physical properties while a rating of 1 would indicate structural flaws causing considerably decreased physical properties. Normally, the visual dispersion ratings indicate the following levels of compound quality.

Visual dispersion rating	Dispersion classification
9-10	excellent
8	good
7	acceptable
5-6	doubtful
3-4	poor
1-2	very poor

3 Method A - Visual microscopic or photographic/microscopic inspection

3.1 Apparatus

3.1.1 Razor blade - single edge, attached to a sample cutter.

3.1.2 Sample cutter comprising a lever mechanism which provides a vertical cutting action and a razor blade holder.

3.1.3 Binocular microscope (30 x), or

3.1.4 Binocular microscope (30 x) equipped with a standard self developing (Polaroid type) camera.

3.1.5 Illuminator, microscopical-type, with high luminous intensity.

3.2 Test piece

3.2.1 Vulcanized compound

Cut a test piece, using the sample cutter, with a cross section of approximately 5 x 8 mm. Do not touch the surface to be used for rating. The razor blade shall be replaced before the edge wears such that the cut becomes lined.

For cured compounds the razor blade should be at standard laboratory temperature.

3.2.2 Unvulcanized compound

The compound shall first be compressed to remove most of the air holes since, even in small amounts, they can have the appearance of poorly dispersed carbon black and can therefore affect the rating. To accomplish this, press the rubber into a slab between thin sheets of plastic in a mould at a pressure of about 1 kPa for 5 min at 105 °C. Care should be taken to avoid excessive flow during this step. The surface to be examined should, as far as possible, be free from distortion and blemishes. To achieve this the cutting edge of the tool should be free from defects and the distortion of the sample shall be minimized by applying the cutting pressure evenly and slowly, with a sharp razor blade, heated to approximately 100 °C. However, even with all these precautions the evaluation of a cured specimen of the same compound may give a different result.

3.3 Procedure

Examine the prepared test piece under the binocular microscope with oblique illumination (at an angle of incidence 30°) to accentuate surface detail. The illuminator shall preferentially be placed parallel to the direction of cutting since any cutting lines will show less.

Compare the size and frequency of carbon black agglomerates in the specimens (showing up as surface bumps or depressions) to the standard photographs.

NOTE 1 - If a microscope with a self developing camera is used, the dispersion can be rated through a side by side comparison with the standard photographs using the photograph obtained. This provides a permanent record of the test piece appearance in a matter of minutes.

Assign the most closely matched numerical rating to each compound being rated in whole numbers. For closer matching use fractional ratings; 5 1/2 would indicate a rating between 5 and 6.

A rating of 10 indicates a state of dispersion having near maximum physical properties while a rating of 1 would indicate structural flaws causing decreased physical properties.

4 Method B - Split-field microscopic inspection

Method B is a test method which determines the degree of dispersion of carbon black in rubber compounds by means of a split-field video camera arrangement, which is used to rate the test piece against a set of transparencies or electronically stored standard references. The assessment of carbon black dispersion using the split-field optical microscopic technique is accomplished by a co-projection of the standard references images and the test piece image on a video-monitor using a black and white video camera.

The references are identical to the photographic standards referred to in Method A.

4.1 Apparatus

4.1.1 Razor blade, see 3.1.1.

4.1.2 Sample cutter, see 3.1.2.

4.1.3 Equipment for split-field representation. The instrument is based on the so-called split-field optical microscopic technique where the photographic 10-grade standard is projected side by side with the reflected image of the test piece surface. The reference pictures are mounted on a rotary disc or are electronically stored and can be operated so that the reference standard matches the appearance of the test piece surface.

A video or a CCD camera could be used together with a monitor to provide a side by side composite picture of test piece and standard. (Figures 2a and 2b).

4.2 Test piece

The test pieces needed for Method B shall be prepared in the same manner as described in 3.2.

4.3 Procedure

Activate the instrument and allow for the specified warm-up time.

Place the test piece with the fresh cut surface against the test piece holder. The test piece shall be placed the same way as in the cutter, with the cutting direction vertical.

Examine the prepared test piece in the instrument with oblique illumination (at an angle of incidence 30°). Compare the size and frequency of carbon black agglomerates in the test piece (showing up as bumps or depressions) to the standard photographs by direct side by side comparison. Chose amongst the stored standard reference pictures, the standard photography that closest matches the image of the test piece by systematically switching the reference pictures in the side position to the image under examination.

Assign the most closely matched numerical rating to each compound being rated in whole numbers. For closer matching use fractional ratings; 5 1/2 would indicate a rating between 5 and 6.

A rating of 10 indicates a state of dispersion having near maximum physical properties while a rating of 1 would indicate structural flaws causing decreased physical properties.

5 Number of tests

Preferably more than one test on different parts of each test specimen should be taken.

6 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) a complete identification of the material tested, including type, whether vulcanized or unvulcanized, source, manufacturer's code number and previous history;

c) whether Method A or B was used;

d) list the rating on the basis of the 10 to 1 scale by the photographic standards. Use fractional ratings when necessary. If more than one operator has rated the material, the number of observations and the average rating should be reported;

e) the date of test.

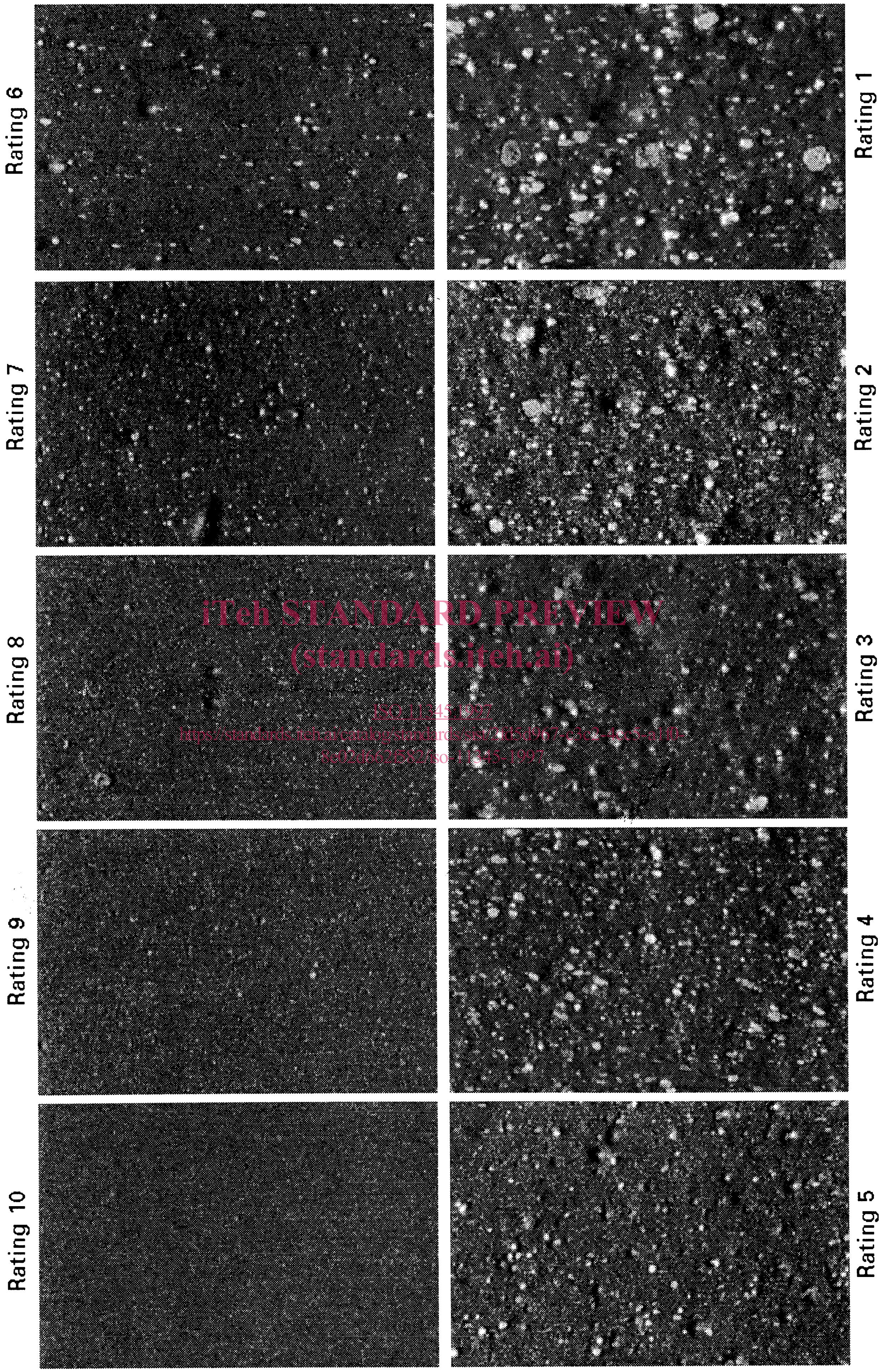

Figure 1 — Photographic standards