
**Rubber — Assessment of carbon black
and carbon black/silica dispersion —
Rapid comparative methods**

*Caoutchouc — Évaluation de la dispersion du noir de carbone et du noir
de carbone/silice — Méthodes comparatives rapides*

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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

This second edition cancels and replaces the first edition (ISO 11345:1997), which has been technically revised.

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Introduction

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

The methods described in this International Standard make 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 four standards each comprising ten photographs 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 and silica 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. Five alternative methods are described.

The addition of a higher magnification and photographic reference standards for silica was in response to a request from tyre manufacturers and filler producers. Method E was specially requested by manufacturers of extruded profiles for the automotive industry.

Equipment with electronically stored standards may be supplied by:

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Rubber — Assessment of carbon black and carbon black/silica dispersion — Rapid comparative methods

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 qualitative visual methods for the rapid and comparative assessment of the degree of macrodispersion of carbon black and carbon black/silica in rubber. Ratings are made relative to a set of standard reference photographs, and the results are expressed on a numerical scale from 1 to 10.

In addition, a method is given for rating the presence of large agglomerates on a numerical scale (ratings 1 to 10).

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2 Normative references (standards.iteh.ai)

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1382, *Rubber — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1382 apply.

4 Principle

The carbon black or carbon black/silica filled rubber compound is cut to expose a fresh surface for examination under magnification.

Five methods are described:

- **Method A:** Visual microscopic or photographic microscopic inspection with $\times 30$ magnification (for carbon black).
- **Method B:** Split-field microscopic inspection with $\times 30$ magnification (for carbon black).
- **Method C:** Visual microscopic or photographic microscopic inspection with $\times 100$ magnification (for carbon black or carbon black/silica).
- **Method D:** Split-field microscopic inspection with $\times 100$ magnification (for carbon black or carbon black/silica).
- **Method E:** Large-agglomerate count with $\times 100$ magnification (for carbon black).

In methods A to D, the dispersion level is compared with a series of ten hard-copy or electronically stored standard references under oblique illumination at 30° (to accentuate detail) and with an effective magnification of × 30 for methods A and B (see Annex A) and × 100 for methods C and D (see Annexes B to E), and then rated numerically from 10 (excellent) to 1 (very poor).

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

Visual dispersion rating	Dispersion classification
9 to 10	Excellent
8	Good
7	Acceptable
5 to 6	Doubtful
3 to 4	Poor
1 to 2	Very poor

In method E, the presence of agglomerates is analysed using an image-processing system under oblique illumination at 30° and with an effective magnification of × 100, and then rated numerically from 10 (excellent) to 1 (very poor). The rating 10 is based on the absence of clusters with a diameter of 23 µm or larger, indicating that the agglomerates are much smaller than this. The rating 1 corresponds to the maximum number of large agglomerates which can be achieved in practice.

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5 Number of tests

A minimum of five tests on different parts of each test piece shall be carried out.

6 Method A — Visual microscopic or photographic microscopic inspection with × 30 magnification

6.1 General

Method A is a test method which determines the degree of dispersion of carbon black in rubber compounds by means of visual microscopic or photographic microscopic inspection, which is used to rate the test piece against a set of transparencies or electronically stored standard references.

6.2 Apparatus

6.2.1 Razor blade, with a single edge, attached to a sample cutter.

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

6.2.3 Binocular microscope (× 30), or 6.2.4.

6.2.4 Binocular microscope (× 30), equipped with a standard self-developing or digital camera with at least 2 megapixel resolution.

6.2.5 Illuminator, microscope type, with high luminous intensity.

6.3 Test piece

6.3.1 Vulcanized compound

Using the sample cutter, cut out a test piece with a cross-section of approximately 8 mm thickness and 10 mm width. Do not touch the surface to be used for rating. The razor blade shall be replaced before the edge wears to the extent that the cut becomes lined.

6.3.2 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. Take care to avoid excessive flow during this step. The surface to be examined shall, as far as possible, be free from distortion and blemishes. To achieve this, the cutting edge of the tool shall be free from defects and the distortion of the sample shall be minimized by applying the cutting pressure evenly and slowly with the sharp razor blade heated to approximately 100 °C. However, even with all these precautions, the evaluation of a cured test piece of the same compound may give a different result.

6.4 Procedure

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

Compare the size and frequency of any carbon black agglomerates in the test piece (showing up as surface bumps or depressions) with those in the reference photographs (see Annex A).

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

Assign to each compound being assessed the most closely matching numerical rating, using 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 representing near-optimum physical properties while a rating of 1 indicates the presence of structural flaws, resulting in inferior physical properties.

7 Method B — Split-field microscopic inspection with × 30 magnification

7.1 General

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

The reference images are identical to the photographic references used in method A.

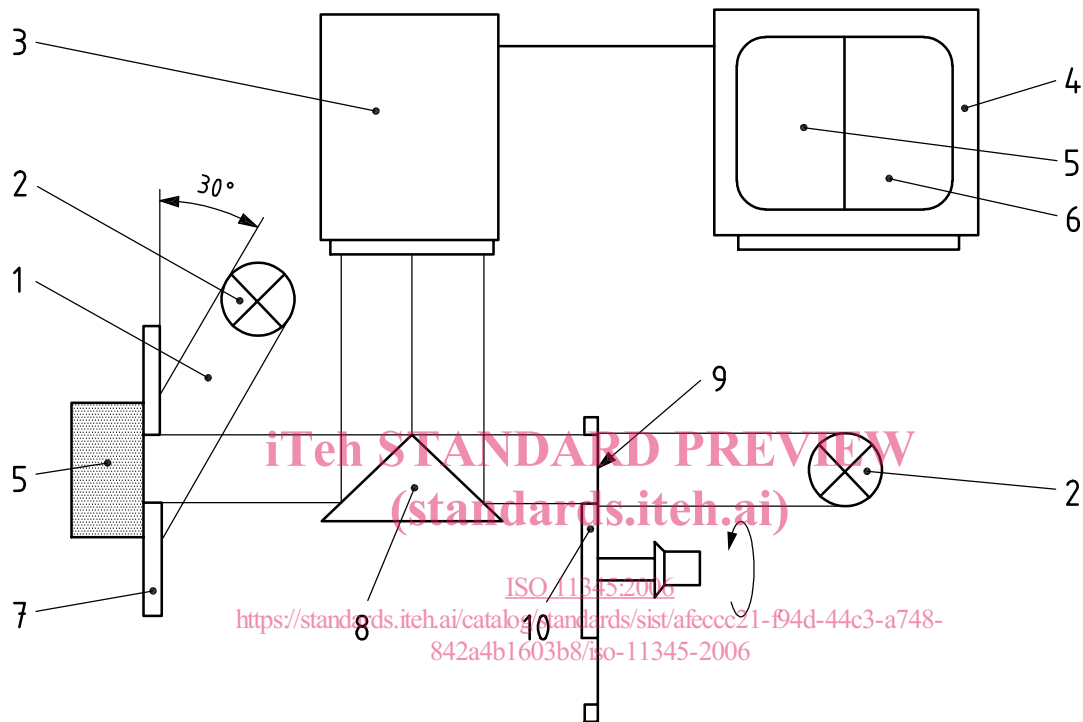
7.2 Apparatus

7.2.1 Razor blade, with a single edge, attached to a sample cutter.

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

7.2.3 Equipment for split-field representation. The instrument is based on the so-called split-field optical-microscope technique in which each of the 10 references can be projected side by side with the reflected image of the test piece surface. The references are mounted on a rotary disc (in the case of transparencies) or are electronically stored (in the case of digital images) and can be viewed successively until the reference which best matches the appearance of the test piece surface is found.

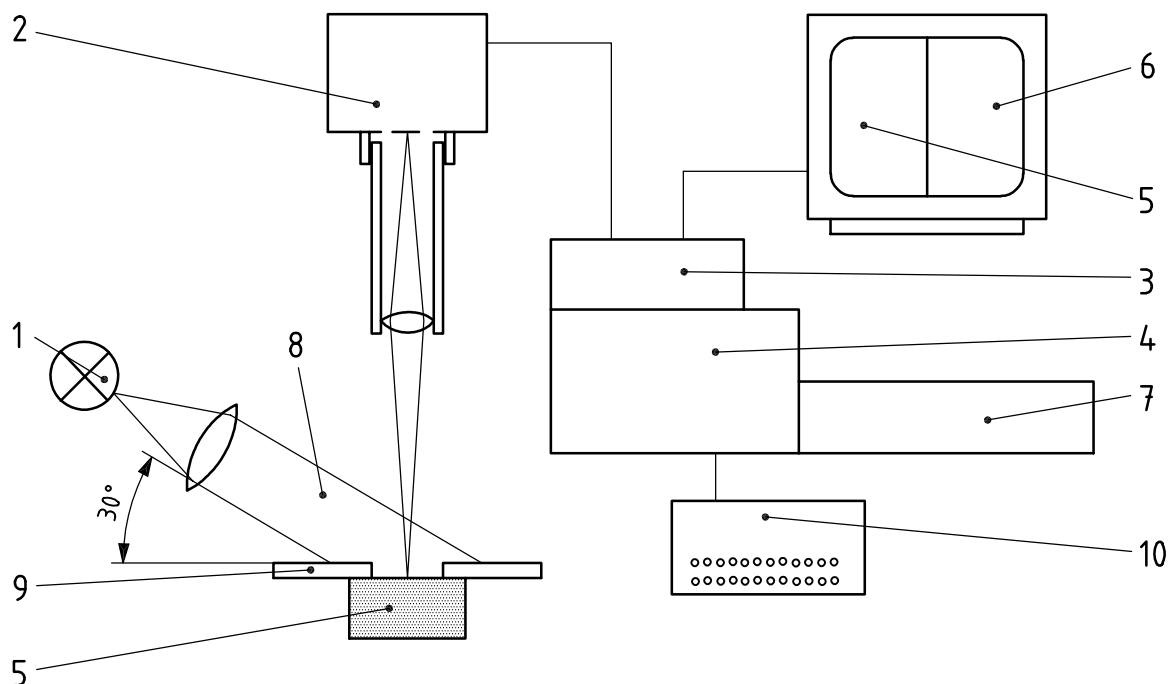
A video camera or CCD camera is used together with a monitor to provide a composite side-by-side picture of the test piece and a reference (see Figures 1 and 2).



Key

- | | |
|----------------|--------------------------|
| 1 light beam | 6 reference |
| 2 lamp | 7 test piece holder |
| 3 video camera | 8 prism |
| 4 TV monitor | 9 reference transparency |
| 5 test piece | 10 transparency carrier |

Figure 1 — Apparatus for methods B and D with a video camera

**Key**

- | | |
|-----------------|-------------------------------------|
| 1 lamp | 6 reference |
| 2 CCD camera | 7 digital memory (reference images) |
| 3 electronics | 8 light beam |
| 4 microcomputer | 9 test piece holder |
| 5 test piece | 10 keyboard |

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Figure 2 — Apparatus for methods B and D with a CCD camera

7.3 Test piece

Prepare the test piece for method B in the same manner as described in 6.3.

7.4 Procedure

Switch on the instrument and allow it to warm up for the specified time.

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

Examine the prepared test piece in the instrument with oblique illumination (at an angle of incidence of 30°). Compare the size and frequency of any carbon black agglomerates in the test piece (showing up as bumps or depressions) to the size and frequency of the agglomerates in the reference images by direct side-by-side comparison. Choose, from the reference images, the one that most closely matches the image of the test piece by successively projecting the reference images beside the image of the test piece under examination.

Assign the most closely matching numerical rating to each compound being assessed 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 representing near-optimum physical properties while a rating of 1 indicates the presence of structural flaws, resulting in inferior physical properties.

8 Method C — Visual microscopic or photographic microscopic inspection with × 100 magnification

8.1 General

Method C determines the degree of dispersion of carbon black or carbon black/silica in rubber compounds by means of visual microscopic or photographic microscopic inspection, which is used to rate the test piece against a set of transparencies or electronically stored standard references.

8.2 Apparatus

8.2.1 **Razor blade**, with a single edge, attached to a sample cutter.

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

8.2.3 **Binocular microscope** (× 100), or 8.2.4.

8.2.4 **Binocular microscope** (× 100), equipped with a standard self-developing or digital camera with at least 2 megapixel resolution.

8.2.5 **Illuminator**, microscope type, with high luminous intensity.

8.3 Test piece

Prepare the test piece for method C in the same manner as described in 6.3.

8.4 Procedure

Examine the prepared test piece under the binocular microscope with oblique illumination (at an angle of incidence of 30°) to accentuate surface detail. The illuminator should preferably be placed so that the light beam is parallel to the direction of cutting, so that any cutting lines will show less.

Compare the size and frequency of any carbon black or carbon black/silica agglomerates in the test piece (showing up as surface bumps or depressions) to the size and frequency of the agglomerates in the reference images. Use the set of references that is closest to the main type of filler used in the compound. (The relationships between the sets of references are illustrated in Figure 3.)

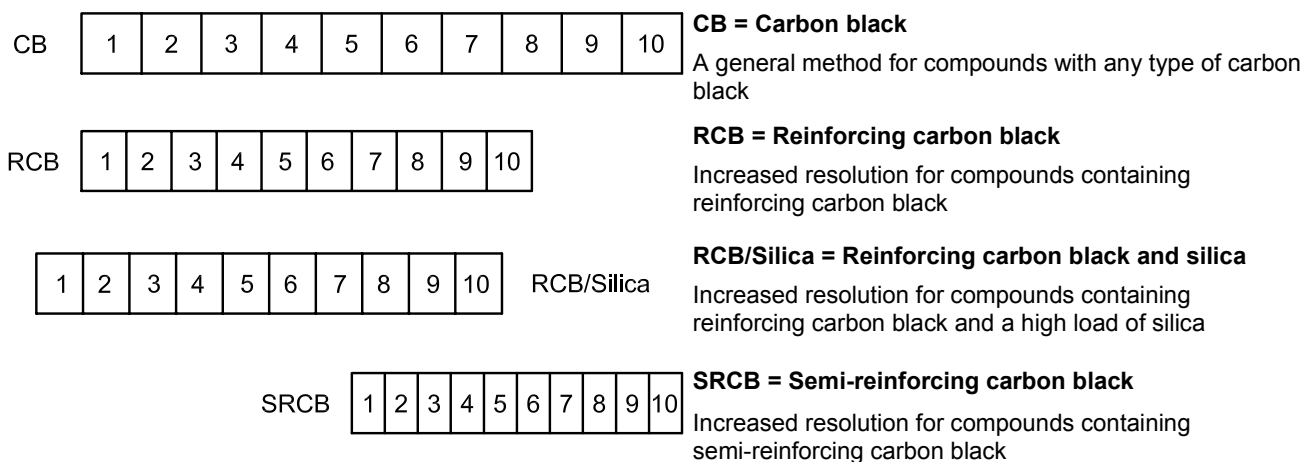


Figure 3 — The principle of the photographic references used in methods C and D