



SLOVENSKI STANDARD
SIST EN 14469-2:2004

01-september-2004

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Pigments and extenders - Testing of colouring materials in plasticized polyvinyl chloride (PVC-P) - Part 2: Preparation of test specimens

Pigmente und Füllstoffe - Prüfung von Farbmitteln in weichmacherhaltigem Polyvinylchlorid (PVC-P) - Teil 2: Herstellen der Probenkörper

Pigments et matieres de charge - Essai des matieres colorantes dans le chlorure de polyvinyle plastifié (PVC-P) - Partie 2: Préparation des éprouvettes

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Ta slovenski standard je istoveten z: EN 14469-2:2004

ICS:

83.040.30	Pomožni materiali in aditivi za polimerne materiale	Auxiliary materials and additives for plastics
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EUROPEAN STANDARD

EN 14469-2

NORME EUROPÉENNE

EUROPÄISCHE NORM

April 2004

ICS 83.040.30

English version

Pigments and extenders - Testing of colouring materials in plasticized polyvinyl chloride (PVC-P) - Part 2: Preparation of test specimens

Pigments et matières de charge - Essai des matières colorantes dans le chlorure de polyvinyle plastifié (PVC-P) - Partie 2: Préparation des éprouvettes

Pigmente und Füllstoffe - Prüfung von Farbmitteln in weichmacherhaltigem Polyvinylchlorid (PVC-P) - Teil 2: Herstellen der Probenkörper

This European Standard was approved by CEN on 2 February 2004.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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EN 14469-2:2004 (E)**Foreword**

This document (EN 14469-2:2004) has been prepared by Technical Committee CEN/TC 298 "Pigments and extenders", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2004, and conflicting national standards shall be withdrawn at the latest by October 2004.

Annex A is informative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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1 Scope

This Part of EN 14469 specifies a procedure for preparing test samples for the testing of colouring materials¹⁾ in plasticized polyvinyl chloride (PVC-P) materials. It describes the manner in which basic mixtures as defined in EN 14469-1 and pigments and pigment preparations are to be used to prepare test samples for the testing of particular pigment properties. The standard is applicable to inorganic and organic pigments, and also to pigment preparations in powder, paste or granular form (see annex A (informative)).

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN ISO 15528, *Paints, varnishes and raw materials for paints and varnishes - Sampling (ISO 15528:2000)*.

3 Apparatus

3.1 Mixing vessel

3.2 Two-roll mill

which can be heated, with rolls whose separation can be adjusted, roll diameters shall be between 80 mm and 200 mm, ratio of the speeds of rotation of the two rollers shall be between 1:1,1 and 1:1,2.

NOTE The rolls used should preferably have a chromed surface.

3.3 Plate press (if required according to 5.3)

which can be heated and cooled.

4 Sampling

Representative samples of the colouring materials to be tested shall be taken as described in EN ISO 15528.

5 Preparation of the test specimens

5.1 Premixing

The quantity of colouring material specified for the particular test sample is mixed for example for 5 min in a shaking mixer with the PVC basic mixture on which the test sample is based. Alternatively, and in the particular case of colouring materials in paste form, it is recommended that premixing be carried out by hand using a spatula in a polyethylene or polypropylene beaker, until the mixture appears homogeneous.

NOTE When testing pigment not susceptible to reagglomeration under pressure, premixing can be dispensed with.

1) For definition of colouring material, see EN 971-1.

EN 14469-2:2004 (E)**5.2 Two-roll milling**

The mix is placed on the two-roll mill, the roll surfaces of which have been adjusted to a temperature of (160 ± 5) °C. A temperature difference between the rolls is permissible if it is within these limits. The amount of mix is judged so that once a milled sheet has formed there is always a rotating bank of molten material in the gap between the rolls. The gap between the rolls is to be adjusted so that the milled sheet has a uniform thickness of 0,4 mm to 0,5 mm.

The sheet is formed in such a way that the whole of the material forms a continuous sheet on the front roll. During the rolling process, the milled sheet shall be continuously turned over or removed and reapplied, so as to give thorough mixing.

NOTE When the colouring material has not been premixed, it should be added gradually and uniformly to the rolling bank.

After the mix has been applied it should be worked for 200 roll revolutions. The rolling time shall be at least 5 min and not exceed 10 min.

The milled sheet is then drawn off. To this end, it is permissible to change the gap between the rolls and, if desired, the speed of rotation and friction.

5.3 Pressing of test specimens

It may be necessary to press the milled sheet in order to improve the surface quality, e.g. to achieve high gloss or to create a sufficiently thick sample for photometric measurement.

The sections of the sheet and a spacer frame of the desired thickness, are placed between high-gloss chromed plates in a plate press heated to a temperature between 165 °C and 170 °C, and pressed for not more than 2 min. The pressed sheet is then cooled rapidly.

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Annex A (informative)

Explanations

The production of sheets on two-roll mills is customary in the laboratories concerned. The manner in which the pigments are incorporated into the basic mixtures, and the process conditions, are important for the degree of dispersion obtained. This, in turn, is a decisive factor in determining the properties of the coloured materials. To obtain reproducible results, suitable conditions were determined through extended experimental work. Variations in laboratory apparatus have been taken into account in this standard, as has ease of implementation.

The following remarks apply to individual sections of the standard:

Clause 1

When testing preparations, it should be borne in mind that the carrier material on which the preparation is based can alter the overall properties of the basic mixture. If the carrier for the pigment preparation is incompatible with the basic mixture, miscibility is frequently also poor. On the other hand if miscibility is good, any effect which the carrier material may have on the properties of the basic mixture can usually only be detected by specific tests. Only then is it possible to arrive at decisions concerning the usability of pigment preparation.

Clause 3

The standard takes account of the variations found in roll diameters of conventional laboratory two-roll mills.

Clause 5.1

The manner in which the pigment is incorporated into the basic mixtures prior to two-roll milling has a marked effect on the colour results obtained after the roll milling of the sheet. No additional grinding of the pigment should take place in the mixing procedure. Incorporation by the method described ensures that this is so. Dispersing of the pigments should not take place until the sheets are milled. The milling time and the number of rotations, and also the setting of the gap between the rolls, are therefore important.

Clause 5.3

It is recommended that the milled sheets are then pressed. However, this should be done under conditions which as far as possible avoid any thermal degradation. The conditions specified take account of this requirement.

NOTE Where photometric measurement is carried out directly on the sheet on the mill, pressing can be dispensed with.

When basic mixture B is used, the minimum layer thickness required for photometric evaluation is 1 mm.

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Bibliography

EN 971-1, *Paints and varnishes - Terms and definitions for coating materials – Part 1: General terms.*

EN 14469-1, *Pigments and extenders – Testing of colouring materials in plasticized polyvinyl chloride (PVC-P) – Part 1: Composition and preparation of basic mixtures.*

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