BRITISH STANDARD

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Pigments and extenders — Testing of colouring materials in plasticized polyvinyl chloride (PVC-P) —

Part 1: Composition and preparation of basic mixtures

The European Standard EN 14469-1:2004 has the status of a British Standard

ICS 83.040.30

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National foreword

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Pigments and extenders - Testing of colouring materials in plasticized polyvinyl chloride (PVC-P) - Part 1: Composition and preparation of basic mixtures

Pigments et matières de charge - Essai des matières colorantes dans le chlorure de polyvinyle plastifié (PVC-P) - Partie 1: Composition et préparation des mélanges de base

Pigmente und Füllstoffe - Prüfung von Farbmitteln in weichmacherhaltigem Polyvinylchlorid (PVC-P) - Teil 1: Zusammensetzung und Herstellen der Grundmischungen

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Foreword

This document (EN 14469-1:2004) 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of an identical text or by endorsement, at the latest by November 2004, and conflicting national standards shall be withdrawn at the latest by November 2004.

Annex A is informative.

This document includes a Bibliography.

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Introduction

In this standard percent (%) in relation to contents indicates percentage proportion by mass.

1 Scope

This Part of EN 14469 specifies a procedure for producing basic mixtures for the testing of colouring materials[1)] in plasticized polyvinyl chloride (PVC-P) materials, together with the composition of these basic mixtures.

It distinguishes between:

- basic mixture A (transparent basic mixture)

- basic mixture B (white basic mixture)

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 14469-2, *Pigments and extenders — Testing of colouring materials in plasticized polyvinyl chloride (PVC-P) — Part 2: Preparation of test specimens.*

EN ISO 489, *Plastics — Determination of refractive index (ISO 489:1999).*

EN ISO 591-1, *Titanium dioxide pigments for paints — Part 1: Specifications and methods of test (ISO 591-1:2000).*

EN ISO 1060-1, *Plastics — Homopolymer and copolymer resins of vinyl chloride — Part 1: Designation system and basis for specifications (ISO 1060-1:1998).*

EN ISO 1628-2, *Plastics - Determination of the viscosity of polymers in dilute solution using capillary viscometers — Part 2: Poly(vinyl chloride) resins (ISO 1628-2:1998).*

EN ISO 2114, *Plastics (polyester resins) and paints and varnishes (binders) - Determination of partial acid value and total acid value (ISO 2114:2000).*

EN ISO 12185, *Crude petroleum and petroleum products — Determination of density — Oscillating U-tube method (ISO 12185:1996).*

ISO 6271, *Clear liquids — Estimation of colour by the platinum-cobalt scale.*

3 Constituents of the basic mixtures

3.1 General

The quality of all constituents of the basic mixtures prepared according to this standard shall be such that the samples produced from these basic mixtures as defined in EN 14469-2 are free from any inhomogenity. All raw materials used shall meet the specified values. Their trade names shall be mentioned in the product specification.

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

3.2 Vinyl chloride homopolymer

Vinyl chloride homopolymer (suspension polymer for general use as defined in EN ISO 1060-1). Principal properties as defined in Table 1.

Table 1 – Vinyl chloride homopolymer

Property	Value	Test as defined in
K value	70 ± 1	EN ISO 1628-2

3.3 Plasticizer

Diisodecyl phthalate (DIDP)[2] , stabilized, with values as defined in Table 2.

Table 2 – Diisodecyl phthalate

Property	Value	Test as defined in
Refractive index n_D^{20}	1,4850 to 1,4860	EN ISO 489
Colour value	≤ 200	ISO 6271

3.4 Epoxidized soybean oil

Epoxidized soybean oil (ESO)[3] with values as defined in Table 3.

Table 3 – Epoxidized soybean oil

Property		Value	Test as defined in
Refractive index n_D^{20}		1,4720 to 1,4740	EN ISO 489
Iodine colour value ICV	mg/100ml	≤ 3	By agreement between the interested parties [a]
Content of epoxide oxygen	%	6,2 to 6,8	By agreement between the interested parties [b]

[a] See DIN 6162.

[b] See ASTM D 1652

2) For plasticizer codes see EN ISO 1043-1.

3) For plasticizer codes see EN ISO 1043-1.

3.5 Stabilizer

Liquid Ba/Zn stabilizer system with values as defined in Table 4.

Table 4 – Ba/Zn stabilizer system

Property	Value	Test as defined in
Barium content %	10,2 to 12,2	[a]
Zinc content %	1,95 to 2,35	[a]
Refractive index $n\frac{20}{D}$	1,4970 to 1,5010	EN ISO 489
[a] Determination by atomic absorption spectrometry		

Any other Ba/Zn stabilizer system which gives the same test results may be used instead of this system.

3.6 Lubricant

Stearic acid with values as defined in Table 5.

Table 5 – Stearic acid

Property	Value	Test as defined in
Density ρ (80 °C) g/ml	0,840 to 0,850	EN ISO 12185
Acid value AV mg KOH/g	206 to 211	EN ISO 2114

3.7 Titanium dioxide pigment

Titanium dioxide pigment with values as defined in Table 6.

Table 6 – Titanium dioxide pigment

Property	Value	Test as defined in
Titanium dioxide (TiO_2) content %	92 to 98	EN ISO 591-1
Rutile content of the titanium dioxide part in the pigment %	≥ 98	By agreement between the interested parties
Silicon dioxide (SiO_2) content and/or aluminium oxide (Al_2O_3) content %	2 to 8	By agreement between the interested parties

4 Apparatus

High-speed mixer, adjustable, heatable and coolable, peripheral speed between 15 m/s and 35 m/s.

5 Composition of the basic mixtures

5.1 Basic mixture A (transparent basic mixture)

65,0	parts by mass of vinyl chloride homopolymer	as defined in 3.2
33,5	parts by mass of plasticizer	as defined in 3.3
1,5	parts by mass of epoxidized soybean oil	as defined in 3.4
1,3	parts by mass of stabilizer	as defined in 3.5
0,2	part by mass of lubricant	as defined in 3.6
101,5	parts by mass = total	

5.2 Basic mixture B (white basic mixture)

65,00	parts by mass of vinyl chloride homopolymer	as defined in 3.2
33,50	parts by mass of plasticizer	as defined in 3.3
1,50	parts by mass of epoxidized soybean oil	as defined in 3.4
1,30	parts by mass of stabilizer	as defined in 3.5
0,20	part by mass of lubricant	as defined in 3.6
5,34	parts by mass of titanium dioxide pigment	as defined in 3.7
106,84	parts by mass = total	

NOTE The titanium dioxide pigment content of the basic mixture has been set so that approximately 5 parts by mass of titanium dioxide pigment are present in 100 parts by mass of finished basic mixture.

6 Preparation of basic mixtures

PVC, stabilizer and lubricant are premixed in a high-speed mixer until the mix temperature is 70 °C. In the case of basic mixture B, the titanium dioxide pigment is then added and mixed for about 2 min (if the mixing time is longer there is a risk of greying due to metal abrasion). The premixed amounts of plasticizer and epoxidized soybean oil are then added as a fine stream while the mixer is running. The homogeneous mixture, the temperature of which has risen to about 100 °C as a result of the mixing procedure, is cooled to room temperature with continuing agitation.

The basic mixtures shall not be stored for longer than 2 years, and shall be hermetically sealed and protected from exposure to light.

Annex A
(informative)

Explanations

When testing colouring materials in polyvinyl chloride, the thermal instability of pure PVC makes it necessary to begin by preparing basic mixtures with a number of additives.

In interlaboratory tests by the "PVC basic mixture" plasticized PVC basic mixtures stabilized with a Ba/Zn stabilizer system gave the best stability. The intention is to avoid any adverse effect on the pigmented samples due to colour change in the basic mixtures.

The basic mixtures include a lubricant to prevent plate-out. Its nature and amount were assessed on the basis of interlaboratory tests, in such a way as practically to eliminate adverse effects under the test conditions proposed.

The vinyl chloride homopolymer chosen was a suspension polymer, since this is currently the most widely used. Without exception, the films produced from the basic mixtures gave good results on exposure to heat and in lightfastness tests.

Bibliography

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

EN ISO 1043-1, *Plastics — Symbols and abbreviated terms — Part 1: Basic polymers and their special characteristics (ISO 1043-1:2001)*.

ASTM D 1652, *Standard test methods for epoxy content of epoxy resins.*

DIN 6162, Determination of iodine colour number

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