

	Plastics Vinyl chloride homopolymer and copolymer resins Sieve analysis in water (ISO 1624 : 2001) English version of DIN EN ISO 1624	DIN EN ISO 1624
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ICS 83.080.20

Supersedes October 1997 edition.

Kunststoffe – Vinylchlorid-Homo- und Copolymerisate –
 Siebanalyse im Wasser (ISO 1624 : 2001)

European Standard EN ISO 1624 : 2001 has the status of a DIN Standard.

A comma is used as the decimal marker.

National foreword

This standard has been published in accordance with a decision taken by CEN/TC 249 to adopt, without alteration, International Standard ISO 1624 as a European Standard.

The responsible German body involved in its preparation was the *Normenausschuss Kunststoffe* (Plastics Standards Committee), Technical Committee *PVC-Formmassen*.

DIN ISO 565 is the standard corresponding to International Standard ISO 565 referred to in clause 2 of the EN.

Amendments

This standard differs from the October 1997 edition in that the drying temperature has been increased from 80 °C to 110 °C and precision data have been included.

Previous edition

DIN EN ISO 1624: 1997-10.

National Annex NA

Standard referred to

(and not included in **Normative reference**)

DIN ISO 565 Test sieves – Metal wire cloth, perforated metal plate and electroformed sheet – Nominal sizes of openings (ISO 565 : 1990)

EN comprises 8 pages.

English version

Plastics

Vinyl chloride homopolymer and copolymer resins

Sieve analysis in water
(ISO 1624 : 2001)

Plastiques – Résines d'homopolymères et de copolymères de chlorure de vinyle – Analyse granulométrique par tamisage sous courant d'eau (ISO 1624 : 2001)

Kunststoffe – Vinylchlorid-Homo- und Copolymerisate – Siebanalyse im Wasser (ISO 1624 : 2001)

This European Standard was approved by CEN on 2001-12-15.

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 Management Centre or to any CEN member.

The European Standards exist 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 Management Centre has the same status as the official versions.

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Management Centre: rue de Stassart 36, B-1050 Brussels

Foreword

International Standard
ISO 1624 : 2001 Plastics – Vinyl chloride homopolymer and copolymer resins – Sieve analysis in water, which was prepared by ISO/TC 61 ‘Plastics’ of the International Organization for Standardization, has been adopted by Technical Committee CEN/TC 249 ‘Plastics’, the Secretariat of which is held by IBN, as a European Standard.
This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, and conflicting national standards withdrawn, by June 2002 at the latest.
In accordance with the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard:
Austria, Belgium, the Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, the Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 1624 : 2001 was approved by CEN as a European Standard without any modification.

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1 Scope

This International Standard specifies a method for the determination of the sieve retention of vinyl chloride homopolymer and copolymer resins. Control of this characteristic can help to ensure consistency of supply and predictable processing behaviour.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

3 Term and definition

For the purposes of this International Standard, the following term and definition apply.

3.1

sieve retention

the percentage, by mass, of resin remaining on the sieve after a sieve analysis test

4 Principle

A test portion is sieved under a stream of water, using standard-aperture sieves.

NOTE Sieving under a stream of water gives truer results than a dry sieving method in which static electricity interferes. This method is particularly suited to emulsion resins.

5 Reagent

5.1 **Wetting agent**, for example a 5 % to 10 % solution of sodium alkylsulfonate.

6 Apparatus

6.1 Set of two sieves, diameter 200 mm to 300 mm, height 30 mm to 50 mm, having mesh sizes 0,063 mm and 0,250 mm, and fitted with deflectors to prevent loss of sample by spray (see Figure 1).

NOTE Other mesh sizes may be used provided that

- the prescribed sieves are also used;
- new sieves are chosen from the series provided in ISO 565.

6.2 Balance, accurate to $\pm 0,01$ g, range and size sufficient to accommodate the sieves and the filtered resin [see 7.1 b)].

6.3 Oven, capable of being maintained at $110\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

6.4 Filter, placed between the water supply tap and the apparatus, and intended to exclude impurities in the water supply.

6.5 Sprinkling device, for one or, better, several sieves, designed so that:

- the mesh and inside walls of the sieve are thoroughly sprinkled over their entire surface;
- the resin is agitated by the jets, e.g. by using jet apertures inclined at an angle to the screen of the sieve;
- the inclination of the jets is such as to prevent track formation in the resin, e.g. by crossing the jet directions;
- the sprayed water passes immediately through the screen and no bulk accumulation of water occurs on it during the sieving of resins.

The water shall finally pass into a dark-coloured vessel to check for the absence of resin particles in the water.

Figure 1 shows a schematic apparatus including a rotating sprinkling device for several sieves, and is given as an example meeting the requirements of this International Standard.

6.6 Filter funnel or filter crucible with adaptor, diameter 40 mm and mean pore size $20\text{ }\mu\text{m}$ to $40\text{ }\mu\text{m}$.

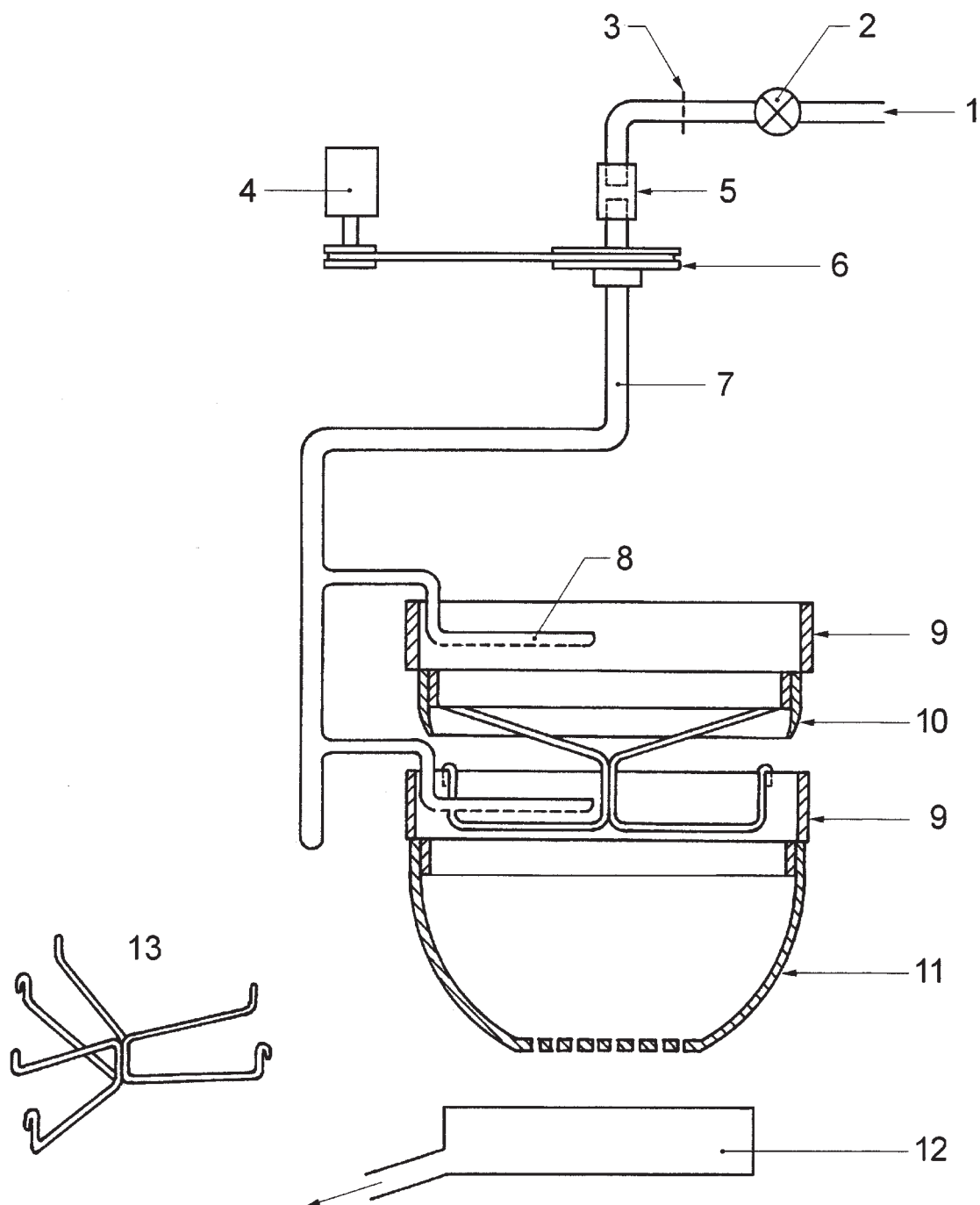
7 Procedure

7.1 General

This International Standard permits several variations, namely:

- a) It is possible
 - either to conduct a single test using two superimposed screens, provided that a sprinkler device is fitted to each sieve (see Figure 1) (recommended method),
 - or to carry out two tests on two separate test portions, using a different sieve each time.
- b) It is possible to dry the residue
 - either in a filter funnel or in a filter crucible (recommended method),
 - or directly on the sieve.

The latter method requires the sieves to be resistant to repeated heating and cooling (for example, they should be constructed from stainless steel).



Key

- | | |
|------------------------------------|--|
| 1 Water | 8 1-mm-thick slotted spray plate (ID 3 mm to 4 mm) |
| 2 Flow-regulating valve | 9 Sieve |
| 3 Filter | 10 Deflector |
| 4 Motor | 11 Bowl with perforated bottom |
| 5 Rotating joint | 12 Dark-coloured dish |
| 6 Pulley controlling spray plates | 13 Stainless-steel sieve support |
| 7 \varnothing int. 3 mm to 10 mm | |

Figure 1 — Sketch of multiple-sieve spraying device

7.2 Sieving

If, at the end of the operation, it is desired to weigh the resin on the sieve, weigh the sieves to the nearest 0,1 g after drying them in the oven (6.3), at $110\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, for 1 h and cooling in a desiccator.

Place the sieve or sieves below the sprinkling device and arrange the bowl with a perforated bottom and the dark-coloured dish beneath them to take the water away to a sink. If several superimposed sieves are used, they shall be arranged from top to bottom according to decreasing mesh size.

Weigh, to the nearest 0,1 g, approximately 25 g (mass m) of the resin into a 400 ml beaker. Add a suitable quantity of wetting agent (5.1) followed by two portions, each of approximately 50 ml, of water, mixing carefully after each addition.

NOTE The quantity of wetting agent added depends on the type of resin being tested. For some resins composed of very fine particles, it may be necessary to add the wetting-agent solution progressively, mixing it in with a glass rod to obtain a paste, which is then diluted gradually before pouring it on the upper sieve, where the resin should then not form lumps or agglomerates.

Set the sprinkler system in operation, taking care that the meshes are well moistened. Introduce the prepared suspension, in one or several goes, into the upper sieve and carefully rinse the beaker several times, pouring the washings into the upper sieve each time.

Apply the fastest rate of water consistent with the avoidance of overflowing or splashing.

Complete the sieving by hand spraying until the drain-off liquid in the dark-coloured dish is free from particles.

Lift each sieve and, using water jets, transfer to the next lower sieve any particles that might have remained adhering to its underside.

7.3 Drying and weighing

7.3.1 Drying on a filter bed

Wash the filter funnel or filter crucible, dry at $110\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, and weigh to the nearest 0,1 g after cooling in a desiccator.

Transfer the residue on each sieve quantitatively, using water jets, to this funnel or crucible fitted on a vacuum filtering flask.

Dry the funnel or crucible (with the residue from one sieve) at $110\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ in the oven to constant mass. Weigh to the nearest 0,1 g after cooling in a desiccator.

Calculate the mass of residue by subtracting the mass of the funnel or crucible from the mass of the funnel or crucible plus residue.

7.3.2 Drying on the sieve

Place each sieve, with its residue, in the oven, maintained at $110\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, and dry to constant mass. Weigh to the nearest 0,1 g after cooling in a desiccator.

Calculate the mass of residue by subtracting the mass of the sieve determined as described in 7.2 from the mass of the sieve plus residue.

7.4 Number of determinations

Each determination shall be carried out in duplicate. Do not take the results into consideration unless the following conditions are fulfilled:

a) Residue (calculated as indicated in clause 8) not less than 5 %

The two measurements are acceptable if the difference between their absolute values is less than or equal to 2 %.

b) Residue (calculated as indicated in clause 8) less than 5 %

The two measurements are acceptable if the difference between their absolute values is less than or equal to 1 %.

If these conditions are not fulfilled, further duplicate determinations shall be carried out.

8 Expression of results

8.1 For the case when two superimposed sieves are used:

Let

m_1 and m_1' be the masses, in grams, of residue on the 0,250 mm mesh sieve in each of the determinations;

m_2 and m_2' be the masses, in grams, of residue on the 0,063 mm mesh sieve in each of the determinations.

Calculate the total residue on both sieves in each determination, M_T and M_T' :

$$M_T = m_1 + m_2 \quad \text{and} \quad M_T' = m_1' + m_2'$$

Calculate the arithmetic mean \bar{m}_1 of m_1 and m_1' and the arithmetic mean \bar{M}_T of M_T and M_T' .

8.2 If two sieves are used separately in separate runs, direct measurement is made of m_1 and m_1' and of M_T and M_T' , from which \bar{m}_1 and \bar{M}_T can be obtained directly.

8.3 Using the values of \bar{m}_1 and \bar{M}_T obtained, calculate the percentages of residue retained by each sieve from the following equations:

$$\text{Residue retained by 0,250 mm sieve (R 250)} = \frac{\bar{m}_1}{\bar{M}} \times 100$$

$$\text{Residue retained by 0,063 mm sieve (R 63)} = \frac{\bar{M}_T}{\bar{M}} \times 100$$

where \bar{M} is the average mass, in grams,

- either of the two test portions in the case where two sieves are used superimposed in the same test run;
- or of the two test portions (used for each sieve) where the two sieves are used in separate test runs.

8.4 Express the results as the values of R 250 and R 63, as follows:

a) For residues greater than or equal to 5 %

The result, expressed as a percentage, is rounded to the nearest whole number.

b) For residues less than 5 %

The result, expressed as a percentage, is rounded to the first decimal place.

9 Precision

Interlaboratory trials conducted on three resins in four laboratories have shown that the repeatability s_r (within the same laboratory) and the reproducibility s_R (between different laboratories) depend to a certain extent on the sieve mesh, as shown in Table 1.

Table 1 — Precision data

	Mesh size		
	> 63 μm	> 106 μm	> 250 μm
	Sieve retention (%)		
s_r	0,06	0,04	0,01
s_R	0,17	0,05	0,03

10 Test report

The test report shall include the following information:

- a) a reference to this International Standard;
- b) all details necessary for complete identification of the test resin;
- c) the mass, in grams, of sample used for the test;
- d) the percentage retained on each sieve;
- e) the date of the test.