

Adhesives — Examination and preparation of samples for testing

The European Standard EN 1067:2005 has the status of a
British Standard

ICS 83.180

National foreword

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Adhesives - Examination and preparation of samples for testing

Adhésifs - Examen et préparations des échantillons pour
essais

Klebstoffe - Untersuchung und Vorbereitung von Proben
zur Prüfung

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Foreword

This European Standard (EN 1067:2005) has been prepared by Technical Committee CEN/TC 193 “Adhesives”, the secretariat of which is held by AENOR.

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of an identical text or by endorsement, at the latest by June 2006, and conflicting national standards shall be withdrawn at the latest by June 2006.

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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 European Standard specifies both the procedure for preliminary examination of a single sample as received for testing, and the procedure for preparing a test sample by blending and reduction of a series of samples representative of a consignment or bulk of adhesives or related product.

It is intended for use in conjunction with EN ISO 15605.

2 Normative references

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

EN 923, *Adhesives – Terms and definitions*

EN ISO 15605, *Adhesives – Sampling (ISO 15605:2000)*

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

ISO 8213, *Chemical products for industrial use – Sampling techniques – Solid chemical products in the form of particles varying from powders to coarse lumps*

3 Terms and definitions

For the purposes of this European Standard, the definitions given in EN 923 apply.

4 Sample container

4.1 Condition of container

Any defects in the sample container or any visible leakage shall be recorded. If it is possible that the contents have been affected, the sample shall be rejected unless it is being taken specifically to determine the extent to which the adhesive has been affected.

4.2 Opening of container

Some adhesives and related products (such as primers) are prone to develop gas or vapour pressure during storage. Care, appropriate to the situation, shall be taken in opening the container, particularly if bulging of the lid or the bottom of the container is observed.

If such phenomena occur, they shall be noted in the report of preliminary examination (see Clause 12 f)).

All packing materials (for example polystyrene chips, saw-dust) and other debris from the outer surface of the container shall be removed, particularly around the closure. The container shall be opened with care so as not to disturb the contents.

5 Preliminary procedure for fluid products consisting of a single homogeneous liquid phase or two liquid phases ¹⁾

5.1 Visual examination

5.1.1 Ullage

Record the approximate ullage, i.e. the air-space above the contents of the container, expressed as a percentage of the total capacity of the container.

5.1.2 Surface skin

Record the presence of any surface skin and its type, i.e. whether continuous, whether hard or soft and whether thin or moderately or excessively thick. If any skin is present detach it as completely as possible from the sides of the container and remove it, if necessary by sieving. Record the ease of removal.

5.1.3 Consistency

Record whether the sample is thixotropic or whether gelling has taken place, taking care not to confuse gelling and thixotropy.

NOTE Both thixotropic and gelled adhesives have a jelly-like consistency, but whereas the consistency of the former is markedly reduced by stirring or shaking, the consistency of a gelled adhesive cannot be reduced in this way.

5.1.4 Separation of phases

Record any separation of the sample into layers, for example water and resinous matter.

5.1.5 Visible impurities

If there are any visible impurities, record their presence and remove them if possible.

5.1.6 Sediment

If there is any appreciable sediment, record its presence and appearance.

5.1.7 Clarity

Record the clarity and colour of the sample.

5.1.8 Odour

Record any odour detected.

NOTE Care should be taken with regard to any resultant health hazards. Before handling the product the precautions indicated on its safety data sheet should be referred to.

5.2 Mixing

Thoroughly stir the sample and incorporate any slight sediment.

¹⁾ These correspond to products of type A and B in EN ISO 15605.

6 Preliminary procedure for fluid products consisting of one or two liquid phases together with one or more solid phases ²⁾

6.1 Visual examination

6.1.1 Ullage

Record the approximate ullage, i.e. the air-space above the contents of the container, expressed as a percentage of the total capacity of the container.

6.1.2 Surface skin

Record the presence of any surface skin and its type, i.e. whether continuous, whether hard or soft, and its approximate thickness. If any skin is present, detach it as completely as possible from the sides of the container and remove it, if necessary by sieving. Record the ease of removal.

6.1.3 Consistency

Record whether the adhesive is thixotropic or whether gelling has taken place, taking care not to confuse gelling and thixotropy.

NOTE Both thixotropic and gelled adhesives have a jelly-like consistency, but whereas the consistency of the former is markedly reduced by stirring or shaking, the consistency of a gelled adhesive cannot be reduced in this way.

6.1.4 Separation of phases

Record any separation of the samples into phases.

6.1.5 Settling

Record the type of settling, for example soft, hard or hard-dry. If the settling is hard and appears dry and crumbly inside a lump when cut with a clean palette knife, describe it as 'hard-dry'.

6.1.6 Extraneous matter

Remove any extraneous matter in the adhesive as carefully as possible and record its presence.

6.1.7 Odour

Record any odour detected.

NOTE Care should be taken with regard to any resultant health hazards.

6.2 Mixing

6.2.1 Limitations

Samples which have gelled or show hard-dry settling (see 6.1.3 and 6.1.6) cannot be effectively made homogeneous and shall therefore not be used for testing purpose.

²⁾ These correspond to products of type C in EN ISO 15605.

6.2.2 General

During all the operations specified in 6.2.3 to 6.2.5 care shall be taken to ensure minimum loss of solvent.

To this end, all the operations shall be carried out as rapidly as practicable and consistent with satisfactory mixing.

6.2.3 Removal of skin

If the original sample contained skin, remove any remnants by straining the incorporated sample under its own weight, through a sieve conforming to ISO 565, of nominal aperture 125 µm unless otherwise specified.

6.2.4 No occurrence of hard settling

Mix the sample thoroughly, even if there is no perceptible settling.

NOTE If the sample is less than 250 g a palette knife is preferred as an alternative to the stouter stirrer normally used for larger samples.

Firmly replace the lid of the container and thoroughly shake the contents, inverting the container as this is being done. Repeat the alternate stirring and shaking until the contents are completely homogeneous. As an added precaution, it is recommended that the mixing be completed by pouring the contents into a clean container and back again several times. At all times during the sample preparation avoid, as far as possible, entrainment of air. The samples shall be free from air bubbles before use.

6.2.5 Occurrence of hard settling

The examination of a sample in which hard settling has occurred (but not hard-dry settling, see 6.2.1) shall be completed in accordance with the procedure detailed below.

Pour all the fluid medium into a clean container. Remove the settled pigment from the bottom of the container with a palette knife and mix thoroughly.

When a uniform consistency has been achieved, return the medium to the original container, a small portion at a time, carefully incorporating each addition before the next is made. Complete the reincorporation by pouring from one container to the other several times (see 6.2.4). Ensure that the sample is free from air bubbles before use.

7 Preliminary procedure for viscous products³⁾

Examine these products in general as for fluid products under Clause 6.

NOTE Where mixing appears to be necessary to ensure homogeneity, a small, heavy-duty mixer can be used.

8 Preliminary procedure for products in powder form⁴⁾

No special procedure is normally required for these products. Unusual features such as abnormal colour, the presence of large or hard lumps, or the presence of foreign matters shall be recorded.

3) These correspond to products of type D in EN ISO 15605.

4) These correspond to products of type E in EN ISO 15605.

9 Preliminary procedure for solid products not in powder form ⁵⁾

No special procedure is normally required for these products. Unusual features such as abnormal colour, the presence of foreign matter shall be recorded.

10 Blending and reduction of a series of samples

10.1 General

In cases where a series of samples have been taken from a homogeneous product, either test these separately, or combine them to produce a reduced sample as specified in 10.2 to 10.5.

10.2 Fluid products (of type A, B or C)

After thoroughly mixing each sample as specified in Clauses 5 and 6, pour or otherwise transfer the samples into a clean, dry container and thoroughly mix them by stirring or shaking. When the mixed sample appears to be homogeneous, take a reduced sample in accordance with EN ISO 15605. Place the reduced sample in one or more clean, dry containers allowing 5 % ullage, then close, label and if necessary seal the containers.

NOTE 1 Because anaerobic adhesives are adversely affected by absence of air, sample containers for these materials should be more than 50 % full.

NOTE 2 Moisture curing or sensitive adhesives could react during the sampling procedure or storage. After sampling, sample containers for moisture curing or sensitive adhesives should be covered with dry nitrogen or inert gas.

10.3 Viscous products (of type D)

It is not possible to specify any single, generally applicable procedure. Therefore treat each case on its merits, taking into account the mechanical aids available, the difficulty of mixing viscous materials, the possible loss of volatile constituents, (see 10.2, notes 1 and 2).

10.4 Products in powder form (of type E)

Empty the contents of the various sample containers into a clean, dry container of suitable size and mix thoroughly. Reduce the sample down to a suitable size (1 kg to 2 kg) by quartering either manually or by means of a rotary sample divider or a riffle sample divider in accordance with ISO 8213, then place the reduced sample in one or more clean, dry containers.

Close, label and, if necessary, seal the containers, (see 10.2, note 2).

10.5 Solid products not in powder form (type F)

It is not possible to specify any single, generally applicable procedure. Therefore treat each case on its merits following as close as possible the indications given in 10.4.

NOTE Samples of adhesives in film form should be rolled up, together with any protective film, and placed in a polyethylene bag that is then sealed and put in a sample container.

11 Labelling of sample containers

The label of the sample container shall include:

⁵⁾ These correspond to products of type F in EN ISO 15605.

- a) name of the manufacturer and a description of the product;
- b) date of manufacture;
- c) consignor;
- d) size and particulars of the consignment;
- e) place of sampling, the date of sampling, the name of the sampler;
- f) reference number or numbers of the batch, storage tank, drum, etc. from which the sample or samples have been drawn;
- g) date of blending, the name of the blender;
- h) reference to this European Standard, i.e. EN 1067.

NOTE For dangerous materials, the label should comply with EU Directives on classification and labelling of dangerous substances and preparations.

If the sample is despatched to another laboratory, a delivery note shall be sent with it, repeating the details given on the label and also, if necessary, a report of the preliminary examination (see Clause 12).

12 Report of preliminary examination

The report shall contain at least the following information:

- a) reference to this European Standard, i.e. EN 1067;
- b) description of the sample, as indicated on the label (see Clause 11);
- c) appearance, clarity, and other similar visual characteristics;
- d) description of the skin formation observed, and of the sieving procedure adopted (if any);
- e) description of the settling observed, and of the mixing and reincorporation method adopted if any (see 5.2 and 6.2);
- f) other preliminary observations, as indicated in Clauses 4 to 9.

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