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Adhesives — Sampling

Adhésifs — Échantillonnage



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Contents

	Page
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Safety	2
5 Types of adhesive and related product	3
6 Sampling equipment	3
7 Types of sampling situation	5
8 Methods of sampling	5
9 Labelling and sealing of samples	7
10 Sampling report	8
11 Storage and use of samples	8

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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 15605 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 11, *Products*.

Introduction

Correct sampling is a skilled operation and the various procedures should be carried out with great care by samplers having the required knowledge and experience. The general instructions in this International Standard are intended to supplement this knowledge and experience and are applicable to most situations; however, some products require special sampling precautions not specified in this International Standard and therefore special vigilance is needed on the part of samplers to take note of unusual characteristics exhibited by the products. It is also essential that samplers observe special precautions in accordance with manufacturers' instructions and safety regulations.

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Adhesives — Sampling

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all 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 international or national regulatory conditions.

1 Scope

This International Standard specifies methods for sampling adhesives and related products, in order to obtain uniform samples of convenient size which are adequately representative of the product being sampled. The sample or samples so obtained are suitable for examination and preparation prior to testing.

The methods are applicable to products which require the addition and mixing in of catalysts or hardeners prior to their application, by sampling each component separately. The methods are not applicable, however, to sampling from a stream of adhesive (in this case the samples can be prepared in accordance with ISO 8213).

This International Standard does not apply to animal glues.

2 Normative references

The following normative documents contain 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 editions of the normative documents 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 472:1999, *Plastics — Vocabulary*.

ISO 8213:1986, *Chemical products for industrial use — Sampling techniques — Solid chemical products in the form of particles varying from powders to coarse lumps*.

ISO 15528:2000, *Paints, varnishes and raw materials for paints and varnishes — Sampling*.

3 Terms and definitions

For the purposes of this International Standard, the terms and definitions given in ISO 472, and the following apply.

3.1

batch

quantity of a particular adhesive (or component of an adhesive system) manufactured at a single location over a defined period of time and under conditions which are presumed to be uniform

3.2

delivery

quantity consisting of one or more batches or parts of batches of a particular adhesive delivered at any one time and covered by a single delivery note or shipping document

3.3

sample

any quantity of adhesive taken from and representative of a batch or delivery and intended to provide information necessary for assessing the characteristics of that material

3.4

gross sample

sample as collected from a batch or delivery

NOTE A reduced sample can be obtained by a method which reduces the quantity of the gross sample without changing its composition or physical state.

3.5

laboratory sample

final sample, ready for examination or testing

3.6

reference sample

sample, prepared at the same time as and identical with the laboratory sample, accepted by the parties to a contract and which is held for use as a laboratory sample in the event of a dispute

3.7

storage sample

retained sample

sample, prepared at the same time as and identical with the laboratory sample, intended for possible future use as a laboratory sample

3.8

test sample

sample, prepared from the laboratory sample, from which the test portions are withdrawn or taken and used for examination or testing

NOTE The test sample can consist of two or more separate components of a multi-component adhesive system.

4 Safety

4.1 A number of hazards exist in the sampling of adhesives, particularly those containing volatile solvents. They include the flammability of such solvents, the danger of explosion (of a vapour and air mixture) and physiological effects. The precautions indicated on the safety data sheet shall be referred to before handling a product.

Many adhesives are formulated with flammable solvents and the following precautions shall be taken. Care shall be taken that all sampling equipment used for these materials is made of material that is not liable to induce sparking from static electricity. It is good practice to connect large containers to earth. All regulations regarding "controlled" or "flammable" areas in which the samples are being taken shall be strictly followed.

The danger of explosion requires the same precautions as outlined above.

Vapour from solvents can be harmful and precautions should be taken to avoid its inhalation. Contact with the skin, spillage on clothing, etc., should be avoided as far as possible during sampling. The correct treatment for any harmful material should be known beforehand and the appropriate antidote should be at hand. Some constituents can also be absorbed through the skin.

4.2 It is recommended that two persons be present when samples are being taken from large vessels such as storage tanks, road tanks or rail tanks.

4.3 Before sampling from rail tanks, it shall be ensured that no shunting operations are likely.

4.4 Except in the case of anaerobic adhesives, the containers shall be filled between 80 % and 90 % of their total capacity; this allows, on the one hand, for the high coefficient of expansion of certain liquid adhesives, and for the

need ultimately to mix the samples thoroughly to obtain representative test samples and avoids, on the other hand, too large an air space which can deleteriously affect some adhesives. Because anaerobic adhesives are adversely affected by the absence of air, sample containers for these materials shall be filled no more than 50 % full.

Moisture-reactive adhesives or hygroscopic adhesives can react during the sampling procedure or storage. After sampling, sample containers for moisture-curing adhesives or hygroscopic adhesives can be covered with dry nitrogen or another inert gas.

Light-sensitive adhesives can be kept in a lightproof container (amber glass provides only partial protection against the action of light). It is essential that they are exposed to light for the minimum time.

Samples of adhesives in film form shall be rolled up, together with their protective film, if any, and placed in a polyethylene bag which is then sealed and put in a sample container (see 6.5).

5 Types of adhesive and related product

The sampling procedures appropriate for use with adhesives and related products depend on the nature and the physical properties of the products. It is convenient to distinguish between the following types of product:

- Type A: Fluid products consisting of a single homogeneous liquid phase.
- Type B: Fluid products consisting of two liquid phases (for example, emulsions).
- Type C: Fluid products consisting of one or two liquid phases together with one or more solid phases (for example, water-based dispersions).
- Type D: Viscous products which usually consist of one or more solid phases with a small amount of a liquid phase (for example, mastics).
- Type E: Products in powder form.
- Type F: Solid products not in powder form (for example, blocks, films, tapes, sheets, granules, etc.).

Some adhesives of Types A to F undergo rapid changes in their properties when exposed to external influences. Examples of such adhesives are:

- light-sensitive adhesives;
- hygroscopic adhesives;
- moisture-reactive adhesives;
- anaerobic adhesives.

Appropriate handling precautions shall therefore be taken (see 8.2.5).

6 Sampling equipment

6.1 General

Sampling equipment is required for two separate operations:

- a) mixing the product to make it as homogeneous as possible;
- b) taking a truly representative sample.

The sampling tools in general use for sampling raw materials, which are described in ISO 15528:2000 and ISO 8213, can also be used for sampling finished products.

6.2 Materials and design

All sampling equipment shall be made of material not subject to deterioration, unaffected by the product being sampled and incapable of contaminating the sample. The design of the equipment shall take into account convenience in use and ease of cleaning (for example, any grooves, acute internal angles, or areas which are inaccessible or difficult to inspect, shall be avoided).

6.3 Apparatus for mixing

Required are broad-bladed stirrers, either mechanical or manual, of suitable length to reach the bottom of the container being sampled.

In certain circumstances, when mixing a product of low flash point or where an explosive hazard exists, the only metal permitted is bronze.

Normal precautionary measures against static electricity shall be taken.

6.4 Apparatus for taking samples

Drawings of designs are given in ISO 15528 or in ISO 8213.

6.4.1 Sampling tubes, of glass or metal.

6.4.2 Small dip cans.

6.4.3 Weighted sampling cans, with valve closures, for taking samples at all levels.

6.4.4 Scoops or spatulas.

6.5 Sample containers

New containers with large apertures shall be used. These shall be of one of the following types:

- a) metal containers of which the interior is not coated with varnish and which are fitted with tight metal closures;
- b) glass containers which can be tightly closed with closures which are not affected by the sample (amber glass provides only partial protection against the action of light);
- c) polyethylene containers which can be tightly closed.

6.6 Labels

Labels or other means of marking or identifying shall be provided.

For dangerous materials, the labels shall conform to relevant regulations.

6.7 Cleaning of sampling equipment

Strict cleanliness shall always be observed. All sampling apparatus shall be dry and free from residue, so as not to contaminate the sample. After each use, the apparatus shall be thoroughly cleaned with the aid, if necessary, of a brush or clean cotton rag, and shall then be rinsed with a suitable solvent in accordance with national regulations.

7 Types of sampling situation

There are two main types of sampling situation, corresponding to the following stages:

- a) When the manufacturing process has been completed but the product is still in the manufacturing vessels, or when it is being transferred into the delivery containers (cans, drums, barrels, etc.).
- b) When sampling from the delivery containers, which can also be bulk transport containers. Such sampling shall take place at the manufacturer's premises or, by special agreement between purchaser and vendor, sampling is permitted at the purchaser's premises, in which case the product shall not have been processed after receipt in any way.

8 Methods of sampling

8.1 Situation 7 a)

8.1.1 When the sampling situation is as described in 7 a), use one of the procedures given in 8.1.2 to 8.1.4.

8.1.2 For fluid products (Types A, B and C): When the sample is to be taken directly from the final manufacturing vessel, the product shall first be thoroughly stirred and the sample taken by means of a weighted sampling can (6.4.3) from different levels in the vessel. When the sample is to be taken in the course of transferring the product to the delivery containers, it shall be made up of small sub-samples taken at regular intervals during the filling process. In the former case, the sample shall be strained before filling the sample container (6.5), using the same procedure as normally used when filling the delivery containers.

NOTE An additional safeguard is to check any relevant parameter, e.g. the relative density or the viscosity, of samples taken from different levels and, if the values obtained are not within agreed tolerances, to continue stirring until the agreed tolerances are met.

If experience has shown that the product is easily homogenized, the sample need only to be taken from one level in the vessel.

8.1.3 For viscous or powdery products (Types D and E): The product shall be examined in the final manufacturing vessel and, if uniform in appearance, the sample shall be taken from various parts of the bulk using a sampling tube (6.4.1) or scoop (see 6.4.4). Alternatively, a number of small samples can be taken at regular intervals during filling of the delivery containers.

8.1.4 For solid products not in powder form (Type F): The product shall be examined in its final packaged form: roll, reel, sheet or block.

NOTE If the product appears to be homogeneous, the packs may be treated as though they were small containers, and the number of samples determined in accordance with Table 1.

8.2 Situation 7 b)

8.2.1 When the sampling situation is as described in 7 b), one of the procedures given in 8.2.2 to 8.2.6 shall be used.

8.2.2 For fluid products (Types A, B and C): If the product is supplied in a bulk transport container, the procedure in 8.1.2 shall be applied for sampling from the final manufacturing vessel. However, in the absence of a circulating pump or other means of agitation, it is usually necessary to take an approximately equal sized sample at different levels in order to obtain a representative sample of the whole.

NOTE 1 An additional safeguard is to check any relevant parameter, e.g. the relative density or the viscosity, of samples taken from different levels and, if the values obtained are not within agreed tolerances, to continue stirring until the agreed tolerances are met.

If the product is supplied in a delivery of containers, the total number of containers shall be noted and then a selection made at random of the containers for sampling. It is recommended that approximately $(n/2)^{1/2}$ containers be

sampled, where n is the total number of containers in the delivery (as a guide, see Table 1). Only sound, unopened containers shall be chosen for sampling, unless it is desired specifically to examine damaged or opened containers.

Table 1 — Minimum number of containers to be taken from a delivery

Total number of containers in the delivery	Minimum number of containers to be taken
1 to 2	All
3 to 8	2
9 to 25	3
26 to 100	5
101 to 500	8
501 to 1 000	13

The following procedure shall then be followed:

- a) Open each selected container in turn and examine for the presence of surface skin, separation of the contents (for example, water or solvent) and the presence of foreign matter.
- b) After removing any surface skin present, insert a stirrer and note the extent and type of any setting, i.e. soft, hard or hard-dry. Note also the presence or absence of any gel, taking care not to confuse gelling and thixotropy.

NOTE 1 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.

- c) Redisperse any settled filler with the stirrer and note the ease of redispersion. Then replace the closure securely, invert the container and leave it inverted while the other containers are examined. Finally, shake and roll the containers to ensure complete redispersion and reincorporation of the filler, preferably using mechanical means.
- d) Re-open the containers and examine for homogeneity. If necessary, continue successively stirring, shaking and rolling until the contents are homogeneous, examining the bottom end of the stirrer for undispersed pigment from time to time during the stirring. Note the time required to attain homogeneity.
- e) When the contents of the containers have become homogeneous, sample them, using a small dip can (6.4.2) to transfer the product to the sample container and leaving an ullage (air space) of about 5 % in the sample container. Close the container sampled and the sample container, clean the outside of the sample container and immediately label it in accordance with 9.1.

8.2.3 For viscous products (Type D): These products are normally supplied in wide-aperture containers. Examine the condition of all the containers and select at random for sampling a number in accordance with Table 1. Open each of the selected containers, remove any protective covering and examine the contents for homogeneity or separation of phases (for example, oil, solvent or water). If the product appears homogeneous (or, if not, when it has been made so by stirring), take a sample from the whole depth of the product in each selected container, using a metal sampling tube (6.4.1) to transfer it to the sample container and leaving an ullage (air space) of about 5 % in the sample container. Close the sample container and label it in accordance with 9.1.

8.2.4 For powdery products (Type E): These products are normally supplied in sacks, paper or plastic bags, drums, barrels or plywood containers. Examine the condition of all the packages and select at random for sampling a number in accordance with Table 1. Open each of the selected packages, take, from various parts of each package, small portions of the product by means of a sampling instrument, and close the packages. Place these portions in sample containers, close the containers, mix the contents thoroughly by shaking and label in accordance with 9.1.

8.2.5 For solid products not in powder form (Type F): It is essential that any surface film or contamination be removed before samples are taken. The sampling procedure is dependent on the form in which the material is received.

Treat uniform blocks of material as though they were small containers and determine the number of samples in accordance with Table 1.

For solids in irregular form, choose pieces at random. Reduce these to a convenient smaller size to make the gross sample.

Film and tape are usually packaged in the form of reels. Treat the reels as though they were small containers and determine the number of samples in accordance with Table 1.

NOTE 1 There is no entirely satisfactory method of sampling a reel because the outer layers of the reel may have been more affected by the environment in which it has been stored than the inner layers. A representative sample can only be obtained by un-reeling all the material and sampling the film or tape at suitable points along its length.

NOTE 2 A sample taken from the beginning of the reel can, however, represent the worst condition of the material when assessed for performance.

8.2.6 The procedures and precautions required for sampling products which undergo rapid changes in their properties when exposed to external influences are dependent on these properties (see 4.4).

Sample containers containing anaerobic products shall be filled no more than 50 % full.

Moisture-reactive adhesives and hygroscopic adhesives shall be sampled in a dry atmosphere.

Light-sensitive adhesives shall not be sampled in direct sunlight.

9 Labelling and sealing of samples

9.1 Labelling

The containers shall be labelled as soon as the samples are taken; the labels shall bear the designation and all other necessary information to enable the samples to be identified without dispute. The labels and marking ink used shall be capable of withstanding the effects of moisture and any solvents contained in the sample. The label shall not be attached to the stopper, but to the neck or body of the container.

The label shall contain the following particulars, or refer uniquely to a separate reference sheet where these data are recorded:

- a) the name of the manufacturer and a description of the product;
- b) the quality and particulars of the delivery (e.g. the appearance of the packaging);
- c) the reference number of the batch, storage tank, barrel, etc.;
- d) the dates of manufacture and of sampling;
- e) the total number of samples taken from the delivery;
- f) the designation and reference number of the sample (where the samples are drawn from a certain number of selected containers, the latter shall be numbered);
- g) the place of sampling;
- h) the name of the sampler;
- i) the expiry date of the product.

For dangerous materials, the labels shall comply with relevant regulations, if requested.

For temperature-sensitive products, the label shall also indicate the recommended storage temperature.

9.2 Sealing

After a sample container has been closed and labelled, it shall be sealed.

10 Sampling report

The sampling report shall include all the information necessary to identify the sample as well as all details which may be useful to those handling it. This report shall include a reference to this International Standard and information on any abnormalities such as:

- a) container defects;
- b) any visible foreign matter;
- c) abnormal odour;
- d) abnormal colour;
- e) marking errors;
- f) non-homogeneity, including the presence of any surface skin before sampling and any straining procedure used before filling the sample container;
- g) any difficulty experienced in combining sub-samples.

11 Storage and use of samples

Samples shall be examined as soon as possible after they have been taken. Some products, in particular those containing water, can deteriorate after they have been stored under extreme temperature conditions. The samples shall therefore be stored in accordance with the adhesive manufacturer's instructions.

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