

INTERNATIONAL STANDARD

ISO
1512

Second edition
1991-12-01

Paints and varnishes — Sampling of products in liquid or paste form

*Peintures et vernis — Échantillonnage des produits sous forme liquide
ou en pâte*

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Reference number
ISO 1512:1991(E)

Foreword

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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.

International Standard ISO 1512 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Sub-Committee SC 9, *General test methods for paints and varnishes*.

This second edition cancels and replaces the first edition (ISO 1512:1974). This edition of ISO 1512 differs from the previous edition by specifically excluding the sampling of coating powders and by including an abbreviated procedure (see 8.2.1.2) when the containers to be sampled are from a single batch. The safety precautions to be considered have also been revised.

Annex A of this International Standard is for information only.

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International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Introduction

This International Standard is one of a series of standards dealing with the sampling and testing of paints, varnishes and related products in liquid or paste form. It does not deal with procedures for the sampling of raw materials used in the manufacture of paints and varnishes: these are described in ISO 842 (see annex A). Also it does not describe the sampling of paints in powder form (coating powders).

Correct sampling is a skilled operation and the various procedures require to be carried out with great care by operators 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 may require special sampling precautions that are not given in this International Standard and therefore special vigilance will be necessary on the part of operators to take note of any unusual characteristics exhibited by those products. It is also essential that operators adhere to any special precautions in accordance with product specifications and national safety regulations.

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Paints and varnishes — Sampling of products in liquid or paste form

1 Scope

This International Standard describes methods for sampling paints, varnishes and related products in liquid or paste form that will provide uniform samples that are of convenient size and adequately represent the product to be tested. The sample or samples so obtained are suitable for examination and preparation in accordance with ISO 1513 prior to testing.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1513:1980, *Paints and varnishes — Examination and preparation of samples for testing*.

3 Definition

For the purposes of this International Standard, the following definition applies.

3.1 batch: The quantity of liquid paint or paste, produced in the final mixing operation after the completion of all production processes, in one large vessel from which smaller containers may be filled for distribution and marketing.

4 Health and safety precautions

4.1 This standard calls for the use of substances and/or procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and in no way absolves the user from statutory obligations relating to health and safety at any stage.

4.2 Particular attention is drawn to the following hazards of sampling paints, varnishes and related products although this list may not necessarily be exhaustive. These hazards include, in addition to explosions and flammability, toxicity by inhalation and/or absorption by the skin. Operators shall always be informed of known dangers and shall be advised to handle such products with due care, using protective equipment where necessary. Attention is drawn to national regulations and requirements concerning safety and hygiene.

5 Types of paints, varnishes and related products

The sampling procedures appropriate for use with paints, varnishes and related products depend on the nature and the physical properties of the products. The following types of product may conveniently be distinguished.

Type A: Fluid products consisting of a single homogeneous liquid phase.

Type B: Fluid products consisting of two liquid phases, such as emulsions.

Type C: Fluid products consisting of one or two liquid phases together with one or more solid phases. Such products are commonly called "paints" but may include enamels, lacquers, some varnishes, etc.

Types D: Viscous products consisting of one or more solid phases with small amounts of a liquid

phase (for example, putties, mastics, luting, cements, and pigment pastes in oil or other binders). This type also includes very viscous resinous materials.

6 Sampling equipment

6.1 General

The sampling tools, which are in general use for sampling raw materials and are described in ISO 842, may also be used for sampling finished products.

In sampling, equipment is required for two separate operations:

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

6.2 Materials and design

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

6.3 Apparatus for mixing

6.3.1 Broad-bladed stirrers, of suitable length, capable of reaching the bottom of the container being sampled. Mechanical stirrers may be used.

Special care shall be taken when a metal stirrer is to be used for stirring a product of low flashpoint or in circumstances where an explosion hazard exists (see 4.2).

NOTE 1 Stirrers constructed from bronze or aluminium may be appropriate.

6.3.2 Paint shakers, suitable for small containers.

6.4 Apparatus for taking samples

6.4.1 Sampling tubes, inert to the product to be sampled; examples are shown in figures 1 to 3 and are described in 6.4.1.1 to 6.4.1.3.

6.4.1.1 The sampling tube shown as an example in figure 1 consists of two concentric metal tubes closely fitted into each other throughout their entire length, so that one tube can be rotated within the

other. A longitudinal opening or series of openings of about one-third of the circumference is cut in both tubes. In one position the tube is open and admits the liquid; by turning the inner tube it becomes a sealed container.

The inner tube is 20 mm to 40 mm in diameter. It may be undivided in its length, in which case the two tubes are provided with V-shaped ports at their lower ends, so placed that liquid contained in the instrument can be drained through them when the longitudinal openings are open.

Alternatively, the inner tube may be divided transversely into from three to ten compartments, in which case the bottom V-shaped ports are omitted. Such an arrangement enables separate samples of liquid to be withdrawn from different depths in the container.

The tube should be of sufficient length to reach the bottom of the container. It is inserted closed, then opened to admit the liquid and finally closed and withdrawn.

6.4.1.2 The sampling tube shown as an example in figure 2 may be used where the liquid to be sampled is known to be homogeneous in character. It consists of a metal or thick-walled glass tube which may vary from 20 mm to 40 mm in diameter and from 400 mm to 800 mm in length. The upper and lower ends are conical and narrow down to about 5 mm to 10 mm. At the upper end there are two rings to assist handling.

To take an individual sample, the tube is first closed at the top with the thumb or a stopper, and lowered until the desired depth is reached. It is opened for a short time to admit the liquid and then closed and withdrawn.

6.4.1.3 The sampling tube shown as an example in figure 3 consists of a metal tube with a valve at the base connected by a central rod to a screw handle at the top. When the handle is screwed down the valve is kept closed. It differs from the tubes previously described in that it is introduced into the liquid with the valve open, allowing the liquid to enter as the tube dips below the surface while the displaced air passes through an air-vent at the top of the tube. When the base of the tube touches the bottom of the container, the valve automatically closes. The handle is then screwed tight so as to keep the valve shut, and the tube containing the sample is withdrawn. The outside of the tube is wiped clean unless a cleaning device is used. Sampling tubes of various lengths are used, one 2 m long, made of aluminium, being convenient for sampling road tanks. This instrument, illustrated in figure 3, is not suitable for use where sediments have accumulated.

6.4.2 Sampling bottle or can.

This instrument consists of a weighted bottle or metal container, with removable stopper or cap to which is attached a suitable chain, pole or cord. This device is lowered to the desired depth where the stopper is removed and the container allowed to fill. An example of a sampling can is shown in figure 4.

This instrument is suitable for sampling vessels and tanks.

6.4.3 Zone sampler, with valve closures, for taking samples at any level.

The instrument shown as an example in figure 5 is suitable for withdrawing bottom samples or zone samples at any level from tanks of liquid. To withdraw a bottom sample, the apparatus is attached to a cord or chain and lowered empty to the bottom of the tank where the central spindle valve automatically opens and the container fills from the bottom. On withdrawal the valve automatically closes again.

To withdraw a sample at any level, the apparatus is lowered empty to the required level and then by means of an additional cord, previously attached to the top of the central valve spindle, the valve is opened and the container filled. The valve is then allowed to close and the container withdrawn.

6.4.4 Sampling scoops.

This instrument, an example of which is shown in figure 6, consists of a D-shaped metal trough divided into compartments along its length, and a shutter that moves vertically along the entire length to open and close compartments. It may be from 25 mm to 50 mm in diameter.

The instrument is inserted closed and the shutter pulled out to admit the liquid; the scoop is then closed and withdrawn.

6.5 Sample containers

New containers of suitable size and with large apertures shall be used. These shall be either:

- a) metal containers, fitted with tight metal closures and free from soldering flux but not coated internally with paint or varnish (see note 2); or
- b) glass containers, fitted with tight closures that are not affected by the sample (see note 3).

NOTES

2 Containers coated internally with varnish are, however, suitable for many water-based products.

3 Dark glass provides a partial protection against the action of light and the contents can be further shielded, if necessary, by external opaque covering or packaging.

6.6 Labels

Labels or other means of legibly marking or identifying samples shall be provided (see clause 9).

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 before drying.

7 Stages for sample collection

Sampling may be carried out at either or both stages of manufacture as follows:

- a) Stage I: Sampling when manufacture of the product has been completed but the product is still in the final manufacturing vessels, or when the final product is being transferred into the delivery containers (cans, drums, barrels, etc.).
- b) Stage II: Sampling from the delivery containers, including bulk transport containers.

8 Method of sampling

Avoid, whenever possible, sampling from products that are foaming or have entrained air.

If the samples are drawn from a number of selected containers, number each container for identification purposes.

8.1 Sampling at stage I [see clause 7, item a)]

8.1.1 For fluid products (types A, B and C)

If the sample is to be taken directly from the final manufacturing vessel, thoroughly stir the product (except if a clear varnish or similar product) (see note 4). Then take samples by means of a zone sampler (6.4.3) from near the top, middle and bottom of the vessel. Do not stir clear varnishes or similar products before sampling from settling tanks.

If a sample is to be taken in the course of transferring the product into the delivery containers, take samples at least at the beginning, middle and end of the filling process.

During the filling process, strain the sample before filling the sample container (6.5), using the same procedure as normally used when filling the delivery

containers unless the material is known to contain insoluble flattening agent(s).

NOTE 4 An additional safeguard is to check the densities of samples taken from the different levels and, if these are not within specified or agreed tolerances, to continue stirring until these tolerances are met.

8.1.2 For viscous products (type D)

Inspect the product in the final manufacturing vessel and, if uniform in appearance, take the sample from near the top, middle and bottom of the vessel using a sampling tube (6.4.1) or scoop (6.4.4). Alternatively, take a number of samples at the beginning, middle and end of filling the delivery containers.

8.2 Sampling at stage II [see clause 7, item b)]

8.2.1 For fluid products (types A, B and C)

8.2.1.1 Containers of volume greater than 5 m³

If the product is supplied in a container of volume greater than 5 m³, apply the same procedure as described in 8.1.1 for sampling from the final manufacturing vessel. However, in the absence of a circulating pump or other means of agitation, take samples of approximately equal size from near the top, middle and bottom of the vessel in order to obtain a representative sample of the whole.

Determine the viscosity, density and colour of the samples and, if the values obtained are within the repeatability limits for the method used, consider each sample to be representative of the product. If agreement is not observed, investigate the cause and take remedial action, e.g. mixing of the material to be sampled.

8.2.1.2 Containers of volume less than 5 m³ — Reduced sampling procedure

When a series of containers, each of less than 5 m³ capacity, can be identified as having originated from the same batch of manufacture, select at random 1 % of the containers. If a fraction results, take the next larger whole number, but select not less than two containers and not more than five containers. Examine the product in each container as described in 8.2.1.3 and for each determine the colour, viscosity and density. If the values obtained are within the repeatability limits for the method used, consider each sample to be representative of the batch. Carry out the full sampling procedure (8.2.1.3) if conformity is not obtained.

8.2.1.3 Containers of volume less than 5 m³ — Full sampling procedure

8.2.1.3.1 If the product is supplied in a consignment of containers, note the total number of containers and then select at random the containers for sampling. Sample from not less than $\sqrt{N/2}$ containers, where N is the total number of containers in the consignment (see table 1). Choose only sound, unopened containers for sampling, unless it is desired specifically to examine damaged or opened containers.

Table 1 — Selection of containers from a consignment

Number of containers in consignment, N	Number of containers to be sampled, n
2 to 8	2
9 to 18	3
19 to 32	4
33 to 50	5
51 to 72	6
73 to 98	7
99 to 128	8
129 to 162	9
163 to 200	10

and thereafter at the rate $n = \sqrt{N/2}$

8.2.1.3.2 Carry out the following procedure (see ISO 1513).

- Open each selected container in turn and examine for the presence of surface skin, separation of the contents (for example, water or solvent) or the presence of foreign matter.
- After completing the removal of any surface skin, if present, insert a suitably sized spatula or paddle into the container and note the extent and type of settling, i.e. soft, hard or hard-dry. Note also the presence or absence of a gel but taking care not to confuse gelling and thixotropy¹⁾.
- Take appropriate action to deal with hard settlement, which is usually a cause of product consignment rejection. With due caution, redisperse soft settlement using an appropriate mechanical stirrer and note the ease of redispersion. Replace the closure securely and, if practicable, invert the container and leave inverted while the other containers are examined. Finally, if practicable, shake and roll the containers, preferably using mechanical means, to effect complete redispersion and reincorporation of the pigment.

1) Both thixotropic and gelled paints and varnishes have a jelly-like consistency, but whereas the consistency of the former is markedly reduced by stirring or shaking, the consistency of a gelled paint or varnish cannot be reduced in this way.

- d) Re-open each selected container and examine for uniformity. Continue with successive 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 uniformity.
 - e) When the contents of the containers have become homogeneous, re-open one of the containers. Use a small sampling bottle or can (6.4.2) to transfer portions of the product to the sample container until filled so that there is an ullage (air-space) of about 5 %. Close the container that has been sampled and the sample container. Clean the sample container on the outside and immediately label in accordance with 9.1. Then sample in the same way from the remaining containers.
- b) the quantity and particulars of the consignment;
 - 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 consignment and, if applicable, the sampling procedure adopted [full or reduced (see 8.2.1)];
 - f) the designation and reference number of the sample;
 - g) the consignor;
 - h) the place of sampling;
 - i) the name of the operator.

8.2.2 For viscous products (type D)²⁾

Examine the condition of all the containers and select at random an appropriate number for sampling in accordance with table 1. Open in turn each of the selected containers, remove any protective covering and inspect the contents for homogeneity or separation of phases (for example phases of oil, solvent or water). If the product appears homogeneous or is made so by stirring, take a single sample through the whole depth of the product in each selected container, using a suitable metal sampling tube (6.4.1) or other suitable equipment. Place the sample in a sample container of suitable size to leave an ullage (air-space) of about 5 %. Close the sample container and immediately label it in accordance with 9.1.

9 Labelling and sealing of samples

9.1 Labelling

Each sample container shall be labelled as soon as the sample has been taken. The label shall bear all the necessary information and be sufficiently legible to enable the sample to be identified without dispute. The label and marking ink used shall be capable of withstanding moisture and any solvents contained in the sample. The label shall be attached to the neck or body of the container, and not to the stopper.

It is recommended that the following information, at least, should be given on the label:

- a) the name of the manufacturer and a description of the product;

9.2 Sealing

After each sample container has been closed and labelled, it shall be sealed in such a way that the contents and label cannot be removed without breaking the seal.

10 Sampling report

The sampling report shall include all the information necessary to identify the sample as well as details which may be useful to those handling it. This report shall include a reference to this International Standard (ISO 1512) and information on any abnormalities such as:

- container defects;
- any visible foreign matter;
- abnormal odour;
- abnormal colour;
- marking errors;
- non-homogeneity, including presence of surface skin (if any) before sampling and any straining procedure used before filling the sample container;
- difficulty in redispersion of solids, etc.;
- any deviation from the sampling procedure described, including any special arrangements agreed between the purchaser and the supplier.

2) These products are normally supplied in containers with a wide aperture.

11 Storage of samples

It is recommended that samples be examined as soon as possible after they have been taken. Some

products, particularly those containing water, may deteriorate after they have been stored under extreme temperature conditions. Therefore, the samples shall be stored according to the instructions of the product manufacturer.

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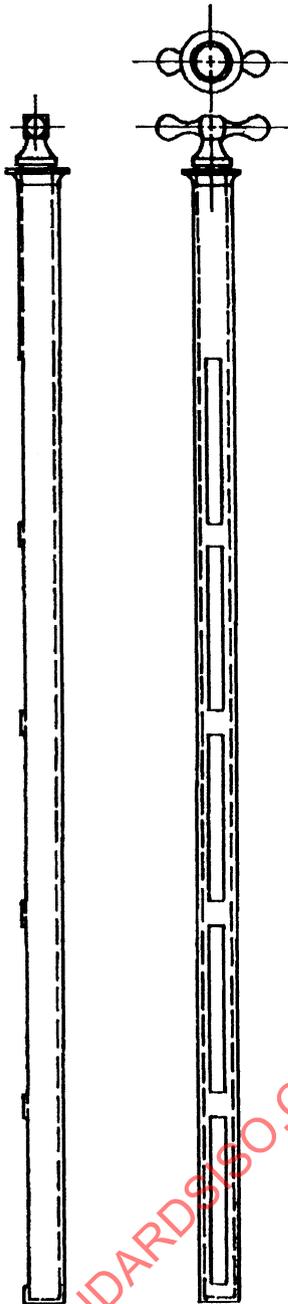


Figure 1 — Sampling tube consisting of two concentric tubes



Figure 2 — Sampling tube with single tube

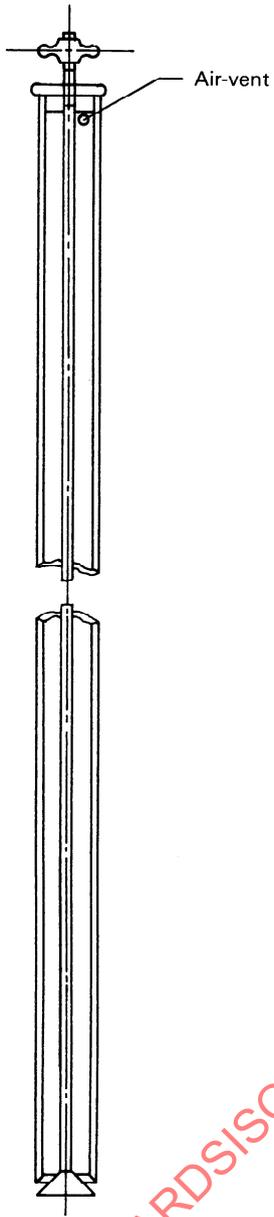


Figure 3 — Valve sampling tube

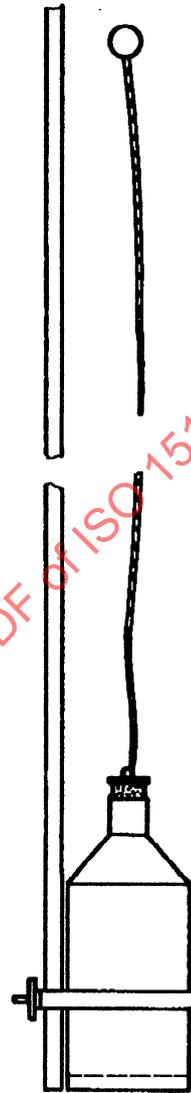


Figure 4 — Sampling can