International Standard 607

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Surface active agents and detergents — Methods of sample division

Agents de surface et détergents — Méthodes de division d'un échantillon

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 607 was developed by Technical Committee ISO/TC 91, *Surface active agents*, and was circulated to the member bodies in August 1978.

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This International Standard cancels and replaces ISO Recommendation R 607-1967, of which it constitutes a technical revision.

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Surface active agents and detergents — Methods of sample division

1 Scope and field of application

This International Standard specifies methods for obtaining a reduced sample of surface active agent or detergent suitable for use with single or mixed products, in the form of powders, pastes or liquids.

The sample reduction process may be required for the following reasons :

a) the preparation of a final sample or a laboratory sample of mass greater than 250 g from a blended bulk sample of mass greater than 500 g;

b) the preparation of several equivalent laboratory samples and/or reference samples and/or storage samples, each of mass greater than 250 g, from a final sample;

c) the preparation of a test sample from a laboratory sample.

2 Reference

ISO 6206, *Chemical products for industrial use — Sampling — Vocabulary.*

3 Definitions[1)]

3.1 bulk sample : A collected set of samples which do not maintain their individual identities.

3.2 blended bulk sample : A collected set of samples blended together to obtain a uniform bulk sample.

3.3 reduced sample : A sample that has been obtained by reducing the quantity of another sample without change of composition.

NOTE — It may also be necessary to reduce the particle size in the course of reducing the quantity.

3.4 final sample : A sample obtained or prepared under the sampling plan for possible subdivision into identical portions for testing, reference or storage.

3.5 laboratory sample : A sample as prepared for sending to the laboratory and intended for inspection or testing.

3.6 reference sample : A sample prepared at the same time as, and equivalent to, the laboratory sample, which is acceptable to the parties concerned, and retained for use as a laboratory sample if a disagreement occurs.

3.7 storage sample : A sample prepared at the same time as, and equivalent to the laboratory sample, and intended for possible future use as a laboratory sample.

3.8 test sample : A sample prepared from the laboratory sample and from which test portions will be taken.

4 Principle

Reduction of a bulk sample by a mechanical process until a reduced sample is obtained.

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1) Definitions based on ISO 6206.

5 Procedure

5.1 Products in powder form

The procedure specified is applicable to powders, including spray dried powders, and particularly, those that contain additives that have been introduced after the drying process.

NOTES

1 In the case of powders containing additives introduced after drying, the physical mixture obtained has a tendency to separate.

2 In the case of washing powders, it is recommended that sampling be carried out under a ventilated hood; if necessary, a mask should be worn.

5.1.1 Apparatus

Any satisfactory apparatus may be used, but the following types are recommended :

5.1.1.1 Conical divider (see figures 1 and 2)

The apparatus shall be constructed in such a way that the two portions of the sample obtained from each dividing operation are quantitatively similar to each other and qualitatively representative of the original sample.

An apparatus which satisfies these conditions is the conical divider (see figure 1) which consists essentially of a hopper (A) from which the sample to be divided runs over the surface of a cone (B) whose apex is situated directly beneath the centre of the lower opening in the hopper. The material running down the cone is diverted to series of receptacles arranged round the circumference of an inverted hopper (C) at the base of the cone (B). Alternate receptacles are connected to one or other of the two outlets at the bottom of the inverted hopper in order to provide two equivalent reduced samples.

5.1.1.2 Rotary sample divider (see figure 3)

A suitable apparatus[1)] comprises a hopper from which the sample falls in a thin stream onto a rotating platform carrying six, or more, identical receivers arranged symmetrically about the vertical axis of rotation so as to collect all of the falling sample. The frequency of rotation exceeds 40 min^{-1}.

NOTE — Take care that the frequency of rotation of the platform is not too high if fine particles are present.

5.1.2 Preparation of the reduced sample using the conical divider (5.1.1.1)

5.1.2.1 Preparation of the final sample

Place a receiver under each of the outlets of the conical divider. Fill the hopper and open the valve fully so as to allow the contents of the hopper to run over the cone, thus dividing the bulk sample into two portions one of which is deposited in each receiver.

Retain one of these portions and discard the other.

Pass a fresh quantity of bulk sample through the conical divider and repeat the operation until all the bulk sample has been divided.

Clean the apparatus and again pass the retained portions, corresponding to half the bulk sample, through the apparatus as described above and repeat this operation until a reduced sample of the required mass has been obtained.

5.1.2.2 Preparation of several equivalent samples

If more than one sample is required, prepare sufficient reduced sample to obtain $2n$ equivalent samples where $2n$ equals or exceeds the number of samples required.

Subsequently, divide the reduced sample into $2n$ equal portions by means of the conical divider. Immediately place the whole of each portion in an airtight bottle or flask.

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5.1.2.3 Preparation of test samples

If test samples are required from the laboratory samples, treat the latter as specified in 5.1.2.1 and 5.1.2.2.

The minimum mass of the test samples shall in no case be less than 10 g, otherwise there is a risk that the test samples may not be truly representative of the bulk sample and, hence, will be unsuitable for test purposes.

5.1.3 Preparation of the reduced sample using the rotary divider (5.1.1.2)

5.1.3.1 Preparation of the final sample

Place on the platform the complete set of receivers, one or more of which shall be clean and empty and shall bear some distinguishing mark. Fill the hopper. Set the rotating platform in motion and allow the bulk sample to fall into the receivers at a uniform rate for at least 2 min. Retain the portion collected in the marked receiver(s) and discard the remainder.

If the mass of the bulk sample is greater than the capacity of the divider, carry out the division in a series of operations. At the end of each operation, combine the sample portion(s) from the marked receiver(s) in a larger container, and continue using the same marked receiver(s) for subsequent divisions until all the sample has been divided.

1) Examples of commercially available apparatus are :

— Pascal Rotary cascade sample divider;

— Retsch type PT sample divider.

Transfer the material collected from the marked receiver(s) to the hopper, and repeat this operation until a sample of the required mass has been obtained.

5.1.3.2 Preparation of several equivalent samples

If more than one sample is required, prepare sufficient reduced sample to obtain n equivalent samples, where n equals or exceeds the number of samples required.

Select an appropriate number, n, of marked receivers, and pass all of the reduced sample through the rotary divider. Immediately place the whole of each portion in an airtight bottle or flask.

5.1.3.3 Preparation of test samples

If test samples are required from the laboratory samples, treat the latter as specified in 5.1.3.1 and 5.1.3.2.

The minimum mass of the test samples shall in no case be less than 10 g, otherwise there is a risk that the test samples may not be truly representative of the bulk sample.

If the mass required is not a suitable fraction of the bulk sample, it may be necessary to combine portions from successive stages of the division.

For example, to reduce a sample of 280 g to 10 g using six receivers, the first division with two marked receivers provides 2 × 47 g. One of the portions may again be divided and two of the resulting portions may be added to the remaining 47 g to give $47 + \left(\frac{2}{6} \times 47\right) \simeq 63$ g. Passing this through the apparatus a third time will give reduced samples of approximately 10 g.

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5.2 Products in paste form

5.2.1 Apparatus

5.2.1.1 Scoop or **spatula**, for sampling.

5.2.1.2 Domestic mixer, provided with a beater, for blending.

It is not possible to specify a mixer suitable for all requirements and any suitable mixer may be used.

In general, it shall be sufficiently powerful so that, when used with a beater of suitable design, the whole of the bulk sample is mixed and a creamy mass attained within 5 min.

5.2.2 Preparation of a reduced sample

Warm the product (bulk sample or laboratory sample) in its original container to 35 to 40 °C, and mix immediately using the domestic mixer (5.2.1.2) for 2 to 3 min until a homogeneous mass is obtained.

The paste shall not be removed from the original container before mixing as this may result in the production of a non-representative sample. It is therefore essential that the bulk sample is in a container which will allow mixing without transfer.

The heating and mixing times shall be as short as possible so as to reduce to a minimum any change in the product. Using the spatula or scoop (5.2.1.1), remove immediately the required quantity of sample and transfer it into an appropriate, previously tared container, fitted with a stopper.

Allow the contents of the container to cool to ambient temperature, and reweigh to obtain the mass of the reduced sample.

NOTE — Contact of the paste with glass vessels readily causes separation of a lye; therefore, once the sample is placed in the vessel, no part should be withdrawn to adjust the mass.

There is a slight loss of moisture during mixing and weighing but experience has shown that, in practice, this is at an acceptable level.

5.3 Liquid products

5.3.1 Apparatus

5.3.1.1 Glass flasks or **weighing pipettes**, for sampling.

5.3.1.2 Manual stirrer (for example, a glass rod).

5.3.1.3 Mechanical stirrer.

5.3.2 Preparation of a reduced sample

5.3.2.1 If the product (bulk sample or laboratory sample) is clear and apparently homogeneous, mix it with the manual stirrer (5.3.1.2); then, using the flask or weighing pipette (5.3.1.1), remove immediately the quantity required for the reduced sample. As little foam as possible shall be produced in the sample during mixing, and any loss of sample by evaporation shall be kept to a minimum.

5.3.2.2 If the product (bulk sample or laboratory sample) is cloudy or contains a sediment, mix it using the mechanical stirrer (5.3.1.3); then remove immediately the required quantity of sample.

5.3.2.3 If the product (bulk sample or laboratory sample) contains a solid deposit, carefully warm the original container to about 30 °C until the sediment can be completely dispersed by stirring or until any crystals disappear, and remove immediately the required quantity of sample.

6 Storage of the reduced sample

It is preferable for the analysis or test to be carried out as soon as possible after sampling but, if this is not possible and according to the intended purpose of the reduced sample, place it immediately in an airtight bottle or flask of glass or plastics materials and determine and record its mass. (Do not use metal containers.) Take care that the reduced sample is preserved as far as possible in its original condition until the analysis or test is carried out.

7 Report

The report shall contain the following information :

a) reference to the method used;

b) the number of types of samples prepared and their mass at the time they were taken;

c) the type of apparatus used;

d) any unusual features noticed during the division procedures;

e) any operation not included in this International Standard or regarded as optional.

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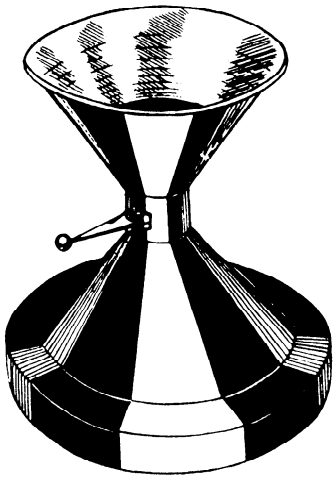

A Hopper

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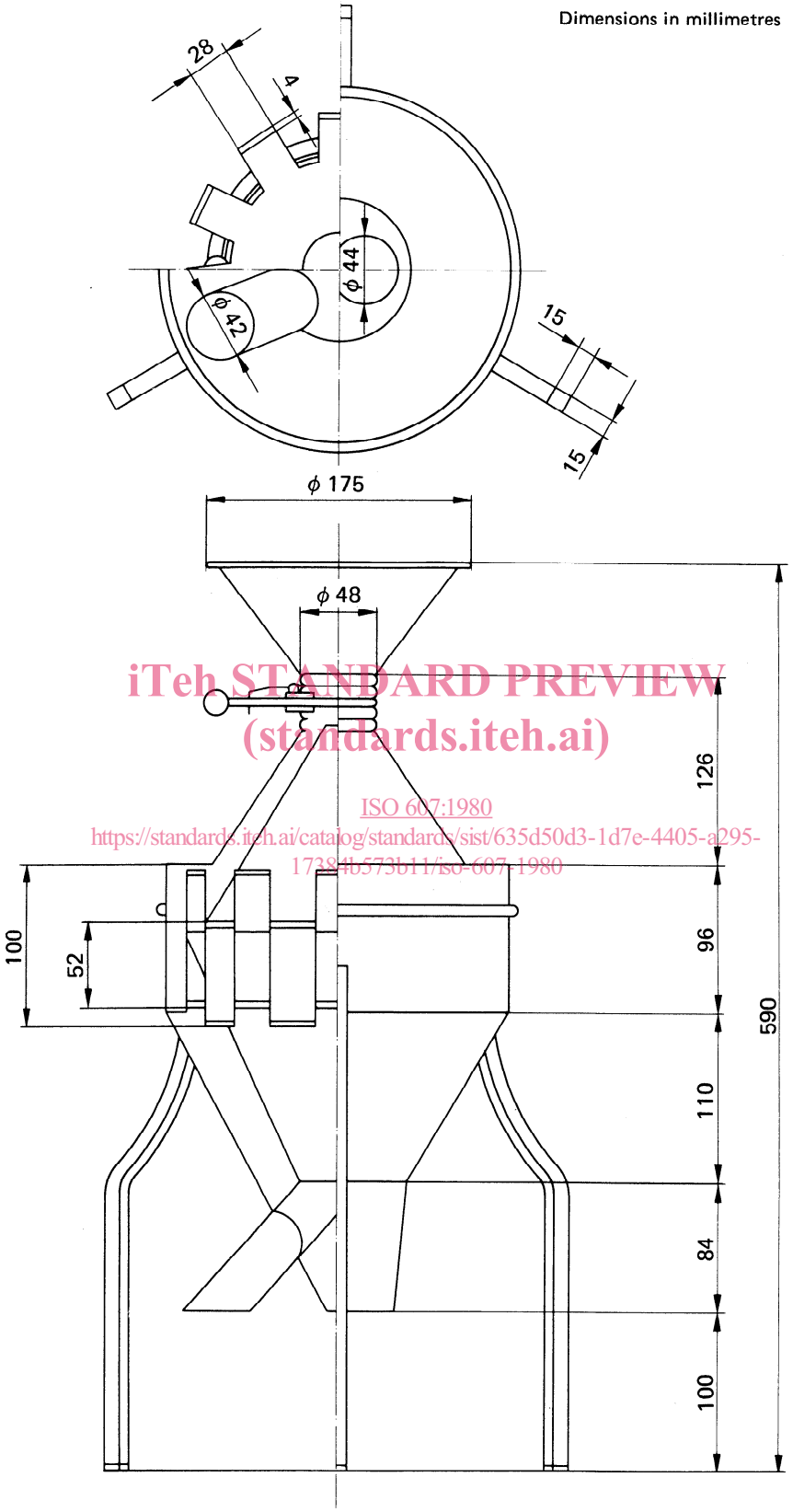
B Cone

C Inverted hopper

Figure 1 — Exploded view of a typical conical divider



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NOTE — Dimensions are given for guidance only.

Figure 2 — Diagram of a typical conical divider

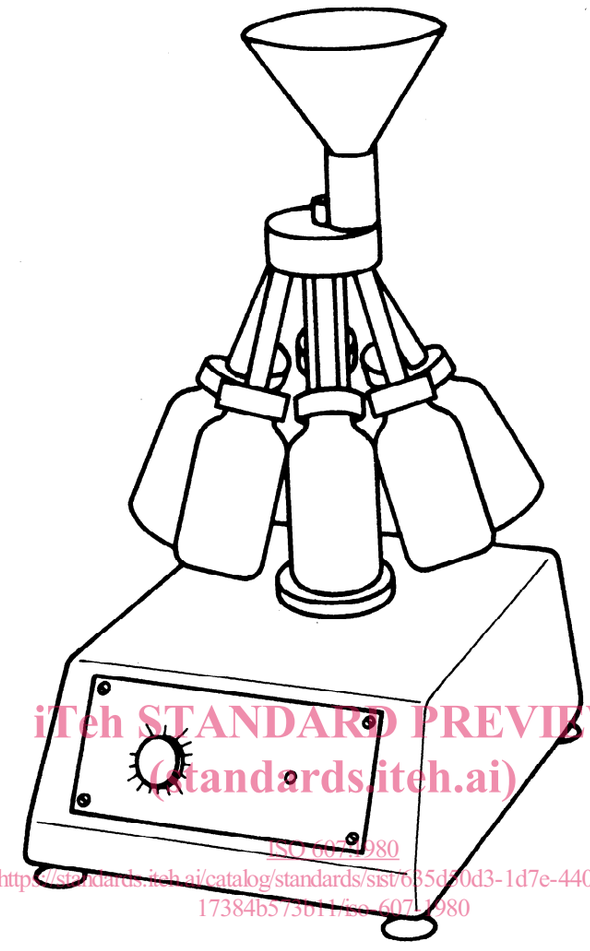

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Figure 3 — Perspective view of a typical rotary sample divider