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INTERNATIONAL STANDARD ISO 4324

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Surface active agents — Powders and granules — Measurement of the angle of repose

Agents de surface — Poudres et granulés — Mesurage de l'angle du talus d'éboulement

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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 4324 was developed by Technical Committee ISO/TC 91, *Surface active agents*, and was circulated to the member bodies in October 1975.

It has been approved by the member bodies of the following countries :

Australia	India	Portugal
Austria	Iran	Romania
Belgium	Italy	South Africa, Rep. of
Brazil	Japan	Spain
Canada	Korea, Rep. of	Switzerland
Egypt, Arab Rep. of	Mexico	Turkey
France	Netherlands	United Kingdom
Germany	New Zealand	U.S.A.
Hungary	Poland	U.S.S.R.

No member body expressed disapproval of the document.

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Surface active agents — Powders and granules — Measurement of the angle of repose

0 INTRODUCTION

Determining the angle of repose of powders and granules may give information on the storage properties of the powder, especially in silos, etc.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a conventional method for the determination of the angle of repose of surface active agents in powder or granular form and of washing powders, free from agglomerates.

This method is also applicable to other powders and granules with comparable properties.

2 REFERENCES

ISO 554, *Standard atmospheres for conditioning and/or testing — Specifications.*

ISO 607, *Surface active agents — Detergents — Methods of sample division.*[1)]

3 DEFINITION

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

angle of repose : The base angle of the cone obtained by flow under the specified conditions.

4 PRINCIPLE

Determination of the angle of repose of the cone obtained by passing a given volume of the product in the form of powder or granules through a special funnel placed at a fixed height above a completely flat and level plate.

5 APPARATUS

Only the dimensions given in the text are mandatory.

5.1 Measuring equipment, consisting of the following parts (see the figure) :

5.1.1 Glass funnel, having an internal stem diameter of 10 mm.

5.1.2 Agitator, comprising two rods placed opposite one another along the whole length of the interior wall of the funnel (5.1.1), and extending into the stem; these rods should be capable of being turned easily by means of a handle.

5.1.3 Base-plate, of minimum length 220 mm and minimum width 158 mm. The base-plate shall be completely rigid and shall have a polished surface or be engraved with concentric circles of diameters of from 10 to 100 mm, the centres of which shall be coaxial with the axis of the funnel.

5.1.4 Transparent plastic vessel, of minimum diameter 100 mm and minimum height 25 mm, placed 75 mm below the lower end of the funnel (5.1.1). Its internal surface shall be slightly rough. It shall be positioned on the base-plate (5.1.3) in such a way that the centre of the vessel and the axis of the funnel coincide, the centring being facilitated by the graduation of the concentric circles on the base-plate.

5.1.5 Funnel support, fixed and positioned so that the axis of the funnel (5.1.1) is the vertical line passing through the centre of the concentric circles engraved on the base-plate (5.1.3).

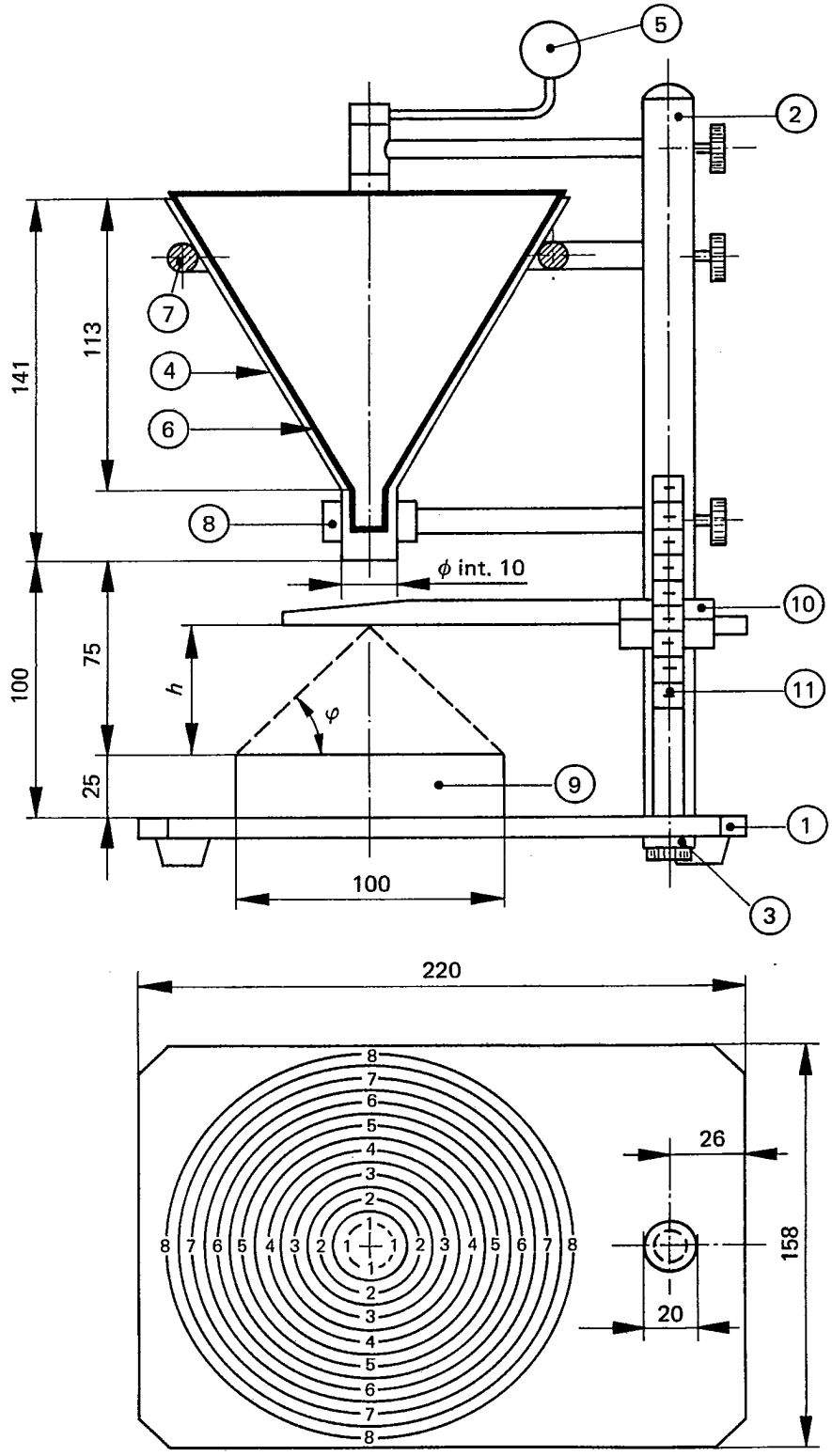
5.1.6 Supporting rod, comprising a scale graduated in millimetres from 0 to 100, starting from the base-plate (5.1.3). A rod integral with a horizontal slide moves on this scale, allowing the height of the cone to be measured.

The whole apparatus shall be protected against vibration.

5.2 Graduated measuring cylinder, of capacity 250 ml, complying with the requirements of ISO 4788.

1) In preparation. (Revision of ISO/R 607.)

Dimensions in millimetres



1. Base-plate
2. Support
3. Fixing screw for the base-plate and support
4. Glass funnel
5. Handle
6. Agitator rod
7. Supporting ring
8. Clip for fixing funnel
9. Transparent plastic vessel
10. Slide for measuring height of cone
11. Millimetre scale

FIGURE — **Apparatus for measuring angle of repose**

6 SAMPLING

The laboratory sample of surface active agent or washing powder shall be prepared and stored in conformity with ISO 607.

7 PROCEDURE

7.1 Preparation of sample and test portion

If the laboratory sample has formed agglomerates after a prolonged period of storage, it may still be examined if it regains the fluid state after the vessel containing it has been inverted.

Take 150 ml of the laboratory sample with the graduated measuring cylinder (5.2).

7.2 Determination

Carry out the measurements in one of the standard atmospheres defined in ISO 554.

Place the test portion in the funnel (5.1.1), having previously blocked the stem. Unblock the stem and allow the powder to flow through, slowly agitating it using the agitator (5.1.2).

NOTES

1 The agitator is not always required, but to achieve the specified reproducibility of the tests, the agitation process should always be included.

2 In the case of sticky products, it may be necessary to complete the discharge by careful use of a spatula.

Two minutes after the flow has ceased, measure the height of the powder cone.

Carry out a minimum of five tests on different portions of the laboratory sample.

8 EXPRESSION OF RESULTS

8.1 Method of calculation

The angle of repose (φ) of the sample is given, in radians, by the formula

$$\varphi = \text{Arctan}\,\frac{2h}{100} = \text{Arctan}\,\frac{h}{50}$$

where h is the height, in millimetres, of the powder cone.

Take as the result the arithmetic average of the five determinations. If the results differ by 5 % or more, repeat the measurements.

8.2 Precision

Comparative analyses on a sample of washing powders, carried out in 13 laboratories, have given the following statistical information :

- mean value of angle ($\bar{\varphi}$) : 0,59 rad (33,80°)
- standard deviation of repeatability (σ_r) : 0,008
- standard deviation of reproducibility (σ_R) : 0,011

8.2.1 *Repeatability*

The maximum deviation found between the results of two determinations carried out on the same product in rapid succession by the same operator using the same apparatus should not exceed $2{,}77\ \sigma_r = 0{,}022$ rad (1,27°) in more than 5 cases out of 100.

8.2.2 *Reproducibility*

The maximum deviation between two results obtained on the same sample, using the same apparatus in two different laboratories, should not exceed $2{,}77\ \sigma_R = 0{,}030$ rad (1,74°) in more than 5 cases out of 100.

9 TEST REPORT

The test report shall include the following particulars :

a) all information necessary for the complete identification of the sample;

b) the reference of the method used (reference to this International Standard);

c) the results obtained and the form in which they are expressed;

d) the test conditions;

e) any operation not included in this International Standard or regarded as optional, as well as any incidents which may have affected the results.