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R 1064

SURFACE ACTIVE AGENTS

DETERMINATION OF APPARENT DENSITY OF PASTES ON FILLING

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BRIEF HISTORY

The ISO Recommendation R 1064, *Surface active agents – Determination of apparent density of pastes on filling*, was drawn up by Technical Committee ISO/TC 91, *Surface active agents*, the Secretariat of which is held by the Association Française de Normalisation (AFNOR).

Work on this question led to the adoption of a Draft ISO Recommendation.

In December 1967, this Draft ISO Recommendation (No. 1424) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Austria	India	Romania
Belgium	Iran	South Africa, Rep. of
Canada	Israel	Spain
Chile	Japan	Sweden
Czechoslovakia	Korea, Rep. of	Switzerland
France	Netherlands	Turkey
Germany	New Zealand	U.A.R.
Greece	Poland	United Kingdom
Hungary	Portugal	Yugoslavia

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in April 1969, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 1064 April 1969

SURFACE ACTIVE AGENTS

DETERMINATION OF APPARENT DENSITY OF PASTES ON FILLING

INTRODUCTION

The volumes of liquid, powdered or granulated substances are defined by the apparent density and the apparent volume. For pasty or gelatinous substances, the corresponding values cannot be obtained by means of a simple physical procedure, as there may be swelling, formation of coacervate, etc.

1. SCOPE

This ISO Recommendation describes a simple method of determining, in the laboratory, the apparent density, on filling, of surface active agents in the form of pastes, ointments and similar products.

2. PRINCIPLE

Introduction, under pressure, of the quantity of a sample required to fill a container of known volume under the conditions of test. Determination, by weighing, of the mass required to fill the vessel.

3. APPARATUS

The Figure gives an example of the assembled apparatus, comprising the following items :

3.1 *A stainless steel tube* with the following dimensions :

- inside diameter : 26 mm
- outside diameter : 30 mm
- height : 188 mm

This tube is provided with an external flange, which forms a stop intended to make contact with the upper edge of the cylindrical vessel (3.3). The length of the part of the tube below the flange is at least 5 mm less than the interior height of the cylindrical vessel (3.3).

3.2 *A stainless steel piston* with the following dimensions :

- outside diameter : 25.9 mm
- mass, approx. : 770 g

and able to move freely inside the tube (3.1). The base is closed and the upper part is provided with a stop which prevents the piston from coming out of the lower end of the tube. A scale pan is fitted at the top of the piston to take additional weights, in order to keep the speed of filling within the prescribed limits.

3.3 *A cylindrical vessel* made of a rigid material which is not attacked by the products being tested, with the following dimensions :

- inside diameter : 30.4 mm
- height, approx. : 70 mm
- capacity : 50 ml at 20 °C.

This vessel closes the lower part of the tube (3.1).

The cylindrical vessel has a flat bottom and its upper edge is polished; the inside diameter is slightly greater than the outside diameter of the steel tube (3.1). The tube can therefore be moved inside the cylindrical vessel, along its axis, with a slight play. The upper end of the vessel is provided with a flat rubber washer placed so that its surface is tightened on the polished edge of the vessel in order to avoid dirtying the edge of the cylindrical vessel by the material.

The washer should have a small split at the side so that it is easy to remove.

3.4 *A platform* provided with a system of levers which enables it to be lowered slowly and evenly.

4. PROCEDURE

Carry out the procedure at 20 ± 2 °C.

4.1 Preparation of the apparatus

Fix the steel tube (3.1) on a support so that it is absolutely vertical.

Push on the cylindrical vessel (3.3), which has been cleaned and weighed, from the bottom until it stops against the flange of the tube (3.1), and place the rubber washer exactly on the upper edge of the vessel (3.3).

4.2 Filling of the apparatus

Fill the tube (3.1) up to 30 mm from the upper edge with the sample to be tested. Introduce the piston (3.2) and place weights on the scale pan of the piston to ensure that the sample will pass steadily down. Lower the cylindrical vessel (3.3) slowly, at a constant rate, using the levers of the platform (3.4). The air contained in the sample may escape through the clearance which exists between the cylindrical vessel (3.3) and the tube (3.1). The sample passes into the cylindrical vessel (3.3) under the pressure of the piston. The rate of descent is adjusted so that the cylindrical vessel is filled in not more than 2 minutes.

When the top of the cylindrical vessel (3.3) is in line with the bottom of the steel tube (3.1), remove the weights placed on the piston (3.2) and place a thin metal plate on the cylindrical vessel (3.3) in order to stop the sample from continuing to flow from the tube (3.1); at the same time, remove any excess from the cylindrical vessel (3.3). The upper surface of the sample thus obtained is flat and level with the upper edge of the cylindrical vessel.

4.3 Measurement

Weigh the cylindrical vessel, to the nearest 0.1 g, after removing the rubber washer; to simplify the measurement, use a counterweight corresponding to that of the cylindrical vessel (3.3).

Carry out five determinations with separate portions of the laboratory sample.

5. EXPRESSION OF RESULTS

5.1 Method of calculation and formula

The apparent density of the sample on filling, at 20 °C, expressed in grammes per millilitre , is

$$\frac{M_1 - M_0}{V}$$

where

M_0 is the mass, in grammes, of the cylindrical vessel;

M_1 is the mass, in grammes, of the cylindrical vessel when filled;

V is the volume, in millilitres, of the cylindrical vessel.

Take as the result the arithmetic mean of the five determinations.

5.2 Repeatability

The difference between the results of two determinations carried out by the same operator should not exceed 0.1 g/ml.

6. TEST REPORT

The test report should indicate the results obtained, stating :

(*a*) the name of the paste;

(*b*) the concentration of active matter;

(*c*) the temperature of filling and measurement if different from 20 °C;

(*d*) the volume of the measuring vessel;

(*e*) the filling time;

(*f*) the mass of sample in the cylindrical vessel.

In addition, the test report should mention all details of procedure not described in this ISO Recommendation, or any which are optional, and any circumstances which may have influenced the results.

The report should give all details required for complete identification of the sample.