UDC 648.181.15

JIS

JAPANESE INDUSTRIAL STANDARD

Testing Methods for Synthetic Detergent

JIS K 3362—1990

Translated and Published

by

Japanese Standards Association

Printed in Japan

In the event of any doubt arising,
the original Standard in Japanese is to be final authority.

Contents

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JAPANESE INDUSTRIAL STANDARD J I S

Testing Methods for Synthetic Detergent K 3362-1990

1. Scope

This Japanese Industrial Standard specifies the testing methods for household synthetic detergents.

Remark: In this Standard the units and numerical values shown in { } are in accordance with the traditional units and are standard values.

2. Common Matters

2.1 Definitions For the purposes of this Standard the main definitions are in accordance with JIS K 3211.

2.2 General Matters General matters common to the test are in accordance with JIS K 0050.

3. Test Items

Test items specified in this Standard are divided into sampling methods, chemical tests, physical tests and detergency evaluation methods and are as follows:

3.1 Sampling Method

3.2 Chemical Tests

(a) Determination of Petroleum Ether Soluble Matter
(b) Ethanol Soluble Matter
(c) Qualitative Test and Determination of Anionic Surface Active Agent
(d) Qualitative Test and Determination of Cationic Surface Active Agent
(e) Qualitative Test and Determination of Nonionic Surface Active Agent
(f) Determination of Urea
(g) Determination of Equivalent to Surface Active
(h) Determination of Sodium Carboxylmethylcellulose
(i) Determination of Peroxide
(j) Determination of Total Phosphate
(k) Determination of Silicate
(l) Determination of Sulfate
(m) Determination of Carbonate
(n) Determination of Chloride
(o) Determination of Zeolite
(p) Confirmation Test of Fluorescent Brightener
(q) Limit Test of Arsenic (As)
(r) Limit Test of Heavy Metal (As Pb)
(s) Determination of Methanol
(t) Determination of Ethanol
(u) Determination of Moisture

Applicable Standards, Correpsonding International Standards: See pages 69 to 71.

3.3 Physical Tests

(a) Grain Size

(b) Apparent Density

(c) pH Value

(d) Surface Tensile Strength

(e) Foaming Power and Stability of Foam

(f) Stability in Hard Water

3.4 Detergency Evaluation Methods

(a) Detergency Evaluation Method for Synthetic Detergent for Cloth

(b) Detergency Evaluation Method for Synthetic Detergent for Kitchen

4. Sampling Method

4.1 Sampling of Representative Sample Form a lot considered to be the same quality such as products manufactured in the same lot, products made within a definite time after continuous manufacturing in the same facility, and the like, corresponding to the number of containers of the lot, take the representative sample of the specified quantity of the number of pieces shown in Table 1 at random according to the suitable method such as random table. However, in case where the containers exceed 1000 pieces, even to its fraction, apply Table 1.

Table 1. Taking Number of Samples

Number of containers	Number of sampling
1 to 10	1
11 to 50	2
51 to 100	3
101 to 500	5
501 to 1000	10

Remark: Relating to the number of sampling, it may be determined according to the process capacity.

4.2 Preparation of Sample Mix the representative samples taken according to 4.1 and according to the profile of products prepare the samples.

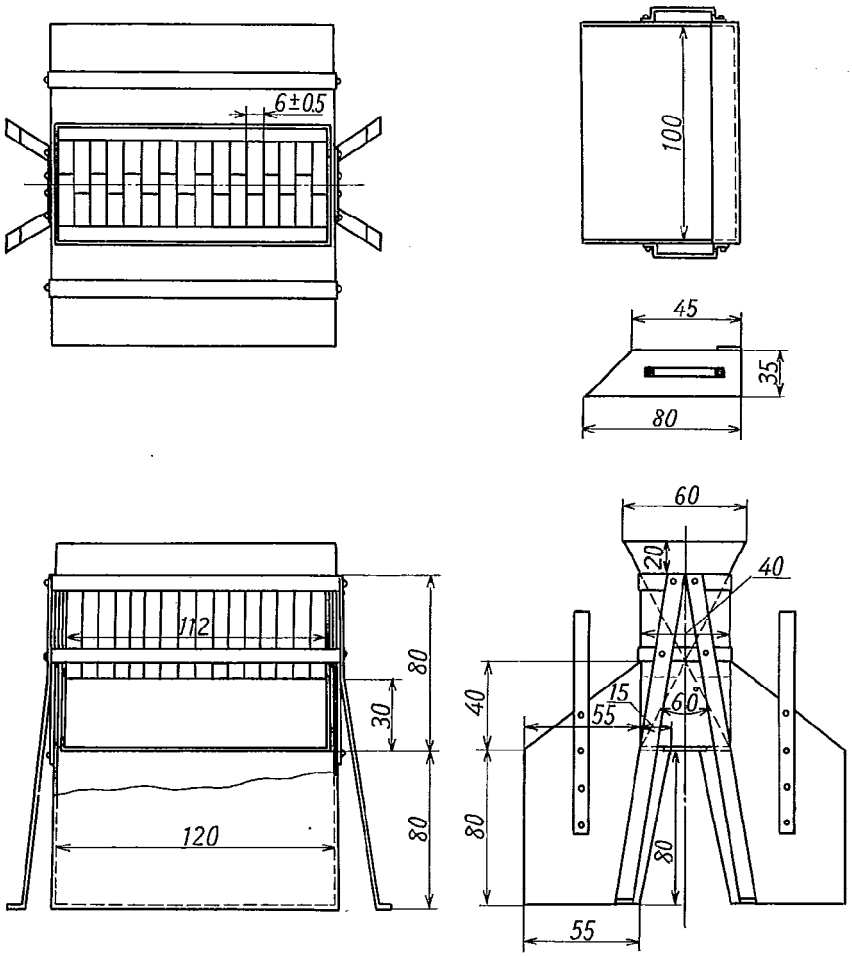
4.2.1 Apparatus The apparatus shall be as follows.

(1) Reduction Apparatus The reduction apparatus of No. 6 of Attached Fig. 4 of JIS M 8100. An example is shown in Fig. 1.

(2) Beaker A beaker made of glass or stainless steel, of 1 *l* in capacity.

Fig. 1. An Example of Reduction Apparatus

Unit: mm



4.2.2 Operation The operation shall be carried out as follows:

(1) Pulverized State or Grain State Sample With taking so care to the representative sample taken in 4.1 as not to break particle itself, loosen the grain having become a specially large lump to mix, apply the total amount to a reduction apparatus and repeat until a suitable quantity sample to be used for test is obtained.

Remarks 1. At the time of using a reduction apparatus, care shall be taken to clogging, shaking method of sample container. In the case of carrying out the repeating operation, if take matters at only one side of reduction apparatus, there is a fear to generate bias, and therefore which is taken shall be selected at random.

2. The pulver state or grain state detergent may have nonuniform property and it is, in some cases, difficult to take representative sample. Particularly the pulver state detergent which is added with other materials after receiving spray, dry and cooling process is physically mixing, therefore each component is in inclination to be separated only by vibration, and therefore it is necessary to apply the total of taken representative sample to reduction apparatus.

(2) Liquid State or Pasty State Sample Transfer the representative sample taken in 4.1 to a beaker, and mix. When the liquid or paste is transparent and uniform, take separately a suitable amount from this and take it as the sample.

In this case, when it is opaque and contains precipitate, while taking so care the air bubbles not to enter, mix by stirring with a glass rod, and if required warm it in water bath at 50 to 60°C([1])([2]). After becoming uniform, while mixing by stirring cool until 15 to 30°C, and take separately a suitable amount to take it as the sample. Enter this sample in a glass bottle or polyethylene bottle with ground-in stopper, and store in a cool, dark place.

Notes ([1]) Products on the market may have pH value of 6.5 or under and, be unsuitable to heat to 50 to 60°C and therefore cares shall be taken to handling.

([2]) In the case of heating, the loss due to evaporation of water shall be taken into consideration, and therefore the mass shall be measured before and after heating.

5. Chemical Tests

5.1 Determination of Petroleum Ether Soluble Matter

5.1.1 Summary As to the petroleum ether soluble matter, in the case where the water-ethanol solution of sample is extracted with petroleum ether, the amount to be solved in petroleum ether is obtained([3]).

Note ([3]) Among the nonionic surface active agents, there are those to be extracted in petroleum ether.

5.1.2 Reagents Reagents shall be as follows:

(1) Petroleum Ether The petroleum ether specified in JIS K 8593, distilled at 30 to 60°C.

(2) Mixed Solution of Water-Ethanol The mixture of ethanol specified in JIS K 8102 with water in equal amount.

(3) Sodium Sulfate (Anhydride) The sodium sulfate specified in JIS K 8987.

(4) 0.5 mol/*l* (N/2) Sodium Hydroxide Solution The 0.5 mol/*l* (N/2) sodium hydroxide solution specified in 4.4 (20.2) of JIS K 8001.

(5) Phenolphthalein Solution (1 w/v %) The phenolphthalein 1 g specified in JIS K 8799 dissolved in ethanol (95) 100 ml.

5.1.3 Operation The operation shall be carried out as follows:

(1) Weigh out the sample approximately 10 g into an Erlenmeyer flask of 300 ml to the nearest 1 mg and dissolve in water-ethanol mixed solution 200 ml. If there are insolubles at this time, filter them.

(2) Add 0.5 mol/*l* (N/2) sodium hydroxide solution 5 ml, and drop phenolphthalein solution (1 w/v %) to confirm that it is alkaline.

(3) Transfer to a separatory funnel of 500 ml, and extract three times by each petroleum ether 50 ml. When the emulsion is generated, add a small amount of ethanol to extinguish.

(4) Join the petroleum ether layer, wash three times with water-ethanol mixed solution of each 30 ml and wash two times with water of each 30 ml, after dehydration with sodium sulfate (anhydride), filter by using dried filter paper into an Erlenmeyer flask of 300 ml of known mass, and wash the filter paper with a small amount of petroleum ether.

(5) Vaporize the petroleum ether on water bath, leave the Erlenmeyer flask in a desiccator to cool until room temperature, feed dry air to inside of Erlenmeyer flask and

after expelling until the odor of remaining petroleum ether extinguishes, weigh the mass.

Remark: Join the residual solution of petroleum ether extraction and washings to preserve and use for 5.3.3 Determination of Soap Content.

5.1.4 Calculation Calculate the petroleum ether solubles according to the following formula.

$$C = \frac{A}{S} \times 100$$

where, C : petroleum ether solubles (%)

A : petroleum ether extraction amount (g)

S : mass of sample (g).

5.2 Determination of Ethanol Solubles

5.2.1 Summary As to ethanol solubles, the sample is dissolved by ethanol and the mass of substances soluble in ethanol when extracted is obtained.

5.2.2 Reagents Reagents shall be as follows:

(1) Ethanol (95) Ethanol specified in JIS K 8102.

(2) Ethanol (99.5) Ethanol specified in JIS K 8101.

5.2.3 Apparatus The apparatus shall be as follows.

(1) Erlenmeyer Flask An Erlenmeyer flask of 300 ml attached with a glass tube of 65 cm or more in length.

(2) Glass Filter The filter plate of fine aperture symbol 4 specified in JIS R 3503.

(3) Receiver for Filtration

(4) The dryer capable of adjusting at 105 ± 2°C.

5.2.4 Operation The operation shall be carried out as follows:

(1) Weigh out the sample approximately 5 g into an Erlenmeyer flask 300 ml to the nearest 1 mg, add ethanol([4]) 100 ml, attach a glass tube and while mixing by shaking at times on the water bath for 30 min to dissolve.

(2) Filter the warm solution as it is by using a glass filter, and add again ethanol (95) 50 ml to the residual of Erlenmeyer flask to dissolve. Filter the warm solution by using a glass filter, and wash throughly the Erlenmeyer flask and glass filter with hot ethanol. Leave to cool to room temperature, transfer the filtrate and washings to one mark volumetric flask 250 ml, and add ethanol (95) up to the marked line. From this, separately take each 100 ml into two beakers of 200 ml of known mass by using a transfer pipet.

(3) Heat the one piece out of them on water bath, after removing ethanol, dry in an oven adjusted at 105 ± 2°C for 1 h and after leaving to cool in a desiccator, measure the mass.

Note ([4]) For pulver state or grain state sample use ethanol (95) and for liquid state or paste state sample use ethanol (99.5).