Singapore Standard 231 : 1984

SPECIFICATION FOR LAUNDRY DETERGENT POWDER FOR HOUSEHOLD USE IN MANUAL WASHING

AMENDMENT NO. 1

August 1994

1. **Page 6, Subclause 2.2**

Delete the existing subclause and table and *substitute* the following :

'**2.2 Detailed Requirements.** The material shall consist essentially of a uniform mixture of surfactants and suitable builders and shall comply with the requirements given in Table 1. The surfactant shall be of either the anionic or non-ionic type, or both. The anionic surfactants shall have a biodegradability of more than 90%.'

Table 1

Characteristic		Requirement	Method of Test in Appendix
1.	Total surfactants, % (m/m)	20.0	A
2.	Biodegradability of synthetic anionic detergents, % (m/m), minimum	90.0	refer to ASTM D 2667 : 1989
3.	Total phosphate (as P_2O_5),% (m/m), maximum	20.0	B
4.	pH at 25°C (for 1% aqueous solution, m/v)	9 - 11	C
5.	Insoluble matter, % (m/m) maximum	1.0	D

2. **Page 9, Appendix A**

(a) *Delete* the existing Appendix A and *replace* as follows :

APPENDIX A

TOTAL SURFACTANTS CONTENT

A.1 DETERMINATION OF TOTAL SURFACTANTS CONTENT

The total surfactants content is obtained by using the following steps:

(a) Determine the non-ionic surfactants content using the method described in A.2.

(b) Determine the anionic surfactants content using the method described in A.3.

(c) Sum up the non-ionic and anionic surfactants contents determined in (a) and (b) to give the total surfactants content.

In the event that the product contains only the non-ionic or anionic surfactants, the content can be obtained using the method described in A.2 or A.3 respectively. The value obtained will then be reported as total surfactants content.

A.2 NON-IONIC SURFACTANTS

A.2.1 Apparatus

A.2.1.1 Ion-exchange column. 25 mm internal diameter and 300 mm length with a stop-cock at the lower end.

A.2.1.2 Reservoir. 150 ml capacity with stop-cock.

A.2.1.3 Oven. Controlled at 100°C to 150°C.

A.2.2 Reagents

A.2.2.1 Ion exchange resin. Amberlite monobed MB-1, analytical grade.

A.2.2.2 Propan-2-o1. Propan-2-o1, 90% (v/v) solution.

A.2.2.3 Methylene blue indicator solution. Dissolve 50 g anhydrous sodium sulphate in about 500 ml water, add 6.5 ml concentrated sulphuric acid, add a solution containing 0.03 g methylene blue and dilute to 1000 ml.

A.2.3 Procedure

A.2.3.1 Half fill the ion-exchange column with 90% propan-2-o1. Insert a plug of cotton wool and push down with a glass rod.

A.2.3.2 Slurry some monobed resin in a beaker with 90% propan-2-o1 and fill the column, allowing the resin to settle while drawing off alcohol through the stop-cock, until the resin is about 50 mm from the top. Adjust the alcohol level to about 10 mm above the surface of the resin.

A.2.3.3 Fix the reservoir above the column and pass 50 ml of 90% propan-2-o1 through the column at about 10 ml per minute, finally allowing the alcohol level to fall to about 10 mm above the surface of the resin.

A.2.3.4 Weigh a 150 ml beaker. Add 5 to 10 g of the sample and re-weigh to the nearest mg. The sample should contain not more than 2 g of anionic active agent.

A.2.3.5 In the case of powders and pastes which are not completely soluble in propan-2-o1, add 50 ml water, heat to boiling and stir for 5 minutes, then cool. To other samples, except liquids containing over 50% water, add 5 ml water.

A.2.3.6 Add 50 ml 100% propan-2-o1 and stir.

A.2.3.7 Transfer the solution to the reservoir, retaining as much as possible any precipitate in the beaker.

A.2.3.8 Allow the solution to flow through the column at 3 ml to 4 ml per minute, collecting the effluent in a 250 ml comical flask or beaker which has been weighed containing 3 to 6 antibumping granules. The resin should be kept covered with liquid throughout the determination.

A.2.3.9 Rinse the original beaker with 50 ml of 90% propan-2-o1 and decant into the reservoir when empty.

A.2.3.10 Repeat the step in A.2.3.9 twice more.

A.2.3.11 Check the effluent for active matter. Pipette 1 ml of the well mixed effluent into a 100 ml stoppered cylinder. Add 9 ml water, 1 ml methylene blue, and 10 ml chloroform. Shake well and allow the layers to separate. If the colour in the lower layer is more intense than in the upper, the whole of the solution must be passed through a new column of resin, washing through with a further 50 ml of 90% propan-2-o1.

A.2.3.12 Place the vessel containing the effluent on the steam bath and evaporate the solvent. Add a few ml of acetone and evaporate to dryness. Repeat until all water appears to have been removed.

A.2.3.13 Dry in the oven at 100 to 105°C for 30 minutes, allow to cool for 15 minutes and weigh.

A.2.3.14 Repeat the step in A.2.3.13 until consecutive weighings agree to within 0.002 g.

$$\%\ \text{Non-ionic} = \frac{\text{Weight of residue}}{\text{Weight of sample}} \times 100$$

The non-ionic residue may be required for further analysis and should be retained.

A.3 SYNTHETIC ANIONIC DETERGENTS (DIRECT TWO-PHASE TITRATION METHOD)

A.3.1 Principle

The anionic active matter is determined in a medium consisting of an aqueous and a chloroform phase, by volumetric titration with a standard cationic-active solution (benzethonium chloride), in the presence of an indicator which consists of a mixture of a cationic dye (dimidium bromide) and an anionic dye (acid blue I).

The anionic-active matter forms a salt with the cationic dye which dissolves in the chloroform to give this layer a red-pink colour.

In the course of the titration, the benzethonium chloride displaces the dimidium bromide from this salt and the pink colour leaves the chloroform layer as the dye passes to the aqueous phase. The benzethonium chloride dissolves in the chloroform layer and colours it blue.

NOTE. Low relative molar mass sulphonates present as hydrotropes (toluene, xylene) do not interfere when present in concentrations up to 15% (m/m) on active material. At higher levels, their influence must be evaluated in each particular case.

Soap, urea and ethylenediaminetetra-acetic acid salts do not interfere.

Typical inorganic components of detergent formulations such as sodium chloride, sulphate, borate, tripolyphosphate, perborate, silicate, etc., do not interfere but bleaching agents other than perborate should be destroyed before the analysis.

A.3.2 Apparatus

A.3.2.1 Glass stoppered measuring cylinders, 100 ml capacity.

A.3.2.2 Burettes, 25 ml and 50 ml capacities.

A.3.2.3 One-mark volumetric flask with ground glass stopper, 1 litre capacity.

A.3.2.4 One-mark pipette, 25 ml capacity.

A.3.3 Reagents

A.3.3.1 Chloroform, $d_{20} = 1.485$, distilling between 59.5°C and 61.5°C.

A.3.3.2 Phenolphthalein, 1% (m/v) solution in 95% (v/v) ethanol.

A.3.3.3 Sulphuric acid, 2.5 M solution.

A.3.3.4 Sulphuric acid, 0.5 M solution.

A.3.3.5 Sodium hydroxide, 1.0 M standard solution.

A.3.3.6 Sodium lauryl sulphate (sodium dodecyl sulphate), 0.004 M standard volumetric solution. Determine the purity of the sodium lauryl sulphate and simultaneously prepare the standard solution.

A.3.3.6.1 Determination of purity of sodium lauryl sulphate. Weigh accurately 5 ± 0.200 g of the sample into a 250 ml round bottom flask with ground glass neck. Add exactly 25 ml of 0.5 M sulphuric acid and reflux under a water condenser. During the first 5 min to 10 min the solution will thicken and tend to foam strongly; control this by removing the source of heat and swirling the contents of the flask. (In order to avoid excessive foaming, instead of refluxing, the solution may be left on a boiling water bath for 60 min). After a further 10 min, the solution clarifies and foaming ceases. Reflux for another 90 min. Remove the source of heat, cool the flask and carefully rinse the condenser with 30 ml of ethanol followed by water. Add a few drops of the phenolphthalein solution and titrate the solution with sodium hydroxide solution.

$$\text{Purity of sodium lauryl sulphate (\%)} = \frac{28.84\ (V_1 - V_0)M_0}{m_1}$$

where:

V_0 = millilitres of sodium hydroxide solution used for the blank test

V_1 = millilitres of sodium hydroxide solution used for the sample

M_0 = exact molarity of the sodium hydroxide solution

m_1 = mass in grams of the sodium lauryl sulphate checked

A.3.3.6.2 0.004 M sodium lauryl sulphate standard volumetric solution. Weigh accurately 1.140 g to 1.160 g of sodium lauryl sulphate and dissolve in 200 ml of water. Transfer to a ground glass stoppered 1 litre one-mark volumetric flask and dilute to volume with water. The molarity of the solution, M_2, is calculated by the formula:

$$M_2 = \frac{m_2 \times \text{purity } (\%)}{288.4 \times 100}$$

where:

m_2 = mass in grams of sodium lauryl sulphate

A.3.3.7 Benzethonium chloride (Benzyl dimethyl - 2 - [2 - p (1,1,3,3,- tetramethylbutyl) phenoxy - ethoxyl] - ethyl ammonium chloride, monohydrate), 0.004 M standard volumetric solution.

$[(CH_3)_3\ C\text{-}CH_2\text{-}C(CH_3)_2\text{-}C_6H_4OCH_2\text{-}CH_2\text{-}OCH_2\text{-}CH_2\text{-}N\ (CH_3)_2\text{-}CH_2\text{-}C_6H_5]\ ^{+}CL^{-}.\ H_2O$

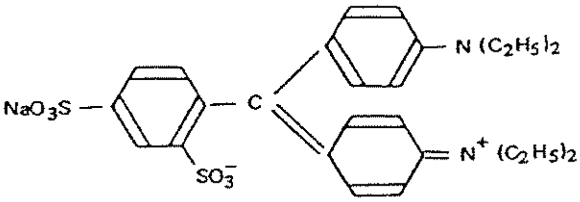
Weigh accurately 1.750 g to 1.850 g of benzethonium chloride and dissolve in water. Transfer to a ground glass stoppered 1 litre one-mark volumetric flask and dilute to volume with water.

NOTE. In order to prepare a 0.004 M solution, dry the benzethonium chloride at 105°C, weigh accurately 1.792 g, dissolve in water and dilute to 1 litre.

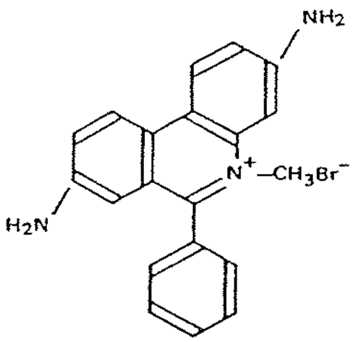
A.3.3.8 Mixed indicator solution*. Add 200 ml of water to 20 ml of the stock solution (A.3.3.8.1) in a 500 ml one-mark volumetric flask. Add 200 ml of 2.5 M sulphuric acid, mix and dilute to volume with water. Store out of direct sunlight.

A.3.3.8.1 Stock solution. This solution is prepared from acid blue I and dimidium bromide.

A.3.3.8.1.1 Acid blue I (mono-sodium -4', 4" -diaminodiethyltriphenyl methane -2, 4-disulphonate). $C_{27}H_{31}N_2O_6S_2Na$



A.3.3.8.1.2 Dimidium bromide (3,8 -diamino-5-methyl-6-phenyl -phenanthridinium bromide). $C_{20}H_{18}BrN_3$



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* This is available commercially in the form of a basic solution, which should be acidified and diluted before use.

A.3.3.8.1.3 Preparation of the stock solution. Weigh accurately 0.5 ± 0.005 g of dimidium bromide into a 50 ml beaker and 0.25 ± 0.005 g of acid blue I into another 50 ml beaker. Add between 20 ml and 30 ml of hot 10% (v/v) aqueous ethanol to each beaker. Stir until dissolved and transfer the solutions to a 250 ml one-mark volumetric flask. Rinse the beakers with 10% (v/v) aqueous ethanol and transfer the rinsings into the volumetric flask. Dilute to the mark with ethanol.

A.3.4 Procedure

A.3.4.1 Standardization of benzethonium chloride solution. Using a pipette, transfer 25 ml of the 0.004 M sodium lauryl sulphate solution to the measuring cylinder; add 10 ml of water, 15 ml of the chloroform and 10 ml of the mixed indicator solution.

Titrate with 0.004 M benzethonium chloride solution; stopper the measuring cylinder after each addition and shake well. The lower layer will be coloured pink. Continue the titration with repeated vigorous shaking. As the end point approaches, the emulsions formed during shaking tend to break easily. Continue the titration drop by drop after each addition of titrant until the end point is reached. This is at the moment when the pink colour is completely discharged from the chloroform layer which becomes a faint greyish blue.

The molarity, M_1 of the benzethonium chloride solution is given by the formula:

$$M_1 = \frac{M_2 \times 25}{V_2}$$

where:

M_2 = molarity of the sodium lauryl sulphate solution

V_2 = volume in millilitres of benzethonium chloride solution added

A.3.4.2 Test portion. Weigh accurately a sample which contains 3 to 5 milliequivalents of anionic-active matter.

NOTE. The table below which has been calculated on the basis of a relative molar mass of 360 may be used as a rough guide.

Table 2. Mass of test portion

Amount of active matter in sample % (m/m)	Mass of test portion g
15	10.0
30	5.0
45	3.2
60	2.4
80	1.8
100	1.4

A.3.4.3 Determination. Dissolve the test portion in water. Add a few drops of phenolphthalein solution and neutralize to a faint pink colour with the sodium hydroxide solution or 0.5 M sulphuric acid solution as required.

Transfer to a 1 litre one-mark volumetric flask and dilute to volume with water. Mix thoroughly and by means of the pipette, transfer 25 ml of this solution to the measuring cylinder. Add 10 ml of water, 15 ml of chloroform and 10 ml of the mixed acid indicator solution. Titrate with the benzethonium chloride solution as described in A.4.1.

A.3.5 Calculation

The content of anionic-active matter, % (m/m) is

$$\frac{V_3 \times M_1 \times 1000 \times W \times 100}{25 \times 1000 \times m_0} = \frac{4 \times V_3 \times M_1 \times W}{m_0}$$

where:

M_0 = mass in grams of the test portion

W = relative molar mass of anionic-active matter

M_1 = molarity of the benzethonium chloride solution

V_3 = volume in millilitres of benzethonium chloride solution used for the titration of a 25 ml aliquot of anionic-active matter solution

3. **Page 14, Appendix B**
Delete the existing Appendix B entirely including Figures 1 and 2.

4. **Page 25, Appendix C**

(a) *Rename* the existing 'Appendix C' as 'Appendix B.'

(b) *Replace* all clauses with the prefix 'C' with the prefix 'B'.

5. **Page 28, Appendix D**

(a) *Rename* the existing 'Appendix D' as 'Appendix C'.

(b) *Replace* all clauses with the prefix 'D' with the prefix 'C'.

6. **Page 29, Appendix E**

(a) *Rename* the existing 'Appendix E' as 'Appendix D'.

(b) *Replace* all clauses with the prefix 'E' with the 'prefix 'D'.