

DEAS 127:2010

UGANDA STANDARD

Specification
for

**Laundry detergent for house-hold use
(Granules and Powders)**

First edition: December 2000

UGANDA NATIONAL BUREAU OF STANDARDS

Price group: E

Descriptors: Detergents

ICS 71.100.40

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IT IS IMPORTANT THAT USERS OF UGANDA STANDARDS ASCERTAIN THAT THEY ARE IN POSSESSION OF THE LATEST AMENDMENTS OR LATEST EDITIONS

In order to keep abreast of technological development Uganda Standards are subject to periodic review.

The following table will assist the user to update the standard

AMENDMENTS

Clause	Amendment No.	Date of Issue	Text affected

In order to keep abreast of technological development Uganda Standards are subject to periodic review.

NOTE:

- 1. Compliance with this Standard does not, of itself confer immunity from legal obligations.**
- 2. A Uganda standard does not purport to include all necessary provisions of a contract. Users are responsible for its correct application.**

Uganda National Bureau of Standards
P.O. Box 6329

Tel:256-41- 222367/9

Fax: 256-41-286123

Website www.unbs.or.ug

E-mail: unbs@starcom.co.ug

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0.0 Forward

0.1 Uganda National Bureau of Standards (UNBS) is a parastatal under the Ministry of Tourism, Trade and Industry established by the Act of Parliament of 1983, of the Laws of Uganda. UNBS is

- (i) a member of International Organisation for Standardisation (ISO) and
- (ii) a contact point for the WHO/FAO Codex Alimentarius Commission on Food Standards, and
- (iii) the National Enquiry Point on TBT/SPS Agreements of the World Trade Organisation (WTO).

0.2 The work of preparing Uganda standards is carried out through Technical Committees. A Technical Committee is established to deliberate on standards in a given field or area and consists of representatives of consumers, traders, academicians, manufacturers, Government and other stakeholders.

0.3 Draft Uganda standards adopted by the Technical Committee are widely circulated to stakeholders and the general public for comments, which are reviewed before recommending them to the National Standards Council for declaration as national standards.

0.4 There are two main groups of detergents namely the soaps and non-soapy detergents. In order to guide the production of non-soapy detergents of well-defined quality and to safeguard consumer interests, it has been felt desirable to formulate this standard

0.5 The soapy detergents are of three types i.e. anionic, cationic, and non-ionic. Anionic non-soapy detergents of the alkyl aryl type such as sodium salts of dodecylbenzene sulphonic acid, are now produced in large quantities.

This Uganda Standard was prepared by the Technical Committee on Chemicals and environment in order to guide the manufacturers, importers and the consumers on the quality of powder detergents.

0.2 COMMITTEE MEMBERSHIP

- Chemistry department – Makerere University Kampala
- Government Chemist laboratories
- National Environment Management Authority (NEMA)
- Chemist Section - Uganda Revenue Authority (URA)
- Uganda Baati
- Nakasero Soap Works Ltd
- Mukwano Industry
- Kakira Sugar works (1985) Limited - Soap Division
- Uganda Consumer Protection Association
- Avis Company Limited
- Mbale Soap works
- Unilever (U) Limited

UGANDA STANDARD FOR GENERAL PURPOSE DETERGENT POWDERS

1.0 SCOPE

This standard prescribes the requirements and methods of sampling and test for synthetic laundry detergents in granulated or powdered form for household hand laundering.

2.0 NORMATIVE REFERENCES

The following standards contain provisions, which, through reference in this text, constitute provisions of this standard. All standards are subject to revision and, since any reference to a standard is deemed to be a reference to the latest edition of that standard, parties to agreements based on this standard are encouraged to take steps to ensure the use of the most recent editions of the standards indicated below. Information on currently valid national and international standards may be obtained from the Uganda national bureau of standards

ISO 4316 Surface active agents – Determination of pH of aqueous solution – Potentiometric method.

ISO 4318 Surface active agents and soaps – Determination of water content – Azeotropic distillation method.

3.0 DEFINITIONS

For the purposes of this standard, the following definitions apply:

3.1 Batch: The material from a single mix or, in the case of a continuous production process, the material from a single day's production.

3.2 Defective: Detergent or a container that fails in one or more respects to comply with the relevant requirements of this standard.

3.3 Lot: That quantity of a detergent bearing the same batch identification, from one manufacturer, and submitted at any one time for inspection and testing.

4.0 REQUIREMENTS

4.1 General

4.1.1 Form

The detergent shall be in the form of granules or a powder, and shall be homogeneous, free-flowing, free from visible impurities, abrasives, and organic solvents, and readily soluble in water. It shall not give rise to excessive dust.

4.1.2 Effect on health:

The detergent shall not be irritating to the normal skin and shall not contain any ingredients in a quantity that is toxic to human beings.

4.2 Odour

Neither the detergent nor a solution of the detergent in hot water (at $60\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$) shall have an objectionable odour. Perfume may be added, in which case, it shall not change its fragrance nor develop an objectionable odour during storage at ambient temperature for six months.

4.3 Colour

The detergent may be coloured provided that the colouring is uniform and does not change appreciably during storage.

4.4 Storage properties

When stored or transported under normal conditions in the original container, the granules or powder shall not cake into hard lumps.

4.5 Dispersibility

The detergent shall be readily dispersible in water.

4.5 Rinsing properties

When tested in accordance with appropriate method the detergent shall be free-rinsing.

4.6 Chloride content

When the detergent is tested in accordance with 6.8, the chloride content (expressed as sodium chloride) shall not exceed 2,0 % (m/m).

4.7 Insoluble matter

When the detergent is tested in accordance with Annex B the (matter insoluble in water) shall not exceed 5.0 % (m/m).

4.8 pH value

The pH value of 10 g/l solution of the detergent, when determined in accordance with Annex B.8 shall be not less than 9,0 and not more than 11,0.

Table 1 – Requirements for synthetic detergents for household use

Characteristics	Requirements	Test Method
1. Moisture and volatile matter content at 105° C per cent by mass (max)	13	Annex B.4
2. [T1]Active ingredient % m/m (min)	12	Annex B.5
3. Matter insoluble in alcohol, % m/m (max)	70	Annex B.6
4. Phosphates (expressed as sodium poly-phosphate) % mass of insoluble matter in alcohol, min	10	Annex B.7
5. pH	9 to 11	Annex B.8
6. Non detergent organic matter, per cent by mass, max	1.0	Annex B.9
7. Matter insoluble in water % m/m max	5.0 0.5[T2]	Annex B.10
8. Carboxyl methyl cellulose (% m/m)	2	Annex B.11

Note: These requirements do not refer to ecologically friendly soaps for which an alternative standard shall apply.[T3]

5.0 SAMPLING, TESTING, AND COMPLIANCE WITH THE STANDARD

5.1 General requirements for sampling

In drawing, preparing, storing and handling samples, the following precautions shall be observed

- 5.1.1 The sample shall be taken in a protected place, not exposed to damp air, dust or soot.
- 5.1.2 The sampling equipment shall be clean and dry when used.
- 5.1.3 The samples, the material being sampled, the sampling instruments and containers for samples shall be protected from adventitious contamination.
- 5.1.4 The samples shall be filled in clean and dry glass container. The sample containers shall be of such a size that they almost completely filled by the sample.
- 5.1.5 Each container shall be filled air-tight after filling and marked with full details of sampling date, batch or code number, name of manufacturer, and other important particulars of the consignment.
- 5.1.6 The samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature, and that they are protected from light.

5.2 Scale of sampling:

- 5.2.1 For ascertaining the conformity of the lot to the requirements of this standard, tests shall be carried out on each lot separately. The number of packages and product units from each container respectively to be selected for drawing the sample shall be in accordance with Table 2.

Table 2- Scale of sampling

Number of packages in the lot N	Number of packages to be selected N
4 to 15	3
16 to 40	4
41 to 65	5
66 to 110	7
111 and above	10

Note: When the size of the lot is 3 packages or less, the number of containers to be selected and the criteria for judging the conformity of the to the specification shall be agreed to between the purchaser and the supplier.

- 5.2.2 The packages (cartons) shall be selected at random, using tables of random numbers. If these are not available, the following procedure shall be applied:

Starting from any package, count all the packages in one order as 1, 2, 3 N, selecting every k^{th} package, where k is the integral part of $N \div n$.
From each package thus selected, draw at random an equal number of cakes so as to obtain a total mass of at least 2 kg.

- 5.3.1 **Inspection** - Inspect the cakes selected for compliance with the requirements of 4.1- 4.4.

6.0 PREPARATION OF TEST SAMPLES

6.1 Gross samples

6.1.1 Powders and granules:

From each one of the packages selected in 5.2 above, draw at random one or more containers. The material in the containers so chosen shall be nearly thrice the quantity required for purpose of test.

6.1.1.1 The material from the containers selected as in 6.1.1 shall be disintegrated if necessary, and mixed thoroughly to give the gross sample for the package.

6.1.1.2 Weigh each bar separately (including any material that may have adhered to the wrapper), and calculate the average mass. Keep one bar for the test in clause 4.0 and table 1. Cut each of the remaining bars into eight parts by means of three cuts at right angles to each other through the middle. Grate finely the whole of two diagonally opposite eighths of each specimen. Mix the gratings and place in a clean, dry, airtight glass container.

6.1.2 Pastes

6.1.2.1 From each of the packages selected in 5.2 above draw at random one or more containers. The material in the containers so chosen shall be nearly thrice the quantity required for purposes of test.

6.1.2.2 The material from the containers selected as in 6.1.2.1 shall be mixed thoroughly to give the gross sample for the package.

6.1.3 Segregate carefully the gross sample (in 6.1.1.1 and 6.1.2.2) of powders and pastes. From the gross sample representing each form of synthetic detergent take a small but equal quantity of material and mix thorough into a composite sample which should be of sufficient size to carry out triplicate testing for all the characteristics specified under 6.3. The composite samples representing each form and type of detergent shall be divided into three equal parts- one for the purchaser, another for the supplier, and the third for the referee.

6.1.4 The remaining portion of the material in each gross samples shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the n selected packages shall be for the purchaser, another for the supplier, and the third for the referee.

6.1.5 All the composite and individual samples shall be transferred to separate containers. These containers shall then be sealed air-tight with stoppers and labelled with full particulars of identification given in 5.1.5

6.2 Referee samples

6.2.1 The referee sample shall consist of a composite sample and a set of individual samples. All the containers shall bear the seals of both the purchaser and the supplier, and shall be kept at a place agreed to between the two parties.

6.2.2 Reference samples shall be used in case of any dispute between the purchaser and the supplier.

6.3 Number of tests

6.3.1 Tests for the determination of active ingredient shall be performed on each of the individual samples

6.3.2 The tests for the determination of the remaining characteristics specified in table 1 shall be conducted on the composite sample.

6.4 Criteria for conformity

6.4.1 For individual samples

For the characteristic which has been determined on the individual sample, the mean (X) and the range of test results shall be calculated as follows:

$$\text{Mean (X)} = \frac{\text{The sum of test results}}{\text{Number of test results}}$$

Range (R) = The difference between the maximum and the minimum value of test results.

- a) if the specification limit for the characteristic is given as a minimum, the value of the expression (X – KR) shall be calculated from the relevant test results (see also 6.4.1 d) if the value so obtained is greater than or equal to the minimum limit, the lot shall be declared as conforming to the requirement for the characteristic.
- b) if the specification limit for the characteristic is given as a maximum, the value of the expression (X + KR) shall be calculated from the relevant test results (see also 6.4.1). If the value so obtained is less than or equal to the maximum limit the lot shall be declared as conforming to the requirement for the characteristic.
- c) If the characteristic has two-sided specification limits, then the values of the expression (X ± KR) shall be calculated from the relevant test results (see also 6.4.1 d). If the value so obtained lies between the two specification limits, the lot shall be declared as conforming to the requirements for the characteristic.
- d) The value of the factor K referred to in 6.4.1 (a) to (c) shall be chosen in accordance with Table 3, depending upon the acceptable quality level that is, the percentage, of non-conforming packages that may be tolerated reasonably

Table 3 - Value of 'K' for achieving different acceptable quality levels

Clause 6.4.1 (d) Acceptable quality level	Value of 'K'
Not more than 3.0 % defectives	0.4
Not more than 1.5 % defectives	0.5
Not more than 0.5 % defectives	0.6

7.0 METHODS OF TEST

7.1 Samples of laundry detergent soap shall be tested in accordance with the methods of test referred to in 4.5 and Table 1.

8.0 PACKING AND MARKING

8.1 Packing

8.1.1 The detergent shall be packed in containers that are strong enough to withstand normal handling and transportation and that will prevent leakage and contamination of the product.

8.1.2 The containers may then be packed in bulk packages. Only detergent from the same batch shall be packed in any one container and, when relevant, in any one bulk package.

8.1.3 Marking

Each container and each bulk package shall bear the following information in prominent,- legible and indelible markings:

a) the manufacturer's name or trade mark or both;

b) words indicating that the product is a general purpose detergent;

c) general instructions for use that are suitable for the purposes specified including a cautionary statement to keep the product out of the reach of children NOTE

Prolonged contact with detergents is harmful to the human skin[T4].

;

d) the batch identification (which may be given in code);

e) the mass of the contents; [T5]

f) in the case of bulk packages, the quantity of containers; and

ANNEX A
(Informative)
RINSING PROPERTIES

- A.1** Accurately weigh (to $\pm 0,001$ g) approximately 0,4 g of the test sample (see 6.2) into a thoroughly cleaned 500 mg conical flask and add 200 mg of the standard hard water (see 6.3).
- A.2** Stopper the flask and shake it vigorously for 1 min.
- A.3** Pour out the solution and rinse the flask by adding 200 ml of the standard hard water, shaking vigorously for 1 min and pouring off the water.
- A.4** Invert the flask and allow to dry.
- A.5** Carry out a blank by repeating the above procedure but omitting the test sample.
- A.6** Compare the two flasks.
- A.7** Consider the detergent to comply with 4.5 if the streaks and marks on the flask used for the test do not exceed those on the flask used for the blank.

ANNEX B

METHODS OF TEST FOR SYNTHETIC DETERGENTS

B.1 QUALITY OF REAGENTS

B.1.1 Unless otherwise specified, chemicals of analytical grade and water, distilled quality, in accordance with TZS 59: 1980 (See clause 2), shall be employed in all the tests.

B.2 QUALITATIVE IDENTIFICATION OF NON-SOAPY DETERGENTS

B.2.1 General

It is recommended that the quantitative examination of a sample is preceded by a quantitative identification of the type of non-soapy detergent present. The procedure given in B.2.4 permits ascertaining whether the materials is based on a soap or a non-soapy detergent. The method described in B.2.5 enables the identification of the type of non-soapy detergent, that is, whether it is cationic, anionic or non-anionic.

B.2.2 Apparatus

B.2.2.1 *Measuring cylinder* – 100 ml capacity, glass stoppered.

B.2.2.2 *Test Tubes*

B.2.3 Reagents

B.2.3.1 *Hydrochloric acid* - 50% (ml/v) solution.

B.2.3.2 *Methyl orange indicator solution* – 0.1% (m/v) solution.

B.2.3.3 Methylene blue reagent - Dissolve 0.5 g of methylene blue in distilled water and make up the volume to 100 ml. To 5 ml of this solution, add 120 ml of one mol sulphuric acid and 50 g of anhydrous sodium sulphate and make up the volume to one litre with distilled water.

B.2.3.4 Chloroform

B.2.3.5 Cetyl dimethyl benzyl ammonium chloride solution – 0.2 % (m/v) solution.

B.2.3.6 Sodium lauryl sulphate solution - 0.2% (m/v) solution.

B.2.4 Procedure for identification of soapy and non-soapy detergents

Take about 0.1 g of the sample in a test-tube, add about 20 ml of water and shake well until dissolution is complete. Add a drop of methyl orange indicator solution, and make it just acidic by adding a few drops of hydrochloric acid solution. If the lather is destroyed and fatty acids separate out, then the material is based on a soap. If the lather persists, then the active matter is non-soapy detergent.

B.2.5 Procedure for identification of the type of non-soapy detergents

Dissolve about 0.1 g of the sample in about 20 ml of water, and take 10 ml of this solution in a measuring cylinder. Add 10 ml of methylene blue reagent and 15 ml of chloroform, shake well and allow to stand. Observe whether the colour is in the chloroform layer or the aqueous layer.

B.2.5.1 If the colour is initially in the chloroform layer (see B.2.5) add 0.1 ml of cetyl dimethyl benzyl ammonium chloride solution, and shake well and allow to stand. If the colour is regained in the chloroform layer, the active matter is anionic. If the colour is transferred to the aqueous layer, the active matter is non-ionic.

B.2.5.2 If the colour is initially in the aqueous layer (see B.2.5) add 0.1 ml of sodium lauryl sulphate solution and shake well and allow to stand. If the colour is retained in the aqueous layer, the active matter is cationic. If the colour is transferred to the chloroform layer, the active matter is non-ionic.

B.3 QUALITATIVE IDENTIFICATION OF ALKYL ARYL SULPHONATES

B.3.1 General

The detection depends on (i) the identification of a non-hydrolysable sulphonate and (ii) the presence of an aromatic ring. The active matter is fused with potassium hydroxide to convert it to the corresponding phenol. The phenol is then coupled with diazotized sulphanilic acid and the formation of a red dye confirms its presence.

B.3.2 Apparatus

B.3.2.1 Nickel crucible

B.3.2.2 Separating funnel

B.3.2.3 Wide-mouthed flat-bottomed flask - 150 ml capacity.

B.3.2.4 Steam-bath

B.3.3 Reagents

B.3.3.1 Hydrochloric acid solution - approximately 3 mol.

B.3.3.2 Ethyl ether

B.3.3.3 Sodium hydroxide solution - approximately 2 mol.

B.3.3.4 Sulphanilic acid

B.3.3.5 Sodium nitrite

B.3.4 Procedure

B.3.4.1 Gently fuse a little of the material with solid potassium hydroxide in a nickel crucible, exercising great care to avoid churning. When the melt is quite clear, cool it, dissolve in distilled water, and transfer the solution to a separating funnel. Acidify with hydrochloric acid, extract with ethyl ether and run off the aqueous phase. Wash the other phase with water until free from mineral acid, and then transfer it to a flask. Remove the ether on a steam-bath and dissolve the residue in sodium hydroxide solution.

B.3.4.2 Diazotize 0.05 g of sulphanilic acid by adding 0.1 g of sodium nitrite and a few drops of hydrochloric acid to a solution in water and pour it into the alkaline solution of the phenol (see B.3.4.1). The formation of a red dye indicated the presence of an aromatic ring and shows that the sulphonic acid group was reached to the nucleus.

B.4 DETERMINATION OF MOISTURE AND VOLATILE MATTER CONTENT

B.4.1 General

Moisture and volatile matter is determined by the oven method.

B.4.2 Apparatus

B.4.2.1 Porcelain or silica dish - 6 to 8 cm in diameter and 2 to 4 cm in depth.

B.4.2.2 Desiccator - containing an efficient desiccant such as phosphorus pentoxide

B.4.2.3 Air oven – preferably electrically heated with temperature control device.

B.4.3 Procedure

Weigh accurately about 5 g of the material into a dry tared dish, and dry to constant mass in an air oven at a temperature of $105^0 \pm 1^0\text{C}$. Cool in a desiccator and weigh. Constant mass shall be considered to have been attained when successive heating for one hour periods shows a difference of not more than 5 mg in the net loss in mass.

B.4.4 Calculation

by

$$\text{Moisture and volatile matter content (at } 105^0\text{C) percent} = \frac{(m - m_0)}{m} \times 100$$

m = mass in g of the material taken for the test, and

m_0 = mass in g of the material after drying.

B.5 DETERMINATION OF ACTIVE INGREDIENT

B.5.1 General

Active matter, namely, the sodium salt of sulphonated alkyl benzene is first separated from inorganic salts, non-detergent organic matter and alkylolamide. It is then neutralised to phenolphthalein, evaporated to dryness, extracted with ethyl alcohol, dried and weighed. Finally, the weighed extract is corrected for the presence of sodium chloride and alkali carbonates.

B.5.2 Apparatus

B.5.2.1 Beakers - 150 ml and 1 000 ml capacity.

B.5.2.2 Buchner flask – 500 ml capacity, fitted with a sintered glass filter funnel (porosity 4).

B.5.2.3 Evaporation basin

B.5.2.4 Separating funnel - 1000 ml capacity.

B.5.2.5 Steam-bath

B.5.2.6 Wide-mouthed flat bottomed flask - 200 ml capacity.

B.5.2.7 Air-oven - preferably electrically heated with temperature control device.

B.5.3 Reagents

B.5.3.1 *Ethyl alcohol* - 30%, 90% and absolute (by volume).

B.5.3.2 *Diethyl ether*

B.5.3.3 *Acetone*

B.5.3.4 *Standard Sulphuric acid* - approximately 0.1 mol.

B.5.3.5 *Standard silver nitrate solution* - approximately 0.1 mol

B.5.3.6 *Phenolphthalein indicator* - 1% solution in 95% (by volume) ethyl alcohol.

B.5.3.7 *Methyl orange indicator* - 0.1% (m/v).

B.5.3.8 *Nitric acid* - concentrated sp. gr. 1.42

B.5.3.9 *Nitro-benzene*

B.5.3.10 *Standard ammonium thiocyanate solution* - approximately 0.1 mol

B.5.3.11 *Ferric ammonium sulphate indicator* - saturated solution

B.5.3.12 *Caustic soda solution* - 10% (m/v).

B.5.4 Procedure

B.5.4.1 Removal of inorganic salts

Weigh accurately about 5 g of the material for products containing about 20% active matter and correspondingly less for products of higher active matter content. Proceed as described in the determination of matter insoluble in alcohol (see B. 6.4). After filtering and washing the residue with hot ethyl alcohol, evaporate the combined filtrate to a small bulk in an evaporating basin.

B.5.4.2 Removal of non-detergent organic matter and alkylolamide

B.5.4.2.1 Dilute the evaporated filtrate (see B.5.4.1) with 50 ml of 90% ethyl alcohol and transfer to a separating funnel. Rinse the evaporating basin once with 50 ml of 90% ethyl alcohol and then four times with 50 ml portions of distilled water. Add each wash in turn to the separating funnel. Add 150 ml of diethyl ether. Swirl gently to ensure adequate mixing and allow the two phases to separate. Run off the aqueous alcoholic layer into a second separating funnel, and extract twice with 75 ml portions of diethyl ether. Transfer the aqueous alcoholic phase into a beaker and combine the three ether extracts.

B.5.4.2.2 Take the combined ether extracts in a clean separating funnel. Wash three times with successive 50 ml portions of distilled water until the phase is free from alcohol; usually, 7 to 10 water washes are necessary.

B.5.4.2.3 Combine the washings and rinsings from (B.5.4.2.2.) with the aqueous alcoholic phase obtained in (B.5.4.2.1).

B.5.4.3 **Determination of active matter**

B.5.4.3.1 Transfer the combined aqueous alcoholic phase and washings from B.5.4.2.3 to a porcelain basin. Neutralise to phenolphthalein and evaporate on a steam-bath until the volume is reduced to about 25 ml. Add an equal volume of absolute alcohol and evaporate to dryness. The solution shall remain just pink to phenolphthalein throughout evaporation.

B.5.4.3.2 To ensure that the residue is completely anhydrous add 30 ml of absolute alcohol and again evaporate to dryness. Extract the residue with 30 ml of hot 96% ethyl alcohol, stirring and breaking up the solid matter in the dish with a glass rod. Allow the solid matter to settle and decant the hot alcoholic solution through a sintered glass filter fitted to a Buchner flask to which suction is applied. Extract the residue in the dish with six further consecutive 30 ml portions of hot 96% ethyl alcohol. Pass each extract in turn through the sintered glass filter. Finally, wash the residue in the sintered glass, filter three times with about 20 ml of hot 96% ethyl alcohol from the jet of a wash bottle.

B.5.4.3.3 Transfer the filtrate and washings in the Buchner flask to a tared wide-mouthed flat-bottomed flask, evaporate nearly to dryness on a water-bath and drive off the remaining solvent by directing a gentle stream of dry air into the flask whilst continuously rotating the latter on the water-bath. A thin film of active matter, easy to dry is thereby obtained. Add 10 ml of acetone, evaporate and remove the last trace of solvent as described above. Cool in a desiccator and weigh.

B.5.4.3.4 Heat the flask for not more than five minutes in an air-oven at a temperature of $100 \pm 1^{\circ}\text{C}$, gently blow out with a current of air, cool and re-weigh. Repeat this drying process until the difference between two successive weighings does not exceed 3 mg. Record this mass as *m*.

B.5.4.3.5 The extract obtained in B.5.4.3.4 contains the active matter, some sodium chloride and possibly a trace of alkali carbonates which may have passed through the filter in the presence of the detergent.

B.5.4.4 **Determination of alkali carbonates**

B.5.4.4.1 Dissolve the extract (see B.5.4.3.4) in cold distilled water. Add a few drops of methyl orange indicator and titrate with standard sulphuric acid to the methyl orange end point.

B.5.4.4.2 **Calculation**

mass in g of sodium carbonate, $m_2 = 0.106 VM$

where

V = volume in ml of standard sulphuric acid solution used and

M = molarity of the standard sulphuric acid solution.

B.5.4.4.3 Reserve the solution for the estimation of chlorides

B.5.4.5 **Determination of chlorides**

B.5.4.5.1 To the solution remaining after the estimation of alkali carbonates (see B.5.4.4.3), add 2 ml of concentrated nitric acid and 20 ml of standard silver nitrate

solution. Add 3 ml of nitro-benzene and shake vigorously. Titrate with standard ammonium thiocyanate solution using ferric ammonium sulphate as indicator.

B.5.4.5.2 Calculation

mass in g of sodium chloride

$$m_3 = 0.0585 (20M - VM_1)$$

where

M= Molarity of the standard silver nitrate solution

V= volume in ml of standard ammonium thiocyanate solution used, and

M₁ = molarity of the standard ammonium thiocyanate solution

B.5.4.6 Calculation

$$\text{Active matte, percent by mass} = 100 \times \frac{(m_1 - m_2 - m_3)}{m_0}$$

where

m₁ = mass in g of the alcoholic extract (see B.5.4.3.4).

m₂ = mass in g of sodium carbonate (see B.5.4.4.2).

m₃ = mass in g of sodium chloride (see B.5.4.5.2) and

m₀ = mass in g of the material taken for the test.

B.6 DETERMINATION OF MATTER INSOLUBLE IN ALCOHOL

B.6.1 General

By matter insoluble in alcohol is generally meant the inorganic salts, such as phosphates, sulphates silicates and carbonates which are usually present in non-soapy detergent preparations. These can be separated from active matter, non-detergent organic matter, etc by extracting the material with 96% ethanol.

B.6.2 Apparatus

B.6.2.1 *Beaker* - 150 ml capacity

B.6.2.2 *Steam bath*

B.6.2.3 *Buchner flask* - 500 ml capacity, fitted with sintered glass funnel (porosity 4).

B.6.2.4 *Air-oven* - preferably electrically heated with temperature control device.

B.6.3 Reagents

B.6.3.1 *Ethyl alcohol* - freshly boiled 96% or higher by volume.

B.6.4 Procedure

B.6.4.1 Weigh accurately about 5 g of the material into a beaker, and digest with 50 ml of ethyl alcohol by heating on a steam-bath for about 2 minutes. Stir and break up any hard lump with a glass rod flattened at one end. Allow the solid matter to settle and decant the

hot alcoholic solution through a sintered glass filter funnel fitted to a Buchner flask to which suction is applied. Repeat the alcoholic digestion in a similar manner with 5 further consecutive 30 ml portions of boiling ethyl alcohol. Filter each extract a turn through the same sintered glass funnel and finally wash the residue several times with hot ethyl alcohol to remove all the alcohol solubles. Dry the sintered glass funnel with the residue in an air-oven at a temperature of $105 \pm 2^{\circ}\text{C}$ until a constant mass is obtained.

B.6.4.2 Even after digestion with five 30 ml portions of boiling ethyl alcohol, the alcohol insoluble portion may sometimes be found to be sticky. In that case, treat it further with more boiling ethyl alcohol until it is free from active matter and the alcohol insoluble portion is no longer sticky.

B.6.5 Calculation

$$\text{matter insoluble in alcohol, per cent by mass} = \frac{m_1}{m} \times 100$$

where

m_1 = mass in g of matter insoluble in alcohol, and

m = mass in g of material taken for the test.

B.7 DETERMINATION OF PHOSPHATES IN MATTER INSOLUBLE IN ALCOHOL

B.7.1 General

The sample is oxidised by gently heating with sodium nitrate. Silica is removed and the condensed phosphates are hydrolysed and precipitated as ammonium phosphomolybdate by the addition of ammonium molybdate. The precipitate is washed with dilute potassium nitrate solution and the phosphorus in the washed precipitate is determined by titration with standard sodium hydroxide solution using phenolphthalein as an indicator.

B.7.2 Apparatus

B.7.2.1 *Silica dish* – 7 cm diameter

B.7.2.2 *Beaker* – 250 ml capacity

B.7.2.3 *Buchner flask* - 500 ml capacity with a sintered glass filter funnel.

B.7.2.4 *Volumetric flask* – 500 ml capacity.

B.7.2.5 *Funnel* - 7.5 cm diameter

B.7.2.6 *Wide-mouthed flat-bottomed flask* - 500 ml capacity.

B.7.3 Reagents

B.7.3.1 *Sodium nitrate*

B.7.3.2 *Hydrochloric acid* - (i) concentrated and (ii) 1:1 dilution.

B.7.3.3 *Ammonium molybdate reagent*

B.7.3.4 *Nitric acid* - sp. gr. 1.4

B.7.3.4.1 *Preparation of ammonium molybdate reagent*

Dissolve 90 g of ammonium molybdate in hot distilled water. Add 240 g of ammonium nitrate and stir to dissolve. Cool and add 30 ml of concentrated ammonia solution (sp. gr. 0.880). Dilute to 1 litre.

B.7.3.5 *Potassium nitrate* - 1.25% solution in distilled water.

B.7.3.6 *Sodium hydroxide* - 1.0 mol accurately standardised.

B.7.3.7 *Sulphuric acid* - 1.0 mol accurately standardised.

B.7.3.8 *Phenolphthalein solution* - one per cent solution m/v in ethyl alcohol.

B.7.4 Procedure

Accurately weigh about 1.5 of the sample in a silica dish and add a small amount of sodium nitrate, mix well and heat gently over a Bunsen burner until the sample is completely oxidised. Cool and add 15 ml of concentrated hydrochloric acid and evaporate to dryness. Add further 15 ml of concentrated hydrochloric acid and repeat the evaporation procedure. Finally extract the residue in 25 ml of 1:1 hydrochloric acid and filter through sintered glass crucible. Wash the crucible with 25 ml of dilute hydrochloric acid and then wash 4 times with 50 ml of distilled water. Collect the filtrate and washings and make up to 250 ml in a volumetric flask.

Pipette 50 ml aliquot from the volumetric flask in 250 ml beaker. Add 10 ml of nitric acid and boil for 15 minutes. Cool and add 100 ml of distilled water and adjust the temperature of the solution to between 40 and 45 °C. Add 50 ml of ammonium molybdate solution (previously heated to 40 °C) slowly with constant stirring. Allow to stand for 30 minutes. Filter the precipitate through a quantitative filter paper and wash with 1.25% potassium nitrate solution till 5 ml of the filtrate with 1 drop of phenolphthalein does not require more than 3 to 5 drops of 0.1 mol caustic potash to produce pink colour. Transfer the filter paper with the precipitate to 500ml wide-mouth flat-bottom flask and add 100 ml of distilled water. Heat over a water-bath for 15 minutes, cool and titrate with 1 mol sodium hydroxide using 1 ml of phenolphthalein till the pink colour just appears. Add 2 ml in excess. Shake well, heat to 60°C in a water-bath. Cool and back titrate against 1 mol sulphuric acid till the pink colour just disappears. Note the volume of normal sodium hydroxide to react with the precipitate.

B.7.5 Calculation

$$\text{Total phosphorus as tripolyphosphate on the original sample X, percent} = \frac{V \times M \times 0,001 \times 349 \times 100 \times 368}{M \times 50 \times 93}$$

B.7.5.1 Total phosphorus as tripolyphosphate on the original sample X, per cent.

Where

V = volume of sodium hydroxide required to react with the precipitate

M = Molarity of sodium hydroxide, and

m = mass of sample taken for test.

B.7.5.2 Total phosphorus as tripolyphosphate on alcohol insoluble per cent

$$\text{Total phosphorus as tripolyphosphate on alcohol insoluble per cent} = \frac{100 \times X}{Y}$$

Where

X = total phosphorus as tripolyphosphate in the original sample per cent

Y = matter insoluble in alcohol per cent.

B.8 DETERMINATION OF pH

B.8.1 General

pH determination should be made in an acid-free atmosphere.

B.8.2 Apparatus

B.8.2.1 pH Meter Any standard electrometric instrument, equipped with a low sodium error glass electrode. The instrument shall be calibrated and standardised with standard buffer solutions (see B. 8.3.2) before use.

B.8.2.2 Volumetric flask - 1 000 ml capacity

B.8.3 Reagents

B.8.3.1 Distilled Water

Distilled water shall be boiled thoroughly or purged with carbon dioxide-free air to remove carbon dioxide and shall be protected with soda lime or soda asbestos while cooling and in storage. The pH of this water shall be between 6.2 and 7.2 at 30 °C. The residue on evaporation when heated at 105 °C for one hour shall not exceed 0.5 mg per litre.

B.8.3.2 Standard buffer solution

Any two suitable buffer solutions within the pH range of 9 to 11 at 30 °C for calibrating the pH meter.

B.8.4 Procedure

Weigh 10 ± 0.001 g of the material and transfer to a 1 litre volumetric flask. Partially fill the flask with distilled water and agitate until the sample is completely dissolved. Adjust the temperature of the solution and the distilled water to 30 ± 0.5 °C and fill to the calibration mark with distilled water. Stopper the flask, mix thoroughly, and allow the solution to stand at a temperature of 30 °C for two hours prior to measuring the pH. Measure the pH of the solution using a glass electrode.

B.9 DETERMINATION OF NON-DETERGENT ORGANIC MATTER

B.9.1 General

The term non-detergent organic matter includes hydrocarbons, fatty alcohols and perfumes. Using petroleum ether and under the conditions prescribed, non-detergent organic matter only is extracted leaving any alkylamide present in the material..

B.9.2 Apparatus

B.9.2.1 Evaporating basin

B.9.2.2 Separating funnels - 1 000 ml capacity

B.9.2.3 Wide-mouthed flat bottomed flask - 200 ml capacity

B.9.2.4 Buchner flask - 500 ml capacity, fitted with a sintered glass filter funnel (porosity 4).

B.9.3 Reagents

B.9.3.1 *Ethyl alcohol* - 50%, 70%, 90% and 96% (by volume),

B.9.3.2 *Petroleum ether* - Boiling range 40 to 60 °C non-volatile residue at 80° C maximum 0.001%.

B.9.4 Procedure

B.9.4.1 *Removal of inorganic salts*

Weigh accurately about 5 g of the material in 150 ml squat beaker. Extract with 50 ml of hot 90% ethanol by heating on the steam bath for about 2 minutes stirring and breaking up any hard lumps with a glass rod flattened at the end.

B.9.4.1.1 Allow the solid matter to settle and decant the hot alcoholic solution through a sintered glass filter funnel (porosity 4) fitted to a 500 ml Buchner flask to which suction is applied. Repeat the extraction in a similar manner with five further consecutive 30 ml quantities of boiling 90% ethanol. Pass each extract in turn through the filter into the flask.

B.9.4.2 Transfer quantitatively all the combined filtrate from the Buchner flask to 1 litre separating funnel and rinse the flask four times with 40 ml quantities of distilled water, transferring each wash in turn to the separating funnel. Add 100 ml of petroleum ether, swirl gently to ensure adequate mixing and allow the two phases to separate. Run off the aqueous alcoholic layer into a second separating funnel, and extract with 75 ml of petroleum ether. Combine the three ether extracts in the first separating funnel. Rinse each of the two empty funnels with a few millilitres petroleum ether and add the rinsings to the combined ether extracts.

B.9.4.3 Wash the combined ether extracts and rinsings (see B.9.4.2) with four successive 50 ml portions of 70% ethyl alcohol shaking and removing the alcoholic phase each time. Transfer the ether layer in stages to a tared flask and evaporate off the solvent. Add 10 ml of acetone and evaporate off the solvent. Rotate the flask on a steam-bath during the operation. Cool the flask to about 60 to 65 °C, gently blow out the last traces of solvent with a current of dry air, cool in a desiccator and weigh.

B.9.5 Calculation

$$\text{Non-detergent organic matter percent by mass} = \frac{m_1}{m} \times 100$$

Where

m_1 = mass in g of the non-detergent organic matter in the flask, and

m = mass g of the material taken for the test

B.10 DETERMINATION OF MATTER INSOLUBLE IN WATER

B.10.1 Procedure

Starting with a fresh portion of the material, proceed as described under B.6.4 but do not dry or weigh the matter insoluble in alcohol. After filtering and washing the residue thoroughly with hot ethyl alcohol, change the receiver extract the residue with successive portions of distilled water at about 60 °C and wash the residue several times to remove all the water solubles. Dry the sintered glass funnel with the residue in an air-oven at a temperature of 105 ± 2 °C until a constant mass is obtained.

B.10.2 Calculation

$$\text{matter insoluble in water, percent by mass} = \frac{m_1}{m} \times 100$$

where

m_1 = mass in g of matter insoluble in water, and

m = mass in g of material taken for the test.

B.11 DETERMINATION OF CARBOXYMETHYLCELLULOSE

B.11.1 Principle

The method is based on hydrolysis and dehydration of the carboxymethylcellulose to furfural derivatives which produce a green colour with a solution of anthrone in 60% sulphuric acid. The method is not specific for carboxymethylcellulose as most cellulose derivatives and also other carbohydrates such as sucrose, react similarly. The intensity of the colour depends upon the degree of substitution of the carboxymethylcellulose, being less intense with higher degrees and it is essential to prepare a standardisation graph from similar material to that in the sample, or to express the result in terms of an arbitrary standard.

B.11.2 Reagents

The reagents shall be of recognised analytical reagent quality. Distilled water or water of at least equal purity shall be used.

- a) Kieselguhr
- b) Sulphuric acid, concentrated, sp. gr. – 1.84
- c) Sulphuric acid, dilute. Add 60 ml of the concentrated sulphuric acid slowly to 40 ml of water, cooling well dilute to 100 ml with water when cold.
- d) Anthrone solution. Dissolve 0.2 g of anthrone in about 50 ml of the concentrated sulphuric acid, which has been added to 5 ml of water. Cool and dilute to 200 ml with the concentrated sulphuric acid. Allow the solution to stand for four hours before use. Do not use a solution that is more than 24 hours old.

Before use, dilute 60 ml of the above solution to 100 ml, adding the strong solution to the requisite amount of water, cooling well and diluting to volume with water when cold.

B.11.3 Apparatus

- a) One mark volumetric flask, of capacity 50, 100, and 200 ml.
- b) Filter sintered glass porosity No. 4
- c) Spectrophotometer, set to read at 550 m μ for absorptiometer with suitable filter, and 1 cm glass cell.

B.11.4 Procedure

Dissolve 1 g of the surface-active agent in the diluted sulphuric acid and dilute to 100 ml with this acid. If per-salts are present, heat the weighed sample for two hours in an oven at 150 °C, then allow to cool before dissolving in the acid. Filter the solution through the sintered glass filter, using a little kieselguhr to aid filtration. Mix 5 ml of the filtered solution in a 50 ml one-mark volumetric flask with 30 ml of the anthrone solution. Heat

the mixture in a boiling water bath for 15 minutes. Cool and dilute to the mark with the diluted sulphuric acid. Measure the optical density of the solution in a 1 cm cell at 625 m μ against a blank prepared from 30 ml of the reagent diluted to 50 ml with the diluted sulphuric acid.

Prepare a standardising graph from figures obtained by treating known amounts of carboxymethylcellulose by the above procedure.

B.11.5 Calculation

Calculate the percentage of carboxymethylcellulose in the sample by reference to the graph.

ANNEX C

Informative

Bibliography

SABS 892, Specification for general purpose detergent (granules and powders)

TZS 39: 1979 Synthetic detergent for household - Specification