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PROJET DE NORME CAMEROUNAISE PNC 3209 : 2026 - CT 28

2026

SYNTHETIC DETERGENT POWDERS — PART 2: COMPOSITION-BASED HOUSEHOLD HAND WASH

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PROJET DE NORME CAMEROUNAISE

ENQUETE PUBLIQUE N° : 19

Durée de l'enquête Du 21/09/2026 Au 19/10/2026

Edition et diffusion par l'Agence des Normes et de la Qualité
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DN-R2/PR-01/ENR-02
16/12/2015

**Synthetic detergent powders — Part 2: Composition-based
household hand wash**



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Introduction

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

Synthetic detergents are of three types: anionic, cationic and non-ionic. Anionic synthetic detergents of the alkyl aryl type, such as dodecylbenzene sulfonic acid, are now produced in fairly large quantities and hence priority has been given for the standardization of this material.

It has been discovered that equally good cleaning can be achieved at relatively low detergent levels. With the advance of technology, other equally effective, environmentally safe and cheaper builders can be used instead of Sodium Tripolyphosphate (STPP) which is currently being used. Phosphates have been known to bring eutrophication in water bodies and companies world-over are doing away with them and encouraging use of alternative builders such as zeolites and carbonates, and therefore for this reason the requirement for levels of STPP are being progressively phased out in the standard with a view of eliminating them in the near future.

The standard is more composition based than performance based and for that reason performance measures for cleaning efficiency and detergency tests have been included. This edition emphasizes on the biodegradability of the surfactants used to manufacture detergent powders and a method for determining the same has been developed.

ENQUÊTE PUBLIQUE N° 19

Synthetic detergent powders — Part 2: Composition-based household hand wash

1 Scope

This African Standard specifies the requirements and methods of sampling and test for synthetic detergents for household use.

This standard does not cover machine wash and industrial detergent powders.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 672, *Soaps — Determination of moisture and volatile matter content — Oven method*

ISO 673, *Soaps — Determination of content of ethanol-insoluble matter*

ISO 2870, *Surface active agents — Detergents — Determination of anionic-active matter hydrolysable and non-hydrolysable under acid conditions*

ISO 2871-1, *Surface active agents — Detergents — Determination of cationic-active matter content — Part 1: High-molecular-mass cationic-active matter*

ISO 2871-2, *Surface active agents — Detergents — Determination of cationic-active matter content — Part 2: Cationic-active matter of low molecular mass (between 200 and 500)*

ISO 4313, *Washing powders — Determination of total phosphorous (V) oxide content — Quinoline phosphomolybdate gravimetric method*

ISO 4316, *Surface active agents — Determination of pH of aqueous solution — Potentiometric method*

ISO 4317, *Surface-active agents and detergents — Determination of water content — Karl Fischer methods*

3 Definitions and abbreviations

For the purpose of this standard the following definitions apply.

3.1

batch

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

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

4 Requirements

4.1 General requirements

4.1.1 The active ingredient shall be the anionic, cationic or non-ionic. The formulation may contain one or more of the builders or additives given in Annex A. The active ingredient used shall be biodegradable.

4.1.2 The detergent shall be homogenous, free flowing powder, free from visible impurities, abrasives and organic solvents and shall be readily soluble in water.

4.1.3 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.

4.1.4 The material shall be a free flowing powder, and shall not give an unpleasant odour and shall give good lather.

4.1.5 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 objectionable odour. Perfume may be added, in which case, the perfume shall not change its fragrance nor develop an objectionable odour during storage at ambient temperature for six months.

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

4.2 Specific quality requirements

The product shall also comply with the specific quality requirements given in Table 1, when tested by the appropriate method prescribed.

Table 1 — Specific quality requirements for synthetic detergents for household use

S/No	Characteristics	Requirements	Method of test
(i)	Moisture and volatile matter content at $150\text{ }^{\circ}\text{C}$, % by mass, max	13	ISO 672 ISO 4317
(ii)	Active ingredient, % by mass, min	12	ISO 2870 ISO 2871-1 ISO 2871-2
(iii)	Matter insoluble in alcohol, % by mass, max	80	ISO 673
(iv)	Phosphate (expressed as sodium tripoly-phosphate), % by mass of matter insoluble in alcohol, min	15	ISO 4313
(v)	pH of 1 % solution (m/v), at $25 \pm 2\text{ }^{\circ}\text{C}$	9 to 11	ISO 4316
(vi)	Non-detergent organic matter, % by mass, max	1.0	Annex B
(vii)	Matter insoluble in water ^{a)} , % by mass, max	0.5	Annex C

^{a)} For the case of non-phosphate based laundry detergents, MIW shall be 15 % by mass maximum.

4.3 Storage properties

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

5 Packaging and marking

5.1 Packaging

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.

5.2 Marking

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

- a) name of the product as "synthetic detergent powder";
- b) manufacturer's name and physical address;

NOTE The name, physical address of the distributor/supplier and trade mark may be added as required.

- c) batch or code number;
- d) net content;
- e) country of origin;
- f) instructions for use;
- g) date of manufacture; and
- h) best before date.

6 Sampling

Sampling shall be done in accordance with Annex D.

**Annex A
(informative)**

List of suggested builders and additives

The following is a list of suggested builders and additives:

- a) trisodium phosphate;
- b) sodium carbonate;
- c) sodium sulfate;
- d) tetrasodium pyrophosphate;
- e) sodium tripolyphosphate;
- f) sodium hexametaphosphate;
- g) sodium carboxy methyl cellulose;
- h) sodium silicate;
- i) optical brighteners;
- j) lather boosters;
- k) hydrotopes;
- l) perfumes;
- m) preservatives;
- n) chelating agents (sequestering agents);
- o) colours;
- p) perborates;
- q) enzymes;
- r) bactericides;
- s) zeolites;
- t) common salt;
- u) urea;
- v) magnesium sulphate;
- w) polymers; and
- x) any other internationally accepted builder cleared by the national standards body in the respective country.

Annex B (normative)

Determination of non-detergent organic matter

B.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 alkylolamide present in the material.

B.2 Apparatus

B.2.1 Evaporating basin

B.2.2 Separating funnels, 1 000-mL capacity

B.2.3 Wide mouthed flat-bottomed flask, 200-mL capacity

B.2.4 Buchner flask, 500-mL-capacity fitted with a sintered glass filter funnel (porosity 4)

B.3 Reagents

B.3.1 Ethyl alcohol, 50 %, 70 %, 90 % and 96% (by volume)

B.3.2 Petroleum ether, boiling range 40 °C to 60 °C non-volatile residue at 80 °C maximum 0.001%

B.3.3 Acetone, non-volatile residue at 80 °C maximum 0.001 %

B.4 Procedure

B.4.1 Weigh accurately about 5 g of the material in a 150-mL squat beaker. Extract with 50 mL of hot 90 % ethanol by heating on the steam bath for about 2 min stirring and breaking up any hard lumps with a glass rod flattened at the end.

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.4.2 Transfer quantitatively all the combined filtrate from the Buchner flask to a 1-L 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. Repeat the extraction of the aqueous alcoholic phase in the third separating funnel with a further 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 rinsing to the combined ether extracts.

B.4.3 Wash the combined ether extracts and rinsing (see B.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 °C to 65 °C, gently blow out the last traces of solvent with a current of dry air, cool in a desiccator and weigh.

B.5 Calculation

The non-detergent organic matter is expressed as follows:

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

m_1 is the mass, in grams, of the non-detergent organic matter in the flask; and

m is the mass, in grams, of the material taken for the test.

Annex C (normative)

Determination of matter insoluble in water

C.1 Procedure

C.1.1 Starting with a fresh portion of the material, 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 min. 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 five further consecutive 30-ml portions of boiling ethyl alcohol. Filter each extract in 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 $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ until a constant mass is obtained.

C.1.2 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. 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 $60\text{ }^{\circ}\text{C}$, 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 $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ until a constant mass is obtained.

C.2 Calculation

The matter insoluble in water is expressed as follows:

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

where

m_1 is the mass, in grams, of matter insoluble in water; and

m is mass, in grams, of material taken for the test.

Annex D (normative)

Sampling procedure for powder detergents

D.1 General requirements

D.1.1 In drawing preparing, storing and handling samples, the precautions in D.1.2 – D.1.7 shall be observed.

D.1.2 Samples shall not be taken from places exposed to damp air, dust or soot.

D.1.3 The sampling instruments shall be clean and dry when used.

D.1.4 The samples, the material being sampled, the sampling instruments and the containers for samples shall be protected from adventitious contamination.

D.1.5 The samples shall be placed in clean and dry glass containers. The sample containers shall be of such a size that they are almost completely filled by the sample.

D.1.6 Each container shall be sealed airtight after filling, and marked with full details of sampling which include, date of sampling, batch or code number, name of manufacturer, and other important particulars of the consignment.

D.1.7 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.

D.2 Scale of sampling

D.2.1 In a single consignment, all the packages containing non-soapy detergents of the same type and form, and drawn from the same batch of manufacture, shall constitute a lot. If the consignment consists of packages containing non-soapy detergents of different types and forms, then the packages containing non-soapy detergents of the same type form and batch of manufacture shall be grouped together, and such group shall constitute a separate lot.

D.2.2 For ascertaining the conformity of the lot to the requirements prescribed in this standard, tests shall be carried out on each lot separately. The number (n) of packages to be selected for drawing the samples shall depend upon the size (N) of the lot and shall be in accordance with Table D.1.

Table D.1 — Scale of sampling

No. 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 lot to the specifications should be as agreed on between the purchaser/inspector and the supplier.	

D.2.3 The packages shall be selected at random and to ensure randomness of selection, a random

number table shall be used. In case such tables are not available, the procedure given below may be adopted.

Starting from any package, count all the packages in one order as 1, 2, 3, ..., up to r and so on, where r is the integral part of N/n , (N being the lot size and n the number of packages to be selected). Every r^{th} package thus counted shall be withdrawn to give a sample for the purposes of test.

D.3 Preparation of gross samples, test sample and reference sample

D.3.1 Gross sample

From each one of the packages selected as in D.2, 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 as indicated in D.4.

The powder from the containers selected shall be disintegrated, if aggregated, and mixed thoroughly to give the gross sample for the package.

D.3.2 Test sample

D.3.2.1 Segregate carefully the gross samples of powders. From the gross representing each form of synthetic detergent take a small but equal quantity of material and mix thoroughly into a composite sample which should be of a size sufficient to carry out triplicate testing for all the characteristics specified under D.4. The composite samples representing each form and type of synthetic detergent shall be divided into three equal parts, one for the purchaser/inspector, another for the supplier, and the third for the referee.

D.3.2.2 The remaining portion of the material in each of the gross samples shall be divided into three equal pans, each forming an individual sample. One set of individual samples, representing the n selected packages shall be for the purchaser/inspector, another for the supplier, and the third for reference.

D.3.2.3 All the composite and individual samples shall be transferred to separate containers. These containers shall then be sealed airtight with stoppers, and labelled with full particulars of identification given in D.1.6.

D.3.3 Reference samples

D.3.3.1 The reference samples shall consist of a composite sample and a set of individual samples. All the containers shall bear the seals of both the purchaser/inspector and the supplier, and shall be kept at a place agreed to between the two parties.

D.3.3.2 Reference samples shall be used in case of any dispute between the purchaser/inspector and the supplier.

D.4 Number of tests

D.4.1 Tests for the determination of active ingredient shall be performed on each of the individual samples.

D.4.2 Tests for the determination of other requirements specified in Table 1 shall be conducted on the composite sample.

D.5 Criteria for conformity

D.5.1 For individual samples

D.5.1.1 For the characteristic, which has been determined on the individual sample, the mean ($\bar{X}$) and the range (R) of test results shall be calculated as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{Sum of test results}}{\text{Number of test results}}$$

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

D.5.1.2 If the specification limit for the characteristic is given as a minimum, the value of the expression ($\bar{X} - KR$) shall be calculated from the relevant test results (see also D.5.1.5). 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.

D.5.1.3 If the specification limit for the characteristic is given as a maximum, the value of the expression ($\bar{X} + KR$) shall be calculated from the relevant test results (see also D.5.1.5). 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.

D.5.1.4 If the characteristic has two-sided specification limits, then the values of the expression ($\bar{X} \pm KR$) shall be calculated from the relevant test results (see also D.5.1.5). If the value so obtained lies between the two specification limits, the lot shall be declared as conforming to the requirement for the characteristic.

D.5.1.5 The value of the factor K referred to in D.5.1.2 - D.5.1.4 shall be chosen in accordance with Table D.2, depending upon the acceptable quality level, that is, the percentage, of non-conforming packages that may be tolerated reasonably.

Table D.2 — Value of 'K' for achieving different acceptable quality levels

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

D.5.2 For composite sample

For declaring the conformity of the lot to the requirements of all the remaining characteristics determined on the composite sample, the test results for each one of the characteristics shall satisfy the relevant requirement given in Table 1 of this standard

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