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ANNEXES 1 to 8

ANNEXES

to the

proposal for a Regulation of the European Parliament and of the Council

on detergents and surfactants, amending Regulation (EU) 2019/1020 and repealing Regulation (EC) No 648/2004

{SEC(2023) 170 final} - {SWD(2023) 113 final} - {SWD(2023) 114 final} -
{SWD(2023) 115 final}

ANNEX I

BIODEGRADABILITY REQUIREMENTS REFERRED TO IN ARTICLE 4

ULTIMATE BIODEGRADABILITY CRITERIA AND TEST METHODS FOR SURFACTANTS AND SURFACTANTS IN DETERGENTS

1. The reference method for laboratory testing of surfactant ultimate biodegradability in this Regulation is based on the EN ISO standard 14593: 1999 (CO_2 headspace test).

2. Surfactants and surfactants contained in detergents shall be ultimately biodegradable as determined in accordance with the criteria laid down in point 3.

3. Surfactants and surfactants contained in detergents shall be considered as ultimately biodegradable if they meet one of the following criteria:

   (a) the level of biodegradability (mineralisation) is at least 60 % within 28 days measured in accordance with one of the following test methods:

      (i) EN ISO Standard 14593: 1999 — Water quality — Evaluation of ultimate aerobic biodegradability of organic compounds in aqueous medium — Method by analysis of inorganic carbon in sealed vessels (CO_2 headspace test);

      (ii) method C.4.-C Carbon dioxide (CO_2) Evolution Test (Modified Sturm Test), described in Part C, Part IV, of the Annex to Commission Regulation (EC) No 440/2008[1];

      (iii) method C.4-D, manometric respirometry test, described in Part C, Part V, of the Annex to Regulation (EC) No 440/2008;

      (iv) method C.4-E, closed bottle test, described in Part C, Part VI, of the Annex to Regulation (EC) No 440/2008;

      (v) method C.4-F Ministry of International Trade and Industry, Japan (M.I.T.I.) described in Part C, Part VII, of the Annex to Regulation (EC) No 440/2008;

      (vi) ISO 10708: 1997 — Water quality — Evaluation in an aqueous medium of the ultimate aerobic biodegradability of organic compounds — Determination of biochemical oxygen demand in a two-phase closed bottle test.

   (b) the level of biodegradability (mineralisation) is at least 70% within 28 days measured in accordance with one of the following test methods:

      (i) method C.4-A DOC die-away test described in Part C, Part II, of the Annex to Regulation (EC) No 440/2008;

      (ii) method C.4-B, modified OECD screening test described in Part C, Part III, of the Annex to Regulation (EC) No 440/2008.

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[1] Commission Regulation (EC) No 440/2008 of 30 May 2008 laying down test methods pursuant to Regulation (EC) No 1907/2006 of the European Parliament and of the Council on the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH) (OJ L 142, 31.5.2008, p. 1).

Pre-adaptation shall not be used and the 10-day window principle shall not be applied in any of the test methods referred to in points (a) and (b) .

4. The tests referred to in point 3 shall be conducted by laboratories meeting any of the following conditions:

(a) the laboratories are complying with the principles of good laboratory practice provided for in Directive 2004/10/EC of the European Parliament and of the Council[2] or international standards recognised as being equivalent;

(b) the laboratories are accredited in accordance with the standard for laboratories referred to in Regulation (EC) No 765/2008.

[2] Directive 2004/10/EC of the European Parliament and of the Council of 11 February 2004 on the harmonisation of laws, regulations and administrative provisions relating to the application of the principles of good laboratory practice and the verification of their applications for tests on chemical substances (OJ L 50, 20.2.2004, p. 44).

ANNEX II

REQUIREMENTS FOR DETERGENTS CONTAINING MICROORGANISMS REFERRED TO IN ARTICLE 5

1. Micro-organisms intentionally added to detergents shall comply with the following conditions:

   (a) shall have an American Type Culture Collection (ATCC) number, belong to a collection of an International Depository Authority (IDA) or have had their DNA identified in accordance with a "Strain identification protocol" (using 16S ribosomal DNA sequencing or an equivalent method);

   (b) shall belong to both of the following:

      (i) Risk Group I as defined by Directive 2000/54/EC – biological agents at work;

      (ii) The Qualified Presumption of Safety (QPS) list issued by the European Food Safety Authority (EFSA).

   This point shall not apply to micro-organisms intentionally added to detergents placed on the market for research and development purposes.

2. The following pathogenic micro-organisms shall not be present in any of the strains included in the finished product when screened using the indicated test methods or equivalent:

   (a) *E. coli*, test method ISO 16649-3:2005;

   (b) *Streptococcus (Enterococcus)*, test method ISO 21528-1:2004;

   (c) *Staphylococcus aureus*, test method ISO 6888-1;

   (d) *Bacillus cereus*, test method ISO 7932:2004 or ISO 21871;

   (e) *Salmonella*, test method ISO 6579:2002 or ISO 19250.

3. Intentionally added micro-organisms shall not be genetically modified microorganisms.

4. Intentionally added micro-organisms shall be, with the exception of intrinsic resistance, susceptible to each of the major antibiotic classes, namely aminoglycoside, macrolide, beta-lactam, tetracycline and fluoroquinolones, in accordance with the European Committee on Antimicrobial Susceptibility Testing (EUCAST) disk diffusion method or equivalent.

5. When placed on the market, detergents containing micro-organisms shall have a standard plate count equal to or greater than $1x10^5$ colony-forming units (CFUs) per ml in accordance with ISO 4833-1:2014.

6. The minimum shelf life of a detergent containing micro-organisms shall not be lower than 24 months and the microbial count shall not decrease by more than 10 % every 12 months in accordance with ISO 4833-1:2014.

7. Micro-organisms contained in detergents that are placed on the market in a spray format shall pass the acute inhalation toxicity test in accordance with the test method B.2., described in Part B of the Annex to Regulation (EC) No 440/2008.

8. Detergents containing micro-organisms shall not be placed on the market in a refill format.

9. All claims made by the manufacturer regarding the actions of the micro-organisms contained in the product shall be supported by third-party testing.

10. It is prohibited to claim or suggest on the label or by any other communication that the detergent has an antimicrobial or disinfecting effect, unless the detergent complies with Regulation (EU) No 528/2012.

11. The tests referred to in points 2, 5, 6, 7 and 9 shall be conducted by laboratories meeting any of the following conditions:

(a) the laboratories are complying with the principles of good laboratory practice provided for in Directive 2004/10/EC of the European Parliament and of the Council[3] or international standards recognised as being equivalent;

(b) the laboratories are accredited in accordance with the standard for laboratories referred to in Regulation (EC) No 765/2008.

[3] Directive 2004/10/EC of the European Parliament and of the Council of 11 February 2004 on the harmonisation of laws, regulations and administrative provisions relating to the application of the principles of good laboratory practice and the verification of their applications for tests on chemical substances (OJ L 50, 20.2.2004, p. 44).

ANNEX III

LIMITATIONS ON THE CONTENT OF PHOSPHATES AND OTHER PHOSPHORUS COMPOUNDS REFERRED TO IN ARTICLE 6

Detergent	Limitations
Consumer laundry detergents	Shall not be placed on the market if the total content of phosphorus is equal to or greater than 0,5 grams in the recommended quantity of the detergent to be used in the main cycle of the washing process for a standard washing machine load as defined in Part B of Annex V for hard water: – for 'normally soiled' fabrics in the case of heavy-duty detergents, – for 'lightly soiled' fabrics in the case of detergents for delicate fabrics.
Consumer automatic dishwasher detergents	Shall not be placed on the market if the total content of phosphorus is equal to or greater than 0,3 grams in the standard dosage as defined in Part B of Annex V.

ANNEX IV

CONFORMITY ASSESSMENT PROCEDURE REFERRED TO IN ARTICLE 7(2)

Module A - Internal production protocol

1. Description of the module

Internal production control is the conformity assessment procedure whereby the manufacturer fulfils the obligations laid down in points 2, 3 and 4, and ensures and declares on his or her sole responsibility that the detergent or surfactant concerned satisfy the requirements of this Regulation that apply to them.

2. Technical documentation

2.1. The manufacturer shall establish the technical documentation. The documentation shall make it possible to assess conformity of the detergent or surfactant with the relevant requirements, and shall include an adequate analysis and assessment of the risks.

2.2. The technical documentation shall specify the applicable requirements and cover, as far as relevant for the assessment, the design, manufacture and intended use of the detergent or surfactant. The technical documentation shall contain, where applicable, at least the following elements:

(a) a general description of the detergent or surfactant and a description of the intended use;

(b) the test reports demonstrating the compliance with Annex I and, where applicable, with Annexes II and III;

(c) a list of test methods used to demonstrate compliance with the requirements of this Regulation ;

(d) results of calculations made and examinations carried out;

(e) an ingredient data sheet which meets the following requirements:

(i) lists all intentionally added substances and preservatives referred to in Part A of Annex V;

(ii) the common chemical name or IUPAC name and, where available, the INCI name, the CAS number, and the European Pharmacopoeia name, is given for each ingredient;

(iii) all substances are listed in order of decreasing abundance by weight, and the list is sub-divided into the following weight percentage ranges:

(1) 10 % or more,

(2) 1 % or over, but less than 10 %,

(3) 0,1 % or over, but less than 1 %,

(4) less than 0,1 %.

For the purposes of point (e), a perfume, an essential oil, or a colouring agent shall be considered to be a single component.

3. Manufacturing

The manufacturer shall take all measures necessary so that the manufacturing process and its monitoring ensure compliance of the detergent or surfactant with the technical documentation referred to in point 2 and with the requirements of this Regulation that apply to them.

ANNEX V

LABELLING REQUIREMENTS

PART A – LABELLING OF CONTENTS

The information to be included on the labels of detergents and surfactants made available on the market

1. The weight percentage ranges ‘less than 5 %’, ‘5 % or over but less than 15 %’, ‘15 % or over but less than 30 %’, ‘30 % and more’, shall be used to indicate the content of the constituents listed below where they are added in a concentration above 0,2 % by weight:
   (a) phosphates,
   (b) phosphonates,
   (c) anionic surfactants,
   (d) cationic surfactants,
   (e) amphoteric surfactants,
   (f) non-ionic surfactants,
   (g) oxygen-based bleaching agents,
   (h) chlorine-based bleaching agents,
   (i) EDTA and salts thereof,
   (j) NTA (nitrilotriacetic acid) and salts thereof,
   (k) phenols and halogenated phenols,
   (l) paradichlorobenzene,
   (m) aromatic hydrocarbons,
   (n) aliphatic hydrocarbons,
   (o) halogenated hydrocarbons,
   (p) soap,
   (q) zeolites,
   (r) polycarboxylates.
2. The following classes of constituents, if added, shall be listed irrespective of their concentration:
   (a) enzymes,
   (b) micro-organisms,
   (c) optical brighteners,
   (d) perfumes.
3. Preservatives shall be listed, using where possible the system referred to in Article 33 of Regulation (EC) No 1223/2009, irrespective of their concentration, provided that they meet the following conditions:

(a) contribute to the qualification of the detergent as a treated article within the meaning of Article 3(1), point (l), of Regulation (EU) No 528/2012;

(b) are labelled on a constituent of the detergent.

The condition listed in point (b) of the first subparagraph does not have to be met where preservatives do not exceed the elicitation thresholds referred to in point 3.4.3.3. / table 3.4.6. of Annex I to Regulation (EC) No 1272/2008 or they no longer have a preservation function in the final product even in synergies with other preservatives.

4. If added at concentrations exceeding 0,01 % by weight, the allergenic fragrances that are listed in entries 45, 67-92 and [X] to [X] of Annex III to Regulation (EC) No 1223/2009, shall be labelled using the system referred to in Article 33 of that Regulation. The first sentence shall not apply to allergenic fragrances that meet the labelling thresholds under Regulation (EC) No 1272/2008.

5. The requirements referred to in points 1 to 4 shall not apply to professional detergents and surfactants, provided that the equivalent information to that required in those points is provided in section 15 of the safety data sheet drawn up in accordance with Article 31 of Regulation (EC) No 1907/2006.

6. In addition to the information listed in points 1 to 5, as applicable, the label of detergents containing micro-organisms shall bear the following information:

(a) an indication or a precautionary statement that the product is not to be used on surfaces in contact with food;

(b) an indication of the shelf life of the product;

(c) use instructions or special precautions, where relevant.

PART B – LABELLING OF DOSAGE INFORMATION

The information to be included on the label of consumer laundry detergents and consumer automatic dishwasher detergents

1. The label of consumer laundry detergents shall contain the following information:

(a) the recommended quantities and/or dosage instructions expressed in millilitres or grams appropriate to a standard washing machine load, for soft, medium and hard water hardness levels and making provision for one or two cycle washing processes,

(b) for heavy-duty detergents, the number of standard washing machine loads of 'normally soiled' fabrics, and, for detergents for delicate fabrics, the number of standard washing machine loads of 'lightly soiled' fabrics, that can be washed with the contents of the package using water of medium hardness, corresponding to 2,5 millimoles $CaCO_3$/l,

(c) the capacity of any measuring cup, if provided, shall be indicated in millilitres or grams, and markings shall be provided to indicate the dose of detergent appropriate for a standard washing machine load for soft, medium and hard water hardness levels,

2. For the purposes of point 1, the standard washing machine loads shall be 4,5 kg dry fabric for heavy-duty detergents and 2,5 kg dry fabric for light-duty detergents. A detergent shall be considered to be a heavy-duty detergent unless the claims of the

manufacturer predominantly promote fabric care, namely low temperature wash, delicate fibres and colours.

3. The label of consumer automatic dishwasher detergents shall indicate the standard dosage expressed in grams or millilitres or number of tablets for the main washing cycle for normally soiled tableware in a fully loaded 12 place settings dishwasher, adjusting the standard dosage, where relevant, for soft, medium, and hard water hardness.

PART C – DIGITAL LABELLING

The following content information referred to in part A, may be provided on the digital label only, in accordance with Article 16(1), second subparagraph, in the manner specified in that part:

(a) anionic surfactants;

(b) cationic surfactants;

(c) amphoteric surfactants;

(d) non-ionic surfactants;

(e) phosphates;

(f) phosphonates;

(g) soap.

PART D – SIMPLIFIED DOSAGE INFORMATION FOR CONSUMER LAUNDRY DETERGENTS

The simplified dosage grid shall contain the following information:

(a) basic instructions for use, where relevant;

(b) the recommended quantities based on medium/average water hardness and different degrees of fabric soiling; and

(c) an indication of the washing machine load.

ANNEX VI

PRODUCT PASSPORT

The product passport shall include the following information:

(a) the unique product identifier of the detergent or surfactant;

(b) the name, the address of the manufacturer or the manufacturer's authorised representative as well the manufacturer's unique operator identifier;

(c) the identification of detergent or surfactant allowing traceability, including a colour image of sufficient clarity to enable the identification of the detergent or surfactant;

(d) the commodity code under which the detergent or surfactant is classified at the moment the product passport is created, as set out in Council Regulation (EEC) No 2658/87[4];

(e) references to Union legal acts that the detergent or surfactant complies with;

(f) a full list of substances intentionally added in the detergent or surfactant and of preservatives labelled in accordance with part A, point 3, first subparagraph, point (b), of Annex V, using the International Nomenclature of Cosmetic Ingredients, or where it is not available, the European Pharmacopoeia name and, when also the latter is not available, the common chemical name or International Union of Pure and Applied Chemists name.

The obligation referred to in point (f) shall not apply to professional detergents, or to surfactants for professional detergents, for which a safety data sheet referred to in Article 31 of Regulation (EC) No 1907/2006 is available.

[4] Council Regulation (EEC) No 2658/87 of 23 July 1987 on the tariff and statistical nomenclature and on the Common Customs Tariff (OJ L 256, 7.9.1987, p. 1).

ANNEX VII

TEST METHODS REFERRED TO IN ARTICLE 22(2)

1. REFERENCE METHOD (CONFIRMATORY TEST)

1.1. Definition

This method describes a laboratory model of the activated sludge and secondary settler which is designed to simulate municipal sewage treatment. Improved state-of-the-art operating conditions can be applied to this test method as described in EN ISO 11733.

1.2. Equipment needed for measurement

The method of measurement employs the small-activated sludge plant shown in Figure 1, and in greater detail in Figure 2. The equipment consists of a sewage vessel A for synthetic sewage, dosing pump B, aeration vessel C, settling vessel D, air-lift pump E to recycle the activated sludge, and vessel F for collecting the treated effluent.

Vessels A and F must be of glass or suitable plastic and hold at least twenty-four litres. Pump B must provide a constant flow of synthetic sewage to the aeration vessel; this vessel, during normal operation, contains three litres of mixed liquor. A sintered aeration cube G is suspended in the vessel C at the apex of the cone. The quantity of air blown through the aerator shall be monitored by means of a flow meter H.

1.3. Synthetic sewage

A synthetic sewage is employed for the test. Dissolve in each litre of tap water:

– 160 mg peptone;

– 110 mg meat extract;

– 30 mg urea, $CO(NH_2)_2$;

– 7 mg sodium chloride, NaCl;

– 4 mg calcium chloride, $CaCl_2.2H_2O$;

– 2 mg magnesium sulphate, $MgSO_4.7H_2O$;

– 28 mg of di-potassium hydrogen phosphate, K_2HPO_4;

– and 10 ± 1 mg of the surfactant.

The synthetic sewage is freshly prepared daily.

1.4. Preparation of samples

Uncompounded surfactants are examined in the original state. Active content of surfactant samples must be determined in order to prepare the synthetic sewage (point 1.3).

1.5. Operation of equipment

Initially, fill aeration vessel C and settling vessel D with synthetic sewage. The height of the vessel D should be so fixed that the volume contained in the aeration vessel C is three litres. Inoculation is made by introducing 3 ml of a secondary effluent of good quality, freshly collected from a treatment plant dealing with a

predominantly domestic sewage. The effluent must be kept under aerobic conditions in the period between sampling and application. Then set the aerator G, air-lift E and dosing device B in operation. The synthetic sewage must pass through the aeration vessel C at a rate of one litre per hour; this gives a mean retention time of three hours.

The rate of aeration should be so regulated that the contents of vessel C are kept constantly in suspension and the dissolved oxygen content is at least 2 mg/l. Foaming must be prevented by appropriate means. Anti-foaming agents that inhibit the activated sludge or contain surfactants must not be used. The air-lift pump E must be set so that the activated sludge from the settling vessel is continually and regularly recycled to aeration vessel C. Sludge which has accumulated around the top of the aeration vessel C, in the base of the settling vessel D, or in the circulation circuit must be returned to the circulation at least once each day by brushing or some other appropriate means. When the sludge fails to settle, its settleability may be increased by the addition of 2 ml portions of a 5 % solution of ferric chloride, repeated as necessary.

The effluent from the settling vessel D is accumulated in vessel F for twenty-four hours, following which a sample is taken after thorough mixing. Vessel F must then be carefully cleaned.

1.6. Checking measuring equipment

The surfactant content (in mg/l) of the synthetic sewage is determined immediately before use.

The surfactant content (in mg/l) of the effluent collected over twenty-four hours in vessel F should be determined analytically by the same method, immediately after collection: otherwise the samples must be preserved, preferably by freezing. The concentrations must be determined to the nearest 0,1 mg/l surfactant

As a check on the efficiency of the process, the chemical oxygen demand (COD) or the dissolved organic carbon (DOC) of the glass fibre filtered effluent accumulated in vessel F and of the filtered synthetic sewage in vessel A is measured at least twice per week.

The reduction in COD or DOC should level off when a roughly regular daily surfactant degradation is obtained at the end of the running-in period shown in Figure 3.

The content of dry matter in the activated sludge contained in the aeration vessel should be determined twice a week in g/l. If it is more than 2,5 g/l, the excess activated sludge must be discarded.

The degradation test is performed at room temperature; this should be steady and kept between 19-24 ° C.

1.7. Calculation of biodegradability

The percentage degradation of surfactant must be calculated every day on the basis of the surfactant content in mg/l of the synthetic sewage and of the corresponding effluent accumulated in vessel F.

The degradability values thus obtained should be presented graphically as in Figure 3.

The degradability of the surfactant should be calculated as the arithmetic mean of the values obtained over the twenty-one days that follow the running-in and acclimatisation period, during which degradation has been regular and the operation of the plant trouble-free. In any event the duration of the running-in period should not exceed six weeks.

The daily degradation values are calculated to the nearest 0,1 % but the final result is given to the nearest whole number.

In some cases it may be permissible to reduce the frequency of sampling but at least fourteen results collected over the twenty-one days which follow the running-in period should be used in calculating the average.

2. DETERMINATION OF ANIONIC SURFACTANTS IN BIODEGRADABILITY TESTS

2.1. Principle

The method is based on the fact that the cationic dye methylene blue forms blue salts with anionic surfactants (MBAS), which can be extracted with chloroform. To eliminate interference, the extraction is first effected from alkaline solution and the extract is then shaken with acidic methylene blue solution. The absorbency of the separated organic phase is measured photometrically at the wavelength of maximum absorption of 650 nm.

2.2. Reagents and equipment

2.2.1. Buffer solution pH 10

Dissolve 24 g sodium bicarbonate, $NaHCO_3$ AR, and 27 g anhydrous sodium carbonate (Na_2CO_3) AR in deionised water and dilute to 1000 ml.

2.2.2. Neutral methylene blue solution

Dissolve 0,35 g methylene blue AR in deionised water and dilute to 1000 ml. Prepare the solution at least twenty-four hours before use. The absorbency of the blank chloroform phase, measured against chloroform must not exceed 0,015 per 1 cm of layer thickness at 650 nm.

2.2.3. Acidic methylene blue solution

Dissolve 0,35 g methylene blue AR in 500 ml deionised water and mix with 6,5 ml H_2SO_4 (d = 1,84 g/ml). Dilute to 1000 ml with deionised water. Prepare the solution at least twenty-four hours before use. The absorbency of the blank chloroform phase, measured against chloroform must not exceed 0,015 per 1 cm of layer thickness at 650 nm.

2.2.4. Chloroform (trichloromethane) AR freshly distilled

2.2.5. Dodecyl benzene sulphonic acid methyl ester

2.2.6. Ethanolic potassium hydroxide solution, KOH 0,1 M

2.2.7. Ethanol pure, C_2H_5OH

2.2.8. sulphuric acid, H_2SO_4 0,5 M

2.2.9. Phenolphthalein solution

Dissolve 1 g phenolphthalein in 50 ml ethanol and add 50 ml deionised water while stirring continuously. Filter off any precipitate obtained.

2.2.10. Methanolic hydrochloric acid: 250 ml hydrochloric acid AR and 750 ml methanol

2.2.11. Separating funnel, 250 ml

2.2.12. Graduated flask, 50 ml

2.2.13. Graduated flask, 500 ml

2.2.14. Graduated flask, 1000 ml

2.2.15. Round-bottomed flask with ground glass stopper and reflux condenser, 250 ml; boiling granules

2.2.16. pH meter

2.2.17. Photometer for measurements at 650 nm, with 1 to 5 cm cells

2.2.18. Qualitative grade filter paper

2.3. Procedure

The samples for analysis must not be taken through a layer of foam.

After thorough cleaning with water, the equipment used for the analysis must be thoroughly rinsed with methanolic hydrochloric acid (point 2.2.10) and then with deionised water before using.

Filter the activated sludge plant influent and effluent to be examined immediately on sampling. Discard the first 100 ml of the filtrates.

Place a measured volume of the sample, neutralised if necessary, into a 250 ml separating funnel (point 2.2.11). The volume of sample should contain between 20 and 150 g of MBAS. At the lower MBAS content, up to 100 ml of sample may be used. When using less than 100 ml, dilute to 100 ml with deionised water. Add to the sample 10 ml of buffer solution (point 2.2.1), 5 ml of neutral methylene blue solution (point 2.2.2) and 15 ml of chloroform (point 2.2.4). Shake the mixture uniformly and not too vigorously for one minute. After phase separation, run the chloroform layer into a second separating funnel, containing 110 ml of deionised water and 5 ml of acidic methylene blue solution (point 2.2.3). Shake the mixture for one minute. Pass the chloroform layer through a cotton-wool filter previously cleaned and wetted with chloroform into a graduated flask (point 2.2.12).

Extract the alkaline and acid solutions three times, using 10 ml of chloroform for the second and third extractions. Filter the combined chloroform extracts through the same cotton wool filter and dilute to the mark in the 50 ml flask (point 2.2.12) with chloroform used for rewashing the cotton wool. Measure the absorbency of the chloroform solution with a photometer at 650 nm in 1 to 5 cm cells against chloroform. Run a blank determination through the whole procedure.

2.4. Calibration curve

Prepare a calibration solution from the standard substance dodecylbenzene sulphonic acid methyl ester (tetrapropylene type mol. wt. 340) after saponification into the potassium salt. The MBAS is calculated as sodium dodecyl benzene sulphonate (mol. wt. 348).

From a weighing pipette, weigh 400 to 450 mg of dodecyl-benzene-sulphonic-acid-methyl-ester (point 2.2.5) to the nearest 0,1 mg in a round-bottomed flask and add 50 ml of ethanolic potassium hydroxide solution (point 2.2.6) and some boiling granules. After mounting the reflux condenser, boil for one hour. After cooling, wash

the condenser and ground glass joint with about 30 ml of ethanol, and add these washings to the contents of the flask. Titrate the solution with sulphuric acid against phenolphthalein until it becomes colourless. Transfer this solution to a 1000 ml graduated flask (point 2.2.14), dilute to the mark with deionised water and mix.

Part of this surfactant stock solution is then further diluted. Withdraw 25 ml, transfer to a 500 ml graduated flask (point 2.2.13), dilute to the mark with deionised water and mix.

This standard solution contains:

$$\frac{E \times 1{,}023\,\text{mg MBAS per ml}}{20\,000}$$

where E is the sample weight in mg.

To establish the calibration curve, withdraw 1, 2, 4, 6, 8 ml portions of the standard solution and dilute each to 100 ml with deionised water. Then proceed as stated under point 2.3 including a blank determination.

2.5. Calculation of results

The amount of anionic surfactant (MBAS) in the sample is read from the calibration curve (point 2.4). The MBAS content of the sample is given by:

$$\frac{\text{mg MBAS} \times 1\,000}{V} = \text{MBAS mg/l}$$

where: V = ml volume of the sample used.

Express the results as sodium dodecylbenzene sulphonate (MW 348).

2.6. Expression of results

Express the results as MBAS mg/l to the nearest 0,1.

3. DETERMINATION OF NON-IONIC SURFACTANTS IN BIODEGRADATION TEST LIQUORS

3.1. Principle

Surface active agents are concentrated and isolated by gas stripping. In the sample used, the quantity of non-ionic surfactant should be in the range 250-800 g.

The stripped surfactant is dissolved in ethyl acetate.

After phase separation and evaporation of the solvent, the non-ionic surfactant is precipitated in aqueous solution with modified Dragendorff reagent ($KBiI_4$ + $BaCl_2$ + glacial acetic acid).

The precipitate is filtered, washed with glacial acetic acid and dissolved in ammonium tartrate solution. The bismuth in the solution is titrated potentiometrically with pyrrolidinedithiocarbamate solution at pH 4-5 using a bright platinum indicator electrode and a calomel or silver/silver chloride reference electrode. The method is applicable to non-ionic surfactants containing 6-30 alkylene oxide groups.

The titration result is multiplied by the empirical factor of 54 for conversion to the reference substance nonylphenol condensed with 10 mols ethylene oxide (NP 10).

3.2. Reagents and Equipment

Reagents are to be made up in deionised water.

3.2.1. Pure ethyl acetate, freshly distilled.

3.2.2. Sodium bicarbonate, $NaHCO_3$ AR.

3.2.3. Dilute hydrochloric acid [20 ml concentrated acid (HCl) diluted to 1000 ml with water]

3.2.4. Methanol AR, freshly distilled, stored in a glass bottle.

3.2.5. Bromocresol purple, 0,1 g in 100 ml methanol.

3.2.6. Precipitating agent: the precipitating agent is a mixture of two volumes of solution A and one volume of solution B. The mixture is stored in a brown bottle and can be used for up to one week after mixing.

3.2.6.1. Solution A

Dissolve 1,7 g bismuth nitrate, $BiONO_3.H_2O$ AR, in 20 ml glacial acetic acid, and make up to 100 ml with water. Then dissolve 65 g potassium iodide AR in 200 ml water. Mix these two solutions in a 1000 ml measuring flask, add 200 ml glacial acetic acid (point 3.2.7) and make up to 1000 ml with water.

3.2.6.2. Solution B

Dissolve 290 g barium chloride, $BaCl_2.2H_2O$ AR, in 1000 ml of water.

3.2.7. Glacial acetic acid 99-100 % (lower concentrations are unsuitable).

3.2.8. Ammonium tartrate solution: mix 12,4 g tartaric acid AR and 12,4 ml of ammonia solution AR (d = 0,910 g/ml) and make up to 1000 ml with water (or use the equivalent amount of ammonium tartrate AR).

3.2.9. Dilute ammonia solution: 40 ml ammonia solution AR (d = 0,910 g/ml) diluted to 1000 ml with water.

3.2.10. Standard acetate buffer: dissolve 40 g solid sodium hydroxide AR, in 500 ml water in a beaker and allow to cool. Add 120 ml glacial acetic acid (point 3.2.7). Mix thoroughly, cool and transfer to a 1000 ml volumetric flask. Make up to the mark with water.

3.2.11. Pyrrolidinedithiocarbamate solution (known as 'carbate solution'): dissolve 103 mg sodium pyrrolidinedithiocarbamate, $C_5H_8NNaS_2.2H_2O$, in about 500 ml water, add 10 ml of n-amyl alcohol AR and 0,5 g $NaHCO_3$ AR, and make up to 1000 ml with water.

3.2.12. Copper sulphate solution (for standardisation of point 3.2.11).

STOCK SOLUTION

Mix 1,249 g copper sulphate, $CuSO_4.5H_2O$ AR, with 50 ml 0,5 M sulphuric acid and make up to 1000 ml with water.

STANDARD SOLUTION

Mix 50 ml stock solution with 10 ml 0,5 M H_2SO_4 and make up to 1000 ml with water.

3.2.13. Sodium chloride AR.

3.2.14. Gas-stripping apparatus (see Figure 5). The diameter of the sintered disc must be the same as the internal diameter of the cylinder.

3.2.15. Separating funnel, 250 ml.

3.2.16. Magnetic stirrer with magnet 25-30 mm.

3.2.17. Gooch crucible, diameter of the perforated base = 25 mm, Type G4.

3.2.18. Circular glass-fibre filter papers, 27 mm diameter with fibre diameter 0,3-1,5 m.

3.2.19. Two filter flasks with adapters and rubber collars, 500 and 250 ml respectively.

3.2.20. Recording potentiometer fitted with a bright platinum indicator electrode and a calomel or silver/silver chloride reference electrode with a 250 mV range, with automatic burette of 20-25 ml capacity, or alternative manual equipment.

3.3. Method

3.3.1. Concentration and separation of the surfactant

Filter the aqueous sample through a qualitative filter paper. Discard the first 100 ml of the filtrate.

Into the stripping apparatus, previously rinsed with ethyl acetate, place a measured quantity of the sample, such that it contains between 250-800 g non-ionic surfactant.

To improve the separation add 100 g sodium chloride and 5 g sodium bicarbonate.

If the volume of the sample exceeds 500 ml, add these salts to the stripping apparatus in solid form, and dissolve by passing nitrogen or air through.

If a smaller-sized sample is used, dissolve the salts in 400 ml water and then add to the stripping apparatus.

Add water to bring the level to the upper stopcock.

Cautiously add 100 ml ethyl acetate on top of the water.

Fill the wash-bottle in the gas-line (nitrogen or air) two-thirds full with ethyl acetate.

Pass a gas stream of 30-60 l/h through the apparatus; the use of a flowmeter is recommended. The rate of aeration must be increased gradually at the beginning. The gas rate must be so adjusted that the phases remain noticeably separate to minimise the mixing of the phases and the solution of the ethyl acetate in the water. Stop the gas flow after five minutes.

If there is a reduction of more than 20 % in the volume of the organic phase through solution in water, the sublation must be repeated paying special attention to the rate of gas flow.

Run off the organic phase into a separating funnel. Return any water in the separating funnel from the aqueous phase — it should only be a few ml — to the stripping apparatus. Filter the ethyl acetate phase through a dry qualitative filter paper into a 250 ml beaker.

Put a further 100 ml ethyl acetate into the stripping apparatus and again pass nitrogen or air through for five minutes. Draw off the organic phase into the separating funnel

used for the first separation, reject the aqueous phase and run the organic phase through the same filter as the first ethyl acetate portion. Rinse both the separating funnel and the filter with about 20 ml ethyl acetate.

Evaporate the ethyl acetate extract to dryness using a water-bath (fume cupboard). Direct a gentle stream of air over the surface of the solution to accelerate the evaporation.

3.3.2. Precipitation and filtration

Dissolve the dry residue from 3.3.1 in 5 ml methanol, add 40 ml water and 0,5 ml dilute HCl (point 3.2.3) and stir the mixture with a magnetic stirrer.

To this solution add 30 ml of precipitating agent (point 3.2.6) from a measuring cylinder. The precipitate forms after repeated stirring. After stirring for ten minutes leave the mixture to stand for at least five minutes.

Filter the mixture through a Gooch crucible, the base of which is covered with a glass-fibre filter paper. First wash the filter under suction with about 2 ml glacial acetic acid. Then thoroughly wash the beaker, magnet, and crucible with glacial acetic acid, of which about 40-50 ml is necessary. It is not necessary to quantitatively transfer the precipitate adhering to the sides of the beaker, to the filter, because the solution of the precipitate for the titration is returned to the precipitating beaker, and the remaining precipitate will then be dissolved.

3.3.3. Dissolution of the precipitate

Dissolve the precipitate in the filter crucible by the addition of hot ammonium tartrate solution (about 80 ° C) (point 3.2.8) in three portions of 10 ml each. Allow each portion to stand in the crucible for some minutes before being sucked through the filter into the flask.

Put the contents of the filter flask into the beaker used for the precipitation. Rinse the sides of the beaker with a further 20 ml of tartrate solution to dissolve the rest of the precipitate.

Carefully wash the crucible, adapter and filter flask with 150-200 ml water, and return the rinsing water to the beaker used for the precipitation.

3.3.4. The titration

Stir the solution using a magnetic stirrer (point 3.2.16), add a few drops of bromocresol purple (point 3.2.5) and add the dilute ammonia solution (point 3.2.9) until the colour turns violet (the solution is initially weakly acid from the residue of acetic acid used for rinsing).

Then add 10 ml standard acetate buffer (point 3.2.10), immerse the electrodes in the solution, and titrate potentiometrically with standard ‘carbate solution’ (point 3.2.11), the burette tip being immersed in the solution.

The titration rate should not exceed 2 ml/min.

The endpoint is the intersection of the tangents to the two branches of the potential curve.

It will be observed occasionally that the inflection in the potential curve becomes flattened; this can be eliminated by carefully cleaning the platinum electrode (by polishing with emery paper).

3.3.5. Blank determinations

At the same time run a blank determination through the whole procedure with 5 ml methanol and 40 ml water, according to the instructions in point 3.3.2. The blank titration should be below 1 ml, otherwise the purity of the reagents (points 3.2.3, 3.2.7, 3.2.8, 3.2.9, 3.2.10) is suspect, especially their content of heavy metals, and they must be replaced. The blank must be taken into account in the calculation of the results.

3.3.6. Control of the factor of the 'carbate solution'

Determine the factor for the carbate solution on the day of use. To do this, titrate 10 ml of the copper sulphate solution (point 3.2.12) with 'carbate solution' after the addition of 100 ml water and 10 ml standard acetate buffer (point 3.2.10). If the amount used is a ml, the factor f is:

$$f = \frac{10}{a}$$

and all the results of the titration are multiplied by this factor.

3.4. Calculation of results

Every non-ionic surfactant has its own factor, depending on its composition, particularly on the length of the alkene oxide chain. The concentration of non-ionic surfactant is expressed in relation to a standard substance — a nonyl phenol with ten ethylene oxide units (NP 10) — for which the conversion factor is 0,054.

Using this factor the amount of surfactant present in the sample is found expressed as mg of NP 10 equivalent, as follows:

(b — c) xfx 0,054 = mg non-ionic surfactant as NP 10

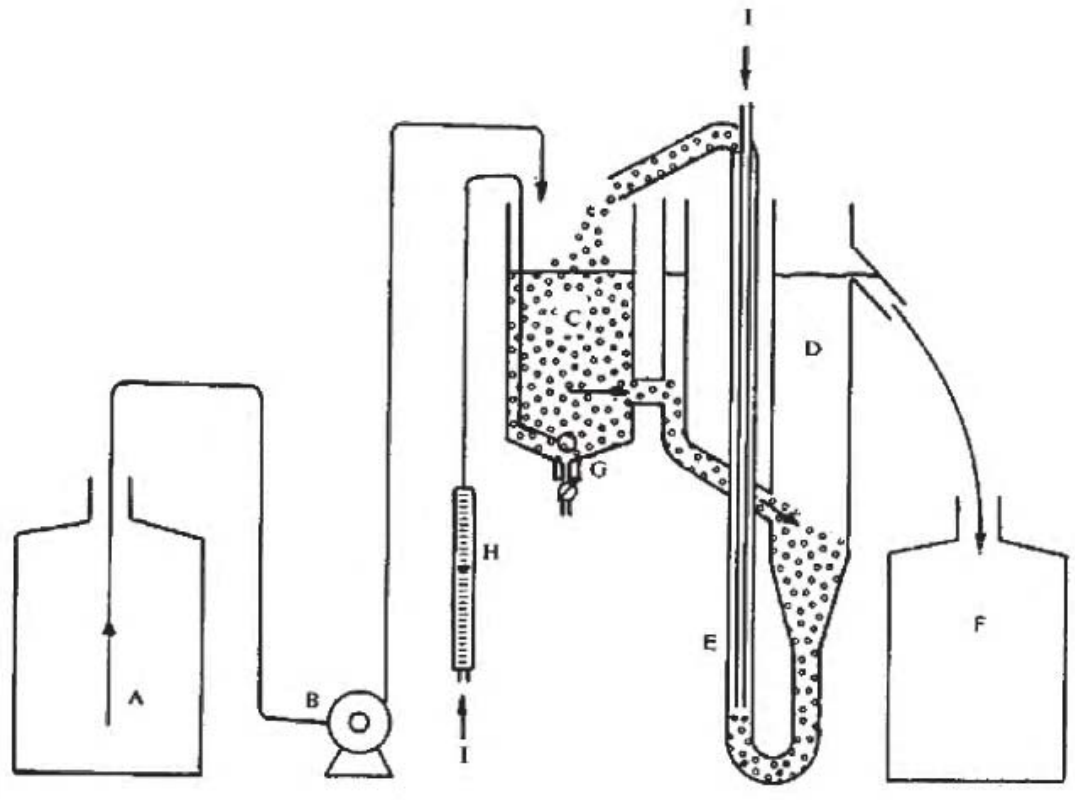
where:

b	=	volume of 'carbate solution' used by the sample (ml),
c	=	volume of 'carbate solution' used by the blank (ml),
f	=	factor of the 'carbate solution'.

3.5. Expression of results

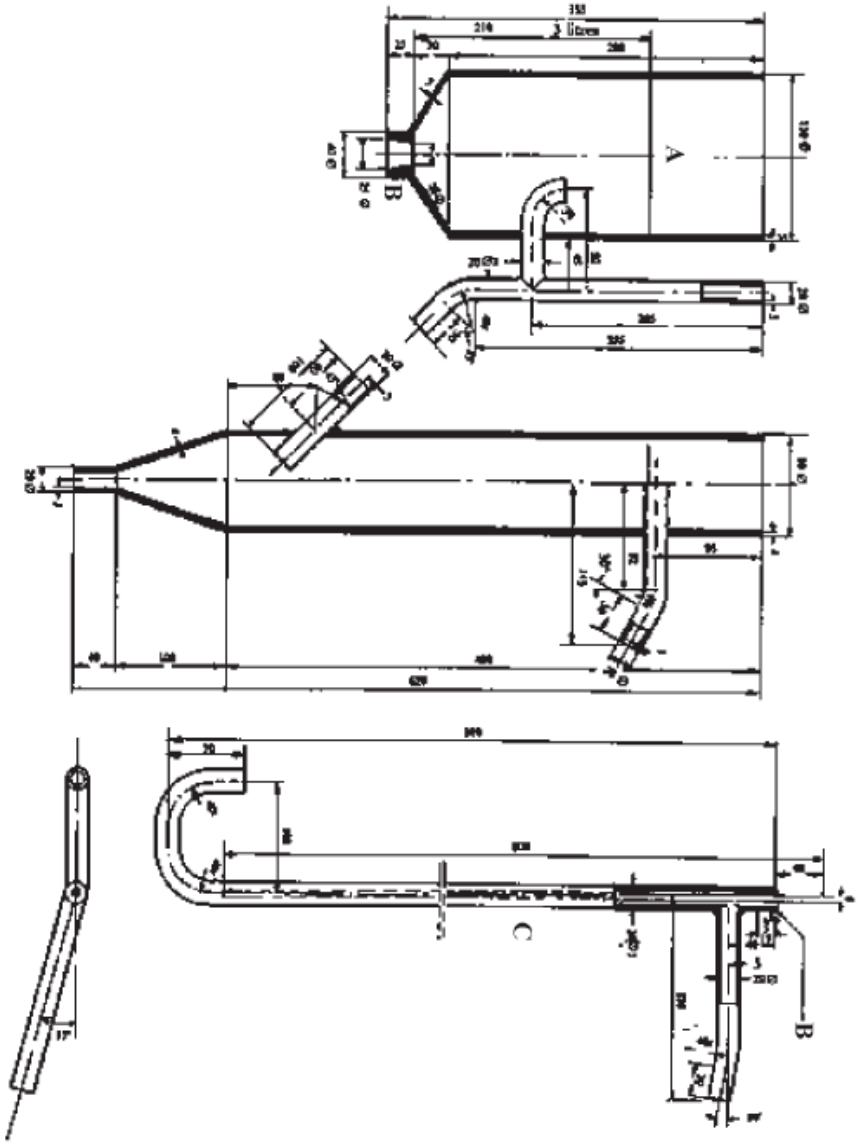
Express the results in mg/l as NP 10 to the nearest 0,1.

Figure 1 Activated sludge plant: overviews



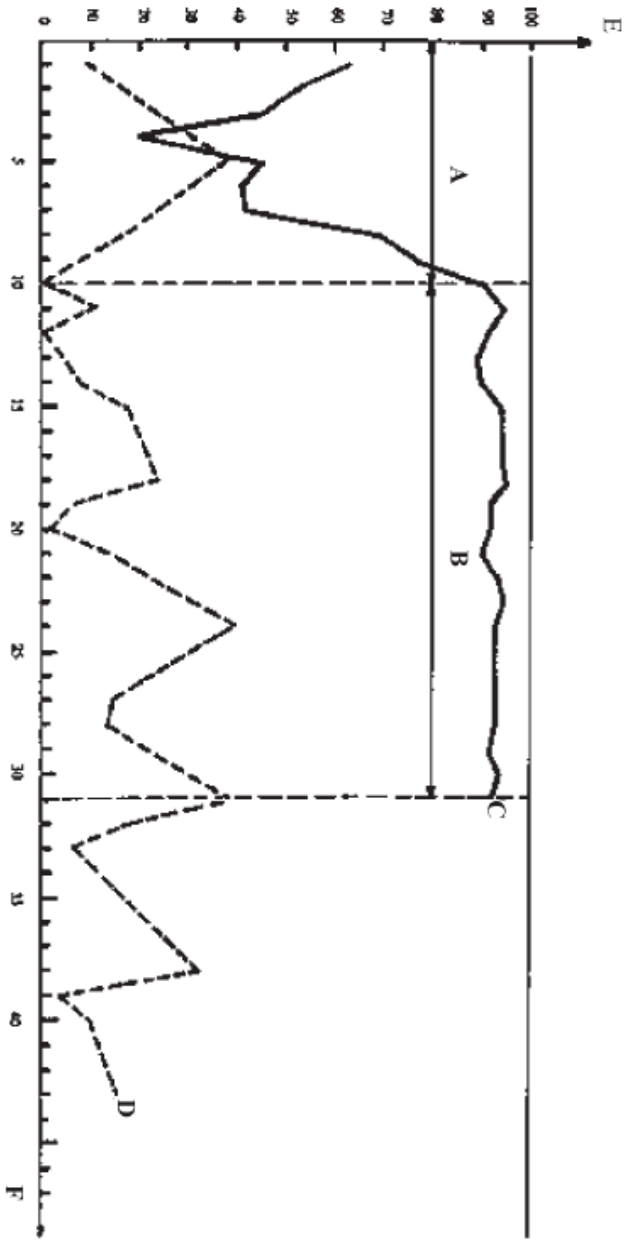
A	Storage vessel
B	Dosing device
C	Aeration chamber (three litres capacity)
D	Settling vessel
E	Air-lift pump
F	Collector
G	Sintered aerator
H	Air-flow meter
I	Air

Figure 2 Activated sludge plant: detail (dimensions in millimetres)



A	Liquid level
B	Hard PVC
C	Glass or waterproof plastic (hard PVC)

Figure 3 Calculation of biodegradability - Confirmatory test



A	Running-in period
B	Period used for calculation (twenty-one days)
C	Readily biodegradable surfactant
D	Surfactant not readily biodegradable
E	Biodegradation (%)
F	Time (days)

ANNEX VIII

CORRELATION TABLE

Regulation (EC) No 648/2004	This Regulation
Article 1(1)	Article 1(1)
Article 1(2)	-
Article 2(1)	Article 2, point (1)
Article 2(1a)	Article 2, point (2)
Article 2(1b)	Article 2, point (3)
Article 2(2)	-
Article 2(3)	Article 2, point (6)
Article 2(4)	Article 2, point (7)
Article 2(5)	Article 2, point (8)
Article 2(6)	Article 2, point (11)
Article 2(7)	-
Article 2(8)	Article 2, point (12)
Article 2(9)	Article 2, point (14)
Article 2(9a)	Article 2, point (13)
Article 2(10)	Article 2, point (15)
Article 2(11)	-
Article 2(12)	Article 2, point (5)
Article 3(1)	Article 3(1) and Article 4(2)
Article 3(2)	-
Article 3(3)	Article 7(1)
Article 4(1)	Article 4(1)
Article 4(2)	-
Article 4(3)	-
Article 4a	Article 6
Article 5(1)	-
Article 5(2)	-
Article 5(3)	-
Article 5(4)	-
Article 5(5)	-
Article 5(6)	-
Article 6(1)	-

Article 6(2)	-
Article 6(3)	-
Article 6(4)	-
Article 7	-
Article 8(1)	-
Article 8(2)	-
Article 8(3)	-
Article 8(4)	-
Article 9(1)	Article 8(2)
Article 9(2)	-
Article 9(3)	Article 7(6)
Article 10(1)	-
Article 10(2)	Article 22(2)
Article 11(1)	Article 1(2), point (b)
Article 11(2) and (3)	Article 15(3)
Article 11(4)	Article 15(4)
Article 11(5)	Article 15(5)
Article 11(6)	-
Article 12	Article 28
Article 13	Article 26
Article 13a(1)	Article 27(1)
Article 13a(2)	Article 27(2)
Article 13a(3)	Article 27(3)
Article 13a(4)	Article 27(5)
Article 13a(5)	Article 27(6)

Article 14(1)	Article 3(2)
Article 14(2)	-
Article 14(3)	-
Article 14(4)	-
Article 14(5)	-
Article 15(1), first subparagraph	Article 24(1)
Article 15(1), second subparagraph	Article 24(3)
Article 15(2)	Article 25(4)
Article 16(1)	-
Article 16(2)	-
Article 17	Article 33
Article 18	Article 29
Article 19	Article 35