COMMON POSITION (EC) No 64/2003

adopted by the Council on 4 November 2003

with a view to the adoption of a Regulation (EC) No .../2003 of the European Parliament and of the Council of ... on detergents

(2003/C 305 E/02)

(Text with EEA relevance)

THE EUROPEAN PARLIAMENT AND THE COUNCIL OF THE EUROPEAN UNION,

Having regard to the Treaty establishing the European Community, and in particular Article 95 thereof,

Having regard to the proposal from the Commission [1],

Having regard to the Opinion of the European Economic and Social Committee [2],

Acting in accordance with the procedure laid down in Article 251 of the Treaty [3],

Whereas:

(1) Council Directives 73/404/EEC of 22 November 1973 on the approximation of the laws of the Member States relating to detergents [4], Council Directive 73/405/EEC of 22 November 1973 on the approximation of the laws of the Member States relating to methods of testing the biodegradability of anionic surfactants [5], Council Directive 82/242/EEC of 31 March 1982 on the approximation of the laws of the Member States relating to methods of testing the biodegradability of non-ionic surfactants [6], Council Directive 82/243/EEC of 31 March 1982 amending Directives 73/405/EEC on the approximation of the laws of the Member States relating to methods of testing the biodegradability of anionic surfactants [7] and Council Directive 86/94/EEC of 10 March 1986 amending for the second time Directive 73/404/EEC on the approximation of the laws of the Member States relating to detergents [8], have been significantly amended on several occasions. It is desirable, for reasons of clarity and rationalisation, that the provisions in question should be recast by bringing them all together in a single text. Commission Recommendation 89/542/EEC [9], as regards labelling provisions concerning detergents and cleaning products, should also be included in the single text.

(2) Since the objective of this Regulation, to ensure the internal market in detergents, cannot be sufficiently achieved by the Member States, if there is no common technical criteria throughout the Community, and can therefore be better achieved at Community level, the Community may adopt measures, in accordance with the principle of subsidiarity as set out in Article 5 of the Treaty. In accordance with the principle of proportionality, as set out in that Article, this Regulation does not go beyond what is necessary in order to achieve that objective. A Regulation is the appropriate legal instrument as it imposes directly on manufacturers precise requirements to be implemented at the same time and in the same manner throughout the Community; in the area of technical legislation, uniformity of application in the Member States is needed, and this can only be guaranteed by a Regulation.

(3) A new definition for detergents is needed to cover equivalent uses and be in line with developments at Member State level.

(4) It is necessary to introduce a surfactant definition, which was lacking in the existing legislation.

(5) It is important to give a clear and precise description of the relevant types of biodegradability.

(6) Measures concerning detergents should be adopted to ensure the functioning of the internal market and avoid restricting the competition in the Community.

(7) As confirmed by the Commission White Paper on the strategy for a future Chemical Policy appropriate measures concerning detergents should ensure a high level of environmental protection, especially of the aquatic environment.

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[1] COM(2002) 485 final — COD 2002/216.

[2] OJ C 95, 23.4.2003, p. 24.

[3] Opinion of the European Parliament of 10 April 2003 (not yet published in the Official Journal), Council Common position of 4 November 2003 and Decision of the European Parliament of ... (not yet published in the Official Journal).

[4] OJ L 347, 17.12.1973, p. 51. Directive as last amended by Regulation (EC) No 807/2003 (OJ L 122, 16.5.2003, p. 36).

[5] OJ L 347, 17.12.1973, p. 53. Directive as amended by Directive 82/243/EEC (OJ L 109, 22.4.1982, p. 18).

[6] OJ L 109, 22.4.1982, p. 1.

[7] OJ L 109, 22.4.1982, p. 18.

[8] OJ L 80, 25.3.1986, p. 51.

[9] OJ L 291, 10.10.1989, p. 55.

(8) Detergents are already subject to certain Community provisions concerning the manufacture, proper handling, usage and labelling, in particular with reference to Commission Recommendation 89/542/EEC and Commission Recommendation 98/480/EC of 22 July 1998 concerning good environmental practice for household laundry detergents ([1]). Directive 1999/45/EC of the European Parliament and of the Council of 31 May 1999 concerning the approximation of the laws, regulations and administrative provisions of the Member States relating to the classification, packaging and labelling of dangerous preparations ([2]) applies to detergents.

(9) Ditallow-dimethyl-ammonium-chloride (DTDMAC) and Nonylphenol (including ethoxylates derivatives-APEs) are priority substances undergoing at Community level Risk Assessment activities, in accordance with Council Regulation (EEC) No 793/93 of 23 March 1993 on the evaluation and control of the risks of existing substances ([3]), and if necessary adequate strategies to limit the risks of exposure to these substances should therefore be recommended and implemented in the framework of other Community provisions.

(10) The existing legislation on biodegradability of surfactants in detergents only covers primary biodegradability ([4]) and it is only applicable to anionic ([5]) and non-ionic ([6]) surfactants; therefore it should be replaced by new legislation, which lays the main emphasis on ultimate biodegradability and meets the important concerns related to the potential toxicity of persistent metabolites.

(11) This requires the introduction of a new set of tests based on EN ISO standards and OECD guidelines, which governs the granting of direct permission for placing detergents on the market.

(12) To provide a high level of protection of the environment, detergents not fulfilling requirements laid down by this Regulation should not be placed on the market.

(13) On 25 November 1999 the Scientific Committee on Toxicity, Ecotoxicity and the Environment has issued an opinion on biodegradability of surfactants in detergents and relevance of test-methods for regulatory control in this area.

(14) The existing requirements regarding primary biodegradability should be maintained on a second hierarchy level and supplemented by a complementary risk assessment, for those surfactants failing ultimate biodegradability tests; furthermore surfactants failing primary biodegradability tests should not obtain marketing authorisation by way of derogation.

(15) The primary biodegradability requirements should be extended to all surfactants, in particular cationic and amphoteric, whilst allowing the possibility of applying instrumental analyses in those cases in which semi-specific analytical methods are not suitable.

(16) The determination of biodegradability test-methods and the record-keeping of lists of derogations are technical matters and should be revised taking into account technical and scientific developments as well as regulatory developments.

(17) Test-methods should produce data that give sufficient assurance of aerobic biodegradability of surfactants in detergents.

(18) Methods to test biodegradability of surfactants in detergents may produce variable results. In such cases they should be complemented by additional assessments in order to determine the risks of continued use.

(19) Provisions should also be set out to place on the market in exceptional cases surfactants in detergents failing ultimate biodegradability tests and this should take place on the basis of all relevant information to ensure environmental protection and on a case by case basis.

(20) The measures necessary for the implementation of this Regulation should be adopted in accordance with Council Decision 1999/468/EC of 28 June 1999 laying down the procedures for the exercise of implementing powers conferred on the Commission ([7]).

(21) It is appropriate to recall that other horizontal legislation is applicable to detergent surfactants, in particular Council Directive 76/769/EEC of 27 July 1976 on the approximation of the laws, regulations and administrative provisions of the Member States relating to restrictions on the marketing and use of certain dangerous substances and preparations ([8]) by which the marketing and use of dangerous substances covered by this Regulation might be banned or restricted, Council Directive 67/548/EEC of 27 June 1967 on the approximation of laws, regulations and administrative provisions relating to the classification, packaging and labelling of dangerous substances ([9]), Commission Directive 93/67/EEC of 20 July 1993, laying down the principles on assessment of risks to man and the environment of substances notified in

([1]) OJ L 215, 1.8.1998, p. 73.

([2]) OJ L 200, 30.7.1999, p. 1. Directive as amended by Commission Directive 2001/60/EC (OJ L 226, 22.8.2001, p. 5).

([3]) OJ L 84, 5.4.1993, p. 1.

([4]) Directives 73/404/EEC and 86/94/EEC.

([5]) Directives 73/405/EEC and 82/243/EEC.

([6]) Directive 82/242/EEC.

([7]) OJ L 184, 17.7.1999, p. 23.

([8]) OJ L 262, 27.9.1976, p. 201. Directive as last amended by Directive 2003/53/EC of the European Parliament and of the Council (OJ L 178, 17.7.2003, p. 24).

([9]) OJ 196, 16.8.1967, p. 1. Directive as last amended by Regulation (EC) No 807/2003.

accordance with Council Directive 67/548/EEC [1], Regulation (EEC) No 793/93, and Commission Regulation (EC) No 1488/94 of 28 June 1994 laying down the principles for the assessment of risks to man and the environment of existing substances [2], Directive 98/8/EC of the European Parliament and of the Council of 16 February 1998 concerning the placing of biocidal products on the market [3], Council Directive 87/18/EEC of 18 December 1986 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 application for tests on chemical substances [4], Council Directive 88/320/EEC of 7 June 1988 on the inspection and verification of good laboratory practice (GLP) [5] and Council Directive 86/609/EEC of 24 November 1986 on the approximation of laws, regulations and administrative provisions of the Member States regarding the protection of animals used for experimental and other scientific purposes [6].

(22) It should be the responsibility of manufacturers to refrain from marketing detergents not complying with this Regulation and to have at the disposal of the national authorities the technical files for all substances and preparations covered by this Regulation; this should also apply to surfactants that have failed to pass the tests mentioned in Annex III.

(23) Manufacturers should be able to request a derogation from the Commission, which should have the possibility to grant such derogation in accordance with the procedure referred to in Article 12(2).

(24) Member States' competent authorities should be able to apply control measures to detergents on the market, but should avoid repeating tests made by the competent laboratories.

(25) The existing labelling provisions for detergents should be continued, including those in Recommendation 89/542/EEC, which are included in this Regulation in order to fulfil the objective of modernising the rules on detergents. Specific labelling is introduced to inform consumers about fragrance substances and preservation agents that are present in detergents. Medical personnel should be able to obtain from the manufacturer upon request a full listing of all ingredients of a detergent to assist them investigate whether a causal link exists between the development of an allergic response and exposure to a particular chemical substance, and Member States should be able to require that such a listing is also made available to a specific public body designated to provide this information to medical personnel.

(26) All the above points call for new legislation replacing the existing legislation; however, for a certain period, Member States can continue to apply their existing laws.

(27) The technical Annexes to this Regulation should be adapted in accordance with the procedure referred to in Article 12(2).

(28) Detergents complying with this Regulation should be allowed to be placed on the market without prejudice to other relevant Community provisions.

(29) In order to ensure the protection of man and the environment from unforeseen risks of detergents, a safeguard clause is needed.

(30) The tests specified for the biodegradability of surfactants should be carried out in laboratories meeting an internationally recognised standard, namely EN/ISO/IEC/17025 or the principles of good laboratory practice; it would not be justified to ask for the application of this latter requirement to existing surfactants to the extent that the available tests on them had been performed before the entering into force of the above standard and still provide a comparable level of scientific quality.

(31) The issues relating to anaerobic biodegradation, the biodegradation of the main non-surfactant organic detergent ingredients, and phosphate content, which are not dealt with in this Regulation should be examined by the Commission and, where this is justified, a proposal should be presented to the European Parliament and the Council.

(32) The five Directives and the Commission Recommendation mentioned in recital (1) which are replaced by this Regulation should be repealed,

HAVE ADOPTED THIS REGULATION:

Article 1

Objectives and scope

1. This Regulation establishes rules designed to achieve the free movement of detergents and surfactants for detergents in the internal market while, at the same time, ensuring a high degree of protection of the environment and human health.

2. For this purpose, this Regulation harmonises the following rules for the placing on the market of detergents and of surfactants for detergents:

— the biodegradability of surfactants in detergents;

[1] OJ L 227, 8.9.1993, p. 9.

[2] OJ L 161, 29.6.1994, p. 3.

[3] OJ L 123, 24.4.1998, p. 1.

[4] OJ L 15, 17.1.1987, p. 29. Directive as amended by Commission Directive 1999/11/EC (OJ L 77, 23.3.1999, p. 8).

[5] OJ L 145, 11.6.1988, p. 35. Directive as last amended by Commission Directive 1999/12/EC (OJ L 77, 23.3.1999, p. 22).

[6] OJ L 358, 18.12.1986, p. 1.

— restrictions or bans on surfactants on grounds of biodegradability;

— the additional labelling of detergents, including fragrance allergens and

— the information that manufacturers must hold at the disposal of the Member States competent authorities and medical personnel.

Article 2

Definitions

For the purpose of this Regulation:

1. 'Detergent' means any substance or preparation containing soaps and/or other surfactants intended for washing and cleaning processes. Detergents may be in any form (liquid, powder, paste, bar, cake, moulded piece, shape, etc.) and marketed for or used in household, or institutional or industrial purposes.

   Other products to be considered as detergents are:

   — 'Auxiliary washing preparation', intended for soaking (pre-washing), rinsing or bleaching clothes, household linen, etc.;

   — 'Laundry fabric-softener', intended to modify the feel of fabrics in processes which are to complement the washing of fabrics;

   — 'Cleaning preparation', intended for domestic all purposes cleaners and/or other cleaning of surfaces (e.g.: materials, products, machinery, mechanical appliances, means of transport and associated equipment, instruments, apparatus, etc.);

   — 'Other cleaning and washing preparations', intended for any other washing and cleaning processes.

2. 'Washing' means the cleaning of laundry, fabrics, dishes and other hard surfaces.

3. 'Cleaning' has the meaning defined by EN ISO 862.

4. 'Substance' means chemical elements and their compounds in the natural state or obtained by any production process, including any additive necessary to preserve the stability of the products and any impurity deriving from the process used, but excluding any solvent which may be separated without affecting the stability of the substance or changing its composition.

5. 'Preparation' means mixture or solution composed of two or more substances.

6. 'Surfactant' means any organic substance and/or preparation used in detergents, which has surface-active properties and which consists of one or more hydrophilic and one or more hydrophobic groups of such a nature and size that it is capable of reducing the surface tension of water, and of forming spreading or adsorption monolayers at the water-air interface, and of forming emulsions and/or microemulsions and/or micelles, and of adsorption at water-solid interfaces.

7. 'Primary biodegradation' means the structural change (transformation) of a surfactant by micro-organisms resulting in the loss of its surface-active properties due to the degradation of the parent substance and consequential loss of the surface-active property as measured by test methods listed in Annex II.

8. 'Ultimate aerobic biodegradation' means the level of biodegradation achieved when the surfactant is totally used by micro-organisms in the presence of oxygen resulting in its breakdown to carbon dioxide, water and mineral salts of any other elements present (mineralisation), as measured by test methods listed in Annex III, and new microbial cellular constituents (biomass).

9. 'Placing on the market' means introducing onto the Community market, thereby making available to third parties, whether in exchange for payment or not. Import into the Community customs territory shall be deemed to be placing on the market.

10. 'Manufacturer' means the natural or legal person responsible for placing a detergent or a surfactant for a detergent on the market; in particular, a producer, an importer, a packager working for his own account, or any person changing the characteristics of a detergent or of a surfactant for a detergent, or creating or changing the labelling thereof, shall be deemed to be a manufacturer. A distributor who does not change the characteristics, labelling or packaging of a detergent, or of a surfactant for a detergent, shall not be deemed to be a manufacturer, except where he acts as an importer.

11. 'Medical personnel' means a registered medical practitioner, or a person working under the direction of a registered medical practitioner, acting to provide patient care, make a diagnosis, or administer treatment and who is bound by professional confidentiality.

12. 'Industrial and institutional detergent' means a detergent for washing and cleaning outside the domestic sphere, carried out by specialised personnel using specific products.

Article 3

The placing on the market

1. When placed on the market, detergents and surfactants for detergents referred to in Article 1, shall conform with the conditions, characteristics and limits laid down in this Regulation and its Annexes and, where relevant, with Directive 98/8/EC and with any other relevant Community legislation. Surfactants that are also active substances within the meaning of Directive 98/8/EC and that are used as disinfectants are exempt from the provisions of Annexes II, III, IV and VIII of this Regulation provided:

(a) they are listed in Annex I or I A of Directive 98/8/EC, or

(b) they are constituents of biocidal products authorised under Article 15(1) or 15(2) of Directive 98/8/EC, or

(c) they are constituents of biocidal products allowed under the transitional measures or subject to the 10 year Work Programme provided for in Article 16 of Directive 98/8/EC.

Instead, such surfactants are deemed to be disinfectants and the detergents of which they are ingredients are subject to the labelling provisions for disinfectants of Annex VII A.

2. Manufacturers of detergents and/or of surfactants for detergents shall be established within the Community.

3. Manufacturers shall be responsible for the conformity of detergents and/or of surfactants for detergents with the provisions of this Regulation and its Annexes.

Article 4

Limitations based on the biodegradability of surfactants

1. Under this Regulation, surfactants and detergents containing surfactants that meet the criteria for ultimate aerobic biodegradation as laid down in Annex III may be placed on the market without further limitations relating to biodegradability.

2. If a detergent contains surfactants for which the level of ultimate aerobic biodegradation is less than that stipulated in Annex III, manufacturers of industrial or institutional detergents containing surfactants, and/or of surfactants for industrial or institutional detergents may ask for derogation. Requests for derogation shall be made and decided in accordance with Articles 5, 6 and 9.

3. The level of 'primary biodegradability' shall be measured for all surfactants in detergents failing ultimate aerobic biodegradation tests. Detergent surfactants, for which the level of 'primary biodegradability' is less than that stipulated in Annex II, shall not be granted derogation.

Article 5

Granting of derogation

1. The request by a manufacturer for derogation shall be made by sending an application to the competent authority of the Member State concerned, referred to in Article 8(1), and to the Commission, providing evidence relating to the criteria mentioned under Article 6(1). Member States can make the request for derogation dependent upon the payment to the Member State competent authority of a fee. Such fees, if any, shall be levied in a non-discriminatory way and shall not exceed the cost of processing the application.

2. Applications shall include a technical file supplying all the information and justifications necessary for evaluating the safety aspects related to the specific use of surfactants in detergents failing to comply with the biodegradability limits, as set out in Annex III.

In addition to the results of tests stipulated in Annex III, the technical file shall include information and results of tests, as stipulated in Annexes II and IV.

The tests laid down in Annex IV point 4 shall be carried out on the basis of a tiered approach. The tiered approach will be defined in a technical guidance document to be adopted in accordance with the procedure of Article 12(2). This guidance document will also specify, where appropriate, those tests for which the principles of good laboratory practice should be applied.

3. The competent authority of the Member State, receiving applications for derogation according to paragraphs 1 and 2, shall examine the requests, evaluate their compliance with the conditions for derogation and inform the Commission about the results within six months of receiving the complete application.

If the competent authority of the Member State deems it necessary, for the evaluation of the risk which may be caused by a substance and/or a preparation, it shall ask, within three months of receiving the application, for further information, verification and/or confirmatory tests concerning these substances and/or preparations or their transformation products, of which they have been notified or have received information under this Regulation. The time period for the evaluation of the dossier by the competent authority of the Member State will start only after the dossier is completed with the additional information. If the requested information is not provided within 12 months, the application shall be considered incomplete and thus invalid. In such a case Article 6(2) shall not apply.

4. The Commission may grant derogation in accordance with the procedure referred to in Article 12(2). If necessary, before granting derogation the Commission shall evaluate further the matters indicated in paragraph 3. It shall take its decision within 12 months of receiving the evaluation from the Member State, except in the case of Article 5(4) and (6) of Decision 1999/468/EC where the period shall be 18 months.

5. Such derogations may allow, limit or severely restrict the placing on the market and the use of surfactants as ingredients in detergents, depending on the results of the complementary risk assessment, as defined in Annex IV. They may include a phase-out period for placing on the market and the use of surfactants as ingredients in detergents.

6. The Commission shall publish the list of surfactants that have obtained derogation, with the corresponding conditions or limitations of use, as provided in Annex V.

Article 6

Refusal of derogation

1. Where the Commission considers refusing to grant a derogation, it shall do so in accordance with the procedure referred to in Article 12(2), and on the basis of the following criteria:

— use in high volumes;

— use in wide-dispersive applications, rather than in low-dispersive applications;

— socio-economic benefits do not outweigh the impact on human health and the environment.

2. As long as the Commission has not decided on a request for derogation, the placing on the market and use of the surfactant in question may be maintained, provided the manufacturer can show that the surfactant was already in use on the Community market at the date of entry into force of this Regulation and that the request for derogation was made within two years from that date.

3. If the Commission refuses to grant a derogation, it shall do so within 12 months of receiving from a Member State the evaluation mentioned in Article 5(3), except in the case of Article 5(4) and (6) of Decision 1999/468/EC where the period shall be 18 months. It may set a transitional period during which the placing on the market and use of the surfactant in question shall be phased-out. This transitional period shall not exceed two years from the date of the Commission Decision.

4. The Commission shall publish in Annex VI the list of surfactants that have been identified as not complying with this Regulation.

Article 7

Testing of surfactants

All tests referred to in Articles 3 and 4 and in Annexes II, III, IV and VIII shall be conducted in compliance with the standards mentioned in Annex I.1 and in accordance with testing requirements under Article 10(5) of Regulation (EEC) No 793/93. For this purpose, it is sufficient to apply either the EN ISO/IEC standard or the principles of good laboratory practice, except for those tests for which the principles of good laboratory practice have been made mandatory. In cases where surfactants are used in detergents which were placed on the market before the entry into force of the above standard, existing tests that were performed using the best scientific knowledge available, and that were performed to a standard comparable to those of the standards mentioned in Annex I may be accepted on a case-by-case basis. The manufacturer or the Member State may submit to the Commission any case over which there is doubt or dispute. A decision shall then be taken in accordance with the procedure laid down in Article 12(2).

Article 8

Duties of the Member States

1. Member States shall appoint the competent authority or authorities responsible for communicating and exchanging information relating to the management of this Regulation and inform the Commission of the full name and address of these authorities.

2. Each Member State shall notify to the other Member States and the Commission the list of approved laboratories, with full name and address, that are competent and authorised to carry out the tests required by this Regulation. Member States shall demonstrate the competence of the above laboratories according to the standard EN ISO/IEC 17025 mentioned in Annex I.1. This requirement shall be deemed to be fulfilled if the Member State has verified the compliance of laboratories with the principles of good laboratory practice in accordance with Article 2 of Directive 88/320/EEC.

3. Where a Member State competent authority has grounds for believing that an approved laboratory does not possess the competence referred to in paragraph 2, it shall raise the matter in the Committee referred to in Article 12. If the Commission decides that the laboratory does not possess the required competence, the name of the approved laboratory shall be removed from the list referred to in paragraph 4. Article 15(2) shall apply, except in the case of laboratories claiming compliance with the requirements of good laboratory practice, for which the non-compliance provisions of Articles 5 and 6 of Directive 88/320/EEC shall apply.

4. The Commission shall publish the lists of competent authorities, mentioned in paragraph 1, and of approved laboratories, mentioned in paragraph 2, once a year in the *Official Journal of the European Union* to the extent that changes have occurred.

Article 9

Information to be provided by manufacturers

1. Without prejudice to Article 17 of Directive 1999/45/EC, manufacturers placing on the market the substances and/or preparations covered by this Regulation shall hold at the disposal of the competent authorities of the Member States:

— information on one or more results of the tests mentioned in Annex III;

— for those surfactants failing to pass tests mentioned in Annex III, and for which a request for derogation was made as referred to in Article 5:

(i) a technical file on results of the tests mentioned in Annex II,

(ii) a technical file on results of the tests and information mentioned in Annex IV.

2. Whenever substances and/or preparations covered by this Regulation are placed on the market, the manufacturer shall be responsible for the correct performance of the relevant tests mentioned above. He shall also have available documentation on the testing carried out to demonstrate compliance with this Regulation, and to show that he is allowed to benefit from the property rights concerning the test results, other than for those test results already in the public domain.

3. Manufacturers placing on the market the preparations covered by this Regulation shall, upon request, make available without delay and free of charge, to any medical personnel, an ingredient datasheet as stipulated in Annex VII C.

This is without prejudice to the right of a Member State to request that such a datasheet be made available to a specific public body to which the Member State has assigned the task of providing this information to medical personnel.

The information contained in the datasheet shall be kept confidential by the specific public body and by the medical personnel, and shall be used only for medical purposes.

Article 10

Control measures

1. Member States' competent authorities may apply, as appropriate, all necessary control measures to detergents placed on the market which ensure the compliance of the product with the provisions of this Regulation. The reference method shall be the test and analytical methods referred to in Annex VIII. These control measures shall not oblige manufacturers to repeat tests made by laboratories fulfilling the conditions indicated in Article 8(2), or to pay for any repeat or additional test, provided the initial test has shown compliance of detergents, or surfactants used as ingredients in detergents, with this Regulation.

2. In cases of concern that a test according to the methods listed in Annex II, III, IV or VIII has produced false positive results, the Member State competent authorities shall notify the Commission and the Commission shall, in accordance with the procedure laid down in Article 12(2), verify those results and take the necessary measures.

Article 11

Labelling

1. The provisions of this Article are without prejudice to the provisions relating to the classification, packaging and labelling of dangerous substances and preparations in Directives 67/548/EEC and 1999/45/EC.

2. The following information must appear in legible, visible and indelible characters on the packaging in which the detergents are put up for sale to the consumer:

(a) the name and trade name of the product;

(b) the name or trade name or trademark and full address and telephone number of the party responsible for placing the product on the market;

(c) the address and telephone number from which the datasheet referred to in Article 9(3) can be obtained.

The same information must appear on all documents accompanying detergents transported in bulk.

3. The packaging of detergents shall indicate the content, according to the specifications provided for in Annex VII A. It shall also indicate instructions for use and special precautions, if required.

4. Additionally, the packaging of detergents sold to the general public intended to be used as laundry detergents shall bear the information provided for in Annex VII B.

5. In cases where a Member State has a national requirement to label in the national language(s), the manufacturer and distributor shall comply with that requirement for the information specified in paragraphs 3 and 4.

6. Paragraphs 1 to 5 are without prejudice to existing national rules according to which graphic representations of fruits which may lead the user into error as to the use of liquid products, shall not appear on the packaging in which the detergents are put up for sale to the consumer.

Article 12

Committee procedure

1. The Commission shall be assisted by a committee.

2. Where reference is made to this paragraph, Articles 5 and 7 of Decision 1999/468/EC shall apply, having regard to the provisions of Article 8 thereof.

The period laid down in Article 5(6) of Decision 1999/468/EC shall be set at three months.

3. The committee shall adopt its Rules of procedure.

Article 13

Adaptation of the Annexes

1. The amendments necessary for adapting the Annexes shall be adopted in accordance with the procedure referred to in Article 12(2), and shall, wherever possible, use European Standards.

2. In particular, the amendments or additions necessary for applying the rules of this Regulation to solvent-based detergents shall be adopted in accordance with the procedure referred to in Article 12(2).

Article 14

Free movement clause

Member States shall not prohibit, restrict or impede the placing on the market of detergents, and/or of surfactants for detergents, which comply with the requirements of this Regulation, on grounds that are dealt with in this Regulation.

Article 15

Safeguard clause

1. Where a Member State has justifiable grounds for believing that a specific detergent, although complying with the requirements of this Regulation, constitutes a risk to safety or health of humans or of animals or a risk to the environment, it may temporarily prohibit the placing on the market of that detergent in its territory or make it temporarily subject to special conditions.

It shall immediately inform the other Member States and the Commission thereof, giving the reasons for its decision.

2. After consultation of the Member States, or, if appropriate, of the relevant technical or scientific committee of the Commission, a decision shall be taken on the matter within ninety days in accordance with the procedure referred to in Article 12(2).

Article 16

Legislation to be repealed

1. The following Directives are hereby repealed with effect from . . . (*)

— Directive 73/404/EEC;

— Directive 73/405/EEC;

— Directive 82/242/EEC;

— Directive 82/243/EEC and

— Directive 86/94/EEC.

2. Recommendation 89/542/EEC is hereby repealed with effect from . . . (*).

3. References made to the repealed Directives shall be construed as being references to this Regulation.

4. On the day of entry into force of this Regulation, Member States shall repeal their laws, regulations and administrative provisions adopted pursuant to the Directives provided for in paragraph 1 or to the Recommendation referred to in paragraph 2.

Article 17

Sanctions

1. No later than . . . (*), Member States shall establish:

— appropriate legal or administrative measures in order to deal with any infringment of this Regulation and;

— dissuasive, effective and proportionate sanctions for any such infringment.

This shall include measures allowing them to detain consignments of detergents that fail to comply with this Regulation.

2. They shall immediately inform the Commission thereof.

Article 18

Entry into force

This Regulation shall enter into force . . . (**).

(*) Date of entry into force of this Regulation.

(**) 18 months after its publication in the *Official Journal of the European Union*.

This Regulation shall be binding in its entirety and directly applicable in all Member States.

Done at . . .

For the European Parliament	*For the Council*
The President	*The President*

ANNEX I

Standards of accreditation, good laboratory practice and animal protection concerning the laboratories that are competent and authorised to provide the necessary service for checking compliance of detergents with the requirements of this Regulation and its Annexes

1. Standards applicable at the level of the laboratories:

   EN ISO/IEC 17025, General requirements for the competence of testing and calibration laboratories;

   Directive 87/18/EEC of the Council of 18 December 1986 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 application for tests on chemical substances ([1]);

   Council Directive 86/609/EEC.

2. Standards applicable at the level of the accreditation bodies and good laboratory practice monitoring authorities:

   EN 45003, Calibration and testing laboratory accreditation system, general requirements for operation and recognition;

   Directive 88/320/EEC.

([1]) OJ L 15, 17.1.1987, p. 29. Directive as last amended by Commission Directive 1999/11/EC (OJ L 77, 23.3.1999, p. 8).

ANNEX II

Primary biodegradability test methods for surfactants in detergents

Primary biodegradability is measured by the determination in biodegraded liquors of the remaining level of parent surfactants. This Annex begins with a list of the test-methods common to all classes of surfactants, and then lists under headings A to D the analytical test procedures specific to each class of surfactant.

The pass criterion for primary biodegradability shall be a level of at least 80 %, as measured according to the test methods below.

The reference method for the laboratory testing of surfactants in this Regulation is based on the Confirmatory test procedure in the OECD method, described in Annex VIII.1. Changes to the Confirmatory test procedure are permissible provided that they comply with EN ISO 11733.

TEST METHODS

1. The OECD method published in the OECD's technical report of 11 June 1976 on the 'Proposed Method for the Determination of the Biodegradability of Surfactants in Synthetic Detergents'.

2. The method in use in France, approved by the 'arrêté du 24 décembre 1987' published in the Journal officiel de la République française of 30 December 1987, p. 15385, and by the standard NF 73-260 of June 1981, published by the Association française de normalisation (AFNOR).

3. The method in use in Germany, established by the 'Verordnung über die Abbaubarkeit anionischer und nichtionischer grenzflächenaktiver Stoffe in Wasch- und Reinigungsmitteln' of 30 January 1977, published in the Bundesgesetzblatt of 1977, Part I, p. 244, as set out in the Regulation amending that Regulation of 4 June 1986, published in the Bundesgesetzblatt of 1986, Part I, p. 851.

4. The method in use in the United Kingdom called the 'Porous Pot Test' and described in Technical Report No 70 (1978) of the Water Research Centre.

5. The 'Confirmatory test procedure' in the OECD method, described in Annex VIII.1 (including possible changes in operating conditions as proposed in EN ISO 11733). This is also the reference method used for the settlement of litigation.

A. ANALYTICAL METHODS FOR ANIONIC SURFACTANTS

The determination of anionic surfactants in the tests shall be done by the Methylene Blue Active Substance (MBAS) analysis according to the criteria established in Annex VIII.2. For those anionic surfactants not reacting to the abovementioned MBAS method, or if it seems more appropriate for reasons of efficiency or precision, appropriate specific instrumental analyses such as high performance liquid chromatography (HPLC) or gas chromatography (GC) are to be applied. Samples of the pure surfactant of interest shall be provided by the manufacturer to the competent authorities of the Member States upon request.

B. ANALYTICAL METHODS FOR NON-IONIC SURFACTANTS

The determination of non-ionic surfactants in the tests shall be done by the Bismuth Active Substance (BiAS) method, according to the analytical procedure established in Annex VIII.3. For those non-ionic surfactants not reacting to the abovementioned BiAS method, or if it seems more appropriate for reasons of efficiency or precision, appropriate specific instrumental analyses such as HPLC or GC are to be applied. Samples of the pure surfactant of interest shall be provided by the manufacturer to the competent authorities of the Member States upon request.

C. ANALYTICAL METHODS FOR CATIONIC SURFACTANTS

The determination of cationic surfactants in the tests shall be done by the Disulfine Blue Active Substance (DBAS) analysis according to the following DBAS procedures: The method in use in the Federal Republic of Germany, (1989) DIN 38 409 — Ausgabe: 1989-07. For those cationic surfactants not reacting to the abovementioned test method, or if it seems more appropriate for reasons of efficiency or precision (this must be justified) appropriate specific instrumental analyses such as HPLC or GC are to be applied. Samples of the pure surfactant of interest shall be provided by the manufacturer to the competent national authorities of the Member States upon request.

D. ANALYTICAL METHODS FOR AMPHOTERIC SURFACTANTS

The determination of amphoteric surfactants in the tests shall be done by analysis following the procedures listed below:

1. If cationics absent:

   The method in use in the Federal Republic of Germany, (1989) DIN 38 409-Teil 20.

2. Otherwise:

   Orange II method (Boiteux, 1984).

   For those amphoteric surfactants not reacting to the above-mentioned tests, or if it seems more appropriate for reasons of efficiency or precision (this must be justified) appropriate specific instrumental analyses such as HPLC or GC are to be applied. Samples of the pure surfactant of interest shall be provided by the manufacturer to the competent authorities of the Member States upon request.

ANNEX III

Ultimate biodegradability (mineralisation) test methods for surfactants in detergents

A. 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).

Surfactants in detergents shall be considered as biodegradable if the level of biodegradability (mineralisation) measured according to one of the five following tests ([1]) is at least 60 % within twenty-eight days:

1. 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). Pre-adaptation is not to be used. The ten days window principle is not applied. (Reference method).

2. Method of the Directive 67/548/EEC Annex V.C.4-C (Carbon dioxide (CO_2) Evolution Modified Sturm Test): Pre-adaptation is not to be used. The ten days window principle is not applied.

3. Method of the Directive 67/548/EEC Annex V.C.4-E (Closed Bottle): Pre-adaptation is not to be used. The ten days window principle is not applied.

4. Method of the Directive 67/548/EEC Annex V.C.4-D (Manometric Respirometry): Pre-adaptation is not to be used. The ten days window principle is not applied.

5. Method of the Directive 67/548/EEC Annex V.C.4-F (MITI: Ministry of International Trade and Industry-Japan): Pre-adaptation is not to be used. The ten days window principle is not applied.

B. Depending on the physical characteristics of the surfactant, one of the methods listed below might be used if appropriately justified ([2]). It should be noted that the pass criterion of at least 70 % of these methods is to be considered as equivalent to the pass criterion of at least 60 % referred to in methods listed in point A. The adequacy of the choice of the methods listed below shall be decided on a case by case confirmation, in accordance with Article 5 of this Regulation.

1. Method of the Directive 67/548/EEC Annex V.C.4-A (Dissolved Organic Carbon DOC Die-Away): Pre-adaptation is not to be used. The ten days window principle is not applied. The pass criteria for biodegradability measured according to the test shall be at least 70 % within twenty-eight days.

2. Method of the Directive 67/548/EEC Annex V.C.4-B (Modified OECD Screening-DOC Die-Away): Pre-adaptation is not to be used. The ten days window principle is not applied. The pass criteria for biodegradability measured according to the test shall be at least 70 % within twenty-eight days.

NB: All the abovementioned methods, taken from Council Directive 67/548/EEC, can also be found in the publication 'Classification, Packaging and Labelling of Dangerous Substances in the European Union', Part 2: 'Testing Methods'. European Commission 1997. ISBN 92-828-0076-8.

([1]) These five tests are identified as the most suitable for surfactants.

([2]) The DOC methods could give results on the removal and not on the ultimate biodegradation. The Manometric Respirometry and the MITI would not be appropriate in some cases because the high initial test concentration could be inhibitory.

ANNEX IV

Complementary risk assessment for surfactants in detergents

For those surfactants for which an environmental risk assessment is available in the context of Directive 93/67/EEC, or Regulation (EEC) No 793/93 and Regulation (EC) No 1488/94, and Technical Guidance Documents, this risk assessment shall be considered together with the complementary risk assessment run in the scope of this Regulation.

The complementary risk assessment run in the scope of this Regulation, in case it is likely that recalcitrant metabolites are produced, shall be considered in the context of assessments made on the basis of Directive 93/67/EEC or Regulation (EEC) No 793/93. This is to be assessed case by case and in particular on the basis of the results of the tests referred to in part 3.

The study shall cover the aquatic environmental compartment. Additional information relating to specific risk assessment concerns might be required by the Committee referred to in Article 12(2) on a case by case basis. Additional information might include other environmental compartments such as sewage sludge and soil. A tiered approach shall be adopted concerning the information required for the technical file referred to in Articles 5 and 9. The file shall contain at least the information described under points 1, 2 and 3 below.

However, to minimise testing, and especially to avoid unnecessary animal testing, the additional studies listed under point 4.2.2 should be requested only where such information is necessary and proportionate. In case of dispute concerning the extent of additional information required, a Decision may be taken in accordance with the procedure laid down in Article 12(2).

As noted in Article 13, the guidelines included in this Annex for the Decisions on derogation may be adapted as appropriate on the basis of the accumulated experience.

1. *Identity of the surfactant* (in accordance with the provisions laid down by Annex VII.A of Directive 67/548/EEC).

1.1. Name

1.1.1. Names in the IUPAC nomenclature

1.1.2. Other names

1.1.3. CAS number and CAS name (if available)

1.1.4. EINECS ([1]) or ELINCS ([2]) numbers (if available)

1.2. Molecular and structural formula

1.3. Composition of the surfactant

2. *Information on the surfactant*

2.1. Quantities of the surfactant used in detergents

2.2. The information on use patterns given in this section shall be sufficient to allow an approximate but realistic estimate of function and environmental exposure to the surfactant as associated with its use in detergents. It shall include the following:

— importance of the application (societal value),

— use conditions (release scenario),

— use volume,

— availability and suitability of alternatives (performance and economic considerations),

— assessment of relevant environmental information.

3. *Information on the potential recalcitrant metabolites*

Toxicity information on test liquors shall be provided. If no data are available on residue identity, the information referred to in point 4.2.1 may be requested, depending on the potential risk, the importance and the quantity of the surfactant used in detergents. In conflicting cases concerning this information, a Decision may be taken in accordance with the procedure laid down in Article 12(2).

([1]) European Inventory of Existing Commercial Substances.

([2]) European List of Notified Chemical Substances.

4. *Additional studies*

4.1. Biodegradability tests

4.1.1. Pre-adapted inoculum

Any of the tests described in Annex III, may be run with pre-adapted inoculum in order to provide evidence of the relevance of pre-adaptation for the surfactant.

4.1.2. Inherent Biodegradability Tests

At least one of the tests referred to below shall be included:

— method of the Directive 67/548/EEC, Annex V.C.12 (Modified SCAS test),

— method of the Directive 67/548/EEC, Annex V.C.9 (Zahn-Wellens).

Failure to pass the inherent biodegradability test would indicate potential for persistency which may be considered, in general terms, as sufficient to prohibit the placing on the market of such a surfactant except in cases where the criteria set out in Article 6 indicate that there is no justification for refusing a derogation.

4.1.3. Activated Sludge Simulation Biodegradability Tests

The following tests referred below shall be included:

— method of the Directive 67/548/EEC, Annex V.C.10 (including possible changes in operating conditions as proposed in EN ISO 11733).

Failure to pass the activated sludge simulation biodegradability test would indicate potential for the release of the metabolites by sewage treatment, which may be considered, in general terms, as evidence of need for a more complete risk assessment.

4.2. Toxicity testing of biodegradation test liquors

Toxicity information on test liquors is to be provided on:

4.2.1. Chemical and physical information, such as:

— identity of the metabolite (and analytical means by which it was obtained);

— key physical chemical properties (water solubility, Octanol: Water partition coefficient (Log Po/w, etc.).

4.2.2. Effects on organisms. Tests to be conducted in compliance with the principles of good laboratory practice.

Fish: The test recommended is that in Annex V.C.1 of Directive 67/548/EEC

Daphnia: The test recommended is that in Annex V.C.2 of Directive 67/548/EEC

Algae: The test recommended is that in Annex V.C.3 of Directive 67/548/EEC

Bacteria: The test recommended is that in Annex V.C.11 of Directive 67/548/EEC

4.2.3. Degradation

Biotic: The test recommended is that in Annex V.C.5 of Directive 67/548/EEC

Abiotic: The test recommended is that in Annex V.C.7 of Directive 67/548/EEC. The information to be provided will consider as well the potential of metabolites for bio-concentration and their partitioning to the sediment phase.

Moreover, if some metabolites are suspected for endocrine disrupting activity, it is recommended to determine if these have potential to result in adverse affects as soon as validated testing schemes to assess such adverse effects are available.

NB: All the abovementioned tests can also be consulted in the publication Classification, Packaging and Labelling of Dangerous Substances in the European Union; Part 2: 'Testing Methods'. European Commission 1997. ISBN 92-828-0076-8.

ANNEX V

List of surfactants that have obtained a derogation

The following detergent surfactants passing tests stipulated in Annex II, but not passing tests stipulated in Annex III, may be placed on the market by means of derogation stipulated in Article 5 and in accordance with the procedure laid down in Article 12(2):

Name in the IUPAC nomenclature	EINECS or ELINCS number	CAS number and CAS name	Limitations

'EINECS' means the European Inventory of Existing Commercial Substances. This inventory contains the definitive list of all substances deemed to be on the Community market on 18 September 1981.

'ELINCS' means the list of new substances as defined in Council Directive 92/32/EEC of 30 April 1992 amending for the seventh time Directive 67/548/EEC on the approximation of the laws, regulations and administrative provisions relating to the classification, packaging and labelling of dangerous substances [1].

[1] OJ L 154, 5.6.1992, p. 1.

ANNEX VI

List of banned or restricted detergent surfactants

The following detergent surfactants have been identified as not complying with the provisions of this Regulation:

Name in the IUPAC nomenclature	EINECS or ELINCS number	CAS number and CAS name	Limitations

ANNEX VII

Labelling and ingredient datasheet

A. Labelling for contents

The following provisions on labelling shall apply to the packaging of detergents sold to the general public.

The following 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:

— phosphates,

— phosphonates,

— anionic surfactants,

— cationic surfactants,

— amphoteric surfactants,

— non-ionic surfactants,

— oxygen-based bleaching agents,

— chlorine-based bleaching agents,

— EDTA and salts thereof,

— NTA (nitrilotriacetic acid) and salts thereof,

— phenols and halogenated phenols,

— paradichlorobenzene,

— aromatic hydrocarbons,

— aliphatic hydrocarbons,

— halogenated hydrocarbons,

— soap,

— zeolites,

— polycarboxylates.

The following classes of constituent, if added, shall be listed irrespective of their concentration:

— enzymes,

— disinfectants.

If added, preservation agents shall be listed, irrespective of their concentration, using where possible the common nomenclature established under Article 8 of Council Directive 76/768/EEC of 27 July 1976 on the approximation of laws of the Member States relating to cosmetic products ([1]).

([1]) OJ L 262, 27.9.1976, p. 169. Directive as last amended by Commission Directive 2003/83/EC (OJ L 238, 25.9.2003, p. 23).

If added, as such, at concentrations exceeding 0,01 % by weight, the allergenic fragrances that appear on the list of substances in Annex III, Part 1 of Directive 76/768/EEC, as a result of its amendment by Directive 2003/15/EC of the European Parliament and of the Council [1] to include the allergenic perfume ingredients from the list first established by the Scientific Committee on Cosmetics and Non Food Products (SCCNFP) in its opinion SCCNFP/0017/98, shall be listed using the nomenclature of that Directive, as shall any other fragrances that are subsequently added to Annex III, Part 1 of Directive 76/768/EEC by adaptation of that Annex to technical progress.

If individual risk-based concentration limits for the fragrance allergens are subsequently established by the SCCNFP, the Commission shall propose the adoption, in accordance with Article 12(2), of such limits to replace the limit of 0,01 % mentioned above.

For detergents intended to be used in the industrial sector, and not made available to members of the general public, the abovementioned requirements do not have to be fulfilled if the equivalent information is provided by means of technical data sheets, safety data sheets, or in a similar appropriate manner.

B. Labelling for dosage information

As prescribed in Article 11(4), the following provisions on labelling shall apply to the packaging of detergents sold to the general public. The packaging of detergents sold to the general public intended to be used as laundry detergents shall bear the following information:

— 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 classes and making provision for one or two cycle washing processes;

— 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;

— 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 classes.

The standard washing machine loads are 4,5 kg dry fabric for heavy-duty detergents and 2,5 kg dry fabric for low-duty detergents in line with the definitions of Commission Decision 1999/476/EC of 10 June 1999 establishing the Ecological Criteria for the award of the Community eco-label to Laundry Detergents [2]. A detergent shall be considered to be a heavy-duty detergent unless the claims of the manufacturer predominantly promotes fabric care i.e. low temperature wash, delicate fibres and colours.

C. Ingredient datasheet

The following provisions shall apply to the listing of ingredients on the datasheet referred to in Article 9(3).

The datasheet shall list the name of the detergent and of the manufacturer.

All ingredients shall be listed; in order of decreasing abundance by weight, and the list shall be sub-divided into the following weight percentage ranges:

— 10 % or more,

— 1 % or over, but less than 10 %,

— 0,1 % or over, but less than 1 %,

— less than 0,1 %.

Impurities shall not be considered to be ingredients. The common chemical name or IUPAC [3] name, the CAS number, and, where available, the INCI [4] name, and the European Pharmacopoeia name, shall be given for each ingredient.

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[1] OJ L 66, 11.3.2003, p. 26.

[2] OJ L 187, 20.7.1999, p. 52. Decision as last amended by Decision 2003/200/EC (OJ L 76, 22.3.2003, p. 25).

[3] International Union of Pure and Applied Chemistry.

[4] International Nomenclature Cosmetic Ingredient.

ANNEX VIII

TEST METHODS AND ANALYTICAL METHODS

The following test and analytical methods apply to control procedures for detergents on the market carried out by Member States:

1. **Reference method** (confirmatory test)

1.1. Definition

This method describes a laboratory model of the activated sludge + secondary settler which is designed to simulate municipal sewage treatment. The conditions described are those from the Directives that preceded this Regulation. 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 (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 1 000 ml.

2.2.2. Neutral methylene blue solution

Dissolve 0,35 g methylene blue AR in deionised water and dilute to 1 000 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 1 000 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, 1 000 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 (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 (2.2.11). The volume of sample should contain between 20 and 150 µg 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 (2.2.1), 5 ml of neutral methylene blue solution (2.2.2) and 15 ml of chloroform (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 (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 (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 (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 (2.2.5) to the nearest 0,1 mg in a round-bottomed flask and add 50 ml of ethanolic potassium hydroxide solution (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 1 000 ml graduated flask (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 (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 item 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 (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 µg.

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 1 000 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 1 000 ml measuring flask, add 200 ml glacial acetic acid (3.2.7) and make up to 1 000 ml with water.

3.2.6.2. Solution B

Dissolve 290 g barium chloride, $BaCl_2.2H_2O$ AR, in 1 000 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 1 000 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 1 000 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 (3.2.7). Mix thoroughly, cool and transfer to a 1 000 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 1 000 ml with water.

3.2.12. Copper sulphate solution (for standardisation of 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 1 000 ml with water.

STANDARD SOLUTION

Mix 50 ml stock solution with 10 ml 0,5 M H_2SO_4 and make up to 1 000 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 μm.

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 μg 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 (3.2.3) and stir the mixture with a magnetic stirrer.

To this solution add 30 ml of precipitating agent (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) (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 (3.2.16), add a few drops of bromocresol purple (3.2.5) and add the dilute ammonia solution (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 (3.2.10), immerse the electrodes in the solution, and titrate potentiometrically with standard 'carbate solution' (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 3.3.2. The blank titration should be below 1 ml, otherwise the purity of the reagents (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 (3.2.12) with 'carbate solution' after the addition of 100 ml water and 10 ml standard acetate buffer (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:

$(b-c) \times f \times 0{,}054$ = 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.

4. **Preliminary treatment of anionic surfactants to be tested**

4.1. Preliminary notes

4.1.1. Treatment of samples

The treatment of anionic surface-active agents and formulated detergents prior to the determination of primary biodegradability in the confirmatory test is:

Products	Treatment
Anionic surfactants	None
Formulated detergents	Alcoholic extraction followed by separation of the anionic surfactants by ion exchange

The purpose of the alcoholic extraction is to eliminate the insoluble and inorganic ingredients of the commercial product, which in some circumstances might upset the biodegradability test.

4.1.2. Ion-exchange procedure

Isolation and separation of anionic surface active agents from soap, non-ionic and cationic surfactants are required for correct biodegradability tests.

This is achieved by an ion-exchange technique using a macro-porous exchange resin and suitable eluants for fractional elution. Thus soap, anionic and non-ionic surfactants may be isolated in one procedure.

4.1.3. Analytical control

After homogenising, the concentration of anionic surfactants in the synthetic detergent is determined according to the MBAS analytical procedure. The soap content is determined by a suitable analytical method.

This analysis of the products is necessary to calculate the quantities required for preparing fractions for the biodegradability test.

Quantitative extraction is not necessary; however, at least 80 % of the anionic surfactants should be extracted. Usually, 90 % or more is obtained.

4.2. Principle

From a homogeneous sample (powders, dried pastes and dried liquids) an ethanol extract is obtained which contains the surfactants, soap and other alcohol-soluble constituents of the synthetic detergent sample.

The ethanol extract is evaporated to dryness, dissolved in an isopropanol/water mixture and the solution obtained is passed through a strongly acidic cation exchange/macro-porous anion exchange combination heated to 50 °C. This temperature is necessary to prevent the precipitation of any fatty acids which may be present in acidic media.

Any non-ionic surfactants remain in the effluent.

Soap fatty acids are separated by extraction with ethanol containing CO_2. The anionic surfactants are then obtained as ammonium salts, by elution with an aqueous isopropanolic solution of ammonium bicarbonate. These ammonium salts are used for the degradation test.

Cationic surfactants that might upset the biodegradability test and the analytical procedure are eliminated by the cation exchanger placed above the anion exchanger.

4.3. Chemicals and equipment

4.3.1. Deionised water

4.3.2. Ethanol, 95 % (v/v) C_2H_5OH (permissible denaturant: methyl ethyl ketone or methanol)

4.3.3. Isopropanol/water mixture (50/50 v/v):

— 50 parts by volume isopropanol, $CH_3CHOH.CH_3$, and

— 50 parts by volume water (4.3.1)

4.3.4. Solution of carbon dioxide in ethanol (approximately 0,1 % CO_2): using a delivery tube with a built-in sinter, pass carbon dioxide, CO_2, through the ethanol (4.3.2) for ten minutes. Use fresh solutions only

4.3.5. Ammonium bicarbonate solution (60/40 v/v): 0,3 mol NH_4HCO_3 in 1 000 ml of an isopropanol/water mixture consisting of 60 parts by volume isopropanol and 40 parts by volume water (4.3.1)

4.3.6. Cation exchanger (KAT), strongly acidic, resistant to alcohol (50-100 mesh)

4.3.7. Anion exchanger (AAT), macro-porous, Merck Lewatit MP 7080 (70-150 mesh) or equivalent

4.3.8. Hydrochloric acid, 10 % HCl (w/w)

4.3.9. 2 000 ml round-bottomed flask with ground glass stopper and reflux condenser

4.3.10. 90 mm diameter suction filter (heatable) for filter papers

4.3.11. 2 000 ml filter flask

4.3.12. Exchange columns with heating jacket and tap: inner tube 60 mm in diameter and 450 mm in height (see *Figure 4*)

4.3.13. Water-bath

4.3.14. Vacuum drying oven

4.3.15. Thermostat

4.3.16. Rotary evaporator

4.4. Preparation of extract and separation of anionic active agents

4.4.1. Preparation of extract

The quantity of surfactants necessary for the biodegradation test is about 50 g MBAS.

Normally, the quantity of product to be extracted will not exceed 1 000 g, but it may be necessary to extract further quantities of sample. For practical reasons, the quantity of product used should in most cases be limited to 5 000 g in preparing extracts for the biodegradation test.

Experience has shown that there are advantages in using a number of small extractions rather than one large extraction. The exchanger quantities specified are designed for a working capacity of 600-700 mmoles of surfactants and soap.

4.4.2. Isolation of alcohol-soluble constituents

Add 250 g of the synthetic detergent to be analysed to 1 250 ml ethanol, heat the mixture to boiling point and reflux for one hour with stirring. Pass the hot alcoholic solution through a coarse-pored suction filter heated to 50 °C and filter rapidly. Wash the flask and suction filter with approximately 200 ml hot ethanol. Collect the filtrate and filter washings in a filter flask.

In the case of pastes or liquid products to be analysed, make sure that not more than 55 g anionic surfactants and 35 g soap are contained in the sample. Evaporate this weighed sample to dryness. Dissolve the residue in 2 000 ml ethanol and proceed as described above.

In the case of powders of low apparent density (< 300 g/l) it is recommended to increase the ethanol ratio in the relation 20:1. Evaporate the ethanolic filtrate to dryness, preferably by means of a rotary evaporator. Repeat the operation if a greater quantity of extract is required. Dissolve the residue in 5 000 ml isopropanol/water mixture.

4.4.3. Preparation of ion-exchange columns

CATION-EXCHANGE COLUMN

Place 600 ml cation-exchange resin (4.3.6) in a 3 000 ml beaker and cover by adding 2 000 ml hydrochloric acid (4.3.8). Allow to stand for at least two hours, with occasional stirring.

Decant the acid and transfer the resin into the column (4.3.12) by means of deionised water. The column should contain a glass-wool plug.

Wash the column with deionised water at a rate of 10-30 ml/min until the eluate is free of chloride.

Displace the water with 2 000 ml isopropanol/water mixture (4.3.3) at a rate of 10-30 ml/min. The exchange column is now ready for operation.

ANION-EXCHANGE COLUMN

Place 600 ml anion-exchange resin (4.3.7) in a 3 000 ml beaker and cover by adding 2 000 ml deionised water.

Allow the resin to swell for at least two hours.

Transfer the resin into the column by means of deionised water. The column should contain a glass-wool plug.

Wash the column with 0,3 M ammonium bicarbonate solution (4.3.5) until free of chloride. This requires about 5 000 ml solution. Wash again with 2 000 ml deionised water. Displace the water with 2 000 ml isopropanol/water mixture (4.3.3) at a rate of 10-30 ml/min. The exchange column is now in the OH-form and ready for operation.

4.4.4. Ion-exchange procedure

Connect the exchange columns so that the cation-exchange column is placed on top of the anion-exchange column.

Heat the exchange columns to 50 °C using thermostatic control.

Heat 5 000 ml of the solution obtained in item 4.4.2 to 60 °C and pass the solution through the exchanger combination at a rate of 20 ml/min. Wash the columns with 1 000 ml hot isopropanol/water mixture (4.3.3).

To obtain the anionic surface active agents (MBAS), disconnect the KAT column. Using 5 000 ml ethanol/CO_2 solution at 50 °C (4.3.4), elute the soap fatty acids out of the KAT column. Reject the eluate.

Then elute the MBAS out of the AAT column with 5 000 ml ammonium bicarbonate solution (4.3.5). Evaporate the eluate to dryness using a steam bath or in a rotary evaporator.

The residue contains the MBAS (as ammonium salt) and possible non-surfactant anionics that have no detrimental effect on the biodegradation test. Add deionised water to the residue until a definite volume is obtained and determine the MBAS content in an aliquot. The solution is used as a standard solution of the anionic synthetic detergents for the biodegradation test. The solution should be kept at a temperature below 5 °C.

4.4.5. Regeneration of ion exchange resins

The cation exchanger is rejected after use.

Passing an additional quantity of ammonium bicarbonate solution (4.3.5) down the column at a flow rate of approximately 10 ml/min until the eluate is free from anionic surfactants (methylene blue test) regenerates the anion-exchange resin.

Then pass 2 000 ml isopropanol/water mixture (4.3.3) down the anion exchanger to wash. The anion exchanger is again ready for operation.

5. Preliminary treatment of non-ionic surfactants to be tested

5.1. Preliminary notes

5.1.1. Treatment of samples

The treatment of non-ionic surface-active agents and formulated detergents prior to the determination of primary biodegradability in the confirmatory test is:

Products	Treatment
Non-ionic surfactants	None
Formulated detergents	Alcoholic extraction followed by separation of the non-ionic surfactants by ion exchange

The purpose of the alcoholic extraction is to eliminate the insoluble and inorganic ingredients of the commercial product, which in some circumstances might upset the biodegradability test.

5.1.2. Ion-exchange procedure

Isolation and separation of non-ionic surface active agents from soap, anionic and cationic surfactants are required for correct biodegradability tests.

This is achieved by an ion exchange technique using a macro-porous exchange resin and suitable eluants for fractional elution. Thus soap, anionic and non-ionic surfactants may be isolated in one procedure.

5.1.3. Analytical control

After homogenising, the concentration of anionic and non-ionic surfactants in the detergent is determined according to the MBAS and BiAS analytical procedure. The soap content is determined by a suitable analytical method.

This analysis of the product is necessary to calculate the quantities required for preparing fractions for the biodegradability tests.

Quantitative extraction is not necessary; however, at least 80 % of the non-ionic surfactants should be extracted. Usually, 90 % or more is obtained.

5.2. Principle

From a homogeneous sample (powders, dried paste and dried liquids) an ethanol extract is obtained which contains the surfactants, soap and other alcohol-soluble constituents of the detergent sample.

The ethanol extract is evaporated to dryness, dissolved in an isopropanol/water mixture and the solution obtained is passed through a strongly acidic cation exchange/macro-porous anion exchange combination heated to 50 °C. This temperature is necessary to prevent the precipitation of any fatty acids which may be present in acidic media. The non-ionic surfactants are obtained from the effluent by evaporation.

Cationic surfactants, which might upset the degradation test and the analytical procedure, are eliminated by the cation exchanger placed above the anion exchanger.

5.3. Chemicals and equipment

5.3.1. Deionised water

5.3.2. Ethanol, C_2H_5OH 95 % (v/v) (permissible denaturant: methyl-ethyl ketone or methanol)

5.3.3. Isopropanol/water mixture (50/50 v/v):

— 50 parts by volume isopropanol, $CH_3CHOH.CH_3$, and

— 50 parts by volume water (5.3.1)

5.3.4. Ammonium bicarbonate solution (60/40 v/v):

0,3 mol NH_4HCO_3 in 1 000 ml of an isopropanol/water mixture consisting of 60 parts by volume isopropanol and 40 parts by volume water (5.3.1)

5.3.5. Cation exchanger (KAT), strongly acidic, resistant to alcohol (50-100 mesh)

5.3.6. Anion exchanger (AAT), macro-porous, Merck Lewatit MP 7080 (70-150 mesh) or equivalent

5.3.7. Hydrochloric acid, 10 % HCl w/w

5.3.8. 2 000 ml round-bottomed flask with ground glass stopper and reflux condenser

5.3.9. 90 mm diameter suction Filter (heatable) for filter papers

5.3.10. 2 000 ml filter flask

5.3.11. Exchange columns with heating jacket and tap: inner tube 60 mm in diameter and 450 mm in height (see *Figure 4*)

5.3.12. Water-bath

5.3.13. Vacuum drying oven

5.3.14. Thermostat

5.3.15. Rotary evaporator

5.4. Preparation of extract and separation of non-ionic active agents

5.4.1. Preparation of extract

The quantity of surfactant necessary for the degradation test is about 25 g BiAS.

In preparing extracts for the degradation tests, the quantity of product to be used should be limited to a maximum of 2 000 g. Therefore it may be necessary to carry out the operation two or more times in order to obtain sufficient quantity for the degradation tests.

Experience has shown that there are advantages in using a number of small extractions rather than one large extraction.

5.4.2. Isolation of alcohol-soluble constituents

Add 250 g of the synthetic detergent to be analysed to 1 250 ml ethanol and heat the mixture to boiling point and reflux for one hour with stirring. Pass the hot alcoholic solution through a coarse-pored suction filter heated to 50 °C and filter rapidly. Wash the flask and suction filter with approximately 200 ml hot ethanol. Collect the filtrate and filter washings in a filter flask.

In the case of pastes or liquid products to be analysed, make sure that not more than 25 g anionic surfactants and 35 g soap are contained in the sample. Evaporate this weighed sample to dryness. Dissolve the residue in 500 ml ethanol and proceed as described above.

In the case of powders of low apparent density (< 300 g/l) it is recommended to increase the ethanol ratio in the relation 20:1.

Evaporate the ethanolic filtrate to complete dryness, preferably by means of rotary evaporator. Repeat the operation if a greater quantity of extract is required. Dissolve the residue in 5 000 ml isopropanol/water mixture.

5.4.3. Preparation of ion-exchange columns

CATION-EXCHANGE COLUMN

Place 600 ml cation-exchange resin (5.3.5) in a 3 000 ml beaker and cover by adding 2 000 ml hydrochloric acid (5.3.7). Allow to stand for at least two hours, with occasional stirring.

Decant the acid and transfer the resin into the column (5.3.11) by means of deionised water. The column should contain a glass-wool plug. Wash the column with deionised water at a rate of 10-30 ml/min until the eluate is free of chloride.

Displace the water with 2 000 ml isopropanol/water mixture (5.3.3) at a rate of 10-30 ml/min. The exchange column is now ready for operation.

ANION-EXCHANGE COLUMN

Place 600 ml anion-exchange resin (5.3.6) in a beaker and cover by adding 2 000 ml deionised water. Allow the resin to swell for at least two hours. Transfer the resin into the column by means of deionised water. The column should contain a glass-wool plug.

Wash the column with 0,3 M ammonium bicarbonate solution (5.3.4) until free of chloride. This requires about 5 000 ml solution. Wash again with 2 000 ml deionised water.

Displace the water with 2 000 ml isopropanol/water mixture (5.3.3) at a rate of 10-30 ml/min. The exchange column is now in the OH form and ready for operation.

5.4.4. Ion-exchange procedure

Connect the exchange columns so that the cation-exchange column is placed on top of the anion-exchange column. Heat the exchange columns to 50 °C using thermostatic control. Heat 5 000 ml of the solution obtained in item 5.4.2 to 60 °C and pass the solution through the exchanger combination at a rate of 20 ml/min. Wash the columns with 1 000 ml hot isopropanol/water mixture (5.3.3).

To obtain the non-ionic surfactants collect the filtrate and filter washings and evaporate to dryness, preferably by means of a rotary evaporator. The residue contains the BiAS. Add deionised water until a defined volume is obtained and determine the BiAS content in an aliquot. The solution is used as a standard solution of non-ionic surfactants for the degradation test. The solution should be kept at a temperature below 5 °C.

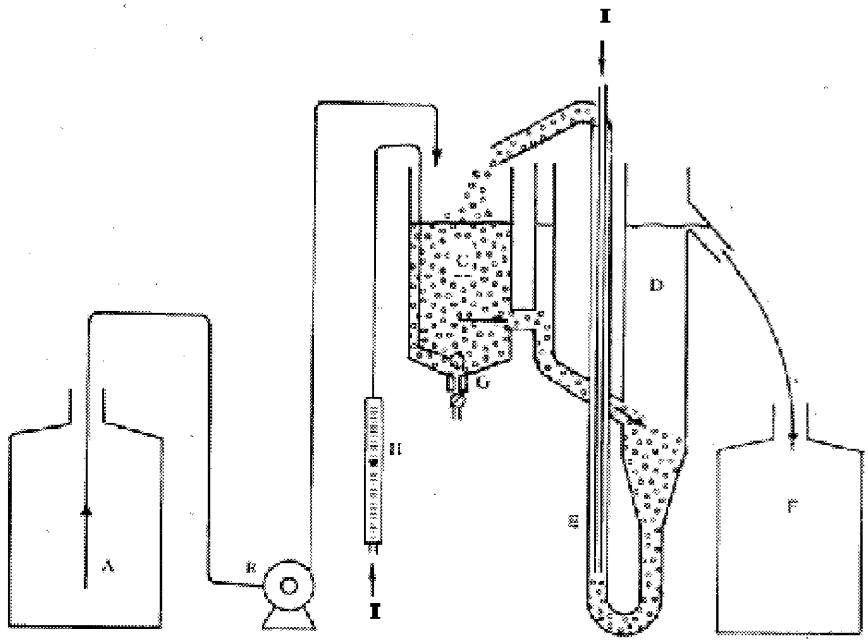
5.4.5. Regeneration of ion exchange resins

The cation exchanger is rejected after use.

Passing about 5 000-6 000 ml of ammonium bicarbonate solution (5.3.4) down the column at a flow rate of approximately 10 ml/min until the eluate is free from anionic surfactants (methylene blue test) regenerates the anion-exchange resin. Then pass 2 000 ml isopropanol/water mixture (5.3.3) down the anion exchanger to wash. The anion exchanger is again ready for operation.

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

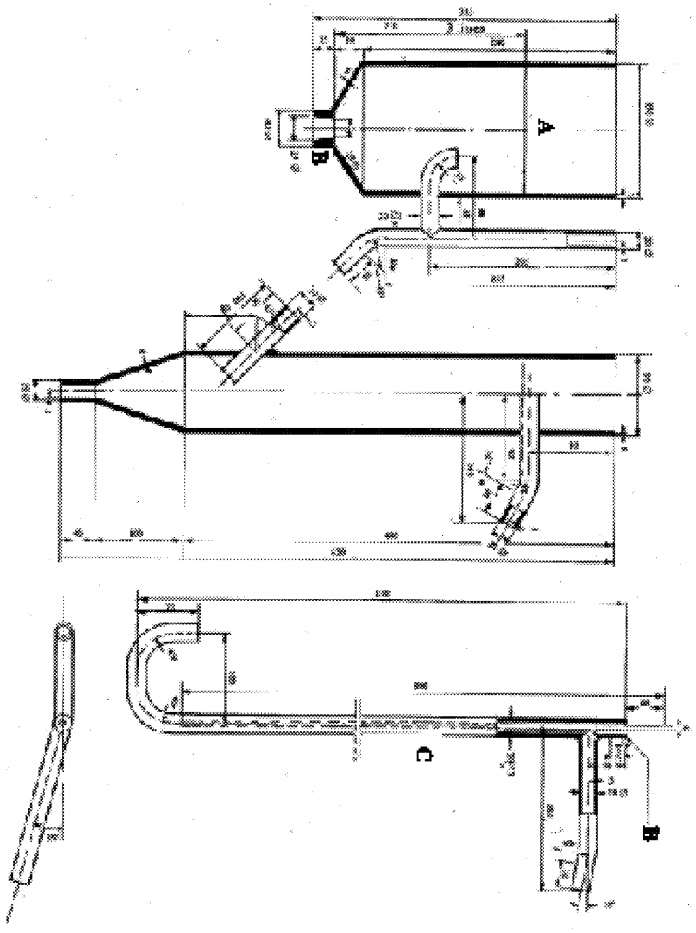
H. Air-flow meter

I. Air

Figure 2

Activated sludge plant: detail

(dimensions in millimetres)



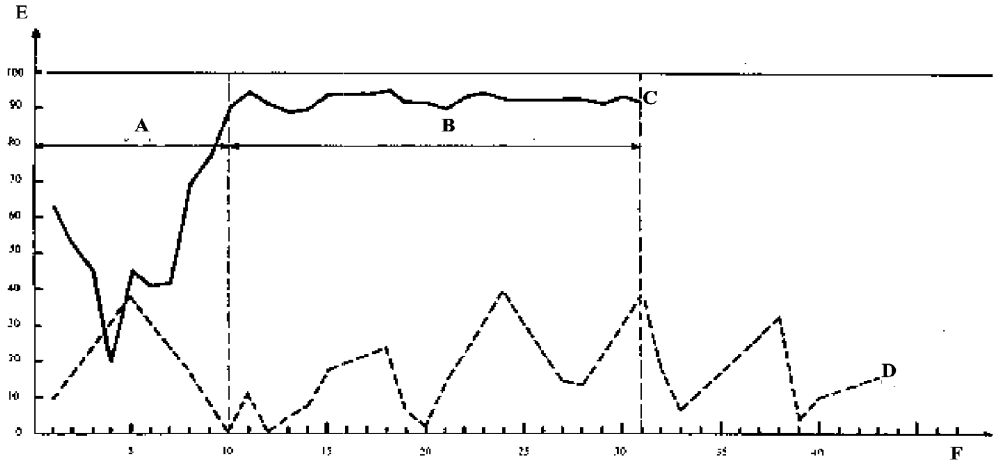
A. Liquid level

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)

Figure 4

Heated exchange column

(dimensions in millimetres)

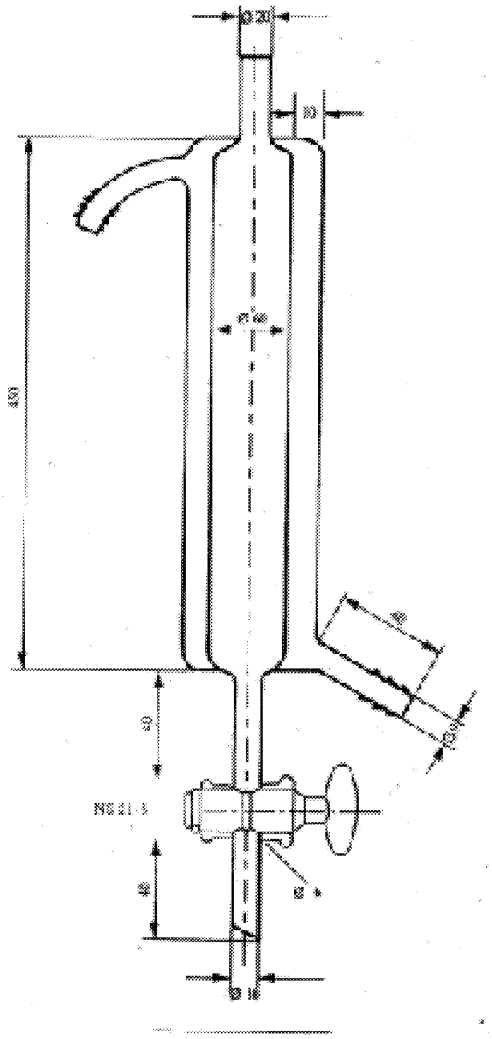
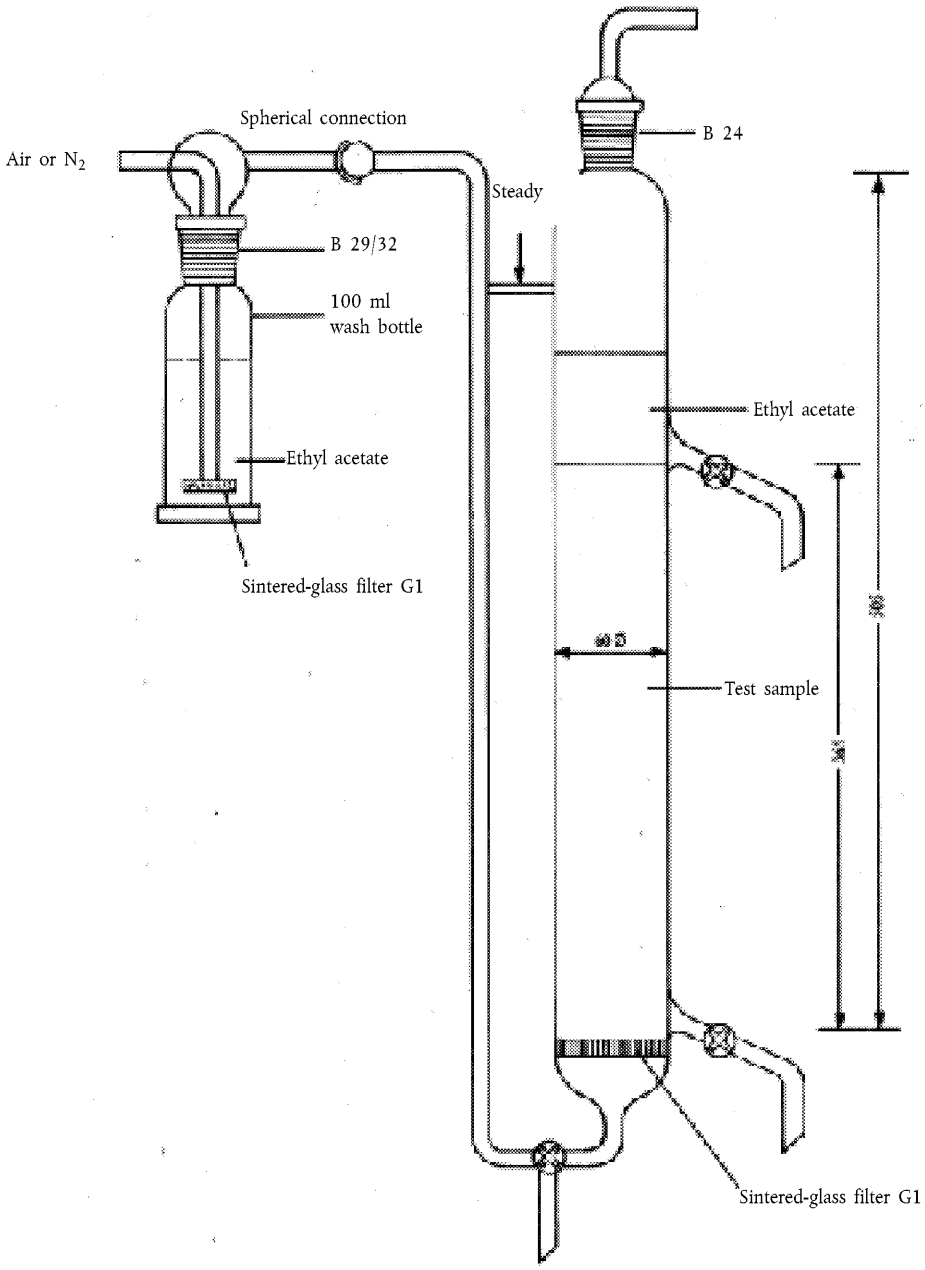


Figure 5

Gas stripping apparatus

(dimensions in millimetres)



STATEMENT OF THE COUNCIL'S REASONS

I. INTRODUCTION

On 4 September 2002 the Commission presented its proposal (12319/02 ENT 129 ENV 507 CODEC 1146).

On 26 February 2003 the Economic and Social Committee delivered its Opinion (Opinion CESE 282/2003 ([1])).

On 10 April 2003 the European Parliament concluded its first reading, adopting a number of amendments (doc. 8310/03 CODEC 448 ENT 59 ENV 213).

On 19 May 2003 the Council (Competitiveness) reached a political agreement by qualified majority on the Presidency text set out in doc. 9096/03 ENT 78 ENV 263 CODEC 610, which after legal/linguistic revision, is now the text of the Common Position, set out in doc. 10595/03.

On 4 November 2003 the Council agreed, pursuant to Article 251 par. 2 of the Treaty, the Common position and the Statement of the Council's reasons.

II. OBJECTIVE

The objective of this Regulation is to ensure the free movement of detergents in the Internal Market while at the same time providing a high level of protection for human health and for the environment. In particular it modernises the existing detergent directives by including provisions on the biodegradability of surfactants. The proposed new biodegradability tests ensure a higher level of environmental protection and are applicable to all types of surfactants in detergents. Furthermore, it makes binding the requirements for the products covered by this Regulation specified in Commission Recommendation 89/542/EEC on labelling of detergents and cleaning products and on specific information to consumers on the presence of fragrance substances in detergents.

III. ANALYSIS OF THE COMMON POSITION AS SET OUT IN DOC. 10595/03

1. General

The Common Position modifies the Commission Proposal by attempting to cater for the following requirements:

— better definition of the parties intervening in the authorisation or derogation process;

— clarification of the scope (inter alia by defining the boundaries with respect to biocidal products);

— need to inform and to protect consumers;

— providing clearer guidelines for approved laboratories and for testing methods.

Amendments 2, 4, 8, 17, and 53 of the E.P. were accepted with their original wording, as these amendments contribute to clarify the text and improve some definitions.

Amendments 3, 18, 27, 34, 35, 36, 37, 45, 46, 52, 55 and 57 were accepted in part, or in substance, as they either further clarify the text or some parts thereof, or else because they add provisions which may ease implementation of the Regulation.

All other amendments were rejected (see also item IV hereafter).

([1]) OJ C 95, 23.4.2003, p. 24.

2. **New elements contained in the Common Position as compared with the Commission proposal**

Article 1 — Objectives and scope

This Article has been modified, strengthening the importance of biodegradability for surfactants and underlining the importance of the information, contained in the technical file, that manufacturers must hold at the disposal of the Member States. It has also been stressed that, besides the environment, one of the objectives is to protect the human health, as requested by the E.P. in Amendment 53.

Amendment 15 of the E.P. was rejected.

Article 2 — Definitions

Besides some redrafting to clarify some definitions (as requested by E.P. in Amendment 46), new definitions were added for 'medical personnel' and for 'industrial and institutional detergent' (as requested by E.P. in Amendment 18). The Amendments 16, 17 and 46 were partially taken into account, though with a different wording.

Article 3 — The placing on the market

The distinction between detergents and surfactants, on the one hand, and biocidal products, on the other hand, has been included in this Article.

Article 4 — Limitations on biodegradability of surfactants

The link between aerobic biodegradation of surfactants and limitations as regard placing on the market has been clarified.

Amendments 20, 55/Rev and 56 of the E.P. were rejected.

Article 5 — Granting of derogations

Derogations are now subject to fees, a tiered approach for tests has been introduced and various deadlines have been added to this Article.

Parts of Amendment 57 of the E.P. were included.

Article 6 — Refusal of derogation

Refusal of derogation will henceforth be decided by the Commission assisted by the Committee set up by Art. 12. Also, deadlines have been introduced together with the obligation to publish a list of surfactant not complying with this Regulation.

Amendment 47 of the E.P. was rejected.

Article 7 — Testing of surfactants

Testing requirements have been clarified.

Amendment 25 of the E.P. was rejected.

Article 8 — Duties of the Member States

Criteria for appointing approved laboratories have been modified, by introducing a reference to the norm EN ISO/IEC 17025 and to good laboratory practices.

Article 9 — Information to be provided by manufacturers

This Article has been modified by slightly rewording it and by introducing the notion of a public body which, in a Member State, may circulate the information received to medical personnel.

Amendment 26 of the E.P. was rejected, whilst Amendment 27 was partially taken into account.

Article 10 — Control measures

This Article has been slightly reworded for clarity and the obligation for the Commission to verify false positive results has been introduced.

Article 11 — Labelling

Amendments to this Article were made to better identify the party responsible for placing on the market the detergent and to allow for national provisions forbidding displaying on the package of liquid products symbols (e.g. fruits) which might lead the consumer into error as to the use of the product (essentially to protect children).

Amendments 28 and 48 of the E.P. were rejected.

Article 12 — Committee procedure

The text was changed to the standard legal text for this type of legislative Articles

Article 13 — Adaptation of the annexes

Minor drafting changes were made to this Article, also stressing *inter alia* that all amendments to rules on solvent-based detergents will be made by Committee procedure (Article 12).

Amendments 29 and 30 of the E.P. were rejected.

Article 14 — Free movement clause

A legal clarification as to when products complying with this Regulation may be prevented from being placed on the market was introduced.

Amendment 59 of the E.P. was rejected.

Article 15 — Safeguard clause

Amendments 32, 33 and 60 were rejected.

Article 16 (Legislation to be replaced) and 17 (Sanctions for non-compliance)

Minor legal redrafting changes were made to both Articles.

Annex I — Standards for accreditation

References to good laboratory practices (GLP) and to animal protection were added.

Annex II — Testing methods

It was specified that in certain cases high performance liquid cromatography (HPLC) or gas cromatography (GC) should be used.

Amendments 34, 35, 36 and 37 of the E.P. were accepted. Amendment 61 was rejected.

Annex III — Biodegradability tests

Footnotes were renumbered.

Amendments 49 and 62 of the E.P. were rejected.

Annex IV

A tiered approach for the technical file was introduced, the relation between failing a test and refusing a derogation was reworded and the header of a section dedicated to biodegradability tests was reworded. Some footnotes were renumbered.

Amendment 39 of the E.P. was rejected.

Annex V

A footnote was renumbered.

Annex VII

Annex VII was deleted. Amendment 40 of the E.P. was rejected.

Annex VIII — Labelling and ingredient datasheet

An amendment was made to include salts of some compounds previously already listed.

The prescription to list allergenic fragrances if superior to a 0,01 % concentration was introduced. Consumer information as to the expected number of loads which may be washed with a package content and the standard dose recommended for a specific type of wash was added. Some footnotes were renumbered.

Amendments 41, 42, 43 and 44 of the E.P. were rejected. Amendment 45 was partially taken into account.

Recital 1

Amendment 1 of the E.P. asking for a new Recital 1 was rejected.

Recital 9

A minor drafting change was made, as Amendment 2 of the E.P. was accepted.

Recital 14

An amendment linking surfactants failing some test and a complementary risk assessment was introduced. It includes most of Amendment 3 of the E.P.

Recital 16

The reference to considering the determination of criteria for derogations as a technical matter was deleted.

Amendment 52 of the E.P. was rejected.

Recital 18

Following Amendment 4 of the E.P., a minor redrafting was made by breaking the sentence into two and by underlining the need for additional assessments.

Recital 20

An amendment was made to refer to the Committee procedure by way of a standard legal text.

Recital 21

The list of horizontal legislation applicable was substantially extended.

Amendment 5 of the E.P. was rejected.

Recital 23

Amendment 6 of the E.P. was rejected.

Recital 24

Amendment 7 of the E.P. was rejected.

Recital 25

Changes were made to reflect the new terminology (medical personnel), as asked in Amendment 8 of the E.P., and to reflect the amendment to Art. 9 par.3.

Recital 27

Amendment 9 of the E.P. was rejected.

Recital 28

Amendment 10 of the E.P. was rejected.

Recital 30

It was specified that good laboratory practices (GLP) are an alternative to using the EN ISO/IEC 17025 norm.

Recital 31

A clarification was made on which areas may be examined again by the Commission prior to proposing new legislation.

Amendments 11, 12, 13 and 14 of the E.P. were rejected.

IV. **CONCLUSION**

The Common Position clarifies further the text of the draft Regulation, improves the definition of the procedural aspects of its implementation, and further reinforces the consumer's information and protection in this product area.

Amendments 2, 3, 4, 8, 18, 34, 35, 36, 37, 46 (partially), 53, 57 (partially) of the European Parliament were accepted in full or partially, while amendments 1, 5, 6, 7, 9, 10 to 17, 20, 24 to 30, 32, 33, 38 to 45, 47 to 49, 52, 55 to 62 were rejected.