

Specification for scouring powder

AMENDMENT NO. 2

January 2013

1. Page 4, Foreword

Insert the following acknowledgement after reference item 8:

Appendix H and I were extracted with permission from Annex II and Annex III respectively of the EC Regulation No. 648/2004 of the European Parliament and of the Council of 31 March 2004.

2. Page 5, 3.1 Description

Replace Paragraph 1 of the subclause with the following text:

The scouring powder shall consist essentially of a finely divided abrasive together with anionic surface active agent (surfactants) with suitable additives and builders. The abrasive shall be powdered hard natural rock. The surface active agent shall be biodegradable. If there is a need to test for biodegradability, the tests should be conducted in accordance with the methods indicated in Appendix H or I.

3. Page 6, Table 1

Amend 'Synthetic anionic detergents, % (m/m)' to 'Anionic surface active agents (surfactants), % (m/m)'.

4. Page 7, Clause 8 Marking

Delete the Note and the SISIR mark.

5. New appendices

Insert the following two new informative appendices after Appendix G.

Appendix H (informative)

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 Appendix 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 criteria 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 standard is based on the Confirmatory test procedure in the OECD method, described in Annex VIII.1 of EC Regulation No. 648/2004 of the European Parliament and of the Council of 31 March 2004. 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 'arrete du 24 decembre 1987' published in the Journal officiel de la Republique francaise of 30 December 1987, p. 15385, and by the standard NF 73-260 of June 1981, published by the Association francaise de normalization (AFNOR).
- (3) The method in use in Germany, established by the 'Verordnung uber die Abbaubarkeit anionischer und nichtionischer grenzflachenaktiver 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 of EC Regulation No. 648/2004 of the European Parliament and of the Council of 31 March 2004 (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 of EC Regulation No. 648/2004 of the European Parliament and of the Council of 31 March 2004.

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 upon request.

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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 of EC Regulation No. 648/2004 of the European Parliament and of the Council of 31 March 2004.

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 surfactants of interest shall be provided by the manufacturer to the competent authorities 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 DGAS procedures:

The method in use in the Federal Republic of Germany, (1989) DIN 38 409 – Ausgabe: 1989-07.

For the cationic surfactants not reacting to the abovementioned test method, or if it seems more appropriate for reasons of efficiency or precision (this shall 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 upon request.

D. Analytical methods for amphoteric surfactants

The determination of amphoteric surfactants in the test 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 abovementioned tests, or if it seems more appropriate for reasons of efficiency or precision (this shall 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 upon request.

Appendix I (informative)

Ultimate biodegradability (mineralisation) test methods for surfactants in detergents

- A. The reference method for laboratory testing of surfactant ultimate biodegradability in this Standard is based on the EN ISO standard 14593: 1999 (CO₂ headspace test).

Surfactants in detergents shall be considered as biodegradable if the level of biodegradability (mineralisation) measured according to one of the following tests ⁽¹⁾ is at least 60% within 28 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 (CO₂ headspace test). Pre-adaptation is not to be used. The 10-day window principle is not applied (reference method).
- (2) Directive 67/548/EEC method, Annex V.C.4-C (Carbon dioxide (CO₂) Evolution modified Sturm test): pre-adaption is not to be used. The 10-day window principle is not applied.
- (3) Directive 67/548/EEC method, Annex V.C.4-E (Closed Bottle): pre-adaption is not to be used. The 10-day window principle is not applied.
- (4) Directive 67/548/EEC method, Annex V.C.4-D (Manometric Respirometry): pre-adaption is not to be used. The 10-day window principle is not applied.
- (5) Directive 67/548/EEC method, Annex V.C.4-F (MITI: Ministry of International Trade and Industry, Japan): pre-adaption is not to be used. The 10-day window principle is not applied.
- (6) ISO 10708: 1997 – Water quality – Evaluation in an aqueous medium of the ultimate aerobic biodegradability of organic compounds – Determination of biomedical oxygen demand in a two-phase closed bottle test. Pre-adaption is not to be used. The 10-day window principle is not applied.

- B. Depending on the physical characteristics of the surfactant, one of the methods listed below may 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 Clause 5 of this standard.

- (1) Directive 67/548/EEC method, Annex V.C.4-A (dissolved organic carbon DOC die-away): pre-adaption is not to be used. The 10-day window principle is not applied. The pass criteria for biodegradability measured according to the test shall be at least 70% within 28 days.
- (2) Directive 67/548/EEC method, Annex V.C.4-B (modified OECD screening-DOC die-away): pre-adaption is not to be used. The 10-day window principle is not applied. The pass criteria for biodegradability measured according to the test shall be at least 70% within 28 days.

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