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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 December 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 above-mentioned 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.

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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 (CO₂ 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 (CO₂ 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 (CO₂) 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.

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