II

(Acts whose publication is not obligatory)

COUNCIL

COUNCIL DIRECTIVE

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 and amending Directive 73/404/EEC

(82/242/EEC)

THE COUNCIL OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community, and in particular Article 100 thereof,

Having regard to the proposal from the Commission (1),

Having regard to the opinion of the European Parliament (2),

Having regard to the opinion of the Economic and Social Committee (3),

Whereas the methods of testing in force in the Member States, while pursuing the same objective, differ in certain respects and are thus detrimental to the proper functioning of the common market;

Whereas Council Directive 73/404/EEC of 22 November 1973 on the approximation of the laws of the Member States relating to detergents (4) provides in Article 4 for the adoption of Directives specifying methods of testing as well as relevant tolerances to enable compliance with the requirements of that Directive to be established; whereas 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) specified such methods and tolerances for anionic surfactants;

Whereas, to enable Member States to determine the level of biodegradability of non-ionic surfactants, it is advisable to employ methods of testing already in use for this purpose in certain Member States; whereas, however, in the event of disputes, biodegradability should be tested by a common reference method;

Whereas, in regard to the approximation of the laws of the Member States relating to detergents, suitable tolerances for measuring biodegradability should be laid down, as provided for in Article 4 of Council Directive 73/404/EEC, in order to safeguard against the unreliability of methods of testing which could lead to rejection decisions having considerable economic consequences; whereas a rejection decision must be taken only if the results obtained by an analytical method mentioned in Article 2 show a level of biodegradability lower than 80 %;

(1) OJ No C 104, 28. 4. 1980, p. 112.
(2) OJ No C 197, 4. 8. 1980, p. 66.
(3) OJ No C 310, 30. 11. 1981, p. 7.
(4) OJ No L 347, 17. 12. 1973, p. 51.
(5) OJ No L 347, 17. 12. 1973, p. 53.

Whereas for the time being small quantities of certain non-ionic surfactants of low biodegradability must be used for some purposes because of technical problems and in order to prevent other undesirable effects on health and the environment; whereas it will nevertheless be necessary to have the opportunity of reviewing the use of these surfactants of low biodegradability in the light of technical progress;

Whereas technical progress necessitates a rapid adaptation of the technical requirements specified by the Directives on detergents; whereas, to facilitate implementation of the necessary measures designed to achieve this, a procedure should be established providing for close collaboration between the Member States and the Commission by means of a Committee for the adaptation to technical progress of the Directives on the removal of technical barriers to trade in detergents,

HAS ADOPTED THIS DIRECTIVE:

Article 1

This Directive concerns the methods of testing the biodegradability of non-ionic surfactants present in the detergents defined in Article 1 of Directive 73/404/EEC.

Article 2

In accordance with the provisions of Article 4 of Directive 73/404/EEC, the Member States shall prohibit the placing on the market and use on their territory of a detergent if the level of biodegradability of the non-ionic surfactants contained in such detergent is less than 80 % determined in accordance with one of the following methods:

— the OECD method, published in the OECD technical report of 11 June 1976 on the 'Proposed Method for the Determination of the Biodegradability of Surfactants used in Synthetic Detergents',

— 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*, 1977, Part I, page 244, as set out in the Regulation amending that Regulation of 18 June 1980, published in the *Bundesgesetzblatt*, 1980, Part I, page 706,

— the method in use in France, approved by Decree of 28 December 1977 published in the *Journal officiel de la République française* of 18 January 1978, and experimental standard T 73-270 March 1974, published by the Association française de normalisation (AFNOR),

— the method in use in the United Kingdom called the 'Porous Pot Test', as described in Technical Report No 70/1978 of the Water Research Centre.

Article 3

Under the procedure laid down in Article 5 (2) of Directive 73/404/EEC, the laboratory opinion on non-ionic surfactants shall be based on the reference method (confirmatory test procedure) described in the Annex to this Directive.

Article 4

The amendments required for adapting the Annex to technical progress shall be adopted in accordance with the procedure laid down in Article 7b of Directive 73/404/EEC.

Article 5

The following Articles shall be inserted in Directive 73/404/EEC:

'Article 2a

1. Until 31 March 1986:

(a) Member States may exempt the following products from the requirements of paragraph 1 of Article 2: low-foaming alkene oxide additives on such substances as alcohols, alkylphenols, glycols, polyols, fatty acids, amides or amines used in dish-washing products;

(b) the requirements of paragraph 1 of Article 2 shall not apply to alkali-resistant terminally blocked alkyl and alkyl-aryl polyglycol ethers and substances of the type referred to in paragraph (a), used in cleaning agents for the food, beverage and metal-working industries.

2. Paragraph 1 shall apply to the abovementioned non-ionic surfactants which

come on the market after 30 September 1983 only if they have a higher level of biodegradability than existing products for the same application.

3. The use of the non-ionic surfactants benefiting from a temporary derogation mentioned in paragraphs 1 and 2 must not, under normal conditions of use, be harmful to human or animal health.

Article 7a

1. A Committee shall be established for the adaptation to technical progress of Directives for removing technical barriers to trade in the detergents sector, hereinafter called "the Committee", which shall consist of representatives of the Member States under the chairmanship of a representative of the Commission.

2. The Committee shall establish its rules of procedure.

Article 7b

1. Where recourse is had to the procedure defined in this Article, the matter shall be referred to the Committee by its chairman, on his own initiative or at the request of the representative of a Member State.

2. The Commission representative shall submit to the Committee a draft of the measures to be taken. The Committee shall deliver its opinion on the draft within a period which may be fixed by the chairman according to the urgency of the matter. A qualified majority of votes as laid down in Article 148 (2) of the Treaty shall be required before the Committee can deliver its opinion.

The chairman shall not vote.

3. (a) The Commission shall adopt the proposed measures where they are in accordance with the opinion of the Committee.

(b) Where the proposed measures are not in accordance with the opinion of the Committee, or if no opinion is delivered, the Commission shall submit to the Council without delay a proposal on the measures to be adopted. The Council shall decide by a qualified majority.

(c) If the Council has not acted within three months of the date on which the proposal was submitted, the proposed measures shall be adopted by the Commission.

Article 7c

1. In accordance with the procedure laid down in Article 7b,

— the references to test methods in the Directives referred to in Article 4 shall, if necessary, be brought up to date or supplemented by other references to test methods established in other Member States,

— the reference methods (confirmatory test) in the Annexes to the Directives referred to in Article 4 shall be modified in order to adapt them to technical progress.

2. These adaptations should not have the effect of modifying in a negative fashion the biodegradability requirements of surfactants, already laid down in accordance with Article 4.'

Article 6

1. The Member States shall bring into force the provisions necessary to comply with this Directive within a period of 18 months following its notification. They shall forthwith inform the Commission thereof.

2. Member States shall communicate to the Commission the text of the provisions of national law which they adopt in the field covered by this Directive.

Article 7

This Directive is addressed to the Member States.

Done at Brussels, 31 March 1982.

For the Council

The President

P. de KEERSMAEKER

ANNEX

DETERMINATION OF THE BIODEGRADABILITY OF NON-IONIC SURFACTANTS

Reference method (confirmatory test)

CHAPTER 1

1.1. **Definition**

Non-ionic surface active agents in the sense of this Directive are those surface active agents which, after passage through cationic and anionic ion exchangers, are determined as bismuth-active substance (BiAS) according to the analytical procedure described in Chapter 3.

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 storage 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 24 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 should be monitored by means of a flowmeter 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, $CaCl_2 \cdot 2H_2O$,
2 mg magnesium sulphate, $MgSO_4 \cdot 7H_2O$,
28 mg of dipotassium hydrogen phosphate K_2HPO_4
and 10 ± 1 mg BiAS.

The BiAS is extracted from the product to be tested by the method given in Chapter 2. The synthetic sewage is freshly prepared daily.

1.4. **Preparation of samples**

1.4.1. Uncompounded surfactants may be examined in the original state. The BiAS content must be determined in order to prepare the synthetic sewage (1.3).

1.4.2. Formulated products are analyzed for BiAS, MBAS and soap content. They must be subjected to an alcoholic extraction and to a separation of the BiAS (see Chapter 2). The BiAS content of the extract must be known in order to prepare the synthetic sewage.

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 seration 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 which inhibit the activated sludge or contain BiAS 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 settlebility 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 24 hours, following which a sample is taken after thorough mixing. Vessel F must then be carefully cleaned.

1.6. **Checking measuring equipment**

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

The BiAS content (in mg/l) of the effluent collected over 24 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 BiAS.

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 BiAS 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 292 and 297 K (19-24 °C).

1.7. **Calculation of biodegradability**

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

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

The degradability of the BiAS should be calculated as the arithmetic mean of the figures obtained over the 21 days which follow the running-in 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 14 results collected over the 21 days which follow the running-in period should be used in calculating the average.