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COUNCIL DIRECTIVE

of 22 November 1973

on the approximation of the laws of the Member States relating to methods of testing the biodegradability of anionic surfactants

(73/405/EEC)

(OJ L 347, 17.12.1973, p. 53)

Amended by:

	Official Journal		
	No	page	date
►**M1** Council Directive of 31 March 1982 (82/243/EEC)	L 109	18	22.4.1982

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COUNCIL DIRECTIVE

of 22 November 1973

on the approximation of the laws of the Member States relating to methods of testing the biodegradability of anionic surfactants

(73/405/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 Council Directive of 22 November 1973 ([1]) on the approximation of the laws of the Member States relating to detergents, and in particular Article 4 thereof;

Having regard to the proposal of the Commission;

Having regard to the Opinion of the European Parliament ([2]);

Having regard to the Opinion of the Economic and Social Committee ([3]);

Whereas, to enable Member States to determine the level of biodegradability of the anionic surfactant, it is advisable to employ methods of testing already in use for this purpose in certain Member States; whereas, however, biodegradability must be tested by a common method in the event of a dispute;

Whereas as concerns the approximation of the laws of the Member States relating to detergents, suitable tolerances for measuring biodegradability should be determined as provided for likewise in Article 4 of Council Directive of 22 November 1973, in order to take account of the unreliability of testing methods which could lead to rejection decisions having important economic consequences and whereas a rejection decision must only be taken therefore if an analysis shows a level of biodegradability less than 80%,

HAS ADOPTED THIS DIRECTIVE:

Article 1

This Directive concerns the methods of testing the biodegradability of anionic surfactants ►**M1** present in detergents such as those referred to in Article 1 of Directive 73/404/EEC ◄.

▼M1

Article 2

Pursuant to Article 4 of Directive 73/404/EEC relating to detergents, Member States shall prohibit the placing on the market and use on their territory of a detergent if the biodegradability of the anionic surfactants contained therein 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,

([1]) See page 51 of this Official Journal (SIC! OJ No L 347, 17. 12. 1973, p. 51.)
([2]) OJ No C 10, 5. 2. 1972, p. 29.
([3]) OJ No C 89, 23. 8. 1972, p. 13.

▼**M1**

— 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, pages 514 and 515, and experimental standard T 73-260 of June 1981 published by the 'Association française de normalisation' (AFNOR),

— 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.

Article 3

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

Article 3a

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.

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Article 4

1. Member States shall put into force the legal, statutory and administrative measures necessary to comply with this Directive within eighteen months of its notification and shall forthwith inform the Commission thereof.

2. Member States shall ensure that the Commission be informed of the text of the main provisions of national law they adopt in the field covered by this Directive.

Article 5

This Directive is addressed to the Member States.

▼M1

ANNEX

DETERMINATION OF THE BIODEGRADABILITY OF ANIONIC SURFACTANTS

Reference method (confirmatory test)

CHAPTER 1

1.1. **Definition**

Anionic surface active agents in the sense of this Directive are those surface active agents which, after passage through cationic and anionic ion exchangers, are separated by fractional elution and determined as methylene blue active substance (MBAS) 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 ($CaCl_2.2H_2O$),

2 mg magnesium sulphate ($MgSO_4.7H_2O$),

28 mg of dipotassium hydrogen phosphate (K_2HPO_4)

and 20 ± 2 mg MBAS

The MBAS 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 MBAS content must be determined in order to prepare the synthetic sewage (1.3).

1.4.2. Formulated products are analyzed for MBAS and soap content. They must be subjected to an alcoholic extraction and separation of MBAS (see Chapter 2). The MBAS 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 vessel D should be so fixed that the volume contained in 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 aerator G, air lift E and dosing device B in operation. The synthetic sewage must pass through aeration vessel C at the rate of one litre per hour; this gives a mean retention time of three hours.

▼M1

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 MBAS must not be used. 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 aeration vessel C, in the base of 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 sludge fails to settle, its density may be increased by the addition of 2 ml portions of a 5% solution of ferric chloride, repeated as necessary.

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

The MBAS 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 MBAS.

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 MBAS biodegradation is obtained, i.e. at the end of the running-in period shown in Figure 3.

The content of dry matter of the suspended solids in the activated sludge in the aeration tank 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 test is performed at room temperature; this should be steady and kept between 292 and 297 K (19-24 ºC).

1.7. **Calculation of biodegradability**

The percentage degradation of MBAS must be calculated every day on the basis of the MBAS 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 MBAS 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.

CHAPTER 2

PRELIMINARY TREATMENT OF PRODUCTS TO BE TESTED

2.1. **Preliminary notes**

2.1.1. *Treatment of samples*

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

Products	Treatment
Anionic surfactants	None