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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 $\text{CO}(\text{NH}_2)_2$,
 - 7 mg sodium chloride NaCl ,
 - 4 mg calcium chloride, $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$,
 - 2 mg magnesium sulphate, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$,
 - 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 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 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 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 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.

CHAPTER 2

PRELIMINARY TREATMENT OF PRODUCTS TO BE TESTED

2.1. Preliminary notes

2.1.1. Treatment of samples

The treatment of non-ionic surface active agents and formulated detergents prior to the determination of 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.

2.1.2. Ion-exchange procedure

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

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

2.1.3. Analytical control

After homogenizing, 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 to prepare 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.

2.2. Principle

From an 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 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/macroporous anion exchange combination heated to 323 K (50 °C). This high 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.

2.3. Chemicals and equipment

2.3.1. Deionized water

2.3.2. Ethanol, 95 % (v/v) C₂H₅OH

(permissible denaturant: methyl-ethyl ketone or methanol)

- 2.3.3. Isopropanol/water mixture (50/50 v/v):
50 parts by volume isopropanol ($\text{CH}_3\text{CHOH} \cdot \text{CH}_3$) and
50 parts by volume water (2.3.1)
- 2.3.4. 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 (2.3.1)
- 2.3.5. Cation exchanger (KAT), strongly acidic, resistant to alcohol (50-100 mesh)
- 2.3.6. Anion exchanger (AAT), macro-porous, Merck Lewatit MP 7080 (70-150 mesh) or
equivalent
- 2.3.7. Hydrochloric acid, 10 % HCl w/w
- 2.3.8. 2 000 ml round-bottomed flask with ground glass stopper and reflux condenser
- 2.3.9. 90 mm diameter suction filter (heatable) for filter papers
- 2.3.10. 2 000 ml filter flask
- 2.3.11. Exchange columns with heating jacket and tap: inner tube 60 mm in diameter and 450 mm
in height (Figure 4)
- 2.3.12. Water-bath
- 2.3.13. Vacuum drying oven
- 2.3.14. Thermostat
- 2.3.15. Rotary evaporator

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

2.4.1. Preparation of extract

The quantity of surface active agents 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.

2.4.2. Isolation of alcohol-soluble constituents

Add 250 g of the synthetic detergent to be analyzed 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 323 K (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 analyzed, 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 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.

2.4.3. *Preparation of ion-exchange columns*

Cation-exchange column

Place 600 ml cation-exchange resin (2.3.5) in a 3 000 ml beaker and cover by adding 2 000 ml hydrochloric acid (2.3.7). Allow to stand for at least two hours, with occasional stirring. Decant the acid and transfer the resin into the column (2.3.11) by means of deionized water. The column should contain a glass wool plug. Wash the column with deionized 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 (2.3.3) 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 (2.3.6) in a beaker and cover by adding 2 000 ml deionized water. Allow the resin to swell for at least two hours. Transfer the resin into the column by means of deionized water. The column should contain a glass wool plug.

Wash the column with 0.3 M ammonium bicarbonate solution (2.3.4) until free of chloride. This requires about 5 000 ml solution. Wash again with 2 000 ml deionized water. Displace the water with 2 000 ml isopropanol/water mixture (2.3.3) at a rate of 10-30 ml/min. The exchange column is now in the OH form and ready for operation.

2.4.4. *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 323 K (50 °C) using thermostatic control. Heat 5 000 ml of the solution obtained in item 2.4.2 to 333 K (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 (2.3.3).

To obtain the non-ionic surface active agents, collect the filtrate and filter washings and evaporate to dryness, preferably by means of a rotary evaporator. The residue contains the BiAS. Add deionized water until a defined volume is obtained and determine the BiAS content as in item 3.3 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 278 K (5 °C).

2.4.5. *Regeneration of ion-exchange resins*

The cation exchanger is rejected after use.

The anion-exchange resin is regenerated by passing about 5 000-6 000 ml of ammonium bicarbonate solution (2.3.4) down the column at a flow rate of approximately 10 ml/min until the eluate is free from anionic surfactants (methylene blue test). Then pass 2 000 ml isopropanol/water mixture (2.3.3) down the anion exchanger to wash. The anion exchanger is again ready for operation.

CHAPTER 3

DETERMINATION OF NON-IONIC SURFACE ACTIVE AGENT 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 phaser separation and evaporation of the solvent, the non-ionic surfactant is precipitated in aqueous solution with modified Dragendorff reagent ($\text{KBiJ}_4 + \text{BaCl}_2 + \text{glacial acetic acid}$).

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 deionized 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 AR ($\text{BiONO}_3 \cdot \text{H}_2\text{O}$) 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 ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) 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 ($d = 0.910 \text{ g/ml}$) 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 ($d = 0.910 \text{ g/ml}$) 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 ($\text{C}_5\text{H}_8\text{NNaS}_2 \cdot 2\text{H}_2\text{O}$) 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 standardization of 3.2.11).

Stock solution

Mix 1.249 g copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) 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 G 4.
- 3.2.18. Circular glass-fibre filter papers, 27 mm diameter with fibre diameter 0.5-1.5 μm .
- 3.2.19. Two filter flasks with adaptors 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 inclusion of a rotameter 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 minimize 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 on 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 diluted 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 10 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. *Solution of the precipitate*

Dissolve the precipitate in the filter crucible by the addition of hot ammonium tartrate solution (about 80 °C, 353 K) (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, adaptor 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 diluted ammonia solution (3.2.9) until the colour turns violet (the solution is 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 titrations 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 10 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:

$$(b-c) \cdot f \cdot 0.054 = \text{mg non-ionic surfactant as NP 10}$$

where: b = volume of 'carbate solution' used by the sample (ml),

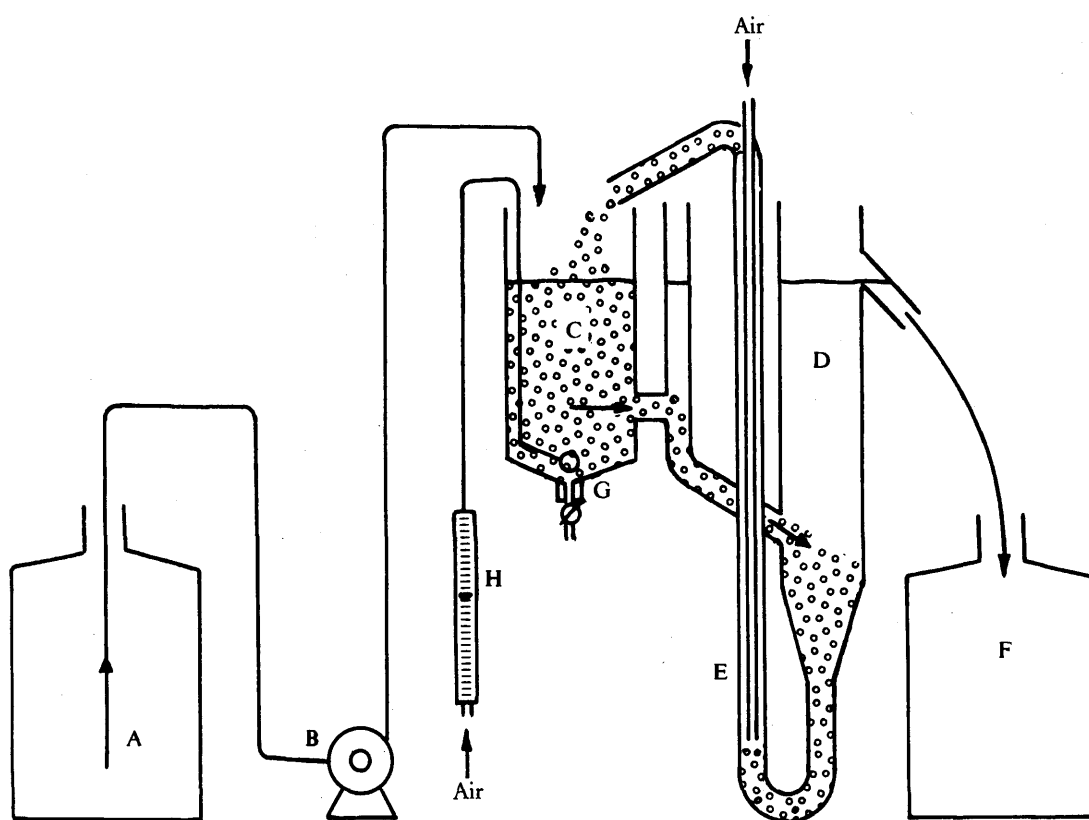
c = volume of 'carbate solution' used by the blank (ml),

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.

Figure 1



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

Figure 2

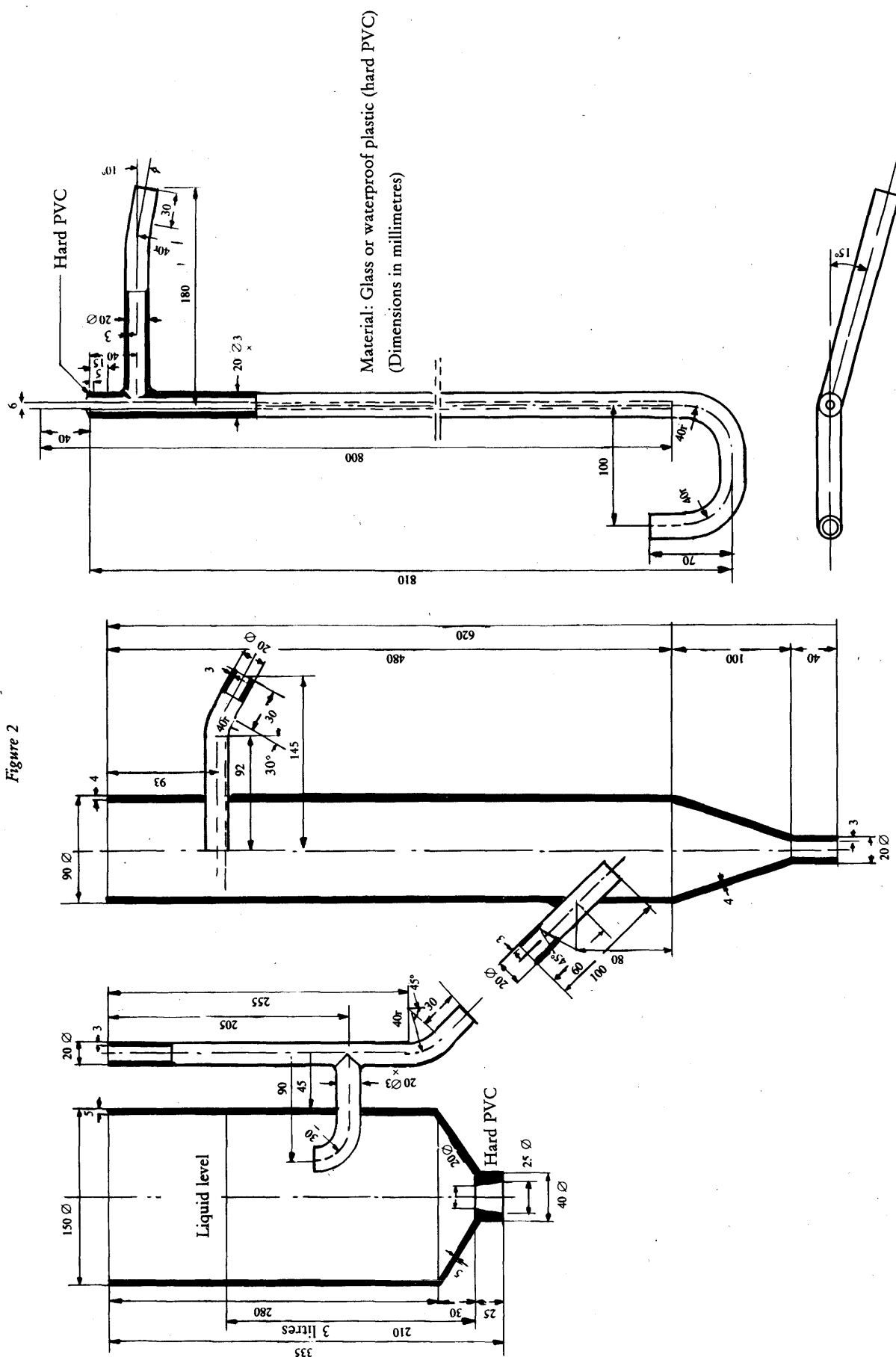


Figure 3

Calculation of biodegradability — Confirmatory test

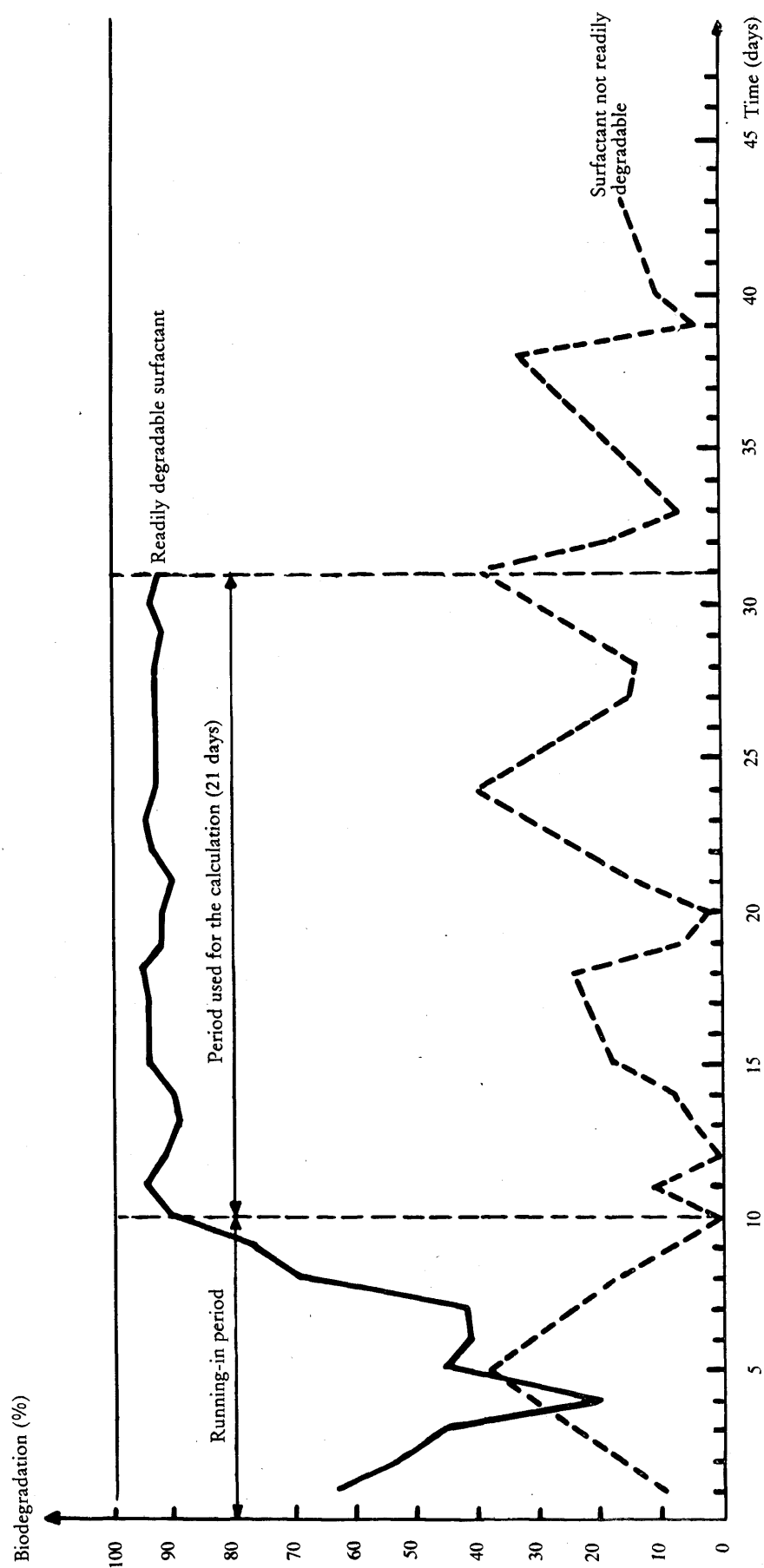


Figure 4

Heated exchange column

(Dimensions in millimetres)

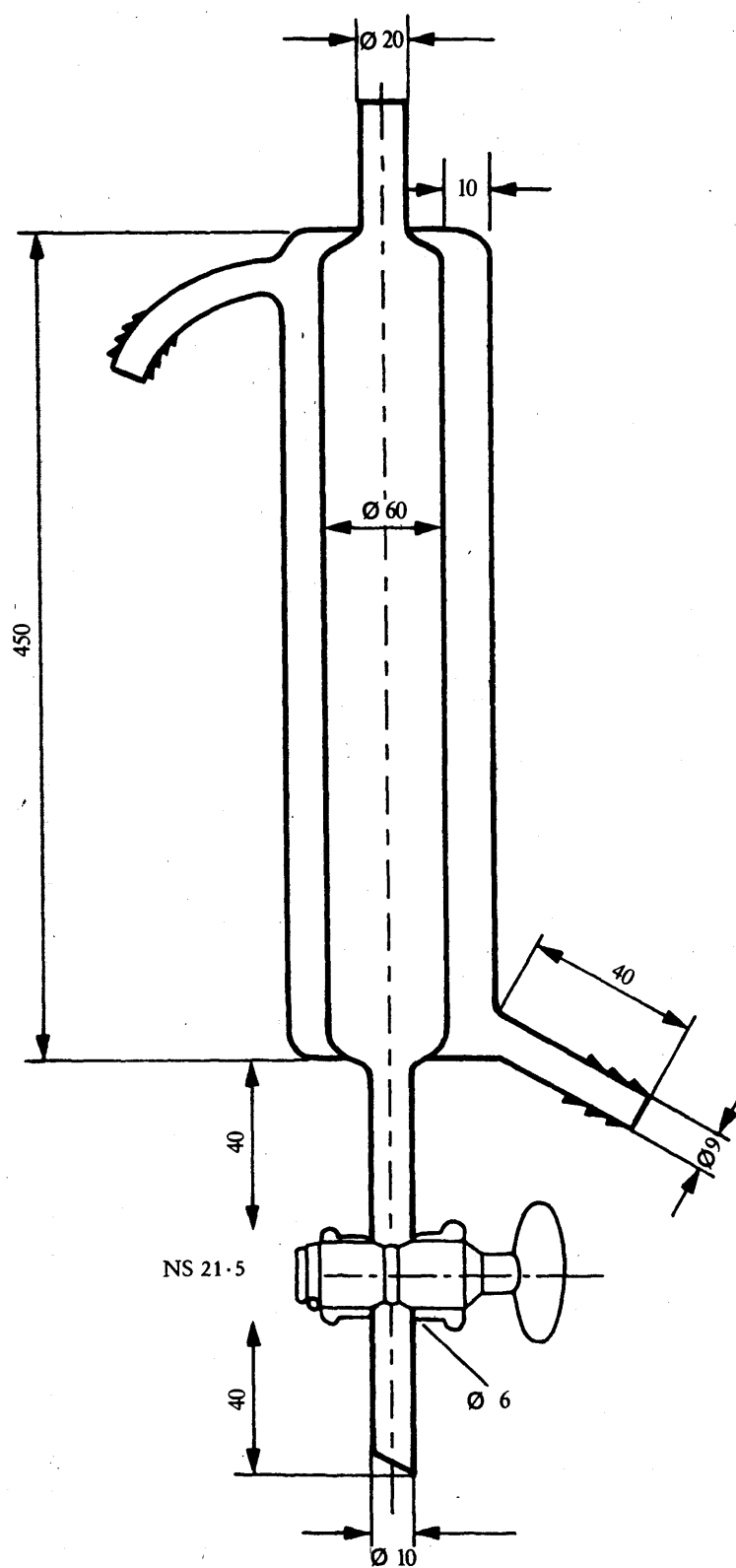
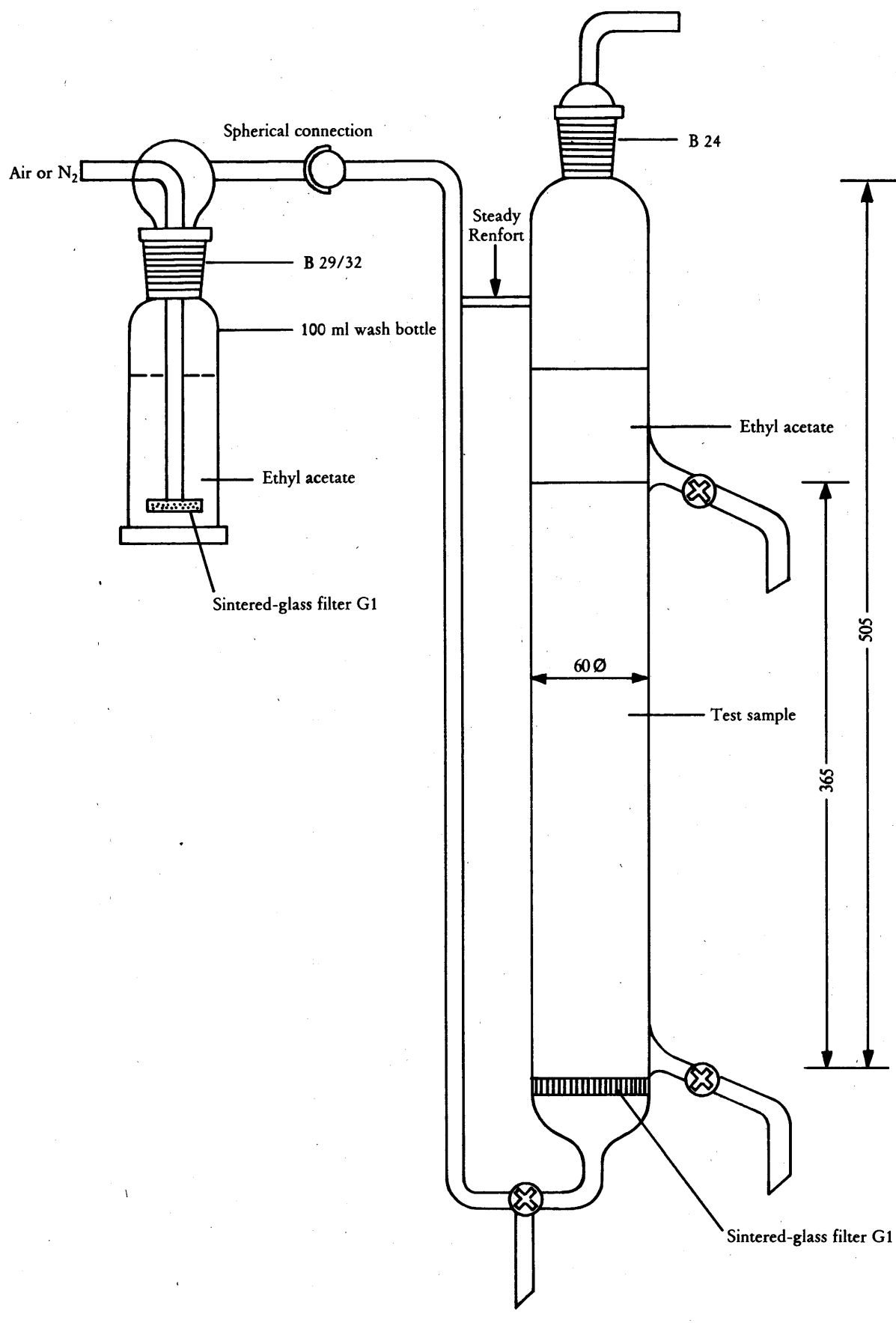


Figure 5

Gas-stripping apparatus
(Dimensions in millimetres)



COUNCIL DIRECTIVE

of 31 March 1982

amending Directive 73/405/EEC on the approximation of the laws of the Member States relating to methods of testing the biodegradability of anionic surfactants

(82/243/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 Council Directive 73/405/EEC ⁽⁴⁾ must be adapted to take account of the latest developments in science and technology, thus necessitating:

- the updating of the references relating to the methods listed in Article 2,
- the addition to Article 2 of a further method of measurement which is in use in the United Kingdom,
- the improvement of the 'confirmatory test procedure' provided for in the event of a dispute;

Whereas, pursuant to Article 4 of Council Directive 73/404/EEC of 22 November 1973 on the approximation of the laws of the Member States relating to detergents ⁽⁵⁾, suitable tolerances for the measurement of biodegradability should be determined in order to take due account of the unreliability of test methods which could result in rejection decisions with important economic consequences; whereas a rejection decision may therefore only be taken where the results obtained with a test method specified in Article 2 of Directive 73/405/EEC reveal a biodegradability of less than 80 %;

Whereas some confusion has arisen as to the scope of Directive 73/405/EEC and it is necessary to make quite clear that the Directive applies only to surfactants used in detergents; whereas it is also necessary to make quite clear that it is the level of biodegradability of the anionic surfactants contained in a detergent, and not the level of biodegradability of the detergent itself, which is dealt with in Article 2;

Whereas the necessary amendment and amplification of the Annex to Directive 73/405/EEC will be carried out under the procedure provided for in Article 3a thereof,

HAS ADOPTED THIS DIRECTIVE:

Article 1

Directive 73/405/EEC is hereby amended as follows:

1. The following is added to Article 1: 'present in detergents such as those referred to in Article 1 of Directive 73/404/EEC'.
2. Articles 2 and 3 are replaced by the following:

'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

⁽¹⁾ OJ No C 112, 14. 5. 1981, p. 4.

⁽²⁾ OJ No C 172, 13. 7. 1981, p. 111.

⁽³⁾ OJ No C 310, 30. 11. 1981, p. 7.

⁽⁴⁾ OJ No L 347, 17. 12. 1973, p. 53.

⁽⁵⁾ OJ No L 347, 17. 12. 1973, p. 51.

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, 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.

3. The following Article shall be inserted:

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.

4. The Annex is replaced by the Annex to this Directive.

Article 2

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

Article 3

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 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 $\text{CO}(\text{NH}_2)_2$,
7 mg sodium chloride (NaCl),
4 mg calcium chloride ($\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$),
2 mg magnesium sulphate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$),
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.

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 °C).

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
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.

2.1.2. Ion-exchange procedure

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

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

2.1.3. Analytical control

After homogenizing, 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 to prepare 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.

2.2. Principle

From an 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 323 K (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 elution with ethanol containing CO₂. 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 which might upset the biodegradability test and the analytical procedure, are eliminated by the cation exchanger placed above the anion exchanger.

2.3. Chemicals and equipment

- 2.3.1. Deionized water
- 2.3.2. Ethanol, 95 % (v/v) C_2H_5OH
(permissible denaturant: methyl ethyl ketone or methanol)
- 2.3.3. Isopropanol/water mixture (50/50 v/v):
50 parts by volume isopropanol ($CH_3CHOH \cdot CH_3$) and
50 parts by volume water (2.3.1)
- 2.3.4. 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 (2.3.2) for 10 minutes. Use fresh solutions only
- 2.3.5. 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 (2.3.1)
- 2.3.6. Cation exchanger (KAT), strongly acidic, resistant to alcohol (50-100 mesh)
- 2.3.7. Anion exchanger (AAT), macro-porous, Merck Lewatit MP 7080 (70-150 mesh) or equivalent
- 2.3.8. Hydrochloric acid, 10 % HCl (w/w)
- 2.3.9. 2 000 ml round-bottomed flask with ground glass stopper and reflux condenser
- 2.3.10. 90 mm diameter suction filter (heatable) for filter papers
- 2.3.11. 2 000 ml filter flask
- 2.3.12. Exchange columns with heating jacket and tap: inner tube 60 mm in diameter and 450 mm in height (Figure 4)
- 2.3.13. Water-bath
- 2.3.14. Vacuum drying oven
- 2.3.15. Thermostat
- 2.3.16. Rotary evaporator

2.4. Preparation of extract and separation and anionic active agents**2.4.1. 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.

2.4.2. Isolation of alcohol-soluble constituents

Add 250 g of the synthetic detergent to be analyzed 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 323 K (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 analyzed, make sure that not more than 55 g anionic surfactant 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 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.

2.4.3. *Preparation of ion-exchange columns*

Cation-exchange column

Place 600 ml cation-exchange resin (2.3.6) in a 3 000 ml beaker and cover by adding 2 000 ml hydrochloric acid (2.3.8). Allow to stand for at least two hours, with occasional stirring. Decant the acid and transfer the resin into the column (2.3.12) by means of deionized water. The column should contain a glass wool plug. Wash the column with deionized 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 (2.3.3) 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 (2.3.7) in a 3 000 ml beaker and cover by adding 2 000 ml deionized water. Allow the resin to swell for at least two hours. Transfer the resin into the column by means of deionized water. The column should contain a glass wool plug.

Wash the column with 0.3 M ammonium bicarbonate solution (2.3.5) until free of chloride. This requires about 5 000 ml solution. Wash again with 2 000 ml deionized water. Displace the water with 2 000 ml isopropanol/water mixture (2.3.3) at a rate of 10-30 ml/min. The exchange column is now in the OH-form and ready for operation.

2.4.4. *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 323 K (50 °C) using thermostatic control. Heat 5 000 ml of the solution obtained in item 2.4.2 to 333 K (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 (2.3.3).

To obtain the anionic surface active agents (MBAS), disconnect the KAT column. Using 5 000 ml ethanol/CO₂ solution (323 K; 50 °C) (2.3.4), 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 (2.3.5). Evaporate the eluate to dryness on a steam bath or in a rotary evaporator. The residue contains the MBAS (as ammonium salt) and possible non-surfactant anionics which have no detrimental effect on the biodegradation test. Add deionized water to the residue until a definite volume is obtained and determine the MBAS content in an aliquot as in Chapter 3. 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 278 K (5 °C).

2.4.5. *Regeneration of ion-exchange resins*

The cation exchanger is rejected after use.

The anion-exchange resin is regenerated by passing an additional quantity of ammonium bicarbonate solution (2.3.5) down the column at a flow rate of approximately 10 ml/min until the eluate is free from anionic surfactants (methylene blue test). The pass 2 000 ml isopropanol/water mixture (2.3.3) down the anion exchanger to wash. The anion exchanger is again ready for operation.

CHAPTER 3

DETERMINATION OF ANIONIC SURFACE ACTIVE AGENTS IN BIODEGRADABILITY TEST

3.1. Principle

The method is based on the fact that the cationic dye methylene blue forms blue salts with anionic surfactants which can be extracted with chloroform. To eliminate interferences, the extraction is first effected from alkaline solution and the extract is then shaken with acidic methylene blue solution. The absorbance of the separated organic phase is measured photometrically at the wavelength of maximum absorption of 650 nm.

3.2. Reagents and equipment

3.2.1. Buffer solution pH 10:

dissolve 24 g sodium bicarbonate (NaHCO_3) AR and 27 g anhydrous sodium carbonate (Na_2CO_3) AR in deionized water and dilute to 1 000 ml.

3.2.2. Neutral methylene blue solution:

dissolve 0.35 g methylene blue (AR) in deionized water and dilute to 1 000 ml. Prepare the solution at least 24 hours before use. The absorbance of the blank chloroform phase, measured against chloroform, must not exceed 0.015 per 1 cm of layer thickness at 650 nm.

3.2.3. Acidic methylene blue solution:

dissolve 0.35 g methylene blue (AR) in 500 ml deionized water and mix with 6.5 ml H_2SO_4 ($d = 1.84 \text{ g/ml}$). Dilute to 1 000 ml with deionized water. Prepare the solution at least 24 hours before use. The absorbance of the blank chloroform phase, measured against chloroform, must not exceed 0.015 per 1 cm of layer thickness at 650 nm.

3.2.4. Chloroform (Trichloromethane) (AR) freshly distilled

3.2.5. Dodecyl benzene sulphonic acid methyl ester

3.2.6. Ethanolic potassium hydroxide solution, KOH 0.1 M

3.2.7. Ethanol pure, $\text{C}_2\text{H}_5\text{OH}$ 3.2.8. Sulphuric acid, H_2SO_4 0.5 M

3.2.9. Phenolphthalein solution:

dissolve 1 g phenolphthalein in 50 ml ethanol and add 50 ml deionized water while stirring continuously. Filter off any precipitate obtained.

3.2.10. Methanolic hydrochloric acid: 250 ml hydrochloric acid AR and 750 ml methanol

3.2.11. Separating funnel, 250 ml

3.2.12. Graduated flask, 50 ml

3.2.13. Graduated flask, 500 ml

3.2.14. Graduated flask, 1 000 ml

3.2.15. Round-bottomed flask with ground glass stopper and reflux condenser, 250 ml; boiling granules

3.2.16. pH meter

3.2.17. Photometer for measurements at 650 nm, with 1 to 5 cm cells

3.2.18. Qualitative grade filter paper

3.3. 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 (3.2.10) and then with deionized 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, neutralized if necessary, into a 250 ml separating funnel (3.2.11). 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 deionized water. Add to the sample 10 ml of buffer solution (3.2.1), 5 ml of neutral methylene blue solution (3.2.2) and 15 ml of chloroform (3.2.4). 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 deionized water and 5 ml of acidic methylene blue solution (3.2.3). 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 (3.2.12).

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 (3.2.12) with chloroform used for rewashing the cotton wool. Measure the absorbance 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.

3.4. Calibration curve

Prepare a calibration solution from the standard substance dodecyl benzene 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 (3.2.5) to the nearest 0.1 mg in a round-bottomed flask and add 50 ml of ethanolic potassium hydroxide solution (3.2.6) 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 (3.2.14), dilute to the mark with deionized water and mix.

Part of this surfactant stock solution is then further diluted. Withdraw 25 ml, transfer to a 500 ml graduated flask (3.2.13), dilute to the mark with deionized water and mix.

This standard solution contains $\frac{E \times 1.023}{20\,000}$ mg MBAS per ml, where E is the sample weight in mg.

To establish the calibration curve, withdraw 1, 2, 4, 6, 8 ml each of the standard solution and dilute each to 100 ml with deionized water. Then proceed as stated under item 3.3 including a blank determination.

3.5. Calculation of results

The amount of anionic surfactant (MBAS) in the sample is read from the calibration curve (3.4). The MBAS content of the sample is given by:

$$\frac{\text{mg MBAS} \times 1\,000}{V} = \text{MBAS mg/l}$$

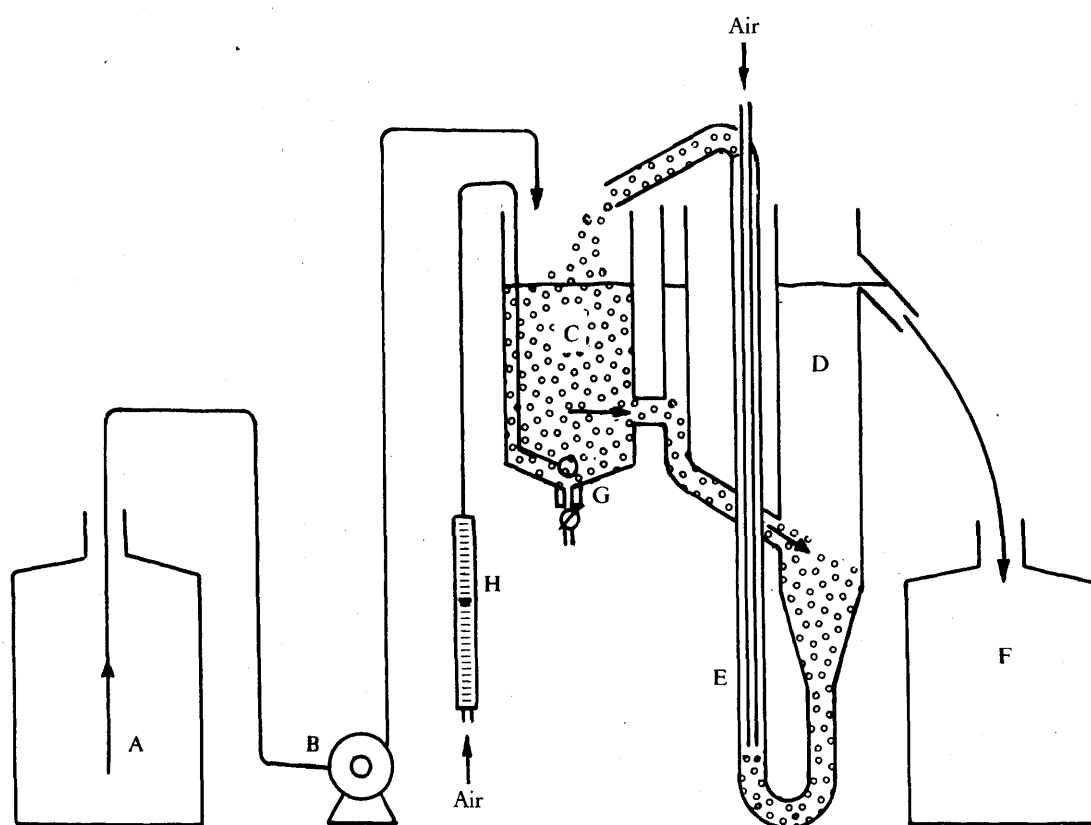
where V = ml volume of the sample used.

Express the results as sodium dodecyl benzene sulphonate (MW 348).

3.6. Expression of results

Express the results as MBAS mg/l to the nearest 0.1.

Figure 1



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

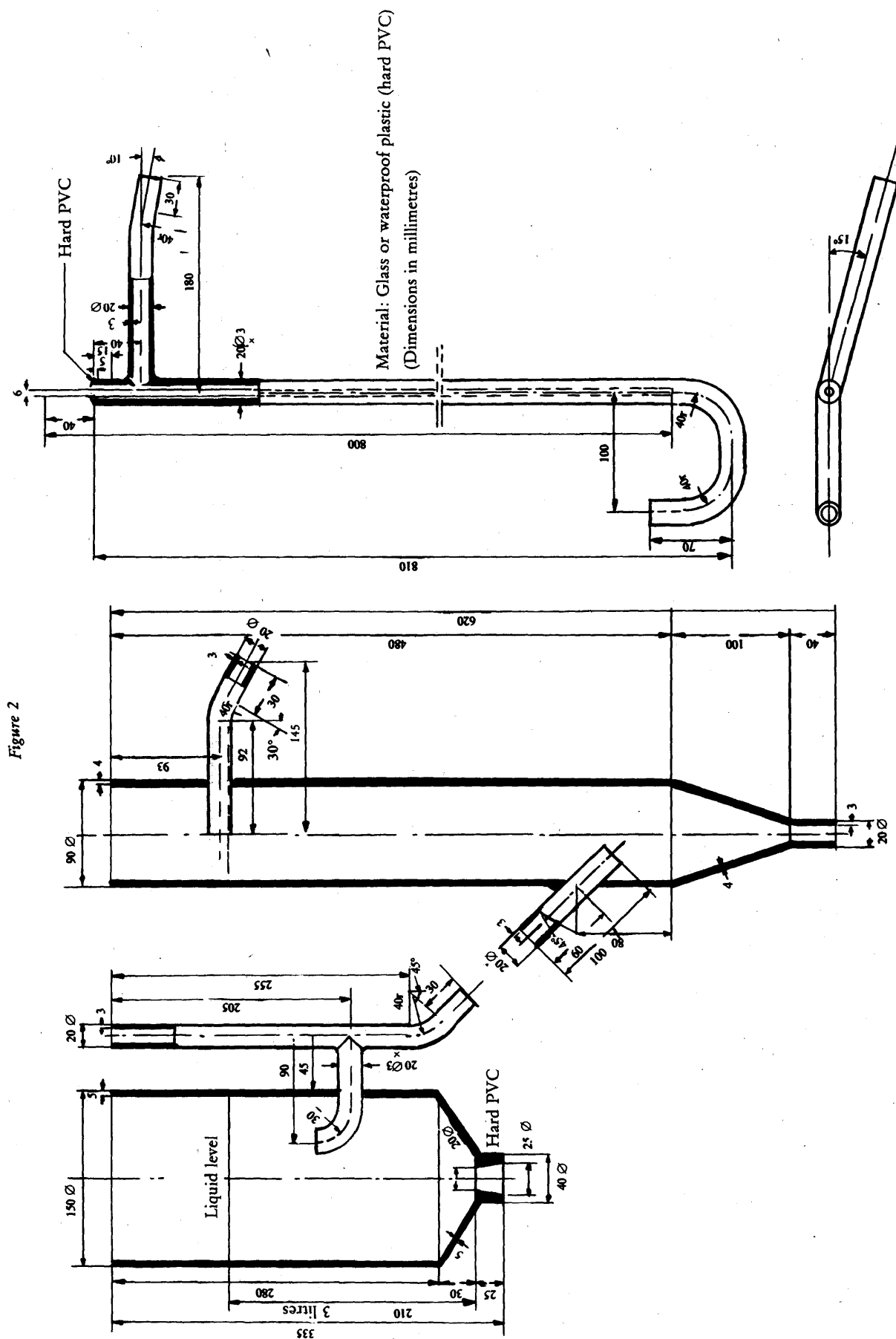


Figure 3
Calculation of biodegradability — Confirmatory test

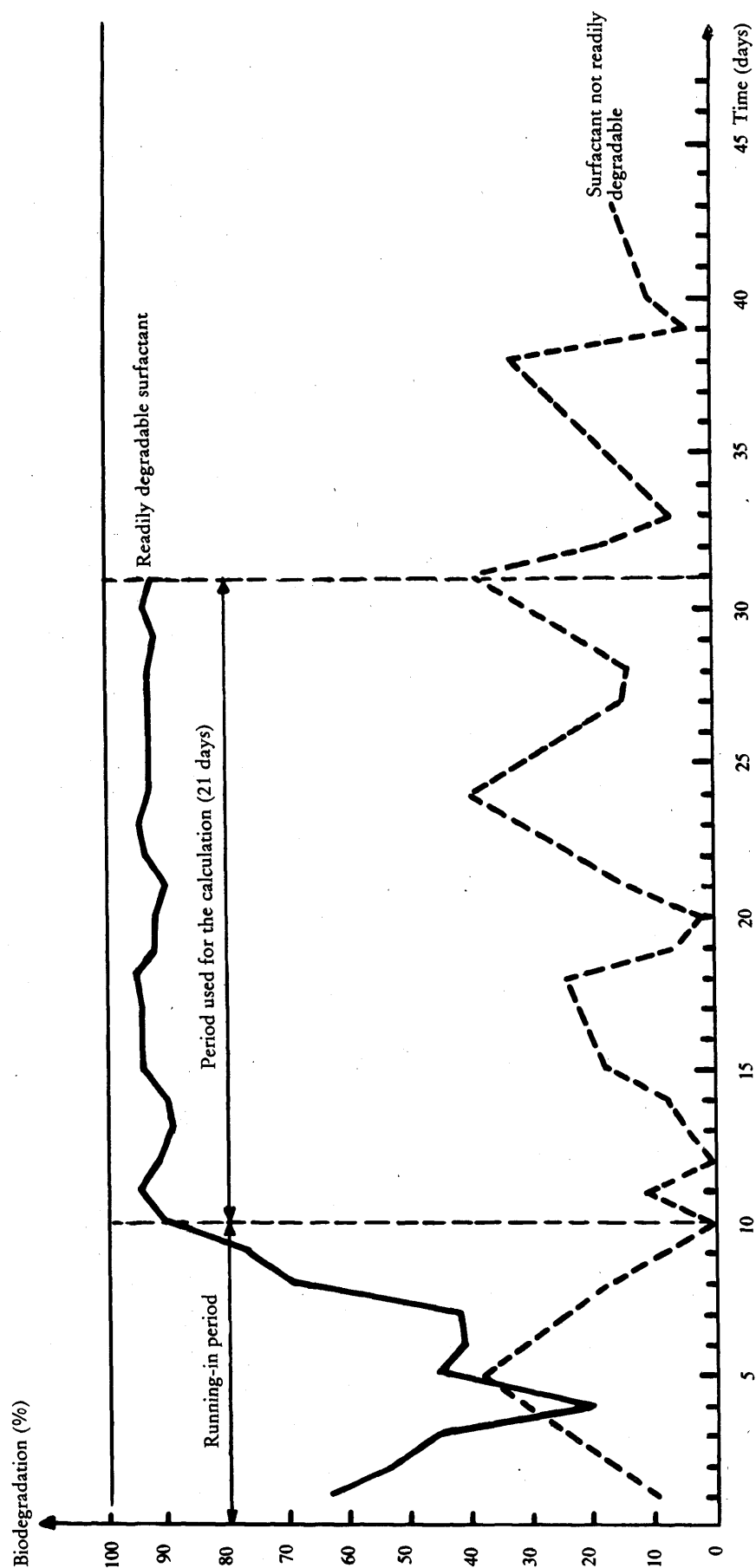
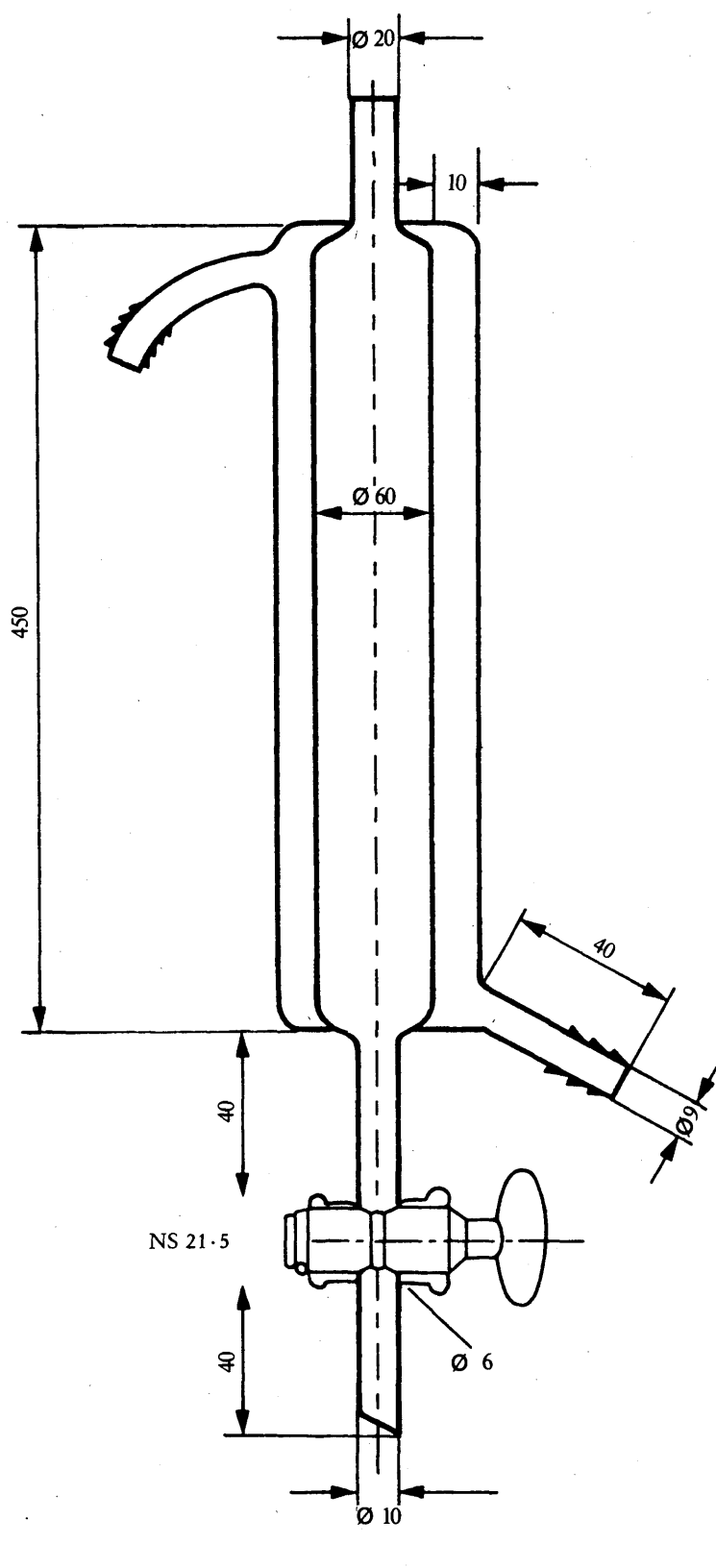


Figure 4

Heated exchange column
(Dimensions in millimetres)



COMMISSION

COMMISSION DIRECTIVE

of 17 March 1982

adapting to technical progress Council Directive 76/756/EEC on the approximation of the laws of the Member States relating to the installation of lighting and light-signalling devices on motor vehicles and their trailers

(82/244/EEC)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Directive 70/156/EEC of 6 February 1970 on the approximation of the laws of the Member States relating to the type-approval of motor vehicles and their trailers ⁽¹⁾, as last amended by Directive 80/1267/EEC ⁽²⁾ and by the Act of Accession of Greece, and in particular Article 11 thereof,

Having regard to Council Directive 76/756/EEC of 27 July 1976 on the approximation of the laws of the Member States relating to the installation of lighting and light-signalling devices on motor vehicles and their trailers ⁽³⁾, as amended by Commission Directive 80/233/EEC ⁽⁴⁾, and in particular Article 4 thereof,

Whereas, in the light of experience gained and in view of technical progress, it is now possible not only to supplement certain requirements and bring them more into line with actual test conditions, but also to render them more stringent with a view to increasing the safety of both the occupants of the vehicles and other road users;

Whereas the measures provided for in this Directive are in accordance with the opinion of the Committee for the Adaptation to Technical Progress of Directives on the Removal of Technical Barriers to Trade in the Motor Vehicles Sector,

HAS ADOPTED THIS DIRECTIVE:

Article 1

Annexes I and II to Directive 76/756/EEC are hereby amended as set out in the Annex to this Directive.

Article 2

1. With effect from 1 October 1982, no Member State may, on grounds relating to the installation of the lighting and light-signalling devices, whether mandatory or optional, listed in Items 1.5.7 to 1.5.20 of Annex I to Directive 76/756/EEC:

— refuse, in respect of a type of vehicle, to grant EEC type-approval, to issue the document referred to in the last indent of Article 10 (1) of Directive 70/156/EEC, or to grant national type-approval, or

— prohibit the entry into service of vehicles,

if the installation of the said lighting and light-signalling devices on this type of vehicle or on these vehicles complies with the provisions of this Directive.

2. With effect from 1 January 1983, Member States:

— shall no longer issue the document referred to in the last indent of Article 10 (1) of Directive 70/156/EEC in respect of a type of vehicle in which the installation of the said lighting and light-signalling devices does not comply with the provisions of this Directive,

— may refuse to grant national type-approval in respect of a type of vehicle in which the installation of the said lighting and light-signalling devices does not comply with the provisions of this Directive.

⁽¹⁾ OJ No L 42, 23. 2. 1970, p. 1.

⁽²⁾ OJ No L 375, 31. 12. 1980, p. 34.

⁽³⁾ OJ No L 262, 27. 9. 1976, p. 1.

⁽⁴⁾ OJ No L 51, 25. 2. 1980, p. 8.

3. With effect from 1 October 1984, Member States may prohibit the entry into service of vehicles in respect of which a certificate pursuant to Article 10 of Directive 70/156/EEC concerning the installation of the said lighting and light-signalling devices was issued after 1 October 1979, where the installation of these devices does not comply with the provisions of this Directive.

Article 3

Member States shall bring into force the provisions necessary in order to comply with this Directive not

later than 1 October 1982 and shall forthwith inform the Commission thereof.

Article 4

This Directive is addressed to the Member States.

Done at Brussels, 17 March 1982.

For the Commission

Karl-Heinz NARJES

Member of the Commission

ANNEX

Amendments to the Annex to Directive 76/756/EEC

ANNEX I: INSTALLATION OF LIGHTING AND LIGHT-SIGNALLING DEVICES

Item 1.1 is hereby amended as follows:

'1.1. Vehicle type with regard to the installation of lighting and light-signalling devices

Vehicle type with regard to the installation of lighting and light-signalling devices means vehicles which do not differ in the essential respects mentioned in Items 1.1.1-1.1.4.

The following are likewise considered not to be "vehicles of a different type": vehicles which differ within the meaning of Items 1.1.1-1.1.4, but not in such a way as to entail a change in the type, number, positioning and geometric visibility of the lamps and the inclination of the dipped beam prescribed for the vehicle type in question, and vehicles on which optional lamps are fitted or are absent.'

After Item 1.1.2, the following new Items 1.1.3 and 1.1.4 shall be added:

'1.1.3. the headlamp levelling system.

1.1.4. the suspension system'

Item 1.10.4 is hereby amended as follows:

'1.10.4. of side direction indicator lamps, end-outline marker lamps, front and rear position (side) lamps, parking lamps and reflex reflectors'

Item 2.2.4 is hereby amended as follows:

'2.2.4. Lay out drawing(s) for each individual lamp showing the illuminating surfaces as defined in Item 1.6, the axis of reference as defined in Item 1.7 and the centre of reference as defined in Item 1.8.

This information is not necessary in the case of the rear registration plate lamp (Item 1.5.14).'

Item 4.2.6.1 is hereby amended as follows:

'4.2.6.1. After adjustment of the initial inclination, the vertical inclination of the dipped beam shall be measured in static conditions under all the loading conditions defined in Appendix 1. It shall remain between -0.5 and -2.5 % without manual adjustment. Initial adjustment shall be between -1 and -1.5 % in the "unladen vehicle" state with one person in the driving seat. The initial adjustments for each type of vehicle must be specified by the manufacturer and must be indicated in a clearly legible and indelible manner on each vehicle close to either the headlamp or the manufacturer's plate by the symbol shown in Appendix 6.'

Item 4.2.6.2.2 is hereby amended as follows:

'4.2.6.2.2. Devices which are adjusted manually, either continuously or non-continuously, shall nevertheless be permitted, provided they have a stop position at which the lamps can be returned to the initial inclination defined in Item 4.2.6.1 by means of the usual adjusting screws. These manually adjustable devices must be operable from the driving seat. Continually adjustable devices must have reference marks indicating the loading conditions that require adjustment of the dipped beam.

The number of positions on devices which are not continuously adjustable must be such as to ensure compliance, starting from an initial inclination of between -1 and -1.5 %; with the range of values between -0.5 and -2.5 % for the loading conditions defined in Appendix 1. For these devices also, the loading conditions that require adjustment of the dipped beam shall be clearly marked near the control of the device (see Appendix 7).'

After Item 4.2.6.2.2, add:

- '4.2.6.2.3. The measurement of the variation of dipped beam inclination as a function of load must be carried out in accordance with the test procedure set out in Appendix 5.'

Item 4.4.1. is hereby amended as follows:

- '4.4.1. *Presence*
Mandatory on motor vehicles
Optional on trailers.'

Item 4.5.4.1 is hereby amended as follows:

- '4.5.4.1. *Width*

The edge of the illuminating surface furthest from the median longitudinal plane of the vehicle must not be more than 400 mm from the extreme outer edge of the vehicle.

The distance between the inner edges of the two illuminating surfaces shall not be less than 600 mm. Where the vertical distance between the rear direction indicator lamp and the corresponding rear position (side) lamp is not more than 300 mm, the distance between the extreme outer edge of the vehicle and the outer edge of the illuminating surface of the rear direction indicator lamp must not exceed, by more than 50 mm the distance between the extreme outer edge of the vehicle and the outer edge of the illuminating surface of the corresponding rear position (side) lamp.'

Item 4.5.5 is hereby amended as follows:

- '4.5.5. *Geometric visibility*

Horizontal angles: see Appendix 4.

Vertical angles: 15° above and below the horizontal. The vertical angle below the horizontal may be reduced to 5° if the lamps are less than 750 mm above the ground.'

Item 4.5.12 is hereby amended as follows:

- '4.5.12. *Other requirements*

The light shall be a flashing light, flashing 90 ± 30 times per minute.

Operation of the light signal control shall be followed within not more than one second by the emission of light and within not more than one and one half seconds by its first extinction. If a motor vehicle is equipped to draw a trailer, the control of the direction indicator lamps on the drawing vehicle shall also be capable of operating the indicator lamps on the trailer.

In the event of the failure of one direction indicator lamp, which is not due to a short circuit, the others must continue to flash but, under these conditions, the frequency may be different from that specified.

For front direction indicator lamps, the illuminating surface must be not less than 40 mm from the illuminating surface of the dipped-beam headlamps or front fog lamps if any. A smaller distance is permitted if the luminous intensity in the reference axis of the direction indicator lamp is not less than 400 cd.'

Item 4.7.8 is hereby amended as follows:

- '4.7.8. Must not be "combined" with another lamp, unless the rear position lamp is reciprocally incorporated with the stop lamp and combined with the rear registration plate lamp.'

Item 4.7.10 is hereby amended as follows:

'4.7.10. *Electrical connections*

Must light up when the service brake is applied. The stop lamps need not function if the device which starts and/or stops the engine is in a position which makes it impossible for the engine to operate.'

Item 4.7.12 shall be deleted.

After Item 4.8.8, add the following new Item 4.8.8.1:

'4.8.8.1. When the rear position lamps are reciprocally incorporated with the stop lamps, the photometric characteristics of the rear registration plate lamp may be modified during the illumination of the stop lamps.'

Item 4.8.10 is hereby amended as follows:

'4.8.10. *Electrical connections*

No individual specifications.'

Item 4.8.11 is hereby amended as follows:

'4.8.11. *Tell-tale*

Tell-tale optional. If it exists, its function must be carried out by the tell-tale required for the front and rear position (side) lamps.'

Item 4.9.11 is hereby amended as follows:

'4.9.11. *Tell-tale*

Circuit-closed tell-tale mandatory. This tell-tale shall be non-flashing and shall not be required if the instrument panel lighting can only be turned on simultaneously with the front position (side) lamps.'

Item 4.10.11 is hereby amended as follows:

'4.10.11. *Tell-tale*

Circuit-closed tell-tale mandatory. It must be combined with that of the front position (side) lamps.'

Item 4.11.4.1 is hereby amended as follows:

'4.11.4.1. *Width:*

If there is only one rear fog lamp, it must be on the opposite side of the median longitudinal plane of the vehicle to the direction of traffic prescribed in the country of registration; the centre of reference may also be situated on the median longitudinal plane of the vehicle.'

Item 4.11.10 is hereby amended as follows:

'4.11.10. *Electrical connections*

These must be such that the rear fog lamp can operate only when the dipped beam or main beam headlamps or the front fog lamps or a combination of these are switched on. They must be such that when the rear fog lamp is switched on it is capable of operating together with the main beam, the dipped beam and the front fog lamps.

When the rear fog lamp is switched on, operating the main-beam or dipped-beam control shall not extinguish the rear fog lamp.

If the front fog lamps exist, the extinguishing of the rear fog lamp must be possible independently from that of the front fog lamps.'

Item 4.11.11 is hereby amended as follows:

'4.11.11. *Tell-tale*

Circuit-closed tell-tale mandatory. An independant non-flashing warning light.'

After Item 4.11.11 add:

'4.11.12. *Other requirements*

In all cases, the distance between the rear fog lamp and each stop lamp must be greater than 100 mm.'

Item 4.12.10 is hereby amended as follows:

'4.12.10. *Electrical connections*

The connection must allow the parking lamp(s) on the same side of the vehicle to be lit independently of any other lamps.

The parking lamp(s) must be able to function even if the device which starts and/or stops the engine is in a position which makes it impossible for the engine to operate.'

Item 4.12.11 is hereby amended as follows:

'4.12.11. *Tell-tale*

Circuit-closed tell-tale optional. If there is one, it must not be possible to confuse it with the tell-tale for the front and rear position (side) lamps.'

Item 4.13.7 is hereby amended as follows:

'4.13.7. May be "grouped" with other lamps.'

Item 4.13.11 is hereby amended as follows:

'4.13.11. *Tell-tale*

Tell-tale optional. If it exists, its function shall be carried out by the tell-tale required for the front and rear position (side) lamps.'

Item 4.14.4.3 is hereby amended as follows:

'4.14.4.3. Length

At rear of vehicle.'

Item 4.15.4.3 is hereby amended as follows:

'4.15.4.3. Length

At rear of vehicle.'

Item 4.16.4.3 is hereby amended as follows:

'4.16.4.3. Length

At front of vehicle.'

Item 4.16.5 is hereby amended as follows:

'4.16.5. *Geometric visibility*

Horizontal angle: 30° inwards and outwards.

If, on account of adjustable drawbars, it is not possible to satisfy the inward angle requirement of 30°, it may be reduced to 10°.

Vertical angle: 15° above and below the horizontal. The vertical angle below the horizontal may be reduced to 5° in the case of a reflex reflector, less than 750 mm above the ground.'

Item 4.17.4.3 is hereby amended as follows:

4.17.4.3. Length

At least one reflex reflector must be fitted to the middle third of the vehicle, the foremost reflex reflector being not further than 3 m from the front, in the case of trailers, account shall be taken of the length of the drawbar for the measurement of this distance.

The distance between two adjacent reflex reflectors shall not exceed 3 m.

The distance between the rearmost reflex reflector and the rear of the vehicle shall not exceed 1 m.

However, for M₁ category vehicles, it is sufficient to have one reflex reflector fitted within the first third and one within the last third of the vehicle length.'

After Appendix 4, the following new Appendices 5, 6 and 7 shall be added:

Appendix 5

Measurement of the variation of dipped beam inclination as a function of load

1. SCOPE

This Appendix specifies a method for measuring variations in motor vehicle dipped beam inclination, in relation to its initial inclination, caused by changes in vehicle attitude due to loading.

2. DEFINITIONS

2.1. Initial inclination

2.1.1. Stated initial inclination:

the value of the dipped beam initial inclination specified by the motor vehicle manufacturer serving as a reference value for the calculation of permissible variations.

2.1.2. Measured initial inclination:

the mean value of dipped beam inclination or vehicle inclination measured with the vehicle in condition No 1, as defined in Appendix 1, for the category of vehicle under test. It serves as a reference value for the assessment of variations in beam inclination as the load varies.

2.2. Dipped-beam inclination may be defined:

- either as the angle, expressed in milliradians, between the direction of the beam towards a characteristic point on the horizontal part of the cut-off in the luminous distribution of the headlamp and the horizontal plane,
- or by the tangent of that angle, expressed in percentage inclination, since the angles are small (for these small angles, 1 % is equal to 10 mrad).

If the inclination is expressed in percentage inclination, it can be calculated by means of the following formula:

$$\frac{h_1 - h_2}{l} \times 100$$

where:

h_1 is the height above the ground, in millimetres, of the abovementioned characteristic point, measured on a vertical screen perpendicular to the vehicle longitudinal median plane, placed at a horizontal distance l ;

h_2 is the height above the ground, in millimetres, of the centre of reference (which is taken to be the nominal origin of the characteristic point chosen in h_1);

l is the distance, in millimetres, from the screen to the centre of reference.

Negative values denote downward inclination (see Figure 1).

Positive values denote upward inclination.

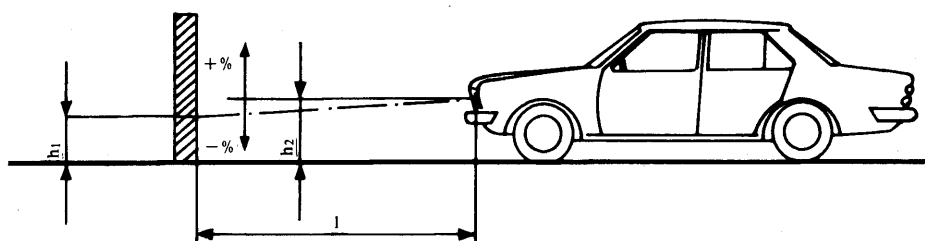


Figure 1

Dipped beam downward inclination of a category M_1 vehicle

Notes:

1. This drawing represents a category M_1 vehicle, but the principle shown applies equally to vehicles of other categories.
 2. Where the vehicle does not incorporate a headlamp levelling system, the variation in dipped beam inclination is identical with the variation in the inclination of the vehicle itself.
3. MEASUREMENT CONDITIONS
 - 3.1. If a visual inspection of the dipped beam pattern on the screen or a photometric method is used, measurement shall be carried out in a dark environment (for example, a dark room) of sufficient area to allow the vehicle and the screen to be placed as shown in Figure 1. Headlamp centres of reference shall be at a distance l from the screen of at least 10 m.
 - 3.2. The ground on which measurements are made shall be as flat and horizontal as possible, so that the reproducibility of measurements of dipped beam inclination can be assured with an accuracy of ± 0.5 mrad (± 0.05 % inclination).
 - 3.3. If a screen is used, its marking, position and orientation in relation to the ground and to the median longitudinal plane of the vehicle, shall be such that the reproducibility of the measurement of the dipped beam inclination can be assured with an accuracy of ± 0.5 mrad (± 0.05 % inclination).
 - 3.4. During measurements, the ambient temperature shall be between 10 and 30 °C.
 4. VEHICLE PREPARATION
 - 4.1. Measurements shall be carried out on a vehicle which has travelled a distance of between 1 000 and 10 000 km, preferably 5 000 km.
 - 4.2. Tyres shall be inflated to the full-load pressure specified by the vehicle manufacturer. The vehicle shall be fully replenished (fuel, water, oil) and equipped with all the accessories

and tools specified by the manufacturer. Full fuel replenishment means that the fuel tank must be filled to not less than 90 % of its capacity as indicated in the information document, a model of which appears in Annex I to Directive 70/156/EEC.

- 4.3. The vehicle shall have the parking brake released and the gearbox in neutral.
- 4.4. The vehicle shall be conditioned for at least 8 h at the temperature specified in Item 3.4.
- 4.5. If a photometric or visual method is used, headlamps with a well-defined dipped beam cut-off should preferably be installed on the vehicle under test in order to facilitate the measurements.

Other methods may be employed to obtain a more precise reading (for example, removal of the headlamp lens).

5. TEST PROCEDURE

5.1. General

The variations in either dipped beam or vehicle inclination, depending on the method chosen, shall be measured separately for each side of the vehicle. The results obtained from both left and right headlamps under all the load conditions specified in Appendix 1, shall be within the limits set out in Item 5.5. The load shall be applied gradually without subjecting the vehicle to excessive shocks.

5.2. Determination of the measured initial inclination

The vehicle shall be prepared as specified in Item 4 and laden as specified in Appendix 1 (first loading condition of the respective vehicle category).

Before each measurement, the vehicle shall be rocked as specified in Item 5.4.

Measurements shall be made three times.

- 5.2.1. If none of the three measured results differs by more than 2 mrad (0.2 % inclination) from the arithmetic mean of the results, that mean shall constitute the final result.
- 5.2.2. If any measurement differs from the arithmetic mean of the results by more than 2 mrad (0.2 % inclination), a further series of 10 measurements shall be made, the arithmetic mean of which shall constitute the final result.

5.3. Measurement methods

Any method may be used to measure variations of inclination provided that the readings are accurate to within ± 0.2 mrad (± 0.02 % inclination).

5.4. Treatment of vehicle in each loading condition

The vehicle suspension and any other part likely to affect dipped beam inclination shall be activated according to the methods described below.

However, the technical authorities and manufacturers may jointly propose other methods (either experimental or based upon calculations), especially when the test poses particular problems, provided such calculations are clearly valid.

5.4.1. *M₁ category vehicles with conventional suspension*

With the vehicle standing on the measuring site and, if necessary, with the wheels resting on floating platforms (which must be used if their absence would lead to restriction of the suspension movement likely to affect the results of measurements), rock the vehicle continuously for at least three complete cycles; for each cycle, first the rear and then the front end of the vehicle is pushed down.

The rocking sequence shall end with the completion of a cycle. Before making the measurements, the vehicle shall be allowed to come to rest spontaneously. Instead of using floating platforms, the same effect can be achieved by moving the vehicle backwards and forwards for at least a complete wheel revolution.

5.4.2. *M₂, M₃ and N category vehicles with conventional suspension*

5.4.2.1. If the treatment method for category M₁ vehicles described in Item 5.4.1 is not possible, the method described in Item 5.4.2.2 or 5.4.2.3 may be used.

5.4.2.2. With the vehicle standing on the measuring site and the wheels on the ground, rock the body by temporarily varying the load.

5.4.2.3. With the vehicle standing on the measuring site and the wheels on the ground, activate the vehicle suspension and all other parts which may affect the dipped beam inclination by using a vibration rig. This can be a vibrating platform on which the wheels rest.

5.4.3. *Vehicles with non-conventional suspension, where the engine has to be running*

Before making any measurement wait until the vehicle has assumed its final attitude with the engine running.

5.5. **Measurements**

The variation of the inclination of the dipped beam shall be assessed for each of the different loading conditions in relation to the measured initial inclination determined in accordance with Item 5.2.

If the vehicle is fitted with a manual headlamp-levelling system, the latter shall be adjusted to the positions specified by the manufacturer for given loading conditions (according to Appendix 1).

5.5.1. To begin with, a single measurement shall be made in each loading condition. Requirements have been met if, for all the loading conditions, the variation in inclination is within the calculated limits (for example, within the difference between the stated initial inclination and the lower and upper limits specified for approval) with a tolerance of 4 mrad (0.4 % inclination).

5.5.2. If the result(s) of any measurement(s) does (do) not lie within the tolerance indicated in Item 5.5.1 or exceed(s) the limit values, a further three measurements shall be made in the loading conditions corresponding to this (these) result(s) as specified in Item 5.5.3.

5.5.3. For each of the above loading conditions:

5.5.3.1. If none of the three measured results differs by more than 2 mrad (0.2 % inclination) from the arithmetic mean of the results, that mean shall constitute the final result.

5.5.3.2. If any measurement differs from the arithmetic mean of the results by more than 2 mrad (0.2 % inclination), a further series of ten measurements shall be made, the arithmetic mean of which shall constitute the final result.

5.5.3.3. If a vehicle is fitted with an automatic headlamp-levelling system which has an inherent hysteresis loop, average results at the top and bottom of the hysteresis loop shall be taken as significant values.

All these measurements shall be made in accordance with Items 5.5.3.1 and 5.5.3.2.

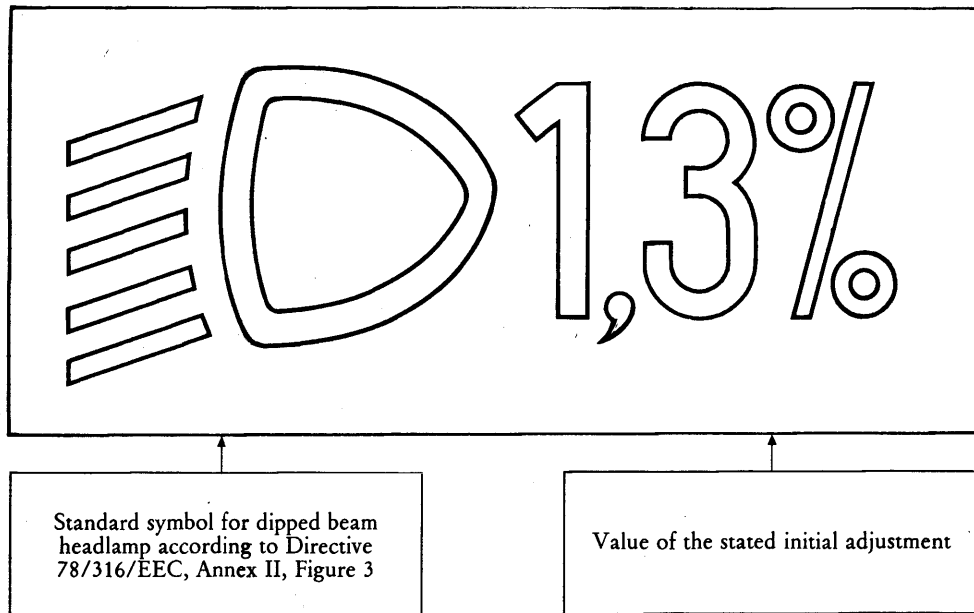
5.5.4. Requirements have been met, if, under all loading conditions, the variation between the measured initial inclination determined in accordance with Item 5.2 and the inclination measured under each loading condition is less than the values calculated in Item 5.5.1 (without safety margin).

5.5.5. If only one of the calculated upper or lower limits of variation is exceeded, the manufacturer shall be permitted to choose a different value for the stated initial inclination, within the limits specified for approval.

Appendix 6

Indication of the stated initial adjustment referred to in Item 4.2.6.1 of Annex I

Example



The size of the symbol and characters is left to the discretion of the manufacturer.'

Appendix 7

The controls for the headlamp-levelling devices referred to in Items 4.2.6.2.2 of Annex I

1. SPECIFICATIONS

1.1. Downward inclination of the dipped beam must in all cases be produced in one of the following ways:

- (a) by moving a control downwards or to the left;
- (b) by rotating a control in a counterclockwise direction;
- (c) by depressing a button (push-pull control).

If several buttons are used to adjust the beam, the button which gives the greatest downward inclination must be installed to the left or below the button(s) for other dipped beam positions.

A rotary control which is installed edge-on, or with only the edge visible, should follow the operating principles of controls of types (a) or (c).

1.1.1. This control must carry symbols indicating clearly the movements corresponding to the downward and upward inclination of the dipped beam.

1.2. The "0" position corresponds to the initial alignment according to Item 4.2.6.1 of Annex I.

1.3. The "0" position which, according to Item 4.2.6.2.2 of Annex I, has to be a "stop position", need not necessarily be at the end of the scale.

1.4. The marks used on the control must be explained in the owner's handbook.

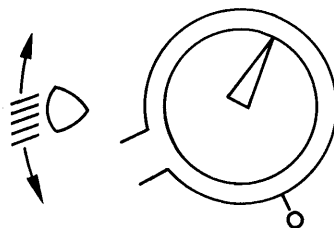
- 1.5. Only the following symbols may be used to identify the controls:



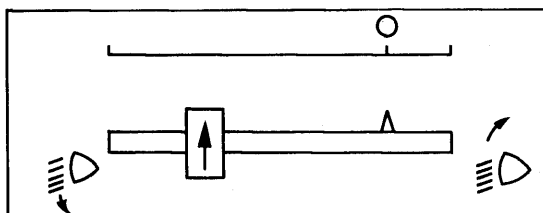
Standard symbol for dipped beam headlamp according to Figure 3 of Annex II to Directive 78/316/EEC, showing the arrows which correspond to the direction of the dipped beam adjustment.

2. EXAMPLES

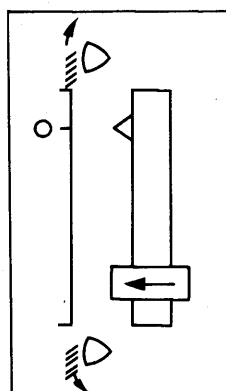
Example 1:



Example 2:



Example 3:



ANNEX II

After Item 5.2 add the following new Item:

'5.2.1. Headlamp levelling device: yes/no (*)'.