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Contents

I *Acts whose publication is obligatory*

.....

II *Acts whose publication is not obligatory*

Commission

89/516/EEC:

- ★ Commission Directive of 1 August 1989 adapting to technical progress Council Directive 76/758/EEC on the approximation of the laws of the Member States relating to the end-outline marker lamps, front position (side) lamps, rear position (side) lamps and stop lamps for motor vehicles and their trailers 1

89/517/EEC:

- ★ Commission Directive of 1 August 1989 adapting to technical progress Council Directive 76/761/EEC on the approximation of the laws of the Member States relating to motor-vehicle headlamps which function as main-beam and/or dipped-beam headlamps and to incandescent electric filament lamps for such headlamps 15

89/518/EEC:

- ★ Commission Directive of 1 August 1989 adapting to technical progress Council Directive 77/538/EEC on the approximation of the laws of the Member States relating to rear fog lamps for motor vehicles and their trailers 24

89/519/EEC:

- ★ Commission Directive of 1 August 1989 supplementing and amending Directive 77/535/EEC on the approximation of the laws of Member States relating to methods of sampling and analysis for fertilizers 30

2

Acts whose titles are printed in light type are those relating to day-to-day management of agricultural matters, and are generally valid for a limited period.

The titles of all other Acts are printed in bold type and preceded by an asterisk.

II

(Acts whose publication is not obligatory)

COMMISSION

COMMISSION DIRECTIVE

of 1 August 1989

adapting to technical progress Council Directive 76/758/EEC on the approximation of the laws of the Member States relating to the end-outline marker lamps, front position (side) lamps, rear position (side) lamps and stop lamps for motor vehicles and their trailers

(89/516/EEC)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

HAS ADOPTED THIS DIRECTIVE:

Having regard to the Treaty establishing the European Economic Community,

Article 1

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 87/403/EEC ⁽²⁾, and in particular Article 11 thereof,

The list of Annexes and Annexes 0, I, II and III to Directive 76/758/EEC are hereby amended as set out in the Annex to this Directive.

Having regard to Council Directive 76/758/EEC of 27 July 1976 on the approximation of the laws of the Member States relating to the end-outline marker lamps, front position (side) lamps, rear position (side) lamps and stop lamps for motor vehicles and their trailers ⁽³⁾, as last amended by Directive 87/354/EEC ⁽⁴⁾, and in particular Article 10 thereof,

Article 2

Whereas, in the light of experience, it is now possible to simplify the EEC component type-approval mark for lamps of this type when these are grouped together with, combined with or reciprocally incorporated in other lamps, and to amend certain provisions to take account of the technical progress made in the meantime;

1. With effect from 1 January 1990, no Member State may:

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

— prohibit the entry into service of the vehicles,

if the end-outline marker lamps, front position (side) lamps, rear position (side) lamps and stop lamps comply with the provisions of this Directive;

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,

(b) — refuse, in respect of a type of end-outline marker lamp, front position (side) lamp, rear position (side) lamp and stop lamp, to grant EEC component type-approval or national type-approval, if these end-outline marker lamps, front position (side) lamps, rear position (side) lamps and stop lamps comply with the provisions of this Directive, or

— prohibit the placing on the market of end-outline marker lamps, front position (side) lamps, rear

⁽¹⁾ OJ No L 42, 23. 2. 1970, p. 1.

⁽²⁾ OJ No L 220, 8. 8. 1987, p. 44.

⁽³⁾ OJ No L 262, 27. 9. 1976, p. 54.

⁽⁴⁾ OJ No L 192, 11. 7. 1987, p. 43.

position (side) lamps and stop lamps which bear the EEC component type-approval mark issued in accordance with the requirements of this Directive.

2. With effect from 1 July 1990 a Member State:

- (a) — shall not issue the document referred to in the third indent of Article 10 (1) of Directive 70/156/EEC in respect of a type of vehicle of which the end-outline marker lamps, front position (side) lamps, rear position (side) lamps and stop lamps do not comply with the provisions of this Directive,
 - may refuse to grant national type-approval in respect of a type of vehicle of which the end-outline marker lamps, front position (side) lamps, rear position (side) lamps and stop lamps do not comply with the provisions of this Directive;
- (b) — shall not grant EEC component type-approval in respect of a type of end-outline marker lamp, front position (side) lamp, rear position (side) lamp and stop lamp if the latter does not comply with the provisions of this Directive,
 - may refuse to grant national component type-approval in respect of a type of end-outline marker lamp, front position (side) lamp, rear position (side) lamp and stop lamp if the latter does not comply with the provisions of this Directive.

3. With effect from 1 October 1995, Member States may prohibit the entry into service of vehicles of which the end-outline marker lamps, front position (side) lamps, rear position (side) lamps and stop lamps do not comply with the provisions of this Directive and the placing on the market of those lamps which do not bear the component type-approval mark issued in accordance with the requirements of this Directive.

4. By way of derogation from the provisions of 2 (b) above, Member States shall continue to recognize EEC

component type-approval granted for a type of end-outline marker lamp, front position (side) lamp, rear position (side) lamp or stop lamp pursuant to the provisions of Directive 76/758/EEC in the case of devices intended to be mounted on vehicles already in service, and may also grant EEC component type-approval for a type of end-outline marker lamp, front position (side) lamp, rear position (side) lamp or stop lamp pursuant to the provisions of Directive 76/758/EEC provided that such devices are intended to be used as replacement parts for vehicles already in service and it is not technically possible for the said devices to comply with the provisions of this Directive.

Article 3

Member States shall bring into force the provisions necessary to comply with this Directive not later than 31 December 1989. They shall forthwith inform the Commission thereof.

The provisions adopted pursuant to the first subparagraph shall make express reference to this Directive.

Article 4

This Directive is addressed to the Member States.

Done at Brussels, 1 August 1989.

For the Commission
Martin BANGEMANN
Vice-President

ANNEX

The list of Annexes is modified as follows:

The text of Annex I is replaced by the following text:

‘— Front position (side) lamps, rear position (side) lamps, stop lamps and end-outline marker lamps; minimum angles required for the light distribution in space’

The text of Annex 0 is modified as follows:

Item 1.4 is deleted:

Items 1.4.1, 1.4.2, 1.4.3, 1.5 and 1.6 are deleted

Insert new items 1.4 and 1.5 as follows:

1.4. Definition of terms

The definitions contained in Directive 76/756/EEC relating to:

- light source (for filament lamps),
 - independent lamps,
 - grouped lamps,
 - combined lamps,
 - reciprocally incorporated lamps,
 - device,
 - single-function lamps
 - single lamps
 - two lamps or an even number of lamps,
 - light emitting surface,
 - illuminating surface of a light-signalling device other than a reflex reflector,
 - apparent surface,
 - axis of reference,
 - centre of reference,
- apply to this Directive.

1.5. Front and rear position (side) lamp, stop lamp and end-outline marker lamp of different types

“Front and rear position (side) lamp, stop lamp and end-outline marker lamp of different types” means lamps which differ in each said category in such essential respects as:

- the trade name or mark,
- the characteristics of the optical system, (levels of intensity, light distribution angles, type of filament lamp, etc.),
- the system used to reduce illumination at night — in the case of stop lamps with two levels of intensity.’

Item 5.5., read:

‘5.5. Front and rear position (side) lamps which are grouped or combined or reciprocally incorporated may also be used as end-outline marker lamps.’

Items 6 to 9 are replaced by the following items:

6. INTENSITY OF LIGHT EMITTED

6.1. In the reference axis, the light emitted by each of the two devices supplied must be of not less than the minimum intensity and of not more than the maximum intensity specified below:

(1)	Minimum intensities cd	Maximum values in cd when used as:		
		Single-function lamp	Single function lamp marked "D" (Annex III item 4.3.6)	Total for the assembly of two lamps (Annex III item 4.3.6)
6.1.1. Front position (side) lamps, front end-outline marker lamp	4	60 (2)	42 (2)	84 (2)
6.1.2. Front position (side) lamps incorporated in headlamp	4	100 (2)	—	—
6.1.3. Rear position (side) lamps, rear end-outline marker lamp	4	12 (2)	8,5 (2)	17 (2)
6.1.4. Stop lamps				
6.1.4.1. with 1 level of intensity	40	100 (2)	70 (2)	140 (2)
6.1.4.2. with 2 levels of intensity				
6.1.4.2.1. by day	130	520 (2)	364 (2)	728 (2)
6.1.4.2.2. by night	30	80 (2)	56 (2)	112 (2)

(1) The installation of the devices referred to above in power-driven vehicles and their trailers is provided for in the Directive concerning the installation of lighting and light-signalling devices (Directive 76/756/EEC).

(2) The total value of maximum intensity for an assembly of two lamps is given by multiplying by 1.4 the value prescribed for a single-function lamp.

When two single-function lamps having the same function, whether identical or not, are grouped in one device such that the projections of the illuminating surfaces of those single-function lamps on a vertical plane perpendicular to the vehicle's median longitudinal plane occupy not less than 60 % of the smallest rectangle circumscribing the projections of the said illuminating surfaces, such an assembly shall be deemed to be a single lamp for the purpose of installation of a vehicle. In such a case, each single-function lamp shall comply with the minimum intensity required, the admissible maximum intensity shall not be exceeded by both lamps together (last column of the table).

In the case of a single-function lamp containing more than one light source:

The lamp shall comply with the minimum intensity required when any one light source has failed, and when all light sources are illuminated the maximum intensity specified for a single-function lamp may be exceeded, provided that the single-function lamp is not marked "D" and the maximum intensity specified for an assembly of two lamps (last column of the table) is not exceeded.

- 6.2. Outside the reference axis and within the angular fields defined in the diagrams in Annex I the intensity of the light emitted by each of the two devices supplied must:
- 6.2.1. in each direction corresponding to the points in the light distribution table reproduced in Annex IV, be not less than the product of the minimum specified in paragraph 6.1 above by the percentage specified in the said table for the direction in question;
- 6.2.2. in no direction within the space from which the light-signalling device is visible, exceed the maximum specified in paragraph 6.1 above;
- 6.2.3. however, a luminous intensity of 60 cd shall be permitted for rear position (side) lamps reciprocally incorporated with stop lamps (see paragraph 6.1.3 above) below a plane forming an angle of 5° with and downward from the horizontal plane;
- 6.2.4. Moreover,
- 6.2.4.1. throughout the fields defined in the diagrams in Annex I, the intensity of the light emitted must be not less than 0,05 cd for front and rear position (side) lamps and end-outline marker lamps, not less than 0,3 cd for stop lamps with one level of intensity, and for stop lamps with two levels of intensity 0,3 cd by day and 0,07 cd by night.
- 6.2.4.2. if a rear position (side) lamp is reciprocally incorporated with a stop lamp, the ratio between the luminous intensities actually measured of the two lamps when turned on simultaneously and the intensity of the rear position (side) lamp when turned on alone should be at least 5:1 in the field delimited by the straight horizontal lines passing through $\pm 5^\circ$ V and the straight vertical lines passing through $\pm 10^\circ$ H of the light distribution table. Where a stop lamp has two levels of intensity this requirement must be met when the night-time use facility is actuated.
- 6.2.4.3. the provisions of paragraph 2.2 of Annex IV on local variations of intensity must be observed.
- 6.3. the intensities shall be measured while the filament lamp(s) continuously alight. In the case of devices emitting selective-yellow or red light, they shall be measured in coloured light.

- 6.4. In the case of a stop lamp providing two levels of intensity the time that elapses between electrical supply being switched on and the light output measured on the reference axis to reach 90 % of the value measured in accordance with paragraph 6.3 above shall be measured for both the day and the night conditions of use. The time measured for the night conditions of use shall not exceed that measured for the day conditions of use.
- 6.5. Annex IV, to which reference is made in paragraph 6.2.1 above, gives particulars of the methods of measurement to be used.
7. TEST PROCEDURE
- 7.1. All measurements shall be made with a white standard filament lamp of the category prescribed for the device, the supply voltage being so regulated as to produce the nominal luminous flux prescribed for that category of lamp.
- 7.2. However, in the case of a stop lamp for which an additional system is used to obtain the night-time intensity, the voltage applied to the system for measuring the night-time intensity shall be that which was applied to the filament lamp for measuring the daytime intensity ⁽¹⁾.
- 7.3. Where a rear position (side) lamp is reciprocally incorporated with a dual-intensity stop lamp and is designed to operate permanently with an additional system to regulate the intensity of the light emitted, measurement of the light emitted shall be performed with the same voltage applied to the system as would, if applied to the filament lamp, enable the lamp to produce the prescribed normal luminous flux.
- 7.4. The vertical and horizontal outlines of the illuminating surface of a light-signalling device other than a reflex reflector shall be determined and measured in relation to the centre of reference.
8. COLOUR OF LIGHT EMITTED
- The colour of the light emitted shall be within the limits of the coordinates prescribed for the colour in question in Annex V to this Directive.
9. CONFORMITY OF PRODUCTION
- Every device bearing an EEC component type-approval mark must conform to the approved type and comply with the photometric conditions specified in sections 6 and 8. Nevertheless, in the case of a device picked at random from series production, the requirements as to minimum intensity of the light emitted (measured with a standard filament lamp as referred to in section 7) may be limited in each direction to 80 % of the minimum values specified in 6.1 and 6.2.

⁽¹⁾ The functioning and installation conditions of these additional systems will be defined by special provisions.

Annex I is modified as follows:

Title, read:

**'FRONT POSITION (SIDE) LAMPS, REAR POSITION (SIDE) LAMPS, STOP LAMPS AND END-OUTLINE
MARKER LAMPS
MINIMUM ANGLES REQUIRED FOR THE LIGHT DISTRIBUTION IN SPACE (*)'**

'end outline marker lamps' is to be added after 'front position (side) lamps' and 'rear position (side) lamps' in the titles for the first two diagrams.

Annex II is replaced as follows:

'ANNEX II

MODEL EEC COMPONENT TYPE-APPROVAL CERTIFICATE

(Maximum format: A 4 (210 × 297 mm))

Name of administration

Notification concerning the granting, refusal or withdrawal of EEC component type-approval, or the granting, refusal or withdrawal of an extension of EEC component type-approval for a type of end-outline marker lamp, front position (side) lamp, rear position (side) lamp or stop lamp

Component type-approval No

1.	Type of lamp	Intended for use in a composition of two lamps
	front position (side) lamp	yes/no (*)
	rear position (side) lamp	yes/no (*)
	stop lamp	yes/no, one/two (*) level(s) of intensity
	end-outline marker lamp	yes/no (*)

2. Type and number of incandescent lamps

3. Colour of light emitted: red/selective yellow/white (*)

4. Trade name or mark

5. Name and address of manufacturer

6. If applicable, name and address of manufacturer's authorized representative

7. Submitted for EEC component type-approval tests on

8. Technical service conducting EEC component type-approval tests

9. Date of report issued by that service

10. Number of report issued by that service

11. Date of granting/refusal/withdrawal of EEC component type-approval (*)

12. Extension of EEC component type-approval to devices emitting a red/selective yellow/white light (*)

13. Date of granting/refusal/withdrawal of the extension of EEC component type-approval (*)

14. Single EEC component type-approval granted on the basis of paragraph 3.3 of Annex III for a lighting and light-signalling device comprising several lamps, and in particular

15. Where a rear position (side) lamp is reciprocally incorporated with a dual-intensity stop lamp, state whether a voltage-adapting system is provided, and if so what its characteristics are

16. Component type approval granted solely for use as a replacement part on vehicles already in service: yes/no (*)

17. For stop lamps with two levels of intensity, indicate the system used to obtain the night-time intensity (give the main characteristics)

18. Date of refusal/withdrawal of single EEC component type-approval

19. Place

20. Date

21. Signature

22. The attached drawing No shows the geometrical position in which the device is to be mounted on the vehicle and the axis of reference and centre of reference of the device.

23. Remarks

(*) Delete where inapplicable.

Annex III is modified as follows:

Item 1.2, read:

- '1.2. The request for the EEC component type approval of a front position (side) lamp shall state whether that lamp is intended to emit white or selective yellow light, and in the case of an end-outline marker lamp, whether that lamp is intended to emit white or red light.'

Item 1.3, read:

- '1.3. For each type of front position (side) lamp, rear position (side) lamp, stop lamp, and end-outline marker lamp the request shall be accompanied by:'

Item 1.3.2, read:

- '1.3.2. drawings (three copies), in sufficient detail to permit identification of the type of device and showing geometrically the position in which the lamp is to be mounted on the vehicle, the axis of observation to be taken as the axis of reference in the tests (horizontal angle $H = 0^\circ$), the point to be taken as the centre of reference in the said tests, the vertical and horizontal tangents to the illuminating surface and their distances from the centre of reference of the lamp'.

Add the following new item 1.3.3 after item 1.3.2:

- '1.3.3. In the case of a dual-intensity stop lamp, a diagram and statement of the characteristics of the systems providing the two levels of intensity;'

Former item 1.3.3 becomes item 1.3.4, and the following is added to it:

'In the case of a dual-intensity stop lamp the request must, moreover, be accompanied by the samples of the components making up the system providing the two levels of intensity.'

Item 3.2, read:

- '3.2. This number is no longer assigned to another type of front position (side) lamp, stop lamp or an end-outline marker lamp except where EEC component type-approval is extended to another type of device which only differs in respect of the colour of the light emitted.'

Item 3.3, read:

- '3.3. Where EEC component type-approval is requested for a type of lighting and light-signalling device consisting of a front position (side) lamp, a rear position (side) lamp, a stop lamp or an end-outline marker lamp and other lamps, a single EEC component type-approval number may be granted, provided that the lamp fulfills the requirements of this Directive and that each of the other lamps forming part of the type of lighting and light-signalling device for which EEC component type approval has been requested complies with the separate directive applying thereto.'

Item 4.1, read:

- '4.1. All front position (side) lamps, rear position (side) lamps, stop lamps, or end-outline marker lamps conforming to a type that has been component type-approved pursuant to this Directive shall bear an EEC component type-approval mark'.

Item 4.2, read:

- '4.2. This mark shall consist of a rectangle surrounding the lower case letter "e" followed by the distinguishing letter(s) or number of the Member State which has granted the type-approval:
- 1 for Germany,
 - 2 for France,
 - 3 for Italy,
 - 4 for the Netherlands,
 - 6 for Belgium,
 - 9 for Spain,
 - 11 for the United Kingdom,
 - 13 for Luxembourg
 - 18 for Denmark,
 - 21 for Portugal,
 - EL for Greece,
 - IRL for Ireland,

It must also include the EEC component type-approval number which corresponds to the number of the EEC component type-approval certificate issued for the type of device in question (see Annex I), preceded by two figures indicating the sequence number assigned to the most recent major technical amendment to Council Directive 76/758/EEC on the date EEC component type-approval was granted. In this Directive the sequence number is 01.'

Item 4.3.3, read:

- '4.3.3. The letter "S" followed by the figure "1" where the device has one level of intensity, and by the figure "2" when it has two levels of intensity on devices fulfilling the requirements of this Directive in respect of stop lamps;'

Item 4.3.4, read:

- '4.3.4. Depending upon the case the letters "R" and "S 1" or "S 2", separated by a horizontal dash, on devices consisting, at the same time, of a rear position (side) lamp and a stop lamp fulfilling the requirements of this Directive in respect of these lamps;'

After item 4.3.5, add the new item 4.3.6 as follows:

- '4.3.6. On lamps which may be used as single-function lamps as well as in an assembly of two lamps, the additional letter "D" on the right side of the symbol mentioned in paragraphs 4.3.1 to 4.3.4.'

Item 4.6, read:

- '4.6. Examples of EEC component type-approval marks and additional symbols are shown in Appendix 1.'

After item 4.7, the following new items 4.7.1 and 4.7.2 are added:

- '4.7.1. This mark may be placed anywhere on lamps that are grouped, combined or reciprocally incorporated provided that:
- 4.7.1.1. it is visible when the lamps have been installed;
 - 4.7.1.2. no light-transmitting component of the grouped, combined or reciprocally incorporated lamps may be removed without the component type-approval mark being removed at the same time.
- 4.7.2. The identification symbol for each lamp corresponding to each Directive pursuant to which component type-approval was granted, together with the two figures referred to in item 4.2, final indent, above and, where necessary, the additional letter "D" shall be entered:
- 4.7.2.1. on the appropriate light-emitting surface; or
 - 4.7.2.2. in a group, such a way that each of the grouped, combined or reciprocally incorporated lamps may be clearly identified'.

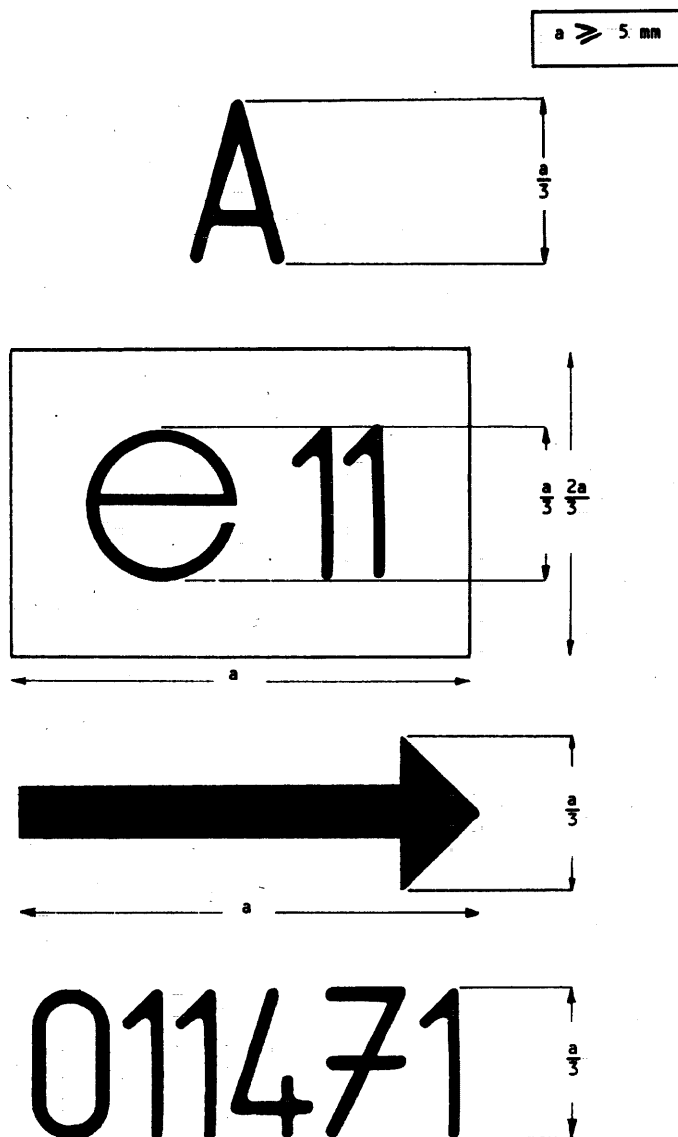
After item 4.8 the following new point 4.9 is added:

- '4.9. Examples of an EEC component type-approval mark for a lamp that is grouped, combined or reciprocally incorporated with other lamps are shown in Annex II.'

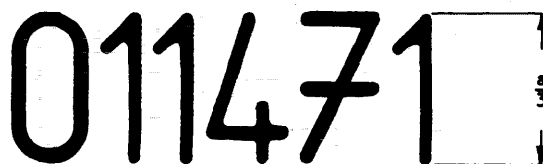
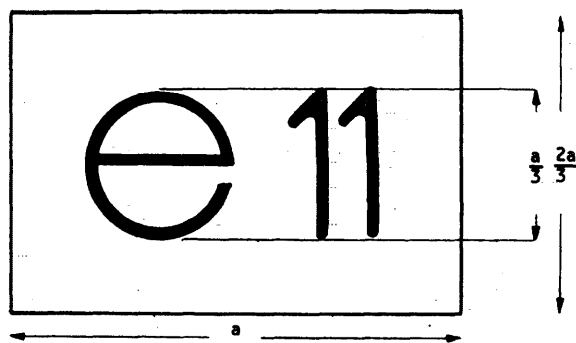
The Appendix is replaced by the following Appendix 1:

Appendix 1

EXAMPLES OF EEC COMPONENT TYPE-APPROVAL MARKS

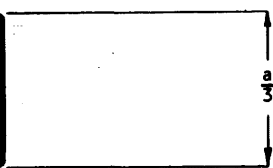


The device bearing the EEC component type-approval mark shown above is a front position (side) lamp, EEC type-approved in the United Kingdom (e 11) under the number 1471. The arrow indicates the side on which the photometric specifications are satisfied up to an angle of 80°H.

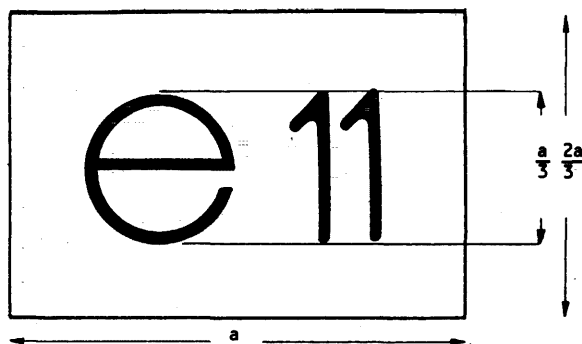


The device bearing the EEC type-approval mark shown above is a rear position (side) lamp, EEC type-approved in the United Kingdom (e 11) under the number 1471. The absence of an arrow means that, both right and left, the photometric specifications are satisfied up to an angle of 80°H.

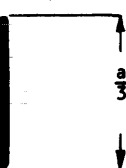
S1



A rectangular box with a height dimension indicated by a vertical arrow on the right side, labeled $\frac{a}{3}$.

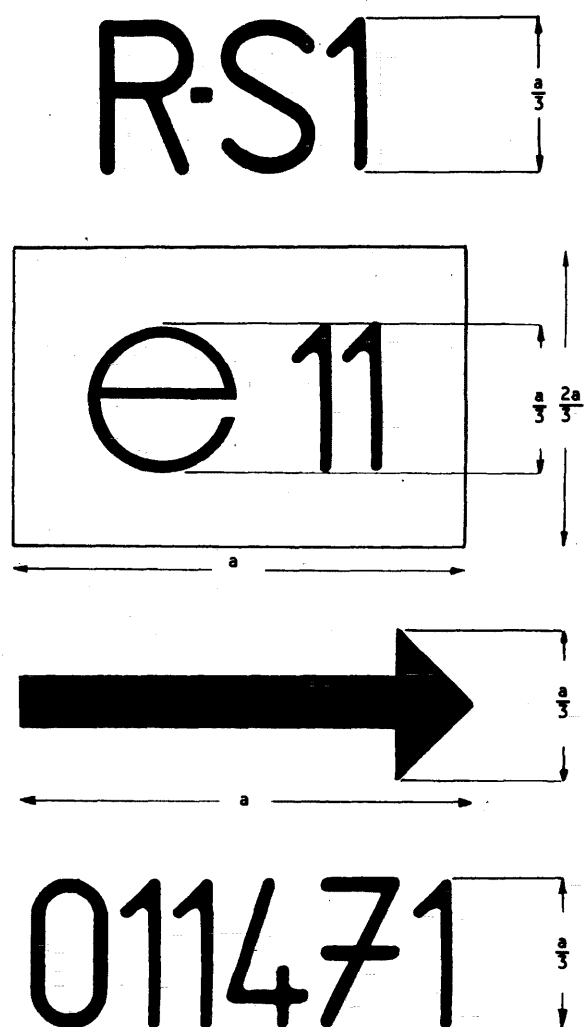


011471



A rectangular box with a height dimension indicated by a vertical arrow on the right side, labeled $\frac{a}{3}$.

The device bearing the EEC type-approval mark shown above is a stop lamp, EEC type-approved in the United Kingdom (e 11) under the number 1471.



The device bearing the EEC type-approval mark shown above is a device comprising both a rear position (side) lamp and a stop lamp, EEC type-approved in the United Kingdom (e 11) under the number 1471. The arrow means that, on the side to which it points, the photometric specifications are satisfied up to an angle of 80°H.'

The following new Appendix 2 is added after Appendix 1:

Appendix 2

EXAMPLES OF SIMPLIFIED MARKING FOR GROUPED, COMBINED OR RECIPROCALLY INCORPORATED LAMPS

Model A

	3333 e 4 →		
	IA 02	2a 01	R 01
	F 00	AR 00	S1 01

Model B

	IA 02 F 00	2a 01 AR 00	R 01 S1 01
		3333 e 4 →	

Model C

IA 02	2a 01	R 01
F 00	AR 00	S1 01

3333

e 4

Note: In the above examples the vertical and horizontal lines indicate the general shape of a lamp unit and do not form part of the component type-approval mark. The three examples of EEC component type-approval marks, models A, B and C represent three possible variants of the marking of a light signalling device when two or more lamps are part of the same device.

The EEC component type-approval mark shows that the device was EEC type-approved in the Netherlands (e 4) under the number 3333 and comprises:

- a reflex-reflector of class 1A EEC component type-approved in conformity with Directive 76/757/EEC;
 - a rear direction indicator lamp of category 2a EEC component type-approved in conformity with Directive 76/759/EEC;
 - a red rear position lamp (R) EEC component type-approved in conformity with the present Directive;
 - a rear fog lamp (F) EEC component type-approved in conformity with Directive 77/538/EEC;
 - a reversing lamp (AR) EEC component type-approved in conformity with Directive 77/539/EEC;
 - a stop lamp (S 1) having one level of intensity EEC component type-approved in conformity with the present Directive.
-

COMMISSION DIRECTIVE

of 1 August 1989

adapting to technical progress Council Directive 76/761/EEC on the approximation of the laws of the Member States relating to motor-vehicle headlamps which function as main-beam and/or dipped-beam headlamps and to incandescent electric filament lamps for such headlamps

(89/517/EEC)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Article 2

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 87/403/EEC ⁽²⁾, and in particular Article 11 thereof,

Having regard to Council Directive 76/761/EEC of 27 July 1976 on the approximation of the laws of the Member States relating to motor-vehicle headlamps which function as main-beam and/or dipped-beam headlamps and to incandescent electric filament lamps for such headlamps ⁽³⁾, as last amended by Directive 87/354/EEC ⁽⁴⁾, and in particular Article 10 thereof,

Whereas, in the light of experience and in view of the current state of the art, certain requirements may now be supplemented and brought more into line with real traffic conditions, thus improving the safety of vehicle occupants 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

The list of Annexes and Annexes I, II, V and VI to Directive 76/761/EEC shall be amended in accordance with the Annex to this Directive. A new Annex VII, also set out in the Annex to this Directive, shall be added.

⁽¹⁾ OJ No L 42, 23. 2. 1970, p. 1.

⁽²⁾ OJ No L 220, 8. 8. 1987, p. 44.

⁽³⁾ OJ No L 262, 27. 9. 1976, p. 96.

⁽⁴⁾ OJ No L 192, 11. 7. 1987, p. 43.

1. With effect from 1 January 1990, no Member State may:

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

— prohibit the entry into service of vehicles,

on grounds relating to the headlamps which function as main-beam and/or dipped-beam headlamps and to the incandescent electric filament lamps for such lamps, hereinafter referred to as 'headlamps' and 'lamps' respectively, if the latter comply with the provisions of this Directive;

- (b) — refuse to grant EEC component type-approval or national component type-approval in respect of the said headlamps and lamps if they comply with the provisions of this Directive, or

— prohibit the placing on the market of headlamps and lamps if the latter bear the EEC component type-approval mark issued in accordance with the provisions of this Directive.

2. With effect from 1 July 1990, Member States:

- (a) — shall not issue the document referred to in the third indent of Article 10 (1) of Directive 70/156/EEC in respect of a type of vehicle of which the headlamps and lamps do not comply with the provisions of this Directive,

— may refuse to grant national type-approval in respect of a type of vehicle of which the said headlamps and lamps do not comply with the provisions of this Directive;

- (b) — shall not grant EEC component type-approval in respect of headlamps and lamps if the latter do not comply with the provisions of this Directive,

— may refuse to grant national component type-approval in respect of the said headlamps and lamps if they do not comply with the provisions of this Directive.

3. With effect from 1 April 1994, Member States may prohibit the entry into service of vehicles of which the

headlamps and lamps do not comply with the provisions of this Directive, and the placing on the market of headlamps and lamps which do not bear the type-approval mark issued in accordance with the provisions of this Directive.

4. By way of derogation from the provisions of 2 (b) above, Member States shall continue to recognize EEC component type-approval granted for a type of headlamp and a type of lamp pursuant to the provisions of Directive 76/761/EEC which are intended to be mounted on vehicles already in service.

Article 3

Member States shall bring into force the provisions necessary to comply with this Directive not later than 31 December 1989. They shall forthwith inform the Commission thereof.

The provisions adopted pursuant to the first subparagraph shall make express reference to this Directive.

Article 4

This Directive is addressed to the Member States.

Done at Brussels, 1 August 1989.

For the Commission
Martin BANGEMANN
Vice-President

ANNEX

The following new Annex VII is added to the List of Annexes, after Annex VI:

'Annex VII — Tests for stability of photometric performance of headlamps in operation'.

Annex I is amended as follows:

Items 1 to 1.1.5 are replaced by the following:

1. DEFINITIONS

1.1 The definitions set out in Directive 76/756/EEC of:

- main-beam headlamp,
 - dipped-beam headlamp,
 - lamp,
 - light source with regard to filament lamps,
 - independent lamps,
 - grouped lamps,
 - combined lamps,
 - reciprocally incorporated lamps,
 - illuminating surface of a lighting device,
 - apparent surface,
 - light-emitting surface,
 - axis of reference,
 - centre of reference,
- shall apply to this Directive.

1.2 Type of lamp

"Type of lamp" means lamps which do not differ in such essential respects as, for example:

- 1.2.1 the trade names or marks;
- 1.2.2 the characteristics of the optical system;
- 1.2.3 the inclusion of additional components capable of altering the optical effects by reflection, refraction or absorption;
- 1.2.4 suitability for right-hand or left-hand traffic or for both traffic systems;
- 1.2.5. ability to provide a dipped beam or a main beam or both.'

In item 5.4, the last paragraph is deleted.

After item 5.4, the following new item 5.5 is added:

'5.5 In order to ensure that the photometric performance of lamps does not change considerably during use, additional tests must be carried out in accordance with the provisions of Annex VII; conformity with the requirements of 5.2 to 5.4 shall be verified visually and, where necessary, by a trial fitting.'

Item 8 is replaced by the following:

8. CONFORMITY OF PRODUCTION

Every headlamp bearing an EEC component type-approval mark shall conform to the approved type and comply with the photometric requirements set out in item 6 above and in item 3 of Annex VII.'

Annex II is amended as follows:

Add the following to item 1:

'C/R, C/R, C/R, C/, C/, C/'

After the item thus amended, the following new items 2 and 3 are added:

- 2. The filament of the dipped-beam headlamp may/may not (*) be lit at the same time as that of the main-beam headlamp and/or another reciprocally incorporated lamp.
- 3. The lamp may be used with (a) filament lamp(s) having a nominal voltage of 6 V, 12 V or 24 V (*)'

Items 2 to 16 are renumbered 4 to 18.

Appendix 4, 3, Notes, is amended as follows:

Item 9 is replaced by the following:

- '9. The contact strips (XIV, XV and XVI) must be placed in the order indicated above and positioned in relation to the positioning lugs either as indicated in the drawing or at an angle of 180° from that position, with a tolerance of $\pm 20^\circ$ in either case.'

Annex VI is amended as follows:

Item 1.2.1.3 is replaced by the following:

- '1.2.1.3 a drawing (three copies), in sufficient detail to permit identification of the type, showing a front view of the lamp with, if necessary, details of the lens moulding, and in cross-section.

The drawing must indicate the limits of the illuminating surface and the position of the EEC component type-approval mark (in particular, the component type-approval number and the category reference(s)).'

After item 2.1.4, the following new item 2.1.5 is added:

- '2.1.5 In all cases, the mode of operation used during the test procedure described in 1.1.1.1 of Annex VII and the voltage(s) permitted in accordance with 1.1.1.2 of Annex VII must be indicated on the EEC component type-approval certificate.

The device must bear the following mark in the appropriate place:

- in the case of headlamps that comply with the provisions of this Directive and are so designed as to preclude any simultaneous lighting of the dipped-beam filament and the filament of any other light source with which it may be reciprocally incorporated, a stroke (/) after the dipped-beam symbol in the component type-approval mark;
- in the case of headlamps that comply with the provisions of Annex VII of this Directive only when under a voltage of 6 V or 12 V, a symbol consisting of the figure 24 marked with a cross (24) affixed near the socket of the filament lamp.'

Item 4.2, read:

- '4.2 This mark shall consist of a rectangle surrounding the letter "e" followed by the distinguishing number or letters of the Member State which has granted type approval:

- 1 for Germany,
- 2 for France,
- 3 for Italy,
- 4 for the Netherlands,
- 6 for Belgium,
- 9 for Spain,
- 11 for the United Kingdom,
- 13 for Luxembourg,
- 18 for Denmark,
- 21 for Portugal,
- EL for Greece,
- IRL for Ireland.

It must also include the EEC component type-approval number which corresponds to the number of the EEC component type-approval certificate issued for the type of headlamp or lamp in question, preceded by one or two figures indicating the serial number assigned to the most recent major technical amendment of Council Directive 76/761/EEC, on the date EEC component type-approval was granted. For the present Directive, the serial number is "2" for filament lamps and "01" for headlamps. That number shall be placed above the rectangle in the case of a headlamp and near the rectangle in the case of a filament lamp.'

After item 4.3.5, the following new item 4.3.6 is added:

- '4.3.6 Marking must, moreover, comply with the provisions of item 2.1.5 of this Annex.'

The Appendix is amended as follows:

After Figure 8, the following new Figures 9 and 10 and their keys are added:

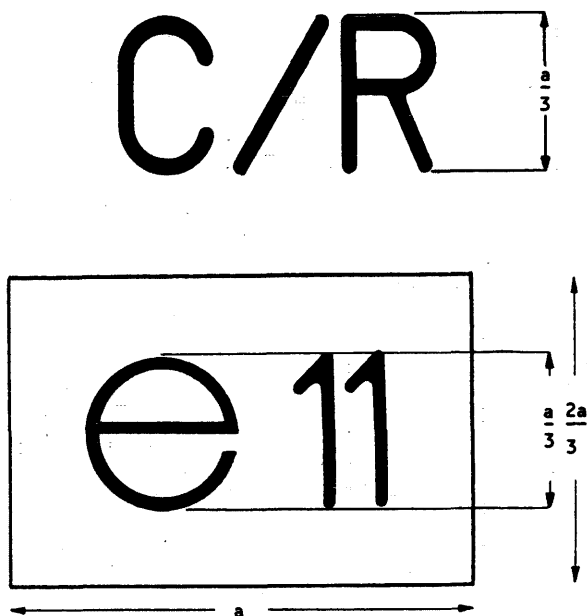


Figure 9

Identification of a headlamp meeting the requirements of this Directive with regard to both the dipped beam and the main beam and designed for right-hand traffic only,

in which the dipped-beam filament cannot be lit at the same time as the main-beam filament of the R 2 filament lamp or that of any other lamp with which it may be reciprocally incorporated.

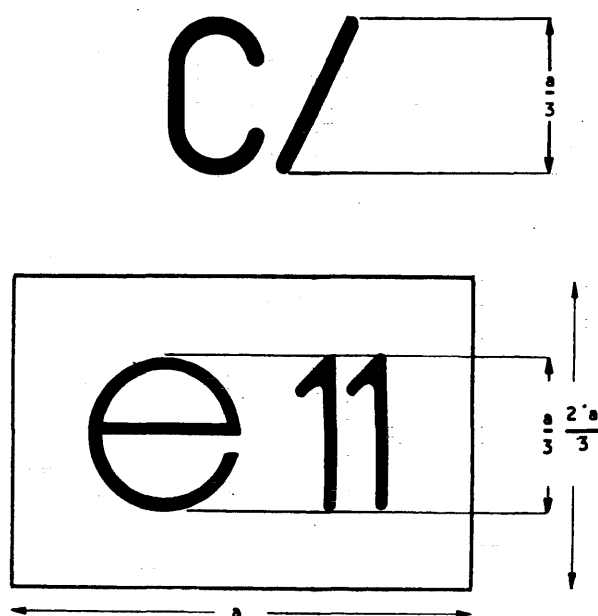


Figure 10

Identification of a headlamp meeting the requirements of this Directive with regard to the dipped beam only and designed for right-hand traffic only,

The following new Annex VII is inserted after Annex VI:

'ANNEX VII

TESTS FOR STABILITY OF PHOTOMETRIC PERFORMANCE OF HEADLAMPS IN OPERATION

Compliance with the requirements of this Annex is not sufficient for the approval of headlamps incorporating lenses of plastic material.

TESTS ON COMPLETE HEADLAMPS

Once the photometric values have been measured in accordance with the provisions of this Directive, at points $E_{\max}$ for the main beam and points HV, 50R, and B50L for the dipped beam, (or HV, 50L, B50R for headlamps designed for driving on the left), a complete headlamp sample shall be tested for stability of photometric performance in operation. The term 'complete headlamp' shall be understood to mean the complete lamp itself including those surrounding body parts and lamps which could influence its thermal dissipation.

1. TEST FOR STABILITY OF PHOTOMETRIC PERFORMANCE

The tests shall be carried out in a dry and still atmosphere at an ambient temperature of $23^{\circ}\text{C} \pm 5^{\circ}\text{C}$, the complete headlamp being mounted on a base representing the correct installation on the vehicle.

1.1 Clean headlamp

The headlamp shall be operated for 12 hours as described in 1.1.1 and checked as prescribed in 1.1.2.

1.1.1 Test procedure

The headlamp shall be operated for the specified time so that:

- 1.1.1.1 (a) in cases where only one light source (main-beam or dipped-beam) is to be type-approved, the corresponding filament is lit for the prescribed time, (**);
- (b) in the case of a reciprocally incorporated main-beam headlamp and dipped-beam headlamp (dual-filament lamp or two filament lamps):
- if the applicant declares that the headlamp is to be used with a single filament lit at a time (*), the test shall be carried out in accordance with this condition, and each specified light source shall be lit (**) for half the time specified in 1.1;
 - in all other cases (*) (**), the headlamp shall be subjected to the following cycle until the time specified is reached:
15 minutes, dipped-beam filament lit,
5 minutes, all filaments lit,
- (c) in the case of grouped light functions (sources), all the individual sources shall be lit simultaneously for the time specified for individual light sources, (a) also taking into account the use of reciprocally incorporated light sources; (b) according to the manufacturer's specifications.

1.1.1.2 Test voltage

The voltage shall be so adjusted as to supply a wattage 15 % higher than the rated wattage specified in the Directive for 6 V or 12 V filament lamps, and 26 % higher than the rated wattage for 24 V filament lamps.

The applied wattage shall in all cases comply with the corresponding value of a filament lamp of 12 V rated voltage, except where the applicant specifies that the headlamp may be used at a different voltage. In the latter case, the test shall be carried out using the filament lamp with the highest wattage that can be used.

(*) Should two or more lamp filaments be simultaneously lit when headlamp flashing is used, this shall not be considered as being normal simultaneous use of the filaments.

(**) When the headlamp tested is grouped or reciprocally incorporated with the position (side) lamps, the latter shall be lit for the duration of the test. If the device comprises a direction indicator lamp, the latter shall be lit in flashing operation mode with an on/off time ratio of one to one.

- 1.1.2 *Test results*
- 1.1.2.1 *Visual inspection*
- Once the headlamp has been stabilized to the ambient temperature, the headlamp lens and the external lens, if any, shall be cleaned with a clean, damp cotton cloth. It shall then be inspected visually; no distortion, deformation, cracking or change in colour of either the headlamp lens or the external lens, if any, shall be noticeable.
- 1.1.2.2 *Photometric test*
- To comply with the requirements of this Directive, the photometric values shall be verified at the following points:
- Dipped-beam:
- 50 R, B 50 L and HV for headlamps made or adjusted for right-hand traffic;
50 L, B 50 R and HV for headlamps made or adjusted for left-hand traffic;
- Main-beam:
- Point of $E_{\max}$
- A further adjustment may be made to allow for any deformation of the headlamp base due to heat (for the setting of the cut-off line, see section 2).
- A 10 % discrepancy, including the tolerances of photometric measurement procedure is permissible between the photometric characteristics and the values measured prior to the test.
- 1.2 *Dirty headlamp*
- After being tested as provided in 1.1 above, the headlamp shall be prepared as prescribed in 1.2.1 then operated for one hour as described in 1.1.1 and checked as prescribed in 1.1.2.
- 1.2.1 *Preparation of the headlamp*
- 1.2.1.1 *Text mixture*
- The mixture of water and a polluting agent to be applied to the headlamp shall be composed of nine parts (by weight) of silica sand with a grain size of between 0 and 100 μm , one part (by weight) of powdered vegetable charcoal with a grain size of between 0 and 100 μm , 0,2 part (by weight) of NaCMC and an appropriate quantity of distilled water, the conductivity of which is lower than 1 mS/m.
- The mixture must not be more than 14 days old.
- 1.2.1.2 *Application of the test mixture to the headlamp*
- The test mixture shall be uniformly applied to the entire light emitting surface of the headlamp and then left to dry. This procedure shall be repeated until the illumination value has dropped to 15 to 20 % of the values measured for each of the following points in the conditions described in this Annex.
- Point of $E_{\max}$ in main-beam photometric distribution for a main-beam/dipped-beam headlamp;
- Point of $E_{\max}$ in main-beam photometric distribution for a dipped-beam headlamp only,
50 R and 50 V (*) for a dipped-beam headlamp only, made or adjusted for right-hand traffic;
50 L and 50 V (*) for a dipped-beam headlamp only, made or adjusted for left-hand traffic.
- 1.2.1.3 *Measuring equipment*
- The measuring equipment shall be equivalent to that used during headlamp component type-approval tests. A standard (reference) filament lamp shall be used for the photometric verifications.
2. *TEST FOR CHANGE IN VERTICAL POSITION OF THE CUT-OFF LINE UNDER THE INFLUENCE OF HEAT*
- This test consists in verifying that the vertical drift under the influence of heat of the cut-off line of a dipped-beam headlamp in operation does not exceed a specified value.
- After having been tested in accordance with section 1, the headlamp shall be subjected to the test described in 2.1, without being removed from or readjusted in relation to its test fixture.

(*) 50 V is situated 375 mm below H on the vertical line V-V where the screen is at a distance of 25 m.

2.1 Test

The test shall be carried out in a dry and still atmosphere at an ambient temperature of $23^{\circ}\text{C} \pm 5^{\circ}\text{C}$.

Using a mass-production filament lamp that has been aged for at least one hour, the headlamp shall be operated on dipped beam without being dismantled from or readjusted in relation to its test fixture. (For the purpose of this test, the voltage shall be adjusted as specified in 1.1.1.2. The position of the horizontal part of the cut-off line (between vv and the vertical line passing through point B 50 L for right-hand traffic or B 50 R for left-hand traffic) shall be verified 3 minutes (r_3) and 60 minutes (r_{60}) respectively after operation.

The measurement of the change in the cut-off line position as described above shall be carried out by any method giving acceptable accuracy and reproducible results.

2.2. Test results

2.2.1. The result expressed in milliradians (mrad) shall be deemed acceptable for a dipped-beam headlamp only where the absolute value

$\Delta r_I = [r_3 - r_{60}]$ recorded on the headlamp is not more than 1,0 mrad ($\Delta r_I \leq 1,0 \text{ mrad}$).

2.2.2. However, if that value is more than 1,0 mrad, but not more than 1,5 mrad ($1,0 \text{ mrad} < \Delta r_I \leq 1,5 \text{ mrad}$), a second headlamp shall be tested as described in 2.1 after being subjected three times in succession to the cycle described below, in order to stabilize the position of the mechanical parts of the headlamp on a base representative of the correct installation on the vehicle:

— operation of the dipped-beam headlamp for one hour (the voltage being adjusted as specified in 1.1.1.2),

— period of rest for one hour.

The headlamp type shall be deemed acceptable if the mean value of the absolute values Δr_I measured on the first sample and Δr_{II} on the second sample is not more than 1,0 mrad

$$\left(\frac{\Delta r_I + \Delta r_{II}}{2} \leq 1,0 \text{ mrad} \right)$$

3. CONFORMITY OF PRODUCTION

One of the sampled headlamps shall be tested according to the procedure described in 2.1 after being subjected three times in succession to the cycle described in 2.2.2.

The headlamp shall be deemed acceptable if Δr does not exceed 1,5 mrad.

If that value is worse than 1,5 mrad, but not more than 2,0 mrad, a second headlamp shall be subjected to the test, after which the mean of the absolute values recorded on both samples shall not exceed 1,5 mrad.

COMMISSION DIRECTIVE

of 1 August 1989

adapting to technical progress Council Directive 77/538/EEC on the approximation of the laws of the Member States relating to rear fog lamps for motor vehicles and their trailers

(89/518/EEC)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Directive 70/156/EE 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 87/403/EEC⁽²⁾, and in particular Article 11 thereof,Having regard to Council Directive 77/538/EEC of 28 June 1977 on the approximation of the laws of the Member States relating to rear fog lamps for motor vehicles and their trailers⁽³⁾, as last amended by Directive 87/354/EEC⁽⁴⁾, and in particular Article 10 thereof,

Whereas, in the light of experience and in view of the current state of the art, it is now possible to simplify the EEC component type-approval mark for such lamps, where they are grouped together with, combined with or reciprocally incorporated in other lamps;

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 0 and II to Directive 77/538/EEC are amended in accordance with the Annex to this Directive.

Article 2

With effect from 1 January 1990, no Member State may:

- (a) — refuse, in respect of a type of vehicle, to grant EEC type-approval, to issue the document referred to in the third indent of Article 10(1) of Directive 70/156/EEC, or to grant national type-approval, or
- prohibit the entry into service of vehicles, on grounds relating to the rear fog lamps if the latter comply with the provisions of this Directive;
- (b) — refuse to grant EEC component type-approval or national component type-approval in respect of the said rear fog lamps if they comply with the provisions of this Directive, or
- prohibit the placing on the market of rear fog lamps if the latter bear the EEC component type-approval mark issued in accordance with the provisions of this Directive.

Article 3

Member States shall bring into force the provisions necessary to comply with this Directive not later than 31 December 1989. They shall forthwith inform the Commission thereof.

The provisions adopted pursuant to the first subparagraph shall make express reference to this Directive.

Article 4

This Directive is addressed to the Member States.

Done at Brussels, 1 August 1989.

For the Commission
Martin BANGEMANN
Vice-President

⁽¹⁾ OJ No L 42, 23. 2. 1970, p. 1.⁽²⁾ OJ No L 220, 8. 8. 1987, p. 44.⁽³⁾ OJ No L 220, 29. 8. 1977, p. 60.⁽⁴⁾ OJ No L 192, 11. 7. 1987, p. 43.

ANNEX

Annex 0 is amended as follows:

Items 1 to 1.4 are replaced by the following items 1 and 1.1:

'1. DEFINITIONS

1.1 The definitions set out in Directive 76/756/EEC of:

- rear fog lamp,
 - lamp,
 - light source with regard to filament lamps,
 - independent lamps,
 - grouped lamps,
 - combined lamps,
 - reciprocally incorporated lamps,
 - device,
 - illuminating surface of a signalling lamp other than a reflex reflector,
 - apparent surface,
 - light-emitting surface,
 - axis of reference,
 - centre of reference
 - single lamp,
- shall apply to this Directive.'

Items 1.5 to 1.5.4 are renumbered 1.2 to 1.2.4.

Item 3.4 is replaced by the following:

'3.4 The illuminating surface of the lamp shall not exceed 140 cm².'

Item 6, '2854 K' is replaced by '2856 K'

In the German version the abbreviation for the International Commission on Illumination should read: 'CIE'.

Annex II is amended as follows:

Item 1.2.2 is replaced by the following:

'1.2.2. drawings (three copies) in sufficient detail to permit identification of the type and showing, geometrically, the position in which the device is to be mounted on the vehicle, the axis of observation to be taken as the axis of reference in the tests (horizontal angle $H = 0^\circ$, vertical angle $V = 0^\circ$), the point to be taken as the centre of reference in the said tests, the vertical and horizontal tangents to the illuminating surface and their distance from the centre of reference of the lamp.'

In item 3.3 'mark' should be replaced by 'number'

Item 4.2 is replaced by the following:

'4.2 This mark shall consist of a rectangle surrounding the letter "e" followed by the distinguishing number or letters of the Member State which has granted type-approval:

- 1 for Germany,
- 2 for France,
- 3 for Italy,
- 4 for the Netherlands
- 6 for Belgium,
- 9 for Spain,
- 11 for the United Kingdom,
- 13 for Luxembourg,
- 18 for Denmark,
- 21 for Portugal,
- EL for Greece,
- IRL for Ireland

It must also include the EEC component type-approval number which corresponds to the number of the EEC component type-approval certificate issued for the type of rear fog lamp in question (see Annex I), preceded by two figures indicating the sequence number assigned to the most recent major technical amendment of Council Directive 77/538/EEC, on the date when EEC component type-approval was delivered. For the present Directive, this number is '00'.

Item 4.6, read:

'4.6 Examples of the EEC component type-approval mark and the additional symbol are shown in Appendix 1.

The component type approval numbers in accordance with appendix 1 to Directive 77/538/EEC, in its initial form i.e. without sequence number "00", may however be retained provided that the technical requirements relating to rear fog lamps are not modified.'

Item 4.7, read:

'4.7 Where a single EEC component type-approval number is issued, as under 3.3, for a type of lighting and light-signalling device comprising a rear fog lamp that is grouped, combined or reciprocally incorporated with other lamps, one EEC component type-approval mark only may be affixed, consisting of:

- a rectangle surrounding the letter "e" followed by the distinguishing number or letters of the Member State which has granted the type approval,
- the EEC component type-approval number and, where necessary, the appropriate arrow.'

After item 4.7, the following new items are added:

'4.7.1 This EEC component type-approval mark may be placed anywhere on lamps that are grouped, combined or reciprocally incorporated, provided that:

4.7.1.1 it is visible when the lamps have been installed;

4.7.1.2 no light-transmitting component of the grouped, combined or reciprocally incorporated lamps may be removed without the EEC component type-approval mark being removed at the same time.

4.7.2 The identification symbol for each lamp corresponding to each Directive pursuant to which EEC component type-approval was granted, together with the two figures indicating the sequence number referred to in item 4.2, final indent, above and, where necessary, the additional letter "D" must be indicated;

4.7.2.1 either on the appropriate light-emitting surface;

4.7.2.2 or in a group, such that each of the grouped, combined or reciprocally incorporated lamps may be clearly identified.'

Item 4.9 is replaced by the following.

'4.9 Examples of an EEC component type-approval mark for a lamp that is grouped, combined or reciprocally incorporated with other lamps are shown in Appendix 2.'

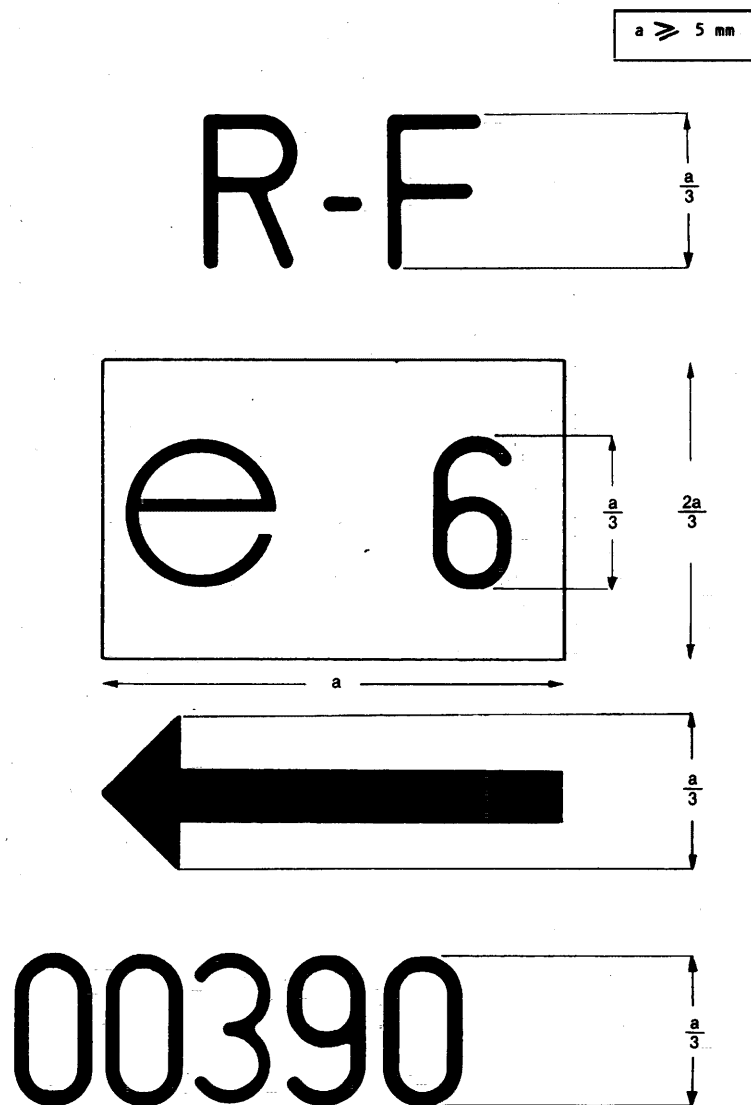
Appendix 1 is amended as follows.

In the title, the word 'Example' is replaced by the word 'Examples'.

Above the example of an EEC component type-approval mark, the words 'Figure 1' are inserted.

After Figure 1, the following Figure 2 is added:

Figure 2



The device bearing the EEC component type-approval mark shown above is a device comprising a rear position (side) lamp and a rear fog lamp, EEC type-approved in Belgium (e 6) under the number 00390.

The arrow indicates the side on which the photometric measurements on the rear position (side) lamp are satisfied up to an angle of 80°H .

Appendix 2 is replaced by the following:

Appendix 2

EXAMPLES OF SIMPLIFIED MARKING FOR GROUPED, COMBINED OR RECIPROCALLY INCORPORATED LAMPS

Model A

	3333 e 4 		
	IA 02	2a 01	R 01
	F 00	AR 00	S1 01

Model B

	IA 02 F 00	2a 01 AR 00	R 01 S1 01
		3333 e 4 	

Model C

IA 02	2a 01	R 01			
F 00	AR 00	S1 01			

3333

e 4

Note: In the above examples the vertical and horizontal lines depict the general shape of a lamp unit and do not form part of the component type-approval mark.

The three examples of EEC component type-approval marks shown above, namely models A, B and C, represent three possible alternatives for marking a lighting device where two or more lamps form part of the same set of grouped, combined or reciprocally incorporated lamps. They show that the device in question was EEC type-approved in the Netherlands (e 4) under the number 3333 and consists of:

- a class IA reflex reflector, EEC type-approved pursuant to Directive 76/757/EEC;
 - a category 2a rear direction indicator, EEC type-approved pursuant to Directive 76/759/EEC;
 - a red rear position (side) lamp (R), EEC type-approved pursuant to Directive 76/758/EEC;
 - a rear fog lamp (F), EEC type-approved pursuant to this Directive;
 - a reversing lamp (AR), EEC type-approved pursuant to Directive 77/539/EEC;
 - a stop lamp (S1), EEC type-approved pursuant to Directive 76/758/EEC.
-

COMMISSION DIRECTIVE

of 1 August 1989

supplementing and amending Directive 77/535/EEC on the approximation of the laws of Member States relating to methods of sampling and analysis for fertilizers

(89/519/EEC)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Directive 76/116/EEC of 18 December 1975 on the approximation of the laws of the Member States relating to fertilizers ⁽¹⁾, as last amended by Council Directive 89/284/EEC ⁽²⁾, and in particular Article 9 (2) thereof,

Whereas Directive 89/284/EEC supplements and amends Directive 76/116/EEC in respect of the calcium, magnesium, sodium and sulphur content of fertilizers;

Whereas Commission Directive 77/535/EEC ⁽³⁾, as last amended by Directive 87/566/EEC ⁽⁴⁾, provides for official controls for EEC fertilizers for the purpose of checking compliance with the requirements imposed by the Community provisions concerning the quality and composition of fertilizers; whereas that Directive should be supplemented so that fertilizers to which Council Directive 89/284/EEC relates can also be checked; whereas it is necessary to replace methods 5.1, 5.2, 5.3 and 5.4; whereas general provisions relating to methods for analysing fertilizers applicable to all methods should be added to the general observations of Annex II;

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

HAD ADOPTED THIS DIRECTIVE:

Article 1

1. General provisions relating to methods for analysing fertilizers of Annex I to this Directive are added to the general observations of Annex II to Directive 77/535/EEC.

2. The methods 8.1 to 8.10 in Annex II to this Directive are added to Annex II to Directive 77/535/EEC.

3. Methods 5.1 to 5.4 set out in Annex II to Directive 77/535/EEC are deleted.

Article 2

1. Member States shall take measures necessary to comply with this Directive by 1 September 1990 and shall forthwith inform the Commission thereof.

The provisions adopted pursuant to the first indent shall make express reference to this Directive.

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

Article 3

This Directive is addressed to the Member States.

Done at Brussels, 1 August 1989

For the Commission
Martin BANGEMANN
Vice-President

⁽¹⁾ OJ No L 24, 30. 1. 1976, p. 21.

⁽²⁾ OJ No L 111, 22. 4. 1989, p. 34.

⁽³⁾ OJ No L 213, 22. 8. 1977, p. 1.

⁽⁴⁾ OJ No L 342, 4. 12. 1987, p. 32.

ANNEX I

'GENERAL PROVISIONS RELATING TO METHODS OF ANALYSING FERTILIZERS**1. Reagents**

Unless otherwise specified in the method of analysing, all of the reagents must be analysis-pure (a. p.). Where trace elements are to be analysed the purity of the reagents must be checked by means of a blank test. Depending upon the result obtained, it might be necessary to conduct a further purification.

2. Water

The dissolution, dilution, rinsing or washing operations mentioned in the methods of analysis without stating precisely what the nature of the solvent or diluent is, imply the use of water. Normally, the water will have to be demineralized or distilled. In these specific instances, as mentioned in the method of analysis, that water will have to be subjected to specific purification processes.

3. Laboratory equipment

In view of the equipment normally used in inspection laboratories, the apparatus described in the methods of analysis is restricted to special instruments and apparatus or to such demanded by any specific requirements. This equipment must be perfectly clean, above all where small quantities are to be determined. The laboratory will have to ensure the accuracy of any graduated glassware used by referring to the standards.

ANNEX II

Methods 8

SECONDARY NUTRIENTS

Method 8.1

EXTRACTION OF TOTAL CALCIUM, TOTAL MAGNESIUM, TOTAL SODIUM AND TOTAL SULPHUR
IN THE FORM OF SULPHATES

1. SCOPE

This document defines the procedure for extracting total calcium, total magnesium and total sodium, and for extracting the total sulphur present in the form of sulphates, so that the same extract may be used for the determination of each nutrient required.

2. FIELD OF APPLICATION

This method applies to EEC fertilizers, for which a declaration of the total calcium, total magnesium, total sodium, and total sulphur in the form of sulphates is provided for in Council Directive 89/284/EEC ⁽¹⁾.

3. PRINCIPLE

Solubilization by boiling in dilute hydrochloric acid.

4. REAGENTS

4.1 Diluted hydrochloric acid:

One volume of hydrochloric acid ($d = 1,18$) plus one volume of water.

5. APPARATUS

Electric hot plate with adjustable temperature.

6. PREPARATION OF THE SAMPLE

See method 1 in Commission Directive 77/535/EEC.

7. PROCEDURE

7.1 Test sample

Calcium, magnesium, sodium and sulphur in the form of sulphates are extracted from a test sample of five grams weighed to within one milligram.

However, when the fertilizer contains more than 15 % of sulphur (S) i. e. 37,5 % SO_3 , and more than 18,8 % of calcium (Ca) i. e. 26,3 % CaO , the extraction of calcium and sulphur is carried out on a test sample of one gram, weighed to within one milligram. Place the test sample in a 600 millilitre beaker.

7.2 Preparation of the solution

Add approximately 400 millilitres of water and, taking care when the sample contains a significant quantity of carbonates, 50 millilitres of dilute hydrochloric acid (4.1) a small amount at a time. Bring to the boil and maintain for 30 minutes. Allow to cool, stirring occasionally. Decant

⁽¹⁾ OJ No L 111, 22. 4. 1989, p. 34.

quantitatively into a 500 millilitre graduated flask. Make up to volume with water, and mix. Pass through a dry filter into a dry container, discarding the initial portion. The extract must be completely transparent. Stopper if the filtrate is not used immediately.

Method 8.2

EXTRACTION OF TOTAL SULPHUR PRESENT IN VARIOUS FORMS

1. SCOPE

This document defines the procedure for extracting the total sulphur contained in fertilizers in elemental form and/or in other chemical combinations.

2. FIELD OF APPLICATION

This method applies to EEC fertilizers for which a declaration of the total sulphur present in various forms (elemental, thiosulphate, sulphite, sulphate) is provided for in the Council Directive 89/284/EEC.

3. PRINCIPLE

Elemental sulphur is converted in an alkaline medium into polysulphides and thiosulphate; these, together with any sulphites which may be present, are then oxidized with hydrogen peroxide. The various forms of sulphur are thus converted into sulphate which is determined by precipitation of barium sulphate (method 8.9).

4. REAGENTS

4.1 Diluted hydrochloric acid:

One volume of hydrochloric acid ($d = 1,18$) plus one volume of water.

4.2 Sodium hydroxide solution, NaOH, 30 % minimum ($d = 1,33$).

4.3 Hydrogen peroxide solution, 30 % w/w.

4.4 Aqueous solution of barium chloride $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$, 122 gram per litre.

5. APPARATUS

Electric hot plate with adjustable temperature.

6. PREPARATION OF THE SAMPLE

See method 1.

7. PROCEDURE

7.1 Test sample

Weigh out to within one milligram a quantity of fertilizer containing between 80 and 350 milligrams of sulphur (S) or 200 and 875 milligrams of SO_3 .

As a rule (where $\text{S} < 15\%$), weigh out 2,5 grams. Place the test sample in a 400 millilitre beaker.

7.2 Oxidation.

Add 20 millilitres of sodium hydroxide solution (4.2) and 20 millilitres of water. Cover with a watch glass. Boil for five minutes on the hot plate (5.1). Remove from the hot plate. Using a jet of hot water, collect the sulphur sticking to the sides of the beaker and boil for 20 minutes. Leave to cool.

Add 2 millilitres increments of hydrogen peroxide (4.3) until no reaction is observed. Six to eight of hydrogen peroxide will be necessary. Allow oxidation to continue for one hour, then bring to the boil for half an hour. Leave to cool.

7.3 Preparation of the solution to be analysed.

Add approximately 50 millilitres of water and 50 millilitres of the hydrochloric acid solution (4.1).

— *If the level of sulphur (S) is less than 5 %:*

filter into a 600 millilitre beaker. Wash the residue on the filter several times with cold water. After washing, check for the absence of sulphate in the last drops of the filtrate using a barium chloride solution (4.4). The filtrate must be perfectly clear. Sulphate is determined on the whole of the filtrate in accordance with method 8.9.

— *If the level of sulphur (S) is at above 5 %:*

transfer quantitatively into a 250 millilitre volumetric flask, make up to volume with water and mix. Filter through a dry filter into a dry container; the filtrate must be completely clear. Stopper if the solution is not to be used immediately. Determine sulphates on an aliquot of this solution by precipitation in the form of barium sulphate (method 8.9).

Method 8.3

EXTRACTION OF WATER-SOLUBLE CALCIUM, MAGNESIUM, SODIUM AND SULPHUR (IN THE FORM OF SULPHATES)

1. SCOPE

This document defines the procedure for extracting water-soluble calcium, magnesium, sodium and sulphur (in the form of sulphates), so that the same extract can be used to determine each nutrient required.

2. FIELD OF APPLICATION

This method applies solely to fertilizers for which a declaration of the water soluble calcium, magnesium, sodium and sulphur (in the form of the sulphates) is provided for in Council Directive 89/284/EEC.

3. PRINCIPLE

The nutrients are solubilized in boiling water.

4. REAGENTS

Distilled or demineralized water of equivalent quality.

5. APPARATUS

Electric hot plate with adjustable temperature.

6. PREPARATION OF THE SAMPLE

See method 1.

7. PROCEDURE

7.1 Test sample.

(a) Where fertilizers contain no sulphur or where they contain, at the same time, no more than 3 % of the sulphur (S) i. e. 7,5 % SO_3 and no more than 4 % of calcium (Ca) i. e. 5,6 % CaO , weigh out five grams of fertilizer to within one milligram.

- (b) Where fertilizers contain more than 3 % of sulphur (S) and more than 4 % of calcium (Ca), weigh out one gram of fertilizer to within one milligram.

Place the test sample in a 600 millilitre beaker.

7.2

PREPARATION OF THE SOLUTION.

Add approximately 400 millilitres of water and boil for 30 minutes. Allow to cool, stirring occasionally, and decant quantitatively into a 500 millilitre graduated flask. Make up to volume with water and mix.

Filter through a dry filter into a dry container. Discard the initial portions of the filtrate. The filtrate must be completely transparent.

Stopper if the solution is not to be used immediately.

Method 8.4

EXTRACTION OF WATER-SOLUBLE SULPHUR WHERE THE SULPHUR IS IN VARIOUS FORMS

1. SCOPE

This document defines the procedure for extracting the water-soluble sulphur contained in fertilizers in various forms.

2. FIELD OF APPLICATION

This method applies to fertilizers for which a declaration of the water-soluble sulphur trioxide is provided for in Directive 89/284/EEC.

3. PRINCIPLE

The sulphur is dissolved in cold water and converted into sulphate by oxidation with hydrogen peroxide in an alkaline medium.

4. REAGENTS

4.1 Diluted hydrochloric acid:

One volume of hydrochloric acid ($d = 1,18$) plus one volume of water.

4.2 Sodium hydroxide solution containing at least 30 % NaOH ($d = 1,33$).

4.3 Hydrogen peroxide solution, 30 % w/w.

5. APPARATUS

5.1 500 millilitre graduated Stohmann flask.

5.2 Rotary shaker, 30 to 40 turns per minute.

5.3 Electric hot plate with adjustable temperature.

6. PREPARATION OF THE SAMPLE

See method 1.

7. PROCEDURE

7.1 Test sample

- (a) Where fertilizers contain a maximum of 3 % of sulphur (S) i. e. 7,5 % SO_3 , together with a maximum of 4 % of calcium (Ca) i. e. 5,6 % CaO , weigh out 5 grams of fertilizer to within one milligram.

- (b) Where fertilizers contain more than 3 % of sulphur (S) together with more than 4 % of calcium (Ca), weigh out one gram of fertilizer to within one milligram.

Place the test sample in a 500 millilitre flask (5.1).

7.2 Preparation of the solution

Add approximately 400 millilitres of water. Stopper. Shake (5.2) for 30 minutes. Make up to volume with water and mix. Pass through a dry filter into a dry container. Stopper if the solution is not to be used immediately.

7.3 Oxidation of the aliquot portion to be analysed

Take an aliquot portion of the extraction solution not exceeding 50 millilitre and, if possible, containing between 20 and 100 milligrams of sulphur (S).

Make up the volume to 50 millilitres with water, if necessary. Add three millilitres of sodium hydroxide solution (4.2) and two millilitres of hydrogen peroxide solution (4.3). Cover with a watch glass and boil gently for one hour on the hot plate (5.3). Keep adding one millilitre increments of hydrogen peroxide solution for as long as the reaction continues (maximum quantity five millilitres).

Then leave to cool. Remove the watch glass and wash the underside into the beaker. Add approximately 20 millilitres of dilute hydrochloric acid (4.1). Make up to approximately 300 millilitres with water.

Determine the content of sulphates on the whole of the oxidized solution in accordance with method 8.9.

Method 8.5

EXTRACTION AND DETERMINATION OF ELEMENTAL SULPHUR

WARNING

This method of analysis involves the use of carbon disulphide (CS₂). Special safety measures must therefore be taken, in particular with regard to:

- the storage of CS₂,
- protective equipment for staff,
- occupational hygiene,
- prevention of fires and explosions,
- disposal of the reagent.

This method requires a highly skilled staff and a suitably equipped laboratory.

1. SCOPE

This defines the procedure for extracting and determining the elemental sulphur contained in fertilizers.

2. FIELD OF APPLICATION

This method applies to EEC fertilizers for which a declaration of the total sulphur in elemental form is provided for in Council Directive 89/284/EEC.

3. PRINCIPLE

After the removal of soluble compounds, elemental sulphur is extracted by using carbon disulphide, followed by gravimetric determination of the sulphur extracted.

4. REAGENTS

Carbon disulphide.

5. APPARATUS

5.1 100 millilitre extraction flask with ground glass stopper.

5.2 Soxhlet apparatus, with the appropriate filter elements.

- 5.3 Vacuum rotary evaporator.
- 5.4 Electric oven, fan assisted, set at 90 ± 2 °C.
- 5.5 Porcelain petri dishes, five to seven centimetres in diameter, not exceeding five centimetres in height.
- 5.6 Electric hot plate with adjustable temperature.

6. PREPARATION OF THE SAMPLE

See method 1.

7. PROCEDURE

7.1 Test sample

Weigh out five to ten grams of the sample to an accuracy of one milligram and place in the thimble of the Soxhlet apparatus (5.2).

7.2 Extraction of the sulphur

Wash thoroughly the contents with hot water to remove all soluble compounds. Dry in the oven at 90 °C (5.4) for at least one hour. Place the filter in the Soxhlet apparatus (5.2).

Place a few glass beads in the flask of the apparatus (5.1) and weigh (P_0), then add 50 millilitres of carbon disulphide (4.1).

Connect the apparatus and leave the elemental sulphur to be extracted for six hours. Switch off the heat and, after cooling, disconnect the flask. Connect the flask to the rotary evaporator (5.3) and evaporate until the contents of the flask have solidified in a spongy mass.

Dry the flask in the oven at 90 °C (5.4) (generally one hour is necessary) until a constant weight is obtained (P_1).

7.3 Determination of the purity of the elemental sulphur

Certain substances may have been extracted by the carbon disulphide at the same time as the elemental sulphur. The purity of the elemental sulphur is determined as follows:

homogenize the contents of the flask as thoroughly as possible, and remove two to three grams, weighed to an accuracy of one milligram (n). Place in the Petri dish (5.5). Weigh dish and contents together (P_2). Place on the hot plate (5.6) set at a temperature not exceeding 220 °C so as not to cause combustion of the sulphur. Continue sublimation for three or four hours until a constant weight is obtained (P_3).

NB: For some fertilizers, it may not be necessary to determine how pure the sulphur is. In this case, omit step 7.2.

8. EXPRESSION OF RESULTS

The percentage elemental sulphur (S) content of the fertilizer is as follows:

$$\text{Impure S (\% of the fertilizer)} = \frac{P_1 - P_0}{m} \times 100$$

$$\text{Purity of sulphur extracted (\%)} = \frac{P_2 - P_3}{n} \times 100$$

$$\text{Pure S (\% of the fertilizer)} = \frac{(P_1 - P_0)(P_2 - P_3)}{m \times n} \times 100$$

Where:

m = the mass of the test sample of fertilizer in grams,

P_0 = the mass of the Soxhlet flask in grams,

P_1 = the mass of the Soxhlet flask and the impure sulphur after drying,

n = the mass of the impure sulphur to be purified in grams,

P_2 = the mass of the Petri dish,

P_3 = the mass of the Petri dish after sublimation of the sulphuring.

Method 8.6

MANGANIMETRIC DETERMINATION OF EXTRACTED CALCIUM FOLLOWING PRECIPITATION IN THE FORM OF OXALATE

1. SCOPE

This document defines the procedure for determining the calcium in fertilizer extracts.

2. FIELD OF APPLICATION

This method applies to EEC fertilizers for which a declaration of the total and/or water-soluble calcium is provided for in Council Directive 89/284/EEC.

3. PRINCIPLE

Precipitation of the calcium contained in an aliquot of the extraction solution in the form of an oxalate, which is determined by titration using potassium permanganate.

4. REAGENTS

4.1 Diluted hydrochloric acid:

One volume of hydrochloric acid ($d = 1,18$) and one volume of water.

4.2 1:10 dilute sulphuric acid:

one volume of sulphuric acid ($d = 1,84$) in ten volumes of water.

4.3 1:1 dilute ammonia solution:

one volume of ammonia ($d = 0,88$) and one volume of water.

4.4 Saturated solution of ammonia oxalate ($(\text{NH}_4)_2 \text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$) at ambient temperature (approximately 40 grams per litre).

4.5 Citric acid solution, 30 % (m/v).

4.6 Ammonium chloride solution, 5 % (m/v).

4.7 Solution of bromothymol blue in ethanol, at 95 %, 0,1 % (m/v).

4.8 Solution of bromocresol green in ethanol, at 95 % 0,04 % (m/v).

4.9 Standard solution of potassium permanganate, 0,02 M.

5. APPARATUS

5.1 Filter crucible with 5 to 20 μ porosity sintered glass.

5.2 Hot water bath.

6. PREPARATION OF THE ALIQUOT TO BE ANALYSED

Using a pipette, take an aliquot portion of the extraction solution obtained by method 8.1 or 8.3, containing between 15 and 50 milligrams of Ca (= 21 to 70 milligrams of CaO). Let the volume of this aliquot be v_2 . Pour into a 400 millilitre beaker. If necessary, neutralize (turning of indicator (4.7) from green to blue) with a few drops of the ammonia solution (4.3).

Add one millilitre of the citric acid solution (4.5) and five millilitres of ammonium chloride solution (4.6).

7. PRECIPITATION OF THE CALCIUM OXALATE

Add approximately 100 millilitres of water. Bring to the boil, add eight to 10 drops of indicator solution (4.8) and, slowly, 50 millilitres of a hot ammonium oxalate solution (4.4). If a precipitate forms, dissolve by adding a few drops of hydrochloric acid (4.1). Neutralize very slowly with

ammonia solution (4.3) while stirring continuously to a pH of 4.4 to 4.6 (turning of indicator (4.8) from green to blue). Place the beaker in a boiling hot water bath (5.2) for approximately 30 minutes.

Remove the beaker from the bath, leave standing for an hour and filter into the crucible (5.1).

8. TITRATION OF THE OXALATE PRECIPITATE

Wash the beaker and crucible until the excess ammonium oxalate has been completely removed (this can be checked by the absence of chloride in the washing water). Place the crucible in the 400 millilitre beaker and dissolve the precipitate with 50 millilitres of hot sulphuric acid (4.2). Add water to the beaker in order to obtain a volume of approximately 100 millilitres. Bring to a temperature of 70 to 80 °C and titrate drop by drop with a permanganate solution (4.9) until the pink colour lasts for a minute. Let this volume be n .

9. EXPRESSION OF RESULTS

The calcium (Ca) content of the fertilizer is as follows:

$$\text{Ca} (\%) = n \times 0,2004 \times \frac{t}{0,02} \times \frac{v_1}{v_2 \times m}$$

Where:

n = the number of millilitres of permanganate used,

m = the mass of the test sample in grams,

v_2 = the aliquot volume in millilitres,

v_1 = the volume of the extraction solution in millilitres,

t = the molarity of the permanganate solution in moles per litre.

$$\text{CaO} (\%) = \text{Ca} (\%) \times 1,400.$$

Method 8.7

DETERMINATION OF MAGNESIUM BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

1. SCOPE

This document defines the procedure for determining the magnesium in fertilizer extracts.

2. FIELD OF APPLICATION

This method applies to EEC fertilizer extracts obtained by methods 8.1 and 8.3, for which a declaration of the total magnesium and/or water soluble magnesium is required, with the following exceptions:

fertilizers listed in the Annex to the Directive on secondary nutrients (89/284/EEC):

type 4 (kieserite),

type 5 (magnesium sulphate) and

type 7 (kieserite with potassium sulphate)

to which method 8.8 applies.

The method set out below applies to all fertilizer extracts containing elements in quantities that might interfere with the complexometric determination of magnesium.

3. PRINCIPLE

Determination of magnesium by atomic absorption spectrophotometry after appropriate dilution of the extract.

4. REAGENTS

4.1 Hydrochloric acid, 1 M solution

4.2 Hydrochloric acid, 0,5 M solution

4.3 Standard solution of magnesium, 1,00 milligrams per millilitre.

4.3.1 Dissolve 1,013 grams of magnesium sulphate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) in the 0,5 M hydrochloric acid solution (4.2).

- 4.3.2. Weigh out 1,658 grams of magnesium oxide (MgO), previously calcined to remove all traces of carbonation. Place in a beaker with 100 millilitres of water and 120 millilitres of 1 M hydrochloric acid (4.1). When it has dissolved, decant quantitatively into a 1 000 millilitre graduated flask. Make up the volume by adding and mix.

or:

- 4.3.3. Commercial standard solution.

The laboratory is responsible for testing such solutions.

- 4.4. Strontium chloride solution.

Dissolve 75 grams of strontium chloride ($\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$) in a hydrochloric acid solution (4.2) and make up to 500 millilitres with the same acid solution.

5. APPARATUS

Spectrophotometer fitted for atomic absorption, with a magnesium lamp, set at 285,2 nm.

Air-acetylene flame.

6. PREPARATION OF THE SAMPLE

See methods 8.1 and 8.3.

7. PROCEDURE

- 7.1. If the fertilizer has a declared magnesium (Mg) content of more than 6 % (i. e. 10 % as MgO), take 25 millilitres (V_1) of the extraction solution (6). Transfer into a 100 millilitre graduated flask, and make up to volume with water and mix. The dilution factor is $D_1 = 100/V_1$.

- 7.2. Using a pipette, take 10 millilitres of the extraction solution (6) or the solution (7.1). Transfer into a 200 millilitre graduated flask. Make up to volume with the 0,5 M hydrochloric acid solution (4.2) and mix. The dilution factor is 200/10.

- 7.3. Dilute this solution (7.2) with the 0,5 M hydrochloric acid solution (4.2) so as to obtain a concentration in the optimum working field of the spectrophotometer (5.1). V_2 is the volume of the sample in 100 millilitres. The dilution factor is $D_2 = 100/V_2$.

The final solution should contain 10 % v/v of the strontium chloride solution (4.4).

- 7.4. Preparation of blank solution

Prepare a blank solution by repeating the whole procedure from the extraction (method 8.1 or 8.3), omitting only the test sample of fertilizer.

- 7.5. Preparation of calibration solutions

By diluting the standard solution (4.3) with the 0,5 M hydrochloric acid, prepare at least five calibration solutions of increasing concentration within the optimum measuring range of the apparatus (5.1).

These solutions should contain 10 % v/v of the strontium chloride solution (4.4).

- 7.6. Measurement

Set up the spectrophotometer (5.1) at a wavelength of 285,2 nm.

Spray, successively, the calibration solutions (7.5), the sample solution (7.3) and the blank solution (7.4), washing the instrument through with the solution to be measured next. Repeat this operation three times. Plot the calibration curve using the mean absorbances of each of the calibrations (7.5) as the ordinates and the corresponding concentration of magnesium in $\mu\text{g/ml}$ as the abscissae. Determine the concentration of magnesium in the sample (7.3), X_s , and blank (7.4), X_b , by reference to the calibration curve.

8. EXPRESSION OF RESULTS

Calculate the amount of magnesium (Mg) or magnesium oxide (MgO) in the sample by reference to the calibration solutions and taking into consideration the blank.

The percentage of magnesium (Mg) in the fertilizer is equal to:

$$\text{Mg (\%)} = \frac{(X_s - X_b) D_1 (200/10) D_2 500.100}{1\,000.1\,000\,M}$$

X_s = the concentration of the solution to be analysed recorded on the calibration curve, in $\mu\text{g/ml}$.

X_b = the concentration of the blank solution as recorded on the calibration curve, in $\mu\text{g/ml}$.

D_1 = the dilution factor when the solution is diluted (7.1).

It is equal to four if 25 millilitres are taken.

It is equal to one when the solution is not diluted.

D_2 = the dilution factor in 7.3.

M = the mass of the test sample at the time of extraction.

$$\text{MgO (\%)} = \text{Mg (\%)} / 0,6$$

Method 8.8

DETERMINATION OF MAGNESIUM BY COMPLEXOMETRY

1. SCOPE

This document defines the procedure for the determination of magnesium in fertilizer extracts.

2. FIELD OF APPLICATION

This method applies to the following EEC fertilizer extracts for which the determination of total magnesium and/or water-soluble magnesium is provided for:

- fertilizers listed in Directive 76/116/EEC: straight nitrogenous fertilizers, type 1 b (calcium magnesium nitrate), type 7 (magnesium sulphonitrate), type 8 (nitrogenous fertilizers with magnesium) and straight potassic fertilizers, type 2 (enriched kainite), type 4 (potassium chloride containing magnesium), type 6 (potassium sulphate containing magnesium salt),
- fertilizers listed in the Annex to the Directive on secondary nutrients (89/284/EEC).

3. PRINCIPLE

The magnesium is solubilized by methods 8.1 and/or 8.3. First titration: with EDTA of Ca and Mg in the presence of Eriochrome black T. Second titration: with EDTA of Ca in the presence of calcein or of calcone carbonic acid. Determination of magnesium by difference.

4. REAGENTS

4.1 Standard 0,05 molar solution of magnesium:

4.1.1 Dissolve 1,232 grams of magnesium sulphate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) in the 0,5 M hydrochloric acid solution (4.11) and make up to 100 millilitres with the same acid.

or:

4.1.2 Weigh out 2,016 grams of magnesium oxide, previously calcined to remove all traces of carbonation. Place it in a beaker with 100 millilitres of water.

Stir in approximately 120 millilitres of approximately 1 M hydrochloric acid (4.12).

After dissolution, transfer quantitatively into a graduated 1 000 millilitre flask. Make up to volume and mix.

One millilitre of these solutions should contain 1,216 milligrams of Mg (= 2,016 milligrams of MgO).

The laboratory is responsible for testing the strength of this standard solution.

4.2 0,05 molar solution of EDTA

Weigh out 18,61 grams of the dihydrated disodium salt of ethylenediaminetetraacetic ($\text{C}_{10}\text{H}_{14}\text{N}_2\text{Na}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$), place it in a 1 000 millilitre beaker and dissolve in 600 to 800 millilitres

of water. Transfer the solution quantitatively into a graduated 1 000 millilitre flask. Make up the volume and mix. Check this solution with the standard solution (4.1) by taking a sample of 20 millilitres of the latter and by titration according to the analytical procedure described at (7.2).

One millilitre of the EDTA solution should correspond to 1,216 milligrams of Mg (= 2,016 milligrams of MgO) and to 2,004 milligrams of Ca (= 2,804 milligrams CaO) (see remarks 10.1 and 10.6).

4.3 0,05 molar standard solution of calcium

Weigh out 5,004 grams of dry calcium carbonate. Place it in a beaker with 100 millilitres of water. Progressively stir in 120 millilitres of approximately 1 M hydrochloric acid (4.12).

Bring to the boil in order to drive off the carbon dioxide, cool, transfer quantitatively into a graduated one-litre flask, make up the volume with water and mix. Check this solution against the EDTA solution (4.2) following analytical procedure (7.3). One millilitre of this solution should contain 2,004 milligrams of Ca (= 2,804 milligrams of CaO) and should correspond to one millilitre of the 0,05 molar EDTA solution (4.2).

4.4 Calcein indicator

Carefully mix in a mortar one gram of calcein with 100 grams of sodium chloride. Use 10 milligrams of this mixture. The indicator changes from green to orange. Titration must be carried out until an orange is obtained which is free from green tinges.

4.5 Calcon carbonic acid indicator.

Dissolve 400 milligrams of calcon carbonic acid in 100 millilitres of methanol. This solution may only be kept for approximately four weeks. Use three drops of this solution. The indicator changes from red to blue. Titration must be carried out until a blue is obtained which is free from red tinges.

4.6 Eriochrome black-T indicator

Dissolve 300 milligrams of Eriochrome black-T in a mixture of 25 millilitres of propanol-1 and 15 millilitres of triethanolamine. This solution may only be kept for approximately four weeks. Use three drops of this solution. This indicator turns from red to blue and titration must be carried out until a blue is obtained which is free from red tinges. It changes colour only when magnesium is present. If necessary add one millilitre of the standard solution (4.1).

When both calcium and magnesium are present the EDTA first forms a complex with the calcium and then with the magnesium. In that case two elements are determined concurrently.

4.7 Potassium cyanide solution

Aqueous solution of KCN at 2 %. (Do not pipette by mouth and see 10.7).

4.8 Solution of potassium hydroxide and potassium cyanide

Dissolve 280 grams of KOH and 66 grams of KCN in water, make up the volume to one litre and mix.

4.9 pH 10,5 buffer solution

In a 500 millilitre graduated flask, dissolve 33 grams of ammonium chloride in 200 millilitres of water, add 250 millilitres of ammonia (d = 0,91) make up the volume with water and mix. Test the pH of the solution regularly.

4.10 Diluted hydrochloric acid: one volume of hydrochloric acid (d = 1,18) plus one volume of water.

4.11 Hydrochloric acid solution approximately 0,5 M.

4.12 Hydrochloric acid solution approximately 1 M.

4.13 Sodium hydroxide solution 5 M.

5. APPARATUS

5.1 Magnetic or mechanical stirrer.

5.2 pH meter.

6. CONTROL TEST

Carry out a determination on aliquot parts of solutions (4.1 and 4.3) such that the Ca/Mg ratio is approximately equal to that of the solution to be analysed. To this end take (a) of standard solution (4.3) and (b - a) of standard solution (4.1). (a) and (b) are the numbers of millilitre EDTA solution used in the two titrations performed on the solution to be analysed. This procedure is correct only if the solutions of EDTA, calcium and magnesium are exactly equivalent. If this is not the case, it is necessary to make corrections.

7. PREPARATION OF THE SOLUTION TO BE ANALYSED

See methods 8.1 and 8.3.

8. DETERMINATION

8.1. Aliquot samples to be taken.

The aliquot part will as far as possible contain between nine and 18 milligrams of magnesium (= 15 to 30 milligrams of MgO).

8.2. Titration in the presence of Eriochrome black-T

Pipette an aliquot part (8.1) of the solution to be analysed into a 400 millilitre beaker. Neutralize the surplus acid with the 5 M sodium hydroxide solution (4.12) using the pH meter. Dilute with water to approximately 100 millilitres. Add 5 millilitres of the buffer solution (4.9). The pH measured by the meter must be $10,5 \pm 0,1$. Add 2 millilitres of the potassium cyanide solution (4.7) and three drops of the Eriochrome black-T indicator (4.6). Titrate with the EDTA solution (4.2). Stirring gently with the stirrer (5.1) (see 10.2, 10.3 and 10.4). Let 'b' be the number of millilitres of 0,05 molar EDTA solution.

8.3. Titration in the presence of calcein or of calcon carbonic acid

Pipette an aliquot part of the solution to be analysed equal to that taken from the above titration and place it in a 400 millilitres beaker. Neutralize the surplus acid with the 5 M sodium hydroxide solution (4.13) using the pH meter. Dilute with water to about 100 millilitres. Add 10 millilitres of the KOH/KCN solution (4.8) and the indicator (4.4) or (4.5). Stirring gently with the stirrer (5.1) titrate with the EDTA solution (4.2) (see 10.2, 10.3 and 10.4). Let 'a' be the number of millilitres of 0,05 molar EDTA solution.

9. EXPRESSION OF THE RESULTS

For the EEC fertilizers to which the method is applicable (5 grams of fertilizer in 500 millilitres of extract), the percentage content of the fertilizer is:

$$\text{MgO (\% in the fertilizer)} = \frac{(b - a) \times T}{M}$$

$$\text{Mg (\% in the fertilizer)} = \frac{(b - a) \times T'}{M}$$

Where:

a = the number of millilitres of 0,05 molar EDTA solution used for the titration in the presence of calcein or calcon carbonic acid,

b = the number of millilitres of 0,05 molar EDTA solution used for the titration in the presence of Eriochrome black-T,

M = the mass of the sample present in the aliquot part taken (in grams),

T = $0,2016 \times$ molarity of the EDTA solution/0,05 (see 4.2),

T' = $0,1216 \times$ molarity of the EDTA solution/0,05 (see 4.2).

10. REMARKS

10.1. The stoichiometric EDTA-metal ratio in the complexometric analyses is always 1:1 whatever the valency of the metal and in spite of the fact that EDTA is quadrivalent. The EDTA titration solution and the standard solutions will therefore be molar and not normal.

- 10.2. Complexometric indicators are often sensitive to air. The solution may lose colour during titration. In this case, one or two drops of indicator must be added. This is true particularly in the case of eriochrome black and calcon carbonic acid.
- 10.3. The metal-indicator complexes are often relatively stable and it may take some time for the colour to change. The last drops of EDTA must therefore be added slowly and a drop of 0,05 molar solution of magnesium (4.1) or calcium (4.3) added to ensure that the colour change has not already taken place. This is particularly true in the case of the eriochrome-magnesium complex.
- 10.4. The turning of the indicator must be observed not vertically, but horizontally across the solution and the beaker must be placed against a white background in a well-lit position. The turning of the indicator may also be observed easily by placing the beaker on frosted glass lit moderately from below (25 watt lamp).
- 10.5. This analysis requires a certain amount of experience. The task will involve, *inter alia*, observing the colour changes of standard solutions 4.1 and 4.3. It is recommended that the determinations be carried out by the same laboratory chemist.
- 10.6. If an EDTA solution of guaranteed strength is used (Titrisol, Normex, for example) this may simplify the control of the equivalence of standard solutions 4.1, 4.2 and 4.3.
- 10.7. The solutions containing potassium cyanide must not be poured down the sink until the cyanide has been converted into a harmless compound, for example, by oxidization with sodium hypochlorite following alkalization.

Method 8.9

DETERMINATION OF SULPHATES

1. SCOPE

This document defines the procedure for determining the sulphur present in fertilizer extracts in the form of sulphates.

2. FIELD OF APPLICATION

This method applies to the determination of sulphates present in the extractions performed in methods 8.1, 8.2, 8.3 and 8.4.

3. PRINCIPLE

Gravimetric determination as barium sulphate.

4. REAGENTS

4.1. Diluted hydrochloric acid:

One volume of hydrochloric acid ($d = 1,18$) and one volume of water.

4.2. Barium chloride solution $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$: 122 grams per litre.

4.3. Silver nitrate solution: 5 grams per litre.

5. APPARATUS

5.1. Porcelain crucibles.

5.2. Hot water bath.

5.3. Drying oven set at $105^\circ\text{C} \pm 1^\circ\text{C}$.

5.4. Electric oven set at $800^\circ\text{C} \pm 50^\circ\text{C}$.

6. PROCEDURE

6.1. Sampling of the solution

Pipette an aliquot part of one of the extraction solutions indicated at 2 containing between 20 and 100 milligrams of S or 50 and 250 milligrams of SO₃.

Place this aliquot in a beaker of suitable capacity. Add 20 millilitres of dilute hydrochloric acid (4.1). Make up to about 300 millilitres with water.

6.2. Preparation of the precipitate

Bring the solution to the boil. Add, drop by drop, about 20 millilitres of the barium chloride solution (4.2) while stirring the solution vigorously. Boil for a few minutes.

Place the beaker, covered with a watch glass, in a boiling hot water bath (5.2) for an hour. Then leave standing hot (± 60 °C) until the supernatant liquor is clear. Decant the clear solution through a slow filtration ash-free filter. Wash the precipitate several times with hot water. Continue to wash the precipitate on the filter until the filtrate is chloride free. This can be checked by using silver nitrate solution (4.3).

6.3. Incineration and weighing of the precipitate

Place the filter paper and precipitate in a porcelain crucible (5.1) previously weighed to the nearest 0,1 milligrams. Dry in the oven (5.3) and ash at approximately 800 °C for half an hour (5.4). Allow to cool in a desiccator and weigh to within 0,1 milligrams.

7. EXPRESSION OF THE RESULTS

One milligram of barium sulphate corresponds to 0,137 milligrams of S or to 0,343 milligrams of SO₃.

The percentage S content of the fertilizer is as follows:

$$S (\%) = w \times 0,0137 \times \frac{v_1}{v_2 \times m}$$

$$SO_3 (\%) = S (\%) \times 2,5$$

Where:

w = the mass of the barium sulphate precipitate in milligrams,

v₁ = the volume of the extraction solution in millilitres,

v₂ = the aliquot volume in millilitres,

m = the mass of the test sample in grams.

Method 8.10

DETERMINATION OF THE SODIUM EXTRACTED

1. SCOPE

This document defines the procedure for determining the sodium in fertilizer extracts.

2. FIELD OF APPLICATION

This method applies to EEC fertilizers for which a declaration of the sodium is provided for in Council Directive 89/284/EEC.

3. PRINCIPLE

Following suitable dilution of the extract obtained via method 8.1 and/or 8.3, the sodium content of the solution is determined by flame-emission spectrophotometry.

4. REAGENTS

4.1. Diluted hydrochloric acid:

One volume of hydrochloric acid for analysis ($d = 1,18$) plus one volume of water.

4.2. Aluminium nitrate $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$.4.3. Caesium chloride, CsCl .4.4. Anhydrous sodium chloride, NaCl .

4.5. Caesium chloride and aluminium nitrate solution

Dissolve in water 50 grams of caesium chloride (4.3) and 250 grams of aluminium nitrate (4.2) in a 1 000 millilitres graduated flask. Make up to volume with water and mix.

4.6. Standard sodium solution of one milligram/millilitre of Na.

Dissolve in water 2,542 grams of sodium chloride (4.4) in a 1 000 millilitre graduated flask. Add 10 millilitres of hydrochloric acid (4.1). Make up to volume with water and mix.

5. APPARATUS

Spectrophotometer equipped for flame emission, set at 589,3 nm.

6. CALIBRATION SOLUTIONS

6.1. Place 10 millilitres of standard solution (4.6) in a 250 millilitre graduated flask. Make up to volume and mix. Concentration of solution: 40 $\mu\text{g}/\text{ml}$ of Na.6.2. Place 0, 5, 10, 15, 20, 25 millilitres of the intermediate solution (6.1) in 100 millilitres graduated flasks. Add 10 millilitres of the solution (4.5). Make up to volume and mix. Concentration of solutions: 0, 2, 4, 6, 8, 10 $\mu\text{g}/\text{ml}$ of Na.

7. PREPARATION OF SOLUTIONS TO BE MEASURED

Depending upon the foreseeable sodium content of the extraction solution as in method 8.1 or 8.3 (five grams of fertilizer in 500 millilitres), carry out the dilutions in accordance with the following table:

Na_2O (%)	Na (%)	Intermediate dilution		Final dilution		Degree of dilution
		Sample (ml) (v_2)	Dilution to ml (v_3)	Sample (ml) (v_4)	Dilution to ml	
3—5	2,2—3,7	10	50	10	100	50
5—10	3,7—7,4	10	100	10	100	100
10—20	7,4—15	10	100	5	100	200
20—38	15—28	5	100	5	100	400

Make up the intermediate dilution with water. For the final dilution add ten millilitres of the solution (4.5) to the 100 millilitre graduated flask.

For a test sample of one gram multiply the volume of the final dilution (v_4) by five.

8. DETERMINATION

Prepare the spectrophotometer (5.1) for the measurements at 589,3 nm. Calibrate the instrument by measuring the response of the calibration solutions (6.2). Then adjust the sensitivity of the instrument to use its entire scale when the most concentrated calibration solution is used. Then measure the response of the sample solution to be analysed (7). Repeat this operation three times.

9.

CALCULATION OF RESULTS

Draw a calibration curve by plotting the average response for each calibration solution along the ordinate and the corresponding concentrations, expressed in μg per millilitre on the abscissa. Determine from this the sodium concentration of the test solution. Calculate the quantity of sodium from the standard solutions taking account of the levels of dilution. Express the results as a percentage of the sample.

The percentage sodium (Na) content of the fertilizer is as follows:

$$\text{Na (\%)} = x \cdot \frac{v_3}{v_4} \frac{v_1}{v_2} \frac{10^{-2}}{m}$$

$$\text{Na}_2\text{O (\%)} = \text{Na (\%)} \times 1,348$$

Where:

x = the concentration of the solution introduced into the spectrophotometer in $\mu\text{g/ml}$,

v_1 = the volume of the extraction solution in millilitres,

v_2 = the aliquot volume in the intermediate dilution in millilitres,

v_3 = the volume of intermediate dilution in millilitres,

v_4 = the aliquot volume in ml of the final dilution (in 100 millilitres),

m = The mass of the test sample in grams.'
