Legislation

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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.

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II *Acts whose publication is not obligatory*

Commission

I

(Acts whose publication is obligatory)

COUNCIL REGULATION (EEC) No 1108/93

of 4 May 1993

laying down certain provisions for the application of the Bilateral Agreements on agriculture between the Community, of the one part, and Austria, Finland, Iceland, Norway and Sweden, of the other part

THE COUNCIL OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community, and in particular Article 113 thereof,

Having regard to the proposal from the Commission,

Whereas the Community signed Bilateral Agreements concerning certain agricultural arrangements in the form of exchanges of letters with Austria, Finland, Iceland, Norway, Sweden and Switzerland in Oporto on 2 May 1992;

Whereas those Agreements were signed at the same time as the Agreement on the European Economic Area between the European Economic Community, the European Coal and Steel Community and the Member States thereof, of the one part, and the Member States of the European Free Trade Association, of the other part, hereinafter referred to as the 'EEA Agreement'; whereas the objective of all parties was to ensure that the EEA Agreement and the Bilateral Agreements on agriculture entered into force at the same time;

Whereas, as a result of the decision of the Swiss Confederation not to ratify the EEA Agreement, agreements in the form of exchanges of letters were signed on 17 March 1993 between the Community, of the one part, and Austria, Finland, Iceland, Norway and Sweden, of the other part, with a view to applying the Bilateral Agreements on agriculture signed on 2 May 1992 with those countries as from 15 April 1993 and until such time as the EEA Agreement enters into force; whereas the agreements of 17 March 1993 also provide that, except where the Contracting Parties decide otherwise, those provisional agreements are to expire on 1 January 1994 if the EEA Agreement does not enter into force on that date;

Whereas, by Decision of 15 March 1993 (¹), the Council approved the said agreements;

Whereas the implementing provisions for the application of various provisions of those agreements must be laid down,

HAS ADOPTED THIS REGULATION :

Article 1

As regards agricultural products listed in Annex II to the Treaty and covered by the rules of a common organization of the market, the implementing provisions for the. Bilateral Agreements on agriculture signed on 2 May 1992 in Oporto between the European Economic Community, of the one part, and Austria, Finland, Iceland, Norway and Sweden, of the other part, shall be adopted in accordance with the procedure laid down in Article 30 of Council Regulation (EEC) No 804/68 of 27 June 1968 on the common organization of the market in milk and milk products (²) in the corresponding Articles of the other regulations on the common organization of the markets. Where the application of the agreements calls for close cooperation with the signatory States, the Commission may take any measures necessary to ensure such cooperation.

Article 2

This Regulation shall enter into force on the day following its publication in the *Official Journal of the European Communities.*

This Regulation shall be applicable from 15 April 1993.

(¹) OJ No L 109, 1. 5. 1993, p. 1.

(²) OJ No L 148, 28. 6. 1968, p. 1. Regulation as last amended by Regulation (EEC) No 2071/92 (OJ No L 215, 30. 7. 1992, p. 1).

This Regulation shall be binding in its entirety and directly applicable in all Member States.

Done at Brussels, 4 May 1993.

<div align="right">

For the Council

The President

J. TRØJBORG

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COMMISSION REGULATION (EEC) No 1109/93

of 6 May 1993

fixing the import levies on cereals and on wheat or rye flour, groats and meal

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Regulation (EEC) No 2727/75 of 29 October 1975 on the common organization of the market in cereals (¹), as last amended by Regulation (EEC) No 1738/92 (²), and in particular Article 13 (5) thereof,

Having regard to Council Regulation (EEC) No 3813/92 of 28 December 1992 on the unit of account and the conversion rates to be applied for the purposes of the common agricultural policy (³), and in particular Article 5 thereof,

Whereas the import levies on cereals, wheat and rye flour, and wheat groats and meal were fixed by Commission Regulation (EEC) No 762/93 (⁴) and subsequent amending Regulations;

Whereas, in order to make it possible for the levy arrangements to function normally, the representative market rate established during the reference period from 5 May 1993, as regards floating currencies, should be used to calculate the levies;

Whereas it follows from applying the detailed rules contained in Regulation (EEC) No 762/93 to today's offer prices and quotations known to the Commission that the levies at present in force should be altered to the amounts set out in the Annex hereto,

HAS ADOPTED THIS REGULATION:

Article 1

The import levies to be charged on products listed in Article 1 (a), (b) and (c) of Regulation (EEC) No 2727/75 shall be as set out in the Annex hereto.

Article 2

This Regulation shall enter into force on 7 May 1993.

This Regulation shall be binding in its entirety and directly applicable in all Member States.

Done at Brussels, 6 May 1993.

For the Commission

René STEICHEN

Member of the Commission

(¹) OJ No L 281, 1. 11. 1975, p. 1.
(²) OJ No L 180, 1. 7. 1992, p. 1.
(³) OJ No L 387, 31. 12. 1992, p. 1.
(⁴) OJ No L 79, 1. 4. 1993, p. 11.

ANNEX

to the Commission Regulation of 6 May 1993 fixing the import levies on cereals and on wheat or rye flour, groats and meal

(ECU/tonne)

CN code	Third countries ([8])
0709 90 60	140,42 ([2]) ([3])
0712 90 19	140,42 ([2]) ([3])
1001 10 00	182,73 ([1]) ([5])
1001 90 91	144,46
1001 90 99	144,46 ([9])
1002 00 00	156,10 ([6])
1003 00 10	142,21
1003 00 20	142,21
1003 00 80	142,21 ([9])
1004 00 00	116,24
1005 10 90	140,42 ([2]) ([3])
1005 90 00	140,42 ([2]) ([3])
1007 00 90	145,64 ([4])
1008 10 00	58,17 ([9])
1008 20 00	102,53 ([4])
1008 30 00	62,71 ([5])
1008 90 10	([7])
1008 90 90	62,71
1101 00 00	214,84 ([9])
1102 10 00	231,14
1103 11 30	295,27
1103 11 50	295,27
1103 11 90	230,38

([1]) Where durum wheat originating in Morocco is transported directly from that country to the Community, the levy is reduced by ECU 0,60/tonne.

([2]) In accordance with Regulation (EEC) No 715/90 the levies are not applied to products imported directly into the French overseas departments, originating in the African, Caribbean and Pacific States.

([3]) Where maize originating in the ACP is imported into the Community the levy is reduced by ECU 1,81/tonne.

([4]) Where millet and sorghum originating in the ACP is imported into the Community the levy is applied in accordance with Regulation (EEC) No 715/90.

([5]) Where durum wheat and canary seed produced in Turkey are transported directly from that country to the Community, the levy is reduced by ECU 0,60/tonne.

([6]) The import levy charged on rye produced in Turkey and transported directly from that country to the Community is laid down in Council Regulation (EEC) No 1180/77 (OJ No L 142, 9. 6. 1977, p. 10), as last amended by Regulation (EEC) No 1902/92 (OJ No L 192, 11. 7. 1992, p. 3), and Commission Regulation (EEC) No 2622/71 (OJ No L 271, 10. 12. 1971, p. 22), as amended by Regulation (EEC) No 560/91 (OJ No L 62, 8. 3. 1991, p. 26).

([7]) The levy applicable to rye shall be charged on imports of the product falling within CN code 1008 90 10 (triticale).

([8]) No levy applies to OCT originating products according to Article 101 (1) of Decision 91/482/EEC.

([9]) Products falling within this code, imported from Poland, Czechoslovakia or Hungary under the Interim Agreements concluded between those countries and the Community, and in respect of which EUR.1 certificates issued in accordance with Regulation (EEC) No 585/92 have been presented, are subject to the levies set out in the Annex to that Regulation.

COMMISSION REGULATION (EEC) No 1110/93

of 6 May 1993

fixing the premiums to be added to the import levies on cereals, flour and malt

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Regulation (EEC) No 2727/75 of 29 October 1975 on the common organization of the market in cereals (¹), as last amended by Regulation (EEC) No 1738/92 (²), and in particular Article 15 (6) thereof,

Having regard to Council Regulation (EEC) No 3813/92 of 28 December 1992 on the unit of account and the conversion rates to be applied for the purposes of the common agricultural policy (³), and in particular Article 5 thereof,

Whereas the premiums to be added to the levies on cereals and malt were fixed by Commission Regulation (EEC) No 3874/92 (⁴) and subsequent amending Regulations;

Whereas, in order to make it possible for the levy arrangements to function normally, the representative market rate established during the reference period from 5 May

1993, as regards floating currencies, should be used to calculate the levies;

Whereas, on the basis of today's cif prices and cif forward delivery prices, the premiums at present in force, which are to be added to the levies, should be altered to the amounts set out in the Annex hereto,

HAS ADOPTED THIS REGULATION:

Article 1

The premiums referred to in Article 15 of Regulation (EEC) No 2727/75 to be added to the import levies fixed in advance in respect of cereals and malt coming from third countries shall be as set out in the Annex hereto.

Article 2

This Regulation shall enter into force on 7 May 1993.

This Regulation shall be binding in its entirety and directly applicable in all Member States.

Done at Brussels, 6 May 1993.

For the Commission
René STEICHEN
Member of the Commission

(¹) OJ No L 281, 1. 11. 1975, p. 1.
(²) OJ No L 180, 1. 7. 1992, p. 1.
(³) OJ No L 387, 31. 12. 1992, p. 1.
(⁴) OJ No L 390, 31. 12. 1992, p. 121.

ANNEX

to the Commission Regulation of 6 May 1993 fixing the premiums to be added to the import levies on cereals, flour and malt

A. Cereals and flour

(ECU/tonne)

CN code	Current 5	1st period 6	2nd period 7	3rd period 8
0709 90 60	0	0	0	0
0712 90 19	0	0	0	0
1001 10 00	0	0	0	0
1001 90 91	0	7,72	7,72	11,39
1001 90 99	0	7,72	7,72	11,39
1002 00 00	0	0	0	0
1003 00 10	0	0	0	0
1003 00 20	0	0	0	0
1003 00 80	0	0	0	0
1004 00 00	0	0	0	0
1005 10 90	0	0	0	0
1005 90 00	0	0	0	0
1007 00 90	0	0	0	0
1008 10 00	0	0	0	0
1008 20 00	0	0	0	0
1008 30 00	0	0	0	0
1008 90 90	0	0	0	0
1101 00 00	0	10,80	10,80	15,95

B. Malt

(ECU/tonne)

CN code	Current 5	1st period 6	2nd period 7	3rd period 8	4th period 9
1107 10 11	0	13,74	13,74	20,27	20,27
1107 10 19	0	10,27	10,27	15,15	15,15
1107 10 91	0	0	0	0	0
1107 10 99	0	0	0	0	0
1107 20 00	0	0	0	0	0

COMMISSION REGULATION (EEC) No 1111/93

of 6 May 1993

fixing the minimum levies on the importation of olive oil and levies on the importation of other olive oil sector products

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Regulation No 136/66/EEC of 22 September 1966 on the establishment of a common organization of the market in oils and fats (¹), as last amended by Regulation (EEC) No 2046/92 (²), and in particular Article 16 (2) thereof,

Having regard to Council Regulation (EEC) No 1514/76 of 24 June 1976 on imports of olive oil originating in Algeria (³), as last amended by Regulation (EEC) No 1900/92 (⁴), and in particular Article 5 thereof,

Having regard to Council Regulation (EEC) No 1521/76 of 24 June 1976 on imports of olive oil originating in Morocco (⁵), as last amended by Regulation (EEC) No 1901/92 (⁶), and in particular Article 5 thereof,

Having regard to Council Regulation (EEC) No 1508/76 of 24 June 1976 on imports of olive oil originating in Tunisia (⁷), as last amended by Regulation (EEC) No 413/86 (⁸), and in particular Article 5 thereof,

Having regard to Council Regulation (EEC) No 1180/77 of 17 May 1977 on imports into the Community of certain agricultural products originating in Turkey (⁹), as last amended by Regulation (EEC) No 1902/92 (¹⁰), and in particular Article 10 (2) thereof,

Having regard to Council Regulation (EEC) No 1620/77 of 18 July 1977 laying down detailed rules for the importation of olive oil from Lebanon (¹¹),

Whereas by Regulation (EEC) No 3131/78 (¹²), as amended by the Act of Accession of Greece, the Commission decided to use the tendering procedure to fix levies on olive oil ;

Whereas Article 3 of Council Regulation (EEC) No 2751/78 of 23 November 1978 laying down general rules for fixing the import levy on olive oil by tender (¹³) specifies that the minimum levy rate shall be fixed for each of the products concerned on the basis of the situation on the world market and the Community market and of the levy rates indicated by tenderers ;

Whereas, in the collection of the levy, account should be taken of the provisions in the Agreements between the Community and certain third countries ; whereas in particular the levy applicable for those countries must be fixed, taking as a basis for calculation the levy to be collected on imports from the other third countries ;

Whereas, pursuant to Article 101 (1) of Council Decision 91/482/EEC of 25 July 1991 on the association of the overseas countries and territories with the European Economic Community (¹⁴), no levies shall apply on imports of products originating in the overseas countries and territories ;

Whereas application of the rules recalled above to the levy rates indicated by tenderers on 3 and 4 May 1993 leads to the minimum levies being fixed as indicated in Annex I to this Regulation ;

Whereas the import levy on olives falling within CN codes 0709 90 39 and 0711 20 90 and on products falling within CN codes 1522 00 31, 1522 00 39 and 2306 90 19 must be calculated from the minimum levy applicable on the olive oil contained in these products; whereas, however, the levy charged for olive oil may not be less than an amount equal to 8 % of the value of the imported product, such amount to be fixed at a standard rate ; whereas application of these provisions leads to the levies being fixed as indicated in Annex II to this Regulation,

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(¹) OJ No 172, 30. 9. 1966, p. 3025/66.
(²) OJ No L 215, 30. 7. 1992, p. 1.
(³) OJ No L 169, 28. 6. 1976, p. 24.
(⁴) OJ No L 192, 11. 7. 1992, p. 1.
(⁵) OJ No L 169, 28. 6. 1976, p. 43.
(⁶) OJ No L 192, 11. 7. 1992, p. 2.
(⁷) OJ No L 169, 28. 6. 1976, p. 9.
(⁸) OJ No L 48, 26. 2. 1986, p. 1.
(⁹) OJ No L 142, 9. 6. 1977, p. 10.
(¹⁰) OJ No L 192, 11. 7. 1992, p. 3.
(¹¹) OJ No L 181, 21. 7. 1977, p. 4.

(¹²) OJ No L 370, 30. 12. 1978, p. 60.
(¹³) OJ No L 331, 28. 11. 1978, p. 6.
(¹⁴) OJ No L 263, 19. 9. 1991, p. 1.

HAS ADOPTED THIS REGULATION:

Article 1

The minimum levies on olive oil imports are fixed in Annex I.

Article 2

The levies applicable on imports of other olive oil sector products are fixed in Annex II.

Article 3

This Regulation shall enter into force on 7 May 1993.

This Regulation shall be binding in its entirety and directly applicable in all Member States.

Done at Brussels, 6 May 1993.

For the Commission
René STEICHEN
Member of the Commission

ANNEX I

Minimum import levies on olive oil (¹)

(ECU/100 kg)

CN code	Non-member countries
1509 10 10	79,00 (²)
1509 10 90	79,00 (²)
1509 90 00	92,00 (³)
1510 00 10	77,00 (²)
1510 00 90	122,00 (⁴)

(¹) No levy applies to OCT originating products according to Article 101 (1) of Decision 91/482/EEC.

(²) For imports of oil falling within this CN code and produced entirely in one of the countries listed below and transported directly from any of those countries to the Community, the levy to be collected is reduced by :

(a) Lebanon : ECU 0,60 per 100 kg ;

(b) Tunisia : ECU 12,69 per 100 kg provided that the operator furnishes proof of having paid the export tax applied by that country ; however, the repayment may not exceed the amount of the tax in force ;

(c) Turkey : ECU 22,36 per 100 kg provided that the operator furnishes proof of having paid the export tax applied by that country ; however, the repayment may not exceed the amount of the tax in force ;

(d) Algeria and Morocco : ECU 24,78 per 100 kg provided that the operator furnishes proof of having paid the export tax applied by that country ; however, the repayment may not exceed the amount of the tax in force.

(³) For imports of oil falling within this CN code :

(a) produced entirely in Algeria, Morocco or Tunisia and transported directly from any of those countries to the Community, the levy to be collected is reduced by ECU 3,86 per 100 kg ;

(b) produced entirely in Turkey and transported directly from that country to the Community, the levy to be collected is reduced by ECU 3,09 per 100 kg.

(⁴) For imports of oil falling within this CN code :

(a) produced entirely in Algeria, Morocco or Tunisia and transported directly from any of those countries to the Community, the levy to be collected is reduced by ECU 7,25 per 100 kg ;

(b) produced entirely in Turkey and transported directly from that country to the Community, the levy to be collected is reduced by ECU 5,80 per 100 kg.

ANNEX II

Import levies on other olive oil sector products (¹)

(ECU/100 kg)

CN code	Non-member countries
0709 90 39	17,38
0711 20 90	17,38
1522 00 31	39,50
1522 00 39	63,20
2306 90 19	6,16

(¹) No levy applies to OCT originating products according to Article 101 (1) of Decision 91/482/EEC.

COMMISSION REGULATION (EEC) No 1112/93

of 6 May 1993

laying down detailed rules for the application of the supplementary trade mechanism to trade in the beef and veal sector between the Community as constituted at 31 December 1985 and Spain and Portugal and repealing Regulations (EEC) No 3810/91 and (EEC) No 3829/92

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to the Act of Accession of Spain and Portugal, and in particular Articles 83 and 251 thereof,

Having regard to Council Regulation (EEC) No 3817/92 of 28 December 1992 laying down general rules for applying the supplementary trade mechanism to deliveries in Spain of products other than fruit and vegetables [1], and in particular Article 9 thereof,

Having regard to Council Regulation (EEC) No 744/93 of 17 March 1993 laying down general rules for applying the supplementary trade mechanism to deliveries in Portugal of products other than fruit and vegetables [2], and in particular Article 1 thereof,

Having regard to Council Regulation (EEC) No 3792/85 of 20 December 1985 laying down the arrangements applying to trade in agricultural products between Spain and Portugal [3], as last amended by Regulation (EEC) No 3296/88 [4], and in particular Article 13 thereof,

Whereas Commission Regulation (EEC) No 3810/91 [5], as last amended by Regulation (EEC) No 894/93 [6], set for 1993 the indicative ceilings mentioned in Articles 83 and 251 of the Act of Accession of Spain and Portugal; whereas these ceilings are set on the basis of estimated Spanish and Portuguese production and consumption of the products in question in the beef and veal sector and a forward timetable for trade with the rest of the Community;

Whereas Article 5 of Council Regulation (EEC) No 3792/85 lays down that imports of these products into Portugal from Spain are subject to the supplementary trade mechanism in accordance with Articles 249 to 252 of the Act of Accession;

Whereas Article 1 (3) of Regulation (EEC) No 3817/92 lays down that the issue of STM licences need not neces-

sarily be subject to the lodging of a security; whereas that possibility was provided for, in particular, to facilitate trade in the products concerned; whereas use should be made of that possibility to ensure that no security need be lodged when applications for the STM licences concerned are submitted;

Whereas in order to regulate trade more satisfactorily by taking account of the varying levels of sensitivity of the Spanish and Portuguese markets at different times of the year, and in particular lower demand in the second and third quarters, the annual quantities should be split up into two-month periods;

Whereas, when laying down the detailed rules for the lodging of applications and the issuing of licences, derogations should be made both from Commission Regulation (EEC) No 3719/88 of 16 November 1988 laying down common detailed rules for the application of the system of import and export licences and advance fixing certificates for agricultural products [7], as last amended by Regulation (EEC) No 2101/92 [8], and from Commission Regulation (EEC) No 574/86 of 28 February 1986 laying down detailed rules for the application of the supplementary trade mechanism [9], as last amended by Regulation (EEC) No 3296/88;

Whereas in order to simplify the procedure for issuing STM licences an automatic system of issue should be laid down along with the introduction of a tighter notification system and a mechanism for monitoring the use of the licences issued;

Whereas it should be laid down that Community traders may only export certain beef and veal products to Spain and Portugal under certain restrictive conditions regarding in particular the length of time they have been established in the trade;

Whereas, with regard to imports into Portugal from third countries, certain aspects of the arrangements applicable to STM import licences laid down by Regulation (EEC) No 3817/92 should be clarified; whereas, for this purpose, the application of Commission Regulation (EEC)

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[1] OJ No L 387, 31. 12. 1992, p. 12.
[2] OJ No L 77, 31. 3. 1993, p. 11.
[3] OJ No L 367, 31. 12. 1985, p. 7.
[4] OJ No L 293, 27. 10. 1988, p. 7.
[5] OJ No L 357, 28. 12. 1991, p. 53.
[6] OJ No L 93, 17. 4. 1993, p. 8.

[7] OJ No L 331, 2. 12. 1988, p. 1.
[8] OJ No L 210, 25. 7. 1992, p. 18.
[9] OJ No L 57, 1. 3. 1986, p. 1.

No 2377/80 of 4 September 1980 on special detailed rules for the application of the system of import and export licences in the beef and veal sector (¹), as last amended by Regulation (EEC) No 3662/92 (²), and of the other provisions regarding the various special import arrangements, is the most appropriate for the import arrangements for beef and veal;

Whereas it is desirable, for the sake of clarity, to set out in a new Regulation the detailed rules for the application of the STM in respect of Spain and Portugal and to repeal Commission Regulations (EEC) No 3810/91 and (EEC) No 3829/92 (³);

Whereas the measures provided for in this Regulation are in accordance with the opinion of the Management Committee for Beef and Veal,

HAS ADOPTED THIS REGULATION:

Article 1

1. The indicative ceilings relating to certain products in the beef and veal sector which may be imported into Spain from the Community as constituted at 31 December 1985 shall be those set out in Annex I hereto.

2. The indicative ceilings relating to certain products in the beef and veal sector which may be imported into Portugal from the Community as constituted at 31 December 1985 and from Spain shall be those set out in Annex II hereto.

3. If in the course of the same calendar year the total quantity for which applications are submitted in respect of a two-month period is less than the quantity available, the quantity remaining shall be added to the quantity available in respect of the following two-month period.

Article 2

Notwithstanding:

(a) Article 15 of Regulation (EEC) No 3719/88, applications for STM licences shall be lodged each working day up to 1 p.m. and the licences shall be issued automatically on the following day, subject to the provisions of Article 3;

(b) the first and second subparagraphs of Article 6 (2) of Regulation (EEC) No 574/86, the Member States shall notify the Commission before 1 p.m. every Monday of the quantity for which licence applications have been lodged the previous week; however, the Member

States shall notify the Commission each day of the quantity for which licences have been issued on the previous day where the said licences cover more than 2 000 head and 200 head per day respectively for Spain and Portugal;

(c) Article 6 (1) of Regulation (EEC) No 574/86, the first copy of the licence shall be handed to the applicant or sent to the address stated in the application.

Article 3

1. Where the notifications referred to in Article 2 (b) indicate that the rate at which licences are being issued threatens to use up completely the quantities remaining available, the Commission shall take special measures to allow the Member States to suspend the issue of licences until the following two-month period.

2. Proof of the use of licences must be provided in the month following the end of their validity; if examination of such proof indicates that the licences issued have not been completely used up the Member State may take the measures which it deems appropriate and which may include the refusal to issue new licences.

Article 4

1. Applicants must be natural or legal persons who at the time the application is submitted are entered in a public register of a Member State and have been engaged for at least 12 months in an activity in the field of trade in live animals of the bovine species other than pure-bred breeding animals.

2. Licence applications shall be considered only if an applicant declares in writing that he has not lodged and undertakes not to lodge any application concerning the same product in any Member State other than that where his present application is lodged; if an applicant lodges applications in two or more Member States, none of the applications shall be considered.

3. All applications from one applicant shall be regarded as a single application.

Article 5

The sum of the quantities stated in the STM licences applied for by a given trader in any one day shall not exceed 200 head per Member State of destination.

Article 6

STM licences as provided for in Articles 1 and 3 of Regulation (EEC) No 744/93 shall be valid for 10 days from the actual date of issue, in acordance with Article 21 (2) of Regulation (EEC) No 3719/88. This period of validity shall be increased to 15 days in the case of duly justified sea transport.

(¹) OJ No L 241, 13. 9. 1980, p. 5.
(²) OJ No L 370, 19. 12. 1992, p. 43.
(³) OJ No L 387, 31. 12. 1992, p. 45.

However, STM licences shall be valid for 30 days if the products are released to the market in the Azores or Madeira.

Article 7

1. The provisions of Regulation (EEC) No 574/86 shall apply, in so far as is necessary, for the supplementary trade mechanism provided for in Regulation (EEC) No 3817/92, save as otherwise provided in paragraph 2.

2. For the purpose of applying Regulation (EEC) No 3817/92 and Article 7 of Regulation (EEC) No 574/86, copy No 4 of the Internal Community transit document, stamped by the office of destination, for use in accordance with the provisions of Article 3 (3) (b) of Council Regulation (EEC) No 2726/90 (¹), shall be considered a declaration of entry for consumption in Spain and Portugal.

The provisions of the previous subparagraph shall not preclude the use of simplified Community transit procedures. They may not, however, be treated as a ground for checks at the frontier.

Article 8

1. Spain and Portugal shall notify the Commission of the quantities of products actually imported in each two-month period, broken down by product, no later than 45 days after the end of the period concerned.

2. Spain and Portugal shall notify the Commission no later than 15 October each year of the forecast production and consumption in each of those Member States for the following year.

Article 9

Notwithstanding Regulation (EEC) No 574/86, in the case of Portugal STM import licences as provided for in Article 1 (1) of Regulation (EEC) No 3817/92 shall be subject to the provisions of Regulation (EEC) No 2377/80 and to the other provisions regarding the various special import arrangements.

The communications referred to in Article 10 (8) of Regulation (EEC) No 574/86 shall specify the quantities applied for under each set of import arrangements.

Article 10

Regulations (EEC) No 3810/91 and (EEC) No 3829/92 are hereby repealed.

Article 11

This Regulation shall enter into force on the day of its publication in the *Official Journal of the European Communities.*

It shall apply with effect from 7 May 1993.

This Regulation shall be binding in its entirety and directly applicable in all Member States.

Done at Brussels, 6 May 1993.

For the Commission
René STEICHEN
Member of the Commission

(¹) OJ No L 262, 26. 9. 1990, p. 1.

ANNEX I

Group I	CN code	Description	Indicative ceiling 1993	
1	0102 90	Live animals of the bovine species other than pure-bred breeding animals and animals for bullfights (head)	275 000 head	
			of which : January/February :	60 000
			March/April :	60 000
			May/June :	35 000
			July/August :	35 000
			September/October :	40 000
			November/December :	45 000

ANNEX II

Group I	CN code	Description	Indicative ceiling 1993	
1	ex 0102 90	Live animals of the domestic bovine species other than pure-bred breeding animals for bullfights (head)	22 000 head	
			of which : January/February :	5 000
			March/April :	5 000
			May/June :	2 000
			July/August :	2 000
			September/October :	4 000
			November/December :	4 000

COMMISSION REGULATION (EEC) No 1113/93

of 6 May 1993

setting specific rules on compensatory payments on certain irrigated arable crops

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Regulation (EEC) No 1765/92 of 30 June 1992 establishing a support system for producers of certain arable crops (¹), as last amended by Regulation (EEC) No 364/93 (²), and in particular Articles 12 and 16 thereof,

Whereas Article 3 of Regulation (EEC) No 1765/92 allows regionalization plans to differentiate between irrigated and non-irrigated areas;

Whereas in certain parts of the Community irrigated and non-irrigated areas are intermixed to such an extent that it is not always possible to define distinct homogeneous production zones; whereas it would be appropriate in such areas to differentiate production yields, within a single production region, on the basis not of geography but of whether or not irrigation is employed;

Whereas the resulting difference in compensatory payment amounts could well stimulate an expansion of irrigation for the purpose of obtaining higher support and hence result in greater expenditure; whereas in order not to jeopardize the effectiveness of the scheme, by analogy with Article 2 of Regulation (EEC) No 1765/92 the area eligible for compensatory payments at the rate for irrigated land should be restricted to a ceiling corresponding to the irrigated area of the region in question over the period 1989, 1990 and 1991;

Whereas in certain cases investment may have been made after the reference period uses for setting the ceiling but before Regulation (EEC) No 1765/92 entered into force; whereas suitable provision should therefore be made in regard to certain newly irrigated areas;

Whereas at the present time most Member States have adopted provisions in regard to irrigation and have notified them to interested parties; whereas application of the provisions of this Regulation in regard to land set-aside could well call into question the set-aside plans already made by interested parties; whereas those provisions should therefore be introduced progressively;

Whereas the Joint Management Committee for Cereals, Oils and Fats, and Dried Fodder has not delivered an opinion within the period set by its chairman,

HAS ADOPTED THIS REGULATION:

Article 1

In regions where irrigation is traditionally of importance but because the irrigated areas are scattered or intermixed with non-irrigated areas the regionalization plan cannot delimit distinct homogeneous zones, compensatory payments for irrigated arable crops may be calculated using a specific yield.

Article 2

If Article 1 is applied, the historic regional average yield notified under the first and second subparagraphs of Article 3 (2) of Regulation (EEC) No 1765/92 shall be broken down into an irrigated and a non-irrigated yield. For that purpose the same years shall be used as are used for determining the past regional average yield. The past yield of the region may not be increased as a result of the differentiation.

Article 3

1.　Member States shall set rules for determining whether an area is irrigated in the course of the crop year. Those rules shall include at least:

— a list of arable crops on which compensatory payment at the irrigated yield rate may be granted,

— specification of the irrigation equipment that the farmer must hold; this must be commensurate with the area involved,

— the irrigation period to be taken into account.

(¹) OJ No L 181, 1. 7. 1992, p. 12.
(²) OJ No L 42, 19. 2. 1993, p. 3.

2. In their 'area' aid applications, producers shall separate irrigated and non-irrigated areas. Member States shall check that applications for irrigated payment satisfy the rules indicated in paragraph 1. Should they not do so the penalties laid down in Commission Regulation (EEC) No 3887/92 (¹) shall be applied according to the area involved.

Article 4

1. Compensatory payment at the yield level set for irrigated arable crops shall be given within a limit set for each production region as delimited in the regionalization plan.

2. For each production region the limit shall be the average area irrigated in the years 1989, 1990 and 1991 for the growing of cereals, oil seeds or protein crops, plus any newly irrigated areas devoted to those crops from 1992 onwards as a result of investment made before 1 August 1992.

3. Member States shall transmit the detailed information indicated in paragraph 2 to the Commission, together with supporting documents showing that the rules are met. They shall in particular specify the areas irrigated for each type of crop from 1989 to 1991 and the areas newly irrigated that are devoted to qualifying crops, together with their location and the date when implementation of the investment commenced.

4. The Commission shall examine the information provided by Member States and satisfy itself that the supporting documents are adequate. It shall get the ceilings using the procedures laid down in Article 12 (1) of Regulation (EEC) No 1765/92.

Article 5

1. If the area for which compensatory payment is applied for on the basis of a specific irrigated yield plus the corresponding set-aside area exceeds the ceiling indicated in Article 4 compensatory payment at the rate for the irrigated yield shall be proportionately reduced for the region in question. However for the 1993/94 marketing year a reduction shall be made only in accordance with the margin (if any) by which the irrigated crops alone — the set-aside area being discounted — exceed the limit.

2. Should both the ceiling indicated in this Regulation and a base area as defined in Article 2 of Regulation (EEC) No 1765/92 be exceeded on the same occasion, only the higher of the two reductions provided for shall be made.

Article 6

In regions where the provisions of this Regulation are applied :

(a) the question whether or not a producer qualified as a small producer as defined in Article 8 of Regulation (EEC) No 1765/92 shall be settled according to the content of his 'area' application or a whole, and to the irrigated and non-irrigated yields ;

(b) compensatory payments for arable crops made under the general or the simplified scheme shall be made on the basis of the irrigated yield, for the corresponding areas, and of the non-irrigated yield, for other areas ;

(c) compensatory payments for set-aside land shall be made on the basis of the average yield for the region.

Article 7

This Regulation shall enter into force on the seventh day following its publication in the *Official Journal of the European Communities.*

This Regulation shall be binding in its entirety and directly applicable in all Member States.

Done at Brussels, 6 May 1993.

For the Commission
René STEICHEN
Member of the Commission

(¹) OJ No L 391, 31. 12. 1992, p. 36.

COMMISSION REGULATION (EEC) No 1114/93

of 6 May 1993

amending Regulation (EEC) No 846/93 introducing a countervailing charge on apples originating in Chile

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Regulation (EEC) No 1035/72 of 18 May 1972 on the common organization of the market in fruit and vegetables (¹), as last amended by Regulation (EEC) No 638/93 (²), and in particular the second subparagraph of Article 27 (2) thereof,

Whereas Commission Regulation (EEC) No 846/93 (³), as last amended by Regulation (EEC) No 1074/93 (⁴), introduced a countervailing charge on apples originating in Chile;

Whereas Article 26 (1) of Regulation (EEC) No 1035/72 laid down the conditions under which a charge intro-

duced in application of Article 25 of that Regulation is amended; whereas, if those conditions are taken into consideration, the countervailing charge on the import of apples originating in Chile, must be altered,

HAS ADOPTED THIS REGULATION:

Article 1

In Article 1 of Regulation (EEC) No 846/93 'ECU 12,28' is hereby replaced by 'ECU 16,97'.

Article 2

This Regulation shall enter into force on 7 May 1993.

This Regulation shall be binding in its entirety and directly applicable in all Member States.

Done at Brussels, 6 May 1993.

For the Commission

René STEICHEN

Member of the Commission

(¹) OJ No L 118, 20. 5. 1972, p. 1.
(²) OJ No L 69, 20. 3. 1993, p. 7.
(³) OJ No L 88, 8. 4. 1993, p. 30.
(⁴) OJ No L 108, 1. 5. 1993, p. 123.

II

(Acts whose publication is not obligatory)

COMMISSION

COMMISSION DIRECTIVE 93/1/EEC

of 21 January 1993

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

(Analysis methods for trace elements)

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 Directive 89/530/EEC (²), and in particular Article 9 (2) thereof,

Whereas Article 8a of the Treaty establishes an area without internal frontiers in which the free movement of goods, persons, services and capital is ensured;

Whereas Directive 89/530/EEC supplements and amends Directive 76/116/EEC in respect of the trace elements boron, cobalt, copper, iron, manganese, molybdenum and zinc in fertilizers;

Whereas Commission Directive 77/535/EEC (³), as last amended by Directive 89/519/EEC (⁴), provides for official controls for Community fertilizers for the purpose of checking compliance with the requirements imposed by Community provisions concerning the quality and composition of fertilizers; whereas Directive 77/535/EEC should be supplemented so that fertilizers to which Directive 89/530/EEC applies can also be checked;

Whereas, in view of the scope and effects of the proposed action, the Community measures provided for by this Directive are not only necessary but also indispensable for

the attainment of the stated objectives, whereas these objectives cannot be achieved by Member States individually, and whereas their attainment at Community level is, in fact, already provided for by Directive 76/116/EEC;

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,

HAS ADOPTED THIS DIRECTIVE:

Article 1

The text set out in the Annex to this Directive is hereby added to Annex II to Directive 77/535/EEC.

The methods are applicable to Community fertilizers for the determination of each trace element the declared content of which is less than or equal to 10 %.

Article 2

1.   Member States shall bring into force the provisions necessary to comply with this Directive by 31 December 1993. They shall immediately inform the Commission thereof.

When Member States adopt these provisions, these shall contain a reference to this Directive or shall be accompanied by such reference at the time of their official publication. The procedure for such reference shall be adopted by Member States.

(¹) OJ No L 24, 30. 1. 1976, p. 21.
(²) OJ No L 281, 30. 9. 1989, p. 116.
(³) OJ No L 213, 22. 8. 1977, p. 1.
(⁴) OJ No L 265, 12. 9. 1989, p. 30.

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, 21 January 1993.

For the Commission

Martin BANGEMANN

Member of the Commission

ANNEX

'Methods 9

TRACE ELEMENTS

Method 9.1

EXTRACTION OF TOTAL TRACE ELEMENTS

1. **SCOPE**

   This method defines the procedure for extracting the following trace elements : total boron, total cobalt, total copper, total iron, total manganese, total molybdenum and total zinc. The aim is to carry out the minimum number of extractions, making use wherever possible of the same extract to determine the total level of each of the trace elements listed above.

2. **FIELD OF APPLICATION**

   This procedure concerns EEC fertilizers covered by Council Directive 89/530/EEC (¹) containing one or more of the following trace elements : boron, cobalt, copper, iron, manganese, molybdenum and zinc. It is applicable to each trace element the declared content of which is less than or equal to 10 %.

3. **PRINCIPLE**

   Dissolution in boiling dilute hydrochloric acid.

   Note : The extraction is empirical and may not be quantitative depending on the product or the other constituents of the fertilizer. In particular, in the case of certain manganese oxides, the quantity extracted may be substantially smaller than the total quantity of manganese which the product contains. It is the responsibility of the fertilizer manufacturers to ensure that the declared content actually corresponds to the quantity extracted under the conditions pertaining to the method.

4. **REAGENTS**

   4.1. Dilute hydrochloric acid (HCl) solution, about 6 M :

   Mix 1 volume of hydrochloric acid (ρ = 1,18 g/ml) with 1 volume of water.

   4.2. Concentrated ammonia solution (NH₄OH, ρ = 0,9 g/ml)

5. **APPARATUS**

   Electric hotplate with variable temperature control.

   Note : Where the boron content of an extract is to be determined, do not use borosilicate glassware. As the method involves boiling, teflon or silica is preferable. Rinse the glassware thoroughly if it has been washed in detergents containing borates.

6. **PREPARATION OF THE SAMPLE**

   See Method 1 (Directive 77/535/EEC, OJ No L 213, 22. 8. 1977, p. 1).

7. **PROCEDURE**

   7.1. Test sample

   Take a quantity of fertilizer weighing between 2 and 10 g depending on the declared content of element in the product. The following table shall be used to obtain a final solution which, after appropriate dilution, will be within the measuring range for each method. Samples should be weighed to within 1 mg.

Declared content of trace element in the fertilizer (%)	<0,01	0,01-<5	≥5-10
Mass of test sample (g)	10	5	2
Mass of element in the sample (mg)	1	0,5-250	100-200
Volume of extract V (ml)	250	500	500
Concentration of element in extract (mg/l)	4	1-500	200-400

Place the sample in a 250 ml beaker.

(¹) OJ No L 281, 30. 9. 1989, p. 116.

7.2.   Preparation of the solution

If necessary moisten the sample with a little water, add 10 ml of dilute hydrochloric acid (4.1) per gram of fertilizer carefully, in small amounts, then add about 50 ml of water. Cover the beaker with a watchglass and mix. Bring to the boil on the hotplate and boil for 30 minutes. Allow to cool, stirring occasionally. Transfer quantitatively to a 250 or 500 ml volumetric flask (see Table). Make up to volume with water and mix thoroughly. Filter through a dry filter into a dry container. Discard the first portion. The extract must be perfectly clear.

It is recommended that the determinations be carried out without delay on aliquot portions of the clear filtrate, if not the containers should be stoppered.

Remark : Extracts in which the boron content has to be determined : Adjust the pH to between 4 and 6 with concentrated ammonia (4.2).

8.   DETERMINATION

The determination of each trace element is to be carried out on the aliquot portions indicated in the method for each individual trace element.

If necessary, remove organic chelating or complexing substances from an aliquot portion of the extract by using Method 9.3. In the case of determination by atomic absorption spectrometry, such removal may not be necessary.

Method 9.2

EXTRACTION OF WATER-SOLUBLE TRACE ELEMENTS

1.   SCOPE

This method defines the procedure for extracting water-soluble forms of the following trace elements : boron, cobalt, copper, iron, manganese, molybdenum and zinc. The aim is to carry out the minimum number of extractions, making use wherever possible of the same extract to determine the level of each of the trace elements listed above.

2.   FIELD OF APPLICATION

This procedure concerns EEC fertilizers covered by Directive 89/530/EEC containing one or more of the following trace elements : boron, cobalt, copper, iron, manganese, molybdenum and zinc. It is applicable to each trace element the declared content of which is less than or equal to 10 %.

3.   PRINCIPLE

The trace elements are extracted by shaking the fertilizer in water at 20 °C ± 2 °C.

Note : The extraction is empirical and may or may not be quantitative.

4.   REAGENTS

4.1.   Dilute hydrochloric acid (HCI) solution, about 6 M :

Mix 1 volume of hydrochloric acid (ρ = 1,18 g/ml) with 1 volume of water.

5.   APPARATUS

5.1.   Rotary shaker set at about 35 to 40 rpm.

5.2.   pH-meter.

Note : Where the boron content of the extract is to be determined, do not use borosilicate glassware. Teflon or silica is preferable for this extraction. Rinse the glassware thoroughly if it has been washed in detergents containing borates.

6.   PREPARATION OF THE SAMPLE

See Method 1 (Directive 77/535/EEC, (OJ No L 213, 22. 8. 1977, P. 1)).

7.   PROCEDURE

7.1.   Test sample

Take a quantity of fertilizer weighing between 2 and 10 g depending on the declared content of the element in the product. The following table shall be used to obtain a final solution which, after appropriate dilution, will be within the measuring range for each method. The samples should be weighed to within 1 mg.

Declared content of trace element in the fertilizer (%)	<0,01	0,01-<5	≥5-10
Mass of test sample (g)	10	5	2
Mass of element in the test sample (mg)	1	0,5-250	100-200
Volume of extract V (ml)	250	500	500
Concentration of element in extract (mg/l)	4	1-500	200-400

Place the sample in a 250 or 500 ml flask (according to the Table).

7.2.   Preparation of the solution

Add about 200 ml of water to the 250 ml flask or 400 ml of water to the 500 ml flask.

Stopper the flask well. Shake vigorously by hand to disperse the sample, then place the flask on the shaker and shake for 30 minutes.

Make up to volume with water and mix thoroughly.

7.3.   Preparation of the test solution

Filter immediately into a clean, dry flask. Stopper the flask. Carry out the determination immediately after filtering.

NB : If the filtrate gradually becomes cloudy, make another extraction following 7.1 and 7.2 in a flask of volume Ve. Filter into a calibrated flask of volume W which has previously been dried and has received 5,00 ml of dilute hydrochloric acid (4.1). Stop the filtration at the exact moment when the calibration mark is reached. Mix thoroughly.

Under these conditions the value of V in the expression of results is :

$$V = Ve \times W / (W - 5).$$

The dilutions in the expression of results depend on this value of V.

8.   DETERMINATION

The determination of each trace element is carried out on the aliquot portions indicated in the method for each individual trace element.

If necessary, remove organic chelating or complexing substances from an aliquot portion by using Method 9.3. In the case of determination by atomic absorption spectrometry, such removal may not be necessary.

Method 9.3

REMOVAL OF ORGANIC COMPOUNDS FROM FERTILIZER EXTRACTS

1.   SCOPE

This method defines a procedure for removing organic compounds from fertilizer extracts.

2.   FIELD OF APPLICATION

This procedure is applicable to analysing samples of fertilizers extracted by Methods 9.1 and 9.2 for which a declaration of total and/or water-soluble element is required by Directive 89/530/EEC.

Note : The presence of small quantities of organic matter usually does not affect determinations by means of atomic absorption spectrometry.

3. PRINCIPLE

The organic compounds in an aliquot portion of the extract are oxidized with hydrogen peroxide.

4. REAGENTS

4.1. Dilute hydrochloric acid (HCl) solution, about 0,5 M :

Mix 1 volume of hydrochloric acid (ρ = 1,18 g/ml) with 20 volumes of water.

4.2. Hydrogen peroxide solution (30 % H_2O_2, ρ = 1,11 g/ml), free from trace elements

5. APPARATUS

Electric hotplate with variable temperature control.

6. PROCEDURE

Take 25 ml of the extract solution obtained by Method 9.1 or Method 9.2 and place in a 100 ml beaker. In the case of Method 9.2, add 5 ml of the dilute hydrochloric acid solution (4.1). Then add 5 ml of the hydrogen peroxide solution (4.2). Cover with a watchglass. Allow oxidation to occur at room temperature for about one hour, then bring gradually to boiling and boil for half an hour. If necessary, add a further 5 ml of the hydrogen peroxide to the solution once it has cooled. Then boil to remove the excess hydrogen peroxide. Allow to cool and transfer quantitatively to a 50 ml volumetric flask and make up to volume. Filter where necessary.

Account should be taken of this dilution when taking aliquot portions and calculating the percentage of trace element in the product.

Method 9.4

DETERMINATION OF TRACE ELEMENTS IN FERTILIZER EXTRACTS BY ATOMIC ABSORPTION SPECTROMETRY

(GENERAL PROCEDURE)

1. SCOPE

This document defines a general procedure for determining the levels of certain trace elements in fertilizer extracts by atomic absorption spectrometry.

2. FIELD OF APPLICATION

This procedure is applicable to analysing samples of fertilizers extracted by Methods 9.1 and 9.2 for which a declaration of total and/or water-soluble element is required by Directive 89/530/EEC.

Adaptations of this procedure for the various trace elements are detailed in the methods defined specifically for each element.

Note : In most cases the presence of small quantities of organic matter will not affect determinations by atomic absorption spectrometry.

3. PRINCIPLE

After the extract has been treated where necessary to reduce or eliminate interfering chemical species, the extract is diluted so that its concentration is in the optimum range of the spectrometer at a wavelength suitable for the trace element to be determined.

4. REAGENTS

4.1. Dilute hydrochloric acid solution (HCl), about 6 M :

Mix one volume of hydrochloric acid (ρ = 1,18 g/ml) with 1 volume of water.

4.2. Dilute hydrochloric acid solution (HCl), about 0,5 M :

Mix one volume of hydrochloric acid (ρ = 1,18 g/ml) with 20 volumes of water.

4.3. Lanthanum salt solutions (10 g of La per litre).

This reagent is used for determinations of cobalt, iron, manganese and zinc. It can be prepared either:

(a) with lanthanum oxide dissolved in hydrochloric acid (4.1). Place 11,73 g of lanthanum oxide (La_2O_3) in 150 ml of water in a 1 litre volumetric flask and add 120 ml of 6 M hydrochloric acid (4.1). Allow to dissolve and then make up to 1 litre with water and mix thoroughly. This solution is approximately 0,5 M in hydrochloric acid; or

(b) with solutions of lanthanum chloride, sulphate or nitrate. Dissolve 26,7 g of lanthanum chloride heptahydrate ($LaCl_3$ $7H_2O$) or 31,2 g of lanthanum nitrate hexahydrate [$La(NO_3)_3$ $6H_2O$] or 26,2 g of lanthanum sulphate nonahydrate ($La_2(SO_4)_3$ $9H_2O$] in 150 ml of water, then add 85 ml of 6 M hydrochloric acid (4.1). Allow to dissolve and then make up to 1 litre with water. Mix thoroughly. This solution is approximately 0,5 M in hydrochloric acid.

4.4. Calibration solutions

For the preparation of these, see the individual determination method for each trace element.

5. APPARATUS

Atomic absorption spectrometer fitted with sources emitting radiation characteristic of the trace elements to be determined.

The analyst must follow the manufacturer's instructions and be familiar with the apparatus. The apparatus must allow background correction so that it can be used whenever necessary (Co and Zn). The gases to be used are air and acetylene.

6. PREPARATION OF THE SOLUTION TO BE ANALYSED

6.1. Preparation of extract solutions of the trace elements to be determined

See Method 9.1 and/or 9.2 and, if appropriate, 9.3.

6.2. Treatment of the test solution

Dilute an aliquot portion of the extract obtained by Method 9.1, 9.2 or 9.3 with water and/or hydrochloric acid (4.1) or (4.2) so as to obtain, in the final solution for measurement, a concentration of the element to be determined that is appropriate to the calibration range used (7.2) and a hydrochloric acid concentration of at least 0,5 M and not more than 2,5 M. This operation may require one or more successive dilutions.

Take an aliquot portion of the final solution obtained by dilution of the extract, let (a) be its volume in ml, and pour into a 100 ml volumetric flask. When determining the cobalt, iron, manganese or zinc content, add 10 ml of the lanthanum salt solution (4.3). Make up to volume with the 0,5 M hydrochloric acid solution (4.2) and mix thoroughly. This is the final solution for measurement. Let D be the dilution factor.

7. PROCEDURE

7.1. Preparation of a blank solution

Prepare a blank solution by repeating the whole procedure from the extraction stage, omitting only the test sample of fertilizer.

7.2. Preparation of calibration solutions

From the working calibration solution prepared using the method given for each individual trace element, prepare in 100 ml volumetric flasks a series of at least five calibration solutions of increasing concentration within the optimum measuring range of the spectrometer. If necessary, adjust the concentration of hydrochloric acid to bring it as close as possible to that of the diluted test solution (6.2). For determining cobalt, iron, manganese or zinc add 10 ml of the same lanthanum salt solution (4.3) as used in 6.2. Make up to volume with the 0,5 M hydrochloric acid solution (4.2) and mix thoroughly.

7.3. Determination

Prepare the spectrometer (5) for the determination and adjust to the wavelength given in the method for the individual trace element concerned.

Spray three times in succession the calibration solutions (7.2), the test solution (6.2) and the blank solution (7.1), noting each result and flushing the instrument with distilled water between individual sprayings.

Construct the calibration curve by plotting the average spectrometer reading for each calibration solution (7.2) along the ordinate and the corresponding concentration of the element, expressed in μg/ml, along the abscissa.

From this curve, determine the concentrations of the relevant trace element in the test solution x_s (6.2) and in the blank solution x_b (7.1), expressing these concentrations in μg per ml.

8. EXPRESSION OF RESULTS

The percentage of trace element (E) in the fertilizer is equal to:

$$E \ (\%) = [(x_s - x_b) \times V \times D] / (M \times 10^4).$$

If method (9.3) has been used:

$$E \ (\%) = [(x_s - x_b) \times V \times 2D] / (M \times 10^4),$$

where:

E   is the amount of the trace element determind, expressed as a percentage of the fertilizer;

x_s is the concentration of the test solution (6.2), in μg/ml;

x_b is the concentration of the blank solution (7.1), in μg/ml;

V   is the volume of the extract obtained by Method 9.1 or 9.2, in ml;

D   is the factor corresponding to the dilution carried out in (6.2);

M   is the mass of the test sample taken in accordance with Method 9.1 or 9.2, in grams.

Calculation of dilution factor D:

If (a1), (a2), (a3), ., ., ., (ai) and (a) are the aliquot portions and (v1), (v2), (v3), ., ., ., (vi) and (100) are the volumes in ml corresponding to their respective dilutions, the dilution factor D will be equal to:

$$D = (v1/a1) \times (v2/a2) \times (v3/a3) \times . \times . \times . \times (vi/ai) \times (100/a).$$

Method 9.5

DETERMINATION OF BORON IN FERTILIZER EXTRACTS BY MEANS OF SPECTROPHOTOMETRY WITH AZOMETHINE-H

1. SCOPE

This method describes a procedure for determining boron in fertilizer extracts.

2. FIELD OF APPLICATION

This procedure is applicable to analysing samples of fertilizers extracted by Methods 9.1 and 9.2 for which a declaration of total and/or water-soluble boron is required by Directive 89/530/EEC.

3. PRINCIPLE

In an azomethine-H solution, borate ions form a yellow complex the concentration of which is determined by molecular absorption spectrometry at 410 nm. Interfering ions are masked with EDTA.

4. REAGENTS

4.1. EDTA buffer solution

Place in a 500 ml volumetric flask containing 300 ml of water:

— 75 g of ammonium acetate (NH_4OOCCH_3);

— 10 g of disodium salt of ethylene diamine tetraacetic acid (Na_2EDTA);

— 40 ml of acetic acid (CH_3COOH, $\rho = 1,05$ g/ml).

Make up to volume with water and mix thoroughly. The pH of the solution, checked by means of a glass electrode, must be $4,8 \pm 0,1$.

4.2. Azomethine-H solution

Place in a 200 ml volumetric flask

— 10 ml of the buffer solution (4.1);
— 400 mg of azomethine-H ($C_{17}H_{12}NNaO_8S_2$);
— 2 g of asorbic acid ($C_6H_8O_6$).

Make up to volume and mix thoroughly. Do not prepare large quantities of this reagent as it is stable for only a few days.

4.3. Boron calibration solutions

4.3.1. Boron stock solution (100 µg/ml)

Dissolve 0,5719 g of boric acid (H_3BO_3) in water in a 1 000 ml volumetric flask. Make up to volume with water and mix thoroughly. Transfer to a plastic bottle for storage in a refrigerator.

4.3.2. Boron working solution (10 µg/ml)

Place 50 ml of stock solution (4.3.1) in a 500 ml volumetric flask. Make up to volume with water and mix thoroughly.

5. APPARATUS

Spectrometer fitted for molecular absorption with cells having a 10 mm optical path and set to a wavelength of 410 nm.

6. PREPARATION OF THE SOLUTION TO BE ANALYSED

6.1. Preparation of the boron solution

See Methods 9.1 and/or 9.2 and, if appropriate, 9.3.

6.2. Preparation of the test solution

Dilute an aliquot portion of extract (6.1) to obtain a boron concentration as specified in 7.2. Two successive dilutions may be necessary. Let D be the dilution factor.

6.3. Preparation of the correction solution

If the test solution (6.2) is coloured, prepare a corresponding correction solution by placing in a plastic flask 5 ml of test solution (6.2), 5 ml of EDTA buffer solution (4.1) and 5 ml of water and mix thoroughly.

7. PROCEDURE

7.1. Preparation of the blank solution

Prepare a blank solution by repeating the whole procedure from the extraction stage, omitting only the test sample of fertilizer.

7.2. Preparation of the calibration solutions

Transfer 0, 5, 10, 15, 20 and 25 ml of the working calibration solution (4.3.2) to a series of 100 ml volumetric flasks. Make up to 100 ml with water and mix thoroughly. These solutions contain between 0 and 2,5 µg/ml of boron.

7.3. Colour development

Transfer 5 ml of the calibration solutions (7.2), test solutions (6.2) and blank (7.1) to a series of plastic flasks. Add 5 ml of the EDTA buffer solution (4.1). Add 5 ml of the azomethine-H solution (4.2).

Mix thoroughly and allow the colour to develop in the dark for 2 $^1/_2$ to three hours.

7.4. Determination

Measure the absorbance of the solutions obtained at 7.3 and if appropriate the correction solution (6.3) against water at a wavelength of 410 nm. Rinse the cells with water before each new reading.

8. EXPRESSION OF RESULTS

Plot a calibration curve of the concentration of the calibration solutions (7.2) along the abscissa and the absorbance given by the spectrophotometer (7.4) along the ordinate.

Read off the calibration curve the concentration of boron in the blank (7.1), the concentration of boron in the test solution (6.2) and, if the test solution is coloured, the corrected concentration of the test solution. To calculate the latter, subtract the absorbance of the correction solution (6.3) from the absorbance of the test solution (6.2) and determine the corrected concentration of the test solution. Note the concentration of the test solution (6.2), with or without correction, $X(x_s)$ and of the blank (x_b).

The percentage of boron in the fertilizer is given by:

$$B \% = [(x_s - x_b) \times V \times D] / (M \times 10^4)$$

If Method 9.3 is used:

$$B \% = [(x_s - x_b) \times V \times 2D] / (M \times 10^4)$$

where:

B   is the quantity of boron expressed as a percentage of the fertilizer;

x_s is the concentration (μg/ml) in the test solution (6.2), with or without correction;

x_b is the concentration (μg/ml) in the blank (7.1);

V   is the volume in ml of extract obtained in accordance with Method 9.1 or 9.2;

D   is the factor corresponding to the dilution carried out in 6.2;

M   is the mass in grams of the test sample taken in accordance with Method 9.1 or 9.2.

Calculation of the dilution factor D : if (a1) and (a2) are succesive aliquot portions and (v1) and (v2) are the volumes corresponding to their respective dilutions, the dilution factor D is given by:

$$D = (v1/a1) \times (v2/a2).$$

Method 9.6

DETERMINATION OF COBALT IN FERTILIZER EXTRACTS BY ATOMIC ABSORPTION SPECTRO-
METRY

1. SCOPE

This method describes a procedure for determining cobalt in fertilizer extracts.

2. FIELD OF APPLICATION

This procedure is applicable to analysing samples of fertilizers extracted by Methods 9.1 and 9.2 for which a declaration of total and/or water-soluble cobalt is required by Directive 89/530/EEC.

3. PRINCIPLE

After suitable treatment and dilution of the extracts, the cobalt content is determined by atomic absorption spectrometry.

4. REAGENTS

4.1. Hydrochloric acid solution, about 6 M.

See Method 9.4 (4.1).

4.2. Hydrochloric acid solution, about 0,5 M.

See Method 9.4 (4.2).

4.3. Lanthanum salt solutions (10 g of La per litre)

See Method 9.4 (4.3).

4.4. Cobalt calibration solutions

4.4.1. Cobalt stock solution (1 000 µg/ml)

In a 250 ml beaker, weigh to the nearest 0,1 mg, 1 g of cobalt, add 25 ml of 6 M hydrochloric acid (4.1) and heat on a hotplate until the cobalt is completely dissolved. When cool, transfer quantitatively to a 1 000 ml volumetric flask. Make up to volume with water and mix thoroughly.

4.4.2. Cobalt working solution (100 µg/ml)

Place 10 ml of the stock solution (4.4.1) in a 100 ml volumetric flask. Make up to volume with 0,5 M hydrochloric acid solution (4.2) and mix thoroughly.

5. APPARATUS

Atomic absorption spectrometer : see Method 9.4, (5). The instrument must be equipped with a source of rays characteristic of cobalt (240,7 nm). The spectrometer must allow background correction to be made.

6. PREPARATION OF THE SOLUTION TO BE ANALYSED

6.1. Cobalt extract solution

See Methods 9.1 and/or 9.2 and, if appropriate, 9.3.

6.2. Preparation of the test solution

See Method 9.4 (6.2). The test solution must contain 10 % (v/v) of a lanthanum salt solution (4.3).

7. PROCEDURE

7.1. Preparation of blank solution

See Method 9.4 (7.1). The blank must contain 10 % (v/v) of the lanthanum salt solution used in 6.2.

7.2. Preparation of calibration solutions

See Method 9.4 (7.2).

For an optimum determination range of 0 to 5 µg/ml of cobalt, place 0, 0,5, 1, 2, 3, 4 and 5 ml respectively of working solution (4.4.2) in a series of 100 ml volumetric flasks. If necessary adjust the hydrochloric acid concentration as closely as possible to that of the test solution. Add to each flask 10 ml of the lanthanum salt solution used in 6.2. Make up to 100 ml with 0,5 M hydrochloric acid solution (4.2) and mix thoroughly. These solutions contain 0, 0,5, 1, 2, 3, 4 and 5 µg/ml respectively of cobalt.

7.3. Determination

See Method 9.4 (7.3). Prepare the spectrometer (5) for measurement at a wavelength of 240,7 nm.

8. EXPRESSION OF RESULTS

See Method 9.4 (8).

The percentage of cobalt in the fertilizer is given by :

$$Co \% = [(x_s - x_b) \times V \times D] / (M \times 10^4)$$

If Method 9.3 is used :

$$Co \% = [(x_s - x_b) \times V \times 2D] / (M \times 10^4)$$

where :

Co   is the quantity of cobalt expressed as a percentage of the fertilizer;

x_s is the concentration in µg/ml of the test solution (6.2);

x_b is the concentration in µg/ml of the blank solution (7.1);

V   is the volume in ml of extract obtained in accordance with Method 9.1 or 9.2;

D   is the factor corresponding to the dilution carried out in 6.2;

M   is the mass in grams of the test sample taken in accordance with Method 9.1 or 9.2.

Calculation of the dilution factor D : if (a1), (a2), (a3), ., ., ., (ai) and (a) are aliquot portions and (v1), (v2), (v3), ., ., ., (vi) and (100) are the volumes in ml corresponding to their respective dilutions, the dilution factor D is given by :

$$D = (v1/a1) \times (v2/a2) \times (v3/a3) \times.\times.\times.\times. \times (vi/ai) \times (100/a).$$

Method 9.7

DETERMINATION OF COPPER IN FERTILIZER EXTRACTS BY ATOMIC ABSORPTION SPECTRO-METRY

1. SCOPE

   This method describes a procedure for determining copper in fertilizer extracts.

2. FIELD OF APPLICATION

   This procedure is applicable to analysing samples of fertilizers extracted by Methods 9.1 and 9.2 for which a declaration of total and/or water-soluble copper is required by Directive 89/530/EEC.

3. PRINCIPLE

   After suitable treatment and dilution of the extracts, the copper content is determined by atomic absorption spectrometry.

4. REAGENTS

4.1. Hydrochloric acid solution, about 6 M

   See Method 9.4, (4.1).

4.2. Hydrochloric acid solution, about 0,5 M

   See Method 9.4, (4.2).

4.3. Hydrogen peroxide solution (30 % H_2O, $\rho = 1,11$ g/ml), free from trace elements.

4.4. Copper calibration solutions

4.4.1. Copper stock solution (1 000 µg/ml)

   In a 250 ml beaker weigh to the nearest 0,1 mg, 1g of copper, add 25 ml of 6M hydrochloric acid (4.1) add 5 ml hydrogen peroxide solution (4.3) and heat on a hotplate until the copper is completely dissolved. Transfer quantiatively to a 1 000 ml volumetric flask. Make up to volume with water and mix thoroughly.

4.4.2. Copper working solution (100 µg/ml)

   Place 20 ml of the stock solution (4.4.1) in a 200 ml volumetric flask. Make up to volume with 0,5 M hydrochloric acid solution (4.2) and mix thoroughly.

5. APPARATUS

   Spectrometer equipped for atomic absorption : see Method 9.4 (5). The instrument must be fitted with a source of rays characteristics of copper (324,8 nm).

6. PREPARATION OF THE SOLUTION TO BE ANALYSED

6.1. Copper extract solution

   See Methods 9.1 and/or 9.2 and, if appropriate, 9.3.

6.2. Preparation of the test solution

   See Method 9.4, (6.2).

7. PROCEDURE

7.1. Preparation of blank solution

   See Method 9.4, (7.1).

7.2. Preparation of calibration solutions

   See Method 9.4, (7.2).

   For an optimum determination range of 0 to 5 µg/ml of copper, place 0, 0,5, 1, 2, 3, 4 and 5 ml respectively of working solution (4.4.2) in a series of 100 ml volumetric flasks. If necessary adjust the hydrochloric acid concentration as closely as possible to that of the test solution (6.2). Make up to 100 ml with 0,5 M hydrochloric acid solution (4.2) and mix thoroughly. These solutions contain 0, 0,5, 1, 2, 3, 4 and 5 µg/ml respectively of copper.

7.3. Determination

See Method 9.4, (7.3). Prepare the spectrometer (5) for measurement at a wavelength of 324,8 nm.

8. EXPRESSION OF RESULTS

See Method 9.4, (8).

The percentage of copper in the fertilizer is given by:

$$Cu \% = [(x_s - x_b) \times V \times D] / (M \times 10^4)$$

If Method 9.3 is used:

$$Cu \% = [(x_s - x_b) \times V \times 2D] / (M \times 10^4)$$

where:

Cu   is the quantity of copper expressed as a percentage of the fertilizer;

x_s is the concentration in $\mu g/ml$ of the test solution (6.2);

X_b is the concentration in $\mu g/ml$ of the blank solution (7.1);

V   is the volume in ml of extract obtained in accordance with Method 9.1 or 9.2;

D   is the factor of the dilution carried out in 6.2;

M   is the mass in grams of the test sample taken in accordance with Method 9.1 or 9.2.

Calculation of the dilution factor D : if (a1), (a2), (a3) ., ., ., (ai) and (a) are aliquot portions and (v1), (v2), (v3), ., ., ., (vi) and (100) are the volumes in ml corresponding to their respective dilutions, the dilution factor D is given by:

$$D = (v1/a1) \times (v2/a2) \times (v3/a3) \times . \times . \times . \times (vi/ai) \times (100/a)$$

Method 9.8

DETERMINATION OF IRON IN FERTILIZER EXTRACTS BY ATOMIC ABSORPTION SPECTRO-
METRY

1. SCOPE

This method describes a procedure for determining iron in fertilizer extracts.

2. FIELD OF APPLICATION

This procedure is applicable to analysing samples of fertilizers extracted by Methods 9.1 and 9.2 for which a declaration of total and/or water-soluble iron is required by Directive 89/530/EEC.

3. PRINCIPLE

After suitable treatment and dilution of the extract, the iron content is determined by atomic absorption spectrometry.

4. REAGENTS

4.1. Hydrochloric acid solution, about 6 M

See Method 9.4 (4.1).

4.2. Hydrochloric acid solution, about 0,5 M

See Method 9.4 (4.2).

4.3. Hydrogen peroxide solution (30 % H_2O_2, $\rho = 1,11$ g/ml) free from trace elements.

4.4. Lanthanum salt solutions (10 g of La per litre)

See Method 9.4, (4.3).

4.5. Iron calibration solutions

4.5.1. Iron stock solution (1 000 µg/ml)

In a 500 ml beaker, weigh to the nearest 0,1 mg, 1 g of pure iron wire, add 200 ml of 6 M hydrochloric acid (4.1) and 15 ml of hydrogen peroxide solution (4.3). Heat on a hotplate until the iron is completely dissolved. When cool, transfer quantitatively to a 1 000 ml volumetric flask. Make up to volume with water and mix thoroughly.

4.5.2. Iron working solution (100 µg/ml)

Place 20 ml of the stock solution (4.5.1) in a 200 ml volumetric flask. Make up to volume with the 0,5 M hydrochloric acid solution (4.2) and mix thoroughly.

5. APPARATUS

Atomic absorption spectrometer : see Method 9.4 (5). The instrument must be fitted with a source of rays characteristic of iron (248,3 nm).

6. PREPARATION OF THE SOLUTION TO BE ANALYSED

6.1. Iron extract solution

See Methods 9.1 and/or 9.2 and, if appropriate, 9.3.

6.2. Preparation of the test solution

See Method 9.4 (6.2). The test solution must contain 10 % (v/v) of a lanthanum salt solution.

7. PROCEDURE

7.1. Preparation of blank solution

See Method 9.4 (7.1). The test solution must contain 10 % (v/v) of the lanthanum salt solution used in 6.2.

7.2. Preparation of calibration solutions

See Method 9.4 (7.2).

For an optimum determination range of 0 to 10 µg/ml of iron, place 0, 2, 4, 6, 8 and 10 ml respectively of working solution (4.5.2) in a series of 100 ml volumetric flasks. If necessary adjust the hydrochloric acid concentration as closely as possible to that of the test solution. Add 10 ml of the lanthanum salt solution used in 6.2. Make up to volume with 0,5 M hydrochloric solution (4.2) and mix thoroughly. These solutions contain 0, 2, 4, 6, 8 and 10 µg/ml respectively of iron.

7.3. Determination

See Method 9.4 (7.3). Prepare the spectrometer (5) for measurement at a wavelength of 248,3 nm.

8. EXPRESSION OF RESULTS

See Method 9.4 (8).

The percentage of iron in the fertilizer is given by :

$$Fe \% = [(x_s - x_b) \times V \times D)] / (M \times 10^4)$$

If Method 9.3 is used :

$$Fe \% = [(x_s - x_b) \times V \times 2D)] / (M \times 10^4)$$

where :

F_e is the quantity of iron expressed as a percentage of the fertilizer ;

x_s is the concentration in µg/ml of the test solution (6.2) ;

x_b is the concentration in µg/ml of the blank solution (7.1) ;

V   is the volume in ml of extract obtained in accordance with Method 9.1 or 9.2 ;

D   is the factor of the dilution carried out in 6.2 ;

M   is the mass in grams of the test sample taken in accordance with Method 9.1 or 9.2.

Calculation of the dilution factor D : if (a1), (a2), (a3), ., ., ., (ai) and (a) are aliquot portions and (v1), (v2), (v3), ., ., ., (vi) and (100) are the volumes in ml corresponding to their respective dilutions, the dilution factor D is given by :

$$D = (v1/a1) \times (v2/a2) \times (v3/a3) \times . \times . \times . \times (vi/ai) \times (100/a).$$

Method 9.9

DETERMINATION OF MANGANESE IN FERTILIZER EXTRACTS BY ATOMIC ABSORPTION SPECTROMETRY

1. SCOPE

    This method describes a procedure for determining manganese in fertilizer extracts.

2. FIELD OF APPLICATION

    This procedure is applicable to analysing samples of fertilizers extracted by Methods 9.1 and 9.2 for which a declaration of total and/or water-soluble manganese is required by Directive 89/530/EEC.

3. PRINCIPLE

    After suitable treatment and dilution of the extracts, the manganese level is determined by atomic absorption spectrometry.

4. REAGENTS

4.1. Hydrochloric acid solution, about 6 M

    See Method 9.4, (4.1).

4.2. Hydrochloric acid solution, about 0,5 M

    See Method 9.4, (4.2).

4.3. Lanthanum salt solutions (10 g of La per litre)

    See Method 9.4, (4.3).

4.4. Manganese calibration solutions

4.4.1. Manganese stock solution (1 000 µg/ml)

    In a 250 ml beaker, weigh to the nearest 0,1 mg, 1 g of manganese, add 25 ml of 6 M hydrochloric acid solution (4.1). Heat on a hotplate until the manganese is completely dissolved. When cool, transfer quantitatively to a 1 000 ml volumetric flask. Make up to volume with water and mix thoroughly.

4.4.2. Manganese working solution (100 µg/ml)

    Dilute 20 ml of the stock solution (4.4.1) in the 0,5 M hydrochloric acid solution (4.2) in a 200 ml volumetric flask. Make up to volume with the 0,5 M hydrochloric acid solution (4.2) and mix thoroughly.

5. APPARATUS

    Atomic absorption spectrometer : see Method 9.4 (5). The apparatus must be fitted with a source of lines characteristic of manganese (279,6 nm).

6. PREPARATION OF THE SOLUTION TO BE ANALYSED

6.1. Manganese extract solution

    See Methods 9.1 and/or 9.2 and, if appropriate, 9.3.

6.2. Preparation of the test solution

    See Method 9.4, (6.2). The test solution must contain 10 % by volume of lanthanum salt solution (4.3).

7. PROCEDURE

7.1. Preparation of the blank solution

    See Method 9.4 (7.1). The blank solution must contain 10 % by volume of the lanthanum salt solution used in 6.2.

7.2.　Preparation of the calibration solutions

See Method 9.4 (7.2).

For an optimum interval of 0 to 5 µg/ml manganese, place 0, 0,5, 1, 2, 3, 4 and 5 ml, respectively, of the working solution (4.4.2) in a series of 100 ml volumetric flasks. Where necessary, adjust the hydrochloric acid concentration to bring it as close as possible to that of the test solution. To each flask add 10 ml of the lanthanum salt solution used in 6.2. Make up to 100 ml with the 0,5 M hydrochloric acid solution (4.2) and mix thoroughly. These solutions contain 0, 0,5, 1, 2, 3, 4 and 5 µg/ml manganese respectively.

7.3.　Determination

See Method 9.4 (7.3). Prepare the spectrometer (5) for measurements at a wavelength of 279,6 nm.

8.　EXPRESSION OF RESULTS

See Method 9.4 (8).

The percentage of manganese in the fertilizer is as follows:

$$Mn\ \% = [(x_s - x_b) \times V \times D] / (M \times 10^4)$$

If Method 9.3 has been used:

$$Mn\ \% = [(x_s - x_b) \times V \times 2D] / (M \times 10^4)$$

where:

Mn　is the quantity of manganese expressed as a percentage of the fertilizer;

x_s　is the concentration in µg/ml of the test solution (6.2);

x_b　is the concentration in µg/ml of the blank solution (7.1);

V　is the volume in ml of the extract obtained using Method 9.1 or 9.2;

D　is the factor corresponding to the dilution performed in 6.2;

M　is the mass in g of the test sample taken using Method 9.1 or 9.2.

Calculation of dilution factor D : where (a1), (a2), (a3), ., ., ., (ai) and (a) are aliquot portions and (v1), (v2), (v3), ., ., ., (vi) and (100) the volumes in ml corresponding to their respective dilutions, dilution factor D will be equal to:

$$D = (v1/a1) \times (v2/a2) \times (v3/a3) \times . \times . \times . \times (vi/ai) \times (100/a).$$

Method 9.10

DETERMINATION OF MOYLBDENUM IN FERTILIZER EXTRACTS BY SPECTOPHOTOMETRY OF A COMPLEX WITH AMMONIUM THIOCYANATE

1.　SCOPE

This method describes a procedure for determining molybdenum in fertilizer extracts.

2.　FIELD OF APPLICATION

This procedure is applicable to analysing samples of fertilizers extracted by Methods 9.1 and 9.2 for which a declaration of total and/or water-soluble molybdenum is required by Directive 89/530/EEC.

3.　PRINCIPLE

Molybdenum (V) forms a complex $[MoO(SCN)_5]$-- in an acid medium with SCN- ions.

The complex is extracted with n-butyl acetate. Interfering ions such as those of iron remain in the aqueous phase. The yellow-orange colour is determined by molecular absorption spectrometry at 470 nm.

4.　REAGENTS

4.1.　Dilute hydrochloric acid solution (HCl), about 6 M

See method 9.4 (4.1).

4.2.　Copper solution (70 mg/l) in 1,5 M hydrochloric acid

Dissolve 275 mg of copper sulphate ($CuSO_4$ $5H_2O$) weighed to within 0,1 mg in 250 ml of the 6 M hydrochloric acid solution (4.1) in a 1 000 ml volumetric flask. Make up to volume with water and mix thoroughly.

4.3. Ascorbic acid solution (50 g/l)

Dissolve 50 g of ascorbic acid ($C_6H_8O_6$) in water in a 1 000 ml volumetric flask. Make up to volume with water, mix thoroughly and keep in a refrigerator.

4.4. n-butyl acetate

4.5. Ammonium thiocyanate solution, 0,2 M

Dissolve 15,224 g of NH_4SCN in water in a 1 000 ml volumetric flask. Make up to volume with water; mix thoroughly and store in a dark-coloured bottle.

4.6. Stannous chloride solution (50 g/l) in 2 M hydrochloric acid

This solution must be perfectly clear and prepared immediately before use. Very pure stannous chloride must be used otherwise the solution will not be clear.

To prepare 100 ml of solution, dissolve 5 g of ($SnCl_2 2H_2O$) in 35 ml of 6 M HCl solution (4.1). Add 10 ml of the copper solution (4.2). Make up to volume with water and mix thoroughly.

4.7. Molybdenum calibration solutions

4.7.1. Molybdenum stock solution (500 µg/ml)

Dissolve 0,920 g of ammonium molybdate [($NH_4)_6Mo_7O_{24}$ $4H_2O$] weighed to within 0,1 mg in the 6 M hydrochloric acid (4.1) in a 1 000 ml volumetric flask. Make up to volume with that solution and mix thoroughly.

4.7.2. Molybdenum intermediate solution (25 µg/ml)

Place 25 ml of the stock solution (4.7.1) in a 500 ml volumetric flask. Make up to volume with 6 M hydrochloric acid (4.1) and mix thoroughly.

4.7.3. Molybdenum working solution (2,5 µg/ml)

Place 10 ml of the intermediate solution (4.7.2) in a 100 ml volumetric flask. Make up to volume with 6 M hydrochloric acid (4.1) and mix thoroughly.

5. APPARATUS

5.1. Spectrometer fitted for molecular absorption with cuvettes having a 20 mm optical path and set to a wavelength of 470 nm.

5.2. 200 or 250 ml separating funnels.

6. PREPARATION OF THE SOLUTION TO BE ANALYSED

6.1. Molybdenum extract solution

See Methods 9.1 and/or 9.2 and, if appropriate, 9.3.

6.2. Preparation of the test solution

Dilute an aliquot portion of the extract (6.1) with 6 M hydrochloric acid solution (4.1) so as to obtain an appropriate molybdenum concentration. Let D be the dilution factor.

Take an aliquot portion (a) from the extract solution containing 1 to 12 µg molybdenum and place it in the separating funnel (5.2). Make up to 50 ml with the 6 M hydrochloric acid solution (4.1).

7. PROCEDURE

7.1. Preparation of the blank solution

Prepare a blank solution by repeating the whole procedure from the extraction stage, omitting only the test sample of fertilizer.

7.2. Preparation of the series of calibration solutions

Prepare a series of at least six calibration solutions of increasing concentration corresponding to the optimum response range of the spectrometer.

For the interval 0-12,5 µg molybdenum, place 0, 1, 2, 3, 4 and 5 ml, respectively, of the working solution (4.7.3) in the separating funnels (5.2). Make up to 50 ml with 6 M hydrochloric acid (4.1). The funnels contain, respectively, 0, 2,5, 5, 7,5, 10 and 12,5 µg molybdenum.

7.3. Development and separation of the complex

To each separating funnel (6.2, 7.1 and 7.2), add in the following order:

— 10 ml of the copper solution (4.2)
— 20 ml of the ascorbic acid solution (4.3);

mix thoroughly and wait for two or three minutes. Then add:

— 10 ml of n-butyl acetate (4.4), using a precision pipette;
— 20 ml of the thiocyanate solution (4.5).

Shake for one minute to extract the complex in the organic phase; allow to precipitate; after the separation of the two phases, draw off the entire aqueous phase and discard it; then wash the organic phase with:

—10 ml of the stannous chloride solution (4.6).

Shake for one minute. Allow to precipitate and draw off the entire aqueous phase. Collect the organic phase in a test tube; this will make it possible to collect the drops of water in suspension.

7.4. Determination

Measure the absorbencies of the solutions obtained at 7.3 at a wavelength of 470 nm using the 0 μg/ml molybdenum calibration solution (7.2) as a reference.

8. EXPRESSION OF RESULTS

Construct the calibration curve by plotting the corresponding masses of molybdenum in the calibration solutions (7.2) expressed in μg along the abscissa and the corresponding values of the absorbancies (7.4) given by the spectrometer reading along the ordinate.

From this curve determine the mass of molybdenum in the test solution (6.2) and the blank solution (7.1). These masses are designated (x_s) and (x_b) respectively.

The percentage of molybdenum in the fertilizer is:

$$Mo \% = [(x_s - x_b) \times V/a \times D] / (M \times 10^4)$$

If Method 9.3 has been used:

$$Mo \% = [(x_s - x_b) \times V/a \times 2D] / (M \times 10^4)$$

where:

Mo   is the quantity of molybdenum expressed as a percentage of the fertilizer;
a   is the volume in ml of the aliquot taken from the last dilute solution (6.2);
x_s is the Mo mass in μg in the test solution (6.2);
x_b is the Mo mass in μg in the blank solution (7.1) the volume of which corresponds to the volume (a) of the aliquot of the test solution (6.2);
V   is the volume in ml of the extract solution obtained in accordance with Method 9.1 or 9.2;
D   is the factor corresponding to the dilution performed in 6.2;
M   is the mass in g of the test sample taken in accordance with Method 9.1 or 9.2.

Calculation of the dilution factor D: where (a1), (a2) are successive aliquot portions and (v1), (v2) are the volumes corresponding to their respective dilutions, the dilution factor D will be:

$$D = (v1/a1) \times (v2/a2).$$

Method 9.11

DETERMINATION OF ZINC IN FERTILIZER EXTRACTS BY ATOMIC ABSORPTION SPECTRO-METRY

1. SCOPE

This method describes a procedure for determining zinc in fertilizer extracts.

2. FIELD OF APPLICATION

This procedure is applicable to analysing samples of fertilizers extracted by Methods 9.1 and 9.2 for which a declaration of total and/or water-soluble zinc is required by Directive 89/530/EEC.

3.   PRINCIPLE

After suitable treatment and dilution of the extracts, the zinc level is determined by atomic absorption spectrometry.

4.   REAGENTS

4.1.   Hydrochloric acid solution, about 6 M

See Method 9.4 (4.1).

4.2.   Hydrochloric acid solution, about 0,5 M

See Method 9.4 (4.2).

4.3.   Lanthanum salt solutions (10 g of La per litre)

See Method 9.4 (4.3).

4.4.   Zinc calibration solutions

4.4.1.   Zinc stock solution (1 000 µg/ml)

In a 1 000 ml volumetric flask dissolve 1 g of zinc powder or flakes weighed to within 0,1 mg in 25 ml of 6 M hydrochloric acid (4.1). When completely dissolved, make up to volume with water and mix thoroughly.

4.4.2.   Zinc working solution (100 µg/ml)

In a 200 ml volumetric flask, dilute 20 ml of the stock solution (4.4.1) in 0,5 M hydrochloric acid solution (4.2). Make up to volume with the 0,5 M hydrochloric acid solution and mix thoroughly.

5.   APPARATUS

Atomic absorption spectrometer : See Method 9.4, (5). The apparatus must be fitted with a source of lines characteristic of zinc (213,8 nm). the spectrometer must allow background correction to be made.

6.   PREPARATION OF THE SOLUTION TO BE ANALYSED

6.1.   Zinc extract solution

See Methods 9.1 and/or 9.2 and, if appropriate, 9.3.

6.2.   Preparation of the test solution

See Method 9.4, (6.2). The test solution must contain 10 % by volume of lanthanum salt solution.

7.   PROCEDURE

7.1.   Preparation of the blank solution

See Method 9.4, (7.1). The blank solution must contain 10 % by volume of the lanthanum salt solution used in 6.2.

7.2.   Preparation of the calibration solutions

See Method 9.4, (7.2).

For an optimum interval of 0 to 5 µg/ml of zinc, place 0, 0,5, 1, 2, 3, 4 and 5 ml, respectively, of the working solution (4.4.2) in a series of 100 ml volumetric flasks. Where necessary, adjust the concentration of hydrochloric acid to bring it as close as possible to that of the test solution. Add 10 ml of the lanthanum salt solution used in (6.2) to each volumetric flask. Make up to 100 ml with the 0,5 M hydrochloric acid solution (4.2) and mix thoroughly. These solutions contain, respectively : 0, 0,5, 1, 2, 3, 4, and 5 µg/ml of zinc.

7.3.   Determination

See Method 9.4 (7.3). Prepare the spectrometer (5) for measurements at a wavelength of 213,8 nm.

8. EXPRESSION OF RESULTS

See Method 9.4 (8).

The percentage of zinc in the fertilizer is equal to :

$$Zn \% = [(x_s - x_b) \times V \times D] / (M \times 10^4)$$

If method 9.3 has been used :

$$Zn \% = [(x_s - x_b) \times V \times 2D] / (M \times 10^4)$$

where :

Z_n is the quantity of zinc expressed as a percentage of the fertilizer ;

x_s is the concentration in μg/ml of the test solution (6.2) ;

x_b is the concentration in μg/ml of the blank solution (7.1) ;

V is the volume in ml of the extract solution obtained in accordance with Method 9.1 or 9.2 ;

D is the factor corresponding to the dilution performed in (6.2) ;

M is the mass in g of the test sample taken in accordance with Method 9.1 or 9.2.

Calculation of the dilution factor D : where (a1), (a2), (a3), ., ., ., (ai) and (a) are successive aliquot portions and (v1), (v2), (v3), ., ., ., (vi) and (100) are the volumes corresponding to their respective dilutions, the dilution factor D will be :

$$D = (v1/a1) \times (v2/a2) \times (v3/a3) \times . \times . \times . \times (vi/ai) \times (100/a).'$$