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⁽¹⁾ Text with EEA relevance

I

(Acts whose publication is obligatory)

COMMISSION REGULATION (EC) No 1995/98
of 18 September 1998
establishing the standard import values for determining the entry price of certain
fruit and vegetables

THE COMMISSION OF THE EUROPEAN COMMUNITIES,
Having regard to the Treaty establishing the European Community,

Having regard to Commission Regulation (EC) No 3223/94 of 21 December 1994 on detailed rules for the application of the import arrangements for fruit and vegetables ⁽¹⁾, as last amended by Regulation (EC) No 1498/98 ⁽²⁾, and in particular Article 4 (1) 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⁾, as last amended by Regulation (EC) No 150/95 ⁽⁴⁾, and in particular Article 3 (3) thereof,

Whereas Regulation (EC) No 3223/94 lays down, pursuant to the outcome of the Uruguay Round multilateral trade negotiations, the criteria whereby the Commission fixes the standard values for imports from third

countries, in respect of the products and periods stipulated in the Annex thereto;

Whereas, in compliance with the above criteria, the standard import values must be fixed at the levels set out in the Annex to this Regulation,

HAS ADOPTED THIS REGULATION:

Article 1

The standard import values referred to in Article 4 of Regulation (EC) No 3223/94 shall be fixed as indicated in the Annex hereto.

Article 2

This Regulation shall enter into force on 19 September 1998.

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

Done at Brussels, 18 September 1998.

For the Commission

Franz FISCHLER

Member of the Commission

⁽¹⁾ OJ L 337, 24. 12. 1994, p. 66.

⁽²⁾ OJ L 198, 15. 7. 1998, p. 4.

⁽³⁾ OJ L 387, 31. 12. 1992, p. 1.

⁽⁴⁾ OJ L 22, 31. 1. 1995, p. 1.

ANNEX

to the Commission Regulation of 18 September 1998 establishing the standard import values for determining the entry price of certain fruit and vegetables

(ECU/100 kg)

CN code	Third country code ⁽¹⁾	Standard import value
0702 00 00	060	36,5
	999	36,5
0702 00 05	060	95,0
	999	95,0
0709 90 70	052	103,6
	999	103,6
0805 30 10	388	82,6
	512	81,0
	524	78,9
	528	72,1
	999	78,6
0806 10 10	052	92,4
	064	56,4
	400	193,4
	624	101,9
	999	111,0
0808 10 20, 0808 10 50, 0808 10 90	388	52,6
	400	66,1
	508	42,5
	512	80,3
	524	27,7
	528	86,6
	800	207,5
	999	80,5
0808 20 50	052	92,7
	064	55,2
	388	90,6
	528	86,7
0809 30 10, 0809 30 90	999	81,3
	052	122,5
	999	122,5
0809 40 05	052	54,6
	060	48,7
	064	59,6
	066	53,3
	068	50,8
	091	55,6
	400	109,0
	624	180,7
	999	76,5

⁽¹⁾ Country nomenclature as fixed by Commission Regulation (EC) No 2317/97 (OJ L 321, 22. 11. 1997, p. 19). Code '999' stands for 'of other origin'.

COMMISSION REGULATION (EC) No 1996/98
of 18 September 1998

on the issue of import licences for rice originating in the ACP States and the overseas countries and territories against applications submitted in the first five working days of September 1998 pursuant to Regulation (EC) No 2603/97

THE COMMISSION OF THE EUROPEAN COMMUNITIES,
Having regard to the Treaty establishing the European Community,

Having regard to Commission Regulation (EC) No 2603/97 of 16 December 1997 laying down the detailed rules of application for the import of rice from the ACP States and for the import of rice from the overseas countries and territories (OCT)⁽¹⁾, as amended by Regulation (EC) No 1595/98⁽²⁾, and in particular Article 9(2) thereof,

Whereas, pursuant to Article 9(2) of Regulation (EC) No 2603/97, the Commission must decide within 10 days of the final date for notification by the Member States the extent to which applications can be granted and must fix the available quantities for the following tranche and, where necessary, for the additional tranche for October;

Whereas examination of the quantities for which applications have been submitted shows that licences should be issued for the quantities applied for reduced, where

appropriate, by the percentages set out in the Annex hereto,

HAS ADOPTED THIS REGULATION:

Article 1

Import licences for rice against applications submitted during the first five working days of September 1998 pursuant to Regulation (EC) No 2603/97 and notified to the Commission shall be issued for the quantities applied for reduced, where appropriate, by the percentages set out in the Annex hereto.

Article 2

This Regulation shall enter into force on 19 September 1998.

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

Done at Brussels, 18 September 1998.

For the Commission

Franz FISCHLER

Member of the Commission

⁽¹⁾ OJ L 351, 23. 12. 1997, p. 22.

⁽²⁾ OJ L 208, 24. 7. 1998, p. 21.

*ANNEX***Regulation (EC) No 2603/97**

Reduction percentages to be applied to quantities applied for under the tranche for September 1998:

Origin	Reduction (%)
ACP (Article 2(1)) — CN codes 1006 10 21 to 1006 10 98, 1006 20 and 1006 30	32,4212 ⁽¹⁾
ACP + OCT (Article 7) — ACP: CN codes 1006 10 21 to 1006 10 98, 1006 20 and 1006 30 — OCT: CN code 1006	84,3306

⁽¹⁾ Issue for the quantity applied for.

COMMISSION REGULATION (EC) No 1997/98
of 18 September 1998
on issuing A2 export licences for fruit and vegetables

THE COMMISSION OF THE EUROPEAN COMMUNITIES,
Having regard to the Treaty establishing the European Community,

Having regard to Commission Regulation (EC) No 2190/96 of 14 November 1996 on detailed rules for implementing Council Regulation (EC) No 2200/96 as regards export refunds on fruit and vegetables ⁽¹⁾, as last amended by Regulation (EC) No 1287/98 ⁽²⁾, and in particular Article 3(4) thereof,

Whereas Commission Regulation (EC) No 1875/98 ⁽³⁾ sets the indicative refund rates and the indicative quantities for A2 export licences, other than those applied for in the context of food aid;

Whereas, for tomatoes, oranges, lemons and apples for destination group X in view of the economic situation in the various destination groups indicated in the Annex to Regulation (EC) No 1875/98 and taking account of information received by operators via their applications for A2 licences, the definitive refund rates should be set at a different rate from the indicative rates; whereas the percentages for the issuing of licences for the quantities applied for should also be set; whereas the definitive rates may not be more than double the indicative rates;

Whereas, pursuant to Article 3(5) of Regulation (EC) No 2190/96, applications for rates in excess of the cor-

responding definitive rates shall be considered null and void,

HAS ADOPTED THIS REGULATION:

Article 1

1. For A2 export licences for which applications have been submitted pursuant to Article 1 of Regulation (EC) No 1875/98, the actual date of application referred to in the second subparagraph of Article 3(1) of Regulation (EC) No 2190/96 is hereby set at 21 September 1998.

2. The licences referred to in the first paragraph shall be issued at the definitive refund rates and at the percentages for the quantities applied for as indicated in the Annex to this Regulation.

3. Pursuant to Article 3(5) of Regulation (EC) No 2190/96, applications referred to in the first paragraph for rates in excess of the corresponding definitive rates set out in the Annex shall be considered null and void.

Article 2

This Regulation shall enter into force on 21 September 1998.

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

Done at Brussels, 18 September 1998.

For the Commission

Franz FISCHLER

Member of the Commission

⁽¹⁾ OJ L 292, 15. 11. 1996, p. 12.

⁽²⁾ OJ L 178, 23. 6. 1998, p. 11.

⁽³⁾ OJ L 243, 2. 9. 1998, p. 3.

ANNEX

Product	Destination or group of destinations ⁽¹⁾	Definitive refund rates (ECU/tonne net)	Percentages for the issuing of licences
Tomatoes	F	30	100 %
Oranges	XYC	30	97 %
Lemons	F	10	100 %
Table grapes	F	25	100 %
Apples	X	24	100 %
	Y	—	—
Peaches and nectarines	E	—	—

⁽¹⁾ The destination codes are defined as follows:

X: Norway, Iceland, Greenland, Faeroes, Poland, Hungary, Romania, Bulgaria, Albania, Estonia, Latvia, Lithuania, Bosnia-Herzegovina, Croatia, Slovenia, Former Yugoslav Republic of Macedonia, Federal Republic of Yugoslavia (Serbia and Montenegro), Malta.

Y: Armenia, Azerbaijan, Belarus, Georgia, Kazakhstan, Kyrgyzstan, Moldova, Russia, Tajikistan, Turkmenistan, Uzbekistan, Ukraine, destinations mentioned in Article 34 of Commission Regulation (EEC) No 3665/87, as amended.

C: Switzerland, Czech Republic, Slovakia.

E: All destinations except Switzerland.

F: All destinations.

COMMISSION REGULATION (EC) No 1998/98
of 18 September 1998
on tenders submitted under the third invitation to tender for beef put up for sale
in Regulation (EC) No 1324/98

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Community,

Having regard to Council Regulation (EEC) No 805/68 of 27 June 1968 on the common organisation of the market in beef and veal ⁽¹⁾, as last amended by Regulation (EC) No 1633/98 ⁽²⁾, and in particular Article 7(3) thereof,

Whereas quantities of beef fixed by Commission Regulation (EC) No 1324/98 ⁽³⁾ have been offered for sale by periodic invitation to tender;

Whereas, pursuant to Article 9 of Commission Regulation (EEC) No 2173/79 ⁽⁴⁾, as last amended by Regulation (EC) No 2417/95 ⁽⁵⁾, minimum sales prices for the meat offered for tender are to be fixed in the light of the tenders received; whereas, for the third invitation to tender referred to in Article 2(1)(c) of Regulation (EC) No

1324/98, the tenders received do not allow minimum prices to be fixed;

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

No action shall be taken on the basis of the tenders submitted under the invitation to tender referred to in Article 2(1)(c) of Regulation (EC) No 1324/98.

Article 2

This Regulation shall enter into force on 19 September 1998.

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

Done at Brussels, 18 September 1998.

For the Commission

Franz FISCHLER

Member of the Commission

⁽¹⁾ OJ L 148, 28. 6. 1968, p. 24.

⁽²⁾ OJ L 210, 28. 7. 1998, p. 17.

⁽³⁾ OJ L 183, 26. 6. 1998, p. 38.

⁽⁴⁾ OJ L 251, 5. 10. 1979, p. 12.

⁽⁵⁾ OJ L 248, 14. 10. 1995, p. 39.

COMMISSION REGULATION (EC) No 1999/98
of 18 September 1998
fixing the minimum selling prices for beef put up for sale under the invitation to
tender referred to in Regulation (EC) No 1824/98

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Community,

Having regard to Council Regulation (EEC) No 805/68 of 27 June 1968 on the common organisation of the market in beef and veal ⁽¹⁾, as last amended by Regulation (EC) No 1633/98 ⁽²⁾, and in particular Article 7(3) thereof,

Whereas tenders have been invited for certain quantities of beef fixed by Commission Regulation (EC) No 1824/98 ⁽³⁾;

Whereas, pursuant to Article 9 of Commission Regulation (EEC) No 2173/79 ⁽⁴⁾, as last amended by Regulation (EC) No 2417/95 ⁽⁵⁾, the minimum selling prices for meat put up for sale by tender should be fixed, taking into account tenders submitted;

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

The minimum selling prices for beef for the invitation to tender held in accordance with Regulation (EC) No 1824/98 for which the time limit for the submission of tenders was 7 September 1998 are as set out in the Annex hereto.

Article 2

This Regulation shall enter into force on the day of 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, 18 September 1998.

For the Commission

Franz FISCHLER

Member of the Commission

⁽¹⁾ OJ L 148, 28. 6. 1968, p. 24.

⁽²⁾ OJ L 210, 28. 7. 1998, p. 17.

⁽³⁾ OJ L 236, 22. 8. 1998, p. 8.

⁽⁴⁾ OJ L 251, 5. 10. 1979, p. 12.

⁽⁵⁾ OJ L 248, 14. 10. 1995, p. 39.

*ANEXO — BILAG — ANHANG — ΠΑΡΑΡΤΗΜΑ — ANNEX — ANNEXE — ALLEGATO —
BIJLAGE — ANEXO — LIITE — BILAGA*

Estado miembro	Productos	Precio mínimo expresado en ecus por tonelada
Medlemsstat	Produkter	Mindestpreise i ECU/ton
Mitgliedstaat	Erzeugnisse	Mindestpreise, ausgedrückt in ECU/Tonne
Κράτος μέλος	Προϊόντα	Ελάχιστες πωλήσεις εκφραζόμενες σε Ecu ανά τόνο
Member State	Products	Minimum prices expressed in ECU per tonne
État membre	Produits	Prix minimaux exprimés en écus par tonne
Stato membro	Prodotti	Prezzi minimi espressi in ecu per tonnellata
Lidstaat	Producten	Minimumprijzen uitgedrukt in ECU per ton
Estado-membro	Produtos	Preço mínimo expresso em ecus por tonelada
Jäsenvaltio	Tuotteet	Vähimmäishinnat ecuina tonnia kohden ilmaistuna
Medlemsstat	Produkter	Minimipriser i ecu per ton

**Carne con hueso — Kød, ikke udbenet — Fleisch mit Knochen — Κρέατα με κόκαλα — Bone-in beef
— Viande avec os — Carni non disossate — Vlees met been — Carne corm osso — Luullinen
naudanliha — Kött med ben**

ESPAÑA	— Cuartos traseros	1 525
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COMMISSION REGULATION (EC) No 2000/98
of 18 September 1998
on the issuing of import licences for bananas under the tariff quota for the fourth
quarter of 1998 and on the submission of new applications
(Text with EEA relevance)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Community,

Having regard to Council Regulation (EEC) No 404/93 of 13 February 1993 on the common organisation of the market in bananas ⁽¹⁾, as last amended by Regulation (EC) No 1637/98 ⁽²⁾, and in particular Article 20 thereof,

Whereas Commission Regulation (EEC) No 1442/93 ⁽³⁾, as last amended by Regulation (EC) No 1409/96 ⁽⁴⁾, lays down detailed rules for the application of the arrangements for importing bananas into the Community; whereas Commission Regulation (EC) No 478/95 ⁽⁵⁾, as amended by Regulation (EC) No 702/95 ⁽⁶⁾, lays down additional rules for the application of the tariff quota arrangements laid down in Articles 18 and 19 of Regulation (EEC) No 404/93;

Whereas Article 9(3) of Regulation (EEC) No 1442/93 lays down that where, in the case of a given origin, for a country or group of countries referred to in Annex I to Regulation (EC) No 478/95, the quantities covered by import licence applications from one or more of the categories of operators exceed the quantity available, a reduction percentage to be applied to applications shall be set;

Whereas the quantities available for import under the tariff quota are laid down for the fourth quarter of 1998 in Commission Regulation (EC) No 1646/98 ⁽⁷⁾;

Whereas in the case of the quantities covered by licence applications that are either less than or equal to the quantities available, licences are issued for the quantities applied for; whereas, however, for certain origins, the quantities applied for exceed the quantities available set out in the Annex to Regulation (EC) No 1646/98; whereas, therefore, a reduction percentage should be set

to be applied to each licence application for the origin or origins involved and category of licence in question;

Whereas, the maximum quantity for which such licence applications may still be submitted should be set taking account of the available quantities fixed by Regulation (EC) No 1646/98 and the applications accepted at the end of the application period whereas Regulation (EC) No 478/95 on additional rules for the application of Regulation (EEC) No 404/93 as regards the tariff quota arrangements for imports of bananas into the Community and amending Regulation (EEC) No 1442/93 applies;

Whereas this Regulation should apply immediately to permit licences to be issued as quickly as possible;

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

HAS ADOPTED THIS REGULATION:

Article 1

Import licences shall be issued under the tariff quota arrangements for the import of bananas for the fourth quarter of 1998 for the quantity indicated in the licence application, multiplied by reduction coefficients of 0,6837, 0,7653, 0,4645 and 0,8904 for applications indicating the origins 'Columbia', 'Costa Rica', 'Dominican Republic' and 'Côte d'Ivoire' respectively.

Article 2

The quantities for which licence applications may still be lodged in respect of the fourth quarter of 1998 are laid down in the Annex hereto.

Article 3

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

⁽¹⁾ OJ L 47, 25. 2. 1993, p. 1.

⁽²⁾ OJ L 210, 28. 7. 1998, p. 28.

⁽³⁾ OJ L 142, 12. 6. 1993, p. 6.

⁽⁴⁾ OJ L 181, 20. 7. 1996, p. 13.

⁽⁵⁾ OJ L 49, 4. 3. 1995, p. 13.

⁽⁶⁾ OJ L 71, 31. 3. 1995, p. 84.

⁽⁷⁾ OJ L 210, 28. 7. 1998, p. 55.

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

Done at Brussels, 18 September 1998.

For the Commission

Franz FISCHLER

Member of the Commission

ANNEX

(in tonnes)

	Quantities available for new requests
NICARAGUA	48 351,916
VENEZUELA	23 837,117
OTHERS	83 290,442
BELIZE	7 950,000
CAMEROON	3 969,200
Other ACP	818,533

COMMISSION REGULATION (EC) No 2001/98
of 18 September 1998
providing for the rejection of applications for export licences in relation to cereal
products

THE COMMISSION OF THE EUROPEAN COMMUNITIES,
Having regard to the Treaty establishing the European Community,

Having regard to Council Regulation (EEC) No 1766/92 of 30 June 1992 on the common organisation of the market in cereals ⁽¹⁾, as last amended by Commission Regulation (EC) No 923/96 ⁽²⁾,

Having regard to Commission Regulation (EC) No 1162/95 of 23 May 1995 laying down special detailed rules for the application of the system of import and export licences for cereals and rice ⁽³⁾, as last amended by Regulation (EC) No 444/98 ⁽⁴⁾, and in particular Article 7(3) thereof,

Whereas the quantity covered by applications for advance fixing of refunds for flour of common wheat and spelt could give rise to speculation; whereas it has therefore

been decided to reject all applications for export licences for this product made on 17 September 1998,

HAS ADOPTED THIS REGULATION:

Article 1

In accordance with Article 7(3) of Regulation (EC) No 1162/95, applications for export licences with advance fixing of refunds for products falling within CN code 1101 00 15 made on 17 September 1998 shall be rejected.

Article 2

This Regulation shall enter into force on 19 September 1998.

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

Done at Brussels, 18 September 1998.

For the Commission

Franz FISCHLER

Member of the Commission

⁽¹⁾ OJ L 181, 1. 7. 1992, p. 21.

⁽²⁾ OJ L 126, 24. 5. 1996, p. 37.

⁽³⁾ OJ L 117, 24. 5. 1995, p. 2.

⁽⁴⁾ OJ L 56, 26. 2. 1998, p. 12.

COMMISSION DIRECTIVE 98/64/EC

of 3 September 1998

establishing Community methods of analysis for the determination of amino-acids, crude oils and fats, and olaquinox in feedingstuffs and amending Directive 71/393/EEC

(Text with EEA relevance)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Community,

Having regard to Council Directive 70/373/EEC of 20 July 1970 on the introduction of Community methods of sampling and analysis for the official control of feedingstuffs⁽¹⁾, as last amended by the Act of Accession of Austria, Finland and Sweden, and in particular Article 2 thereof,

Whereas Directive 70/373/EEC stipulates that official controls of feedingstuffs for the purpose of checking compliance with the requirements arising under the laws, regulations and administrative provisions governing their quality and composition must be carried out using Community methods of sampling and analysis;

Whereas Council Directive 79/373/EEC of 2 April 1979 on the marketing of compound feedingstuffs⁽²⁾, as last amended by Commission Directive 97/47/EC⁽³⁾, and Council Directive 93/74/EEC of 13 September 1993 on feedingstuffs intended for particular nutritional purposes⁽⁴⁾, as last amended by Directive 96/25/EC⁽⁵⁾, stipulate that amino-acids and crude oils and fats are to be set out on the feedingstuffs label;

Whereas Council Directive 70/524/EEC of 23 November 1970 concerning additives in feedingstuffs⁽⁶⁾, as last amended by Commission Directive 98/19/EC⁽⁷⁾, stipulates that the olaquinox content must be indicated on the labelling where this substance is added to compound feedingstuffs;

Whereas the second Commission Directive 71/393/EEC of 18 November 1971 establishing Community methods of analysis for the official control of feedingstuffs⁽⁸⁾, as last amended by Commission Directive 84/4/EEC⁽⁹⁾, sets

out methods for analysis for, *inter alia*, the determination of crude oils and fats; whereas it is appropriate to modify the method described;

Whereas Community methods of analysis must be established for checking these substances;

Whereas the measures provided for in this Directive are in accordance with the opinion of the Standing Committee for Feedingstuffs,

HAS ADOPTED THIS DIRECTIVE:

Article 1

The Member States shall provide that analyses conducted with a view to official controls of the amino-acid, crude oil and fat, and olaquinox content of feedingstuffs are carried out using the methods set out in the Annex hereto.

Article 2

In the Annex to Commission Directive 71/393/EEC, point '4. Determination of crude oils and fats' is replaced by part B of the annex to this Directive.

Article 3

1. Member States shall bring into force the laws, regulations or administrative provisions necessary to comply with this Directive by 31 December 1998. They shall immediately inform the Commission thereof.

They shall apply the measures from 1 January 1999.

When Member States adopt these measures, they 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 L 170, 3. 8. 1970, p. 2.

⁽²⁾ OJ L 86, 6. 4. 1979, p. 30.

⁽³⁾ OJ L 211, 5. 8. 1997, p. 45.

⁽⁴⁾ OJ L 237, 22. 9. 1993, p. 23.

⁽⁵⁾ OJ L 125, 23. 5. 1996, p. 35.

⁽⁶⁾ OJ L 270, 14. 12. 1970, p. 1.

⁽⁷⁾ OJ L 96, 28. 3. 1998, p. 39.

⁽⁸⁾ OJ L 279, 20. 12. 1971, p. 7.

⁽⁹⁾ OJ L 15, 18. 1. 1984, p. 28.

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

Article 4

This Directive shall enter into force on the 20th day following its publication in the *Official Journal of the European Communities*.

Article 5

This Directive is addressed to the Member States.

Done at Brussels, 3 September 1998.

For the Commission

Franz FISCHLER

Member of the Commission

ANNEX

PART A

DETERMINATION OF AMINO ACIDS

1. Purpose and scope

This method is for the determination of free (synthetic and natural) and total (peptide bound and free) amino acids in feedingstuffs, using an amino acid analyzer. It is applicable to the following amino acids: cyst(e)ine, methionine, lysine, threonine, alanine, arginine, aspartic acid, glutamic acid, glycine, histidine, isoleucine, leucine, phenylalanine, proline, serine, tyrosine and valine.

The method does not distinguish between the salts of amino acids and it cannot differentiate between D and L forms of amino acids. It is not valid for the determination of tryptophan or hydroxy analogues of amino acids.

2. Principle

2.1. Free amino acids

The free amino acids are extracted with diluted hydrochloric acid. Coextracted nitrogenous macromolecules are precipitated with sulfosalicylic acid and removed by filtration. The filtered solution is adjusted to pH 2,20. The amino acids are separated by ion exchange chromatography and determined by reaction with ninhydrin with photometric detection at 570 nm.

2.2. Total amino acids

The procedure chosen depends on the amino acids under investigation. Cyst(e)ine and methionine must be oxidised to cysteic acid and methionine sulphone respectively prior to hydrolysis. Tyrosine must be determined in hydrolysates of unoxidized samples. All the other amino acids listed in paragraph 1 can be determined in either the oxidised or unoxidised sample.

Oxidation is performed at 0 °C with a performic acid/phenol mixture. Excess oxidation reagent is decomposed with sodium disulphite. The oxidised or unoxidised sample is hydrolysed with hydrochloric acid ($c = 6 \text{ mol/l}$) for 23 hours. The hydrolysate is adjusted to pH 2,20. The amino acids are separated by ion exchange chromatography and determined by reaction with ninhydrin using photometric detection at 570 nm (440 nm for proline).

3. Reagents

Double distilled water or water of equivalent quality must be used (conductivity $< 10 \mu\text{S}$).

- 3.1. Hydrogen peroxide, $w = 30 \%$.
- 3.2. Formic acid, $w = 98\text{-}100 \%$.
- 3.3. Phenol.
- 3.4. Sodium disulfite.
- 3.5. Sodium hydroxide.
- 3.6. 5-Sulfosalicylic acid dihydrate.
- 3.7. Hydrochloric acid, density approximately 1,18 g/ml.
- 3.8. tri-Sodium citrate dihydrate.
- 3.9. 2,2'-Thiodiethanol (thiodiglycol).
- 3.10. Sodium chloride.
- 3.11. Ninhydrin.
- 3.12. Light petroleum, boiling range 40-60 °C.
- 3.13. Norleucine, or other compound suitable for use as internal standard.
- 3.14. Nitrogen gas ($< 10 \text{ ppm oxygen}$).

- 3.15. 1-Octanol.
- 3.16. Amino acids.
- 3.16.1. Standard substances listed under paragraph 1. Pure compounds containing no water of crystallisation. Dry under vacuum over P_2O_5 or H_2SO_4 for 1 week prior to use.
- 3.16.2. Cysteic acid.
- 3.16.3. Methionine sulphone.
- 3.17. Sodium hydroxide solution, $c = 7,5 \text{ mol/l}$:
Dissolve 300 g NaOH (3.5) in water and make up to 1 litre.
- 3.18. Sodium hydroxide solution, $c = 1 \text{ mol/l}$:
Dissolve 40 g NaOH (3.5) in water and make up to 1 litre.
- 3.19. Formic acid-phenol solution:
Mix 889 g formic acid (3.2) with 111 g water and add 4,73 g phenol (3.3).
- 3.20. Hydrolysis mixture, $c = 6 \text{ mol HCl/l}$ containing 1 g phenol/l:
Add 1 g phenol (3.3) to 492 ml HCl (3.7) and make up to 1 litre with water.
- 3.21. Extraction mixture, $c = 0,1 \text{ mol HCl/l}$ containing 2 % thiodiglycol:
Take 8,2 ml HCl (3.7), dilute with approximately 900 ml water, add 20 ml thiodiglycol (3.9) and make up to 1 litre with water, (do not mix 3.7 and 3.9 directly).
- 3.22. 5-Sulfosalicylic acid, $\beta = 6 \%$:
Dissolve 60 g 5-sulfosalicylic acid (3.6) in water and make up to 1 litre with water.
- 3.23. Oxidation mixture (Performic acid-phenol):
Mix 0,5 ml hydrogen peroxide (3.1) with 4.5 ml formic acid-phenol solution (3.19) in a small beaker. Incubate at 20-30 °C for 1 hour in order to form performic acid, then cool on an ice-water bath (15 min) before adding to the sample.
Caution: Avoid contact with skin and wear protective clothing.
- 3.24. Citrate buffer, $c = 0,2 \text{ mol Na}^+/\text{l}$, pH 2,20:
Dissolve 19,61 g sodium citrate (3.8), 5 ml thiodiglycol (3.9), 1 g phenol (3.3) and 16,50 ml HCl (3.7) in approximately 800 ml water. Adjust pH to 2,20. Make up to 1 litre with water.
- 3.25. Elution buffers, prepared according to conditions for the analyzer used (4.9).
- 3.26. Ninhydrin reagent, prepared according to conditions for the analyzer used (4.9).
- 3.27. Standard solutions of amino acids. These solutions should be stored below 5 °C.
- 3.27.1. Stock standard solution of amino acids (3.16.1). $c = 2,5 \mu\text{mol/ml}$ of each in hydrochloric acid. May be obtained commercially.
- 3.27.2. Stock standard solution of cysteic acid and methionine sulphone, $c = 1,25 \mu\text{mol/ml}$.
Dissolve 0,2115 g cysteic acid (3.16.2) and 0,2265 g methionine sulphone (3.16.3) in citrate buffer (3.24) in a 1 litre graduated flask and make up to mark with citrate buffer. Store below 5 °C for not more than 12 months. This solution is not used if the stock standard solution (3.27.1) contains cysteic acid and methionine sulphone.
- 3.27.3. Stock standard solution of the internal standard e.g. norleucine, $c = 20 \mu\text{mol/ml}$.
Dissolve 0,6560 g norleucine (3.13) in citrate buffer (3.24) in a graduated flask and make up to 250 ml with citrate buffer. Store below 5 °C for no more than 6 months.
- 3.27.4. Calibration solution of standard amino acids for use with hydrolysates, $c = 5 \text{ nmol}/50 \mu\text{l}$ of cysteic acid and methionine sulphone and $c = 10 \text{ nmol}/50 \mu\text{l}$ of the other amino acids.

Dissolve 2,2 g sodium chloride (3.10) in 100 ml beaker with 30 ml citrate buffer (3.24). Add 4,00 ml stock standard solution of amino acids (3.27.1), 4,00 ml stock standard solution of cysteic acid and methionine sulphone (3.27.2) and 0,50 ml stock standard solution of internal standard (3.27.3) if used. Adjust pH to 2,20 with sodium hydroxide (3.18).

Transfer quantitatively to a 50 ml graduated flask and make up to the mark with citrate buffer (3.24) and mix.

Store below 5 °C for not more than 3 months.

See also observations 9.1.

- 3.27.5. Calibration solution of standard amino acids for use with hydrolysates prepared according to paragraph 5.3.3.1. and for use with extracts (5.2). The calibration solution is prepared according to 3.27.4 omitting sodium chloride.

Store below 5 °C for not more than 3 months.

4. Apparatus

- 4.1. 100 or 250 ml round bottomed flask fitted with a reflux condenser.
- 4.2. 100 ml borosilicate glass bottle with screw cap with rubber/teflon liner (e.g. Duran, Schott) for use in the oven.
- 4.3. Oven with forced ventilation and a temperature regulator with an accuracy better than ± 2 °C.
- 4.4. pH-meter (three decimal places).
- 4.5. Membrane filter (0,2 μ m).
- 4.6. Centrifuge.
- 4.7. Rotary vacuum evaporator.
- 4.8. Mechanical shaker or magnetic stirrer.
- 4.9. Amino acid analyzer or HPLC equipment with ion exchange column, device for ninhydrin, post column derivatization and photometric detector.

The column is filled with sulfonated polystyrene resins capable of separating the amino acids from each other and from other ninhydrin-positive materials. The flow in the buffer and ninhydrin lines is provided by pumps having a flow stability of $\pm 0,5$ % in the period covering both the standard calibration run and the analysis of the sample.

With some amino acid analyzers hydrolysis procedures can be used in which the hydrolysate has a sodium concentration of $c = 0,8$ mol/l and contains all the residual formic acid from the oxidation step. Others do not give a satisfactory separation of certain amino acids if the hydrolysate contains excess formic acid and/or high sodium ion concentrations. In this case the volume of acid is reduced by evaporation to approx. 5 ml after the hydrolysis and prior to pH adjustment. The evaporation should be performed under vacuum at 40 °C maximum.

5. Procedure

5.1. Preparation of the sample

The sample is ground to pass through a 0,5 mm sieve. Samples high in moisture must be either air-dried at a temperature not exceeding 50 °C or freeze dried prior to grinding. Samples with a high fat content should be extracted with light petroleum (3.12) prior to grinding.

5.2. Determination of free amino acids in feedingstuffs and premixtures

Weigh to the nearest 0,2 mg an appropriate amount (1-5 g) of the prepared sample (5.1), into a conical flask and add 100,0 ml of extraction mixture (3.21). Shake the mixture for 60 min using a mechanical shaker or a magnetic stirrer (4.8). Allow the sediment to settle and pipette 10,0 ml of the supernatant solution into a 100 ml beaker.

Add 5,0 ml of sulfosalicylic acid solution (3.22), with stirring and continue to stir with the aid of magnetic stirrer for 5 minutes. Filter or centrifuge the supernatant in order to remove any precipitate. Place 10,0 ml of the resulting solution into a 100 ml beaker and adjust the pH to 2,20 using sodium hydroxide solution (3.18), transfer to a volumetric flask of appropriate volume using citrate buffer (3.24), and make up to the mark with the buffer solution (3.24).

If an internal standard is being used add 1,00 ml of internal standard (3.27.3) for each 100 ml final solution and make up to the mark with the buffer solution (3.24).

Proceed to the chromatography step according to paragraph 5.4.

If the extracts are not being examined the same day, they must be stored below 5 °C.

5.3. Determination of total amino acids

5.3.1. Oxidation

Weigh to the nearest 0,2 mg from 0,1 to 1 g of the prepared sample (5.1) into:

- a 100 ml round-bottomed flask (4.1) for open hydrolysis (5.3.2.3) or,
- a 250 ml round-bottomed flask (4.1) if a low sodium concentration is required (5.3.3.1) or,
- a 100 ml bottle fitted with a screw cap (4.2), (for closed hydrolysis 5.3.2.4).

The weighed sample portion should have a nitrogen content of about 10 mg and a moisture content not exceeding 100 mg.

Place the flask/bottle in an ice-water bath and cool to 0 °C, add 5 ml of oxidation mixture (3.23) and mix using a glass spatula with a bent tip. Seal the flask/bottle containing the spatula with an air-tight film, place the ice-water bath containing the sealed container in a refrigerator at 0 °C and leave for 16 hours. After 16 hours remove from the refrigerator and decompose the excess oxidation reagent by the addition of 0,84 g of sodium disulfite (3.4).

Proceed to 5.3.2.1.

5.3.2. Hydrolysis

5.3.2.1. Hydrolysis of oxidised samples

To the oxidised sample prepared according to 5.3.1 add 25 ml of hydrolysis mixture (3.20) taking care to wash down any sample residue adhering to the sides of the vessel and the spatula. Depending on the hydrolysis procedure being used, proceed according to 5.3.2.3 or 5.3.2.4.

5.3.2.2. Hydrolysis of unoxidised samples

Weigh into either a 100 ml or a 250 ml round-bottom flask (4.1) or a 100 ml bottle fitted with a screw cap (4.2), to the nearest 0,2 mg, from 0,1 to 1 g of the prepared sample (5.1). The weighed sample portion should have a nitrogen content of about 10 mg. Carefully add 25 ml of hydrolysis mixture (3.20) and mix with the sample. Proceed according to either 5.3.2.3 or 5.3.2.4.

5.3.2.3. Open hydrolysis

Add 3 glass beads to the mixture in the flask (prepared in accordance with 5.3.2.1 or 5.3.2.2) and boil with continuous bubbling under reflux for 23 hours. On completion of hydrolysis, wash the condenser down with 5 ml of citrate buffer (3.24). Disconnect the flask and cool it in an ice bath. Proceed according to 5.3.3.

5.3.2.4. Closed hydrolysis

Place the bottle containing the mixture prepared in accordance with 5.3.2.1 or 5.3.2.2 in an oven (4.3) at 110 °C. During the first hour in order to prevent a build up of pressure (due to the evolution of gaseous substances) and to avoid explosion, place the screw cap over the top of the vessel. *Do not close the vessel with the cap.* After one hour close the vessel with the cap and leave in the oven (4.3) for 23 hours. On completion of hydrolysis, remove the bottle from the oven, carefully open the cap of the bottle and place the bottle in an ice-water bath. Leave to cool.

Depending on the procedure for pH adjustment (5.3.3), quantitatively transfer the contents of the bottle to a 250 ml beaker or a 250 ml round-bottom flask, using citrate buffer (3.24).

Proceed according to 5.3.3.

5.3.3. Adjustment of pH

Depending on the sodium tolerance of the amino acid analyzer (4.9) proceed according to 5.3.3.1 or 5.3.3.2 for the pH adjustment.

5.3.3.1. For chromatographic systems (4.9) requiring a low sodium concentration:

It is advisable to use an internal stock standard solution (3.27.3) when amino acid analyzers requiring a low sodium concentration are employed (when the acid volume has to be reduced).

In this case add 2,00 ml of the internal stock standard solution (3.27.3) to the hydrolysate before the evaporation.

Add 2 drops of 1-octanol (3.15) to the hydrolysate obtained in accordance with paragraph 5.3.2.3 or 5.3.2.4.

Using a rotary evaporator (4.7) reduce the volume to 5-10 ml under vacuum at 40 °C. If the volume is accidentally reduced to less than 5 ml the hydrolysate must be discarded and the analysis recommenced.

Adjust the pH to 2,20 with sodium hydroxide solution (3.18) and proceed to paragraph 5.3.4.

5.3.3.2. For all other Amino Acid Analyzers (4.9)

Take the hydrolysates obtained in accordance with 5.3.2.3 or 5.3.2.4 and partly neutralise them by carefully adding with stirring, 17 ml of sodium hydroxide solution (3.17), ensuring that the temperature is kept below 40 °C.

Adjust the pH to 2,20 at room temperature using sodium hydroxide solution (3.17) and finally sodium hydroxide solution (3.18). Proceed to 5.3.4.

5.3.4. Sample solution for chromatography

Quantitatively transfer the pH adjusted hydrolysate (5.3.3.1 or 5.3.3.2) with citrate buffer (3.24) to a 200 ml graduated flask, and make up to the mark with buffer (3.24).

If an internal standard has not already been used, add 2,00 ml of internal standard (3.27.3) and make up to the mark with citrate buffer (3.24). Mix thoroughly.

Proceed to the chromatography step (5.4).

If the sample solutions are not being examined the same day they must be stored below 5 °C.

5.4. *Chromatography*

Before chromatography bring the extract (5.2) or hydrolysate (5.3.4) to room temperature. Shake the mixture and filter a suitable amount through a 0,2 µm membrane filter (4.5). The resulting clear solution is subjected to ion exchange chromatography, using an amino acid analyzer (4.9).

The injection may be performed manually or automatically. It is important that the same quantity of solution $\pm 0,5\%$ is added to the column for the analysis of standards and samples except when an internal standard is used, and that the sodium:amino acid ratios in the standard and sample solutions are as similar as is practicable.

In general the frequency of calibration runs depends on the stability of the ninhydrin reagent and the analytical system. The standard or sample is diluted with citrate buffer (3.24) to give a peak area of the standard of 30-200 % of the sample amino acid peak area.

The chromatography of amino acids will vary slightly according to the type of analyzer employed and resin used. The chosen system must be capable of separating the amino acids from each other and from the ninhydrin-positive materials. In the range of operation the chromatographic system should give a linear response to changes in the amounts of amino acids added to the column.

During the chromatography step the valley:peak height ratios mentioned below apply, when an equimolar solution (of the amino acids being determined) is analyzed. This equimolar solution must contain at least 30 % of the maximum load of each amino acid which can be accurately measured with the amino acid analyser system (4.9).

For separation of threonine-serine the valley:peak height ratio of the lower of the two overlapping amino acids on the chromatogram should not exceed 2:10 (if only cyst(e)ine, methionine, threonine and lysine are determined, insufficient separation from adjoining peaks will adversely influence the determination). For all other amino acids the separation must be better than 1:10.

The system must ensure that lysine is separated from 'lysine artifacts' and ornithine.

6. **Calculation of results**

The area of the sample and standard peaks is measured for each individual amino acid and the amount, in g amino acid per kg sample, is calculated.

$$\frac{A \times E \times MW \times F}{B \times W \times 1\,000} = \text{g amino acid per kg sample}$$

If an internal standard is used multiply by: $\frac{D}{C}$

- A = peak area, hydrolysate or extract
 B = peak area, calibration standard solution
 C = peak area, internal standard in hydrolysate or extract
 D = peak area, internal standard, calibration standard solution
 MW = molecular weight of the amino acid being determined
 E = concentration of standard in $\mu\text{mol/ml}$
 W = sample weight (g) (corrected to original weight if dried or defatted)
 F = ml total hydrolysate (5.3.4) or ml calculated total dilution volume of extract (6.1).

Cystine and cysteine are both determined as cysteic acid in hydrolysates of oxidized sample, but calculated as cystine ($\text{C}_6\text{H}_{12}\text{N}_2\text{O}_4\text{S}_2$, MW 240,30 by using MW 120,15 ($= 0,5 \times 240,30$).

Methionine is determined as methionine sulphone in hydrolysates of oxidized sample, but calculated as methionine by using MW of methionine: 149,21.

Added free methionine is determined after extraction as methionine, for the calculation the same MW is used.

- 6.1. The total dilution volume of extracts (F) for determination of free amino acids (5.2) is calculated as following:

$$F = 100 \text{ ml} \times \frac{(10 \text{ ml} + 5 \text{ ml})}{10 \text{ ml}} \times \frac{V_{\text{ml}}}{10 \text{ ml}}$$

V = volume of final extract

7. Evaluation of the method

The method has been tested in an intercomparison made at international level in 1990 using four different feedstuffs (mixed pig feed, broiler compound, protein concentrate, premixture). The results, after elimination of outliers, of mean and standard deviation are given in the table below:

Means in g/kg

Reference Material	Amino acid			
	Threonine	Cyst(e)ine	Methionine	Lysine
Mixed pig feed	6,94 n = 15	3,01 n = 17	3,27 n = 17	9,55 n = 13
Broiler compound	9,31 n = 16	3,92 n = 18	5,08 n = 18	13,93 n = 16
Protein concentrate	22,32 n = 16	5,06 n = 17	12,01 n = 17	47,74 n = 15
Premixture	58,42 n = 16	—	90,21 n = 16	98,03 n = 16

n = number of participating laboratories

7.1. Repeatability

The repeatability expressed as 'within laboratory standard deviation' of the above mentioned intercomparison is given in the tables below:

Within laboratory standard deviation (S_i) in g/kg

Reference material	Amino acid			
	Threonine	Cyst(e)ine	Methionine	Lysine
Mixed pig feed	0,13 n = 15	0,10 n = 17	0,11 n = 17	0,26 n = 13
Broiler compound	0,20 n = 16	0,11 n = 18	0,16 n = 18	0,28 n = 16
Protein concentrate	0,48 n = 16	0,13 n = 17	0,27 n = 17	0,99 n = 15
Premixture	1,30 n = 16	—	2,19 n = 16	2,06 n = 16

n = number of participating laboratories

Coefficient of variation (%) for within laboratory standard deviation (S_i)

Reference material	Amino acid			
	Threonine	Cyst(e)ine	Methionine	Lysine
Mixed pig feed	1,9 n = 15	3,3 n = 17	3,4 n = 17	2,8 n = 13
Broiler compound	2,1 n = 16	2,8 n = 18	3,1 n = 18	2,1 n = 16
Protein concentrate	2,7 n = 16	2,6 n = 17	2,2 n = 17	2,4 n = 15
Premixture	2,2 n = 16	—	2,4 n = 16	2,1 n = 16

n = number of participating laboratories

7.2. Reproducibility

The results for between laboratory standard deviation by the above mentioned intercomparison are given in the table below:

Between laboratory standard deviation (S_R) in g/kg

Reference material	Amino acid			
	Threonine	Cyst(e)ine	Methionine	Lysine
Mixed pig feed	0,28 n = 15	0,30 n = 17	0,23 n = 17	0,30 n = 13
Broiler compound	0,48 n = 16	0,34 n = 18	0,55 n = 18	0,75 n = 16
Protein concentrate	0,85 n = 16	0,62 n = 17	1,57 n = 17	1,24 n = 15
Premixture	2,49 n = 16	—	6,20 n = 16	6,62 n = 16

n = number of participating laboratories

Coefficient of variation (%) for between laboratory standard deviation (S_R)

Reference material	Amino acid			
	Threonine	Cyst(e)ine	Methionine	Lysine
Mixed pig feed	4,1 n = 15	9,9 n = 17	7,0 n = 17	3,2 n = 13
Broiler compound	5,2 n = 16	8,8 n = 18	10,9 n = 18	5,4 n = 16
Protein concentrate	3,8 n = 16	12,3 n = 17	13,0 n = 17	3,0 n = 15
Premixture	4,3 n = 16	—	6,9 n = 16	6,7 n = 16

n = number of participating laboratories

8. Use of reference materials

The correct application of the method shall be verified by making replicate measurements of certified reference materials when available. Calibration with certified amino acid calibration solution is recommended.

9. Observations

- 9.1. Because of differences between amino acid analysers the final concentrations of the calibration solutions of standard amino acids (see 3.27.4 and 3.27.5) and of the hydrolysate (see 5.3.4) should be taken as a guideline.

The range of linear response of the apparatus has to be checked for all amino acids.

The standard solution is diluted with citrate buffer to give peak areas in the middle of the range.

- 9.2. Where high performance liquid chromatographic equipment is used to analyse the hydrolysates, the experimental conditions must be optimised in accordance with the manufacturer's recommendations.
- 9.3. By applying the method to feedingstuffs containing more than 1 % chloride (concentrate, mineral feeds, supplementary feeds) underestimation of methionine could occur and special treatment has to be done.

PART B

DETERMINATION OF CRUDE OILS AND FATS

1. Purpose and scope

This method is for the determination of crude oils and fats in feedingstuffs. It does not cover the analysis of oil seeds and oleaginous fruit defined in Council Regulation 136/66/EEC of 22 September 1966.

The use of the two procedures described below depends on the nature and composition of the feedingstuff and the reason for carrying out the analysis.

1.1. Procedure A — Directly extractable crude oils and fats

This method is applicable to feed materials of plant origin, except those included within the scope of Procedure B.

1.2. Procedure B — Total crude oils and fats

This method is applicable to feed materials of animal origin and to all compound feeds. It is to be used for all materials from which the oils and fats cannot be completely extracted without prior hydrolysis (eg glutens, yeast, potato proteins and products subject to processes such as extrusion, flaking and heating).

1.3. *Interpretation of results*

In all cases where a higher result is obtained by using Procedure B than by Procedure A, the result obtained by Procedure B shall be accepted as the true value.

2. **Principle**

2.1. *Procedure A*

The sample is extracted with light petroleum. The solvent is distilled off and the residue dried and weighed.

2.2. *Procedure B*

The sample is treated under heating with hydrochloric acid. The mixture is cooled and filtered. The residue is washed and dried and submitted to the determination according to Procedure A.

3. **Reagents**

- 3.1. Light petroleum, boiling range: 40 to 60 °C. The bromine value must be less than 1 and the residue on evaporation less than 2 mg/100 ml.
- 3.2. Sodium sulfate, anhydrous.
- 3.3. Hydrochloric acid, $c = 3 \text{ mol HCl/l}$
- 3.4. Filtration aid, e.g. Kieselguhr, Hyflo-supercel.

4. **Apparatus**

- 4.1. Extraction apparatus. If fitted with a siphon (Soxhlet apparatus), the reflux rate should be such as to produce about 10 cycles per hour; if of the non-siphoning type, the reflux rate should be about 10 ml per minute.
- 4.2. Extraction thimbles, free of matter soluble in light petroleum and having a porosity consistent with the requirements of point 4.1.
- 4.3. Drying oven, either a vacuum oven set at $75^\circ\text{C} \pm 3^\circ\text{C}$ or an air-oven set at $100^\circ\text{C} \pm 3^\circ\text{C}$.

5. **Procedure**

5.1. *Procedure A (see point 8.1)*

Weigh 5 g of the sample to the nearest 1 mg, transfer it to an extraction thimble (4.2) and cover with a fat-free wad of cotton wool.

Place the thimble in an extractor (4.1) and extract for six hours with light petroleum (3.1). Collect the light petroleum extract in a dry, weighed flask containing fragments of pumice stone⁽¹⁾.

Distil off the solvent. Dry the residue maintaining the flask for one and a half hours in the drying oven (4.3). Leave to cool in a desiccator and weigh. Dry again for 30 minutes to ensure that the weight of the oils and fats remains constant (loss in weight between two successive weighings must be less than 1 mg).

5.2. *Procedure B*

Weigh 2,5 g of the sample to the nearest 1 mg (see point 8.2), place in a 400 ml beaker or a 300 ml conical flask and add 100 ml of hydrochloric acid (3.3) and fragments of pumice stone. Cover the beaker with a watch glass or fit the conical flask with a reflux condenser. Bring the mixture to a gentle boil over a low flame or a hot-plate and keep it there for one hour. Do not allow the product to stick to the sides of the container.

Cool and add a quantity of filtration aid (3.4) sufficient to prevent any loss of oil and fat during filtration. Filter through a moistened, fat-free, double filter paper. Wash the residue in cold water until a neutral filtrate is obtained. Check that the filtrate does not contain any oil or fats. Their presence indicates that the sample must be extracted with light petroleum, using Procedure A, before hydrolysis.

Place the double filter paper containing the residue on a watch glass and dry for one and a half hours in the air oven (4.3) $100^\circ\text{C} \pm 3^\circ\text{C}$.

Place the double filter paper containing the dry residue in an extraction thimble (4.2) and cover with a fat-free wad of cotton wool. Place the thimble in an extractor (4.1) and proceed as indicated in the second and third paragraphs of point 5.1.

⁽¹⁾ Where the oil or fat has to undergo subsequent quality tests, replace the fragments of pumice stone by glass beads.

6. Expression of result

Express the weight of the residue as a percentage of the sample.

7. Repeatability

The difference between the results of two parallel determinations carried out on the same sample by the same analyst should not exceed:

- 0,2 % in absolute value, for contents of crude oils and fats lower than 5 %,
- 4,0 % relative to the highest result for contents of 5 to 10 %,
- 0,4 % in absolute value, for contents above 10 %.

8. Observations

- 8.1. For products with a high content of oils and fats, which are difficult to crush or unsuitable for drawing a homogeneous reduced test sample, proceed as follows.

Weigh 20 g of the sample to the nearest 1 mg and mix with 10 g or more of anhydrous sodium sulfate (3.2). Extract with light petroleum (3.1) as indicated in point 5.1. Make up the extract obtained to 500 ml with light petroleum (3.1) and mix. Take 50 ml of the solution and place in a small, dry, weighed flask containing fragments of pumice stone⁽¹⁾. Distil off the solvent, dry and proceed as indicated in the last paragraph of point 5.1.

Eliminate the solvent from the extraction residue left in the thimble, crush the residue to a fineness of 1 mm, return it to the extraction thimble (do not add sodium sulfate) and proceed as indicated in the second and third paragraphs of point 5.1.

Calculate the content of oils and fats as a percentage of the sample by using the following formula:

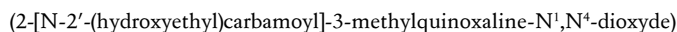
$$(10 a + b) \times 5$$

where:

a = mass in grams of the residue after the first extraction (aliquot part of the extract),

b = mass in grams of the residue after the second extraction.

- 8.2. For products low in oils and fats the test sample may be increased to 5 g.
- 8.3. Pet foods containing a high content of water may need to be mixed with anhydrous sodium sulfate prior to hydrolysis and extraction as per Procedure B.
- 8.4. In paragraph 5.2 it may be more effective to use hot water in place of cold water to wash the residue after filtration.
- 8.5. The drying time of 1,5 h may need to be extended for some feedingstuffs. Excessive drying should be avoided as this can lead to low results. A microwave oven can also be used.
- 8.6. Pre-extraction by Procedure A prior to hydrolysis and re-extraction by Procedure B is recommended if the crude oil/fat content is greater than 15 %. To some extent this depends on the nature of the feedingstuff and the nature of the oil-fat in the feedingstuff.

PART C**DETERMINATION OF OLAQUINDOX****1. Purpose and scope**

This method is for the determination of olaquinox in feedingstuffs. The lower limit of determination is 5 mg/kg.

2. Principle

The sample is extracted by a water-methanol mixture. The content of olaquinox is determined by reversed-phase high-performance liquid chromatography (HPLC) using an UV detector.

⁽¹⁾ Where the oil or fat has to undergo subsequent quality tests, replace the fragments of pumice stone by glass beads.

3. Reagents

3.1. Methanol.

3.2. Methanol, HPLC grade

3.3. Water, HPLC grade

3.4. Mobile phase for HPLC:

Water (3.3) -methanol (3.2) mixture, 900 + 100 (V + V).

3.5. Standard substance: pure olaquinox 2-[N-2'-(hydroxyethyl)carbamoyl]-3-methylquinoxaline-N¹,N⁴-dioxide, E 851.

3.5.1. Olaquinox stock standard solution, 250 µg/ml

Weigh to the nearest 0,1 mg 50 mg of olaquinox (3.5) in a 200 ml graduated flask and add ca. 190 ml water. Then place the flask for 20 min into an ultrasonic bath (4.1). After ultrasonic treatment bring the solution to room temperature, make up to the mark with water and mix. Wrap the flask with aluminum foil and store in a refrigerator. This solution must be prepared fresh each month.

3.5.2. Olaquinox intermediate standard solution, 25 µg/ml

Transfer 10,0 ml of the stock standard solution (3.5.1) into a 100 ml graduated flask, make up to the mark with the mobile phase (3.4) and mix. Wrap the flask with aluminium foil and store in a refrigerator. This solution must be prepared fresh each day.

3.5.3. Calibration solutions:

Into a series of 50 ml graduated flask transfer 1,0, 2,0, 5,0, 10,0, 15,0 and 20,0 ml of the intermediate standard solution (3.5.2). Make up to the mark with the mobile phase (3.4) and mix. Wrap the flask with aluminium foil. These solutions correspond to 0,5, 1,0, 2,5, 5,0, 7,5 and 10,0 µg of olaquinox per ml respectively.

These solutions must be prepared fresh each day.

4. Apparatus

4.1. Ultrasonic bath

4.2. Mechanical shaker

4.3. HPLC equipment with variable wavelength ultraviolet detector or diode array detector

4.3.1. Liquid chromatographic column, 250 mm × 4 mm, C18, 10 µm packing, or equivalent

4.4. Membrane filters, 0,45 µm

5. Procedure

Note: Olaquinox is light sensitive. Carry out all procedures under subdued light or use amber glassware.

5.1. General

5.1.1. A blank feed should be analysed to check that neither olaquinox nor interfering substances are present.

5.1.2. A recovery test should be carried out by analysing the blank feed which has been fortified by addition of a quantity of olaquinox, similar to that present in the sample. To fortify at a level of 50 mg/kg, transfer 10,0 ml of the stock standard solution (3.5.1) to a 250 ml conical flask and evaporate the solution to ca. 0,5 ml. Add 50 g of the blank feed, mix thoroughly and leave for 10 min mixing again several times before proceeding with the extraction step (5.2).

Note: For the purpose of this method the blank feed should be similar in type to that of the sample and olaquinox should not be detected.

5.2. Extraction

Weigh to the nearest 0,01 g, approximately 50 g of the sample. Transfer to a 1 000 ml conical flask, add 100 ml of methanol (3.1) and place the flask for 5 minutes in the ultrasonic bath (4.1). Add 410 ml water and leave in the ultrasonic bath for further 15 minutes. Remove the flask from the ultrasonic bath, shake it for 30 minutes on the shaker (4.2) and filter through a folded filter. Transfer 10,0 ml of the filtrate into a 20 ml graduated flask, make up to the mark with water and mix. An aliquot is filtered through a membrane filter (4.4). (see 9. Observation) Proceed to the HPLC determination (5.3).

5.3. HPLC determination

5.3.1. Parameters:

The following conditions are offered for guidance, other conditions may be used provided that they give equivalent results.

Analytical column (4.3.1)

Mobile phase (3.4): water (3.3) — methanol (3.2) mixture, 900 + 100 (V + V)

Flow rate: 1,5-2 ml/min.

Detection wavelength: 380 nm

Injection volume: 20 µl-100 µl

Check the stability of the chromatographic system, injecting several times the calibration solution (3.5.3) containing 2,5 µg/ml, until constant peak heights and retention times are achieved.

5.3.2. Calibration graph

Inject each calibration solution (3.5.3) several times and determine the mean peak height (areas) for each concentration. Plot a calibration graph using the mean peak heights (areas) of the calibration solutions as the ordinates and the corresponding concentrations in µg/ml as the abscissae.

5.3.3. Sample solution

Inject the sample extract (5.2) several times using the same volume as taken for the calibration solutions and determine the mean peak height (area) of the olaquinox peaks.

6. Calculation of the results

From the mean height (area) of the olaquinox peaks of the sample solution determine the concentration of the sample solution in µg/ml by reference to the calibration graph (5.3.2).

The olaquinox content *w* in mg/kg of the sample is given by the following formula:

$$w = \frac{c \times 1\,000}{m}$$

in which:

c = olaquinox concentration of the sample extract (5.2) in µg/ml

m = mass of the test portion in g (5.2).

7. Validation of the results

7.1. Identity

The identity of the analyte can be confirmed by co-chromatography, or by using a diode-array detector by which the spectra of the sample extract (5.2) and the calibration solution (3.5.3) containing 5,0 µg/ml are compared.

7.1.1. Co-chromatography

A sample extract (5.2) is fortified by addition of an appropriate amount of calibration solution (3.5.3). The amount of added olaquinox should be similar to the amount of olaquinox found in the sample extract.

Only the height of the olaquinox peak should be enhanced after taking into account both the amount added and the dilution of the extract. The peak width, at half of its height, must be within $\pm 10\%$ of the original width of the olaquinox peak of the unfortified sample extract.

7.1.2. Diode array detection

The results are evaluated according to the following criteria:

- (a) The wavelength of maximum absorption of the sample and of the standard spectra, recorded at the peak apex on the chromatogram, must be within a margin determined by the resolving power of the detection system. For diode-array detection this is typically within ± 2 nm.

- (b) Between 220 and 400 nm, the sample and standard spectra recorded at the peak apex of the chromatogram, must not be different for those parts of the spectrum within the range 10-100 % of relative absorbance. This criterion is met when the same maxima are present and at no observed point the deviation between the two spectra exceeds 15 % of the absorbance of the standard analyte.
- (c) Between 220 and 400 nm, the spectra of the upslope, apex and downslope of the peak produced by the sample extract must not be different from each other for those parts of the spectrum within the range 10-100 % of relative absorbance. This criterion is met when the same maxima are present and when at all observed points the deviation between the spectra does not exceed 15 % of the absorbance of the spectrum of the peak apex.

If one of these criteria is not met the presence of the analyte has not been confirmed.

7.2. *Repeatability*

The difference between the results of two parallel determinations carried out on the same sample must not exceed 15 % relative to the higher result for olaquinox contents between 10 and 20 mg/kg.

7.3. *Recovery*

For a fortified blank sample the recovery should be at least 90 %.

8. **Results of a collaborative study**

An EC collaborative study was arranged in which four piglet feed samples including one blank feed were analysed by up to 13 laboratories. The results are given below:

	Sample 1	Sample 2	Sample 3	Sample 4
L	13	10	11	11
n	40	40	44	44
mean (mg/kg)	—	14,6	48,0	95,4
(S _r) (mg/kg)	—	0,82	2,05	6,36
(S _R) (mg/kg)	—	1,62	4,28	8,42
CV _r (%)	—	5,6	4,3	6,7
CV _R (%)	—	11,1	8,9	8,8
Nominal content (mg/kg)	—	15	50	100
recov. (%)	—	97,3	96,0	95,4

L : number of laboratories
 n : number of single values
 S_r : standard deviation of repeatability
 S_R : standard deviation of reproducibility
 CV_r : coefficient of variation of repeatability
 CV_R : coefficient of variation of reproducibility.

9. **Observation**

Although the method has not been validated for feeds containing more than 100 mg/kg of olaquinox, it may be possible to obtain satisfactory results by taking a smaller sample weight and/or diluting the extract (5.2) to reach a concentration within the range of the calibration graph (5.3.2).

COMMISSION DIRECTIVE 98/65/EC

of 3 September 1998

adapting to technical progress Council Directive 82/130/EEC on the approximation of the laws of the Member States concerning electrical equipment for use in potentially explosive atmospheres in mines susceptible to firedamp

(Text with EEA relevance)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

HAS ADOPTED THIS DIRECTIVE:

Having regard to the Treaty establishing the European Community,

Having regard to Council Directive 82/130/EEC of 15 February 1982 on the approximation of the laws of the Member States concerning electrical equipment for use in potentially explosive atmospheres in mines susceptible to firedamp⁽¹⁾, as last amended by Commission Directive 94/44/EC⁽²⁾, and in particular Article 7 thereof,

Whereas, in view of the present state of technical progress, it is now necessary to adapt the contents of the harmonised standards referred to in Annex A to Directive 82/130/EEC;

Whereas, in view of the present state of the standardisation of the types of protection involved, it is necessary to provide for the first and second editions of the standards for electrical equipment for use in potentially explosive atmospheres to be used in parallel;

Whereas certificates issued on the basis of the first edition standards listed in Annex A to Directive 82/130/EEC, as amended, are referred to as 'generation D certificates', and certificates issued on the basis of the second edition standards listed in Annex I to this Directive are referred to as 'generation E certificates'; whereas 'generation D certificates' and 'generation E certificates' are to be used in parallel;

Whereas Directive 94/9/EC of the European Parliament and the Council of 23 March 1994 on the approximation of the laws of the Member States concerning equipment and protective systems intended for use in potentially explosive atmospheres⁽³⁾ stipulates that Directive 82/130/EEC be repealed as from 1 July 2003;

Whereas the measures provided for in this Directive are in accordance with the opinion of the Restricted Committee of the Safety and Health Commission for the Mining and other Extractive Industries,

Article 1

Directive 82/130/EEC is hereby amended as follows:

1. in the first sentence of Annex A, the reference to the 'table below' shall be replaced by the 'tables below';
2. Annex I to the present Directive shall be added to Annex A;
3. Annex II to the present Directive shall be added to Annex B.

Article 2

Member States shall bring into force the laws, regulations and administrative provisions necessary to comply with this Directive not later than 31 December 1999 and shall forthwith 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 the Member States.

Member States shall communicate to the Commission the texts of the provisions of national law which they have already adopted or adopt in the field governed by this Directive.

Article 3

This Directive is addressed to the Member States.

It shall enter into force on the 20th day following its publication in the *Official Journal of the European Communities*.

Done at Brussels, 3 September 1998.

For the Commission

Pádraig FLYNN

Member of the Commission

⁽¹⁾ OJ L 59, 2. 3. 1982, p. 10.

⁽²⁾ OJ L 248, 23. 9. 1994, p. 22.

⁽³⁾ OJ L 100, 19. 4. 1994, p. 1.

ANNEX I

Certificates issued on the basis of the standards listed in the table below shall be referred to as 'generation E certificates'. The letter E shall appear at the beginning of the serial number of each such certificate.

EUROPEAN STANDARDS
(drawn up by Cenelec, 35 rue de Stassart, B-1050 Brussels)

Number	Title	Edition	Date
EN 50014	Electrical apparatus for potentially explosive atmospheres: General requirements	2	December 1992
EN 50015	Electrical apparatus for potentially explosive atmospheres: Oil immersion 'o'	2	April 1994
EN 50016	Electrical apparatus for potentially explosive atmospheres: Pressurised apparatus 'p'	2	October 1995
EN 50017	Electrical apparatus for potentially explosive atmospheres: Powder filling 'q'	2	April 1994
EN 50018	Electrical apparatus for potentially explosive atmospheres: Flameproof enclosure 'd'	2	August 1994
EN 50019	Electrical apparatus for potentially explosive atmospheres: Increased safety 'e'	2	March 1994
EN 50020	Electrical apparatus for potentially explosive atmospheres: Intrinsic safety 'i'	2	August 1994

*ANNEX II***Amendments and supplements made to the European Standards listed in Annex A to this Directive (second editions of European Standards)***Appendix 1***ELECTRICAL APPARATUS FOR POTENTIALLY EXPLOSIVE ATMOSPHERES OF GROUP I****GENERAL REQUIREMENTS**

(European standard EN 50014)

Replace the text of 7.3.1 of European Standard EN 50014 (December 1992) by the following text:

‘7.3.1. Electrical apparatus of Group I

Enclosures of plastic materials with a surface area projected in any direction of more than 100 cm², or containing exposed metallic parts with a capacitance to earth of more than 3 pF, under the most unfavourable conditions in practice, shall be so designed that under normal conditions of use, maintenance and cleaning, danger of ignition due to electrostatic charges is avoided.

This requirement shall be satisfied:

- either by suitable selection of the material: its insulation resistance, measured according to the method given in 23.4.7.8 of this European standard, shall not exceed:
 - 1 GΩ at 23 ± 2 °C and 50 ± 5 % relative humidity,
 - 100 GΩ under extreme service conditions of temperature and humidity specified for the electrical apparatus; the “X” shall then be placed after the certificate reference, as indicated in, 27.2.9,
- or by the size, shape, layout or by other protective methods. The non appearance of dangerous electrostatic charges shall then be verified by actual tests for ignition of an air methane mixture containing 8,5 ± 0,5 % of methane.

If, however, the danger of ignition cannot be avoided in the design, a warning label shall indicate the safety measures to be applied in service’.

*Appendix 3***ELECTRICAL APPARATUS FOR POTENTIALLY EXPLOSIVE ATMOSPHERES OF GROUP I****INTRINSIC SAFETY 'i'****Intrinsically safe electrical systems**

Note: In mines susceptible to firedamp in the Federal Republic of Germany, the word 'Anlage' is used instead of 'System'.

1. Scope

- 1.1. This Annex contains the specific requirements for construction and testing of intrinsically safe electrical systems all or parts of which are intended for installation in potentially explosive atmospheres of mines susceptible to firedamp, in order to ensure that such electrical systems will not cause an explosion in the surrounding atmosphere.
- 1.2. This Annex supplements European Standard EN 50020 'Intrinsic safety "i"' (second edition, August 1994) the requirements of which apply to the construction and testing of intrinsically safe electrical apparatus and associated electrical apparatus.
- 1.3. This Annex does not take the place of installation rules for intrinsically safe electrical apparatus, associated electrical apparatus and intrinsically safe electrical systems.

2. Definitions

- 2.1. The following definitions, specific to intrinsically safe electrical systems, are applicable in this Annex. They supplement the definitions which are in European Standards EN 50 014 'General requirements' and EN 50020 'Intrinsic safety "i"'.
 - 2.2. Intrinsically safe electrical system

An assembly of items of electrical apparatus defined in a descriptive system document in which the interconnecting circuits, or parts of such circuits, intended for use in a potentially explosive atmosphere, are intrinsically safe circuits, and which meet the requirements of this Annex.

- 2.3. Certified intrinsically safe electrical system

An electrical system conforming to 2.2 for which a testing station has issued a system certificate, certifying that the type of electrical system complies with this Annex.

Note 1: It is not necessary for each electrical apparatus in an intrinsically safe electrical system to be certified individually, but it must suitably be identifiable.

Note 2: In so far as the national regulations for installation make it possible, electrical systems conforming to 2.2, for which the knowledge of the electrical parameters of the items of certified intrinsically safe electrical apparatus, certified associated electrical apparatus, non-certified devices conforming to 1.3 of European Standard EN 50014 'General requirements', and the knowledge of the electrical and physical parameters of the components and interconnecting wiring permits the unambiguous deduction that intrinsic safety is conserved, can be installed without additional certificates.

- 2.4. Accessory

Electrical apparatus which contains only components for connecting and switching intrinsically safe circuits and which do not prejudice the intrinsic safety of the system, such as terminal boxes, junction boxes, plugs and sockets and similar items, switches, etc.

3. *Categories of intrinsically safe electrical systems*

- 3.1. Intrinsically safe electrical systems, or parts thereof, shall be placed in one of the two categories: 'ia' or 'ib'. The requirements of this Annex apply to both categories, unless otherwise specified.

Note: Intrinsically safe electrical systems, or parts thereof, may have categories different from those of the intrinsically safe electrical apparatus and associated electrical apparatus included in the system or part thereof. Also different parts of an intrinsically safe electrical system may have different categories.

3.2. Category 'ia'

Intrinsically safe electrical systems, or parts thereof are of category 'ia' if they comply with the requirements for intrinsically safe electrical apparatus of category 'ia' (see 5.2 of European Standard EN 50020 'Intrinsic safety'), except that the intrinsically safe electrical system as a whole shall be considered as a single item of electrical apparatus.

3.3. Category 'ib'

Intrinsically safe electrical systems, or parts thereof, are of category 'ib' if they comply with the requirements for electrical apparatus of category 'ib' (see 5.3 of European Standard EN 50020 'Intrinsic safety') except that the intrinsically safe electrical system as a whole shall be considered as a single item of electrical apparatus.

4. *Interconnecting wiring in an intrinsically safe electrical system*

- 4.1. The electrical parameters and all characteristics of the interconnecting wiring specific to an intrinsically safe electrical system, in so far as intrinsic safety depends on them, shall be specified in the certification documents for that electrical system.

- 4.2. Where a multicore cable contains interconnections which are parts of more than one intrinsically safe circuit the cable shall meet the following requirements:

- 4.2.1. The radial thickness of the insulation shall be appropriate to the diameter of the conductor. In the case of polyethylene, the minimum radial thickness shall be 0,2 mm.

- 4.2.2. Before leaving the manufacturer's works, the multicore cable shall be submitted to AC dielectric tests specified either in 4.2.2.1 or in 4.2.2.2. The success of these tests shall be attested by a tests certificate issued by the cable manufacturer.

- 4.2.2.1. Either each core, before assembly into the cable, is tested at a voltage value (rms) equal to 3 000 V + (2 000 times the radial thickness of the insulation in mm) V; the assembled cable:

- is firstly tested at a voltage value (rms) equal to 500 V applied between all the armourings or screens of the cable joined together electrically and the bundle of all cores joined together electrically, and
- is secondly tested at a voltage value (rms) equal to 1 000 V applied between a bundle comprising one half of the cable cores and a bundle comprising the other half of the cores.

- 4.2.2.2. Or the assembled cable:

- is firstly tested at a voltage value (rms) equal to 1 000 V applied between all the armourings or screens of the cable joined together electrically and the bundle of all cores joined together electrically, and
- is secondly tested at a voltage value (rms) equal to 2 000 V applied in succession between each core of the cable and the bundle formed by all the other cores joined together electrically.

- 4.2.3. The tests prescribed in 4.2.2 shall be carried out with an a.c. voltage of substantially sinusoidal wave form at a frequency between 48 and 62 Hz given by a transformer of appropriate power, taking into account the cable capacity. In the case of dielectric tests on assembled cables, the voltage shall be increased steadily to the specified value in a period not less than 10 seconds and then maintained for at least 60 seconds.

These tests are carried out by the cable manufacturer.

- 4.3. No fault between the cores of a multicore cable shall be taken into account if one of the two following requirements is satisfied:
- 4.3.1. The cable conforms to 4.2 and each individual intrinsically safe circuit is enclosed in a conducting screen providing at least 60 % coverage.
- Note:* The eventual connection of the screen to earth or frame will be specified in the installation rules.
- 4.3.2. The cable conforms to 4.2, is effectively protected against damage and each intrinsically safe circuit within the cable has, in normal operation, a peak voltage of equal to or less than 60 volts.
- 4.4. Where a multicore cable conforms with 4.2 but not with 4.3 and contains only intrinsically safe circuits forming parts of a single intrinsically safe electrical system, faults shall be considered between up to four cores of the cable in addition to the application of either 3.2 or 3.3.
- 4.5. Where a multicore cable conforms with 4.2 but not with 4.3 and contains intrinsically safe circuits forming parts of different intrinsically safe electrical systems, each intrinsically safe circuit contained in the cable shall have a safety factor of at least four times that required by either 3.2 or 3.3.
- 4.6. Where a multicore cable does not comply with 4.2 and 4.3, any number of faults between the cores of the cable shall be taken into account in addition to the application of 3.2 or 3.3.
- 4.7. The certification documents of the intrinsically safe electrical system shall specify the conditions of use resulting from the application of 4.3 to 4.6.
5. *Accessories used in intrinsically safe electrical systems*
- The accessories which are listed in the certification documents as parts of an intrinsically safe electrical system shall meet:
- points 7 and 8 of European Standard EN 50014 'General requirements',
 - points 6 and 12.2 of European Standard EN 50020 'Intrinsic safety "i"'.
- Their marking shall bear at least the manufacturer's name or its registered trade mark.
- Note:* The use of noncertified accessories is subject to the installation requirements.
6. *Type tests*
- Intrinsically safe electrical systems shall be type tested in accordance with the type test requirements of point 10 of European Standard EN 50020 'Intrinsic safety "i"', but taking into account point 4 of this Annex.
7. *Marking of intrinsically safe electrical systems*
- Certified intrinsically safe electrical systems shall be marked by the holder of the certificate of that system on at least one of the electrical apparatus set on a strategic position. The marking shall include the minimal marking of 27.6 of European Standard EN 50 014 'General requirements' and the letters 'SYST'.
-

COMMISSION DIRECTIVE 98/66/EC

of 4 September 1998

amending Directive 95/31/EC laying down specific criteria of purity concerning sweeteners for use in foodstuffs

(Text with EEA relevance)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Community,

Having regard to Council Directive 89/107/EEC of 21 December 1988 on the approximation of the laws of the Member States concerning food additives authorised for use in foodstuffs intended for human consumption ⁽¹⁾, as amended by European Parliament and Council Directive 94/34/EC ⁽²⁾, and in particular Article 3(3)(a) thereof,

After consulting the Scientific Committee on Food,

Whereas European Parliament and Council Directive 94/35/EC of 30 June 1994 on sweeteners for use in foodstuffs ⁽³⁾, as amended by Directive 96/83/EC ⁽⁴⁾, lists those substances which may be used as sweeteners in foodstuffs;

Whereas Commission Directive 95/31/EC of 5 July 1995 laying down specific criteria of purity concerning sweeteners for use in foodstuffs ⁽⁵⁾ sets out the purity criteria for the sweeteners mentioned in Directive 94/35/EC;

Whereas it is necessary, in the light of technical progress, to amend the purity criteria set out in Directive 95/31/EC for isomalt (E 953); whereas it is consequently necessary to adapt that Directive;

Whereas it is necessary to take into account the specifications and analytical techniques for sweeteners as set out in the Codex Alimentarius by the Joint FAO/WHO Expert Committee on Food Additives (JECFA);

Whereas food additives, prepared by production methods or starting materials significantly different from those included in the evaluation of the Scientific Committee for Food, or different from those mentioned in this Directive, shall be submitted for evaluation by the Scientific Committee on Food with a view to full evaluation with emphasis on the purity criteria;

Whereas the measures provided for in this Directive are in accordance with the opinion of the Standing Committee for Foodstuffs,

HAS ADOPTED THIS DIRECTIVE:

Article 1

In the Annex to Directive 95/31/EC, the text concerning E 953 — isomalt is replaced by the text in the Annex to this Directive.

Article 2

Member States shall bring into force the laws, regulations and administrative provisions necessary to comply with the Directive before 1 July 1999. 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.

Article 3

This Directive shall enter into force on the 20th day following that of its publication in the *Official Journal of the European Communities*.

Article 4

This Directive is addressed to the Member States.

Done at Brussels, 4 September 1998.

For the Commission

Martin BANGEMANN

Member of the Commission

⁽¹⁾ OJ L 40, 11. 2. 1989, p. 27.

⁽²⁾ OJ L 237, 10. 9. 1994, p. 1.

⁽³⁾ OJ L 237, 10. 9. 1994, p. 3.

⁽⁴⁾ OJ L 48, 19. 2. 1997, p. 16.

⁽⁵⁾ OJ L 178, 28. 7. 1995, p. 1.

ANNEX

‘E 953 — ISOMALT

Synonyms

Hydrogenated isomaltulose, hydrogenated palatinose.

Definition*Chemical name*

Isomalt is a mixture of hydrogenated mono- and disaccharides whose principal components are the disaccharides:

6-O- α -D-Glucopyranosyl-D-sorbitol (1,6-GPS) and
1-O- α -D-Glucopyranosyl-D-mannitol dihydrate (1,1-GPM)*Chemical formula*6-O- α -D-Glucopyranosyl-D-sorbitol: $C_{12}H_{24}O_{11}$
1-O- α -D-Glucopyranosyl-D-mannitol dihydrate: $C_{12}H_{24}O_{11} \cdot 2H_2O$ *Relative molecular mass*6-O- α -D-Glucopyranosyl-D-sorbitol: 344,32
1-O- α -D-Glucopyranosyl-D-mannitol dihydrate: 380,32*Assay*Content not less than 98 % of hydrogenated mono- and disaccharides and not less than 86 % of the mixture of 6-O- α -D-Glucopyranosyl-D-sorbitol and 1-O- α -D-Glucopyranosyl-D-mannitol dihydrate determined on the anhydrous basis.**Description**

Odourless, white, slightly hygroscopic, crystalline mass.

Identification*A. Solubility*

Soluble in water, very slightly soluble in ethanol.

B. Thin layer chromatography

Examine by thin layer chromatography using a plate coated with an approximately 0,2 mm layer of chromatographic silica gel. The principal spots in the chromatogram are those of 1,1-GPM and 1,6-GPS.

Purity*Water content*

Not more than 7 % (Karl Fischer Method)

Sulphated ash

Not more than 0,05 % expressed on the dry weight basis

D-Mannitol

Not more than 3 %

D-Sorbitol

Not more than 6 %

Reducing sugars

Not more than 0,3 % expressed as glucose on the dry weight basis

Nickel

Not more than 2 mg/kg expressed on the dry weight basis

Arsenic

Not more than 3 mg/kg expressed on the dry weight basis

Lead

Not more than 1 mg/kg expressed on the dry weight basis

Heavy metals (as Pb)

Not more than 10 mg/kg expressed on the dry weight basis.’

II

(Acts whose publication is not obligatory)

COUNCIL

COUNCIL DECISION

of 15 September 1998

appointing the members of the Economic and Social Committee for the period
from 21 September 1998 to 20 September 2002

(98/545/EC, Euratom)

THE COUNCIL OF THE EUROPEAN UNION,

Having regard to the Treaty establishing the European Community, and in particular Article 194 thereof,

Having regard to the Treaty establishing the European Atomic Energy Community, and in particular Article 166 thereof,

Whereas the terms of office of the current members of the Economic and Social Committee expire on 20 September 1998⁽¹⁾ and whereas the members of that Committee should be appointed for a further period of four years as from 21 September 1998;

Whereas each Member State has submitted a list containing twice as many candidates as there are seats allotted to its nationals;

Whereas the Committee shall be composed of representatives of the various categories of economic and social activity, in particular producers, farmers, carriers, workers, dealers, craftsmen, professional occupations and the general public; whereas the composition of the Committee shall take account of the need to ensure adequate representation of the various categories of economic and social activity;

Having consulted the Commission,

HAS DECIDED AS FOLLOWS:

Article 1

The persons listed in the Annex to this Decision are hereby appointed members of the Economic and Social Committee for the period from 21 September 1998 to 20 September 2002.

Article 2

This Decision shall be published in the *Official Journal of the European Communities*. It shall take effect on the date of its adoption.

Done at Brussels, 15 September 1998.

For the Council

The President

W. SCHÜSSEL

⁽¹⁾ OJ L 257, 5. 10. 1994, p. 20.

*ANEXO — BILAG — ANHANG — ΠΑΡΑΡΤΗΜΑ — ANNEX — ANNEXE — ALLEGATO —
BIJLAGE — ANEXO — LIITE — BILAGA*

Lista de los miembros del Comité Económico y Social

Liste over medlemmerne af Det Økonomiske og Sociale Udvalg

Liste der Mitglieder des Wirtschafts- und Sozialausschusses

Κατάλογος των μελών της Οικονομικής και Κοινωνικής Επιτροπής

List of the members of the Economic and Social Committee

Liste des membres du Comité économique et social

Elenco dei membri del Comitato economico e sociale

Lijst van leden van het Economisch en Sociaal Comité

Lista dos membros do Comité Económico e Social

Talous- ja sosiaalikomitean jäsenten luettelo

Förteckning över ledamöter i Ekonomiska och sociala kommittén

BELGIË / BELGIQUE / BELGIEN

Wilfried BEIRNAERT
Directeur-generaal VBO

Paul VERHAEGHE
Afgevaardigd beheerder
Federatie van de Voedingsindustrie

Guido RAVOET
Directeur-generaal
Belgische Vereniging van Banken

Alfons DE VADDER
Directeur-generaal
Federatie van Belgische Distributieondernemingen

Étienne de PAUL de BARCHIFONTAINE
Secrétaire général de l'Alliance agricole belge

Kris PEETERS
Secretaris-generaal NCMV

Guy HAAZE
Nationaal voorzitter van de Algemene Centrale der Liberale Vakbonden van België

Josly PIETTE
Secrétaire général de la Confédération des syndicats chrétiens de Belgique

Thierry DOCK
Collaborateur au service d'études CSC

Arthur LADRILLE
Président honoraire
Centrale d'alimentation FGTB

Paul GRUSELIN
Conseiller
Département des relations internationales FGTB

Jean-François HOFFELT
Secrétaire général de la Fédération belge des coopératives «Febecoop»

DANMARK

Sekretariatschef Helle BUNDGAARD
Dansk Industri

International sekretær Søren KARGAARD
FTF

Afdelingschef Leif ERLAND NIELSEN
Landbrugsrådet

Økonom Henrik HOFMAN
Arbejderbevægelsens Erhvervsråd
Økonom Lisbeth BAASTRUP SØRENSEN
Funktionærernes- og Tjenestemændenes Fællesråd
Cand. jur. Birte JOHANSEN
Dansk Arbejdsgiverforening
Afdelingschef Bo GREEN
Det Danske Handelskammer — Børsen
Sekretær Ib WISTISEN
LO
Konsulent John SVENNINGSEN
LO i Danmark — Bruxelles kontoret

DEUTSCHLAND

Manfred DIMPER
Abteilungsleiter bei der Arbeitsgemeinschaft der Verbraucher
Ursula ENGELN-KEFER
Stellvertretende Vorsitzende des Deutschen Gewerkschaftsbundes
Gräfin Soscha zu EULENBURG
Vorstandsmitglied der Bundesarbeitsgemeinschaft der Freien Wohlfahrtspflege und Vizepräsidentin des Deutschen Roten Kreuzes
Dipl.-Volkswirt Rainer FRANZ
Beauftragter des Bundesverbandes der Deutschen Industrie für den WSA
Dr. Göke FRERICH
Präsidiumsmitglied des Bundesverbandes des Deutschen Groß- und Außenhandels
Prof. Dr. med. Christoph FUCHS
Hauptgeschäftsführer der Bundesärztekammer und des Deutschen Ärztetages
Michael GEUENICH
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