

**COMMISSION REGULATION (EC) No 625/2003
of 2 April 2003**

amending Regulation (EC) No 1623/2000 laying down detailed rules for implementing Council Regulation (EC) No 1493/1999 on the common organisation of the market in wine with regard to market mechanisms

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Community,

Having regard to Council Regulation (EC) No 1493/1999 of 17 May 1999 on the common organisation of the market in wine ⁽¹⁾, as last amended by Regulation (EC) No 2585/2001 ⁽²⁾, and in particular Articles 26, 33 and 36 thereof,

Whereas:

- (1) Chapter I of Title I of Commission Regulation (EC) No 1623/2000 ⁽³⁾, as last amended by Regulation (EC) No 1795/2002 ⁽⁴⁾, lays down the detailed rules for applying the aid scheme for the use of grapes, grape must, concentrated grape must or rectified concentrated grape must. Experience has shown that the comestible products not eligible under the scheme should be specified in greater detail, the administrative burden placed on users and processors of juice should be lightened and adequate arrangements should be introduced to ensure that proper use of the juice is monitored. The percentage of quantities of comestible products which must be checked must be higher than under other schemes because the product is often used in a Member State other than the one where the aid is paid.
- (2) Under the aid scheme for must used to increase the alcoholic strength of wine products, the reference to the method of determining alcoholic strength should be corrected. To facilitate the work of the Member States, they should be responsible for the administration of aid applications. To allow detailed and effective checks, checking arrangements should be specified.
- (3) To ensure that problem cases are treated equally, the provisions relating to payment of the aid provided for under the various aid schemes covered by Regulation (EC) No 1623/2000 should be harmonised.
- (4) To allow detailed and effective checks to be carried out in the case of aid for the private storage of wine, checking arrangements should be laid down and the tolerance margins for verifying the alcoholic strength of grape must, concentrated grape must and rectified concentrated grape must should be clarified. To facilitate the payment of advances under this measure, the related administrative procedure must be changed.
- (5) As regards the arrangements for the distillation of by-products of wine-making, it is fair to modulate aid and prices according to the type of by-product concerned.

The flat-rate aid and the flat-rate price should therefore be abolished. In addition, in order to respond to structural changes in the sector, the Member States concerned should be allowed to extend the exemption from the obligation to deliver by-products for distillation to certain categories of producers.

- (6) If a Member State decides to vary the buying-in price paid to producers according to yield under the distillation arrangements referred to in Article 28 of Regulation (EC) No 1493/1999, it should be possible to extend the deadline by which the aid is to be paid to distillers.
- (7) To ensure that financial transactions under crisis distillation as provided for in Article 30 of Regulation (EC) No 1493/1999 are properly implemented, it should be confirmed that the advance on the price to be paid to distillers by the intervention agency is to be treated the same as the aid provided for in the case of other distillations.
- (8) For all distillation measures, the provision relating to the use of part of the wine checked as being representative of all wine delivered for distillation should be abolished. In addition, the advances paid for crisis distillation and treated as aid should be included in the system of penalties, and the same arrangements should be introduced for all the different distillations to ensure that producers are paid the minimum price for their wine where the distiller fails to do so.
- (9) Experience has shown that the arrangements for disposing of alcohol obtained under the different distillation measures and held by the intervention agencies should be adjusted. The deadlines for removing alcohol should therefore be fixed on a case-by-case basis where large quantities are involved. To enhance marketing opportunities the present geographical limitations on sales of alcohol should be removed. The conditions for checking the destination of alcohol used in the fuel sector should also be specified.
- (10) Since managing the intervention measures requires the Member States to provide the Commission with a great deal of information, the rules for doing so should be laid down.
- (11) Regulation (EC) No 1623/2000 should therefore be amended.

⁽¹⁾ OJ L 179, 14.7.1999, p. 1.

⁽²⁾ OJ L 345, 29.12.2001, p. 10.

⁽³⁾ OJ L 194, 31.7.2000, p. 45.

⁽⁴⁾ OJ L 272, 10.10.2002, p. 15.

- (12) Some amendments aim to clarify existing provisions or to provide additional information and are favourable to market operators. They should therefore apply retroactively.
- (13) Other amendments aim to improve the conditions governing isolated measures for disposing of alcohol. They should therefore apply from the publication of this Regulation.
- (14) By far the majority of amendments provide for technical changes in market management measures. To avoid disrupting the current wine year, these amendments should apply from the next wine year.
- (15) The measures provided for in this Regulation are in accordance with the opinion of the Management Committee for Wine,

HAS ADOPTED THIS REGULATION:

Article 1

Regulation (EC) No 1623/2000 is hereby amended as follows:

1. Chapter I of Title I is replaced by the following:

CHAPTER I

MANUFACTURE OF GRAPE JUICE

Article 3

Purpose of the aid

Aid under Article 35(1)(a) of Regulation (EC) No 1493/1999 shall be granted to processors who:

- (a) being themselves producers or groups of producers, process or have processed on their behalf grapes from their own harvest and grape must and concentrated grape must obtained entirely from their own grape crop into grape juice; or
- (b) buy directly or indirectly from producers or groups of producers grapes produced in the Community and grape must and concentrated grape must for the purpose of processing them into grape juice.

The grape must and concentrated grape must used must come from grapes produced in the Community.

Article 4

Manufacture of other comestible products from grape juice

The grape juice or concentrated grape juice obtained may be processed into any comestible products other than wine products as referred to in Annex I to Regulation (EC) No 1493/1999 or the products referred to in Article 35(1)(b) and (c) of that Regulation.

Article 5

Technical requirements relating to products

1. The raw materials for the manufacture of grape juice referred to in Article 3 must be of sound and fair merchantable quality and suitable for processing into grape juice.
2. The grape must used and the must obtained from grapes used must have a density at 20 °C of between 1,055 and 1,100 grams per cubic centimetre.
3. At the time it is used to manufacture comestible products, the grape juice must comply with Council Directive 2001/112/EC (*).

Article 6

Administrative rules applicable to processors for inspection purposes

1. Processors who undertake operations for the manufacture of grape juice throughout the current wine year shall submit, before the start of each wine year, a grape juice processing schedule to the competent authority of the Member State. If the processor is undertaking operations for the manufacture of grape juice for the first time after the start of a wine year the schedule must be drawn up before operations commence.

The schedule shall contain the following information:

- (a) the type of the raw materials intended for processing (grapes, grape must or concentrated grape must);
- (b) the place where the grape must and concentrated grape must intended for processing are stored;
- (c) the place where processing is to be carried out.

2. Processors who carry out operations for the manufacture of grape juice only on specified dates shall submit, at least three working days before the operations are to start, a processing statement to the competent authority of the Member State.

The processing statement shall contain the following information:

- (a) the information required under the second subparagraph of paragraph 1;
- (b) the quantity of grapes, grape must or concentrated grape must intended for processing;
- (c) the density of the grape must and concentrated grape must;
- (d) the starting date and probable duration of processing operations.

The statement must cover a minimum quantity of:

- (a) 1,3 tonnes for grapes;
- (b) 10 hectolitres for grape must;
- (c) 3 hectolitres for concentrated grape must.

3. In addition to the information referred to in paragraphs 1 and 2, Member States may ask processors to provide further information.

4. The competent authority of the Member State shall stamp the schedules and statements provided for in paragraphs 1 and 2 and return a copy to the processor.

5. As an exception to paragraphs 1, 2 and 3, Member States may introduce simplified procedures for processors who use no more than 5 tonnes of grapes or 40 hl of grape must or 12 hl of concentrated grape must each wine year.

6. Processors shall keep stock records showing the following information taken from the accompanying documents or registers referred to in Article 70 of Regulation (EC) No 1493/1999:

- (a) the quantities and the density of the raw materials entering their premises each day and, where applicable, the name and address of the seller;
- (b) the quantities and density of raw materials used each day;
- (c) the quantities of grape juice produced each day;
- (d) the quantities of grape juice leaving their premises each day and the name and address of the consignee, or the quantities of grape juice used each day by the processor.

The supporting documents relating to the stock records shall be made available to the inspection authorities on the occasion of any checks.

Article 7

Administrative rules applicable to users for inspection purposes

1. For the purposes of this Chapter, "user" means any operator carrying out one of the following operations: bottling, packaging or presenting the grape juice or concentrated grape juice, storing with a view to sale to one or more undertakings responsible for carrying out the preceding or following operations, or preparing other comestible products from that juice.

Such operations may also be carried out by processors as referred to in Article 3.

2. Users shall submit to the competent authority of the place of unloading a written undertaking not to process the grape juice into the wine products referred to in Annex I to Regulation (EC) No 1493/1999 or the products referred to in Article 35(1)(b) and (c) of that Regulation.

The Member States shall lay down the requirements for submitting such undertakings. However, the undertaking must be submitted before the grape juice or concentrated grape juice is used, and no later than four months after the submission of the aid application referred to in Article 8 of this Regulation.

Export shall be regarded as compatible with such undertakings.

3. Where processors dispatch the grape juice to a user within the Community:

- (a) processors shall indicate on the accompanying document referred to in Article 70(1) of Regulation (EC) No 1493/1999 whether the manufacture of the grape juice has been or will be subject to an aid application from them and the actual or planned date of submission of the application;
- (b) users shall send the accompanying document to the competent authority of the place of unloading no later than 15 days after receipt of the product;
- (c) where users dispatch the juice they have received to another operator within the Community they shall ensure that that operator signs their written undertaking and shall submit it to the competent authority within the time limit set in the second subparagraph of paragraph 2;
- (d) the competent authority, after having received the written undertaking, shall stamp the accompanying document and send a copy of the stamped accompanying document to the processor of the grape juice in question no later than 30 days after receipt of the undertaking.

4. Under Article 2 of Commission Regulation (EC) No 2729/2000 (**), during the wine year the competent authorities shall check by sampling, on the basis of a risk analysis, to ensure that the undertaking referred to in paragraph 2 of this Article has been respected. The checks shall relate to at least 10 % of the quantities covered by applications for the stamping of the accompanying documents provided for in paragraph 3(d) of this Article received during the previous wine year.

Article 8

Aid applications

1. Processors as referred to in Article 6(1) shall submit aid applications to the competent authority of the Member State no later than six months after the end of the wine year. These shall be accompanied by the following documents:

- (a) a copy of the processing schedule referred to;
- (b) a copy of the stock records referred to in Article 6(6) or a summary thereof; the Member States may require such copies and summaries to be certified by an authority responsible for inspections.

Member States may require further documentation.

2. Processors as referred to in Article 6(2) shall submit aid applications to the competent authority of the Member State no later than six months after the end of the processing operations. These shall be accompanied by the following documents:

- (a) a copy of the processing statement referred to;
- (b) a copy of the stock records referred to in Article 6(6) or a summary thereof; the Member States may require such copies and summaries to be certified by an authority responsible for inspections.

Aid applications shall indicate the quantity of raw materials actually processed and the day on which the processing operations were completed.

3. The processor shall submit to the competent authority of the Member State, no later than six months after submitting the aid application:

- (a) a copy of the accompanying document stamped by the competent authority as laid down in Article 7(3)(d);
- (b) a copy of the accompanying document bearing the customs stamp certifying export.

4. As an exception to paragraphs 1 and 2, Member States may introduce simplified procedures for processors who use no more than 5 tonnes of grapes or 40 hl of grape must or 12 hl of concentrated grape must each wine year. These procedures must be completed no later than six months after the end of the wine year.

Article 9

Aid amounts and rules

1. Aid for the use of grapes, grape must and concentrated grape must shall be fixed per unit of quantity of raw material actually used as follows:

- (a) for grapes: EUR 4,952 per 100 kg;
- (b) for grape must: EUR 6,193 per hl;
- (c) for concentrated grape must: EUR 21,655 per hl.

2. Except in cases of *force majeure*, no aid shall be payable on quantities of raw materials in excess of the following ratio between the raw material and the grape juice obtained:

- (a) 1,3 for grapes, in 100 kg per hl;
- (b) 1,05 for must, in hl per hl;
- (c) 0,30 for concentrated must, in hl per hl.

Where the product obtained is concentrated grape juice, those ratios shall be multiplied by five.

Article 10

Payment of the aid

The competent authority shall pay the aid within three months of the date of presentation of the required documentation referred to in Article 8.

Article 11

Advance payments

1. Processors may request advance payment of an amount equal to the aid referred to in Article 9 calculated for the raw materials which they can prove have entered their premises, provided they have lodged a security with the competent authority. That security shall be 120 % of the amount of the advance.

2. Advances shall be paid by the competent authority within three months of presentation of proof that the security has been lodged. However, no advances shall be paid before 1 January of the wine year concerned.

3. After the competent authority has checked the required documentation referred to in Article 8 the security referred to in paragraph 1 of this Article shall be released in full or in part in accordance with the procedure laid down in Article 19 of Regulation (EEC) No 2220/85.

Article 11a

Penalties and cases of *force majeure*

1. If the processor presents the required documentation referred to in Article 8 late, but within six months of expiry of the deadline referred to in that Article, the aid shall be reduced by 30 %.

If the processor presents the documentation after the six months referred to above have elapsed, no aid shall be paid.

2. If a check reveals that the user has not complied with the undertakings referred to in Article 7(2) and (3) the aid shall be recovered from the processor. If the user is domiciled in a Member State other than that where the processor is domiciled the Member State concerned shall notify the Member State where the processor is domiciled of the non-compliance without delay.

3. Except in cases of *force majeure*, if processors are found not to have complied with the obligations imposed on them under this Chapter other than the obligation to process into grape juice the raw materials covered by the aid application the aid shall be reduced. The Member State concerned shall set the rate of that reduction.

4. If the quantity of raw material actually used is between 95 % and 99,9 % of the quantity for which the advance was paid the security referred to in Article 11 shall be forfeit for the quantity not processed during the wine year.

Except in cases of *force majeure*, if the quantity of raw material actually used is less than 95 % of the quantity for which the advance was paid, the security shall be forfeit in its entirety.

5. In cases of *force majeure*, the competent authority of the Member State shall determine the measures which it deems appropriate in the light of the reasons invoked and shall inform the Commission thereof.

(*) OJ L 10, 12.1.2002, p. 58.

(**) OJ L 316, 15.12.2000, p. 16.'

2. Article 13(2) is replaced by the following:

'2. The potential alcoholic strength of the products listed in paragraph 1 shall be determined by applying the figures in the table of equivalence in Annex I hereto to the readings at 20 °C from a refractometer used in accordance with the method laid down in the Annex to Commission Regulation (EC) No 558/93 (*).

A tolerance of 0,2 shall be allowed in checks by the competent authorities.

(*) OJ L 58, 11.3.1993, p. 50.'

3. The following third paragraph is added to Article 14:

'However, the Member States may provide for several aid applications to be submitted covering some of the operations to increase alcoholic strength.'

4. The following Article 14a is added:

'Article 14a

Checks

1. The competent authorities of the Member States shall take all necessary steps to ensure the checks needed in order to verify in particular the identity and volume of the product used to increase the alcoholic strength and compliance with Annex V, points C and D, to Regulation (EC) No 1493/1999.

2. Producers shall be obliged to allow the checks referred to in paragraph 1 at any time.'

5. Article 16 is replaced by the following:

'Article 16

Payment of the aid

The competent authority shall pay the aid no later than 31 August following the end of the current wine year.'

6. Article 29(1)(c) is replaced by the following:

'(c) the reading obtained at a temperature of 20 °C with a refractometer used in accordance with the method referred to in the Annex to Regulation (EEC) No 558/93. A tolerance shall be permitted of 0,5 for grape must and 1 for concentrated grape must and rectified concentrated grape must.'

7. Article 34(2) is replaced by the following:

'2. Without prejudice to paragraph 6, products under contract may undergo only such oenological treatments and processes as are necessary for their preservation. A variation shall be permitted in the volume entered in the

contract which may not exceed 2 % for wine and 3 % for grape must, concentrated grape must and rectified concentrated grape must. When there has been a change of vat, the variation permitted is set at 3 % to 4 % respectively.'

8. The following Article 35a is added:

'Article 35a

Checks

1. The competent authorities of the Member States shall take any measures required to ensure the checks necessary to verify the identity and volume of the product covered by the contract and compliance with Article 34.

2. Producers shall be obliged to allow the checks referred to in paragraph 1 at any time.'

9. Article 37 is replaced by the following:

'Article 37

Payment of the aid

1. The competent authority shall pay the aid no later than three months after the expiry date of the storage contract.

2. In cases where a contract has been terminated in accordance with Articles 33 or 35, the aid payable shall be in proportion to the actual duration of the contract. The competent authority shall pay the aid no later than three months after the day on which the contract is terminated.'

10. Article 38 is replaced by the following:

'Article 38

Advance payments

1. Producers may request that an advance be paid to them provided that they have lodged a security for 120 % of the advance in favour of the intervention agency. Without prejudice to Article 32, the amount of the advance shall be calculated on the basis of the amount of the aid for the product in question, as referred to in Article 25.

2. Advances shall be paid by the competent authority within three months of presentation of proof that the security has been lodged.

3. Once the aid is paid by the competent authority, the security referred to in paragraph 1 shall be released.

Where entitlement to the aid is lost in accordance with Article 36(1)(a), the whole security shall be forfeit.

Where the application of Article 36(1)(b) results in an aid amount that is lower than the sum already paid, the security shall be reduced by 120 % of the amount paid in excess of the aid due. Securities reduced in this way shall be released not later than three months after the date of expiry of the contract.

The Member States shall make the necessary adjustments where the clause provided for in Article 29(5)(i) is applied.'

11. The third paragraph of Article 43 is replaced by the following:

'For the purposes of the distillation operations referred to in this Title, Member States shall take the necessary steps to ensure compliance with the obligation laid down in the second subparagraph.'

12. Article 45 is amended as follows:

- (a) In paragraph 1 the following second subparagraph is added:

'The Member States may stipulate that this delivery must take place before the date referred to in the first subparagraph.'

- (b) Paragraph 2 is deleted.

13. Article 46(3)(a)(ii) is replaced by the following:

'(ii) in wine-growing zone C: two litres of pure alcohol, by actual or potential strength/100 kilograms where it is obtained from varieties listed in the classification of vine varieties for the administrative unit concerned as varieties other than wine-grape varieties; litres of pure alcohol, by actual or potential strength/100 kilograms where it is obtained from varieties listed in the classification for the administrative unit concerned solely as wine-grape varieties;'

14. Article 48 is replaced by the following:

'Article 48

Aid to be paid to distillers

1. The aid referred to in Article 27(11)(a) of Regulation (EC) No 1493/1999 shall be fixed, per % vol. of alcohol and per hectolitre of product obtained by distillation, as follows:

- (a) for neutral alcohol:

- obtained from marc: EUR 0,8453,
- obtained from wine and wine lees: EUR 0,4106;

- (b) for spirits distilled from marc and distillates or raw alcohol obtained from marc having an alcoholic strength of at least 52 % vol.: EUR 0,3985;

- (c) for wine spirits and raw alcohol obtained from wine and wine lees: EUR 0,2777.

Where the distiller provides evidence that the distillate or raw alcohol obtained by distillation of marc was used otherwise than as spirits distilled from marc, a further amount of EUR 0,3139/% vol./hl shall be paid.

2. No aid shall be due for quantities of wine delivered for distillation exceeding the producer's obligation as provided for in Article 45 by more than 2 %.'

15. Article 49 is amended as follows:

- (a) paragraph 2 is replaced by the following:

'2. Producers who, during the wine year in question, do not produce more than 25 hectolitres of wine or must themselves on their own premises shall be free not to deliver any quantities.'

- (b) the following paragraph 4 is added:

'4. Under Article 27(8) of Regulation (EC) No 1493/1999, the Member States may provide, in respect of all or part of their territory, that producers who do not exceed a production level to be established, and produced by themselves on their own premises, may discharge their obligation to deliver the by-products referred to in paragraphs 3 and 6 of that Article by withdrawing those products under supervision. However, that production level may not exceed 80 hl of wine or must.'

16. The first paragraph of Article 58 is replaced by the following:

'Producers subject to one of the obligations referred to in Articles 45 and 54 who have delivered at least 90 % of the quantity of product which they are obliged to deliver before 15 July of the current wine year may discharge their obligation by delivering the remaining quantity before a date to be fixed by the competent national authority, which may not be after 31 July of the following wine year.'

17. Article 60 is amended as follows:

- (a) in paragraph 5 the following second subparagraph is added:

'In the case of distillation as referred to in Article 28 of Regulation (EC) No 1493/1999, and where the Member State differentiates the buying-in price according to yield per hectare as provided for in Article 55(2), the period referred to in the first subparagraph shall be seven months.'

- (b) paragraph 6 is deleted.

18. Article 62 is amended as follows:

- (a) paragraph 2 is replaced by the following:

'2. The price to be paid to the distiller by the competent authority for the product delivered shall be fixed in % vol./hl as follows:

- (a) distillation under Article 27 of Regulation (EC) No 1493/1999:

- raw alcohol obtained from marc: EUR 1,872,
- raw alcohol obtained from wine and wine lees: EUR 1,437;

- (b) distillation under Article 28 of Regulation (EC) No 1493/1999:

- raw alcohol obtained from wine: EUR 1,799.

Where alcohol is stored on the premises where it has been produced, the above prices shall be reduced by EUR 0,5 per hectolitre of alcohol.'

- (b) paragraph 3 is deleted.

19. The following third paragraph is added to Article 67(1):

'The advance on the price to be paid to distillers by the intervention agency, which may be provided for under the distillation referred to in Article 30 of Regulation (EC) No 1493/1999, shall be the same as the aid provided for in the second subparagraph.'

20. Article 73(2) is deleted.

21. Article 74 is amended as follows:

(a) paragraph 4 is replaced by the following:

'4. The intervention agency shall recover from the producer an amount equal to all or part of the aid or the advance to be paid to the distiller in cases where a producer does not meet the requirements laid down by the Community rules for the distillation operation in question, for one of the following reasons:

- (a) failure to present the harvest, production or stock declaration within the time allowed;
- (b) presentation of a harvest, production or stock declaration found by the competent authority of the Member State to be incomplete or inaccurate, where the missing or incorrect data are essential for application of the measure concerned;
- (c) failure to meet the obligations laid down in Article 37 of Regulation (EC) No 1493/1999 where the infringement is detected or notified to the distiller after payment of the minimum price on the basis of previous declarations.

In the case provided for in point (a) of the first subparagraph the amount to be recovered shall be determined in accordance with the rules laid down in Article 12 of Commission Regulation (EC) No 1282/2001 (*).

In the case provided for in point (b) of the first subparagraph the amount to be recovered shall be determined in accordance with the rules laid down in Article 13 of Regulation (EC) No 1282/2001.

In the case provided for in point (c) of the first subparagraph the amount to be recovered shall be all the aid or the advance paid to the distiller.

(*) OJ L 176, 29.6.2001, p. 14.'

(b) the following paragraph 5 is added:

'5. If it is found that the distiller has not paid the purchase price to the producer within the deadline laid down in Article 65(7), the intervention agency shall pay the producer, before 1 June following the wine year concerned, an amount equal to the aid or advance, where appropriate through the intermediary of the intervention agency in the producer's Member State. In that case, no aid or advance shall be due to the distiller.'

22. Article 86 is replaced by the following:

'Article 86

Opening tendering procedures

The Commission, acting in accordance with the procedure laid down in Article 75 of Regulation (EC) No 1493/1999,

may issue one or more invitations to tender every quarter for export to certain third countries for end-use in the motor-fuel sector only. The alcohol concerned must be imported and dehydrated in a third country for end-use in the motor-fuel sector in a third country only.'

23. Article 91 is amended as follows:

(a) the third subparagraph of paragraph 7 is replaced by the following:

'The removal order shall state the date by which the alcohol must be physically removed from the storehouse of the intervention agency concerned. The deadline for removal may not be more than eight days from the date of issue of the removal order. However, where a removal order covers more than 25 000 hl the deadline may be more than eight days later but no more than 15 days later.'

(b) paragraph 10 is replaced by the following:

'10. The alcohol must be physically removed from the storehouses of each intervention agency concerned by a deadline to be fixed when the invitation to tender is issued in accordance with the procedure laid down in Article 75 of Regulation (EC) No 1493/1999.'

24. Article 93(6) is replaced by the following:

'6. The alcohol must be physically removed from the storehouses of each intervention agency concerned by a deadline to be fixed when a public sale is opened, in accordance with the procedure laid down in Article 75 of Regulation (EC) No 1493/1999.'

25. Article 95(3) is deleted.

26. The following paragraph 4 is added to Article 101:

'4. Without prejudice to paragraph 1, when the alcohol is exported to third countries for end-use in the motor-fuel sector only, the checks on its actual use shall be carried out up to the moment when the alcohol is mixed with a denaturing agent in the country of destination.

Where the alcohol is disposed of for use as bio-ethanol in the Community, those checks shall be carried out up to the moment when the alcohol is delivered to an oil undertaking using bio-ethanol or to an approved firm as indicated in Article 92, where the supervision referred to in subparagraph 3 is guaranteed from the moment when the alcohol is delivered to that approved firm.

In the cases provided for in the first and second subparagraphs, the alcohol concerned must remain under the supervision of an official body which guarantees its use in the motor fuel sector under special tax arrangements which require that end-use.'

27. The following Article 102a is added to Title IV:

'Article 102a

Derogation from the payment deadlines

As an exception to the provisions governing the payment deadlines granted to the competent authority of the Member States pursuant to this Regulation, if that authority has justified doubts as to the eligibility of the beneficiary to the aid, it shall carry out the necessary checks and payment shall not be effected until eligibility to the aid has been acknowledged.'

28. Article 103 is replaced by the following:

'Article 103

Notifications to the Commission

1. In the case of aid for the private storage of wine and must under Title III, Chapter I, of Regulation (EC) No 1493/1999, the Member States shall communicate:

- (a) no later than 31 December of the wine year following that in which the contracts were concluded, the quantities of grape must processed into concentrated grape must or rectified concentrated grape must during the period of validity of the contract, and the quantities so obtained;
- (b) by 5 March of the current wine year at the latest, the quantities of products under contract at 16 February.

2. In the case of distillation under Articles 27, 28 and 30 of Regulation (EC) No 1493/1999, the Member States shall communicate at the end of October, December, February, April, June and August:

- (a) the quantities of wine, wine lees and wine fortified for distillation that have been distilled in the previous two months;
- (b) the quantities of alcohol, broken down into neutral alcohol, raw alcohol and spirits distilled from wine,
 - produced during the previous period,
 - taken over by the intervention agencies during the previous period,
 - disposed of by the intervention agencies during the previous period and the percentage of those quantities exported and the selling prices charged,
 - held by the intervention agencies at the end of the previous period.

3. Concerning the disposal of alcohol taken over by the intervention agencies, as referred to in Article 31 of Regulation (EC) No 1493/1999, the Member States shall communicate at the end of each month:

- (a) the quantities of alcohol physically removed during the previous month under a tendering procedure;
- (b) the quantities of alcohol physically removed during the previous month following a public sale.

4. In the case of distillation under Article 29 of Regulation (EC) No 1493/1999, the Member States shall communicate at the end of each month:

- (a) the quantities of wine distilled during the previous month;
- (b) the quantities of alcohol which qualified for secondary aid during the previous month.

5. In the case of aid for concentrated and rectified concentrated must used for enrichment as provided for in Article 34 of Regulation (EC) No 1493/1999, the Member States shall communicate no later than 31 December of the wine year following the current one:

- (a) the number of producers who have received aid;
- (b) the quantities of wine enriched;
- (c) the quantities of concentrated grape must and rectified concentrated grape must used for enrichment, expressed in terms of potential alcoholic strength by volume per hectolitre and broken down by the wine-growing zone of origin.

6. In the case of aid for manufacturing grape juice and other comestible products from such grape juice as provided for in Article 35(1)(a) in Regulation (EC) No 1493/1999, the Member States shall communicate no later than 30 April in respect of the previous wine year:

- (a) the quantities of raw materials covered by aid applications, broken down by type;
- (b) the quantities of raw materials on which aid has been granted, broken down by type.

7. In the case of aid for manufacturing certain products in the United Kingdom and Ireland as provided for in Article 35(1)(b) and (c) of Regulation (EC) No 1493/1999, the Member States shall communicate no later than 30 April in respect of the previous wine year:

- (a) the quantities of grape must and concentrated grape must covered by aid applications, broken down by wine-growing zone of origin;
- (b) the quantities of grape must and concentrated grape must on which aid has been granted, broken down by wine-growing zone of origin;
- (c) the prices paid by manufacturers and operators for the grape must and concentrated grape must.

8. The Member States shall communicate:

- (a) no later than 30 April for the previous wine year, the cases where distillers and makers of fortified wine for distillation have failed to fulfil their obligations and the action taken in consequence;
- (b) 10 days before the end of each quarter, the action taken in response to appeals on the grounds of *force majeure* and the steps taken in those instances by the competent authorities in cases covered by this Regulation.'

29. The title of Annex I is replaced by the following:

Article 2

'Table of equivalence between the potential alcoholic strength and the readings obtained at 20 °C with a refractometer used in accordance with the method laid down in the Annex to Regulation (EEC) No 558/93.'

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

Article 1(7), (16), (19), (20), (21) and (29) shall apply from 1 August 2000.

30. Annex IV is replaced by the text in the Annex to this Regulation.

Article 1(1), (2), (3), (4), (5), (6), (8), (9), (10), (11), (12), (13), (14), (15), (17), (18), (27) and (28) shall apply from 1 August 2003.

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

Done at Brussels, 2 April 2003.

For the Commission

Franz FISCHLER

Member of the Commission

ANNEX

'ANNEX IV

COMMUNITY ANALYSIS METHOD FOR NEUTRAL ALCOHOL

I. GENERAL

For the purposes of this Annex:

- (a) the repeatability limit is the value below which the absolute difference between two single test results obtained under the same conditions (same operator, same apparatus, same laboratory and a short interval of time) may be expected to lie with a specified probability;
- (b) the reproducibility limit is the value below which the absolute difference between two single test results obtained under different conditions (different operators, different apparatus and/or different laboratories and/or different time) may be expected to lie with a specified probability.

The term "single test result" means the value obtained when the standardised test method is applied fully once to a single sample. Unless otherwise stated, the probability shall be 95 %.

II. METHODS

Introduction

1. PREPARATION OF THE SAMPLE FOR ANALYSIS

1.1. General

The volume of laboratory sample intended for analysis must normally be 1,5 l unless a larger quantity is required for a specific determination.

1.2. Sample preparation

The sample shall be made homogeneous before analysis.

1.3. Preservation

The prepared sample shall always be kept in an air-tight and moisture-tight container and stored so that deterioration is prevented; in particular seals of cork, rubber and plastic should not come into direct contact with the alcohol and the use of sealing wax is expressly prohibited.

2. REAGENTS

2.1. Water

2.1.1. Wherever mention is made of water for solution, dilution or washing purposes, dilution or washing purposes, distilled water, or demineralised water of a least equivalent purity shall be used.

2.1.2. Wherever reference is made to "solution" or "dilution", without further indication of a reagent, an aqueous solution is intended.

2.2. Chemicals

All chemicals shall be of analytical reagent quality except where otherwise specified.

3. EQUIPMENT

3.1. List of equipment

The list of equipment contains only those items with a specialised use and items with a particular specification.

3.2. Analytical balance

Analytical balance means a balance with a sensitivity of 0,1 mg or better.

4. EXPRESSION OF RESULTS

4.1. Results

The result stated in the analytical report is the mean value obtained from at least two determinations, the repeatability (r) of which is satisfactory.

4.2. Calculation of results

Except where otherwise specified, the results shall be calculated as g per hl of ethanol at 100 % vol.

4.3. Number of significant figures

The result shall not contain more significant figures than are justified by the precision of the method of analysis used.

Method 1: Determination of alcohol content

The alcoholic strength by volume of the alcohol shall be determined in accordance with existing national provisions or, in the event of dispute, by means of alcoholometers or hydrometers as defined in Council Directive 76/765/EEC of 27 July 1976 on the approximation of the laws of the Member States relating to alcoholometers and alcohol hydrometers ⁽¹⁾.

It shall be expressed as percentage by volume, as prescribed by Council Directive 76/766/EEC of 27 July 1976 on the approximation of the laws of the Member States relating to alcohol tables ⁽²⁾.

Method 2: Evaluation of colour and/or clarity

1. SCOPE AND FIELD OF APPLICATION

The method enables the colour and/or clarity of neutral alcohol to be assessed.

2. DEFINITION

The colour and/or clarity: the colour and/or clarity as assessed by the procedure specified.

3. PRINCIPLE

The colour and clarity are assessed visually by comparison with water against a white background and a black background respectively.

4. APPARATUS

Glass cylinders, colourless, at least 40 cm in height.

5. PROCEDURE

Place two glass cylinders (4) on the white background or black background and fill one cylinder with sample to a depth of approximately 40 cm and the other with water to the same depth.

Observe the sample from above i.e. through the length of the cylinder, and compare it with the comparison cylinder.

⁽¹⁾ OJ L 262, 27.9.1976, p. 143.

⁽²⁾ OJ L 262, 27.9.1976, p. 149.

6. INTERPRETATION

Assess the colour and/or clarity of the sample when observed as set out in 5.

Method 3: Determination of permanganate clearing time

1. SCOPE AND FIELD OF APPLICATION

The method determines the permanganate clearing time of neutral alcohol.

2. DEFINITION

The permanganate clearing time, as determined by the method specified, is the number of minutes required for the colour of the sample to match that of the colour standard after adding 1 ml of a 1 mmol/l potassium permanganate solution to 10 ml of the sample.

3. PRINCIPLE

The time for the colour of the sample, after addition of potassium permanganate, to match that of a colour standard is determined and defined as the permanganate clearing time.

4. REAGENTS

4.1. Potassium permanganate solution, 1 mmol/l. Prepare immediately before use.

4.2. Colour solution A (red)

— Weigh accurately 59,50 g $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$,

— prepare a mixture of 25 ml hydrochloric acid ($P^{20} = 1,19 \text{ g/ml}$) and 975 ml water,

— add the cobalt chloride to some of the HCl/water mixture in a 1 000 ml volumetric flask and make up to the mark with the rest of the mixture at 20 °C.

4.3. Colour solution B (yellow)

— Weigh accurately 45,00 g $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$,

— prepare a mixture of 25 ml hydrochloric acid ($P^{20} = 1,19 \text{ g/ml}$) and 975 ml water and then proceed with the weighed quantity of ferric chloride as for colour solution A.

4.4. Colour standard solution

Pipette 13 ml of colour solution A and 5,5 ml colour solution B into a 100 ml volumetric flask and make up to the mark with water at 20 °C.

Note:

Colour solutions A and B may be stored in the dark at 4 °C for several months; the colour standard should be freshly prepared from time to time.

5. APPARATUS

5.1. 100 ml Nessler tubes of colourless transparent glass, graduated to 50 ml, with ground-glass stopper, or test tubes, colourless, circa 20 mm diameter.

5.2. Pipettes, 1, 2, 5, 10 and 50 ml.

5.3. Thermometer, range up to 50 °C in 0,1 or 0,2 °C.

5.4. Analytical balance.

5.5. Water bath, thermostatically controlled at $20 \pm 0,5$ °C.

5.6. Volumetric flasks, 100 and 1 000 ml with ground glass stoppers.

6. PROCEDURE

- 6.1. — Pipette 10 ml of the sample into a test tube or 50 ml in a Nessler tube,
 - place in water bath at 20 °C,
 - add 1 ml or 5 ml, depending on the sample quantity used, of 1 mmol/l KMnO_4 solution, mix and leave in the water bath at 20 °C,
 - note the time,
 - pipette 10 ml of the colour standard into a test tube of the same diameter or 50 ml of the colour standard into a Nessler tube,
 - observe the change of colour of the sample and compare it with the colour standard against a white background from time to time,
 - note the time at which the colour of the sample becomes the same as that of the colour standard.

Note:

Take care not to expose the sample solution to direct sunlight during the test.

7. EXPRESSION OF RESULTS

- 7.1. Interpretation of the time of clearing shall be the time required for the colour of the sample tube to match that of the standard tube.

For a neutral alcohol that time must be at least 18 minutes at a temperature of 20 °C.

- 7.2. Repeatability

The difference in the times of two tests, carried out simultaneously or in rapid succession, by the same analyst, on the same sample, under the same conditions, shall not exceed two minutes.

8. NOTES

- 8.1. Traces of manganese dioxide have a catalysing effect on the reaction, ensure that pipettes and test tubes are used which have been scrupulously cleaned and reserved exclusively for the purpose. Clean them with hydrochloric acid and rinse thoroughly with water, no brown coloration shall be visible on the glass apparatus.
- 8.2. The quality of the water to prepare the dilute permanganate solution (4.1) shall be carefully monitored; it must not consume any permanganate. If the required quality cannot be obtained, distilled water should be brought to the boil and a small quantity of permanganate added in order to obtain a very slightly pink coloration. This should then be cooled and used for the dilution.
- 8.3. For some samples the decoloration may occur without passing through the exact shade of the reference solution.
- 8.4. The permanganate test may be distorted when the alcohol sample for analysis has not been stored in a perfectly clean glass flask, sealed with either a ground glass stopper which has been rinsed with alcohol or another stopper encased in tin or aluminium.

Method 4: Determination of aldehydes

1. SCOPE AND FIELD OF APPLICATION

The method determines aldehydes, expressed as acetaldehyde, in neutral alcohol.

2. DEFINITION

The aldehyde content: the content of aldehydes, expressed as acetaldehyde, as determined by the method specified.

3. PRINCIPLE

The colour obtained after the reaction of the sample with Schiff's reagent is compared with standard solutions having a known acetaldehyde content.

4. REAGENTS

p-rosaniline hydrochloride (basic fuchsin)

sodium sulphite or anhydrous sodium metabisulphite

hydrochloric acid, density $p^{20} = 1.19$ g/ml

powdered active carbon

starch solution, prepared from 1 g soluble starch and 5 mg HgI_2 (preservative) which are suspended in a little cold water, mixed with 500 ml boiling water, boiled for 5 minutes and filtered when cold.

iodine solution, 0,05 mol/l

1-amino-ethanol $CH_3CH(NH_2)OH$ (MW 61.08)

Schiff's reagent

- Dissolve 5,0 g powdered p-rosaniline hydrochloride with about 1 000 ml hot water in a 2 000 ml volumetric flask,
- leave in the water bath until completely dissolved if necessary,
- dissolve 30 g anhydrous sodium sulphite (or an equivalent quantity of sodium metabisulphite) in circa 200 ml water and add to the cool p-rosaniline solution,
- leave to stand for about 10 minutes,
- add 60 ml hydrochloric acid ($p^{20} = 1,19$ g/ml),
- where the solution is colourless — a slight degree of brown colouration can be ignored — make up to the mark with water,
- if necessary filter with a little active carbon over a folded filter to render the solution colourless.

Notes:

- (1) The Schiff's reagent should be prepared at least 14 days before it is used.
- (2) The free SO_2 content in the reagent should be between 2,8 and 6,0 mmol/100 ml, the pH must be 1.

Determination of free SO_2

- Pipette 10 ml Schiff's reagent into a 250 ml Erlenmeyer flask,
- add 200 ml water,
- add 5 ml starch solution,
- titrate with 0,05 mol/l iodine solution to starch end-point,

if the free SO_2 content is outside the indicated range it should either be:

- raised with a calculated quantity of sodium metabisulphite (0,126 g Na_2SO_3 /100 ml reagent per mmol SO_2 lacking), or
- lowered by bubbling air through the reagent.

Calculation of free SO_2 in the reagent.

mmol free SO_2 /100 ml reagent:

$$= \frac{\text{consumed ml iodine solution (0,05 mol/l)} \cdot 3,2 \cdot 100}{64 \cdot 10}$$

$$= \frac{\text{consumed ml iodine solution (0,05 mol/l)}}{2}$$

Important:

If other methods are used to prepare the Schiff's reagent, the sensitivity of the reagent should be checked so that during the test:

- there is no colouration with the aldehyde-free reference alcohol,
- the pink colouration should be visible from 0,1 g acetaldehyde per hl alcohol at 100 % vol.

(3) Purification of commercial 1-amino-ethanol

- Dissolve 5 g 1-amino-ethanol completely in circa 15 ml absolute ethanol,
- add circa 50 ml dry diethyl ether (1-amino-ethanol precipitates),
- leave for several hours in a refrigerator,
- filter off the crystals and wash with dry diethyl ether,
- dry for three to four hours in a desiccator over sulphuric acid in a partial vacuum.

Note:

The cleaned 1-amino-ethanol must be white; if not, repeat the recrystallisation process.

5. APPARATUS

- 5.1. Colorimetric tubes, each provided with a ground-glass stopper, capacity 20 ml.
- 5.2. 1 ml, 2 ml, 3 ml, 4 ml, 5 ml and 10 ml pipettes.
- 5.3. Water bath, thermostatically controlled at $20 \pm 0,5$ °C.
- 5.4. Spectrophotometer with cuvettes of 50 mm path length.

6. PROCEDURE

6.1. Preliminary remark

When using this method to determine aldehyde content ensure that the sample's alcohol content is at least 90,0 % vol. If not it must be raised by adding corresponding amounts of aldehyde-free ethanol.

6.2. Calibration curve

- Weigh 1,3860 g purified and dried 1-amino-ethanol accurately on an analytical balance.
- Place in a 1 000 ml volumetric flask and add aldehyde-free ethanol-free ethanol, make up to the mark at 20 °C. The solution content 1 g/l acetaldehyde.
- Prepare the dilution series in two stages to produce 10 reference solutions containing 0,1 to 1,0 mg acetaldehyde per 100 ml solution.
- Determine the absorbance values for these reference solutions according to 6.3 and construct graph.

6.3. Determining the aldehyde content

- Pipette 5 ml of the sample into a colorimetric tube.
- Add 5 ml water, mix and keep at a constant temperature of 20 °C.
- At the same time make a blank using 5 ml aldehyde-free ethanol at 96 % vol, add 5 ml water and keep at a temperature of 20 °C.
- Then add 5 ml Schiff's reagent to each tube, close with ground-glass stoppers and shake well.
- Keep in water bath for 20 minutes at 20 °C.
- Put contents into cuvettes.
- Determine absorbance values at 546 nm.

Notes:

- (1) To determine the aldehyde figures it is necessary to check the validity of the calibration curves by comparison with test solution; if not, the calibration curve must be prepared again.
- (2) Ensure that the blank is always colourless.

7. EXPRESSION OF RESULTS

7.1. Formula and method of calculation

Construct a graph of optical density against concentration of acetaldehyde and determine the concentration in the sample by reference to this plot.

The content of aldehydes, expressed as acetaldehyde, in g/hl ethanol at 100 % vol is given by

$$\frac{100 \cdot A}{T}$$

where:

A = the content, in g per hl of acetaldehyde in the sample solution as determined by reference to the standard curve,

T = the alcoholic strength by volume of the sample as determined by method 1.

7.2. Repeatability

The difference between the results of two determinations, carried out simultaneously or in rapid succession, by the same analyst, on the same sample, under the same conditions, shall not exceed 0,1 g aldehyde per hl Ethanol at 100 % vol.

Method 5: Determination of higher alcohols

1. SCOPE AND FIELD OF APPLICATION

The method determines budget alcohols, expressed as 2-methylpropan-1-ol, in neutral alcohol.

2. DEFINITION

The higher alcohol content: the content of higher alcohols, expressed as 2-methylpropan-1-ol as determined by the method specified.

3. PRINCIPLE

The absorbance of the coloured products resulting from the reaction of higher alcohols and an aromatic aldehyde in hot dilute sulphuric acid (Komarowsky reaction) are determined at 560 nm, corrected for the presence of any aldehyde in the sample and then compared with that produced by 2-methylpropan-1-ol reacting under the same conditions.

4. REAGENTS

4.1. Salicyl aldehyde solution, 1 % mas. Prepare by adding 1 g of salicyl aldehyde to 99 g of ethanol at 96 % vol (which is free of fusel oil).

4.2. Sulphuric acid, concentrated, density 1,84 g/m³.

4.3. 2-methylpropan-1-ol.

4.4. Standard 2-methylpropan-1-ol solutions

Dilute 2-methylpropan-1-ol (4.3) with an aqueous solution of 96 % vol ethanol to give a series of standards containing 0,1, 0,2, 0,4, 0,6 and 1,0 g of 2-methylpropan-1-ol per hl of solution.

4.5. Standard acetaldehyde solutions.

Prepare the standard acetaldehyde solutions as described in section 6.2 of method 4.

4.6. Ethanol 96 % vol free from higher alcohols and aldehydes.

5. APPARATUS

- 5.1. UV-VIS spectrophotometer, capable of determining absorbance of solutions at 560 nm.
- 5.2. Spectrophotometer cuvettes, 10, 20 and 50 mm pathlengths.
- 5.3. Water bath, thermostatically regulated at $20 \pm 0,5$ °C.
- 5.4. Colorimetric tubes made of thick glass Pyrex or similar with ground-glass stoppers, content circa 50 ml.

6. PROCEDURE

6.1. Aldehyde content

Determine the aldehyde content, expressed as acetaldehyde, in the sample using methanol 4.

6.2. Calibration curve: 2-methylpropan-1-ol

Pipette 10 ml of each of the 2-methylpropan-1-ol standards (4.4) into 50 ml glass cylinders each fitted with ground glass stoppers. Pipette 1 ml of the salicyl aldehyde solution (4.1) into the cylinders and then 20 ml of sulphuric acid (4.2). Mix the contents thoroughly by carefully tilting the cylinders backwards and forwards several times (care being taken to lift the stopper in occasionally). Leave for 10 minutes at room temperature and then put in the water bath (5.3) at $20 \pm 0,5$ °C. After 20 minutes pour the contents into a series of spectrophotometer cuvettes.

Exactly 30 minutes after adding the sulphuric acid determine the absorbance of the solutions at 560 nm using water in the reference cuvette of the spectrophotometer.

Construct a calibration curve of absorbance against 2-methylpropan-1-ol concentration.

6.3. Calibration curve — aldehydes

Repeat 6.2 but replacing the 10 ml of each of the 2-methylpropan-1-ol standards by 10 ml of each of the acetaldehyde standards.

Construct a calibration curve of absorbance at 560 nm against acetaldehyde concentration.

6.4. Sample determination

Repeat 6.2 but replacing the 10 ml of the 2-methylpropan-1-ol standards by 10 ml of the sample.

Determine the absorbance of the sample.

7. EXPRESSION OF RESULTS

7.1. Formula and method of calculation

- 7.1.1. Correct the absorbance of the sample by subtracting the value of the absorbance corresponding to the aldehyde concentration in the sample (obtained from the calibration curve constructed under 6.3).
- 7.1.2. Determine the concentration of higher alcohols, expressed as 2-methylpropan-1-ol, in the sample from the calibration curve constructed under 6.2 but using the correct absorbance (7.1.1).
- 7.1.3. The concentration of higher alcohols, expressed as 2-methylpropan-1-ol in g per hl ethanol at 100 % vol, is given by:

$$\frac{A \cdot 100}{T}$$

where:

A = the concentration of higher alcohols in the sample as calculated in 7.1.2.

T = the alcoholic strength by volume of the sample as determined by method 1.

7.2. Repeatability

The difference between the results of two determinations, when carried out simultaneously or in rapid succession, by the same analyst, on the same sample, under the same conditions, shall not exceed 0,2 g per hl ethanol at 100 % vol.

Method 6: Determination of total acidity

1. SCOPE AND FIELD OF APPLICATION

The method determines the total acidity, expressed as acetic acid, of neutral alcohol.

2. DEFINITION

The total acidity content, expressed as acetic acid: the content of total acidity, expressed as acetic acid, as determined by the method specified.

3. PRINCIPLE

The sample, after de-gassing, is titrated against standard sodium hydroxide solution and the acidity calculated as acetic acid.

4. REAGENTS

4.1. Sodium hydroxide solutions, 0,01 mol/l and 0,1 mol/l, stored so that contact with carbon dioxide is minimised.

4.2. Indigo carmine solution (A)

— Weigh 0,2 g indigo carmine,

— dissolve in 40 ml water and make up to 100 g with ethanol. Phenol red solution (B)

— Weigh 0,2 g phenol red,

— dissolve in 6 ml sodium hydroxide 0,1 mol/l and make up to mark with water in a 100 ml volumetric flask.

5. APPARATUS

5.1. Burette or automatic titrater.

5.2. Pipette, 100 ml.

5.3. Round-bottom flask with ground-glass stopper, 250 ml.

5.4. Reflux condenser with ground-glass stopper.

6. PROCEDURE

— Pipette 100 ml of the sample into the 250 ml round-bottom flask,

— add boiling stones and briefly heat to boiling in the reflux condenser,

— add one drop of each of indicator solutions A and B to the hot solution,

— then titrate with sodium hydroxide 0,01 mol/l until the first signs of change from greenish-yellow to violet.

7. EXPRESSION OF RESULTS

7.1. Formula and method of calculation

The content of total acidity, expressed as acetic acid, in g per hl ethanol at 100 % vol is given by:

$$\frac{V \cdot 60}{T}$$

where:

V = the number of ml of 0,01 mol/l sodium hydroxide required for neutralisation.

T = the alcoholic strength by volume of the sample as determined by method 1.

7.2. Repeatability

The difference between the results of two determinations, carried out simultaneously or in rapid succession, by the same analyst, on the same sample, under the same conditions, shall not exceed 0,1 g per hl ethanol at 100 % vol.

Method 7: Determination of esters

1. SCOPE AND FIELD OF APPLICATION

The method determines esters, expressed as ethyl acetate, in neutral alcohol.

2. DEFINITION

The ester content: the content of esters, expressed as ethyl acetate, as determined by the method specified.

3. PRINCIPLE

Esters react quantitatively with hydroxylamine hydrochloride in alkaline solution to form hydroxylamic acids. These then form coloured complexes with ferric ions in acid solution. The optical densities of these complexes are measured at 525 nm.

4. REAGENTS

4.1. Hydrochloric acid 4 mol/l.

4.2. Ferric chloride solution, 0,37 mol/l in 1 mol/l hydrochloric acid.

4.3. Hydroxylamine Hydrochloride, 2 mol/l. Store in a refrigerator.

4.4. Sodium hydroxide solution, 3,5 mol/l.

4.5. Ethyl acetate standard solutions containing 0,0, 0,2, 0,4, 0,6, 0,8 and 1,0 g ethyl acetate per hl ester-free ethanol at 96 % vol.

5. APPARATUS

5.1. Spectrophotometer with 50 mm pathlength cuvettes.

6. PROCEDURE

6.1. Calibration curve

- Weigh accurately 1,0 g ethyl acetate on an analytical balance,
- add ester-free alcohol in a 1 000 ml volumetric flask and make up to the mark at 20 °C,
- prepare dilution series in two stages to produce 20 reference solutions containing 0,1 to 2,0 mg ethyl acetate per 100 ml solution,
- determine absorbance values for the reference solution in accordance with 6.2 and construct a graph.

6.2. Determination of ester content

- Pipette 10 ml of the sample into test-tubes fitted with ground-glass stoppers,
- add 2 ml hydroxylamine hydrochloride solution,
- at the same time prepare a blank using 10 ml ester-free ethanol at 96 % vol and 2 ml hydroxylamine hydrochloride solution,
- then add 2 ml sodium hydroxide to each solution, close the tubes with ground-glass stoppers and shake well,
- keep for 15 minutes at 20 °C in a water bath,
- add 2 ml hydrochloric acid to each tube, shake briefly,
- add 2 ml ferric chloride solution, mix well,
- pour contents into cuvettes,
- Determine absorbance values at 525 nm.

7. EXPRESSION OF RESULTS

7.1. Formula and method of calculation

Plot the optical densities of the standards against their concentrations.

The ester content (expressed as ethyl acetate = A) corresponding to the absorbance value is read off on the graph and calculated according to the formula:

$$\frac{A \cdot 100}{T}$$

and given in g per hl ethanol at 100 % vol,

where T = alcohol content of the sample in % vol determination as described in method 1.

7.2. Repeatability

The difference between the results of two determinations, carried out simultaneously or in rapid succession, by the same analyst, on the same sample, under the same conditions, should not exceed 0,1 g esters, as ethyl acetate, per hl ethanol at 100 % vol.

Method 8: Determination of volatile nitrogen bases

1. SCOPE AND FIELD OF APPLICATION

The method determines volatile nitrogen bases, expressed as nitrogen, in neutral alcohol.

2. DEFINITION

The volatile nitrogen bases content: the content of volatile nitrogen bases, expressed as nitrogen, as determined by the method specified.

3. PRINCIPLE

The sample is evaporated to a small volume in the presence of sulphuric acid and the ammonia content then determined using the conway micro-diffusion technique.

4. REAGENTS

4.1. Sulphuric acid, 1 mol/l.

4.2. Boric acid indicator solution. Dissolve 10 g of boric acid, 8 mg of bromocresol green and 4 mg of methyl red in 30 % vol propan-2-ol and make up to 1 000 ml with 30 % vol propan-2-ol.

4.3. Potassium hydroxide solution, 500 g/l; carbon dioxide free.

4.4. Hydrochloric acid, 0,02 mol/l.

5. APPARATUS

5.1. Evaporating dish, of sufficient capacity to accept 50 ml of sample.

5.2. Water bath.

5.3. Conway flask with tightly fitting lid; see figure 1 for description and suggested dimensions.

5.4. Micro-burette, capacity 2 to 5 ml, graduated in 0,01 ml.

6. PROCEDURE

6.1. Pipette 50 mls of the sample (with an anticipated nitrogen content of less than 0,2 g per hl sample take 200 ml of sample) into a glass dish, add 1 ml of 1 mol/l sulphuric acid (4.1), place the dish (5.1) on a water bath (5.2) and evaporate until there is about 1 ml remaining.

- 6.2. Pipette 1 ml of the boric acid indicator solution (4.2) into the inner chamber of the Conway flask (5.3) and wash the residue liquid from the evaporation process (6.1) into the outer chamber. Slightly tilt the Conway flask and add about 1 ml of the potassium hydroxide solution (4.3) to the outer chamber as quickly as possible but as far from the majority of the liquid in the outer chamber as possible. Immediately seal the Conway flask by covering with a tightly fitting lid smeared with grease.
- 6.3. Mix the two solutions in the outer chamber taking care that there is no liquid spillage from either chamber to the other. Allow to stand for two hours.
- 6.4. Titrate the ammonia in the inner chamber against 0,02 mol/l hydrochloric acid (4.4) using a micro-burette (5.4) to neutralisation. The volume of acid used should be between 0,2 and 0,9 ml; let the volume of acid used be V_1 ml.
- 6.5. Carry out a blank titration by repeating sections 6.1 to 6.4 but replacing the 50 ml of sample in section 6.1 by the same volume of water. Let the volume of hydrochloric acid used be V_2 ml.

7. EXPRESSION OF RESULTS

7.1. Formula and method of calculation.

The content of volatile nitrogen bases, in g per hl ethanol at 100 % vol, calculated and expressed as nitrogen, is given by:

$$\frac{(V_1 - V_2) \cdot 2\,800}{E \cdot T}$$

Where

V_1 = the volume, in ml, of hydrochloric acid used to neutralize the sample.

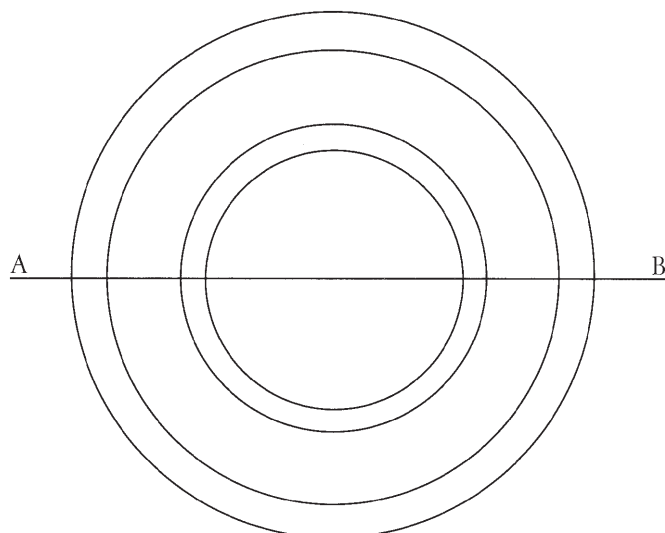
V_2 = the volume, in ml, of hydrochloric acid used in the blank test.

T = the alcoholic strength by volume of the sample as determined by method 1.

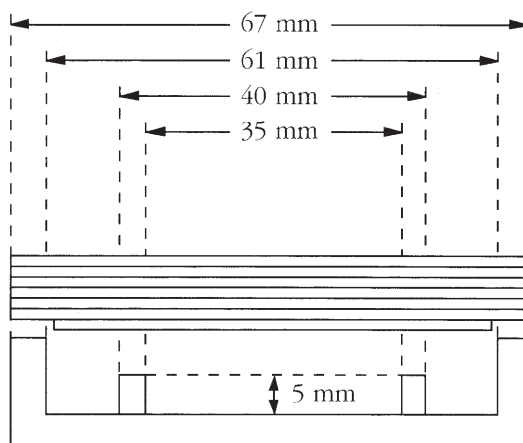
E = the quantity of sample used in ml.

7.2. Repeatability

The difference between the results of two determinations, carried out simultaneously or in rapid succession, by the same analyst, on the same sample, under the same conditions, shall not exceed 0,05 g per hl ethanol at 100 % vol.



Top view of flask



Vertical Section on line A – B

Typical dimension given

Figure 1: Conway flask

Method 9: Determination of methanol

1. SCOPE AND FIELD OF APPLICATION

The method determines the content of methanol in neutral alcohol.

2. DEFINITION

The methanol content: the content of methanol as determined by the method specified.

3. PRINCIPLE

The methanol concentration is determined by direct injection of sample into a gas liquid chromatography apparatus.

4. PROCEDURE

Any GLC method is suitable provided that the gas chromatographic column and conditions employed are capable of achieving a clear separation between methanol, acetaldehyde, ethanol and ethyl acetate. The limit of detection of methanol in ethanol shall be less than 2 g/hl.

5. REPEATABILITY

The difference between the results of two determinations, carried out simultaneously or in rapid succession, by the same analyst, on the same sample, under the same conditions, shall not exceed 2 g methanol per hl ethanol at 100 % vol.

Method 10: Determination of dry residue

1. SCOPE AND FIELD OF APPLICATION

The method determines the dry residue content of neutral alcohol.

2. DEFINITION

The dry residue content: the dry matter content as determined by the method specified.

3. PRINCIPLE

An aliquot of the sample is dried at 103 °C and the residue determined gravimetrically.

4. APPARATUS

4.1. Water bath, boiling.

4.2. Evaporating dish of suitable capacity.

4.3. Desiccator, containing freshly activated silica gel (or an equivalent desiccant) with a moisture content indicator.

4.4. Analytical balance.

4.5. Oven, thermostatically controlled at 103 ± 2 °C.

5. PROCEDURE

Accurately weigh, to the nearest 0,1 mg, a clean dry evaporating dish (4.2) (m_0). Pipette in several operations if necessary a suitable volume of sample into the dish (100 — 250 ml) (V_0 ml). Place the dish with sample on the boiling water bath (4.1) and allow to dry. Place in the oven (4.5) at 103 ± 2 °C for 30 minutes and then transfer dish with residue into a desiccator (4.3). Allow the dish to cool for 30 minutes and then weigh, to the nearest 0,1 mg, the dish with residue (M_1).

6. EXPRESSION OF RESULTS

6.1. Formula and method of calculation

The content of dry residue, to g per hl of ethanol at 100 % vol is given by:

$$\frac{(M_1 - M_0) \cdot 10^7}{V_0 \cdot T}$$

where:

M_0 = the mass, in g, of the clean dry dish,

M_1 = the mass, in g, of the dish and residue after drying,

V_0 = the volume of sample taken for drying, and

T = the alcohols strength by volume of the sample as determined by method 1.

6.2. Repeatability

The difference between the results of two determinations, carried out simultaneously or in rapid succession, by the same analyst, on the same sample, under the same conditions, shall not exceed 0,5 g per hl of ethanol at 100 % vol.

Method 11: Limit test for the absence of furfural

1. SCOPE AND FIELD OF APPLICATION
The method detects furfural in neutral alcohol.
2. DEFINITION
The detection of the limit tests concentration of furfural: the limit test result as determined by the method specified.
3. PRINCIPLE
The alcohol sample is mixed with aniline and glacial acetic acid. The presence of furfural is indicated by a salmon pink colour appearing in the solution within 20 minutes of mixing.
4. REAGENTS
 - 4.1. Aniline, freshly distilled.
 - 4.2. Acetic acid, glacial.
5. APPARATUS
Tubes, fitted with ground glass stoppers.
6. PROCEDURE
Pipette 10 ml of the sample into a tube (5); add 0,5 ml of aniline and 2 ml of glacial acetic acid. Shake the tube and contents to mix.
7. EXPRESSION OF RESULTS
 - 7.1. Limit test interpretation
If the time of development of any salmon pink colouration in the tube is less than 20 minutes the test is positive and the sample contains furfural.
 - 7.2. Observations
The results of two limit tests, carried out simultaneously or in rapid succession, by the same analyst, on the same sample, under the same conditions, shall be identical.

Method 12: UV test

1. SCOPE
This method determines the optical transparenence of neutral alcohol.
2. PRINCIPLE
The optical transparenence of the sample in the wavelength range 220 to 270 nm is measured against a defined reference substance of high optical transparenence.
3. APPARATUS
 - 3.1. UV-VIS spectrophotometer
 - 3.2. Quartz cuvettes, 10 mn path length, same spectrum transmission.
4. REAGENTS
n-Hexane for spectroscopy.
5. PROCEDURE
 - Rinse clean cuvettes with sample solution and then pour in the sample; dry the outside of the cuvettes,
 - treat reference cuvette in the same way with n-Hexane and fill,
 - determine absorbence values and construct graph.

6. EXPRESSION OF RESULTS

The absorbance values found at 270, 240, 230 and 220 nm may not exceed the following figures: 0,02, 0,08, 0,18 and 0,3.

The absorbance curve must be smooth and regular.

Method 13: Determination of ^{14}C content in ethanol

1. METHOD TO DETERMINE TYPE OF ALCOHOL

Determination of the ^{14}C content in ethanol permits a distinction to be made between alcohol from fossil fuels (synthesis alcohol) and alcohol from recent raw materials (fermentation alcohol).

2. DEFINITION

The ^{14}C content of ethanol is understood to be the ^{14}C content determined using the method described here.

The natural ^{14}C content in the atmosphere (the reference value), which is absorbed by living vegetation by assimilation, is not a constant value. The reference value is therefore determined on ethanol from raw materials of the most recent vegetation period. This annual reference value is determined each year by collaborative analyses organised by the Community Bureau of References and the Joint Research Centre, Ispra

3. PRINCIPLE

The ^{14}C content of samples containing alcohol with at least 85 % mass ethanol is determined directly by liquid scintillation count.

4. REAGENTS

4.1. Toluene scintillator

5,0 g 2,5-diphenyloxazole (PPO)

0,5 g p-bis-[4-methyl-5-phenyloxazolyl(2)]-benzene (dimethyl-POPOP) in 1 litre analytical grade toluene.

Commercial, ready-to-use toluene scintillators of this composition can also be used.

4.2. ^{14}C standard

n-Hexadecane ^{14}C with an activity of about 1×10^6 dpm/g (approximately $1,67 \times 10^6$ cBq/g) and a guaranteed accuracy of determined activity of ± 2 % rel.

4.3. ^{14}C -free ethanol

Synthesis alcohol from raw materials of fossil origin with at least 85 % mass ethanol, to determine the background.

4.4. Alcohol from recent raw materials of the most recent vegetation period with at least 85 % mass ethanol as reference material.

5. APPARATUS

5.1. Multi-channel liquid scintillation spectrometer with processor and automatic external standardisation and display of the external standard/channel ratio (usual design: three meter channels and two external standard channels).

5.2. Low-potassium counter tubes suitable for the spectrometer, with dark screw-tops containing a polyethylene insert.

5.3. Volumetric pipettes, 10 ml.

5.4. Automatic dosing device 10 ml.

5.5. 250 ml round-bottom flask with ground-glass stopper.

- 5.6. Alcohol distillation apparatus with heating mantle, e.g. type Micko.
- 5.7. Microliter syringe 50 μ l.
- 5.8. Pycnometer funnel, pycnometers, 25 ml and 50 ml.
- 5.9. Thermostat with a temperature stability of $\pm 0,01$ $^{\circ}$ C.
- 5.10. Official alcohol tables in accordance with the Council Directive of 27 July 1976 on the approximation of the laws of Member States relating to alcohol tables, published by the Commission of the European Communities (ISBN 92-825-0146-9).

6. PROCEDURE

6.1. Adjusting the equipment

The equipment should be adjusted according to the manufacturer's instructions. Measuring conditions are optimal when the value E_2/B , the quality index, is at its maximum.

E = efficiency

B = background

Only two meter channels are optimised. The third is left fully open for control purposes.

6.2. Selection of counter tubes

A larger number of counter tubes than will later be needed are each filled with 10 ml of 14 C-free synthesis ethanol and 10 ml toluene scintillator. Each is measured for at least $\times 100$ minutes. Tubes whose backgrounds vary by more than ± 1 % rel. from the mean are discarded. Only tubes new from the factory and from the same batch may be used.

6.3. Determination of the external standard/channel ratio (ESCR)

During the process of setting the channels (6.1) the ESCR is determined using the appropriate computer program when the efficiency is determined. The external standard used is 137 caesium, which is already built-in by the manufacturer.

6.4. Preparation of sample

Samples having an ethanol content of at least 85 % mass and free from impurities, which absorb at wavelengths below 450 nm can be measured. The low residue of esters and aldehydes is not a problem. After the first few ml have been discarded the sample is distilled direct into the pycnometer and the alcohol content of the sample is determined by pycnometry. The values to be determined are taken from the Official Alcohol Tables.

7. MEASUREMENT OF SAMPLES USING EXTERNAL STANDARD

7.1. Slightly extinguished samples such as those described in 6.4 with an ESCR of around 1.8 may be measured via the ESCR, which provides a measure of the efficiency.

7.2. Measurement

10 ml each of the samples prepared according to 6.4 is pipettes into a selected counter tube checked for background and 10 ml of toluene scintillator is added via an automatic dosing device. The samples in the tubes are homogenised by suitable rotary movements; the liquid must not be allowed to wet the polyethylene insert in the screw-top. A tube containing 14 C-free fossil ethanol is prepared in the same way to measure the background. To check the relevant annual 14 C value a duplicate of recent ethanol from the latest vegetation period is prepared, a tube being mixed with internal standard, see 8.

The control and background samples are placed at the beginning of the measurement series, which should contain no more than 10 samples for analysis. Total measuring time per sample is at least 2×100 minutes, with the individual samples being measured in part stages of 100 minutes so that any equipment drift or other defect can be detected. (One cycle therefore corresponds to a measuring interval of 100 minutes per sample).

Background and control samples should be freshly prepared every four weeks.

This method requires little time and material and is particularly suitable for non-specialist laboratories processing large numbers of samples.

In the case of slightly extinguished samples (ESCR circa 1,8) the efficiency is only negligibly affected by the change in this value. If the change is within $\pm 5\%$ rel. the same efficiency can be expected. For more greatly extinguished samples, such as denatured alcohols, the efficiency can be established via the extinction correction graph. If an appropriate computer program is not available the internal standard must be used, and this gives an unambiguous result.

8. MEASURING SAMPLES USING INTERNAL STANDARD HEXADECANE¹⁴C

8.1. Procedure

Control and background samples (recent and fossil ethanol) and the unknown material are each measured as duplicates. One sample of the duplicate is prepared in a non-selected tube and an accurately dosed quantity (30 μ l) of hexadecane¹⁴C is added as internal standard (added activity around 26 269 dpm/gC approximately 43 782 cBq/gC). For the sample preparation and measuring time of the other samples see 7.2, but the measuring time for the samples with the internal standard can be reduced to about five minutes by presetting at 10^5 pulses. One duplicate each of background and control samples is used per measuring series; these are placed at the beginning of the measuring series.

8.2. Handling the internal standard and counter tubes

To prevent contamination when measuring with the internal standard these must be stored and handled well away from the area where the samples for analysis are prepared and measured. After measurement the tubes checked for background may be re-used. The screw-tops and tubes containing the internal standard are disposed of.

9. EXPRESSION OF THE RESULTS

9.1. The unit of activity of a radio-active substance is the becquerel; 1 Bq = 1 decay/sec.

Indication of specific radio-activity is expressed as becquerels relative to one gram carbon = Bq/gC.

To obtain more practical results it is best to express the results in centi-becquerels = cBq/gC.

The descriptions and formulae used in the literature, based on dpm, may be retained for the time being. To obtain corresponding figures in cBq merely multiply the dpm figure by 100/60.

9.2. Expression of results with external standard

$$\text{cBq/g C} = \frac{(\text{cpm}_{\text{pr}} - \text{cpm}_{\text{NE}}) \cdot 1,918 \cdot 100}{V \cdot F \cdot Z \cdot 60}$$

9.3. Expression of results with internal standard

$$\text{cBq/g C} = \frac{(\text{cpm}_{\text{pr}} - \text{cpm}_{\text{NE}}) \cdot \text{dpm}_{\text{IS}} \cdot 1,918 \cdot 100}{(\text{cpm}_{\text{IS}} - \text{cpm}_{\text{pr}}) \cdot V \cdot F \cdot 60}$$

9.4. Abbreviations

cpm_{pr} = the mean sample count rate over the total measuring time.

cpm_{NE} = the mean background pulse rate calculated in the same way.

cpm_{IS} = the amount of added internal standard added (calibration radioactivity dpm).

dpm_{IS} = the quantity of internal standard added (calibration radioactivity dpm).

V = the volume of the samples used in ml.

F = the content in grammes pure alcohol per ml corresponding to its concentration.

Z = the efficiency corresponding to the ESCR value.

1,918 = the number of grammes alcohol per gramme carbon.

10. RELIABILITY OF THE METHOD

10.1. Repeatability (r)

$$r = 0,632 \text{ cBq/g C}; S_{(r)} = \pm 0,223 \text{ cBq/g C}$$

10.2. Comparability (R)

$$R = 0,821 \text{ cBq/g C}; S_{(R)} = \pm 0,290 \text{ cBq/g C.}$$
