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(Acts whose publication is obligatory)

COMMISSION REGULATION (EEC) No 2984/78

of 17 November 1978

determining Community methods for the analysis of wines and repealing Regulation (EEC) No 1539/71

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Regulation (EEC) No 816/70 of 28 April 1970 laying down additional provisions for the common organization of the market in wine (1), as last amended by Regulation (EEC) No 1861/78 (2), and in particular Article 39 thereof,

Having regard to Council Regulation (EEC) No 817/70 of 28 April 1970 laying down special provisions for quality wines produced in specified regions (3), as last amended by Regulation (EEC) No 2211/77 (4), and in particular Article 11 (3) thereof,

Whereas Article 39 (2) of Regulation (EEC) No 816/70 prescribes the adoption of methods of analysis for establishing the composition of the products indicated in Article 1 of that Regulation and of rules for checking whether these products have been subjected to treatments in violation of authorized oenological practice;

Whereas Article 11 (1) of Regulation (EEC) No 817/70 provides for an analytical test, including at the least an assessment of the characteristics, as listed in the Annex to that Regulation, of the quality wine psr in question;

Whereas the verification of the particulars on documents concerning the products in question calls for the introduction of uniform methods of analysis to ensure that accurate and comparable information is obtained; whereas, consequently, these methods should be compulsory for all commercial transactions and all verification procedures; whereas, however, in view of control requirements and the trade's limited facilities, a restricted number of usual procedures should be admitted enabling the requisite factors to be determined rapidly and with reasonable accuracy;

Whereas generally recognized methods such as those developed under the 1954 International Convention for the Unification of Methods for the Analysis and Appraisal of Wines, which are set out in the Annex to Commission Regulation (EEC) No 1539/71 of 19 July 1971 determining Community methods for the analysis of wines (5) may usefully be retained;

Whereas, however, Regulation (EEC) No 1539/71 indicates the various analysis methods recognized by the Convention but does not describe them in detail; whereas consequently a large number of interested persons are forced to deal with these methods in a language other than their official language; whereas this is a considerable obstacle to a proper understanding of Community law; whereas all the methods of analysis recognized by Community law should therefore be published in all the official languages of the Community in the Annex to this Regulation;

Whereas, pending the adoption of Community provisions on the subject, it is essential to permit Member States, in order to assess whether a product has been subjected to treatments or practices which are not in accordance with Community provisions, to fix the levels of the constituents which

(1) OJ No L 99, 5. 5. 1970, p. 1.
(2) OJ No L 215, 4. 8. 1978, p. 1.
(3) OJ No L 99, 5. 5. 1970, p. 20.
(4) OJ No L 256, 7. 10. 1977, p. 1.
(5) OJ No L 163, 21. 7. 1971, p. 41.

indicate that certain oenological practices have been used and to require the use of tables enabling a comparison of analysis data; whereas in the absence of a method for detecting the improper enrichment of wine, it is desirable to authorize the Member States, until 31 August 1979 at the latest, to determine the reduced dry matter by the method which they used before 19 July 1971 only in the context of checking the proportion of alcohol to dry matter; whereas it is therefore necessary to repeal Regulation (EEC) No 1539/71;

Whereas 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

1. The Annex to this Regulation sets out Community methods, applying to commercial transactions and all control operations, for

— establishing the composition of the products indicated in Article 1 of Regulation (EEC) No 816/70,

— checking whether these products have been subjected to treatments in violation of authorized oenological practice.

2. For products for which reference methods and customary methods are laid down, the results obtained by the application of the reference methods shall prevail.

3. The methods of analysis referred to in Article 39 (3) of Regulation (EEC) No 816/70 may be used for analyzing substances for which no method of analysis is specified in the Annex to this Regulation or for supplementing or supporting the results given by the reference method.

4. Until Community provisions are adopted on the levels of the constituents which indicate that certain oenological practices have been employed and on tables enabling a comparison of the analysis data, the Member States may, in order to assess whether a product has been subjected to treatments or practices which are not in accordance with Community provisions,

— determine the levels of constituents in that product,

— stipulate the use of tables enabling a comparison of analysis data,

— until 31 August 1979 at the latest, determine the reduced dry matter by the method which they used before 19 July 1971 only in the context of checking the proportion of alcohol to dry matter.

Article 2

Regulation (EEC) No 1539/71 is hereby repealed.

Article 3

This Regulation shall enter into force on 1 January 1979.

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

Done at Brussels, 17 November 1978.

For the Commission

Finn GUNDELACH

Vice-President

ANNEX

1. DENSITY AND SPECIFIC GRAVITY AT 20 °C

1.1. DEFINITIONS

The density and the specific gravity of wine and of must are determined at 20 °C.

The density (ϱ) is the mass per unit volume of wine or must at 20 °C. It is expressed in grams per cubic centimetre ([1]).

The specific gravity, d_{20}^{20} or d, is the ratio of the density of wine (or of must) at 20 °C to the density of water at the same temperature.

On the certificates of analysis the characteristic considered shall be specified using only the symbols defined above. It is preferable to use only density at 20 °C.

Conventionally, the density and the specific gravity shall be subject to correction for the effect of sulphur dioxide and any preservative added at the time of sampling for stabilizing the wine or must sample.

1.2. PRINCIPLE OF METHODS

Reference method: measurement by pycnometry, results accurate to 0·0001.

First usual method: measurement by hydrometry, results accurate to 0·0003.

Second usual method: measurement by densimetry by the hydrostatic balance, results accurate to 0·0003.

Preliminary treatment

Any excess carbon dioxide in the wine or must may be removed by stirring 250 ml in a one litre flask or by filtration at reduced pressure through 2 g of cotton wool.

If the wine or must is cloudy it should either be filtered or centrifuged in sealed tubes The use of either procedure should be stated on the certificate of analysis.

1.3. REFERENCE METHOD — MEASUREMENT BY A PYCNOMETER — APPARATUS

A Pyrex pycnometer, of approximately 100 ml capacity, with a ground glass jointed thermometer calibrated in 10ths of a degree from 10 to 30 °C, is used.

The pycnometer comprises a side tube of 15 mm length and of 1 mm (maximum) inside diameter ending in a conical ground joint. This side tube may be capped by a 'receiver plug' consisting of a conical ground joint tube terminating in a drawn-out portion. This plug serves as an expansion chamber. The thermometer should be carefully checked and the two ground joints of the apparatus should be prepared with great care. They may be greased lightly with a trace of silicone.

A vessel of the same outside volume as the pycnometer is used as a tare. It is filled almost completely with a solution of a metal salt such as sodium chloride, potassium chromate, etc., of a specific gravity such that the mass of this standard is equal to the mass of the pycnometer filled with a liquid of density 1·01 ([2]). This vessel is sealed in a flame. Its outside capacity should be equal to the outside capacity of the filled pycnometer to within 1 ml.

Taring of the pycnometer

The empty pycnometer shall be tared using a balance with a sensitivity of 0·1 mg and a maximum range of at least 200 g, using precision weights.

([1]) Or grams per millilitre since 1 ml is now considered as identical to 1 cm^3.

([2]) The pycnometer thus calibrated is ready for measuring specific gravities which are less than or slightly greater than that of water, such as the specific gravities of hydroalcoholic distillates, of dry wines. For the measurement of liquids of a specific gravity appreciably greater than one, such as sweet wines and musts, the same tare shall be used with the addition of a brass weight of 20 g. The volume of this extra load is small and the variations in the buoyancy of the air on it are negligible. In the case of these liquids the density need not be measured with the high accuracy necessary for hydroalcoholic distillates (measurement of alcoholic strength).

Add the mass of air contained in the empty and dry pycnometer (1) to the weights, p, placed beside the pycnometer. The corrected tare of the empty pycnometer is thus obtained.

1·2 g per litre may be assumed as the density of air (2) for the calculation of the mass of air m contained in the pycnometer.

The mass of the air m contained in the apparatus is obtained by multiplying the volume of the pycnometer in millilitres by 0·0012. As it is not necessary to know m with an accuracy greater than to the nearest 0·5 mg, this volume is numerically equal to $p - p'$, p' being the weight required to balance the pycnometer filled with water.

Tare the water-filled pycnometer at t °C, t being the temperature at which the operation is carried out as shown on the pycnometer thermometer, measured to 0·02 °C, and p' being the weight in grams required to establish equilibrium.

Calculate the volume of the pycnometer at 20 °C (3) by multiplying the mass of water at t °C contained in the pycnometer at t °C by the factor F given in Table I. This table gives the reciprocals of the densities of water at t °C corrected for the expansion of the Pyrex pycnometer between t and 20 °C.

The knowledge of this volume is necessary for the measurement of the densities of liquids.

If the specific gravity is to be calculated, it is necessary to know the mass of the water at 20 °C contained in the pycnometer at 20 °C. This is obtained by multiplying the volume at 20 °C by 0·998203, the density of water at 20 °C.

The calibration operation should be carried out in triplicate to check that there is no error in this fundamental operation (4).

The tare of the empty pycnometer and the volume at 20 °C (and consequently the mass of the water at 20 °C) are characteristic of the equipment. Because a tare of the same volume as the full pycnometer is used it is not necessary to determine them at each measurement. It is recommended, however, to check them periodically, e.g. annually.

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(1) The correction for the buoyancy of the air is thus made once and for all.

(2) Strictly speaking, it would be necessary to note the temperature of the pycnometer and the atmospheric pressure and to calculate the density of the air under these conditions. In practice such a calculation would be necessary only when working at an altitude higher than 1 400 m and at a temperature above 30 °C. A variation of 10% in the density of the air produces an error of $5 \cdot 10^{-6}$ in the specific gravity of a wine distillate and a maximum error of 0·005 in the alcoholic strength measured; this is negligible.

(3) The volume so calculated is not strictly accurate, as the buoyancy of the air on the weights recorded has not been taken into account, these weights generally not being corrected for this buoyancy. However it is preferable to maintain this non-corrected, if fictitious, value so as to avoid introducing a correction of the same nature but inverse in the actual measurements. The real volume can be calculated by multiplying the fictitious volume by 0·999857.

(4) It is possible to avoid any calculation by calibrating the pycnometer filled with water at 20 or 15 °C, the temperature reading being made by magnifying glass to the nearest 0·02 °C including any correction for the thermometer scale.

TABLE I

F factors

by which the mass of the water contained in the Pyrex pycnometer at t °C has to be multiplied to calculate the volume of the pycnometer at 20 °C.

t °C	F	t °C	F	t °C	F	t °C	F	t °C	F	t °C	F	t °C	F
10·0	1·000398	13·0	1·000691	16·0	1·001097	19·0	1·001608	22·0	1·002215	25·0	1·002916	28·0	1·003704
·1	1·000406	·1	1·000703	·1	1·001113	·1	1·001627	·1	1·002238	·1	1·002941	·1	1·003731
·2	1·000414	·2	1·000714	·2	1·001128	·2	1·001646	·2	1·002260	·2	1·002966	·2	1·003759
·3	1·000422	·3	1·000726	·3	1·001144	·3	1·001665	·3	1·002282	·3	1·002990	·3	1·003787
·4	1·000430	·4	1·000738	·4	1·001159	·4	1·001684	·4	1·002304	·4	1·003015	·4	1·003815
10·5	1·000439	13·5	1·000752	16·5	1·001175	19·5	1·001703	22·5	1·002326	25·5	1·003041	28·5	1·003843
·6	1·000447	·6	1·000764	·6	1·001191	·6	1·001722	·6	1·002349	·6	1·003066	·6	1·003871
·7	1·000456	·7	1·000777	·7	1·001207	·7	1·001741	·7	1·002372	·7	1·003092	·7	1·003899
·8	1·000465	·8	1·000789	·8	1·001223	·8	1·001761	·8	1·002394	·8	1·003117	·8	1·003928
·9	1·000474	·9	1·000803	·9	1·001239	·9	1·001780	·9	1·002417	·9	1·003143	·9	1·003956
11·0	1·000483	14·0	1·000816	17·0	1·001257	20·0	1·001800	23·0	1·002439	26·0	1·003168	29·0	1·003984
·1	1·000492	·1	1·000829	·1	1·001273	·1	1·001819	·1	1·002462	·1	1·003194	·1	1·004013
·2	1·000501	·2	1·000842	·2	1·001290	·2	1·001839	·2	1·002485	·2	1·003222	·2	1·004042
·3	1·000511	·3	1·000855	·3	1·001306	·3	1·001859	·3	1·002508	·3	1·003247	·3	1·004071
·4	1·000520	·4	1·000868	·4	1·001323	·4	1·001880	·4	1·002531	·4	1·003273	·4	1·004099
11·5	1·000530	14·5	1·000882	17·5	1·001340	20·5	1·001900	23·5	1·002555	26·5	1·003299	29·5	1·004128
·6	1·000540	·6	1·000895	·6	1·001357	·6	1·001920	·6	1·002578	·6	1·003326	·6	1·004158
·7	1·000550	·7	1·000909	·7	1·001374	·7	1·001941	·7	1·002602	·7	1·003352	·7	1·004187
·8	1·000560	·8	1·000923	·8	1·001391	·8	1·001961	·8	1·002625	·8	1·003379	·8	1·004216
·9	1·000570	·9	1·000937	·9	1·001409	·9	1·001982	·9	1·002649	·9	1·003405	·9	1·004245
12·0	1·000580	15·0	1·000951	18·0	1·001427	21·0	1·002002	24·0	1·002672	27·0	1·003432	30·0	1·004275
·1	1·000591	·1	1·000965	·1	1·001445	·1	1·002023	·1	1·002696	·1	1·003458		
·2	1·000601	·2	1·000979	·2	1·001462	·2	1·002044	·2	1·002720	·2	1·003485		
·3	1·000612	·3	1·000993	·3	1·001480	·3	1·002065	·3	1·002745	·3	1·003513		
·4	1·000623	·4	1·001008	·4	1·001498	·4	1·002086	·4	1·002769	·4	1·003540		
12·5	1·000634	15·5	1·001022	18·5	1·001516	21·5	1·002107	24·5	1·002793	27·5	1·003567		
·6	1·000645	·6	1·001037	·6	1·001534	·6	1·002129	·6	1·002817	·6	1·003594		
·7	1·000656	·7	1·001052	·7	1·001552	·7	1·002151	·7	1·002842	·7	1·003621		
·8	1·000668	·8	1·001067	·8	1·001570	·8	1·002172	·8	1·002866	·8	1·003649		
·9	1·000679	·9	1·001082	·9	1·001589	·9	1·002194	·9	1·002891	·9	1·003676		

Procedure

Carefully fill the pycnometer with the liquid at ambient temperature and place in the thermally insulated enclosure; shake the liquid two or three times by inverting the container until the temperature reading on the thermometer is constant. Accurately adjust the level to the upper rim of the lateral tube. Wipe the lateral tube dry, put the receiver plug on and measure the temperature t °C with the same care as previously; carefully wipe the pycnometer dry and then weigh, p″ being the weight to give equilibrium.

The difference between the corrected tare and the weight recorded gives the masses of the liquid in the apparatus (corrected for buoyancy of the air).

Calculate the apparent density at t °C by dividing this mass by the volume at 20 °C, and the apparent specific gravity d^t_{20} by dividing this same mass by the mass of the water contained at 20 °C in the pycnometer.

1. *Correction for temperature*

   Correct ϱ_t and d^t_{20} using one of the following correction tables in accordance with the nature of the liquid measured: dry wine (Table II), natural or concentrated must (Table III), sweet wine (Table IV), alcohol-free residue (wine freed of alcohol by distillation and the residue from the distillation made up to the original volume) (Table II). In the compilation of these tables the expansion of the liquids and of the container between t and 20 °C has been taken into account.

2. *Corrections made necessary by the presence of preservatives*

   The density must subsequently be corrected for the effect of sulphur dioxide by using the formula:

$$\varrho_{20} = \varrho'_{20} - 0{\cdot}0006S$$

   where:

   ϱ'_{20} is the observed density,

   ϱ_{20} is the corrected density,

   S is the total quantity of sulphur dioxide in milligrams per litre.

   For sodium salicylate subtract 0·00043 (concentration in grams per litre)

   For salicylic acid subtract 0·00031 (concentration in grams per litre).

   The specific gravity d^{20}_{20} should be subject to identical corrections.

TABLE II

Temperature corrections c to the density of dry wines and of alcohol free dry wines measured in a Pyrex glass pycnometer at t °C to relate the result to 20 °C

$$\varrho_{20} = \varrho_t \pm \frac{c}{1\,000} \begin{cases} - \text{ if t °C is lower than 20 °C} \\ + \text{ if t °C is higher than 20 °C} \end{cases}$$

Temperature (°C)	Alcoholic strengths																							
	0	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27
10	1·59	1·64	1·67	1·71	1·77	1·84	1·91	2·01	2·11	2·22	2·34	2·46	2·60	2·73	2·88	3·03	3·19	3·35	3·52	3·70	3·87	4·06	4·25	4·44
11	1·48	1·53	1·56	1·60	1·64	1·70	1·77	1·86	1·95	2·05	2·16	2·27	2·38	2·51	2·63	2·77	2·91	3·06	3·21	3·36	3·53	3·69	3·86	4·03
12	1·36	1·40	1·43	1·46	1·50	1·56	1·62	1·69	1·78	1·86	1·96	2·05	2·16	2·27	2·38	2·50	2·62	2·75	2·88	3·02	3·16	3·31	3·46	3·61
13	1·22	1·26	1·28	1·32	1·35	1·40	1·45	1·52	1·59	1·67	1·75	1·83	1·92	2·01	2·11	2·22	2·32	2·44	2·55	2·67	2·79	2·92	3·05	3·18
14	1·08	1·11	1·13	1·16	1·19	1·23	1·27	1·33	1·39	1·46	1·52	1·60	1·67	1·75	1·84	1·93	2·03	2·11	2·21	2·31	2·42	2·52	2·63	2·74
15	0·92	0·96	0·97	0·99	1·02	1·05	1·09	1·13	1·19	1·24	1·30	1·36	1·42	1·48	1·55	1·63	1·70	1·78	1·86	1·95	2·03	2·12	2·21	2·30
16	0·76	0·79	0·80	0·81	0·84	0·86	0·89	0·93	0·97	1·01	1·06	1·10	1·16	1·21	1·26	1·32	1·38	1·44	1·51	1·57	1·64	1·71	1·78	1·85
17	0·59	0·61	0·62	0·63	0·65	0·67	0·69	0·72	0·75	0·78	0·81	0·85	0·88	0·95	0·96	1·01	1·05	1·11	1·15	1·20	1·25	1·30	1·35	1·40
18	0·40	0·42	0·42	0·43	0·44	0·46	0·47	0·49	0·51	0·53	0·55	0·57	0·60	0·63	0·65	0·68	0·71	0·74	0·77	0·81	0·84	0·87	0·91	0·94
19	0·21	0·21	0·22	0·22	0·23	0·23	0·24	0·25	0·26	0·27	0·28	0·29	0·30	0·32	0·33	0·34	0·36	0·37	0·39	0·41	0·42	0·44	0·46	0·47
20																								
21	0·21	0·22	0·22	0·23	0·23	0·24	0·25	0·26	0·27	0·28	0·29	0·30	0·31	0·32	0·34	0·36	0·37	0·38	0·40	0·41	0·43	0·44	0·46	0·48
22	0·44	0·45	0·46	0·47	0·48	0·49	0·51	0·52	0·54	0·56	0·59	0·61	0·63	0·66	0·69	0·71	0·74	0·77	0·80	0·83	0·87	0·90	0·93	0·97
23	0·68	0·70	0·71	0·72	0·74	0·76	0·78	0·80	0·83	0·86	0·90	0·93	0·96	1·00	1·03	1·08	1·13	1·17	1·22	1·26	1·31	1·37	1·41	1·46
24	0·93	0·96	0·97	0·99	1·01	1·03	1·06	1·10	1·13	1·18	1·22	1·26	1·31	1·36	1·41	1·47	1·52	1·58	1·64	1·71	1·77	1·84	1·90	1·97
25	1·19	1·23	1·25	1·27	1·29	1·32	1·36	1·40	1·45	1·50	1·55	1·61	1·67	1·73	1·80	1·86	1·93	2·00	2·08	2·16	2·24	2·32	2·40	2·48
26	1·47	1·51	1·53	1·56	1·59	1·62	1·67	1·72	1·77	1·83	1·90	1·96	2·03	2·11	2·19	2·27	2·35	2·44	2·53	2·62	2·72	2·81	2·91	3·01
27	1·75	1·80	1·82	1·85	1·89	1·93	1·98	2·04	2·11	2·18	2·25	2·33	2·41	2·50	2·59	2·68	2·78	2·88	2·98	3·09	3·20	3·31	3·42	3·53
28	2·04	2·10	2·13	2·16	2·20	2·25	2·31	2·38	2·45	2·53	2·62	2·70	2·80	2·89	3·00	3·10	3·21	3·32	3·45	3·57	3·69	3·82	3·94	4·07
29	2·34	2·41	2·44	2·48	2·53	2·58	2·65	2·72	2·81	2·89	2·99	3·09	3·19	3·30	3·42	3·53	3·65	3·78	3·92	4·05	4·19	4·33	4·47	4·61
30	2·66	2·73	2·77	2·81	2·86	2·92	3·00	3·08	3·17	3·27	3·37	3·48	3·59	3·72	3·84	3·97	4·11	4·25	4·40	4·55	4·70	4·85	4·92	5·17

Note: This table can be used for converting the specific gravity d^t_{20} to the specific gravity d^{20}_{20}.

TABLE III

Temperature corrections c to the density of natural musts and of concentrated musts measured with a Pyrex glass pycnometer or hydrometer to relate the result to 20 °C

$$\varrho_{20} = \varrho_t \pm \frac{c}{1\,000} \begin{cases} - \text{ if t °C is lower than 20 °C} \\ + \text{ if t °C is higher than 20 °C} \end{cases}$$

Temperature (°C)	Densities																					
	1·05	1·06	1·07	1·08	1·09	1·10	1·11	1·12	1·13	1·14	1·15	1·16	1·18	1·20	1·22	1·24	1·26	1·28	1·30	1·32	1·34	1·36
10	2·31	2·48	2·66	2·82	2·99	3·13	3·30	3·44	3·59	3·73	3·88	4·01	4·28	4·52	4·76	4·98	5·18	5·42	5·56	5·73	5·90	6·05
11	2·12	2·28	2·42	2·57	2·72	2·86	2·99	3·12	3·25	3·37	3·50	3·62	3·85	4·08	4·29	4·48	4·67	4·84	5·00	5·16	5·31	5·45
12	1·92	2·06	2·19	2·32	2·45	2·58	2·70	2·82	2·94	3·04	3·15	3·26	3·47	3·67	3·85	4·03	4·20	4·36	4·51	4·65	4·78	4·91
13	1·72	1·84	1·95	2·06	2·17	2·27	2·38	2·48	2·58	2·69	2·78	2·88	3·05	3·22	3·39	3·55	3·65	3·84	3·98	4·11	4·24	4·36
14	1·52	1·62	1·72	1·81	1·90	2·00	2·09	2·17	2·26	2·34	2·43	2·51	2·66	2·82	2·96	3·09	3·22	3·34	3·45	3·56	3·67	3·76
15	1·28	1·36	1·44	1·52	1·60	1·67	1·75	1·82	1·89	1·96	2·04	2·11	2·24	2·36	2·48	2·59	2·69	2·79	2·88	2·97	3·03	3·10
16	1·05	1·12	1·18	1·25	1·31	1·37	1·43	1·49	1·55	1·60	1·66	1·71	1·81	1·90	2·00	2·08	2·16	2·24	2·30	2·37	2·43	2·49
17	0·80	0·86	0·90	0·95	1·00	1·04	1·09	1·13	1·18	1·22	1·26	1·30	1·37	1·44	1·51	1·57	1·62	1·68	1·72	1·76	1·80	1·84
18	0·56	0·59	0·62	0·66	0·68	0·72	0·75	0·77	0·80	0·83	0·85	0·88	0·93	0·98	1·02	1·05	1·09	1·12	1·16	1·19	1·21	1·24
19	0·29	0·31	0·32	0·34	0·36	0·37	0·39	0·40	0·42	0·43	0·44	0·45	0·48	0·50	0·52	0·54	0·56	0·57	0·59	0·60	0·61	0·62
20																						
21	0·29	0·30	0·32	0·34	0·35	0·37	0·38	0·40	0·41	0·42	0·44	0·46	0·48	0·50	0·53	0·56	0·58	0·59	0·60	0·61	0·62	0·62
22	0·58	0·61	0·64	0·67	0·70	0·73	0·76	0·79	0·81	0·84	0·87	0·90	0·96	1·00	1·05	1·09	1·12	1·15	1·18	1·20	1·22	1·23
23	0·89	0·94	0·99	1·03	1·08	1·12	1·16	1·20	1·25	1·29	1·33	1·37	1·44	1·51	1·57	1·63	1·67	1·73	1·77	1·80	1·82	1·84
24	1·20	1·25	1·31	1·37	1·43	1·49	1·54	1·60	1·66	1·71	1·77	1·82	1·92	2·01	2·10	2·17	2·24	2·30	2·36	2·40	2·42	2·44
25	1·51	1·59	1·66	1·74	1·81	1·88	1·95	2·02	2·09	2·16	2·23	2·30	2·42	2·53	2·63	2·72	2·82	2·89	2·95	2·99	3·01	3·05
26	1·84	1·92	2·01	2·10	2·18	2·26	2·34	2·42	2·50	2·58	2·65	2·73	2·87	3·00	3·13	3·25	3·36	3·47	3·57	3·65	3·72	3·79
27	2·17	2·26	2·36	2·46	2·56	2·66	2·75	2·84	2·93	3·01	3·10	3·18	3·35	3·50	3·66	3·80	3·93	4·06	4·16	4·26	4·35	4·42
28	2·50	2·62	2·74	2·85	2·96	3·07	3·18	3·28	3·40	3·50	3·60	3·69	3·87	4·04	4·21	4·36	4·50	4·64	4·75	4·86	4·94	5·00
29	2·86	2·98	3·10	3·22	3·35	3·47	3·59	3·70	3·82	3·93	4·03	4·14	4·34	4·53	4·72	4·89	5·05	5·20	5·34	5·46	5·56	5·64
30	3·20	3·35	3·49	3·64	3·77	3·91	4·05	4·17	4·30	4·43	4·55	4·67	4·90	5·12	5·39	5·51	5·68	5·84	5·96	6·08	6·16	6·22

Note: This table can be used for converting the specific gravity d^t_{20} to the specific gravity d^{20}_{20}.

TABLE IV

Temperature corrections c to the density of wines of 13% vol or more measured with a Pyrex glass pycnometer at t °C to relate the result to 20 °C

$$\varrho_{20} = \varrho_t \pm \frac{c}{1\,000} \begin{cases} - \text{ if t °C is lower than 20 °C} \\ + \text{ if t °C is higher than 20 °C} \end{cases}$$

Temperature (°C)	Wines of 13 % vol							Wines of 15 % vol							Wines of 17 % vol						
	Densities							Densities							Densities						
	1·000	1·020	1·040	1·060	1·080	1·100	1·120	1·000	1·020	1·040	1·060	1·080	1·100	1·120	1·000	1·020	1·040	1·060	1·080	1·100	1·120
10	2·36	2·71	3·06	3·42	3·72	3·96	4·32	2·64	2·99	3·36	3·68	3·99	4·30	4·59	2·94	3·29	3·64	3·98	4·29	4·60	4·89
11	2·17	2·49	2·80	2·99	3·39	3·65	3·90	2·42	2·73	3·05	3·34	3·63	3·89	4·15	2·69	3·00	3·32	3·61	3·90	4·16	4·41
12	1·97	2·25	2·53	2·79	3·05	3·29	3·52	2·19	2·47	2·75	3·01	3·27	3·51	3·73	2·42	2·70	2·98	3·24	3·50	3·74	3·96
13	1·78	2·02	2·25	2·47	2·69	2·89	3·09	1·97	2·21	2·44	2·66	2·87	3·08	3·29	2·18	2·42	2·64	2·87	3·08	3·29	3·49
14	1·57	1·78	1·98	2·16	2·35	2·53	2·70	1·74	1·94	2·14	2·32	2·52	2·69	2·86	1·91	2·11	2·31	2·50	2·69	2·86	3·03
15	1·32	1·49	1·66	1·82	1·97	2·12	2·26	1·46	1·63	1·79	1·95	2·10	2·25	2·39	1·60	1·77	1·93	2·09	2·24	2·39	2·53
16	1·08	1·22	1·36	1·48	1·61	1·73	1·84	1·18	1·32	1·46	1·59	1·71	1·83	1·94	1·30	1·44	1·58	1·71	1·83	1·95	2·06
17	0·83	0·94	1·04	1·13	1·22	1·31	1·40	0·91	1·02	1·12	1·21	1·30	1·39	1·48	1·00	1·10	1·20	1·30	1·39	1·48	1·56
18	0·58	0·64	0·71	0·78	0·84	0·89	0·95	0·63	0·69	0·76	0·83	0·89	0·94	1·00	0·69	0·75	0·82	0·89	0·95	1·00	1·06
19	0·30	0·34	0·37	0·40	0·43	0·46	0·49	0·33	0·37	0·40	0·43	0·46	0·49	0·52	0·36	0·39	0·42	0·46	0·49	0·52	0·54
20																					
21	0·30	0·33	0·36	0·40	0·43	0·46	0·49	0·33	0·36	0·39	0·43	0·46	0·49	0·51	0·35	0·39	0·42	0·45	0·48	0·51	0·54
22	0·60	0·67	0·73	0·80	0·85	0·91	0·98	0·65	0·72	0·78	0·84	0·90	0·96	1·01	0·71	0·78	0·84	0·90	0·96	1·01	1·07
23	0·93	1·02	1·12	1·22	1·30	1·39	1·49	1·01	1·10	1·20	1·29	1·38	1·46	1·55	1·10	1·19	1·29	1·38	1·46	1·55	1·63
24	1·27	1·39	1·50	1·61	1·74	1·84	1·95	1·37	1·49	1·59	1·72	1·84	1·95	2·06	1·48	1·60	1·71	1·83	1·95	2·06	2·17
25	1·61	1·75	1·90	2·05	2·19	2·33	2·47	1·73	1·87	2·02	2·17	2·31	2·45	2·59	1·87	2·01	2·16	2·31	2·45	2·59	2·73
26	1·94	2·12	2·29	2·47	2·63	2·79	2·95	2·09	2·27	2·44	2·62	2·78	2·94	3·10	2·26	2·44	2·61	2·79	2·95	3·11	3·26
27	2·30	2·51	2·70	2·90	3·09	3·27	3·44	2·48	2·68	2·87	3·07	3·27	3·45	3·62	2·67	2·88	3·07	3·27	3·46	3·64	3·81
28	2·66	2·90	3·13	3·35	3·57	3·86	4·00	2·86	3·10	3·23	3·55	3·77	3·99	4·20	3·08	3·31	3·55	3·76	3·99	4·21	4·41
29	3·05	3·31	3·56	3·79	4·04	4·27	4·49	3·28	3·53	3·77	4·02	4·26	4·49	4·71	3·52	3·77	4·01	4·26	4·50	4·73	4·95
30	3·44	3·70	3·99	4·28	4·54	4·80	5·06	3·68	3·94	4·23	4·52	4·79	5·05	5·30	3·95	4·22	4·51	4·79	5·07	5·32	5·57

Temperature (°C)	Wines of 19 % vol							Wines of 21 % vol						
	Densities							Densities						
	1·000	1·020	1·040	1·060	1·080	1·100	1·120	1·000	1·020	1·040	1·060	1·080	1·100	1·120
10	3·27	3·62	3·97	4·30	4·62	4·92	5·21	3·62	3·97	4·32	4·66	4·97	5·27	5·56
11	2·99	3·30	3·61	3·90	4·19	4·45	4·70	3·28	3·61	3·92	4·22	4·50	4·76	5·01
12	2·68	2·96	3·24	3·50	3·76	4·00	4·21	2·96	3·24	3·52	3·78	4·03	4·27	4·49
13	2·40	2·64	2·87	3·09	3·30	3·51	3·71	2·64	2·88	3·11	3·33	3·54	3·74	3·95
14	2·11	2·31	2·51	2·69	2·88	3·05	3·22	2·31	2·51	2·71	2·89	3·08	3·25	3·43
15	1·76	1·93	2·09	2·25	2·40	2·55	2·69	1·93	2·10	2·26	2·42	2·57	2·72	2·86
16	1·43	1·57	1·70	1·83	1·95	2·08	2·18	1·56	1·70	1·84	1·97	2·09	2·21	2·32
17	1·09	1·20	1·30	1·39	1·48	1·57	1·65	1·20	1·31	1·41	1·50	1·59	1·68	1·77
18	0·76	0·82	0·88	0·95	1·01	1·06	1·12	0·82	0·88	0·95	1·01	1·08	1·13	1·18
19	0·39	0·42	0·45	0·49	0·52	0·55	0·57	0·42	0·46	0·49	0·52	0·55	0·58	0·61
20														
21	0·38	0·42	0·45	0·48	0·51	0·54	0·57	0·41	0·45	0·48	0·51	0·54	0·57	0·60
22	0·78	0·84	0·90	0·96	1·02	1·07	1·13	0·84	0·90	0·96	1·02	1·08	1·14	1·19
23	1·19	1·28	1·38	1·47	1·55	1·64	1·72	1·29	1·21	1·48	1·57	1·65	1·74	1·82
24	1·60	1·72	1·83	1·95	2·06	2·18	2·29	1·73	1·85	1·96	2·08	2·19	2·31	2·42
25	2·02	2·16	2·31	2·46	2·60	2·74	2·88	2·18	2·32	2·47	2·62	2·76	2·90	3·04
26	2·44	2·62	2·79	2·96	3·12	3·28	3·43	2·53	2·81	2·97	3·15	3·31	3·47	3·62
27	2·88	3·08	3·27	3·42	3·66	3·84	4·01	3·10	3·30	3·47	3·69	3·88	4·06	4·23
28	3·31	3·54	3·78	4·00	4·22	4·44	4·64	3·56	3·79	4·03	4·25	4·47	4·69	4·89
29	3·78	4·03	4·27	4·52	4·76	4·99	5·21	4·06	4·31	4·55	4·80	5·04	5·27	5·48
30	4·24	4·51	4·80	5·08	5·36	5·61	5·86	4·54	4·82	5·11	5·39	5·66	5·91	6·16

Numerical examples

The setting-up of pycnometer constants

Calculations	*Numerical examples*
1. Weighing of pycnometer filled with air: Tare = air-filled pycnometer + p	p = 104·9454 g
2. Weighing of pycnometer filled with pure water at ambient temperature t °C: Tare = pycnometer + water at t °C + p′	t °C = 16·20 °C p′ = 1·1616 g t °C = 20·50 °C p′ = 1·2396 g t °C = 22·30 °C p′ = 1·2777 g
3. Calculation of the mass m of air contained in the air-filled pycnometer: In practice the volume of the pycnometer is equal to p — p′ to 5/1 000. Hence m = 0·0012 (p — p′)	m = 0·1244 g The two other measurements give the same figure to 0·0001
4. Characteristics to retain: p + m = tare of empty pycnometer	p + m = 104·9454 + 0·1244 tare of the empty pycnometer = 105·0698 g

p + m — p′ = mass of pure water contained at t °C in the pycnometer (column 3)

t °C read	t °C corrected (1)	3	4
—	—	—	—
16·20	16·07	103·9082	104·0233
20·50	20·27	103·8302	104·0226
22·30	22·04	103·7921	104·0229

(p + m — p′) × F = volume of the pycnometer at 20 °C (column 4)	volume at 20 °C of the pycnometer = 104·0229 ml
(p + m — p′) × F × 0·998203 = mass of the pure water contained at 20 °C in the pycnometer	weight of the pure water at 20 °C = 103·8360 g

Determination of the density of a dry wine

Calculation	*Numerical example*
Tare = pycnometer + wine at t °C + p″	t °C = 18·00 °C t °C = 17·80 °C corrected p″ = 1·2622 g
$\varrho_t = \frac{p + m - p''}{\text{volume at } 20\,^\circ C}$	ϱ 17·80 °C = $\frac{105{\cdot}0698 - 1{\cdot}2622}{104{\cdot}0229}$ ϱ 17·80 °C = 0·99793
To calculate ϱ_{20} reference is made to Table II of corrections for temperature for dry wines	$\varrho_{20} = 0{\cdot}99793 - \frac{0{\cdot}54}{1\,000} = 0{\cdot}99739$

(1) Correction of thermometer.

1.4. FIRST USUAL METHOD

Measurement by a hydrometer — apparatus

The apparatus used shall comply with the following characteristics.

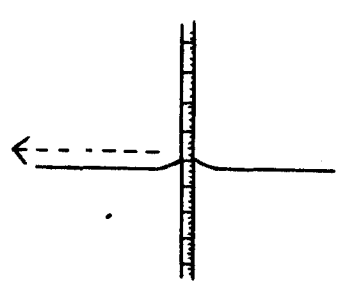
The apparatus shall have a cylindrical bulb, a stem of circular section of minimum diameter 8 mm. For dry wines the apparatus shall be graduated from 0·9830 to 1·0030 with graduation marks every 0·0010 and 0·0002. Each mark at 0·0010 shall be separated by at least 5 mm from the next corresponding mark. For the measurement of the specific gravity of de-alcoholized wines, sweet wines and musts, a set of five hydrometers graduated from 1·0000 to 1·0300, 1·0300 to 1·0600, 1·0600 to 1·0900, 1·0900 to 1·1200 and 1·1200 to 1·1500 shall be used. These hydrometers shall be graduated in densities at 20 °C with marks at 0·0010 and 0·0005, each mark at 0·0010 being separated by at least 3 mm from the next corresponding mark.

These hydrometers shall be graduated so as to be read at the 'top of the meniscus'. To avoid any confusion with hydrometers graduated at 15 °C and normally read at the liquid surface, an indication of the graduation in density at 20 °C and of the reading at the 'top of the meniscus' shall be carried either on the graduated scale or on a strip of paper enclosed in the bulb.

These hydrometers shall be calibrated by a government department.

The result shall be expressed to four decimal places to the nearest three units of the fourth decimal place (± 0·0003).

A calibrated thermometer graduated in minimum steps of 0·5 °C shall be used.



Determination of density — procedure

Pour 250 ml of wine into a level glass cylinder of 36 mm internal diameter and 320 mm height. Place a hydrometer and thermometer in the cylinder.

Read the observed temperature t °C one minute after mixing the sample well. Remove the thermometer and read the apparent density from the hydrometer after a further minute.

The density at 20 °C is then calculated using Table V, VI or VII (1).

Correct the density for the effect of added sulphur dioxide and other preservatives in the same manner as described for the reference method.

(1) In these tables the correction c is given to two decimal places to enable it to be usable if the density were to be measured with a pycnometer of ordinary glass.
For measurement by hydrometer, the correction c shall be expressed by a single decimal.

TABLE V

Temperature corrections c to the density of dry wines and of dry wines freed of alcohol measured with a hydrometer or a pycnomete of ordinary glass at t °C to relate the result to 20 °C

$$\varrho_{20} = \varrho_t \pm \frac{c}{1\,000} \begin{cases} - \text{ if t °C is lower than 20 °C} \\ + \text{ if t °C is higher than 20 °C} \end{cases}$$

Temperature (°C)	Alcoholic strengths																							
	0	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27
10	1·45	1·51	1·55	1·58	1·64	1·70	1·78	1·88	1·98	2·09	2·21	2·34	2·47	2·60	2·75	2·90	3·06	3·22	3·39	3·57	3·75	3·93	4·12	4·31
11	1·35	1·40	1·43	1·47	1·52	1·58	1·65	1·73	1·83	1·93	2·03	2·15	2·26	2·38	2·51	2·65	2·78	2·93	3·08	3·24	3·40	3·57	3·73	3·90
12	1·24	1·28	1·31	1·34	1·39	1·44	1·50	1·58	1·66	1·75	1·84	1·94	2·04	2·15	2·26	2·38	2·51	2·63	2·77	2·91	3·05	3·19	3·34	3·49
13	1·12	1·16	1·18	1·21	1·25	1·30	1·35	1·42	1·49	1·56	1·64	1·73	1·82	1·91	2·01	2·11	2·22	2·33	2·45	2·57	2·69	2·81	2·95	3·07
14	0·99	1·03	1·05	1·07	1·11	1·14	1·19	1·24	1·31	1·37	1·44	1·52	1·59	1·67	1·75	1·84	1·93	2·03	2·13	2·23	2·33	2·44	2·55	2·66
15	0·86	0·89	0·90	0·92	0·95	0·98	1·02	1·07	1·12	1·17	1·23	1·29	1·35	1·42	1·49	1·56	1·63	1·71	1·80	1·88	1·96	2·05	2·14	2·23
16	0·71	0·73	0·74	0·76	0·78	0·81	0·84	0·87	0·91	0·96	0·99	1·05	1·10	1·15	1·21	1·27	1·33	1·39	1·45	1·52	1·59	1·66	1·73	1·80
17	0·55	0·57	0·57	0·59	0·60	0·62	0·65	0·67	0·70	0·74	0·77	0·81	0·84	0·88	0·92	0·96	1·01	1·05	1·11	1·15	1·20	1·26	1·31	1·36
18	0·38	0·39	0·39	0·40	0·41	0·43	0·44	0·46	0·48	0·50	0·52	0·55	0·57	0·60	0·62	0·65	0·68	0·71	0·74	0·78	0·81	0·85	0·88	0·91
19	0·19	0·20	0·20	0·21	0·21	0·22	0·23	0·24	0·25	0·26	0·27	0·28	0·29	0·30	0·32	0·33	0·35	0·36	0·38	0·39	0·41	0·43	0·44	0·46
20																								
21	0·21	0·22	0·22	0·23	0·23	0·24	0·25	0·25	0·26	0·27	0·29	0·29	0·31	0·32	0·34	0·35	0·36	0·38	0·39	0·41	0·43	0·44	0·46	0·48
22	0·43	0·45	0·45	0·46	0·47	0·49	0·50	0·52	0·54	0·56	0·58	0·60	0·63	0·65	0·68	0·71	0·73	0·77	0·80	0·83	0·86	0·89	0·93	0·96
23	0·67	0·69	0·70	0·71	0·72	0·74	0·77	0·79	0·82	0·85	0·88	0·91	0·95	0·99	1·03	1·07	1·12	1·16	1·21	1·25	1·30	1·35	1·40	1·45
24	0·91	0·93	0·95	0·97	0·99	1·01	1·04	1·07	1·11	1·15	1·20	1·24	1·29	1·34	1·39	1·45	1·50	1·56	1·62	1·69	1·76	1·82	1·88	1·95
25	1·16	1·19	1·21	1·23	1·26	1·29	1·33	1·37	1·42	1·47	1·52	1·57	1·63	1·70	1·76	1·83	1·90	1·97	2·05	2·13	2·21	2·29	2·37	2·45
26	1·42	1·46	1·49	1·51	1·54	1·58	1·62	1·67	1·73	1·79	1·85	1·92	1·99	2·07	2·14	2·22	2·31	2·40	2·49	2·58	2·67	2·77	2·86	2·96
27	1·69	1·74	1·77	1·80	1·83	1·88	1·93	1·98	2·05	2·12	2·20	2·27	2·35	2·44	2·53	2·63	2·72	2·82	2·93	3·04	3·14	3·25	3·37	3·48
28	1·97	2·03	2·06	2·09	2·14	2·19	2·24	2·31	2·38	2·46	2·55	2·63	2·73	2·83	2·93	3·03	3·14	3·26	3·38	3·50	3·62	3·75	3·85	4·00
29	2·26	2·33	2·37	2·40	2·45	2·50	2·57	2·64	2·73	2·82	2·91	2·99	3·11	3·22	3·34	3·45	3·58	3·70	3·84	3·97	4·11	4·25	4·39	4·54
30	2·56	2·64	2·67	2·72	2·77	2·83	2·90	2·98	3·08	3·18	3·28	3·38	3·50	3·62	3·75	3·88	4·02	4·16	4·30	4·46	4·61	4·76	4·92	5·07

Note: This table can be used for converting the specific gravity d^t_{20} to the specific gravity d^{20}_{20}.

TABLE VI

Temperature corrections c to the density of natural musts and concentrated musts measured at t °C with a pycnometer or a hydromete of ordinary glass to relate the result to 20 °C

$$\varrho_{20} = \varrho_t \pm \frac{c}{1\,000} \begin{cases} - \text{ if t °C is lower than 20 °C} \\ + \text{ if t °C is higher than 20 °C} \end{cases}$$

Temperature (°C)	Densities																					
	1·05	1·06	1·07	1·08	1·09	1·10	1·11	1·12	1·13	1·14	1·15	1·16	1·18	1·20	1·22	1·24	1·26	1·28	1·30	1·32	1·34	1·36
10	2·17	2·34	2·52	2·68	2·85	2·99	3·16	3·29	3·44	3·58	3·73	3·86	4·13	4·36	4·60	4·82	5·02	5·25	5·39	5·56	5·73	5·87
11	2·00	2·16	2·29	2·44	2·59	2·73	2·86	2·99	3·12	3·24	3·37	3·48	3·71	3·94	4·15	4·33	4·52	4·69	4·85	5·01	5·15	5·29
12	1·81	1·95	2·08	2·21	2·34	2·47	2·58	2·70	2·82	2·92	3·03	3·14	3·35	3·55	3·72	3·90	4·07	4·23	4·37	4·52	4·64	4·77
13	1·62	1·74	1·85	1·96	2·07	2·17	2·28	2·38	2·48	2·59	2·68	2·77	2·94	3·11	3·28	3·44	3·54	3·72	4·86	3·99	4·12	4·24
14	1·44	1·54	1·64	1·73	1·82	1·92	2·00	2·08	2·17	2·25	2·34	2·42	2·57	2·73	2·86	2·99	3·12	3·24	3·35	3·46	3·57	3·65
15	1·21	1·29	1·37	1·45	1·53	1·60	1·68	1·75	1·82	1·89	1·97	2·03	2·16	2·28	2·40	2·51	2·61	2·71	2·80	2·89	2·94	3·01
16	1·00	1·06	1·12	1·19	1·25	1·31	1·37	1·43	1·49	1·54	1·60	1·65	1·75	1·84	1·94	2·02	2·09	2·17	2·23	2·30	2·36	2·42
17	0·76	0·82	0·86	0·91	0·96	1·00	1·05	1·09	1·14	1·18	1·22	1·25	1·32	1·39	1·46	1·52	1·57	1·63	1·67	1·71	1·75	1·79
18	0·53	0·56	0·59	0·63	0·65	0·69	0·72	0·74	0·77	0·80	0·82	0·85	0·90	0·95	0·99	1·02	1·06	1·09	1·13	1·16	1·18	1·20
19	0·28	0·30	0·31	0·33	0·35	0·36	0·38	0·39	0·41	0·42	0·43	0·43	0·46	0·48	0·50	0·52	0·54	0·55	0·57	0·58	0·59	0·60
20																						
21	0·28	0·29	0·31	0·33	0·34	0·36	0·37	0·39	0·40	0·41	0·43	0·44	0·46	0·48	0·51	0·54	0·56	0·57	0·58	0·59	0·60	0·60
22	0·55	0·58	0·61	0·64	0·67	0·70	0·73	0·76	0·78	0·81	0·84	0·87	0·93	0·97	1·02	1·06	1·09	1·12	1·15	1·17	1·19	1·19
23	0·85	0·90	0·95	0·99	1·04	1·08	1·12	1·16	1·21	1·25	1·29	1·32	1·39	1·46	1·52	1·58	1·62	1·68	1·72	1·75	1·77	1·79
24	1·15	1·19	1·25	1·31	1·37	1·43	1·48	1·54	1·60	1·65	1·71	1·76	1·86	1·95	2·04	2·11	2·17	2·23	2·29	2·33	2·35	2·37
25	1·44	1·52	1·59	1·67	1·74	1·81	1·88	1·95	2·02	2·09	2·16	2·22	2·34	2·45	2·55	2·64	2·74	2·81	2·87	2·90	2·92	2·96
26	1·76	1·84	1·93	2·02	2·10	2·18	2·25	2·33	2·41	2·49	2·56	2·64	2·78	2·91	3·03	3·15	3·26	3·37	3·47	3·55	3·62	3·68
27	2·07	2·16	2·26	2·36	2·46	2·56	2·65	2·74	2·83	2·91	3·00	3·07	3·24	3·39	3·55	3·69	3·82	3·94	4·04	4·14	4·23	4·30
28	2·39	2·51	2·63	2·74	2·85	2·96	3·06	3·16	3·28	3·38	3·48	3·57	3·75	3·92	4·08	4·23	4·37	4·51	4·62	4·73	4·80	4·86
29	2·74	2·86	2·97	3·09	3·22	3·34	3·46	3·57	3·69	3·80	3·90	4·00	4·20	4·39	4·58	4·74	4·90	5·05	5·19	5·31	5·40	5·48
30	3·06	3·21	3·35	3·50	3·63	3·77	3·91	4·02	4·15	4·28	4·40	4·52	4·75	4·96	5·16	5·35	5·52	5·67	5·79	5·91	5·99	6·04

Note: This table can be used for converting the specific gravity d^t_{20} to the specific gravity d^{20}_{20}.

TABLE VII

Temperature corrections c to the density of wines of 13% vol or more measured with a hydrometer or a pycnometer of ordinary glass at t °C to relate the result to 20 °C

$$\varrho_{20} = \varrho_t \pm \frac{c}{1\,000} \begin{cases} - \text{ if t °C is lower than 20 °C} \\ + \text{ if t °C is higher than 20 °C} \end{cases}$$

Temperature (°C)	Wines of 13 % vol						
	Densities						
	1·000	1·020	1·040	1·060	1·080	1·100	1·120
10	2·24	2·58	2·93	3·27	3·59	3·89	4·18
11	2·06	2·37	2·69	2·97	3·26	3·63	3·78
12	1·87	2·14	2·42	2·67	2·94	3·17	3·40
13	1·69	1·93	2·14	2·37	2·59	2·80	3·00
14	1·49	1·70	1·90	2·09	2·27	2·44	2·61
15	1·25	1·42	1·59	1·75	1·90	2·05	2·19
16	1·03	1·17	1·30	1·43	1·55	1·67	1·78
17	0·80	0·90	1·00	1·09	1·17	1·27	1·36
18	0·54	0·61	0·68	0·75	0·81	0·86	0·92
19	0·29	0·33	0·36	0·39	0·42	0·45	0·48
20							
21	0·29	0·32	0·35	0·39	0·42	0·45	0·47
22	0·57	0·64	0·70	0·76	0·82	0·88	0·93
23	0·89	0·98	1·08	1·17	1·26	1·34	1·43
24	1·22	1·34	1·44	1·56	1·68	1·79	1·90
25	1·61	1·68	1·83	1·98	2·12	2·26	2·40
26	1·87	2·05	2·22	2·40	2·56	2·71	2·87
27	2·21	2·42	2·60	2·80	3·00	3·18	3·35
28	2·56	2·80	3·02	3·25	3·47	3·67	3·89
29	2·93	3·19	3·43	3·66	3·91	4·14	4·37
30	3·31	3·57	3·86	4·15	4·41	4·66	4·92

Temperature (°C)	Wines of 15 % vol						
	Densities						
	1·000	1·020	1·040	1·060	1·080	1·100	1·120
10	2·51	2·85	3·20	3·54	3·86	4·02	4·46
11	2·31	2·61	2·93	3·21	3·51	3·64	4·02
12	2·09	2·36	2·64	2·90	[illegible]	3·27	3·61
13	1·88	2·12	2·34	2·56	2·78	2·88	3·19
14	1·67	1·86	2·06	2·25	2·45	2·51	2·77
15	1·39	1·56	1·72	1·88	2·03	2·11	2·32
16	1·06	1·27	1·40	1·53	1·65	1·77	1·88
17	0·87	0·98	1·08	1·17	1·26	1·35	1·44
18	0·60	0·66	0·73	0·80	0·86	0·91	0·97
19	0·32	0·36	0·39	0·42	0·45	0·48	0·51
20							
21	0·32	0·35	0·38	0·42	0·45	0·48	0·50
22	0·63	0·69	0·75	0·81	0·87	0·93	0·98
23	0·97	1·06	1·16	1·25	1·34	1·42	1·51
24	1·32	1·44	1·54	1·66	1·78	1·89	2·00
25	1·66	1·81	1·96	2·11	2·25	2·39	2·52
26	2·02	2·20	2·37	2·54	2·70	2·85	3·01
27	2·39	2·59	2·78	2·98	3·17	3·35	3·52
28	2·75	2·89	3·22	3·44	3·66	3·86	4·07
29	3·16	3·41	3·65	3·89	4·13	4·36	4·59
30	3·55	3·81	4·10	4·38	4·66	4·90	5·16

Temperature (°C)	Wines of 17 % vol						
	Densities						
	1·000	1·020	1·040	1·060	1·080	1·100	1·120
10	2·81	3·15	3·50	3·84	4·15	4·45	4·74
11	2·57	2·89	3·20	3·49	3·77	4·03	4·28
12	2·32	2·60	2·87	3·13	3·39	3·63	3·84
13	2·09	2·33	2·55	2·77	2·98	3·19	3·39
14	1·83	2·03	2·23	2·42	2·61	2·77	2·94
15	1·54	1·71	1·87	2·03	2·18	2·32	2·47
16	1·25	1·39	1·52	1·65	1·77	1·89	2·00
17	0·96	1·06	1·16	1·26	1·35	1·44	1·52
18	0·66	0·72	0·79	0·86	0·92	0·97	1·03
19	0·35	0·38	0·41	0·45	0·48	0·51	0·53
20							
21	0·34	0·38	0·41	0·44	0·47	0·50	0·53
22	0·68	0·75	0·81	0·87	0·93	0·99	1·04
23	1·06	1·15	1·25	1·34	1·42	1·51	1·59
24	1·43	1·55	1·65	1·77	1·89	2·00	2·11
25	1·80	1·94	2·09	2·24	2·39	2·52	2·66
26	2·18	2·36	2·53	2·71	2·86	3·02	3·17
27	2·58	2·78	2·97	3·17	3·36	3·54	3·71
28	2·97	3·21	3·44	3·66	3·88	4·09	4·30
29	3·40	3·66	3·89	4·13	4·38	4·61	4·82
30	3·82	4·08	4·37	4·65	4·93	5·17	5·42

Temperature (°C)	Wines of 19 % vol						
	Densities						
	1·000	1·020	1·040	1·060	1·080	1·100	1·120
10	3·14	3·48	3·83	4·17	4·48	4·78	5·07
11	2·87	3·18	3·49	3·78	4·06	4·32	4·57
12	2·58	2·86	3·13	3·39	3·65	3·88	4·10
13	2·31	2·55	2·77	2·99	3·20	3·41	3·61
14	2·03	2·23	2·43	2·61	2·80	2·96	3·13
15	1·69	1·86	2·02	2·18	2·33	2·48	2·62
16	1·38	1·52	1·65	1·78	1·90	2·02	2·13
17	1·06	1·16	1·26	1·35	1·44	1·53	1·62
18	0·73	0·79	0·85	0·92	0·98	1·03	1·09
19	0·38	0·41	0·44	0·48	0·51	0·52	0·56
20							
21	0·37	0·41	0·44	0·47	0·50	0·53	0·56
22	0·75	0·81	0·87	0·93	0·99	1·04	1·10
23	1·15	1·30	1·34	1·43	1·51	1·60	1·68
24	1·55	1·67	1·77	1·89	2·00	2·11	2·23
25	1·95	2·09	2·24	2·39	2·53	2·67	2·71
26	2·36	2·54	2·71	2·89	3·04	3·20	3·35
27	2·79	2·99	3·18	3·38	3·57	3·75	3·92
28	3·20	3·44	3·66	3·89	4·11	4·32	4·53
29	3·66	3·92	4·15	4·40	4·64	4·87	5·08
30	4·11	4·37	4·66	4·94	5·22	5·46	5·71

Temperature (°C)	Wines of 21 % vol						
	Densities						
	1·000	1·020	1·040	1·060	1·080	1·100	1·120
10	3·50	3·84	4·19	4·52	4·83	5·12	5·41
11	3·18	3·49	3·80	4·09	4·34	4·63	4·88
12	2·86	3·13	3·41	3·67	3·92	4·15	4·37
13	2·56	2·79	3·01	3·23	3·44	3·65	3·85
14	2·23	2·43	2·63	2·81	3·00	3·16	3·33
15	1·86	2·03	2·19	2·35	2·50	2·65	2·80
16	1·51	1·65	1·78	1·91	2·03	2·15	2·26
17	1·15	1·25	1·35	1·45	1·54	1·63	1·71
18	0·79	0·85	0·92	0·98	1·05	1·10	1·15
19	0·41	0·44	0·47	0·51	0·54	0·57	0·59
20							
21	0·41	0·44	0·47	0·51	0·54	0·57	0·59
22	0·81	0·88	0·94	1·00	1·06	1·10	1·17
23	1·25	1·34	1·44	1·63	1·61	1·70	1·78
24	1·68	1·80	1·90	2·02	2·13	2·25	2·36
25	2·11	2·25	2·40	2·55	2·69	2·83	2·97
26	2·55	2·73	2·90	3·07	3·22	3·38	3·54
27	3·01	3·20	3·40	3·59	3·78	3·96	4·13
28	3·46	3·69	3·93	4·15	4·36	4·58	4·77
29	3·95	4·20	4·43	4·68	4·92	5·15	5·36
30	4·42	4·68	4·97	5·25	5·53	5·77	6·02

CONVERSION OF THE SPECIFIC GRAVITY TO THE DENSITY

TABLE VIII

Calculation of the density ϱ_{20} from the specific gravity d_{20}^{20} and conversely (1)

$$\varrho_{20} = 0{\cdot}998203\ d_{20}^{20} \text{ and } d_{20}^{20} = 1{\cdot}001800\ \varrho_{20}$$

More simply: $\varrho_{20} = d_{20}^{20} - \frac{c}{1\,000}$

Specific gravities	0·960	0·980	1·000	1·020	1·040	1·060	1·080	1·100
Corrections, c	1·73	1·76	1·80	1·84	1·87	1·91	1·94	1·98
Specific gravities	1·120	1·140	1·160	1·180	1·200	1·220	1·240	1·260
Corrections, c	2·02	2·05	2·09	2·12	2·16	2·20	2·23	2·27
Specific gravities		1·280	1·300	1·320	1·340	1·360	1·380	1·400
Corrections, c		2·30	2·34	2·38	2·41	2·45	2·48	2·52

RELATIONSHIP BETWEEN THE SPECIFIC GRAVITIES CORRECTED FOR THE BUOYANCY OF AIR AND THE UNCORRECTED SPECIFIC GRAVITIES

TABLE IX

Conversion of specific gravities measured in air to specific gravities measured in vacuum

$$d\ (\text{vacuum}) = d\ (\text{air}) + \frac{c}{1\,000}, \text{ valid for 15 and 20 °C}$$

d	c	d	c
0·80	0·24	1·01	— 0·01
0·81	0·23	1·02	— 0·02
0·82	0·22	1·03	— 0·04
0·83	0·20	1·04	— 0·05
0·84	0·19	1·05	— 0·06
0·85	0·18	1·06	— 0·07
0·86	0·17	1·07	— 0·08
0·87	0·16	1·08	— 0·10
0·88	0·15	1·09	— 0·11
0·89	0·13	1·10	— 0·12
0·90	0·12	1·11	— 0·13
0·91	0·11	1·12	— 0·15
0·92	0·10	1·13	— 0·16
0·93	0·09	1·14	— 0·17
0·94	0·07	1·15	— 0·18
0·95	0·06	1·16	— 0·19
0·96	0·05	1·17	— 0·20
0·97	0·04	1·18	— 0·22
0·98	0·02	1·19	— 0·23
0·99	0·01	1·20	— 0·24

(1) The specific gravities 15/4° and 20/4° are for practical purposes identical to the densities at 15 and 20 °C. Strictly, they differ by $27 \cdot 10^{-6}$, the density of water at 4 °C being equal to 0·999973 g/ml or g/cm³ (1 ml being defined at present as being equal to 1 cm³).

Hence $\varrho_{20} = 0{\cdot}998203\ d_{20}^{20}$ (or $d_{20}^{20} = 1{\cdot}001800\ \varrho_{20}$).

Whilst $d_4^{20} = 0{\cdot}998230\ d_{20}^{20}$ (or $d_{20}^{20} = 1{\cdot}001773\ d_4^{20}$).

Similarly $d_4^{15} = 0{\cdot}999130\ d_{15}^{15}$ (or $d_{15}^{15} = 1{\cdot}000870\ d_4^{15}$).

1.5. SUPPLEMENT TO THE REFERENCE METHOD — MEASUREMENT BY PYCNOMETER ON SINGLE-PAN BALANCE

Principle

The reference method for the determination of the density of musts and wines and for the determination of the alcoholic strength of wines requires the use of a two-pan balance and a pycnometer with its tare bottle of the same external volume to eliminate the effect of air. This simple technique may also be applied when a single-pan balance is used subject to a supplementary weighing of the tare bottle to take account of the variations of the buoyancy of air:

— when establishing the pycnometer constants: weight of the empty pycnometer, volume at 20 °C, the weight T_0 of the tare bottle is determined;

— when determining density, the weight T_1 of the tare bottle is again derived and the weight of the pycnometer filled with liquid is corrected for the mathematical value of the difference between T_0 and T_1. The remainder of the calculations are unchanged.

Procedure

1. *Standardization of the pycnometer*

Establish the following constant characteristics of the pycnometer:

— its weight evacuated, P_0,

— its volume at 20 °C, V_{20},

— at the same time, record accurately the weight of the tare bottle T_0.

The weight of the tare bottle T_0 is obtained by weighing to the nearest 0·1 mg.

The weight of the pycnometer evacuated, P_0, is the weight P of the clean and dry pycnometer, filled with air, less the mass m of the air which it contains. Obtain the mass m of the air by multiplying the density of the air by the volume of the pycnometer. Under average conditions of temperature and pressure assume a value of 0·0012 g/ml for the density of air. For the calculation of m, consider that the volume of the pycnometer is numerically equal to $P_1 - P$, P_1 being the weight of the pycnometer filled with water obtained at the time of the determination of the weight of water in the pycnometer at t °C.

Hence: $P_0 = P - m$, where $m = 0{\cdot}0012\ (P_1 - P)$.

To obtain the volume at 20 °C of the pycnometer, determine the weight P_1 of the pycnometer, filled up to the mark with degassed water at t °C. This temperature must be measured to 0·02 °C.

The mass M_t of the water at t °C contained in the pycnometer is equal to:

$$M_t = P_1 - P_0$$

Multiply M_t by the factor F (product of the reciprocal of the density of the water at t °C and the ratio of the volumes of the pycnometer at t °C and at 20 °C) given in Table I (page 5) to obtain the volume of the pycnometer at 20 °C:

$$V_{20} = F\ (P_1 - P_0)$$

2. *Determination of a density*

Weigh the tare bottle to the nearest 0·1 mg; let this weight be T_1.

Fill the pycnometer with liquid (must, wine or distillate) at t °C and weigh; let this be P_2.

Calculate the difference $dT = T_1 - T_0$; this represents the variation in the buoyancy of air.

The mass L_t of the liquid contained in the pycnometer at t °C is equal to $L_t = P_2 - (P_0 + dT)$.

Note must be taken of the sign of dT. The apparent density ϱ_t is equal to the ratio of L_t to V_{20}.

$$\varrho_t = \frac{P_2 - (P_0 + dT)}{V_{20}}$$

Once the apparent density at t °C is known, the density at 20 °C of the liquid under test (dry wine, natural or concentrated must, sweet wine, alcohol-free residue) is determined as specified on page 6.

Numerical example

1. Standardization of the pycnometer:

weight of tare bottle:	$T_0 = 171{\cdot}9160$
weight of pycnometer filled with air:	$P = 67{\cdot}7913$
weight of pycnometer filled with water at 21·65 °C:	$P_1 = 169{\cdot}2715$
mass of air contained in the pycnometer:	$m = 0{\cdot}0012\,(169{\cdot}2715 - 67{\cdot}7913) = 0{\cdot}1218$
weight of empty pycnometer:	$P_0 = 67{\cdot}7913 - 0{\cdot}1218 = 67{\cdot}6695$
volume of the pycnometer at 20 °C:	$V_{20} = 1{\cdot}002140\,(169{\cdot}2715 - 67{\cdot}6695)$ $= 101{\cdot}8194$ ml or $0{\cdot}1018194$ dm³

2. Determination of the apparent density of a wine:

weight of tare bottle at the time of the determination:	$T_1 = 171{\cdot}9178$
pycnometer full of wine at 18 °C:	$P_2 = 169{\cdot}2799$
variation in the buoyancy of air:	$dT = 171{\cdot}9178 - 171{\cdot}9160$ $= + 0{\cdot}0018$
mass of the wine at 18 °C:	$L_t = 169{\cdot}2799 - (67{\cdot}6695 + 0{\cdot}0018) = 101{\cdot}6086$
apparent density of the wine at 18 °C:	$\varrho_{18} = \frac{101{\cdot}6086}{101{\cdot}8194} = 0{\cdot}99793$ g/ml

3. Calculation of the density at 20 °C:

proceed as specified on page 5.

SECOND USUAL METHOD

Densimetry by the hydrostatic balance

Apparatus and calibration of the float

The hydrostatic balance, of maximum capacity not less than 100 g, must have a sensitivity of 0·1 mg.

Under each pan is fixed a Pyrex (1) glass float of volume at least 20 ml. A volume of 50 or 100 ml is preferable. These two identical floats must be suspended by a thread of diameter not exceeding 0·1 mm.

The float suspended beneath the right-hand pan must be able to be introduced into a cylindrical test-tube bearing a mark indicating the level. This test-tube must have an internal diameter at least 6 mm larger than that of the float. The float must be capable of being contained completely in the volume of the test tube situated below the mark; the surface of the liquid to be measured must be traversed only by the suspension thread. The temperature of the liquid present in the test-tube is measured by a thermometer graduated in divisions of 0·2 °C.

Calibration of the apparatus

With the two floats in air one finds the balanced position by placing weights marked p on the right-hand pan.

The test-tube is filled with pure water up to the mark and the temperature, t °C, is measured after shaking and allowing to stand for two to three minutes. The balanced position is found once again by means of marked weights placed on the right-hand pan. Let these weights be p′.

The volume (2) of the float at 20 °C is equal to $V_{20} = (p' - p)\,(F + 0{\cdot}0012)$;

(1) Floats made of ordinary glass may be used; one of these, the one which is to be immersed in the liquid of which the density is being measured, may contain a thermometer for measuring the temperature of the liquid. But in that case, the calibration of the float must be carried out at 20 °C, because the F coefficients have not been calculated for vessels or floats of ordinary glass, which has a higher coefficient of expansion than Pyrex glass.

(2) This volume is imaginary because the air pressure on the marked weights has not been taken into account. It is preferable to keep this imaginary volume in order not to have to introduce a correction, of the same nature but in the opposite sense, to the principal weighing. The true volume may be obtained by multiplying V_{20} by 0·999857.

where:

F is the factor given in Table I,

p and V_{20} are the characteristics of the float, because they do not vary with the air pressure, which is compensated for by the calibrated float of the left-hand pan.

Procedure

The right-hand float is immersed in the test-tube filled with wine (or must) up to the mark. Let t °C be the temperature of the wine (or must) and p″ be the marked weights which re-establish the balanced position. The apparent density, ϱ_t, is given by the formula:

$$\varrho_t = \frac{(p'' - p)}{V} + 0{\cdot}0012$$

This density is adjusted to 20 °C by using one of Tables II to IV, if the float is of Pyrex glass, or one of Tables V to VII if the float is of ordinary glass.

The use of an automatic hydrostatic balance is authorized if this leads to measurement of the density at 20 °C with the same accuracy.

2. ALCOHOLIC STRENGTH BY VOLUME

(degrees alcohol by volume)

2.1. DEFINITION

The alcoholic strength by volume is expressed by the symbol '% vol'. It is equal to the number of litres of ethanol (1) in 100 litres of product at a temperature of 20 °C (2).

2.2. PRINCIPLE OF METHODS

(a) For wines:

(aa) reference method: double distillation and measurement of the specific gravity of the distillate with a pycnometer. Strength expressed to the nearest 0·05% vol;

(bb) usual methods:

— first usual method: single distillation of the sample which has been made alkaline and measurement of strength with a hydrometer: strength expressed to the nearest 0·1% vol,

— second usual method: single distillation of the sample which has been made alkaline and measurement of strength by the hydrostatic balance: strength expressed to the nearest 0·1% vol.

(b) For musts and fermentating musts with an alcoholic strength not exceeding 6% vol:

(aa) reference method: measurement of alcohol by chemical means;

(bb) usual methods: the methods shall be those given under (a) (bb).

2.3. REFERENCE METHOD FOR WINES

Equipment

1. Distillation equipment: this consists of a 1 000 ml round-bottomed flask with ground joints, fitted with a rectifying column of 20 cm length, or, if not available, a Kjeldahl bulb to prevent splashing. To avoid any overheating of the volatile material, the flask, which should be heated by gas, should be placed on a plate provided with a hole of 8 cm diameter. The volatile substances are passed into a West (3) condenser placed vertically. The distillate is directed towards the bottom of the receiving flask using a drawn-out tube. In summer this flask should be immersed in ice water.

   Any other type of distillation apparatus and any other method of collection of the volatile alcoholic material may be used, or the alcohol may be steam-distilled, provided the apparatus used corresponds to the following test: 200 ml of a 10% vol ethanol-water mixture distilled five times in succession should have an alcoholic strength of at least 9·9% vol after the last distillation, i.e. the loss of alcohol occurring during each distillation must not be greater than 0·02% vol.

2. Pycnometer: this piece of apparatus is identical with that specified for the measurement of the density; the tare shall have a weight which is just greater (no more than 1 or 2 g) than that of the pycnometer filled with water. It shall be standardized as specified for that measurement in 'Density and specific gravity' (page 3).

Procedure

Remove the bulk of any carbon dioxide from young and sparkling wines by stirring 250 ml of the wine in a 500 ml bottle which has been previously siliconized internally with three drops of a 1% solution and dried.

1. Distillation: measure out the wine to be analyzed into a 200 ml graduated flask with a neck of maximum 12 mm diameter and record its temperature. Smaller or larger volumes of wine may be used, but at least equal to 150 ml.

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(1) As in practice the ethanol is not separated from its homologues occurring in small quantities in the wine, the whole of the volatile alcohols will be measured as ethanol. Similarly, the alcohol from esters will be included in the alcoholic strength by volume.

(2) The quantity of alcohol may also be expressed in grams per litre at 20 °C by multiplying the alcoholic strength by volume by the factor 7·8924.

(3) This condenser consists of a thin glass cylinder placed in a sleeve of glass which has an internal diameter greater by 1 mm than the external diameter of the cylinder to be cooled. Cold water is thus circulated rapidly against the surface to be cooled.

Transfer the wine to the distillation flask into which has been placed some inert porous material (unglazed porcelain, glass frit, pumice, etc.). Rinse the flask four times with 5 ml of water. Add 10 ml of milk of lime (120 g of CaO per litre). The colouring matter of the wine should be changed by the alkalinity. In the case of very acid, sour, etc., wines add the lime until the wine is clearly alkaline to phenolphthalein used externally. The distillate is collected in a 200 ml flask, to which approximately 10 ml of pure water has been previously added and into which should dip the drawn-out tube connected to the condenser.

A volume equal to approximately three quarters of the initial volume should be distilled and collected.

Redistill the distillate after 1 ml of 10% sulphuric acid and some fragments of dry inert porous material have been added to the flask and the flask has been rinsed four more times with 5 ml of water.

Collect the second distillate in the same flask which was used for measuring the volume of the wine and into which 10 ml of distilled water have been placed. After shaking, make the distillate up to volume at a temperature identical with the initial temperature to within $\pm$ 2 °C.

2. Measurement of the density of the distillate: carefully fill the pycnometer with the distillate at ambient temperature; place the pycnometer in the thermally insulated enclosure; shake the liquid two or three times by inverting the container until the temperature reading on the thermometer is constant. Adjust the level accurately to the upper rim of the side tube. Wipe the side tube dry, put on the receiver plug and measure the temperature t °C with the same care as previously; carefully wipe the pycnometer dry and weigh, p″ being the weight.

   The difference $p + m - p''$ between the corrected tare and the weight p″ gives the mass of the liquid contained in the pycnometer (corrected for buoyancy of air).

   The apparent density ϱ_t is then calculated by dividing this mass by the volume at 20 °C.

3. Use of the table of apparent density ϱ_t of water-alcohol mixtures taken from the international table: find the smallest density greater than ϱ_t in the table on the horizontal line corresponding to the temperature in whole units immediately below t °C. Use the tabular difference read below this density for calculating the density ϱ at this temperature in whole units.

   On the line of this temperature read, in whole units, the difference between the density ϱ' of the table immediately above ϱ and the calculated density. Divide this difference by the tabular difference read on the right of the density ϱ'. The quotient gives the decimal portion of the alcoholic strength, whereas the whole unit portion of this strength is shown at the head of the column where the density ϱ' is located.

   Note: Calculations may be avoided during routine determinations by calculating one particular table for each pycnometer giving the weights p″ which re-establish equilibrium on the balance for each degree of alcoholic strength and each temperature (in 1 °C units).

Calculate this table using the table of apparent densities by whole degrees of alcoholic strength and whole degrees of temperature by multiplying the densities of the table by volume of the pycnometer at 20 °C and by subtracting these densities from the weight p + m.

If necessary the weights p″ for 0·1 % vol of alcoholic strength and 0·1 °C units of temperature may be obtained by interpolation for the table which has been calculated for whole % vol of alcoholic strength and for 1 °C units of temperature.

Example of the calculation of the alcoholic strength of a wine

The constants of the pycnometer have been determined and calculated as specified in 'Density and specific gravity' (page 3).

Calculations	*Numerical example*
1. Weighing of pycnometer filled with distillate.	
Tare = pycnometer + distillate at t °C + p″	t °C = 18·90 °C t °C corrected = 18·70 °C p″ = 2·8074 g
p + m — p″ = mass of distillate at t °C	105·0698 — 2·8074 = 102·2624 g
Apparent density at t °C $\varrho_t = \frac{p + m - p''}{\text{volume of pycnometer at } 20\,^{\circ}C}$	$\varrho_{18\cdot70} = \frac{102\cdot2624}{104\cdot0229} = 0\cdot983076$
2. Calculation of alcoholic strength.	On the line 18 °C of the table of apparent densities, the smallest value greater than the observed density of 0·983076 is 0·98398 in column 11% vol
Refer to the table of apparent densities of water-alcohol mixtures at different temperatures, as indicated above.	The density at 18 °C is: $(98307\cdot6 + 0\cdot7 \times 22)\ 10^{-5} = 0\cdot98323$ $0\cdot98398 - 0\cdot98323 = 0\cdot00075$ The decimal portion of the % vol of alcoholic strength is 75/116 = 0·65. The alcoholic strength is 11·65% vol.

INTERNATIONAL ALCOHOLIC STRENGTH BY VOLUME AT 20 °C

Table of densities of hydroalcoholic mixtures — Pyrex pycnometer — densities at t °C, corrected for buoyancy of air

Alcoholic strength by % vol

Temperature (°C)

	0		1		2		3		4		5		6		7		8		9		10		11	
0	999·64	1·50	998·14	1·44	996·70	1·40	995·30	1·35	993·95	1·30	992·65	1·24	991·41	1·19	990·22	1·14	989·08	1·10	987·98	1·05	986·93	1·00	985·93	0·95
	+ 0·07		+ 0·06		+ 0·06		+ 0·06		+ 0·06		+ 0·06		+ 0·06		+ 0·05		+ 0·04		+ 0·03		+ 0·02		+ 0·01	
1	999·71	1·51	998·20	1·44	996·76	1·40	995·36	1·35	994·01	1·30	992·71	1·24	991·47	1·20	990·27	1·15	989·12	1·11	988·01	1·06	986·95	1·01	985·94	0·97
	+ 0·05		+ 0·05		+ 0·04		+ 0·04		+ 0·04		+ 0·04		+ 0·03		+ 0·03		+ 0·02		+ 0·02		+ 0·01		0·00	
2	999·76	1·51	998·25	1·45	996·80	1·40	995·40	1·35	994·05	1·30	992·75	1·25	991·50	1·20	990·30	1·16	989·14	1·11	988·03	1·07	986·96	1·02	985·94	0·98
	+ 0·03		+ 0·03		+ 0·03		+ 0·02		+ 0·02		+ 0·02		+ 0·02		+ 0·01		+ 0·01		0·00		0·01		0·02	
3	999·79	1·51	998·28	1·45	996·83	1·41	995·42	1·35	994·07	1·30	992·77	1·25	991·52	1·21	990·31	1·16	989·15	1·12	988·03	1·08	986·95	1·03	985·92	1·00
	+ 0·02		+ 0·02		+ 0·01		+ 0·02		+ 0·01		+ 0·01		0·00		0·00		0·01		0·02		0·03		0·04	
4	999·81	1·51	998·30	1·46	996·84	1·40	995·44	1·36	994·08	1·30	992·78	1·26	991·52	1·21	990·31	1·17	989·14	1·13	988·01	1·09	986·92	1·04	985·88	1·00
	0·00		0·00		0·00		0·00		0·01		0·02		0·02		0·02		0·02		0·03		0·04		0·05	
5	999·81	1·51	998·30	1·46	996·84	1·40	995·44	1·37	994·07	1·31	992·76	1·26	991·50	1·21	990·29	1·17	969·12	1·14	987·98	1·10	986·88	1·05	985·83	1·01
	0·01		0·01		0·01		0·02		0·01		0·02		0·03		0·04		0·05		0·05		0·05		0·06	
6	999·80	1·51	998·29	1·46	996·83	1·41	995·42	1·36	994·06	1·32	992·74	1·27	991·47	1·22	990·25	1·18	989·07	1·14	987·93	1·10	986·83	1·06	985·77	1·03
	0·03		0·03		0·03		0·03		0·04		0·04		0·04		0·05		0·06		0·07		0·08		0·09	
7	999·77	1·51	998·26	1·46	996·80	1·41	995·39	1·37	994·02	1·32	992·70	1·27	991·43	1·23	990·20	1·19	989·01	1·15	987·86	1·11	986·75	1·07	985·68	1·03
	0·05		0·04		0·04		0·05		0·05		0·05		0·05		0·06		0·06		0·07		0·08		0·09	
8	999·72	1·50	998·22	1·46	996·76	1·42	995·34	1·37	993·97	1·32	992·65	1·27	991·38	1·24	990·14	1·19	988·95	1·16	987·79	1·12	986·67	1·08	985·59	1·05
	0·05		0·06		0·06		0·06		0·06		0·06		0·07		0·07		0·08		0·09		0·10		0·11	
9	999·67	1·51	998·16	1·46	996·70	1·42	995·28	1·37	993·91	1·32	992·59	1·28	991·31	1·24	990·07	1·20	988·87	1·17	987·70	1·13	986·57	1·09	985·48	1·06
	0·07		0·07		0·07		0·07		0·07		0·08		0·08		0·09		0·09		0·10		0·11		0·12	
10	999·60	1·51	998·09	1·46	996·63	1·42	995·21	1·37	993·84	1·33	992·51	1·28	991·23	1·25	989·98	1·20	988·78	1·17	987·60	1·14	986·46	1·10	985·36	1·06
	0·09		0·09		0·09		0·08		0·09		0·09		0·10		0·10		0·11		0·11		0·12		0·13	
11	999·51	1·51	998·00	1·46	996·54	1·41	995·13	1·38	993·75	1·33	992·42	1·29	991·13	1·25	989·88	1·21	988·67	1·18	987·49	1·15	986·34	1·11	985·23	1·07
	0·10		0·09		0·09		0·10		0·10		0·11		0·11		0·11		0·12		0·13		0·13		0·14	
12	999·41	1·50	997·91	1·46	996·45	1·42	995·03	1·38	993·65	1·34	992·31	1·29	991·02	1·25	989·77	1·22	988·55	1·19	987·36	1·15	986·21	1·12	985·09	1·09
	0·11		0·11		0·11		0·11		0·11		0·11		0·12		0·12		0·13		0·14		0·15		0·16	
13	999·30	1·50	997·80	1·46	996·34	1·42	994·92	1·38	993·54	1·34	992·20	1·30	990·90	1·25	989·65	1·23	988·42	1·20	987·22	1·16	986·06	1·13	984·93	1·09
	0·12		0·12		0·12		0·13		0·13		0·13		0·13		0·14		0·14		0·15		0·16		0·16	
14	999·18	1·50	997·68	1·46	996·22	1·43	994·79	1·38	993·41	1·34	992·07	1·30	990·77	1·26	989·51	1·23	988·28	1·21	987·07	1·17	985·90	1·13	984·77	1·11
	0·13		0·14		0·14		0·13		0·13		0·14		0·14		0·15		0·16		0·16		0·17		0·18	
15	999·05	1·51	997·54	1·46	996·08	1·42	994·66	1·38	993·28	1·35	991·93	1·30	990·63	1·27	989·36	1·24	988·12	1·21	986·91	1·18	985·73	1·14	984·59	1·12
	0·15		0·14		0·14		0·15		0·15		0·15		0·16		0·16		0·17		0·17		0·18		0·19	
16	998·90	1·50	997·40	1·46	995·94	1·43	994·51	1·38	993·13	1·35	991·78	1·31	990·47	1·27	989·20	1·25	987·95	1·21	986·74	1·19	985·55	1·15	984·40	1·13
	0·16		0·16		0·16		0·16		0·16		0·17		0·17		0·18		0·18		0·19		0·19		0·20	
17	998·74	1·50	997·24	1·46	995·78	1·43	994·35	1·38	992·97	1·36	991·61	1·31	990·30	1·28	989·02	1·25	987·77	1·22	986·55	1·19	985·36	1·16	984·20	1·14
	0·17		0·17		0·17		0·16		0·17		0·17		0·18		0·18		0·19		0·20		0·21		0·22	
18	998·57	1·50	997·07	1·46	995·61	1·42	994·19	1·39	992·80	1·36	991·44	1·32	990·12	1·28	988·84	1·26	987·58	1·23	986·35	1·20	985·15	1·17	983·98	1·14
	0·18		0·18		0·18		0·19		0·19		0·19		0·19		0·20		0·20		0·20		0·21		0·22	
19	998·39	1·50	996·89	1·46	995·43	1·43	994·00	1·39	992·61	1·36	991·25	1·32	989·93	1·29	988·64	1·26	987·38	1·23	986·15	1·21	984·94	1·18	983·76	1·16
	0·19		0·19		0·19		0·19		0·19		0·19		0·20		0·20		0·21		0·22		0·23		0·24	
20	998·20	1·50	996·70	1·46	995·24	1·43	993·81	1·39	992·42	1·36	991·06	1·33	989·73	1·29	988·44	1·27	987·17	1·24	985·93	1·22	984 71	1·19	983·52	1·16

	0		1		2		3		4		5		6		7		8		9		10		11	
20	998·20	1·50	996·70	1·46	995·24	1·43	993·81	1·39	992·42	1·36	991·06	1·33	989·73	1·29	988·44	1·27	987·17	1·24	985·93	1·22	984·71	1·19	983·52	1·16
	0·20		0·20		0·20		0·20		0·21		0·21		0·21		0·22		0·22		0·23		0·24		0·24	
21	998·00	1·50	996·50	1·46	995·04	1·43	993·61	1·40	992·21	1·36	990·85	1·33	989·52	1·30	988·22	1·27	986·95	1·25	985·70	1·23	984·47	1·19	983·28	1·18
	0·21		0·21		0·21		0·21		0·21		0·22		0·22		0·23		0·24		0·24		0·24		0·26	
22	997·79	1·50	996·29	1·46	994·83	1·43	993·40	1·40	992·00	1·37	990·63	1·33	989·30	1·31	987·99	1·28	986·71	1·25	985·46	1·23	984·23	1·21	983·02	1·18
	0·22		0·22		0·23		0·23		0·23		0·23		0·24		0·24		0·24		0·25		0·26		0·25	
23	997·57	1·50	996·07	1·47	994·60	1·43	993·17	1·40	991·77	1·37	990·40	1·34	989·06	1·31	987·75	1·28	986·47	1·26	985·21	1·24	983·97	1·20	982·77	1·20
	0·24		0·23		0·23		0·23		0·24		0·24		0·24		0·25		0·26		0·26		0·27		0·29	
24	997·33	1·49	995·84	1·47	994·37	1·43	992·94	1·41	991·53	1·37	990·16	1·34	988·82	1·32	987·50	1·29	986·21	1·26	984·95	1·25	983·70	1 22	982·48	1·20
	0·24		0·25		0·24		0·25		0·24		0·25		0·26		0·26		0·26		0·27		0·28		0·28	
25	997·09	1·50	995·59	1·46	994·13	1·44	992·69	1·40	991·29	1·38	989·91	1·35	988·56	1·32	987·24	1·29	985·95	1·27	984·68	1·26	983·42	1·22	982·20	1·21
	0·25		0·25		0·26		0·25		0·26		0·26		0·26		0·26		0·28		0·28		0·28		0·30	
26	996·84	1·50	995·34	1·47	993·87	1·43	992·44	1·41	991·03	1·38	989·65	1·35	988·30	1·32	986·98	1·31	985·67	1·27	984·40	1·26	983·14	1·24	981·90	1·22
	0·26		0·26		0·26		0·27		0·27		0·27		0·27		0·28		0·28		0·29		0·30		0·30	
27	996·58	1·50	995·08	1·47	993·61	1·44	992·17	1·41	990·76	1·38	989·38	1·35	988·03	1·33	986·70	1·31	985·39	1·28	984·11	1·27	982·84	1·24	981·60	1·23
	0·27		0·27		0·27		0·27		0·28		0·28		0·29		0·29		0·29		0·30		0·31		0·32	
28	996·31	1·50	994·81	1·47	993·34	1·44	991·90	1·42	990·48	1·38	989·10	1·36	987·74	1·33	986·41	1·31	985·10	1·29	983·81	1·28	982·53	1·25	981·28	1·23
	0·28		0·28		0·28		0·29		0·28		0·29		0·29		0·30		0·31		0·31		0·31		0·32	
29	996·03	1·50	994·53	1·47	993·06	1·45	991·61	1·41	990·20	1·39	988·81	1·36	987·45	1·34	986·11	1·32	984·79	1·29	983·50	1·28	982·22	1·26	980·96	1·24
	0·28		0·29		0·29		0·29		0·30		0·30		0·31		0·31		0·31		0·32		0·32		0·33	
30	995·75	1·51	994·24	1·47	992·77	1·45	991·32	1·42	989·90	1·39	988·51	1·37	987·14	1·34	985·80	1·32	984·48	1·30	983·18	1·28	981·90	1·27	980·63	1·25
	0·30		0·30		0·30		0·30		0·31		0·31		0·31		0·31		0·32		0·33		0·34		0·34	
31	995·45	1·51	993·94	1·47	992·47	1·45	991·02	1·43	989·59	1·39	988·20	1·37	986·83	1·34	985·49	1·33	984·16	1·31	982·85	1·29	981·56	1·27	980·29	1·26
	0·31		0·31		0·31		0·32		0·31		0·32		0·32		0·33		0·33		0·34		0·35		0·36	
32	995·14	1·51	993·63	1·47	992·16	1·46	990·70	1·42	989·28	1·40	987·88	1·37	986·51	1·35	985·16	1·33	983·83	1·32	982·51	1·30	981·21	1·28	979·93	1·26
	0·31		0·31		0·32		0·32		0·32		0·33		0·33		0·34		0·35		0·35		0·35		0·35	
33	994·83	1·51	993·32	1·48	991·84	1·46	990·38	1·42	988·96	1·41	987·55	1·37	986·18	1·36	984·82	1·34	983·48	1·32	982·16	1·30	980·86	1·28	979·58	1·28
	0·32		0·33		0·33		0·33		0·35		0·34		0·35		0·35		0·34		0·35		0·36		0·37	
34	994·51	1·52	992·99	1·48	991·51	1·46	990·05	1·44	988·61	1·40	987·21	1·38	985·83	1·36	984·47	1·33	983·14	1·33	981·81	1·31	980·50	1·29	979·21	1·28
	0·33		0·33		0·34		0·35		0·34		0·35		0·35		0·35		0·36		0·36		0·36		0·37	
35	994·18	1·52	992·66	1·49	991·17	1·47	989·70	1·43	988·27	1·41	986·86	1·38	985·48	1·36	984·12	1·34	982·78	1·33	981·45	1·31	980·14	1·30	978·84	1·29
	0·34		0·35		0·35		0·35		0·35		0·35		0·35		0·36		0·36		0·37		0·37		0·38	
36	993·84	1·53	992·31	1·49	990·82	1·47	989·35	1·43	987·92	1·41	986·51	1·38	985·13	1·37	983·76	1·34	982·42	1·34	981·08	1·31	979·77	1·31	978·46	1·29
	0·35		0·35		0·36		0·35		0·36		0·36		0·37		0·37		0·38		0·37		0·39		0·39	
37	993·49	1·53	991·96	1·50	990·46	1·46	989·00	1·44	987·56	1·41	986·15	1·39	984·76	1·37	983·39	1·35	982·04	1·33	980·71	1·33	979·38	1·31	978·07	1·30
	0·36		0·36		0·36		0·37		0·37		0·37		0·37		0·37		0·38		0·39		0·38		0·39	
38	993·13	1·53	991·60	1·50	990·10	1·47	988·63	1·44	987·19	1·41	985·78	1·39	984·39	1·37	983·02	1·36	981·66	1·34	980·32	1·32	979·00	1·32	977·68	1·31
	0·36		0·37		0·37		0·37		0·38		0·38		0·38		0·39		0·38		0·39		0·40		0·40	
39	992·77	1·54	991·23	1·50	989·73	1·47	988·26	1·45	986·81	1·41	985·40	1·39	984·01	1·38	982·63	1·35	981·28	1·35	979·93	1·33	978·60	1·32	977·28	1·32
	0·37		0·37		0·38		0·39		0·38		0·39		0·39		0·39		0·40		0·39		0·40		0·41	
40	992·40	1·54	990·86	1·51	989·35	1·48	987·87	1·44	986·43	1·42	985·01	1·39	983·62	1·38	982·24	1·36	980·88	1·34	979·54	1·34	978·20	1·33	976·87	1·32

Temperature (°C)

	10		11		12		13		14		15		16		17		18		19		20		21	
0	986·93	1·00	985·93	0·95	984·96	0·92	984·06	0·88	983·18	0·84	982·34	0·80	981·54	0·78	980·76	0·75	980·01	0·73	979·28	0·72	978·56	0·70	977·86	0·70
	+ 0·02		+ 0·01		0·01		0·01		0·03		0·04		0·07		0·08		0·10		0·12		0·14		0·17	
1	986·95	1·01	985·94	0·97	984·97	0·92	984·05	0·90	983·15	0·85	982·30	0·83	981·47	0·79	980·68	0·77	979·91	0·75	979·16	0·74	978·42	0·73	977·69	0·72
	+ 0·01		0·00		0·01		0·03		0·04		0·07		0·08		0·10		0·12		0·14		0·16		0·18	
2	986·96	1·02	985·94	0·98	984·96	0·94	984·02	0·91	983·11	0·88	982·23	0·84	981·39	0·81	980·58	0·79	979·79	0·77	979·02	0·76	978·26	0·75	977·51	0·74
	0·01		0·02		0·04		0·06		0·06		0·07		0·09		0·11		0·13		0·15		0·17		0·19	
3	986·95	1·03	985·92	1·00	984·92	0·95	983·97	0·92	983·05	0·89	982·16	0·86	981·30	0·83	980·47	0·81	979·66	0·79	978·87	0·78	978·09	0·77	977·32	0·77
	0·03		0·04		0·04		0·06		0·07		0·09		0·10		0·12		0·14		0·16		0·18		0·20	
4	986·92	1·04	985·88	1·00	984·88	0·97	983·91	0·93	982·98	0·91	982·07	0·87	981·20	0·85	980·35	0·83	979·52	0·81	978·71	0·80	977·91	0·79	977·12	0·79
	0·04		0·05		0·06		0·07		0·09		0·10		0·12		0·14		0·15		0·17		0·19		0·22	
5	986·88	1·05	985·83	1·01	984·82	0·98	983·84	0·95	982·89	0·92	981·97	0·89	981·08	0·87	980·21	0·84	979·37	0·83	978·54	0·82	977·72	0·82	976·90	0·80
	0·05		0·06		0·08		0·09		0·10		0·12		0·13		0·14		0·17		0·19		0·21		0·22	
6	986·83	1·06	985·77	1·03	984·74	0·99	983·75	0·96	982·79	0·94	981·85	0·90	980·95	0·88	980·07	0·87	979·20	0·85	978·35	0·84	977·51	0·83	976·68	0·83
	0·08		0·09		0·09		0·10		0·12		0·13		0·15		0·16		0·18		0·19		0·21		0·23	
7	986·75	1·07	985·68	1·03	984·65	1·00	983·65	0·98	982·67	0·95	981·72	0·92	980·80	0·89	979·91	0·89	979·02	0·86	978·16	0·86	977·30	0·85	976·45	0·85
	0·08		0·09		0·11		0·13		0·13		0·14		0·15		0·18		0·19		0·21		0·23		0·25	
8	986·67	1·08	985·59	1·05	984·54	1·02	983·52	0·98	982·54	0·96	981·58	0·93	980·65	0·92	979·73	0·90	978·83	0·88	977·95	0·88	977·07	0·87	976·20	0·87
	0·10		0·11		0·12		0·12		0·14		0·16		0·18		0·19		0·21		0·22		0·24		0·26	
9	986·57	1·09	985·48	1·06	984·42	1·02	983·40	1·00	982·40	0·98	981·42	0·95	980·47	0·93	979·54	0·92	978·62	0·89	977·73	0·90	976·83	0·89	975·94	0·89
	0·11		0·12		0·12		0·14		0·16		0·17		0·18		0·20		0·20		0·23		0·24		0·26	
10	986·46	1·10	985·36	1·06	984·30	1·04	983·26	1·02	982·24	0·99	981·25	0·96	980·29	0·95	979·34	0·92	978·42	0·92	977·50	0·91	976·59	0·91	975·68	0·91
	0·12		0·13		0·14		0·16		0·16		0·17		0·19		0·20		0·23		0·25		0·27		0·29	
11	986·34	1·11	985·23	1·07	984·16	1·06	983·10	1·02	982·08	1·00	981·08	0·98	980·10	0·96	979·14	0·95	978·19	0·94	977·25	0·93	976·32	0·93	975·39	0·92
	0·13		0·14		0·16		0·16		0·18		0·19		0·21		0·22		0·24		0·25		0·27		0·28	
12	986·21	1·12	985·09	1·09	984·00	1·06	982·94	1·04	981·90	1·01	980·89	1·00	979·89	0·97	978·92	0·97	977·95	0·95	977·00	0·95	976·05	0·94	975·11	0·95
	0·15		0·16		0·16		0·18		0·19		0·20		0·21		0·23		0·24		0·26		0·28		0·30	
13	986·06	1·13	984·93	1·09	983·84	1·08	982·76	1·05	981·71	1·02	980·69	1·01	979·68	0·99	978·69	0·98	977·71	0·97	976·74	0·97	975·77	0·96	974·81	0·96
	0·16		0·16		0·18		0·18		0·20		0·22		0·23		0·24		0·26		0·27		0·28		0·30	
14	985·90	1·13	984·77	1·11	983·66	1·08	982·58	1·07	981·51	1·04	980·47	1·02	979·45	1·00	978·45	1·00	977·45	0·98	976·47	0·98	975·49	0·98	974·51	0·98
	0·17		0·18		0·19		0·20		0·21		0·22		0·24		0·25		0·26		0·28		0·30		0·32	
15	985·73	1·14	984·59	1·12	983·47	1·09	982·38	1·08	981·30	1·05	980·25	1·04	979·21	1·01	978·20	1·01	977·19	1·00	976·19	1·00	975·19	1·00	974·19	1·00
	0·18		0·19		0·20		0·22		0·22		0·24		0·24		0·27		0·28		0·30		0·31		0·32	
16	985·55	1·15	984·40	1·13	983·27	1·11	982·16	1·08	981·08	1·07	980·01	1·04	978·97	1·04	977·93	1·02	976·91	1·02	975·89	1·01	974·88	1·01	973·87	1·02
	0·19		0·20		0·21		0·22		0·23		0·24		0·26		0·27		0·29		0·30		0·32		0·33	
17	985·36	1·16	984·20	1·14	983·06	1·12	981·94	1·09	980·85	1·08	979·77	1·06	978·71	1·05	977·66	1·04	976·62	1·03	975·59	1·03	974·56	1·02	973·54	1·04
	0·21		0·22		0·22		0·23		0·25		0·26		0·27		0·28		0·29		0·31		0·32		0·35	
18	985·15	1·17	983·98	1·14	982·84	1·13	981·71	1·11	980·60	1·09	979·51	1·07	978·44	1·06	977·38	1·05	976·33	1·05	975·28	1·04	974·24	1·05	973·19	1·05
	0·21		0·22		0·24		0·24		0·25		0·26		0·28		0·29		0·31		0·32		0·34		0·35	
19	984·94	1·18	983·76	1·16	982·60	1·13	981·47	1·12	980·35	1·10	979·25	1·09	978·16	1·07	977·09	1·07	976·02	1·06	974·96	1·06	973·90	1·06	972·84	1·06
	0·23		0·24		0·24		0·26		0·27		0·28		0·29		0·30		0·31		0·33		0·34		0·36	
20	984·71	1·19	983·52	1·16	982·36	1·15	981·21	1·13	980·08	1·11	978·97	1·10	977·87	1·08	976·79	1·08	975·71	1·08	974·63	1·07	973·56	1·08	972·48	1·08

	10		11		12		13		14		15		16		17		18		19		20		21	
20	984·71	1·19	983·52	1·16	982·36	1·15	981·21	1·13	980·08	1·11	978·97	1·10	977·87	1·08	976·79	1·08	975·71	1·08	974·63	1·07	973·56	1·08	972·48	1·08
	0·24		0·24		0·26		0·26		0·27		0·28		0·29		0·31		0·33		0·34		0·36		0·37	
21	984·47	1·19	983·28	1·18	982·10	1·15	980·95	1·14	979·81	1·12	978·69	1·11	977·58	1·10	976·48	1·10	975·38	1·09	974·29	1·09	973·20	1·09	972·11	1·09
	0·24		0·26		0·26		0·28		0·29		0·30		0·31		0·33		0·33		0·35		0·36		0·37	
22	984·23	1·21	983·02	1·18	981·84	1·17	980·67	1·15	979·52	1·13	978·39	1·12	977·27	1·12	976·15	1·10	975·05	1·11	973·94	1·10	972·84	1·10	971·74	1·12
	0·26		0·25		0·27		0·28		0·29		0·31		0·32		0·33		0·35		0·35		0·37		0·39	
23	983·97	1·20	982·77	1·20	981·57	1·18	980·39	1·16	979·23	1·15	978·08	1·13	976·95	1·13	975·82	1·12	974·70	1·11	973·59	1·12	972·47	1·12	971·35	1·13
	0·27		0·29		0·29		0·29		0·30		0·31		0·33		0·33		0·35		0·37		0·38		0·40	
24	983·70	1·22	982·48	1·20	981·28	1·18	980·10	1·17	978·93	1·16	977·77	1·15	976·62	1·13	975·49	1·14	974·35	1·13	973·22	1·13	972·09	1·14	970·95	1·14
	0·28		0·28		0·29		0·31		0·32		0·33		0·33		0·35		0·36		0·37		0·39		0·40	
25	983·42	1·22	982·20	1·21	980·99	1·20	979·79	1·18	978·61	1·17	977·44	1·15	976·29	1·15	975·14	1·15	973·99	1·14	972·85	1·15	971·70	1·15	970·55	1·16
	0·28		0·30		0·31		0·31		0·32		0·33		0·35		0·36		0·37		0·39		0·40		0·41	
26	983·14	1·24	981·90	1·22	980·68	1·20	979·48	1·19	978·29	1·18	977·11	1·17	975·94	1·16	974·78	1·16	973·62	1·16	972·46	1·16	971·30	1·16	970·14	1·17
	0·30		0·30		0·31		0·32		0·33		0·34		0·35		0·36		0·38		0·39		0·40		0·42	
27	982·84	1·24	981·60	1·23	980·37	1·21	979·16	1·20	977·96	1·19	976·77	1·18	975·59	1·17	974·42	1·18	973·24	1·17	972·07	1·17	970·90	1·18	969·72	1·18
	0·31		0·32		0·32		0·33		0·34		0·35		0·36		0·38		0·38		0·40		0·41		0·43	
28	982·53	1·25	981·28	1·23	980·05	1·22	978·83	1·21	977·62	1·20	976·42	1·19	975·23	1·19	974·04	1·18	972·86	1·19	971·67	1·18	970·49	1·20	969·29	1·20
	0·31		0·32		0·33		0·34		0·35		0·36		0·37		0·38		0·40		0·40		0·42		0·43	
29	982·22	1·26	980·96	1·24	979·72	1·23	978·49	1·22	977·27	1·21	976·06	1·20	974·86	1·20	973·66	1·20	972·46	1·19	971·27	1·20	970·07	1·21	968·86	1·22
	0·32		0·33		0·34		0·35		0·36		0·37		0·38		0·40		0·41		0·43		0·44		0·45	
30	981·90	1·27	980·63	1·25	979·38	1·24	978·14	1·23	976·91	1·22	975·69	1·21	974·48	1·22	973·26	1·21	972·05	1·21	970·84	1·21	969·63	1·22	968·41	1·23
	0·34		0·34		0·35		0·36		0·37		0·38		0·40		0·40		0·41		0·42		0·44		0·45	
31	981·56	1·27	980·29	1·26	979·03	1·25	977·78	1·24	976·54	1·23	975·31	1·23	974·08	1·22	972·86	1·22	971·64	1·22	970·42	1·23	969·19	1·23	967·96	1·24
	0·35		0·36		0·36		0·37		0·38		0·39		0·39		0·40		0·42		0·43		0·44		0·46	
32	981·21	1·28	979·93	1·26	978·67	1·26	977·41	1·25	976·16	1·24	974·92	1·23	973·69	1·23	972·46	1·24	971·22	1·23	969·99	1·24	968·75	1·25	967·50	1·25
	0·35		0·35		0·37		0·37		0·38		0·39		0·40		0·42		0·42		0·44		0·45		0·46	
33	980·86	1·28	979·58	1·28	978·30	1·26	977·04	1·26	975·78	1·25	974·53	1·24	973·29	1·25	972·04	1·24	970·80	1·25	969·55	1·25	968·30	1·26	967·04	1·27
	0·36		0·37		0·37		0·38		0·39		0·40		0·41		0·42		0·43		0·44		0·46		0·47	
34	980·50	1·29	979·21	1·28	977·93	1·27	976·66	1·27	975·39	1·26	974·13	1·25	972·88	1·26	971·62	1·25	970·37	1·26	969·11	1·27	967·84	1·27	966·57	1·29
	0·36		0·37		0·38		0·39		0·39		0·40		0·42		0·42		0·44		0·46		0·46		0·48	
35	980·14	1·30	978·84	1·29	977·55	1·28	976·27	1·27	975·00	1·27	973·73	1·27	972·46	1·26	971·20	1·27	969·93	1·28	968·65	1·27	967·38	1·29	966·09	1·30
	0·37		0·38		0·38		0·39		0·40		0·41		0·42		0·44		0·45		0·45		0·47		0·48	
36	979·77	1·31	978·46	1·29	977·17	1·29	975·88	1·28	974·60	1·28	973·32	1·28	972·04	1·28	970·76	1·28	969·48	1·28	968·20	1·29	966·91	1·30	965·61	1·32
	0·39		0·39		0·40		0·40		0·41		0·42		0·43		0·44		0·45		0·47		0·48		0·49	
37	978·38	1·31	978·07	1·30	976·77	1·29	975·48	1·29	974·19	1·29	972·90	1·29	971·61	1·29	970·32	1·29	969·03	1·30	967·73	1·30	966·43	1·31	965·12	1·33
	0·38		0·39		0·40		0·41		0·42		0·43		0·44		0·45		0·46		0·47		0·49		0·50	
38	979·00	1·32	977·68	1·31	976·37	1·30	975·07	1·30	973·77	1·30	972·47	1·30	971·17	1·30	969·87	1·30	968·57	1·31	967·26	1·32	965·94	1·32	964·62	1·34
	0·40		0·40		0·41		0·42		0·42		0·43		0·44		0·45		0·47		0·48		0·49		0·50	
39	978·60	1·32	977·28	1·32	975·96	1·31	974·65	1·30	973·35	1·31	972·04	1·31	970·73	1·31	969·42	1·32	968·10	1·32	966·78	1·33	965·45	1·33	964·12	1·36
	0·40		0·41		0·41		0·42		0·43		0·44		0·45		0·46		0·47		0·48		0·49		0·51	
40	978·20	1·33	976·87	1·32	975·55	1·32	974·23	1·31	972·92	1·32	971·60	1·32	970·28	1·32	968·96	1·33	967·63	1·33	966·30	1·34	964·96	1·35	963·61	1·37

Température (°C)

	20		21		22		23		24		25		26		27		28		29		30		31	
0	978·56	0·70	977·86	0·70	977·16	0·69	976·47	0·71	975·76	0·71	975·05	0·72	974·33	0·75	973·58	0·77	972·81	0·80	972·01	0·83	971·18	0·87	970·31	0·90
	0·14		0·17		0·19		0·22		0·24		0·26		0·29		0·31		0·34		0·36		0·39		0·41	
1	978·42	0·73	977·69	0·72	976·97	0·72	976·25	0·73	975·52	0·73	974·79	0·75	974·04	0·77	973·27	0·80	972·47	0·82	971·65	0·86	970·79	0·89	969·90	0·92
	0·16		0·18		0·20		0·23		0·25		0·28		0·30		0·32		0·34		0·37		0·39		0·41	
2	978·26	0·75	977·51	0·74	976·77	0·75	976·02	0·75	975·27	0·76	974·51	0·77	973·74	0·79	972·95	0·82	972·13	0·85	971·28	0·88	970·40	0·91	969·49	0·95
	0·17		0·19		0·22		0·23		0·26		0·28		0·31		0·33		0·36		0·38		0·40		0·42	
3	978·09	0·77	977·32	0·77	976·55	0·76	975·79	0·78	975·01	0·78	974·23	0·80	973·43	0·81	972·62	0·85	971·77	0·87	970·90	0·90	970·00	0·93	969·07	0·98
	0·18		0·20		0·22		0·25		0·27		0·29		0·31		0·34		0·36		0·38		0·40		0·43	
4	977·91	0·79	977·12	0·79	976·33	0·79	975·54	0·80	974·74	0·80	973·94	0·82	973·12	0·84	972·28	0·87	971·41	0·89	970·52	0·92	969·60	0·96	968·64	1·00
	0·19		0·22		0·23		0·26		0·27		0·30		0·33		0·35		0·37		0·39		0·42		0·44	
5	977·72	0·82	976·90	0·80	976·10	0·82	975·28	0·81	974·47	0·83	973·64	0·85	972·79	0·86	971·93	0·89	971·04	0·91	970·13	0·96	969·18	0·98	968·20	1·01
	0·21		0·22		0·25		0·26		0·29		0·31		0·33		0·35		0·37		0·40		0·42		0·44	
6	977·51	0·83	976·68	0·83	975·85	0·83	975·02	0·84	974·18	0·85	973·33	0·87	972·46	0·86	971·58	0·91	970·67	0·94	969·73	0·97	968·76	1·00	967·76	1·03
	0·21		0·23		0·25		0·28		0·30		0·32		0·34		0·36		0·38		0·40		0·42		0·44	
7	977·30	0·85	976·45	0·85	975·60	0·86	974·74	0·86	973·88	0·87	973·01	0·89	972·12	0·90	971·22	0·93	970·29	0·96	969·33	0·99	968·34	1·02	967·32	1·06
	0·23		0·25		0·27		0·28		0·31		0·33		0·35		0·37		0·40		0·42		0·43		0·46	
8	977·07	0·87	976·20	0·87	975·33	0·87	974·46	0·89	973·57	0·89	972·68	0·91	971·77	0·92	970·85	0·96	969·89	0·98	968·91	1·00	967·91	1·05	966·86	1·07
	0·24		0·26		0·28		0·30		0·31		0·34		0·35		0·38		0·39		0·41		0·44		0·46	
9	976·83	0·89	975·94	0·89	975·05	0·89	974·16	0·90	973·26	0·92	972·34	0·92	971·42	0·95	970·47	0·97	969·50	1·00	968·50	1·03	967·47	1·07	966·40	1·09
	0·24		0·26		0·28		0·30		0·33		0·34		0·37		0·39		0·41		0·43		0·45		0·46	
10	976·59	0·91	975·68	0·91	974·77	0·91	973·86	0·93	972·93	0·93	972·00	0·95	971·05	0·97	970·08	0·99	969·09	1·02	968·07	1·05	967·02	1·08	965·94	1·12
	0·27		0·29		0·30		0·33		0·34		0·36		0·38		0·40		0·42		0·44		0·46		0·47	
11	976·32	0·93	975·39	0·92	974·47	0·94	973·53	0·94	972·59	0·95	971·64	0·97	970·67	0·99	969·68	1·01	968·67	1·04	967·63	1·07	966·56	1·09	965·47	1·13
	0·27		0·28		0·31		0·32		0·34		0·36		0·38		0·40		0·42		0·44		0·45		0·48	
12	976·05	0·94	975·11	0·95	974·16	0·95	973·21	0·96	972·25	0·97	971·28	0·99	970·29	1·01	969·28	1·03	968·25	1·06	967·19	1·08	966·11	1·12	964·99	1·15
	0·28		0·30		0·31		0·33		0·35		0·37		0·39		0·41		0·43		0·45		0·47		0·49	
13	975·77	0·96	974·81	0·96	973·85	0·97	972·88	0·98	971·90	0·99	970·91	1·01	969·90	1·03	968·87	1·05	967·82	1·08	966·74	1·10	965·64	1·14	964·50	1·17
	0·28		0·30		0·32		0·34		0·36		0·38		0·40		0·41		0·43		0·45		0·47		0·49	
14	975·49	0·98	974·51	0·98	973·53	0·99	972·54	1·00	971·54	1·01	970·53	1·03	969·50	1·04	968·46	1·07	967·39	1·10	966·29	1·12	965·17	1·16	964·01	1·19
	0·30		0·32		0·34		0·35		0·37		0·39		0·40		0·42		0·44		0·46		0·48		0·49	
15	975·19	1·00	974·19	1·00	973·19	1·00	972·19	1·02	971·17	1·03	970·14	1·04	969·10	1·06	968·04	1·09	966·95	1·12	965·83	1·14	964·69	1·17	963·52	1·21
	0·31		0·32		0·34		0·36		0·37		0·39		0·41		0·43		0·45		0·46		0·48		0·51	
16	974·88	1·01	973·87	1·02	972·85	1·02	971·83	1·03	970·80	1·05	969·75	1·06	968·69	1·08	967·61	1·11	966·50	1·13	965·37	1·18	964·21	1·20	963·01	1·22
	0·32		0·33		0·35		0·37		0·39		0·40		0·42		0·44		0·45		0·48		0·50		0·50	
17	974·56	1·02	973·54	1·04	972·50	1·04	971·46	1·05	970·41	1·06	969·35	1·08	968·27	1·10	967·17	1·12	966·05	1·16	964·89	1·18	963·71	1·20	962·51	1·24
	0·32		0·35		0·36		0·37		0·39		0·41		0·43		0·45		0·47		0·48		0·49		0·52	
18	974·24	1·05	973·19	1·05	972·14	1·05	971·09	1·07	970·02	1·08	968·94	1·10	967·84	1·12	966·72	1·14	965·58	1·17	964·41	1·19	963·22	1·23	961·99	1·25
	0·34		0·35		0·36		0·39		0·40		0·42		0·43		0·45		0·47		0·48		0·50		0·52	
19	973·90	1·06	972·84	1·06	971·78	1·08	970·70	1·08	969·62	1·10	968·52	1·11	967·41	1·14	966·27	1·16	965·11	1·18	963·93	1·21	962·72	1·25	961·47	1·27
	0·34		0·36		0·38		0·39		0·41		0·42		0·45		0·46		0·47		0·49		0·51		0·52	
20	973·56	1·08	972·48	1·08	971·40	1·09	970·31	1·10	969·21	1·11	968·10	1·14	966·96	1·15	965·81	1·17	964·64	1·20	963·44	1·23	962·21	1·26	960·95	1·29

Temperature (C)

	20		21		22		23		24		25		26		27		28		29		30		31	
20	973·56	1·08	972·48	1·08	971·40	1·09	970·31	1·10	969·21	1·11	968·10	1·14	966·96	1·15	965·81	1·17	964·64	1·20	963·44	1·23	962·21	1·26	960·95	1·29
	0·36		0·37		0·38		0·40		0·42		0·44		0·45		0·46		0·49		0·50		0·52		0·53	
21	973·20	1·09	972·11	1·09	971·02	1·11	969·91	1·12	968·79	1·13	967·66	1·15	966·51	1·16	965·35	1·20	964·15	1·21	962·94	1·25	961·69	1·27	960·42	1·31
	0·36		0·37		0·40		0·41		0·42		0·44		0·45		0·48		0·49		0·51		0·52		0·54	
22	972·84	1·10	971·74	1·12	970·62	1·12	969·50	1·13	968·37	1·15	967·22	1·16	966·06	1·19	964·87	1·21	963·66	1·23	962·43	1·26	961·17	1·29	959·88	1·32
	0·37		0·39		0·40		0·42		0·43		0·45		0·47		0·48		0·49		0·51		0·53		0·55	
23	972·47	1·12	971·35	1·13	970·22	1·14	969·08	1·14	967·94	1·17	966·77	1·18	965·59	1·20	964·39	1·22	963·17	1·25	961·92	1·28	960·64	1·31	959·33	1·33
	0·38		0·40		0·41		0·42		0·44		0·45		0·47		0·49		0·51		0·52		0·54		0·55	
24	972·09	1·14	970·95	1·14	969·81	1·15	968·66	1·16	967·50	1·18	966·32	1·20	965·12	1·22	963·90	1·24	962·66	1·26	961·40	1·30	960·10	1·32	958·78	1·35
	0·39		0·40		0·42		0·43		0·45		0·47		0·48		0·49		0·51		0·53		0·54		0·55	
25	971·70	1·15	970·55	1·16	969·39	1·16	968·23	1·18	967·05	1·20	965·85	1·21	964·64	1·23	963·41	1·26	962·15	1·28	960·87	1·31	959·56	1·33	958·23	1·37
	0·40		0·41		0·42		0·44		0·46		0·47		0·49		0·50		0·51		0·53		0·54		0·57	
26	971·30	1·16	970·14	1·17	968·97	1·18	967·79	1·20	966·59	1·21	965·38	1·23	964·15	1·24	962·91	1·27	961·64	1·30	960·34	1·32	959·02	1·36	957·66	1·38
	0·40		0·42		0·43		0·45		0·46		0·48		0·49		0·51		0·53		0·54		0·56		0·56	
27	970·90	1·18	969·72	1·18	968·54	1·20	967·34	1·21	966·13	1·23	964·90	1·24	963·66	1·26	962·40	1·29	961·11	1·31	959·80	1·34	958·46	1·36	957·10	1·40
	0·41		0·43		0·45		0·46		0·47		0·48		0·50		0·52		0·54		0·56		0·57		0·59	
28	970·49	1·20	969·29	1·20	968·09	1·21	966·88	1·22	965·66	1·24	964·42	1·26	963·16	1·28	961·88	1·31	960·57	1·33	959·24	1·35	957·89	1·38	956·51	1·41
	0·42		0·43		0·45		0·47		0·49		0·50		0·52		0·53		0·53		0·55		0·56		0·58	
29	970·07	1·21	968·86	1·22	967·64	1·23	966·41	1·24	965·17	1·25	963·92	1·28	962·64	1·29	961·35	1·31	960·04	1·35	958·69	1·36	957·33	1·40	955·93	1·42
	0·44		0·45		0·46		0·47		0·49		0·50		0·51		0·53		0·55		0·55		0·58		0·58	
30	969·63	1·22	968·41	1·23	967·18	1·24	965·94	1·26	964·68	1·26	963·42	1·29	962·13	1·31	960·82	1·33	959·49	1·35	958·14	1·39	956·75	1·40	955·35	1·44
	0·44		0·45		0·46		0·48		0·49		0·51		0·52		0·53		0·55		0·57		0·58		0·60	
31	969·19	1·23	967·96	1·24	966·72	1·26	965·46	1·27	964·19	1·28	962·91	1·30	961·61	1·32	960·29	1·35	958·94	1·37	957·57	1·40	956·17	1·42	954·75	1·44
	0·44		0·46		0·47		0·48		0·50		0·51		0·53		0·54		0·55		0·57		0·58		0·59	
32	968·75	1·25	967·50	1·25	966·25	1·27	964·98	1·29	963·69	1·29	962·40	1·32	961·08	1·33	959·75	1·36	958·39	1·39	957·00	1·41	955·59	1·43	954·16	1·46
	0·45		0·46		0·48		0·49		0·50		0·52		0·53		0·55		0·57		0·57		0·59		0·61	
33	968·30	1·26	967·04	1·27	965·77	1·28	964·49	1·30	963·19	1·31	961·88	1·33	960·55	1·35	959·20	1·38	957·82	1·39	956·43	1·43	955·00	1·45	953·55	1·47
	0·46		0·47		0·49		0·50		0·51		0·53		0·54		0·56		0·56		0·59		0·59		0·60	
34	967·84	1·27	966·57	1·29	965·28	1·29	963·99	1·31	962·68	1·33	961·35	1·34	960·01	1·37	958·64	1·38	957·26	1·42	955·84	1·43	954·41	1·46	952·95	1·49
	0·46		0·48		0·49		0·51		0·52		0·53		0·55		0·56		0·58		0·58		0·60		0·62	
35	967·38	1·29	966·09	1·30	964·79	1·31	963·48	1·32	962·16	1·34	960·82	1·36	959·46	1·38	958·08	1·40	956·68	1·42	955·26	1·45	953·81	1·48	952·33	1·50
	0·47		0·48		0·50		0·51		0·53		0·54		0·55		0·57		0·58		0·60		0·61		0·62	
36	966·91	1·30	965·61	1·32	964·29	1·32	962·97	1·34	961·63	1·35	960·28	1·37	958·91	1·40	957·51	1·41	956·10	1·44	954·66	1·46	953·20	1·49	951·71	1·51
	0·48		0·49		0·50		0·52		0·53		0·55		0·56		0·57		0·59		0·60		0·61		0·62	
37	966·43	1·31	965·12	1·33	963·79	1·34	962·45	1·35	961·10	1·37	959·73	1·38	958·35	1·41	956·94	1·43	955·51	1·45	954·06	1·47	952·59	1·50	951·09	1·53
	0·49		0·50		0·51		0·52		0·54		0·55		0·57		0·58		0·59		0·60		0·62		0·63	
38	965·94	1·32	964·62	1·34	963·28	1·35	961·93	1·37	960·56	1·38	959·18	1·40	957·78	1·42	956·36	1·44	954·92	1·46	953·46	1·49	951·97	1·51	950·46	1·54
	0·49		0·50		0·52		0·53		0·54		0·56		0·57		0·58		0·60		0·61		0·62		0·64	
39	965·45	1·33	964·12	1·36	962·76	1·36	961·40	1·38	960·02	1·40	958·62	1·41	957·21	1·43	955·78	1·46	954·32	1·47	952·85	1·50	951·35	1·53	949·82	1·55
	0·49		0·51		0·52		0·54		0·55		0·56		0·58		0·59		0·60		0·62		0·63		0·64	
40	964·96	1·35	963·61	1·37	962·24	1·38	960·86	1·39	959·47	1·41	958·06	1·43	956·63	1·44	955·19	1·47	953·72	1·49	952·23	1·51	950·72	1·54	949·18	1·57

INTERNATIONAL ALCOHOLIC STRENGTH BY % VOL AT 20 °C

Table of densities of hydroalcoholic mixtures — ordinary glass apparatus — densities at t °C, corrected for buoyancy of air

Alcoholic strength by % vol

Temperature (°C)

	0		1		2		3		4		5		6		7		8		9		10		11	
0	999·34	1·52	997·82	1·45	996·37	1·39	994·98	1·35	993·63	1·29	992·34	1·24	991·10	1·18	989·92	1·15	988·77	1·09	987·68	1·05	986·63	1·00	985·63	0·96
	+ 0·09		+ 0·09		+ 0·09		+ 0·08		+ 0·08		+ 0·08		+ 0·07		+ 0·05		+ 0·05		+ 0·04		+ 0·03		+ 0·02	
1	999·43	1·52	997·91	1·45	996·46	1·40	995·06	1·35	993·71	1·29	992·42	1·25	991·17	1·20	989·97	1·15	988·82	1·10	987·72	1·06	986·66	1·01	985·65	0·97
	+ 0·06		+ 0·06		+ 0·06		+ 0·06		+ 0·06		+ 0·05		+ 0·05		+ 0·04		+ 0·03		+ 0·02		+ 0·02		+ 0·01	
2	999·49	1·52	997·97	1·45	996·52	1·40	995·12	1·35	993·77	1·30	992·47	1·25	991·22	1·21	990·01	1·16	988·85	1·11	987·74	1·06	986·68	1·02	985·66	0·98
	+ 0·05		+ 0·05		+ 0·04		+ 0·04		+ 0·04		+ 0·04		+ 0·03		+ 0·03		+ 0·03		+ 0·02		0·00		0·01	
3	999·54	1·52	998·02	1·46	996·56	1·40	995·16	1·35	993·81	1·30	992·51	1·26	991·25	1·21	990·04	1·16	988·88	1·12	987·76	1·08	986·68	1·03	985·65	0·99
	+ 0·03		+ 0·03		+ 0·03		+ 0·03		+ 0·02		+ 0·02		+ 0·02		+ 0·01		0·00		0·01		0·01		0·02	
4	999·57	1·52	998·05	1·46	996·59	1·40	995·19	1·36	993·84	1·30	992·53	1·26	991·27	1·22	990·05	1·17	988·88	1·13	987·75	1·08	986·67	1·04	985·63	1·00
	+ 0·02		+ 0·02		+ 0·02		+ 0·02		+ 0·02		+ 0·01		0·00		0·00		0·00		0·01		0·02		0·03	
5	999·59	1·52	998·07	1·46	996·61	1·40	995·21	1·36	993·85	1·31	992·54	1·27	991·27	1·22	990·05	1·17	988·88	1·14	987·74	1·09	986·65	1·05	985·60	1·02
	0·00		0·00		0·00		0·01		0·01		0·01		0·01		0·02		0·03		0·03		0·04		0·06	
6	999·59	1·52	998·07	1·46	996·61	1·41	995·20	1·36	993·84	1·31	992·53	1·27	991·26	1·23	990·03	1·18	988·85	1·14	987·71	1·10	986·61	1·07	985·54	1·02
	0·01		0·01		0·01		0·01		0·01		0·02		0·02		0·02		0·03		0·04		0·05		0·06	
7	999·58	1·52	998·06	1·46	996·60	1·41	995·19	1·36	993·83	1·32	992·51	1·27	991·24	1·23	990·01	1·19	988·82	1·15	987·67	1·11	986·56	1·08	985·48	1·04
	0·03		0·03		0·03		0·03		0·04		0·04		0·05		0·05		0·06		0·07		0·07		0·08	
8	999·55	1·52	998·03	1·46	996·57	1·41	995·16	1·37	993·79	1·32	992·47	1·28	991·19	1·23	989·96	1·20	988·76	1·16	987·60	1·11	986·49	1·09	985·40	1·05
	0·04		0·04		0·04		0·04		0·04		0·04		0·05		0·06		0·06		0·06		0·08		0·08	
9	999·51	1·52	997·99	1·46	996·53	1·41	995·12	1·37	993·75	1·32	992·43	1·29	991·14	1·24	989·90	1·20	988·70	1·16	987·54	1·13	986·41	1·09	985·32	1·06
	0·06		0·06		0·06		0·06		0·06		0·07		0·07		0·07		0·08		0·09		0·10		0·11	
10	999·45	1·52	997·93	1·46	996·47	1·41	995·06	1·37	993·69	1·33	992·36	1·29	991·07	1·24	989·83	1·21	988·62	1·17	987·45	1·14	986·31	1·10	985·21	1·07
	0·07		0·06		0·06		0·07		0·07		0·07		0·07		0·08		0·09		0·10		0·10		0·11	
11	999·38	1·51	997·87	1·46	996·41	1·42	994·99	1·37	993·62	1·33	992·29	1·29	991·00	1·25	989·75	1·22	988·53	1·18	987·35	1·14	986·21	1·11	985·10	1·08
	0·09		0·09		0·09		0·09		0·09		0·09		0·10		0·11		0·11		0·11		0·12		0·13	
12	999·29	1·51	997·78	1·46	996·32	1·42	994·90	1·37	993·53	1·33	992·20	1·30	990·90	1·26	989·64	1·22	988·42	1·18	987·24	1·15	986·09	1·12	984·97	1·09
	0·09		0·09		0·09		0·09		0·10		0·10		0·10		0·10		0·11		0·12		0·13		0·14	
13	999·20	1·51	997·69	1·46	996·23	1·42	994·81	1·38	993·43	1·33	992·10	1·30	990·80	1·26	989·54	1·23	988·31	1·19	987·12	1·16	985·96	1·13	984·83	1·10
	0·11		0·11		0·11		0·11		0·11		0·12		0·12		0·13		0·13		0·14		0·15		0·16	
14	999·09	1·51	997·58	1·46	996·12	1·42	994·70	1·38	993·32	1·34	991·98	1·30	990·68	1·27	989·41	1·23	988·18	1·20	986·98	1·17	985·81	1·14	984·67	1·11
	0·12		0·12		0·12		0·12		0·12		0·12		0·13		0·13		0·14		0·14		0·15		0·16	
15	998·97	1·51	997·46	1·46	996·00	1·42	994·58	1·38	993·20	1·34	991·86	1·31	990·55	1·27	989·28	1·24	988·04	1·20	986·84	1·18	985·66	1·15	984·51	1·12
	0·13		0·13		0·13		0·13		0·14		0·14		0·14		0·15		0·15		0·17		0·17		0·18	
16	998·84	1·51	997·33	1·46	995·87	1·42	994·45	1·39	993·06	1·34	991·72	1·31	990·41	1·28	989·13	1·24	987·89	1·22	986·67	1·18	985·49	1·16	984·33	1·13
	0·14		0·14		0·14		0·14		0·14		0·15		0·15		0·15		0·16		0·17		0·17		0·18	
17	998·70	1·51	997·19	1·46	995·73	1·42	994·31	1·39	992·92	1·35	991·57	1·31	990·26	1·28	988·98	1·25	987·73	1·22	986·50	1·18	985·32	1·17	984·15	1·14
	0·15		0·15		0·16		0·16		0·16		0·16		0·17		0·17		0·18		0·18		0·19		0·19	
18	998·55	1·51	997·04	1·47	995·57	1·42	994·15	1·39	992·76	1·35	991·41	1·32	990·09	1·28	988·81	1·26	987·55	1·23	986·32	1·19	985·13	1·17	983·96	1·15
	0·17		0·16		0·16		0·16		0·16		0·16		0·17		0·18		0·18		0·19		0·20		0·21	
19	998·38	1·50	996·88	1·47	995·41	1·42	993·99	1·39	992·60	1·35	991·25	1·33	989·92	1·29	988·63	1·26	987·37	1·24	986·13	1·20	984·93	1·18	983·75	1·16
	0·18		0·18		0·18		0·18		0·19		0·19		0·19		0·20		0·21		0·21		0·22		0·23	
20	998·20	1·50	996·70	1·47	995·23	1·42	993·81	1·40	992·41	1·35	991·06	1·33	989·73	1·30	988·43	1·27	987·16	1·24	985·92	1·21	984·71	1·19	983·52	1·17
	0·19		0·19		0·19		0·19		0·19		0·20		0·20		0·21		0·21		0·22		0·23		0·23	
21	998·01	1·50	996·51	1·47	995·04	1·42	993·62	1·40	992·22	1·36	990·86	1·33	989·53	1·31	988·22	1·27	986·95	1·25	985·70	1·22	984·48	1·19	983·29	1·17
	0·20		0·20		0·19		0·20		0·20		0·20		0·21		0·21		0·22		0·22		0·23		0·24	
22	997·81	1·50	996·31	1·46	994·85	1·43	993·42	1·40	992·02	1·36	990·66	1·34	989·32	1·31	988·01	1·28	986·73	1·25	985·48	1·23	984·25	1·20	983·05	1·18
	0·21		0·21		0·21		0·21		0·21		0·22		0·22		0·22		0·23		0·24		0·24		0·25	
23	997·60	1·50	996·10	1·46	994·64	1·43	993·21	1·40	991·81	1·37	990·44	1·34	989·10	1·31	987·79	1·29	986·50	1·26	985·24	1·23	984·01	1·21	982·80	1·19
	0·21		0·21		0·22		0·22		0·22		0·22		0·23		0·23		0·23		0·24		0·25		0·26	
24	997·39	1·50	995·89	1·47	994·42	1·43	992·99	1·40	991·59	1·37	990·22	1·35	988·87	1·31	987·56	1·29	986·27	1·27	985·00	1·24	983·76	1·22	982·54	1·20
	0·23		0·23		0·23		0·23		0·24		0·24		0·24		0·25		0·25		0·25		0·26		0·27	
25	997·16	1·50	995·66	1·47	994·19	1·43	992·76	1·41	991·35	1·37	989·98	1·35	988·63	1·32	987·31	1·29	986·02	1·27	984·75	1·25	983·50	1·23	982·27	1·21
	0·23		0·23		0·23		0·24		0·24		0·24		0·24		0·25		0·26		0·27		0·27		0·28	
26	996·93	1·50	995·43	1·47	993·96	1·44	992·52	1·41	991·11	1·37	989·74	1·35	988·39	1·33	987·06	1·30	985·76	1·28	984·48	1·25	983·23	1·24	981·99	1·22
	0·25		0·25		0·25		0·25		0·25		0·26		0·26		0·26		0·27		0·28		0·29		0·29	
27	996·68	1·50	995·18	1·47	993·71	1·44	992·27	1·41	990·86	1·38	989·48	1·35	988·13	1·33	986·80	1·31	985·49	1·29	984·20	1·26	982·94	1·24	981·70	1·23
	0·25		0·25		0·26		0·26		0·26		0·26		0·27		0·28		0·28		0·28		0·29		0·30	
28	996·43	1·50	994·93	1·48	993·45	1·44	992·01	1·41	990·60	1·38	989·22	1·36	987·86	1·34	986·52	1·31	985·21	1·29	983·92	1·27	982·65	1·25	981·40	1·23
	0·26		0·27		0·27		0·27		0·27		0·28		0·28		0·28		0·29		0·29		0·30		0·31	
29	996·17	1·51	994·66	1·48	993·18	1·44	991·74	1·41	990·33	1·39	988·94	1·36	987·58	1·34	986·24	1·32	984·92	1·29	983·63	1·28	982·35	1·26	981·09	1·24
	0·27		0·27		0·27		0·28		0·28		0·28		0·28		0·29		0·29		0·30		0·31		0·32	
30	995·90	1·51	994·39	1·48	992·91	1·45	991·46	1·41	990·05	1·39	988·66	1·37	987·29	1·34	985·95	1·32	984·63	1·30	983·33	1·29	982·04	1·27	980·77	1·25
	0·29		0·29		0·29		0·29		0·30		0·30		0·30		0·31		0·31		0·32		0·32		0·32	
31	995·61	1·51	994·10	1·48	992·62	1·45	991·17	1·42	989·75	1·39	988·36	1·37	986·99	1·35	985·64	1·33	984·31	1·30	983·01	1·29	981·72	1·27	980·45	1·26
	0·29		0·29		0·29		0·29		0·30		0·31		0·31		0·31		0·31		0·32		0·33		0·34	
32	995·32	1·51	993·81	1·48	992·33	1·45	990·88	1·42	989·45	1·40	988·05	1·37	986·68	1·35	985·33	1·33	984·00	1·31	982·69	1·30	981·39	1·28	980·11	1·26
	0·30		0·31		0·31		0·31		0·31		0·31		0·31		0·32		0·33		0·33		0·34		0·34	
33	995·02	1·52	993·50	1·48	992·02	1·45	990·57	1·43	989·14	1·40	987·74	1·37	986·37	1·36	985·01	1·34	983·67	1·31	982·36	1·31	981·05	1·28	979·77	1·27
	0·30		0·31		0·31		0·31		0·31		0·32		0·33		0·33		0·33		0·34		0·34		0·35	
34	994·72	1·53	993·19	1·48	991·71	1·45	990·26	1·43	988·83	1·41	987·42	1·38	986·04	1·36	984·68	1·34	983·34	1·32	982·02	1·31	980·71	1·29	979·42	1·28
	0·32		0·32		0·32		0·33		0·33		0·33		0·33		0·33		0·33		0·34		0·34		0·35	
35	994·40	1·53	992·87	1·48	991·39	1·46	989·93	1·43	988·50	1·41	987·09	1·38	985·71	1·36	984·35	1·34	983·01	1·33	981·68	1·31	980·37	1·30	979·07	1·29
	0·32		0·32		0·33		0·33		0·33		0·33		0·34		0·34		0·35		0·35		0·36		0·37	
36	994·08	1·53	992·55	1·49	991·06	1·46	989·60	1·43	988·17	1·41	986·76	1·39	985·37	1·36	984·01	1·35	982·66	1·33	981·33	1·32	980·01	1·31	978·70	1·29
	0·33		0·34		0·34		0·34		0·35		0·35		0·35		0·35		0·36		0·36		0·36		0·37	
37	993·75	1·54	992·21	1·49	990·72	1·46	989·26	1·44	987·82	1·41	986·41	1·39	985·02	1·37	983·65	1·35	982·30	1·33	980·97	1·32	979·65	1·32	978·33	1·30
	0·34		0·34		0·35		0·36		0·36		0·36		0·36		0·36		0·37		0·38		0·38		0·38	
38	993·41	1·54	991·87	1·50	990·37	1·47	988·90	1·44	987·46	1·41	986·05	1·39	984·66	1·37	983·29	1·36	981·93	1·34	980·59	1·32	979·27	1·32	977·95	1·31
	0·35		0·35		0·36		0·36		0·36		0·37		0·37		0·37		0·37		0·38		0·38		0·39	
39	993·06	1·54	991·52	1·51	990·01	1·47	988·54	1·44	987·10	1·41	985·68	1·39	984·29	1·37	982·92	1·36	981·56	1·34	980·22	1·33	978·89	1·33	977·56	1·31
	0·35		0·36		0·36		0·37		0·38		0·38		0·38		0·38		0·38		0·39		0·39		0·39	
40	992·71	1·55	991·16	1·51	989·65	1·48	988·17	1·45	986·72	1·42	985·30	1·39	983·91	1·37	982·54	1·36	981·18	1·35	979·83	1·33	978·50	1·33	977·17	1·32

Temperature (°C)

	10		11		12		13		14		15		16		17		18		19		20		21	
0	986·63	1·00	985·63	0·96	984·67	0·92	983·75	0·87	982·88	0·84	982·04	0·81	981·23	0·77	980·46	0·75	979·71	0·73	978·98	0·72	978·26	0·70	977·56	0·70
	+ 0·03		+ 0·02		+ 0·01		0·00		0·02		0·04		0·05		0·07		0·09		0·11		0·13		0·15	
1	986·66	1·01	985·65	0·97	984·68	0·93	983·75	0·89	982·86	0·86	982·00	0·82	981·18	0·79	980·39	0·77	979·62	0·75	978·87	0·74	978·13	0·72	977·41	0·72
	+ 0·02		+ 0·01		0·00		0·01		0·03		0·04		0·06		0·08		0·10		0·12		0·14		0·17	
2	986·68	1·02	985·66	0·98	984·68	0·94	983·74	0·91	982·83	0·87	981·96	0·84	981·12	0·81	980·31	0·79	979·52	0·77	978·75	0·76	977·99	0·75	977·24	0·74
	0·00		0·01		0·02		0·04		0·05		0·06		0·08		0·10		0·12		0·14		0·16		0·18	
3	986·68	1·03	985·65	0·99	984·66	0·96	983·70	0·92	982·78	0·88	981·90	0·86	981·04	0·83	980·21	0·81	979·40	0·79	978·61	0·78	977·83	0·77	977·06	0·76
	0·01		0·02		0·03		0·04		0·05		0·07		0·08		0·10		0·12		0·14		0·16		0·18	
4	986·67	1·04	985·63	1·00	984·63	0·97	983·66	0·93	982·73	0·90	981·83	0·87	980·96	0·85	980·11	0·83	979·28	0·81	978·47	0·80	977·67	0·79	976·88	0·79
	0·02		0·03		0·05		0·06		0·08		0·09		0·11		0·13		0·14		0·16		0·18		0·20	
5	986·65	1·05	985·60	1·02	984·58	0·98	983·60	0·95	982·65	0·91	981·74	0·89	980·85	0·87	979·98	0·84	979·14	0·83	978·31	0·82	977·49	0·81	976·68	0·81
	0·04		0·06		0·06		0·07		0·08		0·10		0·11		0·13		0·15		0·17		0·19		0·21	
6	986·61	1·07	985·54	1·02	984·52	0·99	983·53	0·96	982·57	0·93	981·64	0·90	980·74	0·89	979·85	0·86	978·99	0·85	978·14	0·84	977·30	0·83	976·47	0·83
	0·05		0·06		0·08		0·09		0·10		0·12		0·14		0·15		0·17		0·19		0·20		0·22	
7	986·56	1·08	985·48	1·04	984·44	1·00	983·44	0·97	982·47	0·95	981·52	0·92	980·60	0·90	979·70	0·88	978·82	0·87	977·95	0·85	976·10	0·85	976·25	0·85
	0·07		0·08		0·09		0·10		0·11		0·12		0·14		0·16		0·18		0·19		0·21		0·23	
8	986·49	1·09	985·40	1·05	984·35	1·01	983·34	0·98	982·36	0·96	981·40	0·94	980·46	0·92	979·54	0·90	978·64	0·88	977·76	0·87	976·89	0·87	976·02	0·87
	0·08		0·08		0·09		0·11		0·13		0·14		0·15		0·16		0·18		0·20		0·22		0·24	
9	986·41	1·09	985·32	1·06	984·26	1·03	983·23	1·00	982·23	0·97	981·26	0·95	980·31	0·93	979·38	0·92	978·48	0·90	977·56	0·89	976·67	0·89	975·78	0·89
	0·10		0·11		0·12		0·13		0·14		0·16		0·17		0·18		0·19		0·21		0·23		0·25	
10	986·31	1·10	985·21	1·07	984·14	1·04	983·10	1·01	982·09	0·99	981·10	0·96	980·14	0·94	979·20	0·93	978·27	0·92	977·35	0·91	976·44	0·91	975·53	0·91
	0·10		0·11		0·12		0·13		0·15		0·16		0·17		0·19		0·21		0·23		0·25		0·27	
11	986·21	1·11	985·10	1·08	984·02	1·05	982·97	1·03	981·94	1·00	980·94	0·97	979·97	0·96	979·01	0·95	978·06	0·94	977·12	0·93	976·19	0·93	975·26	0·92
	0·12		0·13		0·14		0·15		0·16		0·17		0·19		0·21		0·22		0·24		0·26		0·27	
12	986·09	1·12	984·97	1·09	983·88	1·06	982·82	1·04	981·78	1·01	980·77	0·99	979·78	0·98	978·80	0·96	977·84	0·96	976·88	0·95	975·93	0·94	974·99	0·94
	0·13		0·14		0·15		0·16		0·17		0·19		0·20		0·21		0·23		0·24		0·26		0·28	
13	985·96	1·13	984·83	1·10	983·73	1·07	982·66	1·05	981·61	1·03	980·58	1·00	979·58	0·99	978·59	0·98	977·61	0·97	976·64	0·97	975·67	0·96	974·71	0·96
	0·15		0·16		0·17		0·18		0·19		0·20		0·22		0·23		0·24		0·26		0·27		0·29	
14	985·81	1·14	984·67	1·11	983·56	1·08	982·48	1·06	981·42	1·04	980·38	1·02	979·36	1·00	978·36	0·99	977·37	0·99	976·38	0·98	975·40	0·98	974·42	0·98
	0·15		0·16		0·17		0·18		0·19		0·20		0·22		0·24		0·26		0·27		0·28		0·30	
15	985·66	1·15	984·51	1·12	983·39	1·09	982·30	1·07	981·23	1·05	980·18	1·04	979·14	1·02	978·12	1·01	977·11	1·00	976·11	0·99	975·12	1·00	974·12	1·00
	0·17		0·18		0·19		0·20		0·21		0·22		0·23		0·25		0·26		0·28		0·30		0·31	
16	985·49	1·16	984·33	1·13	983·20	1·10	982·10	1·08	981·02	1·06	979·96	1·05	978·91	1·04	977·87	1·02	976·85	1·02	975·83	1·01	974·82	1·01	973·81	1·02
	0·17		0·18		0·19		0·20		0·21		0·23		0·24		0·25		0·27		0·29		0·30		0·31	
17	985·32	1·17	984·15	1·14	983·01	1·11	981·90	1·09	980·81	1·08	979·73	1·06	978·67	1·05	977·62	1·04	976·58	1·04	975·54	1·02	974·52	1·02	973·50	1·04
	0·19		0·19		0·20		0·22		0·24		0·25		0·26		0·27		0·28		0·29		0·31		0·33	
18	985·13	1·17	983·96	1·15	982·81	1·13	981·68	1·11	980·57	1·09	979·42	1·07	978·41	1·06	977·35	1·05	976·30	1·05	975·25	1·04	974·21	1·04	973·17	1·05
	0·20		0·21		0·22		0·23		0·24		0·25		0·26		0·27		0·29		0·30		0·32		0·34	
19	984·93	1·18	983·75	1·16	982·59	1·14	981·45	1·12	980·33	1·10	979·23	1·08	978·15	1·07	977·08	1·07	976·01	1·06	974·94	1·05	973·89	1·06	972·83	1·06
	0·22		0·23		0·24		0·24		0·25		0·26		0·28		0·29		0·30		0·31		0·33		0·35	
20	984·71	1·19	983·52	1·17	982·35	1·14	981·21	1·13	980·08	1·11	978·97	1·10	977·87	1·08	976·79	1·08	975·71	1·08	974·63	1·07	973·56	1·08	972·48	1·08
	0·23		0·23		0·23		0·25		0·26		0·28		0·29		0·31		0·32		0·33		0·35		0·36	
21	984·48	1·19	983·29	1·17	982·12	1·16	980·96	1·14	979·82	1·13	978·69	1·11	977·58	1·10	976·48	1·09	975·39	1·09	974·30	1·09	973·21	1·09	972·12	1·09
	0·23		0·24		0·25		0·26		0·27		0·28		0·29		0·31		0·32		0·33		0·35		0·36	
22	984·25	1·20	983·05	1·18	981·97	1·17	980·70	1·15	979·55	1·14	978·41	1·12	977·29	1·12	976·17	1·10	975·07	1·10	973·97	1·10	972·86	1·10	971·76	1·11
	0·24		0·25		0·26		0·27		0·28		0·29		0·30		0·31		0·33		0·34		0·35		0·37	
23	984·01	1·21	982·80	1·19	981·61	1·18	980·43	1·16	979·27	1·15	978·12	1·13	976·99	1·13	975·86	1·12	974·74	1·11	973·63	1·12	972·51	1·12	971·39	1·13
	0·25		0·26		0·27		0·28		0·29		0·30		0·31		0·32		0·33		0·35		0·36		0·38	
24	983·76	1·22	982·54	1·20	981·34	1·19	980·15	1·17	978·98	1·16	977·82	1·14	976·68	1·14	975·54	1·13	974·41	1·13	973·28	1·13	972·15	1·14	971·01	1·14
	0·26		0·27		0·28		0·29		0·30		0·31		0·32		0·33		0·35		0·36		0·38		0·39	
25	983·50	1·23	982·27	1·21	981·06	1·20	979·86	1·18	978·68	1·17	977·51	1·16	976·36	1·15	975·21	1·15	974·06	1·14	972·92	1·15	971·77	1·15	970·62	1·15
	0·27		0·28		0·29		0·29		0·30		0·31		0·33		0·34		0·35		0·37		0·38		0·39	
26	983·23	1·24	981·99	1·22	980·77	1·20	979·57	1·19	978·38	1·18	977·20	1·17	976·03	1·16	974·87	1·16	973·71	1·16	972·55	1·16	971·39	1·16	970·23	1·17
	0·29		0·29		0·30		0·31		0·32		0·33		0·34		0·36		0·37		0·38		0·39		0·41	
27	982·94	1·24	981·70	1·23	980·47	1·21	979·26	1·20	978·06	1·19	976·87	1·18	975·69	1·18	974·51	1·17	973·34	1·17	972·17	1·17	971·00	1·18	969·82	1·18
	0·29		0·30		0·30		0·31		0·32		0·33		0·35		0·36		0·38		0·39		0·40		0·41	
28	982·65	1·25	981·40	1·23	980·17	1·22	978·95	1·21	977·74	1·20	976·54	1·20	975·34	1·19	974·15	1·19	972·96	1·18	971·78	1·18	970·60	1·19	969·41	1·20
	0·30		0·31		0·32		0·33		0·34		0·35		0·36		0·37		0·38		0·39		0·40		0·42	
29	982·35	1·26	981·09	1·24	979·85	1·23	978·62	1·22	977·40	1·21	976·19	1·21	974·98	1·20	973·78	1·20	972·58	1·19	971·39	1·19	970·20	1·21	968·99	1·21
	0·31		0·32		0·33		0·34		0·35		0·36		0·37		0·38		0·38		0·40		0·42		0·43	
30	982·04	1·27	980·77	1·25	979·52	1·24	978·28	1·23	977·05	1·22	975·83	1·21	974·62	1·21	973·41	1·21	972·20	1·21	970·99	1·21	969·78	1·22	968·56	1·23
	0·32		0·32		0·33		0·34		0·35		0·36		0·37		0·38		0·39		0·40		0·42		0·43	
31	981·72	1·27	980·45	1·26	979·19	1·25	977·94	1·24	976·70	1·23	975·47	1·22	974·25	1·22	973·03	1·22	971·81	1·22	970·59	1·23	969·36	1·23	968·13	1·24
	0·33		0·34		0·34		0·35		0·36		0·37		0·38		0·39		0·40		0·42		0·43		0·45	
32	981·39	1·28	980·11	1·26	978·85	1·26	977·59	1·25	976·34	1·24	975·10	1·23	973·87	1·23	972·64	1·23	971·41	1·24	970·17	1·24	968·93	1·25	967·68	1·26
	0·34		0·34		0·35		0·35		0·36		0·37		0·39		0·40		0·41		0·42		0·43		0·45	
33	981·05	1·28	979·77	1·27	978·50	1·26	977·24	1·26	975·78	1·25	974·73	1·25	973·48	1·24	972·24	1·24	971·00	1·25	969·75	1·25	968·50	1·27	967·23	1·27
	0·34		0·35		0·36		0·37		0·38		0·39		0·40		0·41		0·42		0·43		0·45		0·45	
34	980·71	1·29	979·42	1·28	978·14	1·27	976·87	1·27	975·60	1·26	974·34	1·26	973·08	1·25	971·83	1·25	970·58	1·26	969·32	1·27	968·05	1·27	966·78	1·29
	0·34		0·35		0·36		0·37		0·38		0·39		0·40		0·41		0·43		0·44		0·45		0·47	
35	980·37	1·30	979·07	1·29	977·78	1·28	976·50	1·28	975·22	1·27	973·95	1·27	972·68	1·26	971·42	1·27	970·15	1·27	968·88	1·28	967·60	1·29	966·31	1·30
	0·36		0·37		0·37		0·38		0·38		0·39		0·40		0·42		0·43		0·44		0·45		0·47	
36	980·01	1·31	978·70	1·29	977·41	1·29	976·12	1·28	974·84	1·28	973·56	1·28	972·28	1·28	971·00	1·28	969·72	1·28	968·44	1·29	967·15	1·31	965·84	1·31
	0·36		0·37		0·38		0·39		0·40		0·41		0·42		0·43		0·44		0·45		0·46		0·47	
37	979·65	1·32	978·33	1·30	977·03	1·30	975·73	1·29	974·44	1·29	973·15	1·29	971·86	1·29	970·57	1·29	969·28	1·29	967·99	1·30	966·69	1·32	965·37	1·32
	0·38		0·38		0·39		0·39		0·40		0·41		0·42		0·43		0·44		0·46		0·47		0·48	
38	979·27	1·32	977·95	1·31	976·64	1·30	975·34	1·30	974·04	1·30	972·74	1·30	971·44	1·30	970·14	1·30	968·84	1·31	967·53	1·31	966·22	1·33	964·89	1·34
	0·38		0·39		0·39		0·40		0·41		0·42		0·43		0·44		0·45		0·46		0·48		0·49	
39	978·89	1·33	977·56	1·31	976·25	1·31	974·94	1·31	973·63	1·31	972·32	1·31	971·01	1·31	969·70	1·31	968·39	1·32	967·07	1·33	965·74	1·34	964·40	1·36
	0·39		0·39		0·40		0·41		0·42		0·42		0·43		0·45		0·47		0·48		0·49		0·50	
40	978·50	1·33	977·17	1·32	975·85	1·32	974·53	1·32	973·21	1·31	971·90	1·32	970·58	1·33	969·25	1·33	967·92	1·33	966·59	1·34	965·25	1·35	963·90	1·37

Temperature (°C)

	20		21		22		23		24		25		26		27		28		29		30		31	
0	978·26	0·70	977·56	0·70	976·86	0·69	976·17	0·70	975·47	0·72	974·75	0·72	974·03	0·74	973·29	0·77	972·52	0·80	971·72	0·83	970·89	0·87	970·02	0·90
	0·13		0·15		0·17		0·20		0·22		0·24		0·27		0·30		0·32		0·35		0·37		0·39	
1	978·13	0·72	977·41	0·72	976·69	0·72	975·97	0·72	975·25	0·74	974·51	0·75	973·76	0·77	972·99	0·79	972·20	0·83	971·37	0·85	970·52	0·89	969·63	0·93
	0·14		0·17		0·19		0·21		0·24		0·26		0·29		0·31		0·34		0·36		0·38		0·41	
2	977·99	0·75	977·24	0·74	976·50	0·74	975·76	0·75	975·01	0·76	974·25	0·78	973·47	0·79	972·68	0·82	971·86	0·85	971·01	0·87	970·14	0·92	969·22	0·96
	0·16		0·18		0·20		0·23		0·25		0·27		0·29		0·32		0·34		0·36		0·38		0·40	
3	977·83	0·77	977·06	0·76	976·30	0·77	975·53	0·77	974·76	0·78	973·98	0·80	973·18	0·82	972·36	0·84	971·52	0·87	970·65	0·89	969·76	0·94	968·82	0·98
	0·16		0·18		0·21		0·23		0·25		0·28		0·30		0·32		0·34		0·36		0·39		0·42	
4	977·67	0·79	976·88	0·79	976·09	0·79	975·30	0·79	974·51	0·81	973·70	0·82	972·88	0·84	972·04	0·86	971·18	0·89	970·29	0·92	969·37	0·96	968·40	1·00
	0·18		0·20		0·22		0·24		0·26		0·28		0·30		0·33		0·35		0·38		0·40		0·41	
5	977·49	0·81	976·68	0·81	975·87	0·81	975·06	0·81	974·25	0·83	973·42	0·84	972·58	0·86	971·71	0·88	970·83	0·92	969·91	0·95	968·97	0·98	967·99	1·02
	0·19		0·21		0·23		0·25		0·27		0·30		0·33		0·34		0·37		0·39		0·41		0·43	
6	977·30	0·83	976·47	0·83	975·64	0·83	974·81	0·84	973·97	0·85	973·12	0·87	972·25	0·88	971·37	0·91	970·46	0·94	969·52	0·96	968·56	1·00	967·56	1·04
	0·20		0·22		0·24		0·26		0·28		0·30		0·32		0·35		0·37		0·39		0·41		0·43	
7	976·10	0·85	976·25	0·85	975·40	0·85	974·55	0·86	973·69	0·87	972·82	0·89	971·93	0·91	971·02	0·93	970·09	0·96	969·13	0·98	968·15	1·02	967·13	1·06
	0·21		0·23		0·25		0·27		0·29		0·31		0·33		0·35		0·37		0·39		0·42		0·44	
8	976·89	0·87	976·02	0·87	975·15	0·87	974·28	0·88	973·40	0·89	972·51	0·91	971·60	0·93	970·67	0·95	969·72	0·98	968·74	1·01	967·73	1·04	966·69	1·08
	0·22		0·24		0·26		0·28		0·30		0·32		0·34		0·36		0·39		0·41		0·43		0·45	
9	976·67	0·89	975·78	0·89	974·89	0·89	974·00	0·90	973·10	0·91	972·19	0·93	971·26	0·95	970·31	0·98	969·33	1·00	968·33	1·03	967·30	1·06	966·24	1·09
	0·23		0·25		0·27		0·29		0·31		0·33		0·35		0·37		0·39		0·41		0·43		0·45	
10	976·44	0·91	975·53	0·91	974·62	0·91	973·71	0·92	972·79	0·93	971·86	0·95	970·91	0·97	969·94	1·00	968·94	1·02	967·92	1·05	966·87	1·08	965·79	1·11
	0·25		0·27		0·28		0·30		0·32		0·34		0·36		0·38		0·40		0·42		0·44		0·45	
11	976·19	0·93	975·26	0·92	974·34	0·93	973·41	0·94	972·47	0·95	971·52	0·97	970·55	0·99	969·56	1·02	968·54	1·04	967·50	1·07	966·43	1·09	965·34	1·13
	0·26		0·27		0·29		0·31		0·33		0·35		0·37		0·39		0·40		0·42		0·44		0·46	
12	975·93	0·94	974·99	0·94	974·05	0·95	973·10	0·96	972·14	0·97	971·17	0·99	970·18	1·01	969·17	1·03	968·14	1·06	967·08	1·09	965·99	1·11	964·88	1·15
	0·26		0·28		0·30		0·32		0·34		0·36		0·38		0·39		0·41		0·43		0·45		0·47	
13	975·67	0·96	974·71	0·96	973·75	0·97	972·78	0·98	971·80	0·99	970·81	1·01	969·80	1·02	968·78	1·05	967·73	1·08	966·65	1·11	965·54	1·13	964·41	1·17
	0·27		0·29		0·31		0·33		0·35		0·37		0·38		0·40		0·42		0·44		0·45		0·47	
14	975·40	0·98	974·42	0·98	973·44	0·99	972·45	1·00	971·45	1·01	970·44	1·02	969·42	1·04	968·38	1·07	967·31	1·10	966·21	1·12	965·09	1·15	963·94	1·19
	0·28		0·30		0·32		0·33		0·35		0·37		0·39		0·41		0·43		0·45		0·47		0·49	
15	975·12	1·00	974·12	1·00	973·12	1·00	972·12	1·02	971·10	1·03	970·07	1·04	969·03	1·06	967·97	1·09	966·88	1·12	965·76	1·14	964·62	1·17	963·45	1·20
	0·30		0·31		0·33		0·35		0·36		0·38		0·40		0·42		0·44		0·45		0·47		0·49	
16	974·82	1·01	973·81	1·02	972·79	1·02	971·77	1·03	970·74	1·05	969·69	1·06	968·63	1·08	967·55	1·11	966·44	1·13	965·31	1·16	964·15	1·19	962·96	1·22
	0·30		0·31		0·33		0·35		0·37		0·38		0·40		0·42		0·43		0·45		0·47		0·49	
17	974·52	1·02	973·50	1·04	972·46	1·04	971·42	1·05	970·37	1·06	969·31	1·08	968·23	1·10	967·13	1·12	966·01	1·15	964·86	1·18	963·68	1·21	962·47	1·24
	0·31		0·33		0·34		0·36		0·38		0·40		0·42		0·43		0·45		0·47		0·48		0·50	
18	974·21	1·04	973·17	1·05	972·12	1·06	971·06	1·07	969·99	1·08	968·91	1·10	967·81	1·11	966·70	1·14	965·56	1·17	964·39	1·19	963·20	1·23	961·97	1·26
	0·32		0·34		0·35		0·36		0·38		0·40		0·42		0·44		0·46		0·47		0·49		0·50	
19	973·89	1·06	972·83	1·06	971·77	1·07	970·70	1·09	969·61	1·10	968·51	1·11	967·39	1·13	966·26	1·16	965·10	1·18	963·92	1·21	962·71	1·24	961·47	1·28
	0·33		0·35		0·37		0·39		0·40		0·41		0·42		0·45		0·46		0·48		0·50		0·52	
20	973·56	1·08	972·48	1·08	971·40	1·09	970·31	1·10	969·21	1·11	968·10	1·13	966·97	1·14	965·81	1·17	964·64	1·20	963·44	1·23	962·21	1·26	960·95	1·29

Temperature (°C)

	20		21		22		23		24		25		26		27		28		29		30		31	
20	973·56	1·08	972·48	1·08	971·40	1·09	970·31	1·10	969·21	1·11	968·10	1·13	966·97	1·16	965·81	1·17	964·64	1·20	963·44	1·23	962·21	1·26	960·95	1·29
	0·35		0·36		0·37		0·39		0·40		0·42		0·44		0·45		0·47		0·49		0·50		0·52	
21	973·21	1·09	972·12	1·09	971·03	1·11	969·92	1·11	968·81	1·13	967·68	1·15	966·53	1·17	965·36	1·19	964·17	1·22	962·95	1·24	961·71	1·28	960·43	1·31
	0·35		0·36		0·38		0·39		0·41		0·43		0·44		0·46		0·48		0·49		0·51		0·52	
22	972·86	1·10	971·76	1·11	970·65	1·12	969·53	1·13	968·40	1·15	967·25	1·16	966·09	1·19	964·90	1·21	963·69	1·23	962·46	1·26	961·20	1·29	959·91	1·32
	0·35		0·37		0·39		0·40		0·42		0·43		0·45		0·46		0·48		0·50		0·52		0·53	
23	972·51	1·12	971·39	1·13	970·26	1·13	969·13	1·15	967·98	1·16	966·82	1·18	965·64	1·20	964·44	1·23	963·21	1·25	961·96	1·28	960·68	1·30	959·38	1·33
	0·36		0·38		0·39		0·41		0·42		0·44		0·46		0·48		0·49		0·51		0·52		0·54	
24	972·15	1·14	971·01	1·14	969·87	1·15	968·72	1·16	967·56	1·18	966·38	1·20	965·18	1·22	963·96	1·24	962·72	1·27	961·45	1·29	960·16	1·32	958·84	1·34
	0·38		0·39		0·40		0·42		0·44		0·45		0·46		0·48		0·50		0·51		0·53		0·54	
25	971·77	1·15	970·62	1·15	969·47	1·17	968·30	1·18	967·12	1·19	965·93	1·21	964·72	1·24	963·48	1·26	962·22	1·28	960·94	1·31	959·63	1·33	958·30	1·36
	0·38		0·39		0·41		0·42		0·44		0·46		0·48		0·49		0·50		0·52		0·53		0·55	
26	971·39	1·16	970·23	1·17	969·06	1·18	967·88	1·20	966·68	1·21	965·47	1·23	964·24	1·25	962·99	1·27	961·72	1·30	960·42	1·32	959·10	1·35	957·75	1·38
	0·39		0·41		0·42		0·44		0·45		0·46		0·48		0·50		0·51		0·52		0·53		0·55	
27	971·00	1·18	969·82	1·18	968·64	1·20	967·44	1·21	966·23	1·22	965·01	1·25	963·76	1·27	962·49	1·28	961·21	1·31	959·90	1·33	958·57	1·37	957·20	1·40
	0·40		0·41		0·43		0·44		0·46		0·48		0·49		0·50		0·52		0·53		0·55		0·56	
28	970·60	1·19	969·41	1·20	968·21	1·21	967·00	1·23	965·77	1·24	964·53	1·26	963·27	1·28	961·99	1·30	960·69	1·32	959·37	1·35	958·02	1·38	956·64	1·41
	0·40		0·42		0·43		0·45		0·46		0·48		0·49		0·50		0·52		0·54		0·55		0·56	
29	970·20	1·21	968·99	1·21	967·78	1·23	966·55	1·24	965·31	1·26	964·05	1·27	962·78	1·29	961·49	1·32	960·17	1·34	958·83	1·36	957·47	1·39	956·08	1·43
	0·42		0·43		0·45		0·46		0·47		0·48		0·50		0·52		0·53		0·54		0·56		0·58	
30	969·78	1·22	968·56	1·23	967·33	1·24	966·09	1·25	964·84	1·27	963·57	1·29	962·28	1·31	960·97	1·33	959·64	1·35	958·29	1·38	956·91	1·41	955·50	1·44
	0·42		0·43		0·44		0·45		0·47		0·49		0·51		0·52		0·53		0·55		0·56		0·58	
31	969·36	1·23	968·13	1·24	966·89	1·25	965·64	1·27	964·37	1·29	963·08	1·31	961·77	1·32	960·45	1·34	959·11	1·37	957·74	1·39	956·35	1·43	954·92	1·45
	0·43		0·45		0·46		0·48		0·49		0·50		0·51		0·52		0·54		0·56		0·57		0·58	
32	968·93	1·25	967·68	1·26	966·43	1·27	965·16	1·28	963·88	1·30	962·58	1·32	961·26	1·33	959·93	1·36	958·57	1·39	957·18	1·40	955·78	1·44	954·34	1·47
	0·43		0·45		0·47		0·48		0·50		0·51		0·52		0·54		0·55		0·56		0·58		0·59	
33	968·50	1·27	967·23	1·27	965·96	1·28	964·68	1·30	963·38	1·31	962·07	1·33	960·74	1·35	959·39	1·37	958·02	1·40	956·62	1·42	955·20	1·45	953·75	1·48
	0·45		0·45		0·47		0·49		0·50		0·51		0·52		0·54		0·55		0·56		0·58		0·60	
34	968·05	1·27	966·78	1·29	965·49	1·30	964·19	1·31	962·88	1·32	961·56	1·34	960·22	1·37	958·85	1·38	957·47	1·41	956·06	1·44	954·62	1·47	953·15	1·49
	0·45		0·47		0·48		0·49		0·50		0·52		0·54		0·55		0·57		0·58		0·59		0·60	
35	967·60	1·29	966·31	1·30	965·01	1·31	963·70	1·32	962·38	1·34	961·04	1·36	959·68	1·38	958·30	1·40	956·90	1·42	955·48	1·45	954·03	1·48	952·55	1·50
	0·45		0·47		0·48		0·49		0·51		0·53		0·54		0·55		0·57		0·59		0·60		0·61	
36	967·15	1·31	965·84	1·31	964·53	1·32	963·21	1·34	961·87	1·36	960·51	1·37	959·14	1·39	957·75	1·42	956·33	1·44	954·89	1·46	953·43	1·49	951·94	1·51
	0·46		0·47		0·48		0·50		0·52		0·53		0·55		0·56		0·57		0·58		0·60		0·61	
37	966·69	1·32	965·37	1·32	964·05	1·34	962·71	1·36	961·35	1·37	959·98	1·39	958·59	1·40	957·19	1·43	955·76	1·45	954·31	1·48	952·83	1·50	951·38	1·52
	0·47		0·48		0·50		0·51		0·52		0·54		0·55		0·57		0·58		0·59		0·60		0·61	
38	966·22	1·33	964·89	1·34	963·55	1·35	962·20	1·37	960·83	1·39	959·44	1·40	958·04	1·42	956·62	1·44	955·18	1·46	953·72	1·49	952·23	1·51	950·72	1·54
	0·48		0·49		0·51		0·52		0·53		0·54		0·56		0·57		0·58		0·60		0·61		0·62	
39	965·74	1·34	964·40	1·36	963·04	1·36	961·68	1·38	960·30	1·40	958·90	1·42	957·48	1·43	956·05	1·45	954·60	1·48	953·12	1·50	951·62	1·52	950·10	1·55
	0·49		0·50		0·51		0·53		0·54		0·55		0·56		0·58		0·60		0·61		0·62		0·64	
40	965·25	1·35	963·90	1·37	962·53	1·38	961·15	1·39	959·76	1·41	958·35	1·43	956·92	1·45	955·47	1·47	954·00	1·49	952·51	1·51	951·00	1·54	949·46	1·56

FOR MUSTS AND FERMENTING MUSTS, MEASUREMENT OF ALCOHOL BY CHEMICAL PROCEDURE

This method should only be used with beverages of low alcoholic content (less than 6% vol) and with samples where only a small volume is available.

Solutions

(a) Potassium dichromate solution: dissolve 33·600 g of potassium dichromate analytical reagent quality in distilled water and make to one litre at 20 °C. (This solution corresponds to an international alcoholic strength ([1]).)

1 ml of this solution oxidizes 7·8924 mg of alcohol.

(b) Ammonium ferrous sulphate solution: dissolve 135 g of ammonium ferrous sulphate and 20 ml of pure sulphuric acid in water and make to one litre. This solution is equivalent to approximately half its volume of dichromate solution when freshly prepared; it will then slowly oxidize.

Frequently restandardize this solution against the dichromate solution which keeps well in a ground glass stoppered bottle. Carry out the titration in the same way as for the alcohol determination, except that the 10 ml of alcohol dilution is replaced by an equal volume of pure water. This compensates for the small quantities of organic substances which sometimes are present in the sulphuric acid.

(c) Potassium permanganate solution: dissolve 1·088 g of potassium permanganate in water and make up to one litre with water.

(d) Dilute sulphuric acid: gradually add, with stirring, 500 ml of pure sulphuric acid to 500 ml of distilled water. After cooling make up to one litre with distilled water.

(e) Ferrous o-phenanthroline reagent: dissolve 0·695 g of $FeSO_4, 7\,H_2O$ in 100 ml of water and add 1·485 g of o-phenanthroline monohydrate. Heat to assist dissolution. This solution, bright red in colour, keeps very well.

Procedure

1. *Distillation.* Separate the alcohol by distillation, using the equipment described above (page 16). Calculate the volumes of the sample and of the distillate so that the alcoholic strength of the latter should be of the order of 1% vol; it must be less than 1·8% vol.

Assumed alcoholic strength	Volume of liquid ([1]) to be used V (ml)	Volume of water to be added before distillation (ml)	Volume to which the distillate should be made up (ml)
Less than 0·8% vol	200	0	100
0·8 to 1·5% vol	100	20	100
1·5 to 3% vol	50	70	100
3 to 6% vol	25	100	100
6 to 9% vol	20	100	100
9 to 18% vol	20	200	200
18 to 22% vol	20	200	250

([1]) Wine or must.

([1]) The weight of dichromate is 33·791 g/l of solution at 15 °C for the French alcoholic strength at 15°/15° and 33·611 g/l of solution for the German alcoholic strength at 20°/20°.
1 ml of the solution with 33·791 g/l oxidizes 7·93634 mg of alcohol.
1 ml of the solution with 33·611 g/l oxidizes 7·8940 mg of alcohol.
These relationships between dichromate and alcohol are theoretical, and have been calculated according to the specific gravities or densities of pure alcohol (100% vol) indicated in the alcoholic strength tables. They are liable to vary as a function of the atomic weights of the elements which are revised each year. The figures given above have been calculated according to the 1961 table. These variations are of no practical importance, as they are minimal.

Transfer a volume V of liquid, wine or must to be analyzed, which has been made alkaline by the addition of a slight excess of 4N milk of lime and the prescribed volume of water, to the steam-distillation flask described previously. Collect the distillate in a measuring flask of equal volume to that specified in the last column of the above table. Fill up to the mark with distilled water.

One can also dilute the distillate, obtained by one of the routine methods (page 27), by taking 50, 25 or 20 ml of the distillate and making up to 100, 200 or 250 ml.

2. *Oxidation:* pipette 20 ml of standard potassium dichromate solution and 20 ml of 50 % v/v sulphuric acid into a 250 ml wide-necked flask fitted with a ground-glass stopper. Mix with stirring. Pipette 10 ml of distillate into the flask. Stopper the flask, the stopper having been previously moistened with a drop of concentrated sulphuric acid, shake and allow to stand for at least 30 minutes with occasional shaking.

3. *Titration:* rinse the stopper and neck and titrate the excess dichromate with standard ferrous ammonium sulphate from a calibrated burette using four drops of o-phenanthroline as indicator added when the green coloration of the solution turns to green-blue. The end-point is indicated when the colour changes from blue-green to a red-brown.

   As the colour change is frequently slightly overshot, return to the exact end-point using a potassium permanganate solution of 1·083 g/l. Subtract one tenth of the volume of this solution used from the volume of the ferrous sulphate solution, the difference being n.

   Carry out a blank experiment using a similar bottle containing the same quantities of the same reagents except that the 10 ml of distillate have been replaced by 10 ml of pure water. The volume of the ferrous solution used is n′.

Calculations

n′ ml of ferrous solution reduces 20 ml of dichromate solution which oxidizes 200 mm² or 0·2 ml of pure alcohol.

1 ml of ferrous solution has the same reducing power as $\frac{0{\cdot}2}{n'}$ ml of alcohol.

n′ — n ml of ferrous solution have the same reducing power as $0{\cdot}2\,\frac{n' - n}{n'}$ ml of alcohol in the 10 ml of distillate which in turn contains alcohol of $\frac{10}{100}$ of the volume V of liquid sample, if the distillate has been made up to 100 ml.

Hence, $0{\cdot}2\,\frac{n' - n}{n'}$ ml of alcohol are contained in $\frac{V}{10}$ ml of liquid.

The alcoholic strength of this liquid is:

$$\frac{200}{V}\ \frac{(n' - n)}{n'}$$

When the distillate has been made up to 200 or 250 ml this formula becomes:

$$\frac{400\ (n' - n)}{V \quad n'} \text{ or } \frac{500\ (n' - n)}{V \quad n'}$$

Notes:

1. The 10 and 20 ml pipettes and the burette used for the measurement must be certified and accurate to at least one part per 1 000.

2. The measurement of the acetic acid formed permits checking the chromometric determination of the alcohol and the identification of that alcohol. Methanol, in particular, is oxidized to carbon dioxide and water under the conditions where ethanol is converted to acetic acid; the oxidation of the former requires at least 12 hours however, whereas that of ethanol is complete in 30 minutes (1).

   It should be noted that 1 ml of $K_2Cr_2O_7$ of concentration 33·600 g/l oxidizes 3·6596 mg of methanol.

   To measure the acetic acid, steam-distill and collect the distillate. The volume of the distillate should be equal to at least 10 times the volume of the solution in which the chromometric titration has been carried out and to which a small excess of ferrous sulphate has been added.

   1 ml 0·1N NaOH corresponds to 4·607 mg of ethanol.

   1 ml $K_2Cr_2O_7$ of concentration 33·600 g/l thus corresponds to 1·71 ml of 0·1N NaOH.

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(1) Under the conditions specified above, the acetic acid formed resists the chromic mixture indefinitely and the prolonged oxidation thus does not alter the stoichiometric relation between the ethanol and the dichromate.

2.4. USUAL METHODS FOR WINES AND MUSTS

FIRST USUAL METHOD

Measurement by a hydrometer

Apparatus

1. Distillation apparatus: the apparatus is similar to that described for the reference method.

   As for the reference method other types of equipment may be used. In particular steam distillation may be employed for the distillation of the alcohol, which speeds the operation, provided that the apparatus and procedure fulfil the conditions specified for the reference method (see page 16).

2. Hydrometer: the hydrometer used shall comply with the specifications of Council Directive 66/765/EEC of 27 July 1976 relating to alcoholometers and alcohol hydrometers.

   This apparatus shall be calibrated by a government department.

   A calibrated thermometer graduated in 1/10th degrees from 0 to 30 °C certified to 1/20th degree shall be used.

   Carry out the test in an upright cylindrical tube of 36 mm diameter and 320 mm height. The inside diameter of the cylinder must exceed the bulb diameter of the hydrometer by at least 6 mm.

Procedure

1. *Distillation:* free young or sparkling wines of the bulk of their carbon dioxide by shaking 260 ml of the wine in a 500 ml bottle which has previously been siliconized internally.

   Measure out the wine to be analyzed into a 250 ml flask with an internal neck diameter not greater than 12 mm. Transfer to a distillation flask. Rinse the flask four times with 5 ml of water. Add 10 ml of milk of lime (120 g of CaO per litre). The colouring matter of the wine should be changed by the alkalinity. In the case of very acid, sour wines, etc. add the lime until the wine is clearly alkaline to phenolphthalein used externally. Add, if necessary, some inert porous matter and a drop of an aqueous solution, of one part per 100 of water-soluble silicone to prevent frothing. Collect the distillate in the same flask as that used for measuring out the wine, and into which has been placed 10 ml of distilled water.

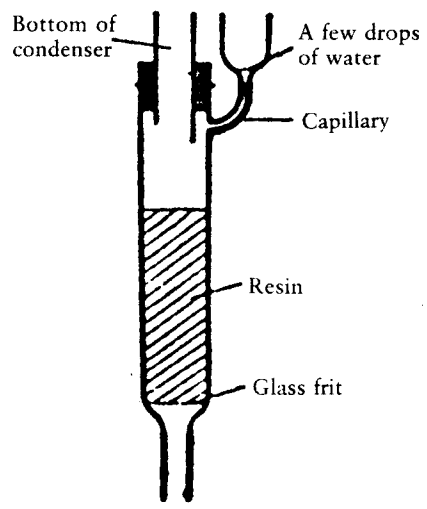
   Distill at least 200 ml. Shake the flask and make up to the mark at the same temperature as initially (to within 5 °C).

   Note:

   Neutralization may be avoided by the use of 10 g of a strongly basic ion exchange resin (50 to 100 mesh) suitably washed with distilled water and supported on a glass frit to retain fine particles.

   This resin retains acetic acid, free and combined sulphur dioxide and carbon dioxide.

   10 g of resin is effective for 30 distillations when used for normal wines. Carry out regeneration using a 5 % sodium hydroxide solution and six flushings with distilled water (see drawing).



2. *Measurement of specific gravity of the distillate:* pour the distillate into a level glass cylinder of 36 mm internal diameter and 320 mm height. Keep the cylinder vertical. Place a thermometer and hydrometer in the cylinder. Read the thermometer one minute after shaking, so ensuring equilibrium temperature of cylinder, thermometer, hydrometer and distillate has been reached. Withdraw the thermometer and read the apparent degree of alcoholic strength after one minute. Take at least three readings using a magnifying glass. Correct the apparent degree measured at t °C for the effect of the temperature with the following table.

   When making the observations the temperature should not differ from ambient by more than 5 °C. If a greater difference is observed it may be avoided by using a vacuum flask.

INTERNATIONAL ALCOHOLIC STRENGTH BY VOLUME AT 20 °C

Table of corrections to be applied to the apparent degree of alcoholic strength to correct for the effect of the temperature

Add to or deduct from the apparent degree of alcoholic strength at t °C (glass hydrometer) the correction indicated below

Temperature (°C)		Apparent degree of alcoholic strength at t ° C																
		0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Add	0	0·76	0·77	0·82	0·87	0·95	1·04	1·16	1·31	1·49	1·70	1·95	2·26	2·62	3·03	3·49	4·02	4·56
	1	0·81	0·83	0·87	0·92	1·00	1·09	1·20	1·35	1·52	1·73	1·97	2·26	2·59	2·97	3·40	3·87	4·36
	2	0·85	0·87	0·92	0·97	1·04	1·13	1·24	1·38	1·54	1·74	1·97	2·24	2·54	2·89	3·29	3·72	4·17
	3	0·88	0·91	0·95	1·00	1·07	1·15	1·26	1·39	1·55	1·73	1·95	2·20	2·48	2·80	3·16	3·55	3·95
	4	0·90	0·92	0·97	1·02	1·09	1·17	1·27	1·40	1·55	1·72	1·92	2·15	2·41	2·71	3·03	3·38	3·75
	5	0·91	0·93	0·98	1·03	1·10	1·17	1·27	1·39	1·53	1·69	1·87	2·08	2·33	2·60	2·89	3·21	3·54
	6	0·92	0·94	0·98	1·02	1·09	1·16	1·25	1·37	1·50	1·65	1·82	2·01	2·23	2·47	2·74	3·02	3·32
	7	0·91	0·93	0·97	1·01	1·07	1·14	1·23	1·33	1·45	1·59	1·75	1·92	2·12	2·34	2·58	2·83	3·10
	8	0·89	0·91	0·94	0·98	1·04	1·11	1·19	1·28	1·39	1·52	1·66	1·82	2·00	2·20	2·42	2·65	2·88
	9	0·86	0·88	0·91	0·95	1·01	1·07	1·14	1·23	1·33	1·44	1·57	1·71	1·87	2·05	2·24	2·44	2·65
	10	0·82	0·84	0·87	0·91	0·96	1·01	1·08	1·16	1·25	1·35	1·47	1·60	1·74	1·89	2·06	2·24	2·43
	11	0·78	0·79	0·82	0·86	0·90	0·95	1·01	1·08	1·16	1·25	1·36	1·47	1·60	1·73	1·88	2·03	2·20
	12	0·72	0·74	0·76	0·79	0·83	0·88	0·93	0·99	1·07	1·15	1·24	1·34	1·44	1·56	1·69	1·82	1·96
	13	0·66	0·67	0·69	0·72	0·76	0·80	0·84	0·90	0·96	1·03	1·11	1·19	1·28	1·38	1·49	1·61	1·73
	14	0·59	0·60	0·62	0·64	0·67	0·71	0·74	0·79	0·85	0·91	0·97	1·04	1·12	1·20	1·29	1·39	1·49
	15	0·51	0·52	0·53	0·55	0·58	0·61	0·64	0·68	0·73	0·77	0·83	0·89	0·95	1·02	1·09	1·16	1·24
	16	0·42	0·43	0·44	0·46	0·48	0·50	0·53	0·56	0·60	0·63	0·67	0·72	0·77	0·82	0·88	0·94	1·00
	17	0·33	0·33	0·34	0·35	0·37	0·39	0·41	0·43	0·46	0·48	0·51	0·55	0·59	0·62	0·67	0·71	0·75
	18	0·23	0·23	0·23	0·24	0·25	0·26	0·27	0·29	0·31	0·33	0·35	0·37	0·40	0·42	0·45	0·48	0·51
	19	0·12	0·12	0·12	0·12	0·13	0·13	0·14	0·15	0·16	0·17	0·18	0·19	0·20	0·21	0·23	0·24	0·25
Deduct	21		0·13	0·13	0·13	0·14	0·14	0·15	0·16	0·17	0·18	0·19	0·19	0·20	0·22	0·23	0·25	0·26
	22		0·26	0·27	0·28	0·29	0·30	0·31	0·32	0·34	0·36	0·37	0·39	0·41	0·44	0·47	0·49	0·52
	23		0·40	0·41	0·42	0·44	0·45	0·47	0·49	0·51	0·54	0·57	0·60	0·63	0·66	0·70	0·74	0·78
	24		0·55	0·56	0·58	0·60	0·62	0·64	0·67	0·70	0·73	0·77	0·81	0·85	0·89	0·94	0·99	1·04
	25		0·69	0·71	0·73	0·76	0·79	0·82	0·85	0·89	0·93	0·97	1·02	1·07	1·13	1·19	1·25	1·31
	26		0·85	0·87	0·90	0·93	0·96	1·00	1·04	1·08	1·13	1·18	1·24	1·30	1·36	1·43	1·50	1·57
	27			1·03	1·07	1·11	1·15	1·19	1·23	1·28	1·34	1·40	1·46	1·53	1·60	1·68	1·76	1·84
	28			1·21	1·25	1·29	1·33	1·38	1·43	1·49	1·55	1·62	1·69	1·77	1·85	1·93	2·02	2·11
	29			1·39	1·43	1·47	1·52	1·58	1·63	1·70	1·76	1·84	1·92	2·01	2·10	2·19	2·29	2·39
	30			1·57	1·61	1·66	1·72	1·78	1·84	1·91	1·98	2·07	2·15	2·25	2·35	2·45	2·56	2·67
	31			1·75	1·80	1·86	1·92	1·98	2·05	2·13	2·21	2·30	2·39	2·49	2·60	2·71	2·83	2·94
	32			1·94	2·00	2·06	2·13	2·20	2·27	2·35	2·44	2·53	2·63	2·74	2·86	2·97	3·09	3·22
	33				2·20	2·27	2·34	2·42	2·50	2·58	2·67	2·77	2·88	2·99	3·12	3·24	3·37	3·51
	34				2·41	2·48	2·56	2·64	2·72	2·81	2·91	3·02	3·13	3·25	3·38	3·51	3·65	3·79
	35				2·62	2·70	2·78	2·86	2·95	3·05	3·16	3·27	3·39	3·51	3·64	3·78	3·93	4·08
	36				2·83	2·91	3·00	3·09	3·19	3·29	3·41	3·53	3·65	3·78	3·91	4·05	4·21	4·37
	37					3·13	3·23	3·33	3·43	3·54	3·65	3·78	3·91	4·04	4·18	4·33	4·49	4·65
	38					3·36	3·47	3·57	3·68	3·79	3·91	4·03	4·17	4·31	4·46	4·61	4·77	4·94
	39					3·59	3·70	3·81	3·93	4·05	4·17	4·30	4·44	4·58	4·74	4·90	5·06	5·23
	40						3·82	3·94	4·06	4·18	4·31	4·44	4·57	4·71	4·86	5·02	5·19	5·36	5·53

Temperature (°C)		Apparent degree of alcoholic strength at t °C																
		14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30
Add	0	3·49	4·02	4·56	5·11	5·65	6·16	6·63	7·05	7·39	7·67	7·91	8·07	8·20	8·30	8·36	8·39	8·40
	1	3·40	3·87	4·36	4·86	5·35	5·82	6·26	6·64	6·96	7·23	7·45	7·62	7·75	7·85	7·91	7·95	7·96
	2	3·29	3·72	4·17	4·61	5·05	5·49	5·89	6·25	6·55	6·81	7·02	7·18	7·31	7·40	7·47	7·51	7·53
	3	3·16	3·55	3·95	4·36	4·77	5·17	5·53	5·85	6·14	6·39	6·59	6·74	6·86	6·97	7·03	7·07	7·09
	4	3·03	3·38	3·75	4·11	4·48	4·84	5·17	5·48	5·74	5·97	6·16	6·31	6·43	6·53	6·59	6·63	6·66
	5	2·89	3·21	3·54	3·86	4·20	4·52	4·83	5·11	5·35	5·56	5·74	5·89	6·00	6·10	6·16	6·20	6·23
	6	2·74	3·02	3·32	3·61	3·91	4·21	4·49	4·74	4·96	5·16	5·33	5·47	5·58	5·67	5·73	5·77	5·80
	7	2·58	2·83	3·10	3·36	3·63	3·90	4·15	4·38	4·58	4·77	4·92	5·05	5·15	5·24	5·30	5·34	5·37
	8	2·42	2·65	2·88	3·11	3·35	3·59	3·81	4·02	4·21	4·38	4·52	4·64	4·74	4·81	4·87	4·92	4·95
	9	2·24	2·44	2·65	2·86	3·07	3·28	3·48	3·67	3·84	3·99	4·12	4·23	4·32	4·39	4·45	4·50	4·53
	10	2·06	2·24	2·43	2·61	2·80	2·98	3·16	3·33	3·48	3·61	3·73	3·83	3·91	3·98	4·03	4·08	4·11
	11	1·88	2·03	2·20	2·36	2·52	2·68	2·83	2·98	3·12	3·24	3·34	3·43	3·50	3·57	3·62	3·66	3·69
	12	1·69	1·82	1·96	2·10	2·24	2·38	2·51	2·64	2·76	2·87	2·96	3·04	3·10	3·16	3·21	3·25	3·27
	13	1·49	1·61	1·73	1·84	1·96	2·08	2·20	2·31	2·41	2·50	2·58	2·65	2·71	2·76	2·80	2·83	2·85
	14	1·29	1·39	1·49	1·58	1·68	1·78	1·88	1·97	2·06	2·13	2·20	2·26	2·31	2·36	2·39	2·42	2·44
	15	1·09	1·16	1·24	1·32	1·40	1·48	1·56	1·64	1·71	1·77	1·83	1·88	1·92	1·96	1·98	2·01	2·03
	16	0·88	0·94	1·00	1·06	1·12	1·19	1·25	1·31	1·36	1·41	1·46	1·50	1·53	1·56	1·58	1·60	1·62
	17	0·67	0·71	0·75	0·80	0·84	0·89	0·94	0·98	1·02	1·05	1·09	1·12	1·14	1·17	1·18	1·20	1·21
	18	0·45	0·48	0·51	0·53	0·56	0·59	0·62	0·65	0·68	0·70	0·72	0·74	0·76	0·78	0·79	0·80	0·81
	19	0·23	0·24	0·25	0·27	0·28	0·30	0·31	0·33	0·34	0·35	0·36	0·37	0·38	0·39	0·40	0·40	0·41
Deduct	21	0·23	0·25	0·26	0·28	0·29	0·30	0·31	0·33	0·34	0·35	0·35	0·37	0·38	0·38	0·39	0·39	0·40
	22	0·47	0·49	0·52	0·55	0·57	0·60	0·62	0·65	0·67	0·70	0·72	0·74	0·75	0·76	0·78	0·79	0·80
	23	0·70	0·74	0·78	0·82	0·86	0·90	0·93	0·97	1·01	1·04	1·07	1·10	1·12	1·15	1·17	1·18	1·19
	24	0·94	0·99	1·04	1·10	1·15	1·20	1·25	1·29	1·34	1·39	1·43	1·46	1·50	1·53	1·55	1·57	1·59
	25	1·19	1·25	1·31	1·37	1·43	1·49	1·56	1·62	1·68	1·73	1·78	1·83	1·87	1·90	1·94	1·97	1·99
	26	1·43	1·50	1·57	1·65	1·73	1·80	1·87	1·94	2·01	2·07	2·13	2·19	2·24	2·28	2·32	2·35	2·38
	27	1·68	1·76	1·84	1·93	2·01	2·10	2·18	2·26	2·34	2·41	2·48	2·55	2·61	2·66	2·70	2·74	2·77
	28	1·93	2·02	2·11	2·21	2·31	2·40	2·49	2·58	2·67	2·76	2·83	2·90	2·98	3·03	3·08	3·13	3·17
	29	2·19	2·29	2·39	2·50	2·60	2·70	2·81	2·91	3·00	3·09	3·18	3·26	3·34	3·40	3·46	3·51	3·55
	30	2·45	2·56	2·67	2·78	2·90	3·01	3·12	3·23	3·34	3·44	3·53	3·62	3·70	3·77	3·84	3·90	3·95
	31	2·71	2·83	2·94	3·07	3·19	3·31	3·43	3·55	3·67	3·78	3·88	3·98	4·07	4·15	4·22	4·28	4·33
	32	2·97	3·09	3·22	3·36	3·49	3·62	3·74	3·87	4·00	4·11	4·22	4·33	4·43	4·51	4·59	4·66	4·72
	33	3·24	3·37	3·51	3·65	3·79	3·92	4·06	4·20	4·33	4·45	4·57	4·68	4·79	4·88	4·97	5·04	5·10
	34	3·51	3·65	3·79	3·94	4·09	4·23	4·37	4·52	4·66	4·79	4·91	5·03	5·15	5·25	5·34	5·42	5·49
	35	3·78	3·93	4·08	4·23	4·38	4·53	4·69	4·84	4·98	5·12	5·26	5·38	5·50	5·61	5·71	5·80	5·87
	36	4·05	4·21	4·37	4·52	4·68	4·84	5·00	5·16	5·31	5·46	5·60	5·73	5·86	5·97	6·08	6·17	6·25
	37	4·33	4·49	4·65	4·82	4·98	5·15	5·31	5·48	5·64	5·80	5·95	6·09	6·22	6·33	6·44	6·54	6·63
	38	4·61	4·77	4·94	5·12	5·29	5·46	5·63	5·80	5·97	6·13	6·29	6·43	6·57	6·69	6·81	6·92	7·01
	39	4·90	5·06	5·23	5·41	5·59	5·77	5·94	6·12	6·30	6·47	6·63	6·78	6·93	7·06	7·18	7·29	7·39
	40	5·19	5·36	5·53	5·71	5·90	6·08	6·26	6·44	6·62	6·80	6·97	7·13	7·28	7·41	7·54	7·66	7·76

SECOND USUAL METHOD

Densimetry by the hydrostatic balance

The distillation apparatus and its method of operation are the same as for the first usual method.

The apparatus, the calibration of the float and the procedure have been described in connection with the measurement of the density, but the measurement of density of the distillate must be more accurate than that of wine. It should be obtained at least to the nearest 5×10^{-5}. In order to achieve this result one should use a float of at least 50 ml and take all necessary precautions to avoid any evaporation of the alcohol and to know the temperature, t °C, of the distillate to at least the nearest 0·1 °C.

The formulae for the calculation have also been mentioned already.

Knowing p'_t at t °C, one determines the alcoholometric titre by means of one of the density tables, which one selects according to the nature of the glass used in the float, following the same interpolation procedure as for the reference method.

2.5. SUPPLEMENT TO THE REFERENCE METHOD FOR THE MEASUREMENT OF THE DENSITY OF THE DISTILLATE

MEASUREMENT BY PYCNOMETER ON SINGLE-PAN BALANCE

Principle

The principle of the method of determining the density of the distillate is given in 'Density and specific gravity'.

Numerical example

Determine the constants of the pycnometer and calculate as described in 'Density and specific gravity'.

Calculations

1. Weighing of the pycnometer filled with distillate:

Weight of tare bottle: $T_1 = 171{\cdot}9178$

Pycnometer filled with distillate at 20·50 °C: $P_2 = 167{\cdot}8438$

Variation in the buoyancy of air: $dT = 171{\cdot}9178 - 171{\cdot}9160 = + 0{\cdot}0018$

Mass of the distillate at 20·50 °C: $L_t = 167{\cdot}8438 - (67{\cdot}6695 + 0{\cdot}0018) = 100{\cdot}1725$

Apparent density of the distillate: $\varrho\ 20{\cdot}50\ °C = \dfrac{100{\cdot}1725}{100{\cdot}8194} = 0{\cdot}983825$

2. Calculation of alcoholic strength:

Refer to the table of apparent densities of water-alcohol mixtures at different temperatures, as indicated above.

On the line 20 °C of the table of apparent densities, the smallest value greater than the observed density of 0·983825 is 0·98471 in column 10 % vol

The density at 20 °C is:

$(98382{\cdot}5 + (0{\cdot}50 \cdot 24))\ 10^{-5} = 0{\cdot}983945$

$0{\cdot}98471 - 0{\cdot}983945 = 0{\cdot}000765$

The decimal portion of the % vol of alcoholic strength is 76·5/119 = 0·64

The alcoholic strength is 10·64 % vol

3. TOTAL DRY EXTRACT

Total dry matter

3.1. DEFINITION

The total dry extract or the total dry matter includes all matter which is non-volatile under specified physical conditions. These physical conditions must be such that the matter forming the extract is not materially altered while the test is being carried out.

The sugar-free extract is the difference between total dry extract and the total sugars.

The reduced extract is the difference between the total dry extract and the total sugars in excess of 1 g/l, potassium sulphate in excess of 1 g/l, any mannitol present and any other chemical substances which may have been added to the wine.

The residual extract is the sugar-free extract less the fixed acidity expressed as tartaric acid.

The extract is expressed in grams per litre and it should be determined to within the nearest 0·5 g.

3.2. PRINCIPLE OF THE METHOD

Single method: measurement by a densimeter

The total dry extract is calculated indirectly from the specific gravity of the alcohol-free residue, or wine from which the alcohol has been removed and which has been made up to the initial volume with water.

This dry extract is expressed in terms of the quantity of sucrose which, when dissolved in water and made up to a volume of one litre, gives a solution of the same specific gravity as the alcohol-free residue. This quantity is shown in Table I.

Method of calculation

The specific gravity 20/20 d_r of the 'alcohol-free residue' is calculated using Tabarie's formula:

$$d_r = d_v - d_a + 1{\cdot}000$$

where:

d_v = specific gravity of the wine at 20 °C (corrected for volatile acidity) (1);

d_a = specific gravity at 20 °C of a water-alcohol mixture of the same alcohol strength as the wine.

d_r may also be calculated from the densities at 20 °C, ϱ_v of wine and ϱ_a of the water-alcohol mixture of the same strength by the formula:

$$d_r = 1{\cdot}0018\ (\varrho_v - \varrho_a) + 1{\cdot}000$$

where the coefficient 1·0018 approximates to one when ϱ_v is below 1·05.

Table I should be used for calculating the dry extract from the specific gravity d_r.

For musts, the total dry extract should be calculated from the specific gravity d_{20}^{20} using the same table.

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(1) Before carrying out this calculation, the specific gravity (or density) of the wine measured as specified above should be corrected for the effect of the volatile acidity using the formula:

$d_v = d_{20}^{20} - 0{\cdot}0000086 \cdot a$ or $\varrho_v = \varrho_{20} - 0{\cdot}0000086 \cdot a$

where a is the volatile acidity expressed in milliequivalents per litre.

TABLE I

for the calculation of the content of extract

(g/l)

Specific gravity to two decimal places	Third decimal place of the specific gravity									
	0	1	2	3	4	5	6	7	8	9
	Grams of extract per litre									
1·00	0	2·6	5·1	7·7	10·3	12·9	15·4	18·0	20·6	23·2
1·01	25·8	28·4	31·0	33·6	36·2	38·8	41·3	43·9	46·5	49·1
1·02	51·7	54·3	56·9	59·5	62·1	64·7	67·3	69·9	72·5	75·1
1·03	77·7	80·3	82·9	85·5	88·1	90·7	93·3	95·9	98·5	101·1
1·04	103·7	106·3	109·0	111·6	114·2	116·8	119·4	122·0	124·6	127·2
1·05	129·8	132·4	135·0	137·6	140·3	142·9	145·5	148·1	150·7	153·3
1·06	155·9	158·6	161·2	163·8	166·4	169·0	171·6	174·3	176·9	179·5
1·07	182·1	184·8	187·4	190·0	192·6	195·2	197·8	200·5	203·1	205·8
1·08	208·4	211·0	213·6	216·2	218·9	221·5	224·1	226·8	229·4	232·0
1·09	234·7	237·3	239·9	242·5	245·2	247·8	250·4	253·1	255·7	258·4
1·10	261·0	263·6	266·3	268·9	271·5	274·2	276·8	279·5	282·1	284·8
1·11	287·4	290·0	292·7	295·3	298·0	300·6	303·3	305·9	308·6	311·2
1·12	313·9	316·5	319·2	321·8	324·5	327·1	329·8	332·4	335·1	337·8
1·13	340·4	343·0	345·7	348·3	351·0	353·7	356·3	359·0	361·6	364·3
1·14	366·9	369·6	372·3	375·0	377·6	380·3	382·9	385·6	388·3	390·9
1·15	393·6	396·2	398·9	401·6	404·3	406·9	409·6	412·3	415·0	417·6
1·16	420·3	423·0	425·7	428·3	431·0	433·7	436·4	439·0	441·7	444·4
1·17	447·1	449·8	452·4	455·2	457·8	460·5	463·2	465·9	468·6	471·3
1·18	473·9	476·6	479·3	482·0	484·7	487·4	490·1	492·8	495·5	498·2
1·19	500·9	503·5	506·2	508·9	511·6	514·3	517·0	519·7	522·4	525·1
1·20	527·8	530·5	533·3	536·0	538·7	541·4	544·1	546·8	549·6	552·3
1·21	555·0	557·7	560·4	563·1	565·8	568·5	571·2	573·9	576·6	579·3
1·22	582·0	584·8	587·5	590·2	593·0	595·7	598·4	601·1	603·9	606·6
1·23	609·3	612·1	614·8	617·5	620·3	623·0	625·7	628·4	631·2	633·9
1·24	636·6	639·4	642·1	644·9	647·6	650·3	653·1	655·8	658·6	661·3
1·25	664·0	666·8	669·5	672·3	675·0	677·7	680·5	683·2	686·0	688·7
1·26	691·4	694·2	697·0	699·8	702·5	705·3	708·1	710·8	713·6	716·4
1·27	719·1	721·9	724·7	727·4	730·2	732·9	735·7	738·5	741·2	744·0
1·28	746·7	749·5	752·3	755·1	757·8	760·6	763·4	766·1	768·9	771·7
1·29	774·4	777·2	780·0	782·8	785·6	788·3	791·1	793·9	796·7	799·5
1·30	802·3	805·0	807·8	810·6	813·4	816·2	819·0	821·8	824·6	827·4
1·31	830·2	833·1	835·9	838·7	841·5	844·3	847·1	849·9	852·7	855·5
1·32	858·3	861·2	864·0	866·8	869·6	872·4	875·3	878·1	880·9	883·7
1·33	886·5	889·4	892·2	895·0	897·9	900·7	903·5	906·4	909·2	912·0
1·34	914·8	917·7	920·5	923·3	926·2	929·0	931·8	934·7	937·5	940·3
1·35	943·1	—	—	—	—	—	—	—	—	—

Interpolation table

Fourth decimal place of the specific gravity	1	2	3	4	5	6	7	8	9
Grams of extract per litre	0·26	0·52	0·78	1·04	1·30	1·56	1·82	2·08	2·34

4. REDUCING SUGARS

4.1. DEFINITION

The reducing sugars, comprising all of the sugars exhibiting ketonic and aldehydic functions, are determined using their reducing action on an alkaline copper salt solution.

Their determination requires two successive operations: clarification and titration.

4.2. PRINCIPLE OF THE SINGLE METHODS

(a) *Clarification*

The clarification is carried out:

— for red wines: with basic lead acetate,

— for other products: with zinc ferrocyanide.

(b) *Measurement*

After the clarified wine has reacted with a specified quantity of an alkaline copper salt solution, the excess copper ions are determined iodimetrically.

4.3. CLARIFICATION

Before any measurement it is necessary to remove from the wine reducing agents other than sugars by the following methods.

(a) **For red wines:**

— clarification by basic lead acetate.

Solutions

Basic lead acetate solution:

neutral lead acetate	300 g,
lead oxide	100 g,
distilled water	700 g.

Mix and allow to stand for several days, stirring from time to time. Filter. The solution obtained should have a specific gravity of 1·32.

Procedure

Add (n — 0·5) ml of normal sodium hydroxide solution (n being the volume of 0·1N solution used for measuring the total acidity of 5 ml of wine) to 50 ml of wine in a 100 ml volumetric flask. Then add 2·5 ml of 0·1N acetic acid solution and, with stirring, 2·5 ml of basic lead acetate solution. Leave for 15 minutes, add 5 ml of a 7·6% disodium hydrogen phosphate solution; stir the mixture and then add distilled water without making up to the mark. Allow to stand for 15 minutes and make the contents of the flask up to the mark. Filter.

1 ml of filtrate corresponds to 0·5 ml of wine.

(b) **For the other products:**

— clarification by zinc ferrocyanide.

Solutions

Potassium ferrocyanide solution I:

potassium ferrocyanide:	150 g,
water to	1 000 ml.

Zinc sulphate solution II:

zinc sulphate	300 g,
water to	1 000 ml.

Procedure

Add a volume of wine (or of must or mistelle) into a 100 ml volumetric flask. The volume required is shown below.

1. *Musts and mistelles:* prepare a 10 % solution of the liquid to be analyzed and take 10 ml of the diluted sample.

   1 ml of filtrate corresponds to 0·01 ml of must or mistelle.

2. *Sweet wines, whether or not fortified, having a density between 1·005 and 1·038:* prepare a 20 % dilution of the liquid to be analyzed and take 20 ml of the diluted sample.

   1 ml of filtrate corresponds to 0·04 ml of sweet wine.

3. *Semi-sweet wines having a density between 0·997 and 1·006:* take 20 ml of the non-diluted wine.

   1 ml of filtrate corresponds to 0·20 ml of semi-sweet wine.

4. *Dry wines:* take a sample of 50 ml of the non-diluted wine. Add 5 ml of solution I, potassium ferrocyanide and 5 ml of solution II, zinc sulphate, to the sample. Mix and make up to the mark. Allow to stand for 10 minutes and filter.

   1 ml of filtrate corresponds to 0·50 ml of dry wine.

4.4. DETERMINATION OF SUGARS

Titration of a standard solution of invert sugar

Every chemist must check his procedure using a standard solution of invert sugar. Prepare this solution according to the following method:

Invert sugar solution containing 10 g per litre.

Place the following in a one litre flask:

sucrose pure and dry	9·50 g,
water approx.	100 ml,
hydrochloric acid pure (d 1·18 to 1·19)	5 ml.

Leave for seven days at 10 to 15 °C, or three days at 20 to 25 °C, after which the sugar has inverted; then make up the solution to 1 000 ml at 20 °C. This acid solution keeps well for one month. Shortly before use, neutralize the bulk of the free acid with sodium hydroxide solution (the prepared solution is approximately 0·06N acid).

Note:

The standard invert sugar solution may be prepared more quickly as follows:

heat the sucrose solution, acidified as above, in a 200 ml flask on a waterbath maintained at 60 °C to a temperature of 50 °C; maintain the flask and contents at 50 °C for 15 minutes; allow to cool in air and by immersion in cold water; transfer to a one litre volumetric flask and make up to volume at 20 °C.

To test the chosen procedure, determine the invert sugar at several dilutions covering all the concentrations over which it is to be applied.

DETERMINATION OF EXCESS OF COPPER IONS BY IODOMETRY

The quantity of copper precipitated by the action of the clarified solution on the alkaline copper salt solution is determined by iodometry.

Solutions

Alkaline copper salt solution:

copper sulphate analytical reagent grade $CuSO_4 \cdot 5H_2O$	25 g,
citric acid	50 g,
sodium carbonate crystalline, $10H_2O$	388 g,
water to	1 000 ml.

Dissolve the copper sulphate in 100 ml of water, the citric acid in 200 ml of water and the sodium carbonate in 250 ml of hot water. Mix the citric acid and the sodium carbonate solutions. Add the copper sulphate solution and make up to one litre.

Potassium iodide solution, 30 % :

potassium iodide	30 g,
water to	100 ml.

Store in a brown bottle.

Sulphuric acid, 25 % :

sulphuric acid pure	25 ml,
distilled water to	100 ml.

Pour the acid into the water, allow to cool and make up to 100 ml with distilled water.

Starch solution, 5 g/l, containing 200 g of sodium chloride per litre as preservative. Boil this solution for 10 minutes during preparation.

Sodium thiosulphate 0·1N.

Procedure

Mix 25 ml of clarified solution and 25 ml of alkaline copper salt solution in a 300 ml conical flask. This volume of sugar solution must not contain more than 60 mg of invert sugar.

Add a few grains of pumice stone and bring the mixture to boiling within two minutes. Fit a reflux condenser to the flask and boil for exactly 10 minutes.

Cool the flask immediately in cold running water. Add, when cool, 10 ml of 30 % potassium iodide solution, 25 ml of 25 % sulphuric acid and 2 ml of starch solution.

Titrate the iodine with the 0·1N sodium thiosulphate solution, the titration being n.

Carry out a blank titration in which the 25 ml of sugar solution is substituted by 25 ml of distilled water. The volume of thiosulphate used is n′.

Calculations

The quantity of sugar expressed as invert sugar contained in the test sample is given in the following table as a function of the number (n′ — n) ml of thiosulphate used.

The sugar content of the wine is expressed in grams of invert sugar per litre, account being taken of the dilutions made in the course of clarification and of the volume of the test sample.

Relation between the volume of 0·1N sodium thiosulphate solution (n′ — n) in millilitres and the quantity of reducing sugar in milligrams

$Na_2S_2O_3$ ml 0·1N	Reducing sugars mg	Difference	$Na_2S_2O_3$ ml 0·1N	Reducing sugars mg	Difference
1	2·4	2·4	13	33·0	2·7
2	4·8	2·4	14	35·7	2·8
3	7·2	2·5	15	38·5	2·8
4	9·7	2·5	16	41·3	2·9
5	12·2	2·5	17	44·2	2·9
6	14·7	2·6	18	47·1	2·9
7	17·2	2·6	19	50·0	3·0
8	19·8	2·6	20	53·0	3·0
9	22·4	2·6	21	56·0	3·1
10	25·0	2·6	22	59·1	3·1
11	27·6	2·7	23	62·2	
12	30·3	2·7			

5. SUCROSE

5.2. PRINCIPLE OF METHODS

(a) **Reference method**

(aa) For qualitative testing:

thin layer chromatography: the sucrose is identified in the musts and in the wines after separation of glucose and fructose by thin layer chromatography on a plate coated with silica G powder buffered with sodium acetate. The developing agent (thiobarbituric acid and trichloroacetic acid) is incorporated in the solvent (ethyl acetate, iso-propanol and water). When sucrose is heated in acid medium, hydroxymethylfurfural is produced which on reacting with thiobarbituric acid gives a yellow-orange coloration.

(bb) For measurement after inversion:

the sucrose is determined by comparison of the reducing power before and after inversion with hydrochloric acid of the solution, obtained after clarification of the wine. The clarified solution is prepared according to the procedures described under 'Reducing sugars'.

The measurement of the reducing sugars before and after inversion is carried out by the method described under 'Reducing sugars'.

(b) **Usual method**

(aa) For qualitative testing:

colorimetric method: the wine is clarified using lead acetate, magnesia and potassium permanganate at pH 8 to 9. The solution obtained is reacted at 100 °C with diphenylamine in a hydrochloric and acetic acid medium. The condensation product obtained in the presence of sucrose is extracted into chloroform which forms a blue colour.

(bb) For measurement after inversion see under (a) (bb) above.

5.3. QUALITATIVE TESTING

REFERENCE METHOD

Thin-layer chromatography method

Apparatus:

— apparatus required for thin-layer chromatography:
  — layer spreader,
  — plate holder,
  — chromatography tank,
  — micrometric syringe or micro-pipette,
  — oven with air circulation.

Solution and reagents:

— silica G powder,

— sodium acetate in 0·02 M solution (1),

— solvent with incorporated developer.

The following mixture is prepared:

— ethyl acetate 65 parts,

— isopropanol 30 parts,

— water five parts.

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(1) Preparation of plates: mix 30 g of silica G powder and 60 ml of 0·02 M sodium acetate to obtain a homogeneous suspension. Spread onto plates to give a thickness of 0·30 mm.

The following reagents are freshly prepared:

— 0·3 % of 2-thiobarbituric acid,

— 5·0 % of crystalline trichloroacetic acid.

Standard solutions:

— sucrose solution 0·05 % w/v,

— sucrose and reducing sugar solutions.

The following solutions are prepared:

— 'A': sucrose solution 0·5 %,

— 'B': solution containing 5 % of glucose and 5 % of fructose.

Mix 1 ml of solution A with 1, 2, 3, 4 and 5 ml respectively of solution B in 10 ml volumetric flasks. Make up the solutions to 10 ml with distilled water. A series of reference solutions containing rising percentages of reducing sugars is thus obtained.

Preparation of sample

When the wine is strongly coloured, treat it with decolorizing charcoal before inoculating the plate.

The chromatogram run

Apply the liquids to be examined to a starting line situated 2·5 cm from the edge of the plate to be dipped into the solvent, at 3 cm from the lateral edge (sides) of the plate and 3 cm from one another.

Deposit between 1 to 5 μl of sample depending on the content of reducing sugars. Each spot should not contain more than 0·25 mg of reducing sugars. Additionally, apply 5 μl of the standard solutions to the plate. In order to obtain spots of small diameter it is recommended that solutions are applied repeatedly in small amounts, drying after each application with a weak jet of cold air.

The silica layer should be removed over a width of 0·5 cm on the lateral edges of the plate before placing the plate into the chromatography tank containing the solvent. Ensure that the atmosphere in the tank is saturated with solvent.

Allow the solvent to run more than 16 cm from the starting line.

Dry the plate in a current of air, after withdrawing from the tank and place in the oven for 15 minutes at 105 °C parallel with the current of air.

In the presence of sucrose, a yellow-orange spot appears, the Rf of which is identical with that of sucrose in the standard solutions. Glucose and fructose give a similarly coloured spot, the Rf of which is greater than that of sucrose.

5.4. USUAL METHOD

Colorimetric method

Solutions:

— neutral lead acetate, crystalline,

— heavy magnesium oxide, powder,

— potassium permanganate solution, two per 1 000,

— chloroform,

— diphenylamine reagent. Add 10 ml of diphenylamine 10 % in absolute ethanol to 20 ml of glacial acetic acid and 70 ml of hydrochloric acid, analytical reagent grade (d_4^{20} : 1·18 to 1·19).

The purity of the reagent should be checked; place 2 ml of diphenylamine reagent, to which has been previously added 2 ml of distilled water, on the boiling waterbath for five minutes. Cool the solution rapidly by immersion in cold water and extract with 1 ml of chloroform. No blue coloration should appear.

Procedure

Dissolve 100 to 200 mg of magnesium oxide and approximately 200 mg of neutral lead acetate in 10 ml of water and place on the waterbath, maintained at 90 to 95 °C, for three to five minutes. Add 2 ml of wine or must, which should be clear and have a total sugar content of less than 1 %.

If necessary dilute the sample to bring its sugar content within this limit.

Check that sufficient lead acetate has been added but that there is no excess, which would reduce the sensitivity of the reaction. Add dropwise a concentrated solution of neutral lead acetate to the supernatant clear liquid until no more precipitate is formed. Stir the mixture and maintain on the waterbath until the supernatant liquid becomes clear. Then add a saturated solution of sodium sulphate to precipitate the excess lead.

Add 0·5 ml of potassium permanganate solution and maintain the mixture on the waterbath (90 to 95 °C) for 10 minutes.

Cool rapidly by immersion in cold water and filter.

A yellow coloration may develop, but this does not interfere with the reaction.

Place 2 ml of the filtrate in a test tube and add 2 ml of diphenylamine reagent; place on a boiling waterbath for exactly five minutes. Cool by immersion in cold water, add 1 ml of chloroform and immediately extract the colour.

Dry wines, to which no sucrose has been added, give, at most, a very slight grey-blue colour; a clear blue colour develops in the presence of sucrose.

Sweet wines, musts and concentrated musts produce, in the absence of sucrose, a golden yellow colour and in the presence of sucrose, a clear green colour.

The reaction is sensitive to 0·2 mg of sucrose in the test sample.

5.5. MEASUREMENT AFTER INVERSION

SINGLE REFERENCE AND USUAL METHOD

Procedure

Place, in two identical flasks A and B, the same test sample of clarified solution obtained using the procedure described under 'Reducing sugars'. Add a volume of concentrated hydrochloric acid to flasks A and B in the proportion 0·3 ml per 10 ml of sugar-containings olution. Immediately add the same volume of 12N sodium hydroxide solution, i.e. 0·3 ml per 10 ml of sugar solution, into flask A and determine the reducing sugars by the procedure described under 'Reducing sugars'.

Place flask B, with the acidified sugar-containing solution, in a boiling waterbath for two minutes. Allow the vessel to cool in air for 15 minutes. Then add the same volume of 12N sodium hydroxide as added to flask A and determine the reducing sugars.

The difference between the quantities of reducing sugars obtained from these two determinations is multiplied by 0·95 to give the sucrose content of the test sample. This concentration is expressed in grams per litre of wine, account being taken of the dilutions that may have been made in the course of clarification and of the volume of the test sample of sugar solution being examined.

6. ASH CONTENT

6.1. DEFINITION

The ash content is defined to be all of those products remaining after igniting the residue obtained after evaporation of the wine; the ignition is carried out such that all of the cations (excluding ammonia) are converted into carbonates or other anhydrous mineral salts.

6.2. PRINCIPLE OF THE METHOD

Ignition of the wine extract is carried out at a temperature between 500 and 550 °C until complete oxidation of the carbon is obtained.

The quantity of ash should be expressed in grams per litre and be determined to the nearest 0·03 g.

6.3. SINGLE REFERENCE AND USUAL METHOD

Pipette 20 ml of wine into a flat-bottomed platinum dish of 70 mm diameter and 25 mm height, previously tared on a balance sensitive to 0·1 mg. Carefully evaporate the liquid, preferably on a waterbath. Heat the residue gently on a hot plate or under an infrared evaporator (for approximately 30 minutes) until carbonization commences. When evaporation has ceased, place the dish in an electric furnace with continuous ventilation and heated to 525 ± 25 °C. Withdraw the dish after carbonization (normally five minutes), add 5 ml of water, evaporate the mixture on a waterbath or under an evaporator and again heat the residue to 525 °C.

If combustion (oxidation) of cabonized particles is not complete after 15 minutes, repeat the washing operation, evaporation of the water and re-ignition.

For wines with a high sugar content, add if necessary a few drops of pure vegetable oil to the extract before the first ashing to prevent excessive foaming. In this case prolong the ignition for a further 15 minutes.

Weigh the dish after cooling it in a desiccator. The weight of the ash is multiplied by 50 and reported as grams per litre.

7. ALKALINITY OF THE ASH

7.1. DEFINITIONS

Total alkalinity of the ash is defined as the sum of cations other than ammonium combined with the organic acids of the wine.

Partial alkalinity of the ash is the alkalinity from the carbonates, oxides and silicates only in the ash.

Alkalinity per gram of ash (or alkalinity value) is calculated by dividing the total alkalinity expressed in grams of potassium carbonate by the weight of the ash.

7.2. PRINCIPLE OF METHODS

(a) **Total alkalinity**

Treatment with excess sulphuric acid and back titration of the excess using methyl orange as indicator.

(b) **Partial alkalinity**

Treatment with excess sulphuric acid and back titration of the excess in the presence of cerous ions to a pH of 4·5.

Expression of results

The alkalinity of the ash is expressed either in milliequivalents per litre and determined to the nearest 0·5, or in grams of potassium carbonate per litre.

7.3. (a) TOTAL ALKALINITY

SINGLE REFERENCE AND USUAL METHOD

Procedure

Weigh the ash and add 10 ml of 0·1N sulphuric acid and place the dish on a boiling waterbath for 15 minutes; repeatedly rub the bottom of the dish with a glass rod in order to assist dissolution of the less readily soluble material. Add two drops of methyl orange, one part per 1 000, and titrate the excess sulphuric acid against 0·1N sodium hydroxide until the colour of the indicator changes to yellow. Let the volume of sodium hydroxide used be n ml.

Calculations

The alkalinity of ash is equal to:

— 5 (10 — n) expressed in milliequivalents per litre,

— 0·345 (10 — n) expressed in grams of potassium carbonate per litre.

The alkalinity of 1 g of ash (alkalinity value) is equal to the ratio of the potassium carbonate alkalinity to the weight of the ash.

7.4. (b) PARTIAL ALKALINITY

SINGLE REFERENCE AND USUAL METHOD

Solutions

(a) Mixed indicator: dissolve 0·04 g of methyl orange in 20 ml of distilled water, and dissolve 0·2 g of bromocresol green and 0·04 g of methyl red in 80 ml of ethanol. Mix the two solutions. The colour change to grey takes place at pH 4·5.

(b) Cerium chloride solution: dissolve 10 g of analytical reagent grade $CeCl_3$, $7H_2O$ in sufficient distilled water (in a graduated cylinder) to make 12·5 ml of solution.

Procedure

Dissolve the ash from 20 ml of wine in 10 ml of 0·1N sulphuric acid. Bring the solution to the boil in order to drive off the CO_2. Add, after cooling, two drops of mixed indicator, two drops (or four in the case of wines rich in phosphates) of cerium chloride, and titrate with 0·1N sodium hydroxide until the colour of the indicator changes to grey. In order to check that sufficient cerium chloride has been added, add a further drop of cerium chloride solution, and if the indicator changes from red to orange, continue the titration with 0·1N sodium hydroxide until the colour changes to grey.

Calculations

As above.

8. TOTAL ACIDITY

8.1. DEFINITION

The total acidity is the sum of the titratable acids when the wine is titrated to pH 7 against a standard alkaline solution. Carbon dioxide and free and combined sulphur dioxide are not included in the total acidity. Carbon dioxide is removed by stirring when cold under vacuum.

8.2. PRINCIPLE OF METHODS

Reference method: Potentiometric titration.

Usual method: Titration with bromothymol blue as indicator.

Expression of results

The total acidity is expressed in milliequivalents per litre to the nearest two milliequivalents or in grams of tartaric acid per litre.

8.3. REFERENCE METHOD

Procedure

(a) Preliminary elimination of carbon dioxide and of sulphur dioxide.

Free the wine of dissolved carbon dioxide and sulphur dioxide present. To achieve this, place 100 ml of wine in a 500 ml flask fitted with a reflux condenser. Replace the air in the flask by nitrogen and then boil the wine for one hour while passing a slow stream of nitrogen. Cool the apparatus without interrupting the nitrogen supply.

(b) Titration.

Pipette 20 ml of wine into a beaker and add 0·1N sodium hydroxide until the pH as measured at 20 °C with a glass electrode is 7. The addition of the sodium hydroxide solution should be carried out slowly and the solution should be constantly stirred. Duration of titration must be at least five minutes.

Expression of results

The result is expressed in milliequivalent per litre or in grams of tartaric acid per litre.

8.4. USUAL METHOD

Expel the carbon dioxide from the wine by stirring 50 ml in a one litre flask while applying a vacuum produced by a water-jet pump. Stirring should take place for one to two minutes or until liberation of the dissolved gas is completed. Take a 10 ml aliquot for the total acidity and a 20 ml aliquot for the volatile acidity determination.

Note:

As the total acidity of the wine is measured at pH 7 with bromothymol blue as indicator, a preliminary test assists in the production of a suitable end-point colour at pH 7.

Allowance is made for the acidity of the free and combined sulphur dioxide by using the formula whereby the volumes n′ and n″ of 0·01N iodine are required for the oxidation of free and combined sulphur dioxides found in the volatile acidity distillate from 20 ml of wine (see determination of volatile acidity).

Solutions

(a) 0·05N sodium hydroxide solution, free from CO_2

(b)	Bromothymol blue	4 g
	Neutral ethanol	200 ml
	Dissolve and add:	
	— water free of CO_2	200 ml
	— N Sodium hydroxide sufficient to produce blue-green colour (pH 7)	7·5 ml approx.
	— water to	1 000 ml

(c) Buffer solution pH 7:

— monopotassium phosphate, KH_2PO_4	107·3 g
— sodium hydroxide N	500 ml
— water to	1 000 ml

Procedure

1. Preliminary test: end-point colour determination: place 25 ml of boiled distilled water into a crystallizing dish of 12 cm diameter, add 1 ml of bromothymol blue (4 g/l) and 5 ml of carbon dioxide free wine. Neutralize with sufficient 0·05N sodium hydroxide so that the colour changes to green-blue. Then add 5 ml of the pH 7 buffer solution.

2. Actual measurement: Place 30 ml of boiled distilled water, 1 ml of bromothymol blue (4 g/l), 5 ml of carbon dioxide free wine into a crystallizing dish of 12 cm diameter, and add 0·05N sodium hydroxide to produce an identical colour to that in the control.

   Let the titration be n.

Calculations

The total acidity is expressed in milliequivalents per litre:

$10\ n - 0{\cdot}35\ n' - 0{\cdot}25\ n''$

or in grams of tartaric acid per litre

$0{\cdot}75\ (n - 0{\cdot}035\ n' - 0{\cdot}025\ n'')$.

Notes:

1. In the case of white, rosé or almost colourless red wines the quantity of wine may be doubled. 10 ml of wine and 0·1N sodium hydroxide are then used; the formulae for the calculation remain the same.

2. When several wines of the same origin are to be examined a single end-point colour may be used for all the measurements. The absence of a change of colour on the addition of further buffer will verify that the end-point has been reached.

9. VOLATILE ACIDITY

9.1. DEFINITION

The volatile acidity derives from the fatty acids of the acetic series present in wine either in the free state or combined as a salt.

9.2. PRINCIPLE OF THE METHOD

Single referee and routine method. Separation of the volatile acids is obtained by steam distillation and redistillation of the distillate. The wine is acidified with tartaric acid (approx. 0·5 g for each 20 ml) prior to steam distillation. Precautions must be taken to avoid the carbon dioxide in the distillate. The indicator used is phenolphthalein. The acidity of distilled free and combined sulphur dioxide is not calculated as volatile acidity and should be deducted from the acidity of the distillate as well as any acidity from sorbic acid.

9.3. EXPRESSION OF RESULTS

The volatile acidity is expressed in milliequivalents per litre and is determined to the nearest 0·5 milliequivalent.

SINGLE REFEREE AND USUAL METHOD

Principle of the apparatus and its control

The apparatus consists of a steam generator, a bubbler into which the wine sample is placed, arectifying column and a condenser.

In view of the large variety and type of equipment which have been or which could be proposed, and in view of the difficulties which the adoption of a single type of equipment for all users in every country would present, it is preferable and rational to specify in the following manner the minimum conditions which must be met by any equipment or procedure permitting the accurate determination of volatile acidity as defined:

1. The steam produced by the generator must be sufficiently free of carbon dioxide such that 250 ml of distillate to which has been added 0·1 ml of N/10 sodium hydroxide and two drops of phenolphthalein (1 : 100) exhibits a stable coloration for at least 10 seconds.
2. Under normal conditions of use, the recovery of an aqueous solution acetic acid placed into the bubbler instead of wine should be at least 99·5 % in the distillate.
3. Not more than 5 % of lactic acid, added as a normal solution to replace the wine in a similar control experiment, should appear in the distillate under the conditions which produce a 99·5 % recovery of acetic acid.

Any apparatus and procedure which permits these three tests to be carried out successively and successfully fulfils the requirements of official international equipment and procedure.

The two types of apparatus described below meet the specified conditions: the first is suitable for laboratories dealing with limited numbers of samples, the second is intended for laboratories where a large number of determinations of volatile acidity have to be carried out daily and as quickly as possible.

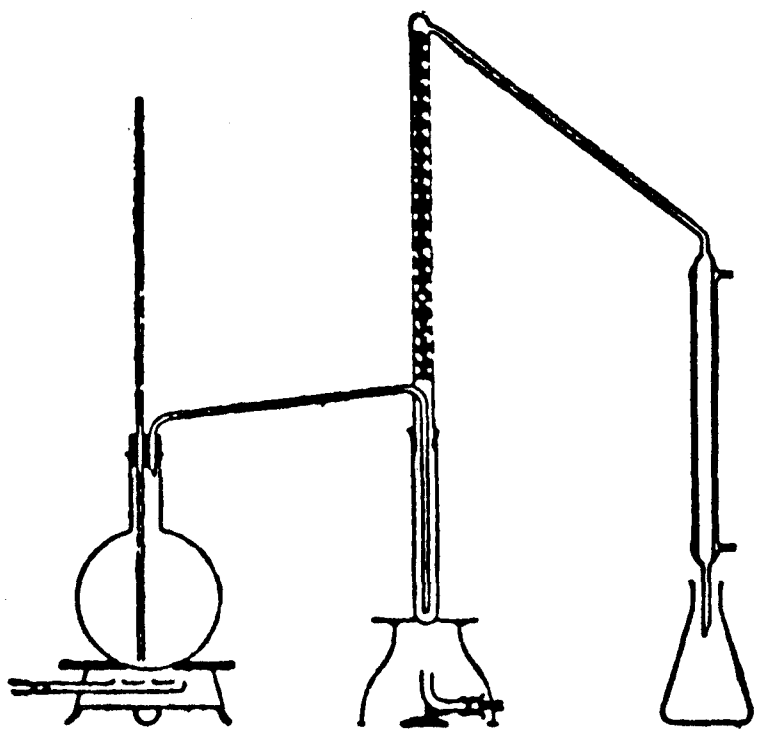
1. SMALL APPARATUS

Steam is produced in a 1 500 ml flask. The bubbler consists of a cylindrical tube of 3 cm diameter and 27 cm height. This tube is heated either electrically or by a flame; the base of the bubbler tube is inserted in the 29 mm diameter hole cut in a sheet-metal disc of 15 cm diameter and under which the flame is played. This prevents any overheating of extractive material in the wine. Control the auxiliary heating so that the volume of wine does not change by more than 5 ml during the course of the steam-distillation. The steam feed tube should end 1 cm above the bottom of the bubbler. Pass volatile acids in the steam into a rectifier column comprising a cylindrical tube of 20 mm diameter and 50 cm height containing a helix of corrugated stainless steel sheet No 100 at a pitch of 15 mm.

Place a West condenser, of 40 cm effective length, vertically to condense the vapour.

Procedure

The steam generator is filled with clear lime water or baryta water. Place 20 ml of wine, which has been freed of carbon dioxide by stirring under vacuum at room temperature, in the bubbler. Add approximately 0·5 g of tartaric acid. Distil and collect 250 ml of the distillate in 12 to 15 minutes.



Small apparatus for the distillation of the volatile acids

Titration

Titrate the acidity against 0·1N sodium hydroxide using two drops of phenolphthalein indicator (1 : 100 in neutral alcohol); let the volume of titrant used be n ml. Immediately restore the acid conditions after completion of the acidimetric titration by adding a drop of concentrated hydrochloric acid. Then add 2 ml of starch paste, a crystal of potassium iodide and titrate the free sulphur dioxide against an 0·01N iodine solution; let the volume used be n′ ml. Next add 20 ml of a saturated borax solution (the solution becomes pale pink) and again add 0·01N iodine solution until the starch paste becomes coloured. Let the volume of iodine used in this second titration, which represents the sulphur dioxide combined with acetaldehyde, be n″.

The volatile acidity expressed in milliequivalents per litre is equal to:

$$5\ (n - 0{\cdot}1\ n' - 0{\cdot}05\ n'')$$

Notes:

1. With samples preserved with salicylic acid, distil this acid, determine colorimetrically at a pH of $3 \pm 0{\cdot}5$, and subtract its value from this figure.

2. Compensation must be made for the presence of sorbic acid in wine as under the conditions of the experiment it is almost completely distilled. It is determined in a small portion of the distillate taken before titration. The volume of this distillate (250 to 300 ml) may be measured in a measuring cylinder.

   Take 0·5 ml of the distillate and transfer to a 1 cm silica spectrophotometric cell and add 1·5 ml of solution A below (1); and mix:

Solution A	$NaHCO_3$	0·5 g
	$CuSO_4, 5H_2O$	0·001 g
	Water to	1 000 ml

Measure the optical density at 256 nm after a few minutes against water and a standard sorbic acid solution of 20 mg/l (2), 0·5 ml of which is placed in a 1 cm cell with 1·5 ml of solution A and mixed.

This method is applicable to concentrations of 10 to 300 mg/l sorbic acid. The accuracy of the determination is approximately 1 %.

1 g of sorbic acid is equivalent to 8·92 milliequivalents and 0·438 g of sulphuric acid. For any wine to which 200 mg/l sorbic acid has been added, the appropriate correction for the volatile acidity is 1·8 milliequivalents per litre of wine.

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(1) This solution is intended to encourage the oxidation of the distilled sulphur dioxide by the atmospheric oxygen, because sulphur dioxide exhibits an appreciable absorption at 256 nm.

(2) Prepare this solution by placing 20 mg of sorbic acid into a litre flask with 900 ml of hot water, stir while cooling and make up to one litre. Alternatively dissolve 26·8 mg of potassium sorbate in cold water and make up to one litre with cold water.

2. RAPID DISTILLATION APPARATUS

The steam generator consists of a copper boiler of 20 litre capacity fed continuously with clear dilute lime water. It is heated moderately vigorously so that one kilogram of steam is produced in 12 minutes at a pressure not greater than that of a 50 cm water column.

The bubbler has a rectifier column attached to it. It consists of a U-tube, one branch of which is the steam supply tube with an inside diameter of 10 mm connected to the steam pipe from the boiler. The second branch consists of a cylindrical vessel of 26 mm diameter and 100 mm height. The steam is initially dispersed at the base of the vessel through a finned plate. To this cylinder are fused two bulbs, one of 500 ml and the other of 1 000 ml, on top of one another. The wine is introduced through a lateral nozzle fitted with a plug. There is an opening at the lowest point of the U-tube through which residues are removed at the completion of a distillation. Throughout the distillation this opening is kept closed with a rubber bung attached to a lever. The cylindrical part is heated electronically by a nickel-chrome steel strip (element); 350 W is produced using a 24 V supply. The water is evaporated at the same rate as it is condensing in the rectifier column.

The rectifier column is a column with a helix of corrugated stainless steel gauze No 100 and is 80 cm high with an internal diameter of 20 mm.

The condenser consists of a stainless steel tube with thin water jacket.

This apparatus may be provided with different control and interlocking devices to prevent faulty operation and to allow semi-automatic functioning if considered appropriate. 250 ml of distillate can be obtained in four minutes using this apparatus.

The measurement takes place as described previously.

10. FIXED ACIDITY

The fixed acidity is calculated from the difference between total acidity and volatile acidity.

11. TARTARIC ACID

11.1. PRINCIPLE OF METHODS

(a) **Reference method**

Tartaric acid is precipitated in the form of calcium DL-tartrate and determined gravimetrically. This determination may be completed by a volumetric procedure for comparison. The conditions for precipitation (pH, total volume used, concentrations of precipitating solutions) are such that precipitation of the calcium DL-tartrate is complete whereas the calcium (—) tartrate remains in solution.

When metatartaric acid has been added to the wine, it must first be hydrolized.

(b) **Usual methods**

The tartaric acid, separated using an ion exchange column, is determined colorimetrically in the eluate by measurement of the red colour produced on reaction with vanadic acid. The eluate also contains lactic and malic acids which do not interfere.

Expression of results

The tartaric acid is expressed in milliequivalents per litre and in grams of tartaric acid per litre. Accuracy of determination is 0·5 milliequivalents per litre.

11.2. REFERENCE METHOD

Reagents

Calcium acetate solution containing 10 g/l of calcium:

— calcium carbonate, analytical reagent grade	25 g,
— acetic acid, glacial	40 ml,
— water to	1 000 ml.

Calcium DL-tartrate, crystallized:

Place 20 ml of (+) tartaric acid solution (5 g/l) into a 400 ml beaker. Add 20 ml of ammonium (—) tartrate solution (6·126 g/l) and 6 ml of calcium acetate solution (10 g of calcium per litre).

Allow to stand for two hours to precipitate. Collect the precipitate in a sintered glass crucible of porosity No 4, and wash it three times with distilled water, the total washings to amount to about 30 ml. Dry to constant weight in the oven at 70 °C. Using the quantities of reagent indicated above about 340 mg of crystallized calcium DL-tartrate is obtained. Store in a stoppered bottle.

Precipitation solution (pH 4·75):

— ammonium (—) tartrate	150 mg,
— calcium acetate solution (10 g of calcium per litre)	8·8 ml,
— water to	1 000 ml.

Dissolve the ammonium (—) tartrate in 900 ml of water; add 8·8 ml of calcium acetate solution and make up to one litre. Since calcium DL-tartrate is slightly soluble in this solution, add 5 mg of calcium DL-tartrate per litre, stir for 12 hours and filter.

Procedure

Place 500 ml of precipitation solution and 10 ml of wine into a 600 ml beaker. Mix by rubbing the sides of the vessel with the tip of a glass rod. Leave to precipitate for 12 hours (overnight).

Filter the liquid and precipitate through a weighed sintered glass crucible of porosity No 4 fitted on a clean vacuum flask. Rinse the vessel in which precipitation took place with the filtrate to ensure, that all precipitate is transferred.

Dry to constant weight in an oven at 70 °C. Weigh: Let p be the weight of crystallized calcium DL-tartrate ($CaC_4O_6H_4, 4H_2O$) obtained.

Wines to which metatartaric acid has been added

When analyzing wine to which metatartaric acid has been or is suspected of having been added, proceed by first hydrolyzing this acid as follows:

Place 10 ml of wine and 0·4 ml of glacial acetic acid into a 50 ml conical flask. Place a reflux condenser on top of the flask and boil for 30 minutes. Allow to cool and then transfer the solution in the conical flask to a 600 ml beaker; rinse the flask twice using 5 ml of water each time and then continue as described above.

Metatartaric acid is calculated and included as tartaric acid in the final result.

Calculation and expression of results

One molecule of calcium DL-tartrate corresponds to half a molecule of (+) tartaric acid in the wine.

The quantity of tartaric acid per litre of wine is equal to:

384·5 p expressed in milliequivalents,

28·84 p expressed in grams of tartaric acid,

36·15 p expressed in grams of potassium acid tartrate.

COMPARATIVE VOLUMETRIC ANALYSIS

Reagents

0·2N hydrochloric acid (about 82 g/l)

Complexon III solution, 0·05M:

— complexon III (ethylenediaminetetraacetic acid disodium salt)	18·61 g,
— distilled water to	1 000 ml.

Sodium hydroxide solution, 40 % (w/v):

— sodium hydroxide, analytical reagent grade	40 g,
— distilled water to	100 ml.

Complexometric indicator (1 %):

— 2-hydroxy-1-(2-hydroxy-4-sulpho-1-naphthylazo)-3-naphthoic acid (Patton and Reeder's indicator)	1 g,
— sodium sulphate, anhydrous	100 g.

Procedure

Weigh the precipitate of calcium DL-tartrate contained in the crucible and then dissolve in 10 ml of dilute hydrochloric acid.

Wash the sintered glass crucible with 50 ml of distilled water.

Add 5 ml of 40 % sodium hydroxide solution and 30 mg of indicator. Titrate with 0·05 MEDTA solution. Let the number of millilitres used be n.

Calculation and expression of results

The quantity of tartaric acid per litre of wine is equal to:

5n expressed in milliequivalents,

0·375 n expressed in grams of tartaric acid.

11.3. USUAL METHOD

Solutions

Preliminary treatment of the wine.

— An ion exchange resin of Merck basicity strength III: the exchange resin should be in the acetate form. This is obtained by keeping it for at least 24 hours in 30 % acetic acid. Ideally, however, a more substantial stock is maintained by keeping the resin under 30 % acetic acid (200 ml of 30 % acetic acid is poured over approximately 100 g of exchange resin).

— Acetic acid 30 % : make up 300 ml of glacial acetic acid to one litre with distilled water.

— Acetic acid 0·5 % : make up 5 ml of glacial acetic acid to one litre with distilled water.

— Sodium sulphate solution 7·1 % (0·5 M) : a large volume of this reagent is required for elution. Dissolve 335 g of anhydrous sodium sulphate (Na_2SO_4) in water and make up to five litres with water.

Determination of the tartaric acid.

— Vanadic reagent: dissolve 10 g of ammonium metavanadate in 150 ml of N sodium hydroxide solution. Transfer the solution to a 500 ml volumetric flask and add 200 ml of a 27 % sodium acetate solution. Make up to 500 ml with distilled water.

— Standard 2N H_2SO_4 solution.

— Standard 0·1N H_2SO_4 solution.

— 0·05 M periodic acid solution (0·1N): put 10·696 g of sodium periodate and 50 ml of N sulphuric acid into a one litre volumetric flask and make up to one litre with distilled water.

— Glycerol solution 10 % : take 10 g of analytical reagent, twice distilled glycerol and make up to 100 ml with distilled water.

— Sodium sulphate solution, 7·1 % : dissolve 71 g of anhydrous sodium sulphate in distilled water. Make up to one litre.

— Pure tartaric acid solution, 1 g/l:

tartaric acid pure for analysis	0·500 g,
sodium hydroxide solution, N	6·66 ml,
sodium sulphate solution 7·1 % to	500 ml.

PROCEDURE

Preparation of the ion exchange column

Use a glass tube of 10 to 11 mm internal diameter and approximately 300 mm long with a drain tap attached at its end by means of a drilled rubber bung. The glass tube of the tap must not extend above the inner surface of the bung so avoiding any dead space.

Cover the rubber bung with a glass-wool plug of about 2 to 3 mm height, fill the tube with water and then allow to drain to approximately 5 mm above the glass-wool. Fill the tube with approximately 10 ml of a suspension of ion exchange resin in its acetate form, avoiding air bubbles. To achieve this, suspend the exchange resin, kept in 30% acetic acid, by stirring and pour rapidly into the tube through a funnel. Allow the ion exchange resin to settle. Place a small plug of glass-wool on top of the resin to prevent it being disturbed during subsequent washings. The exchange resin may only be used once.

For subsequent determinations remove the spent exchange resin by blowing out with water from a tap. The glass-wool plug at the bottom will normally be retained.

Separation of the tartaric acid using an ion exchange column

Open the drain tap fully and allow 30 % acetic acid to flow down the column to within approximately 2 to 3 mm of the upper glass-wool plug. Add approximately 10 ml of 0·5 % acetic acid and mark the liquid level with a grease pencil. Open the tap wide and allow the liquid to drain again down to within 2 to 3 mm of the upper glass-wool plug. Repeat this washing operation four more times with approximately 10 ml 0·5 % acetic acid, refilling the column each time to the reference mark.

After the last wash close the tap and pour 10 ml of the wine or must on to the exchange resin. (The presence of sugar, alcohol, colouring matter or tannins may be ignored.)

Pass the wine through drop by drop so that a flow rate of one to 1·5 drops per second (corresponding to a flow of 25 to 30 ml in 10 minutes) is not exceeded; stop the flow just above the level of the upper

glass-wool plug. Fill the tube again with 0·5 % acetic acid up to the reference mark. Allow to drain at the same rate as previously and subsequently wash seven times in the same manner using 10 ml of water each time, taking care to wash down the walls of the tube to the reference mark on each occasion.

Close the tap as soon as the liquid level is just below the upper glass-wool plug during the last washing.

Use a graduated flask of 100 ml as a receiver for the following operation. Elute the acids absorbed to the exchange material with a 0·5 M sodium sulphate solution. For this purpose fill a tubulated bottle with 0·5 M sodium sulphate solution and connect its lower tube to the ion exchange column tube with rubber tubing (using an adjustable screw-clip, a drawn-out tube and a rubber bung). When the two pieces of apparatus are joined, open the screw-clip, raise the rubber bung until the tube containing the ion exchange resin is filled to a height of approximately 10 cm with sodium sulphate solution. The two pieces of apparatus will now be joined. When the tap is opened sodium sulphate solution will run out at the same rate as the tube refills. The flow is regulated at a rate of two to three drops per second until the receiver flask has been filled up to its calibration mark.

Determination of tartaric acid

Place 20 ml of the eluate in two 100 ml conical flasks *a* and *b*.

Use flask *a* for the determination on sample and flask *b*, for the blank experiment, in which the tartaric acid has been destroyed by periodic acid.

Pipette into flask *a* 2 ml of 2N H_2SO_4, 5 ml of 0·1N H_2SO_4 and 1 ml of 10 % glycerol

Pipette into flask *b* 2 ml of 2N H_2SO_4, 5 ml of 0·05N periodic acid; allow to stand for 15 minutes and add 1 ml of 10 % glycerol solution to destroy the excess of periodic acid. Allow to stand for two minutes.

With stirring, pipette 5 ml of the vanadic reagent first into flask *b* and then immediately afterwards into flask *a*. Mix. Immediately start a stop-watch and pour part of the contents of flasks *a* and *b* into two matched 10 mm spectrophotometer cells. After one minute 30 seconds record the optical density of the liquid from flask *a* measured at 490 nm using that from flask *b* as the blank.

Plotting the calibration curve

Pipette 10, 20, 30, 40 and 50 ml aliquots of the standard tartaric acid solution preparation (see page 49) (1 g/l) into calibrated 100 ml flasks and make up to volume with the 7·1 % sodium sulphate solution. These solutions correspond to wine eluates containing 1, 2, 3, 4 and 5 g/l of tartaric acid.

Place 20 ml of each of the standard solutions of tartaric acid in two conical 100 ml flasks *a* and *b* and treat as described above for the wine eluate.

A graph of the optical densities of these solutions as a function of their tartaric acid concentration is a straight line curving slightly inwards towards the origin.

Draw this part of the calibration curve more carefully by repeating the determinations using solutions containing 0·1, 0·2, 0·3, 0·4, 0·5, 0·6, 0·7, 0·8, 0·9 and 1 g/l of tartaric acid.

12. CITRIC ACID

12.1. PRINCIPLE (single method)

Citric acid is precipitated by the barium ion in an aqueous alcoholic alkaline medium. After decoloration of the barium precipitate in aqueous solution, by activated charcoal, and oxidation by lead (IV) acetate, a yellow compound is formed in the presence of diazosulphanilic acid which is determined colorimetrically at 420 nm.

12.2. REAGENTS (1)

Concentrated ammonia: (C 20 = 0·910).

Barium chloride solution, 20 % : dissolve 20 g barium chloride, $BaCl_2 \cdot 2H_2O$ in distilled water and make up to 100 ml.

Washing liquid: mix 140 ml water with 300 ml alcohol (96 % by volume).

Sodium sulphate solution, 7·1 % w/v: dissolve 71 g anhydrous sodium sulphate in water and make up to one litre.

Activated charcoal.

Sodium acetate solution, 27 % : dissolve 270 g anhydrous sodium acetate in water and make up to one litre.

Sodium nitrite solution, 2 % : dissolve 2 g crystalline sodium nitrite in water and make up to 100 ml (this solution may be stored for a long period in a brown bottle).

Sulphanilic acid solution: dissolve 1·5 g sulphanilic acid in 50 ml glacial acetic acid and make up to 250 ml with water. Shake frequently after the preparation and allow to stand overnight. Store in brown bottles. This solution keeps indefinitely.

Saturated solution of lead (IV) acetate: place about 50 g lead (IV) acetate (lead tetracetate, moistened with glacial acetic acid) in a brown bottle and add approximately 250 ml glacial acetic acid. Shake and allow the non-dissolved part of the lead tetra-acetate to settle so that a clear supernatant liquid is obtained. Providing it is stored in a brown bottle, the solution keeps indefinitely. Further saturated solution can be prepared by adding glacial acetic acid to the residue and continuing as above.

Lead (IV) acetate solution, 1 % : Introduce 5 ml of the saturated lead (IV) acetate solution into a 250 ml conical flask and add all together 50 ml of 10 % potassium iodide solution. Titrate immediately with 0·1N sodium thiosulphate in the presence of starch solution. Let n be the number of millilitres added. Take a volume of saturated lead (IV) acetate solution equal to $\frac{2{\cdot}25 \times 1\,000}{n}$ ml and make up to one litre with glacial acetic acid. Store in brown bottles. This solution may be kept indefinitely.

12.3. PROCEDURE

Isolation of citric acid

Pipette into a 40 to 50 ml centrifuge tube 5 ml wine or must, 1 ml concentrated ammonia and 1 ml 20 % barium chloride. Mix with a glass rod. After approximately two minutes add 15 ml alcohol (96 % by volume) and mix again with the glass rod. Wait five minutes and then centrifuge for three to four minutes. Discard the supernatant liquid and then wash the precipitate as follows: take 2 ml of the washing liquid using a 2 ml pipette; apply the point of the pipette to the top of the tube wall, empty the pipette by blowing out while at the same time rotating the tube about its axis. Mix the precipitate and washing liquid to form a suspension using a glass rod fitted with a rubber covered tip. Rinse the rubber tip twice with a further 5 ml washing liquid, blowing fiercely down the pipette so that the jet of liquid removes any precipitate adhering to the rubber. Centrifuge again and discard the clear supernatant solution. Wash the precipitate a second time.

Cover the precipitate, still wet with the alcoholic washing liquid, with about 10 ml of the 7·1 % sodium sulphate solution. Place the centrifuge tube in a boiling waterbath for 10 minutes and crush the precipitate with a glass rod so that no lumps remain.

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(1) The reagents used must be very pure; analytical reagent grade chemicals are to be employed. No precipitation or coloration should be observed when the operations described below are carried out in the absence of citric acid.

Transfer quantitatively the contents of the still-warm tube to a 50 ml volumetric flask using the 7·1 % sodium sulphate solution. Allow to cool and make up to the mark with sodium sulphate solution, mix and pour the contents of the flask into a 100 ml conical flask containing 0·2 g activated charcoal. Stir. Allow to stand for five minutes and filter through a sintered glass crucible.

The filtrate is the test solution containing the citric acid from the wine or must at 10-fold dilution.

Determination of citric acid

Take two 50 ml conical flasks (a) and (b) and introduce 10 ml 27 % sodium acetate solution into each. Pipette 2 ml of the test solution to each of the flasks.

Prepare the diazotization solution by the addition, with shaking, of 5 ml of sulphanilic acid to 1 ml sodium nitrite solution.

Pipette into flask (a) 2 ml of the diazotization solution while swirling the flask continuously and then 5 ml glacial acetic acid; pipette into flask (b) 2 ml of the diazotization solution and 5 ml of 1 % lead (IV) acetate solution. Immediately start a stopclock.

After approximately five minutes, filter the contents of flasks (a) and (b) through fluted filter papers to aid rapid filtration. After nearly 13 minutes, pour the filtrates into two parallel sided curvettes with 30 mm path length and place in the spectrophotometer. After exactly 13 minutes, measure the absorbance at 420 nm of the liquid from flask (b) using the liquid from flask (a) as a blank.

If it is found that the test solution contains a citric acid concentration such that the absorbance is too great, dilute the test solution further with the 7·1 % sodium sulphate solution.

Calculate the citric acid concentration in the solution by reference to the standard curve giving the concentration of citric acid, $C_6H_8O_7$ as a function of absorbance.

Preparation of the standard curve

Dissolve 273·4 mg of pure citric acid monohydrate $C_6H_8O_7 \cdot H_2O$ (= 250 mg $C_6H_8O_7$) in a 7·1 % sodium sulphate solution. Make up to 100 ml with this solution. Pipette 10 ml of the solution into a further 100 ml volumetric flask and make up to volume with the sodium sulphate solution. Using this solution, prepare the solutions diluted as follows, making up to volume with the 7·1 % sodium sulphate solution in each case:

5 ml made up to 200 ml,
5 ml made up to 100 ml,
5 ml made up to 50 ml,
10 ml made up to 50 ml,
20 ml made up to 50 ml.

These different solutions correspond to test solutions from wines containing 0·0625, 0·125, 0·250, 0·500 and 1 g/l citric acid ($C_6H_8O_7$).

Pipette 2 ml of each of these dilutions into the flasks marked (a) and (b) repeat the above operations.

Obtain the zero point of the curve by pipetting 2 ml of the sodium sulphate solution into flasks (a) and (b) and repeat the above operations.

Obtain the zero point point of the curve by pipetting 2 ml of the sodium sulphate solution into flasks (a) and (b) and proceed as previously described.

The graph of the absorbances of these solutions as a function of the citric acid content is not linear but slightly curved.

13. SULPHUR DIOXIDE

13.1. DEFINITIONS

Free sulphur dioxide includes molecular SO_2 and that combined as HSO_3, SO_3^2 and H_2SO_3.

Combined sulphur dioxide is the difference between total and free sulphur dioxide.

13.2. PRINCIPLE OF METHODS

(a) **Reference method**

The sulphur dioxide is carried over by a current of air or nitrogen; it is fixed and oxidized by being bubbled into a diluted and neutral solution of hydrogen peroxide. The sulphuric acid formed is determined by titration with a standard solution of sodium hydroxide.

Entrainment at low temperature (10 °C) ensures extraction and determination of the free sulphur dioxide only. At a higher temperature (approximately 100 °C) the total sulphur dioxide is determined.

(b) **Usual method**

(aa) *Free sulphur dioxide:* direct iodometric titration with a blank titration on the same wine sample in which the free sulphur dioxide has been combined with an excess of acetaldehyde or propionaldehyde.

(bb) *Combined sulphur dioxide:* iodometric titration after double alkaline hydrolysis of the wine where the free sulphur dioxide has been oxidized during the iodometric titration.

Expression of results

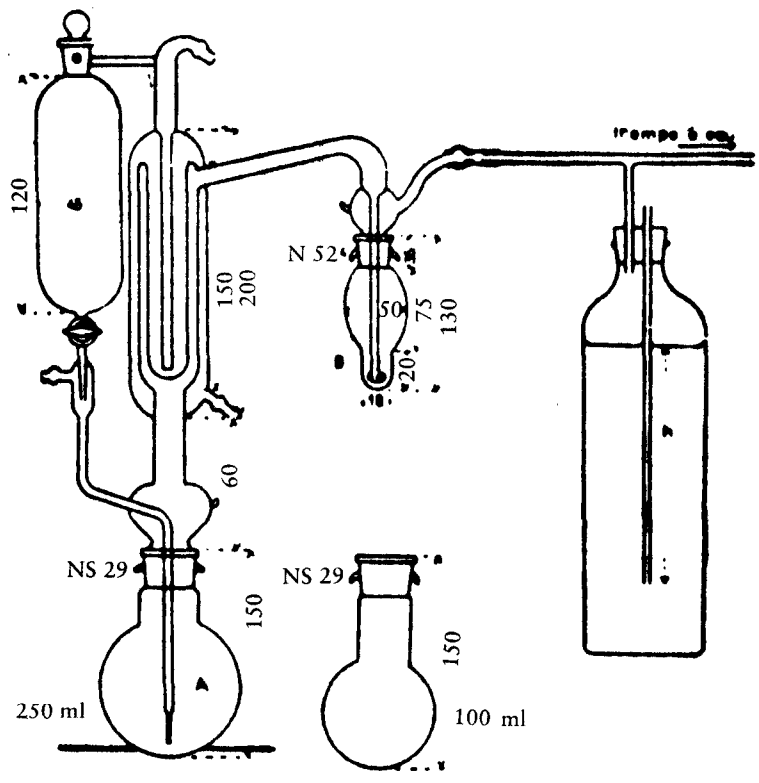
The quantity of sulphur dioxide is expressed in milligrams of sulphur dioxide per litre and determined to the nearest 10 mg/l.

13.3. REFERENCE METHOD

FREE AND COMBINED SULPHUR DIOXIDE

Apparatus

The apparatus used should conform to the diagram shown below with particular attention paid to the condenser.



The dimensions are indicated in millimetres. The internal diameters of the four concentric tubes which form the condenser are 45, 34, 27 and 10 mm.

The gas feed tube to the bubbler 'B' ends in a small sphere of 1 cm diameter with 20 holes of 0·2 mm diameter. Alternatively the tube may also end in a glass frit plate which produces a large number of very small bubbles thus ensuring good contact between the gaseous and liquid phases.

Ensure that the gas flow through the apparatus is approximately 40 litres per hour. The bottle on the right of the illustration is intended to restrict the vacuum effect of the water vacuum pump to 20 to 30 cm water. Install a flowmeter with semi-capillary tube between the bubbler and the bottle to regulate the vacuum.

Solutions

Phosphoric acid at 25 % (w/v).

Hydrogen peroxide (at 0·3 volume).

Indicator reagent:

— methyl red 100 mg,

— methylene blue 50 mg,

— alcohol 50 % 100 ml.

Sodium hydroxide 0·01N.

Procedure

Maintain the wine at 20 °C in a full and stoppered bottle for four days before the determination of the free and combined sulphur dioxide. This precaution is unnecessary if the sulphur dioxide only is to be measured.

Determination of free sulphur dioxide

Introduce into the 100 ml flask 'A' of the entrainment apparatus, 10 ml of wine and 5 ml of 25 % phosphoric acid. Assemble the apparatus immediately. If a larger volume (20 or 50 ml) of a wine of very low sulphur dioxide concentration is to be analyzed, use a flask of 250 ml capacity for A.

Maintain flask A at a temperature of 10 °C.

Place 2 to 3 ml of hydrogen peroxide at 0·3 volume (previously neutralized with 0·01N sodium hydroxide) and two drops of indicator reagent into the bubbler B.

Connect the bubbler to the apparatus and bubble air (or nitrogen) through for 12 to 15 minutes. The free sulphur dioxide carried over is oxidized to sulphuric acid. Remove the bubbler from the apparatus and titrate the acid which has formed against the 0·01N sodium hydroxide solution.

Let the number of millilitres used be n.

Determination of combined sulphur dioxide

When the determination of the free sulphur dioxide has been completed, again place 2 to 3 ml of neutralized hydrogen peroxide in the bubbler B. Heat the wine contained in flask A to boiling point with a small flame of 4 to 5 cm height which should directly touch the base of the flask. Do not put the flask on a metal plate, but on a disc provided with a hole of approximately 30 mm diameter to prevent any overheating of the extractive matter of the wine on the walls of the flask.

Simmer whilst passing a current of air (or nitrogen). Within 12 to 15 minutes the combined sulphur dioxide has been carried over and oxidized. Determine the sulphuric acid which has formed by titration with 0·01N sodium hydroxide solution.

Let the number of ml used be n′.

Determination of total sulphur dioxide

Obtain the total sulphur dioxide from the sum of the free and combined sulphur dioxide.

The total sulphur dioxide may also be determined directly by heating the test sample of 10 ml of wine to boiling point whilst a current of air or nitrogen is passed.

The free and combined sulphur dioxide are carried over simultaneously; the sulphuric acid formed corresponds to the total sulphur dioxide.

The number of millilitres of 0·01N sodium hydroxide used is designated n′′′.

Calculation

Free sulphur dioxide in milligrams per litre: 32 n.

Combined sulphur dioxide in milligrams per litre: 32 n′.

Total sulphur dioxide in milligrams per litre: 32 (n + n′) or 32 n‴.

Note:

If the wine contains more than 2 g/l of acetic acid (especially in vinegar) determine the acetic acid that may distil into the bubbler. This may occur if the gas flow is too great. After titrating the contents of the bubbler add a crystal of tartaric acid to it, put in the volatile acidity distillation apparatus and pass steam through. Deduct the volatile acidity obtained from the acidimetric determination of the sulphur dioxide.

13.4. USUAL METHOD

FREE AND COMBINED SULPHUR DIOXIDE

TOTAL SULPHUR DIOXIDE

Solutions

(a) Sodium hydroxide 4N (160 g/l).

(b) Sulphuric acid 1 : 10 by volume (180 g/l).

(c) Starch solution 2·5 g/l. Crush 2·5 g of soluble starch and 10 mg of mercuric iodide in a mortar with enough water to obtain a fluid paste. Transfer to one litre of strongly boiling water and keep boiling for 10 minutes. The liquid produced should be clear. Filter if necessary.

(d) Iodine 0·05N.

(e) Sodium thiosulphate 0·01N.

Procedure

Put into a 500 ml conical flask:

— 50 ml of wine,

— 3 ml of H_2SO_4 1/10,

— 5 ml of starch paste,

— 30 mg of sodium edetate (complexon III).

Titrate with 0·05N iodine until the blue colour, transient at first, persists clearly for 10 to 15 seconds (1). Let the volume of iodine used be n ml.

Add 8 ml of 4N sodium hydroxide solution, shake the mixture once and leave in contact for five minutes. Add, with vigorous stirring, in one operation, 10 ml of 10% v/v sulphuric acid. Titrate immediately with 0·05N iodine. Let the volume of iodine used be n′ (1).

Add 20 ml of 4N soda, shake once and leave in contact for five minutes. Dilute with 200 ml of ice-cold water.

Add, with vigorous stirring, 30 ml of 10% sulphuric acid in one operation. Titrate the free sulphur dioxide with 0·05N iodine. Let the volume of iodine used be n″ (1).

As certain substances are oxidized by iodine in acid solution, more accurate determinations may be required to assess the iodine used. If so, excess acetaldehyde or propionaldehyde are added before the addition of iodine. Add to 50 ml of wine in a 300 ml conical flask 5 ml of an acetaldehyde solution (7 g/l) or 5 ml of a propionaldehyde solution (10 g/l).

Stopper the flask and allow to stand for at least 30 minutes. Add 3 ml of sulphuric acid 1/10 and titrate with 0·05N iodine with starch solution as indicator. Let the volume of iodine used be n‴ (2).

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(1) If the precise end-point of the starch has been passed, carefully back titrate with 0·01 N thiosulphate.

(2) n‴ is generally small: 0·2 to 0·3 ml 0·05N. If ascorbic acid has been added to the wine, n‴ will be much higher and it is possible to measure approximately the amount from the value of n‴ on the basis of 1 ml of 0·05N iodine oxidizing 4·4 mg of ascorbic acid. By determining n‴ it is possible to detect the presence of residual ascorbic acid in amounts greater than 20 mg/l in wines to which it has been added.

Calculation

$n - n'''$ corresponds to free SO_2.
The wine contains: $32 (n - n''')$ mg of free SO_2 per litre.
$n' + n''$ corresponds to combined SO_2.
The wine contains: $32 (n' + n'')$ mg of combined SO_2 per litre.
$n + n' + n'' - n'''$ corresponds to total SO_2.
The wine contains: $32 (n + n' + n'' - n''')$ mg of total SO_2 per litre.

Notes:

1. For red wines with low SO_2 concentrations, more dilute iodine may be used, for example, 0·02. Replace the coefficient 32 by 12·8 in the above formulae.
2. For red wines illumination from below with a beam of yellow light from an ordinary electric light bulb and a solution of potassium chromate or from a sodium vapour lamp is useful. Carry out the determination in a dark room for the end-point to be visible.
3. If the quantity of sulphur dioxide is close to the legal limit determine the total sulphur dioxide by the reference method and complete this determination by gravimetric determination of the sulphate ions.
4. If the determination of free sulphur dioxide is particularly required, carry out the analysis at 20 °C on a sample kept for four days in anaerobic conditions at 20 °C.

14. pH OF WINE AND OF MUST

14.2. PRINCIPLE OF THE METHOD

The difference in potential between two electrodes immersed in the liquid under test is measured. One of these two electrodes has a potential which is a certain function of the pH of the liquid, the other has a fixed and known potential and constitutes the reference electrode.

The pH must be determined to ± 0·05 pH unit.

14.3. SINGLE REFERENCE AND USUAL METHOD

Apparatus and reagents

— *pH meter with a scale calibrated in pH units and suitable electrodes.* Keep the glass electrodes in distilled water. Keep the saturated potassium chloride calomel electrodes in a solution of saturated potassium chloride.

— *Potassium acid phthalate solution 0·1 M* (20·42 g/l).

— *Hydrochloric acid solution 0·1 M.*

— *Buffer solutions.* Saturated solutions of pure potassium acid tartrate (at least 5·7 g/l at 20 °C) (this solution can be kept up to two months by adding 0·1 g of thymol per 200 ml).

pH { 3·57 at 20 °C
3·56 at 25 °C
3·55 at 30 °C

— *Potassium acid phthalate solution 0·05 M* (10·211 g of analytical reagent potassium acid phthalate per litre at 20 °C) (maximum keeping period is two months).

pH { 3·998 at 10 °C
3·999 at 15 °C
4·003 at 20 °C
4·008 at 25 °C
4·015 at 30 °C

— Solution containing:

analytical reagent grade monopotassium phosphate KH_2PO_4	3·402 g,
analytical reagent grade dipotassium phosphate K_2HPO_4	4·354 g (1),
water to	1 000 ml.

(Maximum keeping period is two months.)

pH { 6·90 at 15 °C
6·88 at 20 °C
6·86 at 25 °C
6·85 at 30 °C

Procedure

1. *Zero setting of the equipment*

   Carry out before any determination, following the manufacturer's instructions given for the particular equipment used.

2. *Calibration of the pH meter*

   Dip the electrode into the buffer solution of pH 3·57 at 20 °C and adjust the pointer on the pH scale to this value with the control button. Discard the buffer solution and rinse the electrode carefully with distilled water.

   Dip the electrode into the buffer solution of pH 4·00, and at 20 °C the pointer should indicate the value 4 on the pH scale. Rinse the electrode carefully again.

3. *Determination*

   Dip the electrode into the wine or the grape juice, the temperature of which should be between 20 and 25 °C and as close as possible to 20 °C. Read the pH value directly off the scale.

   Carry out at least two determinations on the same wine.

   The arithmetic mean of two determinations which do not differ by more than 0·1 is reported.

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(1) Or 3·55 g of disodium phosphate Na_2HPO_4 dried for two hours at 110 to 130 °C.

15. SORBIC ACID

15.1. PRINCIPLE

Sorbic acid, which is steam-volatile (volatility constant 0·59) is determined in the distillate, after making slightly alkaline by adding limewater and evaporating to dryness to remove interfering substances, by either untraviolet absorption spectrophotometry, or by colorimetry after oxidation to malonic aldehyde which is then condensed with thiobarbituric acid to give a red coloured complex.

Concentration of less than 20 mg/l must be confirmed with thin-layer chromatography (sensitivity: 5 mg/l).

Preparation of the distillate

Prepare the distillate as described in the procedure for the determination of the volatile acidity by distilling and collecting 320 to 330 ml from a 20 ml sample of wine to which 2 g of tartaric acid has been added to ensure that all of the sorbic acid is distilled (1).

15.2. ULTRAVIOLET SOLUTION ABSORPTION SPECTROPHOTOMETRY

Reagents

Limewater, approximately 0·04N.

Copper sulphate solution, 50 mg/l:

$CuSO_4 \cdot 5H_2O$ pure	50 mg,
H_2SO_4 pure	0·1 ml,
Water to	1 000 ml.

Sorbic acid solution, 20 mg/l: place 20 mg of sorbic acid in a one litre volumetric flask, add 900 ml of hot water, shake, allow to cool and make up to one litre with water. It is also possible to dissolve 26·8 mg of potassium sorbate in cold water and make up to volume.

Procedure

Place 5 ml of distillate in an evaporating dish of 55 mm diameter, add 1 ml of clear limewater (approximately 0·04N) and one drop of copper sulphate solution. Evaporate to dryness on a boiling waterbath. Dissolve the residue in distilled water and make up to volume in a 20 ml volumetric flask.

Measure the absorbance at 256 nm against a blank of 1 ml of limewater containing one drop of copper sulphate solution in 20 ml.

Determine the concentration of sorbic acid by reference to a straight line calibration graph prepared with solutions containing 0·5, 1, 2·5 and 5 mg of sorbic acid per litre, the absorbance of which have been determined using distilled water as a blank. Prepare the calibration solutions by diluting the 20 mg/l sorbic acid solution with water.

Calculation

Let the concentration of sorbic acid in the solution in the cell (i.e. the concentration obtained from the spectrophotometric determination) be e mg/l.

If V ml is the volume (approximately 330 ml) of the distillate obtained from 20 ml of wine, the wine will contain $0{\cdot}2 \cdot e \cdot V$ mg of sorbic acid per litre (i.e. $66 \cdot e$ mg/l if exactly 330 ml of distillate has been collected).

If the concentration of sorbic acid determined is less than or equal to 20 mg/l, immediately carry out an identification of the sorbic acid in the wine by thin layer chromatography (2).

If the presence of sorbic acid is confirmed, recommence the determination using the following procedure:

Take a sample of 50 ml of the distillate and place it in an evaporating dish with 3 ml of clear limewater (approximately 0·04N) and five drops of the copper sulphate solution. Evaporate to dryness on a boiling

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(1) For the volatile acidity determination it is sufficient to collect only 250 ml of distillate, but to ensure that all of the sorbic acid has distilled over 320 to 330 ml must be collected. The determination of sorbic acid as a correction for the volatile acidity determination (see note 2 of the method on volatile acidity) recovers 96 to 97 % of the sorbic acid, when the volume of distillate collected is 250 ml.

(2) A full description of such a method will be given in a future Community Regulation.

waterbath. Dissolve the residue in distilled water and make up to volume in a 20 ml volumetric flask. Shake. Measure the absorbance of this solution against a block of 3 ml of limewater and five drops of copper sulphate solution diluted to 20 ml.

Calculation

Let the concentration of sorbic acid determined be e mg/l.

If V ml is the volume (approximately 330 ml) of the distillate obtained from 20 ml of wine, the wine will contain 0·02 · e · V mg of sorbic acid per litre.

15.3. COLORIMETRIC DETERMINATION

Reagents

Limewater approximately 0·04N.

Sulphuric acid solution, N.

Potassium dichromate solution, 0·1N

Thiobarbituric acid solution, 0·2 % (w/v): dissolve 200 mg of thiobarbituric acid in distilled water by heating to 60 to 80 °C. Cool and make up to 100 ml. Unless the solution is perfectly clear it must be filtered. It has to be prepared when required.

Sorbic acid solution, 20 mg/l: place 20 mg of sorbic acid in a one-litre volumetric flask, add 900 ml of hot water, shake, allow to cool and make up to one litre with water. It is also possible to dissolve 26·8 mg of potassium sorbate in cold water and make up to volume.

Procedure

Place 5 ml of the distillate in an evaporating dish with 1 ml of clear limewater (approximately 0·004N). Evaporate to dryness on a boiling waterbath. Dissolve the residue in distilled water and make up to volume in a 10 ml volumetric flask including the rinsing water from the dish. Shake.

Introduce successively into a test tube:

— 1 ml of this distillate (i.e. the 50 % dilution),

— 1 ml of distilled water,

— 1 ml of sulphuric acid,

— 0·2 ml of potassium dichromate solution, 0·1 N.

Put the test tube into a boiling waterbath for exactly five minutes. Rapidly cool the tube in a bath of ice-cold water and add 2 ml of the thiobarbituric acid solution, 0·2 %.

Put the tube into the boiling waterbath again for exactly 10 minutes, remove and then cool it in a bath of ice-cold water.

A pink coloration will form. Measure its absorption within 10 minutes following the removal of the test tube from the waterbath, but ensure that the test tube solution is completely cold.

The times of the heating and the use of the bath of ice-cold water must be rigidly observed, otherwise the intensity of the colours produced is affected. Development of the colours continues especially in summer temperatures and even at ordinary temperatures.

Measure the absorbance of the coloured solution at 532 nm against a blank prepared using 2 ml of distilled water to which the same quantities of reagents as those specified above have been added.

Determine the concentration of sorbic acid in the distillate by reference to a straight line calibration graph prepared with solutions containing 0·5, 1, 2·5 and 5 mg/l of sorbic acid, prepared from the 20 mg/l solution. Treat 1 ml of each of these solutions in the same manner as for this distillate.

Calculation

Let the concentration of sorbic acid contained in the solution placed in the cell, and determined by spectrophotometry, be e mg/l.

If V ml is the volume (approximately 330 ml) of the distillate obtained from 20 ml of wine, the wine contains 0·1 · e · V mg of sorbic acid per litre (i.e. 33 · e mg/l of sorbic acid, if exactly 330 ml of distillate has been collected).

If the concentration of sorbic acid determined is less than or equal to 20 mg/l, immediately carry out an identification of sorbic acid in the wine by thin layer chromatography (1).

If the presence of sorbic acid is confirmed, recommence the determination using the following procedure:

Take a sample of 50 ml of the distillate and place it in an evaporating dish with 3 ml of limewater (approximately 0·04N). Evaporate to dryness on a boiling waterbath. Dissolve the residue in distilled water and make up to volume in a 20 ml volumetric flask including the rinsing water. Shake.

Introduce successively into a test tube:

— 1 ml of this (2·5 times concentrated) distillate,

— 1 ml of distilled water,

— 1 ml of sulphuric acid solution, N,

— 0·2 ml of potassium dichromate solution, 0·1N.

Continue the determination as previously.

Calculation

Let the concentration of sorbic acid contained in the solution placed in the cell and determined by spectrophotometry be e mg/l.

If V ml is the volume (approximately 330 ml) of the distillate obtained from 20 ml of wine, the wine will contain $0{\cdot}02 \cdot e \cdot V$ mg of sorbic acid per litre.

(1) A full description of such a method will be given in a future Community Regulation.

16. ASCORBIC ACID

16.1. PRINCIPLE OF METHODS

(a) **Determination of total ascorbic acid**

The ascorbic acid is oxidized by iodine to dehydroascorbic acid which is then precipitated using 2,4-dinitrophenylhydrazin to produce a bis (2,4-dinitrophenylhydrazone). After separation by thin layer chromatography and dissolution in acetic acid medium the red-coloured derivative is determined colorimetrically at 500 nm.

(b) **Determination of active ascorbic acid (rapid test method)**

The quantity of active, i.e. still reducing, ascorbic acid can be approximated by its direct iodometric determination in the wine provided any free sulphur dioxide present has been combined with acetaldehyde or propionaldehyde.

16.2. DETERMINATION OF THE TOTAL ASCORBIC ACID

Reagents

— Metaphospheric acid solution 30 % (w/v): crush a few pieces of vitreous metaphosphoric acid in a mortar. Weigh 30 g and wash rapidly by covering the pieces with distilled water and stir, discard the washing water. Dissolve the washed acid in distilled water with stirring. Make up to 100 ml. The solution obtained has a metaphosphoric acid concentration of approximately 30 %. Keep in the refrigerator for a maximum period of one week.

— Metaphosphoric acid solution, 3 %: this solution is prepared on the day of use by dilution of the 30 % metaphosphoric acid solution.

— Metaphosphoric acid solution, 1 %: this solution is prepared on the day of use by dilution of the 30 % metaphosphoric acid solution.

— Polyamide suspension: slurry 10 g of polyamide powder, suitable for chromatography with 60 ml of distilled water and allow to stand for two hours (this quantity is sufficient for four determinations).

— Thiourea.

— Iodine solution, 0·1N.

— 2,4-Dinitrophenylhydrazine solution, 6 g/100 ml in a mixture of equal parts of acetic and sulphuric acids. First suspend the 2,4-dinitrophenylhydrazine in 50 ml of glacial acetic acid and then add concentrated sulphuric acid $\left(d_4^{20} = 1{\cdot}84\right)$ to dissolve.

— Ethyl acetate to which has been added 2 % (v/v) of glacial acetic acid.

— Chloroform.

— Silica gel G for chromatography.

— Soluble starch in 5 % solution.

— Solvent:

ethyl acetate	50 parts,
chloroform	60 parts,
glacial acetic acid	five parts.

Mix and allow the solvent to stand for 12 hours before use.

— L-ascorbic acid.

Apparatus

Equipment for producing thin layer chromatography plates.

Glass plates 20 × 20 cm.

Preparation of plates for chromatography. Suspend 30 g of silica gel in 70 ml of an aqueous solution of soluble starch, 0·5 %. Spread this suspension on the plates to give a thickness of 0·3 mm. This quantity is sufficient for five plates of size 20 × 20 cm. Dry the plates in air for approximately 15 minutes and then activate them by placing them for one hour 30 minutes in an oven at 105 °C.

Procedure

Oxidation to dehydroascorbic acid. Place 50 ml of wine in a 100 ml volumetric flask, add 15 ml of the polyamide suspension and make up to volume with metaphosphoric acid solution, 3 %. Allow to stand for one hour with frequent shaking. Filter through a fluted filter. Place 20 ml of filtrate into a 50 ml centrifuge tube fitted with a ground-glass stopper. Add 1 ml of 0·1N iodine solution. Mix and after one minute reduce the excess iodine by the addition of approximately 25 mg of thiourea.

Formation and extraction of the bis (2,4-dinitrophenylhydrazone) derivative of diketogulonic acid

Place the tube into a waterbath with the temperature maintained between 5 and 10 °C; add 4 ml of the 2,4-dinitrophenylhydrazine solution. Mix carefully, avoiding any wetting of the glass stopper. Place the completely sealed tube in a bath of 20 °C for approximately 16 hours (overnight).

Introduce 15 ml of ethyl acetate, containing 2 % of ascetic acid, into the centrifuge tube. Close the tube with its ground-glass stopper and shake vigorously for 30 seconds. Centrifuge for five minutes at 1 000 to 1 200 r/min.

Pipette 10 ml of the ethyl acetate extract into a conical flask fitted with a ground-glass stopper.

Introduce a further 5 ml of ethyl acetate containing 2 % of acetic acid into the centrifuge tube; again shake for 30 seconds and centrifuge for five minutes at 1 000 to 1 200 r/min. Pipette 5 ml of the ethyl acetate extract into the conical flask containing the 10 ml from the first extraction. Mix.

Separation of the bis (2,4-dinitrophenylhydrazone) by chromatography

Apply 0·2 ml of the ethyl acetate extract to the whole of a starting line situated 2 cm from the edge of the plate but leave a margin of 2 cm at the sides of the plate. Introduce solvent to a depth of approximately 1 cm into a chromatography tank and allow the atmosphere to become saturated with solvent vapour before introducing the plate. Allow the solvent to run to the top edge of the plate. (This chromatographic separation should be carried out within two hours after carrying out the extraction.)

Dry the plates for one hour under a ventilated hood. Hold the plate in a vertical position on a sheet of glazed paper and with a spatula scrape off (perpendicularly to the direction of the run) the red-coloured zone, which is characteristic of the bis (2,4-dinitrophenylhydrazone) derivative. Ensure that any current of air is excluded during this operation. Transfer the powdered product obtained, without loss, into a small ground-glass stoppered weighing bottle containing 4 ml of glacial acetic acid. Allow to stand for 30 minutes stirring frequently during this time. Filter through a small fluted filter paper directly into a 10 mm thick spectrophotometric cell but return the first 25 to 30 drops to the filter paper in order to obtain a perfectly clear filtrate.

Measure the absorbance of this solution at 500 nm using acetic acid in the reference cell and determine the concentration by reference to the calibration curve.

Preparation of the calibration curve

Prepare a solution of L-ascorbic acid of concentration 1 g per 100 ml using the 1 % metaphosphoric acid solution solvent.

Dilute 5, 10 and 15 ml of this solution to 100 ml using the same solution of metaphosphoric acid as the diluent.

Carry out the reactions described above with 50 ml of each of these dilutions which contain 50, 100, 150 mg of ascorbic acid per litre to prepare the calibration curve.

A graph of the absorbance as a function of the concentration of the solution should be a straight line through the origin.

16.3. RAPID TEST METHOD

Determination of active ascorbic acid

Solutions

— Acetaldehyde solution 6·9 g/l: prepare by distillation of metaldehyde or of paraldehyde in the presence of sulphuric acid and titrate by the sodium sulphite method. Adjust the solution to a concentration of 6·9 g/l. 1 ml of this solution combines with 10 mg of sulphur dioxide.

— Sulphuric acid diluted 1 : 10 by volume (180 g/l).

— Starch solution, 5 g/l: add to this solution 200 g of sodium chloride to preserve; boil for 10 minutes at the time of preparation.

— Iodine, 0·05N.

Procedure

Place 50 ml of wine into a 300 ml conical flask and add 5 ml of the acetaldehyde solution (6·9 g/l) (1). Stopper the flask and allow to stand for at least 30 minutes. Add 3 ml of sulphuric acid (1/10 v/v) and titrate with 0·05N iodine using the starch solution as indicator. Let the volume of iodine used be n

1 ml of 0·05N iodine oxidizes 4·4 mg of ascorbic acid.

The wine contains approximately 88 n mg of ascorbic acid per litre.

Notes:

1. Certain substances in the wine are oxidized by iodine in acid medium, but the volume of 0·05N iodine consumed under the above conditions is normally small (0·2 to 0·3 ml). This method also permits detection of wines to which more than 20 mg ascorbic acid per litre has been added and which has not changed into oxidation products.

2. In the case of red wines it is advantageous to illuminate the wine from below using a beam of yellow light obtained by passing light from an electric light bulb through a solution of potassium chromate or by using a sodium vapour lamp. The apparatus must be used in a darkroom to observe the transparency of the wine which becomes opaque when the end-point is reached.

(1) It is also possible to use 5 ml of a 10 g/ propionaldehyde solution.

17. SODIUM

PRINCIPLE OF THE SINGLE METHOD

Sodium is determined in the same manner as potassium i.e., using a flame photometer to measure the intensity of the 589·0 and 589·6 nm radiation emitted from the flame of a burner into which wine diluted by at least 1 : 10 is aspirated.

Apparatus

As the method is comparative, the sensitivity of the photometer and the temperature of the flame are not specified but it is advisable to use an air-butane flame for the measurement of the alkali metals to reduce interference from other cations.

Calibrate the photometer using appropriate dilutions of a reference solution containing 20 mg of sodium per litre (0·869 milliequivalents per litre) and various anions, cations and organic material in quantities similar to those in an average wine which has been diluted 1 : 10 with water.

Solutions

Reference solution	
Alcohol absolute	10 ml
Citric acid	700 mg
Sugar	300 mg
Glycerol	1 000 mg
Potassium hydrogen tartrate	481·3 mg
Calcium chloride dry	10 mg
Magnesium chloride dry	10 mg
Sodium chloride dry	50·84 mg
Water to	1 000 ml

Dilution solution	
Alcohol absolute	10 ml
Citric acid	700 mg
Sugar	300 mg
Glycerol	1 000 mg
Potassium hydrogen tartrate	481·3 mg
Calcium chloride dry	10 mg
Magnesium chloride dry	10 mg
Water to	1 000 ml

Prepare the solutions by dissolving 481·3 mg of pure potassium hydrogen tartrate in 0·5 litre of very hot water, mix this solution with 400 ml of water containing the other chemicals already dissolved; make up to one litre with water. Preserve solutions by adding two drops of allyl isothiocyanate.

Add in the case of sweet wines and must samples, a quantity of sugar similar to that in the wine (or the must) after it has been diluted 1 : 10 with water.

Add, in the case of wines which are salicylated to 1 g/l, 86·4 mg of salicylic acid per litre to the reference and dilution solutions, because this material interferes considerably in the determination.

Procedure

Calibrate the photometer using the pure reference solution and various dilutions of the reference solution (to be 1 : 20, 1 : 10, 1 : 2, 3 : 4 v/v dilutions) using the dilution solution as diluent.

Carry out the determination on wine which has been diluted 1 : 10 with water.

If necessary further dilute the diluted wine with the dilution solution so that the galvanometer reading does not exceed full scale deflection.

18. IRON

18.1. PRINCIPLE OF METHODS

(a) **Reference method:** spectrophotometric determination of the total iron with o-phenanthroline after digestion with hydrogen peroxide.

(b) **First usual method:** spectrometric determination.

(c) **Second usual method:** atomic absorption spectrophotometry.

18.2. REFERENCE METHOD

1. DIGESTION

Solutions

Hydrogen peroxide (100 to 110 volumes) free of iron.

Hydrochloric acid, normal, free of iron.

Ammonia, pure; specific gravity 0·92.

Pumice stone grains, treated with hydrochloric acid, 1 : 2 dilution, at boiling temperature and washed with distilled water.

Procedure

There are two variations of the procedure which are to be used depending on the sugar content of the wine sample:

(a) for dry wines or wines with moderate sugar content (up to 50 g/l of sugar): introduce into a Kjeldhal flask of 100 ml capacity a few grains of pumice, 20 ml of the wine sample and 10 ml of hydrogen peroxide (100 vol). Concentrate the liquid on the sand bath to a volume of 2 to 3 ml. Cool the residue and add dropwise using a burette 3 to 4 ml of ammonia, or more if necessary, taking care not to wet the walls of the flask. It is necessary to add a sufficient excess, which is detectable by smell, to make the liquid alkaline and then to precipitate any metal hydroxides. The speed of reaction between the hydrogen peroxide and ammonia must be controlled by either cooling the flask with cold water or by moderate heating on a waterbath. Cool and then add normal hydrochloric acid to the alkaline liquid so that any precipitated hydroxides are just dissolved; transfer the solution obtained to a volumetric flask of 100 ml capacity. Rinse the Kjeldahl flask with normal acid. Add the rinsings to the volumetric flask and then make up to volume with the acid;

(b) for wines with high sugar content: the oxidizing treatment for wines with high sugar content (50 to 200 g/l) differs slightly from the procedure given above in that the sample of 20 ml of wine is treated with 20 ml of hydrogen peroxide.

Liquids containing more than 200 g/l of sugar must be diluted 1 : 2 or possibly 1 : 4 before oxidation.

Note:

Blank tests should be carried out with distilled water and the volume of hydrogen peroxide required for the treatment of the wine samples.

2. SPECTROPHOTOMETRIC MEASUREMENT

Solutions

— Hydroquinone solution, 2·5 %, in water containing 10 ml of concentrated sulphuric acid per litre. This solution must be stored in a brown flask in the refrigerator and discarded as soon as there is any darkening of solution.

— Sodium sulphite solution, 20 %, prepared from neutral, anhydrous sodium sulphite.

— Ammonium acetate solution, 20 %.

— o-Phenanthroline solution, 0·5 %, in 96 % vol alcohol.

— Standardized iron III solution, 1 g/l of iron. Weigh 8·6341 g of ferric ammonium sulphate and put in a one-litre volumetric flask; dissolve the salt in normal hydrochloric acid. Dissolution is not immediate and it is necessary to stir periodically. Make up the solution when clear to volume with the same acid. The concentration of this solution must be checked.

Reduction of iron III and formation of coloured complex

Take two test samples each of 20 ml from the 100 ml hydrochloric acid wine sample solution and place each in a 50 ml volumetric flask provided with ground-glass stopper. Add 2 ml of hydroquinone solution, 2 ml of sulphite solution and 1 ml of o-phenanthroline solution to both flasks. Allow to stand for 15 minutes during which time reduction takes place at a pH of approximately 1 to 2; Then add 10 ml of ammonium acetate solution; the formation of the coloured ferrous o-phenanthroline complex appears rapidly; make up to volume with distilled water. Use these solutions for the colorimetric determination.

Colorimetric measurements: calculation of results

Measure the absorbance at 508 nm. If a spectrophotometer with monochromator is not available use a Wratten filter No 75 blue-green.

Prepare a calibration curve using iron concentrations over the range 0 to 200 g in 50 ml. In general the use of 10 mm cells will be sufficient. However, the use of cells with different path lengths may make it possible to avoid dilutions and to increase the accuracy of measurement.

Prepare a calibration curve for each cell under the above conditions. Draw up the curves so that the optical densities observed correspond to numbers of micrograms of iron contained in a 50 ml test volume taken for the colorimetric measurement. This volume contains the 20 ml of acid solution from the 100 ml of the acid solution prepared following the oxidation of the wine sample. This 100 ml is prepared from a test sample of 20 ml of wine.

If n is the number of micrograms of iron contained in 50 ml of the solution used for the colorimetric determination and which correspond to 20 ml of the acid solution or to 4 ml of wine, the concentration of iron in the wine sample is 0·25 n mg per litre.

Range of application and accuracy of the procedure

The procedure described above, with variable cell path lengths, permits measurement without dilution of the iron concentration if, within a range of 1·25 to 75 mg/l for a test sample of 20 ml of wine, by increasing the size of the test sample, the lower limit may be reduced to approximately 0·5 mg/l.

The accuracy of the determination is 10% for iron concentration from 1·25 to 5 mg of iron per litre of wine, and less than 5% for quantities exceeding 5 mg/l.

18.3. FIRST USUAL METHOD

Principle

Iron in the wine sample is reduced by hydroxylamine hydrochloride and then treated with o-phenanthroline. The absorbance at 508 nm is measured and the concentration of iron in the sample is calculated from a calibration curve.

With very coloured red wine samples it may be necessary to use a solution prepared from the ashed sample.

Solutions

Solution 1:	
hydrochloric acid concentrated (d = 1·19)	170 ml,
hydroxylamine hydrochloride	10 g,
water to	1 000 ml.
Solution 2:	
o-phenanthroline	1 g,
water to	100 ml.
Solution 3:	
ammonium acetate	180 g,
water to	1 000 ml.

Note:

The presence of traces of iron in the water or in the reagents does not affect the accuracy of the results.

The acid solution of hydroxylamine chlorohydrate is stable for several months (in contrast to the hydroquinone solutions).

Procedure

Place 10 ml of wine in each of two 50 ml volumetric flasks and add 10 ml of solution 1. Pipette 1 ml of solution 2 into one of the flasks. Allow to stand for 15 minutes and then add 10 ml of solution 3 to each flask. On this addition the solution contained in the flask to which the phenanthroline (solution 2) has been added will turn an orange-red colour, whereas the colour of the second flask (blank) will remain unchanged or tend towards a greyish colour.

Make up to volume with water and determine photometrically the absorbance of the solution in the first flask by comparison with the solution in the second flask and using a filter with transmission maximum of approximately 508 nm. The result of the reading is converted into milligrams of iron per litre of wine using the calibration curve which is prepared for all future determinations as described below.

Preparation of the calibration curve

Dissolve, with heating, 1 g of pure iron wire in 250 ml of 20% sulphuric acid taking care to eliminate losses by splashing. When the reaction is complete, cool and make the volume up to one litre. This solution is the stock solution which is stable indefinitely.

The stock solution may also be prepared from Mohr's salt of known purity. For Mohr's salt of 100% purity the equivalence is: 1 g of iron = 7·018 g of Mohr's salt (weighed in air).

Take 20 ml of the stock solution and make up to one litre. Using this diluted solution, which contains 0·02 mg/ml of iron, prepare solutions equivalent to iron concentrations of between 0·4 and 4 mg/l in wine by pipetting between 1 and 10 ml of the dilute solution into a series of 50 ml volumetric flasks.

Next pipette 10 ml of solution 1 and 1 ml of solution 2 into each flask. Allow to stand for 15 minutes and then carry out the instructions given in the general procedure, i.e. add 10 ml of solution 3 and sufficient water to make up to volume. Photometric readings are then taken by comparison with a blank prepared by mixing 10 ml of solution 1, 10 ml of solution 3 and sufficient water to make up the volume to 50 ml [1].

Plot the transmission values obtained against the iron concentrations, expressed in milligrams per litre, on semilogarithmic paper and draw the straight line which should pass through the points so plotted.

Correction of the calibration curve

Sometimes correctly established the reference points do not form a straight line. This is generally caused by inadequate sensitivity of the photometer used. Under these circumstances, and in order to avoid repeated measurements, the results already obtained may still be used by applying the method of least squares to calculate the most probable values of the constants A and k in the equation $Y = \log y = A - kx$, where y = transmission in % and x = iron content (expressed in mg/l of wine).

Once this relation has been established, the values 1 and 2 corresponding to values x_1 and x_2 chosen arbitrarily (e.g. $x_1 = 0$ and $x_2 = 10$) may be calculated. The corrected calibration curve is constructed by drawing on semilogarithmic paper the straight line passing through the coordinate points $(x_1 Y_1)$ and (x_2, Y_2).

It is to be noted that the straight line thus drawn will pass through the origin only if the reagents used contained no iron (in the absence of phenanthroline such reagents have little influence on the transmission).

Special cases

Wines with a high iron concentration

It is then advantageous but not absolutely necessary to use a photometer with cells of different path lengths and to construct a calibration curve for each cell path length. It should however be noted that the Beer-Lambert law applies in the range 20 to 200 g (per 50 ml). If the concentration is too high to allow a correct assessment of the absorbance, repeat the measurement using a smaller sample [2]. It is also possible to dilute (e.g. 1 : 2 or 1 : 5) the solutions in the two flasks using a freshly prepared mixture of:

solution 1: one part,
solution 3: one part,
water: three parts.

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[1] As in the procedure used for the wire, no phenanthroline (solution 2) is introduced into the blank solution. Care must be taken that the filter is used for these readings as for the determinations with wine samples.

[2] The transmission values most suitable for measurement are between 30 and 70 %.

Highly coloured red wines

In the case of very strongly coloured wine samples the method whilst theoretically as accurate will nevertheless lose some sensitivity, as the percentage of the total absorbance resulting from phenanthroline complex will be low thus leading to unreliable results. It is advisable when analyzing these samples to carry out the measurement on a smaller sample of wine or else to use the ash. If a procedure such as is described below is adopted, the ashing process should not involve any losses greater than 5 %.

Determination of iron after ashing the wine

Pipette 10 ml of wine into a thin-walled, flat-bottomed platinum dish of approximately 8 cm diameter and 1·5 to 2 cm height. Evaporate on a waterbath and dry in an oven at 110 to 120 °C. Ash using a very moderate flame, taking care to avoid any fusion of the ash. In order to eliminate the last carbonized particles it may be necessary to take up the ash in a few millilitres of water, dry again and repeat the ashing process. After cooling, moisten the ash and dissolve it in 10 ml of solution 1. Place the dish on a waterbath. After three to five minutes transfer the liquid to a 50 ml volumetric flask, rinse the dish three times with 2 to 3 ml of water and collect the rinsings in the flask. After cooling add 1 ml of solution 2. Into a second 50 ml flask pipette 10 ml of solution 1 [1]. Wait 15 minutes and then proceed with the measurement as described in the general procedure, i.e. add 10 ml of solution 3 to the two flasks and sufficient water to make up to volume and then determine the transmission photometrically.

18.4. SECOND USUAL METHOD

1. Principle

The method is based on the use of atomic absorption spectrophotometry to determine the iron content of solutions obtained after direct dilution of the sample.

2. Reagents

1. metallic iron,
2. nitric acid, concentrated, 65 % (d = 1·413),
3. nitric acid, dilute (1 + 1),
4. water, doubly-distilled.

3. Apparatus and other equipment

1. calibrated pipette,
2. micropipette,
3. volumetric flask,
4. atomic absorption spectrophotometer,
5. iron hollow cathode lamp,
6. fuel gas: acetylene, support gas: air or nitrous oxide.

4. Procedure

1. *Preparation of the sample*

Pipette 20 ml of the sample into a 100 ml volumetric flask and make up to the volume with doubly-distilled water.

Determine the absorbance of the diluted sample using the atomic absorption spectrophotometer set at a wavelength of 248·3 nm. Calculate the concentration of the metal in solution using the calibration graph prepared as described below.

2. *Preparation of the calibration graph*

Weigh 1·00 g of metallic iron and transfer to a 1 000 ml volumetric flask. Add only as much dilute nitric acid as is necessary to dissolve the metal. Add 10 ml of concentrated nitric acid and make up to the volume with doubly-distilled water.

This solution contains 1 000 mg/l of iron (solution A). Pipette 10 ml of solution A into a 100 ml volumetric flask and make up to the volume with doubly-distilled water. This solution contains 100 mg/l of iron (solution B).

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[1] But not on solution 2 (phenanthroline).

Pipette 1, 2 and 5 ml of solution B into a series of 100 ml volumetric flasks and make up to volume with doubly-distilled water. The above solutions contain, respectively, 1, 2 and 5 mg/l of iron. Determine the absorbance using the spectrophotometer as described under 4.1 above and construct a calibration graph.

5. **Expression of results**

Express the concentration of iron in the sample in milligrams per litre after calculation using the formula $c \times n$:

where:

c = concentration of the metal in the diluted sample determined using the calibration graph,

n = the dilutions carried out on the sample.

6. Notes

1. Select the solutions for construction of the calibration graph and dilution of the sample appropriate to the sensitivity of the apparatus to be employed and the concentration of the iron present in the sample.
2. Re-check at least one part of the calibration graph every time a series of determinations is carried out.

19. COPPER

19.1. PRINCIPLE OF THE SINGLE METHOD

The method is based on the use of atomic absorption spectrophotometry to determine the copper content of solutions obtained after direct dilution or ashing of the sample.

19.2. REAGENTS

1. Metallic copper.
2. Nitric acid concentrated 65 % (d = 1·40).
3. Nitric acid dilute (1 + 1).
4. Water, doubly-distilled.

19.3. APPARATUS AND OTHER EQUIPMENT

1. Calibrated pipette.
2. Micropipette.
3. Flask.
4. Platinum dish.
5. Atomic absorption spectrophotometer.
6. Copper hollow cathode lamp.
7. Fuel gas: acetylene, support gas: air or nitrous oxide.

19.4. PROCEDURE

1. **Preparation of the sample**

   Pipette 20 ml of the sample into a 100 ml volumetric flask and make up to volume with doubly-distilled water. Determine the absorbance of the distilled sample using an atomic absorption spectrophotometer set at a wavelength of 324·8 nm. Calculate the concentration of the metal in solution using the calibration graph prepared as described below.

2. **Preparation of the calibration graph**

   Weigh 1 000 g of metallic copper, transfer the quantity to a 1 000 ml volumetric flask. Add just sufficient diluted acid to dissolve the metal. Add 10 ml of concentrated nitric acid and make up to volume with doubly-distilled water. This solution contains 1 000 mg/l of copper (solution A).

   Pipette 10 ml of solution A and transfer to a 100 ml flask and make up to the volume with doubly-distilled water: this solution contains 100 mg/l of copper (solution B).

   Pipette 0·5, 1 and 2 ml of solution B into 100 ml volumetric flasks and make up to the volume with doubly-distilled water.

   Thus solutions containing 0·5, 1 and 2 mg/l respectively of copper are produced. Determine the absorbance values of those solutions as described in 4.1 and construct the calibration graph.

19.5. EXPRESSION OF THE RESULTS

Express the results in milligrams per litre after calculation using the formula $c \times n$:

where:

c = concentration of the metal in the diluted sample determined using the calibration graph,

n = the dilutions carried out on the sample.

19.6. NOTES

1. Select the solutions for construction of the calibration graph and dilution of the sample appropriate to the sensitivity of the apparatus to be employed and the concentration of the copper present in the sample.
2. Re-check at least one part of the calibration graph every time a series of determinations is carried out.

3. Proceed as follows when concentrations of very low copper in the sample to be analyzed are expected; place 100 ml of the sample in a platinum dish and evaporate on a waterbath until it becomes syrupy. Add 2·5 ml of concentrated sulphuric acid dropwise and cover the bottom of the dish completely. Carefully ash the residue on an electric hotplate or over a low flame; then place the dish into a muffle furnace set at 500 ± 25 °C and leave for about one hour. After cooling, moisten the ash with 1 ml of concentrated nitric acid while crushing it with a glass rod; allow the mixture to evaporate and ash again as before. Place the dish in the muffle furnace again for 15 minutes, repeat the treatment with the concentrated nitric acid at least three times. Dissolve the ash by adding 1 ml of concentrated nitric acid and 2 ml of doubly-distilled water to the dish and transfer to a 10 ml flask. Wash the dish three times using 2 ml of doubly-distilled water each time. Finally make up to volume with doubly-distilled water.

20. SILVER

20.1. PRINCIPLE

The method is based on the use of atomic absorption spectrophotometry to determine the silver content of solutions obtained after ashing of the sample.

20.2. REAGENTS

1. Silver nitrate.
2. Nitric acid, concentrated, 65 % (d = 1·40).
3. Nitric acid, dilute 1 + 9.
4. Water, doubly-distilled.

20.3. APPARATUS AND OTHER EQUIPMENT

1. Platinum dish.
2. Volumetric flask.
3. Calibrated pipette.
4. Atomic absorption spectrophotometer.
5. Silver hollow cathode lamp.
6. Fuel gas: acetylene, support gas: air.

20.4. PROCEDURE

1. **Preparation of the sample**

   Place 20 ml of the sample in a platinum dish and evaporate to dryness over a boiling waterbath. Ash in a muffle furnace at 500 to 525 °C. Moisten the white ash with 1 ml of concentrated nitric acid. Evaporate over a waterbath and then add 1 ml of nitric acid and evaporate a second time. Add 5 ml of diluted nitric acid and heat slightly until dissolved and make up to 5 ml with water. Determine the absorbance of the ashed sample using an atomic absorption spectrophotometer set at a wavelength of 328·1 nm and with an air-acetylene flame.

2. **Preparation of the calibration graph**

   Weigh out 1·5750 g of silver nitrate and place in a 1 000 ml volumetric flask; make up to volume with dilute nitric acid (1 + 9) (solution A).

   Pipette 10 ml of solution A into a 1 000 ml volumetric flask and make up to volume with dilute nitric acid (solution B). Pipette 2, 4, 6, 8, 10 and 20 ml of solution B into a series of 100 ml volumetric flasks and make up to volume with dilute nitric acid.

   The above solutions contain 0·20, 0·40, 0·60, 0·80, 1·0 and 2·0 mg/l of silver.

   Determine the absorbance using the spectrophotometer as described under 20.4 (1) above and construct a calibration graph.

20.5. EXPRESSION OF RESULTS

Express the concentration of silver in the sample in milligrams per litre after calculation using the formula $\frac{C}{4}$:

where:

C = concentration of the metal in the diluted sample determined using the calibration graph.

20.6. NOTES

1. Select the concentration of the solutions for construction of the calibration graph, the volume of the sample taken and the final volume of the ashed sample to be appropriate to the sensitivity of the apparatus to be employed and the concentration of silver present in the sample.
2. Re-check at least one part of the calibration graph every time a series of determinations is carried out.

21. CYANIDE DERIVATIVES

21.1. PRINCIPLE OF METHODS

(a) **Rapid test method**

Analysis of wines treated with potassium ferrocyanide. Analysis for the absence of ferric ferrocyanide in suspension or in deposit. Analysis for the absence of formation of ferric ferrocyanide by addition of a ferric salt to the acidified wine. Analysis for the presence of iron by precipitation with a mixture of potassium ferrocyanide and ferricyanide added to the acidified wine.

(b) **Usual methods**

Argentometric estimation of the total hydrocyanic acid liberated by acid hydrolysis and distillation. Argentometric estimation of the free hydrocyanic acid distilled in vacuo or entrained in an air stream at ambient temperatures.

Such cyanide derivatives are expressed in milligrams of hydrocyanic acid per litre and determined to the nearest 0·1 mg.

The traces of free hydrocyanic acid and of hydrocyanic acid liberated by distillation in acid medium from various natural wine compounds are determined colorimetrically using a pyridine phenylpyrazolone reagent.

21.2. RAPID TEST METHOD

Analysis of wines treated with potassium ferrocyanide

1. Examination for traces of ferric ferrocyanide in suspension: shake the sample and place 20 ml of wine in a 30 ml concial centrifuge tube. Centrifuge for 15 minutes at 3 500 r/min. The deposit obtained after centrifugation should be completely free of blue particles (1).

2. Examination for trace concentrations of ferrocyanide ions in solution: pipette 20 ml of clear wine, 1 ml of hydrochloric acid (50 % v/v) and then add one drop of ferric ammonium sulphate solution (15 %) to a 30 ml conical centrifuge tube. Stir and allow to stand for at least 24 hours before centrifuging for 15 minutes at 3 500 r/min. The deposit obtained after centrifugation should be completely free of blue ferric ferrocyanide particles.

3. Examination for the presence of iron ions in the wine: pipette 20 ml of wine and 1 ml of 50 % (v/v) (2) hydrochloric acid into a test tube. Add one drop of 10 % potassium ferrocyanide solution and one drop of a freshly prepared 10 % solution of potassium ferricyanide. A blue coloration or a blue precipitate appearing in less than 30 minutes indicates a positive result. Confirmation of a blue precipitate can be obtained by filtration through a fluted filter of 5 cm diameter and rinsing twice with 5 ml of water.

21.3. USUAL METHODS

I. ARGENTOMETRIC DETERMINATION OF THE TOTAL HYDROCYANIC ACID CONTENT

Apparatus

Place a 300 ml round-bottomed flask on an iron sheet of 200 to 300 mm with a hole cut in it of 50 mm diameter. Incline the neck of the flask. Join the flask with ground-glass jointed tube to the upper end of a West condenser (3) which has a cooling jacket of length 350 mm.

Attach to the lower end of this condenser, which is placed vertically, an adapter with a drawn-out portion to conduct the distillate to the bottom of a 50 ml receiver flask. Completely immerse this flask in ice water.

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(1) Centrifuging may be replaced by filtration through iron-free paper (diameter 5 cm) or preferably through a membrane filter (pore diameter: 0·45μ).

(2) Hydrochloric acid (diluted to 50 %) may be easily obtained free of iron by diluting pure commercial hydrochloric acid with an equal volume of water and distilling the solution.

(3) This condenser consists of a thin glass cylinder inside a glass jacket; the internal diameter is only 1 mm greater than the external diameter of the cylinder to be cooled. Thus cold water is circulated rapidly against the wall to be cooled.

Solutions

Sulphuric acid, dilute. Add 200 ml of sulphuric acid to sufficient water to produce 1 000 ml of solution.

Phenol red solution. Dissolve 0·05 g of phenol red in 1·4 ml of a sodium hydroxide solution, 0·1N; make up the solution to 1 000 ml.

Potassium iodide solution. Dissolve 250 g of potassium iodide in water and make up to one litre with water.

Silver nitrate solution, 0·001N. Add 0·5 ml of concentrated nitric acid to 10 ml of 0·1N silver nitrate solution and make up to one litre with water. Sodium hydroxide, 1N — iron-free.

Procedure

Introduce 100 ml of filtered wine (or in the unfiltered state if the determination of hydrocyanic acid contained any blue turbidity is also required), add approximately 0·005 g of cupric chloride and 10 ml of diluted sulphuric acid. Place 5 ml of a normal solution of pure sodium hydroxide, iron-free, into the receiver flask. Distil about 50 ml into the receiver flask.

Transfer the distillate to a 400 ml beaker and place on a boiling waterbath (1); accelerate the evaporation by directing a fairly strong current of cold air produced by a blower across the surface of the alkaline liquid. Reduce the volume to 5 to 7 ml; this will take about 30 minutes (ensure that the volume is never reduced to less than 5 ml).

Filter the cooled solution through a small filter (2), collecting the filtrate in a cylindrical tube of 20 mm diameter and 180 mm length. Wash beaker and filter with a few millilitres of water.

Place the glass tube on a black base and allow a beam of white light to fall on it from the side. The liquid must be perfectly clear (3).

Add two drops of phenol red solution (4) and then one drop of potassium iodide solution to aid observation of the end point. Titrate with 0·001N silver nitrate solution until a slight but persistent turbidity is observed. Let n be the volume of titrant used.

Carry out a blank titration in a similar tube containing 5 ml of normal sodium hydroxide solution, two drops of phenol red (4), one drop of potassium iodide and a quantity of pure water sufficient to obtain an identical volume to that of the above. Add sufficient 0·001N silver nitrate solution to obtain the same turbidity as above. Let the volume be n′ (5).

Calculation

1 ml of the silver nitrate solution 0·001N corresponds to 54μg of hydrocyanic acid; 0·54 (n — n′) mg is therefore the total hydrocyanic acid contained in one litre of wine. Express the result to one decimal place.

Regard as significant only those results where n — n′ is greater than 0·5 ml.

If n — n′ is greater than 20 ml, repeat the procedure using one hundredth normal silver nitrate.

If n — n′ is greater than 10 ml, detect and determine the free hydrocyanic acid by the following process (II).

II. MEASUREMENT OF FREE HYDROCYANIC ACID

Apparatus

The apparatus consists of two one litre flasks A and B connected by a (ground-glass jointed) straight tube of 30 mm diameter and 300 mm length. One of the flasks (A) is provided with a tap which can be connected to a vacuum pump or to which a small funnel can be attached using a plastic connector.

The straight tube connecting the flasks is attached to such apparatus to enable rotation of the assembly at a moderate speed about its axis.

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(1) The waterbath should be electrically heated and not by gas as the combustion product of the latter contains traces of hydrocyanic acid. Tobacco smoke similarly contains hydrocyanic acid and smoking during this evaporation is therefore prohibited.

(2) Filtration is unnecessary if the filtrate is quite clear; it is required if the laboratory air contains dust.

(3) Certain wines, such as liqueur wines, etc., give a distillate which is not clear even after filtration; in such cases the distillate must be placed in a 200 ml distillation flask, made up to 30 ml with distilled water, and distilled while still alkaline, discarding the first 15 ml of the distillate. Cool the contents of the flask, acidify with approximately 5 ml diluted sulphuric acid and resume the distillation, collecting the distillate in 5 ml of a normal solution of sodium hydroxide. Distil approximately 5 ml of the liquid which will then be clear.

(4) This addition is optional. Some analysts consider it easier to observe the appearance of turbidity in a pink solution than in a colourless one.

(5) n′ is equal 0·05 or 0·1 ml if the volume of water used is less than 10 ml. To obtain a discernable end point, the volume used should be as small as possible and hence any dilutions should as far as possible be avoided during the main operation.

Procedure

Introduce 5 ml of sodium hydroxide solution into flask B and 5 ml of water into flask A. Connect the two flasks by ground-glass joint and evacuate the apparatus while slightly heating the two flasks thus evaporating a little water so that the steam produced removes the last traces of air. Close the tap and disconnect the vacuum pump. Dip flask B into cold water, then into ice water. Carefully introduce 100 ml of wine into flask A while avoiding any entry of air.

Immerse flask A in a dish containing warm water, thus making the wine boil. Commence the distillation by slowly turning the apparatus about its axis.

Distil approximately two thirds of the contents of flask A (normally requiring about 20 minutes), then reverse the apparatus and immerse flask B into warm water and flask A into ice water. Redistil in the inverse direction approximately one third of the contents of flask B.

Flask A contains the alcohol, aldehydes, esters and the ferrocyanide ions together with the residue from the wine.

Next carry out the argentometric determination of the hydrocyanide acid in the contents of flask B as specified in section I.

III. DETERMINATION OF TRACES OF TOTAL HYDROCYANIC ACID

Apparatus

The apparatus has been described in section I above.

Solutions

Chloramine T, 1 % in distilled water.

Buffer pH 7·5:

— monopotassium phosphate	94 g,
— sodium hydroxide solution, N	565 ml,
— distilled water to	1 000 ml.

Phospheric acid solution, 1·5N (0·75M). Prepare by dilution 1 : 8 (v/v) of phospheric acid 60 ° Be (d = 1·71) and adjusting to 1·5N by titration with normal sodium hydroxide solution using phenolphthalein as indicator.

Bis (1-phenyl-3-methyl-5-pyrazolone). Dissolve 17·4 g of 1-phenyl-3-methyl-5-pyrazolone in 100 ml of alcohol 95 % v/v, add 25 g of freshly distilled phenylhydrazine and boil under reflux for four hours. Filter the mixture while hot and wash the precipitate several times with alcohol 95 % v/v. The melting point of the product obtained is 320 °C.

Extend the period of boiling under reflux beyond the four hours if insufficient yellow crystals have formed during this time.

Preparation of reagent. Place 150 mg of 1-phenyl-3-methyl-5-pyrazolone in a 100 ml flask and dissolve in 50 ml alcohol 95 % v/v which has been distilled over sodium hydroxide; make up to 100 ml with distilled water. Weigh 20 mg of bis (1-phenyl-3-methyl-5-pyrazolone) and dissolve with continuous stirring in 20 ml of pyridine. Mix the above two solutions by transferring them to a brown glass bottle wrapped in black paper. Keep in refrigerator.

Procedure

Introduce successively into a 300 ml distillation flask 50 ml of wine, 1 to 2 mg of cupric chloride and 5 ml of 0·2N sulphuric acid.

Collect the distillate in a 40 ml flask into which 4 ml of a normal sodium hydroxide solution has been introduced. Distil until 40 ml have been obtained.

Transfer this distillate to a 250 ml beaker, rinsing the receiver flask with 5 ml of distilled water. After noting the liquid level in the beaker, place it on a boiling waterbath, directing a fairly strong current of air across the surface of the liquid. Reduce the volume to 28 to 30 ml in approximately 15 minutes.

Transfer this residue to a 300 ml distillation flask identical with the first flask, rinse the beaker with 5 ml of distilled water and cool the liquid by placing the flask in ice water. Add a small quantity of active starch indicator and 6 to 7 ml of normal sulphuric acid. Oxidize the sulphur dioxide by

adding 0·1N iodine solution until the starch indicator turns blue (1). Decolorize the indicator by adding one or more drops of a 0·01N thiosulphate solution and fit the flask immediately to the distillation apparatus as described in section I.

Distil, slowly at first and then more rapidly, collecting the distillate in a 40 ml flask containing 4 ml of normal sodium hydroxide solution. Stop the distillation when 28 ml of liquid have been collected in the receiver flask.

Introduce into the flask 2 ml of pH 7·5 buffer solution and 3 ml of phosphoric acid 1·5N. Check that the pH is 7·5 using indicator paper and adjust if necessary. Then add 0·3 ml of chloramine T, 1 %, and allow to stand for 90 seconds. Add 6 ml of the pyridinepyrazolone reagent, mix, adjust to 40 ml with distilled water and mix. Determine the optical density after 45 minutes to one hour at 625 nm using either a blank solution or distilled water. Calibrate the apparatus with solutions prepared as above but containing 0·1, 0·2, 0·5 .... 5 µg of hydrocyanic acid in the 40 ml of mixture.

Sensitivities

This method permits the determination of hydrocyanic acid in the range 2 to 300 µg/l of wine.

If a reduced sensitivity is required, 10 or 25 ml of the secondary distillate may be taken and made up to 40 ml. The volume of secondary distillate is placed into another 40 ml flask and adjusted to the same pH by addition of 5 ml of buffer solution and 1·9 ml of phosphoric acid solution if 25 ml has been taken or 0·75 ml of phosphoric acid solution if 10 ml has been taken. Dilute to 40 ml and carry out the remainder of the determination as described above.

When 25 ml of distillate is taken (corresponding to 31·25 ml of wine) the sensitivity range is between 3 and 480 µg of hydrocyanic acid per litre of wine.

When 10 ml of distillate is taken (corresponding to 12·5 ml of wine) the sensitivity range is between 8 and 1 200 µg of hydrocyanic acid per litre of wine.

Regard as significant only those result values greater than 300 µg/l (0·3 mg/l).

IV. DETERMINATION OF TRACES OF FREE HYDROCYANIC ACID

Apparatus

The apparatus, consisting of two one litre flasks connected by a straight tube of 30 mm diameter, has been described in section II.

Procedure

Introduce 4 ml of normal sodium hydroxide solution into flask B and 50 ml of wine into flask A. Carry out the distillation as described in section II and collect the alcohol, aldehyde and esters in flask A.

Dip flask B into ice water for five minutes after dismantling the apparatus. Add 6 ml of normal sulphuric acid and oxidize the sulphur dioxide with iodine (1). Just decolorize the starch indicator with 0·01N thiosulphate solution and distil the hydrocyanic acid in the distillation apparatus under normal pressure as described previously in section I.

The determination of the hydrocyanic acid is carried out as specified above in section III.

(1) In the absence of sulphur dioxide this operation is not required.

22. CARBON DIOXIDE

22.1. PRINCIPLE OF METHODS

(a) **Reference method**

Sulphur dioxide is initially oxidized at low temperature by hydrogen peroxide and a cupric salt; carbon dioxide, released from the wine by a phosphate/phosphoric acid buffer, is entrained by a low temperature current of air produced by a small circulation pump operating in a closed circuit and passed into a standard solution of barium hydroxide. Excess base is then back-titrated against a standard solution of hydrochloric acid using a mixed thymolphthalein/phenolphthalein indicator.

(b) **Routine method**

Wine is agitated inside a bottle of a much larger capacity than the volume of the wine sample taken, thus releasing the bulk of carbon dioxide from the wine until equilibrium between the two phases is established. Measurement of the pressure increase enables an evaluation, sufficient for practical purposes, to be made of the carbon dioxide content of the wine.

22.2. REFERENCE METHOD

Apparatus

Glass apparatus for the extraction of carbon dioxide in closed circuit (fig. 1) consisting of:

— a 250 ml flask (1) fitted with an anti-splash bulb and a burette (5),

— two bubblers (2) and (3),

— a pump (4) ensuring air circulation in the circuit (at a rate of two to 10 bubbles per second).

Reagents

Sodium hydroxide solution, 0·2N.

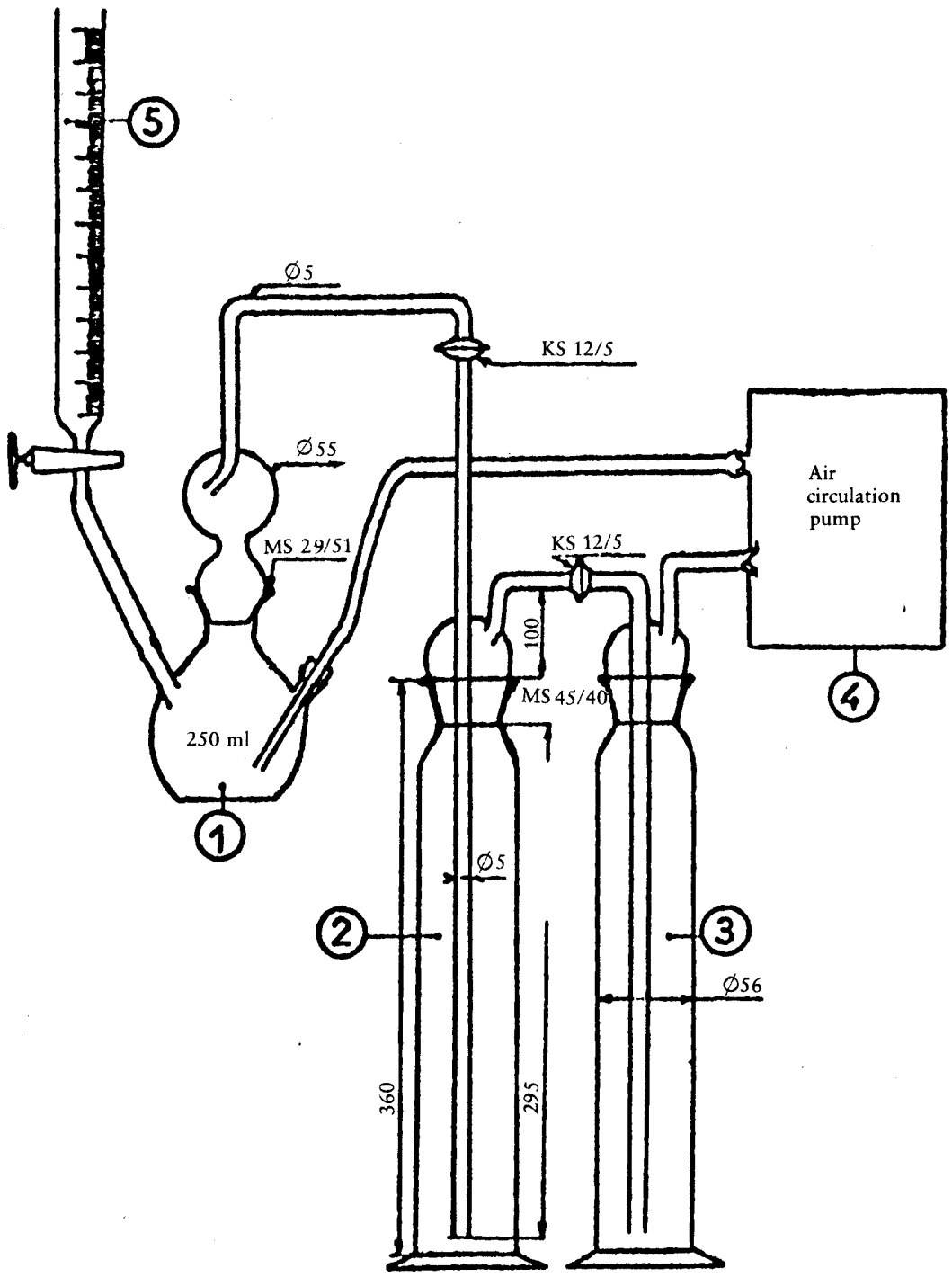
Barium chloride solution, 90 g $BaCl_2 \cdot 2H_2O$ per litre.

Barium hydroxide solution:

— mix two volumes of barium chloride solution at 90 g/l with one volume of sodium hydroxide solution, 0·2N.

Figure 1

Apparatus for the extraction of CO_2 in closed circuit



The quantity of solution used in a determination is 150 ml; it is therefore recommended that a sufficient volume is prepared (five litres for example) and that it is stored in a container fitted with a guard tube to prohibit the entry of carbon dioxide from the air after sampling.

Buffer solution:

— monosodium phosphate $NaH_2PO_4 \cdot 2H_2O$ 400 g,

— phosphoric acid H_3PO_4 (p 20 °C = 1·63) 50 ml,

— boiled distilled water to 1 000 ml.

Sodium hydroxide solution 50 % (w/w):

— aqueous solution (p = 1·525) containing 50 g of sodium hydroxide in 100 g of solution. Use 15 days after preparation thus allowing any sodium carbonate to be deposited. This solution should be clear.

Hydrogen peroxide, 10 vol solution. The solution contains 30·4 g of H_2O_2 per litre; the available oxygen is liberated on catalytic decomposition by MnO_2 in alkaline medium.

Copper sulphate, $CuSO_4 \cdot 5H_2O$, powdered.

Mixed indicator:

— phenolphthalein 1 g,

— thymolphthalein 0·5 g,

— alcohol, 96 % vol to 100 ml.

Hydrochloric acid 0·2N.

Procedure

Wines containing less than 4 g of carbon dioxide per litre.

Place the sample bottle of wine on its side in a refrigerator maintained at 0 °C overnight.

Pipette into the bubbler (2) 150 ml of barium hydroxide solution.

Introduce into the flask (1) a few crystals of copper sulphate and 5 ml of hydrogen peroxide, 10 vol. Using a graduated flask, previously cooled to 0 °C, measure out 50 ml of wine and then carefully introduce the wine into the flask (1). Place the flask in position in the circuit and allow to stand for 10 minutes. Start the pump and introduce into the flask 20 ml of the buffer solution using the burette (5). Continue the pumping for 30 minutes.

Stop the pump and withdraw 50 ml of barium hydroxide solution from the bubbler; titrate against hydrochloric acid 0·2N using the mixed indicator. Let the number of millilitres used be V_2.

It is essential that the titre of 150 ml of the barium hydroxide solution is checked each day of use. Let the number of millilitres of the hydrochloric acid 0·2N required be V_1.

Calculation

CO_2 content of the wine in grams per litre:

$$(V_1 - 3V_2) \cdot 0{\cdot}088$$

Wines containing more than 4 g of carbon dioxide per litre.

Place the sample of wine on its side in a refrigerator maintained at 0 °C overnight. Open the bottle with care. Withdraw 50 ml of liquid and introduce 50 ml of sodium hydroxide 50 % into the bottle by running the solution gently down the wall of the bottle. Reclose the bottle immediately and homogenize the contents by shaking; then place in a waterbath at 20 °C.

Pipette into the bubbler (2) 300 ml of barium hydroxide solution.

Introduce into the flask (1) a few crystals of copper sulphate and 5 ml of hydrogen peroxide, 10 vol.

Carefully pipette 50 ml of wine into the flask. Place the flask in position and allow to stand for 10 minutes.

If the wine contains more than 8 g/l of carbon dioxide reduce the volume of wine taken as the sample to 25 ml.

Start the pump and introduce into the flask (1) 40 ml of buffer solution using the burette (5). Continue the pumping for 30 minutes.

Stop the pump and withdraw 50 ml of barium hydroxide solution from the bubbler (2). Titrate against hydrochloric acid 0·2N using the mixed indicator. Let the number of millilitres used be V_2. Let the number of millilitres of hydrochloric acid 0·2N used for titrating 150 ml barium hydroxide solution be V_1.

Calculation

Measure the volume of liquid remaining in the wine bottle and add to it the volume of the test sample taken for the determination (i.e. 50 or 25 ml). Let the total volume thus determined be V. Caculate the correction factor f required to allow for the 50 ml of the sodium hydroxide solution 50 % introduced into the bottle:

$$f = \frac{V}{V - 50}$$

If the volume of the test sample taken for the determination was 50 ml the carbon dioxide content of the wine, in grams per litre, is given by:

$$(2\ V_1 - 6\ V_2) \cdot 0{\cdot}088 \cdot f$$

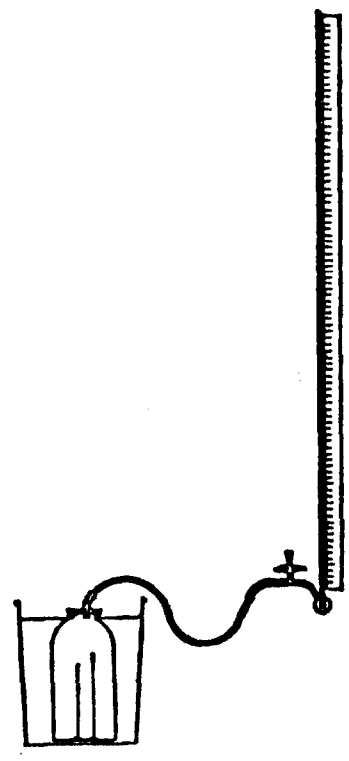
Multiply the result by two if the volume of the test sample taken was 25 ml.

22.3. APPARATUS

1. Water pressure gauge of 1 m length and 1·5 to 2 mm inside diameter with the receiver bulb having a tap so that it can be opened to the atmosphere. The pressure gauge is fitted onto a vertical support and is provided with a movable graduated scale. The receiver bulb contains approximately 3 ml. The zero of the movable, scale is placed on the same level as the water in the pressure gauge stem when the tap is open to the atmosphere.

2. A one litre bottle provided with an internal tube of 30 mm inside diameter and 140 mm height which is sealed to the bottom of the bottle. The bottle is hermetically sealed by a flat screw stopper which is fitted with a tube of 4 mm inside diameter allowing the laboratory bottle to be connected to the pressure gauge with a thick-walled rubber tube of 4 mm inside diameter and 60 cm length. The available capacity of this bottle is 994 ml.

Figure 2



For sparkling and semi-sparkling wines, ciders and beers, use a one litre bottle of the same type but without central tube. The available capacity of this bottle to the stopper is 1 030 ml.

3. An eight to 10 litre vessel with a support thus enabling the one litre bottle to be immersed to its neck. This vessel is filled with water at room temperature. The temperature of the water is measured with a thermometer graduaded in half degrees Celsius.

4. One mark pipettes, of 50, 20, 10 and 1 ml capacity, the delivery tube of which is long enough to reach the bottom of the bottle.

Reagents

Sodium hydroxide solution, 50% w/w, specific gravity 20/20 1·50 to 1·54. Prepare the solution before it is required thus allowing it to clarify (and so be carbonate-free). Mark the bottle which contains this solution with a red 'Poison' label, because the solution is both very toxic and very caustic. When removing 20 or 30 ml of solution (as is required when analyzing samples rich in carbon dioxide) use a pear-shaped suction bulb. When withdrawing a millilitre of sodium hydroxide dip the pipette into the solution. *Never draw off this caustic liquid by mouth.*

Tartaric acid in fairly large crystals of 2 to 4 mm.

Sodium carbonate solution prepared from analytical reagent grade anhydrous sodium carbonate and containing 4·73 g of Na_2CO_3 per litre. This solution gives off its own volume of carbon dioxide if reacted with excess tartaric acid.

Procedure

1. *Still wines* [1]

   Carefully decarbonate 30 to 50 ml of wine sample using a vacuum produced by a water-jet pump for two to three minutes. In the absence of a water-jet pump or a mechanical pump it is sufficient to shake energetically 30 to 50 ml of wine in a one litre bottle for five to 10 seconds.

   Transfer the wine to another bottle of the same capacity and repeat the same operation. The wine will now not contain more carbon dioxide than 2 or 3 ml/l.

   Pour a few millilitres of the decarbonated wine into the laboratory bottle. Shake (for a short time) to distribute the wine over all the internal walls of the bottle; drain quickly, stopper the bottle and place it on the waterbath. After one to two minutes the water level in the pressure gauge will have become steady.

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[1] In summer and with wines relatively rich in carbon dioxide it is advisable to cool the bottle containing the wine sample to 10 °C before opening the bottle to remove the test sample.

Open the tap on the pressure gauge, introduce into the central tube approximately 20 ml of decarbonated wine, and then 50 ml of wine to be analyzed by dipping the pipette to the bottom of the tube thus avoiding rapid delivery. Restopper, close the tap on the pressure gauge and shake vigorously for five to 10 seconds. Stand in a waterbath for one or two minutes and then shake the bottle for two seconds before putting it back on the waterbath. Read the excess pressure h as soon as the water level in the pressure gauge has become steady. Note the temperature t °C of the bath.

Calculation: the height of water in the pressure gauge in millimetres must be multiplied by the factor F to give the quantity of carbon dioxide in millilitres of this gas per litre of wine.

This factor F varies between 1·7 and 1·9 according to the temperature and the capacity of the laboratory bottle. It is given in column 2 of Table I.

To express the content in milligrams per litre of wine, multiply this result by 1·977.

2. *Sparkling and semi-sparkling wines, beer, etc.*

Before commencing, mark on the neck of the bottle of sparkling wine the level of wine in the bottle. Cool the full and stoppered bottle to — 4 °C. Immediately after unstoppering withdraw 30 ml (1) of wine (assume the bottle to contain approximately 800 ml of wine). Immediately introduce 30 ml (1) of sodium hydroxide solution 50 %. Restopper ensuring that the air pocket is not greater than 3 ml. Immediately shake and then bring the bottle to room temperature.

Introduce 1 ml of sodium hydroxide solution 50 % and 10 ml of alkalized wine (case of sparkling wine) or 20 ml of alkalized wine (case of semi-sparkling wine) into the laboratory bottle without central tube. Stopper the bottle and shake to saturate the atmosphere with water and alcohol. Place the bottle in the waterbath and leave for two to three minutes.

Add 4 g of tartaric acid into the bottle, stopper it immediately and close the tap on the pressure gauge. Shake the bottle vigorously to mix the wine with the tartaric acid. The wine will become clear as carbon dioxide is released and the water then rises in the pressure gauge. Place the bottle for one to two minutes in the waterbath, shake it again, replace it in the waterbath and note the excess pressure h on the pressure gauge as soon as the level has become steady. Note the temperature, t °C, of the waterbath.

Determine accurately the quantity Q ml of the wine contained originally in the bottle from the mark inscribed on the neck of the bottle prior to any withdrawal.

Calculations: the quantity of carbon dioxide initially contained in the wine is given by the formula:

$$X = \frac{Q}{Q - 30\ (1)} \cdot F \cdot h \text{ ml } CO_2 \text{ per litre of wine}$$

where:

Q = the volume of wine in the sample before analysis,

F = the factor given in Table I.

This process can be used for evaluating the total carbon dioxide in water, mineral waters and aerated waters.

If the available bottle does not have the capacity given in Table I, the factor F can be calculated by the formula:

$$F = \left(BV + \frac{A}{1 + \alpha t}\right) \frac{1\,000}{10\,350\ V}$$

where:

B = the coefficient of solubility of carbon dioxide in the wine or in the water as given in Table II,

V = the volume of wine introduced into the bottle.

(1) Volume reduced to 20 ml for sparkling wines (30 ml for semi-sparkling wines).

TABLE I

Factors F

Method	1°	2°	2°	3°
Bottle	with central tube	without central tube	without central tube	without central tube
Capacity of bottle A + V ml	994 ml	1 030 ml	1 030 ml	1 030 ml
Nature of liquid analyzed	still wine	semi-sparkling wine	sparkling wine cider, beer, aerated waters	non-aerated waters
Volume of liquid introduced V ml	50 ml	10 ml	20 ml	100 ml
10 °C	1·86	9·55	4·77	0·982
11 °C	1·86	9·51	4·75	0·975
12 °C	1·85	9·48	4·73	0·969
13 °C	1·84	9·45	4·72	0·963
14 °C	1·83	9·42	4·70	0·956
15 °C	1·82	9·39	4·68	0·950
16 °C	1·81	9·35	4·67	0·943
17 °C	1·80	9·32	4·65	0·938
18 °C	1·79	9·29	4·64	0·933
19 °C	1·78	9·26	4·62	0·927
20 °C	1·77	9·23	4·61	0·922
21 °C	1·77	9·20	4·60	0·917
22 °C	1·76	9·17	4·58	0·913
23 °C	1·75	9·14	4·56	0·906
24 °C	1·74	9·11	4·55	0·901
25 °C	1·73	9·08	4·53	0·897
26 °C	1·73	9·04	4·51	0·892
27 °C	1·72	9·01	4·50	0·887
28 °C	1·71	8·98	4·49	0·882
29 °C	1·70	8·95	4·47	0·878
30 °C	1·70	8·92	4·46	0·874

A = the available volume of the bottle less V and, for sparkling wines, less 1 ml of sodium hydroxide 50 % and 2·5 ml, i.e. the volume of 4 g of tartaric acid.

The ratio $\frac{1}{1 + \alpha t}$ is given in Table II (column 2).

Note:

This is the formula by which the values included in Table I have been calculated which are valid for water as well as for wines of 10 to 14 °, still dry wines or still wines containing less than 50 g of sugar per litre, as well as for sparkling wines, semi-sparkling wines and ciders, beers, aerated waters, etc.

N.B.

After each determination renew the atmosphere in the laboratory bottle by filling completely with water and driving out any carbon dioxide that may be present.

TABLE II

Coefficient of solubility B of CO_2 in litres of CO_2 at 0 °C and 760 mm Hg per litre of water or water-alcohol mixtures at t °C

t °C	$\frac{1}{1 + \alpha t}$	B (1) water	B (2) 10 % vol	B (2) 12 % vol	B (2) 14 % vol	B (2) 10 % vol + 50 g sugar/litre
10	0·965	1·194	1·10	1·08	1·05	1·03
11	0·961	1·154	1·06	1·04	1·02	1·00
12	0·958	1·117	1·02	1·00	0·98	0·96
13	0·955	1·083	0·99	0·97	0·95	0·92
14	0·951	1·050	0·95	0·93	0·91	0·89
15	0·948	1·019	0·92	0·90	0·88	0·85
16	0·944	0·985	0·88	0·86	0·84	0·82
17	0·941	0·956	0·85	0·83	0·81	0·79
18	0·938	0·928	0·82	0·80	0·78	0·76
19	0·935	0·902	0·79	0·77	0·75	0·73
20	0·932	0·878	0·76	0·74	0·72	0·71
21	0·929	0·847	0·74	0·72	0·70	0·68
22	0·926	0·820	0·71	0·69	0·66	0·66
23	0·923	0·794	0·69	0·67	0·64	0·63
24	0·920	0·770	0·66	0·64	0·62	0·61
25	0·917	0·759	0·64	0·62	0·60	0·59
26	0·913	0·738	0·62	0·60	0·58	0·57
27	0·910	0·718	0·60	0·58	0·56	0·55
28	0·907	0·700	0·58	0·56	0·54	0·53
29	0·904	0·685	0·56	0·54	0·52	0·51
30	0·901	0·665	0·55	0·53	0·51	0·50

(1) From the table of BOHR.
(2) From the table of AGABALIANTZ.

Checking the method

Before commencing any analyses test the method using the standard solution of sodium carbonate which when reacted with excess tartaric acid releases its volume of carbon dioxide.

Accuracy of the method

This method produces results to an accuracy of 3 to 5 %. This, in practice, is sufficient as carbon dioxide is lost very easily by the wine during the course of decanting, drawing off, etc.

Relation between the pressure and the quantity of carbon dioxide contained in a sparkling wine

The quantity of carbon dioxide contained in a sparkling wine is given by the following formulae:

— in litres of carbon dioxide per litre of wine: $(1 + 0{\cdot}987\ P)\ (0{\cdot}86 - 0{\cdot}01\ A)\ (1 - 0{\cdot}00144\ s)$,

— in grams of carbon dioxide per litre of wine: $1{\cdot}977\ (1 + 0{\cdot}987\ P)\ (0{\cdot}86 - 0{\cdot}01\ A)\ (1 - 0{\cdot}00144\ s)$;

where:

P = the excess pressure of carbon dioxide in the vessel expressed in bars at 20 °C,

A = the alcoholometric titre at 20 °C of the wine,

s = the sugar content of the wine in grams per litre.

Calculation

Let the excess pressure, expressed in bars, determined at t °C by means of an aphrometer, be P_t. To obtain the absolute pressure at t °C, that is Pa_t, expressed in atmospheres, multiply P_t by 0·987 (1 bar = 0·987 atmosphere), and add one.

Calculate the absolute pressure at 20 °C, Pa_{20} expressed in atmospheres from the table given below.

Pa_{20} is related to P, the excess pressure at 20 °C expressed in bars, by the relationship $Pa_{20} = 1 + 0{\cdot}987\ P$, from which P can be then calculated.

The quantity of carbon dioxide contained in a sparkling wine is given by the following formulae:

— in litres of carbon dioxide per litre of water: Pa_{20} (0·86 — 0·01 A) (1 — 0·00144 s) or (1 + 0·987 P) (0·86 — 0·01 A) (1 — 0·00144 s),

— in grams of carbon dioxide per litre of wine: 1·977 Pa_{20} (0·86 — 0·01 A) (1 — 0·00144 s) or 1·977 (1 + 0·987 P) (0·86 — 0·01 A) (1 — 0·00144 s).

Notes:

1. If the aphrometer is graduated in absolute pressure and in atmospheres, multiply the pressure as measured by the coefficient $\frac{Pa_{20}}{Pa_t}$ given by the table below to relate it to 20 °C.

2. If the aphrometer is graduated in excess pressure expressed in atmospheres, add one to the measured value before the correction is made.

   In both cases, having obtained Pa_{20}, one can calculate P (the excess pressure in bars at 20 °C) and the quantity of carbon dioxide contained in the wine either in litres of carbon dioxide or in grams of carbon dioxide per litre of wine.

Relation of the absolute pressure Pa_{20} of a sparkling wine at 20 °C to the absolute pressure Pa_t at t °C

t °C	$\frac{Pa_{20}}{Pa_t}$	t °C	$\frac{Pa_{20}}{Pa_t}$	t °C	$\frac{Pa_{20}}{Pa_t}$
— 5	2·44	9	1·50	23	0·90
— 4	2·36	10	1·44	24	0·87
— 3	2·29	11	1·39	25	0·84
— 2	2·21	12	1·34	26	0·81
— 1	2·14	13	1·29	27	0·78
0	2·07	14	1·24	28	0·75
1	2·00	15	1·20	29	0·72
2	1·93	16	1·16	30	0·70
3	1·86	17	1·11	31	0·68
4	1·80	18	1·07	32	0·66
5	1·73	19	1·03	33	0·64
6	1·67	20	1·00	34	0·62
7	1·61	21	0·97	35	0·60
8	1·55	22	0·93		

Note:

The above expressions are suitable for alcohol titres within the range 0 to 100 g/l. As wines do not have a constant composition, these expressions are only accurate to within 2 to 3 %.

23. ALLYL ISOTHIOCYANATE

23.1. PRINCIPLE OF THE SINGLE METHOD

Allyl isothiocyanate present in wine is collected by distillation and identified by gas chromatography.

23.2. REAGENTS

1. Ethanol, analytical reagent grade.
2. Standard solution: alcoholic solution of allyl isothiocyanate containing 15 mg/l.
3. Freezing mixture consisting of ethanol and dry ice (temperature — 60 °C).

23.3. APPARATUS

1. Distillation apparatus as shown in the figure on page 86. A stream of nitrogen is passed continuously through the apparatus.
2. Heating mantle, thermostatically controlled.
3. Flowmeter.
4. Gaschromatograph fitted with a flame spectrophotometric detector equipped with a selective filter for sulphur compounds (= 394 nm).
5. Steel chromatographic columns with internal diameters of 3 mm and 3 m long filled with carbowax 20 M at 10 % on chronosorb WHP 80 to 100 mesh.
6. Microsyringes, 10 μl.

23.4. METHOD

Put two litres of wine into the distillation flask, introduce a few millilitres of ethanol into the two collecting tubes so that the porous parts of the gas dispersion rods are completely immersed. Cool the two tubes externally with the freezing mixture. Connect the flask to the collecting tubes and begin to flush the apparatus with nitrogen at a rate of three litres per hour. Heat the wine to 80 °C with the heating mantle, distil and collect 40 to 50 ml of distillate.

Preparation of the gas chromatograph

It is recommended that the following conditions are used:

— injector temperature: 200 °C,

— column temperature: 130 °C,

— helium gas carrier flow-rate: 20 ml per minute.

Introduce with a microsyringe a volume of the standard solution such that the peak corresponding to the allyl isothiocyanate can be easily identified on the gas chromatogram.

Introduce an aliquot of the distillate into the chromatograph. A peak with the same retention time as that of the allyl isothiocyanate in the sample.

Under the conditions described above compounds naturally present in the wine will not produce interfering peaks on the chromatogram of the sample solution.

Figure

Apparatus for distillation under a current of nitrogen

