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►B ►C1 **FIRST COMMISSION DIRECTIVE**

of 26 July 1979

laying down Community methods of analysis for testing certain sugars intended for human consumption

(79/796/EEC) ◄

(OJ L 239, 22.9.1979, p. 24)

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FIRST COMMISSION DIRECTIVE

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(79/796/EEC)

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Article 1

1. Member States shall require that the analyses necessary for verification of the criteria set out in Annex I be performed according to the methods described in Annex II to this Directive.

2. Without prejudice to the second subparagraph, the Luff-Schoorl method (Annex II, method 6) shall be used to determine the reducing sugars in the following sugars:

— sugar solution,

— white sugar solution,

— invert sugar solution,

— white invert sugar solution,

— invert sugar syrup,

— glucose syrup,

— dried glucose syrup,

— dextrose monohydrate,

— dextrose anhydrous.

Member States may, however, require the use in their territory of the Lane and Eynon method (Annex II, methods 7 and/or 8 as appropriate) to determine the reducing sugars in one or more of the sugars listed above.

3. If a Member State makes use of the option provided for in the second subparagraph of paragraph 2, it shall forthwith inform the Commission and the other Member States thereof.

Article 2

Member States shall bring into force the laws, regulations or administrative provisions necessary to comply with this Directive not later than 18 months following its notification. They shall forthwith inform the Commission thereof.

Article 3

This Directive is addressed to the Member States.

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ANNEX I

SCOPE OF TOE COMMUNITY METHODS OF ANALYSIS FOR CERTAIN SUGARS INTENDED FOR HUMAN CONSUMPTION

I. Determination of the loss of mass on drying in:

— semi-white sugar — sugar or white sugar — extra-white sugar	(using method 1, Annex II)

II. Dry matter determination in:

II.1.	— glucose syrup — dried glucose syrup — dextrose monohydrate — dextrose anhydrous	(using method 2, Annex II)
II.2.	— sugar solution or white sugar solution — invert sugar solution or white invert sugar solution — invert sugar syrup or white invert sugar syrup	(using method 3, Annex II)

III. Measurement of reducing sugars in:

III.1.	— semi-white sugar	(using method 4, Annex II)
III.2.	— sugar or white sugar — extra-white sugar	(using method 5, Annex II)
III.3.	— sugar solution — white sugar solution — invert sugar solution — white invert sugar solution — invert sugar syrup — white invert sugar syrup	(using method 6 or 7, Annex II)
III.4.	— glucose syrup — dried glucose syrup — dextrose monohydrate — dextrose anhydrous	(using method 6 or 8, Annex II)

IV. Sulphated ash determination in:

— glucose syrup — dried glucose syrup — dextrose monohydrate — dextrose anhydrous	(using method 9, Annex II)

V. Determination of polarization in:

— semi-white sugar — sugar or white sugar — extra-white sugar	(using method 10, Annex II)

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ANNEX II

METHODS OF ANALYSIS TO VERIFY THE COMPOSITION OF CERTAIN SUGARS INTENDED FOR HUMAN CONSUMPTION

INTRODUCTION

1. **Preparation of the sample for analysis**

Thoroughly mix the sample received at the laboratory.

Remove a sub-sample of at least 200 g and transfer immediately to a clean, dry, moisture-tight vessel fitted with an airtight closure.

2. **Reagents and apparatus**

In the description of the apparatus, reference is made only to special instruments and apparatus or to those calling for special standards.

Wherever mention is made of water, this means distilled water or demineralized water of at least equivalent purity.

All reagents shall be of analytical reagent quality unless otherwise specified.

Wherever reference is made to a reagent solution without further qualification, an aqueous solution is meant.

3. **Expression of results**

The result referred to in the official analysis report shall be the mean value of at least two satisfactory replicate determinations.

Unless otherwise stated the results shall be expressed as a percentage by mass of the original sample as received at the laboratory.

The number of significant figures in the result so expressed shall be governed by the precision of the method.

METHOD 1

DETERMINATION OF THE LOSS OF MASS ON DRYING

1. **Scope and field of application**

The method determines the loss of mass on drying in:

— semi-white sugar,

— sugar or white sugar,

— extra-white sugar.

2. **Definition**

‘Loss of mass on drying’: the value of the loss of mass on drying as determined by the method specified.

3. **Principle**

The loss of mass on drying is determined by drying at a temperature of 103 ± 2 °C.

4. **Apparatus**

4.1. *Analytical balance,* accurate to within 0.1 mg.

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4.2. Oven, suitably ventilated, thermostatically controlled, and capable of being maintained at 103 ± 2 °C.

4.3. *Metal weighing dish,* flat-bottomed, resistant to attack by the samples and the conditions of test, diameter at least 100 mm, depth at least 30 mm.

4.4. *Desiccator,* containing freshly activated silica gel or an equivalent desiccant, with a water content indicator.

5. **Procedure**

N.B.: The operations described in sections 5.3 to 5.7 must be performed immediately after opening the sample container.

5.1. Dry the dish (4.3) to constant weight in the oven (4.2) at 103 ± 2 °C.

5.2. Allow the dish to cool in the desiccator (4.4) for at least 30 to 35 minutes and then weigh to the nearest 0.1 mg.

5.3. Weigh accurately, to the nearest 0.1 mg, approximately 20 to 30 g of the sample into the dish.

5.4. Place the dish in the oven (4.2) at 103 ± 2 °C for three hours.

5.5. Allow the dish to cool in a desiccator (4.4) and weigh to the nearest 0.1 mg.

5.6. Replace the dish in the oven at 103 ± 2 °C for 30 minutes.

Allow to cool in the desiccator (4.4) and weigh to the nearest 01 mg. Repeat this operation if the difference between two weighings is more than 1 mg. Should an increase in mass occur, the lowest recorded reading will be used in the calculation.

5.7. Do not exceed four hours total drying time.

6. **Expression of results**

6.1. *Formula and method of calculation*

The loss of mass on drying, as a percentage by mass of the sample, is given by the following formula:

$$\frac{(m_0 - m_1)}{m_0} \times 100$$

where:

m_0 is the initial mass, in grams, of the test portion,

m_1 is the mass, in grams, of the test portion after drying.

6.2. *Repeatability*

The difference between the results of two determinations when carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 0·02 g per 100 g of sample.

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METHOD 2

DETERMINATION OF DRY MATTER

Vacuum oven method

1. **Scope and field of application**

The method determines the dry matter content in:

— glucose syrup,

— dried glucose syrup,

— dextrose monohydrate,

— dextrose anhydrous.

2. **Definition**

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

3. **Principle**

The dry matter is determined at a temperature of 70 ± 1 °C using a vacuum oven at a pressure not exceeding 3·3 kPa (34 mbar). The test portions in the case of glucose syrup or dried glucose syrups, are prepared by mixing with water and kieselguhr before drying.

4. **Reagents**

4.1. *Kieselguhr:* place in a Buchner funnel and purify by repeated washings with dilute hydrochloric acid (1 ml of concentrated acid, density at 20 °C = 1·19 g/ml per litre of water). The treatment is complete when the washings remain definitely acid. Wash with water until the pH value of the filtered water is greater than 4. Dry in an oven at 103 ± 2 °C and store in an airtight container.

5. **Apparatus**

5.1. *Vacuum drying oven,* leak tight, thermostatically controlled and equipped with a thermometer and a vacuum manometer. The oven design must be such that the heat is rapidly transferred to the weighing dishes placed on the shelves.

5.2. *Air-drying train* consisting of a glass tower filled with freshly activated dry silica gel or an equivalent desiccant containing a water content indicator. This tower is mounted in series with a gas scrubber containing concentrated sulphuric acid connected to the air intake of the oven.

5.3. *Vaccum pump* capable of maintaining the presure in the oven at 3·3 kPa (34 mbar) or less.

5.4. *Metal weighing dish,* flat-bottomed, resistant to attack by the samples and the conditions of test, diameter at least 100 mm, depth at least 300 mm.

5.5. *Glass rod* of a length such that it cannot completely fall into the container.

5.6. *Desiccator* containing freshly activated dry silica gel, or an equivalent desiccant, with a water content indicator.

5.7. *Analytical balance* accurate to within 0.1 mg.

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6. **Procedure**

6.1. Pour approximately 30 g of kieselguhr (4.1) into the weighing dish (5.4) equipped with a glass rod (5.5). Place the whole in the oven (5.1) at 70 ± 1 °C and reduce the pressure to 3·3 kPa (34 mbar) or less.

Dry for at least five hours, drawing a slow stream of air into the oven through the drying train. Check the pressure from time to time and correct it if necessary.

6.2. Restore atmospheric pressure in the oven by cautiously increasing the intake of dry air. Immediately place the dish together with the glass rod in the desiccator (5.6). Allow to cool and then weigh.

63. Accurately weigh to the nearest 1 mg approximately 10 g of the sample to be analyzed into a 100 ml beaker.

6.4. Dilute the test portion with 10 ml of warm water and transfer the solution quantitatively into the weighing dish, using the glass rod (5.5).

6.5. Place the dish containing the test portion and the glass rod in the oven and reduce the pressure to 3.3 kPa (34 mbar) or less. Dry at 70 ± 1 °C, allowing a slow stream of dry air to pass through the oven.

The drying operation should proceed for 20 hours; the bulk of the loss should occur towards the end of the first day. It will be necessary to keep the vacuum pump working at a preset pressure and allow a slow stream of dry air to enter the oven so as to maintain a pressure of approximately 3·3 kPa (34 mbar) or less during the night.

6.6. Restore atmospheric pressure in the oven by cautiously increasing the intake of dry air. Immediately place the weighing dish and contents in the desiccator. Allow to cool and then weigh to the nearest 1 mg.

6.7. Continue operation (6.5) for a further four hours. Restore atmospheric pressure in the oven and immediately place the dish in the desiccator. Allow to cool and then weigh. Ascertain whether constant mass has been reached. It is considered that constant mass has been satisfactorily attained if the difference between the two weighings of the same dish does not exceed 2 mg. If the difference is greater, repeat operation 6.7.

6.8. For the determination of the dry matter in dextrose anhydrous or dextrose monohydrate samples the use of kieselguhr and water is not required.

7. **Expression of results**

7.1. *Formula and method of calculation*

The dry matter content, expressed as a percentage by mass of the sample is given by:

$$(m_1 - m_2) \times \frac{100}{m_0}$$

where:

m_0 = the initial mass, in grams, of the test portion,

m_1 = the mass, in grams, of the weighing dish plus the kieselguhr, the glass rod and the residue of the test portion after drying,

m_2 = the mass, in grams, of the weighing dish plus the kieselguhr and the glass rod.

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7.2. *Repeatability*

The difference between the results of two determinations when carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 0·12 g per 100 g of sample.

METHOD 3

DETERMINATION OF TOTAL DRY MATTER

(Refractometric method)

1. **Scope and field of application**

The method determines the dry-matter content in:

— sugar solution,

— white sugar solution,

— invert sugar solution,

— white invert sugar solution,

— invert sugar syrup,

— white invert sugar syrup.

2. **Definition**

'Dry matter content': the content of dry matter as determined by the method specified.

3. **Principle**

The refractive index of a test portion is determined at 20 °C and converted into dry matter content by reference to tables showing the concentration as a function of the refractive index.

4. **Apparatus**

4.1. *Refractometer,* accurate to four decimal places, provided with a thermometer and a water-circulation pump connected to a water-bath thermostatically controlled at 20 ± 0·5 °C.

4.2. *Light source* consisting of a sodium vapour lamp.

5. **Procedure**

5.1. If any crystals are present in the sample, redissolve them by diluting the sample in the ratio 1 : 1 (m/m).

5.2. Measure the refractive index of the sample at 20 °C in the refractometer (4.1).

6. **Expression and calculation of results**

6.1. Calculate the dry matter content from the refractive indices for sucrose solutions at 20 °C in the table given and correct for the presence of invert sugars by adding to the result obtained from the tables, 0.022 for every 1 % of invert sugar present in the sample as analyzed.

6.2. If the sample was diluted to 1: 1 (m/m) with water, the calculated dry matter content must be multiplied by two.

6.3. *Repeatability*

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

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REFERENCE TABLES

Refractive indices *(n)* of sucrose solutions at 20 °C [1]

n (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)
1·3330	0·009	1·3375	3·122	1·3420	6·165	1·3465	9·139	1·3510	12·048
1·3331	0·078	1·3376	3·190	1·3421	6·231	1·3466	9·205	1·3511	12·112
1·3332	0·149	1·3377	3·259	1·3422	6·298	1·3467	9·270	1·3512	12·176
1·3333	0·218	1·3378	3·327	1·3423	6·365	1·3468	9·335	1·3513	12·240
1·3334	0·288	1·3379	3·395	1·3424	6·432	1·3469	9·400	1·3514	12·304
1·3335	0·358	1·3380	3·463	1·3425	6·498	1·3470	9·466	1·3515	12·368
1·3336	0·428	1·3381	3·532	1·3426	6·565	1·3471	9·531	1·3516	12·431
1·3337	0·498	1·3382	3·600	1·3427	6·632	1·3472	9·596	1·3517	12·495
1·3338	0·567	1·3383	3·668	1·3428	6·698	1·3473	9·661	1·3518	12·559
1·3339	0·637	1·3384	3·736	1·3429	6·765	1·3474	9·726	1·3519	12·623
1·3340	0·707	1·3385	3·804	1·3430	6·831	1·3475	9·791	1·3520	12·686
1·3341	0·776	1·3386	3·872	1·3431	6·898	1·3476	9·856	1·3521	12·750
1·3342	0·846	1·3387	3·940	1·3432	6·964	1·3477	9·921	1·3522	12·813
1·3343	0·915	1·3388	4·008	1·3433	7·031	1·3478	9·986	1·3523	12·877
1·3344	0·985	1·3389	4·076	1·3434	7·097	1·3479	10·051	1·3524	12·940
1·3345	1·054	1·3390	4·144	1·3435	7·164[2]	1·3480	10·116	1·3525	13·004
1·3346	1·124	1·3391	4·212	1·3436	7·230	1·3481	10·181	1·3526	13·067
1·3347	1·193	1·3392	4·279	1·3437	7·296	1·3482	10·246	1·3527	13·131
1·3348	1·263	1·3393	4·347	1·3438	7·362	1·3483	10·311	1·3528	13·194
1·3349	1·332	1·3394	4·415	1·3439	7·429	1·3484	10·375	1·3529	13·258
1·3350	1·401	1·3395	4·483	1·3440	7·495	1·3485	10·440	1·3530	13·321
1·3351	1·470	1·3396	4·550	1·3441	7·561	1·3486	10·505	1·3531	13·384
1·3352	1·540	1·3397	4·618	1·3442	7·627	1·3487	10·570	1·3532	13·448
1·3353	1·609	1·3398	4·686	1·3443	7·693	1·3488	10·634	1·3533	13·511
1·3354	1·678	1·3399	4·753	1·3444	7·759	1·3489	10·699	1·3534	13·574
1·3355	1·747	1·3400	4·821	1·3445	7·825	1·3490	10·763	1·3535	13·637
1·3356	1·816	1·3401	4·888	1·3446	7·891	1·3491	10·828	1·3536	13·700
1·3357	1·885	1·3402	4·956	1·3447	7·957	1·3492	10·892	1·3537	13·763
1·3358	1·954	1·3403	5·023	1·3448	8·023	1·3493	10·957	1·3538	13·826
1·3359	2·023	1·3404	5·091	1·3449	8·089	1·3494	11·021	1·3539	13·890
1·3360	2·092	1·3405	5·158	1·3450	8·155	1·3495	11·086	1·3540	13·953
1·3361	2·161	1·3406	5·225	1·3451	8·221	1·3496	11·150	1·3541	14·016
1·3362	2·230	1·3407	5·293	1·3452	8·287	1·3497	11·215	1·3542	14·079
1·3363	2·299	1·3408	5·360	1·3453	8·352	1·3498	11·279	1·3543	14·141
1·3364	2·367	1·3409	5·427	1·3454	8·418	1·3499	11·343	1·3544	14·204
1·3365	2·436	1·3410	5·494	1·3455	8·484	1·3500	11·407	1·3545	14·267
1·3366	2·505	1·3411	5·562	1·3456	8·550	1·3501	11·472	1·3546	14·330
1·3367	2·574	1·3412	5·629	1·3457	8·615	1·3502	11·536	1·3547	14·393
1·3368	2·642	1·3413	5·696	1·3458	8·681	1·3503	11·600	1·3548	14·456
1·3369	2·711	1·3414	5·763	1·3459	8·746	1·3504	11·664	1·3549	14·518
1·3370	2·779	1·3415	5·830	1·3460	8·812	1·3505	11·728	1·3550	14·581
1·3371	2·848	1·3416	5·897	1·3461	8·878	1·3506	11·792	1·3551	14·644
1·3372	2·917	1·3417	5·964	1·3462	8·943	1·3507	11·856	1·3552	14·707
1·3373	2·985	1·3418	6·031	1·3463	9·008	1·3508	11·920	1·3553	14·769
1·3374	3·053	1·3419	6·098	1·3464	9·074	1·3509	11·984	1·3554	14·832

[1] *n* values in these tables are calculated from the equation developed by K. Rosenhauer for ICUMSA, programed and computed by Frank G. Carpenter of UDSA, and published in *Sugar J.* 33, 15-22 (June 1970). Refractive index was measured at 20 °C with 0 line of Na. Brix (% sucrose by weight) was obtained by weighing at 20 °C in air at 760 Torr (mm Hg) pressure and 50 % relative humidity. It replaces the previous table, 47.012, 11th edition, taken from *Intern. Sugar J.* 39, 22s (1937).

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n (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)
1·3555	14·894	1·3610	18·290	1·3665	21·599	1·3720	24·825	1·3775	27·971
1·3556	14·957	1·3611	18·351	1·3666	21·658	1·3721	24·883	1·3776	28·028
1·3557	15·019	1·3612	18·412	1·3667	21·718	1·3722	24·941	1·3777	28·084
1·3558	15·082	1·3613	18·473	1·3668	21·777	1·3723	24·998	1·3778	28·141
1·3559	15·144	1·3614	18·534	1·3669	21·836	1·3724	25·056	1·3779	28·197
1·3560	15·207	1·3615	18·595	1·3670	21·896	1·3725	25·114	1·3780	28·253
1·3561	15·269	1·3616	18·655	1·3671	21·955	1·3726	25·172	1·3781	28·310
1·3562	15·332	1·3617	18·716	1·3672	22·014	1·3727	25·230	1·3782	28·366
1·3563	15·394	1·3618	18·777	1·3673	22·073	1·3728	25·287	1·3783	28·422
1·3564	15·456	1·3619	18·837	1·3674	22·132	1·3729	25·345	1·3784	28·479
1·3565	15·518	1·3620	18·898	1·3675	22·192	1·3730	25·403	1·3785	28·535
1·3566	15·581	1·3621	18·959	1·3676	22·251	1·3731	25·460	1·3786	28·591
1·3567	15·643	1·3622	19·019	1·3677	22·310	1·3732	25·518	1·3787	28·648
1·3568	15·705	1·3623	19·080	1·3678	22·369	1·3733	25·576	1·3788	28·704
1·3569	15·767	1·3624	19·141	1·3679	22·428	1·3734	25·633	1·3789	28·760
1·3570	15·829	1·3625	19·201	1·3680	22·487	1·3735	25·691	1·3790	28·816
1·3571	15·891	1·3626	19·262	1·3681	22·546	1·3736	25·748	1·3791	28·872
1·3572	15·953	1·3627	19·322	1·3682	22·605	1·3737	25·806	1·3792	28·928
1·3573	16016	1·3628	19·382	1·3683	22·664	1·3738	25·863	1·3793	28·984
1·3574	16·078	1·3629	19·443	1·3684	22·723	1·3739	25·921	1·3794	29·040
1·3575	16·140	1·3630	19·503	1·3685	22·781	1·3740	25·978	1·3795	29·096
1·3576	16·201	1·3631	19·564	1·3686	22·840	1·3741	26·035	1·3796	29·152
1·3577	16·263	1·3632	19·624	1·3687	22·899	1·3742	26·093	1·3797	29·208
1·3578	16·325	1·3633	19·684	1·3688	22·958	1·3743	26·150	1·3798	29·264
1·3579	16·387	1·3634	19·745	1·3689	23·017	1·3744	26·207	1·3799	29·320
1·3580	16·449	1·3635	19·805	1·3690	23·075	1·3745	26·265	1·3800	29·376
1·3581	16·511	1·3636	19·865	1·3691	23·134	1·3746	26·322	1·3801	29·432
1·3582	16·573	1·3637	19·925	1·3692	23·193	1·3747	26·379	1·3802	29·488
1·3583	16·634	1·3638	19·985	1·3693	23·251	1·3748	26·436	1·3803	29·544
1·3584	16·696	1·3639	20·045	1·3694	23·310	1·3749	26·493	1·3804	29·600
1·3585	16·758	1·3640	20·106	1·3695	23·369	1·3750	26·551	1·3805	29·655
1·3586	16·819	1·3641	20·166	1·3696	23·427	1·3751	26·608	1·3806	29·711
1·3587	16·881	1·3642	20·226	1·3697	23·486	1·3752	26·665	1·3807	29·767
1·3588	16·943	1·3643	20·286	1·3698	23·544	1·3753	26·722	1·3808	29·823
1·3589	17·004	1·3644	20·346	1·3699	23·603	1·3754	26·779	1·3809	29·878
1·3590	17·066	1·3645	20·406	1·3700	23·661	1·3755	26·836	1·3810	29·934
1·3591	17·127	1·3646	20·466	1·3701	23·720	1·3756	26·893	1·3811	29·989
1·3592	17·189	1·3647	20·525	1·3702	23·778	1·3757	26·950	1·3812	30·045
1·3593	17·250	1·3648	20·585	1·3703	23·836	1·3758	27·007	1·3813	30·101
1·3594	17·311	1·3649	20·645	1·3704	23·895	1·3759	27·064	1·3814	30·156
1·3595	17·373	1·3650	20·705	1·3705	23·953	1·3760	27·121	1·3815	30·212
1·3596	17·434	1·3651	20·765	1·3706	24·011	1·3761	27·178	1·3816	30·267
1·3597	17·496	1·3652	20·825	1·3707	24·070	1·3762	27·234	1·3817	30·323
1·3598	17·557	1·3653	20·884	1·3708	24·128	1·3763	27·291	1·3818	30·378
1·3599	17·618	1·3654	20·944	1·3709	24·186	1·3764	27·348	1·3819	30·434
1·3600	17·679	1·3655	21·004	1·3710	24·244	1·3765	27·405	1·3820	30·489
1·3601	17·741	1·3656	21·063	1·3711	24·302	1·3766	27·462	1·3821	30·544
1·3602	17·802	1·3657	21·123	1·3712	24·361	1·3767	27·518	1·3822	30·600
1·3603	17·863	1·3658	21·183	1·3713	24·419	1·3768	27·575	1·3823	30·655
1·3604	17·924	1·3659	21·242	1·3714	24·477	1·3769	27·632	1·3824	30·711
1·3605	17·985	1·3660	21·302	1·3715	24·535	1·3770	27·688	1·3825	30·766
1·3606	18·046	1·3661	21·361	1·3716	24·593	1·3771	27·745	1·3826	30·821
1·3607	18·107	1·3662	21·421	1·3717	24·651	1·3772	27·802	1·3827	30·876
1·3608	18·168	1·3663	21·480	1·3718	24·709	1·3773	27·858	1·3828	30·932
1·3609	18·229	1·3664	21·540	1·3719	24·767	1·3774	27·915	1·3829	30·987

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n (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)
1·3830	31·042	1·3885	34·040	1·3940	36·970	1·3995	39·834	1·4050	42·635
1·3831	31·097	1·3886	34·094	1·3941	37·023	1·3996	39·885	1·4051	42·685
1·3832	31·152	1·3887	34·148	1·3942	37·075	1·3997	39·937	1·4052	42·736
1·3833	31·207	1·3888	34·202	1·3943	37·128	1·3998	39·988	1·4053	42·786
1·3834	31·262	1·3889	34·256	1·3944	37·180	1·3999	40·040	1·4054	42·836
1·3835	31·317	1·3890	34·310	1·3945	37·233	1·4000	40·091	1·4055	42·887
1·3836	31·372	1·3891	34·363	1·3946	37·286	1·4001	40·142	1·4056	42·937
1·3837	31·428	1·3892	34·417	1·3947	37·338	1·4002	40·194	1·4057	42·987
1·3838	31·482	1·3893	34·471	1·3948	37·391	1·4003	40·245	1·4058	43·037
1·3839	31·537	1·3894	34·524	1·3949	37·443	1·4004	40·296	1·4059	43·088
1·3840	31·592	1·3895	34·578	1·3950	37·495	1·4005	40·348	1·4060	43·138
1·3841	31·647	1·3896	34·632	1·3951	37·548	1·4006	40·399	1·4061	43·188
1·3842	31·702	1·3897	34·685	1·3952	37·600	1·4007	40·450	1·4062	43·238
1·3843	31·757	1·3898	34·739	1·3953	37·653	1·4008	40·501	1·4063	43·288
1·3844	31·812	1·3899	34·793	1·3954	37·705	1·4009	40·553	1·4064	43·338
1·3845	31·867	1·3900	34·846	1·3955	37·757	1·4010	40·604	1·4065	43·388
1·3846	31·922	1·3901	34·900	1·3956	37·810	1·4011	40·655	1·4066	43·439
1·3847	31·976	1·3902	34·953	1·3957	37·862	1·4012	40·706	1·4067	43·489
1·3848	32·031	1·3903	35·007	1·3958	37·914	1·4013	40·757	1·4068	43·539
1·3849	32·086	1·3904	35·060	1·3959	37·967	1·4014	40·808	1·4069	43·589
1·3850	32·140	1·3905	35·114	1·3960	38·019	1·4015	40·860	1·4070	43·639
1·3851	32·195	1·3906	35·167	1·3961	38·071	1·4016	40·911	1·4071	43·689
1·3852	32·250	1·3907	35·220	1·3962	38·123	1·4017	40·962	1·4072	43·739
1·3853	32·304	1·3908	35·274	1·3963	38·175	1·4018	41·013	1·4073	43·789
1·3854	32·359	1·3909	35·327	1·3964	38·228	1·4019	41·064	1·4074	43·838
1·3855	32·414	1·3910	35·380	1·3965	38·280	1·4020	41·115	1·4075	43·888
1·3856	32·468	1·3911	35·434	1·3966	38·332	1·4021	41·166	1·4076	43·938
1·3857	32·523	1·3912	35·487	1·3967	38·384	1·4022	41·217	1·4077	43·988
1·3858	32·577	1·3913	35·540	1·3968	38·436	1·4023	41·268	1·4078	44·038
1·3859	32·632	1·3914	35·593	1·3969	38·488	1·4024	41·318	1·4079	44·088
1·3860	32·686	1·3915	35·647	1·3970	38·540	1·4025	41·369	1·4080	44·138
1·3861	32·741	1·3916	35·700	1·3971	38·592	1·4026	41·420	1·4081	44·187
1·3862	32·795	1·3917	35·753	1·3972	38·644	1·4027	41·471	1·4082	44·237
1·3863	32·849	1·3918	35·806	1·3973	38·696	1·4028	41·522	1·4083	44·287
1·3864	32·904	1·3919	35·859	1·3974	38·748	1·4029	41·573	1·4084	44·337
1·3865	32·958	1·3920	35·912	1·3975	38·800	1·4030	41·623	1·4085	44·386
1·3866	33·013	1·3921	35·966	1·3976	38·852	1·4031	41·674	1·4086	44·436
1·3867	33·067	1·3922	36·019	1·3977	38·904	1·4032	41·725	1·4087	44·486
1·3868	33·121	1·3923	36·072	1·3978	38·955	1·4033	41·776	1·4088	44·535
1·3869	33·175	1·3924	36·125	1·3979	39·007	1·4034	41·826	1·4089	44·585
1·3870	33·230	1·3925	36·178	1·3980	39·059	1·4035	41·877	1·4090	44·635
1·3871	33·284	1·3926	36·231	1·3981	39·111	1·4036	41·928	1·4091	44·684
1·3872	33·338	1·3927	36·284	1·3982	39·163	1·4037	41·978	1·4092	44·734
1·3873	33·392	1·3928	36·337	1·3983	39·214	1·4038	42·029	1·4093	44·783
1·3874	33·446	1·3929	36·389	1·3984	39·266	1·4039	42·080	1·4094	44·833
1·3875	33·500	1·3930	36·442	1·3985	39·318	1·4040	42·130	1·4095	44·882
1·3876	33·555	1·3931	36·495	1·3986	39·370	1·4041	42·181	1·4096	44·932
1·3877	33·609	1·3932	36·548	1·3987	39·421	1·4042	42·231	1·4097	44·981
1·3878	33·663	1·3933	36·601	1·3988	39·473	1·4043	42·282	1·4098	45·031
1·3879	33·717	1·3934	36·654	1·3989	39·525	1·4044	42·332	1·4099	45·080
1·3880	33·771	1·3935	36·706	1·3990	39·576	1·4045	42·383	1·4100	45·130
1·3881	33·825	1·3936	36·759	1·3991	39·628	1·4046	42·433	1·4101	45·179
1·3882	33·879	1·3937	36·812	1·3992	39·679	1·4047	42·484	1·4102	45·228
1·3883	33·933	1·3938	36·865	1·3993	39·731	1·4048	42·534	1·4103	45·278
1·3884	33·987	1·3939	36·917	1·3994	39·782	1·4049	42·585	1·4104	45·327

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n (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)
1·4105	45·376	1·4160	48·061	1·4215	50·691	1·4270	53·269	1·4325	55·798
1·4106	45·426	1·4161	48·109	1·4216	50·738	1·4271	53·316	1·4326	55·844
1·4107	45·475	1·4162	48·158	1·4217	50·786	1·4272	53·362	1·4327	55·889
1·4108	45·524	1·4163	48·206	1·4218	50·833	1·4273	53·408	1·4328	55·935
1·4109	45·574	1·4164	48·254	1·4219	50·880	1·4274	53·455	1·4329	55·980
1·4110	45·623	1·4165	48·302	1·4220	50·928	1·4275	53·501	1·4330	56·026
1·4111	45·672	1·4166	48·350	1·4221	50·975	1·4276	53·548	1·4331	56·071
1·4112	45·721	1·4167	48·399	1·4222	51·022	1·4277	53·594	1·4332	56·116
1·4113	45·770	1·4168	48·447	1·4223	51·069	1·4278	53·640	1·4333	56·162
1·4114	45·820	1·4169	48·495	1·4224	51·116	1·4279	53·686	1·4334	56·207
1·4115	45·869	1·4170	48·543	1·4225	51·164	1·4280	53·733	1·4335	56·253
1·4116	45·918	1·4171	48·591	1·4226	51·211	1·4281	53·779	1·4336	56·298
1·4117	46·967	1·4172	48·639	1·4227	51·258	1·4282	53·825	1·4337	56·343
1·4118	46·016	1·4173	48·687	1·4228	51·305	1·4283	53·871	1·4338	56·389
1·4119	46·065	1·4174	48·735	1·4229	51·352	1·4284	53·918	1·4339	56·434
1·4120	46·114	1·4175	48·784	1·4230	51·399	1·4285	53·964	1·4340	56·479
1·4121	46·163	1·4176	48·832	1·4231	51·446	1·4286	54·010	1·4341	56·525
1·4122	46·212	1·4177	48·880	1·4232	51·493	1·4287	54·056	1·4342	56·570
1·4123	46·261	1·4178	48·928	1·4233	51·540	1·4288	54·102	1·4343	56·615
1·4124	46·310	1·4179	48·976	1·4234	51·587	1·4289	54·148	1·4344	56·660
1·4125	46·359	1·4180	49·023	1·4235	51·634	1·4290	54·194	1·4345	56·706
1·4126	46·408	1·4181	49·071	1·4236	51·681	1·4291	54·241	1·4346	56·751
1·4127	46·457	1·4182	49·119	1·4237	51·728	1·4292	54·287	1·4347	56·796
1·4128	46·506	1·4183	49·167	1·4238	51·775	1·4293	54·333	1·4348	56·841
1·4129	46·555	1·4184	49·215	1·4239	51·822	1·4294	54·379	1·4349	56·887
1·4130	46·604	1·4185	49·263	1·4240	51·869	1·4295	54·425	1·4350	56·932
1·4131	46·652	1·4186	49·311	1·4241	51·916	1·4296	54·471	1·4351	56·977
1·4132	46·701	1·4187	49·359	1·4242	51·963	1·4297	54·517	1·4352	57·022
1·4133	46·750	1·4188	49·407	1·4243	52·010	1·4298	54·563	1·4353	57·067
1·4134	46·799	1·4189	49·454	1·4244	52·057	1·4299	54·609	1·4354	57·112
1·4135	46·848	1·4190	49·502	1·4245	52·104	1·4300	54·655	1·4355	57·157
1·4136	46·896	1·4191	49·550	1·4246	52·150	1·4301	54·701	1·4356	57·202
1·4137	46·945	1·4192	49·598	1·4247	52·197	1·4302	54·746	1·4357	57·247
1·4138	46·994	1·4193	49·645	1·4248	52·244	1·4303	54·792	1·4358	57·292
1·4139	47·043	1·4194	49·693	1·4249	52·291	1·4304	54·838	1·4359	57·337
1·4140	47·091	1·4195	49·741	1·4250	52·338	1·4305	54·884	1·4360	57·382
1·4141	47·140	1·4196	49·788	1·4251	52·384	1·4306	54·930	1·4361	57·427
1·4142	47·188	1·4197	49·836	1·4252	52·431	1·4307	54·976	1·4362	57·472
1·4143	47·237	1·4198	49·884	1·4253	52·478	1·4308	55·022	1·4363	57·517
1·4144	47·286	1·4199	49·931	1·4254	52·524	1·4309	55·067	1·4364	57·562
1·4145	47·334	1·4200	49·979	1·4255	52·571	1·4310	55·113	1·4365	57·607
1·4146	47·383	1·4201	50·027	1·4256	52·618	1·4311	55·159	1·4366	57·652
1·4147	47·431	1·4202	50·074	1·4257	52·664	1·4312	55·205	1·4367	57·697
1·4148	47·480	1·4203	50·122	1·4258	52·711	1·4313	55·250	1·4368	57·742
1·4149	47·528	1·4204	50·169	1·4259	52·758	1·4314	55·296	1·4369	57·787
1·4150	47·577	1·4205	50·217	1·4260	52·804	1·4315	55·342	1·4370	57·832
1·4151	47·625	1·4206	50·264	1·4261	52·851	1·4316	55·388	1·4371	57·877
1·4152	47·674	1·4207	50·312	1·4262	52·897	1·4317	55·433	1·4372	57·921
1·4153	47·722	1·4208	50·359	1·4263	52·944	1·4318	55·479	1·4373	57·966
1·4154	47·771	1·4209	50·407	1·4264	52·990	1·4319	55·524	1·4374	58·011
1·4155	47·819	1·4210	50·454	1·4265	53·037	1·4320	55·570	1·4375	58·056
1·4156	47·868	1·4211	50·502	1·4266	53·083	1·4321	55·616	1·4376	58·101
1·4157	47·916	1·4212	50·549	1·4267	53·130	1·4322	55·661	1·4377	58·145
1·4158	47·964	1·4213	50·596	1·4268	53·176	1·4323	55·707	1·4378	58·190
1·4159	48·013	1·4214	50·644	1·4269	53·223	1·4324	55·752	1·4379	58·235

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n (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)
1·4380	58·279	1·4435	60·716	1·4490	63·108	1·4545	65·460	1·4600	67·771
1·4381	58·324	1·4436	60·759	1·4491	63·152	1·4546	65·502	1·4601	67·813
1·4382	58·369	1·4437	60·803	1·4492	63·195	1·4547	65·544	1·4602	67·854
1·4383	58·413	1·4438	60·847	1·4493	63·238	1·4548	65·587	1·4603	67·896
1·4384	58·458	1·4439	60·891	1·4494	63·281	1·4549	65·629	1·4604	67·938
1·4385	58·503	1·4440	60·935	1·4495	63·324	1·4550	65·672	1·4605	67·979
1·4386	58·547	1·4441	60·979	1·4496	63·367	1·4551	65·714	1·4606	68·021
1·4387	58·592	1·4442	61·023	1·4497	63·410	1·4552	65·756	1·4607	68·063
1·4388	58·637	1·4443	61·066	1·4498	63·453	1·4553	65·798	1·4608	68·104
1·4389	58·681	1·4444	61·110	1·4499	63·496	1·4554	65·841	1·4609	68·146
1·4390	58·726	1·4445	61·154	1·4500	63·539	1·4555	65·883	1·4610	68·187
1·4391	58·770	1·4446	61·198	1·4501	63·582	1·4556	65·925	1·4611	68·229
1·4392	58·815	1·4447	61·241	1·4502	63·625	1·4557	65·967	1·4612	68·270
1·4393	58·859	1·4448	61·285	1·4503	63·668	1·4558	66·010	1·4613	68·312
1·4394	58·904	1·4449	61·329	1·4504	63·711	1·4559	66·052	1·4614	68·353
1·4395	58·948	1·4450	61·372	1·4505	63·754	1·4560	66·094	1·4615	68·395
1·4396	58·993	1·4451	61·416	1·4506	63·797	1·4561	66·136	1·4616	68·436
1·4397	59·037	1·4452	61·460	1·4507	63·840	1·4562	66·178	1·4617	68·478
1·4398	59·082	1·4453	61·503	1·4508	63·882	1·4563	66·221	1·4618	68·519
1·4399	59·126	1·4454	61·547	1·4509	63·925	1·4564	66·263	1·4619	68·561
1·4400	59·170	1·4455	61·591	1·4510	63·968	1·4565	66·305	1·4620	68·602
1·4401	59·215	1·4456	61·634	1·4511	64·011	1·4566	66·347	1·4621	68·643
1·4402	59·259	1·4457	61·678	1·4512	64·054	1·4567	66·389	1·4622	68·685
1·4403	59·304	1·4458	61·721	1·4513	64·097	1·4568	66·431	1·4623	68·726
1·4404	59·348	1·4459	61·765	1·4514	64·139	1·4569	66·473	1·4624	68·768
1·4405	59·392	1·4460	61·809	1·4515	64·182	1·4570	66·515	1·4625	68·809
1·4406	59·437	1·4461	61·852	1·4516	64·225	1·4571	66·557	1·4626	68·850
1·4407	59·481	1·4462	61·896	1·4517	64·268	1·4572	66·599	1·4627	68·892
1·4408	59·525	1·4463	61·939	1·4518	64·311	1·4573	66·641	1·4628	68·933
1·4409	59·569	1·4464	61·983	1·4519	64·353	1·4574	66·683	1·4629	68·974
1·4410	59·614	1·4465	62·026	1·4520	64·396	1·4575	66·725	1·4630	69·016
1·4411	59·658	1·4466	62·070	1·4521	64·439	1·4576	66·767	1·4631	69·057
1·4412	59·702	1·4467	62·113	1·4522	64·481	1·4577	66·809	1·4632	69·098
1·4413	59·746	1·4468	62·156	1·4523	64·524	1·4578	66·851	1·4633	69·139
1·4414	59·791	1·4469	62·200	1·4524	64·567	1·4579	66·893	1·4634	69·181
1·4415	59·835	1·4470	62·243	1·4525	64·609	1·4580	66·935	1·4635	69·222
1·4416	59·879	1·4471	62·287	1·4526	64·652	1·4581	66·977	1·4636	69·263
1·4417	59·923	1·4472	62·330	1·4527	64·695	1·4582	67·019	1·4637	69·304
1·4418	59·967	1·4473	62·373	1·4528	64·737	1·4583	67·061	1·4638	69·346
1·4419	60·011	1·4474	62·417	1·4529	64·780	1·4584	67·103	1·4639	69·387
1·4420	60·056	1·4475	62·460	1·4530	64·823	1·4585	67·145	1·4640	69·428
1·4421	60·100	1·4476	62·503	1·4531	64·865	1·4586	67·186	1·4641	69·469
1·4422	60·144	1·4477	62·547	1·4532	64·908	1·4587	67·228	1·4642	69·510
1·4423	60·188	1·4478	62·590	1·4533	64·950	1·4588	67·270	1·4643	69·551
1·4424	60·232	1·4479	62·633	1·4534	64·993	1·4589	67·312	1·4644	69·593
1·4425	60·276	1·4480	62·677	1·4535	65·035	1·4590	67·354	1·4645	69·634
1·4426	60·320	1·4481	62·720	1·4536	65·078	1·4591	67·396	1·4646	69·675
1·4427	60·364	1·4482	62·763	1·4537	65·120	1·4592	67·437	1·4647	69·716
1·4428	60·408	1·4483	62·806	1·4538	65·163	1·4593	67·479	1·4648	69·757
1·4429	60·452	1·4484	62·849	1·4539	65·205	1·4594	67·521	1·4649	69·798
1·4430	60·496	1·4485	62·893	1·4540	65·248	1·4595	67·563	1·4650	69·839
1·4431	60·540	1·4486	62·936	1·4541	65·290	1·4596	67·604	1·4651	69·880
1·4432	60·584	1·4487	62·979	1·4542	65·333	1·4597	67·646	1·4652	69·921
1·4433	60·628	1·4488	63·022	1·4543	65·375	1·4598	67·688	1·4653	69·962
1·4434	60·672	1·4489	63·065	1·4544	65·417	1·4599	67·729	1·4654	70·003

▼B

n (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)
1·4655	70·044	1·4710	72·280	1·4765	74·480	1·4820	76·646	1·4875	78·777
1·4656	70·085	1·4711	72·320	1·4766	74·520	1·4821	76·685	1·4876	78·816
1·4657	70·126	1·4712	72·361	1·4767	74·560	1·4822	76·724	1·4877	78·854
1·4658	70·167	1·4713	72·401	1·4768	74·599	1·4823	76·763	1·4878	78·892
1·4659	70·208	1·4714	72·441	1·4769	74·639	1·4824	76·802	1·4879	78·931
1·4660	70·249	1·4715	72·482	1·4770	74·678	1·4825	76·841	1·4880	78·969
1·4661	70·290	1·4716	72·522	1·4771	74·718	1·4826	76·880	1·4881	79·008
1·4662	70·331	1·4717	72·562	1·4772	74·758	1·4827	76·919	1·4882	79·046
1·4663	70·372	1·4718	72·602	1·4773	74·797	1·4828	76·958	1·4883	79·084
1·4664	70·413	1·4719	72·643	1·4774	74·837	1·4829	76·997	1·4884	79·123
1·4665	70·453	1·4720	72·683	1·4775	74·876	1·4830	77·036	1·4885	79·161
1·4666	70·494	1·4721	72·723	1·4776	74·916	1·4831	77·075	1·4886	79·199
1·4667	70·535	1·4722	72·763	1·4777	74·956	1·4832	77·113	1·4887	79·238
1·4668	70·576	1·4723	72·803	1·4778	74·995	1·4833	77·152	1·4888	79·276
1·4669	70·617	1·4724	72·843	1·4779	75·035	1·4834	77·191	1·4889	79·314
1·4670	70·658	1·4725	72·884	1·4780	75·074	1·4835	77·230	1·4890	79·353
1·4671	70·698	1·4726	72·924	1·4781	75·114	1·4836	77·269	1·4891	79·391
1·4672	70·739	1·4727	72·964	1·4782	75·153	1·4837	77·308	1·4892	79·429
1·4673	70·780	1·4728	73·004	1·4783	75·193	1·4838	77·347	1·4893	79·468
1·4674	70·821	1·4729	73·044	1·4784	75·232	1·4839	77·386	1·4894	79·506
1·4675	70·861	1·4730	73·084	1·4785	75·272	1·4840	77·425	1·4895	79·544
1·4676	70·902	1·4731	73·124	1·4786	75·311	1·4841	77·463	1·4896	79·582
1·4677	70·943	1·4732	73·164	1·4787	75·350	1·4842	77·502	1·4897	79·620
1·4678	70·984	1·4733	73·204	1·4788	75·390	1·4843	77·541	1·4898	79·659
1·4679	71·024	1·4734	73·244	1·4789	75429	1·4844	77·580	1·4899	79·697
1·4680	71·065	1·4735	73·285	1·4790	75469	1·4845	77·619	1·4900	79·735
1·4681	71·106	1·4736	73·325	1·4791	75·508	1·4846	77·657	1·4901	79·773
1·4682	71·146	1·4737	73·365	1·4792	75·547	1·4847	77·696	1·4902	79·811
1·4683	71·187	1·4738	73·405	1·4793	75·587	1·4848	77·735	1·4903	79·850
1·4684	71·228	1·4739	73·445	1·4794	75·626	1·4849	77·774	1·4904	79·888
1·4685	71·268	1·4740	73·485	1·4795	75·666	1·4850	77·812	1·4905	79·926
1·4686	71·309	1·4741	73·524	1·4796	75·705	1·4851	77·851	1·4906	79·964
1·4687	71·349	1·4742	73·564	1·4797	75·744	1·4852	77·890	1·4907	80·002
1·4688	71·390	1·4743	73·604	1·4798	75·784	1·4853	77·928	1·4908	80·040
1·4689	71·431	1·4744	73·644	1·4799	75·823	1·4854	77·967	1·4909	80·078
1·4690	71·471	1·4745	73·684	1·4800	75·862	1·4855	78·006	1·4910	80·116
1·4691	71·512	1·4746	73·724	1·4801	75·901	1·4856	78·045	1·4911	80·154
1·4692	71·552	1·4747	73·764	1·4802	75·941	1·4857	78·083	1·4912	80·192
1·4693	71·593	1·4748	73·804	1·4803	75·980	1·4858	78·122	1·4913	80·231
1·4694	71·633	1·4749	73·844	1·4804	76·019	1·4859	78·160	1·4914	80·269
1·4695	71·674	1·4750	73·884	1·4805	76·058	1·4860	78·199	1·4915	80·307
1·4696	71·714	1·4751	73·924	1·4806	76·098	1·4861	78·238	1·4916	80·345
1·4697	71·755	1·4752	73·963	1·4807	76·137	1·4862	78·276	1·4917	80·383
1·4698	71·795	1·4753	74·003	1·4808	76·176	1·4863	78·315	1·4918	80·421
1·4699	71·836	1·4754	74·043	1·4809	76·215	1·4864	78·353	1·4919	80·459
1·4700	71·876	1·4755	74·083	1·4810	76·254	1·4865	78·392	1·4920	80·497
1·4701	71·917	1·4756	74·123	1·4811	76·294	1·4866	78·431	1·4921	80·534
1·4702	71·957	1·4757	74·162	1·4812	76·333	1·4867	78·469	1·4922	80·572
1·4703	71·998	1·4758	74·202	1·4813	76·372	1·4868	78·508	1·4923	80·610
1·4704	72·038	1·4759	74·242	1·4814	76·411	1·4869	78·546	1·4924	80·648
1·4705	72·078	1·4760	74·282	1·4815	76·450	1·4870	78·585	1·4925	80·686
1·4706	72·119	1·4761	74·321	1·4816	76·489	1·4871	78·623	1·4926	80·724
1·4707	72·159	1·4762	74·361	1·4817	76·528	1·4872	78·662	1·4927	80·762
1·4708	72·199	1·4763	74·401	1·4818	76·567	1·4873	78·700	1·4928	80·800
1·4709	72·240	1·4764	74·441	1·4819	76·607	1·4874	78·739	1·4929	80·838

▼B

n (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)	*n* (20 °C)	Sucrose (%)
1·4930	80·876	1·4960	82·007	1·4990	83·128	1·5020	84·240	1·5050	85·343
1·4931	80·913	1·4961	82·044	1·4991	83·165	1·5021	84·277	1·5051	85·379
1·4932	80·951	1·4962	82·082	1·4992	83·202	1·5022	84·314	1·5052	85·416
1·4933	80·989	1·4963	82·119	1·4993	83·240	1·5023	84·351	1·5053	85·452
1·4934	81·027	1·4964	82·157	1·4994	83·277	1·5024	84·388	1·5054	85·489
1·4935	81·065	1·4965	82·194	1·4995	83·314	1·5025	84·424	1·5055	85·525
1·4936	81·103	1·4966	82·232	1·4996	83·351	1·5026	84·461	1·5056	85·562
1·4937	81·140	1·4967	82·269	1·4997	83·388	1·5027	84·498	1·5057	85·598
1·4938	81·178	1·4968	82·307	1·4998	83·425	1·5028	84·535	1·5058	85·635
1·4939	81·216	1·4969	82·344	1·4999	83·463	1·5029	84·572	1·5059	85·672
1·4940	81·254	1·4970	82·381	1·5000	83·500	1·5030	84·609	1·5060	85·708
1·4941	81·291	1·4971	82·419	1·5001	83·537	1·5031	84·645	1·5061	85·744
1·4942	81·329	1·4972	82·456	1·5002	83·574	1·5032	84·682	1·5062	85·781
1·4943	81·367	1·4973	82·494	1·5003	83·611	1·5033	84·719	1·5063	85·817
1·4944	81·405	1·4974	82·531	1·5004	83·648	1·5034	84·756	1·5064	85·854
1·4945	81·442	1·4975	82·569	1·5005	83·685	1·5035	84·792	1·5065	85·890
1·4946	81·480	1·4976	82·606	1·5006	83·722	1·5036	84·829	1·5066	85·927
1·4947	81·518	1·4977	82·643	1·5007	83·759	1·5037	84·866	1·5067	85·963
1·4948	81·555	1·4978	82·681	1·5008	83·796	1·5038	84·903	1·5068	86·000
1·4949	81·593	1·4979	82·718	1·5009	83·833	1·5039	84·939	1·5069	86·036
1·4950	81·631	1·4980	82·755	1·5010	83·870	1·5040	84·976	1·5070	86·072
1·4951	81·668	1·4981	82·793	1·5011	83·907	1·5041	85·013	1·5071	86·109
1·4952	81·706	1·4982	82·830	1·5012	83·944	1·5042	85·049	1·5072	86·145
1·5953	81·744	1·4983	82·867	1·5013	83·981	1·5043	85·086	1·5073	86·182
1·4954	81·781	1·4984	82·905	1·5014	84·018	1·5044	85·123	1·5074	86·218
1·4955	81·819	1·4985	82·942	1·5015	84·055	1·5045	85·159	1·5075	86·254
1·4956	81·856	1·4986	82·979	1·5016	84·092	1·5046	85·196	1·5076	86·291
1·4957	81·894	1·4987	83·016	1·5017	84·129	1·5047	85·233	1·5077	86·327
1·4958	81·932	1·4988	83·054	1·5018	84·166	1·5048	85·269	1·5078	86·363
1·4959	81·969	1·4989	83·091	1·5019	84·203	1·5049	85·306	1·5079	86·399

METHOD 4

MEASUREMENT OF REDUCING SUGARS EXPRESSED AS INVERT SUGARS

(Berlin Institute method)

Scope and field of application

1. The method determines the reducing sugar content expressed as invert sugar in semi-white sugar.

2. **Definitions**

‘Reducing sugars expressed as invert sugar’: the content of reducing sugars as determined by the method specified.

3. **Principle**

The sample solution containing reducing sugars is used to reduce a solution of copper II complex. The copper I oxide formed is then oxidized with standard iodine solution, the excess of which is determined by back-titration with standardized sodium thiosulphate solution.

4. **Reagents**

4.1. *Copper II solution* (Muller's solution)

4.1.1. Dissolve 35 g of copper II sulphate, pentahydrate ($CuSO_4.5H_2O$) in 400 ml of boiling water. Allow to cool.

4.1.2. Dissolve 173 g of sodium potassium tartrate tetrahydrate (Rochelle salt or Seignette salt; $KNaC_4H_4O_64H_2O$) and 68 g of anhydrous sodium carbonate in 500 ml of boiling water. Allow to cool.

▼B

4.1.3. Transfer both solutions (4.1.1 and 4.1.2) to a one litre volumetric flask and make up to one litre with water. Add 2 g of activated carbon, shake, allow to stand for several hours and filter through thick filter paper or a membrane filter.

If small amounts of copper I oxide appear during storage, the solution should be re-filtered.

4.2. *Acetic acid solution* 5 mol/litre.

4.3. *Iodine solution* 0·01665 mol/litre (i.e. 0·0333 N, 4·2258 g/litre).

4.4. *Sodium thiosulphate solution* 0·0333 mol/litre.

4.5. *Starch solution:* to one litre of boiling water add a mixture of 5 g of soluble starch slurried in 30 ml of water. Boil for three minutes, allow to cool and add, if required, 10 mg of mercury II iodide as a preservative.

5. **Apparatus**

5.1. *Conical flask,* 300 ml; precision burettes and pipettes.

5.2. *Water-bath,* boiling.

6. **Procedure**

6.1. Weigh a portion of the sample (10 g or less) containing not more than 30 mg of invert sugar in a 300 ml conical flask and dissolve in about 100 ml of water.

Pipette 10 ml of the copper II solution (4.1), into the flask containing the sample solution. Mix the contents of the flask by swirling and place it in the boiling water-bath (5.2) for exactly 10 minutes.

The level of the solution in the conical flask should be at least 20 mm below the level of the water in the water-bath. Cool the flask rapidly in a stream of cold running water. During this operation the solution should not be stirred otherwise atmospheric oxygen will reoxidize some precipitated copper I oxide.

Add 5 ml of 5 mol/litre acetic acid (4.2) by pipette without shaking and immediately add an excess (between 20 and 40 ml) of the iodine solution 0.01665 mol/litre (4.3) from a burette.

Stir to dissolve the copper precipitate. Titrate the excess iodine against the sodium thiosulphate solution 0·0333 mol/litre (4.4) using the starch solution (4.5) as indicator. The indicator is added towards the end of the titration.

6.2. Carry out a blank test with water. This is to be carried out with each new copper II solution (4.4). The titration shall not exceed 0·1 ml.

6.3. Carry out a control test under cold conditions with the sugar solution. Allow to stand at room temperature for 10 minutes to permit any reducing agents such as sulphur dioxide which may by present to react.

7. **Expression of results.**

7.1. *Formula and method of calculation*

Volume of iodine consumed = ml 0·01665 mol/litre iodine added in excess minus ml 0·0333 mol/litre sodium thiosulphate used in titration.

▼B

The volume (in ml) of 0·01665 ml/litre iodine consumed is corrected by subtracting:

7.1.1. The number of ml consumed in the blank test carried out with water (6.2).

7.1.2. The number of ml consumed in the cold test with the sugar solution (6.3).

7.1.3. A value of 2·0 ml for every 10 g of sucrose present in the aliquot used, or a proportionate quantity where the sample contains less than 10 g sucrose (correction for sucrose).

After these corrections are made each ml of iodine solution (4.3) which has reacted corresponds to 1 mg of of invert sugar.

The invert sugar contents, as a percentage of the sample, is given by the formula:

$$\frac{V_1}{10 \times m_o}$$

where:

V_1 = the number of ml of iodine solution (4.3) after correction,

m_o = the mass, in grams, of the sample used.

7.2. *Repeatability*

The difference between the results of two determinations when carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 0·02 g per 100 g of sample.

METHOD 5

MEASUREMENT OF REDUCING SUGARS EXPRESSED AS INVERT SUGAR

(Knight and Allen method)

1. **Scope and field of application**

The method determines the reducing sugar content expressed as invert sugar in:

— sugar or white sugar,

— extra white sugar.

2. **Definition**

‘Reducing sugars expressed as invert sugar’: the content of reducing sugars as determined by the method specified.

3. **Principle**

Copper II reagent is added in excess to the sample solution, reduced and the unreduced portion is back-titrated with EDTA solution.

4. **Reagents**

4.1. *Ethylene diamine tetra-acetic acid solution* (disodium salt) (EDTA) 0·0025 mol/litre: dissolve 0·930 g of EDTA in water and make up to one litre with water.

▼B

4.2. *Murexide indicator solution:* add 0·25 g of murexide to 50 ml of water and mix with 20 ml of a 0·2 g /100 ml aqueous solution of methylene blue.

4.3. *Alkaline copper reagent:* dissolve 25 g of anhydrous sodium carbonate and 25 g of potassium sodium tartrate tetrahydrate in about 600 ml of water containing 40 ml of 1.0 mol/litre sodium hydroxide. Dissolve 6·0 g of copper II sulphate pentahydrate ($CuSO_4.5H_2O$) in about 100 ml of water, and add to the tartrate solution. Dilute to one litre with water.

N.B.: the solution has a limited life (one week).

4.4. *Standard invert sugar solution:* dissolve 23·750 g of pure sucrose (4.5) in about 120 ml of water in a 250 ml graduated flask, add 9 ml of hydrochloric acid (ζ = 1·16) and allow to stand for eight days at room temperature. Make the solution up to 250 ml and check for completion of hydrolysis by a polarimeter or saccharimeter reading in a 200 mm tube. This should be - 11·80° ± 0·05 °S (see Note 8). Pipette 200 ml of this solution into a 2 000 ml graduated flask. Dilute with water and while shaking (to avoid excessive local alkalinity) add 71·4 ml of sodium hydroxide solution (1 mol/litre) in which 4 g of benzoic acid has been dissolved. Make up to 2 000 ml to give a 1 g/100 ml solution of invert sugar. This solution should be approximately pH 3.

This stable stock solution should only be diluted immediately before use.

4.5. *Pure sucrose:* sample of pure sucrose with an invert sugar content not greater than 0·001 g/100 g.

5. **Apparatus**

5.1. *Test tubes,* 150 x 20 mm.

5.2. *White porcelain dish.*

5.3. *Analytical balance,* accurate to within 0·1 mg.

6. **Procedure**

6.1. Dissolve 5 g of sugar sample in 5 ml of cold water in the test tube (5.1). Add 2·0 ml of the copper reagent (4.3) and mix. Immerse the tube in a boiling water bath for five minutes and then cool in cold water.

6.2. Transfer quantitatively the solution in the test tube to the white porcelain dish (5.2) using as little water as possible, add three drops of indicator (4.2) and titrate with EDTA solution (4.1). V_o is the number of ml of EDTA used in the titration.

Just before the end-point the colour of the solution changes from green through grey to purple at the end-point. The purple colour will disappear slowly because of oxidation of copper I oxide to copper II oxide at a rate dependent on the concentration of reduced copper present. The end-point of the titration shall therefore be approached fairly rapidly.

▼B

6.3. Construct a calibration graph by adding known amounts of invert sugar (as solution 4.4 appropriately diluted) to 5 g of pure sucrose (4.5) and add sufficient cold water so that a total of 5 ml of solution is added. Plot the titration volumes (in ml) against the percentage of invert sugar added to the 5 g of sucrose: the resultant graph is a straight line over the range 0·001 to 0·019 g/100 g invert sugar/100 g sample.

7. **Expression of results**

7.1. *Method of calculation*

Read on the calibration curve the percentage of invert sugar corresponding to the value V_o ml of EDTA determined when analyzing the sample.

7.2. When a concentration greater than 0·017 g invert sugar/100 g sample is expected in the sample to be analyzed, the sample size taken in Procedure (6.1) must be appropriately reduced but the analysis sample made up to 5 g with pure sucrose (4.5).

7.3. *Repeatability*

The difference between the results of two determinations when carried out simultaneously or in rapid succession on the same sample, by the same, analyst, under the same conditions, shall not exceed 0·005 g per 100 g of sample.

8. **Note**

Divide by 2·889 to convert °S to polarmetric degrees of arc (precision tubes of 200 mm; light source consisting of a sodium vapour lamp; the instrument must be installed in a room where the temperature may be maintained close to 20 °C).

METHOD 6

DETERMINATION OF REDUCING SUGARS EXPRESSED AS INVERT SUGAR OR DEXTROSE EQUIVALENT

(Luff-Schoorl method)

1. **Scope and field of application**

The method determines:

1.1. The reducing sugars content expressed as invert sugar in:

— sugar solution,

— white sugar solution,

— invert sugar solution,

— white invert sugar solution,

— invert sugar syrup,

— white invert sugar syrup.

1.2. The reducing sugar content, expressed and calculated (on the dry matter) as the dextrose equivalent in:

— glucose syrup,

— dried glucose syrup

▼B

1.3. The reducing sugar content expressed as D-glucose in:

— dextrose monohydrate,

— dextrose anhydrous

2. **Definition**

'Reducing sugars expressed as invert sugars, D-glucose or dextrose equivalent': the content of reducing sugars expressed or calculated as invert sugar, D-glucose or dextrose equivalent as determined by the method specified.

3. **Principle**

The reducing sugars in the sample (clarified if necessary) are heated to boiling point under standardized conditions with a copper II solution, which is partially reduced to copper I. The excess copper II is subsequently determined iodometrically.

4. **Reagents**

4.1. *Carrez solution I:* dissolve 21·95 g of zinc acetate dihydrate ($Zn(CH_3COO)_2.2H_2O$) (or 24 g of zinc acetate trihydrate ($Zn(CH_3COO)_2.3H_2O$) and 3 ml of glacial acetic acid in water and make up to 100 ml with water.

4.2. *Carrez solution II:* dissolve 10·6 g of potassium hexacyanoferrate IT trihydrate K_4 $[Fe(CN)_6]$. $3H_2O$ in water and make up to 100 ml with water.

4.3. *Luff-Schoorl reagent:* prepare the following solutions:

4.3.1. Copper II sulphate solution: dissolve 25 g of iron-free copper II sulphate pentahydrate ($CuSO_4.5H_2O$) in 100 ml water.

4.3.2. Citric acid solution: dissolve 50 g of citric acid monohydrate ($C_6H_8O_7.H_2O$) in 50 ml of water.

4.3.3. Sodium carbonate solution: dissolve 143.8 g of anhydrous sodium carbonate in about 300 ml of warm water and allow to cool.

4.3.4. Add the citric acid solution (4.3.2) to the sodium carbonate solution (4.3.3) in a one litre volumetric flask with gentle swirling. Swirl until effervescence ceases and then add the copper II sulphate solution (4.3.1) and make up to 1 000 ml with water. Allow the solution to stand overnight and then filter if necessary. Check the molarity of the reagent thus obtained by the method described in 6.1 (Cu 0·1 mol/litre; Na_2CO_3 1 mol/litre).

4.4. *Sodium thiosulphate solution,* 0·1 mol/litre.

4.5. *Starch solution:* to one litre of boiling water add a mixture of 5 g of soluble starch slurried in 30 ml of water. Boil for three minutes, allow to cool and add, if required, 10 mg of mercury II iodide as a preservative.

4.6. *Sulphuric acid,* 3 mol/litre.

4.7. *Potassium iodide solution,* 30 % (m/v).

▼B

4.8. *Pumice chips,* boiled in hydrochloric acid, washed free of acid with water and then dried.

4.9. *Isopentanol*

4.10. *Sodium hydroxide,* 0·1 mol/litre.

4.11. *Hydrochlorie acid,* 0·1 mol/litre.

4.12. *Phenolphthalein solution,* 1 % (m/v) in ethanol.

5. **Apparatus**

5.1. *Conical flask,* 300 ml, fitted with a reflux condenser.

5.2. *Stop-watch.*

6. **Procedure**

6.1. *Standardization of the Luff-Schoorl reagent* (4.3)

6.1.1. To 25 ml of Luff-Schoorl reagent (4.3) add 3 g of potassium iodide and 25 ml of 3 mol/litre sulphuric acid (4.6).

Titrate with 0·1 mol/litre sodium thiosulphate (4.4) using starch solution (4.5) as indicator added towards the end of the titration. If the volume of 0·1 mol/litre sodium thiosulphate used is not 25 ml the reagent must be made up afresh.

6.1.2. Pipette 10 ml of the reagent into a 100 ml volumetric flask and dilute to volume with water.

Pipette 10 ml of the diluted reagent into 25 ml of 0·1 mol/litre hydrochloric acid (4.11) in a conical flask and heat for one hour in a boiling water-bath. Cool, make up to the original volume with freshly boiled water and titrate with 0·1 mol/litre sodium hydroxide (4.10) using phenolphthalein (4.12) as indicator.

The volume of 0·1 mol/litre sodium hydroxide (4.10) used must be between 5·5 and 6·5 ml.

6.1.3. Titrate 10 ml of the diluted reagent (6.1.2) with 0·1 mol/litre hydrochloric acid (4.11) using phenolphthalein (4.12) as indicator. The end-point is characterized by the disappearance of the violet colour.

The volume of 0·1 mol/litre hydrochloric acid (4.11) used must be between 6·0 and 7·5 ml.

6.1.4. The pH of the Luff-Schoorl reagent must be between 9·3 and 9·4 at 20 °C.

6.2. *Preparation of the solution*

6.2.1. Accurately weigh, to the nearest 1 mg, 5 g of the sample and transfer quantitatively to a 250 ml volumetric flask, with 200 ml water. Clarify, if necessary, by adding 5 ml of Carrez solution I (4.1) followed by 5 ml of Carrez solution II (4.2). Mix after each addition. Make up to 250 ml with water. Mix well. Filter if necessary.

6.2.2. Dilute the solution (6.2.1) so that 25 ml of the solution contains not less than 15 mg and not more than 60 mg of reducing sugars expressed as glucose.

▼B

6.3. *Titration by the Luff-Schoorl method*

Pipette 25 ml of Luff-Schoorl reagent (4.3) into a 300 ml conical flask (5.1). Pipette 25 ml of the sugar solution (6.2.2) into the conical flask and introduce two pumice chips (4.8). Fit a reflux condenser to the conical flask (5.1) and immediately place the apparatus on an asbestos wire gauze over a Bunsen flame. The gauze shall have a hole cut in the asbestos part of the same diameter as the base of the flask. Heat the liquid to boiling point over a period of about two minutes and simmer gently for exactly 10 minutes. Cool immediately in cold water and after five minutes titrate as follows:

Add 10 ml of potassium iodide solution (4.7) then immediately add with caution (because of effervescence) 25 ml of 3 mol/litre sulphuric acid (4.6). Titrate with 01 mol/litre sodium thiosulphate solution (4.4) until the solution is almost colourless, then add a few ml of starch solution (4.5) as indicator and continue titrating until the blue colour disappears.

Carry out a control test, using 25 ml of water in place of the 25 ml of sugar solution (6.2.2).

7. **Expression of results**

7.1. *Formula and method of calculation*

From the table below, find (interpolating if necessary) the weight of glucose or of invert sugar in mg corresponding to the difference between the two titration readings, expressed in ml of 0·1 mol/litre sodium thiosulphate.

Express the result in terms of invert sugar or D-glucose as percentage (m/m) of the dry matter.

7.2. *Repeatability*

The difference between the results of two titrations when carried out simultaneously or in rapid succession on the same sample by the same analyst, under the same conditions, shall not exceed 0·2 ml.

8. **Note**

A small volume of isopentanol (4·9) may be added before acidifying with sulphuric acid to reduce foaming.

Table of values according to Luff-Schoorl reagent

0·1 mol/litre $Na_2S_2O_3$	Glucose, fructose, invert sugars $C_6H_{12}O_6$	
ml	mg	difference
1	2·4	
2	4·8	2·4
3	7·2	2·4
4	9·7	2·5
5	12·2	2·5
6	14·7	2·5
7	17·2	2·5
8	19·8	2·6
9	22·4	2·6
10	25·0	2·6
11	27·6	2·6
12	30·3	2·7
13	33·0	2·7

▼B

0·1 mol/litre $Na_2S_2O_3$	Glucose, fructose, invert sugars $C_6H_{12}O_6$	
ml	mg	difference
14	35·7	2·7
15	38·5	2·8
16	41·3	2·8
17	44·2	2·9
18	47·1	2·9
19	50·0	2·9
20	53·0	3·0
21	56·0	3·0
22	59·1	3·1
23	62·2	3·1

METHOD 7

MEASUREMENT OF REDUCING SUGARS EXPRESSED AS INVERT SUGAR

(Lane and Eynon constant volume modification)

1. **Scope and field of application**

The method determines the reducing sugars, expressed as invert sugar, in:

— sugar solution,

— white sugar solution,

— invert sugar solution,

— white invert sugar solution,

— invert sugar syrup,

— white invert sugar syrup.

2. **Definition**

'Reducing sugars expressed as invert sugar': the content of reducing sugars as determined by the method specified.

3. **Principle**

The sample solution is titrated at the boiling point against a specified volume of Fehling's solution, using methylene blue as internal indicator.

4. **Reagents**

4.1. *Fehling's solution:*

4.1.1. Solution A:

Dissolve 69·3 g of copper II sulphate pentahydrate ($CuSO_4.5H_{2O)}$ in water and make up to 1 000 ml.

4.1.2. Solution B:

Dissolve 346·0 g of double sodium potassium tartrate tetrahydrate ($KNaC_4H_4O_6.4H_2O$) with 100·0 g of sodium hydroxide in water and make up to 1 000 ml. The clear solution should be decanted from a sediment that may form from time to time.

▼B

Note:

These two solutions should be stored in brown or amber bottles.

4.2. *Sodium hydroxide solution,* 1 mol/litre.

4.3. *Standard invert sugar solution:* dissolve 23·750 g of pure sucrose in about 120 ml of water in a 250 ml graduated flask, add 9 ml of hydrochloric acid (ζ = 1·16) and allow to stand for eight days at room temperature. Make the solution up to 250 ml and check for completion of hydrolysis by a polarimeter or saccharimeter reading in a 200 mm tube. This should be - 11·80° ± 0·05 °S (see note 8). Pipette 200 ml of this solution into a 2 000 ml graduated flask. Dilute with water and while shaking (to avoid excessive local alkalinity) add 71·4 ml of sodium hydroxide solution (1 mol/litre) (4.2) in which 4 g of benzoic acid has been dissolved. Make up to 2 000 ml to give a 1 g/100 ml solution of invert sugar. This solution should be a pH of approximately 3.

This stable stock solution should only be diluted immediately before use.

To make up the 0·25 g/100 ml invert sugar solution, fill a 250 ml graduated flask to the mark with the stock 1 g/100 ml invert solution at 20 °C. Wash the contents of this flask into a 1 000 ml graduated flask and dilute to the mark with water again at 20 °C.

4.4. *Methylene blue solution,* 1 g/100 ml.

5. **Apparatus**

5.1. *Narrow-necked laboratory boiling flasks,* 500 ml.

5.2. *Burette,* 50 ml, with tap and offset tip, graduated to 0·05 ml.

5.3. *Pipettes* graduated at 20, 25 and 50 ml.

5.4. *One mark volumetric flasks,* 250, 1 000 and 2 000 ml.

5.5. *A heating device,* suitable for maintaining boiling according to the conditions described in 6.1, permitting the observation of the end-point colour change without the necessity of removing the boiling flask (5.1) from the source of heat.

5.6. *Stop-watch,* indicating to within at least one second.

6. **Procedure**

6.1. *Standardization of Fehling's solution*

6.1.1. Pipette 50 ml of solution B (4.1.2) and then 50 ml of solution A (4.1.1) into a clean dry beaker and mix well.

6.1.2. Rinse and fill the burette with 0·25 % (0·25 g/100 ml) standard invert sugar solution (4.3).

6.1.3. Pipette a 20 ml aliquot of the mixed solutions A and B (6.1.1) into a 500 ml boiling flask (5.1). Add 15 ml of water to the flask. Run in, from the burette, 39 ml of the invert sugar solution, add a small quantity of anti-bumping granules and mix the contents of the flask by gentle swirling.

▼B

6.1.4. Heat the flask and contents till boiling and allow to boil for exactly two minutes; the flask must not be removed from the heat source during the course of the rest of the procedure, or allowed to cease boiling.

Add three or four drops of methylene blue solution (4.4) at the end of the two-minute boiling period: the solution should be a definite blue colour.

6.1.5. Continue the standardization by adding, from the burette, the standard invert sugar solution in small increments, initially of 0·2 ml; then 0·1 ml and finally in single drops until the end-point is reached. This is indicated by the disappearance of the blue colour imparted by the methylene blue. The solution has then assumed the reddish colour associated with a suspension of copper I oxide.

6.1.6. The end-point should be reached at the end of three minutes from when the solution started to boil. The final titre, V_o, shall be between 39·0 and 41·0 ml. If V_o lies outside these limits, adjust the copper concentration of Fehling's solution A (4.1.1) and repeat the standardization process.

6.2. *Preparation of sample solutions*

The concentration of the sample test solution should be such that it contains between 250 and 400 mg invert sugar per 100 ml.

6.3. *Preliminary test*

6.3.1. A preliminary test must be carried out to ensure that the quantity of water to be added to the 20 ml of mixed solutions A and B is sufficient to ensure that a final volume after titration of 75 ml is obtained.

The same procedure as described in 6.1.4 is carried out except that the sample solution is used instead of the standard invert sugar solution, i.e. 25 ml of the sample solution is run into the flask from the burette. 15 ml of water is added, and the solution is allowed to boil for two minutes and then titrated until the end-point is reached as described in 6.1.5.

6.3.2. If, after the addition of the methylene blue solution, the reddish colour persists, the sample solution used is too concentrated. In this case, the test is discarded but repeated using a less concentrated sample solution.

If more than 50 ml of sample solution are required to obtain the reddish colour, a more concentrated solution of the sample must be used.

Calculate the quantity of water to be added by subtracting the volumes of mixed Fehling's solution (20 ml) and of the sample solution from 75 ml.

6.4. *Final analysis of sample solution*

6.4.1. Pipette into the boiling flask 20 ml of mixed Fehling's solution and the quantity of water determined as in 6.3.

6.4.2. Add, from the burette, the observed titre of the sample solution (as determined in 63) less 1 ml. Add some anti-bumping granules, mix the contents of the flask by swirling, boil the flask and contents and titrate as previously (6.3). The end-point should be reached one minute from the time of addition of the methylene blue solution. Final titre = V_1.

▼B

7. **Expression of results**

7.1. *Formula and method of calculation*

The reducing sugars content of the sample calculation as invert sugar, is given by:

% reducing sugars (as invert sugar =

$$\frac{V_o \times 25 \times f}{C_o \times V_1}$$

where:

C = the concentration of the sample test solution in g per 100 ml.

V_o = the volume in ml of the standard invert solution used in the standardization titration,

V_1 = the volume in ml of the sample test solution used in the accurate analysis in 6.4.2,

f = the correction factor to take account of the sucrose concentration in the sample test solution. Values are shown in the table below:

Sucrose (g in boiling mixture)	Correction factor f
0	1·000
0·5	0·982
1·0	0·971
1·5	0·962
2·0	0·954
2·5	0·946
3·0	0·939
3·5	0·932
4·0	0·926
4·5	0·920
5·0	0·915
5·5	0·910
6·0	0·904
6·5	0·898
7·0	0·893
7·5	0·888
8·0	0·883
8·5	0·878
9·0	0·874
9·5	0·869
10·0	0· 64

Corrections for varying sucrose contents of the sample test solution may be calculated from the table by interpolation.

Note:

The approximate sucrose concentration may be found by subtraction of the dissolved solids concentration due to the invert sugar (estimated for the purposes of this calculation f as 1.0), from the total dissolved solids concentration, expressed as sucrose, obtained from the refractive index of the solution using method three of this document.

▼B

7.2. *Repeatability*

The difference between the results of two determinations, carried out simultaneously or in rapid succession on the same sample by the same analyst under the same conditions, shall not exceed 1·0 % of their arithmetic mean.

8. **Note**

Divide by 2·889 to convert °S to polarmetric degrees of arc (precision tubes of 200 mm; light source consisting of a sodium vapour lamp; the instrument must be installed in a room where the temperature may be maintained close to 20 °C).

METHOD 8

DETERMINATION OF DEXTROSE EQUIVALENT

(Lane and Eynon constant)

1. **Scope and field of application**

This method determines the dextrose equivalent of:

— glucose syrup,

— dried glucose syrup,

— dextrose monohydrate,

— dextrose anhydrous.

2. **Definition**

2.1. 'Reducing power': the reducing sugar content, determined by the method specified, expressed in terms of anhydrous dextrose (D-glucose) and calculated as a percentage by mass of the sample.

2.2. 'Dextrose equivalent': the reducing power, calculated as a percentage by mass of the dry matter in the sample.

3. **Principle**

The test solution is titrated at the boiling point against a specified volume of mixed Fehling's solution, under strictly specified conditions, using methylene blue as an internal indicator.

4. **Reagents**

4.1. *Fehling's solution:*

4.1.1. Solution A:

Dissolve 69·3 g of copper II sulphate pentahydrate ($CuSO_4.5H_2O$) in water and make up to volume in a 1 000 ml volumetric f ask.

4.1.2. Solution B:

Dissolve 346·0 g of sodium potassium tartrate tetrahydrate ($KNaC_4H_4O_6.4H_2O$) and 100 g of sodium hydroxide in water. Make up to volume in a 1 000 ml volumetric flask. Decant the clear solution from any sediment that may from time to time form.

Note:

These two solutions (4.1.1 and 4.1.2) should be stored in brown or amber bottles.

4.1.3. Preparation of the mixed Fehling's solution

Pipette 50 ml of solution B (4.1.2) and then 50 ml of solution A (4.1.1) into a clean dry beaker and mix well.

▼B

Note:

Mixed Fehling's solution shall not be stored but made up afresh every day and standardized (6.1).

4.2. *Anhydrous dextrose* (D-glucose) ($C_6H_{12}O_6$)

This material shall be dried before use for four hours in a vacuum oven at 100 ± 1 °C or less, and an internal pressure of approximately 10 kPa (103 mbar).

4.3. *Standard dextrose solution,* 0·600 g/100 ml

Weigh, to the nearest 0·1 mg, 0·6 g of anhydrous dextrose (4.2), dissolve it in water, transfer the solution quantitatively into a 100 ml volumetric flask (5.4), dilute to the mark and mix.

This solution shall be freshly prepared on each day of use.

4.4. *Methylene blue solution,* 0·1 g/100 ml

Dissolve 0·1 g of methylene blue in 100 ml water.

5. **Apparatus**

5.1. *Narrow necked laboratory boiling flasks,* 250 ml.

5.2. *Burette,* 50 ml, with tap and offset tip, graduated to 0·05 ml.

5.3. *One mark pipettes,* 25 ml and 50 ml.

5.4. *One mark volumetric flasks,* 100 and 500 ml.

5.5. *A heating device,* suitable for maintaining boiling according to the conditions described in 6·1, permitting the observation of the end-point colour change without the necessity of removing the boiling flask (5.1) from the source of heat (see 6.1, note 3).

5.6. *A stop-watch,* indicating to at least the nearest second.

6. **Procedure**

6.1. *Standardization of the Fehling's solution*

6.1.1. Pipette 25 ml of Fehling's solution (4.1.3) into a clean, dry boiling flask (5.1).

6.1.2. Fill the burette (5.2) with standard dextrose solution (4.3) and adjust the meniscus to the zero mark.

6.1.3. Run into the boiling flask (5.1) from the burette 18 ml of standard dextrose solution (4.3). Swirl the flask to mix contents.

6.1.4. Place the boiling flask on the heating device (5.5), previously adjusted so that boiling commences in 120 ± 15 seconds.

The heating device shall not be further adjusted during the whole of the titration (see note 1).

6.1.5. When boiling commences, start the stop-watch from zero.

6.1.6. Boil the contents of the flask for 120 seconds, as timed by the stop-watch.

Add 1 ml of methylene blue solution (4.4) towards the end of this period.

6.1.7. After boiling has continued for 120 seconds (by the stop-watch) start adding standard dextrose solution to the boiling flask (5.1) from the burette (6.1.2) in 0·5 ml increments until the colour of the methylene blue is discharged (see notes 2 and 3).

▼B

Note the total volume of standard dextrose solution added up to and including the penultimate 0·5 ml increment (X ml).

6.1.8. Repeat 6.1.1 and 6.1.2.

6.1.9. Run into the boiling flask (5.1) from the burette a volume of standard dextrose solution equal to (X-0·3) ml.

6.1.10 Repeat 6.1.4, 6.1.5 and 6.1.6.

6.1.11. After boiling has continued for 120 seconds (by the stop-watch), start adding standard dextrose solution to the boiling flask (5.1) from the burette, initially in 0·2 ml increments and finally dropwise, until the colour of the methylene blue is just discharged.

Towards the end of this action the time between successive additions of standard dextrose solution shall be 10 to 15 seconds.

These additions shall be completed within 60 seconds, making the total time to boiling no longer than 180 seconds.

A third titration with a slightly larger, appropriately adjusted, initial addition of standard dextrose solution (6.1.9) may be necessary to achieve this.

6.1.12. Note the volume (V_o ml) of standard dextrose solution used up to the end-point of the final titration (see note 4).

6.1.13. V_o shall be between 19·0 and 21·0 ml standard dextrose solution (4.3).

If V_o lies outside these limits, adjust the concentration of the Fehling's solution A (4.1.1) appropriately and repeat the standardization process.

6.1.14. For the day-to-day standardization of the mixed Fehling's solution, as V_o is known with accuracy, a single titration only is necessary, using an initial addition of (V_o — 0·5) ml standard dextrose solution.

Note 1:

This ensures that once boiling has commenced the evolution of steam is brisk and continuous throughout the whole of the titration process, thus preventing to the maximum possible extent the entrance of air into the titration flask with consquent re-oxidation of its contents.

Note 2:

The disappearance of the colour of the methylene blue is best seen by looking at the upper layers and the meniscus of the contents of the titration flask, as these will be relatively free from the precipiated, red copper I oxide. The colour disappearance is more easily seen when indirect lighting is used. A white screen behind the titration flask is helpful.

Note 3:

The burette should be isolated as much as possible from the source of heat during the determination.

Note 4:

As there is always a personal factor involved, each operator shall carry out his own standardization titration and use his own value of V_o in the calculation (7.1).

▼B

6.2. *Preliminary examination of the prepared sample*

6.2.1. Unless the reducing power (2.1) of the prepared sample is known approximately, it is necessary to carry out a preliminary examination in order to obtain an approximate figure for it so that the mass of the test portion (6.3) can be calculated.

This examination is carried out as follows:

6.2.2. Prepare a 2 % m/v solution of the sample 'Z', having an estimated value.

6.2.3. As 6.1.2, using the sample solution (6.2.2) in place of the standard dextrose solution.

6.2.4. As 6.1.1.

6.2.5. As 6.1.3, using 10·0 ml sample solution instead of 18·0 ml standard dextrose solution.

6.2.6. As 6.1.4.

6.2.7. Heat the contents of the flask to boiling. Add 1 ml methylene blue solution (4.4).

6.2.8. Immediately boiling has started, start the stop-watch (5.6) from zero and commence adding sample solution to the flask from the burette in 1·0 ml increments at intervals of approximately 10 seconds until the blue colour of the methylene blue is discharged.

Note the total volume of sample solution added up to and including the penultimate increment (Y ml).

6.2.9. 'Y' must not exceed 50 ml. If it does, increase the concentration of the sample solution and repeat the titration.

6.2.10. The approximate reducing power of the prepared sample in percent by mass is given by:

$$\frac{60 \times V_0}{Y \times Z}$$

6.3. *Test portion*

Weigh out, to the nearest 0·1 mg, a mass of the prepared sample (mg) which contains between 2·85 and 3·15 g reducing sugars, expressed as anhydrous dextrose (D-glucose) using in the calculation either known approximate figure for the reducing power (2.1) or the approximate figure obtained in 6.2.10.

6.4. *Test solution*

Dissolve the test portion in water and make up to 500 ml in a volumetric flask.

6.5. *Determination*

6.5.1. As 6.1.1.

6.5.2. Fill the burette (5.2) with test solution (6.4) and adjust the meniscus to the zero mark.

6.5.3. Run into the boiling flask from the burette 18·5 ml test solution. Swirl the flask to mix the contents.

6.5.4. As 6.1.4.

6.5.5. As 6.1.5.

6.5.6. As 6.1.6.

6.5.7. As 6.1.7, using test solution in place of standard dextrose solution.

6.5.8. As 6.1.8.

▼B

6.5.9. As 6.1.9, using test solution in place of standard dextrose solution.

6.5.10. As 6.1.10.

6.5.11. As 6.1.11, using test solution in place of standard dextrose solution.

6.5.12. Note the volume (V_1) of test solution used up to the end-point of the final titration.

6.5.13. V_1 shall be between 19·0 and 21·0 ml test solution.

If V_1 lies outside these limits, adjust the concentration of the test solution appropriately and repeat 6.5.1 to 6.5.12.

6.5.14. Carry out two determinations on the same test solution.

6.6. *Dry matter content*

Determine the dry matter content of the prepared sample by method 2.

7. **Expression of results**

7.1. *Formulae and method of calculation*

7.1.1. Reducing power

The reducing power, calculated as a percentage by mass of the prepared sample, is given by:

$$\frac{300 \times V_0}{V_1 \times M}$$

where:

V_0 = the volume, in ml, of the standard dextrose solution (4.3) used in the standardization titration (6.1),

V_1 = the volume, in ml, of the test solution (6.4) used in the determination titration (6.5),

M = the mass, in grams, of the test portion (6.3) used to make 500 ml test solution.

7.1.2. Dextrose equivalent

The dextrose equivalent, calculated as a percentage by mass of the dry matter in the prepared sample, is given by:

$$\frac{RP \times 100}{D}$$

where:

RP = the reducing power, calculated as a percent by mass of the prepared sample (7.1.1),

D = the dry matter content of the prepared sample in percent by mass.

7.1.3. Take as the result the arithmetic mean of the two determinations provided that the requirement concerning repeatability (7.2) is satisfied.

7.2. *Repeatability*

The difference between the results of two determinations when carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 10 % of their arithmetic mean.

▼B

METHOD 9

DETERMINATION OF SULPHATED ASH

1. **Scope and field of application**

The method determines the sulphated ash content in:

— glucose syrup,

— dried glucose syrup,

— dextrose monohydrate,

— dextrose anhydrous.

2. **Definition**

'Sulphated ash content': the content of sulphated ash as determined by the method specified.

3. **Principle**

The residual mass of a test portion is determined after incineration in an oxidizing atmosphere at 525 °C in the presence of sulphuric acid and calculated as a percentage by mass of the sample.

4. **Reagents**

4.1. *Sulphuric acid,* dilute solution: slowly and cautiously add 100 ml of concentrated sulphuric acid (density at 20 °C = 1·84 g/ml; 96 % m/m) to 300 ml water with stirring and cooling.

5. **Apparatus**

5.1. *Electric muffle furnace,* equipped with a pyrometer and capable of operating at a temperature of 525 ± 25 °C.

5.2. *Analytical balance,* accurate to 0·1 mg.

5.3. *Ashing crucibles,* platinum or quartz, of suitable capacity.

5.4. *Desiccator,* containing freshly activated silica gel or an equivalent desiccant with a water content indicator.

6. **Procedure**

Heat a crucible (5.3) to the ashing temperature, cool in a desiccator and weigh. Accurately weigh, to the nearest 01 mg, 5 g of glucose syrup or dried glucose syrup, or about 10 g of dextrose monohydrate or dextrose anhydrous into the crucible.

Add 5 ml of sulphuric acid solution (4.1) (see note 8.1) and carefully heat the sample in the crucible over a flame or on a hotplate until it is completely carbonized. This carbonization process, during which vapours are burnt off from the sample (see note 8.2), should be carried out in a fume cupboard.

Place the crucible (5.3) in the muffle furnace (5.1) heated to 525 ± 25° C until a white ash is obtained. This normally takes two hours (see note 8.3).

Allow the sample to cool for about 30 minutes in a desiccator (5.4) and then weigh.

7. **Expression**

7.1. *Formula and method of calculation*

The sulphated ash content expressed as a percentage by mass of the sample to be analyzed is given by:

$$S = \frac{m_1}{m_0} \times 100$$

where:

m_1 = the mass, in grams, of the ash,

m_0 = the mass, in grams, of the test portion.

▼B

7.2. *Repeatability*

The difference between the results of two determinations when carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 2 % of their arithmetic mean.

8. **Notes**

8.1. The sulphuric acid is added in small quantities to prevent excessive foaming.

8.2. Every relevant precaution must be taken during the first carbonization to prevent losses of sample or of ash through excessive swelling of the sample.

8.3. If the sample is difficult to ash completely (i.e. black particles remain) the crucible should be removed from the muffle furnace and the residue moistened, after cooling, with a few drops of water before being returned to the furnace.

METHOD 10

DETERMINATION OF POLARIZATION

1. **Scope and field of application**

The method determines the polarization in:

— semi-white sugar,

— sugar or white sugar,

— extra-white sugar.

2. **Definition**

The polarization is the rotation of the polarized light plane by a sugar solution with 26 g of sugar per 100 ml contained in a tube of 200 mm in length.

3. **Principle**

The polarization is determined by using a saccharimeter or a polarimeter according to the conditions described in the following method.

4. **Reagents**

4.1. *Clarification agent:* basic lead acetate solution.

Add 560 g of dry basic lead acetate to about 1 000 ml of freshly boiled water. Boil the mixture for 30 minutes and then leave it to stand overnight.

Decant the supernatant liquid and dilute with freshly boiled water to obtain a solution of density of 1·25 g/ml, at 20 °C.

Protect this solution from contact with air.

4.2. *Diethyl ether*

5. **Apparatus**

5.1. *Saccharimeter graduated for the normal weight of 26 g of sucrose, or polarimeter*

This instrument must be installed in a room where the temperature may be maintained close to 20 °C. Calibrate the instrument against standard quartz plates.

5.2. *Light source,* consisting of a sodium vapour lamp.

5.3. *Precision polarimeter tubes,* length 200 mm, error not exceeding ± 0·02 mm.

5.4. *Analytical balance,* accurate to within 0·1 mg,

▼B

5.5. *Individually calibrated 100 ml volumetric flasks stoppered.* Flasks with a real capacity in the range 100·00 ± 0·01 ml may be used without correction. Flasks with a capacity outside those limits must be used with an appropriate correction to adjust the capacity to 100 ml.

5.6. *Water-bath,* controlled thermostatically at 20 ± 0·1 °C.

6. **Procedure**

6.1. *Preparation of the solution*

Weigh as quickly as possible 26 ± 0·002 g of the sample and transfer it quantitatively into a 100 ml volumetric flask (5.5) with approximately 60 ml of water.

Dissolve by swirling but without heating.

Where clarification is necessary, add 0·5 ml of lead acetate reagent (4.1).

Mix the solution by rotating the flask and wash the flask walls, until the volume is such that the meniscus is about 10 mm below the calibration mark.

Place the flask in the water-bath controlled (5.6) at 20 ± 0·1 °C until the temperature of the sugar solution is constant.

Eliminate any bubbles formed at the surface of the liquid with a drop of diethyl ether (4.2).

Make up to volume with water.

Stopper and mix thoroughly by inverting the flask at least three times.

Allow to stand for five minutes.

6.2. *Polarization*

Maintain the temperature at 20 ± 1 °C for all subsequent operations.

6.2.1. Obtain the zero correction of the apparatus.

6.2.2. Filter the sample through a filter paper. Discard the first 10 ml of the filtrate. Collect the next 50 ml of the filtrate.

6.2.3. Wash the polarimeter tube by rinsing twice with the sample solution to be examined (6.2.2).

6.2.4. Fill the tube carefully at 20 ± 0·1 °C with the sample solution to be examined.

Remove all air bubbles when sliding the end-plate into position. Place the filled tube in the cradle of the instrument.

6.2.5. Read the rotation to within 0·05 °S or 0·02 angular degrees. Repeat a further four times. Take the mean of the five readings.

7. **Expression of results**

7.1. *Formula and method of calculation*

The results are expressed in degrees S to the nearest 0·1 °S. To convert the angular degrees into degrees S, the following formula is used:

$$°S = \text{degree of arc} \times 2{\cdot}889$$

7.2. *Repeatability*

The difference between the results of two determinations when carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, and each representing the mean of five readings, must not exceed 0·1 °S.