Method 7.15 – Raw sugar: grain size distribution

1. Rationale

The method is applicable to raw sugars and determines the grain size distribution of affinated raw sugars.

2. Principle

A representative raw sugar sample is washed with methanol to remove the molasses coating followed by washing with diethyl ether for quick drying. The sample is segregated into size fractions in a set of appropriately woven wire cloth sieves. The mass of each size fraction is determined and expressed as a percentage of the sample. The distribution is expressed in terms of the Specific Grain Size (SGS), the Mean Aperture (MA) and the Coefficient of Variance (CV) using the Rens equations.

3. Definitions

3.1 Specific grain size (SGS)

The specific grain size is defined as the mean size of the crystals in the sample expressed in millimetres.

3.2 Mean aperture (MA)

The mean aperture (MA) is defined as the aperture (sieve size) that would retain 50% (m/m) of the sample in millimetres.

3.3 Coefficient of variance (CV)

The coefficient of variance (CV) is defined as the standard deviation of the particle size distribution and is expressed as a percentage of the MA.

4. Apparatus

4.1 **Sample divider** or riffle

4.2 **Top pan balance** readable to 0.01 g

4.3 **Tumbler** operating at approximately 30 rpm

4.4 **Conical flask**: 250 cm^3 wide-neck with a stopper

4.5 **Strainer stopper**

A stopper that fits tightly into the conical flask (approximately 35 mm ϕ). The stopper must have a central hole approximately 12 mm in diameter. At its narrow end, the stopper must have a circular disc of centrifugal screening held connected to the stopper by a number 3 rubber sleeve.

4.6 Buchner flask: 1 000 cm^3

4.7 Stopwatch

4.8 Woven wire cloth sieves

A series of test sieves is required with different nominal operative sizes complete with a receiver pan lid. In particular sieve sizes 1700, 1180, 1000, 600 and 355 µm are used.

4.9 Mechanical shaker

The sieves are mounted onto the mechanical shaker. The shaker must move the sample continually across the entire surface of the mesh of each sieve and incorporate a secondary motion that gives a rising and falling movement to the stack of sieves. The rising and falling action, with a nominal lift of about 5 mm and a frequency of at least 120 times a minute, imparts a jolting action similar to that used in manual shaking.

When a high frequency shaker is used for this analysis, amplitude setting and time of shaking are important. A shaker with a frequency of 3 000 oscillations per minute, an amplitude of 1.5 mm and a shaking time of 15 minutes is recommended.

4.10 Rubber stopper to fit tightly into the Conical flask

4.11 Filter paper: Whatman No. 91 or equivalent, 185 mm ϕ

5. Reagents

5.1 Methanol

Methanol is a flammable solvent and is toxic to humans. Swallowing of methanol will cause blindness, unconsciousness and death.

5.2 Methanol (95% and saturated with sugar)

Dilute the methanol to 95% by adding 5% (V/V) distilled water depending on the amount needed. Saturate the methanol with refined sugar (about 500 g in 2500 cm^3) and filter through a Whatman No. 91 (or equivalent) filter paper before use.

5.3 Methanol (90% and saturated with sugar)

Dilute the methanol to 90% by adding 10% (V/V) distilled water depending on the amount needed. Saturate the methanol with refined sugar (about 500 g in 2500 cm^3) and filter through a Whatman No. 91 (or equivalent) filter paper before use.

5.4 Diethyl ether (saturated with $CaCl_2$)

Diethyl ether (ether) is a flammable solvent and is toxic to humans. Inhalation will cause loss of consciousness. Ether will also form explosive peroxides when exposed to air and sunlight for long periods. Inspect ether containers for leaks to avoid evaporation.

Calcium chloride ($CaCl_2$) is an irritant and should not be swallowed.

Mix diethyl ether with anhydrous calcium chloride ($CaCl_2$) to remove any water present. Add small amounts of calcium chloride until it stops caking. Filter through a Whatman No. 91 (or equivalent) filter paper before use.

6. Procedure

6.1 Sub-sampling

The final sub-sample must be truly representative of the sample. For this purpose the use of a riffle for mixing and sub-sampling is preferable. Mix and sub-divide the sample until a sub-sample of about 100 - 104 g is obtained. This entire sub-sample must be used for the analysis.

6.2 Washing of the sample

Add 100 cm^3 of the 90% saturated methanol to the sub-sample of sugar in the conical flask and stopper. Tumble for 2 minutes every 15 minutes for a period of 1 hour and siphon the methanol through the strainer stopper under vacuum in a fume cupboard, removing as much of the methanol as possible. Add 50 cm^3 of 95% saturated methanol to the flask through the strainer stopper to wash any crystals adhering to the stopper back into the flask. When opening the stopper work on a clean sheet of paper to catch and recover any grain that may escape the flask. Replace the solid stopper, tumble for 2 minutes and remove the methanol as before. Repeat the 95% saturated methanol washing step three more times.

Add 50 cm^3 saturated diethyl ether, tumble for 1 minute and remove the diethyl ether through the strainer stopper under vacuum. Repeat the diethyl ether washing three more times. After draining the final portion of ether, keep the flask under vacuum until the crystals are dry and no longer cling to the sides of the flask. Spread the crystals on a piece of clean paper to air dry for approximately 30 minutes. Return the crystals to the conical flask and shake gently to ensure that the crystals are free-flowing and dry.

6.3 Sieving

Weigh each sieve and the base pan to the nearest 0.01 g on the top pan balance. Assemble the sieves in descending order of aperture size and include the base pan and the lid. In this way the sieve with the largest aperture size should be on top with the lid and the base pan should be at the bottom. Weigh the sub-sample obtained in 6.1 to the nearest 0.01 g and transfer to the top sieve. Replace the lid, attach the stack of sieves to the mechanical shaker and shake for approximately 15 minutes. Remove the stack of sieves from the shaker and carefully reweigh each sieve and base pan with its retained sugar to the nearest 0.01 g.

7. Calculations

7.1 Size fraction percentage of total sample

Determine the amount of sugar retained by each sieve and the base pan from their differences in weights before and after shaking. Sum the amounts retained by each sieve and the base pan. This sum must equal the weight of the test portion used in 6.3 to within 0.6 g. If not the test should be repeated. Express the quantity of sugar remaining on each sieve as a percentage of the total mass of sugar used for the test.

Report the percentage of the sample that has crystals larger than the aperture of the 1000 µm sieve (sum of percentages on 1700, 1180 and 1000 µm sieves) and the percentage of the sample that has crystals smaller than the 600 µm sieve (the sum of the percentages on the base pan and the 355 µm pan).

7.2 Specific grain size (SGS)

Each size fraction percentage is multiplied by the specific surface (U) of the sieve which is the ratio between the total surface of all the particles and the total surface of the same mass of particles in 1 cm diameter. U is calculated using Zunker's formula:

$$U = \frac{4.343}{(\log d_2 - \log d_1)} \times \left(\frac{1}{d_1} - \frac{1}{d_2} \right)$$

where d_1 ≡ smallest aperture (mm)
d_2 ≡ largest aperture (mm)

In the case of the top sieve d_1 = 1.7 mm and d_2 = 3.4 mm (*i.e.* 2 × 1.7 mm).

In the case of the bottom sieve (pan) d_1 = 0.1775 mm (*i.e.* ½ × 0.355 mm) and d_2 = 0.355 mm.

The specific surfaces (U) of the sieves used are indicated in Table 1.

Table 1: Specific surface of the sieves for calculation of the specific grain size (SGS)

Aperture size (mm)	Aperture size (mm)	Specific surface, U
1700	1.700	4.2
1180	1.180	7.1
1000	1.000	9.2
600	0.600	13.1
355	0.355	21.9
pan	pan	40.6

Each factor is multiplied by the percentage of sugar retained on the corresponding sieve. The sum of these products is divided into 1 000 which gives the specific grain size (SGS) in mm.

7.3 Mean aperture (MA) and coefficient of variance (CV) using the Rens calculations

The particle size distribution is assumed to form a bell shaped curve which is best described using a logarithmic scale. To express the distribution on a linear scale the cumulative percentage (y) retained on each sieve can be converted to a corresponding linear value (*z*) by using the following function:

For cumulative percentages, y < 50:

$$z = -34.3 \times \left[\sqrt[1.14]{\ln \frac{50}{y}} - e^{-0.18y} \right]$$

For cumulative percentages, y > 50:

$$z = 34.3 \times \left[\sqrt[1.14]{\ln \frac{50}{(100 - y)}} - e^{-0.18(100-y)} \right]$$

If the cumulative percentage (y) equals 50 then the calculated value (*z*) is 0. The formula is only applied to accumulative percentages greater than 10% and less than 90%.

The linear equation obtained is used to calculate the mean aperture of the sample (mm). The coefficient of variation is calculated by subtracting the aperture retaining 16% of the sample (calculated using the linear equation) from the mean aperture and expressing the result (which is the standard deviation) as a percentage of the mean aperture (%).

8. Expression of results

The following results are reported as required:

Percentage on 1000 µm (%) to the nearest unit
Percentage through 600 µm (%) to the nearest unit
SGS (mm) to two decimal places
MA (mm) to two decimal places
CV (%) to the nearest unit

9. Example

Table 2: Example

	1700 µm	**1180 µm**	**1000 µm**	**600 µm**	**355 µm**	**pan**
Mass of sieve + sugar	552.79	538.48	534.49	563.88	499.78	612.66
Mass of sieve	552.69	536.16	527.53	507.74	472.19	606.75
Mass of sugar	0.10	2.32	6.96	56.14	27.59	5.91
						Total mass of sugar = 99.02
% sugar	0.10	2.34	7.03	56.70	27.86	5.97
U	4.2	7.1	9.2	13.1	21.9	40.6
Product	0.42	16.61	64.68	742.77	610.13	242.38
Accumulative %	0.10	2.44	9.47	66.17	94.03	100.00

U º specific surface of the sieve; Accumulative % = % sugar + all previous % sugars

9.1 Percentages

Percentage on 1000 µm = (0.10 + 2.34 + 7.03)%
= 9.47%

Report as 10%

Percentage through 600 µm = (27.86 + 5.97)%
= 33.83%

Report as 34%

9.2 SGS

Sum of products = 1676.99 mm
SGS = 1000 ÷ 1676.99 mm
= 0.5963 mm

Report as 0.60 mm

9.3 MA and CV

Table 3: Rens calculations

Sieve	Aperture (mm)	Percentage (%)	Accumulative %, y (%)	z
1700 µm	1.70	0.10	0.10	-136.40
1180 µm	1.18	2.34	2.44	-68.30
1000 µm	1.00	7.03	9.47	-47.37
600 µm	0.60	56.70	66.17	14.96
355 µm	0.36	27.86	94.03	54.74
Pan	-	5.97	100.00	-

Linear regression of d and z gives a straight line (R^2 = 0.997) with:

slope = -0.007
constant = 0.711

Therefore

MA = 0.7108 mm
CV = 36.0950%

Report MA as 0.71 mm
Report CV as 36%

10. Precision

The tolerance associated with the analysis is ± 2% for the percentage on 1000 µm, ± 3% for the percentage through 600 µm, ± 0.03 mm for SGS, ± 0.04 mm for MA and ± 5% for CV.

11. References

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