Method 8.11 – Refined sugar: grain size distribution

1. Rationale

The method is applicable to refined and white sugars and determines the grain size distribution of the sample.

2. Principle

A representative sample is segregated into size fractions in a set of appropriately woven wire cloth sieves. The weight 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 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 850, 600, 500 and 355 μm are used for refined sugars.

4.4 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 are recommended.

5. Procedure

5.1 Sub-sampling

- Use a riffle for mixing and sub-dividing the sample. Mix and sub-divide until a sub-sample of about 100 - 104 g is obtained. This entire sub-sample must be used for the analysis. The final sub-sample must be truly representative of the sample.

5.2 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 5.1 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.

6. Calculations

6.1 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 5.2 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 850 μm sieve and the percentage of the sample that has crystals smaller than the 355 μm sieve.

6.2 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 = 0.85 mm and d_2 = 1.70 mm (*i.e.* 2 × 0.85 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 (µm)	Aperture size (mm)	Specific surface, U
850	0.850	8.5
600	0.600	14.1
500	0.500	18.3
355	0.355	23.9
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.

6.3 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 (%).

7. Expression of results

The following results are reported as required:

Percentage on 850 µm (%) to the nearest unit
Percentage through 355 µm (%) to the nearest unit
SGS (mm) to two decimal places
MA (mm) to two decimal places
CV (%) to the nearest unit

8. Example

Table 2: Example

	850 μm	600 μm	500 μm	355 μm	Pan
Mass of sieve + sugar	542.25	540.96	527.72	491.41	619.48
Mass of sieve	529.48	507.8	507.38	472.22	604.78
Mass of sugar	12.77	33.16	20.34	19.19	14.70
Total mass of sugar = 100.16 g					
% sugar	12.75	33.11	20.31	19.16	14.68
U	8.5	14.1	18.3	23.9	40.6
Product	108.38	466.85	371.67	457.92	596.01
Accumulative %	12.75	45.86	66.17	85.33	100.01

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

8.1 Percentages

Percentage on 850 μm = 12.75%

Report as 13%

Percentage through 355 μm = 14.68%

Report as 15%

8.2 SGS

Sum of products = 2000.83
SGS = 1000 ÷ 2000.83
= 0.4999

Report as 0.50 mm

8.3 MA and CV

Table 3: Rens calculations

Sieve	Aperture (mm)	Percentage (%)	Accumulative %, y (%)	z
850 μm	0.850	12.75	12.75	-41.65
600 μm	0.600	33.11	45.86	-4.00
500 μm	0.500	20.31	66.16	14.96
355 μm	0.355	19.16	85.32	38.56
Pan	-	14.68	100.00	-

Linear regression of d and z gives a straight line (R^2 = 0.998) with:

slope = -0.006
constant = 0.588

Therefore

MA = 0.5883 mm
CV = 38.15%

Report MA as 0.59 mm
Report CV as 38%

9. Precision

The tolerances associated with the analysis is ± 2.5% for the percentage on 850 µm, ± 3% for the percentage through 355 µm, ± 0.03 mm for SGS, ± 0.03 mm for MA and ± 2% for CV.

10. References

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