Method 7.9 – Raw sugar: starch by the SASTA Simplified method

1. Rationale

This method is applicable to VHP sugars only and is based on the colorimetric determination of the starch/iodine complex in the presence of sugar. The method is easier but less accurate than the SMRI starch method (Method 7.8) and its use should be restricted to purposes of factory control.

2. Principle

Starch consists predominantly of amylose and amylopectin. This method is mainly sensitive to amylose. The starch is not separated from the sugar and the method will therefore experience interference from the large sugar bulk. The starch is solubilized and reacted with iodine. The colour of the starch/iodine complex is measured spectrophotometrically at a wavelength of 600 nm. A standard graph prepared using a standard potato starch solution is used.

3. Apparatus

3.1 **Spectrophotometer** operating at 600 nm

3.2 **Optical glass cell**: 10 mm

3.3 **Oven** operating at 105 ± 5°C

3.4 **Desiccator** with self-indicating silica gel

3.5 **Analytical balance** readable to 0.0001 g

3.6 **Beakers**: 100 and 5 × 250 cm^3

3.7 **Volumetric flasks**: 5 × 50, 100, 500 and 1 000 cm^3

3.8 **Pipettes**: 5, 10 (graduated), 15 and 20 cm^3

3.9 **Hot plate**

A thermostatically controlled hotplate tray covered with a layer of sand to ensure even boiling must be used.

3.10 **Watch glass**: 100 mm ϕ

3.11 **Moisture dish** with lid: 80 mm ϕ, 10 mm height

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

3.13 **Glass beads**

4. Reagents

4.1 Acetic acid

Acetic acid (CH_3COOH) in its concentrated form (also called glacial acetic acid) is corrosive and flammable and should not be inhaled. Always open in a fume cupboard using gloves and safety glasses. Decant carefully into clean and dry smaller containers for regular use.

4.2 Potasium iodide solution (10%)

Potassium iodide (KI) is an irritant. Wear gloves and safety glasses during use.

The reagent is unstable and must be prepared immediately prior to use.

Weigh 10.0 g potassium iodide and dissolve in distilled water. Transfer to a 100 cm^3 volumetric flask and make to the mark.

4.3 Potassium iodate-acetic acid solution (0.0017 M KIO_3)

Potassium iodate (KIO_3) is explosive and should be kept away from other chemicals. Handle in a fume cupboard while wearing gloves and safety glasses. Toxic fumes may form above 100°C.

Dry the potassium iodate powder overnight in a desiccator before use. Weigh 0.3567 g and dissolve in some distilled water. Add 57 cm^3 glacial acetic acid, transfer to a 1 000 cm^3 volumetric flask and make to the mark.

4.4 Potato starch, BDH

4.5 Refined sugar: first boiling sugar

5. Procedure

5.1 Starch moisture content

Weigh accurately about 1 g potato starch into the moisture dish and dry in the oven at 105 ± 5°C for 1½ hours. Cool in a desiccator and reweigh. From the loss in mass, calculate the moisture content of the starch (6.1). All subsequent masses must be adjusted accordingly to give a known mass of dried starch. The dried sample must be discarded as it will not have the same solubility as fresh starch (retrogradation).

5.2 Preparation of the standard graph

Prepare a stock solution of starch by adding 500 mg fresh potato starch to 10 cm^3 of distilled water in a 100 cm^3 beaker to make a slurry. Pour the slurry into 300 cm^3 boiling distilled water with glass beads being careful to rinse the beaker well with water. Continue boiling for 1 minute. Cool and transfer the solution quantitatively into a 500 cm^3 volumetric flask with distilled water. Make to the mark with distilled water. This is a 1 000 mg/litre solution and 1 cm^3 contains 1 mg starch.

Prepare the standard starch solutions exactly according to the amounts indicated in Table 1 in 250 cm^3 beakers to a volume of about 70 cm^3. Dissolve the sugar by swirling.

Table 1: Standard starch solutions

Standard	Refined sugar (g)	Stock starch aliquot (cm^3)	Starch concentration (mg/litre)
1	25	0	0
2	25	2.5	10
3	25	5.0	20
4	25	7.5	30
5	25	10.0	40

Cover each beaker with a watch glass, place on the hot plate and bring to the boil. Boil gently for 2 minutes then cool in running water.

Transfer the cooled solution to a 100 cm^3 volumetric flask and make to the mark. Pipette 10 cm^3 of this solution into a 50 cm^3 volumetric flask and add 15 cm^3 distilled water. Add 0.5 cm^3 of the potassium iodide (KI) solution and 5 cm^3 of the potassium iodate-acetic acid solution. Mix thoroughly, make to the mark and measure the absorbance at 600 nm in a 10 mm cell, using water as the reference.

5.3 Sample Preparation

Weigh 25.0 ± 0.1 g of the sample sugar into a 250 cm^3 beaker and dissolve in 30 cm^3 hot water. Cover the beaker with a watch glass, place on the hot plate and bring to the boil. Boil gently for 2 minutes and cool in running water.

Transfer the cooled solution to a 100 cm^3 volumetric flask and make to the mark. Pipette 20 cm^3 of this solution into a 50 cm^3 volumetric flask. Prepare a reagent blank using 20 cm^3 distilled water. Add 15 cm^3 distilled water, 0.5 cm^3 of the potassium iodide (KI) solution and 5 cm^3 of the potassium iodate-acetic acid solution. Mix thoroughly, make to the mark and measure the absorbance at 600 nm in a 10 mm cell, using water as the reference.

6. Calculations

6.1 Starch moisture

$$\text{Starch moisture (\%)} = \frac{M_1 - M_2}{M_1} \times 100$$

where M_1 ≡ mass of starch before drying (g)
M_2 ≡ mass of starch after drying (g)

6.2 Standard graph

Subtract the absorbance of the blank (standard 1) from the absorbencies of the other five solutions. Plot these absorbance values (AU) against the starch concentrations (mg/litre), taking the moisture content of the starch into account when calculating the starch concentrations of the standard solutions. This graph should be a straight line passing through the origin. The slope is calculated (absorbance over concentration) and used in the calculation of starch in the samples.

6.3 Samples

Subtract the absorbance of the sample blank from the absorbance of the sample solution to get the absorbance of the sample. Calculate the amount of starch per sample in mg/litre according to the equation below.

$$\text{Starch in sample} = \frac{\text{absorbance}}{\text{slope}} \times \frac{100\ \text{cm}^3}{1000} \times \frac{50\ \text{cm}^3}{\text{aliquot}} \div 0.025\ \text{kg}$$

$$= \frac{\text{absorbance}}{\text{slope} \times \text{aliquot}} \times 200$$

Report to the nearest 5 mg/kg.

7. Example

7.1 Standard graph

Starch moisture = 16.95%

500 mg of starch therefore contains 415.3 mg dry starch

Calculate the starch concentration of each standard in terms of the dry starch according to the following formula (results are indicated in Table 2):

$$\text{Starch concentration (mg/litre)} = \frac{\text{dry mass}}{500\ \text{cm}^3} \times \frac{\text{aliquot}}{100\ \text{cm}^3} \times \frac{20\ \text{cm}^3}{50\ \text{cm}^3} \times 1000$$

Table 2: Calculated standard starch solutions

Standard	Absorbance (AU)	Absorbance (Solution - Blank)	Starch concentration (mg/litre)
1	0.011	-	-
2	0.182	0.171	8.3
3	0.353	0.342	16.6
4	0.524	0.513	24.9
5	0.695	0.684	33.2

From the standard graph, the slope is 0.0206 AU litre/mg.

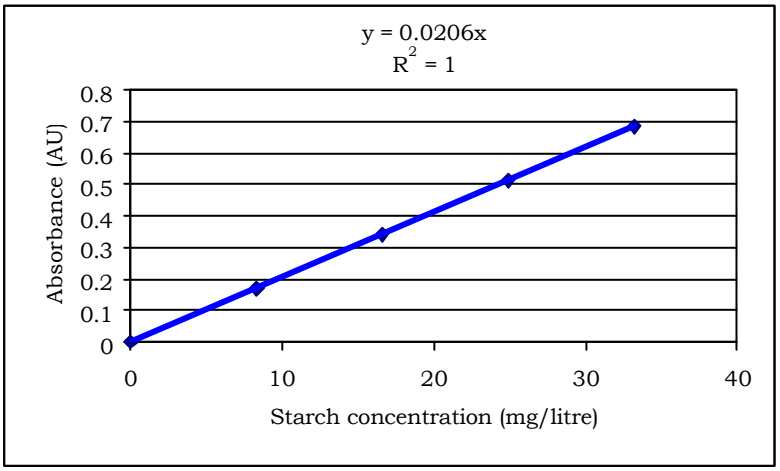

Figure 1: Standard starch curve

6.3 Samples

absorbance of solution = 0.236 AU
absorbance of blank = 0.008 AU
absorbance of sample = (0.236 – 0.008) AU
= 0.228 AU

$$\text{starch in sugar} = \frac{0.228 \text{ AU}}{0.0206 \text{ AU litre/mg} \times 20} \times 200$$

$$= 110.68 \text{ mg/kg}$$

Report as 110 mg/kg

7. References

SASTA (1985). *Laboratory Manual for South African Sugar Factories.* 3rd Edition: 198 - 200, 326 - 327.