



**Australian Government**

**National Measurement  
Institute**

## NITP 15.2 National Instrument Test Procedures for Measuring Instruments for Cane Sugar Quality

Part 1: Self-indicating Polarimetric Saccharimeters

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## PREFACE

NMI's Chief Metrologist has determined that NITP 15.2 Part 1 contains the test procedures for the verification of measuring instruments for cane sugar quality including self-indicating polarimetric saccharimeters.

## ABBREVIATIONS

$CT_{QP}$	correction to the quartz plate certified value when the temperature inside the trough compartment ( $T_t$ ) is not 20 °C (reference temperature as stated on the certificate)
$Error_{20}$	accuracy error associated with each value of $I_{20}$ (expressed in °Z), i.e. indicated value that has been corrected automatically to 20 °C minus the certified value of the quartz plate (i.e. $I_{20} - QP_{20}$ )
$Error_t$	accuracy error associated with each value of $I_t$ (expressed in °Z), i.e. indicated value at $T_t$ minus the value of the quartz plate corrected to $T_t$ (i.e. $I_t - QP_{CTt}$ )
$I_{20}$	value indicated by a saccharimeter with a facility to correct the sample temperature to 20 °C (expressed in °Z)
$I_t$	value indicated by a saccharimeter at temperature $T_t$ (may be expressed in °Z units provided that the quartz plate temperature is 20 °C)
ICUMSA	International Commission for Uniform Methods of Sugar Analysis
MPD	maximum permissible difference (expressed in °Z)
MPE	maximum permissible error (expressed in °Z)
$QP_{20}$	certified sugar value of the quartz plate at 20 °C and specified wavelength (expressed in °Z)
$QP_{CTt}$	value of the certified quartz plate corrected to $T_t$ (i.e. $QP_{20} + CT_{QP}$ )
$T_t$	temperature inside the trough compartment supporting the sample at the time of measurement
°Z	unit of the ICUMSA International Sugar Scale which quantifies the degree of optical rotation in aqueous solution of sucrose

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## EXPLANATION OF TERMS

For metrological terms not listed below, see *General Information for Test Procedures*. For other terms relating to polarimetry, refer to [NMI R 14 Self-indicating Polarimetric Saccharimeters Graduated in Accordance with the ICUMSA International Sugar Scale](#).

### Adjustment

Alteration of the measurement parameters to bring the instrument within the allowable maximum permissible errors for an instrument in use.

### Angular Degree Optical Rotation

Regarding plane-polarised light: the measured rotation of the plane of polarisation during transmission through an aqueous solution in angular degrees ( $^{\circ}$ ).

### Calibration

The set of operations that (under specified conditions) establishes the relationship between the indicated or nominal value of an instrument and the corresponding known value of the measured quantity.

### Certified Value/Sugar Value

The optical rotation value ( $^{\circ}\text{Z}$ ) of a quartz plate with reference to the indicated wavelength and temperature ( $20^{\circ}\text{C}$ ) stated in the Regulation 13 or Regulation 21 certificate.

### Correction

Compensation for an estimated systematic effect, e.g. the temperature of a sample affects the rotation of linear polarised light transmitted through it.

### In-service Inspection

The examination of an instrument by a trade measurement inspector to check that:

- the verification mark is valid; and
- the errors do not exceed the MPEs permitted for in-service inspection.

In-service inspection does not permit the instrument to be marked with a verification mark.

### Indication

The value indicated on a saccharimeter (i.e.  $I_t$  or  $I_{20}$ ).

### Measured Value

The value attributed to the optical rotation of a sample at a specified temperature. The value can be obtained from a single indication or the mean value from repeat measurement cycles. If reporting in  $^{\circ}\text{Z}$  units, it is necessary to correct it to the  $20^{\circ}\text{C}$  reference temperature.

Note: For the purpose of this document, the measured value is based on a single indication.

### Measurement Result

The measured value together with relevant information, e.g. method description, expanded uncertainty etc.

### Optical Rotation

The angular degree optical rotation normalised to the ICUMSA International Sugar Scale (measured in  $^{\circ}\text{Z}$ ). An optical rotation equivalent to that caused by the 'normal sugar solution' (a pure sucrose solution defined by ICUMSA) signifies  $100^{\circ}\text{Z}$  on the International Sugar Scale. An optical rotation equivalent to that caused by pure water signifies  $0^{\circ}\text{Z}$ .

### Polarimetric Saccharimeter

A device that transmits linear polarised light through a sample (typical of which are solutions of sugars such as sucrose) and measures the resulting optical rotation in  $^{\circ}\text{Z}$ .

### Sample/Substance to be Analysed

Optically active substance (e.g. quartz plate, sugar solution prepared with or without clarification products, a mixture based on mill products) that causes the plane of polarisation of transmitted plane-polarised light to be rotated.

### Verification

The examination of an instrument by a trade measurement inspector, servicing licensee or an employee of a servicing licensee (verifier) in order to mark the instrument indicating that it conforms with the relevant test procedures.

**Initial verification** is the verification of a new instrument, which does not bear a verification mark and has never been verified before.

## 1. SCOPE

NITP 15.2 Part 1 describes the test procedures for the verification and in-service inspection of self-indicating polarimetric saccharimeters (hereafter referred to as saccharimeters) to ensure that they measure within the maximum permissible errors (MPEs) specified in the *National Trade Measurement Regulations 2009* (Cth), and comply with their certificate of approval.

Certificates of approval are based on [NMI R 14 Self-indicating Polarimetric Saccharimeters Graduated in Accordance with the ICUMSA International Sugar Scale](#). Refer to NMI R 14 for all metrological and technical requirements.

Note: Saccharimeters have either automatic or manual temperature correction for the environment and sample.

## 2. EQUIPMENT

Record details of the equipment used on the test report.

1. Certificate(s) of approval for the saccharimeter and any ancillary components or additional devices.
2. Appropriate quartz plate reference standards of measurement, namely at least four quartz plates with various certified °Z optical rotation values at the wavelength of the saccharimeter (see Table 1 for selection guidelines). Mandatory rotation values depend on:
  - maximum measuring range of the saccharimeter;
  - optical rotation values of the typical products analysed; and
  - accuracy class of the saccharimeter.

Select additional plates judiciously to fill the remainder of the measuring range and ensure that at least a minimum of four °Z values are checked.

3. Current Regulation 13 or Regulation 21 certificates for all quartz plate reference standards. Uncertainties and variations must not be greater than one-third of the relevant MPE for the accuracy class of the saccharimeter.
4. For saccharimeters with manual temperature correction, a traceable liquid-in-glass or digital thermometer (minimum range 15–35 °C, maximum scale interval 0.1 °C, maximum uncertainty ±0.2 °C).
5. Test report (see Annex A).

## 3. VISUAL INSPECTION

Visually inspect the saccharimeter and record details of the required data and characteristics of the saccharimeter on the test report.

### 3.1 Required Data

1. Test report reference number
2. Date of test
3. Type of test
4. Name of owner/user
5. Address of owner/user
6. Name of contact on premises
7. Address where located
8. Description of saccharimeter
9. Manufacturer
10. Model
11. Operating wavelength

Table 1. Mandatory sugar values in the set of quartz plates for verification

Accuracy class	Number in set	Optical rotation values	Notes
0.1	1	One plate within the typical sugar value of the product routinely analysed	For example +75 °Z for cane juice
	2	+100 °Z or sugar value within 10% of saccharimeter max	Select +100 °Z if in the measuring range
	3	–25 °Z or +25 °Z	Select –25 °Z if in the measuring range
	4	Any value (ideally reducing the largest gap in the range)	Mandatory plate
	5+	Any value	Optional plates
0.05 and 0.02	1–2	At least two plates within the typical sugar value of the product(s) routinely analysed	For example +95 °Z and +98 °Z for sugar analysis
	3	+100 °Z or sugar value within 10% of saccharimeter max	Select +100 °Z if in the measuring range
	4	–25 °Z or +25 °Z	Select –25 °Z if in the measuring range
	5+	Any value	Optional plates

12. Serial number
13. Certificate of approval number
14. Accuracy class
15. Measuring range
16. Length(s) of the pol. tubes routinely used
17. Temperature correction status (automatic or manual)
18. Quartz plate(s) serial number and optical rotation
19. Product type(s) analysed and typical optical rotation range(s)

### 3.2 Characteristics of the Saccharimeter

Saccharimeters shall comply with the following statements.

1. The saccharimeter complies with its certificate of approval.
2. All mandatory descriptive markings are clearly and permanently marked on a data plate.  
Note: Some markings may be required on the indicating device.
3. The saccharimeter is complete.
4. All seals (physical and electronic) are intact.
5. The saccharimeter is on a firm base.
6. The saccharimeter is clean.
7. The saccharimeter is adequately protected against influences that are likely to affect its performance.  
Note: Refer to the certificate of approval or the data plate for any exposure limits to dust, air movement, vibrations, atmospheric conditions, direct radiation (e.g. sunlight), temperature and other influences.
8. For additional indicating devices: they exactly repeat the information on the primary indication.
9. For ticket/label printing devices: they comply with the requirements of General Supplementary Certificate S1/0B.

## 4. TEST PROCEDURES

The following test procedures, together with any test procedures specified in the certificate of approval, determine if the performance of the saccharimeter meets requirements and whether it requires adjustment or service. Record all results on the test report.

Each test procedure is explained as a discrete test. However tests can be combined to expedite the testing procedure. A suggested sequence for testing is shown in clause 5.

### 4.1 Setup

- 4.1.1 Prior to taking measurements, ensure the saccharimeter has been switched on for longer than any minimum warm-up time specified in the certificate of approval or manufacturers' specifications. If no minimum time is specified, warm-up for one hour.
- 4.1.2 While testing, store the set of quartz plates required for testing in the trough compartment. If the quartz plates do not fit in, place them nearby, but away from any source of heat or air currents (e.g. fans).  
Note: During testing, only one quartz plate is in the trough cradle.
- 4.1.3 For saccharimeters with **automatic** temperature correction: ensure it is enabled.

For saccharimeters requiring **manual** temperature correction: place the thermometer in the storage location of the quartz plates until the temperature has stabilised.

### 4.2 Repeatability

The difference between the results of four indications obtained from the same value quartz plate shall not be greater than the absolute value of the maximum permissible difference (MPD) for the accuracy class of the saccharimeter.

- 4.2.1 Select the quartz plate with a sugar value within the optical rotation range of the typical substance routinely analysed.
- 4.2.2 Complete the setup described in clause 4.1.
- 4.2.3 For saccharimeters requiring **manual** temperature correction: record the observed temperature and true temperature ( $T_t$ ) of the storage location on the test report (to 0.1 °C).  
Note: Use the temperature correction table when converting the observed temperature to true temperature.
- 4.2.4 Ensure the trough compartment door is closed and the cradle is empty. Allow the reading of the saccharimeter to stabilise and reset to zero.
- 4.2.5 Place the quartz plate in the trough cradle with minimum handling. Close the lid gently.
- 4.2.6 When the reading stabilises, record the indication to two decimal places ( $I_{20}$  for automatic and  $I_t$  for manual temperature correction) on the test report.

- 4.2.7 Remove the quartz plate from the trough cradle, close the lid and check that the indication returns to zero  $\pm 0.02$  °Z. If the indication does not return to within  $\pm 0.02$  °Z of zero, a fault in the saccharimeter is indicated. The test procedure shall be terminated and not restarted until the condition has been rectified.
- 4.2.8 Repeat steps 4.2.3 to 4.2.7 once.
- 4.2.9 For saccharimeters requiring **manual** temperature correction: place the quartz plate in the storage location, allow it to stabilise at  $T_t$ .
- 4.2.10 Repeat 4.2.3 to 4.2.7 twice.
- 4.2.11 For saccharimeters requiring **manual** temperature correction: the optical rotation value of a certified quartz plate in °Z units is only applicable at the reference temperature of 20 °C, so a correction ( $CT_{QP}$ ) is manually applied to  $QP_{20}$  (certified value at 20 °C).  $CT_{QP}$  depends on  $T_t$  (true temperature of the trough supporting the quartz plates).

For all wavelengths under 880 nm:  
 $CT_{QP} = 0.000\ 144 \times QP_{20} \times (T_t - 20)$

For near-infrared 880 – 882.6 nm:  
 $CT_{QP} = 0.000\ 139 \times QP_{20} \times (T_t - 20)$

**First**, calculate  $QP_{CTt}$  (the value of the quartz plate corrected to  $T_t$ ) by adding  $CT_{QP}$  to the quartz plate certified value,  $QP_{20}$ :

$$QP_{CTt} = QP_{20} + CT_{QP}$$

Note:  $CT_{QP}$  is applied in a different manner to the correction stated in Method 1 and Table XXVI in the BSES Manual<sup>1</sup>.  $C_{tq}$  from Table XXVI adjusts the measured value at temperature  $t_q$  to 20 °C, whereas  $CT_{QP}$  adjusts the certified quartz plate optical rotation at 20 °C to the expected rotation value at temperature  $T_t$ .

**Then**, calculate  $Error_t$  (the error associated with each measurement obtained from the quartz plate) by subtracting  $QP_{CTt}$ , (the corrected value

for the quartz plate at temperature  $T_t$ ) from each  $I_t$  value:

$$Error_t = I_t - QP_{CTt}$$

- 4.2.12 For saccharimeters with **automatic** temperature correction: calculate  $Error_{20}$  (the error associated with each measurement obtained from the quartz plate) by subtracting  $QP_{20}$  (the certified value for the quartz plate) from each  $I_{20}$  value:  $Error_{20} = I_{20} - QP_{20}$
- 4.2.13 Determine the maximum and minimum errors and calculate the absolute value of the difference.
- 4.2.14 Determine whether the difference is within the allowable maximum MPD in Table 2.

Table 2. MPDs for repeatability

Accuracy class	Repeatability MPD (°Z)
0.02	0.02
0.05	0.03
0.1	0.05

### 4.3 Accuracy

- 4.3.1 Select test quartz plates in accordance with clause 2, point 2. The first quartz plate to be measured shall be one with a sugar value within the optical rotation range of the typical substance routinely analysed.
- 4.3.2 Follow steps 4.2.3 to 4.2.7 to obtain a value for  $I_t$  at temperature  $T_t$  (or a value for  $I_{20}$  on saccharimeters with automatic temperature correction).
- 4.3.3 Repeat steps 4.2.3 to 4.2.7 on the remaining quartz plates. Randomise the order. Only a single value of  $I_t$  or  $I_{20}$  is required for each quartz plate selected.
- 4.3.4 For saccharimeters requiring **manual** temperature correction: calculate and record  $Error_t$  (the accuracy error associated with each measurement of each quartz plate) by subtracting  $QP_{CTt}$  (the corrected value for the quartz plate at temperature  $T_t$ ) from the value  $I_t$ :  
 $Error_t = I_t - QP_{CTt}$
- 4.3.5 For saccharimeters with **automatic** temperature correction: calculate and record  $Error_{20}$  (the accuracy error associated with each measurement obtained from each quartz plate) by subtracting  $QP_{20}$  (the certified value for the quartz plate) from each  $I_{20}$  value:  
 $Error_{20} = I_{20} - QP_{20}$

<sup>1</sup> Laboratory Manual for Australian Sugar Mills – Volume 2 Analytical Method and Tables, Method 1 – Calibration of Polarimeter by Quartz Plate Check (1991), BSES Limited

- 4.3.6 Determine whether the results are within the allowable MPE listed in Table 3.

Table 3. MPEs for accuracy

Accuracy class	Accuracy MPE (°Z)
0.02	±0.02
0.05	±0.05
0.1	±0.1

## 5. SUGGESTED SEQUENCE FOR TESTING

- Record details of the reference standards.
- Check the certificate of approval for supplementary tests, e.g. tests for additional indicating devices and printers in General Supplementary Certificate S1/0B. If possible, integrate these in the testing sequence.
- Visually inspect the saccharimeter and record the required data (clause 3.1) and characteristics (clause 3.2).
- Set up the saccharimeter and associated equipment as described in clause 4.1.
- Conduct the repeatability and accuracy tests (clauses 4.2 and 4.3). These tests may be conducted separately, however, to decrease the test time, it is recommended that they are combined. Table 4 contains example sequences for combining the tests on a set comprised of four quartz plates.

Table 4. Example sequences for a test set comprised of four quartz plates

Indication (I)	Example order 1	Example order 2	Example order 3
1	Plate 1 (I1)	Plate 1 (I1)	Plate 1 (I1)
2	Plate 1 (I2)	Plate 1 (I2)	Plate 4
3	Plate 4	Plate 4	Plate 1 (I2)
4	Plate 1 (I3)	Plate 1 (I3)	Plate 1 (I3)
5	Plate 3	Plate 1 (I4)	Plate 3
6	Plate 1 (I4)	Plate 3	Plate 1 (I4)
7	Plate 2	Plate 2	Plate 2

- Carry out required calculations and evaluate the results.
- Conduct any supplementary tests that were not integrated in the test procedures.
- Carry out any remaining activities required to complete the procedure. See *General Information for Test Procedures* for more information. This may include:
  - completing the test report;
  - removing traces of the verification mark from the saccharimeter;
  - applying a verification mark; and
  - applying a seal.

## APPENDIX A. TEST REPORT

The following test report contains the minimum amount of information that must be recorded.

If the certificate(s) of approval requires additional tests, attach pages that record the results of these tests.

Number each page of the test report in the style shown at the top of each page.

**Test Report for Self-indicating Polarimetric Saccharimeters**

Test report reference number..... Date of test.....

Type of test (tick one)       Verification                       In-service inspection

For in-service inspection, record the verification mark.....

Name of owner/user .....

Address of owner/user.....

Name of contact on premises.....

Address where located.....

Description of saccharimeter (select one):

 Mechanical circular polariser                       Optomagnetic polariser                       Quartz wedge polariser Other balancing mechanism (give details) .....

Manufacturer..... Model..... Operating wavelength (nm).....

Serial number ..... Certificate(s) of approval number .....

Measuring range (°Z)                      Minimum ..... Maximum.....

Length(s) of the pol tubes routinely used:

 200 mm                       100 mm                       50 mm                       10 mm

Temperature correction status (select one):

 Automatic                       Manual (automatic facility unavailable/disabled)

Quartz plate(s) serial number and optical rotation:

Serial number ..... Optical rotation (°Z) .....

Serial number ..... Optical rotation (°Z) .....

Product type(s) analysed and typical optical rotation range(s):

Product/sample..... Optical rotation (°Z)    min ..... max.....

Product/sample..... Optical rotation (°Z)    min ..... max.....

Product/sample..... Optical rotation (°Z)    min ..... max.....

Does the saccharimeter comply with its certificate of approval?	<input type="checkbox"/> Yes <input type="checkbox"/> No
Are all mandatory descriptive markings clearly and permanently marked on the data plate?	<input type="checkbox"/> Yes <input type="checkbox"/> No
Is the saccharimeter complete?	<input type="checkbox"/> Yes <input type="checkbox"/> No
Are all seals (physical and electronic) intact?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> n/a
Is the saccharimeter on a firm base?	<input type="checkbox"/> Yes <input type="checkbox"/> No
Is the saccharimeter clean?	<input type="checkbox"/> Yes <input type="checkbox"/> No
Is the saccharimeter adequately protected against influences that are likely to affect its performance?	<input type="checkbox"/> Yes <input type="checkbox"/> No
For additional indicating devices: do they exactly repeat the information on the primary indication?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> n/a
For devices for ticket/label printing: do they comply with the requirements of General Supplementary Certificate S1/0B?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> n/a

**Details of the Standards of Measurement (see clause 2)**

<b>Quartz plates</b>	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>	<b>5</b>	<b>6</b>
Serial number						
Certified optical rotation at saccharimeter wavelength (°Z)						
Expanded uncertainty (k = 2) (°Z)						
Regulation 13 or 21 number						
Regulation 13 or 21 expiry date						
<b>Thermometer</b> (only for saccharimeters with manual temperature correction)						
Serial number						
Smallest scale graduation/interval (°C)						
Uncertainty of corrections (k = 2) (°C)						
Calibration expiry date						

**Test Results for Automatic Temperature Correction (see clauses 4.2 and 4.3)**

Plate number	QP <sub>20</sub> (°Z)	I <sub>20</sub> (°Z)	Error <sub>20</sub> (°Z) (I <sub>20</sub> - QP <sub>20</sub> ) (for comparison with MPE)	Error <sub>20</sub> range (°Z) (for comparison with MPD)
1	1			
	2			
	3			
	4			
2				
3				
4				

**Test Results for Manual Temperature Correction (see clauses 4.2 and 4.3)**

Plate number	QP <sub>20</sub> (°Z)	Observed temp (°C)	True temp T <sub>t</sub> (°C)	I <sub>t</sub>	CT <sub>QP</sub> <sup>*</sup>	QP <sub>CTt</sub> (QP <sub>20</sub> + CT <sub>QP</sub> )	Error <sub>t</sub> (°Z) (I <sub>t</sub> - QP <sub>CTt</sub> ) (for comparison with MPE)	Error <sub>t</sub> range (°Z) (for comparison with MPD)
1	1							
	2							
	3							
	4							
2								
3								
4								

\*  $0.000\ 144 \times QP_{20} \times (T_t - 20)$  or  $0.000\ 139 \times QP_{20} \times (T_t - 20)$

Did the saccharimeter pass the inspection, repeatability and accuracy tests?  Yes  No

Was the saccharimeter verified?  Yes  No

Verifier's name ..... Identification number .....

Signature .....

Comments .....

.....

**Temperature Correction Table to Convert Observed Temperature to True Temperature**

Observed temp (°C)	Correction (°C)		True temp (°C)	Observed temp (°C)	Correction (°C)		True temp (°C)	Observed temp (°C)	Correction (°C)		True temp (°C)
	Certified	Interpolated			Certified	Interpolated			Certified	Interpolated	
14.0				19.7				25.4			
14.1				19.8				25.5			
14.2				19.9				25.6			
14.3				20.0				25.7			
14.4				20.1				25.8			
14.5				20.2				25.9			
14.6				20.3				26.0			
14.7				20.4				26.1			
14.8				20.5				26.2			
14.9				20.6				26.3			
15.0				20.7				26.4			
15.1				20.8				26.5			
15.2				20.9				26.6			
15.3				21.0				26.7			
15.4				21.1				26.8			
15.5				21.2				26.9			
15.6				21.3				27.0			
15.7				21.4				27.1			
15.8				21.5				27.2			
15.9				21.6				27.3			
16.0				21.7				27.4			
16.1				21.8				27.5			
16.2				21.9				27.6			
16.3				22.0				27.7			
16.4				22.1				27.8			
16.5				22.2				27.9			
16.6				22.3				28.0			
16.7				22.4				28.1			
16.8				22.5				28.2			
16.9				22.6				28.3			
17.0				22.7				28.4			
17.1				22.8				28.5			
17.2				22.9				28.6			
17.3				23.0				28.7			
17.4				23.1				28.8			
17.5				23.2				28.9			
17.6				23.3				29.0			
17.7				23.4				29.1			
17.8				23.5				29.2			
17.9				23.6				29.3			
18.0				23.7				29.4			
18.1				23.8				29.5			
18.2				23.9				29.6			
18.3				24.0				29.7			
18.4				24.1				29.8			
18.5				24.2				29.9			
18.6				24.3				30.0			
18.7				24.4				30.1			
18.8				24.5				30.2			
18.9				24.6				30.3			
19.0				24.7				30.4			
19.1				24.8				30.5			
19.2				24.9				30.6			
19.3				25.0				30.7			
19.4				25.1				30.8			
19.5				25.2				30.9			
19.6				25.3				31.0			