



# **Bellingham + Stanley**

## **Technical Bulletin**

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**Bulletin No:** P003

**Title:** *Calibration of polarimeters using sugar solutions*

A pure solution of sucrose in water can, in principle, be used to calibrate a polarimeter. However, much care and strict experimental protocol is needed to ensure sufficient accuracy. This technical bulletin provides some explanation of the practical problems of using such a procedure and some recommendations from Bellingham+Stanley.

### ***The 100 °Z definition***

All B+S polarimeters are equipped with the *International Sugar Scale* (ISS) or 'Z' scale. The 100 °Z point of the ISS scale is defined as the optical rotation of a normal solution of pure sucrose in pure water. A normal solution of sucrose is defined as follows:

26.000g (weighed in air) in 100 ml of aqueous solution

The ISS scale can therefore be used to measure the purity of sugar solutions or, conversely, it can be used to calibrate an instrument, provided the sugar solution is prepared with very high accuracy.

### ***Problems in preparing sugar solutions***

Laboratory chemists will know from experience that volumetric methods are prone to considerable error. Measuring 100.00 ml of a solution is extremely difficult because volumetric flasks are often not accurate and strict temperature control is needed to ensure the correct liquid density. Furthermore, when one substance is dissolved in another, there is a heat (enthalpy) of mixing, sometimes exothermic, sometimes endothermic. This means that the solution temperature will change during mixing. This leads to further problems when trying to make an accurate concentration.

Of course, any solution preparation method must ensure the use of reliable materials: Analar (Analytical Reagent Grade) sucrose (with specified moisture content) and distilled or AR water.

A 1 % error (not uncommon) in solution preparation will give an error of the same order in optical rotation.

### ***ICUMSA method***

ICUMSA (*International Commission for Uniform Methods of Sugar Analysis*) is the recognised international body that approves and publishes all methods for use in the Sugar Industry, world-wide. The following method is relevant

### ***Method GS2/3-1 (1994) – The Braunschweig Method for the Polarisation of White Sugar by Polarimetry***

In this method a strict protocol for the preparation of sugar solutions is described. For example, the method specifies such things as:

- Special 100ml flasks that conform to ICUMSA standards (tolerance  $\pm 0.1$  ml)
- Sample equilibration times
- temperature control and measurement to within  $0.1^{\circ}\text{C}$
- volume correction

Note that this method is aimed at the measurement of test solution using a polarimeter which has been calibrated using a quartz plate. However, it serves to illustrate the special care needed in preparing sugar solutions.

### ***Weighing method***

It is possible to improve the accuracy in preparing sugar solutions by weighing the sugar and water. To do this a relationship is needed relating sugar solution volumetric concentration to weight % or mass fraction. Available by request is a set of data showing such a relationship for sucrose solutions. This is in the form of a spreadsheet which can accept specific calculations; the 26.000 g/ml calculation is illustrated.

### ***B+S Recommendations***

1. B+S do not recommend the use of sugar solutions for calibrating polarimeters; errors in sample preparation are likely to be too large. Also, as the sample decays (inverts) the optical rotation value will change. Therefore only FRESH sucrose solutions may be used to calibrate a polarimeter.
2. For the accurate calibration of polarimeters, B+S recommend the use of a Quartz Control Plate.

They have the advantages:

- The value is reliable and traceable
- A quartz plate is stable and provides a long term reference
- Operator errors are considerably reduced

However, quartz plates still require good temperature control to ensure sufficient accuracy. The operator must ensure that the exact temperature of the plate is known or the instrument is measuring the correct temperature so that any *automatic temperature compensation* (quartz compensation) applied is using the correct plate temperature.

If using an ADP/S instrument manufactured by B+S Ltd., it is important to ensure the plate has thermally equilibrated with the chamber. The temperature sensor in the chamber will then provide a reliable reading for the compensation calculation. To assist this, a 'thermal block' (code 34-241) is available for making a mechanical connection between the plate and the sensor.

Note, ensure the instrument mode is adjusted for quartz temperature compensation'.

***If a quartz plate is not available and sugar solutions are the only means available, it is essential that a very strict preparation procedure is followed***