The Refractive Index Calibration Procedure

When required	Recommended after exchange of the optical unit or RIM board.
Tools required	Laboratory balance
Parts required	DAB/Ph Eur/BP/JP/NF/USP Grade Sucrose Syringe 9301-1446 Syringe needle 9301-0407 Sample filter 5061-3367 PEEK fitting 0100-1516

Steps

1 Preparation of the Sucrose calibration solution.

2 Preparing the pump.

3 Start Refractive Index Calibration.

4 Flushing the degasser and pump.

5 Purging the sample and reference cells.

6 Fill the sample cell with calibration solution.

7 Calibrate Refractive Index.

Preparation of the Sucrose sucrose Calibration Solution

1 To prepare 25 ml of the calibration solution 87.5 mg of the Sucrose sample is required.

2 Add the weighed amount of sample into a suitable volumetric flask.

3 Dispense 10 ml of LC grade water into the flask and shake to dissolve.

4 Dilute the contents of the flask to volume with LC grade water.

Wait five minutes and shake again. The solution is now ready for use.

Preparation of the Pump

1 Fill a suitable solvent bottle with LC grade water.

2 Connect this bottle to Channel A of the pump, A1 if a binary pump.

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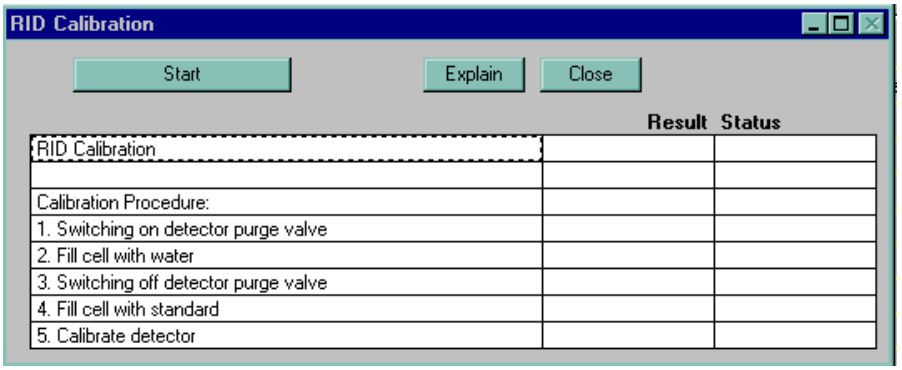
Calibration Procedure for the 1100 Series Refractive Index Detector

Start Refractive Index Calibration

1 From the user interface start the RID Calibration (see Figure 18).
Agilent ChemStation: *Diagnosis - Maintenance - RID Calibration*
Control Module: *System - Tests - RID - Calibrate.*

2 Follow the instructions with reference to the steps below.

Figure 18 **Start Refractive Index Calibration**



Purging the Sample and Reference Cells

1 The purge valve will automatically switch to the On position.

2 Using a syringe or LC pump flush the sample and reference cell with about 20 ml of LC grade water.

3 Click continue and the purge valve will automatically switch to the Off position.

Fill the Sample Cell with Calibration Solution

1 Remove the inlet capillary or flushing syringe from the in port.

2 Take the syringe and fix the needle to the syringe adapter.

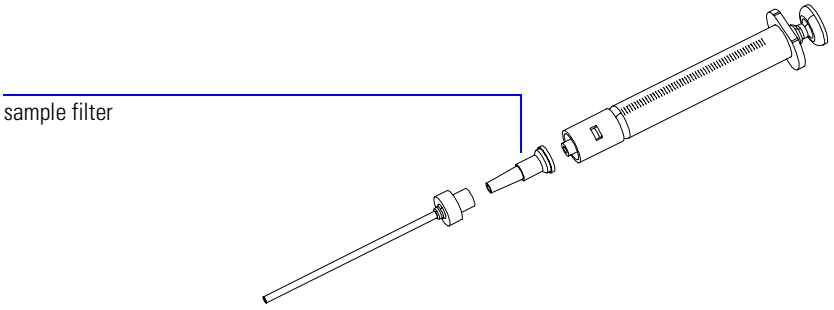
3 Suck about 1.5 ml of the calibration sample into the syringe.

4 Keep the syringe in a horizontal position.

5 Remove the needle.

6 Add the filter to the syringe and fit the needle to filter.

Figure 19 **Syringe with Sample Filter**



7 Lift the needle tip and carefully eject approximately 0.5 ml to remove air out of the syringe and to flush the needle.

8 Add the PEEK fitting to the needle tip and fix both at the flow cell inlet.

NOTE Do not inject the calibration solution without the sample filter.

9 Slowly inject about 1.0 ml and wait for about 10 seconds to inject another 0.1 ml. This will assure that the cell is filled properly.

Calibrate Refractive Index

1 If the detector response differs from the theoretical response of 512,000 nRIU +/- 5,000 nRIU enter the theoretical value (512,000) in the dialog box. If the detector response is within the theoretical response click *OK* (see Figure 20).

NOTE Rinse the sample cell with pure water at a minimum of 1.5 ml/min to flush the Sucrose from the cell and the capillaries. When organic solvent is sequentially applied (without rinsing), a blockage of capillaries may occur.

Figure 20 **Calibrate Refractive Index**