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## Technical Procedure for Glass Refractive Index Measurement System 3 (GRIM 3)

- 1.0 Purpose** - This technical procedure shall be followed for the operation of the GRIM 3.
- 2.0 Scope** - This procedure applies to all Glass Refractive Index Measurement Systems 3 set up for glass samples in the Trace Evidence Section.
- 3.0 Definitions** – N/A
- 4.0 Equipment, Materials, and Reagents**
- 4.1 Equipment**
- Glass Refractive Index Measurement System 3
  - Leica Phase Contrast Microscope
  - Mettler Toledo FT82HT Hot Stage
  - Narrow bandwidth (10 nm, centered on the wavelength of interest  $\pm 5$  nm) light filters
  - Video camera system
  - Processing unit for match-point detection
- 4.2 Materials**
- 19 x 76 mm, 1,0 mm, 100 OT, Microscope slides
  - 18 x 18 – 1, Cover slips
  - Tweezers
  - Kimwipes
  - B2 Locke Scientific Glass Standard
  - B3 Locke Scientific Glass Standard
  - B4 Locke Scientific Glass Standard
  - B6 Locke Scientific Glass Standard
  - B7 Locke Scientific Glass Standard
  - B8 Locke Scientific Glass Standard
  - B9 Locke Scientific Glass Standard
  - B10 Locke Scientific Glass Standard
  - B11 Locke Scientific Glass Standard
  - B12 Locke Scientific Glass Standard
- 4.3 Reagents**
- Immersion liquids, such as Locke oils, Silicone oil Type A, B, and C
  - Calibrated glass standards
  - Acetone
- 5.0 Procedure**
- 5.1 Start-up**

### **5.1.1 Powering Instrument**

- 5.1.1.1** Turn red switch on GRIM 3 main unit ON (this powers the GRIM 3).
- 5.1.1.2** Turn black switch on side of Phase Microscope ON (this powers the light source).
- 5.1.1.3** Turn on computer.
- 5.1.1.4** Allow the system to warm up and stabilize for one hour before taking any measurements.

### **5.2 Calibration/Performance Check**

- 5.2.1** A calibration curve with the B Oil shall be performed annually. Calibration curves with the A and C oils shall be done upon use when the A and C oils are required for the examination.
- 5.2.2** Double click on Stage Manager Icon.
- 5.2.3** Go to menu bar and select Calibrate.
- 5.2.4** Select Calibration Log On.
  - 5.2.4.1** Enter operator information and password.
  - 5.2.4.2** Click OK.
- 5.2.5** Go back to menu bar and select Calibrate.
  - 5.2.5.1** Select the Select Oil-Wavelength.
  - 5.2.5.2** Select (or type in) desired oil and wavelength (e.g., B Oil, 589 nm).
  - 5.2.5.3** Click OK.
- 5.2.6** Go back to Calibrate on menu bar and click Continue Calibration.
- 5.2.7** The calibration page will appear with the previously entered data set. Replace this data with the current data as you proceed with the calibration.
- 5.2.8 Run Performance Check Sample**
  - 5.2.8.1** Crush a shard of B2 glass, immerse it in B oil on a slide, and cover it with a cover slip.
  - 5.2.8.2** Click on B2 row in the middle column, and then click on Measure B2.
  - 5.2.8.3** Choose a fragment with an edge that has sufficient contrast and click on it.

- 5.2.8.3.1 Click on center of edge box and drag it to reposition it.
      - 5.2.8.3.2 Click just inside of edge box to resize it.
    - 5.2.8.4 Adjust the temperature to a few degrees above the match temperature noted in the old data set.
      - 5.2.8.4.1 Click Go To.
      - 5.2.8.4.2 Type in desired temperature and click OK.
    - 5.2.8.5 Once temperature has settled, click on Auto.
    - 5.2.8.6 The measurement will automatically be added to the old data set.
      - 5.2.8.6.1 Click row in middle column.
      - 5.2.8.6.2 Click Edit.
      - 5.2.8.6.3 Click on old measurement, click Delete, and then OK.
      - 5.2.8.6.4 In the calibration dialogue, select Save to save the values.
      - 5.2.8.6.5 Repeat procedure for fragments 2-5.
      - 5.2.8.6.6 After each standard is run, click on the Calibrate button. This will update the calibration values as the calibration is being performed.
    - 5.2.8.7 Repeat 5.2.8.1 through 5.2.8.6 for glass standards B3, B4, B6, B7, B8, B9, B10, B11, and B12.
    - 5.2.8.8 Click on Close to finish the performance check.
    - 5.2.8.9 The correlation coefficient for the calibration curve needs to be at 0.9997 or greater and the dRI[e-5] value shall be within  $\pm 10$  for each standard used. If this is not achieved, an attempt shall be made to calibrate again. After a second attempt, call service engineer if instrument is unable to meet calibration criteria. Once maintenance is performed and this criteria is met, the instrument may be returned to service.

### 5.3 Verification of New Instrumentation

- 5.3.1 Perform the performance check procedure following 5.2.8 using John Locke Glass Standards A, B, and C and using appropriate A, B, and C oils on both new instrument as well as instrument previously verified.
- 5.3.2 Run Locke Glass standards B3 and B7 using Oil B on both the new instrument as well

as the instrument previously verified. Verify that the values are consistent within  $\pm 0.0001$ .

**5.3.3** Run Locke Glass standard B4 on two different days and verify values are consistent within  $\pm 0.0001$ .

**5.3.4** Run two different test cases on the new instrument as well as an instrument verified previously. Verify that the same conclusion would be reached using both instruments. If the same conclusions are obtained, then the instrument may be used in case work. If the same conclusion is not obtained, call the service technician for the new instrument. Once maintenance has been performed, repeat the verification procedure.

## **5.4 Analysis**

### **5.4.1 Opening software and creating a case**

**5.4.1.1** Double click on GRIM 3 icon labeled Glass.

**5.4.1.2** Go to the menu bar and select New Case icon.

**5.4.1.3** Type case reference in casebook folder D:\SBI.

**5.4.1.4** Click OK.

**5.4.1.5** Type Forensic Scientist's name (description of case is optional).

**5.4.1.6** Click OK.

**5.4.1.7** Collapse Glass software and double click on Stage Manager icon.

**5.4.1.7.1** Select Calibration in menu bar.

**5.4.1.7.2** Click on Select Oil-Wavelength.

**5.4.1.7.3** Select desired Oil and wavelength (normally B Oil, and 589 nm).

**5.4.1.7.4** Click Select.

**5.4.1.7.5** Click Close.

**5.4.1.7.6** Exit Stage Manager program.

### **5.4.2 Quality Control Check with Standard**

**5.4.2.1** A standard shall be run and recorded in the Use & QC Check Log - GRIM with each case.

**5.4.2.2** Return to Glass software.

- 5.4.2.2.1 Click Run Standard icon (yellow magnifying glass icon).
- 5.4.2.2.2 Click New.
- 5.4.2.2.3 Select appropriate standard (e.g., B4).
- 5.4.2.2.4 Click OK.
- 5.4.2.2.5 NOTE: if not using B oil, select Other, and fill in the appropriate standard in the item comment box on left column of the dialogue box.
- 5.4.2.3 Prepare selected standard from the known glass standards provided.
- 5.4.2.4 Place slide with glass particles, B oil, and cover slip in the Mettler Hot Stage.
- 5.4.2.5 Click Measure GRIM (the Stage Manager screen appears).
- 5.4.2.6 Select up to four edges with sufficient contrast for analysis.
- 5.4.2.7 Click on edge to place measuring box on glass fragment.
  - 5.4.2.7.1 Click centre of box and drag to reposition.
  - 5.4.2.7.2 Click just inside of box to resize.
- 5.4.2.8 Select Search to determine approximate null temperature, and allow starting temperature to settle. NOTE: If starting temperature is too low, click Go To, type in higher temperature, and click OK.
- 5.4.2.9 When Search is complete, click Auto to measure null point and refractive index. NOTE: If a red line appears on graph, click Cancel (number of edge box). NOTE: Edge count is a measure of contrast. An optimal edge count is greater than 50.
- 5.4.2.10 After analysis is complete, select Save Record (a box with the measurement information appears).
  - 5.4.2.10.1 If measurement is acceptable, click OK.
  - 5.4.2.10.2 If measurement is unacceptable, click on that fragment's row, and then check the Reject box.
- 5.4.2.11 Repeat steps 5.4.2.4-5.4.2.9 for fragments 2-5. NOTE: Check phase rings frequently, and adjust as needed.
- 5.4.2.12 Record RI value of standard in calibration log.

**5.4.3** Run Control (known) glass sample.

**5.4.3.1** Click Run Control icon (green magnifying glass icon).

**5.4.3.2** Click New in left column.

**5.4.3.2.1** Type in reference for glass fragment, i.e. item number (the rest of the information bars are optional).

**5.4.3.2.2** Click OK.

**5.4.3.2.3** Repeat steps **5.4.2.3** through **5.4.2.10** for the control sample.

**5.4.4** Run Recovered (unknown) glass sample.

**5.4.4.1** Click Run Recovered icon (red magnifying glass icon).

**5.4.4.2** Fill in reference information for glass fragment.

**5.4.4.3** Repeat **5.4.2.3** through **5.4.2.10** for the recovered sample.

**5.4.5** Print by selecting File from the menu bar and clicking on Print. Complete the Performance Check Log-GRIM.

**5.5 Shut-down**

**5.5.1** Allow the system to cool.

**5.5.2** Turn all instruments OFF in the reverse order of the ON procedure.

**5.6 Standards and Controls**

- **B2** Locke Scientific Glass Standard
- **B3** Locke Scientific Glass Standard
- **B4** Locke Scientific Glass Standard
- **B6** Locke Scientific Glass Standard
- **B7** Locke Scientific Glass Standard
- **B8** Locke Scientific Glass Standard
- **B9** Locke Scientific Glass Standard
- **B10** Locke Scientific Glass Standard
- **B11** Locke Scientific Glass Standard
- **B12** Locke Scientific Glass Standard

**5.6.1** All standards shall be packaged separately and stored at room temperature next to the instrument.

**5.7 Maintenance** – Record in log any maintenance performed on instrument

## 5.8 Sampling and Sample Selection

**5.8.1** No sampling is performed. When sample selection occurs, the Forensic Scientist shall use knowledge gained from experience and scientific training when making the selection.

**5.8.2** The average of no fewer than 5 measurements shall be made for the B4 standard, known glass sample(s), and recovered glass sample(s). If fewer than 5 readings are possible (e.g., very small samples), it shall be noted in the FA worksheet.

## 5.9 Calculations – N/A

**5.10 Uncertainty of Measurement** - The precision of this method is typically better than the measurable variation of a glass object. The manufacturer reports that repeat measurements can produce results with a standard deviation of 0.00003 over a five-day period, with results typically reported to the nearest 0.00001. The expected variation within a single float source is in the range of  $\pm 0.00004$  for annealed glass and  $\pm 0.00016$  for tempered glass.

**6.0 Limitations** – This method will not differentiate between glasses whose refractive indices differ by fewer than  $\pm 0.00003$ .

## 7.0 Safety

**7.1** Glass slides are sharp.

**7.2** High temperatures may be produced. Care shall be exercised when using this process.

## 8.0 References

Foster and Freeman, Limited. Instrument Manual for GRIM 3. Foster and Freeman, Limited, Evesham, Worcestershire, United Kingdom, 2008.

Reference Glasses and Silicone Oils for Refractive Index Determination. Locke Scientific. Hampshire, United Kingdom, 10.

Glass Refractive Index Determination. Scientific Working Group for Materials Analysis (SWGMA), July 2004.

Standard Test Method for the Automated Determination of Refractive Index of Glass Samples Using the Oil Immersion Method and a Phase Contrast Microscope. American Society for Testing and Materials (ASTM), 1998.

## 9.0 Records

- Use & QC Check Log - GRIM
- Maintenance Log

- Performance Check Log-GRIM

**10.0 Attachments – N/A**

| Revision History |                |                                                                                                  |
|------------------|----------------|--------------------------------------------------------------------------------------------------|
| Effective Date   | Version Number | Reason                                                                                           |
| 09/17/2012       | 1              | Original ISO Document                                                                            |
| 09/30/2013       | 2              | Updated list of controlled documents in 9.0 and controlled form titles throughout the procedure. |
| 10/18/2013       | 3              | Added issuing authority to header                                                                |
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