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 to analyze the refractive index (RI) of 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 ±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
- KBr die set
- Set of Locke Scientific Glass Standards
    - **A1** Locke Scientific Glass Standard
    - **A2** Locke Scientific Glass Standard
    - **A3** Locke Scientific Glass Standard
    - **A4** Locke Scientific Glass Standard
    - **A5** Locke Scientific Glass Standard
    - **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
    - **C1** Locke Scientific Glass Standard
    - **C1** Locke Scientific Glass Standard

4.3 Reagents

- Immersion liquids, such as Locke Silicone Oils Type A, B, and C
- Acetone

5.0 Procedure

5.1 Sample Preparation

5.1.1 If necessary, clean the glass fragment with acetone.

5.1.2 Crush the glass fragment and place a small sample on a slide. Immerse the sample in the appropriate Silicone Oil and cover with a cover slip.

5.1.3 Place the slide on the hot stage and focus the image. Ensure proper phase ring alignment.

5.1.4 Allow the sample and temperature to settle prior to beginning analysis.

5.2 Instrument Notes

5.2.1 Allow the GRIM 3 system to warm up and stabilize for one hour before taking any measurements.

5.2.2 Use the Search option to determine approximate null temperature. When Search is complete, use the Auto option to measure null point and refractive index.

5.2.3 A red line on a graph indicates the measurement was not completed (e.g., fragment moved during analysis).

5.2.4 Edge count is a measure of contrast. An optimal edge count is greater than 50.

5.2.5 Check phase rings frequently and adjust as needed.

5.3 Calibration/Performance Check

5.3.1 A calibration curve with the B Oil shall be performed annually using the B2, B3, B4, B6, B7, B8, B9, B10, B11 and B12 Locke Scientific Glass Standards.

5.3.2 Calibration curves with the A and C oils shall be done upon use when the A and C oils are required for the examination.

5.3.3 The correlation coefficient for the calibration curve needs to be at 0.9997 or greater and the dRI[e-5] value shall be within ±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 criterion is met, the instrument may be returned to service.

5.3.4 Print the calibration curve and place it in the GRIM 3 logbook next to the instrument.

5.4 Verification of New Instrumentation

5.4.1 Perform the calibration/performance check described in **5.3** using Locke Scientific Glass Standards A, B and C with the appropriate A, B and C oils on both the new instrument as well as the instrument previously verified.

5.4.2 Run Locke Scientific Glass Standards B3 and B7 using Silicone Oil B on both the new instrument as well as the instrument previously verified. Verify that the values are consistent within ±0.0001.

5.4.3 Run Locke Scientific Glass Standard B4 on two different days and verify values are consistent within ±0.0001.

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

5.5 Casework Analysis

5.5.1 A glass standard (e.g., B4) shall be run with each case and the RI recorded in the GRIM 3 logbook. This shall also serve as the instrument performance check. The RI shall be consistent within ±0.0001 of the previously recorded RI measurement. Repeat the performance check if the error criterion is not met. If a problem persists, contact the service representative. Once maintenance has been performed, the performance check must pass in order to continue use for casework.

5.5.2 Analyze the Recovered (questioned) glass sample(s) and the Control (known) glass sample(s) following the **Glass Refractive Index Determination** section of the Trace Evidence Section Technical Procedure for the Examination of Glass Evidence.

5.6 Standards and Controls

5.6.1 Set of Locke Scientific Glass Standards

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

5.7 Maintenance – Record any maintenance performed in the instrument logbook.

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.

5.9 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 ±0.00004 for annealed glass and ±0.00016 for tempered glass.

6.0 Limitations – This method will not differentiate between glasses whose refractive indices differ by fewer than ± 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

ASTM Standard E1967-19, 2019, "Standard Test Method for the Automated Determination of Refractive Index of Glass Samples Using the Oil Immersion Method and a Phase Contrast Microscope." ASTM International, West Conshohocken, PA, 2019, DOI: 10.1520/E1967-19, http://www.astm.org.

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.

SWGMAT July 2004. "Glass Refractive Index Determination."

9.0 Records

- Use & QC Check Log
- Maintenance Log
- Performance Check Log

10.0 Attachments – N/A

Revision History		
Effective Date	Version Number	Reason
03/25/2025	7	Combined 5.5.2 and 5.5.3, added reference to Technical Procedure for Examination of Glass Evidence. Updated References.