

TECHNICAL NOTE

Arie Zeichner,¹ Ph.D. and George Feingold,¹ M.Sc.

Improved Sample Holder for Refractive Index Measurements of Small, Single, Glass Fragments

REFERENCE: Zeichner, A. and Feingold, G., "Improved Sample Holder for Refractive Index Measurements of Small, Single, Glass Fragments," *Journal of Forensic Sciences*, JFSCA, Vol. 34, No. 4, July 1989, pp. 1003-1006.

ABSTRACT: A simple method is described to convert the flat microscope slide into an improved sample holder for refractive index determination of single, small, glass fragments and their subsequent easy recovery for additional examinations.

KEYWORDS: forensic science, refractive index, glass, sample holders

Refractive index (RI) measurements have generally been used in the forensic science examination of glass fragments [1,2]. Recently, an automatic instrument Glass Refractive Index Measurement (GRIM), based on a phase contrast microscope and the Mettler hot stage, was developed by Foster and Freeman Ltd., greatly reducing operator fatigue [3].

Usually the sample for RI measurements is prepared as follows: glass fragments (whole or crushed) are mounted in an appropriate silicone oil on a microscope slide and covered with a coverslip [1,3]. Some investigators use depression microscope slides instead of flat ones [2].² However, the use of plano-plano specimen preparations is of paramount importance in phase microscopy. When using intermediate and long working distances, the specimen mount should have parallel walls with flat surfaces and be of good optical quality. Irregular, lens-shaped (hollow ground) slides or wedge-shaped preparations upset the alignment of the phase system. Even with only a small wedge, in which case the coverslip is not quite parallel with the slide, it may be necessary to repeat centering procedures each time the specimen is moved to maintain a good contrast [1,4].

To improve the evidential value obtained by the RI measurements, additional examinations are usually performed, such as various techniques of element analyses [1,5,6], density measurements [1], and identification of toughened glass by change of the RI caused by annealing [7]. In cases in which a very small sample of glass (a single small fragment or very few small fragments) is available, it may be very difficult to divide the sample for all the required

Received for publication 16 July 1988; revised manuscript received 3 Oct. 1988; accepted for publication 12 Oct. 1988.

¹Head and head of the mechanics and metallurgy group, respectively, Toolmarks and Materials Laboratory, Division of Criminal Identification, Israel National Police, Jerusalem, Israel.

²J. Locke, personal communication, Central Research Establishment, Home Office Forensic Science Service, Aldermaston, Reading Berkshire, U.K., 1985.

examinations. In such cases it may be important to recover all of the sample used for RI measurements. The recovery of the crushed sample may be very difficult, if not impossible, if a very small sample was used. Actually, in such cases it is often difficult even to locate glass fragments during RI measurements in the hot stage while using the smallest (to our knowledge) commercially available coverslips (18 by 18 mm).

In this article we wish to report a simple method to convert the flat microscope slide into an improved sample holder for RI determination of single, small, glass fragments and their subsequent easy recovery for additional examinations.

Experimental Procedure

Instrumentation

Measurements of RI were carried out using the GRIM system (Foster and Freeman Ltd.) [3] combined with the Mettler FP52 hot stage and AO Reichert H110TG-PP4 phase star microscope. The microscope was originally equipped with the 6-V (20-W) tungsten halogen illuminator. This illuminator is not sufficient for satisfactory results with the GRIM and therefore a 12-V (100-W) illuminator was adapted to the microscope. A 589-nm interference filter and a $\times 10$ objective were used for all measurements.

Sample Holder

The basic idea to improve the usual microscope slides as sample holders was to modify them in such a way that the glass fragments would be confined to a small area (for example, a 2 to 3-mm circle, similar to the size of the aperture in the hot stage) for their quick localization during RI determination. For this purpose, small steel tablets (about 8- by 8-mm size) with one hole, specially prepared, were glued (Araldite Rapid, Ciba Geigy) to microscope slides (Fig. 1). The thickness of the tablets was 0.2 to 0.3 mm and their large walls smooth and parallel. Such thickness is sufficient for the small glass fragments not to protrude from the opening of the tablet. Smoothness of the walls is important so that small fragments of glass cannot be hidden in the interfaces between the microscope slide and the tablet on the one side and the tablet and the coverslip on the other.

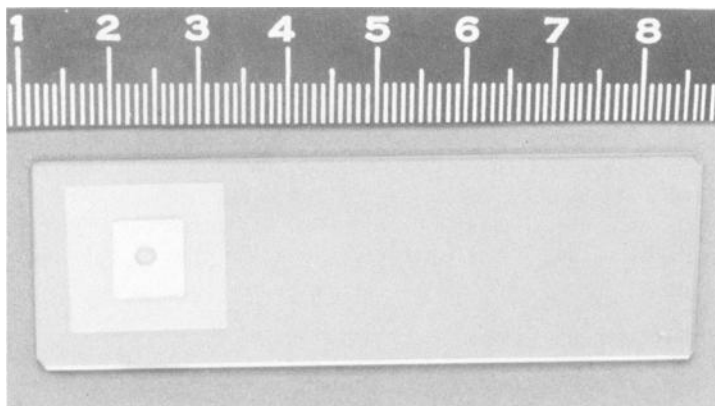


FIG. 1—The improved sample holder with a coverslip.

TABLE 1—Comparison of the match temperatures obtained using the improved sample holder and the usual method of a coverslip on a flat microscope slide.

Oil ^a	Glass ^a	The Usual Method		The New Sample Holder	
		Match Temp., °C	S.D., °C	Match Temp., °C	S.D., °C
B	B 1	41.09	0.16	41.07	0.14
B	B 4	69.81	0.15	69.71	0.14
B	B 9	91.83	0.24	91.28	0.14
B	B 12	115.07	0.22	114.54	0.26

^aStandard glasses and silicone oils were purchased from Locke Scientific, 16 Oakfield Rd., Pamper Heath, Basingstoke, Hampshire, RG26 6DN, England.

Sample Preparation and Recovery of the Glass Fragments Following RI Measurement

One or several (if they may be identified by their different shape or size) small, glass fragments are placed in the hole of the holder and covered with a drop of an appropriate silicone oil. The amount of oil should be large enough to overflow the opening of the hole. This is to prevent formation of air bubbles when covering the hole with a coverslip. Since the hole is small, the glass fragments are found immediately in the view field of the phase microscope.

Following the RI determination, the glass fragments may be recovered as follows: the coverslip is removed (using tweezers) by sliding it from the holder. While observing the hole through the stereomicroscope, petroleum ether, 60 to 80°C, is added dropwise to the hole. Petroleum ether dissolves the silicone oil and the concentration of the latter gradually decreases so that the glass fragments become visible (as a result of a much larger RI difference between petroleum ether and glass). When the solvent dries out, the glass fragments may be removed for subsequent, additional examinations.

It should be stressed that, for precise results, the calibration of the silicone oil and the RI measurements should be performed in the same type of holder.

Differences were found between the match temperatures obtained using the improved sample holder compared to the usual method of a coverslip on a microscope slide. The difference increased as the match temperature rose (Table 1).

Acknowledgment

The authors would like to express their thanks to Rebecca Jayson for her assistance in the preparation of this manuscript.

References

- [1] Saferstein, R., Ed., *Forensic Science Handbook*. Prentice-Hall Inc., Englewood Cliffs, NJ, 1982, pp. 140-183.
- [2] Ojeda, S. M. and De Forest, P. R., "Precise Refractive Index Determination by the Immersion Method, Using Phase Contrast Microscopy and the Mettler Hot Stage," *Journal of Forensic Science Society*, Vol. 12, 1972, pp. 315-329.
- [3] Locke, J. and Underhill, M., "Automatic Refractive Index Measurement of Glass Particles," *Forensic Science International*, Vol. 27, 1985, pp. 247-260.
- [4] "Reference Manual Series One-Ten and One-Twenty Phase STAR Microscopes," AO Reichert Scientific Instruments, Box 123, Buffalo, NY 14240.
- [5] Keeley, R. H. and Christofides, S., "Classification of Small Glass Fragments by X-Ray Microanalysis with the SEM and a Small Sample XRF Spectrometer," *Scanning Electron Microscopy*, Vol. 1, 1979, pp. 459-464.

- [6] Catterick, T. and Hickman, D. A., "Sequential Multi-Element Analysis of Small Fragments of Glass by Atomic-Emission Spectrometry Using an Inductively Coupled Radio Frequency Argon Plasma Source," *Analyst*. Vol. 104, June 1979, pp. 516-524.
- [7] Locke, J., Sanger, D. G., and Gayatri Roopnarine, "The Identification of Toughened Glass by Annealing," *Forensic Science International*. Vol. 20, 1982, pp. 295-301.

Address requests for reprints or additional information to
Arie Zeichner, Ph.D.
Toolmarks and Materials Laboratory
Division of Criminal Identification
Israel National Police
Sheikh Jarrah
Jerusalem, Israel 91906