Validation of an analytical method to measure the refractive index of glass

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Introduction

Glass analyses are carried out by forensic scientists in many criminal cases (burglary, vandalism, hold-ups, road accidents, homicides or assaults with a glass object as a weapon).
A fragment of glass can be identified by its physical properties (thickness, colour ...), its elementary chemical composition but mainly by its refractive index. To perform these analysis the Forensic Science Laboratory of Paris acquired a phase contrast microscope, the "GRIM 3", which working principle is based on the Becke line method: the refractive index is measured with an oil immersion technique.
Before being used in criminal cases and in order to ensure the trustworthiness of the results given by the forensic scientists, this method must be first validated.

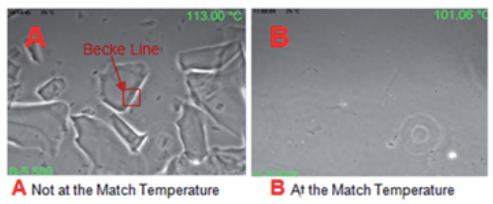
Experimental procedures

The principle of the Becke line method is to immerse the glass particles in silicone oil while varying the temperature of the system (oil-glass) with a hot stage. When the refractive indexes of glass and oil are different, a bright halo along the fragments is observed, this is the Becke line.
At a specific temperature, the glass particles seem to "disappear" in the oil (figure 1). The oil and the glass have exactly the same refractive index at this point, referred to as the "Match Temperature" or point of phase extinction.
Prior to the validation process, an internal calibration of the GRIM 3 was conducted using reference glasses whose refractive indexes are precisely known. Through this internal calibration, the software is able to calculate the refractive index in function of the measured Match Temperature according to the calibration equation "refractive index = f(temperature)".

Results and discussion

The validation strategy set up is based on the French standard AFNOR NF T90-210-2009, which deals with the "Protocol for initial evaluation of performances of a method in a laboratory."
Validation range covers a field commonly used by the Forensic Science Laboratory: 1.46 < RI < 1.56. This validation was only performed with the interference filter of wavelength λ = 589.3 nm (sodium D line).
Different performances of this method were evaluated.
The accuracy was evaluated and validated: observed results correspond to the real value of the reference sample.
The repeatability was tested: 24 measurements were made in a short time interval, The measured standard deviation is lower than 0.00001. This method is repeatable.
The reproducibility was checked. It was proven that three different operators gave results matching each others'.
External parameters, which may impact on the measurements, were also studied. It was shown that the localization of the glass particle in the camera field has no impact on results. However, the study of the method robustness demonstrated that the aging of the oil had an impact on the measurement of the refractive index. Therefore providing the sample is prepared on the day of the analysis, the method is valid.

Figure 1: « Disappearance » of the glass particle in the oil at the Match Temperature



Conclusion

The glass analysis method with the Grim 3 instrument is validated: it can be used to measure the refractive index in a comparative analysis of glass samples.
This analytical method provides highly accurate measurements of refractive index (RI) and has a high repeatability.
However, the statistical retreatment of the refractive index results must be performed cautiously by the forensic science experts. They must confirm their results by combining, as far as possible, refractive index measurements to another elemental technique such as LA ICP-MS.

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