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Refractive-index measurements in the near-IR using an Abbe refractometer

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Abstract. A novel method to measure the refractive index *n* in the near-infrared by simple extensions to a standard Abbe refractometer is described. A technique is derived to correct for the dispersion of the glass prism and experimental results of refractive-index measurements at $\lambda = 830$ nm are compared with published data. These results prove the suitability of the described method, the accuracy being comparable to that of an Abbe refractometer used in the visible range; that is, the refractive index *n* can be measured to an accuracy of $\pm 10^{-4}$. Finally, new refractive-index data at 830 nm are given for methanol, water, acetone, ethanol, cyclohexane, glycol, di-2-ethyl hexyl-sabacate (DEHS), carbon tetrachloride, glycerol, toluene, ethyl salicylate, methyl salicylate and cinnamaldehyde at 20 and 25 °C.

1. Introduction

Most techniques of optical particle characterization require knowledge of the optical properties of the investigated substance. The Abbe refractometer is a standard tool for measuring the refractive index n of a specimen in the visible range [1–3]. This instrument is precise and easy to use, but it cannot be applied to radiation invisible to the human eye, such as infrared (IR) or ultraviolet (UV) light. With the development of laser-light sources and applications in the near-IR there is also an increasing need for rapid and easy measurements of optical properties in this wavelength range. Various techniques to obtain the refractive index at such wavelengths have been described in the literature, but they are all more or less complex and difficult to apply. For a small but certainly not complete survey of some of these techniques, see [4–9]. Reference books [10–12], on the other hand, cannot cover the entire range of matter, illumination wavelength and temperature, likewise they lack data in the UV and IR.

To overcome this deficiency a method was developed which makes a standard Abbe refractometer applicable to the UV or IR. The required instruments are available in every well-equipped laboratory so that this technique can be implemented very easily. Another and even more important advantage arises from the fact that this instrument does not require additional calibration by the user.

Two problems had to be solved in order to attain this goal. First, a method had to be developed to correct for the dispersion of the glass prism so that the measured data could be related to the actual refractive index of the specimen. Second, an experimental set-up had to be designed and tested. These two steps are described in the following; refractive indices measured at $\lambda = 830$ nm are verified by comparison with literature data. Finally, new refractive-index data are presented for several liquids at $\lambda = 830$ nm.

2. Working principles

With an Abbe refractometer, the refractive index n of a liquid or solid sample is determined by using the effect of total reflection [1–3]. The substance to be investigated is placed between two prisms made of highly refracting glass with refractive index N (figure 1). Diffuse light passes through the illumination prism and hits the interface with the specimen under different angles, expressed with respect to the normal of this interface. If the incident angle is above the value $\arcsin(n/N)$ and if $N > n$, then total reflection occurs at this interface [1], and no light is transmitted into the specimen. This case is represented by the dotted lines in figure 1. If the incident angle is below $\arcsin(n/N)$, the light is transmitted through the specimen, the measuring prism and a focusing lens, and thus forms a bright range in the eyepiece of the refractometer (broken lines). At the angle δ, at which the transition from transmission to total reflection occurs, the image displays a sharp separation between the bright and dark ranges (full lines). By varying the observation angle, the user adjusts this separation line to the point of intersection of a reticle. Instead of the angle δ of total reflection, he now reads a measured refractive index n' from a scale. In the next step, he has to apply a correction to n' to compensate for the dispersion of the glass

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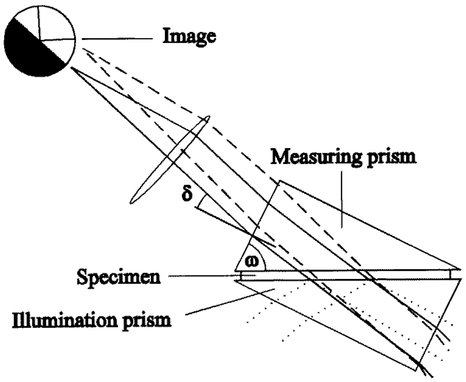

Figure 1. The working principle of an Abbe refractometer.

prism. Thereby, the user obtains the refractive index n of the specimen, measured to within an accuracy of $\pm 10^{-4}$. Correction tables are provided by the manufacturer of the refractometer.

This procedure simplifies the use of the instrument at the expense of a limited application range: the user does not need to deal with the correction, but the Abbe refractometer can only be used at illumination wavelengths for which such correction tables exist. To extend the application range, the relationship between the actually measured parameter, the angle δ of total reflection, and the parameter read from the scale of the refractometer, the refractive index n', must be known. This geometrical relationship is not affected by changing the wavelength of illumination so that it has to be determined only once and can then be applied to any other wavelength. Such a relation will be derived in the following.

3. The correction for the dispersion of the glass prism

The angle δ of total reflection depends on the refractive indices both of the sample and of the prism. These values in turn strongly depend on the wavelength of illumination, which dependence is called dispersion. The change in refractive index with temperature, on the other hand, is much more pronounced for a liquid than it is for a solid, typically by one order of magnitude. In the case of the material used for the glass prisms of the Abbe refractometer, SF13, the gradient $\partial n/\partial t$ is $7.5 \times 10^{-6}\,{}^{\circ}\mathrm{C}^{-1}$ [13]. The effect it has on refractive-index measurements can be neglected as long as the temperature change is less than 13 °C, as is the case in the following measurements.

Equation (1) gives the refractive index n of the sample as a function of the angle δ of total reflection, the refractive index N of the glass prism and the angle ω. This angle is determined by the geometry of the glass prism, figure 1, and is 63° in the present case [13].

$$n_{\lambda,t} = \sin\omega(N_\lambda^2 - \sin^2\delta_{\lambda,t})^{1/2} - \cos\omega\sin\delta_{\lambda,t}. \quad (1)$$

Equation (2) gives a mathematical description of the aforementioned relationship between the measured refractive index n' and the actual refractive index n of the sample as given by the correction tables. Because Δn compensates for the dispersion of the glass prism it also depends on the illumination wavelength. Furthermore, it incorporates the angular expressions of equation (1) so that it is a function of the measured refractive index n', too:

$$n_{\lambda,t} = n'_{\lambda,t} + \Delta n_{\lambda,n'}. \quad (2)$$

The Abbe refractometer B, manufactured by Carl Zeiss, Germany, is calibrated with respect to the sodium D line with $\lambda_D = 589.3$ nm. This means that, at this wavelength, Δn is zero and $n_{\lambda,t}$ is equal to $n'_{\lambda,t}$ in equation (2). Furthermore, it implies that the refractive index n' read from the scale is equivalent to the actual refractive index n of the sample. On replacing n by n' in equation (1), the following relation of n' to δ is obtained:

$$n'_{D,t} = \sin\omega(N_D^2 - \sin^2\delta_{D,t})^{1/2} - \cos\omega\sin\delta_{D,t}. \quad (3)$$

This equation gives the desired relationship between n' and δ. The only remaining unknown is the refractive index of SF13, the glass material used for the prisms. This datum is available from Schott, the glass manufacturer [14]. At $\lambda_D = 589.3$ nm, the refractive index of the prism is $N_D = 1.740\,54$.

From knowledge of this relationship, a correction table can be calculated for any other wavelength by equating (1) with (2) and solving this for Δn:

$$\Delta n_{\lambda,t,n'} = \sin\omega(N_\lambda^2 - \sin^2\delta_{\lambda,t})^{1/2} - \cos\omega\sin\delta_{\lambda,t} - n'_{\lambda,t}. \quad (4)$$

The glass catalogue [14] gives the refractive index N of the glass prism at the desired wavelength, either directly from a table or by interpolation using the Sellmeier formula.

This procedure was verified by computing Δn at $\lambda = 488$ nm, with the refractive index of the glass prism being $N_{488\ nm} = 1.759\,39$. Figure 2 shows the result in comparison with data from a correction table provided by Zeiss [15]. The trend of Δn matches the given values very well but the absolute values are not represented exactly; the calculated curve gives values slightly above data from the correction table. The difference, however, is relatively small, it is within the inaccuracy of the refractometer and never exceeds 10^{-4}. This result confirms the described method for the computation of correction tables.

In the next step, the correction coefficient Δn was calculated for infrared light at $\lambda = 830$ nm, with $N_{830\ nm} = 1.721\,56$. The result, depicted in figure 3, served as the basis for the following measurements.

4. Refractive-index measurements

4.1. The experimental set-up

The experimental set-up is shown in figure 4 and can be described as follows. The standard Abbe refractometer B was connected to a water bath with a temperature control unit to ensure a constant temperature setting. The

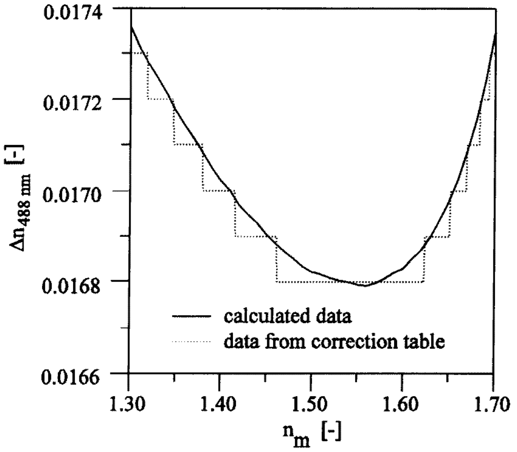

Figure 2. The correction value Δn as a function of n' at λ = 488 nm, showing a comparison between calculated data and data from a correction table.

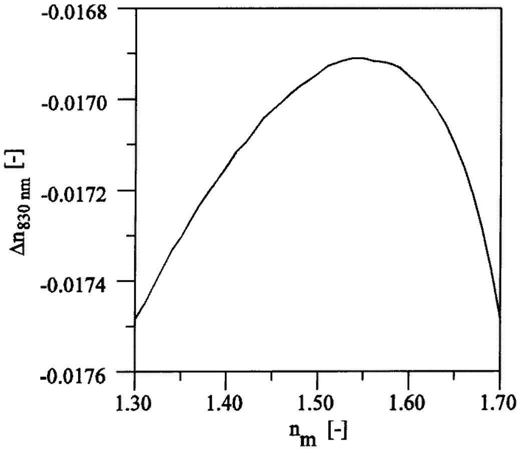

Figure 3. The correction value Δn as a function of n' at λ = 830 nm, calculated data.

temperature was measured at the observation prism of the refractometer. A Sony AVC-D7CE black-and-white CCD camera, normally used in the visible range, was mounted onto the eyepiece of the refractometer. With the infrared filter removed, the CCD chip also received infrared radiation at $\lambda = 830$ nm, coming from a solid-state laser. The CCD image was transmitted to a monitor and the adjustment of the separation line with respect to the cross wires was accomplished by inspection of the monitor. A simple flashlight sufficed to illuminate the scale and thus to read the value of n'. A sheet of transparent paper was inserted between the laser and the refractometer to obtain diffuse illumination. Mounting this sheet onto a rotating disc made the illumination time-variant. This avoided saturation and blooming of CCD pixels and enhanced image contrast significantly, because no speckle patterns could build up.

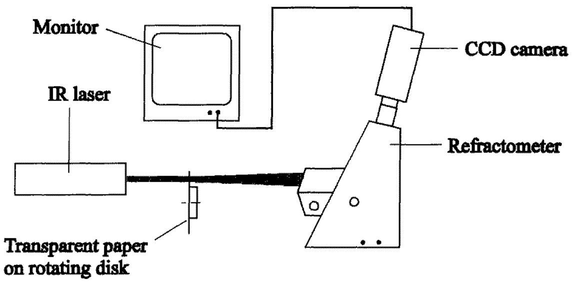

Figure 4. The experimental set-up for refractive-index measurements in the near infrared.

4.2. The experimental verification

The set-up described in section 4.1 was used for refractive-index measurements at $\lambda = 830$ nm; the results were compared with published data [5, 6, 9, 16]. The first column of table 1 lists the liquids used with their refractive indices n in increasing order. The value in the second column is the refractive index n which was obtained by the Abbe refractometer. The measured n' was corrected by application of equation (2) with $\Delta n_{830\ nm}$ from figure 3. The third column shows the data measured by Berg [9], who used the technique of beam displacement [17] for refractive-index measurements at 830 nm. The fourth column contains reference data [5, 6, 16]. Cooper [5] and Moreels *et al* [6] used the principle of the Pulfrich refractometer [1, 3, 13]; Cooper claimed an accuracy of within $\pm 3\times 10^{-4}$ for the measured refractive indices; d'Ans *et al* [16] gave no further specification about the instrument used.

The measurements published by these three groups were not performed at 830 nm so that the results had to be evaluated by a nonlinear regression analysis, using the Cauchy dispersion formula [1, 4–6]

$$n = A + B\lambda^{-2} + C\lambda^{-4}. \qquad (5)$$

Table 1 also gives the values for the constants A, B and C, together with the wavelength range to which the nonlinear regression analysis was applied.

The agreement between refractometer measurements and literature data is very good and lies well within the accuracy of the used instruments. The difference did not exceed 1×10^{-4} when the reference data had to be interpolated and was less than 3×10^{-4} when data extrapolation had to be used. The reason is that data extrapolation causes a higher uncertainty than does interpolation so that the former reference is less certain than the latter. The only exception is cinnamaldehyde with a difference of 11×10^{-4}. This substance is chemically unstable; it polymerizes. For the measurements with the refractometer a freshly distilled sample was used, but the reference sample might have been an old one.

4.3. Additional measurements

Following the confirmation of the method, it was used to measure the refractive indices of several liquids at 20 and 25 °C. These results are summarized in table 2, together with the results from the previous section.

Table 1. Refractive indices of various liquids at λ = 830 nm; comparison with reference data from [5, 6, 9, 16].

Liquid (temperature)	*n*, Abbe refractometer	*n*, [9]	*n*, [5, 6, 16]	Cauchy dispersion constants			Wavelength range (nm)
				A	*B* (10^6 nm^2)	*C* (10^{12} nm^4)	
Water (20 °C)	1.3277	1.3272	1.3276	1.320 43	0.054 30	−0.000 36	486.1–943.0
Acetone (20 °C)	1.3540	1.3530	1.3543	1.349 79	0.003 06	0.000 06	476.5–632.8
Ethanol (20 °C)	1.3568	1.3564	1.3571	1.352 65	0.003 06	0.000 02	476.5–632.8
Cyclohexane (20 °C)	1.4206	1.4205	1.4209	1.415 45	0.003 69	0.000 04	476.5–632.8
Glycerol (25 °C)	1.4659		1.4659	1.457 97	0.005 98	−0.000 36	589.3–1050.0
Ethyl salicylate (25 °C)	1.5069		1.5068	1.493 76	0.008 73	0.000 18	589.3–1050.0
Methyl salicylate (25 °C)	1.5194		1.5195	1.505 13	0.009 71	0.000 15	589.3–1050.0
Cinnamaldehyde (24 °C)	1.5929		1.5940	1.570 08	0.015 23	0.000 84	589.3–1050.0

Table 2. Refractive indices of various liquids at λ = 830 nm. (DEHS or di-2-ethyl hexyl-sabacate, $C_{26}H_{50}O_4$, is an oil used as nebulizer fluid.)

Liquid	*n*, 20 °C	*n*, 25 °C
Methanol	1.3247	1.3228
Water	1.3277	1.3274
Acetone	1.3540	1.3514
Ethanol	1.3568	1.3548
Cyclohexane	1.4206	1.4180
Glycol	1.4229	1.4217
DEHS	1.4447	1.4430
Carbon tetrachloride	1.4534	1.4505
Glycerol	1.4670	1.4659
Toluene	1.4855	1.4828
Ethyl salicylate		1.5069
Methyl salicylate		1.5194
Cinnamaldehyde		1.5929 (at 24 °C)

5. Conclusion

This paper gives a detailed description of a novel and simple method for refractive-index measurements under UV or IR illumination by modifications to a standard Abbe refractometer. The extension of the application range requires the calculation of correction coefficients to compensate for the dispersion of the glass prism, which has been accomplished successfully and is also described herein. Measurements performed under IR light with $\lambda = 830$ nm, together with comparison with published data, confirm the validity of the described technique. The paper closes with refractive-index data of several liquids frequently used in fluids experiments, but for which no such data had previously been published at $\lambda = 830$ nm.

The described technique has several advantages over other methods used for refractive-index measurements under UV or IR illumination. It is easy to use, does not require additional calibration and has a higher accuracy. The wavelength range of application is limited only by the transmittances of lenses, prisms and their coatings, and by the sensitivity of CCD devices. The transmittance of most optical glass types is above 80% from UV light at 400 nm to mid-IR light at 2600 nm [10]. Because the type of coating material used for the lenses and prisms is unknown, no estimate can be given for any limitation possibly imposed upon the application range. Contrary to transmittance, CCD sensitivity already is a problem in the near-IR because standard, silicon-based arrays are confined to a wavelength range between approximately 300 and 1000 nm. This restriction can be overcome by using another CCD sensor better suited to the wavelength of incident light.

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