
**AC 2011-1361: INSTRUMENTATION FOR HIGHLY ACCURATE INDEX
MEASUREMENT OF LIQUID**

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Abstract

The index of refraction is one important optical property of materials. It also provides information to analyze liquids or mixed solutions, such as chemicals, foodstuffs, drinks, and pharmaceuticals. In this work, we would like to introduce a creative but simple method to measure the index of liquid. The measurement accuracy was high, with an error under 5×10^{-5} for DI water. This method presented similar merits as minimum deviation method inherently. However, the simplicities in measurement process and experimental parameters showed great potential for accurate index measurement applications.

Most of the design and developing works of this measurement system was performed by undergraduate students from Physics Department and Department of Optics and Photonics. The hardware integration and the data-acquire automation provided practical learning opportunities for the subjects about optics, electronics, programming, and data analysis. And, the measurement results can also provide further insights about the temperature or concentration related index variation of liquid for the students.

Introduction

The index of refraction is one important optical property of materials. For liquid materials, it also provides information to analyze liquids or mixed solutions, such as chemicals, foodstuffs, drinks, and pharmaceuticals. In general, the instruments to characterize the index of liquids were developed according to the fundamental optical properties such as total internal reflection (Abbe refractometer)¹, diffraction (grating)², interference³, or deflection^{4,5}, etc.

Minimum deviation method (MDM) is one well-known and well-developed index measurement method since 1930.⁶⁻⁹ In this method, the index was deduced by the “minimum deviation angle” of the probe beam when it passed through the material under test. Such a material can be solid or liquid, but it has to be shaped as a prism to deflect the probe beam. Compared with other methods, MDM is inherently a good choice for high accurate absolute index measurement. This property benefits the index characterization of the liquid used in immersion lithography technology, or provides valuable information for exploring phenomena of the dynamically interconverting microdomains of two structural types of water.

The method proposed in this work is also a prism based method. But principally, it applies one well-known physical law, Snell's Law. To be compared with MDM, the measurement process is more accessible. However, they have similar measurement accuracy. Briefly, the integration of this measurement apparatus can be divided by four parts: "system design", "optical system integration", "data acquirement", and "data analysis". Students from Physics Department and Department of Optics and Photonics had been participated in this project in their "Special Topics Study" course. In this paper, we would like to share our approach to realize the physical concept with the community in the hope that this selected topic will be helpful for teaching the principles and methods about index characterization.

Measurement principle and result

As shown in Fig-1, one laser beam is incident on a prism of apex angle α . The first air-prism interface which laser beam enters is regarded as "entrance face" and the one which laser beam exits is regarded as "exit face" respectively. If the laser beam is incident on entrance face with an angle different from zero degree, it will be deflected. In general, the index of material is larger than the index of air. And then, the angle of refraction inside the prism will be smaller than the incident angle on entrance face. The deflected beam will pass through the prism material and arrive at the exit face. Only if the incident angle θ_n on the entrance face reaches a certain value, then the corresponding incident angle on exit face become zero. Since partial reflection effect existed on the interface between two difference index materials, part of the laser beam was reflected. And the reflected beam from exit face passes through the prism and emigrates from entrance face right along the same path but in an opposite direction.

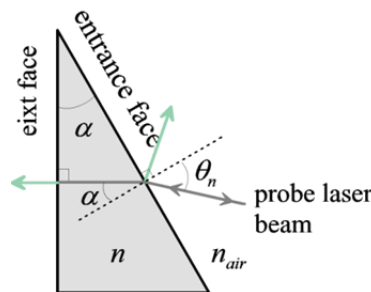


Fig-1 The gray line indicates the optical path as the laser beam is incident normally at the exit face. The green lines with arrow indicate the partial reflected beam and the partial transmitted beam respectively.

Then, simply according Snell's Law, the index n of the prism can be deduced as

$$n = \frac{n_{air} \sin \theta_n}{\sin \alpha}, \quad (1)$$

where n_{air} is the index of air. Theoretically, the maximum measurement range of index is around 2 for a prism of apex angle 30° . Such an index measurement method can be applied to liquid.

The liquid under test has to be filled within a prism shaped cell.

The system diagram is shown in Fig-2. In this project, one He-Ne laser of wavelength 633 nm is introduced as probe laser. The laser beam is expanded and collimated by a telescope configuration consisting of a spatial filter and a convex lens. The beam collimation will prevent the measurement accuracy from being sensitive to the distance between the cell and the convex lens. The pinhole can confine the direction of return beam, and increase the measurement precision. The prism shaped material is installed on a rotational stage. When the laser beam is incident normally on the entrance face (or the exit face), the return beam will pass through the pinhole and detected by photo-diode. The variable slit in front of the photo-diode can reduce the field of view and improves the measurement resolution.

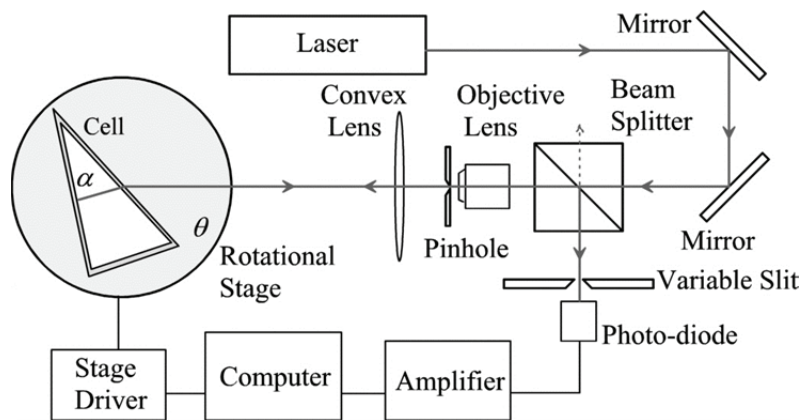


Fig-2 The system diagram.

The origin of the cell's angular position is defined as the position where the laser beam incident on the window's inner surface normally. If the cell is filled with nothing but air, the cell's angular position has to move to α to make the laser beam incident normal on the inner rear surface. However, when the cell is filled with liquid, the cell's angular position has to move to θ_{liquid} to achieve a similar condition on the same surface. If we record these necessary data and put them in equation (1), we can find the index of liquid under test.

In the measurement of liquids, a flat window will be necessary on cell's entrance face. The

flatness and the wedge angle of this window will induce additional error for the index. In general, the error is dominated by wedge angle. If the two surfaces of this window are well parallel to each other, the index of liquid can be deduced as well as equation (1). But if the window has a wedge angle, it would deflect the laser beam and induce a significant measurement error. Considering this unavoidable influence, the deduction of the index has to be corrected as

$$n_{liquid} = \frac{n_g}{\sin \alpha} \sin \left(\sin^{-1} \left(\frac{n_{air} \sin(\theta_{liquid} - \Delta\theta_0)}{n_g} \right) - \delta \right), \quad (2)$$

where $\Delta\theta_0$ is the origin of the cell's angular position, and n_g is the index of window. In this work, the wedge angle δ of the window is 0.012° , and it causes the error on the 4th digit under decimal point.

The measured index of DI water in this project at various temperatures is shown in Fig-3. The temperature varied from 14.9°C to 27.8°C with uncertainty of 0.1°C , and the corresponding measured index varied from 1.33215 to 1.33104. The maximum measurement error was $\pm 3.6 \times 10^{-5}$, and the minimum was $\pm 0.7 \times 10^{-5}$. The measured result at 24.8°C was 1.33130 ($\pm 1.5 \times 10^{-5}$). Moreels had proposed a similar result of 1.33128 for distilled water at 25°C .¹⁰ Our result shows that this system is capable to distinguish the index variation in a half Celsius degree variation.

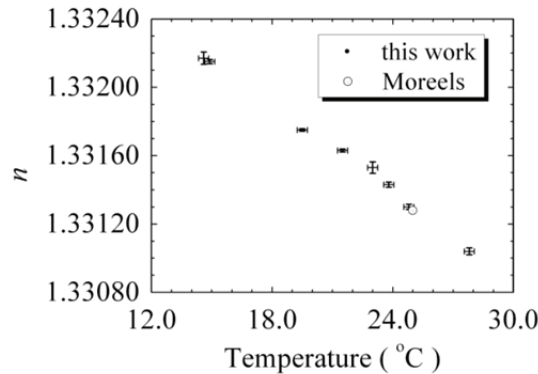


Fig-3 Measured index of DI water for various temperatures at wavelength 633nm.

The index of DI water is depending on the air pressure above the water surface. However, during the measurement, the index uncertainty induced by the uncertainty of air pressure is much smaller than the uncertainty of our system. So we can ignore this effect. The temperature of DI

water is monitored by a thermometer with uncertainty of ± 0.1 °C. And since the absorption coefficient of DI water in this wavelength is small, the laser heating effect can be ignored.

The practices for undergraduate students

The whole system integration process can be divided into 4 parts as shown in Table-1. The corresponding learning subjects include optics, electronics, programming, and data analysis. Each of them can possibly be developed as an education topic, and also can be accessed individually.

Table-1 The four processes of the integration.

Process Descriptions	Sub-items
System design	Optical system design Electronics design
Optical system integration	Characterization of optics Optical signal optimization
Data acquirement	Signal transformation from optical to electronic LabView Programming Goniometer Operation Temperature identification
Data analysis	Curve Fitting Error Estimation

System Design

The design work in this instrument includes two parts: electronics and optics. The well- designed electronics system can improve the data collection more reliably and automatically. And the well-designed optical system can allow more tolerance of installation errors and increase the accuracy of measurement result.

The optical system consists of laser, mirrors, beam splitter, beam expander, and prism shaped cell. Except the cell, all of the others are well-known components which can be found in an educational optical laboratory. Since the main optical path in this method is similar to many common optical systems such as Michelson Interferometer or the pick-up head of CD-ROM

driver, the design work in this system should focus on the possibility of compact-size and the application of handy components. The flexibility to adjust the positions and orientation of the optics should be considered simultaneously.

The design of prism shaped cell is important in this project. The apex angle of the cell limits the maximum measurable index value of this system. In this work, the apex angle is 30 degrees. The “entrance face” (or the “exit face”) is the interface between the cell’s window and DI water. The window materials are both BK7. A proper coating selection increases the ratio of the reflected beam signal from the “entrance face” (or from the “exit face”) to the one from other surfaces in the optical path. The coating on “entrance face” induces 50% reflection and 50% transmission. The coating on “exit face” induces over 95% reflection. The tilt angle of the “exit face” of the cell has better to be adjustable to correct unnecessary vertical beam deflection by the prism shaped liquid. The cell material is Polyacetal (POM).

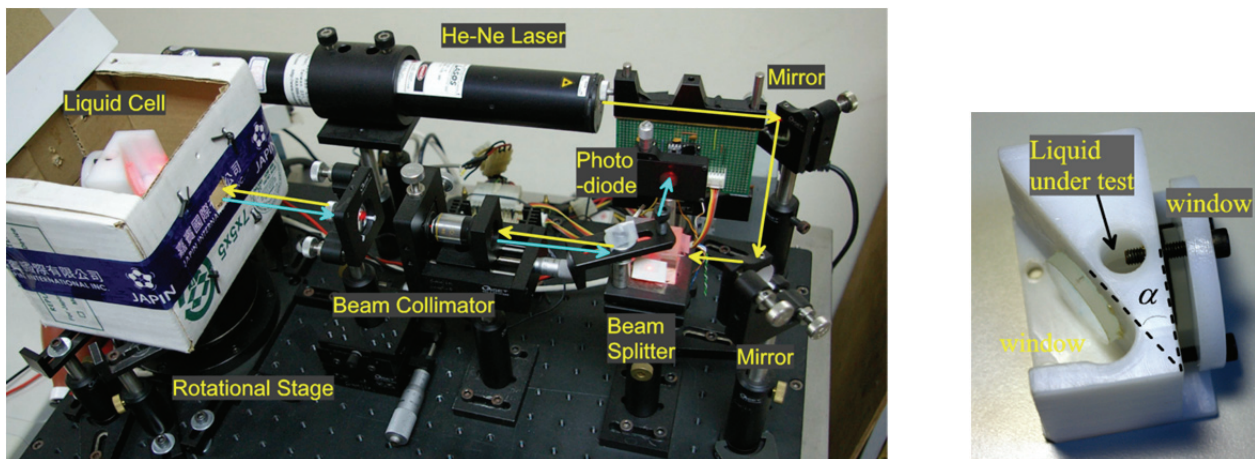


Fig-4 The whole system in this work and the prism shaped cell.

The electronics system consists of photo-diode, amplified electronics, A/D interface, and goniometer controller. All of these electronics are connected (or controlled) by a computer. This computer will acquire signal intensity and rotate the goniometer synchronously. The recorded data will then be analyzed to deduce the precise value of cell’s angular position. Students have to know the specifications of each component and judge whether the system error is permitted or not, and they need to design one flow chart to acquire all the necessary data.

Optical system integration

Since the integration of optics is a new experience for most of the undergraduate students, this

training can provide the experience to align the light with simple optics and their mounts. It also provides the opportunity to find how to setup the optics at a proper position and organize them to be a whole system. Students will have a series of practices, or challenges, to let the laser beam pass through a 10 μm pinhole, or to keep the laser beam pass through all optics in a straight line path. In this integration, students can also build their skill to interpret the information from the observations about laser spot patterns, direction, etc.

Data acquired automation

In this work, we used one monolithic photodiode OPT101 from Texas Instruments to convert the reflected signal intensity into electrical signal. Accompanied with the voltage amplified circuit, this is another small but practical education topic for undergraduate students. The analog signal output from photo-diode is received by A/D interface and recorded by personal computer. One LabView program is developed to synchronize the data acquire operation and the goniometer movement.

Data Analysis

The cell's angular position is deduced by the angular-intensity curve, as shown in Fig-5. Its peak position indicates the correct angular position of the cell. This peak position is deduced by Gaussian curve fitting. Students can practice the curve fitting operation with mathematic tools. The final measurement result has to be summarized and interpreted by a statistic method. The error discussion is an interesting and practical course.

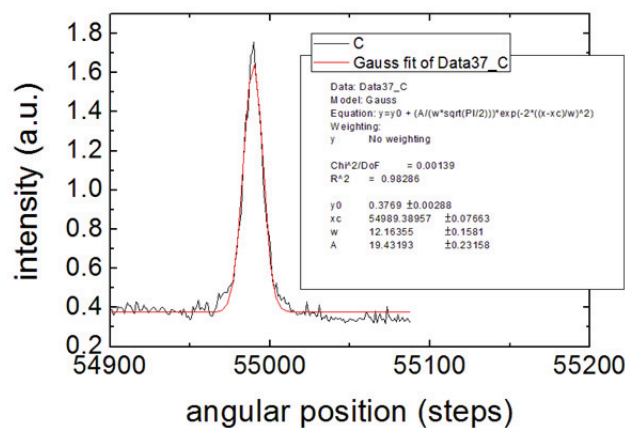


Fig-5 The reflected beam intensity curve (black line). The peak position,

deduced by curve fitting, is the cell's angular position.

Conclusions

In this paper, we would like to share this interesting idea to measure the index of liquid. The measurement result for DI water with this method is accurate, and is capable to distinguish the index variation in half Celsius degree variation. The simplicities in measurement process and experimental parameters showed great potential for accurate index measurement applications. The integration of this instrument can be divided by several learning topics, such as optics, electronics, and data analysis. We hope it is helpful for teaching the principles and the instrumentations about index characterization for solid or liquid materials.

Acknowledgement

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