

# Improved retroreflection method for measuring the refractive index of liquids

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We propose a new method for measuring the refractive index of liquids with high precision; the method is based on use of the optical fiber end face. As an example, we investigated the refractive index of sugar solution under varying conditions tens of times. The results show that this method has the advantage of higher stability and repeatability. The concentration and the temperature-dependent refractive index of the sugar solution is also experimentally studied. © 2010 Optical Society of America

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## 1. Introduction

With the development of science and technology, high-precision measurement takes a more and more important role in our life, especially in scientific studies. The refractive index (RI) is a basic optical property of materials and plays a vital role in many areas of material science, especially with regard to thin film technology [1], integrated optics, and fiber optics [2]. Methods to measure the RIs of liquids can be classified into refraction [3–5] and reflection techniques [6–9]. Recent studies [10,11] provide more detailed discussion of concentration mapping by the measurement of the RI of liquids. Several techniques have been reported for measurement of the concentration and temperature dependence of the RI of liquids [12,13]. A refractometer, which is based on the refraction of light, is widely used for measuring the RI. But when the liquid is not transparent or is translucent, a reflection method must be adopted. A method for measuring an untransparent magnetic fluid based on retroreflection on the fiber-optic end face was proposed by our group [9]. From the experimental data in that paper, although the measurement

precision of the RI is reported to be about  $10^{-4}$  after the measured data are averaged, it is found that the data stability and repeatability are not satisfactory. To improve the experimental results, we suggest a new setup to measure the RI of the liquid by employing a circulator instead of a fiber coupler.

## 2. Method and Analyses

When the measurement method as shown in the literature [9] is applied in the experiment, strong perturbation of the liquid's RI is observed. The modified formula of the RI can be rewritten as

$$n_f + \Delta n_f = \frac{(1 + (k + \Delta k)^2)n_{fc}}{1 - (k + \Delta k)^2} \pm \sqrt{\left(\frac{(1 + (k + \Delta k)^2)n_{fc}}{1 - (k + \Delta k)^2}\right)^2 - n_{fc}^2 - k_f^2}, \quad (1)$$

where

$$k = \left(\frac{P_f - P_0}{P_{air} - P_0}\right) \left(\frac{n_{fc} - n_{air}}{n_{fc} + n_{air}}\right); \quad (2)$$

hence

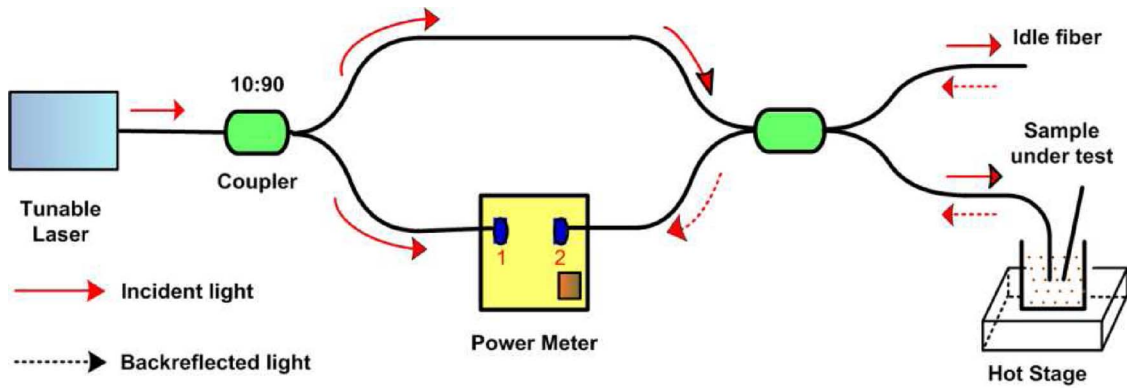


Fig. 1. (Color online) Double-light-paths experimental setup to measure the RI of liquids.

$$\Delta k = \left( \frac{P_f + \Delta P_f - P_0 - \Delta P_0}{P_{\text{air}} + \Delta P_{\text{air}} - P_0 - \Delta P_0} \right) \left( \frac{n_{\text{fc}} - n_{\text{air}}}{n_{\text{fc}} + n_{\text{air}}} \right) - k, \quad (3)$$

where

$$\Delta P_0 = \frac{\Delta P_{\text{water}} - M \Delta P_{\text{air}}}{1 - M}, \quad (4)$$

$$M = \frac{\left( \frac{n_{\text{fc}} - n_{\text{water}}}{n_{\text{fc}} + n_{\text{water}}} \right)^2}{\left( \frac{n_{\text{fc}} - n_{\text{air}}}{n_{\text{fc}} + n_{\text{air}}} \right)^2}, \quad (5)$$

where  $P_0$  and  $\Delta P_0$  are the power and power perturbation of the intrinsic reflection power;  $n_{\text{air}}$ ,  $n_{\text{water}}$ ,  $n_{\text{fc}}$ , and  $n_f$  are the RI of the air, water, optical fiber, and the liquid sample;  $k$ ,  $\Delta k$ ,  $M$  are introduced just to simplify Eq. (1);  $k$  is the extinction coefficient of the liquid;  $P_{\text{air}}$ ,  $P_{\text{water}}$ ,  $P_f$ ,  $\Delta P_{\text{air}}$ ,  $\Delta P_{\text{water}}$ , and  $\Delta P_f$  are the measured reflected powers and their perturbation when the detecting fiber tip is immersed in the air calibration sample, the water calibration sample, and the liquid sample, respectively. On random perturbation of the surrounding circumstances, laser

source power shift, or detector response change,  $\Delta P_{\text{air}}$  and  $\Delta P_{\text{water}}$  are not equal. Similarly,  $\Delta P_f$  and  $\Delta P_0$  cannot be the same. The value of the refractive index of liquid is not accurate because  $\Delta n_f$  cannot be zero.

To weaken the perturbation, a reference light is added to the previous setup [9]. By using the double-light-path measuring technique in Fig. 1, undesirable effects that are caused mainly by possible laser source power shifts and detector response changes are corrected. However, the repeatability of the experimental results is still not satisfactory.

By analyzing the above experimental method, the backreflection powers come from not only the port immersed in the liquid but also the idle fiber port. As the external environment, such as the temperature or the humidity, will change all the time, the retroreflection powers from the idle fiber port seriously perturb the results.

A circulator is employed instead of a fiber coupler as shown in Fig. 2 to solve the above problems. The port connected to the coupler works as the input port of the circulator. The reflection port of it is linked by the powermeter. The output port is immersed in the liquid by the optic fiber end face. The backreflection light comes from just the output port of the circulator

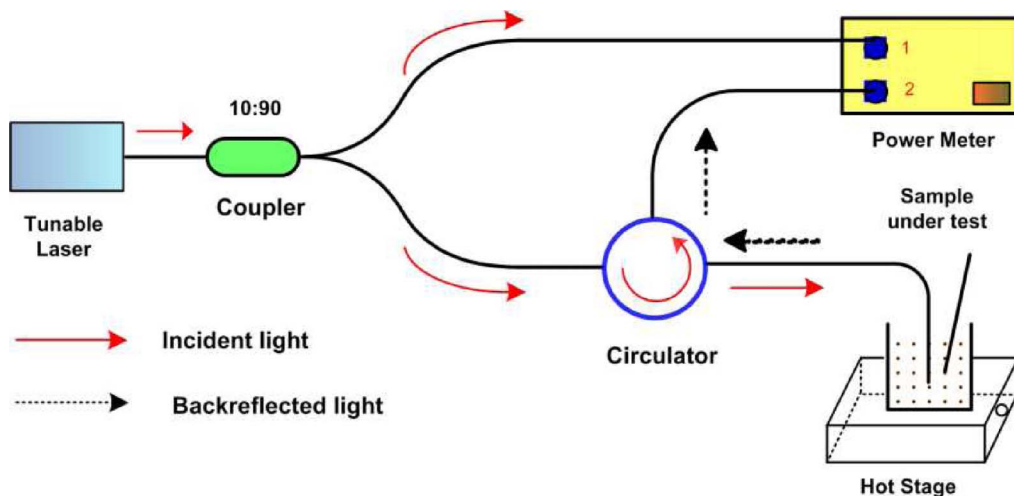


Fig. 2. (Color online) Improved experimental setup to measure the RI of liquids.

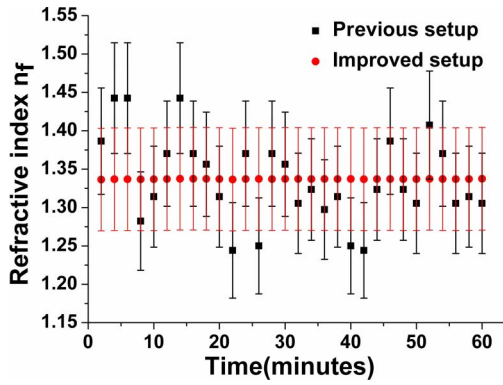


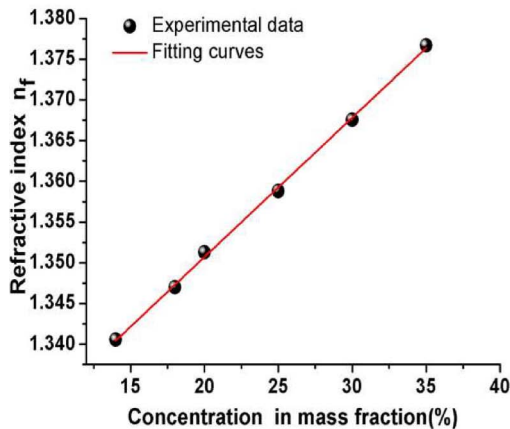
Fig. 3. (Color online) RI of sugar solution for different situations.

because there is no idle fiber port exposed to the surrounding environment. Therefore, the perturbation of  $\Delta n_f$  can be removed, and then the RI of the liquid according to Eq. (1) can be obtained in the following shortened form:

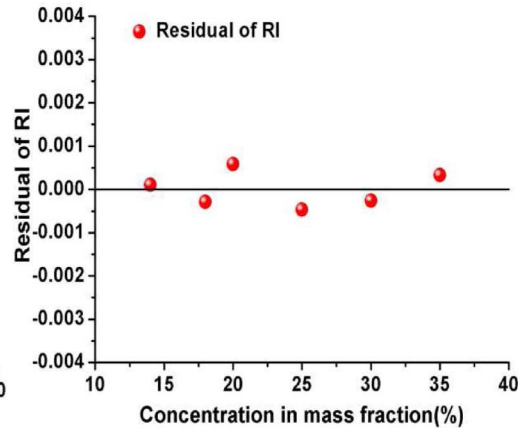
$$n_f = \frac{(1+k^2)n_{fc}}{1-k^2} \pm \sqrt{\left(\frac{(1+k^2)n_{fc}}{1-k^2}\right)^2 - n_{fc}^2 - k_f^2}. \quad (6)$$

### 3. Experimental Results and Discussion

To compare the above improved setup and the previous setup [9], the RI of the sugar solvent at different times is measured by both methods. In our experiments, air and water are chosen as the calibration samples. The operation central wavelength in our experiments is 1550 nm at room temperature. Since the sugar solution is transparent,  $k_f$  in Eq. (6) equals zero. The RI of the sugar solution in different situations at many times is depicted in Fig. 3. It is obvious that the stability of the experimental data is dramatically increased with the improved method. The strong perturbation of the RI of the liquid is attributed to the unstable surrounding circumstances.



(a)



(b)

Fig. 4. (Color online) (a) Concentration-dependent RI of sugar solution. (b) Residual of RI of sugar solution at 23°C.

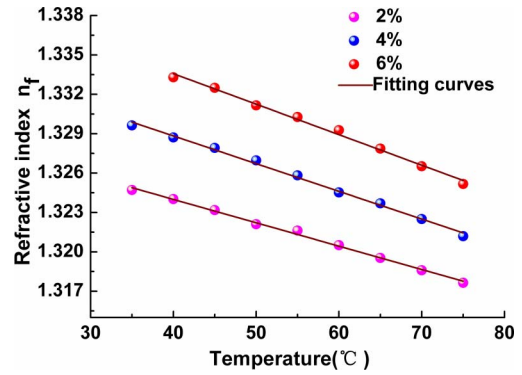


Fig. 5. (Color online) Temperature-dependent RIs of a sugar solution with different concentrations.

Figure 4(a) depicts the concentration-dependent RI of sugar solutions at room temperature when the sugar concentration increases from 14% to 35% (testing points at 14%, 18%, 20%, 25%, 30%, 35%). From the experimental data, we can find that the RI of the sugar solution increase linearly with the concentration. The slope of the linear fitting curves is 0.00171, and the standard error of the slope is  $2.59 \times 10^{-5}$ . Figure 4(b) displays the residual of the RI at different concentrations of sugar solution. It shows the good linear relationship between the measured RI and concentration.

The RI of sugar solution as a function of temperature is depicted in Fig. 5 for three samples with different sugar concentrations. The experimental result shows a linear dependence of the RI of the sugar solution on the temperature at a fixed concentration. It is evident that the RI decreases with the temperature. From the fitting curves with different concentrations, we can see that the slope of the temperature coefficient is  $-1.69 \times 10^{-4}$ ,  $-2.07 \times 10^{-4}$ , and  $-2.3 \times 10^{-4}$  (when the concentration is 2%, 4%, and 6%, respectively) with the order of the standard error as small as  $10^{-6}$ . The temperature coefficient gradient increases with concentration. The temperature coefficient

cient of the RI for different concentrations can be used to calculate the thermal expansion coefficient.

#### 4. Conclusion

An improved method to measure the RI of liquids has been described, in which a circulator is employed instead of a coupler. The measurement results show that the method has a high precision, of the order of  $10^{-4}$ . Comparing the methods between the previous setup and the improved setup demonstrates that the stability is greatly increased in the latter method. Concentration and the temperature-dependent RI of the sugar solution is also measured. The experimental setup we proposed can also serve as a high-precision sensor.

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#### References

1. J.-Q. Xi, M. F. Schubert, J. K. Kim, E. F. Schubert, M. Chen, S.-Y. Lin, W. Liu, and J. A. Smart, "Optical thin-film materials with low refractive index for broadband elimination of Fresnel reflection," *Nat. Photonics* **1**, 176–179 (2007).
2. X. Wu, L. Liu, Y. Zhang, D. Li, W. Wang, and L. Xu, "Low electric power driven thermo-optic multimode interference switches with tapered heating electrodes," *Opt. Commun.* **258**, 135–143 (2006).
3. G. Fishter, "Refractometry," in *Applied Optics and Optical Engineering, Volume IV, Optical Instruments—Part I*, R. Kingslake, ed. (Academic, 1967), p.363
4. K. Schmid and A. Penzkofer, "Refractive-index measurements with a Pellin–Broca prism apparatus," *Appl. Opt.* **22**, 1824–1827 (1983).
5. W. M. b. M. Yunus and A. b. A. Rahman, "Refractive index of solutions at high concentrations," *Appl. Opt.* **27**, 3341–3343 (1988).
6. Y. Lu and A. Penzkofer, "Optical constants measurements of strongly absorbing media," *Appl. Opt.* **25**, 221–225 (1986).
7. J. Chen, X. Chen, R. Xu, Y. Zhu, Y. Shi, and X. Zhu, "Refractive index of aqueous solution of CdTe quantum dots," *Opt. Commun.* **281**, 3578–3580 (2008).
8. C.-B. Kim and C. B. Su, "Measurement of the refractive index of liquids at 1.3 and 1.5 micron using a fibre optic Fresnel ratio meter," *Meas. Sci. Technol.* **15**, 1683–1686 (2004).
9. S. Pu, X. Chen, Y. Chen, W. Liao, L. Chen, and Y. Xia, "Measurement of the refractive index of a magnetic fluid by the retroreflection on the fiber-optic end face," *Appl. Phys. Lett.* **86**, 171904 (2005).
10. A. Schwartz and K. Berglund, "The use of Raman spectroscopy for *in situ* monitoring of lysozyme concentration during crystallization in a hanging drop," *J. Cryst. Growth* **203**, 599–603 (1999).
11. J. Olesberg, M. Arnold, S. Hu, and J. Wiencek, "Temperature-insensitive near-infrared method for determination of protein concentration during protein crystal growth," *Anal. Chem.* **72**, 4985–4990 (2000).
12. A. McPherson, A. Malkin, Y. Kuznetsov, S. Koszelak, M. Wells, G. Jenkins, J. Howard, and G. Lawson, "The effects of microgravity on protein crystallization: evidence for concentration gradients around growing crystals," *J. Cryst. Growth* **196**, 572–586 (1999).
13. S. Miyashita, H. Komatsu, Y. Suzuki, and T. Nakada, "Observation of the concentration distribution around a growing lysozyme crystal," *J. Cryst. Growth* **141**, 419–424 (1994).