

Polarized Photon-Pairs Heterodyne Polarimetry for Ultrasensitive Optical Activity Detection of a Chiral Medium

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A polarized photon-pairs heterodyne polarimetry is proposed in order to measure in an ultrasensitive manner the circular birefringence of a chiral medium via optical rotation detection. A balanced detector is integrated into this polarimeter. Thus, shot-noise-limited detection by this polarimeter can be achieved. Experimentally, the detection sensitivity for the circular birefringence of a glucose–water solution up to $\partial |n_r - n_l| = 2 \times 10^{-11}$ at 10 mg/dL is verified. To our knowledge, this is the highest sensitivity ever measured of a chiral liquid solution based on single traveling sample cell geometry. Finally, when compared to a fiber loop ring-resonator in the frequency domain for a chiral liquid, this polarimeter shows an order of 10^4 enhancement on the sensitivity of natural optical activity measurement.

1. Introduction

Circular birefringence,^{1–3} $|n_r - n_l|$, the difference between two circular polarized refractive indices n_r and n_l is dependent on the difference in the propagation speeds with respect to right and left circular polarized light waves in a chiral medium. However, even in a pure chiral liquid, $|n_r - n_l|$ is usually as low as a few parts per million.^{3,4} Therefore, it can only be measured either by enhancing the signal-to-noise ratio (SNR)^{2,4} or by increasing the propagation length such as when using a Fabry–Perot resonance cavity with multiple reflection mode.^{3,5} In the latter case, the optical rotation angle by sample cell is amplified through multiple reflection within the resonance cavity. Grand et al.⁵ proposed a helicoidal wave in the Fabry–Perot resonator, which is used in conjunction with a pair of quarter wave plates to amplify the optical rotation by a factor proportional to F^2 , which is the square of the finess of the Fabry–Perot cavity. The sensitivity is close to 10^{-6} deg for optical rotation measurement, and this was demonstrated using such an intracavity polarimeter. Thereafter, Poirson et al.⁶ used the same technique for vapor chirality detection. Optical rotation of 10^{-6} deg was thus demonstrated successfully. Recently, Vollmer et al.³ developed a fiber ring-resonator based on a resonance frequency shifting technique to measure the circular birefringence of a limonene liquid. Their sensitivity is 2×10^{-7} for $|n_r - n_l|$ by use of a ring resonator at a fineness of $F = 3.4$. This result implies that the sensitivity for $|n_r - n_l|$ can be enhanced further when a resonator of $F = 500$ is applied in their setup.⁷ An equivalent approach is to increase the SNR of the detected signal in order to enhance the sensitivity of the circular birefringence measurement. This can be done using an optical heterodyne interferometer that is capable at the shot-noise limit of detection. This can be obtained by integrating a balanced detector into the setup.^{8,9} Such an interferometer in association with a narrow band filter and synchronized detection

will allow the further enhancement of sensitivity.^{4,8–10} In this experiment, linear polarized photon pairs (LPPP), which are composed of pairs of correlated linear polarized P and S photons at different temporal frequencies, are generated by a frequency-stabilized Zeeman He–Ne laser.¹⁰ The spatial and temporal coherence of the LPPP are highly correlated when the LPPP undergo common-path propagation in a chiral medium. This introduces an equal optical rotation to the P and S polarizations simultaneously. Thereafter, the rotated LPPP generates two antisymmetric heterodyne signals inherently after the rotated LPPP is divided by a polarized beam splitter and detected by two photo detectors.⁴ This is critical to achieve shot-noise-limited detection coupled with a balanced detector.¹¹ Meanwhile, the optical path dependent phase of LPPP is cancelled out automatically. This results in coherent detection of the heterodyne signal at the same time. Snyder et al.¹² suggested a sub-shot-noise polarimeter that integrates a balanced-detectors scheme with quantum-correlated photon beams generated by an ultrastable optical parametric oscillator (OPO) for optical rotation angle measurement. This setup eliminates not only classical common mode noise, but also the correlated quantum fluctuations of the twin beams, thereby offering sensitivity below the shot-noise level. Recently, this system was successfully demonstrated by Feng et al.^{13,14} However, an ultrastable optical twin beam is required and an imperfect wave plate, polarizer, and cross-talk of the orthogonal polarizations in the OPO cavity will yield a residual beat note or heterodyning. Thus, the system becomes a classical heterodyne polarimeter when the optical rotation is larger than 0.1° .¹³ In this study, an arrangement involving the classical sensitivity of the heterodyne polarimeter in association with LPPP and a balanced detector scheme for optical polarization rotation detection is set up, where a frequency-stabilized Zeeman He–Ne laser is adopted. As a result of its simple geometry, the polarized photon-pairs heterodyne polarimeter (PPHP) is then implemental for optical polarization rotation measurement in real time. Experimentally, the noise floor of PPHP at zero angle rotation is close to that of electronic noise (flat noise) and was demonstrated. Thus, a shot-noise-limited

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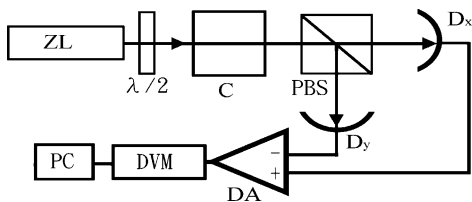


Figure 1. The optical setup of PPHP. ZL, Zeeman laser; $\lambda/2$, half wave plate; C, cuvette; PBS, polarization beam splitter; Dx and Dy, photo detectors; DA, differential amplifier; DVM, digital voltmeter; PC, personal computer.

detection of the PPHP is achieved. If we compare PPHP with a conventional polarimeter in which a Fabry–Perot resonance cavity of multiple-reflection mode is introduced on the optical polarization rotation measurement, this novel polarimeter performs much better than conventional methods, because PPHP is capable of achieving not only shot-noise-limited detection but also a common phase noise rejection mode. In addition, a conversion of an optical polarization rotation angle into the amplitude of an amplitude-modulated (AM) heterodyne signal is operated automatically. As a result, a high SNR and a high modulation index (MI) of the detected signal are generated. Therefore, the sensitivity of this amplitude-sensitive PPHP is greatly improved. Finally, the sensitivity of PPHP on both optical polarization rotation and circular birefringence of glucose–water solution are analyzed.

2. Principle and Experimental Setup

As shown in Figure 1, a Zeeman He–Ne laser emits LPPP in which a pair of correlated linear polarized P and S photons are produced, and their temporal frequencies are ω_p and ω_s , respectively.

Then, a half wave plate (HWP) rotates the P and S waves an equal angle simultaneously when P and S polarizations are aligned parallel to the x-axis and y-axis of the setup, respectively. Thus, the Jones matrices of the P and S waves can be expressed by $A_p e^{i\omega_p t} \begin{bmatrix} 1 \\ 0 \end{bmatrix}$ and $A_s e^{i\omega_s t} \begin{bmatrix} 0 \\ 1 \end{bmatrix}$, where A_p and A_s are the amplitudes of P and S waves, respectively. Once the laser beam transmits through a nonabsorbed chiral medium with an optical thickness l , this will produce an angle rotation $\delta\theta$ of both the P and S polarizations in the x–y plane at same time. Then, a polarized beam splitter (PBS) selects the x and y components of the rotated P and S waves simultaneously such that two optical heterodyned signals are generated at the photo detectors Dx and Dy. Thus

$$I_x = |A_p e^{i(\omega_p t + \phi_p)} \cos \delta\theta - A_s e^{i(\omega_s t + \phi_s)} \sin \delta\theta|^2 \quad (1)$$

$$I_x = A_p^2 \cos^2 \delta\theta + A_s^2 \sin^2 \delta\theta - A_p A_s \sin(2\delta\theta) \times \cos(\Delta\omega t + \Delta\phi) \quad (2)$$

$$I_y = |A_p e^{i(\omega_p t + \phi_p)} \sin \delta\theta + A_s e^{i(\omega_s t + \phi_s)} \cos \delta\theta|^2 \quad (3)$$

$$I_y = A_p^2 \sin^2 \delta\theta + A_s^2 \cos^2 \delta\theta + A_p A_s \sin(2\delta\theta) \times \cos(\Delta\omega t + \Delta\phi) \quad (4)$$

If $A_p \cong A_s = A_0$ is satisfied, then eqs 2 and 4 become antisymmetric to each other inherently.

$$I_x \cong A_0^2 [1 - \sin(2\delta\theta) \cos(\Delta\omega t + \Delta\phi)] \quad (5)$$

$$I_y \cong A_0^2 [1 + \sin(2\delta\theta) \cos(\Delta\omega t + \Delta\phi)] \quad (6)$$

where $\Delta\phi = \phi_p - \phi_s$ is defined and $\Delta\omega = \omega_p - \omega_s$ is the beat frequency of the heterodyne signal. Both ϕ_p and ϕ_s are the optical path dependent phases with respect to the P and S polarizations of LPPP, which undergoes common-path propagation in the chiral medium. A balanced detector scheme is then suggested in Figure 1 that is able to reduce the laser intensity fluctuation effectively. Therefore, the output voltage from a differential amplifier (DA) in the balanced detector scheme becomes

$$|\Delta I^-(\Delta\omega t)| = |I_y - I_x| \cong 2A_0^2 |\sin(2\delta\theta)| \cos(\Delta\omega t + \Delta\phi) \quad (7)$$

Equation 7 belongs to an amplitude-modulated (AM) signal where the optical rotation angle is proportional to the amplitude of the signal. In the case where the amplitude of P and S waves are expressed by $A_s = A_p + \Delta A$ and $\Delta A/A_p \ll 1$, then the residual excess noise from DA becomes $2A_p^3 |\sin(2\delta\theta)|$. It can be ignored when a small optical rotation angle is detected, and the glucose concentration detection in this experiment satisfies the condition. Meanwhile, the phase difference between the pair of polarized photons of LPPP when propagating a distance l in a chiral medium is $\Delta\phi = n/c \Delta\omega l \approx 0$ when the thickness of glass cuvette ($l = 1$ cm) and beat frequency ($\Delta\omega = 2.6$ MHz) of a Zeeman He–Ne laser are used. c is the speed of light in a vacuum, and $n \cong 1.33$ is the refractive index of the solution. Finally, the amplitude of eq 7 is demodulated precisely using the enveloped detection of the solution.

$$|\delta\theta| \cong \frac{1}{2} \sin^{-1} \left(\frac{|\Delta I^-|}{2A_0^2} \right) \quad (8)$$

$$|\delta\theta| \cong \frac{|\Delta I^-|}{4A_0^2} \quad (9)$$

To add eqs 5 and 6

$$|\Delta I^+| = |I_x + I_y| \cong 2A_0^2 \quad (10)$$

Then

$$|\delta\theta| \cong \frac{1}{2} \sin^{-1} \left\{ \frac{|\Delta I^-|}{|\Delta I^+|} \right\} \quad (11)$$

$$|\delta\theta| \cong \frac{|\Delta I^-|}{2|\Delta I^+|} \quad (12)$$

From eq 11 or 12, $\delta\theta$ can be obtained by means of the output intensity from DA and a digital voltmeter (DVM) in real time. According to the relationship between the optical rotation angle and circular birefringence of a chiral medium, $\delta\theta = \pi/\lambda(n_r - n_l)l$ where n_r and n_l are the circular refractive indices with respect to right and left circular polarizations³ and λ is the wavelength of the laser. Then, $|n_r - n_l|$ can be calculated in terms of the optical rotation angle being measured. In order to reduce the excess noise from laser intensity fluctuation effectively, a balanced detector scheme is integrated into the PPHP where shot-noise-limited detection is performed.

3. Results and Discussion

In this setup, a Zeeman He–Ne laser (Agilent, HP 5519A) of 0.25 mW and a beat frequency of 2.6 MHz was used. In order to calibrate the amplitude stability of the polarimeter,

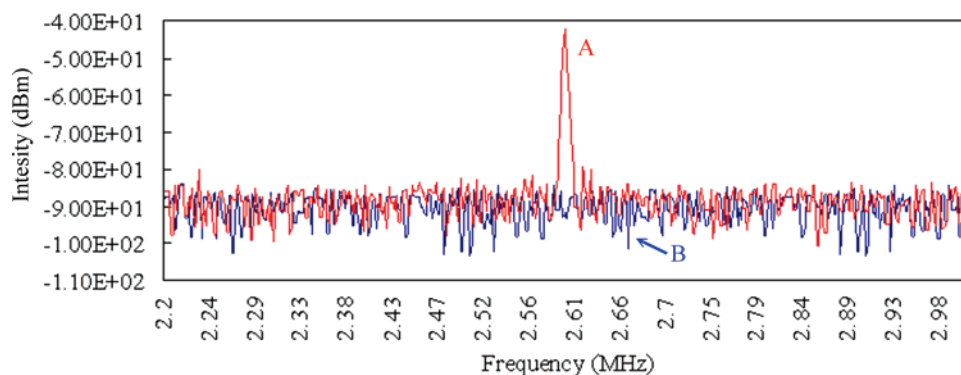


Figure 2. Shot-noise-limited detection of PPHP where curve A is noise floor of detected heterodyne signal at zero glucose concentration (tri-distilled water). Curve B represents the electronic noise level when the laser beam was blocked in the measurement.

distilled water was tested. During the measurement, the HWP was rotated until the output intensity $|\Delta I^-|$ from a DVM (Agilent, HP34401A) is maximized ($|\Delta I^-| = |\Delta I^-|_{\max}$). Thus, $A_0^2 = |\Delta I^-|_{\max}/2$ is obtained. In order to optimize the detection sensitivity of this experiment, the HWP was then adjusted so that the P and S polarizations are rotated to a preset angle, $\delta\theta_0 = 4^\circ$ relative to the x -axis and y -axis of PBS (Agilent, HP10565B), respectively. This introduces a carrier frequency into the heterodyne signal at zero glucose concentration. The output signal from DA (LeCroy DA1855A) becomes and

$$|\Delta I^-(\Delta\omega)| \cong 2A_0^2 |\sin 2(\delta\theta_0 + \delta\theta)| \cos(\Delta\omega t) \quad (13)$$

$$\delta\theta \cong \frac{1}{2} \sin^{-1} \left\{ \frac{|\Delta I^-|}{2A_0^2} \right\} - \delta\theta_0 \quad (14)$$

$$|\Delta I_0^-| \cong 2A_0^2 \sin(2\delta\theta_0) \quad (15)$$

Then, eq 14 becomes

$$\delta\theta \cong \frac{1}{2} \sin^{-1} \left(\frac{|\Delta I^-|}{2A_0^2} \right) - \frac{1}{2} \sin^{-1} \left(\frac{|\Delta I_0^-|}{2A_0^2} \right) \quad (16)$$

Therefore, PPHP not only reduces the excess noise due to laser intensity fluctuation but also converts the optical rotation angle into the amplitude of an AM heterodyne signal. Because of a nonzero optical rotation by presetting HWP a rotation angle $\delta\theta_0$ in this setup, the output signal at zero glucose concentration becomes offset at $2A_0^2 \sin(2\delta\theta_0)$ in eqs 13 and 14. Therefore, the polarimeter is capable of providing the direction of optical rotation angle clockwise or counter-clockwise determined by the output voltage being positive or negative, respectively, compared to the output voltage at zero concentration. However, a limitation on dynamic range of optical rotation is dependent upon the magnitude of $\delta\theta_0$, and the sign from the x -axis. In order to verify the polarimeter at shot-noise-limited detection, tri-distilled water was tested, where the HWP is rotated until the output voltage at $\Delta\omega = 2.6$ MHz is minimized by the use of a spectrum analyzer (Advantest R3361A). Figure 2 shows that the noise floor is at -90 dBm; however, the output voltage at 2.6 MHz is -42 dBm.

The reason for a peak output voltage at 2.6 MHz in Figure 2 is the polarization leakage of the P and S waves of the Zeeman He–Ne laser due to linear birefringence of the cavity that produces the heterodyne signal at the beat frequency.¹⁵ Nevertheless, it is clearly seen in Figure 2 that the noise floor of the heterodyne signal nearly coincides with the electronic noise level when the laser beam is blocked during the measurement. The

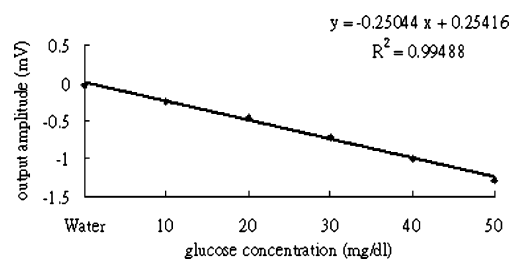


Figure 3. The linearity of the output voltage versus the glucose concentration by PPHP. The size of the error bar in this measurement is smaller than the size of the solid diamond.

small difference between the shot-noise-limited level and electronics noise floor is due to low laser power (0.25 mW) in this experiment. Moreover, the common-path propagation of LPPP in a chiral medium introduces a common phase noise rejection mode such that the laser frequency noise and background induced phase noise are cancelled out automatically. Thus, the conversion from phase noise into amplitude noise is prevented.¹⁶ Furthermore, the amplification factor $2A_0^2$ in eq 7 enlarges the optical rotation angle and improves the detection sensitivity. In this experiment, different concentrations of D-(+)-glucose from zero to 50 mg/dL were measured. Because glucose solution presents two isomers, α -D-glucose and β -D-glucose, the optical rotation angle detection can be measured precisely if the equilibrium mixture of α -D-glucose and β -D-glucose at ratio 36:64 is produced after mutarotation.¹⁷ In this experiment, the time needed to prepare the glucose solutions of different concentrations was longer than the one hour that the equilibrium mixture of glucose is produced properly. Figure 3 shows the experimental results for the linear dependence of the demodulated amplitude of the heterodyne signal versus glucose concentration where $\delta\theta$ becomes 0° when tri-distilled water was tested.

The experimental result shown in Figure 3 verifies the ability of this important information of PPHP apparently. From Figure 3, the negative slope is caused by optical rotation clockwise versus higher concentration of D-(+)-glucose from tri-distilled water in which a preset rotation angle of HWP was adjusted in the calibration process. This is the verification in the experiment of the ability of PPHP to obtain the information about the optically active medium being levorotatory or dextrorotatory. In the meantime, the absolute value of the slope in Figure 3 is proportional to the specific optical rotation $[\alpha]_{25^\circ\text{H}_2\text{O}(\text{pH}7)}^{633}$ of glucose, which can be obtained simultaneously by this polarimeter too.

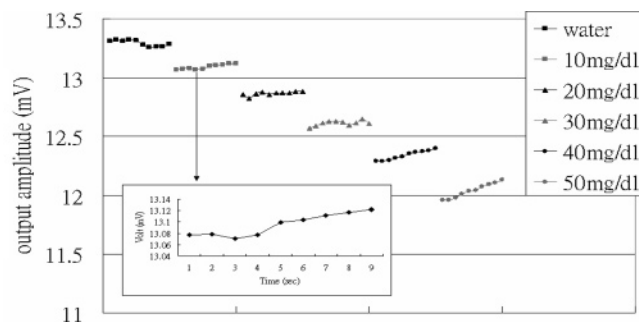


Figure 4. The amplitude response of the heterodyne signal versus the concentration of the glucose–water solution in the range from 0 to 50 mg/dL. The inserted picture is an enlargement of the signal for clarity.

It should be noted that the optical rotation angle is very small in the region from zero to 50 mg/dL in this experiment, and therefore, from eq 12

$$\frac{\partial(|\Delta I^-|)}{|\Delta I^-|} = \frac{\partial(|\delta\theta|)}{|\delta\theta|} \quad (17)$$

According to Biot's law,¹ the variation of the concentration δC is proportional to the optical rotation angle $\delta\theta$ in terms of the relationship

$$\delta\theta = [\alpha]_{T,pH}^{\lambda} \cdot l \cdot \delta C \quad (18)$$

where $[\alpha]_{25^{\circ}\text{H}_2\text{O}(pH7)}^{633} = 45.5 \text{ deg} \cdot \text{mL} \cdot \text{g}^{-1} \cdot \text{dm}^{-1}$ is the specific optical rotation of D-(+)-glucose under the given conditions at $\lambda = 632.8 \text{ nm}$, $T = 25^{\circ}\text{C}$, and $pH = 7$.¹⁸ Then, the sensitivity of polarimeter can be expressed by

$$\frac{\partial(|\delta\theta|)}{l} = \frac{\partial(|\Delta I^-|)}{|\Delta I^-|} [\alpha]_{T,pH}^{\lambda} \cdot \delta C \quad (19)$$

From Figure 4, $\{\partial(|\Delta I^-|)\}/\{|\Delta I^-|\} = 1/\text{SNR} \cong 1/10$ is calculated at a concentration of 10 mg/dL where the variation $\partial(|\Delta I^-|)$ is the standard deviation of measured data of 10 mg/dL at 2.6 MHz beat frequency in Figure 4. The experimental result shown in Figure 4 verifies that α -D-glucose and β -D-glucose are in equilibrium after mutarotation. Otherwise, a big fluctuation on output signal is detected due to a large difference in specific rotations $[\alpha]_D = 112$ for α -D-glucose and $[\alpha]_D = 18.7$ for β -D-glucose isomers in solution.¹⁷ In fact, a small variation of the output signal was observed in Figure 4 which might be caused by the convection of the glucose solution and the temperature variation. Because the thickness of glass cuvette is $l = 1 \text{ cm}$, the sensitivity for optical rotation detection by this novel polarimeter is calculated at $\{\partial(|\delta\theta|)\}/\{l\} = 5.5 \times 10^{-5} \text{ deg/cm}$ under an arrangement involving single traveling sample cell geometry as shown in Figure 1. In addition, on the basis of the relationship of an optical rotation angle $|\delta\theta|$ to circular birefringence $|n_r - n_l|$ in a chiral medium, the sensitivity for circular birefringence becomes

$$\partial|n_r - n_l| = \frac{\partial(|\delta\theta|)}{l} \cdot \frac{\lambda}{\pi} \cong 2 \times 10^{-11} \quad (20)$$

in this experiment. To compare this result with the latest experimental data of $\partial|n_r - n_l| \cong 2 \times 10^{-7}$ obtained by Vollmer et al.³ on limonene using a Fabry–Perot cavity, the sensitivity of this novel polarimeter by single pass geometry is 10^4 higher than their experimental result. Recently, we measured circular birefringence of crystal quartz by this novel polarimeter.⁴ The experimental result demonstrates the sensitivity for circular

birefringence at $\partial|n_r - n_l| = 8.5 \times 10^{-10}$ and the optical rotation angle at $\{\partial|\delta\theta|\}/\{l\} \cong 3 \times 10^{-5} \text{ deg/cm}$. It is comparable to the highest sensitivity published for optical rotation measurement of the conventional polarimeter^{5,6} in which a Fabry–Perot resonance cavity is used. If a Fabry–Perot cavity resonance or a multiple traveling sample cell geometry is integrated into this polarimeter, a factor of $\{4KF^2\}/\{\pi^2\}$ is amplified on sensitivity theoretically where K denotes the Fabry–Perot transmission coefficient. The result implies that 10^{-8} deg/cm on optical polarization rotation is possible when a Fabry–Perot cavity of $F = 150$ is introduced in this polarimeter.⁵ Equivalently, $\partial|n_r - n_l| \cong 10^{-14}$ is anticipated too. On the basis of its simple geometry and high sensitivity on optical rotation detection, the performance of PPHP becomes equivalent to the detection sensitivity of the resonant cavity polarimeter and cavity ring down polarimeter^{6,19} on optical polarization rotation measurement, which are experimentally capable of 10^{-6} deg/cm on sensitivity with a Fabry–Perot resonance cavity. We believe, to our knowledge, that PPHP gives the highest sensitivity for circular birefringence detection by a single traveling sample cell geometry.²⁰ In summary, an ultrahigh-sensitivity PPHP on optical rotation or circular birefringence measurement of a chiral medium in the liquid phase is developed and experimentally demonstrated. The significance and importance of PPHP can provide a complementary method compared with the conventional method on optical rotation detection in chemistry and biological science. The merit of PPHP is not only on its single traveling geometry and high sensitivity but also the significance of PPHP that is potentially able to extend into stereochemistry for non-superimposable asymmetric molecular structure in the future.

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