Capillary refractometer integrated in a microfluidic configuration

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We propose a microfluidic method to measure the refractive index of liquids. This method is based on the dynamic focusing by a capillary when liquids with different refractive indexes are inserted into it. Fabrication of such a refractometer has been done by encapsulating two fibers and a capillary. A calibration method is proposed. © 2008 Optical Society of America

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1. Introduction

The measurement of refractive index has been done through the years by means of refractometers like the Abbe, Jamin, Pulfrich, Rayleigh, Hilgen–Chance, and others [1]. Some of them are based on the critical angle method and others on interferometry. Recently a new class of refractometers has appeared. They are based on microfluidics, the science of fluids confined on the micrometer scale. Some of the new refractometers work with Fabry–Perot microcavities [2], diffraction gratings [3], hollow-core optical waveguides [4], photonic crystal microcavities [5], evanescent field interaction nanowires [6], and microring resonators [7]. Those that use the gratings have arrays of parallel channels, 50 μm wide, filled with fluids. Hollow-core optical waveguides simultaneously confine light and the substance to be measured in the waveguide core. Two dimensional photonic crystals microcavities rely on the shift of resonant wavelength. Interaction of nanowires is done when a tapered fiber, immersed in a transparent soft curable polymer, interacts by means of the evanescent field with a fluid that is confined in a microchannel. These miniaturized photonic devices, which can integrate some optical elements in a single chip, could revolutionize chemical analysis. Fabrication of these new classes of refractometers requires a dedicated use of an expensive fabrication technology, developed and optimized for different research applications i.e., microelectronics.

In this paper we propose a simple method to measure the refractive index of liquids by using a capillary and two fibers embedded in a polymeric matrix. In Section 2 we describe the physical principle of the proposed refractometer. Section 3 shows the materials and layout of the optical configuration. Section 4 describes the method to calibrate the refractometer, and finally in Section 5 we comment and conclude.

2. Physical Principle

Lens maker's equation is a relation among the radii of curvature of the lens (r_1 and r_2), the index of refraction of the lens (n_l) and the medium (n_m), where it is embedded, and the focal distance f. The lens maker's equation for thin lenses is given by the following relation [8]:

$$1/f = (n_l - n_m)((1/r_1) - (1/r_2)). \tag{1}$$

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This equation defines an optical element once the radii and the refractive indices are given. If the lens is immersed in air $n_m = 1$.

Nowadays we can find optical elements that can change their focal distance. These dynamical elements [9–12] have sizes ranging from a few centimeters to big segmented telescope mirrors. Usually small active optical elements comprise piezoelectric actuators that modulate the surface of the mirror. Thus, by changing the radius a dynamical element is obtained. However, another way to implement a dynamical element is by changing the refractive index n_l.

In Fig. 1 the behavior of the focal length as a function of the refractive index of a lens is shown. This behavior was calculated with Eq. (1) supposing $n_m = 1.4126$, $r_1 = 0.615\,\text{mm}$, $r_2 = -0.615\,\text{mm}$. It is interesting to note that when the refractive index of the lens is smaller than that of the surrounding medium, the lens is a negative element. Then, when the refractive indices of the lens and medium are equal, the focal distance is very large, and when the refractive index n_l is larger than n_m, the lens is a positive element.

The situation that describes the lens maker's equation is difficult to achieve when we use liquid lenses. Usually a container is needed to confine the liquid. This container has walls that present a thickness. In Fig. 2 a series of schemes, made by using an optical design program, show the physical situation when a collimated beam of light is sent to a capillary lens ($r_1 = 0.615$, $r_2 = -0.615\,\text{mm}$) embedded in a polymeric matrix with a refractive index of 1.4126. Inner diameter of the lens is $939\,\mu\text{m}$ and outer diameter is $1230\,\mu\text{m}$. Parameter in the drawings is the refractive index of liquid that fills the lens. Capillary container walls consist of silica glass with a refractive index of 1.45851. We can see that the lens behaves like a negative lens for some refractive indices and as a positive one for other indices. Note, if a detector is placed close to the second surface, i.e., after the light leaves the lens, the detector will receive more energy as the refractive index grows. This is the physical basis of the proposed capillary refractometer.

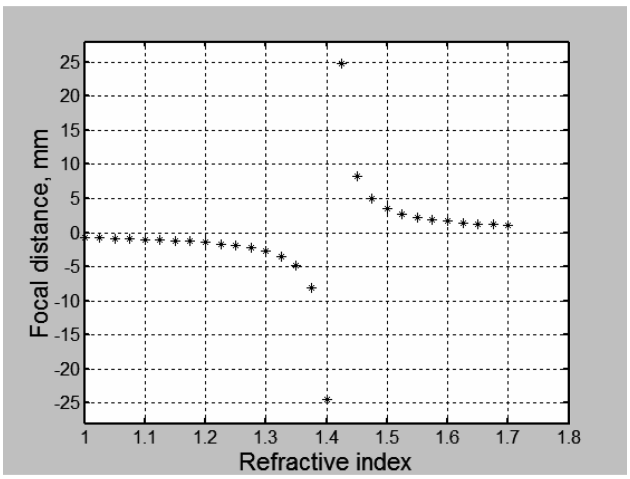

Fig. 1. Behavior of focal distance as a function of lens refractive index calculated with the lens maker's equation. It was supposed that the lens was immersed in a medium with a 1.4126 refractive index.

It should be mentioned that Fig. 2 shows the behavior of light only in the plane of the page. A capillary lens behaves like a cylindrical lens; thus the spatial distribution of light after the capillary has an ellipse cross section. The ellipse will change size as different liquids are introduced into the capillary.

3. Refractometer Layout and Fabrication

To implement the ideas proposed in Section 2, we chose the optical configuration depicted in Fig. 3. Light source for the capillary refractometer was a stabilized He–Ne laser ($\lambda = 632.8\,\text{nm}$). Its beam shows a Gaussian intensity cross section. The fiber that was chosen to send the light to the refractometer was an SM 600 (NA = 0.12). This fiber is designed to work in a single mode regime for red light. This light that traverses the capillary will show an ellipse cross section, as mentioned herein. Intensity in the major axis of the ellipse is more spread than in its minor axis. That means light distribution does not have a symmetry of revolution.

We mentioned that the SM 600 fiber, used to illuminate the capillary, works in a single mode regime. This characteristic should be kept if another fiber is used because otherwise higher order modes will be shown. These modes present nodes in the spatial distribution of light, and measurement of the refractive index will not be possible.

In Fig. 2 we have seen that an elliptical light cone at the output of the capillary changes its diameter as the refractive index of liquid inside changes. In the drawings it is supposed that light comes from infinity. One method to achieve this configuration is by placing the tip of the fiber very far from the capillary tube. Light that arrives at the capillary tube will have a plane wavefront but its intensity will be weak because most of the light will be spread, and the capillary lens will focus on just part of the beam. To avoid this loss of light the optical design program was used to find the distance of where to place the tip of the fiber so that all the light emerging from the fiber is focused by the capillary tube. It was found that a good position to place the tip of the fiber was 1.98 mm from the capillary. In this position light illuminated the central part of the capillary and no light was lost. Maximum refractive index liquid (immersion oil) that was available at measurement time had a value of 1.515. With this refractive index the conjugate distance of the tip of the SM 600 fiber was −9.44 mm. We have this negative value because the face of the feeding fiber is within the focal distance of the capillary filled with immersion oil.

In Fig. 4 the dynamical focusing of a beam of light given by an optical fiber placed at 1.98 mm from the capillary is shown. This figure was calculated with

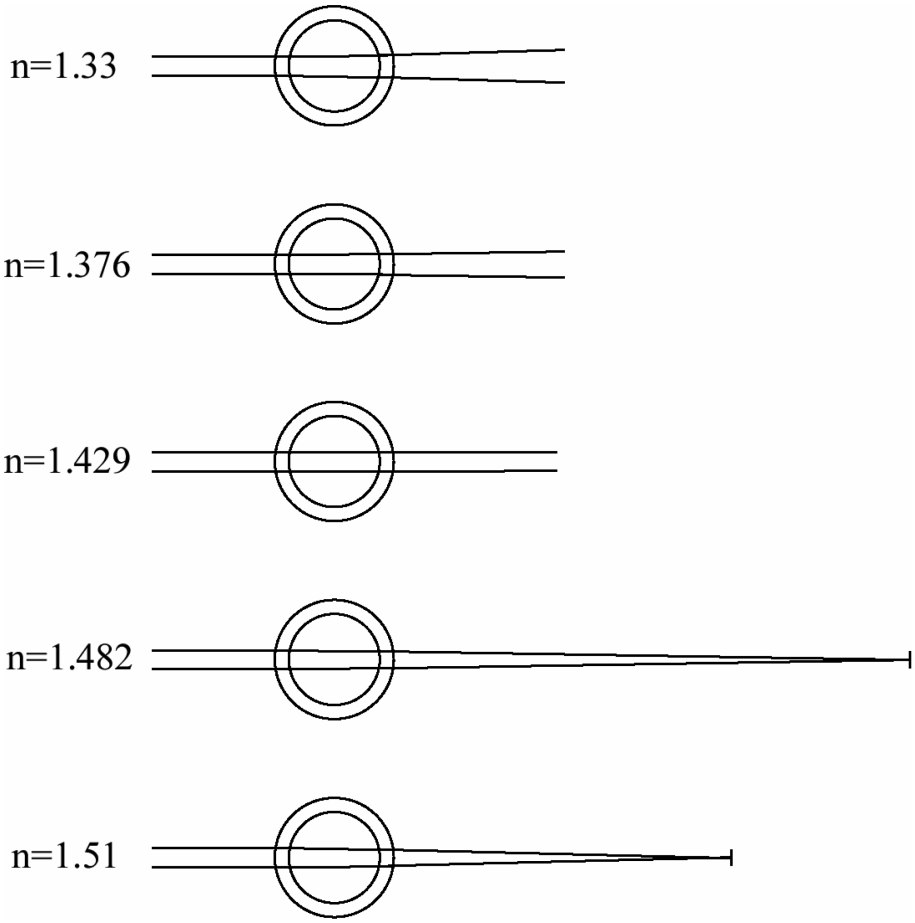

Fig. 2. Diagrams, given by an optical design program, of a capillary lens focusing collimated light. Parameter in the diagrams is the refractive index of the lens. For parameters of the lens see text.

an optical design program. Also in Fig. 4 the diameter of the emerging beam is shown when it leaves the capillary lens for each different refractive index. In these calculations a value of 7.3° for the light cone of the SM 600 was used. This angle is a measured quantity.

Capillary used to convey the liquid was obtained by drawing a fused silica tube in an optical fiber drawing tower. The walls of the capillary were chosen to be thin to have the maximum amount of liquid in the capillary. With this configuration the refraction phenomenon is more noticeable. Capillaries with

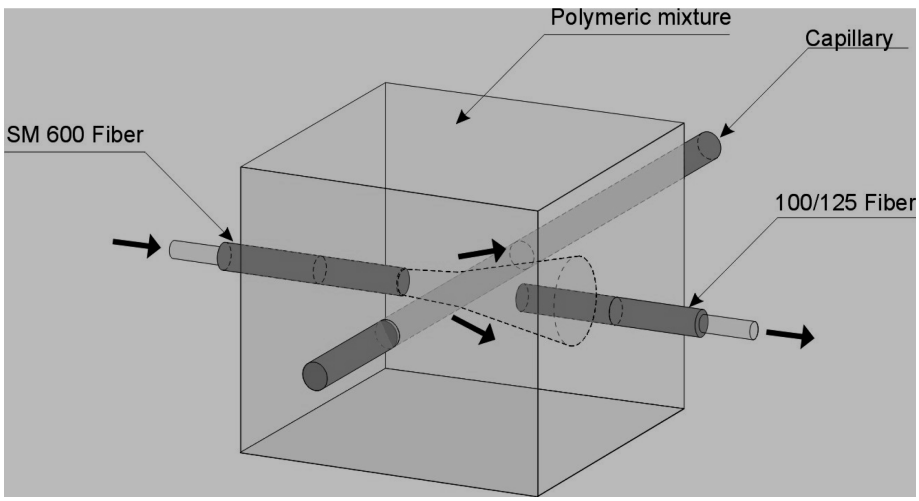

Fig. 3. Scheme showing the layout of the fibers and the capillary to form the capillary refractometer. (Not to scale.)

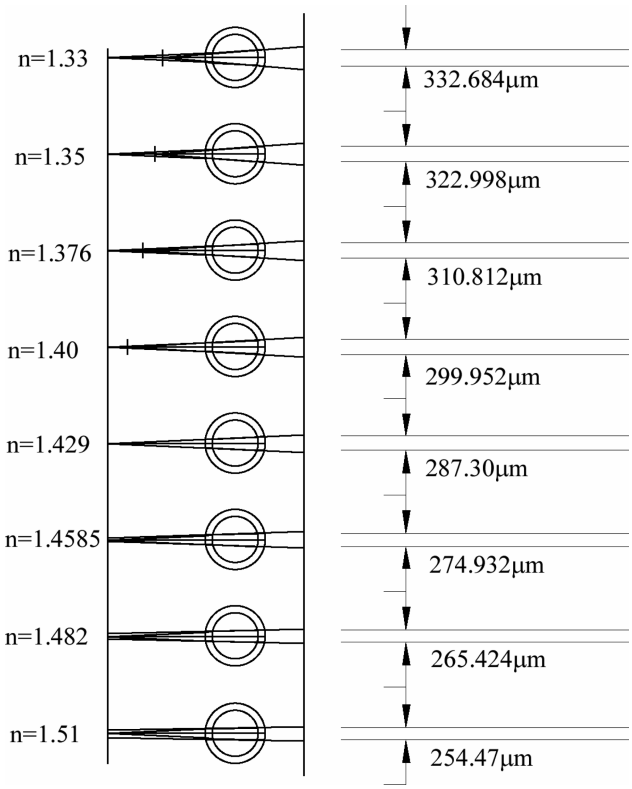

Fig. 4. Diagrams, given by an optical design program, of a capillary lens that focus light from a fiber that is 1.98 mm far from the capillary. Parameter in the diagrams is the refractive index of the liquid that fills the capillary. Diameter for each emerging beam is shown.

several ratios between the inner and outer diameters were fabricated. Finally we have found that one with an outer diameter of 1230 μm and an inner diameter of 939 μm worked better. In Fig. 5 a cross section of the capillary is shown.

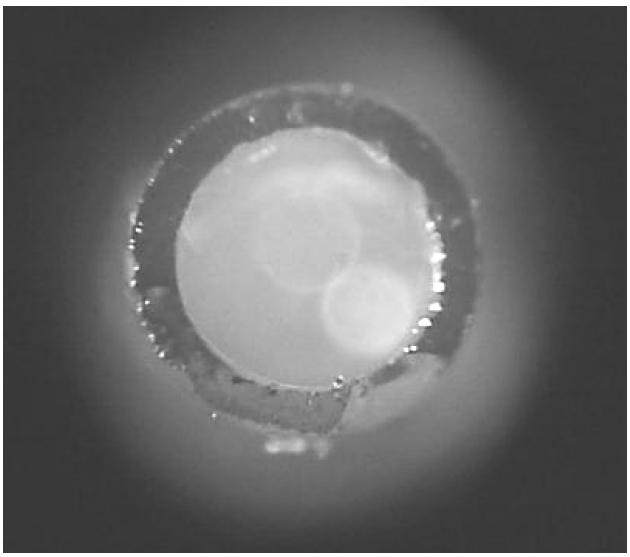

Fig. 5. Cross section of the capillary used in the experiments. Outside diameter is 1.23 mm.

To collect some of the light focused by the capillary, two classes of fibers were considered. One was a single mode fiber (SMF 28) with a core diameter of about 9 μm, and the other a multimode fiber with a core diameter of 100 μm and a clad diameter of 125 μm, i.e., a 100/125 fiber. (The major concern in light coupling between fibers is the poor coupling efficiency.) We made some preliminary experiments and observed that the light gathered by the small core diameter of the SMF 28 was too weak. However, in the 100/125 fiber the intensity of the output signal was high enough to be measured.

Several positions of the input face for the 100/125 fiber, with respect to the capillary, were tried experimentally. Finally it was placed at 3.06 mm from the capillary. This gave us enough intensity when the refractive indices changed.

The method to fix the fibers and the capillary tube was done by placing them in a small plastic box (11 mm × 11 mm × 8 mm). Then the box was filled with a polymeric mixture [13], i.e., components were encapsulated. At this step the mixture has the consistency of heavy syrup. Once the mixture was poured fibers were placed, with respect to the capillary, with micropositioners. Then the mixture was left to cure for 24 h. At the end of the process a polymerized and transparent rigid matrix was found. Figure 6 shows a photograph of one of the refractometers made.

4. Refractometer calibration

Once the capillary refractometer is fabricated a calibration curve for it is obtained with the following method. Light from an He–Ne laser was sent to the refractometer by means of the SM 600 fiber. The output face of the 100/125 fiber was coupled to a detector. Time was let to pass to achieve a stable He–Ne light intensity.

Liquids with different refractive indices were prepared with the following method. It is known [14] that the refraction index for aqueous solutions of sucrose depends on the water/sugar ratios. A series of water/sugar mixtures were prepared with the following arbitrary refractive indices: 1.35, 1.40, and 1.429. These indices were measured with an Abbe refractometer. Besides these mixtures other liquids were obtained with different refractive indices: distilled water, 1.33; isopropyl alcohol, 1.376; ciclohexanol, 1.458; special liquid [15], 1.482; and immersion oil, 1.51. These liquids were chosen not only because of their refractive indices but also because they had low viscosity.

To obtain the calibration curve a few drops of each liquid were placed, one liquid at a time, in one end of the capillary. These drops filled the capillary. Then the intensity of light transmitted by the 100/125 fiber was measured. The graph in Fig. 7 shows the results. It is seen that as the refractive index grows, intensity of light collected by the fiber also grows, indicating the focusing of light by the capillary cylindrical lens. With this curve it is now possible

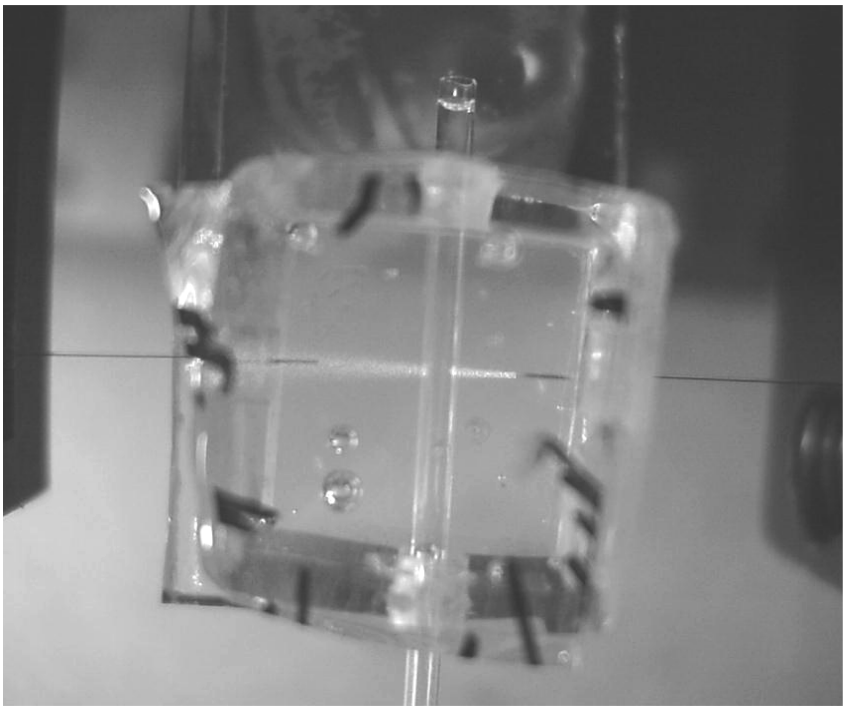

Fig. 6. Capillary refractometer made with two fibers and a capillary. Dimension of the plastic box is about 11 mm × 11 mm × 8 mm. At measurement time liquids are inserted into the capillary.

to know the refractive index of some liquid, inserted into the capillary, by measuring the intensity of the output light.

Software was used to determine the polynomial that gave the best fit to the set of experimental measurements. The formula obtained is $I = 0.132$

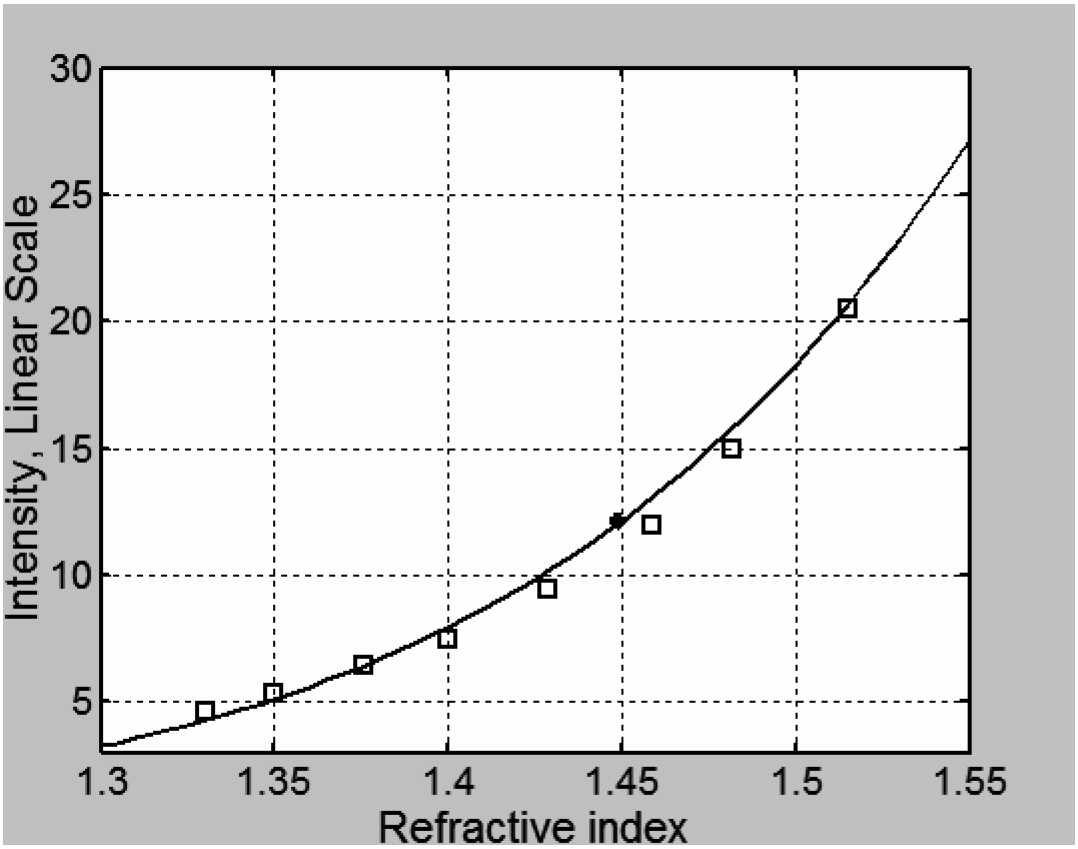

Fig. 7. Calibration curve. Squares show the intensity of light, collected by the 100/125 fiber, versus the refractive index of the liquid in the capillary. Solid curve shows the interpolated curve. Asterisk shows the test liquid.

$r^{12.10513}$, where I is the measured intensity of the light given by the 100/125 fiber, and r is the refractive index. This curve has also been plotted in Fig. 7.

To know the refractive index of a test liquid we inserted some drops of it and measured the intensity. A value of 12.1 was obtained. By using the polynomial a refractive index of 1.449 was obtained. The value of the liquid refractive index measured with an Abbe refractomer was 1.44. There is a difference of 0.009.

5. Comments and Conclusions

Regarding the rigidity of the system fiber—capillary–fiber, when it is immersed in the polymeric matrix, it resulted to be very stable. The matrix fixes optical components well. This matrix, silicone, shows low absorption and scattering, and high transmittance. The only drawback found, when manipulating the polymer, is the appearance of some bubbles. These have no effect in the optical configuration unless one of such bubbles interposes in the optical path.

Due to its small size, the capillary refractometer is transportable. However, the use of an He–Ne laser can restrict its transportability. To improve the system we suggest the use of a laser diode (LD) [16] coupled to a fiber. The fiber should be single mode for the wavelength of the LD.

In the developing of the system it was noticed that the alignment of the system fiber—capillary–fiber should be precise. If the sending fiber was slightly out of the optical axis, the cone of the output light beam would show angular movements when the liquids with different refractive indices are inserted into the capillary. This movement will affect the measurements, because the output of the SM 600 fiber shows a high intensity in the center and low intensity in the periphery, as was mentioned herein. A well aligned system should show only the focusing of light as the refractive index changes. Fortunately positioning of the fibers and capillary has to be done just once.

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