# Laser Photometric Probe for Concentration Measurements in Liquid Dispersions

**H. Schmidt, C. Tsouris**
**E. Eggert, L. L. Tavlarides**
Department of Chemical Engineering
and Materials Science
Syracuse University
Syracuse, NY 13244

Numerous techniques for measuring local concentration in various processes have been developed over the last thirty years. The most important of these techniques provides instantaneous, continuous, and reliable measurements without disturbing the process. Photometric techniques, depending upon light absorbance, are applicable in many industrial process units since the experimental procedure is simple and safe. Further, the use of lasers and fiber optics reduces the space required for the work and increases the accuracy and the consistency of the data.

Fiber-optic technology has played an important role in the development of photometric techniques for local concentration measurements. For example, Bell (1972) used a fiber-optic technique to measure local variations in fluoresced tracer concentration in commercial-scale distillation columns, and Chiba and Kobayachi (1970) and Sit and Grace (1978) used fiber-optic probes to analyze the concentration of various species in bubbles of fluidized beds.

Local concentration measuring techniques in one-phase systems are well established. For a two-phase liquid–liquid system, one has to combine a phase-separation technique with the concentration measuring technique. Zhang et al. (1985) used a simple phase-separation device for the coalescence and separation of the dispersed phase, but they measured the concentration externally, using a spectrophotometer. Niebuhr and Vogelpohl (1980) employed a photometric technique using a fluoresced tracer activated by ultraviolet irradiation. The emitted light was detected by two photomultipliers externally positioned, providing information about a cross-section layer. The disadvantage of their approach is the need for light-transparent equipment. The technique also requires specific tracers, which, in general, are surface-active, causing changes in the system's behavior.

Another approach, by Bensalem (1985), involves development of a photometric probe to measure concentration in the drops that coalesce on the surface of an inverted teflon tunnel. However, our laboratory effort to adapt this separation technique to highly agitated dispersions was unsuccessful. It appears that we still need to identify reliable techniques for in situ concentration measurements of a solute or tracer in unidimensional flow or highly turbulent flow liquid–liquid dispersions. The laser photometric technique described in this work fills this need.

## Experimental

### *Concentration measuring technique*

The laser photometric technique consists of a laser light source, a fiber-optic probe, an electronic device, and the data acquisition system connected with a microcomputer, Figure 1. The laser is a 5mW He-Ne tube emitting light at wavelength 632.6 nm. In order to provide local concentration measurements with minimum process disturbance, the fiber-optic probe must be relatively small. Figure 2 shows the probe, consisting in principle of a two-loop fiber-optic setup. Very thin optical glass fibers (50 $\mu$m dia.) are coated with a layer of glass of lower refraction index to increase reflection inside the light guides, and many such fibers are assembled to create a fiber-optic bundle of 0.42 mm dia. and 500 mm length. Both ends of the fiber-optic bundle are glued, ground, and polished, while half of its length is introduced into a flexible metal tube.

Two of these fiber-optic bundles are assembled together in a rigid stainless-steel tube of 4 mm outer diameter (OD) and 250 mm length, as shown in Figure 1. At the tip of this tube, the two bundles are separately adjusted in two small stainless-steel tubes

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Correspondence concerning this paper should be addressed to L. L. Tavlarides.
H. Schmidt and E. Eggert are presently with Kernforschungszentrum Karlsruhe.

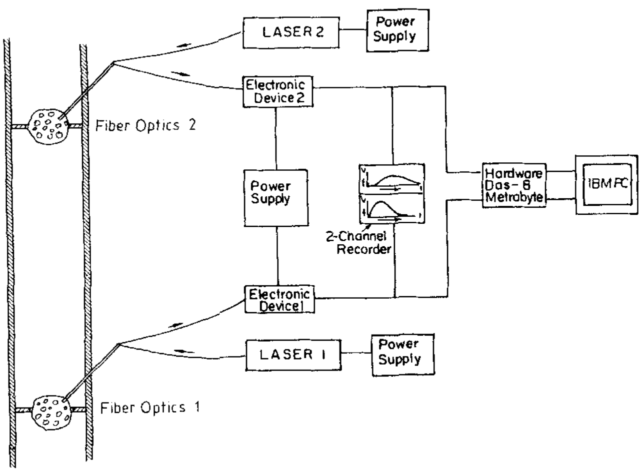


**Figure 1. Laser photometric technique.**

of 0.7 mm OD, building a forked device of 10 mm length. At the tip of each prong, a rectangular isosceles prism is located in such a way that one of the perpendicular faces is sitting on the fiber optics while the free perpendicular faces of both prisms are facing each other. The hypotenuse side is covered with a silver foil to reflect the light. Both prisms are affixed with optical glue.

By using the one fiber-optic bundle as the sender and the other as the receiver, the fiber-optic probe can be used as a photometric device with a light-traveling distance of around 2 mm through the liquid volume. Thus, the part of the probe taking measurements is considerably smaller than the dimensions of the contactor. The small-size probe, however, has the disadvantage of being very sensitive to optical disturbances and therefore, the use of lasers, which are very consistent light sources, is required.

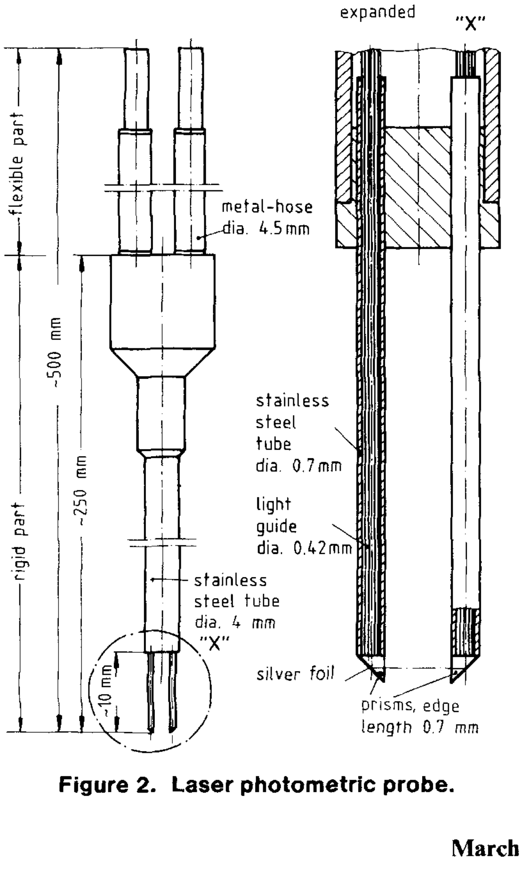


**Figure 2. Laser photometric probe.**

A photodetector (photodiode) located in the electronic device, Figure 1, is chosen according to the light's wavelength in order to optimally convert the input light into an electrical signal. The circuit of the electronic device is divided into four operations:

- The light input with the photo-detector.
- The first amplification step with an amplifier operating in the current/voltage mode.
- The second amplification step with an amplifier operating in the voltage amplification mode.
- The signal output process (see Tsouris, 1988).

The electric signal from the circuit is either recorded by an analog recorder or digitized by an A/D converter and sampled by a data acquisition system.

The intensity of the light, $I$, at the exit of the probe is related to the initial intensity, $I_0$ (at zero concentration), the concentration of the investigated species in the liquid medium, $c$, and the traveling distance through the medium, $x$, by the Lambert-Beer law:

$$I = I_0 \exp(-\epsilon c x) \qquad (1)$$

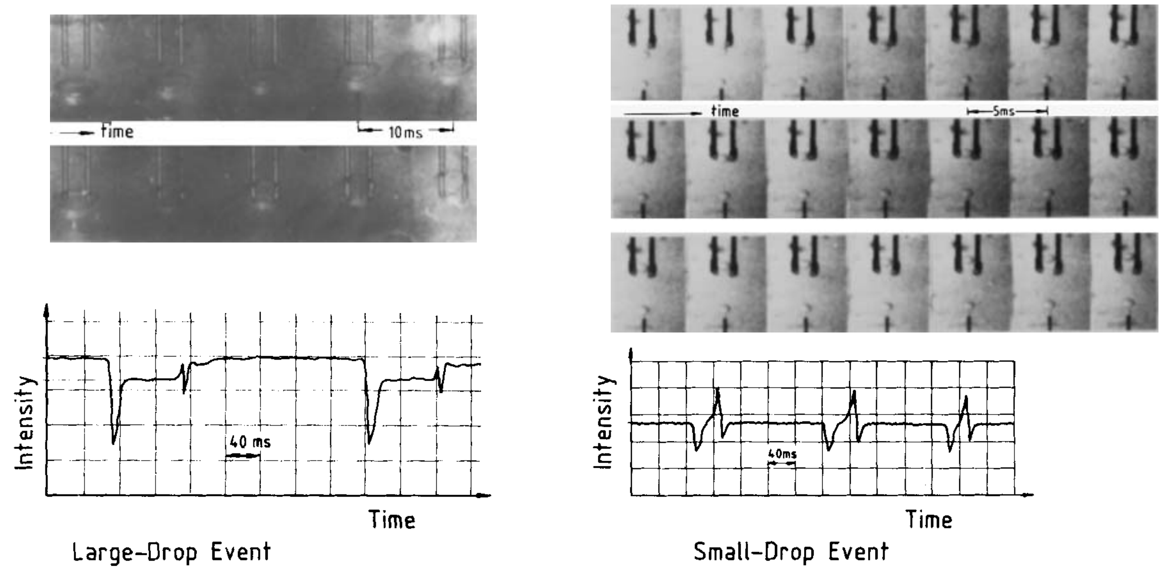
where $\epsilon$ is the molar absorption coefficient, characteristic for each species. Measurements of different concentrations of oil-blue in toluene show that the Lambert-Beer law holds and that the light conversion into voltage and the amplification at the electronic device are carried out linearly.

### *Test of the technique in liquid dispersions*

The laser photometric technique was tested to determine whether it would successfully obtain concentration information on single drops, by recording the signal at different drop sizes and simultaneously taking pictures of the drops, Figure 3. When a large drop approaches the measuring device, the drop interface is deformed by the prisms' edges. As a result, the light beam is reflected at the deformed drop's interface and, consequently, over a short period of time, the light intensity drops significantly. When the prisms are immersed in the drop, absorption causes the undisturbed photometric measurement to be recorded at a lower intensity than for the other phase. A second deformity of the drop interface occurs when the drop moves upward, leaving the two prisms and reentering the continuous phase.

Small drops, on the other hand, which may travel through the measuring zone without wetting the prisms, cannot provide reliable measurements. These disparities mean that this laser photometric technique cannot provide information about individual drops in a polydispersed system. Also, in order that measurements be reliable, one must eliminate the interference of one phase with the other by separating the two phases before taking the measurements. For this purpose, coalescing devices were developed to be applied in different flow regimes. These devices, also called coalescence bodies, collect selectively and continuously samples of one of the two phases.

The accuracy of the technique was tested in the following way. Two sets of known concentration samples were generated: the first for the calibration of the probe according to the Lambert-Beer law and the second for the test of the measurements. Three experiments were conducted, each one at different zero-concentration voltage. The results proved the technique to be satisfactory, and the maximum deviation, around 8% of the actual value, occurred at the lowest concentration.



Large-Drop Event

Small-Drop Event

**Figure 3. Functional test on drop size; signal analysis.**

## Application of the techniques

The laser photometric technique was applied in two types of liquid–liquid contactors with different hydrodynamic characteristics in order to determine the axial dispersion coefficient in pulsed, perforated plate, extraction columns of different geometries (Schmidt; 1983, 1986) and in a multistaged stirred column (Tsouris et al., 1987). Different phase separation devices, according to the hydrodynamic behavior of each contactor, were developed for these applications.

### *Pulsed, perforated plate, column contactor*

The sinusoidal rise and fall of the liquid dispersions, caused by pulsation, in these kinds of contactors act on the dispersed phase in a manner of forced convective-stagnation flow. During upstroke pulsation, the drops move in the vertical direction, while during downstroke pulsation, they stagnate. This flow suggests the configuration of the coalescing device presented in Figure 4a. A needle pointing in the direction of the drop movement collects samples of the drops during upstroke pulsation. The coalescence body is constructed from material wetted by the dispersed phase only. It has dimensions of 28 mm length and 4 mm OD and is adjusted around the probe, so that the two prisms are located close to the needle. The coalesced dispersed phase passes through the coalescence body during upstroke pulsation and returns to the dispersion via bores after the measurements are taken. The sampling cross-section area of the coalescence body is 12 mm$^2$ and the inner liquid volume about 80 mm$^3$. Using the equipment setup shown in Figure 1, measurements were taken with a color tracer stimulus injection at two positions along the column. In Figure 4b, signal $\alpha$ is coming from probe 1, and signal $\omega$ from probe 2. The distance between the two probes is 2.25 m. The high frequencies observed in both signals are due to large drops with large differences in tracer concentration (Schmidt, 1983).

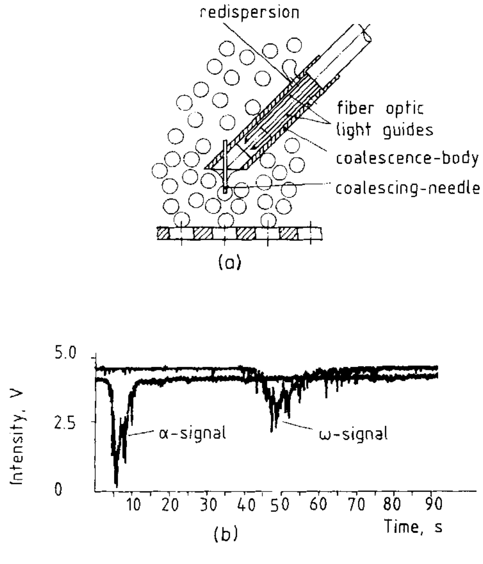


**Figure 4a. Coalescence device for pulsed columns.**
**b. Tracer experiment in pulsed column, 100 mm ID, 4 m length.**
system: TBP $(d)$–$HNO_3(c)$

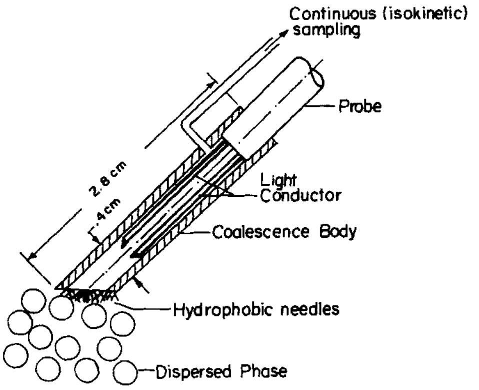


**Figure 5. Coalescence device for stirred columns.**

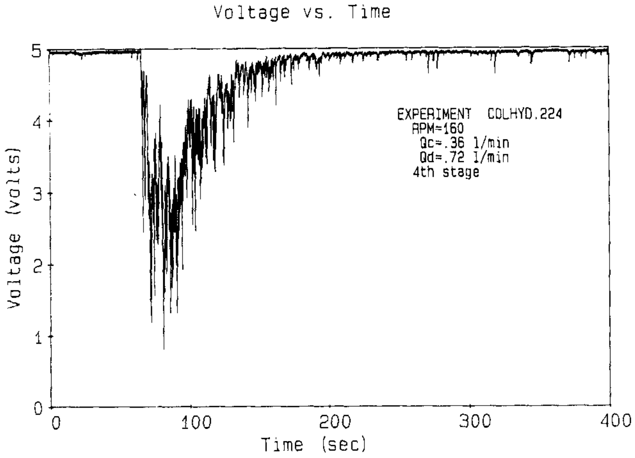


**Figure 6. Tracer experiment in stirred column, 127 mm ID.**
system: toluene(*d*)–water(*c*)

### *Multistage stirred column contactor*

Different flow patterns are obtained in highly agitated systems like the multistaged stirred contactor. The movement of the drops is not axial but has a random motion superimposed upon the local mean flow. The high energy input results in smaller drop sizes than in pulsed columns.

Accordingly, a different design of the coalescence body is employed, as shown in Figure 5. The needle was replaced by a cluster of smaller needles to create a brush at the entrance of the coalescence body. Also, since the flow cannot cause the redispersion of the coalesced droplets, a continuous external sampling of this material from the internal of the coalescence body was employed.

A voltage signal from probe 1, located at the inlet of the fourth stage of a seven-staged contactor, is shown in Figure 6. The signal again has high frequencies because large drops with large differences in tracer concentration are present, as in the pulsed column. When experiments are conducted at 200 rpm, the drop size distribution shifts to smaller diameters, and the high frequencies in the voltage signal disappear (Tsouris et al., 1987).

By using the data acquisition system, the voltage signal may be simultaneously converted into concentration by the Lambert-Beer law, providing the online concentration measurements valuable for controlling the process.

The results of this communication show that the laser photometric probe can provide reliable concentration measurements in liquid–liquid dispersions, which permit characterization of the hydrodynamics and quantification of the performance of equipment processing such systems. Further, this technique can be implemented as an invaluable instrument for controlling extraction processes.

## Acknowledgment

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