PULSED LASER PHOTOTHERMAL SPECTROSCOPY

OF LIQUIDS AND SOLIDS

Progress Report

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Pulsed Laser Photothermal Spectroscopy of Liquids and Solids

Progress Report Abstract

Published papers have compared the advantages of piezoelectric and optical detection of photoacoustic signals in sample liquids and have demonstrated the superior capabilities of thermal lens measurements on time scales as short as a few hundred nanoseconds. Computer programs have been developed to compare theoretical and experimental photoacoustic pulse shapes in liquids with a view to determining triplet state quantum yields and radiationless decay lifetimes with greater accuracy. Non-radiative quantum yields of 1.0, 0.9 and 0.5 have been measured for nonaqueous solutions of Crystal Violet, Evan's Blue and Nile Blue A respectively. Pulsed photothermal radiometry experiments have yielded thermal diffusivity data for a room temperature molten salt system. Microphonic photoacoustic signals obtained by illuminating solid samples with synchrotron X-rays have been enhanced with volatile liquids, and the results have been compared with theory.

Two major objectives were set for the April 1, 1987 to March 31, 1990 grant period:

1. A quantitative comparison was to be made between experimental pulsed laser photothermal spectroscopy curves obtained with sample liquids and curve shapes predicted by theory. Such comparisons would lead to more accurate determinations of quantum yields and lifetimes for radiationless decay processes in liquids.

2. In a parallel study, a pulsed laser system would be used to explore the capabilities of pulsed photothermal radiometry (PPTR) for revealing the properties of opaque solids.

In two early papers[1a,b] we reported a useful combination of the thermal lens and an optical detection photoacoustic technique and a comparison of the advantages of piezoelectric detection and optical detection of photoacoustic effects in liquids. Neither study directly addressed the first of the two objectives described above. Recently, Dr. S. J. Komorowski has developed computer programs in PASCAL for an IBM PC that permit a calculation of the shape of photoacoustic curves in liquids from theoretical expressions.[2,3] Before returning to Poland (from Utah) in December, 1989, he will determine whether the fitting of these computer generated curves to experimental data significantly facilitates the determination of radiationless decay lifetimes in liquids.

Stefan J. Isak, a graduate student, has shown[4] that the thermal lens method is more effective than photothermal beam deflection and piezoelectric detection of acoustic waves for determining submicrosecond radiationless decay lifetimes in liquids. Isak has used a combination of all three photothermal methods to investigate radiationless decay in a group of photothermal sensitizing dyes.[5] These are dyes that lose practically all of
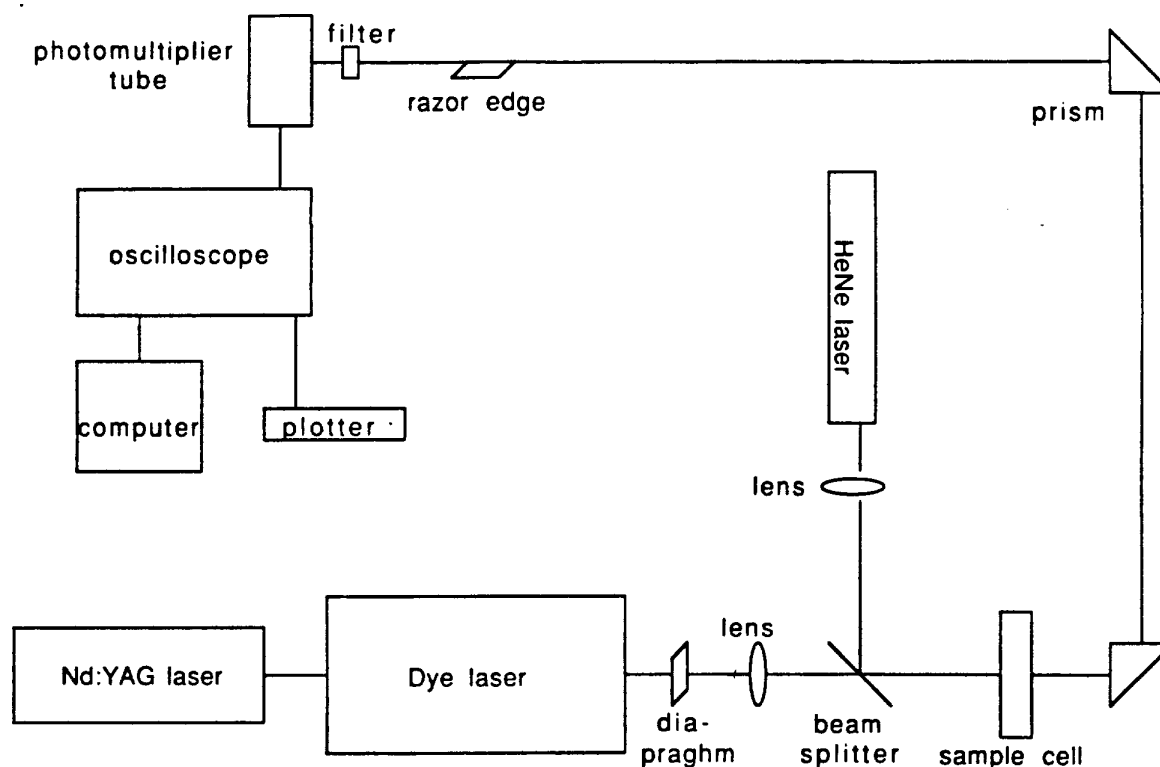
Fig. 1. Apparatus for making thermal lens, beam deflection and photoacoustic measurements in liquid samples.

their electronic excitation energy through radiationless decay rather than by fluorescence. They have been used to selectively kill cancer cells in new laser surgical procedures ("photodynamic therapy") being developed in the University of Utah College of Medicine. Among the dyes being considered are several triphenylmethane dyes such as Crystal Violet and diazo dyes such as Evan's Blue, that absorb red light (in our case at 610 nm.). Red light is transmitted by undyed (nonmalignant) biological tissue.

The experimental set-up used in determining non-radiative quantum yields, ϕ_{nr}, and triplet state lifetimes, τ_T, is shown in Fig. 1. The design allows for several types of photothermal measurements to be made such as thermal lens (TL), photothermal beam deflection (PBD), and photoacoustic (PA) measurements, where the respective signals are all detected optically.

There are several problems with the chemical systems under study that must be resolved before the results can be confidently interpreted. For example, most organic dyes have broad absorption spectra in the visible region. The absorption bands for the dyes we are studying extend from the green into the infrared making most cw laser sources, such as a HeNe or Ar^+ laser, somewhat unsatisfactory probe lasers. Thus the observed PBD signal varies with the power of the HeNe beam, and it is necessary to work at very low signal levels at which essentially no absorption of the HeNe laser probe beam is occurring. It is also necessary to work in the linear absorbing region of the dyes so that we are restricted to the use of very dilute solutions. Fortunately, these dyes have very large extinction coefficients, on the order of tens of thousands. The high extinction coefficients as well as the short (picosecond) excited state lifetimes of some of the dyes combined with the relatively long (nanosecond) pump laser pulse duration are an invitation for non-linear absorption effects. Thus, the behavior of the dyes with respect to the pump beam energy must also be characterized. A plot of PBD signal amplitude versus pump beam energy would ideally yield a straight line through the origin. The shape of the experimental curves in Fig. 2 indicates signal saturation.[6,7] Log/log plots of such data should yield curves whose slope at any one point will be equal to the apparent number of photons being absorbed by the system.[7] For most, but not all, of the dyes we are studying we have obtained an apparent one photon absorption at the lower pump energies. More experimental data will be needed at very low pump energies to avoid extrapolation errors. We have obtained a consistent non-radiative quantum yield of 1.0 for Crystal Violet and reasonable values for non-radiative quantum yields of about 0.9 for Evan's Blue and about 0.5 for Nile Blue A in the solvents studied. A manuscript[8]
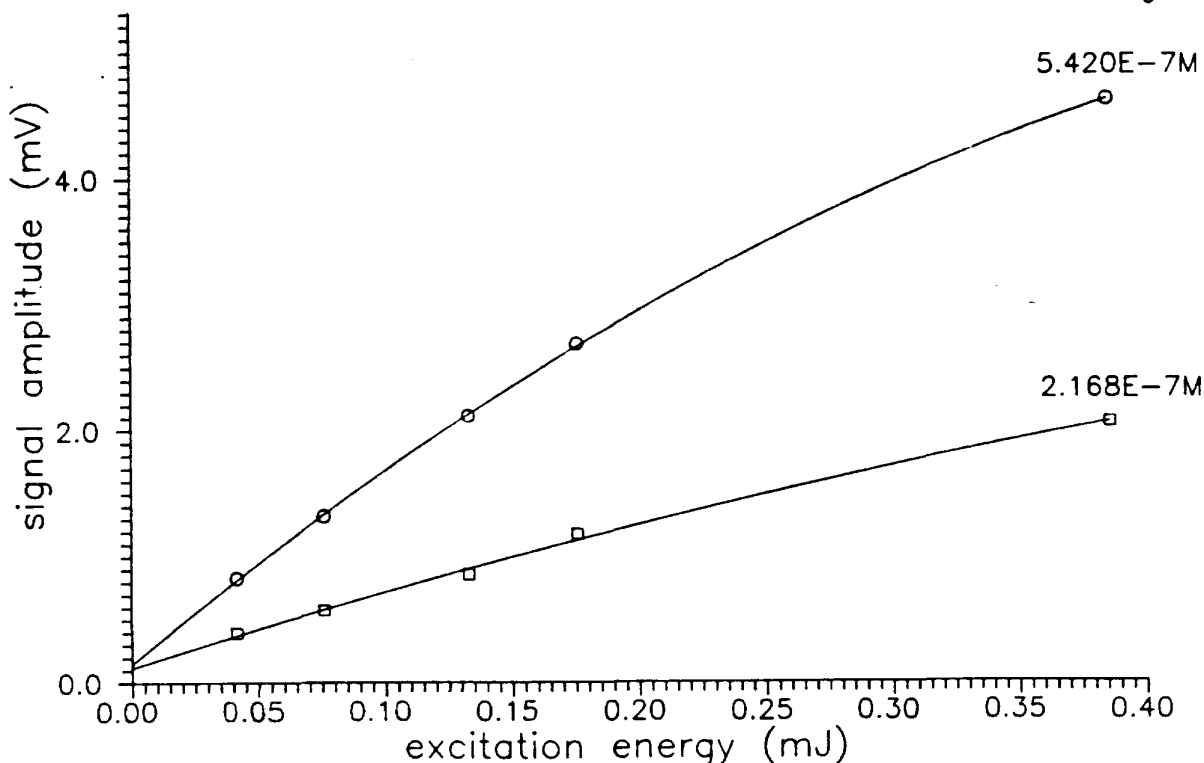
Fig. 2. Pump beam energy versus signal amplitude for Evan's Blue in methanol at two different molar concentrations.

reporting non-radiative quantum yields and triplet state lifetimes for a group of these dyes will be ready for publication in early 1990. Interest in this work will arise from the fact that this type of data is less commonly available for non-fluorescing dyes than it is for fluorescing dyes.

Daryl P. Cobranchi, a graduate student, and Dr. Nelia F. Leite, a postdoctoral coworker from Brazil, carried out a series of pulsed photothermal radiometry (PPTR)[9] experiments that yielded the thermal properties of both solid and liquid samples. A pulsed visible laser source of ~ 10 ns duration was used to excite the samples. In PPTR, this induces a temperature increase in a solid sample. The surface temperature variation is monitored by infrared detection. A schematic of the PPTR experiment is shown in Fig. 3. The signal S(t), for a semi-infinite (i.e. thermally thick) solid is given by[10,11]
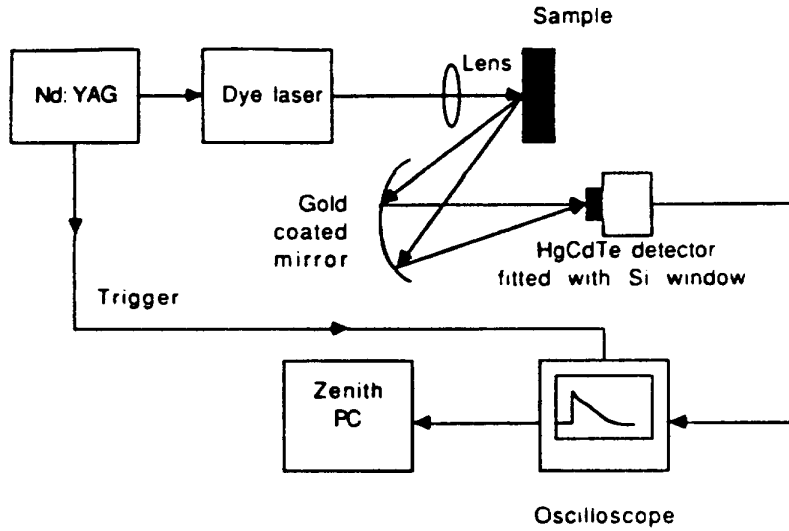
Fig. 3. Schematic of the experimental apparatus for pulsed photothermal radiometry experiments (PPTR).

$$S(t) = \frac{C\alpha\beta}{\beta^2-\alpha^2} [\beta \exp(\alpha^2 Dt) \, \mathrm{erfc}(\alpha^2 Dt)^{1/2} - \alpha \exp(\beta^2 Dt) \, \mathrm{erfc}(\beta^2 Dt)^{1/2}]$$

(1)

where α and β are the bulk and surface absorption coefficients, D is the thermal diffusion coefficient of the sample, erfc is the complementary error function, and C is a constant. In the special case where $\alpha \gg \beta$ equation (1) reduces to

$$S(t) = C' \exp(\beta^2 Dt) \, \mathrm{erfc}(\beta^2 Dt)^{1/2}$$

(2)

It should be noted that equation (1) is symmetric with respect to α and β, i.e. the value of $S(t)$ does not vary if α and β are interchanged. This creates an ambiguity as to which of the coefficients pertains to each property.[10,11] The uncertainty can be resolved by modifying the surface because the infrared absorption should not change with only a surface modification.

We have used the PPTR technique to measure the thermal and optical properties of several solids such as the Y-Ba-Cu-O high temperature super-conductor and a lead brick.[12] However, the application of PPTR to liquids has turned out to be more interesting and has culminated in a paper accepted for publication.[13] In this paper (preprint attached) we have reported thermal diffusivity data for the 1-methyl-3-ethylimidazolium tetrachloroaluminate [$AlCl_3$/MEIC] room temperature molten salt. This liquid has been touted as an electrolyte for batteries.

For most aqueous solutions the PPTR signal is described quite satisfactorily by equation (2). Thus, for example, the thermal diffusivity D of water can be deduced[13] from PPTR measurements on an optically opaque aqueous solution of $KMnO_4$ and then the optical absorption coefficient β can be deduced for an aqueous crystal violet solution from the PPTR curve shown in Fig. 4 using the diffusivity D of water.

Other accomplishments during the grant period that were not forseen by the original research proposal include the following: A review chapter[14] has been prepared on photoacoustic instrumentation; a study[15] of microphonic photoacoustic signal enhancement by volatile liquids is nearing completion; and finally, ^{7}Li-nmr spectroscopy is being used[16] to determine complex ion stability constants in the $AlCl_3$/MEIC molten salt solvent.

Microphonic photoacoustic enhancement by volatile liquids was first reported by workers in India a few years ago.[17] We became interested in the technique in February, 1988 when we made some X-ray photoacoustic measurements at a Japanese synchrotron in collaboration with Prof. Tsutomu Masujima. We have since conducted additional X-ray photoacoustic experiments at the Brookhaven synchrotron (in April and September, 1989).
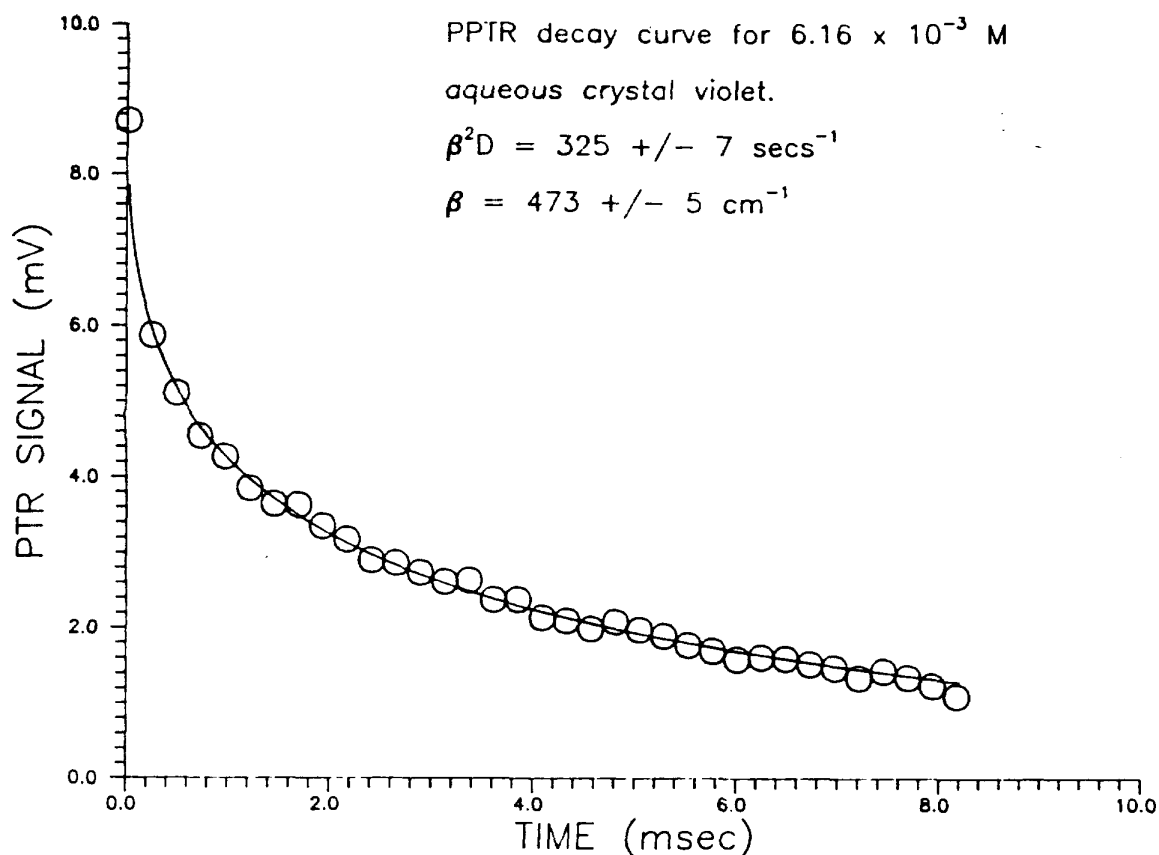
Fig. 4. PPTR curve leading to the optical absorption coefficient β of Crystal Violet in aqueous solution.

The present focus of our effort is the extension of a theory[18] of photoacoustic signal enhancement by volatile liquids from the visible wavelength region for which it was derived to a quantitative accounting for our X-ray photoacoustic data.

Our [7]Li-nmr study is presently focused on the identification of the most appropriate method of calculating complex ion stability constants from [7]Li-nmr data.

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Personnel

Personnel paid from this grant since its inception on April 1, 1987 include: E. M. Eyring, principal investigator; S. J. Komorowski, postdoctoral research associate; N. F. Leite, postdoctoral research associate; S. J. Isak, graduate student; B. A. Garland, graduate student; D. Spencer, undergraduate research assistant.

Publications Acknowledging Support of this Grant Since April 1, 1987:

1. S. J. Komorowski and E. M. Eyring, "Pulse Shapes of Nanosecond Photoacoustic Signals in Liquids Detected by Piezoelectric Foil," J. Appl. Phys. $\underline{62}$, 3066-3069 (1987).

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Posters and Talks Presenting Research Supported by this Grant Since April 1, 1987:

1. S. J. Komorowski, "Comparison of the Sensitivity and Time Resolution of PVF_2 Foil and Beam Deflection Detection Methods for Pulsed Photoacoustic Signals in Liquids," (Contributed Poster), 5th International Topical Meeting on Photoacoustic and Photothermal Phenomena, Heidelberg, W. Germany, July 30, 1987.

2. E. M. Eyring, "Photoacoustic Spectroscopy," (Contributed Talk), CHEMRAWN IV, Keystone, Colorado, October 5-9, 1987.

3. E. M. Eyring, "Pulsed Photoacoustic Spectroscopy in Liquids," (Contributed Talk), DOE Workshop on Advanced Laser Technology for Chemical Measurements, Gaithersburg, MD, November 4-6, 1987.

4. E. M. Eyring, "Fourier Transform Photoacoustic Spectroscopy," (Invited Lecture), Department of Industrial Chemistry, University of Tokyo, Tokyo, Japan, February 24, 1988.

5. E. M. Eyring, "FT-IR PAS," (Invited Lecture), JASCO, Tokyo, Japan, March 7, 1988.

6. E. M. Eyring, "Photoacoustic Detection of Infrared Spectra," (Invited Symposium Talk), 9th ACS Rocky Mountain Regional Meeting, Las Vegas, Nevada, March 29, 1988.

7. E. M. Eyring, "Laser Photothermal Spectroscopic Rate Measurements in Liquids," (Invited Seminar), Department of Chemistry, Tufts University, Medford, Mass., November 15, 1988.

8. E. M. Eyring, "Laser Optoacoustic Techniques," (Invited Seminar), Department of Chemistry, Baylor University, Waco, Texas, February 10, 1989.

9. E. M. Eyring, "Comparison of Photoacoustic and Thermal Lens Kinetic Measurements," (Invited Symposium Talk), American Physical Society Meeting, St. Louis, Missouri, March 22, 1989.

10. E. M. Eyring, "Pulsed Laser Photothermal Radiometry of Solids," (Contributed Talk), DOE Workshop on Advanced Laser Technology for Chemical Measurement, Argonne, IL, May 9-11, 1989.

11. D. P. Cobranchi, "Pulsed Laser Photothermal Radiometry of Some Strongly Absorbing Liquids and Solids," (Contributed Talk), Annual Meeting of Utah Academy of Sciences, Arts and Letters, Utah State University, Logan, UT, May 19, 1989.

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Pulsed Laser Photothermal Radiometry and Photothermal Beam Deflection Spectroscopy: Determination of Thermal Diffusivites of Liquids

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Abstract. A comparison of the relative merits of both pulsed photothermal radiometry (PPTR) and photothermal beam deflection spectroscopy (PBDS) for the determination of thermal diffusivities of liquids is presented. Also reported are data for the thermal diffusivity of the room temperature molten salt system 1-methyl-3-ethylimidazolium tetrachloroaluminate at several temperatures.

Introduction

The thermal diffusivity, D, plays a critical role in the various photothermal spectroscopies. Thus, the photothermal effect has been used to probe the thermal properties of many condensed phase materials including thin polymer films[1], high T_c superconductors[2], and stainless steel sheets at elevated temperatures[3].

In most aqueous solutions the PPTR signal as a function of time is given by[4]

$$S(t) = C \exp(\beta^2 Dt) \; \text{erfc}(\beta^2 Dt)^{1/2} \tag{1}$$

where β is the excitation wavelength absorption coefficient and erfc is the complementary error function.

For photothermal beam deflection spectroscopy, the 1/e signal decay time, τ, is given by[5]

$$\tau = \frac{\omega_o^2}{4D}. \tag{2}$$

Here ω_o is the radius of the focused laser spot on the sample. The present study employs both the above techniques to determine the thermal diffusivity in several aqueous solutions and in the room temperature molten salt system 1-methyl-3-ethylimidazolium tetrachloroaluminate.

Experimental

1-methyl-3-ethylimidazolium chloride (MEIC) was prepared following the method of Wilkes et al.[6]. Aluminum chloride (anhydrous, Fluka) was sublimed under vacuum.

PPTR: The output from a frequency doubled Nd:YAG laser illuminates the sample. The emitted infrared radiation is collected with a gold coated
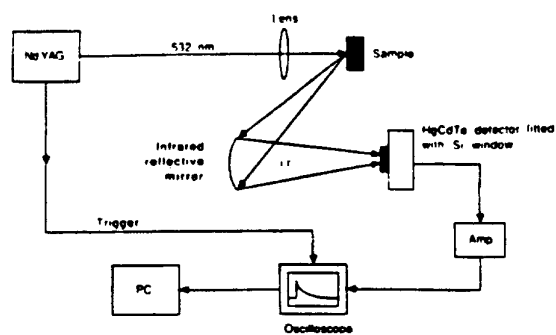
Figure 1. Schematic diagram for   Figure 2. Schematic diagram for
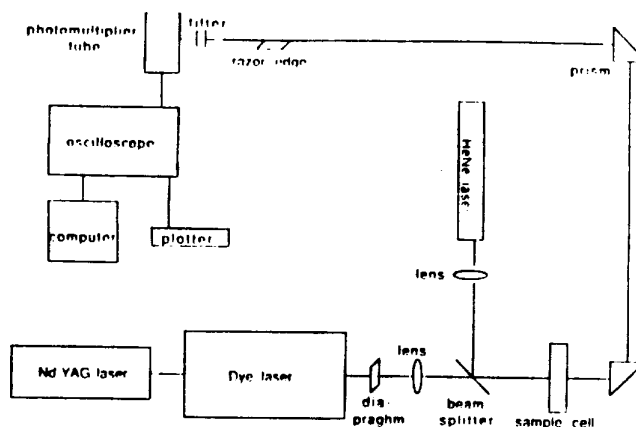   PPTR experiment.   PBDS experiment.

mirror and directed into the liquid nitrogen cooled HgCdTe detector. The
signal is amplified and sent to a digital oscilloscope. A schematic of the
apparatus is shown in Fig. 1.
     PBDS: The Nd:YAG laser pumps a dye laser. The output from the dye
laser is focused co-linearly with the output from a HeNe probe laser. The
fluctuation in position of the HeNe beam is monitored by a fast
photomultiplier tube. This experiment is shown schematically in Fig. 2.

Results and Discussion

     PPTR: A 0.100 molar aqueous solution of $KMnO_4$ was prepared to test the
feasibility of measuring the thermal diffusivity of a liquid. For this
solution the molar absorptivity at 532 nm is 1.94×10^6 cm^2 $mole^{-1}$. Using
this value, the calculated D is $(1.40 \pm 0.02) \times 10^{-3}$ cm^2 sec^{-1} in good
agreement with the accepted value[7] of 1.45×10^{-3} cm^2 sec^{-1}.
     PBDS: A sample of 50 mole % $AlCl_3$/MEIC was placed in a constant
temperature cuvette. After allowing the solvent temperature to equilibrate
for at least 30 minutes, several exponential decay curves were generated by
signal averaging at least 100 laser pulses. After all the data were
gathered, the molten salt was removed from the cuvette without disturbing
its position. Several dilute solutions of colored molecules in various
solvents were then introduced into the cell to determine ω_0 from the known
thermal diffusivities. The spot size ω_0 measured was (57.2 ± 0.4) µm. The
calculated thermal diffusivities are presented in Table 1.
     Both PPTR and PBDS generate accurate values for the thermal diffusivity
of liquids. Each technique has its advantages and disadvantages. The main
advantage of PPTR is the ease of set-up and operation of the experiment.
Alignment of the laser is not critical, and the entire experiment can be set
up in a few minutes. The primary disadvantage is the need to know the
absorption coefficient in the very opaque sample solutions. This drawback
forced us to carry out the PBDS experiment.

Table 1. PBDS thermal diffusivities of 50/50 $AlCl_3$/MEIC room temperature molten salt. Uncertainties are the standard deviations. Calculations employed a value of (57.2 ± 0.4) μm for ω_0.

Temp., °C	τ, msecs	D, cm^2sec^{-1}
10	7.86 ± 0.12	$(1.04 \pm 0.03) \times 10^{-3}$
20	8.24 ± 0.10	$(9.93 \pm 0.04) \times 10^{-4}$
30	9.03 ± 0.24	$(9.06 \pm 0.13) \times 10^{-4}$
40	9.03 ± 0.23	$(9.06 \pm 0.12) \times 10^{-4}$
50	9.02 ± 0.33	$(9.08 \pm 0.22) \times 10^{-4}$
60	8.64 ± 0.41	$(9.47 \pm 0.33) \times 10^{-4}$

PBDS complements PPTR in several ways. Beam deflection works in solutions which are not optically dense and does not require knowledge of the absorbance. This property is important in mixed solvent or molten salt systems where mole fractions are not fixed. The major drawbacks to PBDS are the critical alignment of two lasers and the need for many optical components.

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