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A Study the Techniques of Spectroscopic and effects of Solvents on Rhodamine

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***Abstract-** The purpose of this study is to examine the effect of environmental conditions on the laser dyes in various liquid & solid media. Due to the different fluorescence spectrum of the dye, it is also well known that a dye laser's tunable range is affected by solvent. Laser dye & solvent interact in some way, as evidenced by this finding. Furthermore, rhodamine dyes are affected by different environments. As previously stated, the fluorescence quantum yield of the Rh6G dye in water is reduced, that reduces the output power of Rh6G dye laser. These samples require the dyes to be dissolved in different solvents & doped in different solid hosts & polymers in order to be prepared. Dispersion infrared spectrophotometer, Fourier transform infrared spectrophotometer, Raman spectrophotometer, UV-Vis spectrophotometer, & fluorescence spectrophotometer are some of the spectrometers explored.*

***Keywords-** Lasers, Rhodamine Dyes, Spectroscopy, Chemical Properties, DFT*

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INTRODUCTION

Lasers are used in a wide range of applications today. Microsurgery, communication, and many spectroscopic applications, like Raman spectroscopy for tracking photochemical reactions & ultrafast reaction kinetics, isotope separations, & trace analysis, all make use of them. For example, sodium atoms at concentrations as low as 0.003ng cm–3 can be detected using these techniques. Among the most versatile and successful laser sources known today, dye lasers have made a significant contribution to fundamental physics, chemical chemistry, biology or other fields. In 1966, a laser dye was first reported. In order to generate the necessary emission, traditional laser technology relies on a variety of inorganic materials. Inorganic lasers that emit in the ultraviolet, visible, or infrared ranges have been developed in a variety of configurations. Inorganic lasers are inexpensive and durable, but they come with some drawbacks. Dye lasers, on the other hand, can be tuned to cover the entire visible and near-infrared spectrum, whereas inorganic lasers are limited to a few specific wavelengths & bands. Dye lasers have made a significant contribution to the advancement of laser technology.

SAMPLE PREPARATIONS

Dyes in Solvents

The Exciton, USA laser dyes (rhodamines & coumarins), which are commercially available, were used directly. Depending on the application, dye solutions are prepared in a concentration range of 10-2 to 10-3 M in various solvents (methanol, dirnethylformamide, pyridine, chloroform, benzene, dichloromethane, & acetonitrile). Only spectroscopic/HPLC grade solvents were used in the work reported in the thesis, and no further purification was required unless specifically stated. Deionized & triple-distilled water was used in the process of making the product.

Dye Dispersed in Solid Host

The polymer precursor was mixed with a known amount of dye dissolved in a suitable solvent & permitted to polymerize by adding initiator in order to disperse the dye in polymers like polyethylene glycol (PE), polymethylmetacrylate (PMMA), etc. Once the solution was ready, it was poured into the desired moulds. The solvent in the polymer evaporated over time, leaving behind a solid polymer of the desired shape, which contained dye. Sol-gel glass was also used to study the spectroscopic properties of the dyes. The procedure described in the literature [G. Hungerford 1991] was used to dope the dyes in the sol-gel glass.

Sample Preparation for Infrared Spectroscopy

They are sandwiched between two plates of a high purity salt to analyse liquid samples (commonly sodium chloride, potassium bromide, or calcium fluoride) [M.H. Laurence 1990]. To the infrared, the plates are completely transparent, and thus do not alter the spectra in any way. For salt plates, anhydrous sample & washing solutions are required because some salt plates are highly water soluble. There are two methods for making solid samples: To begin, use a pestle and a marble or agate mortar and pestle to crush the sample with a mulling agent (typically nujol). This method involves applying a thin layer of mull to salt plates, while the second involves grinding the sample finely with potassium bromide. A mechanical die press is used to press the powder mixture into a translucent pellet, which the spectrometer beam can then pass through. Different sample preparation methods will yield slightly different spectra because of the physical differences in the samples.. The cast film method is used to prepare polymeric compounds. In this method, the sample is first dissolved in a non-hygroscopic solvent, and then the resulting solution is analysed. On the KBr or NaCl cell surface, a drop of this solution is deposited. After the solution has been evaporated to a dry state, the cell film can be examined directly for any signs of contamination. Make sure the film isn't too thick or you won't be able to see through it. Qualitative data can be analysed using this method. Pellet & solution spectra were obtained in the current study.

SPECTROSCOPIC TECHNIQUES

The spectroscopic studies of laser dyes are the focus of the study mentioned. The various methods used in this study have been laid out in the following paragraphs.

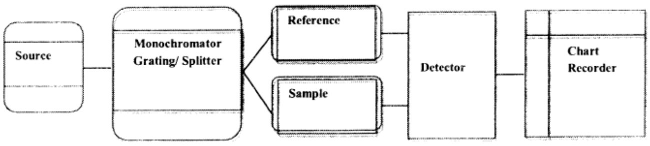


Figure 1: Schematic diagram of dispersive infrared spectrophotometer.

Infrared Spectroscopy

A beam of IR radiation is used to capture the IR spectrum of a sample. To determine the amount of energy absorbed at each wavelength, the transmitted light is analysed. A monochromatic beam, that changes wavelength over time, or a Fourier transform instrument could be used to measure all wavelengths simultaneously. A transmittance or absorbance spectrum could be generated from this, which shows which IR wavelengths the sample absorbed. Analyzing the sample's absorption characteristics reveals information about its molecular structure. Dispersive IR spectrophotometers were used to record IR absorption spectra before Fourier Transform Infra-red (FTIR) spectrophotometers were developed. Figure 1 depicts the schematic diagram of an IR spectrometer with a dispersive double beam. A nichrome wire or rod of silicon carbide is used to generate a range of frequencies in the infrared spectrum. A monochromator is used to separate this frequency range into individual frequencies. The resulting beam is split in half, yielding two new beams. In this case, one of the beams is used as a reference, while the other is passed through the sample itself.

Detection of the intensity difference & subsequent transmission of a proportional signal to the recorder are accomplished by having the two beams meet at the detector. To determine the transmission rate against frequency, a plot known as a wave number plot is used instead (cm-l), Numerous drawbacks exist for dispersive instruments. Slow scanning was the main drawback of dispersive instruments, which led to FTIR spectroscopy's development. As a result, a technique for simultaneously measuring all IR frequencies was required. The interferometer was developed as a device to provide a solution. All of the infrared frequencies are "encoded" into the signal produced by the interferometer. The instrument is able to measure the signal in a short period of time. As a result, scanning time per sample is cut down from several minutes to just a few seconds. When an infrared beam enters the interferometer, a beamsplitter divides it into two separate optical beams. A fiat fixed mirror reflects one light beam. Using a mechanism that moves the mirror a few millimetres away from the beam splitter, the other beam reflects off a flat mirror. The beamsplitter combines the two beams after they have reflected off of their respective mirrors. The path taken by one beam is predetermined in length, while the path taken by the other beam is dynamic due to the movement of the mirror. Both beams "interfere" to produce the signal that comes out of the interferometer. An interferogram is the name given to the resulting signal. In an interferogram, every data point (a function of the mirror position) contains information about every infrared frequency that is coming from the source. This is a unique property of the interferogram. As a result, a measurement of an interferogram requires the simultaneous measurement of all frequencies.

Interferometer measurements are extremely fast because of this. Since a spectrum is a plot of intensity at each individual frequency, the interferogram signal cannot be directly interpreted. This necessitates a means of "decoding" each frequency individually. The Fourier transformation is a well-known mathematical technique for accomplishing this. Afterward, the user is presented with the desired spectral information for analysis by the computer. As a result of using FTIR, the following data can be obtained:

- It is able to identify unknown substances.
- A sample's quality or consistency can be determined by this method.
- it can tell you how much of a particular substance is in the mixture.

Raman Spectroscopy

Studies of materials' vibrational, rotational, or other low frequency modes use this spectroscopic method [D.J. Gardiner 1989]. Monochromatic light from a laser in the visible, near infrared, or near ultraviolet ranges is scattered inelastically (Raman scattering). There is an up- or down-shifting of laser photon energy as a result of laser light's interaction with phonons or other excitations in a system. Information about the system's phonon/vibrational modes can be gleaned from the change in energy. The information gleaned from infrared spectroscopy is similar, but it is also complementary.

Usually, a sample is illuminated with a laser beam in order to record the Raman spectrum. A monochromator is used to filter the light from the illuminated area. There are some wavelengths that are not collected because of Rayleigh scattering, but the rest is dispersed onto a detector. When light hits a molecule & interacts with the electron cloud and the bonds, the Raman effect occurs. Virtual states are created when molecules interact with each other. Because of the spontaneous Raman effect, molecules are excited from their ground state to a virtual energy level, where they are then free to enter a vibrationally excited state.

The molecular polarisation potential must be altered, or the electron cloud must be deformed in relation to the vibrational coordinate, in order to exhibit Raman effect. When the polarizability of a substance changes, the Raman scattering intensity will increase, while the Raman shift will decrease. A technique called spontaneous Raman spectroscopy is used in solid state physics to identify materials. A sample's crystallographic orientation can also be determined using this method. Raman scattering can be used to determine the crystal orientation. Orientation of the crystal can be determined by comparing the polarisation of the Raman scattered light and the polarisation of the laser light to the crystal's structure (specifically, its point group). It is common practise in chemistry to use Raman spectroscopy to obtain information about the vibrational properties of chemical bonds. It serves as a fingerprint that could be used to identify the molecule. A normal coordinate analysis with IR and Raman spectra has been used to identify and assign the vibrational frequency for the three different silicon atoms, namely SiO, Si202, & Si303. Near infrared lasers are commonly used in Raman microscopy of biological & medical specimens. As a result of the development of more sensitive detectors, Raman microscopy can now use a wider range of excitation wavelength for inorganic specimens. Resonance As a result of Raman analysis, it is possible to link normal modes to the observed frequency shifts that they produce. The polarisation of Raman scattered light is useful for gaining insights. In crystal lattices, liquid crystals, & polymer samples, spectral analysis reveals macro molecular orientation. Analysis of art objects using molecular Raman spectroscopy has been found to be non-destructive. For example, it could be used to identify inorganic & organic pigments, as well as binding media & varnishes [P. Vandenabeele, 2000].

Ultraviolet Visible Spectroscopy

Photon spectroscopy in the ultraviolet - visible ranges (UV-Vis) is related to UV-Vis spectroscopy. Thus, the absorption in the visible spectrum is strongly influenced by the colour of the chemicals involved, and is therefore limited to the visible & adjacent (near ultraviolet (UV) & near IR (NIR)) light ranges. Molecules go through electronic transitions in this region of the spectrum of electromagnetic radiation. While fluorescence spectroscopy deals with excited states transitioning to the ground state, this method deals with transitions from the excited state into the ground state.

Analysis of transition metals & highly conjugated organic compounds is routinely carried out using UV-Vis spectrum spectroscopy. A colour could be added to transition metal ion solutions (i.e., absorb visible light). Metal atoms have d electrons that have been excited from one electronic state to another. In the presence of other species, such as anions or ligands, the colour of metal ion solutions can be greatly influenced.

Fluorescence Spectroscopy

Fluorescence from a sample is analysed using an electromagnetic spectroscopy technique. Light is used to excite molecules of certain compounds, causing their electrons to emit lower-energy light. Electron & vibrational states are the primary focus of fluorescence spectroscopy. It's common for a species being studied to have both a low-energy ground electronic state & high-energy excited electronic state, with a variety of vibrational states within each. After a photon is absorbed from its ground electronic state, the excited molecule loses vibrational energy until it reaches the lowest vibrational state in the excited electronic state. At some point during this process, the molecule returns to the ground electronic state and emits another photon. The emitted photons will have different energies & frequencies because molecules can fall into any one of a number of vibrational states in the ground state. Thus, the structure of various vibrational levels could be deduced from an examination of the different light frequencies and relative intensities emitted in fluorescent spectroscopy. Fluorescent light emitted by a sample is measured at various frequencies while the excitation light remains at a constant wavelength. It's termed an emission spectrum.

CHEMICAL PROPERTIES

Organic dyes have been used in textile dyeing, colour photography, & photoconductivity because of their ability to withstand irradiation [N.S. Allen 1980]. Chibisov & Korobov 1983 reviewed photochemical changes in various organic dyes; laser dyes were studied under the following two circumstances.

(a) Reports of dye solution characteristics under actual or simulated lasing conditions.

(b) Research into the mechanism of photochemical degradation.

The effects of oxygen on irradiated solutions & addition of detergent additives were both demonstrated by the Weber group [D. Beer 1972] in dye photobleaching for rhodamine or other laser dyes. The primary dye absorption bands have been bleached, according to early studies and subsequent reports. There is a decrease in the chromophore's ability to lase, which is associated with increased in the dye or photoproduct's absorption at a shorter wavelength. Photolysis in dye laser systems with high pump energies occurs with multiple photon events that are uncommon in dye photochemistry under normal conditions. The following terms describe upper state population generated by sequential two photon excitation:

1. Triplet → Triplet absorption:

$$S_0 \xrightarrow{h\nu} S_1 \dashrightarrow T_1 \xrightarrow{h\nu} T_n$$

2. Excitation to higher excited singlet states

$$S_0 \xrightarrow{h\nu} S_1 \xrightarrow{h\nu} S_n$$

The second mechanism is characterized by photoquenching (reversible loss of fluorescent S species) & irreversible dye losses due to the intervention of upper singlet levels. The first mechanism has a low probability due to low inter-system crossing yields, but triplets have a long lifespan. At high pump powers, where Si species have a large absorption cross section, the singlet mechanism comes into play. Radicals and other photochemical products can build up when the dye is optimistic. R.H. Knipe (1983) offered a classification of multiple photon phenomena that led to secondary chemical reactions via absorption by long-lived photoproducts.

EFFECTS OF SOLVENTS ON RHODAMINE 6G STUDIED

Single-molecule detection & various other applications use the rhodamine 6G (Rh6G) as an active medium. It is currently being used for gas sensing, non-linear optics, textile & paper colours inkjet dyes, stains on biological & forensic samples as well as fluorescent pigments & photo sensitizers [P L. Smart 1977]. Organic thin films doped with Rh6G could also be used in light emitting diodes.

In analysing a molecule's vibrations, vibrational spectroscopy, such as Raman and infrared (IR) spectroscopy, could be extremely useful in providing valuable information about the bonding arrangement. Resonance Raman spectroscopy has also been used to study some biomolecules. As a result, few studies on the molecule have been presented in the literature [Majoube 1991]. To overcome this fluorescence, Rh6G has been explored using the surface enhanced Raman scattering (SERS) method in which intensities are up to a higher order of magnitude.

A solvent's effect on rhodamine dyes' molecular structure, fluorescence, & photo physics is significant. Various optical spectroscopic techniques have investigated the effects of impurities in solvents & water on the structure and properties of rhodamine dyes [A. Sharma,2007]. Even so, no vibrational studies have been found on this specific aspect. The IR and Raman spectra of the Rh6G molecule can be used to examine chemically induced changes in the structure & bonding of the molecule. Changes in these parameters are predicted to be dependent on the chemical interactions that take place. It was our goal in the current study to test the impact that solvents have on the Rh6G molecular structure by observing the changes in spectra that were observed in powder & liquid forms of the compound. Understanding Rh6sensing G's properties & solvent dependence of its fluorescence & lasing properties may be gained from this research. Rh6G-methanol solvent interaction is also investigated by DFT in addition to vibrational spectroscopy.

EXPERIMENTAL

The laser grade Rh6G from Exciton in the United States was used without any further purification steps being taken. However, electronic absorption spectroscopy was used to verify its purity. In addition to the spectroscopic grade solvents, methanol, pyridine & acetonitrile from Qualigens Fine Chemicals, India, were utilised without any purification, except for DMF, which was distilled before use. The SOLAR Til monochromator model MS2004 was used to record the electronic absorption spectrum. In order to get the IR spectra of Rh6G crystal powder, we used the KBr pellet. A few drops of Rh6G (cone ~ 2xl0^{-2} M) solution were placed on a KBr window to record the IR spectra of the dye. Spectra were collected using a Perkin Elmer PE-Rxl FTIR Spectrophotometer to capture the infrared spectrum. All across the experiment, the IR spectrometer's spectral resolution was 1 cm^{-1}. A FabRam JY HORIBA FIR 800 spectrograph equipped with an Olympus Bx41 microscope and an ANDOR Model DU 420-0E-323 CCD detector was used to record the RR & NR spectra. Back scattering geometry was used for the measurements, with incident light polarised in a linear fashion and scattered light detected in an unpolarized fashion. Air-cooled Ar+ & He Ne lasers provided excitation lines at 488.0 & 632.8 nm, respectively. The laser head and sample had 10 mW & 3 mW of Ar+ laser power, respectively. The microscope used a lens of IOx to produce a 3 micron spot on the sample. The measurement process took a total of ten seconds. The He-Ne laser's output power was approximately ~ 2 mW. In order to get a spot size of ~ 2 microns, the lens used was 50x. A few seconds to a few minutes was the range of measurement time.

We removed the fluorescence background from the recorded Raman spectra by first fitting the background with a non-linear curve as explained by Chandra [2003] & Washington [1970] and then subtracting the fitted background from the measured data, which has already been identified in this study [J. Sarkar 2006].

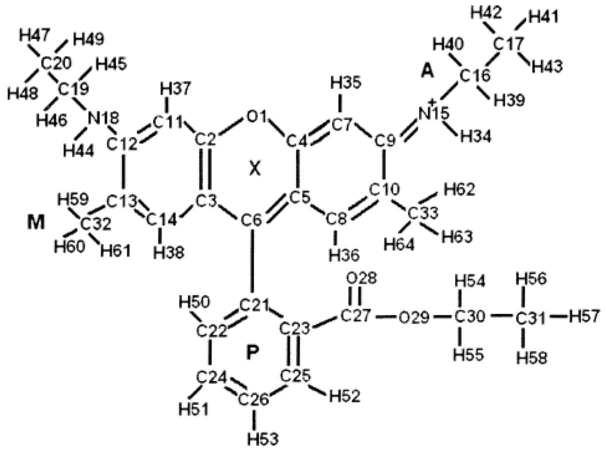


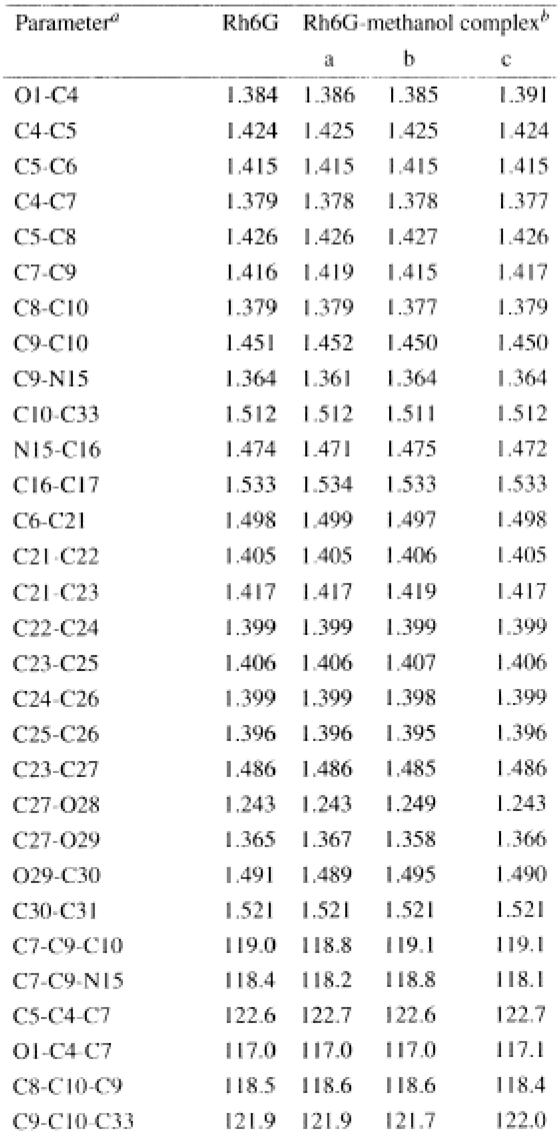
Figure 2: Structure & atomic numbering scheme of rhodamine 6G

RESULTS

DFT Calculations

B3LYP functionals with basis sets of 6-31G are used in DFT calculations to optimise the molecular structure of Rh6G & investigate the interaction between Rh6G and methanol. Gaussian 03 Revision D,01 was used to perform the calculations [M.J. Frisch, G.W. Trucks, & H.B. Schlegel, 2004]. The absence of imaginary wavenumbers has affirmed that the optimised molecule geometry belongs to the minimum potential energy. Table 1 lists the molecule's optimised structural parameters. Compared to the experimental crystal structure data, the bond lengths estimated here are slightly overestimated. Figure 2 depicts the molecule's molecular structure. The molecule's atom labelling scheme is also shown in this diagram.

Table 1: Rh6G or its complexes with methanol, the average molecular parameters



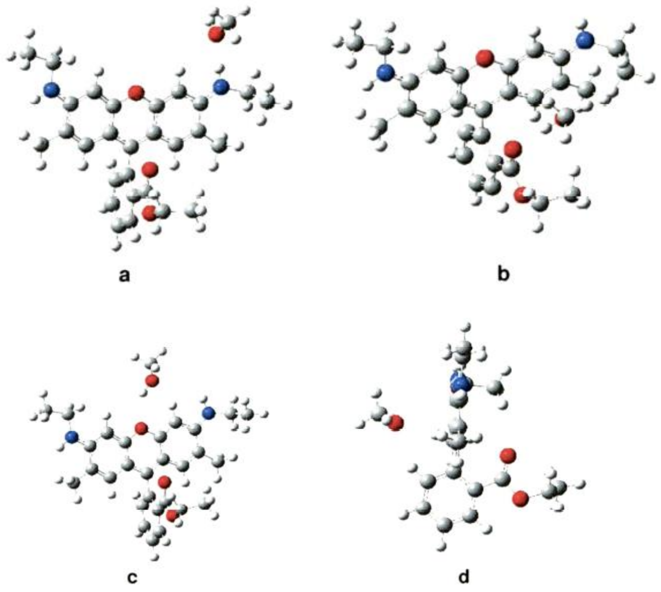
Parameter[a]	Rh6G	Rh6G-methanol complex[b]		
		a	b	c
O1-C4	1.384	1.386	1.385	1.391
C4-C5	1.424	1.425	1.425	1.424
C5-C6	1.415	1.415	1.415	1.415
C4-C7	1.379	1.378	1.378	1.377
C5-C8	1.426	1.426	1.427	1.426
C7-C9	1.416	1.419	1.415	1.417
C8-C10	1.379	1.379	1.377	1.379
C9-C10	1.451	1.452	1.450	1.450
C9-N15	1.364	1.361	1.364	1.364
C10-C33	1.512	1.512	1.511	1.512
N15-C16	1.474	1.471	1.475	1.472
C16-C17	1.533	1.534	1.533	1.533
C6-C21	1.498	1.499	1.497	1.498
C21-C22	1.405	1.405	1.406	1.405
C21-C23	1.417	1.417	1.419	1.417
C22-C24	1.399	1.399	1.399	1.399
C23-C25	1.406	1.406	1.407	1.406
C24-C26	1.399	1.399	1.398	1.399
C25-C26	1.396	1.396	1.395	1.396
C23-C27	1.486	1.486	1.485	1.486
C27-O28	1.243	1.243	1.249	1.243
C27-O29	1.365	1.367	1.358	1.366
O29-C30	1.491	1.489	1.495	1.490
C30-C31	1.521	1.521	1.521	1.521
C7-C9-C10	119.0	118.8	119.1	119.1
C7-C9-N15	118.4	118.2	118.8	118.1
C5-C4-C7	122.6	122.7	122.6	122.7
O1-C4-C7	117.0	117.0	117.0	117.1
C8-C10-C9	118.5	118.6	118.6	118.4
C9-C10-C33	121.9	121.9	121.7	122.0

C5-C8-C10	123.5	123.6	123.4	123.6
C6-C5-C8	124.3	124.3	124.4	124.2
C4-C5-C8	116.0	116.0	116.0	116.0
C3-C6-C5	119.2	119.3	119.3	119.3
C5-C6-C21	120.3	120.3	120.3	120.3
C2-O1-C4	120.6	120.6	120.7	120.6
N15-C16-C17	111.8	111.8	111.5	111.8
C21-C23-C25	119.6	119.6	119.5	119.6
C21-C23-C27	120.0	120.1	121.0	120.0
C21-C22-C24	120.9	120.9	121.1	120.9
C23-C25-C26	120.8	120.8	120.9	120.8
C23-C27-O28	124.2	124.4	124.4	124.2
C23-C27-O29	112.8	112.7	112.9	112.8
C24-C26-C25	119.8	119.8	119.7	119.8
C22-C24-C26	120.0	120.0	120.0	120.0
C27-O29-C30	118.1	118.0	119.2	118.1
O29-C30-C31	110.6	110.6	110.8	110.6
C11-C12-N18-C19	-0.2	-0.2	0.2	0.0
C9-N15-C16-C17	-79.6	-83.8	-68.4	-83.3
C5-C6-C21-C22	-87.1	-86.2	-100.2	-86.3
C7-C9-N15-C16	166.5	171.6	154.3	169.6
C21-C23-C27-O28	-1.0	-0.7	3.0	-0.5
C27-O29-C30-C31	-87.3	-87.1	-87.6	-87.2
H34-O_m		1.792		
H34-O_m-H_m		121.0		
H34-O_m-C_m		126.9		
C16-N15-O_m-C_m		-72.4		
O28-H_m			1.879	
C27-O28-H_m			130.8	
O28-H_m-O_m			173.2	
C27-O28-H_m-O_m			-24.7	
O1-H_m				2.267
H35-O_m				2.094
O1-H_m-O_m				139.5
C_m-O_m-H_m-O1				-147.6

We assumed that different solvent molecules interact with the Rh6G molecule at different locations in order to monitor the effect of methanol on vibrational spectra of Rh6G. At various points on Rh6G, solvent molecules may interact with each other and form stable complexes. Structures of the methanol & Rh6G complexes without symmetry constraints were calculated. Molecular parameters of these compounds have also been calculated by us. Table 1 also shows the average values for these parameters. Figure 2 shows the gas phase interactions between Rh6G & methanol complexes as calculated structures. Determined Rh6G-methanol structures can be regarded as an approximation of the complexes formed in solution, for this reason. On the other hand, as illustrated in Figure 2, the ethoxycarbonyl group attached to the phenyl ring (P) interacts with and forms bonds with the dye molecules primarily at N-H of the ethylamine group (A), central oxygen atom of the xanthene (X) ring (C=0). As calculated molecular parameters show, Rh6G-methanol complexes undergo significant structural changes as a result of methanol's interactions with the dye Rh6G. (table 1). Also, the Rh6binding G's energy (E) with methanol has been estimated by us. Table 2 lists the E values for various Rh6G-methanol complexes. When Rh6G is in close proximity to the N-H of the A groups or/and the C=Q of the P group attached to the P ring, interacting methanol molecules bind strongly with Rh6G. A weak bond between the central oxygen atom of X ring or H atoms of P Ring & Methanol could be inferred from E calculated values. Standard B3LYP functionals also fail to adequately capture weak van der Waals interactions [M.A. Vincent 2008]. Bonding between methanol & Rh6G at various locations was used to investigate the possibility of multiple methanol molecules interacting with a single Rh6G molecule at the same time. Rh6G complexes with 4 (figure 2e) & seven (figure not used) methanol molecules have had their structures optimized. According to binding energies, it is more likely that multiple methanol-methanol & methanol-Rh6G interactions will occur simultaneously than a single methanol-Rh6G interaction.

Table 2: Methanol (E) & Rh6G (E) binding energies calculated at different locations.

Configuration	E (KCal/mol)
a	-13.9
b	-10.3
c	-6.7
d	-6.2



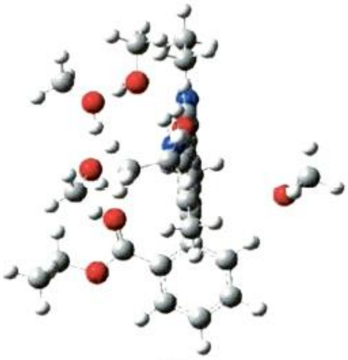


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Figure 2: Intraction between Ph6G & methanol involving: (a) N-H of ethylamine group (b) C=0 of ethoxycarbonyl group and (c) central oxyge atom of xanthenes ring (d) hydrogen atom of the phenyl ring. Trac (e) Show interactio between four methanol molecules & single molecule of Rh6G.

CONCLUSION

The study's original goal was to use spectroscopic methods to look at how the environment affected laser dyes. Laser dyes, & rhodamines have been studied in a variety of settings & environments to achieve this goal (solid hosts, solvents with water impurity). Laser dyes are affected by the environment in which they are used. Many sites/atoms of the rhodamine dyes are found to interact with the solvent molecules in solution, according to research done on rhodamine 6G & rhodamine B. Hydrogen bonds between molecules in dimethylformamide are strengthened when water impurities are added to a solution of rhodamine 6G in the presence of a dimethylformamide solvent.

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