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ENERGY TRANSFER DYE LASER INVESTIGATIONS FROM RHODAMINE 6G TO NILE BLUE IN DIFFERENT SOLVENTS

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ABSTRACT

Photophysical Properties of Rhodamine 6G (Rh6G) and Nile Blue (N.B) dyes in liquid solution, dissolved in Ethanol (ETOH) and propylene carbonate (PC) have been investigated. Propylene carbonate is a new medium for dye laser and has not been used previously as a solvent in energy transfer dye laser. Fluorescence quantum yield and lifetime of these dyes have been calculated in the two mediums. Energy transfer from Rh6G (donor) to N.B (acceptor) in ETOH and PC has been studied by steady-state emission measurements. It has been found that most of the pump energy absorbed by Rh6G is transferred to N.B as useful pump energy. The large values of Energy transfer rate constant $K_{\rm ET}$ and Critical transfer radius R_0 indicate that the dominant mechanism responsible for energy transfer is due to long-range dipole—dipole interaction between excited donor and ground state acceptor molecules. The effect of donor concentration on the Peak Wavelength Emission in the mixtures (Rh6G-N.B) has been studied, for different concentration ratios of Rh6G to N.B, the tuning ranges of 105 nm and 111 nm in ETOH and PC respectively, were achieved. We found a good overlap area that has not been noticed in other previous studies.

Keywords: Energy Transfer, Quantum Yield, Stern Volmer

INTRODUCTION

Dye lasers have been commonly used as coherent light sources since 1966. Organic dyes have wide wavelengths range tunability, and by choosing appropriate dye compounds, dye laser can cover the wavelength range from ultraviolet to infrared (Fukuda *et al.*, 2002). Liquid solutions of laser dyes are versatile sources of coherent tunable radiation with many applications in different fields (Barroso *et al.*, 1998).

However, some dyes show weak absorption of the pump energy resulting in poor lasing efficiency. By increasing the dye concentration in order to increase its absorption, the concentration quenching will occur and leads to decrease in its quantum efficiency. A simple way of overcoming this problem is to use a laser binary dye mixture, so that there is more efficient conversion of the pumping energy (Sesha *et al.*, 2010; Sabry and Mekawey, 1990; Costela *et al.*, 1996). Energy transfer dye lasers (ETDL) using numerous donor acceptor dye pairs have been reported by various investigators during the last three decades.

They have become the subject of intense study because they provide a wide-tunable range, ultra-short laser pulses and improve the efficiency of dye lasers (Sharma *et al.*, 2009; Nedumpara *et al.*, 2008; Dey *et al.*, 2013; Li *et al.*, 2014).

In 1968, soon after the discovery of organic dye lasers, Peterson and Snavelly demonstrated the feasibility of a dye mixture laser with flash lamp excitation (Peterson and Snavely, 1968). In Moller *et al.*, (1971) using N_2 laser pumping obtained an effective excitation transfer from Rh6G to CV and observed an increase in the power output. A simple theoretical model developed by Dienes and Madden, (1973) found to be in good agreement with experimental observations for the Rh6G–CV mixture, a dye pair commonly used for most ETDL studies.

Kumar and Unnikrishnan (2001) also used theoretical calculations to define the suitable energy transfer mechanism responsible for gain enhancement in acceptor. Recent studies have used various dye mixtures which are incorporated into different kinds of solid media to improve ETDL (Sesha *et al.*, 2010; Kailasnath *et al.*, 2008; Yang *et al.*, 2013).

All of those studies use different ways for an ETDL to broaden the spectral range of dye lasers, the most common way to achieve this is to use appropriate medium. In this paper, we study the energy transfer from Rhodamine 6G (Rh6G; donor molecule D) to Nile Blue (N.B; acceptor molecule A) in liquid solutions using a traditional solvent ETOH and a new liquid medium PC which has not been used before in other studies, the energy transfer processes involved are demonstrated in details.

Experimental

Materials

Rhodamine 6G laser dye was obtained from Aldrich, and used as received; the purity of the dye was 99%. Nile blue dye laser was obtained from Aldrich, and used as received, the purity of the dye was 95%. The solvents used in present study Ethanol and Propylene carbonate were of spectroscopic grade, and were used without further purification, and these solvents were chosen because of their low evaporation rate. Refractive index (n), viscosity (η) and Dipole moment (μ) of the pure solvent were taken from the literature (Covington and Dickinson, 1973), and were shown in table 1.

Both dyes are very well soluble in ETOH and PC, freshly prepared solutions were used for all experiments. The chemical structures of the molecules under study are shown in Figure 1

Table 1: Some Physical Properties of the Solvents ETOH and PC

Solvent	Refractive Index (n)	Viscosity in Centi-Poise	Dipole Moment µ
ЕТОН	1.36	1.089	1.69
PC	1.42	2.530	4.98

Figure 1: Molecular Structure of (Rhodamine 6G) Rh6G, Nile Blue (NB) and Propylene Carbonate (PC)

Measurements

Different samples of dyes and dye mixtures were prepared with different concentrations. The absorption spectra of the dyes were recorded using Jasco-Spectrophotometer type V-570 from Ishikama Company. Fluorescence measurements were obtained with excitation at 532 nm using Jasco-Spectrofluorometer type FP-6200 from Ishikama Company.

The peak wavelength emission of the dyes was delineated for three donor/acceptor ratios and different donor concentrations and fixed output wavelength of 532 A from Nd-YAG laser type Surelite 1-10 from Continuum company, the peak wavelengths measured by using spectrometer type S150-11 from solar company.

RESULTS AND DISCUSSION

Spectral Properties of Rh6G and N.B Laser Dyes

Absorption and Emission Spectral Characteristics

The electronic absorption and emission spectra of 1×10^5 mol dm⁻³ Rh6G were carried out in organic solvents of various polarities, namely, ETOH and PC, as shown in figure 2, Rh6G shows no significant

change in both absorption and emission maxima on going from polar to more polar solvents, indicating no change in the dipole moment and the polarity of the dye upon excitation (El-Daly *et al.*, 2004). The electronic absorption and emission spectra of 5×10^{-5} mol dm⁻³ N.B were measured in ETOH and PC, as shown in figure 3.

Both the absorption and emission maxima are red-shifted with increased solvent polarity. The absorption spectrum is more sensitive to change in solvent polarity than the emission spectrum. The maximum wavelength of the absorption peak in N.B shifts by nearly 11 nm when the solvent is changed from ETOH (λ_{ab} =627 nm) to PC (λ_{ab} =638 nm), this long red shift in the position of the absorption spectra indicates an obvious change in dipole moments on going from ground state to an excited state, this change also made a high molar absorption coefficient of N.B in ETOH than in PC that consistent with a large geometry change between electronic ground and excited state.

The emission maxima also shifts to longer wavelengths in solvents of large polarity, however the magnitude of the shift is not as large as that observed in the absorption spectrum. The red shift of the absorption and emission spectrum in polar solvents could mean that the dye is more polar in the excited state (Raju and Varadarajan, 1994). Table 2 summarizes some spectral data of Rh6G and N.B in two different solvents.

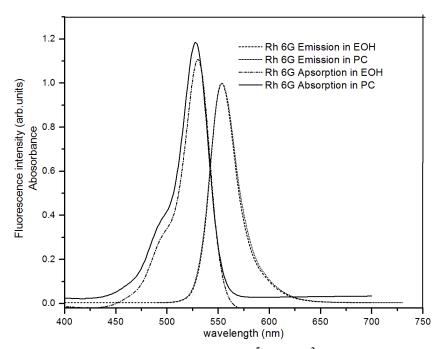


Figure 2: Absorption and Emission Spectra of 1x10⁻⁵mol.dm⁻³(Rh6G) in Ethanol and Propylene Carbonate

Absorption and Emission Cross-Sections

An important indication of the quality of laser dyes is the absorption cross-section σ_a and the emission cross-section σ_e . A higher value of σ_a at the wavelength of pumping laser is desirable for a good laser dye. The emission cross-section σ_e and the absorption cross-section σ_a are calculated according to (Azim *et al.*, 2000):

$$\sigma_{a}(\lambda) = 3.82356 \times 10^{-21} \times \varepsilon \tag{1}$$

$$\sigma_{e}(\lambda) = \lambda_{e}^{4} E(\lambda) \varphi_{f} / 8\pi cn^{2} \tau_{f} \tag{2}$$

Where is λ_e the emission wavelength; n is the refractive index of the solvent; c is the velocity of light; E (λ) is the normalized fluorescence spectrum, ($\int F(v) dv=1$, ϵ is the molar extinction coefficient; ϕ_f the

fluorescence quantum yield, and τ_f is the fluorescence lifetime, The values of σ_a and σ_e for both donor and acceptor at the excitation wavelength (532 nm) are listed in Table 2.

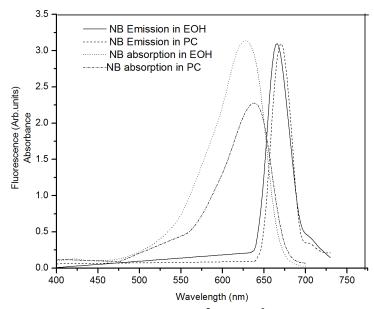


Figure 3: Absorption and Emission Spectra of 5x10⁻⁵mol.dm⁻³(Nile Blue) in Ethanol and Propylene Carbonate

Table 2: Spectral Data of Rh6G and N.B in ETOH and PC

Dye	Rh6G		N.B	
Solvent	ЕТОН	P.C	ЕТОН	P.C
Absorption maximum	530	528	627	638
$\lambda_{(max)}$ nm				
Extinction Coefficient (ε)	1.11	1.18	0.53	0.11
$L.M^{-1}.CM^{-1}(10^5)$				
Absorption Cross- section	4.2	4.3	0.26	0.15
$\sigma_{\rm a} (10^{-16}) {\rm cm}^2$				
Emission maximum	554	554	665	669
$\lambda_{(max)}$ nm				
Emission Cross- section	4.3	3.75	3.36	2.3
$\sigma_{\rm e} (10^{-16}) {\rm cm}^2$				
Fluorescence lifetimes τ_f	3.99	4.08	1.44	0.66
Fluorescence quantum yields ϕ_f	0.95	0.96	0.16	0.12

The calculated value of σ_a for Rh6G in ETOH is same to the value (σ_a =4.2×10⁻¹⁶cm²) in ETOH (Deshpande and Panhalkar, 2002).

Fluorescence Lifetimes and Quantum Yields

The room temperature fluorescence quantum yields ϕ_f were calculated relative to the fluorescence quantum yield of Rh6G in ETOH using the relation (Azim *et al.*, 2000).

$$\varphi_f = \varphi_f(r) \left(\int F_s(v) dv \times A_s \times n_s^2 \right) / \left(\int F_r(v) dv \times A_r \times n_r^2 \right)$$
(3)

Where the indices s and r refer to sample and reference respectively, the integrals represent the corrected fluorescence peak areas, A is the absorbance at the excitation wavelength (532 nm), and n is the refractive index of the solvent. In the determination of the fluorescence quantum yields ϕ_f care was taken to keep the concentrations of all the samples at a level low enough to avoid reabsorption of emitted photons.

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Having the quantum yield, the absorption and the emission spectra, calculation of fluorescence lifetime τ_f is possible, from the expression (Yariv *et al.*, 2001):

$$1/\tau_{f} = 2.88 \times 10^{-9} \,\mathrm{n}^{2} \,\varphi_{f}^{-1} (\int F(v) \,\mathrm{d}v) / (v^{-3} \int F(v) \,\mathrm{d}v) \times \int \epsilon(v) / v \,\mathrm{d}v \tag{4}$$

Where v is the wave number (in cm⁻¹), F (v) is the emitted fluorescence intensity and n is the refractive index of the solvent, $\varepsilon(v)$ is the extinction coefficient of the dye. Based on the absorption and emission spectra, the fluorescence lifetime is calculated.

The fluorescence lifetimes τ_f and quantum yields ϕ_f of Rh6G and N.B have been measured in ETOH and PC and listed in table 2. As shown in Table 2, the fluorescence lifetime τ_f of Rh6G is not strongly affected by solvent polarity.

However, in the case of sample with N.B, both lifetime and quantum yield change drastically with solvent. In particular, the small value of $\tau_f = 0.66$ ns and low $\phi_f = 0.12$ of N.B in P.C contrast with values $\tau_f = 1.44$ ns and $\phi_f = 0.16$ of N.B in ETOH. This decrease of a lifetime and therefore of quantum yield of N.B in going from (ETOH) to more polar (P.C) solvents is attributed to radiation less deactivation of the first excited singlet state. These results of N.B in ETOH are close to $\phi_f = 0.19$ (De Oliveria *et al.*, 2003), and $\tau_f = 1.3$ ns (Kubinyi *et al.*, 2003).

Energy Transfer Dye Laser Characteristics of Rhodamine 6G and Nile Blue

Figure 4 and 5 show the emission spectra of Rh 6G and the absorption spectra of N.B at equal concentrations, $C = 1 \times 10^{-5}$ mol.dm⁻³ in Ethanol and Propylene Carbonate respectively. The overlap spectrum of this pair of D–A molecules, shown in Figure 4 and 5, suggests that energy transfer from Rh 6G to N.B can take place. Various physical phenomena that occur in the dye mixture due to energy transfer and their functional dependence on a number of parameters are described in detail in the following subsections.

Critical Transfer Distance R₀

The strength of interaction between the donor (D^*) and acceptor (A) is usually expressed in terms of the critical transfer distance R_0 . At this distance of separation between donor (D^*) and acceptor (A), the probability of intermolecular energy transfer is just equal to the sum of probabilities for all de-excitation processes of (D^*) (Azim *et al.*, 2000). The critical transfer distance R_0 has been calculated according to the following equation (Katraro *et al.*, 1977).

$$R_o^6 = (9000 \ln (10) K^2 \phi_f / 128 \pi^5 n^4 N_o) \times \int_0^\infty (F_D(v) \varepsilon_A(v) / v^4) dv$$
 (5)

Where ϕ_f is the emission quantum yield of the donor in the absence of the acceptor, $F_D(v)$ is the spectral distribution of the fluorescence of donor normalized to unity, v is the wave number; ϵ_A is the molar extinction coefficient of the acceptor; N_0 is Avogadro's number, n is the refractive index of the solvent; and K^2 is the square of the orientation factor assumed to be 2/3 for solution studies. The values of R_0 for Rh 6G– N.B in Ethanol and Propylene Carbonate are summarized in Table 3. These values are considerably greater than those normally obtained for the collisional energy transfer in which R_0 is in the range of 4–6 Å (El-Daly, 1999).

The values of R_0 for Rh 6G– N.B in Ethanol are greater than those for Rh 6G– N.B in Propylene Carbonate. This is because the overlap area between the absorption spectrum of N.B and emission spectrum of Rh 6G in Ethanol is larger than that between the absorption spectrum of N.B and the emission spectrum of Rh 6G in Propylene Carbonate.

Fluorescence Quenching of Rhodamine 6G by Nile Blue

Figures 6 and 7 show the fluorescence quenching of 1×10^{-6} mol dm⁻³ Rh 6G by N.B at various concentrations in Ethanol and Propylene Carbonate respectively. As shown by increasing the acceptor concentrations, the fluorescence intensity of the donor molecules decreased because of the excitation light absorption of N.B.

This was accompanied by an increase in the fluorescence intensity of the acceptor N.B, whereas the emission spectrum of a mixture of donor and acceptor shows no new band which is an evidence of the absence of exciplex formation between the excited donor and acceptor dye molecules. The quenching rate constant K_{SV} and the energy transfer rate K_{ET} can be determined from the Stern-Volmer relation (Raju and Varadarajan, 1994):

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$$I_{o}/I=1+K_{SV}[A]$$
 (6)
 $K_{ET}=K_{SV}/\tau_{f}$ (7)

Where I_0 , I represent the fluorescence intensity of the donor in the absence and in the presence of acceptor, respectively. [A] is the acceptor concentration, and τ_f is the fluorescence lifetime of the donor in the absence of the acceptor.

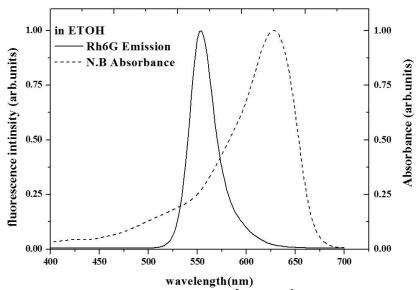


Figure 4: Absorption Spectrum of Nile Blue (1x10⁻⁵mol.dm⁻³) and Emission Spectrum of Rh6G (1x10⁻⁵mol.dm⁻³) in ETOH

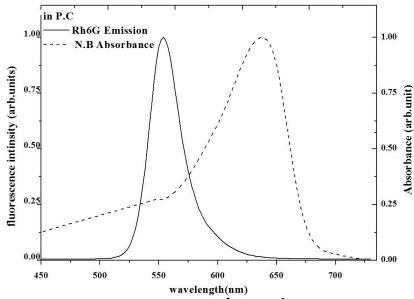


Figure 5: Absorption Spectrum of Nile Blue $(1x10^{-5}mol.dm^{-3})$ and Emission Spectrum of Rh6G The values τ_f are obtained from table 2, and K_{SV} are the slopes of Stern–Volmer plots. Figure 8 shows the Stern–Volmer plots of fluorescence quenching of Rh 6G versus the N.B concentrations in ETOH and PC. The values of K_{SV} and K_{ET} for Rh6G– N.B are listed in Table 3. The higher values of K_{SV} , K_{ET} and R_0 indicate that the dominant mechanism of fluorescence quenching is resonance energy transfer due to long-range dipole–dipole interaction between the excited donor and the ground state acceptor molecules.

The Effect of Donor Concentrations on the Peak Wavelength Emission in the Mixtures (Rh6G-N.B) The concentration effect of the donor (Rh6G) on the Peak Wavelength Emission (λ_{max}) for different donor / acceptor ratios (i.e. D/A= 3, 5, 7) has been studied using second harmonic beam 532 A° from Nd-YAG laser as a pumping source, the result was shown in figure 9 in ETOH and P.C, also an emission spectrum for Rh6G and N.B are shown for comparison. It can be shown from these figures that as the donor concentrations increase, (D/A=3 \rightarrow D/A=7) the Peak Wavelength emission (λ_{max}) of the acceptor largely shifts towards a shorter wavelength, i.e., blue shifted. This indicates that the gain enhancement of N.B due to energy transfer occurred efficiently. Also, the energy transfer dye laser system can operate at low acceptor concentration. These effects practically, expand the spectral range of operation (El-Daly *et al.*, 2013).

Table 3: Measured Values of Photophysical Parameters of Rh 6G and N.B Laser Dyes

Dye	Solvent	Quenching Constant K _{SV} ×10 ³ dm ³ mol ⁻¹	Energy Transfer Rate $K_{ET} \times 10^{12} dm^3 mol^{-1} s^{-1}$	Critical Radius R ₀ (Transfer (A°)
Rh6G-	ЕТОН	35	9		55.5
N.B	P.C	47	11.5		42.4

The dependence of λ_{max} on the concentration of the acceptor could be delineated from Figure 9 and is given by Figure 10. It could be seen that as the concentration of the acceptor increases, the λ_{max} is red shifted. Again, as the donor concentration increases, the emission wavelength of the acceptor shifts towards shorter wavelengths. Thus, most of the excitation energy absorbed by Rh6G is transferred to N.B as a useful pump power making the excitation transfer quite efficient.

The maximum emission wavelength λ_{max} of Rh6G-N.B in ETOH in the ETDL system of 675 nm is achieved at D/A=3 and a donor concentration of 3×10^{-4} mol.dm⁻³. The maximum emission wavelength λ_{max} of Rh6G-N.B in P.C in the ETDL system of 680 nm is achieved at D/A=3 and a donor concentration of 3×10^{-4} mol.dm⁻³. Besides that, a wide tuning band extending from 570-675 nm is realized in ETOH, and from 569- 680 nm in P.C at this ratio.

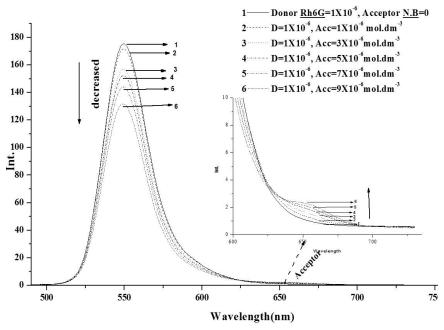


Figure 6: Emission Spectra of 1×10^{-6} mol dm⁻³ Rh6G in ETOH (λ =532 nm) in the Absence and in the Presence of N.B. The Concentrations of N.B are 0, 1, 3, 5, 7 and 9×10^{-6} mol dm⁻³

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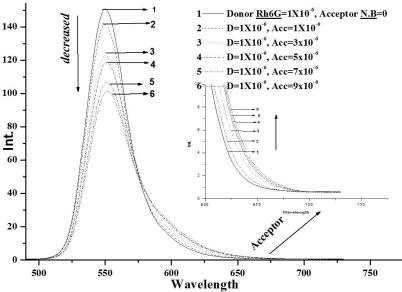


Figure 7: Emission Spectra of 1×10^{-6} mol dm⁻³ R6G in PC (λ =532 nm) in the Absence and in the Presence of N.B. The Concentrations of N.B are 0, 1, 3, 5, 7 and 9×10^{-6} mol dm⁻³

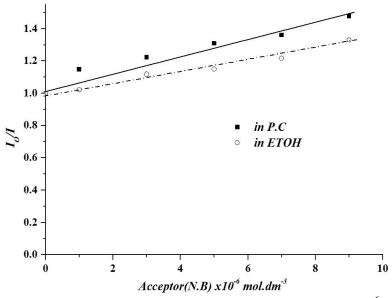


Figure 8: Stern-Volmer Plots for Fluorescence Quenching of Rh6G (1x10⁻⁶ mol.dm⁻³) by Nile Blue in Ethanol and Propylene Carbonate

Conclusion

In this study energy transfer from R6G (donor) to NB (acceptor) in P c and EOH has been studied by emission measurements. Some physical parameters have been calculated and presented. We show that the new medium propylene carbonate PC has a good effect on the efficiency of energy transfer comparing with EOH although the low absorption cross section and quantum yield of NB, the values of energy transfer rate constant K_T and R_o in PC and EOH are found to be 11.5×10^{12} dm³. mol¹.s⁻¹, 42.2 A°, and 9×10^{12} dm³. mol¹.s⁻¹, 55.5 A° respectively for Rh6G, N.B mixture, which indicated that the energy transfer depends primarily on the D-A distance. The energy transfer in this medium shows a new tunable band of 111 nm width, and a good overlap area that have not been reached in previous studies.

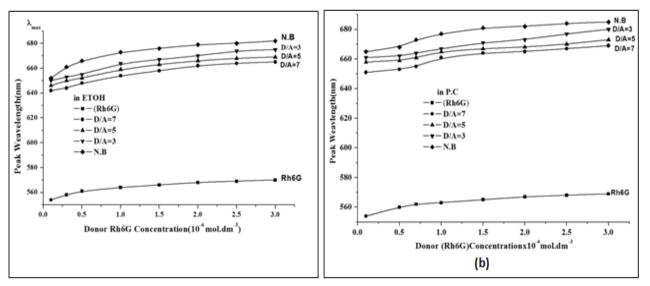


Figure 9: Peak Wavelength Emission as a Function of Donor Concentration for (Rh6G, Nile Blue) Mixture, the Ratios are (3:1),(5:1),(7:1). Also Peak Wavelength Emission for Rh6G Alone and Nile Blue Alone as a Function of Concentrations are Represented in (a) ETOH, (b) P.C

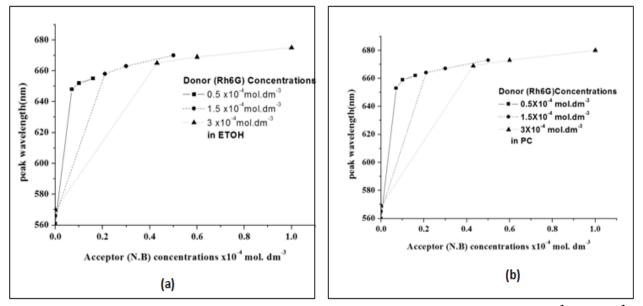


Figure 10: Dependence of λ_{max} on the Acceptor (N.B) Concentration at 1,3 and 5×10^{-3} mol.dm⁻³ Donor (Rh6G) Concentration, when(D/A=3,5,7) in (a) ETOH, (b) P.C

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