Absorption and luminescence studies of Dy^{3+} doped Phosphate glass

M.Reddi Babu[a,b], N.Madhusudhana Rao[a], A.Mohan Babu[a,b], L.Ramamoorthy[b]

[a]School of Advanced Sciences, VIT Univeristy, Vellore, TN, India
[b]Department of Physics, C R Engineering College, Tirupati - 517506, AP, India

Abstract- Absorption and emission properties of Dy^{3+} doped phosphate glass with the chemical composition of 55.5 P_2O_5 + 14.5 SrO + 14 K_2O + 9 Al_2O_3 + 6 KF +1.0 Dy_2O_3 have been investigated. The density and refractive index of the prepared glass were determined by Archimedes principle and using an Abbe refractometer, respectively. Optical absorption spectra were recorded on a Hitachi U-3400 spectrometer in the wavelength of 250-2500 nm. The intensity parameters (Ω_2, Ω_4 and Ω_6) are determined from the Judd-Ofelt analysis have been used to calculate radiative transition probabilities (A_R), life times (τ_R) and branching ratios (β_R) from the exited $^4F_{9/2}$ level to the lower levels of Dy^{3+} ion.

Index Terms- Dy^{3+} ion, Phosphate glass, Rare Earths, JO parameters

I. INTRODUCTION

Rare-earth doped composites are the treasury of optical materials because of they have been extensively used in the preparation of solid state lasers, optical amplifiers, phosphors, memory and display devices. Number of rare-earth doped glasses were prepared using the selected network formers, network modifiers and intermediates by the different researchers [1-10]. The optical properties of rare earth (RE) ions doped in different crystalline and glass matrices are of current interest because they exhibit characteristic lasing transitions.

The host glass plays an important role in the development of RE doped optical devices. In the search of different host glasses, the oxide glasses were identified as the most stable host for practical applications due to their high chemical and thermal stability. Among the various oxide glasses, the phosphate based glasses are identified as good hosts for many RE ions because of their high stimulated emission cross sections, weak upconversion luminescence and lesser probability for energy back transfer which are suitable for various luminescence applications.

In addition, phosphate glass is an excellent material due to its good chemical stability, ion exchange ability, high gain coefficient and wide bandwidth capability. Also, it is very easy to prepare these glasses in different compositions as they also preserve useful properties upon the introduction of significant amount of rare earth ions. In phosphate glasses, Al_2O_3 is often added to modify the glass structure that improves the physical properties and mechanical strength [11]. P_2O_5 based multicomponent glass systems have tremendous potential applications in optical devices such as laser devices, optical amplifiers, fluorescent lamps [11-15]**.** Among different rare-earth ions, the Dy^{3+} ion has been identified as the most efficient ion for obtaining the lasing action, frequency up-conversion and optical fiber amplification [16]. In the present work, we report the absorption and luminescent properties of Dy^{3+} doped P_2O_5–SrO-K_2O-Al_2O_3-KF glasses obtained from the absorption, emission and decay measurements.

II. MATERIALS AND METHOD

2.1 Sample preparation

Dy^{3+} doped phosphate glass with molar compositions of 55.5 P_2O_5 +14.5 SrO +14 K_2O + 9 Al_2O_3 + 6 KF + 1.0 Dy_2O_3 (labeled as PKFSADy10) were prepared by conventional melt quenching technique using the reagent grade $Al(PO_3)_3$, $Sr(PO_3)_2$, KH_2PO_4, KF and Dy_2O_3 chemicals. About 10 gm of batch composition was thoroughly crushed in an agate mortar and the homogeneous mixture was taken in a platinum crucible and heated in an electric furnace at a temperature of 1075 ^{0}C for 45 min. The molten sample was air quenched by pouring onto a preheated thick brass plate. To remove the thermal strains, the glass sample was annealed for 10 hours at 350^{0}C and allowed to cool to room temperature (RT).

2.2 Physical and optical measurements

The density of the glass sample is determined by the Archimedes method using distilled water as immersion liquid. The refractive index (n) was measured on an Abbe refractometer using sodium lamp as light source with 1 – bromonaphthaline ($C_{10}H_7Br$) as contact liquid. Absorption spectra were recorded on a spectrophotometer (Hitachi U-3400) in the wavelength region of 250-2500 nm with a spectral resolution of 1 nm. Emission spectra were obtained by exciting the samples with the 457.9 nm line of an Ar^+ laser. The photoluminescence was detected with a double monochromator equipped with a photo-multiplier tube (PMT) with a spectral resolution of 0.06 nm. For lifetime measurements, a mechanical chopper in conjunction with a multi-channel scalar was employed.

III. THEORTICAL BACKGROUND

The optical absorption spectra of rare-earth ions provide the information regarding the energy level positions and the intensities of various absorption bands to understand their radiative properties. The quantitative computation of the intensities of these transitions has been developed by Judd [17] and Ofelt [18] and their theory is called Judd - Ofelt theory. The radiative properties such as radiation transition probability (A_R),

total radiative transition probability (A_T), radiative life time (τ_R), branching ratio (β_R) and stimulated emission cross-emission ($\sigma(\lambda_P)$) have been calculated using the equations available in the literature [19-26].

IV. RESULTS AND DISCUSSION

4.1 Physical properties

The prepared Dy^{3+} doped phosphate glass has been found to be optically transparent without any formation of bubbles and strains. Using the general chemical formulae, the density (d) and refractive index (n), rare-earth ion concentration (N) and optical path length (l) measured of the glass are presented in the Table 1.

4.2 Absorption spectra and energy level analysis

The optical absorption spectra of Dy^{3+} doped phosphate glass recorded in the UV-VIS and NIR regions are shown in the Figs.1 (a) & (b) respectively, The absorption bands observed at 1685, 1276, 1095, 900, 801, 752, 473, 452, 425, 385, 377, 364 and 349 nm are assigned to different transitions from the $^6H_{15/2}$ ground state to the $^6H_{11/2}$, $^6F_{11/2}$, $^6F_{9/2}$, $^6F_{7/2}$, $^6F_{5/2}$, $^6F_{3/2}$, $^4F_{9/2}$, $^4I_{15/2}$, $^4G_{11/2}$, $^4I_{13/2}$, $^4M_{19/2}$, $^4P_{3/2}$ and $^6P_{7/2}$ excited states respectively. It is observed that the most intense transition $^6H_{15/2} \rightarrow {}^6F_{11/2}$ is centered at around 1276 nm.

Table 2 presents the experimental (E_{exp}) and calculated (E_{cal}) free-ion energy level positions of PKFSADy10 glass along with other host glasses [27-29]. The energy level analysis has been carried out by a f-shell empirical program [30] and the best-fit set of free-ion parameters of have been determined by minimizing the difference between the observed and calculated energies by the standard least-square fit method using the initial free-ion values of Dy^{3+}: $LaCl_3$ [31]. Among various interactions that contribute to total free-ion Hamiltonian, the major contribution occurs from the interelectronic (F^k) and the spin-orbit (ζ) interactions which govern the $^{2S+1}L_J$ level positions. The rest of the terms will only give corrections to the energy of these levels without removing their degeneracy. Hence, during the fitting process, out of 20 free-ion parameters, the only parameters that were allowed to vary are F^K and ζ. Four of the atomic parameters (M^2, M^4, P^2 and P^6) were constrained according to $M^2 = 0.56\ M^0$, $M^4 = 0.38\ M^0$, $P^4 = 0.75\ P^2$, $P^6 = 0.50\ P^2$. The values of $T^2 = 301$, $T^3 = 12$, $T^4 = 14$, $T^6 = -299$, $T^7 = 386$, $T^8 = 350$, $M^0 = 2.50$, and $P^2 = 501$ were fixed in the parameterization. The best fit free-ion parameter values evaluated for PKFSADy10 glass are compared in Table 3. The hydrogenic ratios, F^2/F^4 (~1.42) and F^2/F^6 (~2.03), are more or less same in all the glass systems presented in Table 3, which indicate that the radial integral part of the f-orbitals of Dy^{3+} ions remains unchanged even though the glass compositions are changed. This could be due to the fact that the 4f shell of Dy^{3+} ions is strongly shielded by the $5s^2$ and $5p^6$ orbitals and the total electrostatic effects $\sum_K F^K$ experienced by the Dy^{3+} ion glass is found to be greater than that of PKBAFDy10 [27] and PKBADy10 [28] and PKAZFDy10 [29] glasses.

The nature of bonding properties in the host glass has been calculated from nephelauxetic ratio (β) and the bonding parameter (δ) is defined as [32,33],

$$\delta = [(1- \overline{\beta})/ \overline{\beta}\ ] \times 100$$

$$\overline{\beta} = \frac{(\sum_n \beta)}{N}$$

where , here β is called nephelauxetic ratio = E_c/E_a ; E_c and E_a are the energies of the corresponding transitions in the complex and aquo-ions, respectively and N represents the number of levels that are used to compute the $\overline{\beta}$ values. Based on the local environment, the δ value may be positive or negative which represents covalent or ionic bonding. Table 3 also presents the values of $\overline{\beta}$ and δ for PKFSADy10 glass along with other Dy^{3+} ion doped glass systems [27-29]. The experimental and theoretical oscillator strengths calculated from the JO theory for the Dy^{3+}doped PKFSADy10 glass are presented in Table 4. The evaluated J-O parameters are $\Omega_2 = 6.21X10^{-20}$ cm^2, $\Omega_4 = 1.58X10^{-20}$ cm^2 and $\Omega_6 = 1.38X10^{-20}$ cm^2. Table 5 compares the JO parameters obtained in the present work with those obtained for the other glass systems. For PKFSADy10 glass, the magnitude of parameters follow the trend as $\Omega_2 > \Omega_4 > \Omega_6$. In general, the JO parameters provide information on the nature of bonding between RE ion and surrounding ligands as well as the symmetry of the environment around the Ln ions.

The JO parameters determined from the absorption spectra have been used to predict radiative properties such as, radiative transition probabilities(A_R), lifetimes(τ_R), branching ratios(β_R) and peak stimulated emission cross-sections (σ_R) of Dy^{3+} ions in the titled glass for fluorescent levels for the emission $^4F_{9/2} \rightarrow {}^6H_{J\ (J=11/2,13/2,15/2)}$ transitions are presented in Table 6.

4.3 Luminescence and decay time measurements

Fig. 2 shows the emission spectra of PKFSADy10 glass which consists of three bands at 663 nm, 574 nm and 480 nm corresponding to the $^4F_{9/2} \rightarrow {}^6H_{11/2}$, $^4F_{9/2} \rightarrow {}^6H_{13/2}$ and $^4F_{9/2} \rightarrow {}^6H_{15/2}$ transitions, respectively. From the emission spectra the peak positions (λ_p), effective linewidths (Δ_{eff}) and branching ratios (β_R) have been determined and are given in Table 6 along with β_R values predicted from the JO theory. The luminescence decay from the $^4F_{9/2}$ level of Dy^{3+} ions measured with an excitation of 457.9 nm is shown in Fig. 3. The lifetime of the $^4F_{9/2}$ level has been obtained by taking the first e-folding time.

4.4 Radiative properties

The luminescence quantum efficiency (η) is defined as the ratio of the number of photons emitted to the number of photons absorbed. For lanthanide ion systems it is equal to the ratio of the experimental lifetime (τ_{exp}) to the predicted radiative lifetime (τ_R) of the excited level as given by

$$\eta = \frac{\tau_{exp}}{\tau_R}$$

The quantum efficiency (η) of the $^4F_{9/2}$ fluorescent level has been found to be 65% and the non-radiative decay rate (W_{nr}) for the $^4F_{9/2}$ level is given by,

$$W_{nr} = \frac{1}{\tau_{exp}} - (A_R)_{JO}$$

where $(A_R)_{JO}$ is the total radiative transition probability determined through JO analysis. The W_{nr} for $^4F_{9/2}$ level of Dy^{3+} ions is found to be 457 s^{-1}. In this work, the experimental value of branching ratio (β_{exp}) for $^4F_{9/2} \rightarrow {}^6H_{13/2}$ transition is found to be maximum and is in good agreement with the predicted value (β_{exp}).

V. CONCLUSIONS

The electronic structure of Dy^{3+} ions in phosphate glasses has been deduced from absorption spectra by means of a free-ion Hamiltonian model. The intensities of absorption bands of Dy^{3+} ions in phosphate have also been analyzed with the help of Judd-Ofelt theory. A fairly good agreement between the radiative properties calculated on the basis of the JO theory and experimental values has been noticed. The decay curve of the $^4F_{9/2}$ level of Dy^{3+} in the phosphates glass has been found to be non-exponential.

VI. ACKNOWLEDGEMENTS

One of the Authors Dr.A.Mohan Babu would like to thank the DAE-BRNS, Mumbai for sanctioning the Major Research Project No. 2012/34/72/BRNS and DST-SERB, New Delhi, Sanction Letter. No. SR/FTP/PS-109/2012 for providing the financial support to carry out the present research work.
M. Reddi Babu is also thankful to DAE-BRNS for the financial support through project fellow in the above project.

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AUTHORS

First Author – M. Reddi Babu, School of Advanced Sciences, VIT University,Vellore, Tamilnadu, India.
Email Id: reddibab@gmail.com
Second Author – Dr. N.Madhusudhana Rao, School of Advanced Sciences, VIT University, Vellore, Tamilnadu.
Email Id: nmrao@gmail.com
Third Author – Dr. A.Mohan Babu, Dept. of Physics, C R Engineering College, Tirupati, Chittoor Dt. India
E mail Id: mohanphy57@gmail.com
Fourth Author – Prof. L.Ramamoorthy Dept. of Physics, C R Engineering College, Tirupati, Chittoor Dt. India.
E mail Id: lrmphysics@yahoo.co.in

***Correspondence Author** – Dr. A.Mohan Babu, Dept. of Physics, C R Engineering College, Tirupati, Chittoor Dt. India
E mail Id: mohanphy57@gmail.com

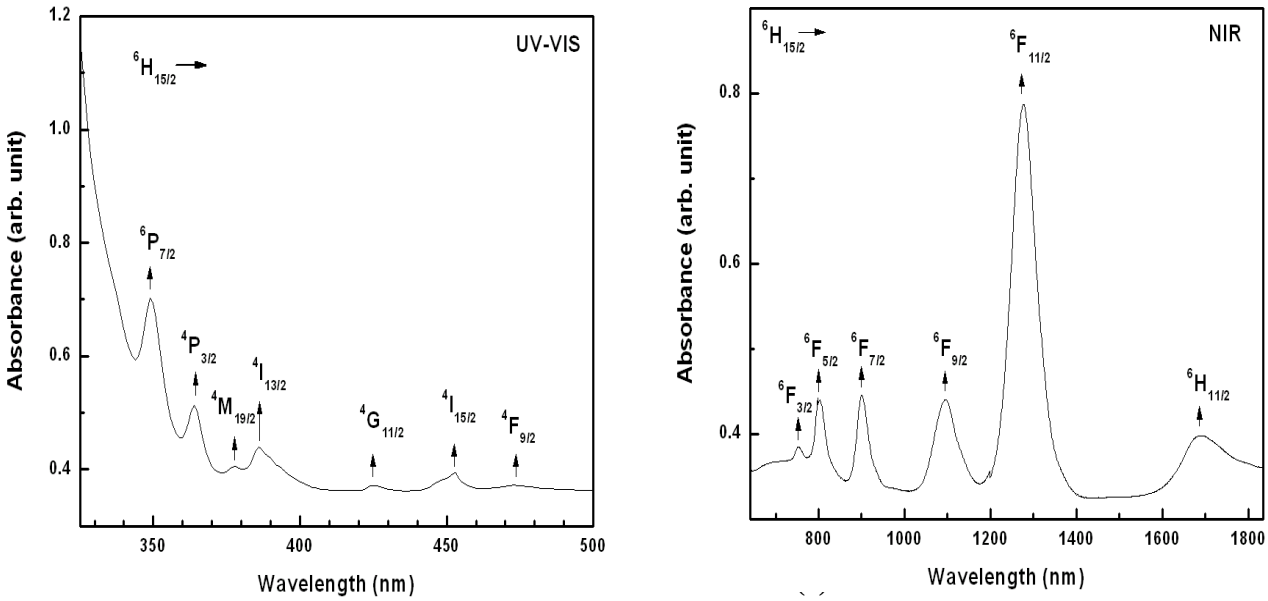

Fig. 1 Optical absorption spectra of PKFSADy10 glass in (a) UV-VIS and (b) NIR regions

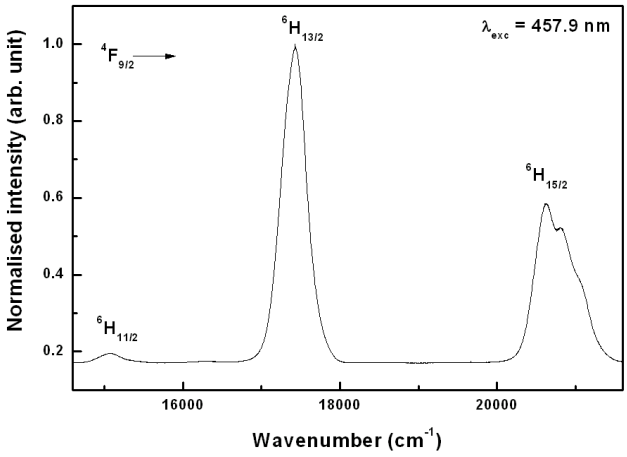

Fig. 2 Emission spectrum of PKFSADy10 glass

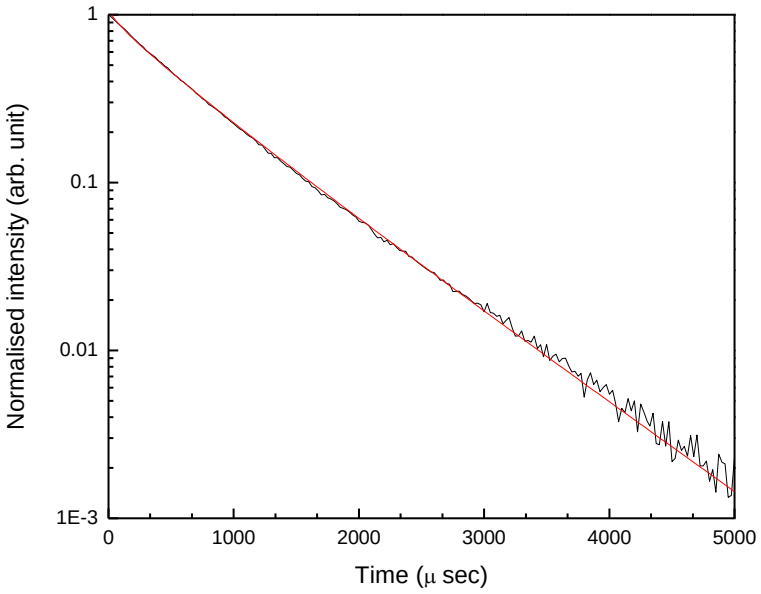

Fig. 3 Decay profile for $^4F_{9/2}$ level of PKFSADy10 glass.

Table 1 Physical properties of 1.0 mol % Dy^{3+}-doped PKFSADy10 glass.

Properties	PKFSADy10
Refractive index, *n*	1.532
Density, *d* (g cm^{-})	2.795
Concentration, *C* (10^{20} ions cm^{-3})	2.622
Optical path length l (cm)	0.312

Table 2 Comparison of experimental (E_{exp}) and calculated (E_{cal}) energies (cm^{-1}) of PKFSADy10 glass along with different glasses.

Transition	PKFSADy10 [Present work]		PKBAFDy10 [27]		PKBADy10 [28]		PKAZDy10 [29]	
	E_{exp}	E_{cal}	E_{exp}	E_{cal}	E_{exp}	E_{cal}	E_{exp}	E_{cal}
$^6H_{15/2}$	0	42	0	43	0	-3	0	12
$^6H_{13/2}$	--	3560	3665	3544	--	3556	--	--
$^6H_{11/2}$	5932	5934	5935	5896	5931	5928	5995	5975
$^6F_{11/2}$	--	7755	7836	7799	--	7722	7874	7884
$^6H_{9/2}$	7834	7835	7826	7769	7819	7831	--	--
$^6F_{9/2}$	9130	9004	9120	9116	9124	9050	9132	9109
$^6H_{7/2}$	--	9289	--	9204	--	9285	--	--
$^6H_{5/2}$	--	10366	--	10268	--	10362	--	--
$^6F_{7/2}$	11106	11068	11104	11067	11038	11036	11,186	11,104
$^6F_{5/2}$	12490	12514	12419	12494	12407	12482	12,500	12,556
$^6F_{3/2}$	13281	13318	13291	13294	13280	13284	13,351	13,361
$^6F_{1/2}$	--	13884	--	13851	--	13851	--	--
$^4F(3)_{9/2}$	21134	21173	21128	21122	21142	21128	21,142	21,176
$^4I(3)_{15/2}$	22095	22162	22198	22189	22075	22157	22,075	22,125
$^4G(4)_{11/2}$	23513	23545	23507	23515	23474	23500	23,529	23,512
$^4M_{21/2}$	--	25204	--	25244	--	25268	--	--
$^4K(1)_{17/2}$	--	25831	25880	25890	--	25826	25,907	25,897
$^4I(3)_{13/2}$	--	25867	--	25833	--	25854	--	--
$^4F(3)_{7/2}$	25918	25882	--	25790	25907	25859	--	--
$^4M_{19/2}$	26477	26376	26448	26443	26455	26426	26,385	26,348
$^4P(2)_{3/2}$	27468	27437	27465	27465	--	27330	27,473	27,431
$^6P_{5/2}$	--	27580	--	27640	27473	27439	--	--
$^4I(3)_{11/2}$	--	28112	--	--	--	28098	--	--
$^6P_{7/2}$	28634	28677	--	--	--	28551	28,653	28,689
$^4M_{15/2}$	--	28700	--	--	--	28738	--	--

Table 3 The best fit free ion parameters (in cm^{-1}), net electrostatic field ($\sum_K F^K$), hydrogeneic ratios (F^2/F^4 & F^2/F^6), the average nephelauxetic ratio ($\overline{\beta}$) and bonding parameter (δ) of Dy^{3+} complexes.

Parameters	**PKFSADy10** [Present work]	**PKBAFDy10[27]**	**PKBADy10[28]**	**PKAZFDy10[29]**
E_{AVG}	56545	56537	56456	56,498
F^2	94009	94252	93490	93,743
F^4	66257	64954	66878	66,398
F^6	46257	45786	45799	45,992
ζ	1969	1942.5	1969	1977
$\sum_K F^K$	206523	204992	206167	206,133
F^2 / F^4	1.42	1.45	1.40	1.41
F^2 / F^6	2.03	2.06	2.01	2.03
$\overline{\beta}$	1.0058	1.0042	1.0043	1.0031
δ	-0.5767	-0.416	-0.424	-0.309

Table 4 Experimental (f_{exp}) and calculated (f_{cal}) oscillator strengths ($x10^{-6}$) of PKFSADy10 glass.

Transition	f_{exp}	f_{cal}
$^6H_{11/2}$	1.44	1.69
$^6F_{11/2}$	10.8	10.77
$^6F_{9/2}$	3.3	3.22
$^6F_{7/2}$	2.18	2.48
$^6F_{5/2}$	2.1	1.1
$^6F_{3/2}$	0.3	0.2
$^4F_{9/2}$	0.11	0.19
$^4I_{15/2}$	0.68	0.57
$^4G_{11/2}$	0.11	0.11
$^4M_{19/2}$	2.89	0.79

Table 5 Comparison of the JO parameters ($\times 10^{-20}$ cm^2) of PKFSADy10 glasss with other Dy^{3+} doped glass.

System	Ω_2	Ω_4	Ω_6	Trend	Reference
PKFSADy10	6.21	1.58	1.38	$\Omega_2 > \Omega_4 > \Omega_6$	Present work
PKBAFDy10	12.34	2.67	2.30	$\Omega_2 > \Omega_4 > \Omega_6$	[27]
PKBADy10	9.72	3.08	1.66	$\Omega_2 > \Omega_4 > \Omega_6$	[28]
PKAZFDy10	14.11	3.07	1.95	$\Omega_2 > \Omega_4 > \Omega_6$	[29]

Table 6 Emission peaks (λ_p, nm), effective band widths (λ_{eff}, nm), radiative transition probabilities (A_R, s^{-1}), peak stimulated emission cross-sections ($\sigma(\lambda_p)$,$\times 10^{-22}$$cm^2$), and experimental and calculated branching ratios(β_R) for the $^4F_{9/2} \rightarrow {}^6H_J$ levels of PKFSADy10 glass.

Level $^4F_{9/2} \rightarrow$	λ_p	$\Delta\lambda_{eff}$	A_R	$\sigma(\lambda_p)$	β_R	
					Exp	Cal
$^6H_{11/2}$	663	51	67	1.41	0.01	0.079
$^6H_{13/2}$	574	10	540	33.54	0.56	0.645
$^6H_{15/2}$	480	18	109	1.79	0.429	0.13