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Spectral, Thermal and FTIR analysis of Pr³⁺ doped in Zinc Lithium Sodium Lead Tungsten Aluminophosphate glasses

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ABSTRACT: Zinc lithium sodium lead tungsten aluminophosphate glasses containing Ho³⁺ in (35-x) P₂O₅:10ZnO:10Li₂O:10Na₂O:10PbO:15WO₃:10Al₂O₃: xPr₆O₁₁ (where x=1, 1.5,2 mol %) have been prepared by melt-quenching method. The amorphous nature of the glasses was confirmed by x-ray diffraction studies. Optical absorption, Excitation, FTIR and fluorescence spectra were recorded at room temperature for all glass samples. Judd-Ofelt intensity parameters Ω_{λ} ($\lambda=2, 4, 6$) are evaluated from the intensities of various absorption bands of optical absorption spectra. Using these intensity parameters various radiative properties like spontaneous emission probability, branching ratio, radiative life time and stimulated emission cross-section of various emission lines have been evaluated.

KEYWORDS: ZLSLTAP Glasses, Optical Properties, Judd-Ofelt Theory, Rare earth ions.

I. INTRODUCTION

Glass is a continuous random network lacking both symmetry and periodicity. Glasses are super cooled liquid and amorphous in nature. Transparent glass-ceramic as host materials for active optical ions have attracted great interest recently due to their potential application in optical devices such as frequency-conversion materials, lasers and optical fiber amplifiers. The oxide glasses generally possess a good mechanical strength, chemical durability, and thermal stability [1-5]. Phosphate glasses combine the attributes of wide transmission region, good glass stability, rare earth ion solubility, high refractive index, high density, excellent chemical and thermal stability [6-8]. Phosphate glass is an extremely promising material for laser and nonlinear applications in optics due to some of its essential characteristic features, such as low phonon maxima, low melting temperature and excellent transparency in the far infrared region [9-12]. The addition of network modifier (NWF) Li₂O is to improve both electrical and mechanical properties of such glasses. An important characteristic of these materials is the relatively low cut off phonon energy compared with other oxide glasses such as silicate or tellurite glasses. ZnO is a wide band gap semiconductor and has received increasing research interest. It is an important multifunctional material due to its specific chemical, surface and microstructural properties [13].

The present work reports on the preparation and characterization of rare earth doped heavy metal oxide (HMO) glass systems for lasing materials. I have studied on the Optical absorption, Excitation and fluorescence properties of Pr³⁺ doped zinc lithium sodium lead tungsten aluminophosphate glasses. The intensities of the transitions for the rare earth ions have been estimated successfully using the Judd-Ofelt theory. The laser parameters such as radiative probabilities(A), branching ratio (β), radiative life time(τ_R) and stimulated emission cross section(σ_p) are evaluated using J.O.intensity parameters(Ω_{λ} , $\lambda=2,4$ and 6).

II. EXPERIMENTAL TECHNIQUES

Preparation of glasses

The following Pr³⁺ doped Zinc lithium sodium lead tungsten aluminophosphate glass samples (35-x) P₂O₅:10ZnO:10Li₂O:10Na₂O:10PbO:15WO₃:10Al₂O₃: xPr₆O₁₁ (where x=1,1.5,2) have been prepared by melt-quenching method. Analytical reagent grade chemical used in the present study consist of P₂O₅, ZnO, Li₂O,Na₂O, PbO,



WO₃, Al₂O₃ and Pr₆O₁₁. All weighed chemicals were powdered by using an Agate pestle mortar and mixed thoroughly before each batch (10g) was melted in alumina crucibles in silicon carbide based an electrical furnace.

Silicon Carbide Muffle furnace was heated to working temperature of 1080⁰C, for preparation of zinc lithium sodium lead tungsten aluminophosphate glasses, for two hours to ensure the melt to be free from gases. The melt was stirred several times to ensure homogeneity. For quenching, the melt was quickly poured on the steel plate & was immediately inserted in the muffle furnace for annealing. The steel plate was preheated to 100⁰C. While pouring; the temperature of crucible was also maintained to prevent crystallization. And annealed at temperature of 350⁰C for 2h to remove thermal strains and stresses. Every time fine powder of cerium oxide was used for polishing the samples. The glass samples so prepared were of good optical quality and were transparent. The chemical compositions of the glasses with the name of samples are summarized in Table 1

Table 1 Chemical composition of the glasses

Sample	Glass composition (mol %)
ZLSLTAP (UD)	35 P ₂ O ₅ :10ZnO:10Li ₂ O:10Na ₂ O:10PbO:15WO ₃ :10Al ₂ O ₃
ZLSLTAP (PR 1)	34 P ₂ O ₅ :10ZnO:10Li ₂ O:10Na ₂ O:10PbO:15WO ₃ :10Al ₂ O ₃ :1 Pr ₆ O ₁₁
ZLSLTAP (PR 1.5)	33.5 P ₂ O ₅ :10ZnO:10Li ₂ O:10Na ₂ O:10PbO:15WO ₃ :10Al ₂ O ₃ :1.5 Pr ₆ O ₁₁
ZLSLTAP (PR 2)	33P ₂ O ₅ :10ZnO:10Li ₂ O:10Na ₂ O:10PbO:15WO ₃ :10Al ₂ O ₃ : 2Pr ₆ O ₁₁

ZLSLTAP (UD)—Represents undoped Zinc Lithium Sodium Lead Tungsten Aluminophosphate glass specimen.

ZLSLTAP (PR) -Represents Pr³⁺ doped Zinc Lithium Sodium Lead Tungsten Aluminophosphate glass specimens.

III. THEORY

3.1 Oscillator Strength

The intensity of spectral lines are expressed in terms of oscillator strengths using the relation [14].

$$f_{\text{expt.}} = 4.318 \times 10^{-9} \int \epsilon(\nu) d\nu \tag{1}$$

where, $\epsilon(\nu)$ is molar absorption coefficient at a given energy ν (cm⁻¹), to be evaluated from Beer–Lambert law.

Under Gaussian Approximation, using Beer–Lambert law, the observed oscillator strengths of the absorption bands have been experimentally calculated, using the modified relation [15].

$$P_m = 4.6 \times 10^{-9} \times \frac{1}{cl} \log \frac{I_0}{I} \times \Delta\nu_{1/2} \tag{2}$$

where c is the molar concentration of the absorbing ion per unit volume, l is the optical path length, $\log I_0/I$ is absorbivity or optical density and $\Delta\nu_{1/2}$ is half band width.



3.2. Judd-Ofelt Intensity Parameters

According to Judd [16] and Ofelt [17] theory, independently derived expression for the oscillator strength of the induced forced electric dipole transitions between an initial J manifold $|4f^N(S, L) J\rangle$ level and the terminal J' manifold $|4f^N(S', L') J'\rangle$ is given by:

$$\frac{8\pi^2 mc \bar{\nu}}{3h(2J+1)n} \frac{1}{n} \left[\frac{(n^2+2)^2}{9} \right] \times S(J, J') \tag{3}$$

where, the line strength S (J, J') is given by the equation

$$S(J, J') = e^2 \sum_{\lambda=2, 4, 6} \Omega_{\lambda} \langle 4f^N(S, L) J || U^{(\lambda)} || 4f^N(S', L') J' \rangle^2 \tag{4}$$

In the above equation m is the mass of an electron, c is the velocity of light, ν is the wave number of the transition, h is Planck's constant, n is the refractive index, J and J' are the total angular momentum of the initial and final level respectively, Ω_{λ} ($\lambda = 2, 4$ and 6) are known as Judd-Ofelt intensity parameters.

3.3. Radiative Properties

The Ω_{λ} parameters obtained using the absorption spectral results have been used to predict radiative properties such as spontaneous emission probability (A) and radiative life time (τ_R), and laser parameters like fluorescence branching ratio (β_R) and stimulated emission cross section (σ_p).

The spontaneous emission probability from initial manifold $|4f^N(S', L') J'\rangle$ to a final manifold $|4f^N(S, L) J\rangle$ is given by:

$$A[(S', L') J'; (S, L) J] = \frac{64 \pi^2 \nu^3}{3h(2J'+1)} \left[\frac{n(n^2+2)^2}{9} \right] \times S(J', \bar{J}) \tag{5}$$

Where, $S(J', J) = e^2 [\Omega_2 || U^{(2)} ||^2 + \Omega_4 || U^{(4)} ||^2 + \Omega_6 || U^{(6)} ||^2]$

The fluorescence branching ratio for the transitions originating from a specific initial manifold $|4f^N(S', L') J'\rangle$ to a final manifold $|4f^N(S, L) J\rangle$ is given by

$$\beta[(S', L') J'; (S, L) J] = \sum_{S, L, J} \frac{A[(S', L) J]}{A[(S', L') J'; (\bar{S}, \bar{L}) \bar{J}]} \tag{6}$$

where, the sum is over all terminal manifolds.

The radiative life time is given by

$$\tau_{rad} = \sum_{S, L, J} A[(S', L') J'; (S, L) J] = A_{Total}^{-1} \tag{7}$$

where, the sum is over all possible terminal manifolds. The stimulated emission cross-section for a transition from an initial manifold $|4f^N(S', L') J'\rangle$ to a final manifold $|4f^N(S, L) J\rangle$ is expressed as

$$\sigma_p(\lambda_p) = \left[\frac{\lambda_p^4}{8\pi c n^2 \Delta\lambda_{eff}} \right] \times A[(S', L') J'; (\bar{S}, \bar{L}) \bar{J}] \tag{8}$$

where, λ_p the peak fluorescence wavelength of the emission band and $\Delta\lambda_{eff}$ is the effective fluorescence line width.

3.4 Nephelauxetic Ratio (β') and Bonding Parameter ($b^{1/2}$)

The nature of the R-O bond is known by the Nephelauxetic Ratio (β') and Bonding Parameters ($b^{1/2}$), which are computed by using following formulae [18, 19]. The Nephelauxetic Ratio is given by

$$\beta' = \frac{\nu_g}{\nu_a} \tag{9}$$

where, ν_a and ν_g refer to the energies of the corresponding transition in the glass and free ion, respectively. The value of bonding parameter ($b^{1/2}$) is given by $b^{1/2} = \left[\frac{1-\beta'}{2} \right]^{1/2}$ (10)

IV. RESULT AND DISCUSSION

4.1. XRD Measurement

Figure 1 presents the XRD pattern of the samples containing show no sharp Bragg’s peak, but only a broad diffuse hump around low angle region. This is the clear indication of amorphous nature with in the resolution limit of XRD instrument.

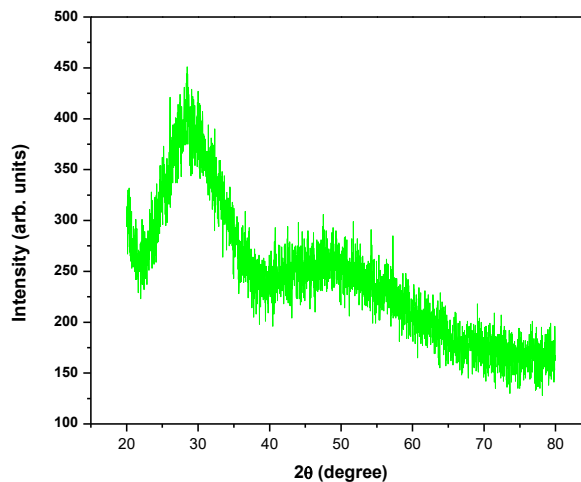


Fig.1: X-ray diffraction pattern of ZLSLTAP PR (01) glass.

4.2 Thermal Properties

Differential thermal analysis checks the heat absorbed by glass samples during heating or cooling. Fig. 2 depicts the DTA thermogram of powdered YZLPCABP sample. The glass transition temperature (T_g), onset crystallization temperature (T_c), crystallization temperature (T_p), melting temperature(T_m), thermal stability (T_s), thermal stability parameter(S),Hurbe’s criterion (H_r) and reduced glass transition temperature(T_{rg}) were calculated[20]. All the determined thermal parameters are given in table 2.

Glass samples	T _g (°C)	T _c (°C)	T _p (°C)	T _m (°C)	T _s (°C)	H _r (°C)	S(°C)	T _{rg} (°C)
ZLSLTAP HO(01)	374	505	545	682	131	0.226	14.01	0.548
ZLSLTAP HO(01)	376	508	548	686	132	0.225	14.04	0.548
ZLSLTAP HO(01)	378	511	550	688	133	0.220	13.72	0.549

$$\text{Thermal stability (T}_s\text{)} = T_c - T_g \quad (11)$$

$$\text{Hruby's criterion (H}_r\text{)} = [(T_p - T_c) / (T_m - T_c)] \quad (12)$$

$$\text{Reduced glass transition temperature (T}_{rg}\text{)} = T_g / T_m \quad (13)$$

$$\text{Thermal stability parameter (S)} = [(T_p - T_c) \times (T_c - T_g)] / T_g \quad (14)$$

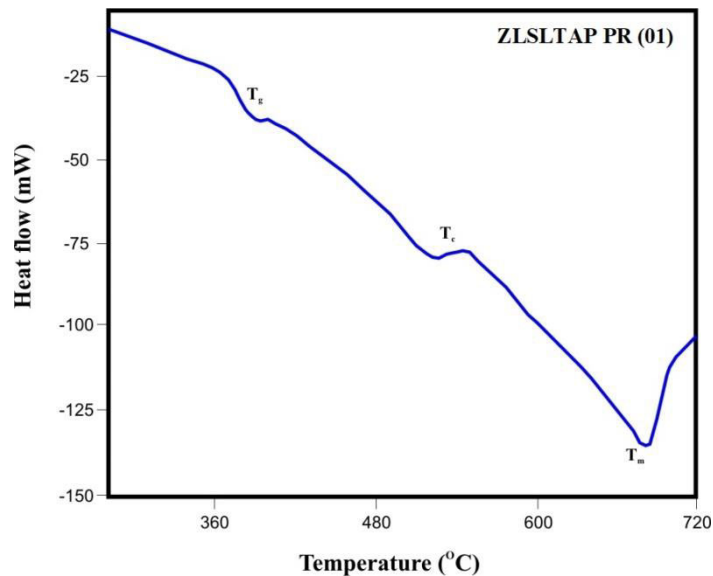


Fig.2: DTA curve of ZLSLTAP PR(01) glass.

4.3 FTIR Transmission spectra

The FTIR spectrum of ZLSLTAP glass samples in the wave number range 450-1300cm⁻¹ is presented in Fig.3 and the possible mechanism bands are tabulated in Table 3

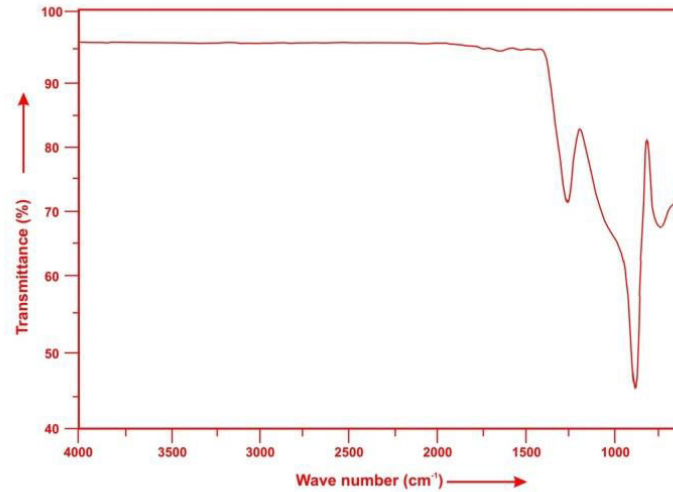


Fig. 3: FTIR spectrum of ZLSLTAP HO (01) glass.

Table3. Assignment of infrared transmission bands of ZLSLTAP HO (01) glass.

Peak position(cm ⁻¹)	Band Assignment
~740	symmetric stretching vibration of P-O-P bonding [21]
~890	Asymmetric stretching vibration of P-O-P bonds [21]
~1269	Asymmetric stretching vibration of P=O and O-P-O bonds [21]

4.4. Absorption spectra

The absorption spectra of ZLSLTAP (PR) glasses, consists of absorption bands corresponding to the absorptions from the ground state ³H₄ of Pr³⁺ ions. Eight absorption bands have been observed from the ground state ³H₄ to excited states ³F₂, ³F₃, ³F₄, ¹G₄, ¹D₂, ³P₀, ³P₁ and ³P₂ for Pr³⁺ doped ZLSLTAP (PR) glasses.

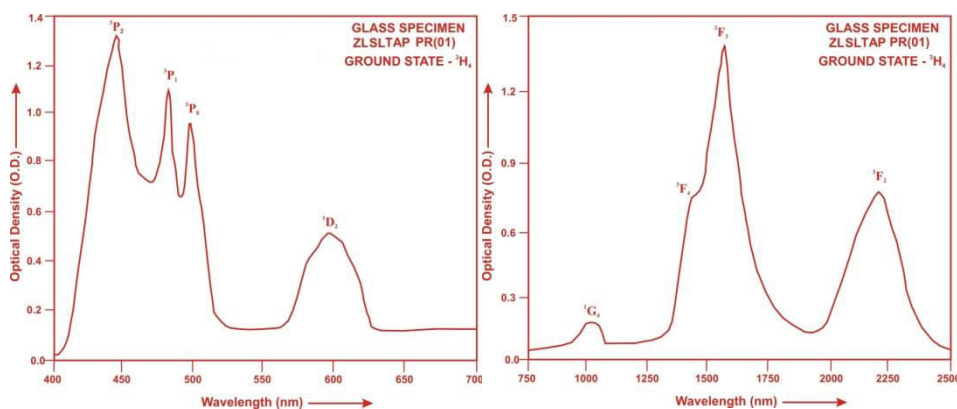


Fig.4: Absorption spectra of ZLSLTAP PR (01) glass.

The experimental and calculated oscillator strengths for Pr³⁺ ions in zinc lithium sodium lead tungsten aluminophosphate glasses are given in Table 4



Table 4. Measured and calculated oscillator strength ($P^m \times 10^{+6}$) of Pr^{3+} ions in ZLSLTAP glasses.

Energy level	Glass ZLSLTAP (PR01)		Glass ZLSLTAP (PR1.5)		Glass ZLSLTAP (PR02)	
3H_4	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$
3F_2	4.45	3.68	3.65	3.02	2.55	2.05
3F_3	6.76	5.84	5.85	4.99	4.85	4.06
3F_4	4.39	3.73	3.45	3.13	2.59	2.59
1G_4	0.45	0.31	0.35	0.26	0.24	0.21
1D_2	2.43	1.05	2.09	0.89	1.64	0.73
3P_0	4.48	1.20	3.69	1.20	2.59	0.90
3P_1	4.73	2.23	3.84	2.11	2.73	21.61
3P_2	12.68	3.48	11.43	2.96	10.52	2.45
R.m.s.deviation	3.6290		3.2319		2.9784	

The various energy interaction parameters like Slater-Condon parameters F_k ($k=2, 4, 6$), Lande' parameter ξ_{4f} and Racah parameters E^k ($k=1, 2, 3$) have been computed. Computed values of Slater-Condon, Lande', Racah, nephelauxetic ratio and bonding parameter for Pr^{3+} doped ZLSLTAP glass specimens are given in **Table 5**.

Table5. Computed values of Slater-Condon, Lande', Racah, nephelauxetic ratio and bonding parameter for Pr^{3+} doped ZLSLTAP glass specimens.

Parameter	Free ion	ZLSLTAP (PR01)	ZLSLTAP(PR1.5)	ZLSLTAP (PR02)
$F_2(cm^{-1})$	322.09	299.99	300.03	299.99
$F_4(cm^{-1})$	44.46	44.25	44.28	44.25
$F_6(cm^{-1})$	4.867	4.411	4.413	4.411
$\xi_{4f}(cm^{-1})$	741.00	858.76	858.24	858.76
$E^1(cm^{-1})$	4728.92	4450.08	4451.79	4450.08
$E^2(cm^{-1})$	24.75	22.01	22.01	22.01
$E^3(cm^{-1})$	478.10	454.69	454.73	454.69
F_4/F_2	0.13805	0.1475	0.1476	0.1475
F_6/F_2	0.01511	0.0147	0.0147	0.0147
E^1/E^3	9.8911	9.787	9.790	9.787
E^2/E^3	0.0518	0.0484	0.0484	0.0484

β'		0.8885	0.8889	0.8885
$b^{1/2}$		0.2362	0.2358	0.2362

Table 6. Judd-Ofelt intensity parameters for Pr³⁺ doped ZLSLTAP glass specimens.

Glass Specimen	$\Omega_2(\text{pm}^2)$	$\Omega_4(\text{pm}^2)$	$\Omega_6(\text{pm}^2)$	Ω_4/Ω_6
ZLSLTAP (PR01)	2.803	1.693	5.320	0.3182
ZLSLTAP (PR1.5)	2.139	1.687	4.449	0.3792
ZLSLTAP (PR02)	1.285	1.267	3.709	0.3416

4.5 Excitation Spectrum

Excitation spectra of ZLSLTAP PR (01) glass recorded at the emission wavelength 395 nm is depicted as figure 5. The excitation spectra consists of three peaks corresponding to the transitions from the ground state ³H₄ to the various excited states ³P₂, ³P₁ and ³P₀ at the wavelengths of 448, 467 and 485 nm respectively. Among these, a prominent excitation band at 448 nm has been selected for the measurement of emission spectrum of Pr³⁺ glass.

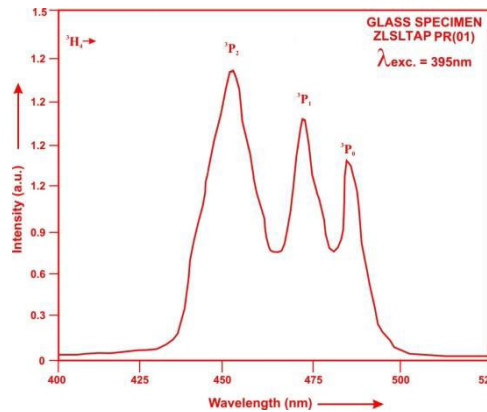


Fig.5: Excitation spectra of ZLSLTAP PR (01) glass.

4.6. Fluorescence Spectrum

The fluorescence spectrum of Pr³⁺ doped in zinc lithium sodium lead tungsten aluminophosphate glass is shown in Figure 6. There are nine broad bands (³P₀→³H₄), (³P₀→³H₅), (¹D₂→³H₄), (³P₀→³H₆), (³P₀→³F₂), (³P₁→³F₃), (¹D₂→³H₅), (³P₀→³F₄) and (¹G₄→³H₅) respectively for glass specimens.

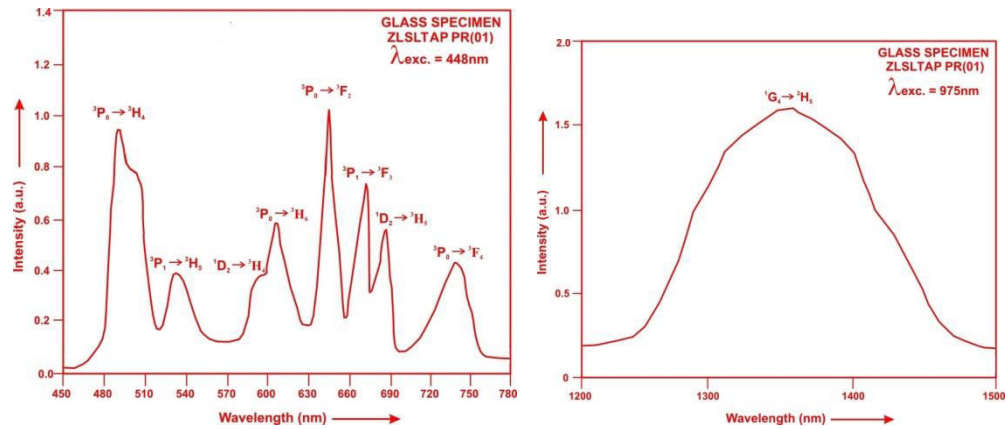


Fig.6: Fluorescence spectrum of ZLSLTAP PR (01) glass.

Table 7. Emission peak wave lengths (λ_p), radiative transition probability (A_{rad}), branching ratio (β_R), stimulated emission crosssection (σ_p), and radiative life time (τ) for various transitions in Pr^{3+} doped ZLSLTAP glasses.

Transition	ZLSLTAP PR 01					ZLSLTAP PR 1.5				ZLSLTAP PR 02			
	λ_{max} (nm)	$A_{rad}(s^{-1})$	β	$\sigma_p (10^{-20} cm^2)$	$\tau_R(\mu s)$	$A_{rad}(s^{-1})$	β	$\sigma_p (10^{-20} cm^2)$	$\tau_R(\mu s)$	$A_{rad}(s^{-1})$	β	$\sigma_p (10^{-20} cm^2)$	$\tau_R (10^{-20} cm^2)$
$^3P_0 \rightarrow ^3H_4$	485	1043.59	0.1011	0.374	968.92	1042.56	0.1191	0.424	1142.53	784.05	0.1271	0.378	1622.27
$^3P_1 \rightarrow ^3H_5$	532	2139.30	0.2073	0.418		1964.77	0.2245	0.405		1552.81	0.2519	0.340	
$^1D_2 \rightarrow ^3H_4$	599	565.39	0.0548	0.219		481.53	0.0550	0.192		395.90	0.0642	0.168	
$^3P_0 \rightarrow ^3H_6$	602	503.46	0.0488	0.269		421.86	0.0482	0.239		352.37	0.0572	0.229	
$^3P_0 \rightarrow ^3F_2$	645	2292.51	0.2221	2.176		1753.67	0.2004	1.854		1055.57	0.1712	1.326	
$^3P_1 \rightarrow ^3F_3$	676	3306.75	0.3204	1.756		2661.25	0.3041	1.511		1686.57	0.2736	1.015	
$^1D_2 \rightarrow ^3H_5$	685	5.586	0.0005	0.0061		5.223	0.0006	0.0063		4.084	0.0007	0.0006	
$^3P_0 \rightarrow ^3F_4$	730	192.89	0.0187	0.1418		192.51	0.0220	0.1478		144.92	0.0235	0.119	
$^1G_4 \rightarrow ^3H_5$	1350	271.31	0.0263	0.865		229.12	0.0262	0.7416		187.92	0.0305	0.625	

V. CONCLUSION

In the present study, the glass samples of composition (35-x): P₂O₅:10ZnO:10Li₂O:10Na₂O:10PbO:15WO₃:10Al₂O₃:xPr₆O₁₁ (where x =1, 1.5, 2 mol %) have been prepared by melt-quenching method. The value of stimulated emission cross-section (σ_p) is found to be maximum for the transition (³P₀→³F₂) for glass ZLSLTAP (PR 01), suggesting that glass ZLSLTAP (PR 01) is better compared to the other two glass systems ZLSLTAP (PR1.5) and ZLSLTAP (PR02).

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