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Spectral, Thermal And Raman Analysis Of Ho^{3+}Doped Borophosphate Glasses With Large Balaji Parameter

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Abstract
Glasses samples containing Ho^{3+} in Yttrium Zinc Lithium Sodalime Barium Antimony Borophosphate Glasses (20-x) P_2O_5: 10ZnO: 10Li_2O: 10CaO: 10Na_2O: 10BaO: 10Sb_2O_3: 10Y_2O_3: 10B_2O_3: xHo_2O_3 (where x=1, 1.5,2 mol %) have been prepared by melt-quenching method. The amorphous nature of the prepared glass samples was confirmed by X-ray diffraction. Optical absorption, Excitation, fluorescence, DTA thermogram and Raman spectra were recorded at room temperature for all glass samples. Judd-Ofelt intensity parameters Ω_λ (λ=2, 4 and 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 (A), branching ratio (β), radiative life time (τ_R) and stimulated emission cross–section (σ_p) of various emission lines have been evaluated.

Keywords: *YZLSLBABP Glasses, Thermal Properties, Raman Spectroscopy, Judd-Ofelt Theory,*

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I. Introduction

Rare earth glasses have attracted much attention, because they have large applications in many fields, such as optical fiber amplifiers, sensors, glass lasers, solid state batteries, optical fiber amplifiers, electro-luminescent devices, up-conversion lasers, memory devices and sensors [1–5].

Among different glasses, phosphate glasses have high transparency, high refractive index and low dispersion rates. Phosphate glasses possess interesting properties high density, high solubility, non-linear optical susceptibilities, and low melting temperature [6-8]. The addition of network modifier (NWF) Li_2O is to improve both electrical and mechanical properties of such glasses. Zinc oxide is added in the glass matrix to increase glass forming ability and to ensure low rates of crystallization in the glass system [9,10]. The host glass materials should have high refractive index with good chemical and thermal stability.

Na_2O is often added to modify the glass structure that improves the physical properties, chemical durability and mechanical strength.Borophosphate glasses provide a number of advantages over other oxide glasses, including strong thermal stability, high thermal stability and high gain density due to high solubility of rare earth ions in phosphate network [11-14].The low glass melting temperature, high thermal stability, good rare earth ion solubility and the optical fiber development compatibility makes the borophosphate glasses suitable candidates for laser and photonic applications [15-18].

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, fluorescence, Raman spectra and DTA thermogram of Ho^{3+} doped yttrium zinc lithium sodalime barium antimony borophosphate 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, λ=2,4 and 6).

II. Experimental Techniques

Preparation of glasses

The following Ho^{3+}doped borophosphate glass samples (20-x)P_2O_5: 10ZnO: 10Li_2O: 10CaO: 10Na_2O: 10BaO: 10Sb_2O_3:10Y_2O_3:10B_2O_3 x Ho_2O_3. (where x=1,1.5 and 2 mol%) have been prepared by melt-quenching method. Analytical reagent grade chemical used in the present study consist of P_2O_5, ZnO, Li_2O,CaO, Na_2O, BaO, Sb_2O_3, Y_2O_3,B_2O_3, and Ho_2O_3. They were thoroughly mixed by using an agate pestle mortar. then melted at 1050^0C by an electrical muffle furnace for 2h., After complete melting, the melts were quickly poured in to a preheated stainless steel mould and annealed at temperature of 250^0C 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 %)
YZLSLBABP (UD)	$20P_2O_5$:10ZnO: 10Li_2O: 10CaO: 10Na_2O: 10BaO: 10Sb_2O_3: 10Y_2O_3:10B_2O_3
YZLSLBABP HO (1.0)	$19P_2O_5$: 10ZnO: 10Li_2O: 10CaO: 10Na_2O: 10BaO: 10Sb_2O_3: 10Y_2O_3: 10B_2O_3: 1 Ho_2O_3
YZLSLBABPHO (1.5)	18.5 P_2O_5: 10ZnO: 10Li_2O: 10CaO: 10Na_2O: 10BaO: 10Sb_2O_3:10Y_2O_3:10B_2O_3:1.5 Ho_2O_3
YZLSLBABP HO (2.0)	$18P_2O_5$: 10ZnO: 10Li_2O: 10CaO: 10Na_2O: 10BaO: 10Sb_2O_3:10Y_2O_3:10B_2O_3:2 Ho_2O_3

YZLSLBABP (UD) -Represents undoped Yttrium Zinc Lithium Sodalime Barium Antimony Borophosphate glass specimen

YZLSLBABP (HO)-Represents Ho^{3+} doped Yttrium Zinc Lithium Sodalime Barium Antimony Borophosphate glass specimens

III. Theory

Oscillator Strength

The intensity of spectral lines are expressed in terms of oscillator strengths using the relation [19].

$$f_{expt.} = 4.318 \times 10^{-9} \int \varepsilon(\nu)\, d\nu \qquad (1)$$

where, ε (*ν)* 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 [20], using the modified relation:

$$P_m = 4.6 \times 10^{-9} \times \frac{1}{cl} \log \frac{I_0}{I} \times \Delta\upsilon_{1/2} \qquad (2)$$

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

Judd-Ofelt Intensity Parameters

According to Judd [21] and Ofelt [22] 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:

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

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

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

λ =2, 4, 6

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 (λ=2,4and 6) are known as Judd-Ofelt intensity.

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:

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

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 many fold $|4f^N (S, L) J\rangle$ is given by

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

S L J

where, the sum is over all terminal manifolds.

The radiative life time is given by

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

S L J

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

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

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

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 [23, 24]. The Nephelauxetic Ratio is given by

$$\beta' = \frac{\nu_g}{\nu_a} \quad (9)$$

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

$$b^{1/2} = \left[\frac{1-\beta'}{2}\right]^{1/2} \quad (10)$$

IV. Result And Discussion

XRD Measurement

Fig.1 presents the XRD pattern of the sample contain – P_2O_5 which is show no sharp Bragg's peak, but only a broad diffuse hump around low angle region. This is the clear indication of amorphous nature within the resolution limit of XRD instrument.

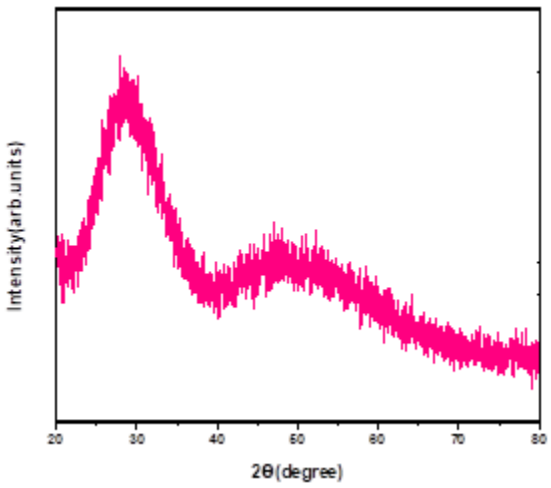

Fig.1: X-ray diffraction pattern of YZLSLBABP HO (1.0) Glass.

Thermal Property

Differential thermal analysis checks the heat absorbed by glass samples during heating or cooling. Fig.2 depicts the DTA thermogram of powdered YZLSLBABP HO (1.0) 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), Balaji Parameter (B_p) and reduced glass transition temperature (T_{rg}) were calculated. All the determined thermal parameters are given in table 2.

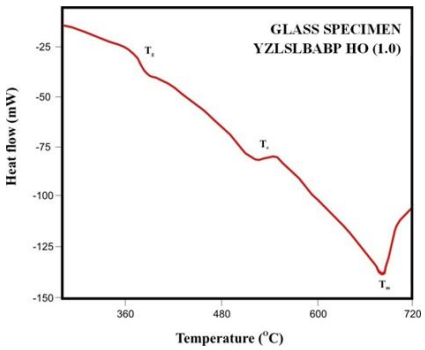

Fig.2: DTA curve of YZLSLBABP HO (1.0) glass.

Table 2: Thermal parameters determined from the DTA traces of YZLSLBABP HO glasses.

Sample Name	T_g (°C)	T_c(°C)	T_p(°C)	T_m(°C)	T_s(°C)	B_P(°C)	H_r(°C)	T_{rg}(°C)
YZLSLBABP HO (1.0)	376	509	545	685	133	3.694	0.208	0.549
YZLSLBABP HO (1.5)	380	510	547	688	130	3.514	0.219	0.552
YZLSLBABP HO (2.0)	384	512	556	692	128	2.909	0.249	0.555

The thermal stability of the glass samples can be calculated by difference between onset crystallization temperature and transition temperature [25].

Thermal stability (T_s) =T_c-T_g (11)

Balaji Parameter can be calculated using

Balaji Parameter (B_p) = $[(T_c-T_g)/(T_p-T_c)$ (12)

Hruby's criterion is calculated using the Hurby's relation [26].

Hruby's criterion (H_r) = $[(T_p-T_c)/(T_m-T_c)$ (13)

Reduced glass transition temperature is given as [27].

Reduced glass transition temperature (T_{rg}) =T_g/T_m (14)

Raman spectra

The Raman spectrum of yttrium zinc lithium sodalime barium antimony borophosphate

YZLSLBABP HO (1.0) glass specimen is recorded and is shown in Fig.3. The spectrum peaks located at 397 and 775 cm^{-1}. The band at 397 cm^{-1} is related to the bending motion of phosphate polyhedral PO_4 units with cation like ZnO as the modifier. The broad band at 775cm^{-1} is due to symmetric stretching of (P–O–P) bridging oxygen bonds in $(P_2O_7)_4$ units.

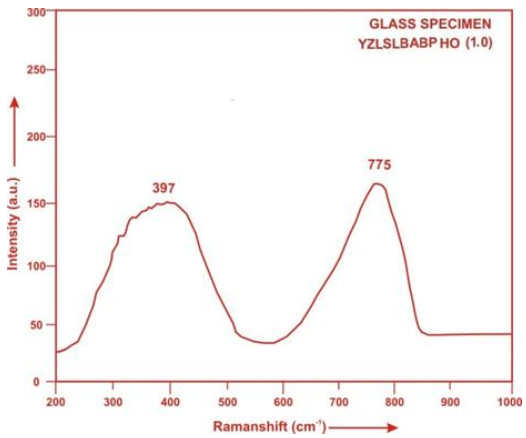

Fig.3: Raman spectrum of YZLSLBABP HO (1.0) glass.

Absorption Spectrum

The absorption spectra of Ho^{3+}doped YZLSLBABP HO (1.0) glass specimens have been presented in Fig.4 in terms of optical density versus wavelength. Twelve absorption bands have been observed from the ground state $^5I_8$ to excited states $^5I_5$, $^5I_4$, $^5F_5$, $^5F_4$, $^5F_3$, $^3K_8$, $^5G_6$, $(^5G,^3G)_5$, $^5G_4$, $^5G_2$, $^5G_3$, and$^3F_4$ for Ho^{3+} doped YZLSLBABP HO (1.0) glass.

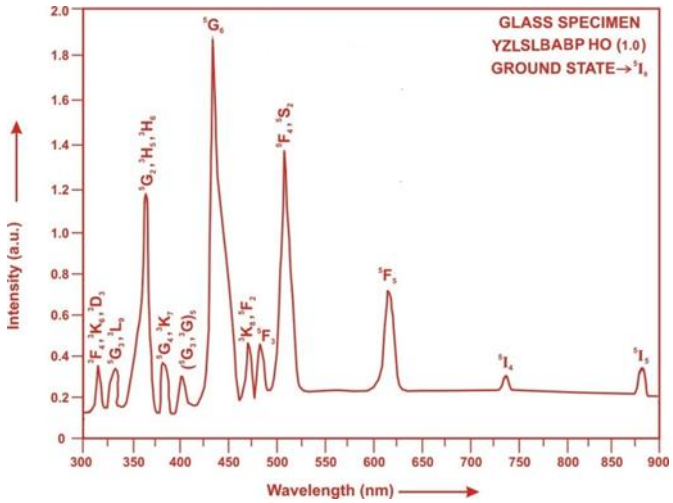

Fig.4: Absorption spectrum of YZLSLBABP HO (1.0) glass.

The experimental and calculated oscillator strength for Ho^{3+} ions in YZLSLBABP glasses are given in **Table 3.**

Table 3: Measured and calculated oscillator strength ($P_m \times 10^{+6}$) of Ho^{3+}ions in YZLSLBABP glasses.

Energy level from $^5I_8$	Glass YZLSLBABP HO (1.0)		Glass YZLSLBABP HO (1.5)		Glass YZLSLBABP HO (2.0)	
	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$
$^5I_5$	0.52	0.25	0.50	0.25	0.47	0.25
$^5I_4$	0.08	0.02	0.07	0.02	0.05	0.02
$^5F_5$	3.78	2.86	3.75	2.83	3.71	2.80
$^5F_4$	4.78	4.44	4.75	4.40	4.71	4.38
$^5F_3$	1.68	2.47	1.64	2.45	1.62	2.44
$^3K_8$	1.47	2.04	1.43	2.01	1.40	1.99
$^5G_6$	26.65	26.59	25.52	25.48	24.62	24.60
$(^5G,^3G)_5$	3.9	1.71	3.86	1.69	3.82	1.67
$^5G_4$	0.09	0.63	0.08	0.62	0.06	0.61
$^5G_2$	5.96	5.64	5.92	5.44	5.87	5.28
$^5G_3$	1.58	1.44	1.55	1.41	1.51	1.39
$^3F_4$	1.42	4.23	1.38	4.18	1.33	4.12
r.m.s. deviation	±1.1221		±1.1221		±1.1225	

Computed values of F_2, Lande′ parameter (ξ_{4f}**),** Nephlauxetic ratio (β') and bonding parameter ($b^{1/2}$) for Ho^{3+} ions in YZLSLBABP glass specimen are given in Table 4.

Table 4: F_2, ξ_{4f}, β' and $b^{1/2}$ parameters for Holmium doped glass specimen.

Glass Specimen	F_2	ξ_{4f}	β'	$b^{1/2}$
Ho^{3+}	358.82	1258.16	0.9337	0.1821

In the Yttrium Zinc Lithium Sodalime Barium Antimony Borophosphate (YZLSLBABP) Ω_2, Ω_4 and Ω_6 parameters decrease with the increase of x from 1 to 2 mol%. The order of magnitude of Judd-Ofelt intensity parameters is $\Omega_2 > \Omega_6 > \Omega_4$ for all the glass specimens. The spectroscopic quality factor (Ω_4/Ω_6) related with the rigidity of the glass system has been found to lie between 0.5924 and 0.6030 in the present glasses.
The values of Judd-Ofelt intensity parameters are given in **Table 5.**

Table 5: Judd-Ofelt intensity parameters for Ho^{3+} doped YZLSLBABP glass specimens.

Glass Specimen	$\Omega_2(pm^2)$	$\Omega_4(pm^2)$	$\Omega_6(pm^2)$	Ω_4/Ω_6	Ref.
YZLSLBABP HO (1.0)	6.589	1.373	2.277	0.6030	P.W.
YZLSLBABP HO (1.5)	6.268	1.350	2.259	0.5976	P.W.
YZLSLBABP HO (2.0)	6.017	1.330	2.245	0.5924	P.W.

TL (HO)	4.980	0.990	2.960	0.3345	[28].
LLF (HO)	2.430	1.670	1.840	0.9076	[29].
ZLAABP (HO)	5.649	1.225	2.044	0.5993	[30].

Excitation Spectrum

The Excitation spectrum of YZLSLBABP HO (1.0) glass has been presented in Fig.5 in terms of Excitation Intensity versus wavelength. The excitation spectrum was recorded in the spectral region 325–525 nm fluorescence at 545nm having different excitation band centered at 349,419, 452, 473and 486 nm are attributed to the $^5G_3$, (^5G, $^3G)_5$, $^5G_6$, $^3K_8$ and $^5F_3$ transitions, respectively. The highest absorption level is $^5G_6$ and is at 452nm.So this is to be chosen for excitation wavelength.

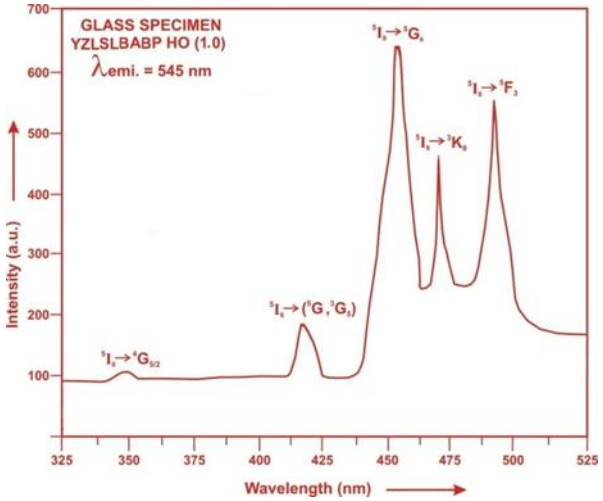

Fig.5: Excitation spectrum of YZLSLBABP HO (1.0) glass.

Fluorescence Spectrum

The fluorescence spectra of Ho^{3+}doped YZLSLBABP HO (1.0) glass specimens have been presented in Fig.6. There are eleven broad bands observed in the Fluorescence spectrum of Ho^{3+}doped yttrium zinc lithium sodalime barium antimony borophosphate glass. The wavelengths of these bands along with their assignments are given in Table 6. The peak with maximum emission intensity appears at 2035 nm and corresponds to the ($^5I_7 \rightarrow {}^5I_8$) transition.

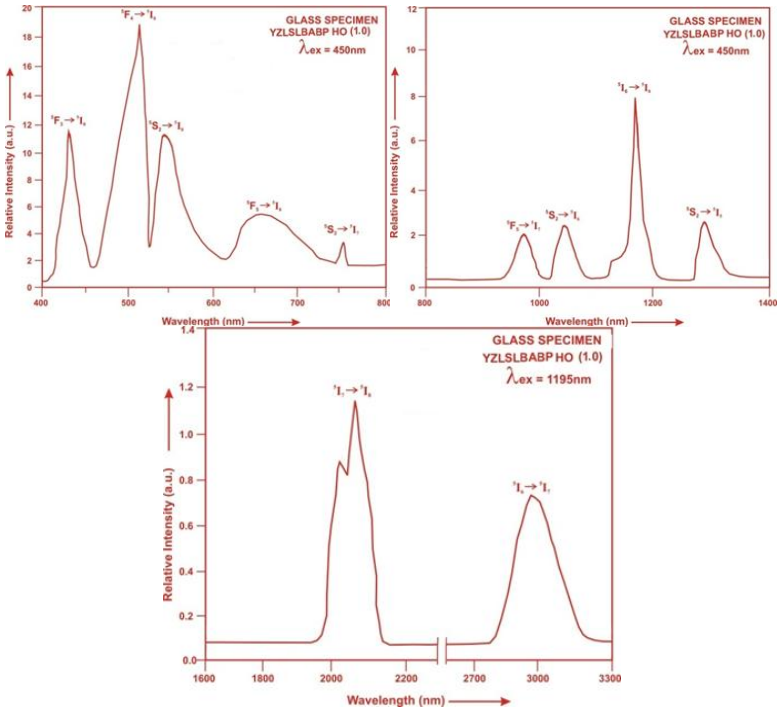

Fig.6: Fluorescence spectrum of YZLSLBABP HO (1.0) glass.

Table 6: Emission peak wave lengths (λ_p),radiative transition probability (A_{rad}),branching ratio (β), stimulated emission cross-section(σ_p) and radiative life time(τ_R) for various transitions in Ho^{3+} doped YZLSLBABP glasses.

Transition		YZLSLBABP HO(1.0)				YZLSLBABP HO(1.5)				YZLSLBABP HO(2.0)			
	λ_{max} (nm)	A_{rad}(s^{-1})	β	σ_p (10^{-20} cm^2)	τ_R(μs)	A_{rad}(s^{-1})	β	σ_p (10^{-20} cm^2)	τ_R (μs)	A_{rad}(s^{-1})	β	σ_p (10^{-20} cm^2)	τ_R (10^{-20} cm^2)
$^5F_3\rightarrow{}^5I_8$	435	4230.03	0.2487	0.589		4204.96	0.2490	0.572		4187.24	0.2492	0.555	
$^5F_4\rightarrow{}^5I_8$	501	6702.68	0.3941	1.239		6655.01	0.2940	1.198		6619.33	0.3940	1.170	
$^5S_2\rightarrow{}^5I_8$	555	1765.67	0.1038	0.445		1755.21	0.1039	0.434		1747.81	0.1040	0.418	
$^5F_5\rightarrow{}^5I_8$	652	1903.85	0.1119	0.740		1887.33	0.1117	0.726		1874.34	0.1115	0.710	
$^5S_2\rightarrow{}^5I_7$	761	1339.49	0.0788	1.123	5880.08	1331.55	0.0788	1.103	5920.98	1325.94	0.0789	1.0809	5951.60
$^5F_5\rightarrow{}^5I_7$	995	445.10	0.0262	1.212		439.82	0.0260	1.176		435.69	0.0253	1.144	
$^5I_6\rightarrow{}^5I_8$	1032	205.20	0.0121	0.696		203.82	0.0121	0.673		202.80	0.0121	0.661	
$^5S_2\rightarrow{}^5I_5$	1195	234.90	0.0138	1.211		233.12	0.0138	1.175		231.81	0.0138	1.143	
$^5S_2\rightarrow{}^5I_6$	1310	62.56	0.0037	0.621		62.17	0.0037	0.599		61.89	0.0037	0.584	
$^5I_7\rightarrow{}^5I_8$	2035	93.30	0.0055	4.693		92.54	0.0055	4.537		91.98	0.0055	4.424	
$^5I_6\rightarrow{}^5I_7$	2925	23.79	0.0014	3.960		23.56	0.0014	3.860		23.38	0.0014	3.756	

V. Conclusion

In the present study, the glass samples of composition (20-x) P2O5: 10ZnO: 10Li2O: 10CaO: 10Na2O: 10BaO: 10Sb2O3: 10Y2O3: 10B2O3: xHo2O3 (where x =1, 1.5and 2mol %) have been prepared by melt-quenching method. The value of stimulated emission cross-section (σp) is found to be maximum for the transition (5I7→5I8) for glass YZLSLBABP HO (1.0), suggesting that glass YZLSLBABP HO (1.0) is better compared to the other two glass systems YZLSLBABP HO (1.2) and YZLSLBABP HO (2.0). The large stimulated emission cross section in tellurite glasses suggests the possibility of utilizing these systems as laser materials. The Raman spectra of glasses revealed the presence of characteristic bonding vibrations of different functional groups.

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