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Spectroscopic properties and blue to ultraviolet upconversion for Ho^{3+} ions in $\text{Cs}_2\text{NaGdCl}_6$ crystals

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Abstract

A spectroscopic study of Ho^{3+} ions in $\text{Cs}_2\text{NaGdCl}_6$ is reported and four strong emission bands (peaked at 493, 545, 587 and 658 nm, respectively) are observed. The 587-nm emission band is assigned to $^5\text{G}_4 \rightarrow ^5\text{I}_6$ transition, in accordance with the assignment made by previous works. The other three emission bands are easily assigned to $^5\text{F}_3 \rightarrow ^5\text{I}_8$, $(^5\text{F}_4, ^5\text{S}_2) \rightarrow ^5\text{I}_8$, and $^5\text{F}_5 \rightarrow ^5\text{I}_8$ transitions, respectively. The emission intensity dependence on Ho^{3+} concentration is also presented. It is found that the first three emission bands are quenched almost completely when the concentration x of Ho^{3+} ions in $\text{Cs}_2\text{NaGd}_{1-x}\text{Ho}_x\text{Cl}_6$ is larger than 0.5, but that of 658-nm emission band always increases with concentration. The two efficient cross-relaxations $^5\text{F}_3 + ^5\text{I}_8 \rightarrow ^5\text{F}_5 + ^5\text{I}_7$ and $^5\text{G}_4 + ^5\text{I}_8 \rightarrow ^5\text{F}_3 + ^5\text{I}_7$ are responsible for the phenomenon. In addition, an exploration of the visible to ultraviolet energy upconversion in $\text{Cs}_2\text{NaGd}_{1-x}\text{Ho}_x\text{Cl}_6$ is carried out. Upon blue ($\lambda_{\text{exc}} = 457.9$ nm) laser excitation, a quite strong ultraviolet upconversion emission band, ranging between 320 and 340 nm, is observed in $\text{Cs}_2\text{NaGd}_{1-x}\text{Ho}_x\text{Cl}_6$ ($x = 0.05$ to 1.0) for the first time. This upconversion emission is attributed to the $^5\text{D}'_4 \rightarrow ^5\text{I}_5$ transition and the excited-state absorption (ESA) upconversion is presumed to be the main mechanism.

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1. Introduction

In the last few decades, in order to characterize the crystal-field (CF) energy levels and the intra-4f-shell spectroscopic transitions of lanthanide ions (Ln^{3+}) situated in a high-symmetry crystalline environment, many optical absorption and emission spectra have been reported for the cubic $\text{Cs}_2\text{NaLnCl}_6$ model system. In the $\text{Cs}_2\text{NaLnCl}_6$ crystal, each Ln^{3+} ion is hexacoordinated to six Cl^- ions forming an exact octahedral (O_h) symmetry. Therefore, due to the presence of the center of symmetry, electric dipole (ED) transitions are rigorously forbidden and the optical spectra are dominated by ED vibronic (EDV) transitions or weak magnetic dipole allowed transitions.

Experimental studies of the optical properties (absorption/emission) of $\text{Cs}_2\text{NaHoCl}_6$ and $\text{Cs}_2\text{Na(Y,Gd)Cl}_6:\text{Ho}^{3+}$ systems have also been reported [1–7]. However, so far, the CF energy levels of Ho^{3+} ions have only been well

assigned up to $26\,134\text{ cm}^{-1}$ in these systems [8], and the study of emission spectra only focuses on those originating from some lower luminescent levels, such as $^5\text{F}_3$, $^5\text{F}_4$, $^5\text{S}_2$ and $^5\text{F}_5$. In addition, some investigations of $\text{Ho}^{3+} \rightarrow \text{Ho}^{3+}$, Sm^{3+} , Yb^{3+} , Tb^{3+} [9] and $\text{Er}^{3+} \leftrightarrow \text{Ho}^{3+}$ [5] energy transfer in $\text{Ln}^{3+}:\text{Cs}_2\text{NaYCl}_6$ systems have also been carried out, also involving only $^5\text{F}_3$, $^5\text{F}_4$, $^5\text{S}_2$ and $^5\text{F}_5$ levels. In the present work, by using laser and high-pressure mercury lamp as excitation sources, we have obtained some emissions from higher luminescent levels and studied their concentration dependence for $\text{Cs}_2\text{NaGd}_{1-x}\text{Ho}_x\text{Cl}_6$ system at room temperature (RT), which, to our best knowledge, has not previously been reported.

Recently, exploration of the visible (VIS) to ultraviolet (UV) energy upconversion of trivalent lanthanide rare-earth (RE) ions has also attracted much attention, mainly because it is very useful for new tunable all-solid-state lasers in the ultraviolet spectral domains [10]. For Ho^{3+} ions, energy upconversion processes were investigated usually under excitation into $^5\text{F}_4$ multiplet ($\lambda_{\text{exc}} \approx 532$ nm) [11], $^5\text{F}_5$ multiplet ($\lambda_{\text{exc}} \approx 640$ nm) [12–18], $^5\text{I}_5$ multiplet ($\lambda_{\text{exc}} \approx 875$ nm) [19], or $^5\text{I}_6$ multiplet ($\lambda_{\text{exc}} \approx 1140$ nm)

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[17–21]. In this work we report the blue to ultraviolet energy upconversion under excitation into 5G_6 multiplet ($\lambda_{\text{exc}}=457.9$ nm) for the first time. The upconversion mechanism is also discussed.

2. Experimental

Powder samples of $\text{Cs}_2\text{NaGd}_{1-x}\text{Ho}_x\text{Cl}_6$ ($x=0.01, 0.05, 0.10, 0.15, 0.25, 0.5, 0.75, 1.0$) were prepared by method E of Morss et al., described in Ref. [22], by evaporating a solution of $2\text{Cs}^+-\text{Na}^+-(1-x)\text{Gd}^{3+}-x\text{Ho}^{3+}$ in HCl to dryness. To reduce their exposure to atmospheric water, all samples were stored in sealed quartz tubes. The Ho_2O_3 and Gd_2O_3 used were of 99.99% purity. All the experiments were conducted at room temperature. The excitation spectra were recorded by a 850 fluorescence spectrophotometer (Hitachi, Japan). A CR599-21 argon ion laser (Coherent, USA) and a high-pressure mercury lamp were used to excite the samples. The emission spectra were recorded using an HRD double grating monochromator (Jobin-Yvon, France).

3. Results and discussion

3.1. Concentration quenching of some emissions

The emission spectra in the range from 420 to 700 nm of Ho^{3+} ions in $\text{Cs}_2\text{NaGd}_{1-x}\text{Ho}_x\text{Cl}_6$ ($x=0.01, 0.05, 0.10, 0.15, 0.25, 0.50, 0.75, 1.0$) are shown in Fig. 1. Here a high-pressure mercury lamp (365 nm) is used as excitation source to match the second strongest excitation peak at 363 nm in the excitation spectrum of 658-nm emission from 5F_5 multiplet (Fig. 2). As mentioned above, all the

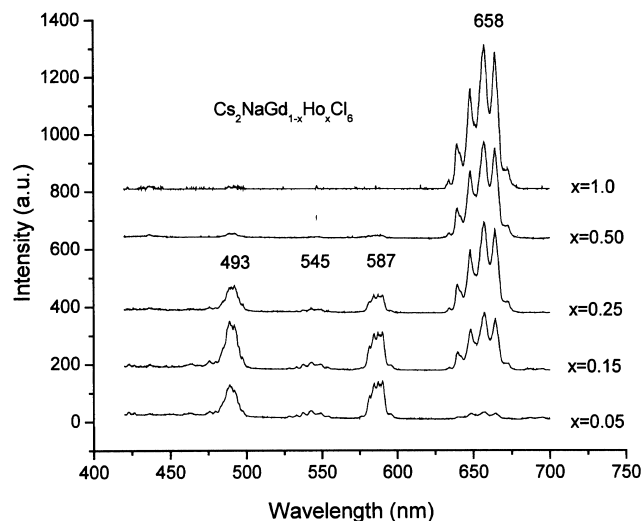


Fig. 1. Emission spectra of $\text{Cs}_2\text{NaGd}_{1-x}\text{Ho}_x\text{Cl}_6$ upon 365-nm excitation at room temperature.

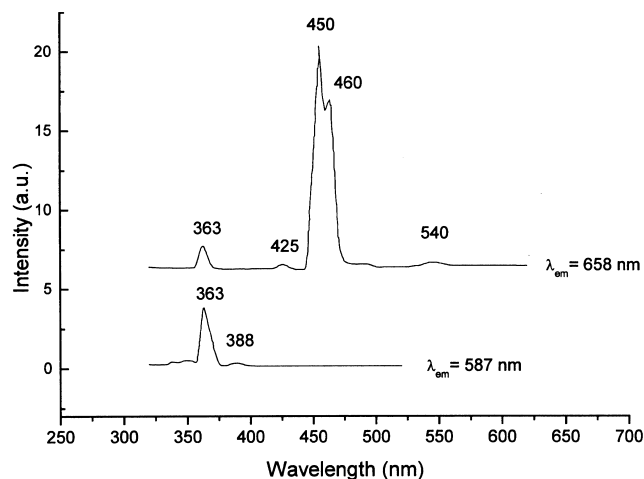


Fig. 2. Excitation spectra for 658- and 587-nm emission of Ho^{3+} ions in $\text{Cs}_2\text{NaGd}_{0.95}\text{Ho}_{0.05}\text{Cl}_6$, $T=300$ K.

transitions recorded in Fig. 1 (as well as in Figs. 3 and 4) are EDV transitions or magnetic dipole allowed transitions. In addition, the phonon side bands are also very notable in all these emission spectra. From Fig. 1 it can be seen that there are four strong emission bands with, respectively, peaks at 493, 545, 587 and 658 nm, three of which, at 493, 545 and 658 nm, can be easily assigned as $^5F_3 \rightarrow ^5I_8$, $^5F_4, ^5S_2 \rightarrow ^5I_8$, $^5F_5 \rightarrow ^5I_8$ transitions, respectively, according to many reports on Ho^{3+} ions. However, reports on 587-nm emission are very scarce. In order to examine its origin, an excitation spectrum of 587-nm emission is recorded and shown in Fig. 2. It is easy to see that the excitation peak at 388 nm corresponds to the absorption transition $^5I_8 \rightarrow ^5G_4 + ^3K_7$, so it is reasonable to assign the 587-nm emission band to $^5G_4 \rightarrow ^5I_6$ transition, which is also consistent with the assignments made in previous studies [23]. Moreover, upon 457.9-nm laser excitation, the emission spectra of Ho^{3+} in $\text{Cs}_2\text{NaGd}_{1-x}\text{Ho}_x\text{Cl}_6$ ($x=0.15$,

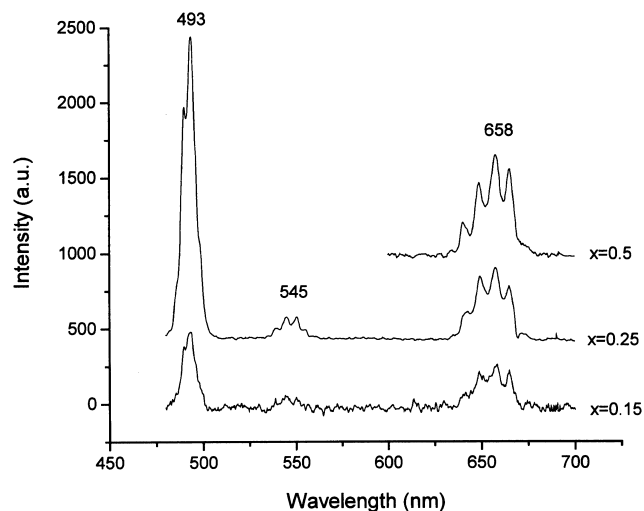


Fig. 3. Emission spectra of $\text{Cs}_2\text{NaGd}_{1-x}\text{Ho}_x\text{Cl}_6$ upon 457.9-nm laser excitation at room temperature.

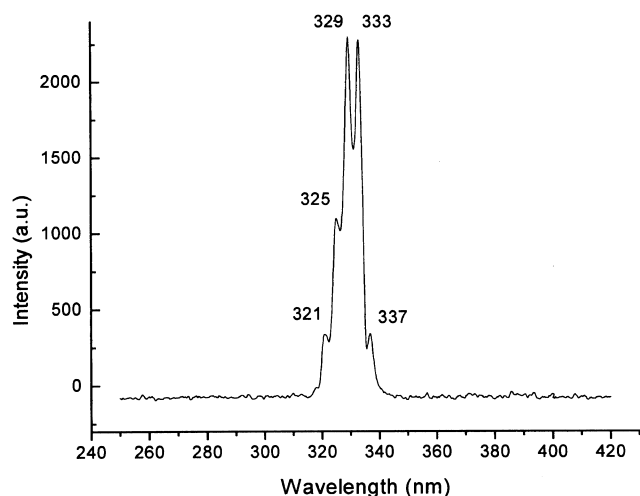


Fig. 4. Blue to ultraviolet energy upconversion fluorescence from the Ho^{3+} ions in $\text{Cs}_2\text{NaGdCl}_6$ under 457.9-nm blue laser excitation, $T=300$ K.

0.25, 0.50) (Fig. 3) consist of three emission bands with peaks at 493, 545 and 658 nm. With this excitation, the 587-nm emission band can not be detected, as the laser used can not excite the $^5\text{G}_4$ levels, confirming again our assignments.

It is worthy of note that the relative intensities of these four emission bands change remarkably with Ho^{3+} concentration (Fig. 1). For the first three emission bands, 493, 545 and 587 nm, when Ho^{3+} ions concentration x is <0.15 , their intensities increase with concentration, however when $x \geq 0.15$, a notable concentration quenching happens. When $x \geq 0.5$, these three emission bands are almost completely quenched, while for the 658-nm emission band, its intensity always increases with concentration. All this emission intensity quenching or increasing can be interpreted by using two very efficient cross relaxations: $^5\text{G}_4 + ^5\text{I}_8 \rightarrow ^5\text{F}_3 + ^5\text{I}_7$ and $^5\text{F}_3 + ^5\text{I}_8 \rightarrow ^5\text{F}_5 + ^5\text{I}_7$, with an energy mismatch of less than one phonon [24]. For high concentration samples both cross relaxations above become very efficient, resulting in the population of $^5\text{G}_4$ level relaxing rapidly to the $^5\text{F}_5$ level, and accordingly three emission bands with peaks at 493, 545 and 587 nm originating in $^5\text{F}_3$, $^5\text{F}_4$, $^5\text{S}_2$ and $^5\text{G}_4$ levels quench, while the emission intensity of the band with peak at 658 nm originating mainly from $^5\text{F}_5$ level increases observably.

3.2. Blue to ultraviolet energy upconversion

For $\text{Cs}_2\text{NaGd}_{1-x}\text{Ho}_x\text{Cl}_6$ ($x=0.05, 0.15, 0.5, 1.0$), upon pumping with 457.9-nm blue laser, all obtained upconversion emission spectra are quite similar (Fig. 4). The upconversion emission lies between 320 and 340 nm with 329 nm being the strongest, which has not previously been reported. Therefore, further investigation of this interesting phenomenon becomes very necessary. Firstly, this upconversion emission can be proved to be definitely from

Ho^{3+} ions as follows: (i) it is not from Gd^{3+} ions because it has also been observed in $\text{Cs}_2\text{NaHoCl}_6$ sample; and (ii) this emission is not laser plasma line or from Er^{3+} impurity ions because it has not been observed when a $\text{Cs}_2\text{NaYCl}_6:5\%\text{Er}$ sample is pumped with the same 457.9-nm laser.

The available data indicate that this upconversion emission does not come from the luminescent levels with energy lower than $34\,000\text{ cm}^{-1}$, so we think it must come from the luminescent levels of Ho^{3+} ion with higher energy. Table 1 shows the extended energy level data of Ho^{3+} ions in $\text{Cs}_2\text{NaGdCl}_6$ crystals, which are calculated using the F-shell empirical programs developed by Reid and the fitted parameters reported by Tanner [8]. In our case the relevant highest level will be at $\sim 44\,000\text{ cm}^{-1}$ when a two-photon upconversion process occurs, so Table 1 only shows the energy levels lower than $45\,000\text{ cm}^{-1}$. According to the energy level data in Table 1, we conclude that the upconversion emission corresponds to $^5\text{D}'_4 \rightarrow ^5\text{I}_5$ transition. The structure of this broad upconversion emission band (phonon side bands) is very complex. The energy level calculation mentioned above shows that the $^5\text{D}'_4$ state at $41\,290\text{ cm}^{-1}$ mainly consists of 32% $^5\text{D}_4$, 43% $^3\text{F}_4$ and 17% $^3\text{H}_4$, and that the $^5\text{I}_5$ state mainly consists of 90.4% $^5\text{I}_5$ and 7.3% $^3\text{H}_5$. So two kinds of channels by which the $^5\text{D}'_4 \rightarrow ^5\text{I}_5$ transition takes place are identified: (i) the phonon assisted (vibronically induced) electronic dipole transitions $^3\text{F}_4 \rightarrow ^3\text{H}_5$ and $^5\text{D}_4 \rightarrow ^5\text{I}_5$, which make main contributions to this upconversion emission transition; and (ii) the pure magnetic dipole and vibronic electronic dipole transition $^3\text{H}_4 \rightarrow ^3\text{H}_5$.

The excited-state absorption (ESA) of blue photons (457.9 nm) from $^5\text{F}_3$ level is believed to be the most probable upconversion mechanism in our case (Fig. 5). When using 457.9-nm ($21\,839\text{ cm}^{-1}$) blue laser as an excitation source, the first step absorption corresponds to the $^5\text{I}_8 \rightarrow ^5\text{G}_6$ transition, and then the population in $^5\text{G}_6$ state relax rapidly to the lower levels of $^5\text{F}_3$ multiplet, which can act as a metastable level because it has a intrinsic lifetime of $\approx 2.8\text{ ms}$ [3]. And then another laser photon (457.9 nm) can be absorbed directly from $^5\text{F}_3$ multiplet, reaching $^5\text{D}_3 + ^5\text{D}_1 + ^3\text{F}_3$ multiplet at $42\,548\text{ cm}^{-1}$ and then relaxing to the luminescent level $^5\text{D}'_4$.

Fig. 6 shows the plot of upconversion emission intensity I_{up} versus laser pump power I_0 . A slope of 1.0 is obtained when the laser beam is focused on the sample $\text{Cs}_2\text{NaGd}_{0.95}\text{Ho}_{0.05}\text{Cl}_6$. The experimental result seems to be a little strange, because in the ESA process where two photons are involved, a slope of 2 is expected (that is $I_{\text{up}} \propto I_0^2$). But actually the slope will change from 2 to 1 with increasing pump power for an ESA process, as concluded by Pollnau et al. after theoretical analysis and some experimental verification [25].

Now we will explain this unusual experimental result using a simple model. Let N_0 , N_1 , N_2 , N_3 , N_4 represent populations in the $^5\text{I}_8$, $^5\text{F}_3$, $^5\text{G}_6$, $^5\text{D}'_4$ and $^5\text{D}_3 + ^5\text{D}_1$ states,

Table 1

Calculated energy level data of Ho^{3+} in $\text{Cs}_2\text{NaGdCl}_6$ using the F-shell empirical programs developed by Reid and the fitted parameters reported by Tanner [8]

$^{2S+1}L_J$ level	Energy (cm^{-1})	$^{2S+1}L_J$ level	Energy (cm^{-1})	$^{2S+1}L_J$ level	Energy (cm^{-1})	$^{2S+1}L_J$ level	Energy (cm^{-1})	$^{2S+1}L_J$ level	Energy (cm^{-1})	$^{2S+1}L_J$ level	Energy (cm^{-1})
5I_8	0		18 520		26 069	5G_2	30 661	3P_0	36 048		38 894
	9		18 524		26 123		30 773				38 897
	35		18 558		26 134			1L_8	36 132		38 901
	208					3D_3	32 972	$+^3F_2$	36 166		38 903
	245	5F_3	20 398	$^5G'_5$	27 361		33 046		36 173		38 910
	281		20 541		27 456		33 173		36 202		
	281		20 618		27 500				36 217	3I_5	39 117
					27 568	3P_1	33 273		36 225		39 128
5I_7	5107	5F_2	20 978						36 235		39 227
	5112		21 103	3H_6	27 608	$^3M_{10}$	33 671		36 269		39 252
	5208				27 617		33 678		36 315		
	5226	3K_8	21 259		27 679		33 679			5D_3	39 529
	5238		21 260		27 787		33 697	3H_5	36 494	$+^3I_6$	39 566
	5271		21 280		27 879		33 706		36 622	$+^5D_1$	39 581
			21 296		27 937		33 750		36 793		39 626
5I_6	8620		21 353				33 751		36 838		39 690
	8628		21 370	$^5F'_2$	28 143		33 752				39 741
	8671		21 376		28 283		33 753	3L_7	37 629		39 762
	8713							$+^3P_2$	37 665		39 766
	8738	5G_6	21 848	3L_9	28 500	3L_8	33 844		37 684		39 800
	8762		21 889	$+^5G_3$	28 524		33 861		37 717		39 910
			21 941		28 555		33 883		37 786		
5I_5	11 191		22 100		28 616		33 999		37 842	$^5D'_4$	41 268
	11 237		22 190		28 650		34 135		37 844		41 278
	11 266		22 217		28 735		34 147		37 869		41 286
	11 291				28 759		34 166				41 326
		5F_1	22 247		28 807			3I_7	38 104		
5I_4	13 238				28 816	$^5G'_4$	34 566		38 117	5D_2	41 511
	13 241	5G_5	23 809		28 996		34 595		38 122		41 617
	13 242		23 820		29 014		34 597		38 185		
	13 446		23 930				34 602		38 205	5D_0	42 161
			23 943	3F_4	29 832				38 225		
5F_5	15 330			$+^3K_6$	29 892	3G_3	35 077			5D_3	42 548
	15 391	5G_4	25 650		29 920		35 161	3F_4	38 250	$+^5D_1$	42 674
	15 541		25 722		29 936		35 178		38 303	$+^3F_3$	42 677
	15 570		25 759		29 938				38 357		42 919
			25 769		29 964	5D_4	35 636		38 416		
5S_2	18 373				29 971		35 742			1D_2	44 854
	18 380	3K_7	26 055		30 017		35 796	3M_9	38 809		44 882
			26 067		30 025		36 001		38 823		
5F_4	18 486		26 068		30 055				38 837		

respectively. These states are correspondingly represented by $|0\rangle$, $|1\rangle$, $|2\rangle$, $|3\rangle$ and $|4\rangle$, respectively. Under steady-state excitation conditions, we have the following approximative equations:

$$\dot{N}_2 = 0 = N_0 A_{02} I_0 - N_2 \sigma_{21} \quad (1)$$

$$\dot{N}_1 = 0 = N_2 \sigma_{21} - N_1 \tau_1^{-1} - N_1 A_{14} I_0 \quad (2)$$

$$\dot{N}_4 = 0 = N_1 A_{14} I_0 - N_4 \sigma_{43} \quad (3)$$

$$\dot{N}_3 = 0 = N_4 \sigma_{43} - N_3 \tau_3^{-1} \quad (4)$$

where τ_1^{-1} and τ_3^{-1} are the total relaxation rates of the

states $|1\rangle$ and $|3\rangle$, respectively, A_{ij} is the absorption transition rate from level i to level j , and σ_{ij} is the non-radiative relaxation rate from level i to level j . The above equations are obtained under the approximation $\sigma_{21} \approx \tau_2^{-1}$ and $\sigma_{43} \approx \tau_4^{-1}$, and a solution of them yields

$$N_3 = \frac{N_0 A_{02} A_{14} \tau_3 I_0^2}{(A_{14} I_0 + \tau_1^{-1})} \quad (5)$$

Because the upconversion emission intensity is proportional to the population N_3 in $^5D'_4$, it is obvious from Eq. (5) that the upconversion signal I_{up} is proportional to the laser pump power I_0 in the case of $A_{14} I_0 > \tau_1^{-1}$, which is possible in our condition where intense laser is used and

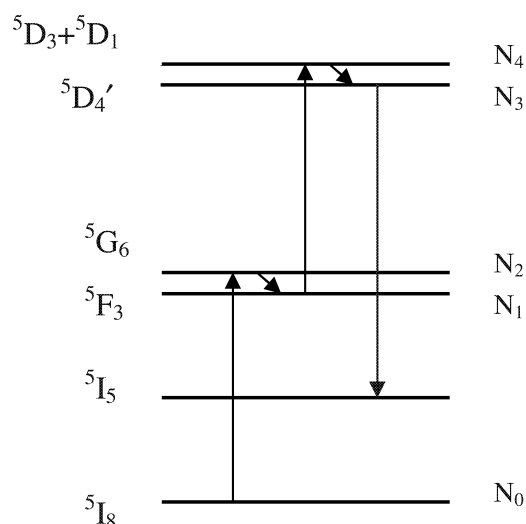


Fig. 5. Upconversion model of Ho^{3+} ion pumped at 457.9 nm.

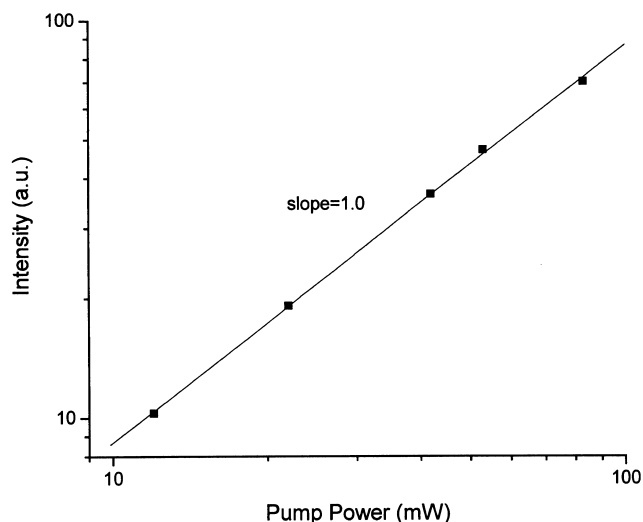


Fig. 6. A double log plot of the upconversion emission intensity versus the pump power of the laser for $\text{Cs}_2\text{NaGd}_{0.95}\text{Ho}_{0.05}\text{Cl}_6$, $\lambda_{\text{em}} = 333$ nm.

A_{14} may also be large enough since the absorption transition from $^5\text{F}_3$ to $^5\text{D}_3 + ^5\text{D}_1$ is an EDV transition, together with the consideration that τ_1 is quite large.

4. Conclusion

This work has presented observed luminescent spectra and their concentration dependence of Ho^{3+} in $\text{Cs}_2\text{NaGdCl}_6$ under 365- and 457.9-nm excitations, as well as an efficient blue to ultraviolet energy upconversion upon pumping with a blue (457.9-nm) laser in this system. It is concluded that the optimum concentration of Ho^{3+} ions is ~ 0.15 for three emission bands with peaks at 493, 545 and 587 nm. The upconversion luminescence is attributed to

the $^5\text{D}'_4 \rightarrow ^5\text{I}_5$ transition. Excited state absorption from $^5\text{F}_3$ state is supposed to be responsible for the phenomenon. The dependence of the upconverted emission intensity on the pump power is also discussed and a slope of 1 is obtained.

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