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**QUENCHING OF THE FLUORESCENCE OF  $\text{Pr}^{3+}$  IONS  
IN APROTIC SOLVENTS**

by  
**A. Watanabe and J.G. Chambers**

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DEPARTMENT OF COMMUNICATIONS  
MINISTÈRE DES COMMUNICATIONS

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*(Informatique Directorate)*



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# QUENCHING OF THE FLUORESCENCE OF $\text{Pr}^{3+}$ IONS IN APROTIC SOLVENTS

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## ABSTRACT

The effects of the composition of the solvent on the non-radiative quenching of the fluorescence of  $\text{Pr}^{3+}$  ions dissolved in aprotic solvents are discussed. The value obtained for the radiative efficiency of the  $^3\text{P}_0$  level was 0.096. Experimental results are also presented for the concentration quenching of the intensity of the  $^1\text{D}_2 \rightarrow ^3\text{H}_4$  transition.

## 1. INTRODUCTION

In the period following the first paper suggesting the possibility of light amplification by stimulated emission<sup>1</sup>, research has been carried out to discover laser materials in solid, gaseous and liquid states. Many solid and gaseous laser materials have been unearthed, but until recently the search for liquid laser materials has not been as productive; one of the few exceptions was the first liquid laser using an organic chelate of trivalent europium<sup>2</sup>. In subsequent years room-temperature laser action was reported in the europium chelate liquid laser<sup>3</sup>, and laser action was also observed in alcohol and aqueous solutions of organic dye molecules<sup>4,5,6,7</sup> and in neodymium ions dissolved in aprotic solvents<sup>8</sup>. Apart from these few exceptions the performance of liquid systems, especially the rare-earth-doped liquids, has been poor compared to that of solid systems. The quantum yield of luminescence of  $\text{Nd}^{3+}$  was found to be as low as  $10^{-5}$  in aqueous solutions<sup>9</sup>, in which radiationless quenching occurs due to the transfer of energy to vibrational states of the solvent molecules. Research on the fluorescence of  $\text{Eu}^{3+}$  in aqueous solutions showed that the fluorescence yields could be greatly enhanced by the deuteration of the solvent<sup>10,11</sup>. By the use of aprotic solvents a fluorescence yield approaching unity has been achieved<sup>12</sup> for  $\text{Nd}^{3+}$  in liquids.

We have studied experimentally the non-radiative quenching of the fluorescence of  $\text{Pr}^{3+}$  ions dissolved in aprotic solvents. The effects of the solvent composition and the rare-earth ion concentration on the fluorescence intensity are discussed, and a value is reported for the radiative efficiency of the  $^3\text{P}_0$  level.

## 2. SOLVENT QUENCHING

Following the preparative procedures and experimental methods described in a previous report<sup>13</sup>, the fluorescence spectra of the  $\text{Pr}^{3+}$  ion were studied in a variety of aprotic solvent systems consisting of a polar solvent, such as  $\text{SeOCl}_2$  or  $\text{POCl}_3$ , and a conjugate acid<sup>8</sup>, such as  $\text{SnCl}_4$ ,  $\text{ZrCl}_4$  or  $\text{TiCl}_4$ . Portions of the fluorescence spectra are shown in Figure 1 for  $\text{Pr}^{3+}$  ions dissolved in a mixture of  $\text{SeOCl}_2$  and  $\text{SnCl}_4$  (top trace) and in a mixture of  $\text{POCl}_3$  and  $\text{TiCl}_4$  (bottom trace). The fluorescence spectra were excited by optically pumping the  $^3\text{H}_4 \rightarrow ^3\text{P}_2$  transition with blue light. The three fluorescence transitions shown in Figure 1 are the  $^1\text{D}_2 \rightarrow ^3\text{H}_4$  transition at  $0.597 \mu\text{m}$ , the  $^3\text{P}_0 \rightarrow ^3\text{H}_6$  transition at  $0.613 \mu\text{m}$  and the  $^3\text{P}_0 \rightarrow ^3\text{F}_2$  transition at  $0.642 \mu\text{m}$ .

The energy level diagram<sup>14</sup> for the  $\text{Pr}^{3+}$  ion is shown in Figure 2. When any of the  $^3\text{P}$  states is excited internal coupling due to non-radiative de-excitation leaves the ion in the  $^3\text{P}_0$  state, since the three levels are close together. The  $^3\text{P}_0$  state is a metastable state, lying approximately  $3600 \text{ cm}^{-1}$  above the  $^1\text{D}_2$  state.

The O-H vibration of a water molecule, which has an energy<sup>15</sup> of approximately  $3000$  to  $4000 \text{ cm}^{-1}$ , is very effective in quenching the fluorescence from the  $^3\text{P}_0$ . Quenching occurs by the excitation of the fundamental or possibly the first-overtone vibration of the O-H bond. The fluorescence from the  $^1\text{D}_2$  level is also quenched by the first-overtone band of the O-H stretching vibration, so that the ratio of the fluorescence intensities from the two levels is relatively unchanged. The total fluorescence intensity is decreased markedly, however, and care must be taken to prevent contamination of the samples by even trace amounts of water, if accurate intensity measurements are to be made. All samples used in our experiments were prepared from dried materials in an evacuable stainless-steel glove box<sup>16</sup>.

It is evident from Figure 1 that the intensity of the  $^1\text{D}_2 \rightarrow ^3\text{H}_4$  transition relative to the intensity of the  $^3\text{P}_0 \rightarrow ^3\text{F}_2$  transition is strongly dependent on the nature of the solvent; the relative intensity changes by a factor of approximately 20 in the two traces. One of the differences between  $\text{SeOCl}_2$  and  $\text{POCl}_3$  lies in the energies of the internal modes of vibration of the two molecules. The infrared transmittance of  $\text{SeOCl}_2$  and  $\text{POCl}_3$  are shown in Figure 3. P-O stretching vibration has an energy<sup>15</sup> of  $1230 \text{ cm}^{-1}$ ; the first-

overtone band of this vibration is seen at approximately  $2500\text{ cm}^{-1}$  and the second overtone at  $3800\text{ cm}^{-1}$ . The fundamental band of the Se-O stretching vibration has an energy<sup>15</sup> of  $907\text{ cm}^{-1}$ ; the second-overtone band of this vibration appears at approximately  $2800\text{ cm}^{-1}$ . It has been shown<sup>17</sup> that, when the energy is too large to be taken away by the excitation of one quantum of vibration, non-radiative quenching of this type in hydrated salts of rare-earth ions takes place by the excitation of a vibrational overtone of one molecule rather than by the simultaneous excitation of the fundamental vibration of several molecules. Since the excitation of vibrational overtones is dependent on the anharmonicity of the intramolecular potential well, there is a very large decrease in the excitation efficiencies of the successive

harmonics. The  $^3P_0$  level can be quenched by the second overtone of the P-O stretching vibration, but requires the excitation of the third overtone of the Se-O stretching vibration. Consequently, non-radiative de-excitation of the  $^3P_0$  level has a lower probability in  $\text{SeOCl}_2$  than in  $\text{POCl}_3$ . The non-radiative de-excitation of the  $^1D_2$  level requires the excitation of the fourth overtone of the P-O vibration or the sixth overtone of the Se-O vibration. The depopulation of the  $^1D_2$  level probably occurs primarily by radiative mechanisms in both solvents (except at high concentrations, as discussed in Section 4).

Since the non-radiative quenching of the  $^3P_0$  level is significantly greater in  $\text{POCl}_3$  than in  $\text{SeOCl}_2$ , all subsequent experiments were carried out on  $\text{Pr}^{3+}$  ions dissolved in a solvent consisting of a 4:1 mixture of  $\text{SeOCl}_2$  and  $\text{SnCl}_4$ .



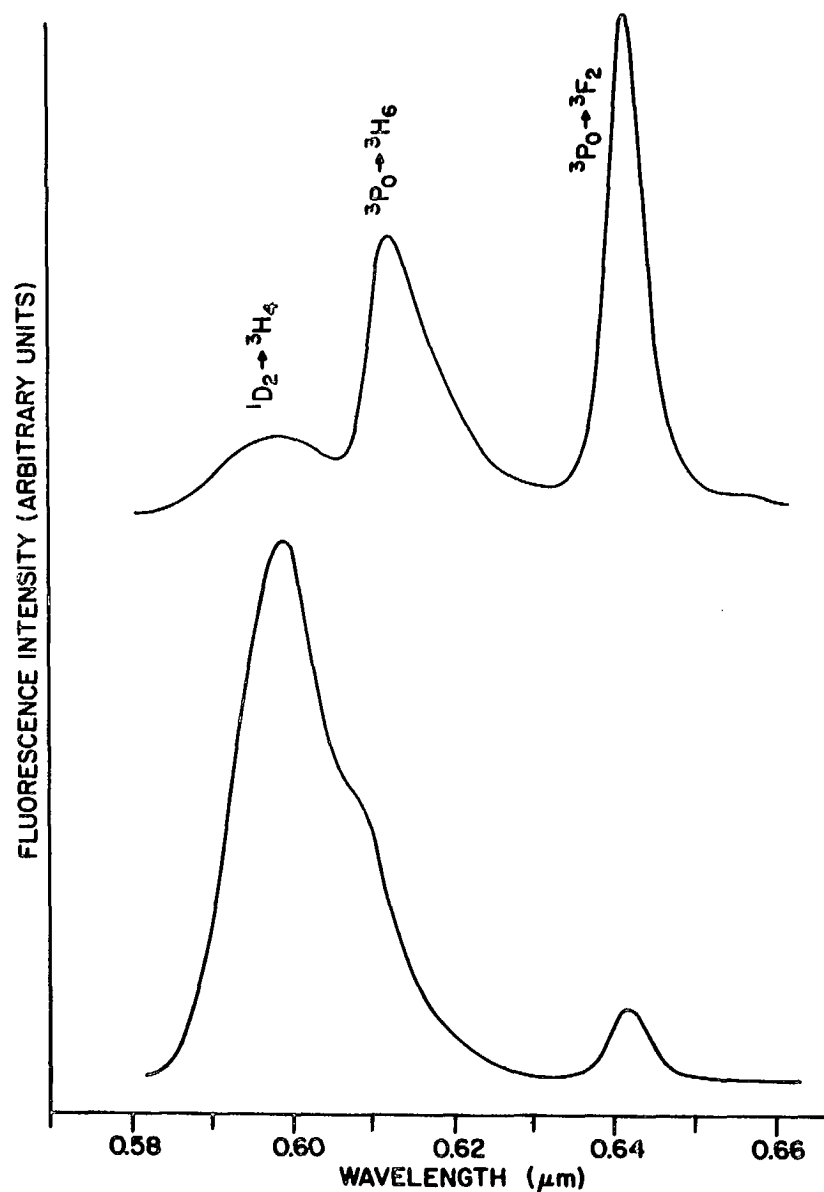


Fig. 1. Portions of the fluorescence spectra of  $\text{Pr}^{3+}$  ions dissolved in two different aprotic solvents, a mixture of  $\text{SeOCl}_2$  and  $\text{SnCl}_4$  (upper trace) and a mixture of  $\text{POCl}_3$  and  $\text{TiCl}_4$  (lower trace). The two traces are not drawn to the same intensity scale.

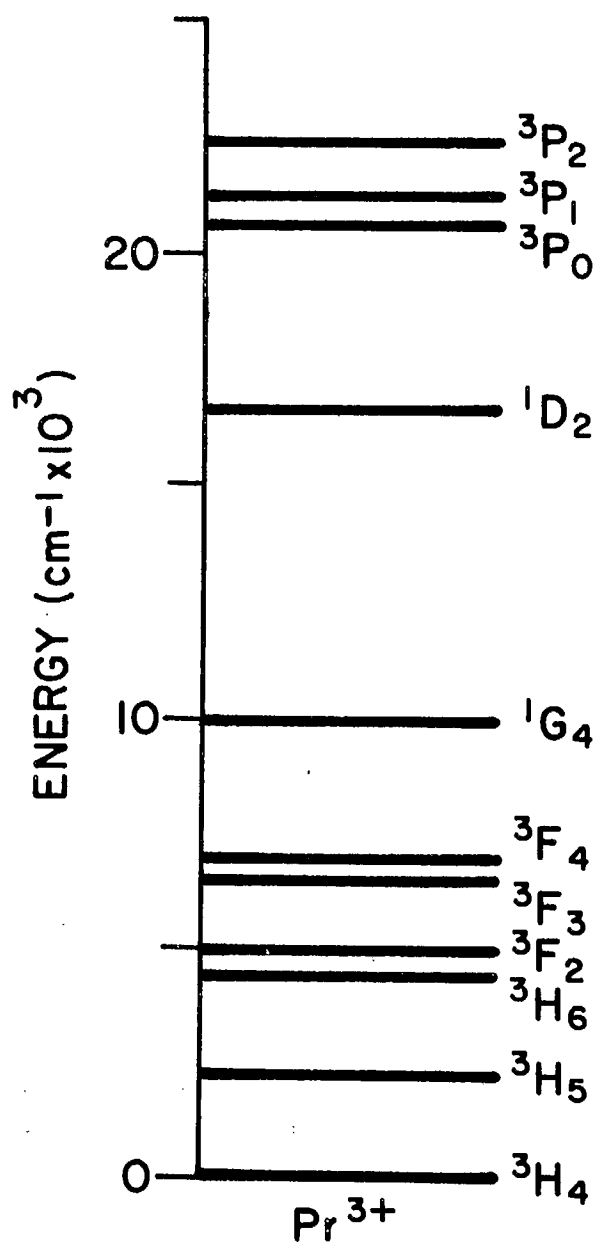


Fig. 2. The free-ion energy levels of the  $\text{Pr}^{3+}$  ion.

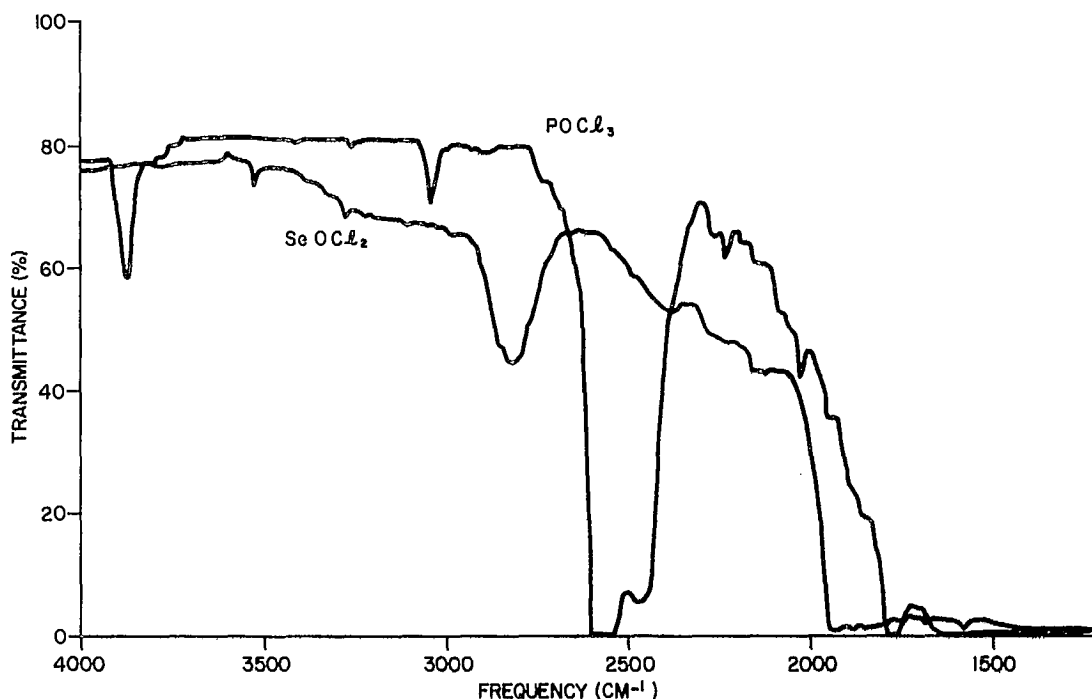


Fig. 3. Portions of the infrared-transmittance curve of  $\text{SeOCl}_2$  and  $\text{POCl}_3$ .

### 3. RADIATIVE EFFICIENCY

A measure of the relative rates of the radiative and the non-radiative mechanisms for depopulating the metastable  $^3\text{P}_0$  state was obtained by calculating the quantum efficiency of fluorescence. The  $^3\text{P}_0$  state is depopulated by non-radiative relaxation to the  $^1\text{D}_2$  level and by radiative relaxation to the  $^3\text{H}_4$ ,  $^3\text{H}_5$ ,  $^3\text{H}_6$ ,  $^3\text{F}_2$ ,  $^3\text{F}_3$  and  $^3\text{F}_4$  levels. The radiative lifetime of the level  $\tau_r$  was calculated by measuring the total coefficient of spontaneous emission  $A$  for all radiative transitions originating from the  $^3\text{P}_0$  level. From the definition of  $A$  it follows<sup>18</sup> that

$$\tau_r = \frac{1}{A} . \quad \text{.....(1)}$$

In a previous note<sup>13</sup> we reported the value  $\tau = 44.7 \mu\text{sec}$  for the radiative lifetime of the  $^3\text{P}_0 \rightarrow ^3\text{H}_4$  transition. The number of photons in all the fluorescence transitions originating in the  $^3\text{P}_0$  level relative to the number of photons emitted in the  $^3\text{P}_0 \rightarrow ^3\text{H}_4$  transition was found by measuring the integrated intensities of the fluorescence lines. The value of the ratio was found to be  $R = 2.38$ , so that the value of the radiative lifetime of the  $^3\text{P}_0$  level is

$$\tau_r = \frac{1}{R} \tau = \frac{1}{2.38} \times 44.7 \mu\text{sec} = 18.8 \mu\text{sec} . \quad \text{.....(2)}$$

The total rate of depopulation of the  $^3P_0$  level was determined by observing the rate of decay of the fluorescence intensity of the  $^3P_0 \rightarrow ^3F_2$  transition with excitation provided by the 0.488  $\mu\text{m}$  radiation of an Ar laser. An optical pulse of width approximately 100 nsec was obtained by optically chopping<sup>19</sup> the beam of a CW laser. The fluorescence lifetime was found to be  $\tau_f = 1.8 \mu\text{sec}$ . The radiative efficiency of the  $^3P_0$  level is therefore given by

$$\eta_r = \frac{\tau_f}{\tau_r} = \frac{1.8}{18.8} = 0.096 \quad \text{.....(3)}$$

This low value for the radiative efficiency shows that non-radiative quenching by the transfer of energy to the intramolecular vibrations of the solvent molecules accounts for more than 90 per cent of the de-excitation of the  $^3P_0$  level, even when the solvent consists of molecules with only relatively low-energy internal modes of vibration, such as the aprotic solvent molecule  $\text{SeOCl}_2$ . The best host for the  $\text{Pr}^{3+}$  would be provided by a material which contains only very heavy atoms. In the solid state,  $\text{LaCl}_3$  is a suitable host for  $\text{Pr}^{3+}$ . A liquid material which is capable of dissolving  $\text{Pr}^{3+}$  ions and contains only heavy atoms is not known.

#### 4. CONCENTRATION QUENCHING

The effect of ion concentration on the fluorescence spectra was studied for  $\text{Pr}^{3+}$  ion concentrations ranging from  $3 \times 10^{-3} \text{ N}$  to  $3 \text{ N}$ . Portions of the fluorescence spectra are shown in Figure 4 for four of the samples. The very marked decrease in the relative intensity of the  $^1D_2 \rightarrow ^3H_4$  transition is apparent in the spectra of the highly concentrated samples. Figure 5 shows the fluorescence intensity per ion as a function of  $\text{Pr}^{3+}$  ion concentration for the  $^3P_0 \rightarrow ^3F_2$  and  $^1D_2 \rightarrow ^3H_4$  transitions. The former shows a variation in the intensity per ion of roughly a factor of 2 over the range of observation. On the other hand the intensity of the  $^1D_2 \rightarrow ^3H_4$  transition varies by 2 orders of magnitude over the same range. The decrease in the intensity of this transition with increasing concentration can be explained in terms of energy transfer between two neighbouring  $\text{Pr}^{3+}$  ions.

Energy transfer between ions<sup>20</sup> can occur either by radiative or by non-radiative processes. For the majority of rare-earth ions radiative transfer is not an effective quenching process<sup>21</sup>. Non-radiative exchange can occur either due to multipolar interactions between the multipolar fields of the ions or due to exchange interactions. Exchange interaction is the electrostatic interaction between the two charge clouds of overlapping ions, and can take place directly or through the interaction with one or two intermediate atoms. Since two interacting ions could be exchange coupled through many intermediate paths, such a process is sometimes called transport quenching to denote the role of the intermediate atoms in the exchange process.

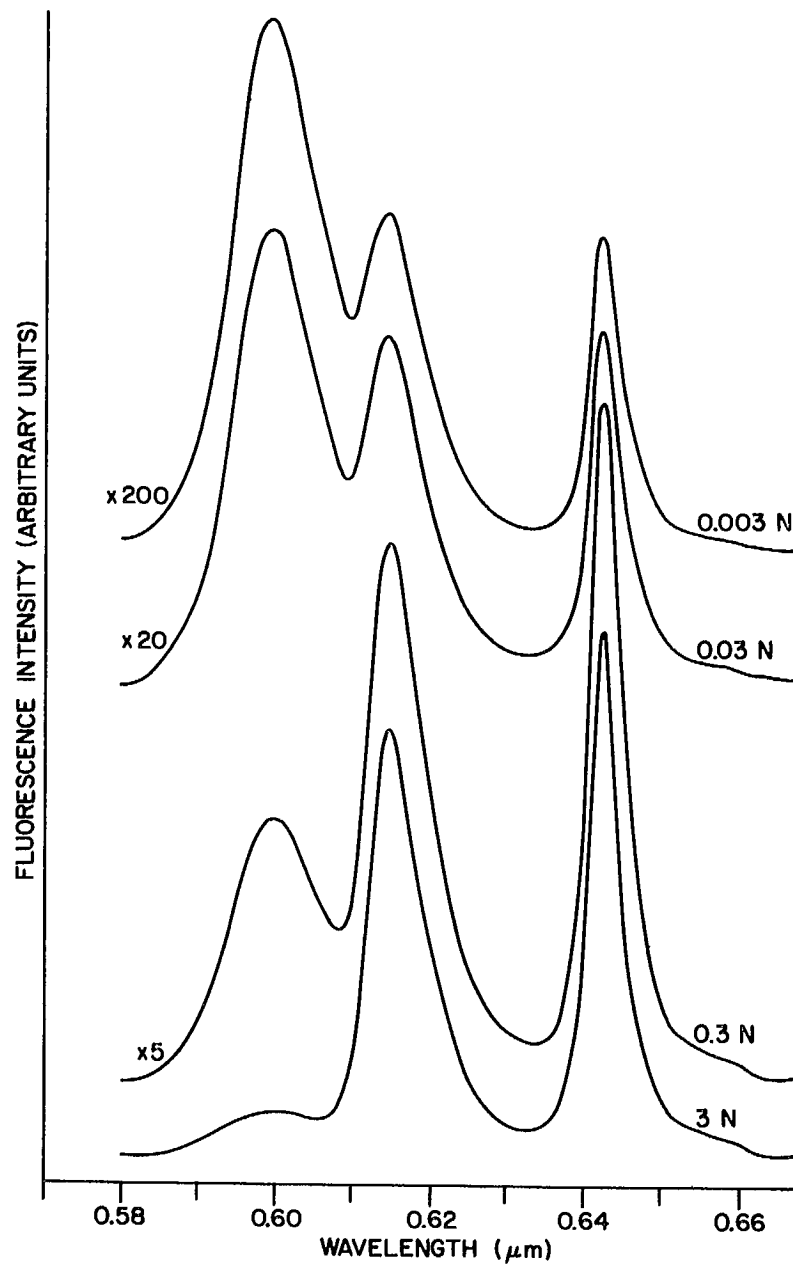


Fig. 4. Portions of the fluorescence spectra of  $\text{Pr}^{3+}$  ions dissolved in a 4:1 mixture of  $\text{SeOCl}_2$  and  $\text{SnCl}_4$  for  $\text{Pr}^{3+}$  ion concentrations of 0.003 N, 0.03 N, 0.3 N and 3 N. The spectra for the lower concentration samples have been amplified by the factors shown at the left end of the spectra.

In the presence of exchange interaction the fluorescence intensity per ion is given<sup>20</sup> by

$$\frac{I}{n} = \left(1 - \frac{n}{n_{\max}}\right)^z, \quad \dots\dots(4)$$

where  $n$  is the number of active ions,  $n_{\max}$  is the total number of ionic sites and  $z$  is the maximum number of contacts that a given ion can have with other active ions in neighbouring sites. It was shown<sup>22</sup> that  $\text{Pr}^{3+}$  ions dissolved in  $\text{SeOCl}_2:\text{SnCl}_4$  are coordinated by a symmetric arrangement of adducted solvent molecules. Thus  $z$  could have a fairly large value. However, the strength of the exchange interaction varies exponentially with the ionic separation<sup>20</sup>, so that it is a very short-ranged effect. Since the separation of two neighbouring ions is probably 10 Å or greater, exchange interaction does not appear to be a likely mechanism for concentration quenching in the present case.

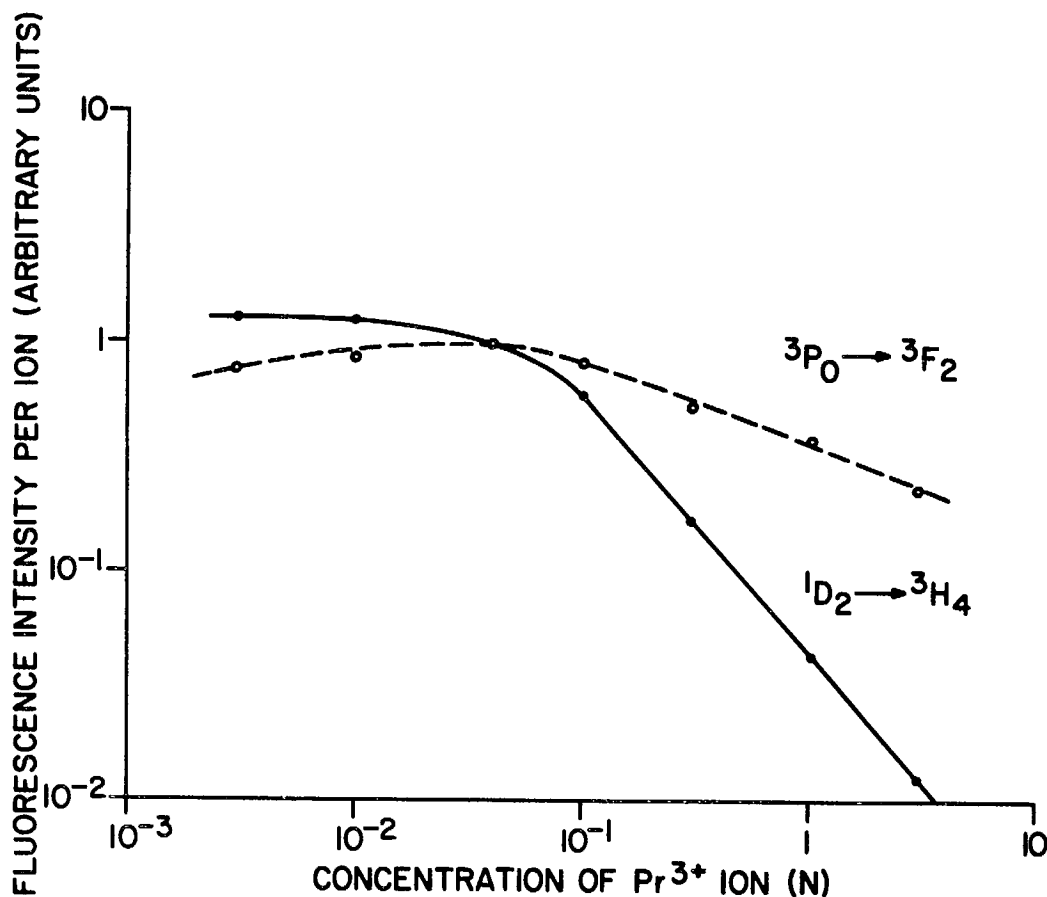


Fig. 5. The fluorescence intensity per ion as a function of  $\text{Pr}^{3+}$  ion concentration for the  ${}^3P_0 \rightarrow {}^3F_2$  transition (dashed line) and the  ${}^1D_2 \rightarrow {}^3H_4$  transition (solid line).

On the other hand multipolar interactions can occur<sup>20</sup> for separations as great as 20 Å. Multipolar transfer can be efficient process for radiative transfer, especially if a resonance condition exists between the two transitions



involved. In this process an ion is non-radiatively de-excited by a certain amount of energy, while at the same time a neighbouring ion is raised in energy by nearly the same amount, with any excess energy being transferred to the surrounding lattice or solvent. In the presence of quenching by multipolar transfer the dependence of the fluorescence intensity per ion on the number of active ions is given by<sup>23</sup>

$$\frac{I}{n} = \left[ 1 + \beta n^{\theta/3} \right]^{-1}, \quad \text{.....(5)}$$

where  $\beta$  is a parameter characterizing the strength of the multipole interaction and  $\theta = 6, 8$  or  $10$  for dipole-dipole, dipole-quadrupole and quadrupole-quadrupole interactions, respectively.

For the  $\text{Pr}^{3+}$  in  $\text{SeOCl}_2:\text{SnCl}_4$  the  $^3\text{P}_0$  level does not appear to be quenched by ion-ion interactions. The combination of transitions that can cause transfer quenching, such as

$$^3\text{P}_0 \leftrightarrow ^1\text{D}_2; ^3\text{H}_4 \leftrightarrow ^3\text{H}_6, \quad \text{.....(6)}$$

differ in energy by  $600 \text{ cm}^{-1}$  or more, so that concentration quenching of the  $^3\text{P}_0$  has a low probability. On the other hand the  $^1\text{D}_2$  level appears to suffer from concentration quenching for concentrations greater than  $0.1 \text{ N}$ . In this case the possible quenching transitions

$$^1\text{D}_2 \leftrightarrow ^3\text{F}_4; ^3\text{H}_4 \leftrightarrow ^1\text{G}_4, \quad \text{.....(7)}$$

are almost exactly in resonance. Also another pair of transitions

$$^1\text{D}_2 \leftrightarrow ^1\text{G}_4; ^3\text{H}_4 \leftrightarrow ^3\text{F}_4, \quad \text{.....(8)}$$

are just slightly off resonance.

The fluorescence intensity per ion of the  $^1\text{D}_2 \leftrightarrow ^3\text{H}_4$  transition of the  $\text{Pr}^{3+}$  ion are shown as a function of the concentration in Figure 6. The dots give the experimental values. The dashed lines show the shape of the exchange-interaction quenching equation (4) for  $z = 1$  and  $20$ . It is obvious from the figure that exchange-interaction quenching cannot account for the observed concentration quenching. The solid line shows the multipolar-transfer quenching equation (5) for  $\theta = 4$ . The value of  $\beta$  does not have a large effect on the shape of the curve; the effect of changing  $\beta$  is to translate the theoretical curve along the horizontal axis. The theoretical curve was calculated with  $\beta = 1$  and was then translated along the horizontal axis to obtain the best fit between experimental and theoretical curves. As discussed above the values  $\theta = 6, 8$  and  $10$  have a definite physical meaning in terms of the nature of the multipolar interaction. In addition, the value  $\theta = 3$  has been observed<sup>24</sup> when quenching takes place by the multipolar interaction of two dissimilar ions A and B and the excitation migrates rapidly over the first set of ions A, so that the quenching process is rate-limited by the transfer from A to close-lying B ions. In the present case only one type of ion appears to contribute

to the fluorescence output, namely,  $\text{Pr}^{3+}$  ions that are fully solvated by  $\text{SeOCl}_2$  molecules. However, there are other ions which have one or more  $\text{H}_2\text{O}$  molecules in their solvation spheres. Although such ions show no fluorescence output due to the very rapid quenching by the high-energy O-H vibrations of the water molecules, it is probable that they contribute to the transfer quenching of the fluorescence intensity of ions surrounded solely by  $\text{SeOCl}_2$  molecules. Thus it is possible that two multipolar quenching processes occur in the present case, one characterized by  $\theta = 3$  and the other by  $\theta = 6, 8$  or  $10$ . The fit of the experimental data to the multipolar quenching equation with  $\theta = 4$  may be fortuitous. Detailed theoretical calculations are necessary in order to explain the nature of the multipolar quenching interaction.

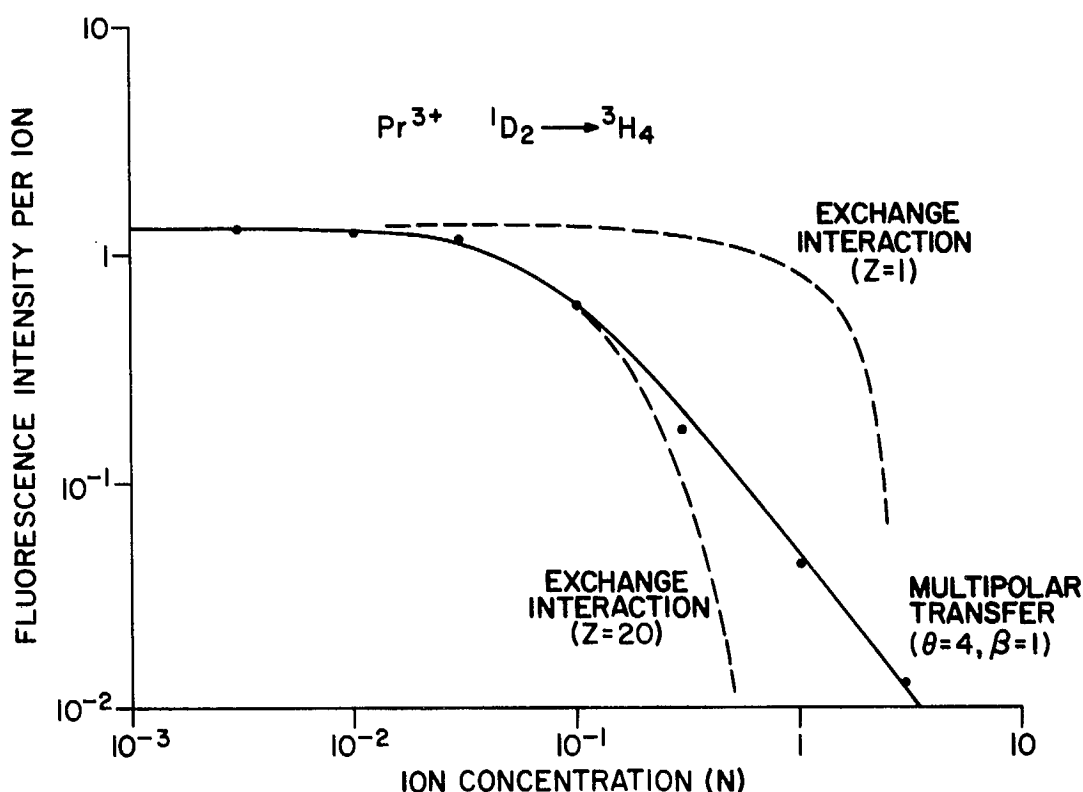


Fig. 6. Experimental and theoretical variation of the fluorescence intensity per ion with the  $\text{Pr}^{3+}$  ion concentration for the  $^1D_2 \rightarrow ^3H_4$  transition. The experimental values are shown by the plotted points. The shape of the exchange quenching equations for  $z = 1$  and  $z = 20$  are shown by the dashed lines. The shape of the multipolar quenching equation for  $\theta = 4$  is shown by the solid line.

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Quenching of the  
fluorescence of  $\text{Pr}^{3+}$  ions in  
aprotic solvents

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