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Singlet–singlet energy transfer from ketone to lanthanide ion β-diketonates as studied by chemiluminescence quenching. First observation of infrared chemiluminescence of neodymium (III) and ytterbium (III) in solution

A.I. Voloshin *, N.M. Shavaleev, V.P. Kazakov

Institute of Organic Chemistry, Ufa Scientific Center of the Russian Academy of Sciences, 71 prosp. Oktyabrya, 450054 Ufa, Russia

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Abstract

Intermolecular singlet–singlet energy transfer from excited adamantanone formed at decomposition of adamantylideneadamantane-1,2-dioxetane (AAD) to the excited levels of β-diketonate ligand in $Ln(TTA)_3{\cdot}2H_2O$ (TTA — thenoyltrifluoroacetone, $Ln = Nd^{3+}$, Yb^{3+}) complexes has been studied by quenching of AAD chemiluminescence (CL) by $Ln(TTA)_3{\cdot}2H_2O$. Chemiexcitation of Nd^{3+} and Yb^{3+} chelates occurs both at intermolecular quenching of AAD CL and at decomposition of $AAD{\cdot}Ln(TTA)_3$ complex followed by emission of infrared light from Nd^{3+} or Yb^{3+} excited ff-levels. The corresponding Nd^{3+} or Yb^{3+} CL spectra were recorded and they coincide with their photoluminescence spectra. The $Yb(TTA)_3{\cdot}2H_2O$ has higher photo- and chemiluminescence efficiencies as compared to $Nd(TTA)_3{\cdot}2H_2O$ due to a larger energy gap between radiative and lower-lying levels in Yb^{3+} as compared to Nd^{3+} leading to a lower efficiency of non-radiative relaxation in Yb^{3+} ion. ©2000 Elsevier Science S.A. All rights reserved.

Keywords: Energy transfer; β-Diketonates; Ytterbium; Neodymium; Dioxetane

1. Introduction

Metal β-diketonates are a convenient model compounds to study the interaction between excited organic molecules and metal complexes, including electron and/or energy transfer processes and photochemical reactions [1–3]. β-Diketonates of lanthanide ions (Ln^{3+}) are of particular interest since some of them (especially Eu^{3+} and Tb^{3+} compounds) possess bright luminescence due to radiative ff-transitions in Ln^{3+} ion in solutions at room temperature [4,5]. Luminescence of Ln^{3+} can be sensitized by energy transfer from suitable excited organic donor formed either by photo excitation [2,4], or in the course of electrochemical [6] or chemical [7–9] reactions generating excited species (chemiexcitation). Lanthanide β-diketonates are often used for elucidation of mechanisms and quantitative characteristics of chemiluminescent reactions and also for the activation (enhancement) of CL intensity in those systems where the primary excited product formed does not emit but can transfer its excitation energy on Ln^{3+} compound [6–9]. The photo- and chemiluminescence of brightly luminescent Eu^{3+} and Tb^{3+} ions in solutions were studied in detail [2,4–8] while there were only a limited number of investigations concerning weakly luminescent Nd^{3+} and Yb^{3+} ions [10–12]. Recent interest to the luminescence of Nd^{3+} and Yb^{3+} complexes in solutions [13–20] is due to their application for infrared luminescence probing in biochemical investigations [13,16]; for liquid lasers based on Ln^{3+} complexes [20]; for analytical determination of individual Ln^{3+} ions [14]. The chemiluminescence of Nd^{3+} and Yb^{3+} ions has not been studied yet. Herein we present the results of first observation of chemiluminescence of *Tris*–thenoyltrifluoroacetonates of Nd^{3+} and Yb^{3+} ($Ln(TTA)_3{\cdot}2H_2O$) in near-infrared region in solution at decomposition of adamantylideneadamantane-1,2-dioxetane (AAD). Moreover, quenching of AAD chemiluminescence by $Ln(TTA)_3{\cdot}2H_2O$ complexes allowed us to study singlet–singlet energy transfer from ketone to β-diketonates of Nd^{3+} and Yb^{3+}.

2. Experimental

Adamantylideneadamantane-1,2-dioxetane was synthesized as in [21]. Adamantanone (Ad=O) was purified

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* Corresponding author. Fax: +7-347-2-35-6066.
E-mail address: chemlum@ufanet.ru (A.I. Voloshin).

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by column chromatography and sublimed. Thenoyltrifluoroacetone (HTTA) was used as received. Synthesis of $Ln(TTA)_3 \cdot 2H_2O$ [22]: 0.8 g of HTTA (3.6×10^{-3} mol) was dissolved in water (650 ml) at 60°C at intense stirring. Solution pH was adjusted to 6–6.5 with NH_4OH. To the obtained HTTA solution was added 9×10^{-4} mol of $LnCl_3 \cdot 6H_2O$ in 3 ml of water. Fine precipitate of $Ln(TTA)_3 \cdot 2H_2O$ formed immediately was stirred for 30 min until flocculation took place and was filtered. Large flakes of $Ln(TTA)_3 \cdot 2H_2O$ were not formed if pH of initial HTTA solution was >7 complicating filtration. Chelates were dried at normal pressure in dessicator, their composition checked by C,H-analysis, deviation from calculated values <0.5%. All experiments were carried out in toluene dried by boiling with metallic sodium and distilled. The equipment used was described elsewhere [8,9].

3. Results and discussion

3.1. Singlet–singlet energy transfer from adamantanone to the ligand excited levels in $Ln(TTA)_3 \cdot 2H_2O$ as studied by quenching of AAD chemiluminescence

Adamantylideneadamantane-1,2-dioxetane is an energy-rich four-membered cyclic peroxide. It neatly decomposes in solution to form the lowest excited singlet ($Ad{=}O_S^*$) and triplet ($Ad{=}O_T^*$) states of adamantanone with corresponding yields (Eq. (1)) [21]:

$$\mathrm{AAD} \xrightarrow{k_1} 1.83\,\mathrm{Ad{=}O} + 0.15\,\mathrm{Ad{=}O_T}^* + 0.02\,\mathrm{Ad{=}O_s}^* \qquad (1)$$

Reaction (1) can be considered as a practically stationary source of excited species since the rate of AAD decomposition (k_1) is less than $10^{-6}\,\mathrm{s}^{-1}$ up to 100°C. Activation parameters of the reaction are $\lg A = 14.1$ and $E_a = 34\,\mathrm{kcal\,mol}^{-1}$ [21]. Spectral composition of the observed chemiluminescence at AAD decomposition is due to the fluorescence of Ad=O, and coincides with its photoluminescence spectrum ($\lambda_{max} = 420$ nm) (Fig. 1). In the presence of chelates $Ln(TTA)_3 \cdot 2H_2O$ or β-diketone HTTA the spectrum of AAD CL in visible (360–700 nm) was not altered while CL intensity was quenched significantly. In the CL quenching experiments the rate constants for monomolecular decomposition of AAD were $<10^{-6}\,\mathrm{s}^{-1}$, and AAD conversion level was <0.5%. In order to prevent inner filter effect the quenching of AAD CL by $Ln(TTA)_3 \cdot 2H_2O$ and HTTA was registered in spectral range $\lambda > 450$ nm since absorption spectra of the quenchers overlap with CL spectrum of AAD (Fig. 1). The quenching obeyed Stern–Volmer equation (Fig. 2). Corresponding Stern–Volmer constants (K_{SV}) of CL quenching are given in Table 1.

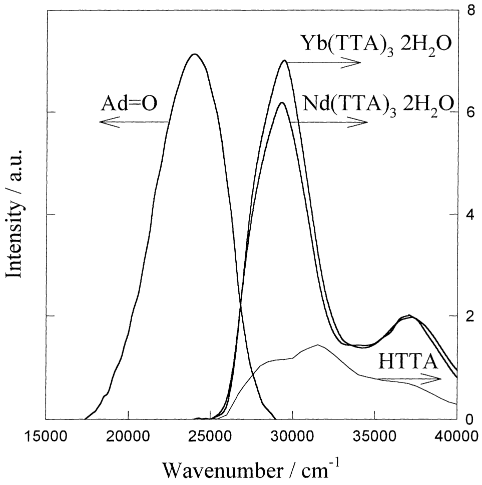

Fig. 1. Fluorescence spectra of Ad=O in CH_3CN. Absorption spectra of $Ln(TTA)_3 \cdot 2H_2O$ in CH_3CN and HTTA in heptane.

Quenching of AAD chemiluminescence by $Ln(TTA)_3 \cdot 2H_2O$ can occur either at intermolecular energy transfer from $Ad{=}O_S^*$ to chelate (Eq. (2)) and/or at complex formation between AAD and $Ln(TTA)_3 \cdot 2H_2O$ that lowers the concentration of free AAD and thus decreases CL intensity (Eq. (3)).

$$\mathrm{Ad{=}O_s}^* + \mathrm{Ln(TTA)_3 \cdot 2H_2O} \xrightarrow{k_q} \mathrm{Ad{=}O} + \mathrm{Ln(TTA)_3 \cdot 2H_2O}^* \qquad (2)$$

$$\mathrm{AAD} + \mathrm{Ln(TTA)_3} \overset{K}{\rightleftarrows} \mathrm{ADD \cdot Ln(TTA)_3} \xrightarrow{k_2} \phi_{\mathrm{Ln}}{}^*[\mathrm{Ln(TTA)_3}{}^* \cdot 2\mathrm{Ad{=}O}] + (1 - \phi_{\mathrm{Ln}}{}^*)[\mathrm{Ln(TTA)_3} \cdot 2\mathrm{Ad{=}O}],$$
$$\phi_{\mathrm{Ln}}{}^* \text{ is the excitation yield of } \mathrm{Ln(TTA)_3} \qquad (3)$$

If only the mechanism (3) is operative, then the observed K_{SV} should be equal to the stability constant of $AAD \cdot Ln(TTA)_3$ complex, $K_{SV} = K$ [8]. Obviously, the formation of $AAD \cdot Ln(TTA)_3$ requires substitution of H_2O molecules with AAD, since Ln^{3+} ion in $Ln(TTA)_3 \cdot 2H_2O$ is already coordination saturated. It should be noted that

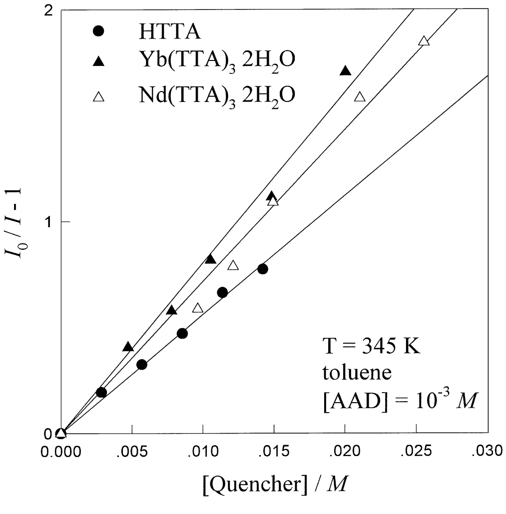

Fig. 2. Stern–Volmer plot for quenching of AAD chemiluminescence by $Ln(TTA)_3 \cdot 2H_2O$ and HTTA.

Table 1
Parameters of quenching of AAD chemiluminescence by $Ln(TTA)_3{\cdot}2H_2O$ and HTTA in toluene. Relative intensities of photo- and chemiluminescence of $Ln(TTA)_3{\cdot}2H_2O$

Quencher	K_{SV}[a] ($l\,mol^{-1}$); 345 K	$k_q \times 10^{-9}$ ($l\,mol^{-1}\,s^{-1}$); 345 K	$J \times 10^{15}$ ($cm^4\,M^{-1}$)	R_0 (Å)	r (Å)	I_T (PL)[b] (a.u.)	I_T (CL)[c] (a.u.)
HTTA	56	6.1	1.1	9.3	4.8	–	–
$Nd(TTA)_3{\cdot}2H_2O$	72	7.9	4.7	11.9	5.9	35	39
$Nd(TTA)_3 + Ad{=}O$[d]	76	8.3	4.7	11.9	5.8	–	33[e]
$Yb(TTA)_3{\cdot}2H_2O$	81	8.9	4.6	11.9	5.7	100	100
$Yb(TTA)_3 + Ad{=}O$[d]	77	8.4	4.1	11.6	5.7	–	154[e]

[a] Error in the determination of K_{SV} is 10%.

[b] Relative photoluminescence intensities of 10^{-3} M $Ln(TTA)_3{\cdot}2H_2O$ toluene solutions, $\lambda_{exc} = 365$ nm (frontal excitation) at room temperature. Intensities can be compared.

[c] Relative chemiluminescence intensities of 3 ml of $[Ln(TTA)_3{\cdot}2H_2O] = 3 \times 10^{-2}$ M, $[AAD] = 10^{-2}$ M toluene solutions at 95°C. Intensities can be compared.

[d] K_{SV} were determined at ratio $[Ad{=}O]/[Ln(TTA)_3{\cdot}2H_2O] = 5$.

[e] $[Ad{=}O] = 0.15$ M. Other conditions as in footnote c.

$[Ln(TTA)_3{\cdot}2H_2O]$ was used in $5 \div 25$ excess relative to [AAD] in CL quenching experiments and the composition of $Ln(TTA)_3{\cdot}2H_2O$ is not changed in toluene solution. It should be expected that in such conditions AAD molecules do not enter inner coordination sphere of Ln^{3+} due to competing H_2O molecules. It is also evidenced by the fact that adamantanone introduced in large excess to [AAD] and $[Ln(TTA)_3{\cdot}2H_2O]$ does not alter the efficiency of CL quenching by $Ln(TTA)_3{\cdot}2H_2O$ (Table 1). Adamantanone binds $Ln(TTA)_3$ into $Ln(TTA)_3{\cdot}nAd{=}O$ ($n = 1$ or 2) thus decreasing the concentration of $AAD{\cdot}Ln(TTA)_3$ [9]. If CL quenching was due to formation of $AAD{\cdot}Ln(TTA)_3$, the presence of Ad=O should have changed the quenching efficiency. But since the values of K_{SV} do not depend on the presence of Ad=O we consider the reaction (3) to be insignificant in the present system.

We think that the observed quenching of AAD CL occurs by allowed intermolecular singlet–singlet energy transfer (S–S) from $Ad{=}O_S{}^*$ to the singlet levels of β-diketonate ligand ($L_S{}^*$) in $Ln(TTA)_3{\cdot}2H_2O$ chelate. The energy transfer is exothermic since $Ad{=}O_S{}^*$ lies at 29 000 cm^{-1}, while $L_S{}^*$ in $Ln(TTA)_3$ is at 26 000 cm^{-1}. Additional evidence for intermolecular S–S energy transfer mechanism comes from the fact that β-diketone HTTA itself also quenches the CL of AAD although complex formation mechanism according to Eq. (3) can not take place with HTTA. The first excited singlet state of HTTA lies at 26 000 cm^{-1}.

According to Forster's equation for dipole–dipole energy transfer, the efficiency of luminescence quenching is directly proportional to the overlap integral (J) between luminescence spectrum of the donor and absorption spectrum of energy acceptor (Fig. 1) [23]: $J = \int I_D(\nu)\varepsilon_A(\nu)\nu^{-4}\,d\nu$, where $I_D(\nu)$ is the luminescence intensity of the donor provided that $\int I_D(\nu)\,d\nu = 1$ and $\varepsilon_A(\nu)$ the molar absorption coefficient of energy acceptor. Actually, K_{SV} is proportional to J (Table 1). If intermolecular energy transfer is the sole quenching mechanism, then the bimolecular rate constants of AAD CL quenching (k_q) can be calculated from equation $k_q = K_{SV}\,\tau_D{}^{-1}$ where τ_D is the lifetime of adamantanone fluorescence in the absence of acceptor. No estimation of τ_D in toluene is available, and it was assumed to be equal to that in hexane [24]; $\tau_D = 9.12$ ns. Calculated values for k_q (Table 1) are close to diffusion limited. Also, in Table 1 are presented the critical energy transfer distances (R_0) calculated from: $R_0 = 9.79 \times 10^3 (k^2 n^{-4} \phi_D J)^{1/6}$ [23], where $k^2 = 2/3$ is the geometric factor; n the refraction index of toluene; $\phi_D = 0.0052$ the quantum yield of fluorescence of Ad=O [25] and the actual distance between donor and acceptor (r) calculated from: $r = R_0 (k_q \tau_D)^{-1/6}$ [23]. The molar absorption coefficient of $Ln(TTA)_3{\cdot}2H_2O$ is higher compared to HTTA (Fig. 1), since the former is composed of three β-diketonate moieties and that results in the increased value of overlap integral and more efficient quenching by $Ln(TTA)_3{\cdot}2H_2O$ as compared to HTTA (Table 1).

Quenching of triplet excited states of organic donors by metal β-diketonates is studied in detail [1,2,4] while quenching of singlet states have almost been left unexplored. One should note the quenching of benzene fluorescence by copper (II) acetylacetonate studied by Marciniak [3]. Investigation of S–S energy transfer from adamantanone to $Ln(TTA)_3{\cdot}2H_2O$ using photoexcitation of ketone is inconvenient since Ad=O cannot be excited selectively, due to a low value of molar absorption coefficient as compared to $Ln(TTA)_3{\cdot}2H_2O$. ε_{max} of Ad=O at 295 nm is only 17 $(M\,cm)^{-1}$ [25], while that for $Ln(TTA)_3{\cdot}2H_2O$ is $\varepsilon \approx 10^4$ $(M\,cm)^{-1}$ (Fig. 1). Thus, chemiluminescence might serve as a convenient approach to study interaction of singlet excited states of organic molecules with metal β-diketonates, since the donor excited state is populated through the energy released in the course of chemical reaction and not through an irradiation with light.

3.2. Spectra and efficiency of photo- and chemiluminescence of β-diketonates of Nd^{3+} and Yb^{3+}

In β-diketonates of luminescent Ln^{3+} ions excitation into ligand absorption band leads to emission of ff-luminescence of Ln^{3+} [5]. Population of excited ff-levels of Ln^{3+} occurs by energy transfer from ligand triplet state ($L_T{}^*$), formed by

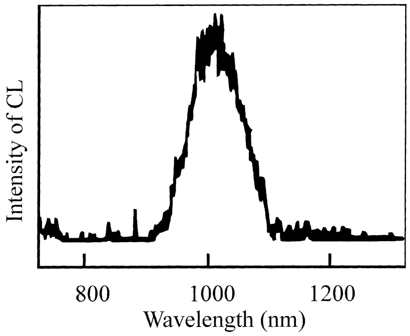

Fig. 3. Chemiluminescence spectrum of Yb^{3+} (uncorrected). $[Yb(TTA)_3{\cdot}2H_2O] = 3\times10^{-2}$ M, $[AAD] = 10^{-2}$ M, $t = 95°C$, toluene. Spectral slit $\Delta\lambda = 20$ nm.

fast intersystem crossing $L_S{}^* \rightarrow L_T{}^*$. It should be noted that direct excitation of Ln^{3+} ion also results in Ln^{3+} emission in Ln^{3+} β-diketonates.

Energy transfer from Ad=O* to $Ln(TTA)_3{\cdot}2H_2O$ (Eqs. (2) and (3)) also forms excited Ln^{3+} β-diketonate with excitation located on ligand $L_S{}^*$ state or directly on the Ln^{3+} ion, and thus we anticipated to observe chemiluminescence of Ln^{3+} ion, since Nd^{3+} and Yb^{3+} β-diketonates exhibited photoluminescence in toluene solution. Actually the chemiluminescence of Nd^{3+} and Yb^{3+} was observed in the system $Ln(TTA)_3{\cdot}2H_2O$–AAD (Figs. 3 and 4). For Nd^{3+} the maxima in CL spectra (λ_{max}) are situated at 880 ± 20 nm ($^4F_{3/2} \rightarrow {}^4I_{9/2}$ transition) and 1060 ± 20 nm ($^2F_{5/2} \rightarrow {}^2I_{7/2}$), and for Yb^{3+} at 980 ± 20 nm ($^2F_{5/2} \rightarrow {}^2F_{7/2}$). Spectra of PL and CL of Nd^{3+} and Yb^{3+} coincide with each other and with the known PL spectra of these ions [10–20].

The integral luminescence intensities (I_T) of $Ln(TTA)_3{\cdot}2H_2O$ corrected for the spectral sensitivity of Cs–O–Ag photocathode [26] used for infrared measurements were calculated in order to compare their luminescence efficiencies (Table 1). Corrected integral intensities of CL and PL for $Yb(TTA)_3{\cdot}2H_2O$ were always higher than those for Nd^{3+} chelate. It can be accounted for by larger energy gap between radiative and lower lying level in Yb^{3+} as compared to Nd^{3+}. In Yb^{3+} the energy gap between $^2F_{5/2}$ and $^2F_{7/2}$ levels is 10 000 cm^{-1}, while for Nd^{3+} the gap between $^4F_{3/2}$ and $^4I_{15/2}$ levels is 5390 cm^{-1}. Smaller energy gap in Nd^{3+} ion results in effective non-radiative energy dissipation through high vibrational modes of C–H and O–H bonds present in the surroundings of Nd^{3+}.

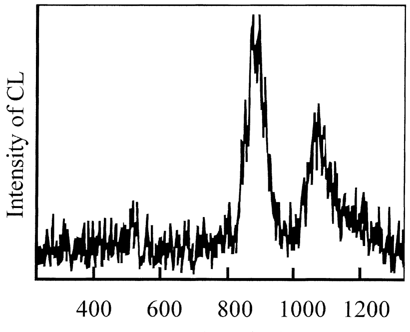

Fig. 4. Chemiluminescence spectrum of Nd^{3+} (uncorrected). $[Nd(TTA)_3{\cdot}2H_2O] = 3\times10^{-2}$ M, $[AAD] = 10^{-2}$ M, $t = 95°C$, toluene, $\Delta\lambda = 20$ nm. Broad band at 1100–1250 nm is an artifact.

3.3. Mechanism of chemiexcitation of Nd^{3+} and Yb^{3+}

Chemiexcitation of Nd^{3+} and Yb^{3+} in the system in question can occur firstly, at intermolecular singlet–singlet energy transfer from $Ad{=}O_S{}^*$ to the β-diketonate ligand $L_S{}^*$ level (Eq. (2)): $Ad{=}O_S{}^* \rightarrow L_S{}^* \rightarrow L_T{}^* \rightarrow Ln^{3+*}$ (vide supra). In that case the observed CL intensity of Ln^{3+} should be directly proportional both to the k_q value (Eq. (2)) and luminescence quantum yield of Ln^{3+} compound.

Secondly, Ln^{3+} can be excited at intermolecular triplet–triplet energy transfer from $Ad{=}O_T{}^*$ to the β-diketonate ligand $L_T{}^*$ level: $Ad{=}O_T{}^* \rightarrow L_T{}^* \rightarrow Ln^{3+*}$, since triplet states of organic donors are effectively quenched by Ln^{3+} β-diketonates and the quenching is accompanied by sensitized Ln^{3+} luminescence [1,2,4]. Energy transfer $Ad{=}O_T{}^* \rightarrow L_T{}^*$ is exothermic, since $Ad{=}O_T{}^*$ is situated at 26 300 cm^{-1} [7], while $L_T{}^*$ in $Ln(TTA)_3{\cdot}2H_2O$ at 20 500 cm^{-1}. However, the probability of intermolecular energy transfer from $Ad{=}O_T{}^*$ should be small due to an extremely short lifetime of $Ad{=}O_T{}^*$: $\tau \approx 0.06$ ns at 90°C [21].

Thirdly, one cannot exclude chemiexcitation of Ln^{3+} at intracomplex energy transfer to Ln^{3+} or ligand levels from $Ad{=}O_T{}^*$ formed at decomposition of AAD in $AAD{\cdot}Ln(TTA)_3$ (Eq. (3)). Although complex formation is an insignificant pathway in the quenching of AAD CL (vide supra), chemiexcitation of Ln^{3+} in $AAD{\cdot}Ln(TTA)_3$ might be very effective to determine the observed Ln^{3+} CL intensity, since the rate of AAD decomposition in $AAD{\cdot}Ln(TTA)_3$ (k_2 in Eq. (3)) is expected to be higher than k_1 by $2 \div 3$ orders of magnitude [9] and energy transfer might occur directly to Ln^{3+} levels. Recently, the efficiency of intracomplex Ln^{3+} chemiexcitation as compared to intermolecular was shown in Pr^{3+} β-diketonates–AAD system [9].

To elucidate the chemiexcitation mechanism we have studied the influence of adamantanone on the CL intensity of Nd^{3+} and Yb^{3+} chelates. If intermolecular energy transfer was the sole chemiexcitation mechanism the addition of Ad=O would enhance Ln^{3+} chemiluminescence intensity since K_{SV} and k_q values for $Ln(TTA)_3{\cdot}n$Ad=O and $Ln(TTA)_3{\cdot}2H_2O$ are equal (Table 1) while luminescence quantum yield of $Ln(TTA)_3{\cdot}n$Ad=O is >3 times higher than that of $Ln(TTA)_3{\cdot}2H_2O$. On the other hand if intracomplex mechanism was the only operative — adamantanone being in excess to AAD would quench Ln^{3+} CL intensity by completely preventing formation of $AAD{\cdot}Ln(TTA)_3$ complex. In the experiment we observed that addition of Ad=O to the $Ln(TTA)_3{\cdot}2H_2O$–AAD system enhances CL intensity for Yb^{3+} and decreases it for Nd^{3+} chelate

(Table 1). Thus it seems that for chelates studied both intermolecular and intracomplex mechanisms are operative. However, with Nd^{3+} chelate the efficiency of intracomplex excitation is higher than intermolecular resulting in decreased CL intensity of Nd^{3+} in the presence of Ad=O while with Yb^{3+} intermolecular excitation is more efficient than intracomplex.

4. Conclusions

Chemiluminescence may serve as a convenient approach to study interaction of singlet excited organic donors with metal complexes especially in those systems where selective photoexcitation of energy donor is impossible. Thus, quenching of AAD chemiluminescence by $Ln(TTA)_3{\cdot}2H_2O$ revealed and allowed to study singlet–singlet energy transfer from adamantanone to β-diketonate ligand excited states. Chemiexcitation of Nd^{3+} and Yb^{3+} chelates occurs both at intermolecular quenching of AAD CL and at decomposition of $AAD{\cdot}Ln(TTA)_3$ complex followed by emission of infrared light from Nd^{3+} or Yb^{3+} excited ff-levels. The corresponding Nd^{3+} or Yb^{3+} CL spectra were recorded and they coincide with their photoluminescence spectra. The $Yb(TTA)_3{\cdot}2H_2O$ has higher photo- and chemiluminescence efficiencies as compared to $Nd(TTA)_3{\cdot}2H_2O$ due to a larger energy gap in Yb^{3+} between radiative and lower-lying level as compared to Nd^{3+}.

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