

The influence of the T_{β} level upon fluorescence and laser properties of aromatic compounds

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Abstract

The fluorescence and laser properties of seven specially chosen aromatic compounds are studied at 293 °C. The quantum yield of fluorescence, γ , decay times, τ_f , of the deaerated and non-deaerated solutions are measured. The oscillator strength, f_e , fluorescence rate constants, k_f , natural lifetimes, τ_0^f , and intersystem crossing rate constants, k_{ST} , are calculated. Some laser parameters are calculated or measured experimentally. It is found that the position of the T_{β} level plays an important role in the fluorescence and laser properties of aromatic compounds. If the T_{β} level is situated below the S_{β} level, it decreases the quantum yield of fluorescence and the decay time and increases the threshold of laser action. If, due to some structural changes of a molecule, the T_{β} level is situated higher than the S_{β} level, then the quantum yield of fluorescence and the decay times are increasing and the threshold of laser action is decreasing. Such influence of the position of the T_{β} level upon fluorescence and laser properties of aromatic compound is explained by the fact that the S_{β} level mixes with the T_{β} level more readily than with other $\tau_{\pi\pi^*}$ levels.

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1. Introduction

It is known that, to a large extent, the spectral-luminescent and laser properties of heteroaromatic compounds are determined by the mutual arrangement of their singlet and triplet levels and their orbital nature [1]. The majority of laser dyes are heteroaromatic compounds [2]. In most cases, the fluorescence properties of an heteroaromatic molecule are determined by three types of electronic transitions: $\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$, and $\pi l \rightarrow \pi^*$ ($l \rightarrow a_n$). Depending on the mutual arrangement of the singlet and triplet levels of πl , π^* and $n\pi^*$ orbital nature, five different spectral-luminescent and laser classes of heteroaromatic molecules can be identified [3–5]. In heteroaromatic compounds, the position of T_{β} level does not play a decisive role.

In the case of pure aromatic molecules, the fluorescence and laser properties are entirely determined by $\pi \rightarrow \pi^*$ transitions. This class of aromatic compounds is also of great interest since some of them are excellent scintillators and

give effective laser oscillation in the UV region of the spectrum [2]. The fluorescence and laser properties of aromatic compounds are determined by the mutual arrangement of the S_{α} and S_{β} levels, but, as it was shown in [5], the position of the T_{β} level also plays an important role. Unfortunately, to date the role of the T_{β} level in aromatic molecule is not fully investigated.

The objective of this study is to further investigate the role of the T_{β} level upon the fluorescence and laser properties of pure aromatic compounds. To illustrate this, the following aromatic compounds are studied in this paper: (1) naphthalene, (2) anthracene, (3) tetracene, (4) *p*-terphenyl, (5) *p*-quaterphenyl, (6) *p*-quinquephenyl and (7) 3,3''-dimethyl-*p*-quaterphenyl.

The structural formulae of the compounds studied are given in Fig. 1.

2. Experimental methods

The compounds studied were recrystallized, sublimized and purity controlled using chromatography. The ultraviolet spectra of the substances were recorded using a SPECORD

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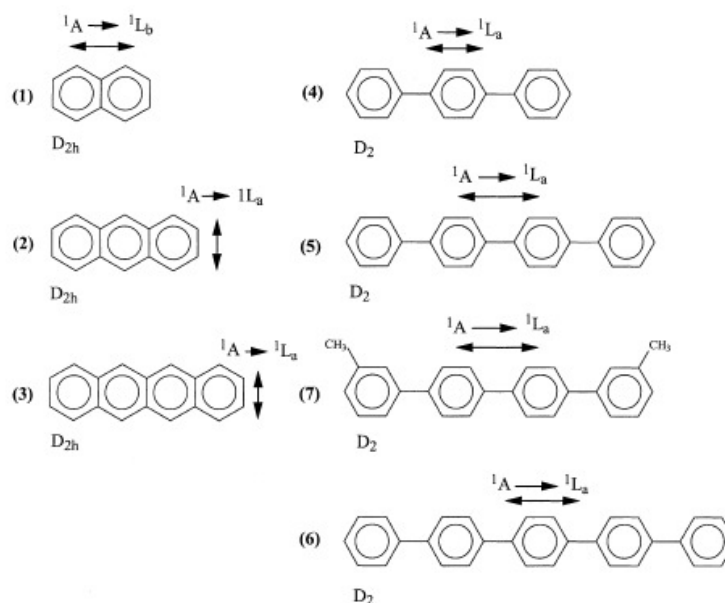


Fig. 1. Structural formulae of compounds studied. The symbol ' \leftrightarrow ' indicates the direction of polarization of the $S_0 \rightarrow S^1$ transition.

M40 spectrometer with spectroquality cyclohexane or benzene as solvent. Compounds (5) and (6), which are of low solubility, were dissolved using a 9:1 cyclohexane/benzene mixture in an ultrasonic USU-0.25 bath. A Hitachi MPF-4 spectrofluorimeter was used to record the fluorescence spectra. The quantum yields of fluorescence were measured using the method described in [6] and a highly diluted solution of 9,10-diphenylanthracene in cyclohexane served as a standard. According to this method, highly diluted solutions of the sample and the standard are excited by monochromatic light of the same frequency and the fluorescence spectra of both compounds are recorded under the same conditions. The quantum yield of the sample is calculated using the formula:

$$\gamma = \frac{\gamma' C_a C_1 \int F(\nu) d\nu}{\int F'(\nu) d\nu} \quad (1)$$

where γ' is the quantum yield of the standard, $C_a = K'(v)/K(v)$ is the absorption correction factor, $K'(v)$ and $K(v)$ are the absorption coefficients of the standard and the sample at the frequency of excitation; $C_1 = e^{-K'(v) \cdot 0.5l} / e^{-K(v) \cdot 0.5l}$ is the intensity of excitation correction factor, with l the length of the absorption cell, $F(\nu)$ and $F'(\nu)$ are the quantum fluorescence functions.

The fluorescence quantum yield of 9,10-diphenylanthracene was in found to be 0.90 [7]. In order to minimize re-absorption effect in cases where there is a large amount of overlap between long-wavelength absorption and fluores-

cence bands, solutions for fluorescence quantum yields and lifetimes measurements were prepared following the recommendations given in [8]. Since some of the substances investigated reveals a very structured long-wavelength absorption band, the spectral bandpass of the excitation monochromator was chosen to be not greater than 0.5 nm in each case. The emission slit width was chosen depending on the fluorescence intensity, but in each case it was not large enough to cause distortion in the fluorescence spectrum.

The decay times of fluorescence, τ_f , were measured using either a SLM-4800S phase fluorimeter or installations based on the stroboscopic principle combined with single photon counting measurements, depending on the value of τ_f . The natural lifetimes were calculated using the formula presented in [9] and modified in [10].

$$\frac{1}{\tau_0^2} = 2.88 \times 10^{-9} n^2 \langle \bar{\nu}_f^{-3} \rangle^{-1} \theta \int \frac{\epsilon(\bar{\nu})}{\bar{\nu}} d\bar{\nu} \quad (2)$$

where $\theta = 9n/(n^2 + 2)^2$ (Lorentz-Lorenz factor), n is the refractive index of the solvent, $\bar{\nu}$ is the frequency in cm^{-1} and $\epsilon(\bar{\nu})$ is the molar extinction coefficient. Deaeration was carried out using the method described in [11]. According to this method, a solution of the sample in a cell is frozen. Then air is evacuated from the cell and replaced by nitrogen gas before the sample is unfrozen. For deep deaeration, the procedure could be repeated. The value of k_{ST} was calculated by taking into account the fact that the fluorescence quantum yield of highly deaerated solutions of photostable

compounds can be determined using Eq. (2), with only intramolecular quenching processes considered:

$$\gamma^* = \frac{k_f}{k_f + k_S + k_{ST}} \quad (3)$$

where γ^* is the fluorescence quantum yield of the deaerated solution and k_S is the internal conversion rate coefficient. From Eq. (3) one obtains:

$$k_S + k_{ST} = \frac{1 - \gamma^*}{\tau_f^*}$$

where τ_f^* is the fluorescence decay time for the deaerated solution. For the Ermolaev–Sveshnikova [12] molecules, k_S is very much less than $k_f + k_{ST}$ and in many cases $k_S \ll k_{ST}$, hence:

$$k_{ST} \approx \frac{1 - \gamma^*}{\tau_f^*}$$

The Stoke's shift values were determined using the formulae:

$$\Delta\tilde{\nu}_{ST} = \tilde{\nu}_a^{c,\xi} - \tilde{\nu}_f^{c,\xi}$$

where

$$\tilde{\nu}_a^{c,\xi} = \frac{\int \tilde{\nu}_a \varepsilon(\tilde{\nu}_a) d\tilde{\nu}_a}{\int \varepsilon(\tilde{\nu}_a) d\tilde{\nu}_a} \quad \text{and} \quad \tilde{\nu}_f^{c,\xi} = \frac{\int \tilde{\nu}_f I(\tilde{\nu}_f) d\tilde{\nu}_f}{\int I(\tilde{\nu}_f) d\tilde{\nu}_f}$$

$\tilde{\nu}_a^{c,\xi}$ and $\tilde{\nu}_f^{c,\xi}$ are the "centre of gravity" or first moment of the long-wave absorption band and fluorescence spectrum respectively. $\tilde{\nu}_a$ and $\tilde{\nu}_f$ are the frequencies in the range of the absorption and fluorescence spectra, $I(\nu_f)$ is the intensity of fluorescence.

The oscillator strength of well resolved long-wave absorption bands was determined using the formula:

$$f_e = 1.3 \times 10^{-8} \theta \int \varepsilon(\tilde{\nu}) d\tilde{\nu} \quad (4)$$

This formula, according to [13], is considered to be appropriate if the oscillator strength of free molecules is calculated from the absorption band of the solution. The oscillator strengths of low intensity or submerged bands were determined using the formula:

$$f_e = \frac{4.514 \nu_a^{c,\xi} \gamma}{n^2 (\tilde{\nu}_f^{c,\xi})^3 \tau_f} \quad (5)$$

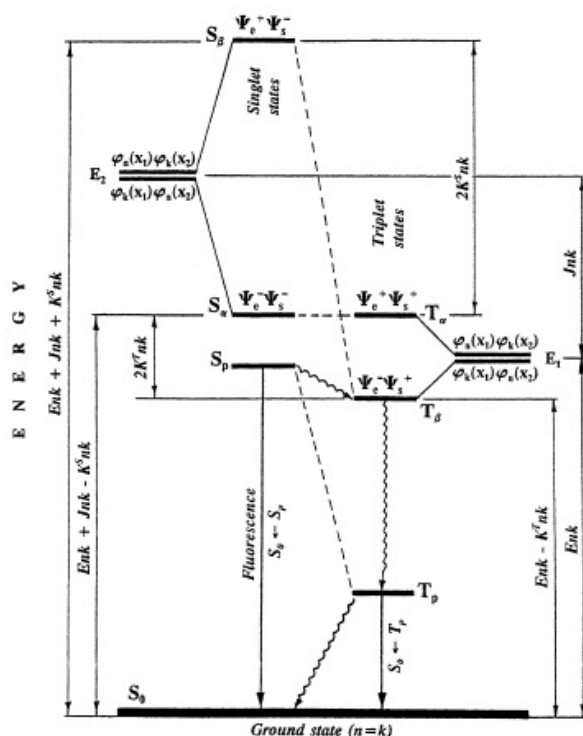


Fig. 2. Quantum mechanical interpretation of the $\pi\pi^*$ orbital nature of S_1 (1B_u), S_2 (1L_u), T_0 (3L_u) and T_1 (3B_u) states and the energy separation between them.

This was obtained by dividing Eq. (4) by a simplified version of Eq. (2), taking into account that

$$(\bar{\nu}^{-3})^{-1} \approx (\bar{\nu}_f^c \bar{\epsilon})^3$$

The error limits determined for the various fluorescence parameters are as follows: quantum yield $\pm 10\%$, decay time $\pm 5\%$, symmetry line frequencies $\pm 60 \text{ cm}^{-1}$, Stokes shifts $\pm 200 \text{ cm}^{-1}$, k_{ST} and k_f values (if $\tau_f < 2 \text{ ns}$) $\pm 15\%$.

The cross-section for absorption at the pumping frequency was calculated by:

$$\sigma_p(\bar{\nu}_p) = 3.82 \times 10^{-21} \epsilon_{10}(\bar{\nu}_p) \quad (6)$$

The spectral Einstein coefficient for the above frequency was found with the formula:

$$B_p(\bar{\nu}_p) = \frac{\sigma_p(\bar{\nu}_p)c}{nh\nu} \quad (7)$$

where c is the speed of light in vacuum and n the refractive index.

The simulated cross-section for stimulated emission was calculated as:

$$\sigma_p(\bar{\nu}_f^{\text{max}}) = 3.82 \times 10^{-21} \epsilon_{10}(\bar{\nu}_f^{\text{max}}) \quad (8)$$

assuming that $\sigma_e(\bar{\nu}_f^{\text{max}}) = \sigma_a(\bar{\nu}_f^{\text{max}})$

The threshold for the laser action was calculated using Eq. (8) [14]

$$U_f^T = \frac{A}{\gamma B_p(\bar{\nu}_p)} \times \frac{K_{\text{loss}}}{N\sigma_e(\bar{\nu}_f^{\text{max}}) - K_{\text{loss}}} \quad (9)$$

where U_f^T is the threshold density of radiation at the pumping frequency, K_{loss} is the loss coefficient, $K_{\text{loss}} = (1 - R)/L$, R coefficient of reflection of the mirror, L the length of the active medium; N the molecular concentration in the active medium,

$$N = \frac{C}{1000} \times N_A,$$

C is the molar concentration, N_A is the Avogadro number.

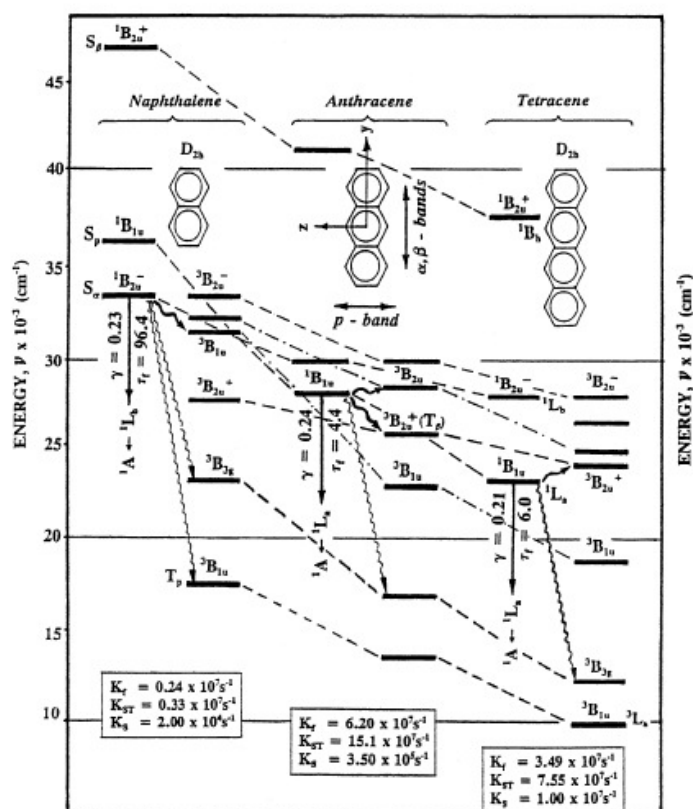


Fig. 3. Simulated singlet and triplet states of naphthalene, anthracene and tetracene. The mixing of states between S^1 and T_1 are shown by wavy arrows. Axes y and z are according to Mulliken [15]. The '+' and the '-' signs denote the symmetric and antisymmetric wave functions respectively. States of the same symmetry are joined by dashed lines.

For pumping, either a XeCl (308 nm) or a N₂ (337 nm) laser was used.

In most cases, the transverse method of pumping was employed.

3. Results and discussion

Spectral—luminescent and hence, laser parameters of pure aromatic compounds are mostly determined by the mutual arrangement of the singlet S_β (1B_g), S_α (1L_g), S_p (1L_a) levels and the corresponding T_β (3B_g), T_α (3L_g), and T_p (3L_a) triplet levels. The S_β state is biconfigurational, symmetric with respect to the electron coordinates and antisymmetric with respect to the spin coordinates ($\Psi_e^+\Psi_s^-$). The S_α state is also biconfigurational but antisymmetric with respect to both the electron and spin coordinates ($\Psi_e^-\Psi_s^-$). The corresponding triplet states T_β and T_α , are of opposite nature: $\Psi_e^-\Psi_s^+$ and $\Psi_e^+\Psi_s^+$ (see Fig. 2). The energy separation between S_β and S_α states and between T_β and T_α are determined by twice the value of the so called electrostatic exchange integrals for the singlet and the triplet states (K_{nk}^S, K_{nk}^T). The quantity $2K_{nk}$ is the Fermi correlation energy. It is much smaller for the triplet states due to there being better correlation of the electron orbits [15]. Thus, the mutual arrangement of S_β , S_α , T_β and T_α levels is determined by the coulomb integral, J_{nk} , and the exchange integrals K_{nk}^S and K_{nk}^T . As one can see from Fig. 2, the T_β level is always associated and situated below the S_α and T_α states. The energy split between S_α and T_β is usually about 3000 cm^{-1} for polycondensed aromatics and about 2500 cm^{-1} for oligophenylene aromatics [16].

The fluorescence parameters of the compounds investigated are given in Table 1 and the laser properties of compound (4–6) in Table 2.

As one can see from Table 1. In the transition from naphthalene to tetracene, the fluorescence rate constant first increases and then decreases: $0.24 \times 10^7\text{ s}^{-1} \rightarrow 6.20 \times 10^7\text{ s}^{-1} \rightarrow 3.49 \times 10^7\text{ s}^{-1}$. The quantum yield remains al-

most constant: $0.23 \rightarrow 0.24 \rightarrow 0.21$. The intersystem rate constant changes in the following way: $0.33 \times 10^7\text{ s}^{-1} \rightarrow 15.10 \times 10^7\text{ s}^{-1} \rightarrow 8.55 \times 10^7\text{ s}^{-1}$. It is easy to understand why k_{ST} is increasing in the transition from naphthalene to anthracene. That is because the transition to the lowest excited level in naphthalene is a forbidden one, while in anthracene the lowest level is allowed, which mixes with triplet levels readily [15].

One could expect that k_{ST} should increase more in the transition from anthracene to tetracene, because the S_p level considerably goes down ($26,580\text{ cm}^{-1} \rightarrow 20,950\text{ cm}^{-1}$). However, this is not happening.

To explain this striking phenomenon a simulation of the singlet and triplet levels of naphthalene, anthracene and tetracene was done using the PPP-CI method. The results are shown in Fig. 3. As one can see from Fig. 3, the T_β ($^3B_{2u}^+$) level of anthracene is situated below the fluorescent level S_p ($^1B_{1u}$) while in tetracene, the T_β level is situated higher than the S_p ($^1B_{1u}$) level. It was shown in [16] earlier that the S_p level mixes with the T_β level readily. This explains why the k_{ST} value of tetracene is less than the k_{ST} value of anthracene.

Compounds (1–3) do not show laser activity. Naphthalene does not show laser activity because its fluorescence state is of S_α nature, which is forbidden and hence its k_f value is very low. Anthracene and tetracene do not show laser activity because of strong reabsorption in the $S^1 \rightarrow S^0$ and the $T^1 \rightarrow T^0$ channels [17]. Now consider compound (4–7) which show laser activity. It seems that laser activity must increase in the transition from compound (4) to (6) because the k_f value is increasing in this row. But this is not happening. The quantum yield of fluorescence in this row is changing as follows: $0.84 \rightarrow 0.81 \rightarrow 0.89$. k_f is steadily increasing ($83.80 \times 10^7\text{ s}^{-1} \rightarrow 94.25 \times 10^7\text{ s}^{-1} \rightarrow 108.70 \times 10^7\text{ s}^{-1}$). k_{ST} is first increasing and then decreasing: ($11.40 \times 10^7\text{ s}^{-1} \rightarrow 20.68 \times 10^7\text{ s}^{-1} \rightarrow 13.41 \times 10^7\text{ s}^{-1}$). Again the behaviour of γ and k_{ST} are strange. It seems also that in the transition from compound (5) to compound (7) γ must decrease, because in many cases CH₃ groups decrease the fluorescence quantum yield [18]. However, strangely

Table 1
Experimental and calculated values of the main fluorescence parameters of diluted cyclohexane solutions of the investigated aromatic compounds

No.	S	ν_{00} (cm^{-1})	$\Delta\nu$ (cm^{-1})	γ	τ_f (ns)	τ_f^{ex} (ns)	γ^*	τ_f^* (ns)	τ_f^{ex*} (ns)	t_0^T (ns)	k_f ($\times 10^{-7}\text{ s}^{-1}$)	k_{ST} ($\times 10^{-7}\text{ s}^{-1}$)	f_0	Nature of S^1
1	D_{2h}	31170	3140	0.23	96.40	417.40	0.42	175.00	418.00	–	0.24	0.33	0.006+	S_α (1L_h)
2	D_{2h}	26580	4100	0.24	4.40	18.30	0.29	4.70	16.20	16.00	6.20	15.10	0.250	S_p (1L_a)
3	D_{2h}	20950	3700	0.21	6.00	28.57	0.29	8.30	28.60	28.60	3.49	8.55	0.190	S_p (1L_a)
4	D_2	31960	7860	0.84	1.00	1.19	0.88	1.05	1.19	1.50	83.80	11.40	2.060	S_p (1L_a)
5	D_2	30100	7640	0.81	0.85	1.05	0.82	0.87	1.06	1.45	94.25	20.68	2.360	S_p (1L_a)
6	D_2	29060	7460	0.89	0.82	0.92	0.89	0.82	0.92	1.15	108.70	13.41	2.600	S_p (1L_a)
7	C_2	29820	7840	0.88	0.90	1.02	0.90	0.92	1.02	1.38	97.83	10.87	2.580	S_p (1L_a)

Headings from left to right: No., compound number; S, symmetry group; $\Delta\nu_{ST}$, Stokes shift; γ , fluorescence quantum yield; τ_f , fluorescence decay time; τ_f^{ex} , experimental natural fluorescence lifetime; τ_f^T , natural lifetime; k_f , fluorescence rate constant; K_{ST} , intersystem crossing rate constant; f_0 , $S_0 \rightarrow S^1$ transition oscillator strength. The nature of S^1 state is given in Clar's notation with Platt's notation in parentheses.

* Parameters for deaerated solutions.

+ Calculated using Eq. (4).

Table 2
Main fluorescence and laser parameters of compound (4–6)

No.	S	$\lambda_{\text{f}}^{\text{max}}$ (nm)	γ	τ_{f} (ns)	k_{IT} ($\times 10^{-7} \text{ s}^{-1}$)	A ($\times 10^{-7} \text{ s}^{-1}$)	Pump Laser	σ_{p} ($\times 10^{17} \text{ cm}^2$)	B_{p} ($\times 10^{-4} \text{ cm}^3 \text{ erg}^{-1} \text{ s}^{-1}$)	σ_{e} ($\times 10^{17} \text{ cm}^2$)	U_{t}^{f} (erg cm^{-3})	U_{t}^{b} (erg cm^{-3})	$\lambda_{\text{f}}^{\text{max}}$ (nm)
4	D ₂	340	0.84	1.00	11.40	83.80	NeCl	2.08	7.24	13.00	213.0	251.0	340
5	D ₂	365	0.81	0.85	20.68	94.25	NeCl	11.46	39.90	15.50	221.3	272.1	365
6	D ₂	380	0.89	0.82	13.41	108.70	NeCl	16.80	58.40	18.00	201.6	220.3	380

Headings from left to right: No., compound number; S, symmetry group; $\lambda_{\text{f}}^{\text{max}}$, Stokes shift between maxima of *p*-band and fluorescence spectrum; γ , fluorescence quantum yield; τ_{f} , fluorescence decay time; k_{IT} , intersystem crossing rate constant; A, Einstein coefficient for spontaneous emission (A_{21}); σ_{p} , absorption cross-section of a molecule at the pumping frequency; B_{p} , Einstein absorption coefficient at the pumping frequency; σ_{e} , emission cross-section at $\lambda_{\text{f}}^{\text{max}}$; U_{t}^{f} , theoretical threshold of laser action; U_{t}^{b} , theoretical threshold of laser action; $\lambda_{\text{f}}^{\text{max}}$, wavelength of broad band laser action.

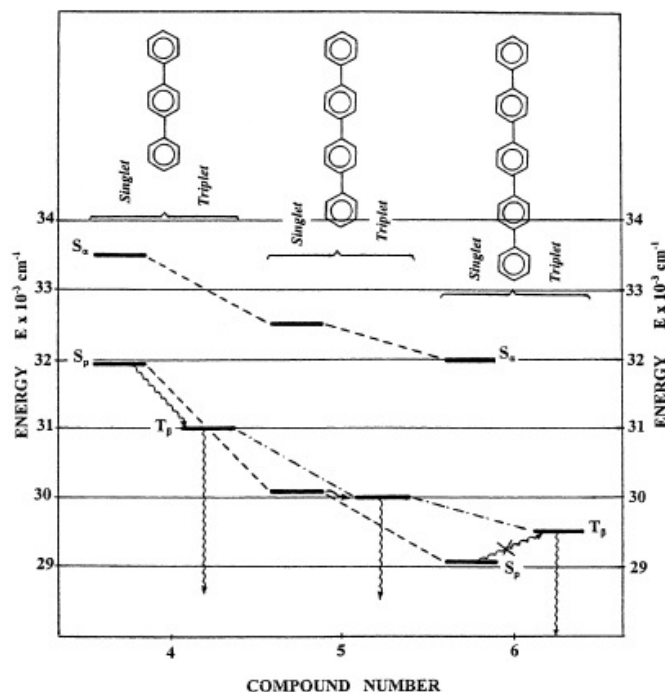


Fig. 4. S_α , S_β and T_β states of fluorescence of compound (4–6). Wavy arrows shows the main channels of disintegration of S_β ($S_{\beta\pi\pi^*}^1$) states. The experimental values of S_α and S_β levels are taken from [19].

enough, γ is increasing. An explanation to this phenomenon also can be given by the position of the T_β level. In Fig. 4, the S_α , S_β and T_β energy levels of compound (4–6) are shown. As one can see from Fig. 4 in *p*-terphenyl the T_β level is 960 cm^{-1} lower than the S_β level. In this row of molecules, the S_β level goes down faster than S_α and related T_β levels, hence the $S_\beta - T_\beta$ energy gap is changing. In *p*-quaterphenyl, the $S_\beta - T_\beta$ energy gap is equal to only 100 cm^{-1} . Therefore, the $S_\beta \rightarrow T_\beta \rightarrow T_1$ disintegration channel becomes more effective than in *p*-terphenyl. As a result of this, the γ value is decreasing from 0.84 to 0.81 and theoretical and experimental values of the threshold are increasing. In the transition from *p*-quaterphenyl to *p*-quinquephenyl, the position of the T_β is changing further: now it is situated higher than the S_β level, and therefore the $S_\beta \rightarrow T_\beta \rightarrow T_1$ disintegration channel is suppressed. Consequently γ is increasing (0.81 \rightarrow 0.89) and the theoretical and experimental value of the thresholds are decreasing ($221.3\text{ erg cm}^{-3} \rightarrow 201.6\text{ erg cm}^{-3}$), ($272.1\text{ erg cm}^{-3} \rightarrow 2220.3\text{ erg cm}^{-3}$) correspondingly.

Improvement of the laser parameters in the transition from compound (5) to compound (7) are also explained by the inversion of T_β and S_β levels. The experimental value of

the threshold for compound (7) is equal to 228.4 erg cm^{-3} . The laser parameters of compound (7) are very close to the parameters of compound (6).

4. Conclusions

From the fluorescence and laser data presented and discussed in the paper, the following conclusions may be drawn. The position of the T_β level exerts great influence upon the fluorescence and laser properties of aromatic compounds. If the T_β level is situated below the S_β level, it mixes with the S_β level readily and because of that the quantum yield of fluorescence and decay time are decreasing and the threshold of laser action is increasing. But if the inversion of the T_β level and the S_β level is happening so that T_β goes higher than S_β , then the quantum yield of fluorescence and decay time are increasing and the threshold of laser action is decreasing. The facts observed gives a better understanding of the transformation of light absorbed by an aromatic molecule. Finally, it is necessary to point out that the most important fluorescence parameters of the compounds studied are in good agreement with the results of earlier investigations [1,20].

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