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Charge-transfer complexations of 1,*n*-di(9-ethylcarbazol-3-yl) alkanes with tetracyanoethylene and tetranitromethane

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ABSTRACT

1,*n*-Di(9-ethylcarbazol-3-yl)alkanes, where $n = 1–5$, as the dichromophoric model compounds of poly-3-vinylcarbazoles were synthesized to examine their complexation behaviors with the electron acceptors tetracyanoethylene (TCNE) and tetranitromethane (TNM). 9,9′-Diethyl-3,3′-dicarbazolyl, di(3-ethylcarbazol-9-yl)methane, and three monomeric analogues were also included for comparison. In dichloromethane solution, the dicarbazoles formed stable 1:1 electron donor–acceptor complexes with TCNE having formation enthalpies around −3.5 kcal/mol. With TNM they formed more weakly bound complexes that showed little dependence on concentration and almost zero dependence on temperature changes having nearly 0 kcal/mol enthalpies of formation. The smaller gap between the two carbazole groups in 1,*n*-di(9-ethylcarbazol-3-yl)alkanes with $n \leq 2$ affected complexation adversely, while such an effect was not observed in the dicarbazoles with $n \geq 3$.

1. Introduction

As a tricyclic heteroaromatic compound with 14 π-electrons, including the unshared electron pair of the nitrogen atom, carbazole has drawn much attention due to the photoconductivity of its polymeric and lower weight systems. Poly-*N*-vinylcarbazole (PVK) is among the most studied photoconducting polymers due to its commercial application in electrophotography [1–4]. PVK itself is not very efficient for electrophotographic purposes because of its low sensitivity in the visible region of the light [5]. However, this sensitivity is increased by the addition of a small amount of certain dyes, such as triphenylmethane, xanthene, acridine, cyanine, merocyanine, or pyrylium dyes, or electron acceptors such as tetracyanoethylene (TCNE), 2,4,7-trinitrofluorene-9-one (TNF), and chloranils [6,7]. PVK comprised with TNF was the first organic photoconductor commercially used for electrophotographic purposes [8]. This photoconductive material is an EDA complex formed between carbazole moieties and TNF molecules. It exhibits low photosensitivity, toxicity due to TNF, and poor mechanical properties [9]. Therefore, other variations of EDA complexes of PVK were investigated, as well as the dye sensitization of PVK [10–13]. Other carbazole-containing polymers have also been prepared for the same purpose [14,15]. Poly-3-vinylcarbazole (P3VK) is an isomeric carbazole-containing photoconducting polymer that has been applied to electrophotograpy [16,17].

In studies conducted to understand the nature of the charge-transfer (CT) complex formation with certain electron acceptors, researchers tend to prefer working on the model compounds of polymers to avoid dealing with the complex spectroscopic properties and other disadvantages of polymers, such as purification, polydispersity, and difficulty in working at microscale levels. For the same reasons, a series of 1,*n*-di(9-carbazolyl)alkanes (DKA) as the model compounds of PVK and 1,*n*-di(9-anthryl)alkanes (DAA) as the model compounds of poly-9-vinylanthracene were synthesized, and their physical and chemical properties were widely investigated [18,19]. We report here the results of a study aimed at investigating thermodynamic properties and stoichiometries of complexation of 1,*n*-di(9-ethylcarbazol-3-yl)alkanes (D1–D5), 9,9′-diethyl-3,3′-dicarbazolyl (D0), di(3-ethylcarbazol-9-yl)methane (D1-9), 9-ethylcarbazole (M1), 9-ethyl-3-methylcarbazole (M2), and 3,9-diethylcarbazole (M3) with TCNE and TNM in dichloromethane (Scheme 1).

2. Experimental

2.1. Instrumentation

All absorbance measurements were recorded on a Hewlett-Packard HP8452A diode array spectrophotometer in 1 cm optical Pyrex sample cells using dichloromethane as the solvent.

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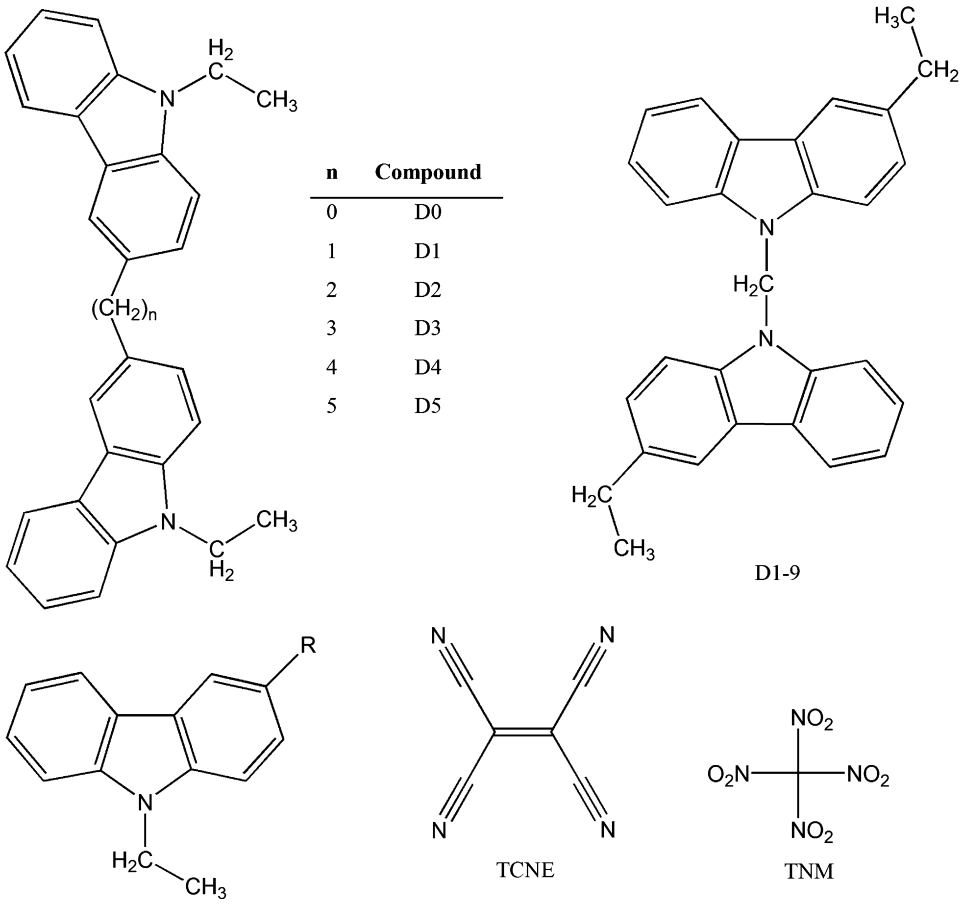


n	Compound
0	D0
1	D1
2	D2
3	D3
4	D4
5	D5

R	Compound
H	M1
CH_3	M2
CH_2CH_3	M3

Scheme 1. Molecular structures of the compounds discussed in this paper.

2.2. Materials

9-Ethylcarbazole (M1) (Aldrich) was purified by column chromatography (80–200 mesh alumina) eluting fractionally with hexane/dichloromethane and by recrystallization from ethanol. 9-Ethyl-3-methylcarbazole (M2) and 3,9-diethylcarbazole (M3) were prepared via the reduction of 9-ethylcarbazole-3-carboxaldehyde (Aldrich) and 9-ethyl-3-acetylcarbazole using $AlCl_3/LiAlH_4$ as the reducing agent in anhydrous ether. 9,9′-Diethyl-3,3′-dicarbazolyl (D0) was prepared according to the literature procedure [20] via oxidation of 9-ethylcarbazole by $FeCl_3$. Di(9-ethylcarbazol-3-yl)methane (D1) was prepared via acid-catalyzed condensation of 9-alkylcarbazoles with formaldehyde [21]. Di(3-ethylcarbazol-9-yl)methane (D1-9) was prepared through the reaction between dibromomethane and potassium salt of 3-ethylcarbazole according to the literature [22]. 1,2-Di(9-ethylcarbazol-3-yl)ethane (D2) was prepared via catalytic (Pd-C) hydrogenation of 1,2-di(9-ethylcarbazol-3-yl)ethane, which was prepared by the reductive coupling of 9-ethylcarbazole-3-caboxaldehyde according to the literature procedure [23]. 1,3-Di(9-ethylcarbazol-3-yl)propane (D3) was prepared via the reduction of 1,3-di(9-ethylcarbazol-3-yl)propenone, which was prepared through a base catalyzed aldol condensation of 9-ethyl-3-acetylcarbazole with 9-ethylcarbazole-3-aldehyde [24]. Literature procedure [25] for the preparation of 3,6-disubstituted carbazoles was modified for the preparation of 1,4-Di(9-ethylcarbazol-3-yl)butane (D4). The procedure involves cross coupling of 3-bromo-9-ethylcarbazole with the di-Grignard reagent of 1,4-dibromobutane in the presence of $NiCl_2$(dppp) catalyst [dppp = l,3-bis(diphenylphosphino)propane]. 1,5-Di(9-ethylcarbazol-3-yl)pentane (D5) was prepared via the catalytic hydrogenation (Pd-C) of 1,5-bis(9-ethylcarbazol-3-yl)-1,3-pentadiene, which was prepared according to literature [26], through a bis-Wittig reaction between 1,3-bis(triphenylphosphonium)propane dibromide and 9-ethylcarbazol-3-carboxaldehyde. TCNE (Aldrich) was purified by successive sublimation and crystallization from dichloromethane. TNM prepared according to the literature procedure [27] and purified by steam distillation followed by repeated extraction with distilled water and freeze–thaw degassing to remove traces of NO_2. Dichloromethane (Quantum Chemical Co.) was purified by distillation over P_2O_5 under argon atmosphere prior to using in the spectrophotometric measurements.

2.3. Absorption measurements

The Benesi–Hildebrand technique [28] was used for the determination of the equilibrium constants and charge-transfer molar extinction coefficients (ε) for the formation of EDA complexes of

the dicarbazolylalkanes with TCNE and TNM. For the carbazole-TCNE CT measurements, in a 1.0 cm UV cuvette 1.0 mL of 0.06 M TCNE:0.75 mM carbazole solution was placed. This was diluted 10 times by addition of increments of 0.1 mL and 10 times by addition of increments of 0.2 mL of the 0.75 mM carbazole solutions, to make total of 20 dilutions. The TCNE–carbazole concentration ratios were varied from about 80:1 to 20:1 through a series of 20 dilutions with stock solutions of carbazoles. Thus, the concentration of the electron donor (carbazole unit) was kept constant whereas the concentration of the electron acceptor (TCNE) decreased throughout the experiment. For the carbazole–TNM CT measurements, the initial concentrations of 4.64 mM carbazole and 0.6 M TNM concentration were used. The carbazole–TNM concentration ratios were varied from about 129:1 to 30:1 by diluting initial solution with 5.0 mM carbazole solutions. Absorbance measurements of the samples containing TNM had to be collected in dim red light due to the rapid photochemical reactions with carbazoles. D0 readily reacts with TNM to give nitration products, even in dark. Hence, it was excluded from the complexation studies with TNM.

Absorbance changes were monitored after each dilution by measuring the optical density (O.D.) at the wavelength of the interest. Average of three runs was taken to minimize the experimental errors. The wavelengths of the maximum absorbance were determined from the UV–vis spectra of the complexation.

All carbazoles formed blue EDA complexes with TCNE and brown complexes with TNM in dichloromethane according to following hypothetical equation.

$$D + A \overset{K}{\rightleftharpoons} D, A$$

The equilibrium constant K for the above reaction can be written as

$$K = \frac{[D, A]}{([A]_0 - [D, A])([D]_0 - [D, A])} \quad (1)$$

The equilibrium constants (K) for EDA complex formation are related to the maximum absorption (A) of the complex, the molar extinction coefficient (ε), and the initial concentrations of donor ($[D]_0$) and acceptor ($[A]_0$). Replacing [D,A] with (A/ε) from Beer–Lambert law and ignoring [D,A] concentration in ($[A]_0 - [D,A]$) term since $[A]_0 \gg [D]_0$, Eq. (1) yields the Benesi–Hildebrand equation.

$$\frac{[D]_0}{A} = (K\varepsilon)^{-1}\left(\frac{1}{[A]_0}\right) + (\varepsilon)^{-1} \quad (2)$$

Ploting $[D]_0/A$ vs. $(1/[A]_0)$, $(K\varepsilon)^{-1}$ as the slope and $(\varepsilon)^{-1}$ as the intercept were obtained. Benesi–Hildebrand plots of carbazole–TCNE and carbazole–TNM EDA complexes are given in Figs. 1 and 2, respectively.

The stoichiometries of the complexation of carbazoles with TCNE were determined using Job plots [29] (method of continuous variation). In 5.0 mL volumetric flasks, 10 mM of carbazole donor and 10 mM of TCNE in dichloromethane were prepared separately. These solutions were mixed in a 1.0 mL volumetric flask in which the mole fraction of the components differed from 0.1 to 0.9. The average absorptions of four different scans of the EDA complexes on each dilution were recorded at the maximum CT wavelength. Job plots of the carbazole–TCNE complexes are given in Fig. 3.

Temperature dependencies of complexations were determined using van't Hoff equation and Beer–Lambert law by measuring absorption spectra of the complexes at six different temperatures, 0 °C, 7 °C, 14 °C, 21 °C, 28 °C, and 35 °C (±1 °C), at λ_{CT}. In a 10 mL volumetric flask, a solution containing 5.0 mM TCNE and 2.5 mM carbazole unit (and 0.4 M TNM and 5 mM carbazole unit) at 21.0 °C was prepared. Then the solution was placed into UV a cell with

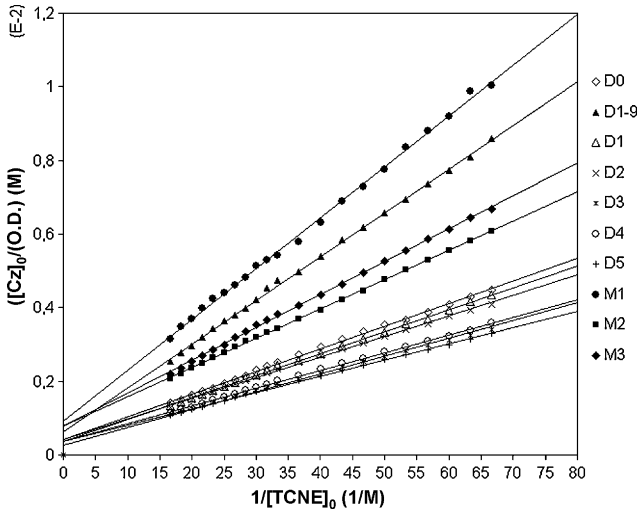


Fig. 1. Benesi–Hildebrand plots of carbazole–TCNE EDA complexes.

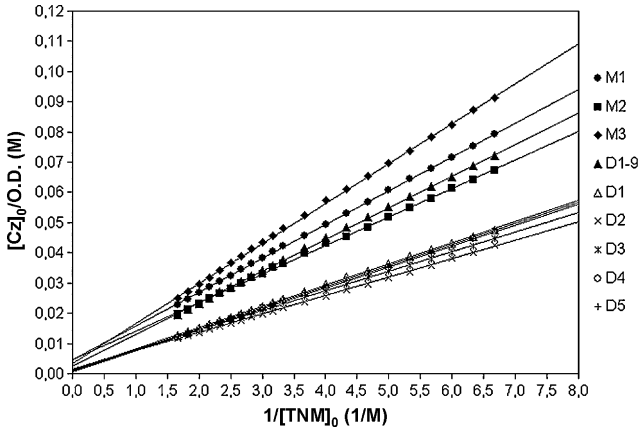


Fig. 2. Benesi–Hildebrand plots of carbazole–TNM EDA complexes.

1 cm path length and equilibrated in water or ice-water baths of the desired temperatures for 15–20 min. The UV cell was dried off with paper wipes prior to every scan and average of three scans were taken for the calculations. The volume changes were measured for these temperatures and concentration changes were taken into account in calculating the thermodynamic constants.

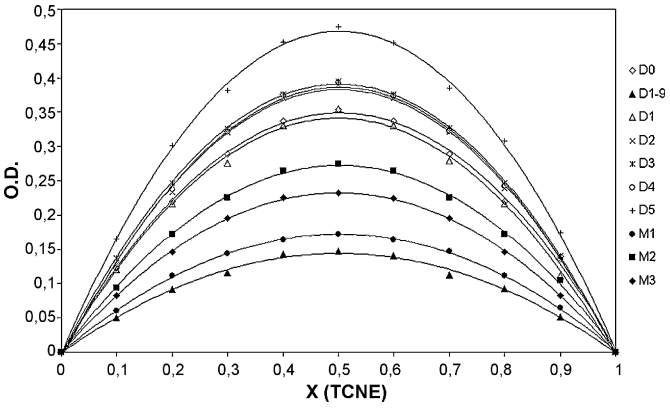


Fig. 3. Job plots of carbazole–TCNE EDA complexes.

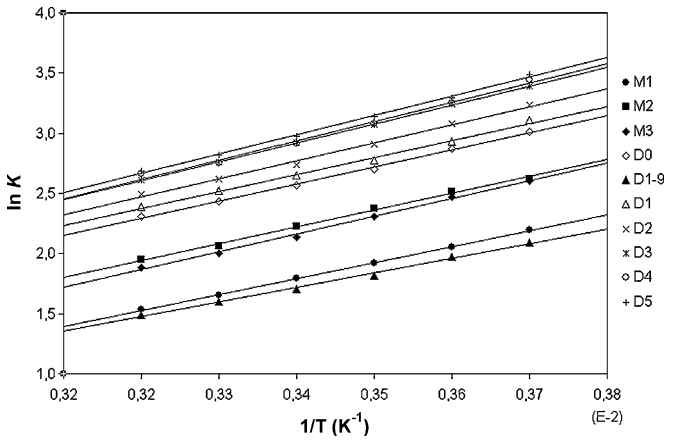


Fig. 4. Van't Hoff plots of carbazole–TCNE EDA complexes (2.5 mM donor-group, 5 mM TCNE).

The integrated form of the van't Hoff equation is

$$-\left(\frac{\Delta H}{R}\right)T^{-1}+\left(\frac{\Delta S}{R}\right)=\ln\left[\frac{A/\varepsilon}{([D]_0-A/\varepsilon)([A]_0-A/\varepsilon)}\right] \quad (3)$$

The value of the molar extinction coefficient, ε, was calculated as 1260 M^{-1} cm^{-1} for carbazole–TCNE complexes and 380 M^{-1} cm^{-1} for carbazole–TNM complexes from the Benesi–Hildebrand plots. A plot of ln K vs. $1/T$ in Eq. (3) would yield $-\Delta H/R$ as the slope and $\Delta S/R$ as the intercept. The van't Hoff plots of carbazoles with TCNE and TNM are given in Figs. 4 and 5, respectively.

3. Results and discussion

Formation of a CT complex results from the association of an electron donor with an electron acceptor. The ionization potential IP of a donor and the electron affinity E_a of an acceptor are the major factors in determining the strength of the complexation. Carbazoles are good electron donors having low IP's around 7.6–8.0 eV [30,31] and TCNE is a good electron acceptor with E_a = 3.17 eV [32]. TNM forms more weakly bound complexes with carbazoles compared to TCNE. The tetrahedral structure of TNM, a σ-acceptor, may prevent a stronger association with planar donor compounds when compared with planar TCNE molecule whose four –CN groups are in conjugation with each other. A CT absorption occurs when one electron from the HOMO (or the lower energy orbitals) of the donor is promoted to the LUMO of the acceptor (Scheme 2). EDA complexes absorb light in a manner different than either donor or acceptor molecules, although the physical properties of the original components are, to a large extent, preserved. When the ground state of an EDA complex absorbs light that matches the excitation energy of the complex ($h\nu_{CT}$), the excited state is formed. In this excited state, an electron is transferred from one of the higher occupied molecular orbitals of the excited donor (D^*) to the lowest unoccupied molecular orbital (LUMO) of the acceptor (A) to form a pair of ions ($D^{\bullet+}$, $A^{\bullet-}$).

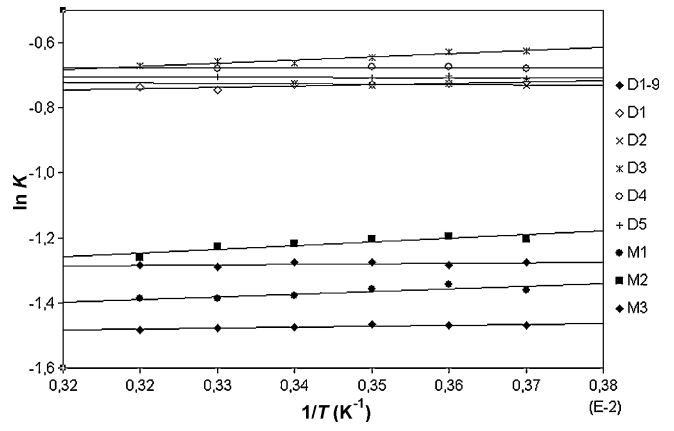
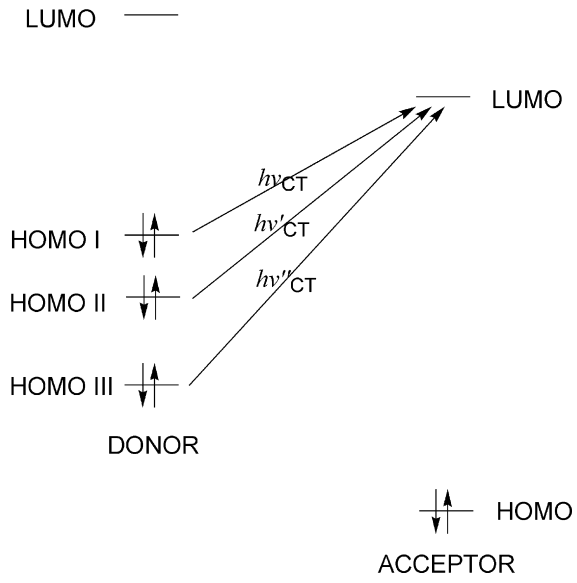


Fig. 5. Van't Hoff plots of carbazole–TNM EDA complexes (5 mM donor group, 0.4 M TNM).



Scheme 2. Representation of CT process between HOMO's of an electron donor and LUMO of an electron acceptor molecule.

The Benesi–Hildebrand technique was used to determine the molar extinction coefficient, ε, and equilibrium constant, K, for the weak donor–acceptor complexes.

The average molar extinction coefficient of carbazole–TCNE complexes was determined to be 1260 M^{-1} cm^{-1}, where that of carbazole–TNM complexes found to be 380 M^{-1} cm^{-1}. These results are consistent with the results of the earlier studies [19]. The values of equilibrium constant, K, ranged between 3.34 and 8.98 M^{-1} for the carbazole–TCNE complexes and between 0.199 and 0.431 M^{-1} for the carbazole–TNM complexes (Table 1). In dicarbazolylalkanes, D1-9 has the smallest K values for its complexations with both TCNE and TNM. D0 (with TCNE) and D1 have the smallest K values among the dicarbazoles that are bridged through carbazole benzene rings. This result is attributable to the higher electron density at nitrogens, which are not allowed to associate freely with the donor molecules in D1-9 due to the proximity of the carbazole rings. Comparably, in D0, where the two carbazole groups are directly attached to each other at the *para* position to the nitrogen atom, and D1, where the two carbazole groups are separated with a methylene group, effect of proximity is far less than it is in D1-9. The presence of electron donating ethyl groups on the carbazole rings of D1-9 had little effect on the complexation compared to its unsubstituted analogue (K = 3.13 M^{-1}; Ref. [19]). The K values of D0 and D1 found to be higher than D1-9 but expectedly lower than M2, M3, and D2–D5. Nearness of carbazole groups in D0 and D1 prevented complexation of each carbazole group independently. The equilibrium constants for M2, M3, and D3–D5 were similar. This indicates that for the dicarbazolyl alkanes where $n \geq 3$ there is little effect of the carbazole rings on each other to prevent formation of complexes with TCNE. It can be concluded that the length of methylene chain has a positive effect on the complexation for the dicarbazoles D1–D3, however, this effect was diminished in the dicarbazoles with $n \geq 3$.

The temperature dependences of complexations were determined by calculating the enthalpy and entropy from Eq. (3). The

Table 1
Thermodynamic properties of EDA complexes of selected carbazoles with TNM in CH_2Cl_2

TCNE	λ[a]	λ_{CT}[b]	$K\varepsilon_{CT}$[c] (M^{-2} cm^{-1})	K[d] (M^{-1})	ΔH (kcal/mol)	ΔS (eu)
M1	347	596	7,235 ± 69	5.74	−3.12 ± 0.03	−7.08 ± 0.11
M2	352	610	12,489 ± 51	9.91	−3.27 ± 0.13	−6.76 ± 0.46
M3	352	614	11,200 ± 74	8.89	−3.48 ± 0.07	−7.56 ± 0.23
D0	347	594	8,079 ± 77	6.41	−3.35 ± 0.05	−6.31 ± 0.19
D1-9	341	598	4,209 ± 30	3.34	−2.85 ± 0.07	−6.31 ± 0.23
D1	352	613	8,377 ± 61	6.65	−3.33 ± 0.07	−6.06 ± 0.23
D2	352	611	8,926 ± 78	7.09	−3.54 ± 0.07	−6.56 ± 0.25
D3	352	612	10,829 ± 70	8.59	−3.70 ± 0.07	−6.82 ± 0.24
D4	352	613	10,376 ± 80	8.24	−3.76 ± 0.13	−6.99 ± 0.45
D5	352	611	11,318 ± 81	8.98	−3.77 ± 0.04	−6.92 ± 0.15
TNM	λ[a]	λ_{CT}[b]	$K\varepsilon_{CT}$[c] (M^{-2} cm^{-1})	K[e] (M^{-1})	ΔH (kcal/mol)	ΔS (eu)
M1	347	534	89.3 ± 0.22	0.234	−0.19 ± 0.06	−3.36 ± 0.20
M2	352	547	105.7 ± 0.64	0.275	−0.27 ± 0.07	−3.35 ± 0.25
M3	352	550	79.6 ± 0.34	0.199	−0.06 ± 0.02	−3.14 ± 0.08
D1-9	341	536	95.8 ± 0.42	0.251	−0.04 ± 0.03	−2.67 ± 0.11
D1	352	549	140.1 ± 0.26	0.187	−0.09 ± 0.03	−1.76 ± 0.10
D2	352	547	163.8 ± 0.36	0.215	0.13 ± 0.07	−0.99 ± 0.23
D3	352	548	154.3 ± 0.34	0.203	−0.23 ± 0.04	−2.08 ± 0.12
D4	352	549	144.4 ± 0.37	0.190	0.00 ± 0.03	−1.35 ± 0.11
D5	352	548	143.9 ± 0.32	0.189	0.02 ± 0.04	−1.35 ± 0.14

[a] Lowest energy absorption maximum of uncomplexed carbazole (nm).
[b] Lowest energy CT maximum (nm).
[c] Acceptor in excess.
[d] $\varepsilon = 1260\ M^{-1}\ cm^{-1}$ at 21 ± 1 °C.
[e] $\varepsilon = 380\ M^{-1}\ cm^{-1}$ at 21 ± 1 °C.

formation enthalpies around −3.5 kcal/mol shows that carbazoles form weakly associated complexes with TCNE. The effects of the alkyl substituents on the carbazole rings and the length of the alkylene separator can be judged from the data (Table 1). It was found in the studies of Haderski et al. [19] that 1,*n*-dicarbazol-*N*-ylalkanes form weak complexes with TCNE having formation enthalpies around −3 kcal/mol. The slightly more favorable ΔH values in this study may be due to the presence of an extra electron donating group (–ethyl) on each carbazole ring in addition to the alkylene chain. The effect of alkyl substituents on the formation enthalpies can be seen more clearly when the ΔH values of monocarbazoles compared where M2 and M3 have more negative enthalpies compared to M1. The formation enthalpies of D0, D1, and D1-9 were the least favorable among the dichromophores.

The near-zero enthalpies of formation for the compounds studied indicate that there is almost no temperature effect on the complexations of carbazoles with TNM. There was no noticeable effect attributable to the substituents or to the number of $–CH_2–$ groups (n) in the spacer connecting the two carbazolyl groups.

The effect of the alkyl substituent on the formation entropies of the TCNE complexes depends on the size of the alkyl group. The methyl group in M2 (-6.76 ± 0.46 eu) has no noticeable effect whereas the ethyl group in M3 (-7.56 ± 0.23 eu) significantly changed the entropy, unfavorably, compared to that of M1 (-7.08 ± 0.11 eu). The chain length in dicarbazoles appears to have a negative effect on the formation entropies. The complex formation for D0, D1, and D1-9 is entropically more favorable compared to D2–D5. However, this effect disappeared among D3–D5, in which the carbazolyl groups are separated with longer alkylene chains.

Entropies of formation for the monocarbazole–TNM complexes were found to be more negative (i.e. more unfavorable) than those for the dicarbazolylalkanes. For the TNM–dicarbazolylalkene complexes the ΔS values of D1–D5 are considerably larger than those of M1–M3 and D1-9. It appears that the two carbazolyl groups complex essentially independently for D1–D5, but not for D1-9. Independent behavior of the two carbazolyl groups would increase the entropy of complexation in D1–D5 by $R \ln 2$ (or about 1.4 eu) compared to the monocarbazole analogues. As seen in Table 1, the experimental values differ by close to this amount. The difference in the complex formation behavior of D1-9 and D1 can be ascribed to the more open geometry of D1, which places the carbazolyl groups farther apart so that acceptors experience less steric interactions with the second carbazolyl group.

4. Conclusion

1,*n*-Di(9-ethylcarbazol-3-yl)alkanes and selected monocarbazoles form weak EDA complexes with TCNE and even weaker EDA complexes with TNM as indicated by the thermodynamic constants ΔH and ΔS. The K values of D0–D2 were found to increase in the same order as the length of their methylene chain, while the K values for D3–D5 were about the same. Relatively larger K values for the complexation of M2 and M3 with TNM and TCNE compared to M1 indicate that electron-donating alkyl substituents on the benzylic rings of carbazoles also favor the complexation. All the compounds form 1:1 carbazole group–TCNE complexes at the concentrations studied. The electron-donating methyl and ethyl substituents in M2 and M3 make the enthalpies more negative (i.e. more favorable for the complex formation) compared to M1. In the dicarbazoles, the complex formation is favored for the compounds with $n \geq 2$. The entropies of formation for the carbazole–TCNE complexes also become more negative (less favorable) in M2 and M3 and D2–D5. Similar trends were observed in carbazole–TNM complexes except that their enthalpies of formation were found to be between −0.27 and +0.13 kcal/mol. The entropies of formation for complexation with TNM are found to be less negative (less unfavorable) than those for TCNE, consistent with their "contact" in nature behavior.

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