SYNTHESIS AND SPECTRAL PROPERTIES OF POLYENE BIS-w,w'-DIAMINOKETONES

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This article describes the synthesis of polyene bis- ω, ω '-diaminoketones (BDAK) from

aminals and aminal-acetals $Me_2N(CH=CH)_nCH$ (I) (n = 2, 3; Y = NMe₂, OMe) [1], and a study Y

of their spectral properties.

We have previously shown [2, 3] that compounds (I) (n = 1) and 1,1,3-trimethoxy-3-dimethylaminopropane (II) are convenient reagents for the synthesis of BDAK

$$\begin{array}{c} 0 & 0 \\ \begin{matrix} & & \\ C \\ (n = 1) \\ (n = 1) \end{matrix} (II) + H_2C \\ \begin{matrix} & \\ H_2C \\ II \\ R^1 \\ R^2 \end{matrix} \rightarrow Me_2N(CH=CH)_nCH=C \\ \begin{matrix} & \\ C \\ C=CH(CH=CH)_mNMe_2 \\ II \\ R^1 \\ R^2 \end{matrix}$$
where $n = m = 1$; R^1 $R^2 = H$, $(CH_2)_k$, $k = 2-4$.

A number of polyconjugated BDAK with n = 1 and m = 2-4 have been obtained by condensing (I) (n - 1) and (II) with cyclic and acyclic α,β -unsaturated ketones and their vinylogs [3]. However, the use of difficultly accessible conjugated ketones as starting materials considerably restricts the usefulness of this method. In addition, the development of methods for the synthesis of polyconjugated BDAK, which are ketocyanines, is of considerable importance in view of the fact that ketocyanines are of promise in quantum electronics [4], and, furthermore, they may be used to obtain cyanine dyes, which are efficient sensitizers [5].

A study of the reactions of (I) (n = 2, 3) with ketones with the aim of synthesizing polyene BDAK showed that aminals and aminal-acetals with n = 2 condensed smoothly with a variety of ketones at both methyl and methylene groups to give the BDAK (III), (V), (VI), and (VIII) (Table 1). It is noteworthy that (III) and (VIII), obtained by reaction of (I) (n = 2) with cyclohexanone or acetone in yields of 60 and 98%, have been synthesized previously in yields of only 30% [6] and 34% [3]. The condensation of ketones with (I) (n = 3) requires more severe conditions, under which the polyene BDAK formed are unstable. Only cyclopentanone and acetone afforded BDAK (IV) and (IX) in very low yields, these compounds containing eight double bonds.

In order to study the relationship of the spectral properties of BDAK to their structure, the following have also been prepared: (VII) by condensation of (I) (n = 1) with the δ -dimethylaminodienone (XIII); (X) by condensation of (I) (n = 2) with β -dimethylaminovinyl methyl ketone; and (XI) and (XII) from (I) (n = 1 and 2) and p-diacetylbenzene. It is noteworthy that, in addition to (XII), the monodimethylaminoketone (XXX) was also formed (ratio 2:1) irrespective of the ratio of the reactants.



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The BDAK and diketones (III)-(XII), their yields, and UV and PMR spectral data are shown in Table 1. The polyene BDAK and diketones were crystalline solids varying in color from red to deep violet, sparingly soluble in organic solvents. The PMR spectra of (III), (V), (VII), (XI), and (XII) (J = 12-15 Hz) show that these compounds, like BDAK previously obtained [2, 3] possess the trans-configuration of the methine protons, and occur as the Strans-conformers.

It has been noted [3, 6] that ketocyanines display positive solvatochromism. Examination of the UV spectral data in $C_{2}H_{5}OH$ and $CHCl_{3}$ shown in Table 1 shows that these BDAK also display positive solvatochromism.* The considerable solvatochromism of ketocyanines, as has been pointed out previously [4, 6], is particularly apparent in proton-donor solvents, owing to the formation of hydrogen-bonded complexes with the keto group, which possesses strong electron-donor properties. In the case of the BDAK (XXII), in fact (Table 2), it will be seen that when the capacity of the solvent for specific solvation (characterized by the parameter $E_{\rm T}$) is increased, $\lambda_{\rm max}$ is shifted bathochromically; for instance, $\Delta\lambda_{\rm max}$ in water and hexane reaches 100 nm.

On acidification of alcoholic solutions, the bathochromic shift reaches as much as 190 nm, the λ_{max} value of 590 nm approaching λ_{max} for the corresponding ethoxynonamethine salt [9].

Me2NCH=CHCH

It follows from the ¹³C NMR spectral data for (XXII), shown in Table 3, that the chemical shifts (CS) of the carbon atoms of the polymethine chain and the carbonyl group also vary with the type of solvent. The greatest change in the CS for the β , δ , α , and γ -carbon atoms occurs between $Cl_2C=CCl_2$ and CD_3OD . These changes in the CS are due primarily to such properties of the solvent as its specific solvation.

It was found that, as in the case of the UV absorption spectra, $\delta^{13}C$ depended on ET (Table 3). The signals for Cg, C_Y, and C_{\delta} were shifted to lower field, and C_{\alpha} to higher field ($\Delta\delta C_{\beta} > \Delta\delta C_{\gamma}$). It is interesting that the change in CS on exchanging CDCl₃ for CD₃OD is similar to that observed between BDAK (XXII) and the salt (XXXI) (Table 3).

It has been noted [4, 10] that ketocyanines containing four double bonds, in which the nitrogen atom forms part of the heterocyclic residue, display intense fluorescence and a large Stokes shift. It was of interest to examine the fluorescence spectra of our polyconjugated BDAK, together with previously obtained BDAK, with alkyl and aryl substituents on the nitrogen atom. For this purpose, the spectra were recorded and the quantum yields of the fluorescence of BDAK in ethanol and hexane determined, and in the cases of (XXII) and (XXIII), in other solvents in addition. These data, together with the λ_{max}^{abs} values for the UV spectra and the values of the Stokes shift, are given in Tables 2 and 4. These data show that the spectra and the quantum fluorescence yields of these BDAK, like those of the absorption spectra, are dependent on the polarity and proton-donor capacities of the solvents.

In consequence of the formation of H-bonded complexes, the fluorescence spectra in proton-donor solvents are shifted toward the red in comparison with the spectra in aprotic, nonpolar solvents, and they display an anomalously high Stokes shift which increases with increasing polyene chain length, reaching values of 170-180 nm, for instance in the series (XIV-XVII) and (XXI-XXVI). All the compounds fluoresced very weakly in hexane ($\varphi_{fl} \sim 10^{-3}$). As the polarity of the solvent increased, the quantum fluorescence yield also increased, the maximum values of φ_{fl} being observed in proton-donor solvents capable of forming hydrogen bonds with the carbonyl group of the BDAK (Table 2).

In many carbonyl compounds, the ${}^{1}\pi\pi^{*}$ - and ${}^{3}n\pi^{*}$ -levels are close to each other, resulting in highly efficient intercombinational transition in the triplet state and, consequently, to low values of φ_{ll} (spectral luminescence group IV) [13].

^{*}A systematic study of the relationship of the UV absorption spectra of BDAK to their structure has been initiated [7].

| Ketones and Diketones (solvent CDC13; for | | |
|--|--------------------------------------|--|
| PMR Spectra of Polyene Bis- ω , ω 'diamino | | |
| 1. Yields, Constants, and UV and 1 | (V), and (VIII), $CDCl_3 + CD_3OD$) | |
| TABLE 1 | (III), | |



On heating (XII) > 195°C, λ_{max} at 500, 360, and 290 disappeared, and and M+ Phc--C₆H₄--CPh deamination, occurring in the mass spectrometer. 265 nm appeared, corresponding to PhackHab. 0 λ_{\max}

Ъу

cyclization accompanied

of intramolecular

a result

as

and 286, probably $M^+ Me_iN(GH=GH)_s Q - C_s H_t - QPh$

m/z 331

TABLE 2. Absorption Maxima $(\lambda_{max}^{abs}, nm)$, Fluorescence Maxima (λ_{max}^{fl}, nm)

| | ····· | | | (XXII) | | (X2 | (III) |
|---|------------------------|----------------------------|--------------------------------------|-----------------------|------------------------|------------------------|-----------------------|
| Solvent | μ, D | E _T , kcal/mole | λ_{\max}^{abs} | λ_{\max}^{fl} | Φfl | λ_{\max}^{abs} | λ_{\max}^{fl} |
| C6H14 C6H12 | 0 | 30,9 31,2 | 400, 415 † 410 † | 455, 480 † | 0,002 | 415, 439 T | 460, 480 † |
| $Cl_2C = CCl_2$ CCl_4 $CH_3C_6H_5$ | 0 0 0,39 | 32,5 33,9 | 420 + 430 + 435 + | 473 487 | 0,006 | 432, 452 † | 473 |
| C_6H_6 CH ₃ COOC ₂ H ₅ | 0 1,78 | 34,5 38,1 | 430 † 445, 430 pl. | 503 | 0,009 | 432, 450 T | 505 |
| CHCl3 CH2Cl2 CH3COCH3 | $1,15 \\ 1,55 \\ 2,88$ | 39,1 41,1 42,2 | 460 460 455, 438 pl | 505 | 0,008 | | |
| ДМФА ДМСО CH ₂ CN | 3,86 3,96 3,37 | 43,8 45,0 46,0 | 460 460 460 460 | 533 510 | 0,08 0.035 | 458 | 536 |
| <i>n</i> -C ₃ H ₇ OH абс. C ₂ H ₅ OH CH ₃ OH | 1,68 1,68 1,66 | 50,7 51,9 55,5 | 480 485 490 | 547 548 558 | 0,09 0,082 0,078 | 480 | 575 |
| $\begin{array}{c} OHCH_2CH_2OH \\ H_2O \\ C_2H_5OH+ \\ 1\% HCI \end{array}$ | 2,28 1,84 | 56,3 63,1 | 492 510 ‡ 590 | 570 572 623 | 0,027 0,025 0,03 | 625 | 672 |

nm), and Quantum Fluorescence Yields $(\varphi_{fl}) \xrightarrow{Me}_{R}$ NCH=CHCH=(1)=CHCH= CHN $\langle \frac{Me}{r}$; R = Me (XXII) and Ph (XXIII) in Various Solvents

*ET values obtained from [8]. †Split.

[‡]Freshly prepared solution; Me,NCH=CHCH=CHCHO, formed on

standing, apparently as a result of hydrolysis, since λ_{max} 510 nm disappears and λ_{max} at 438 and 285 nm appear.

When polar solvents are used, in which complex formation with proton donors is possible, the distribution of levels may vary, as a result of which the ${}^{1}\pi\pi^{*}$ -level may fall below the ${}^{3}n\pi^{*}$ -level, and fluorescence is enhanced (spectral luminescence group (V). An analogous situation is apparently to be found in these BDAK.

As in other luminophores [14], as the length of the polyene chain is increased, the quantum fluorescence yield at first increases, then falls, passing through a maximum when n = 2 and m = 2 or 3 [compounds (XV), (XVI), (XXII), and (XXV)]. The decrease in the quantum yield as the polyene chain length increases is apparently due to a decrease in the rigidity of the molecule, and hence to an increase in the rate of internal conversions.

For a given length of polyene chain, the quantum fluorescence yield decreases on passing from the linear BDAK (XIV)-(XVIII) and (VIII) to the cyclic (III)-(VII) and (XXI)-(XXVII), although λ_{max}^{abs} , λ_{max}^{fl} , and the Stokes shift undergo little change. The introduction of Me substituents, and, more particularly Ph, into the polyene chain substantially reduces the quantum fluorescence yield [cf. compounds (XV), (XIX), and (XX)]; also, it appears, as a result of a reduction in the rigidity of the molecule. Replacement of the methyl groups on nitrogen in (XXII) by phenyl [compounds (XXIII) and (XXIV)] results in an increase in the fluorescence yield, the fluorescence maxima shifting to longer wavelengths (549, 578, and 590 nm). It is interesting that the absorption maxima λ_{max}^{abs} for (XXII), (XXIII), and (XXIV) are the same (490 nm), whereas λ_{max}^{abs} for their protonated forms, obtained by acidifying the alcoholic solutions, are, respectively, 590, 625, and 640 nm, i.e., they vary in the same sequence as the fluorescence maxima.

| Compound | CH2 | NHe2 | cα | с ^у | с'n | с _ð | C=0 | Solvent | E _T , kcal/mole |
|----------------------------------|--------------------------|-------------------------------------|--------|----------------|-------|----------------|--------|----------------------------------|------------------------------|
| (IIXX) | 23,8 and 27,45 | 40,76 | 126,00 | 136,66 | 97,18 | 148,44 | * | $Cl_2C = CCl_2 + 5\% C_6D_6$ | |
| (IIXX) | 23,87 and 27,51 | 39,93 | 125,64 | 137,61 | 96,26 | 149,34 | 185,82 | C ₆ D ₆ | 34,5 |
| (XXII) | 22,88 and 26,58 | 40,53 | 124,61 | 137,69 | 95,43 | 149,94 | 186,61 | CDC1 ₃ | 39,1 |
| (IIXX) | 23,58 and 27,00 | 40,47 | 124,37 | 138,14 | 95,32 | 151,19 | 185,88 | CD ^s CN | 46,0 |
| (IIXX) | 23,91 and 27,3 | 40,86 | 123,88 | 142,10 | 96,58 | 153,37 | 188,44 | CD ₃ OD | 55,5 |
| (XXXI) | 21,5 and 25,1 | 42,39 and 42,30 | 120,1 | 147,8 | 102,0 | 159,4 | | CDC1 _s | |
| $\Delta\delta$ (XXII) = δ | $(inCD_3OD) - \delta(in$ | ר C ₆ D ₆) : | -1,76 | +4,49 | +0,38 | +4,03 | - | | $\Delta E_T 21,0$ |
| $\Delta\delta$ (XXII) = δ | $(inCD_3OD) - \delta(in$ | CDCl ₃): | -0,73 | +4,41 | +1,15 | +3,43 | | | ΔE_{T} 16,4 |
| Δδ=δ(XXXI) |) -ô(XXII) (in (| GDCl ₃) : | 4,5 | +10,1 | +6,6 | +9,5 | | | |
| | | | | | | | | | |

¹³C NMR Spectra of (XXII) in Various Solvents at ~38°C, and the Salt (XXXI), in CDCl₃ TABLE 3. *Signal not accumulated as a result of low concentration due to the low solubility of the sample. +171.6 (COEt), 15.4 and 15.7 (Me), 62.9 and 73.6 (CH₂).

 $(\lambda_{\max}^{f1},$ nm) of Bis- ω , ω '-diaminoketones in Ethanol* A max λ_{\max}^{fl} abs1 Compound n m k R١ \mathbb{R}^2 R³ λ_{\max} Φţι Amax. R1 \R² (XIV) [3] CH_{\bullet} CH3 430 1 47646 0,011 (X) (XV) [2] 32 CH3 1 2 CH_3 450566116 0,031 CH_3 CH₃ 470 543 73 0.122 (372) (460)(88) (~0,001) $\begin{array}{c}
 2 \\
 2 \\
 2 \\
 3
 \end{array}$ 3 (XVI) [3] CH₃ CH₃ **`**507 119 626 0.144(XVII) [3] CH₃ CH₃ 5224 5 3 702180 0.022XVIII) [3] 530 CH₃ CH₃ ≪0,001 CH3 (VIII) 555678 CH₃ 1230,022 R Q R. N-CH=CCH=CHCCH=CHC=CHN R1 (XIX) [11] CH3 500 2 2 $\frac{2}{2}$ CH_3 CH3 0,018 548 48 (XX) [12] CH₃ CH_3 C_6H_5 500 552550,005 (414) (482) (56)(~0,001) 2 C=CH(CH=CH)m--(CH=CH)_{n-1}CH=C (ÈH2)k (XXI) [3] (XXII) [2] CH₃ 2 2 2 2 2 1 3333 CH3 440 491 0,003 51 2 CH₃ CH₃ 490 549 59 0,08 (XXIII) [2] $\frac{\overline{2}}{2}$ CH₃ C₆H₅ 4900,145 578 88 (XXIV) C_6H_2 C₆H₅ 490590 100 0,165 (438)(485)(47) 0,001) (XXV) [3] (XXVI) [3] 2 3 3 CH₃ CH_3 **`**512 630 118 0,056 2 3 3 3 4 CH₃ 532 3 3 2 CH3 705 0.013 173 (V) (VI) (III) 3 CH₃ CH₃ 675 560 115 0.017 CH3 4 CH₃ 702 700 575149 0,007 CH3 $\widetilde{\mathrm{CH}}_3$ 3 585 115 80 0,027 (IV) 4 $\overline{2}$ 4 CH₃ CH_3 625 705 0,004 N(CH=CH)_{n-1}CH CH=CH)m (VII) CH₃ 2 2 CH₃ 469 565 0.057 (390) 505 (446) 626 (XXVII) [3] $\mathbf{2}$ CH₃ 3 CH₃ 121 0,014 (406) (495) (89)CH) (CH=CH)_nN (XI) (XII) CH₃ CH₃ 2 3 CH_3 0,002 460 55090 CH3 530 552 22 0,025

Absorption Spectra (λ_{max}^{abs} , nm) and Fluorescence Spectra TABLE 4.

*Values in hexane given in brackets.

A comparison of the spectral properties of the BDAK (XV) and the diketone (XI) shows them to have very similar absorption and fluorescence maxima, but that the quantum fluorescence yield of the diketone (XI) is much lower.

In the corresponding aromatic ketone (XXVIII) and diketone (XXIX), the absorption and fluorescence λ_{max} values are similarly close in toluene, but the quantum fluorescence yield for the diketone is ten times greater [15].



EXPERIMENTAL*

PMR spectra were obtained on Tesla BS-497 (100 MHz) and Bruker WM-250 (250 MHz) instruments, and ¹³C NMR spectra on a Bruker WM-250. UV spectra were obtained on a Specord UV-VIS instrument. Fluorescence spectra were measured on an Élyumin 2-M (FÉU-38) spectrofluorimeter, with solutions of concentration $(1-2) \cdot 10^{-5}$ mole/liter (optical density 0.1-0.3). Spectra were corrected using a 10^{-4} solution of m-dimethylaminonitrobenzene as standard [16], and the standards for the determination of the relative quantum yields were: Na fluorescence in ethanol ($\varphi_{fl} = 0.85$) and Rhodamine C in ethanol ($\varphi_{fl} = 0.49$) [17], accuracy about 15%.

(I) (n = 2 and 3) were obtained as described in [1]; the proportions of the aminal $[Y = N(CH_3)_2]$ and the aminal-acetal (Y = OCH₃) in (I) (n = 2 and 3) were determined by PMR, being 3:2 (n = 2) and 2:3 (n = 3). The elemental analyses obtained for the new compounds were in agreement with the calculated values.

Polyene BDAK (III)-(VI), (VIII), (IX), and Diketones (XI) and (XII). Reaction conditions, constants, and yields are given in Table 1. A mixture of 1 mole of the ketone and 2 moles of (I) (n = 2 or 3) was heated under nitrogen in the absence of a solvent, and evaporated in vacuo. After adding dry ether and cooling to -30° C, the solid was separated, washed with a small amount of dry acetone, and recrystallized.

The bisdimethylaminodiketone (XII) was formed in admixture with the monodimethylaminodiketone (p-acetyldimethylaminohexatrienobenzene) (XXX) in a ratio of 2:1, according to the PMR spectra. After washing with absolute methanol, the mixture of (XII) and (XXX) was separated by preparative TLC on alumina (grade II), solvent system acetone-hexane, 2:3, eluted with CHCl₃; (XII) was recovered from the original. The UV and PMR spectra for (XII) are given in Table 1. (XXX), mp 137-141°C, UV spectrum (λ_{max} , C₂H₅OH): 516 nm (ϵ 35,300), 266 plateau (ϵ 11,200), PMR spectrum (δ , ppm, CDCl₃): 2.9 (NMe₂), 7.95-8.05 (C₆H₄), 2.64 (COMe), 7.58 (H_β), 6.77 (H_δ), 6.62 (H_ξ), 6.68 (H_α), 6.14 (H_γ), 5.2 (H_ε), J_{α,β} = J_{γ,δ} = 13.8 Hz, J_{β,γ} = J_{δ,ε} = J_{ε,ξ} = 11.8 Hz. Mass spectrum (m/z): 269 (M⁺).

The mass spectrum showed an intense ion at m/z 224, this being M⁺ for p-acetylbenzoylbenzene (XXXII) as a result of partial conversion of (XXX) into (XXXII) in the mass spectrometer.

On heating (160-170°C), (XXX) also apparently undergoes intramolecular cyclization and deamination to give (XXXII), since λ_{max} (in C₂H₅OH) 263 nm appears in the UV spectrum.

 δ -Dimethylaminodienone (XIII). A mixture of 4.3 g of DMF dimethyl acetal and 4 g of 3-methylcyclohexene-2-one was heated at 120-130°C for 2.5 h with simultaneous removal of the methanol formed. Distillation afforded 1.85 g (31%) of (XIII), bp 125-131°C (0.25 mm). Treatment with dry ether gave (XIII) as a yellow crystalline solid, mp 46-49°C, λ max (in C₂H₅OH) 386 nm (ε 60,900), PMR spectrum (δ , ppm, CDCl₃) 6.8 (H $_{\delta}$), 5.66 (H $_{\alpha}$), 5.0 (H $_{\gamma}$), 2.88 (NMe₂), 2.3, and 1.9 [(CH₂)₃], J_Y, δ = 13 Hz.

EXPERIMENTAL

1. Condensation of aminals and aminal-acetals of conjugated ω -dimethylaminoaldehydes with various ketones and aromatic diketones has afforded a number of polyene bis- ω , ω '-dimethylaminoketones (BDAK) containing 4-8 double bonds, together with polyene bis- ω , ω '-dimethyl-aminodiketones.

2. The luminescence spectral properties of BDAK depend on their structure and the solvent used. As the polarity and proton-donor capacity of the solvent are increased, the absorption and fluorescence maxima are shifted toward the red, and the quantum fluorescence yield increases. The Stokes shift increases with increasing polyene chain length, reaching

*D. G. Gusev participated in the experimental work.

values of 170-180 nm in nonsymmetrical BDAK containing six double bonds. The quantum fluorescence yield passes through a maximum as the chain length is increased, when n = 2 and m = 2 or 3.

The ¹³C chemical shifts in BDAK, like the fluorescence and absorption maxima, vary depending on the capacity of the solvent for specific solvation.

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