

SYNTHESIS AND REACTIONS OF FUROCONDENSED SYSTEMS CONTAINING INDOLE SKELETON*

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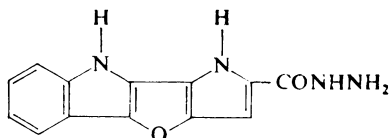
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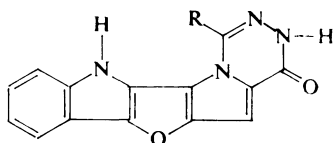
The new 1,2,4-triazolo[3^{'''},4^{'''}:6^{''},1^{''}]-1,2,4-triazino[4^{''},5^{''}:1',5']pyrrolo[2',3':4,5]furo[3,2-*b*]indoles *V* and 1-acetyl-9*H*-pyrrolo[2',3':4,5]furo[3,2-*b*]indole (*IX*) were prepared from 1,9-dihydropyrrolo[2',3':4,5]furo[3,2-*b*]indole-2-carboxhydrazide *I* and 2-(2-nitrophenyl)-4*H*-furo[3,2-*b*]pyrrole-5-carboxylic acid, respectively. Compound *I* gave with triethyl orthoformate or with triethyl orthoacetate 1,2-dihydro-1,2,4-triazino[4^{''},5^{''}:1',5']pyrrolo[2',3':4,5]furo[3,2-*b*]indol-1-one or its methyl analogue *II*. Substances *II* afforded with phosphorus pentasulfide thiones *III* reacting with hydrazine to furnish *IV*. Cyclization of the latter with triethyl orthoformate or orthoacetate led to *V*. Compounds *II* and *III* can also be obtained by a deoxygenative cyclization of *VI* and *VII* with triethyl phosphite.

Recently, the condensed derivatives of triazine¹⁻⁶ and triazole⁷⁻¹⁰ have been intensively studied since some of them reveal biological activity^{11,12}. The preparation of ethyl 2-(2-nitrophenyl)-4*H*-furo[3,2-*b*]pyrrole-5-carboxylate was described in our preceding paper¹³; from this compound pyrrolo[2',3':4,5]furo[3,2-*b*]indoles were synthesized¹⁴. This paper concerning the preparation of some new 1,2,4-triazolo[3^{'''},4^{'''}:6^{''},1^{''}]-1,2,4-triazino[4^{''},5^{''}:1',5']pyrrolo[2',3':4,5]furo[3,2-*b*]indoles (*V*) is a continuation of preceding papers dealing with the synthesis of furan¹⁵, benzo[*b*]furan¹⁵ and arylfuran¹⁶ analogues. The starting 1,9-dihydropyrrolo[2',3':4,5]furo[3,2-*b*]indole-2-carboxhydrazide (*I*) was obtained from the corresponding ester with hydrazine hydrate in ethanol. Two reaction centres of the above-mentioned compound made it possible to prepare 1,2-dihydro-1,2,4-triazino[4^{''},5^{''}:1',5']pyrrolo[2',3':4,5]furo[3,2-*b*]indol-1-ones *IIa,b*, which, with phosphorus pentasulfide furnished the corresponding thiones *III*. These giving 1-hydrazino-1,2,4-triazino[4^{''},5^{''}:1',5']pyrrolo[2',3':4,5]furo[3,2-*b*]indoles *IVa,b* with hydrazine hydrate are anew substances with two reaction centres, which afforded 1,2,4-triazolo[3^{'''},4^{'''}:6^{''},1^{''}]-1,2,4-triazino[4^{''},5^{''}:1',5']pyrrolo[2',3':4,5]furo[3,2-*b*]indoles *Va-Vd*.

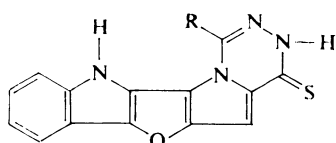
* Part CLXXIX in the series Furan Derivatives; Part CLXXVIII: This Journal 49, 533 (1984).



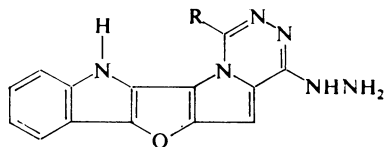
I



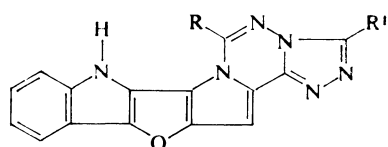
IIa, b



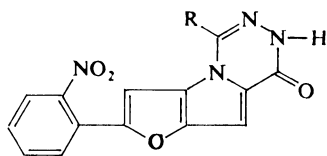
IIIa, b



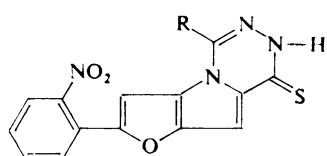
IVa, b



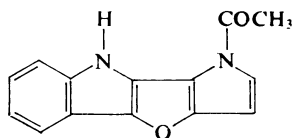
Va-d



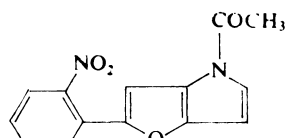
VIa, b



VIIa, b



IX



VIII

In formula II-IV, VIa R = H, b R = CH₃, in formula Va
R=R=H, b R = H, R¹ = CH₃, c R = CH₃, R¹ = H, d R = R¹ = CH₃

Compounds *II* and *III* were alternatively prepared by a deoxygenative cyclization of 7-(2-nitrophenyl)-1,2-dihydrofuro[2',3':4,5]pyrrolo[1,2-*d*]-1,2,4-triazin-1-ones *VIa,b* and thiones *VIIa,b* with triethyl phosphite in 1,2-dichlorobenzene.

Ethyl 2-(2-nitrophenyl)-4*H*-furo[3,2-*b*]pyrrole-5-carboxylate was hydrolyzed with alkali to acid¹³, which served as the starting material for the synthesis of 2-(2-nitrophenyl)-4-acetylfuro[3,2-*b*]pyrrole (*VIII*). Compound *VIII* gave upon deoxygenative cyclization with triethyl phosphite 1-acetyl-9*H*-pyrrolo[2',3':4,5]furo[3,2-*b*]indole (*IX*).

The IR spectra of compounds *Va*–*Vd* revealed absorption bands of C=N triazole and triazine ring bonds at 1 635–1 630 cm⁻¹ and 1 590–1 580 cm⁻¹; the band at lower wavelength is stronger. Bands of the same wavelengths were also found in the spectra of compounds *IVa,b*. The $\nu(\text{C—H})$ and $\nu(\text{C—H})_{\text{arom}}$ bands appeared at 2 910–2 891 and 3 170–3 110 cm⁻¹, respectively. The $\nu(\text{N—H})$ bands occurred at 3 407–3 200 cm⁻¹. Spectra of compounds *VI* and *VII* contained $\nu_{\text{as}}(\text{NO}_2)$ bands at 1 540–1 532 cm⁻¹, and $\nu_{\text{s}}(\text{NO}_2)$ ones at 1 385–1 371 cm⁻¹.

The electron absorption spectra of all compounds showed an intense band in the 332 to 396 nm region and a series of weaker bands at 240 to 300 nm. These bands correspond to the $\pi \rightarrow \pi^*$ electronic transitions. The relatively high λ_{max} values are associated with the extension of the conjugated system, since four to six condensed rings are involved.

The structure of the compounds synthesized was corroborated by ¹H NMR spectrometry: all the spectra contained a proton signal of the pyrrole ring. A long-range coupling constant was observed between protons H₆ and H₉ in compounds *VI* and *VII*, and between protons H₃ and H₆ in derivative *VIII* (⁵*J* = 0.75 Hz). Formation of 1,2,4-triazine derivatives *II*, *III*, *IV*, *VI*, and *VII* was backed by the presence of C₍₄₎—H or C₍₄₎—CH₃ proton signal, and by the change of multiplicity of the C₍₉₎—H or C₍₁₂₎—H proton signal from doublet to a doublet-doublet. Substitution of the oxygen atom in substances *II* and *VI* by sulfur (derivatives *III* and *VII*) was manifested by a down-field shift of the C₍₆₎—H and C₍₉₎—H furo[3,2-*b*]pyrrolo, and C₍₁₂₎—H pyrrole proton signals. The structure of 1,2,4-triazolo-1,2,4-triazine derivatives *V* was evidenced by the presence of C₍₃₎—H, or C₍₃₎—CH₃ proton signals.

EXPERIMENTAL

1*H*,9*H*-Pyrrolo[2',3':4,5]furo[3,2-*b*]indole-2-carboxhydrazide (*I*)

Hydrazine hydrate (80%, 3.5 g) was added to a solution of ethyl 1*H*,9*H*-pyrrolo[2',3':4,5]furo[3,2-*b*]indole-2-carboxylate (2.68 g, 10 mmol) in ethanol (60 ml). The mixture was refluxed for 6 h, cooled and the separated precipitate was filtered off. Yield 2.1 g (81.3%), m.p. 226°C (ethanol). For C₁₃H₁₀N₄O₂ (254.2) calculated: 61.41% C, 3.96% H, 22.04% N; found: 61.45% C,

3.86% H, 21.83% N. IR spectrum (ν_{\max} , cm^{-1}): 1 611 (C=O), 3 285 (NH). UV spectrum, λ_{\max} , nm (log ϵ): 339 (3.58). ^1H NMR spectrum: 6.96 (1 H, s, $\text{C}_{(3)}$ -H), 7.00–7.75 (4 H, m, H_{arom}).

1,2-Dihydro-1,2,4-triazino[4",5":1',5']pyrrolo[2',3':4,5]furo[3,2-b]indol-1-one (*IIa*)

A) 1*H*,9*H*-Pyrrolo[2',3':4,5]furo[3,2-b]indole-2-carboxyhydrazide (2.54 g, 10 mmol) and triethyl orthoformate (2 g, 14 mmol) were refluxed in dimethylformamide (10 ml) for 2.5 h, cooled and the precipitate was filtered off. Yield 1.98 g (79%), m.p. 230°C (decomp., dimethylformamide). For $\text{C}_{14}\text{H}_8\text{N}_4\text{O}_2$ (264.2) calculated: 63.63% C, 3.05% H, 21.20% N; found: 63.48% C, 2.93% H, 21.10% N. IR spectrum, ν_{\max} , cm^{-1} : 1 651 (C=O), 3 407 (NH). UV spectrum, λ_{\max} , nm (log ϵ): 344 (3.06). ^1H NMR spectrum: 7.29 (1 H, d, $\text{C}_{(12)}$ -H), 8.79 (1 H, d, $\text{C}_{(4)}$ -H), 7.00–7.37, and 7.43–7.75 (4 H, m, H_{arom}), $J_{4,12} = 0.8$ Hz. Mass spectrum m/z (%): 264 (100).

4-Methyl-1,2-dihydro-1,2,4-triazino[4",5":1',5']pyrrolo[2',3':4,5]furo[3,2-b]indol-1-one (*IIb*) was obtained in a 76% yield from *I* and triethyl orthoacetate. M.p. 255°C (decomp., dimethylformamide). For $\text{C}_{15}\text{H}_{10}\text{N}_4\text{O}_2$ (278.3) calculated: 64.74% C, 3.62% H, 20.13% N; found: 64.60% C, 3.53% H, 20.32% N. IR spectrum, ν_{\max} , cm^{-1} : 1 655 (C=O), 3 200 (NH). UV spectrum, λ_{\max} , nm (log ϵ): 340 (3.62). ^1H NMR spectrum: 7.17 (1 H, s, $\text{C}_{(12)}$ -H), 2.86 (3 H, s, $\text{C}_{(4)}$ -CH₃), 7.07–7.80 (4 H, m, H_{arom}). Mass spectrum, m/z (%): 278 (100).

7-(2-Nitrophenyl)-1,2-dihydrofuro[2',3':4,5]pyrrolo-[1,2-d]-1,2,3-triazin-1-one (*VIa*) was prepared from 2-(2-nitrophenyl)furo[3,2-b]pyrrole-5-carboxyhydrazide and triethyl orthoformate in a 66% yield, m.p. 306°C (dimethylformamide). For $\text{C}_{14}\text{H}_8\text{N}_4\text{O}_4$ (296.2) calculated: 56.76% C, 2.72% H, 18.91% N; found: 56.51% C, 2.58% H, 19.35% N. IR spectrum, ν_{\max} , cm^{-1} : 1 680 (C=O), 1 537 (NO_{2(as)}), 1 385 (NO_{2(s)}). UV spectrum, λ_{\max} , nm (log ϵ): 341 (3.43). ^1H NMR spectrum: 7.18 (1 H, d, $\text{C}_{(6)}$ -H), 7.27 (1 H, dd, $\text{C}_{(9)}$ -H), 8.91 (1 H, d, $\text{C}_{(4)}$ -H), 7.50–7.96 (4 H, m, H_{arom}), $J_{6,9} = 0.75$ Hz, $J_{4,9} = 0.8$ Hz.

4-Methyl-7-(2-nitrophenyl)-1,2-dihydrofuro[2',3':4,5]pyrrolo[1,2-d]-1,2,4-triazin-1-one (*VIb*) was synthesized using triethyl orthoacetate. Yield 74%, m.p. 259°C (dimethylformamide). For $\text{C}_{15}\text{H}_{10}\text{N}_4\text{O}_4$ (310.3) calculated: 58.07% C, 3.25% H, 18.06% N; found: 57.93% C, 3.16% H, 18.10% N. IR spectrum, ν_{\max} , cm^{-1} : 1 680 (C=O), 1 540 (NO_{2(as)}), 1 370 (NO_{2(s)}). UV spectrum, λ_{\max} , nm (log ϵ): 343 (3.46). ^1H NMR spectrum: 7.14 (1 H, d, $\text{C}_{(6)}$ -H), 7.24 (1 H, d, $\text{C}_{(9)}$ -H), 2.75 (3 H, s, $\text{C}_{(4)}$ -CH₃), 7.33–7.95 (4 H, m, H_{arom}), $J_{6,9} = 0.75$ Hz.

B) Compound *VIa* (2.96 g, 10 mmol) and triethyl phosphite (13 g, 80 mmol) were heated in 1,2-dichlorobenzene (15 ml) at reflux temperature in nitrogen atmosphere for 6 h. The unreacted triethyl phosphite and 1,2-dichlorobenzene were removed under reduced pressure and the residue was crystallized. Yield 1.4 g (52%), m.p. 229°C (decomp., dimethylformamide). According to this method compounds *IIb*, *IIIa* and *IIIb* were prepared from *VIb*, *VIIa* and *VIIb*, respectively.

1,2-Dihydro-1,2,4-triazino[4",5":1',5']pyrrolo[2',3':4,5]furo[3,2-b]indole-1-thione (*IIIa*)

Compound *IIa* (2.64 g, 10 mmol) and phosphorus pentasulfide (2.22 g, 10 mmol) were refluxed in pyridine for 4 h, poured into water (40 ml) and the precipitate was filtered off. Yield 2.1 g (74%), m.p. above 350°C (dioxane). For $\text{C}_{14}\text{H}_8\text{N}_4\text{OS}$ (280.3) calculated: 59.99% C, 2.88% H, 19.98% N, 11.44% S; found: 59.82% C, 2.96% H, 20.59% N, 10.93% S. IR spectrum, ν_{\max} , cm^{-1} : 1 537 (C=S). UV spectrum λ_{\max} , nm (log ϵ): 396 (3.13). ^1H NMR spectrum: 7.45 (1 H, s, $\text{C}_{(12)}$ -H), 9.17 (1 H, s, $\text{C}_{(4)}$ -H), 7.15–7.34 and 7.55–7.91 (4 H, m, H_{arom}).

Following compounds were prepared in an analogous way:

The methyl derivative IIIb; yield 72%, m.p. above 350°C (dioxane). For $C_{15}H_{10}N_4OS$ (294.3) calculated: 61.21% C, 3.43% H, 19.03% N, 10.89% S; found: 61.10% C, 3.51% H, 18.27% N, 11.60% S. IR spectrum, ν_{\max} , cm^{-1} : 1 547 (C=S). UV spectrum, λ_{\max} , nm (log ϵ): 389 (3.04). 1H NMR spectrum: 7.37 (1 H, s, $C_{(12)}-H$), 2.96 (3 H, s, $C_{(4)}-CH_3$), 7.15–7.91 (4 H, m, H_{arom}).

7-(2-Nitrophenyl)-1,2-dihydrofuro[2',3':4,5]pyrrolo[1, 2-d]-1,2,4-triazine-1-thione (VIIa). Yield 79%, m.p. 289°C (dioxane). For $C_{14}H_8M_4O_3S$ (312.3) calculated: 53.84% C, 2.58% H, 17.94% N, 10.27% S; found: 53.71% C, 2.70% H, 17.58% N, 10.63% S. IR spectrum, ν_{\max} , cm^{-1} : 1 552 (C=S), 1 540 ($NO_{2(\text{as})}$), 1 377 ($NO_{2(\text{s})}$). UV spectrum, λ_{\max} , nm (log ϵ): 390 (3.02). 1H NMR: 7.32 (1 H, d, $C_{(6)}-H$), 7.53 (1 H, d, $C_{(9)}-H$), 9.22 (1 H, s, $C_{(4)}-H$), 7.63–7.97 (4 H, m, H_{arom}), $J_{6,9} = 0.75$ Hz.

4-Methyl-7-(2-nitrophenyl)-1,2-dihydrofuro[2',3':4,5]pyrrolo[1,2-d]-1,2,4-triazine-1-thione (VIIb). Yield 84%, m.p. 304°C (dioxane). For $C_{15}H_{10}N_4O_3S$ (326.3) calculated: 55.21% C, 3.09% H, 17.17% N, 9.83% S; found: 55.09% C, 3.16% H, 16.81% N, 10.27% S. IR spectrum, ν_{\max} , cm^{-1} : 1 549 (C=S), 1 532 ($NO_{2(\text{as})}$), 1 371 ($NO_{2(\text{s})}$). UV spectrum, λ_{\max} , nm (log ϵ): 387 (3.06). 1H NMR spectrum: 7.31 (1 H, d, $C_{(6)}-H$), 7.56 (1 H, d, $C_{(9)}-H$), 2.80 (3 H, s, $C_{(4)}-CH_3$), 7.65–8.04 (4 H, m, H_{arom}), $J_{6,9} = 0.75$ Hz.

1-Hydrazino-1,2,4-triazino[4'',5'':1',5']pyrrolo[2',3':4,5]furo[3,2-b]indole (IVa)

Compound IIIa (2.8 g, 10 mmol) and hydrazine hydrate (94%, 15 ml) were stirred at 80°C for 2 h, the mixture was cooled, the precipitate was filtered off and washed with ether. Yield 2.17 g (78%), m.p. over 350°C (dioxane). For $C_{14}H_{10}N_6O$ (278.3) calculated: 60.43% C, 3.62% H, 30.20% N; found: 60.24% C, 3.68% H, 29.95% N. 1H NMR spectrum: 7.08 (1 H, s, $C_{(12)}-H$) 8.43 (1 H, s, $C_{(4)}-H$), 7.00–7.16 and 7.42–7.63 (4 H, m, H_{arom}).

1-Hydrazino-4-methyl-1,2,4-triazino[4'',5'':1',5']pyrrolo[2',3':4,5]furo[3,2-b]indole (IVb). Yield 79%, m.p. above 350°C (dioxane). For $C_{15}H_{12}N_6O$ (292.3) calculated: 61.74% C, 4.14% H, 28.75% N; found: 61.72% C, 4.08% H, 28.43% N. 1H NMR spectrum: 7.01 (1 H, s, $C_{(12)}-H$), 2.38 (3 H, s, $C_{(4)}-CH_3$), 7.01–7.16 and 7.40–7.65 (4 H, m, H_{arom}).

1,2,4-Triazolo[3'',4''':6'',1'']-1,2,4-triazino[4'',5'':1',5']pyrrolo[2',3':4,5]furo[3,2-b]indole (Va)

Compound IVa (2.78 g, 10 mmol) and triethyl orthoformate (4 g, 28 mmol) were heated in dimethylformamide (20 ml) under reflux for 4 h, and the precipitate obtained in a routine way was filtered off. Yield 2.1 g (73%), m.p. 344°C (decomp., dimethylformamide). For $C_{15}H_8N_6O$ (288.3) calculated: 62.50% C, 2.80% H, 29.15% N; found: 62.31% C, 2.69% H, 28.80% N. UV spectrum, λ_{\max} , nm (log ϵ): 345 (3.01). 1H NMR spectrum: 7.25 (1 H, s, $C_{(14)}-H$), 7.95 (1 H, s, $C_{(3)}-H$), 8.78 (1 H, s, $C_{(6)}-H$), 7.05–7.80 (4 H, m, H_{arom}).

Compound Vb: Yield 72%, m.p. 337°C (decomp., dimethylformamide). For $C_{16}H_{10}N_6O$ (302.3) calculated: 63.57% C, 3.33% H, 27.80% N; found: 63.35% C, 3.21% H, 27.34% N. UV spectrum, λ_{\max} , nm (log ϵ): 344 (3.03). 1H NMR spectrum: 7.24 (1 H, s, $C_{(14)}-H$), 8.67 (1 H, s, $C_{(6)}-H$), 2.83 (3 H, s, $C_{(3)}-CH_3$), 7.07–7.76 (4 H, m, H_{arom}).

Compound Vc: Yield 74%, m.p. 356°C (decomp., dimethylformamide). For $C_{16}H_{10}N_6O$ (302.3) calculated: 63.57% C, 3.33% H, 27.80% N; found: 63.42% C, 3.24% H, 27.42% N. UV spectrum, λ_{\max} , nm (log ϵ): 342 (3.14). 1H NMR spectrum: 7.06 (1 H, s, $C_{(14)}-H$), 7.39 (1 H, s, $C_{(3)}-H$), 2.92 (3 H, s, $C_{(6)}-CH_3$), 6.97–7.25 and 7.35–7.65 (4 H, m, H_{arom}).

Compound Vd: Yield 78%, m.p. 349°C (decomp., dimethylformamide). For $C_{17}H_{12}N_6O$ (316.3). Calculated: 64.55% C, 3.82% H, 26.57% N; found: 64.32% C, 3.73% H, 26.31% N. UV spectrum, λ_{max} , nm (log ϵ): 339 (3.59). 1H NMR spectrum: 7.24 (1 H, s, $C_{(14)}-H$), 2.87 (6 H, s, $C_{(3)}-CH_3$, $C_{(6)}-CH_3$), 7.07–7.35 and 7.45–7.75 (4 H, m, H_{arom}).

2-(2-Nitrophenyl)-4-acetylfuro[3,2-*b*]pyrrole (*VIII*)

2-(2-Nitrophenyl)furo[3,2-*b*]pyrrole-5-carboxylic acid (2.72 g, 10 mmol) was heated in acetic anhydride (20 ml) under reflux for 4 h. Acetic anhydride was distilled off under reduced pressure and the residue was crystallized. Yield 2.45 g (90.8%), m.p. 112–113°C (ethanol). For $C_{14}H_{10}N_2O_4$ (270.2) calculated: 62.23% C, 3.73% H, 10.37% N; found: 62.14% C, 3.65% H, 10.54% N. IR spectrum, ν_{max} , cm^{-1} : 1 695 (C=O), 1 537 ($NO_{2(as)}$), 1 375 ($NO_{2(s)}$). UV spectrum, λ_{max} , nm (log ϵ): 335 (4.28). 1H NMR spectrum: 7.41 (1 H, d, $C_{(3)}-H$), 7.58 (1 H, d, $C_{(5)}-H$), 6.59 (1 H, dd, $C_{(6)}-H$), 7.55–8.00 (4 H, m, H_{arom}), $J_{3,6} = 0.75$ Hz, $J_{5,6} = 4$ Hz.

1-Acetyl-9*H*-pyrrolo[2',3':4,5]furo[3,2-*b*]indole (*IX*)

Compound *VIII* (2.70 g, 10 mmol) and triethyl phosphite (8.3 g, 50 mmol) were refluxed under nitrogen. The work-up afforded 1.62 g (68.2%), m.p. 243–244°C (decomp., chloroform). For $C_{14}H_{10}N_2O_2$ (238.8) calculated: 70.59% C, 4.23% H, 11.76% N; found: 70.48% C, 4.12% H, 11.92% N. IR spectrum, ν_{max} , cm^{-1} : 1 690 (C=O). UV spectrum, λ_{max} , nm (log ϵ): 365 (4.35). 1H NMR spectrum: 7.48 (1 H, d, $C_{(2)}-H$), 6.65 (1 H, d, $C_{(3)}-H$), 7.05–7.24 and 7.50–7.67 (4 H, m, H_{arom}), 2.73 (3 H, s, CH_3), $J_{2,3} = 4$ Hz.

Spectral Measurements

The IR spectra of compounds *Via,b* and *VIIa,b* in KBr were measured on a PYE UNICAM SP 100 spectrophotometer; other spectra were taken with a UR 20 (Zeiss, Jena) apparatus. The electron absorption spectra of dioxane or methanolic (compounds *VIII* and *IX*) solutions were recorded with a Specord UV VIS (Zeiss, Jena) instrument at $1 \cdot 10^{-5}$ to $1 \cdot 10^{-4}$ mol l^{-1} concentration at room temperature. The 1H NMR spectra of hexadeuteriodimethyl sulfoxide solutions containing hexamethyldisiloxane were run with Tesla BS 487 C spectrometer operating at 80 MHz, the electron impact mass spectra of *Iia,b* were measured with an AEI 902 S apparatus at an electron energy 70 eV, trap current 100 μA and 200°C ionization energy.

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