1,3-DIPOLAR CYCLOADDITIONS OF N-(5-NITRO-2-FURFURYL)-ISOQUINOLINIUM BROMIDE IN THE SYNTHESIS OF BENZOINDOLIZINES

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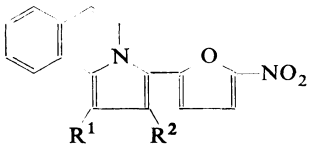
Substituted 3-(5-nitro-2-furyl)benzo[*g*]indolizines *III*—*XII* were synthesized by a 1,3-dipolar cycloaddition of the ylide *II* formed *in situ* from N-(5-nitro-2-furfuryl)isoquinolinium bromide (*I*) and dimethyl butinedioate, diethyl butenedioate, 1-nitro-2-phenylethylene, ethyl 2-propenoate, ethyl 3-(5-nitro-2-furyl)-2-propenoate, 1,3-diphenyl-2-propenone, 1,3-diphenyl-2-propinone, 2-propenenitrile, 1-phenyl-3-(5-nitro-2-furyl)-2-propenone, and methyl 2-cyano-3-(4-nitrophenyl)-2-propenoate. Structure of these products was verified by spectral evidence.

One of our preceding papers[1] dealt with the study of some properties of 5-nitro-2-furfuryl N-onium bromides; especially those obtained from tertiary heterocyclic amines were shown to be relatively strong CH-acids. Deprotonation of the methylene group of these derivatives gave rise to azomethine ylides, which can be *inter alia* employed as 1,3-dipoles[2].

This paper, which can be regarded as a continuation of our contribution[3] to the synthesis of indolizines from 1-pyridinium 5-nitro-2-furyl-methylide, is aimed to the preparation of substituted 3-(5-nitro-2-furyl)benzo[*g*]indolizines *III*—*XII* by a 1,3-dipolar cycloaddition of 2-isoquinolinium 5-nitro-2-furylmethylide (*II*) to compounds with an activated C—C multiple bond.



III—*XII*

Azomethine ylide *II*, formed *in situ* from N-(5-nitro-2-furfuryl)isoquinolinium bromide[1] (*I*) reacted with dimethyl butinedioate, diethyl butenedioate[4], 1-nitro-

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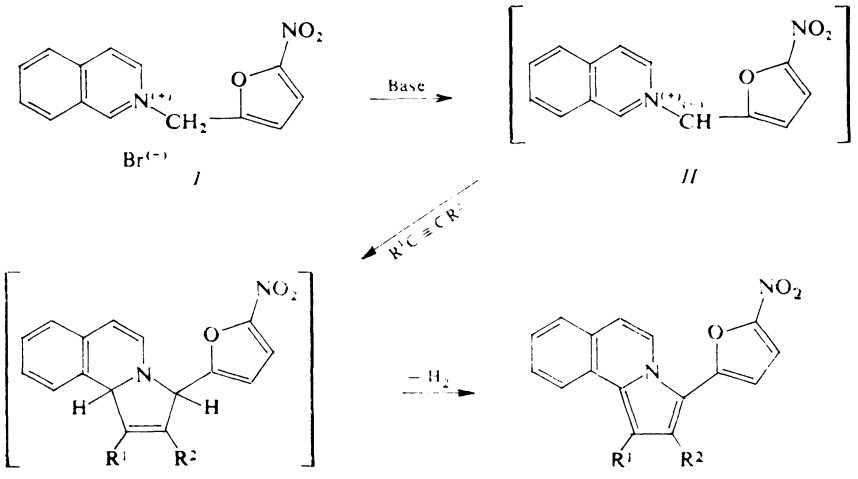
-2-phenylethylene[5], ethyl 2-propenoate, ethyl 3-(5-nitro-2-furyl)-2-propenoate[6-9], 1,3-diphenyl-2-propenone[10], 1,3-diphenyl-2-propinone[11,12], 2-propenenitrile, 1-phenyl-3-(5-nitro-2-furyl)-2-propenone[12], and methyl 2-cyano-3-(4-nitrophenyl)-2-propenoate[13] to give the respective benzo[*g*]indolizines *III*–*XII*. Sodium hydride and potassium carbonate were employed for the generation of the ylide *II* from *I*: the equimolar mixture of *I* and the dipolarophile in dioxane or in chloroform–2-methoxyethanol (10 : 1) with the excess of the base were shaken at room temperature for some days. This reaction was accompanied by formation of a great amount of unidentified polymers, which were separated from the low-molecular products by chromatography on alumina. The high sensitivity of 5-nitrofuran derivatives towards basic reagents, differences in the character of the dipolarophile, and also the low stability of the carbanion *II* generated *in situ* were reflected by yields of these reactions (5–76%). The selection of the proper base is in some cases of great importance. Thus, *e.g.* the yield of 1-cyano-3-(5-nitro-2-furyl)benzo[*g*]indolizine (*X*) raised from 5% when using sodium hydride to 23% with potassium carbonate, but that of 1-ethoxycarbonyl-3-(5-nitro-2-furyl)benzo[*g*]indolizine (*VII*) remained virtually unchanged (23 and 26%, respectively). Sometimes even the increase of temperature was proved substantial. The reaction of dimethyl butinedioate in dioxane leading to *III* ($R^1 = R^2 = COOCH_3$) proceeded *e.g.* at an ambient temperature to 35%, at 50–60°C to 51%, and at reflux to 24% only.

Analogously as in[14-16] and partly also in[17] the cycloaddition of 2-isoquinolinium



SCHEME 1

5-nitro-2-furylmethylide (*II*) to dimethyl butinedioate and 1,3-diphenyl-2-propinone was accompanied by aromatization of the five-membered ring (Scheme 1). Both acetylenic dipolarophiles can also act as aromatization reagents[18], since a partial decomposition takes place due to the action of basis to the salt *I* under formation of nitrofuran derivative. On the other hand, some isoquinolinium azomethine ylides react with activated acetylenes to afford stable dihydroadducts[19], or, one can isolate indolizine in addition to dihydroindolizine and possibly to dimer of the ylide as by-products[17,20].

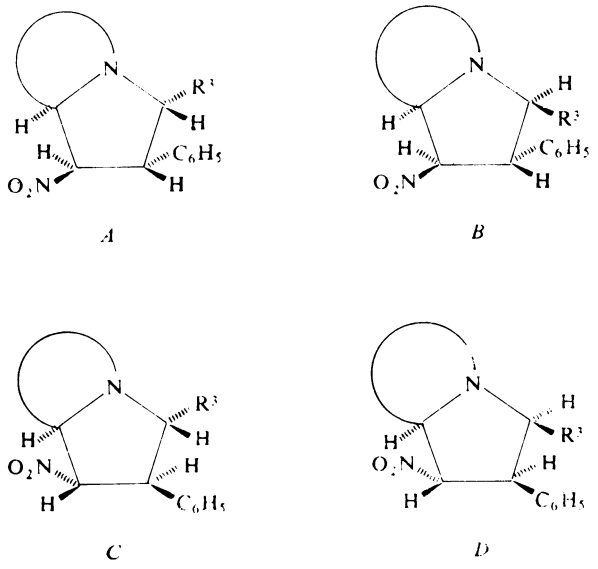
Substituted benzo[*g*]indolizines *IV*–*XII* were also obtained by treatment of the dipole *II* with the eight already mentioned compounds with activated double bond. Neither the corresponding tetrahydro nor the dihydro derivatives could be isolated. These results are in line with those we already published[3] although it is known that addition of olefins to azomethine ylides, generated from various pyridinium and isoquinolinium salts, gives stable 2,3-dihydroindolizines[21], tetrahydroindolizines[22,23], a mixture thereof[24] (as *e.g.* with addition of 2-propenenitrile to 2-isoquinolinium phenacylmethylides), and hexahydroindolizines[14]. The majority of these derivatives formed the corresponding indolizines only by catalysts as palladium over charcoal, or by reagents as 2,3-dichloro-5,6-dicyano-1,4-benzoquinone, chloranil, or $KMnO_4$.

The reaction with 1-nitro-2-phenylethylene led to two products: 2-phenyl-3-(5-nitro-2-furyl)benzo[*g*]indolizine (*V*, 76%), and 1-nitro-2-phenyl-3-(5-nitro-2-furyl)-benzo[*g*]indolizine (*VI*, 10%). This finding can be rationalized by a different mutual orientation of the dipole and dipolarophile at the cycloaddition reaction. The structures of the tetrahydroindolizine intermediate can be drawn by four various formulas *A* – *D*; the enantiomeric pairs *A* and *B*, *C* and *D* differ from each other by arrangement of substituents at $C_{(3)}$ of benzo[*g*]indolizine (Scheme 2). Elimination of nitrous acid and the origination of derivative *V* can occur providing the hydrogen and nitro group are in *trans* position. This requirement met structures *A* and *B*; cleavage of nitrous acid from *C* and *D* is impossible and compound *VI* results from oxidation by air.

Azomethine ylide *II* with methyl 2-cyano-3-(4-nitrophenyl)-2-propenoate yielded an unexpected product, which was identified as 1-cyano-2-(4-nitrophenyl)-3-(5-nitro-2-furyl)benzo[*g*]indolizine (*XII*), $\nu C{\equiv}N$ 2 200 cm^{-1}, *m/z* 224 (M^+).

Product *IX* ($R^1 = COC_6H_5$, $R^2 = C_6H_5$) was isolated from a parallel reaction with compounds with the same substituents having an activated double or triple bond (1,3-diphenyl-2-propenone and 1,3-diphenyl-2-propinone).

The analytical data of substituted 3-(5-nitro-2-furyl)benzo[*g*]indolizines *III*–*XII* agree with their structure (Table I). The spectral parameters are, in principal, quite similar. The UV spectra of *III*–*XII* indicate the presence of a cyclic conjugated system (Table II). The existence of the benzo[*g*]indolizine backbone[17,25,26] was evidenced especially by ^1H NMR spectral data (Table III). The protons H_7, H_8 and H_9 of the fused benzene ring *e.g.* of compound *VII* ($R^1 = COOC_2H_5$, $R^2 = H$)



SCHEME 2

resonated as a multiplet at δ 7·48 – 7·92. The H_{10} proton signal of this ring was downfield shifted (δ 9·57) due to the anisotropic effect of the ethoxycarbonyl group at $C_{(1)}$. The H_5 and H_6 proton signals form two doublets (δ 8·55 and 7·36 ppm, $J_{5,6}$ = = 7·3 Hz). The higher chemical shift value of H_5 is due to the presence of nitrogen adjacent to this carbon atom. The singlet at 7·73 ppm was attributed to H_2 proton. The 5-nitro-2-furyl grouping was manifested by two doublets at 7·28 and 7·78 ppm ($J_{A,B}$ = 3·9 Hz). The H_B proton was downfield shifted due to the electron-withdrawing character of the nitro group. Characteristic signals of the ethyl group are at δ 1·37 and 4·38 ppm. The substituent effect was seen mostly at the chemical shift of protons H_{10} and H_A. Character of the substituent at $C_{(2)}$ must be taken into account[27] when considering the anisotropic effect of the ethoxycarbonyl group on the H_{10} proton shift, as seen from the comparison of chemical shifts of compounds *IV*, *VII* and *VIII*. Type of the substituent at $C_{(2)}$ influences the chemical shift value of the proton H_A of furan ring. Thus, the phenyl group at $C_{(2)}$ of compounds *V*, *VI*, *IX*, *XI* causes the upfield shift of the H_A signal when compared with other derivatives, this being due to its electronic effects and to a possible shielding of benzene ring current. Taking this fact in mind and considering the reported data[28,29], one can presume also in this case the *s-cis* conformation of the furan ring with respect to the $C_{(2)}$—$C_{(3)}$ bond of benzo[*g*]indolizine. The ^{1}H NMR spectrum of *XI* confirms that a mixture

of position isomers in a 2 : 1 ratio was involved (R^1 = COC_6H_5, R^2 = 5-nitro-2-furyl and *vice versa*). Assignment of protons of one of furan rings was impossible, since both have signals in the region of broad multiplet of aromatic protons.

Mass spectra of benzo[*g*]indolizines *III*–*XII* (Table IV) were interpreted to back the structure. All compounds revealed peaks of molecular ions, which, for compounds *IV*, *VI* and *VIII* were the most intense. Base peaks of compounds *III*, *V*, *VII* and *X* were formed by the species $(M—NO_2—CO)^+$; the parent peak of *IX* and *XI* repre-

TABLE I

Substituted 3-(5-nitro-2-furyl)benzo[*g*]indolizines *III*–*XII*

Compound	R^1 R^2	Formula (M_r)	M.p., °C (yield, %)	Calculated/Found		
				% C	% H	% N
III	$COOCH_3$ $COOCH_3$	$C_{20}H_{14}N_2O_7$ (394·3)	191–192[a,b] (51)	60·91 61·12	3·58 3·69	7·11 6·78
IV	$COOC_2H_5$ $COOC_2H_5$	$C_{22}H_{18}N_2O_7$ (422·4)	156–157[c,d] (14)	62·56 62·63	4·29 4·29	6·63 6·66
V	H C_6H_5	$C_{22}H_{14}N_2O_3$ (354·4)	212–213[a,e] (76)	74·57 74·58	3·98 4·37	7·90 7·27
VI	NO_2 C_6H_5	$C_{22}H_{13}N_3O_5$ (399·4)	214–216[a,e] (10)	66·00 66·14	3·52 3·41	10·49 10·24
VII	$COOC_2H_5$ H	$C_{19}H_{14}N_2O_5$ (350·3)	184–185[a,f] (26)	65·14 65·47	4·03 4·07	7·99 7·75
VIII	$COOC_2H_5$ 5-NO_2-2-furyl	$C_{23}H_{15}N_3O_8$ (461·4)	218–220[a,g] (31)	59·88 60·11	3·28 3·36	9·10 9·13
IX	COC_6H_5 C_6H_5	$C_{29}H_{18}N_2O_4$ (458·5)	225–227[a,f] (9·8[h], 10·9[i])	75·98 76·63	3·96 4·40	6·10 6·53
X	CN H	$C_{17}H_9N_3O_3$ (303·3)	244–247[a,e] (23)	67·33 67·61	2·99 3·19	13·85 13·64
XI[j]	COC_6H_5 5-NO_2-2-furyl	$C_{27}H_{15}N_3O_7$ (493·4)	[a,e] (16)	65·72 65·71	3·06 3·05	8·51 8·13
XII	CN 4-nitrophenyl	$C_{23}H_{12}N_4O_5$ (424·4)	274–277[a,e] (5·3)	65·10 65·02	2·85 2·78	13·19 12·87

[a] Crystallized from acetone; [b] eluent chloroform; [c] crystallized from ethanol; [d] eluent chloroform–acetone (11 : 3); [e] benzene–n-hexane (5 : 1); [f] benzene; [g] benzene–n-hexane–acetone (10 : : 2 : 1); [h] addition of the ylide to 1,3-diphenyl-2-propinone; [i] to 1,3-diphenyl-2-propenone; [j] mixture of isomers.

sented the benzoyl radical ion at m/z 105 and that of *XII* the M-32 ion at m/z 392. Compounds, where the molecule of methanol could not be split off a cleavage of NO + H_2 took place; this presumption was proved with compound *X* revealing a metastable transition m* = 242·4 (303 → 271). The main fragmentation pathways were governed by substituents of benzo[*g*]indolizines *III*–*XII*.

As presented, the 1,3-dipolar cycloadditions of the above-mentioned dipolarophiles (with the exception of 1-phenyl-3-(5-nitro-2-furyl)-2-propenone) to 2-isoquinolinium-5-nitro-2-furylmethylide (*II*) are regioselective.

TABLE II

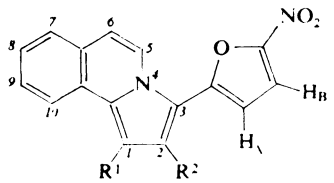
Infrared (cm^{-1}) and UV-VIS spectra of the substituted 3-(5-nitro-2-furyl)benzo[*g*]indolizines *III*–*XII*

Compound	ν(C=O)	$\nu_{as}(NO_2)$ $\nu_s(NO_2)$	Other bands		λ_{max}, nm (log ε)
III	1 715	1 533	1 450	$\delta_{as}(CH_3)$	423
	1 684	1 340	1 367	$\delta_s(CH_3)$	(4·17)
IV	1 697	1 531	1 455	$\delta_{as}(CH_3)$	418
		1 337	1 380	$\delta_s(CH_3)$	(4·10)
V	—	1 528		—	465
		1 333			(4·00)
VI	—	[a]		—	414
		1 330			(4·30)
VII	1 691	1 526	1 450	$\delta_{as}(CH_3)$	444
		1 332	1 385	$\delta_s(CH_3)$	(4·27)
VIII	1 696	1 526	1 448	$\delta_{as}(CH_3)$	409
		1 330	1 355	$\delta_s(CH_3)$	(4·18)
IX	1 626	1 552		—	452
		1 330			(4·23)
X	—	1 525	2 160	ν(C≡N)	432
		1 338			(4·12)
XI	1 650	1 531		—	410
		1 340			(4·34)
XII	—	1 538	2 180	ν(C≡N)	425
		1 328			(4·12)

[a] The band overlapped by ring vibrations at 1 485 cm^{-1}.

TABLE III

^{1}H NMR (δ, ppm, J, Hz) of substituted 3-(5-nitro-2-furyl)benzo[g]indolizines *III*—*XII*



Compound[a]	H_5 $J_{5,6}$	H_6	H_7 H_8 H_9	H_{10}	H_A	H_B[b]	Others
III	8·25d 7·5	7·11d	7·47—7·72m	8·72m	7·10d	7·47d	4·01; 3·89 (OCH_3)
IV	8·34d 7·4	7·36d	7·50—7·90m	8·56m	7·33d	7·91d	4·42; 4·28 (OCH_2) 1·35; 1·23 (CH_3)
V	8·35d 7·4	7·12d	7·30—7·90m	8·26m	6·65d	7·68d	7·30—7·55m (C_6H_5) H_1[c]
VI	8·47d 7·5	7·47d	7·40—7·80m	8·47m	6·55d	7·62d	7·40bs (C_6H_5)
VII	8·55d 7·3	7·36d	7·48—7·92m	9·57m	7·28d	7·78d	4·38 (OCH_2) 1·37 (CH_3) 7·33 (H_2)
VIII	8·25d 7·5	7·32d	7·42—7·56	7·83m	7·01d 7·31d	7·84d 7·82d	4·10 (OCH_2) 1·02 (CH_3)
IX	8·58d 7·3	7·36d	[d]	[d]	6·51d	7·70d	7·21bs (C_6H_5) 7·23—7·87m (COC_6H_5)
X	8·57d	7·42d	7·55—8·00m	8·67m	7·28d	7·80d	7·77s (H_2)
XI[e]	8·38d 7·4	7·34d	[f]	[f]	6·66d	7·47d	C_6H_5[f]
	8·46d 7·4	7·41	[f]	[f]	6·87d 7·07d	7·63d 7·65d	C_6H_5[f]
XII	8·38d 7·3	7·45d	7·55—8·00m	8·77m	6·92d	7·69d	8·31d 7·70d ($C_6H_4NO_2$

[a] Compound *III* measured in deuteriochloroform, compounds *IV*—*XII* in hexadeuteriodimethyl sulfoxide; [b] $J_{A,B}$ = 3·9 Hz; [c] in the multiplet of phenyl group protons 7·30—7·55; [d] in the multiplet of aromatic ring protons; [e] a mixture of isomers; [f] in the multiplet 7·23—8·00.

EXPERIMENTAL

Melting points were measured with a micro hot-stage Boetius, the microanalyses were carried out on an elemental analyser (Carlo Erba, Italy). Purity of products was checked by thin-layer chromatography on Silufol (Kavalier, Czechoslovakia). The IR spectra were recorded with a UR-20 (Zeiss, Jena) spectrophotometer in KBr (0·6 mg of the substance in 300 mg of KBr). The electron absorption spectra in dioxane solutions were measured at room temperature with a UV-VIS (Zeiss, Jena) apparatus. The ^{1}H NMR spectra of deuteriochloroform and hexadeuteriodimethyl sulfoxide solutions containing tetramethylsilane and hexamethyldisiloxane, respectively, were taken with a Tesla BS 487 C apparatus operating at 80 MHz at 25—80°C. The mass spectra were recorded on an MS 902 (AEI, Manchester) spectrometer at a 70 eV ionization energy, trap current 100 μA and 150°C ion source temperature.

TABLE IV

Ten most intense peaks in the mass spectra of 3-(5-nitro-2-furyl)benzo[*g*]indolizines *III*—*XII*

Compound	*m/z* (relative intensity, %)
III	$M^{+\cdot}$ 394 (77), 363 (36), 362 (93), 348 (25), 331 (54), 320 (100), 316 (25), 303 (29), 216 (21), 203 (30)
IV	423 (26), $M^{+\cdot}$ 422 (100), 390 (33), 377 (22), 376 (26), 348 (52), 302 (44), 274 (30), 246 (22), 202 (24)
V	$M^{+\cdot}$ 354 (83), 322 (56), 308 (83), 293 (42), 280 (100), 279 (29), 278 (58), 241 (25), 139 (29), 128 (15)
VI	400 (24), $M^{+\cdot}$ 399 (100), 367 (45), 337 (32), 336 (76), 306 (23), 279 (21), 278 (66), 277 (21), 128 (30)
VII	$M^{+\cdot}$ 350 (87), 318 (60), 304 (47), 276 (100), 273 (40), 248 (72), 203 (27), 202 (27), 164 (15), 139 (25)
VIII	$M^{+\cdot}$ 461 (100), 429 (30), 387 (95), 359 (28), 355 (30), 312 (21), 284 (37), 256 (23), 228 (33), 128 (16)
IX	$M^{+\cdot}$ 458 (52), 428 (26), 427 (76), 413 (19), 350 (24), 279 (29), 272 (14), 270 (29), 105 (100), 77 (38)
X	$M^{+\cdot}$ 303 (63), 271 (52), 257 (23), 243 (19), 230 (21), 229 (100), 219 (17), 192 (17), 191 (17), 164 (17)
XI	494 (23), $M^{+\cdot}$ 493 (77), 461 (29), 447 (34), 419 (64), 416 (20), 343 (20), 315 (21), 105 (100), 77 (50)
XII	425 (25), $M^{+\cdot}$ 424 (88), 393 (25), 392 (100), 378 (31), 350 (62), 332 (38), 317 (31), 304 (50), 305 (75)

N-(5-Nitro-2-furfuryl)isoquinolinium Bromide[1] (*I*)

It was synthesized from furfuryl alcohol *via* 5-nitro-2-furfuryl nitrate and 5-nitro-2-furfuryl bromide. Ethyl 3-(5-nitro-2-furyl)-2-propenoate[9] was obtained by a Wittig reaction from 5-nitro-2-furaldehyde and ethyl chloroacetate. Chloroacetic acid was esterified by ethanol according to[6], ethoxycarbonylmethyltriphenylphosphonium chloride was prepared according to[7], ethoxycarbonylmethylenetriphenylphosphorane according to[8], and 1-phenyl-3-(5-nitro-2-furyl)-2-propenone according to[12].

Substituted 3-(5-Nitro-2-furyl)benzo[*g*]indolizines *III*, *IV*, *VII*—*IX*, *XI*

Sodium hydride (0·48 g, 20 mmol) was added to a stirred and ice-cooled suspension of N-(5-nitro-2-furfuryl)isoquinolinium bromide (3·35 g, 10 mmol) and the dipolarophile (10 mmol) in dioxane (60 ml). The inorganic portion was filtered off after a 3-day stirring at room temperature (at 50 to 60°C for compound *IV*), washed with chloroform (30 ml) and the solvent was removed under diminished pressure. The residue was separated on an alumina-packed column using the proper eluent (Table I). Further work-up gave coloured solids, which were crystallized from a suitable solvent.

Substituted 3-(5-Nitro-2-furyl)benzo[*g*]indolizines *X*, *XII*

Anhydrous potassium carbonate (4 g, 29 mmol) was successively added to a stirred and ice-cooled suspension of N-(5-nitro-2-furfuryl)isoquinolinium bromide (1·76 g, 5 mmol) and the dipolarophile (5 mmol) in chloroform (50 ml) and 2-methoxyethanol (5 ml). The mixture was stirred at an ambient temperature for 3 days and worked up as in the preceding case.

Substituted 3-(5-Nitro-2-furyl)benzo[*g*]indolizines *V*—*VI*

Sodium hydride (0·48 g, 20 mmol) was added to a stirred and cooled suspension of N-(5-nitro-2-furfuryl)isoquinolinium bromide (3·35 g, 10 mmol) and 1-nitro-2-phenylethylene (1·49 g, 10 mmol) in dioxane (70 ml). The mixture was stirred at room temperature for 4 days, the inorganic portion was filtered off, washed with chloroform (20 ml) and the solvent was distilled off under reduced pressure. The residue was separated on an alumina-packed column using benzene–n-hexane (5 : 1) as eluent. The red compound *V* (2·7 g, 76%) was the principal substance; m.p. 212—213°C (acetone). The by-product, *VI* also red, was isolated in a 10% yield (0·4 g), m.p. 214—216°C (acetone).

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