ELECTROPHILIC 3,5-SUBSTITUTION OF 2,4,4,6-TETRAPHENYL-4*H*-PYRAN AND SOME OF ITS 1-HETEROANALOGUES

Marián SCHWARZ*, Pavel ŠEBEK and Josef KUTHAN

Department of Organic Chemistry,
Institute of Chemical Technology, 166 28 Prague 6

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Dedicated to Professor Miloslav Ferles on the occasion of his 70th birthday.

The reaction of dibromine with the heterocycles *I*—*IV* gives corresponding 3,5-dibromo derivatives, while the less selective reactions with dichlorine afforded 3,5-dichloro derivative *XII* only in the case of 4*H*-thiopyran *IV* and trichloro derivative of the probable formula *XIII* in the case of 1,4-dihydropyridine *II*. Dichloro derivative *IX* was obtained on reaction of phosphorus pentrachloride with substrate *I*. Nitration of compounds *I*—*IV* gives 3,5-dinitroderivatives *XV*—*XVIII*, but mononitro derivatives *XVX* and *XX* were also obtained. Iodination of compounds *II*—*IV* with di-iodine was unsuccessful.

Pyran-like heterocyclic compounds generally behave as π-electron donors but no systematic attention has yet been devoted to their electrophilic substitutions[1-6]. In connection with our interest in photochromic derivatives *I*—*IV* we investigated the possibility of the modification of their structure by introducing further chromophorically interesting substituents into their molecules. So far only bromination of 2,4,4,6-tetraphenyl-4*H*-pyran has been described[7], leading to corresponding dibromo derivative of the probable formula *V*. 3,5-Regioselectivity of the electrophilic attack on the molecules of 2,4,4,6-tetraphenyl-1,4-dihydropyridine (*II*) and its 1-methyl derivative *III* was recently demonstrated[8] by deuteriodeprotonation.

In this study we are describing experimental findings concerning bromination, chlorination and nitration of compounds *I*—*IV*, carried out preparatively under mild conditions. The determined molecular structures of all the isolated products correspond to a preferential 3,5-regioselective electrophilic attacks with corresponding reagents, in agreement with the π-electron distribution and the HOMO character in quantum chemical models of the starting heterocycles[1,2,9]. The yields and the properties of the isolated products are surveyed in Table I.

Reaction with a solution of dibromine in carbon disulfide at 20°C leads to a gradual conversion of all investigated compounds *I*—*IV* to corresponding 3,5-dibromo derivatives *V*—*VIII*. In the case of the formation of 3,5-dibromo-2,4,4,6-tetraphenyl-

* Present address: Department of Biochemistry and Radiobiology of the Agricultural University, 949 76 Nitra

-4*H*-pyran (*V*) this is in agreement with an earlier assumption[7]. The reaction with dichlorine takes place under analogous conditions with all the substrates very rapidly and non-selectively, under formation of complex mixtures of reaction

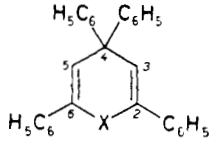
TABLE I

Physico-chemical and spectral characteristics of compounds *V–IX*, *XII*, *XIII*, *XV–XX*

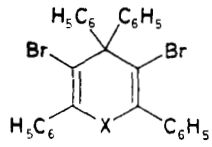
Compound	M.p., °C (Yield, %)	Formula (M.w.)	Calculated/Found				IR[a] $\tilde{\nu}_{max}$, cm^{-1}
			% C	% H	% N	% X[b]	
V	228[c]	$C_{29}H_{20}OBr_2$	63·99	3·70	—	29·35	1 662 1 598 1 575
	(64)	(544·3)	64·00	3·75	—	29·25	
VI	157–160	$C_{29}H_{21}NBr_2$	64·11	3·89	2·58	29·42	1 650 1 600 1 575
	(52)	(543·3)	64·15	3·92	2·62	29·37	3 430[d]
VII	212–215[e]	$C_{30}H_{23}NBr_2$	64·65	4·16	2·51	28·68	1 649 1 602 1 580
	(30)	(557·3)	64·70	4·20	2·56	28·50	
VIII	210–211·5	$C_{29}H_{20}SBr_2$	62·16	3·60	5·72[f]	28·52	1 593 1 573
	(82)	(560·3)	62·11	3·62	5·72[f]	28·52	
IX	222·5–223	$C_{29}H_{20}OCl_2$	76·49	4·43	—	15·57	1 674 1 600 1 580
	(80)	(455·4)	76·27	4·36	—	15·25	
XII	197–198	$C_{29}H_{20}SCl_2$	73·89	4·28	6·80[f]	15·03	1 594 1 575
	(68)	(471·4)	74·15	4·32	6·69[f]	14·90	
XIII	187–189	$C_{29}H_{20}NCl_3$	71·25	4·12	2·86	21·76	1 605 1 598
	(93)	(488·9)	71·21	4·21	2·80	21·64	
XV	oil	$C_{29}H_{20}N_2O_5$	73·10	4·23	5·88	—	1 670 1 598 1 572
	(32)	(476·5)	73·21	4·28	5·80	—	1 520[g] 1 346[g]
XVI	228–232	$C_{29}H_{21}N_3O_4$	73·25	4·45	8·84	—	1 634 1 600 1 578
	(32)	(475·5)	73·30	4·50	8·70	—	1 522[g] 1 350[g] 3 420[d]
XVII	313–315	$C_{30}H_{23}N_3O_4$					1 630 1 598 1 570
	(20)	(489·1689)		489·1698[h]			1 522[g] 1 350[g]
XVIII	280·5–282	$C_{29}H_{20}N_2O_4S$	70·72	4·09	5·68	6·51[f]	1 610 1 595 1 575
	(41)	(492·6)	70·63	4·17	5·76	6·65[f]	1 523[g] 1 340[g]
XIX	295–297	$C_{30}H_{24}N_2O_2$					1 630 1 598 1 570
	(11)	(444·1838)		444·1833[h]			1 518[g] 1 344[g]
XX	190·5–192	$C_{29}H_{21}NSO_2$	77·83	4·72	3·13	7·16[f]	1 595 1 573
	(17)	(447·6)	77·69	4·99	3·03	7·35[f]	1 525[g] 1 360[g]

[a] Valence vibrations in phenyl and heterocyclic rings; [b] % of halogen in the corresponding formula; [c] ref.[4] gives m.p. 215–216°C; [d] NH stretching vibration; [e] decomp.; [f] % of sulfur; [g] NO_2 stretching vibrations; [h] determined by high resolution MS measurements.

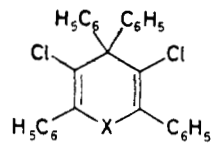
products which have not been investigated systematically. In the case of 1,4-dihydropyridine derivative *X* we managed to isolate the not too stable trichloro derivatives of the probable formula *XIII*. The formation of this product can be explained by



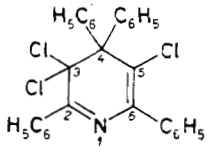
I, X = O
II, X = NH
III, X = NCH_3
IV, X = S



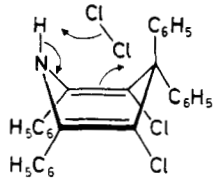
V, X = O
VI, X = NH
VII, X = NCH_3
VIII, X = S



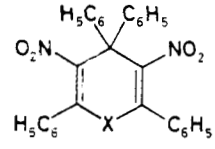
IX, X = O
X, X = NH
XI, X = NCH_3
XII, X = S



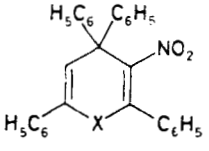
XIII



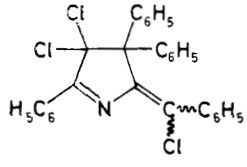
XIV



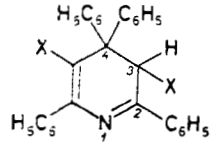
XV, X = O
XVI, X = NH
XVII, X = NCH_3
XVIII, X = S



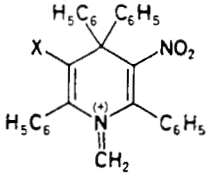
XIX, X = NCH_3
XX, X = S



XXI



XXII, X = Br
XXIII, X = NO_2



XXIV, X = NO_2
XXV, X = H

the sequence *II* → *X* → *III*, using the idea of the reacting supermolecule *XIV* at the last stage of the conversion. N-Methyl derivative *III* is still more reactive with dichlorine. The ^{1}H NMR spectrum of the reaction mixture lacks the methyl signal, which indicates the absence of the assumed product *XI*. 3,5-Dichloro derivative *XII* could be obtained by decreasing the reaction temperature to 0°C and limiting the reaction time to 1 min.

Similarly as in other highly nucleophilic substrates[10,11], 4*H*-pyran *I* undergoes chlorination with phosphorus pentachloride at elevated temperature. Using this procedure we succeeded in obtaining 3,5-dichloro derivative *IX* in high yield (Table I). Attempts at iodination of compounds *I*–*IV* with diiodine were unsuccessful and always led only to a recovery of the starting compounds. This circumstance indicates an addition–elimination mechanism followed by halogenation, where in the case of the reaction with iodine the first addition step is evidently sterically blocked in view of the character of the molecules *I*–*IV*.

TABLE II

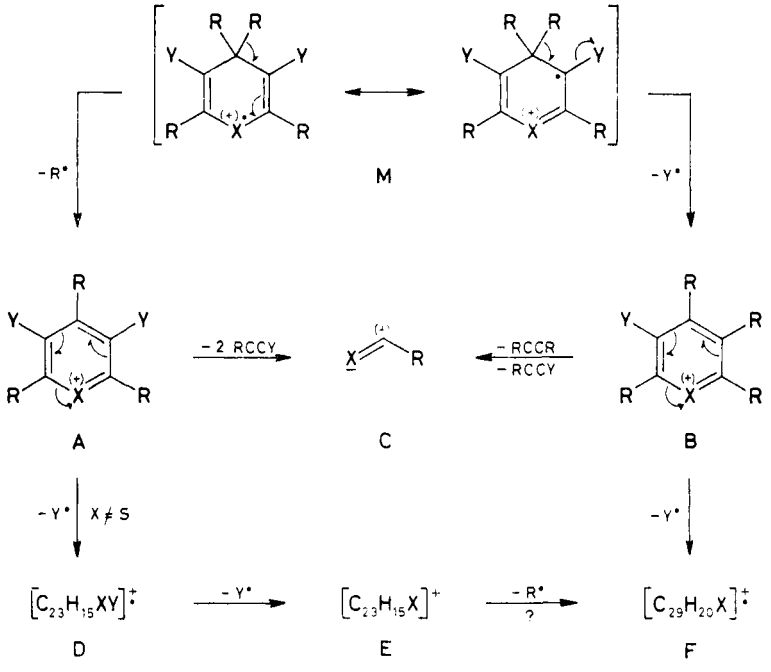
^{1}H NMR spectra of compounds *V*–*VI*, *XII*, *XIII*, *XV*–*XX*

Compound	*ortho*-H_{Ar}	*meta*, *para*-H_{Ar}	NR (R)
V	7·69–7·71 m 7·58–7·61 m	7·32–7·45 m	–
VI	7·73–7·75 m	7·31–7·47 m	5·45 s (H)[a]
VII	7·68–7·72 m	7·28–7·43 m	2·34 s (CH_3)
VIII	7·68–7·80 m	7·24–7·44 m	–
IX	7·60–7·71 m	7·29–7·44 m[b]	–
XII	7·68–7·74 m	7·30–7·48 m	–
XIII	8·05–8·07 m 7·60–7·71 m	7·20–7·53 m	–
XV	7·80–7·82 m	7·25–7·62 m	–
XVI	7·70–7·72 m	7·30–7·52 m	6·30 s (H)
XVII	7·80–7·82 m 7·70–7·72 m	7·32–7·55 m	2·45 s (CH_3)
XVIII	7·60–7·66 m 7·55–7·59 m	7·33–7·51 m	–
XIX	7·65–7·67 m	7·29–7·51 m	2·50 s (CH_3)
XX	7·59–7·69 m 7·49–7·54 m 7·45–7·46 m	7·26–7·45 m	–

[a] The signal of the tautomer *XXII* is at 5·78 s, 1 H (CHBr); [b] signals of *ortho*-H_{Ar} do not form a separate multiplet.

Nitration of substrates *I–IV* with 100% nitric acid already takes place sufficiently rapidly at 0°C. In all experiments we could isolate corresponding 3,5-dinitro derivatives *XV–XVIII* in good yields. In the case of the starting compounds *III* and *IV* we succeeded in obtaining corresponding intermediates substituted with nitro group in position 3, *XIX* and *XX*.

The molecular structure of compounds *V–IX*, *XII* and *XV–XX* follows clearly from their ^{1}H and ^{13}C NMR spectra (Tables II and III) which were interpreted analogously as the spectra[8,12] of compounds *III* and *IV* and by means of INEPT-LR experiments in the case of compound *XVIII*. Owing to the not too stable product of chlorination of 1,4-dihydropyridine *II* formula *XIII* may be considered as the most probable one, but the given spectral data do not exclude some other alternative structures, for example of type *XXI*. In the case of 3,5-dibromo derivative *VI*, similarly as in the case of the starting compound *II* (see ref.[8]) the presence of 3,4-dihydro tautomer *XXII* was detected in the solution phase (approx. 10% at 20°C, with a rise when the temperature was increased), which manifested itself in the ^{1}H NMR spectrum by a singlet of the methine proton in position 3, at δ 5·78. In



SCHEME 1

TABLE III

^{13}C NMR spectra of the synthesized compounds

Compound[a]	X	C-2	C-2(*i*)	C-2(*o*)	C-2(*m*)	C-2(*p*)	C-3	C-4	C-4(*i*)	C-4(*o*)	C-4(*m*)	C-4(*p*)
V		141·37	134·22	127·93	130·73	129·34	104·89	60·15	147·20	127·48	129·43	127·20
VI	—	137·22	136·27	128·48	130·84	128·48	100·92	62·66	142·96	127·22	129·04	126·63
VII	39·21	141·76	137·56	128·48	130·27	128·54	104·66	61·47	144·65	127·35	129·92	126·62
VIII	—	130·45	137·99	127·53	130·52	127·27	115·98	64·69	141·99	128·47	129·02	128·77
IX	—	140·55	132·91	127·66	130·63	129·40	113·16	59·82	146·14	128·03	129·22	127·29
XII	—	129·87	135·96	127·68	130·45	127·31	123·47	63·90	140·76	128·52	129·15	128·87
XIII[b]	—	161·04	137·00	127·98	129·56	128·57	88·32	67·86	142·26	127·86	129·37	128·31
XV	—	139·86	134·78	128·50	129·11	130·47	132·35	55·80	142·80	128·33	128·91	128·16
XVI	—	140·88	132·08	128·37	129·63	131·27	130·15	56·04	140·94	127·53	128·52	127·66
XVII	37·90	141·91	135·71	128·45	129·55	130·86	130·70	55·40	146·30	128·09	128·58	127·72
XVIII	—	140·23	132·09	128·54	129·35	128·69	145·12	59·89	138·36	128·56	129·69	131·16
XIX[c]	38·03	141·09	134·52	128·85	129·73	131·22	130·22	55·14	147·30	128·48	129·02	128·35
XX[d]	—	137·28	132·64	126·37	127·72	128·18	141·95	54·99	141·84	127·17	127·85	127·95

[a] Abbreviations: (*i*)-*ipso*, (*o*)-*ortho*, (*m*)-*meta* and (*p*)-*para*, respectively, the number denotes the position to which phenyl group is attached; [b] additional signals: 128·80 C-5, 139·00 C-6, 134·85 C-6(*o*), 129·30 C-6(*o*), 129·55 C-6(*m*) and 130·55 C-6(*p*); [c] 123·64 C-5, the C-6 signals were not resolved from the C-2 ones; [d] probable assignment; 129·25 C-5, 131·41 C-6, 135·09 C-6(*i*), 125·85 C-6(*o*), 127·35 C-6(*m*), 128·07 C-6(*p*).

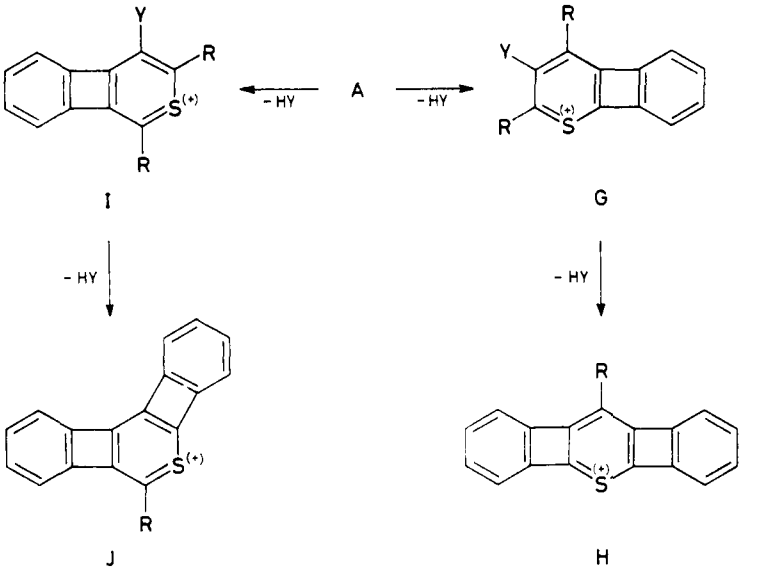
TABLE IV

Mass spectra of the synthesized compounds (70 eV)

Compound	*m/z* (r.i., %), see Schemes 1 and 2
V	544 (~0·1)^{M}, 469 (12), 468 (6); 467 (25), 466 (24), 465 (75)^{A}, 464 (24), 463 (70)^{B}, 388 (4), 387 (3), 386 (8)^{D}, 385 (8), 384 (7)^{F}, 307 (12)^{E}, 279 (15), 278 (13), 277 (7), 276 (8), 232 (5), 202 (9)
VI	541 (5)^{M}, 465 (18), 464 (62)^{A}, 463 (92), 462 (62)^{B}, 461 (80), 385 (20)^{D}, 384 (68), 383 (100)^{F}, 382 (12), 381 (30), 306 (4)^{E}, 305 (65), 304 (16), 303 (13), 302 (13), 301 (7), 278 (8), 277 (12), 276 (18), 191 (10), 190 (11), 189 (10), 184 (18), 183 (16), 138 (9), 104 (5)^{c}, 77 (7), 51 (5)
VII	559 (5), 557 (8), 555 (5)^{M}, 483 (8), 482 (38), 481 (20), 480 (72), 479 (32), 478 (100)^{A}, 477 (30), 476 (71)^{B}, 402 (5), 400 (13), 399 (10)^{D}, 398 (33), 297 (23)^{F}, 396 (15), 320 (15), 320 (30)^{E}, 318 (20), 304 (12), 239 (10), 238 (10), 202 (10), 118 (20)^{C}, 103 (14), 91 (6), 77 (19), 51 (6)
VIII	562 (3), 560 (6)^{M}, 558 (3), 485 (16), 483 (27)^{A}, 481 (100)^{B}, 480 (26), 479 (92), 404 (5), 403 (15), 402 (13), 401 (20)^{G}, 400 (25)^{F}, 399 (12), 325 (10), 326 (22), 323 (42)^{E}, 322 (17), 321 (31)^{H}, 298 (37), 265 (14), 245 (10), 192 (13), 165 (23), 121 (36)^{C}, 97 (23), 69 (47), 54 (53), 52 (38)
IX	456 (~0·5), 454 (3) ^{M}, 452 (1), 422 (4), 421 (43), 419 (100)^{B}, 385 (1), 384 (4)^{F}, 379 (23), 377 (28)^{A}, 344 (2), 342 (4)^{D}, 307 (3)^{E}, 306 (3), 305 (3), 279 (7), 278 (21), 277 (7), 276 (7), 202 (8), 165 (8), 105 (72), 97 (10); 77 (42), 69 (18), 54 (18), 52 (16)
XII	474 (1), 473 (1), 472 (5), 471 (1), 470 (7)^{M}, 439 (5), 438 (15), 437 (47), 436 (37), 435 (100)^{B}, 402 (2), 400 (2)^{F}, 399 (2), 397 (7), 396 (7), 395 (23), 394 (17), 393 (32)^{A}, 395 (8), 358 (5), 357 (20)^{G}, 323 (12)^{E}, 320 (6), 321 (18)^{H}, 289 (9), 165 (13), 121 (22)^{C}, 77 (6)
XV	476 (2)^{M}, 430 (5)^{B}, 400 (5), 399 (7)^{A}, 385 (5), 384 (6)^{F}, 359 (8), 353 (10)^{D}, 313 (12), 307 (15)^{E}, 297 (8), 282 (9), 208 (7), 165 (10), 105 (100)^{C}, 77 (80), 51 (30)
XVI	475 (~0·1), 430 (14), 429 (40)^{B}, 399 (26), 398 (100)^{A}, 383 (12), 382 (10)^{F}, 352 (3)^{D}, 322 (13), 306 (10)^{E}, 295 (10), 279 (5), 278 (5), 277 (5), 276 (6), 202 (10), 189 (8), 165 (8), 104 (15)^{C}, 77 (15), 51 (5)
XVII	489 (35)^{M}, 488 (100), 458 (20), 457 (75), 443 (15)^{B}, 442 (20), 413 (20), 412 (75)^{A}, 397 (8)^{F}, 366 (2)^{D}, 320 (15)^{E}, 319 (20), 304 (8), 277 (5), 118 (30)^{C}, 77 (10), 51 (5)
XVIII	447 (13), 446 (100)^{B}, 417 (3), 415 (33)^{A}, 400 (17)^{F}, 370 (3), 368 (15)^{G}, 352 (12), 339 (10), 323 (25)^{E}, 322 (23), 321 (20)^{H}, 291 (12), 289 (10), 267 (8), 265 (10), 236 (8), 213 (5), 189 (8), 165 (20), 152 (12), 121 (25)^{C}, 105 (20), 97 (23), 83 (27), 71 (30), 69 (36), 54 (49), 52 (38)

contrast to this no spectral manifestation of the analogous tautomer *XXIII* could be observed in solution phase of 3,5-dinitro derivative *XIV* within the limits of determination.

The analysis of the mass spectra of compounds *V–IX*, *XII*, *XV–XVIII* (Table IV) permitted the formulation of common features of the fragmentation mechanism of their molecules under the effect of electron impact (Scheme 1). It is evident that the gradual elimination of 3,5-substituents is also characteristic of them, in addition to the expected[1,2] aromatization of molecular ions. A different course of further fragmentation of the cation A is also observed in 4*H*-thiopyrans *VII*, *XII*, *XVIII*, where elimination of HY molecules takes place under formation of ions of the probable structure G and H, or I and J (Scheme 2) instead of the cleavage of the



(R = C_6H_5 ; Y = Br, Cl, NO_2)

SCHEME 2

radical Y•, which is typical of other cases. N-Methyl-1,4-dihydropyridine derivatives *XVII* and *XIX* are most resistant to fragmentation and they display the main ion species *XXIV* and *XXV*.

EXPERIMENTAL

The temperature data are not corrected. The melting points were determined on a Boetius instrument. The IR spectra were measured on a Perkin–Elmer 325 spectrophotometr, in chloroform,

the NMR spectra (given in ppm δ-scale) on a Bruker AM-400 instrument, in deuteriochloroform. They refer to tetramethylsilane as internal reference, under the conditions described in ref.[8]. The mass spectra were measured on an LKB 9000 instrument or a Jeol 303/DA 5000 instrument (70 eV, direct inlet). The starting compounds *I*—*IV* were prepared according to known procedures[12,13].

Bromination of Compounds *I*—*IV*

Dibromine (0·8 g, 5 mmol) in carbon disulfide (5 ml) was added to a solution of 2·5 mmol of the starting substrate in 20 ml of the same solvent at room temperature and after 5 h stirring the reaction mixture was allowed to stand overnight. After evaporation of the solvent the oily residue usually crystallized out, only the 1-methyl derivative *VII* was isolated by chromatography on silica gel by elution with cyclohexane. The products *V*—*VII* were crystallized from the same solvent, and in the case of 1-thia-analogue *VIII* a mixture of heptane–benzene was used (Table I).

Chlorination of Compounds *II* and *III*

Dry dichlorine was introduced over 2 h into a solution of 1,4-dihydropyridine derivative (2·5 mmol) in carbon disulfide (15 ml) at room temperature and the reaction mixture was then stirred for 5 h and allowed to stand overnight. After evaporation of the solvent the residue was dissolved in benzene and chromatographed on a silica gel column. The isolated trichloro derivative *XIII* was crystallized from cyclohexane (Table I). In the case of starting 1-methylderivative *III* a mixture of oligo derivatives already began to separate in the course of the chlorination. Attempts at crystallization from a mixture of benzene and chloroform or chromatography on silica gel led to decomposition. The ^{1}H NMR spectrum of the mixture originally formed did not contain any methyl signal.

Chlorination of Compounds *I* and *IV*

Carbon disulfide (10 ml) saturated with dichlorine (0·42 g, 5·9 mmol) was added to a solution of the pyran or thiopyran derivative (2·5 mmol) in carbon disulfide (20 ml) under intensive stirring and cooling with ice. The reaction mixture was stirred for 1 min and then decomposed by shaking with an aqueous solution of sodium sulfite. The organic layer was dried over magnesium sulfate and evaporated in a vacuum. In this way 4*H*-pyran *I* afforded a mixture of 10 compounds (HPLC analysis), which was not further separated. 4*H*-Thiopyran *XII* was obtained by chromatography of the raw mixture on a silica gel column (100 g) with benzene and subsequent crystallization from heptane (Table I)

Chlorination of 4*H*-Pyran Derivative *I* with Phosphorus Pentachloride

A mixture of 4*H*-pyran *I* (0·5 g, 1·25 mmol) and phosphorus pentachloride (1·1 g, 5 mmol) was dissolved in phosphorus oxychloride (10 ml) and heated at 120—130°C (bath temperature) for 2 h. The reaction mixture was decomposed by pouring it onto ice and the precipitate formed was filtered off under suction, dried and crystallized from heptane–benzene (Table I).

Attempts at Iodination of Compounds *I*—*IV*

A solution of diiodine (1·28 g, 5 mmol) in carbon disulfide (10 ml) was added to a solution of the starting derivative (2·5 mmol) in the same solvent (10 ml) and the mixture was stirred at room temperature for 5 h. It was then washed with a dilute aqueous solution of sodium sulfite

until the reagent disappeared completely. The organic extracts were evaporated to give only the starting compounds *I*—*IV* in quantitative yield.

Nitration of Compounds *I*—*IV*

Nitric acid (100%, 0·35 g, 5·5 mmol) was added dropwise to a solution of the starting derivative (2·5 mmol) in chloroform (20 ml) under intensive stirring and cooling with ice and the reaction mixture was further stirred at 0°C for 1 h in the case of subtrates *I*—*III*, and for 3 h in the case of substrate *IV*. After washing with 10 ml of icy water the organic layer was separated, washed with water until neutral, dried over magnesium sulfate and evaporated in a vacuum. The oily crude products were chromatographed on a silica gel column (100 g), using benzene for elution in the case of the starting compounds *I*—*III* or heptane–benzene mixture (2 : 1) in the case of substrate *IV*. The first chromatographic fractions contained 3-nitro derivative *XIX* (120 mg) in the case of the starting 1-methyl derivative *III*, while the other compounds gave 3,5-dinitro derivative *XVII* (240 mg), see Table I. In the case of the starting substrate *IV* 3-nitro derivative *XX* (180 mg) and 3,5-dinitro derivative *XVIII* (500 mg) were obtained.

3,5-Dinitro derivatives *XV*—*XVIII* were crystallized from heptane–benzene, and the 3-nitro derivative *XX* from ethanol.

3-Nitro-2,4,4,6-tetraphenyl-4*H*-thiopyran *XX*

The reaction of 4*H*-thiopyran *IV* with 100% HNO_3 was carried out as in the preceding experiment but the reaction time was only 8 min. Chromatography of the crude product on silica gel (100 g) with heptane–benzene (2 : 1) gave 680 mg (61%) of 3-nitro derivative *XX* and 100 mg (8%) of 3,5-dinitro derivative *XVIII*.

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