Tetrahedron Vol 36, pp 3309 to 3315
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0040-4020/80/1115-3309/\$02.00/0

STUDIES ON ORGANOPHOSPHORUS COMPOUNDS†

THIATION OF 3,1-BENZOXATHIAN-4-ONES. NEW ROUTES TO 1,2-BENZISOTHIAZOLE-3(2H)-THIONES AND 3H-1,2-BENZODITHIOL-3-IMINES

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(*Received in UK* 29 *February* 1980)

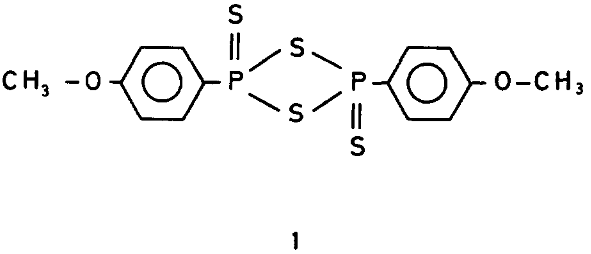
Abstract—3,1-Benzoxathian-4-ones, **2**, when heated with 2,4-bis(4-methoxyphenyl)-1,3,2,4-dithiadiphosphetane-2,4-disulfide, **1**, or with P_4S_{10} give one or more of the following products: 3,1-benzoxathian-4-thione, **3**, 1,3-benzodithian-4-one, **4**, 1,3-benzodithian-4-thione, **5**, and 3H-1,2-benzodithiole-3-thione, **6**. Compounds **2**, when heated with primary and secondary amines and with hydrazines, give 2-mercaptobenzamides, **7**, and 2-mercaptobenzohydrazides, **8**, or their corresponding disulfides, **7′** and **8′**. 3H-1,2-Benzodithiol-3-imines, which are in equilibrium with 1,2-benzisothiazole-3(2H)-thiones, (**9A** $\rightleftarrows$ **9B**), are prepared by two new routes: (a) by allowing **3** or **5** to react with primary amines or hydrazines, (b) by allowing **7**, **8**, **7′** or **8′** to react with **1**.

The reaction of aldehydes with 2-mercaptobenzoic acid smoothly gives 3,1-benzoxathian-4-ones, **2**[1-3], but no basic chemistry of **2** seems to be known. In our search for acylating and thioacylating reagents it was thought that the activated ester **2** and its thiono-derivatives, **3** and **5**, would react with nucleophiles to give acylated (thioacylated) products. We therefore have reacted **2** with 2,4-bis(4-methoxyphenyl)-1,3,2,4-dithiadiphosphetane-2,4-disulfide, **1**, and with P_4S_{10} to obtain **3**–**6**. Compound **1** is a new thiation reagent, which has been found to give very high yields of products when reacted with ketones[4], carboxamides[5-9], esters[10], S-substituted thioesters[10], compounds containing two carbonyl functions[10-15], and different carbonyl-containing heterocycles[11,12,14,15]. Besides the thiation studies this paper reports on the reaction of **2**, **3** and **5** with nucleophiles and also on the reaction of 2-mercaptobenzamides, **7**, with **1** to give new unexpected routes to 3H-1,2-benzodithiol-3-imines, **9A**, and 1,2-benzisothiazole-3(2H)-thiones, **9B**. It is known[6] that salicylic amides, when reacted with **1**, produce salicylic thioamides + P-heterocycles.

RESULTS AND DISCUSSION

By refluxing 3,1-benzoxathian-4-ones, **2**, with 2,4-bis(4-methoxyphenyl)-1,3,2,4-dithiadiphosphetane-2,4-disulfide, **1**, in benzene or toluene, or with P_4S_{10} in toluene or xylene one or more of the following products: 3,1-Benzoxathian-4-thione, **3**, 1,3-benzodithian-4-one, **4**, 1,3-benzodithian-4-thione, **5**, and 3H-1,2-benzodithiole-3-thione, **6**, are isolated (Scheme 1). Based on earlier findings[11,12] it is suggested that **2** is thiated to give **3**, which rearranges to **4**, and a final thiation then gives **5**. Compound **6** is known to be formed from the reaction of P_4S_{10} with different derivatives of 2-mercaptobenzoic acid[16-18]. We have also isolated **6** in high yields from the reaction of **1** with 2-mercaptobenzoic acid, ethyl 2-mercaptobenzoate, 2,2′-dithiobisbenzoic acid, and diethyl 2,2′-dithiobisbenzoate.

Compounds **2** have been allowed to react with different types of nucleophiles but in our hands only amines and hydrazines gave the aldehyde RCHO and the reaction products: 2-Mercaptobenzamides, **7**[19-27], 2-mercaptobenzohydrazides, **8**[28-32], and the



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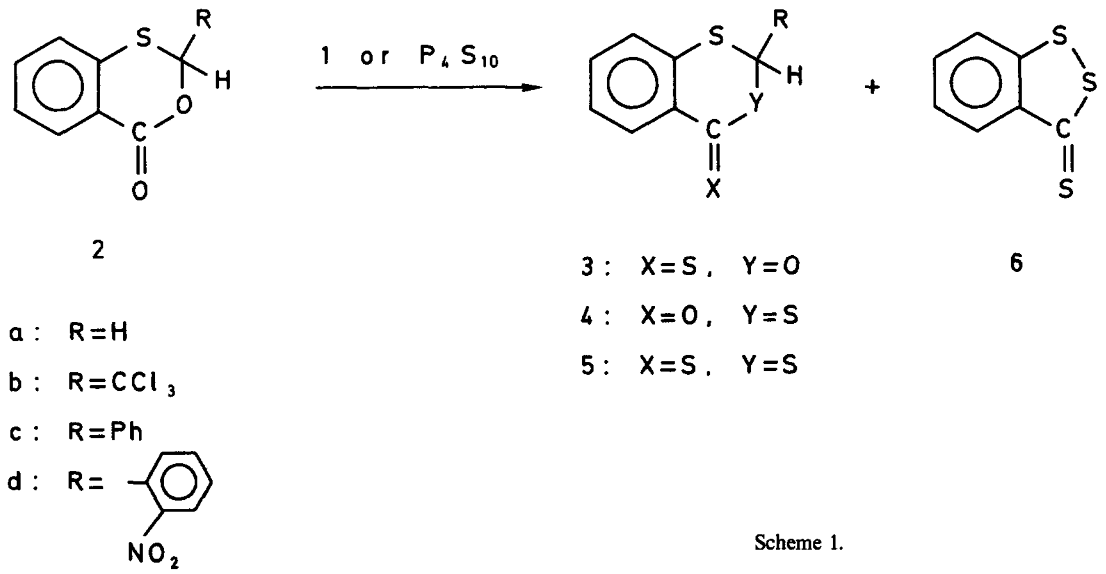
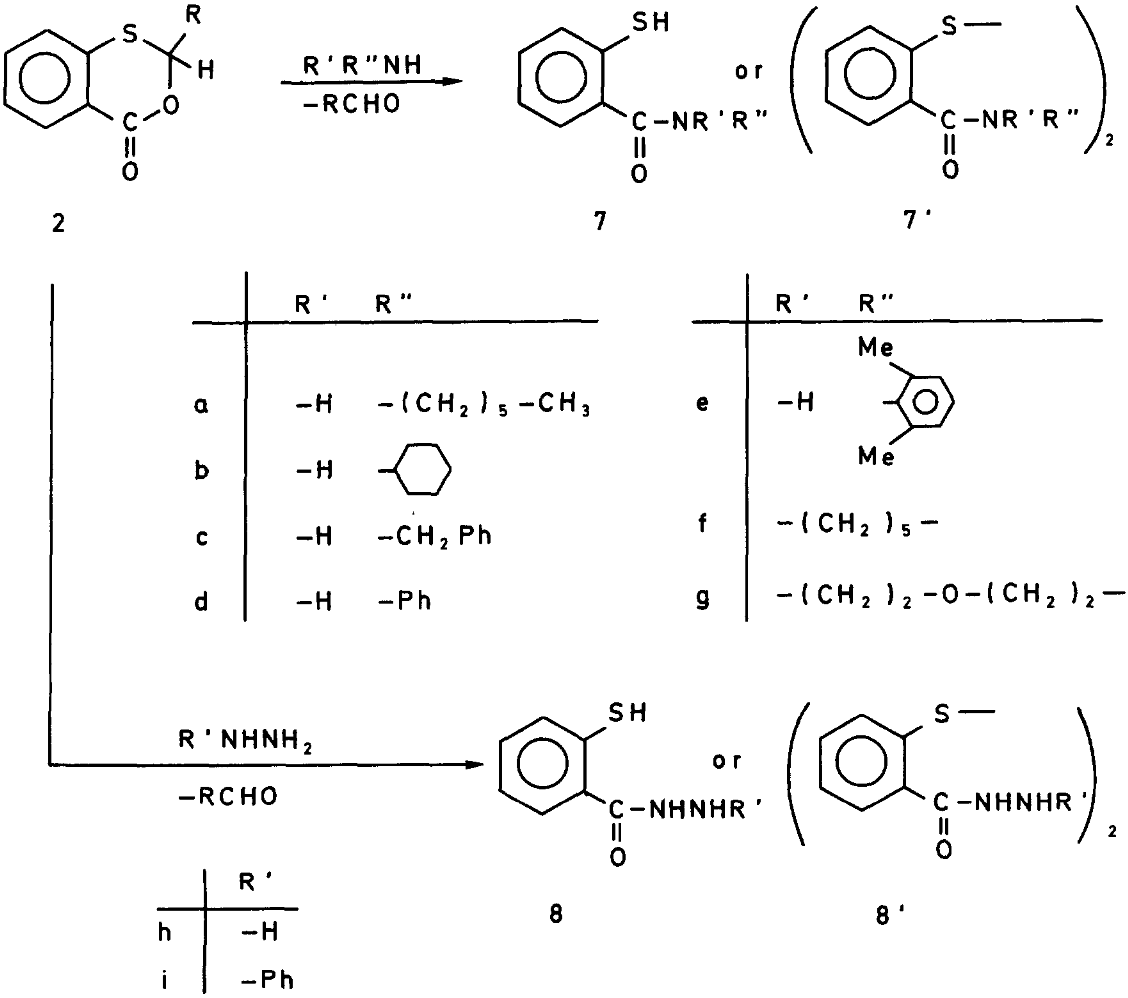
† Part XXXIII. Part XXXII, K. A. Jørgensen, R. Shabana, S. Scheibye and S.-O. Lawesson, *Bull. Soc. Chim. Belg.* **89**, 247 (1980).

§ On leave from Faculty of Science, University of Tanta, Tanta, Egypt.

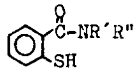
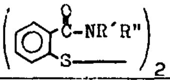
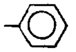
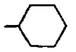
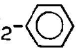
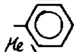
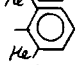
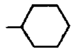
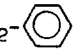
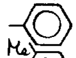
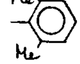


Scheme 1.



Scheme 2.

Table 1. Experimental data for the reaction of 3,1-benzoxathian-4-ones, **2**, with primary and secondary amines

	R′R″NH			Products (% Yields)		
Starting compound	R′	R″	Reaction time (h)			Mp. (°C)
2a	-H		6	7d (29)		110^{19}
2a	-(CH$_2$)$_2$-O-(CH$_2$)$_2$-		2.5		7′g (32)	143^{27}
2b	-H	-(CH$_2$)$_5$-CH$_3$	7		7′a (58)	159^{46}
2b	-H		10		7′b (74)	249^{46}
2b	-H	-CH$_2$-	0.2		7′c^{b}(49)	206^{46}
2b	-H		20		7′d (46)	248^{19}
2b	-H		8		7′e (55)	288^{a}
2b	-(CH$_2$)$_2$-O-(CH$_2$)$_2$-		1.5		7′g (56)	
2c	-H		4.5	7b (76)		124^{47}
2c	-H	-CH$_2$-	0.7	7c^{b}(93)		108^{48}
2c	-H		1.0	7d (30)		
2c	-H		12		7′e (39)	
2c	-(CH$_2$)$_5$-		1.3	7f (89)		69^{27}
2c	-(CH$_2$)$_2$-O-(CH$_2$)$_2$-		3.5		7′g (63)	
2d	-H	-(CH$_2$)$_5$-CH$_3$	4		7′a (58)	

a) Anal. F: C 69.99, H 5.48, N 5.44, S 12.66. Calc. C 70.31, H 5.47, N 5.47, S 12.50 %.

b) In literature 7c and 7′c are sometimes confused.[20,21,23,26]

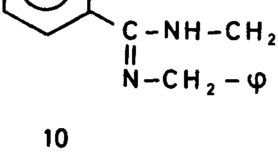
corresponding disulfides **7′** and **8′** (Scheme 2, Tables 1 and 2). Sodium ethoxide, sodium phenoxide and sodium benzyl sulfide only gave the hydrolysis product 2-mercaptobenzoic acid.

Compounds **3** and **5**, being thioacylating reagents, did not give the expected 2-mercapto-*N*-benzylthiobenzamide (reported to be unstable[33,43]; but see also patent literature[44]) when reacted with benzylamine, but a subsequent oxidation produced **9Ac** ⇄ **9Bc** (route a). A by-product from the reaction of **3** (R = H) and benzylamine is *N*,*N*′-dibenzyl 2-mercaptobenzamidine, **10**, characterized by ^{1}H NMR, MS and elemental analyses.

It is a well-known fact that 3H-1,2-benzodithiol-3-imines, **9A** (for R′ = alkyl, NH$_2$) are in equilibrium (known as the Dimroth-rearrangement[38,41]) with 1,2-benzisothiazole-3(2H)-thiones, **9B**, whereas (for R′ = aryl, NHR″, H, OH, OR, and acyl) 3H-1,2-benzodithiol-3-imines, **9A**, are the only isomers found[33–41]. As the assignments of the two structures **9A** and **9B** earlier have been made by means of ^{1}H NMR, IR, and UV spectroscopy[34,40] and X-ray diffraction measurements[42], further comments are unnecessary except that also ^{13}C NMR confirms the structure of **9B** as absorptions at $\delta = 183.8$–185.3 ppm corresponding to a thiocarbonyl carbon are observed.

Another route (b) to **9A** ⇄ **9B** has also been devised: Reaction of *N*-substituted 2-mercaptobenzamides, **7**, or the corresponding disulfides, **7′**, with **1** in anhydrous benzene at 80° or in HMPA at 100° gave as main products **9A** ⇄ **9B**. However, the *N*,*N*-disubstituted 2-mercaptobenzamides (or the disulfides) when heated with **1** or P_4S_{10} under the same conditions as above, only gave 3H-1,2-benzodithiole-3-thione, **6**.

It should also be mentioned that **3** or **5**, when reacted with phenylhydrazine or hydroxylamine, gave the known **9Ai** and **9Aj**, respectively. With **5** (R = ϕ) and phenylhydrazine also 4-phenylhydrazono-2-phenyl-1,3-benzodithian, **11**, was isolated as a by-product. The proof of **11** is based on ^{1}H NMR, ^{13}C NMR, MS, and elemental analyses.



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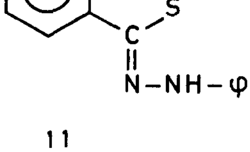

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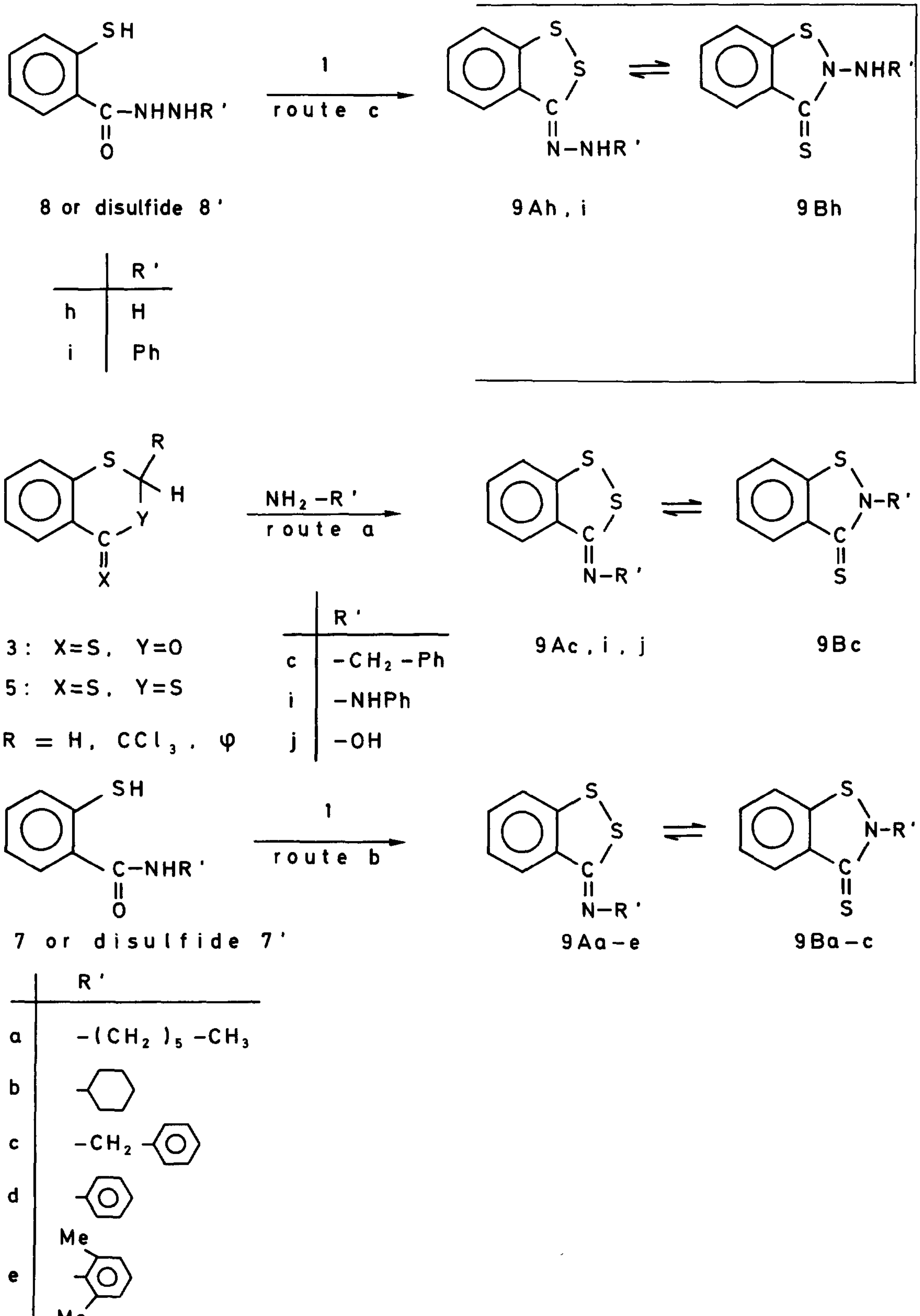
Another new route (c) to **9Ah, i** and **9Bh** is that 2-mercaptobenzohydrazides **8** (R′ = H or ϕ) or the corresponding disulfides are reacted with **1** to give **9Ah** ⇄ **9Bh** and **9Ai** respectively. Also here no 2-mercaptothiobenzohydrazide was isolated.

In conclusion it can be stated that new synthetic ways of preparing **9A** ⇄ **9B** have been worked out and also that 3,1-benzoxathian-4-ones are suitable starting materials for the preparation of 2-mercaptobenzamides and related derivatives.



Scheme 3

Table 2. Experimental data for the reaction of 3,1-benzoxathian-4-ones, **2**, with hydrazines

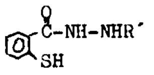
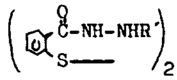
Starting compound	H_2N-NHR′ R′	Reaction time (h)	Products (% Yields) 		Mp. (°C)
2c	-H	24		8′h (36)	217[48]
2c	-Ph	17	8i (56)		186[49]

Table 3. Experimental data for the reactions of **3** and **5** with primary amines and hydrazines and for **7**, **7′**, **8** and **8′** with **1**

Starting compound	Reacted with	Products (% yields)	Reaction time (h)
3a	$PhCH_2NH_2$	9Ac - 9Bc (33)[50]	0.5
3a	$PhNHNH_2$	9Ai (75)[33]	5
3b	$PhCH_2NH_2$	9Ac - 9Bc (58)	0.5
3b	$PhNHNH_2$	9Ai (89)	2
3b	NH_2OH	9Aj (60)[33]	0.1
5c	$PhCH_2NH_2$	9Ac - 9Bc (48)	0.5
5c	$PhNHNH_2$	9Ai (37)	3.5
5c	NH_2OH	9aj (82)	0.1
7b	1/HMPA	9Ab - 9Bb (52)[51]	3.5
7c	1/HMPA	9Ac - 9Bc (45)	1.5
7d	1/HMPA	9Ad (13)[50]	1
7′a	1/HMPA	9Aa - 9Ba (62)[51]	4
7′c	1/HMPA/φH	9Ac - 9Bc (54)	9
7′e	1/HMPA	9Ae (19)	15
8′h	1/HMPA	9Ah - 9Bh (5)[33]	18
8i	1/HMPA	9Ai (50)	1

EXPERIMENTAL

^{1}H NMR spectra were recorded at 60 MHz on a Varian A-60 spectrometer. ^{13}C NMR spectra were recorded at 20 MHz on a Varian CFT-20 spectrometer. TMS was used as internal standard and chemical shifts are expressed in δ-values. $CDCl_3$ or DMSO were used as solvents. IR spectra were recorded on a Beckman IR-18 spectrometer. Mass spectra were recorded on a Micromass 7070 F mass spectrometer operating at 70 eV using direct inlet. Elementary analyses were carried out by Novo Microanalytic Laboratory, Novo Industry A/S, Novo Allé, DK-2880 Bagsværd, supervised by Dr. R. E. Amsler. Silica gel 60 (Merck) was used for chromatography. M.ps are uncorrected.

Compound **1** was prepared as described earlier[4].

3,1-Benzoxathian-4-ones, **2a-d**, were prepared from 2-mercaptobenzoic acid and the respective aldehydes according to known procedures[1-3]. In ^{13}C NMR of **2a-d** the carbon absorptions of the carbonyl groups were found in the range 161.3–164.2 ppm.

Reaction of 3,1-benzoxathian-4-ones, **2**, *with* **1** *and with* P_4S_{10}. 0.01 mole of **2** was refluxed with 0.01 mole of **1** in anhyd benzene or toluene or with 0.01 mole of P_4S_{10} in anhyd toluene or xylene until no more starting material could be detected (tlc). The solvent was evaporated and the mixture subjected to column chromatography on silica gel using ether/light petroleum as eluant.

Reaction of 3,1-benzoxathian-4-one, **2a**, *with* **1** (5 hr). 3,1-Benzoxathian-4-thione, **3a**, yield 68%, m.p. 67°. ^{13}C NMR: C2 70.2, C4 204.9. (Found: C, 52.52; H, 3.26; S, 35.04. $C_8H_6OS_2$ requires: C. 52.72; H, 3.32; S, 35.18%).

Compound **2a** with P_4S_{10} (4 hr) yielded 11% of 1,3-benzodithian-4-thione, **5a**, m.p. 60°. ^{13}C NMR: C2 32.6, C4 220.5 and 3H-1,2-benzodithiole-3-thione, **6**, yield 27%, m.p. 98° (lit.[33] 98°). ^{13}C NMR: C3 216.3.

Reaction of 2-trichloromethyl-3,1-benzoxathian-4-one, **2b**, *with* **1** (16 hr). 2-Trichloromethyl-3,1-benzoxathian-4-thione, **3b**, yield 28%, m.p. 104°. ^{13}C NMR: C2 91.6, C4 202.2. (Found: C, 36.06; H, 1.67; Cl, 35.05; S, 21.74. $C_9H_5Cl_3OS_2$ requires: C, 36.08; H, 1.68; Cl, 35.50; S, 21.40%).

Compound **2b** with P_4S_{10} (4 hr). **3b**, yield 24%. **6**, yield 35%.

Reaction of 2-phenyl-3,1-benzoxathian-4-one, **2c**, *with* **1** (16 hr). 2-Phenyl-1,3-benzodithian-4-thione, **5c**, yield 39%, m.p. 94°. ^{13}C NMR: C2 52.5, C4 219.2. (Found: C, 61.21; H, 3.61; S, 34.94. $C_{14}H_{10}S_3$ requires: C, 61.27; H, 3.67; S, 35.05%). **6**, yield 19%.

Compound **2c** with P_4S_{10} (4 h): **5c**, yield 35%. **6**, yield 15%. *Reaction of* 2-*o*-nitrophenyl-3,1-benzoxathian-4-one, **2d**, with **1** (16 hr): **6**, yield 33%.

Compound **2d** with P_4S_{10} (4 hr). 2-*o*-Nitrophenyl-1,3-benzodithian-4-thione, **5d**, yield 29%, m.p. 142°. ^{13}C NMR: C2 46.4, C4 220.6. (Found: C, 51.73; H, 2.92; N, 4.23; S, 29.95. $C_{14}H_9NO_2S_3$ requires: C, 52.66; H, 2.82; N, 4.39; S, 30.09%). **6**, yield 30%.

Compound **6** from 2-mercaptobenzoic acid, ethyl 2-mercaptobenzoate, 2,2′-dithiobisbenzoic acid, diethyl 2,2′-dithiobisbenzoate or 3H-1,2-benzodithiol-3-one. Same procedure as above for the reaction of **2** with **1**. Reaction time: 2–10 hr, yields 78–98%.

Preparation of 2-*mercaptobenzamides*, **7**, *and* 2-*mercaptobenzohydrazides*, **8**. 0.01 mole of **2**, and 0.03 mole of amine or hydrazine were refluxed in 10 ml anhyd benzene under N_2 until no more starting material could be detected (tlc). After cooling to room temp the mixture was poured into water and acidified with dil HCl. The two phases were separated and the organic phase extracted with dil NaOH aq. After neutralization the ppt was filtered off and dried. Experimental and physical data are given in Tables 1 and 2.

Preparation of 2-*mercaptobenzanilide*, **7d**, *and* 2,2′-*dithiobis*(2,6-*dimethylbenzanilide*, **7′e**. 0.02 mole of **2**, and anilinomagnesium bromide or 2,6-dimethyl-anilinomagnesium bromide (prepared according to the procedure given by Aksonova[45]) were refluxed until no more **2** could be detected (tlc). Working up of the products: same as above.

Reaction of 3,1-*benzoxathian*-4-*thione*, **3**, *and* 1,3-*benzodithian*-4-*thione*, **5**, *with benzylamine, phenylhydrazine and hydroxylamine* (route a). 0.01 mole of **3** or **5** and 0.03 mole of the amine, hydrazine or hydroxylamine were refluxed in anhyd benzene under N_2 until no more starting material could be detected (tlc). The solvent was evaporated and the mixture subjected to column chromatography on silica gel using ether/light petroleum as eluant. Experimental and physical data are given in Table 3.

For **3a** with benzylamine a by-product, N,N′-*dibenzyl*-2-*mercaptobenzamidine*, **10**, was isolated, m.p. 184°. (Found: C, 75.10; H, 6.03; N, 8.49; S, 9.69. $C_{21}H_{20}N_2S$ requires: C, 75.90; H, 6.02; N, 8.43; S, 9.64%). MS: *m/e* 332 (M^+, 52%). Another by-product was **7′c**.

For **5c** with phenylhydrazine 4-*phenylhydrazono*-2-*phenyl*-1,3-*benzodithian*, **11**, was isolated as a by-product, m.p. 148°. ^{13}C NMR: C2 50.3. (Found: C, 68.29; H, 4.59; N, 8.00; S, 18.34. $C_{20}H_{16}N_2S_2$ requires: C, 68.96; H, 4.59; N, 8.04; S, 18.36%). MS: *m/e* 348 (M^+, 100%).

Reaction of N-*substituted* 2-*mercaptobenzamides*, **7**, 2-*mercaptobenzohydrazides*, **8**, *or their disulfides*, **7′** *and* **8′**, *with* **1** (route b). 0.01 mole of **7**, **8**, **7′** and **8′** and 0.01 mole of **1** were heated in anhyd benzene (80°) or in HMPA (100°) until no more starting material could be detected (tlc). The solvent was evaporated and the mixture subjected to column chromatography on silica gel with ether/light petroleum as eluant. The experimental data are given in Table 3.

For **7f**, and **7g**, under the same reaction conditions only **6** was isolated in 72% and 75% yield, respectively.

Acknowledgements—Thanks are expressed to DANIDA for a grant to one of us (A.A.E.-B.).

REFERENCES

[1] A. Senning and S.-O. Lawesson, *Ark. Kemi* **17**, 261 (1961).
[2] A. Senning and S.-O. Lawesson, *Ibid.* **17**, 387 (1961).
[3] A. Senning and S.-O. Lawesson, *Ibid.* **17**, 489 (1961).
[4] B. S. Pedersen, S. Scheibye, N. H. Nilsson and S.-O. Lawesson, *Bull. Soc. Chim. Belg.* **87**, 223 (1978).
[5] S. Scheibye, B. S. Pedersen and S.-O. Lawesson, *Ibid.* **87**, 229 (1978).
[6] S. Scheibye, B. S. Pedersen and S.-O. Lawesson, *Ibid.* **87**, 299 (1978).
[7] H. Fritz, P. Hug, S.-O. Lawesson, E. Logemann, B. S. Pedersen, H. Sauter, S. Scheibye and T. Winkler, *Ibid.* **87**, 525 (1978).
[8] K. Clausen, B. S. Pedersen, S. Scheibye, S.-O. Lawesson and J. H. Bowie, *Org. Mass Spectrometry* **14**, 101 (1979).
[9] K. Clausen, B. S. Pedersen, S. Scheibye, S.-O. Lawesson and J. H. Bowie, *Int. J. Mass Spectrum. Ion Phys.* **29**, 223 (1979).
[10] B. S. Pedersen, S. Scheibye, K. Clausen, and S.-O. Lawesson, *Bull. Soc. Chim. Belg.* **87**, 293 (1978).
[11] S. Scheibye, J. Kristensen and S.-O. Lawesson, *Tetrahedron* **35**, 1339 (1979).
[12] K. Clausen and S.-O. Lawesson, *Bull. Soc. Chim. Belg.* **88**, 305 (1979).
[13] B. S. Pedersen and S.-O. Lawesson, *Tetrahedron* **35**, 2433 (1979).
[14] K. Clausen and S.-O. Lawesson, *Nouv J. Chim.* **4**, 43 (1980).
[15] R. Shabana, S. Scheibye, K. Clausen, S. O. Olesen and S.-O. Lawesson, *Ibid.* **4**, 47 (1980).
[16] F. S. Fowkes and E. W. McClelland, *J. Chem. Soc.* 187 (1941).
[17] E. Klingsberg and A. M. Schreiber, *J. Am. Chem. Soc.* **84**, 2941 (1962).
[18] S. Palazzo and L. I. Giannola, *Atti Accad. Sci. Lett. Arti Palermo*, Parte 1 1971–73 (Pub. 1973), **32**, 21; *Chem. Abstr.* **81**, 49605n (1974).
[19] A. Reissert and E. Manns, *Chem. Ber.* **61B**, 1308 (1928).
[20] I. V. Hopper, J. H. MacGregor and F. J. Wilson, *J. Soc. Dyers Colourists* **57**, 6 (1941).
[21] W. Baker, A. S. El-Nawawy and W. D. Ollis, *J. Chem. Soc.* 3163 (1952).
[22] R. Boudet, *Bull. Soc. Chim. Fr.* 322 (1956).
[23] A. E. Kretov and A. S. Bespalyi, *Zh. Obshch. Khim.* **34**, 1307 (1964); *Chem. Abstr.* **61**, 1862c (1964).
[24] J. S. Hanker, L. Katzoff, H. R. Rosen, M. L. Seligman, H. Ueno and A. M. Seligman, *J. Med. Chem.* **9**, 288 (1966).
[25] G. Y. Paris and C. H. Chambers, *Ibid.* **9**, 971 (1966).
[26] H. Schindlbauer, *Monatsh. Chem.* 1799 (1968).
[27] M. Wolf, J. H. Sellstedt and R. L. Fenichel, *Ger. Offen.* 2,310,572 (1973); *Chem. Abstr.* **79**, 136848w (1973).
[28] L. Katz, L. S. Karger, W. Schroeder and M. S. Cohen, *J. Org. Chem.* **18**, 1380 (1953).
[29] L. Katz, *U.S. Pat.* 2,767,173 (1956); *Chem. Abstr.* **51**, P6704b (1957).
[30] Shenley Industries, Inc., *Brit. Pat.* 767,027 (1957); *Chem. Abstr.* **51**, P17998b (1957).
[31] F. Gialdi, R. Ponci and A. Baruffini, *Farmaco Pavia, Ed. Sci.* **14**, 216 (1959).
[32] F. Boberg and R. Schardt, *Liebigs Ann.* **728**, 44 (1969).
[33] E. W. McClelland and C. E. Salkeld, *J. Chem. Soc.* 1143 (1936).
[34] H. Böshagen, H. Feltkamp and W. Geiger, *Chem. Ber.* **100**, 2435 (1967).
[35] H. Böshagen and W. Geiger, *Ibid.* **101**, 2472 (1968).
[36] Montecatini Edison S.p.A., *Ital. Pat.* 800,015 (1968); *Chem. Abstr.* **70**, 37807q (1969).
[37] G. Ambrosoli, M. P. Ciuti, M. G. Menozzi and M. R. Mingiardi, *Bull. Chim. Farm.* **109**, 251 (1970).
[38] S. Hünig, G. Kiesslich, K.-H. Oette and H. Quast, *Liebigs Ann.* **754**, 46 (1971).
[39] S. Palazzo and L. I. Giannola, *Atti Accad. Sci. Lett. Arti Palermo* Parte 1, **32**, 21 (1971–73; Pub. 1973).
[40] R. Mayer, P. Rosmus and J. Fabian, *J. Chromatog.* **15**, 153 (1964).
[41] M. Wahren, *Z. Chem.* **9**, 241 (1969).
[42] G. D. Andreetti, A. Corradi, P. Domiano and A. Musatti, *Ric. Sci.* **38**, 1104 (1968).
[43] G. Wagner and P. Richter, *Pharmazie* **22**, 611 (1967).
[44] H. C. Stecher, Stecher International S.p.A., *Brit. Pat.* 1,088,498 (1967); *Chem. Abstr.* **68**, P43165d (1968).

[45] V. I. Aksenova, *Sbornik Nauch. Rabot Studentov*, No. 2, 92 (1939); *Chem. Abstr.* **35**, 6238 (1941).
[46] F. Gialdi, R. Ponci and A. Baruffini, Il *Farmaco, Ed. Sci.* **14**, 648 (1959).
[47] R. Ponci, A. Baruffini and F. Gialdi, *Ibid.* **19**, 515 (1964).
[48] F. Gialdi, R. Ponci and A. Baruffini, *Ibid.* **15**, 856 (1960).
[49] S. M. Sicardi, S. Lamdan and C. H. Gaozza, *J. Heterocycl. Chem.* **10**, 1039 (1973).
[50] E. W. McClelland, L. A. Warren and J. H. Jackson, *J. Chem. Soc.* 1582 (1929).
[51] A. Baruffini, P. Borgna, F. Gialdi and R. Ponci, *Il Farmaco, Ed. Sci.* **23**, 3 (1968).