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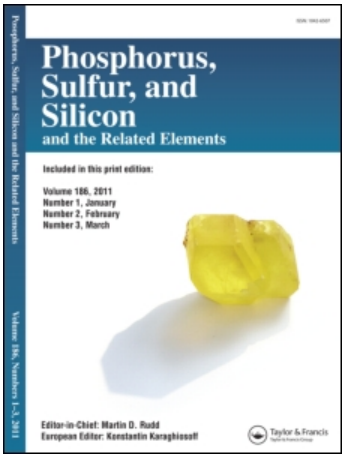

Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

REACTIONS OF N-CHLOROMETHYL-2-THIONO- BENZOXAZOLES AND BENZOTHIAZOLES SYNTHESIS OF *s*-TRIAZOLO- AND 1,2,4,-OXADIAZOLO FUSED SYSTEMS

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To cite this Article Abdel-Rahman, M. A. , Ghattas, A-B. A. G. , El-saraf, G. A. and Khodary, A.(1993) 'REACTIONS OF N-CHLOROMETHYL-2-THIONO- BENZOXAZOLES AND BENZOTHIAZOLES SYNTHESIS OF *s*-TRIAZOLO- AND 1,2,4,-OXADIAZOLO FUSED SYSTEMS', Phosphorus, Sulfur, and Silicon and the Related Elements, 85: 1, 183 — 192
To link to this Article: DOI: 10.1080/10426509308038198
URL: http://dx.doi.org/10.1080/10426509308038198

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Phosphorus, Sulfur, and Silicon, 1993, Vol. 85, pp. 183–192
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REACTIONS OF *N*-CHLOROMETHYL-2-THIONO-BENZOXAZOLES AND BENZOTHIAZOLES SYNTHESIS OF *s*-TRIAZOLO- AND 1,2,4,-OXADIAZOLO FUSED SYSTEMS

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(Received June 9, 1992; in final form September 30, 1993)

N-chloromethyl-2-thiono-benzoxazole 1 and benzothiazole 2 undergo nucleophilic substitution by sulfur, oxygen and nitrogen nucleophiles. Condensation of compounds 1 and 2 with hydrazine, phenylhydrazine and hydroxylamine afforded the *s*-triazolo- and 1,2,4-oxadiazolo fused systems 13–18, respectively. The structures of the resulting products are assigned on the basis of spectral data and elemental analyses.

Key words: N-chloromethyl-2-thionobenzoxazole, N-chloromethyl-2-thionobenzothiazole

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Continuing our studies of the synthetic use of *N*-chloromethyl heterocyclic systems,[1-3] we report the reaction of *N*-chloromethyl benzoxoazole 1 and benzothiazole 2 with some sulfur, oxygen and nitrogen nucleophiles. Condensation of compounds 1 and 2 with hydrazine, phenylhydrazine and hydroxylamine have been studied. These compounds are of synthetic interest as industrial intermediates.[4-7]

Compounds 1 and 2 were prepared from 2-thiono-benzoxazole and benzothiazole, via the corresponding hydroxymethyl derivatives by the action of neat thionyl chloride in 80% and 70% yields, following literature procedures.[8,9]

Treatment of compounds 1 and 2 with aliphatic, aromatic or heterocyclic thiols acting as a sulfur nucleophile in boiling ethanol in presence of sodium ethoxide yielded the corresponding sulfides 3 and 4 in good yields. The structure of products 3 and 4 was proven by elemental analysis, IR and ^{1}H NMR spectra. IR spectra (KBr, cm^{-1}) of compounds 3 and 4 showed the absence of the absorption bands corresponding to C—Cl, while exhibiting the characteristic bands corresponding to C—H (3030–2920), C=N (1630–1590); C=S (1160–1090) and C—S—C (690–670). The ^{1}H NMR spectra of compounds 3 and 4 in $CDCl_3$ or DMSO are in agreement with the proposed structures (cf. Table I).

When compounds 3a, g, h and 4h were allowed to react with mercuric acetate in boiling methanol they afforded the corresponding oxo derivatives 5a–c and 6 in moderate yields.

The IR spectra (KBr, cm^{-1}) of products 5 and 6 showed the absence of the absorption bands corresponding to C=S groups; while exhibiting the characteristic absorption bands corresponding to C=O groups at 1775, 1768, 1770 and 1735. ^{1}H NMR spectra of compounds 5 and 6 are shown in Table II.

Compounds 1 and 2 reacted with sodium phenoxide as an oxygen nucleophile

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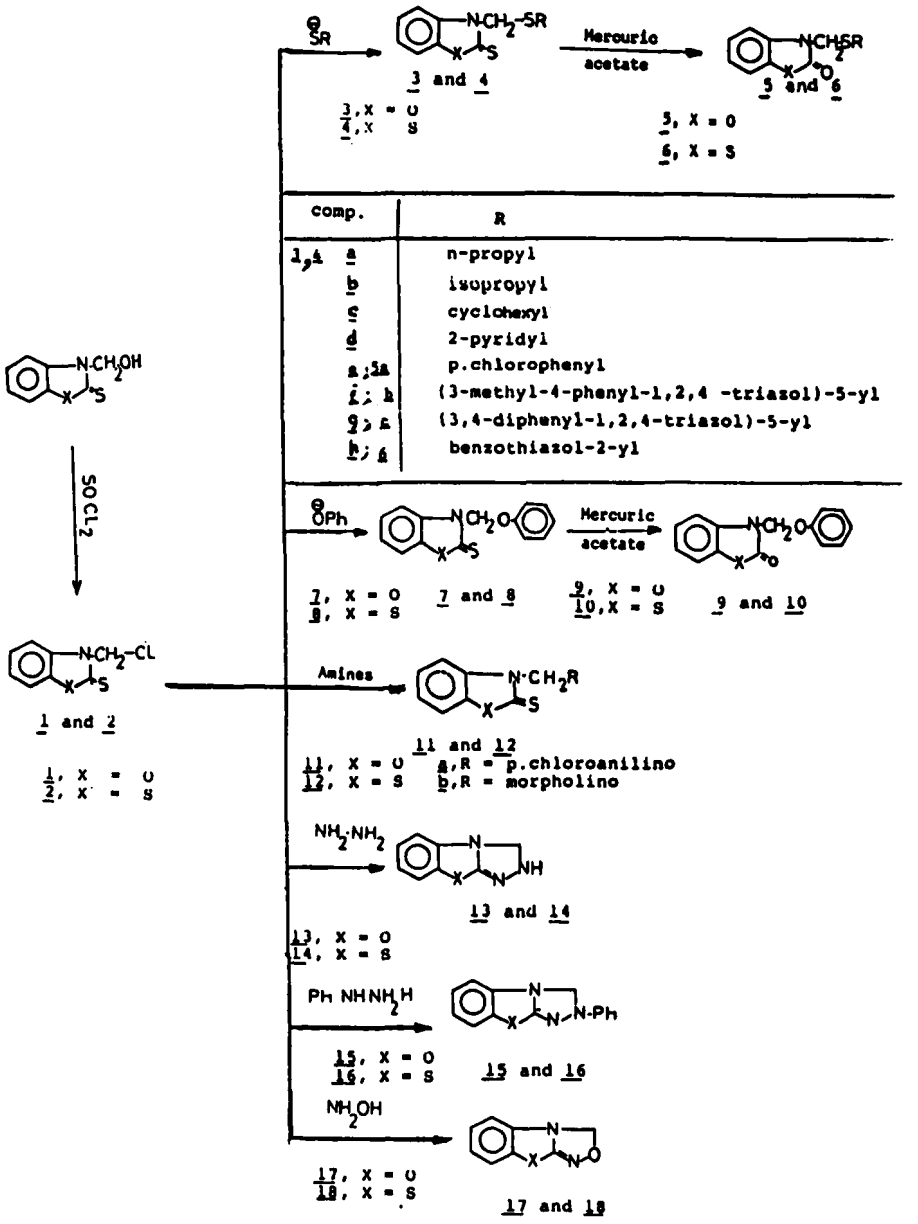

Scheme 1

in refluxing ethanol to yield the corresponding ethers 7 and 8 in good yields.

The IR spectra (KBr, cm^{-1}) of compounds 7 and 8 showed the absence of absorption bands corresponding to C—Cl; while exhibiting the characteristic absorption bands corresponding to C—H (3020–2900), C—O—C (1190–1140) and C=S (1125–1090), respectively. The ^1H NMR spectrum ($CDCl_3$, δ) for compound

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TABLE I

^{1}H NMR spectra of compounds 3 and 4

Comp.	Solvent	^{1}H NMR
3a	$CDCl_3$	7.40-7.20(m,4H,aromatic),5.40(s,2H,N-CH_2-S) ;2.80-2.50(t,2H,S-CH_2) 1.80-1.40(m,2H,-CH_2-CH_3),1.50-0.70 (t,3H,CH_2-CH_3)
3b	$CDCl_3$	7.50-7.20(m,4H,aromatic);5.45(s,2H,N-CH_2-S);3.40-3.10(m,1H,S-CH) 1.50-1.10(d,6H,-C(CH_3)$_2$)
3c	$CDCl_3$	7.41-7.25(m,4H,aromatic);5.45(s,2H,N-CH_2-S);2.10-2.00(m,10H,aliphatic)
3d	$CDCl_3$	8.70-8.30(d,1H,N=CH-);7.70-7.00(m,4H,aromatic) ;6.20(s,2H,N-CH_2-S)
3e	DMSO	7.60-7.10(m,8H,aromatic);6.00(s,2H,N-CH_2-S)
3f	DMSO	7.80-7.10(m,9H,aromatic);5.60(s,2H,N-CH_2-S) ,3.40(s,3H-CH_3)
3g	DMSO	7.60-6.90(m,14H,aromatic);5.70(s,2H,N-CH_2-S)
3h	$CDCl_3$	7.90-7.10(m,8H,aromatic);6.10(s,2H,N-CH_2-S)
4a	$CDCl_3$	7.50-7.20(m,4H,aromatic);5.48(s,2H,N-CH_2-S);2.60-2.50(t,2H,S-CH_2) 1.90-1.50(m,2H,-CH_2-CH_3);1.10-0.80(t,3H,-CH_2-CH_3)
4b	$CDCl_3$	7.60-7.20(m,4H,aromatic);5.44(s,2H,N-CH_2-S) ;1.50-1.20(d,6H,CH(CH_3)$_2$)
4c	$CDCl_3$	7.50-7.20(m,4H,aromatic);5.40(s,2H,N-CH_2-S);3.30-2.90(m,1H,S -CH);2.30-1.20(m,10m, aliphatic)
4d	DMSO	8.50-8.40(d,1H,N=CH),7.70-6.90(m,4H,aromatic); 6.10(s,2H,N-CH_2-S)
4e	$CDCl_3$	8.00-7.30(m,9H,aromatic);6.10(s,2H,N-CH_2-S)
4f	DMSO	7.70-7.10(m,9H,aromatic);5.70(s,2H,N-CH_2-S); 3.50(s,3H,CH_3)
4g	$CDCl_3$	8.00-7.10(m,14H,aromatic);5.70(s,2H,N-CH_2-S)
4h	$CDCl_3$	8.00-7.10(m,8H,aromatic);6.20(s,2H,N-CH_2-S)

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TABLE II

^{1}H NMR spectra of compounds 5 and 6

Comp.	solvent	^{1}H NMR
5a	$CDCl_3$	7.50-7.20(m,4H,aromatic),5.50(s,2H,N-CH_2-S), 3.00-2.60(t,2H,S-CH_2)1.90-1.60(m,2H-CH_2-CH_3); 1.10-1.00(t,3H,-CH_3).
5b	$CDCl_3$	7.90-7.10(m,8H,aromatic); 6.20(s,2H,N-CH_2-S).
5c	DMSO	7.60-7.00(m,14H,aromatic);5.60(s,2H,N-CH-S).
6	$CDCl_3$	8.00-7.10(m,8H,aromatic);6.20(s,2H,N-CH_2-S).

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7 showed signals at (7.8–7.2, m) for nine aromatic protons and at (6.2, s) for two aliphatic protons. A similar pattern has been shown for compound 8 under similar conditions: (7.7–7.1, m) for nine aromatic protons and (6.2, s) for two aliphatic protons.

Transformation of C=S to C=O for 3-phenoxymethyl-2-thiono-benzoxazole 7 and benzothiazole 8 has been achieved by the action of mercuric acetate in boiling methanol giving the corresponding 2-oxo-3-phenoxymethyl-benzoxazole 9 and benzothiazole 10.

The IR spectra (KBr, cm^{-1}) of compounds 9 and 10 showed the absence of absorption bands corresponding to C=S groups, while exhibiting the characteristic absorption bands corresponding to C=O groups at 1735 and 1730. The ^{1}H NMR spectrum ($CDCl_3$, δ) of compound 9 showed signals at 7.6–7.1 (m, 9H, aromatic); 5.8 (s, 2H, N—CH_2—O), while the ^{1}H NMR spectrum ($CDCl_3$, δ) of compound 10 showed signals at: 7.9–7.3 (m, 9H, aromatic) and 6.2 (s, 2H, N—CH_2—O).

Treatment of compounds 1 and 2 with *p*-chloroaniline in boiling benzene in the presence of triethylamine afforded 3-(*p*-chloroanilinomethyl)-2-thiono-benzoxazole 11a and benzothiazole 12a, respectively and when morpholine was used instead of *p*-chloroaniline 3-morpholinomethyl-2-thiono-benzoxazole 11b and benzothiazole 12b, were obtained.

The IR spectra (KBr, cm^{-1}) of compounds 11 and 12 showed the absence of absorption bands corresponding to C—Cl; while exhibiting the characteristic absorption bands corresponding to C—O—C (1190–1120) and C—N—C (1230, 1240). The ^{1}H NMR spectra of compounds 11 and 12 are shown in Table III.

Condensation of compounds 1 and 2 with hydrazine hydrate in boiling xylene afforded 2,3-dihydro-*s*-triazolo(3,4-*b*)-benzoxazole 13 and benzothiazole 14.

The IR spectra (KBr, cm^{-1}) of compounds 13 and 14 showed the absence of absorption bands corresponding to C—Cl; while exhibiting absorption bands of –NH (3350, 3300), C—H (3030, 2995) and C=N (1630, 1610). The ^{1}H NMR spectrum (DMSO, δ) of compound 13 showed signals at: 6.9–6.3 (m, 5H, aromatic

TABLE III

^{1}H NMR ($CDCl_3$) of compounds 11 and 12

Comp.	^{1}H NMR
11a	7.70-6.70(m,9H,aromatic+NH);5.50-5.20(s,2H,N-CH_2-N)
11b	7.60-7.00(m,4H,aromatic);4.60(s,2H,N-CH_2-N), 3.80-3.50(m,4H,O(CH_2)$_2$),2.70-2.50(m,4H,N(CH_2)$_2$)
12a	8.70-7.90(m,9H,aromatic+NH);6.90(s,2H,N-CH_2-N).
12b	7.70-7.90(m,4H,aromatic);4.70(s,2H,N-CH_2-N),3.8-3.60(m,4H,O(CH_2)$_2$);2.90-2.60(m-4H,N(CH_2)$_2$).

+NH); 6.0–5.5 (br, 2H, N—CH_2—N.), while the ^{1}H NMR spectrum (DMSO, δ) of compound 14 showed signals at 7.5–6.5 (m, 5H, aromatic +NH), and 5.5 (s, 2H, N—CH_2—N). Compounds 1 and 2 reacted also with phenylhydrazine hydrochloride in the presence of triethylamine in boiling xylene to give 3(H)-2-phenyl-*s*-triazolo-(3,4-*b*)-benzoxazole 15 and benzothiazole 16, in moderate yields.

The IR spectra (KBr, cm^{-1}) of compounds 15 and 16 showed the absence of the absorption bands corresponding to C—Cl, while exhibiting the characteristic absorption bands corresponding to C—H (3010–2910), and C=N (1940, 1930).

The ^{1}H NMR spectrum ($CDCl_3$, δ) of compound 15 showed signals at 8.2–7.2 (m, 9H, aromatic), 6.5 (s, 2H, N—CH_2—N); while ^{1}H NMR spectrum (DMSO, δ) of compound 16 showed signals at: 8.1–7.1 (m, 9H, aromatic); 6.25 (s, 2H, N—CH_2—N).

The reaction of compounds 1 and 2 with hydroxylamine hydrochloride in presence of triethylamine in boiling xylene afforded 3(H)-1,2,4-oxadizolo(3,4-*b*)-benzoxazole 17 and benzothiazole 18. The IR spectra (KBr, cm^{-1}) of compounds 17 and 18 revealed the absence of absorption bands corresponding to C—Cl and C=S groups, while exhibiting absorption bands corresponding to C=N (1640, 1630) and C—O—C (1150, 1130). The ^{1}H NMR spectrum ($CDCl_3$, δ) of compound 17 showed signals at: 7.7–7.0 (m, 4H, aromatic); 5.35 (s, 2H, N—CH_2—O); while the ^{1}H NMR spectrum ($CDCl_3$, δ) of compound 18 showed signals at 8.1–7.2 (m, 4H, aromatic); 5.50 (s, 2H, N—CH_2—O).

EXPERIMENTAL

The IR spectra (cm^{-1}) was recorded on a Perkin Elmer 137 Spectrophotometer in KBr. ^{1}H NMR spectra were recorded at 60 MHz on a Varian A-60 Spectrometer. The chemical shifts are expressed in δ values (ppm). TMS was used as the internal reference. Elemental analyses were done by Microanalytical Laboratory, Cairo University, Giza, Egypt. All melting points are uncorrected. The time allowed for completion of the reactions and the purity of the prepared compounds were controlled by means of TLC.

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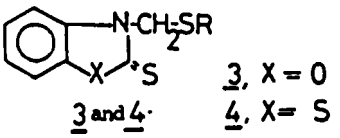
TABLE IV

Physical data of compounds 3 and 4



3 and 4. 3, X = O; 4, X = S

Comp.	R	m.p. °C	Yield %	Formula M.Wt	Analysis calc./found C%	H%	N%
3a	$-CH_2CH_2CH_3$	69-70	65	$C_{11}H_{13}NOS_2$ 239	55.23 55.03	5.44 5.81	5.85 5.75
3b	$-CH(CH_3)_2$	79-80	65	$C_{11}H_{13}NOS_2$ 239	55.23 55.22	5.44 5.49	5.85 5.50
3c		50	65	$C_{14}H_{17}NOS_2$ 247	60.21 60.00	6.09 6.10	5.01 4.90
3d		140	75	$C_{14}H_{10}ClNOS_2$ 307.5	54.63 54.40	3.25 3.00	4.55 4.20
3e		98-100	80	$C_{13}H_{10}N_2OS_2$ 274	56.93 56.84	3.64 3.20	10.21 9.90
3f		183-185	85	$C_{17}H_{14}N_4OS_2$ 354)	57.62 57.51	3.95 3.74	15.81 15.65
3g		145	81	$C_{22}H_{16}N_4OS_2$ 416	63.46 63.10	3.84 3.63	13.46 13.20
3h		120	80	$C_{15}H_{10}N_2OS_3$ 418	54.19 54.00	3.03 3.06	8.48 8.51
4a	$-CH_2CH_2CH_3$	75	60	$C_{11}H_{13}NS_3$ 255	51.76 51.49	5.09 5.00	6.49 6.20
4b	$-CH(CH_3)_2$	85	65	$C_{11}H_{13}NS_3$ 255	51.76 51.46	5.09 5.25	6.49 6.20
4c		60	60	$C_{14}H_{17}NS_3$ 295	56.94 56.76	5.76 5.44	4.74 4.30

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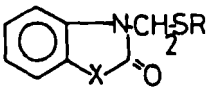
TABLE IV (*continued*)

Comp.	R	m.p. °C	Yield %	Formula M.Wt	Analysis calc./found C%	H%	N%
4d		151	60	$C_{14}H_{10}ClNS_3$ 323.5	51.93 51.79	3.09 2.90	4.32 4.00
4e		101	75	$C_{13}H_{10}N_2S_3$ 290	53.79 53.89	3.44 3.00	9.65 9.53
4f		178	75	$C_{17}H_{14}N_4S_2$ 370	55.13 55.00	3.78 3.40	15.13 15.00
4g		150	75	$C_{22}H_{16}N_4S_3$ 432	61.10 61.50	3.70 3.58	12.90 12.75
4h		128	69	$C_{15}H_{10}N_2S_4$ 334	52.20 52.00	2.89 2.70	8.09 8.00

TABLE V

Physical data of compounds 5 and 6



5 and 6 5, X = O ; 6, X = S

Comp. No.	R	m.p. °C	Yield %	Formula M.Wt	Analysis calc./found C%	H%	N%
5a	$CH_2CH_2CH_3$	58	50	$C_{11}H_{13}NO_2S$ 223	59.09 59.00	5.82 5.70	6.27 6.11
5b		153	60	$C_{17}H_{14}N_4O_2S$ 338	60.35 60.20	4.14 4.00	16.56 16.46
5c		175	55	$C_{22}H_{16}N_4O_2S$ 400	66.00 66.00	4.00 3.90	14.00 13.90
6		130	73	$C_{15}H_{10}N_2OS_3$ 330	54.45 54.63	3.03 3.01	8.48 8.70

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Synthesis of 3-chloromethyl-2-thiono-benzoxazole 1 and 3-chloromethyl-2-thiono-benzothiazole 2 were prepared according to known methods.

Reaction of compounds 1 *and* 2 *with thiols*: (General procedure). The appropriate thiol (0.01 mol) was added to an alcoholic solution of sodium ethoxide [Na, 0.23 g, 0.01 mol in 25 ml ethanol], then compound 1 and/or 2 (0.01 mol) was added in small portions over 10 min. The reaction mixture was refluxed for 3–4 hrs. The solvent was removed under reduced pressure giving the product. The precipitate was washed with cold water, filtered, and crystallized from ethanol to yield the corresponding sulfide 3 and 4 (cf. Table IV).

Reaction of sulfides 3a, g, h *and* 4h *with mercuric acetate*. Mercuric acetate (0.3 g, 0.001 mol) was added to a solution of the appropriate sulfide (0.01 mol) in methanol (50 ml). The reaction mixture was refluxed for 10 hrs, and was filtered off while hot, the solvent was removed under reduced pressure. The precipitated product was crystallized from methanol to give the corresponding oxo derivatives (cf. Table V).

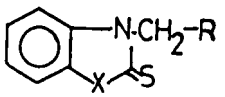
Reactions of compounds 1 *and* 2 *with sodium phenoxide*: (General procedure). To sodium ethoxide in ethanol [Na, 0.23 g, 0.01 mol in 10 ml ethenal], phenol (0.94 g; 0.01 mol) was added, the reaction mixture was heated at 50°C for 10 min, compound 1 and/or 2 (0.01 mol) was added. The mixture was refluxed with stirring for 6 hrs. After cooling to room temperature, the solid was filtered off, washed with water, dried and crystallized from ethanol to yield 3-phenoxymethyl-2-thiono-benzoxazole (7) and 3-phenoxymethyl-2-thiono-benzothiazole (8).

Compound 7 m.p. 80–83°C, yield 45%
Analysis:
Found C, 65.25; H, 4.61; N, 5.35
$C_{14}H_{11}NO_2S$ requires C, 65.36; H, 4.80; N, 5.44

Compound 8: m.p. 79°C, yield 40%
Analysis:
Found C, 63.30; H, 4.09; N, 5.00
$C_{14}H_{11}NOS_2$ requires C, 63.80; H, 4.18; N, 5.32

TABLE VI

Physical data of compounds 11 and 12



11 and 12 11, X=O; 12, X=S

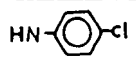
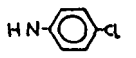
Comp.	R	m.p. °C	Yield %	Formula M.Wt	Analysis calc./found C%	H%	N%
11a		112	75	$C_{14}H_{11}ClN_2OS$ 290.5	57.83 / 57.63	3.78 / 3.50	9.63 / 9.30
11b		80	80	$C_{12}H_{12}N_2O_2S$ 250	57.60 / 57.20	5.60 / 5.20	11.2 / 11.0
12a		120	70	$C_{14}H_{11}ClN_2S_2$ 306.5	54.80 / 54.58	5.58 / 5.48	9.13 / 9.00
12b		86	65	$C_{12}H_{14}N_2OS_2$ 266	54.13 / 54.00	5.20 / 5.20	10.52 / 10.30

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TABLE VII

Physical data of compounds 13–18

Comp.	m.p. °C	yield %	formula M.Wt.	analysis C %	Calc. found % H%	N %
13	160	70	$C_8H_7N_3O$	59.62	4.34	26.08
			161	59.66	4.31	26.50
14	146	75	$C_8H_7N_3S$	54.23	3.95	23.72
			177	54.00	3.66	23.70
15	143	50	$C_{14}H_{11}N_3O$	70.88	4.64	17.72
			237	70.77	4.64	17.78
16	152	52	$C_{14}H_{11}N_3S$	66.40	4.34	16.60
			253	66.29	4.30	16.58
17	150	50	$C_8H_6N_2O_2$	58.53	3.65	17.07
			162	58.49	3.56	17.00
18	161	45	$C_8H_6N_2OS$	53.93	3.37	15.73
			178	53.90	3.30	15.71

Reaction of compounds 7 *and* 8 *with mercuric acetate*: (General procedure). Mercuric acetate (0.3 g, 0.001 mol) was added to a solution of 7 and/or 8 (0.001 mol) in methanol (50 mol). The reaction mixture was refluxed for 10 hrs, and was filtered off while hot, the solvent was removed under reduced pressure. The precipitated product was crystallized from methanol to give oxo derivative 9 and 10.

Compound 9: m.p. 55°C, yield 50%
Analysis:
Found C, 69.60; H, 4.50; N, 5.75
$C_{14}H_{11}NO_3$ Calcd. C, 69.70; H, 4.56; N, 5.80

Compound 10: m.p. 59°C, yield 50%
Analysis:
Found C, 65.30; H, 4.05; N, 5.33
$C_{14}H_{11}NO_2S$ Calcd. C, 65.36; H, 4.28; N, 5.44

Reaction of compounds 1 *and* 2 *with amines*: (General procedure). To a solution of 1 and/or 2 (0.001 mol) in benzene (100 ml), triethylamine (0.1 ml, 0.001 mol), *p*-chloroaniline and/or morpholine (0.001 mol) were added. The reaction mixture was refluxed for 3–4 hrs, cooled to room temperature, the precipitate was filtered off, washed with water, and was crystallized from ethanol to give products 11 and 12 (Table VI).

Reaction of compounds 1 *and* 2 *with hydrazine, phenyl hydrazine, and/or hydroxylamine*: (General procedure). To solution of 1 and/or 2 (0.01 mol) in benzene (50 ml), triethylamine (1.01 ml, 0.01 mol), hydrazine, phenylhydrazine and/or hydroxylamine (0.01 mol) were added. The reaction mixture was stirred at room temperature for 2–3 hrs. The solvent was removed under reduced pressure, xylene (20 ml) was added to the crude product, the reaction mixture was refluxed until the hydrogen sulfide decreased (≈20 hrs). After cooling, the product was filtered off, washed with water and crystallized from benzene/pet. ether (40–60) mixture to yield product (13–18) (cf. Table VII).

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