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SULFENYL HALIDES IN THE SYNTHESIS OF HETEROCYCLES. 2*. CYCLIZATION IN REACTIONS OF HETARENESULFENYL CHLORIDES WITH 3,3-DIMETHYL-1-BUTENE

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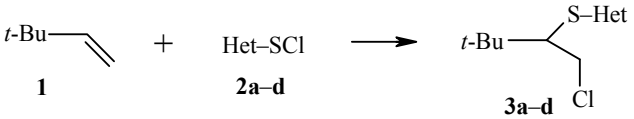
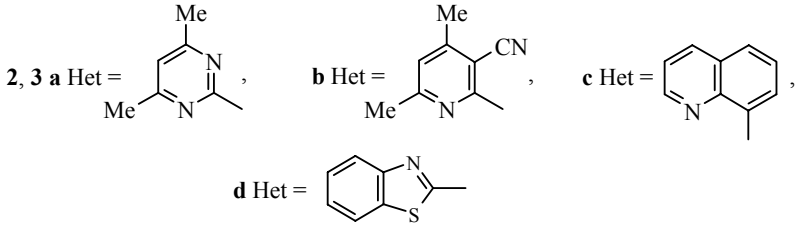
Products of additive cyclization with ring closure at the nitrogen atom of the thiohetaryl unit were synthesized by the interaction of 4,6-dimethylpyrimidine-2-, 3-cyano-4,6-dimethylpyridine-2-, quinoline-8-, and 1,3-benzothiazole-2-sulfenyl chlorides with 3,3-dimethyl-1-butene.

Keywords: alkenes, sulfenyl halides, heterocyclization.

We have formulated and developed a route for the synthesis of sulfur-containing heterocycles based on the interaction of sulfenyl chlorides with unsaturated compounds which occurs by ring closure at the nucleophilic center of the sulfenyl unit [1-6].

In the current work reactions of 3,3-dimethyl-1-butene (**1**) with the hetarenesulfenyl chlorides **2a-d** which contain a potentially nucleophilic nitrogen atom in the hetaryl unit: 4,6-dimethylpyrimidine-2- (**2a**), 3-cyano-4,6-dimethylpyridine-2- (**2b**), quinoline-8- (**2c**), and 1,3-benzothiazole-2-sulfenyl chloride (**2d**).





With the help of ^{1}H NMR spectroscopy it was established that addition products at the double bond contrary to Markovnikov's rule – the corresponding β-chlorosulfides **3a-d** – were obtained rapidly and with quantitative yield from compounds **2a-d** and the alkene **1** at 20°C.

* For part 1, see [1].

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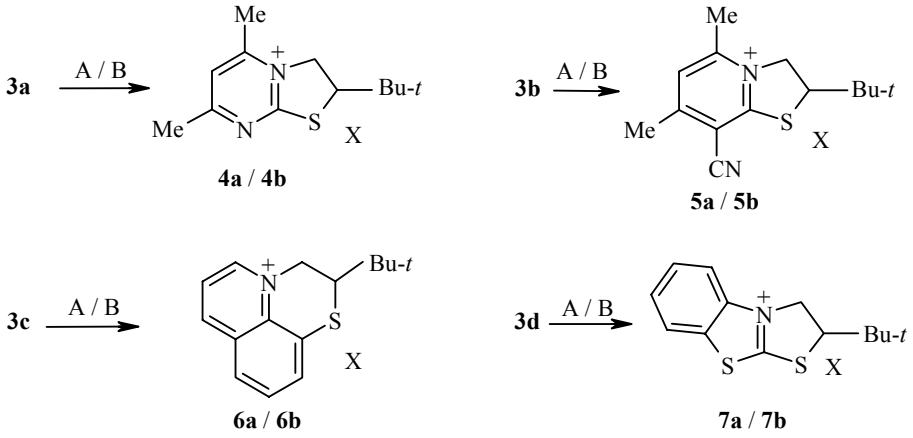
Subsequently compounds **3a-d** were slowly (3-150 days) converted under the same conditions (method A) into the condensed heterocyclic compounds **4a-7a**, the products of intramolecular cyclization with replacement of the chloride anion by the nitrogen atom of the sulfenyl unit (the σ-route of heterocyclization [7]). It was shown with the compound **3d** as example that this process accelerated under higher temperature (Table 1). The β-chloro sulfides **3a-d** underwent intramolecular cyclization at 20°C considerably more rapidly in nitromethane in the presence of lithium perchlorate (method B). The corresponding perchlorates **4b-7b** were formed in this way.



A – in $CHCl_3$, B – in $LiClO_4 – MeNO_2$; **4-7 a** X = Cl^-, **b** X = ClO_4^-

Compounds **4b-6b** were formed directly in an Ad_E process by the reaction of the sulfenyl chlorides **2a-c** with the alkene **1** in nitromethane in the presence of lithium perchlorate at 20°C (the π-route to heterocyclization [7]): from **2a** compound **4b** (83% yield), from **2b** compound **5b** (92% yield), and from **2c** compound **6b** (91% yield).

$$\mathbf{1} + \mathbf{2a–c} \xrightarrow[-LiCl]{LiClO_4 – MeNO_2} \mathbf{4b – 6b}$$

In the case of the sulfenyl chloride **2d** a considerable amount (43%) of the product of tandem rearrangement-cyclization **8** was produced along with compound **7b**.

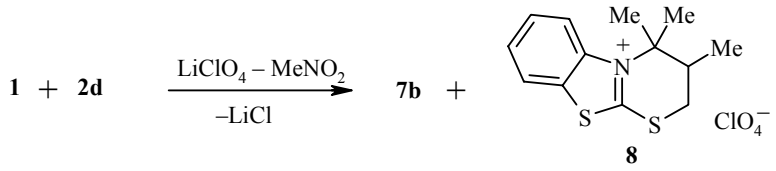
TABLE 1. Conditions and Results of the Conversion of the β-Chloro Sulfides **3a-d** into the Salts **4a,b-7a,b**

Compound	Medium	T, °C	Conversion time, d	Degree of conversion, %	Reaction product	Yield, %
3a	$CHCl_3$	20	20	100	**4a**	87
	$LiClO_4$–$MeNO_2$	20	1	100	**4b**	84
3b	$CHCl_3$	20	3	100	**5a**	97
	$LiClO_4$–$MeNO_2$	20	1	100	**5b**	98
3c	$CHCl_3$	20	21	100	**6a**	96
	$LiClO_4$–$MeNO_2$	20	1	100	**6b**	92
3d	$CHCl_3$	20	150	30	**7a**	22
	$CHCl_3$	61	5	50	**7a**	43
	$LiClO_4$–$MeNO_2$	20	6	25	**7b**	19

TABLE 2. Characteristics of the Compounds Synthesized

Compound	Name	Empirical formula	Found, % / Calculated, %				mp, °C
			C	H	N	S	
3a	4,6-Dimethylpyrimidin-2-yl 1-chloro-3,3-dimethylbutyl-2 sulfide	$C_{12}H_{19}ClN_2S$	55.48 / 55.69	7.27 / 7.40	10.71 / 10.82	12.28 / 12.39	—*
3b	4,6-Dimethyl-3-cyanopyridyl-2 1-chloro-3,3-dimethylbutyl-2 sulfide	$C_{14}H_{19}ClN_2S$	59.20 / 59.45	6.62 / 6.77	9.69 / 9.90	11.25 / 11.34	—*
3c	Quinolyl-8 1-chloro-3,3-dimethylbutyl-2 sulfide	$C_{15}H_{18}ClNS$	64.12 / 64.38	6.35 / 6.48	4.88 / 5.01	11.37 / 11.46	—*
3d	Benzothiazol-2-yl 1-chloro-3,3-dimethylbutyl-2 sulfide	$C_{13}H_{16}ClNS_2$	54.43 / 54.62	5.52 / 5.64	4.77 / 4.90	22.28 / 22.43	134-135
4a	2-(*tert* -Butyl)-5,7-dimethyl-2,3-dihydro-thiazolo[3,2-*a*]pyrimidinium-4 chloride	$C_{12}H_{19}ClN_2S$	55.51 / 55.69	7.31 / 7.40	10.69 / 10.82	12.23 / 12.39	155 (dec.)
5a	2-(*tert* -Butyl)-8-cyano-5,7-dimethyl-2,3-dihydrothiazolo-[3,2-*a*]pyridinium-4 chloride	$C_{14}H_{19}ClN_2S$	59.23 / 59.45	6.68 / 6.77	9.75 / 9.90	11.21 / 11.34	205-207
6a	2-(*tert* -Butyl)-2,3-dihydro[1,4]thiazino[2,3,4-*ij*]quinolinium-4 chloride	$C_{15}H_{18}ClNS$	64.19 / 64.38	6.42 / 6.48	4.93 / 5.01	11.32 / 11.46	173-175
7a	2-(*tert* -Butyl)-2,3-dihydrobenzo[*d*]thiazolo[2,3-*b*][1,3]thiazolium-4 chloride	$C_{13}H_{16}ClNS_2$	54.51 / 54.62	5.57 / 5.64	4.80 / 4.90	22.29 / 22.43	204-206
4b	2-(*tert* -Butyl)-2,3-dihydro-5,7-dimethylthiazolo[3,2-*a*]-pyrimidinium-4 perchlorate	$C_{12}H_{19}ClN_2O_4S$	44.54 / 44.65	5.87 / 5.93	8.54 / 8.68	9.98 / 9.93	228-230
5b	2-(*tert* -Butyl)-8-cyano-5,7-dimethyl-2,3-dihydrothiazolo[3,2-*a*]-pyridinium-4 perchlorate	$C_{14}H_{19}ClN_2O_4S$	48.29 / 48.48	5.45 / 5.52	7.97 / 8.08	9.12 / 9.24	198-200
6b	2-(*tert* -Butyl)-2,3-dihydro[1,4]thiazino[2,3,4-*ij*]quinolinium-4 perchlorate	$C_{15}H_{18}ClNO_4S$	52.28 / 52.40	5.22 / 5.28	4.01 / 4.07	9.21 / 9.32	160 (dec.)
7b	2-(*tert* -Butyl)-2,3-dihydrobenzo[*d*]thiazolo[2,3-*b*][1,3]thiazolium-4 perchlorate	$C_{13}H_{16}ClNO_4S_2$	44.52 / 44.63	4.57 / 4.61	3.92 / 4.00	18.19 / 18.33	230-232
8	3,4,4-Trimethyl-3,4-dihydro-2H-benzo[4,5]thiazolo[2,3-*b*][1,3]-thiazolium-5 perchlorate	$C_{13}H_{16}ClNO_4S_2$	44.48 / 44.63	4.55 / 4.61	3.89 / 4.00	18.27 / 18.33	222-224

* Oil.



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The composition and structure of the compounds synthesized were confirmed by elemental analysis (Table 2), ^{1}H NMR (Table 3), ^{13}C NMR (Table 4) and IR spectroscopy, and in the case of compound **5a** by X-ray crystallography.

TABLE 3. ^{1}H NMR Spectra of Compounds **3a-d**, **4a-7a***, and **8**

Compound	Chemical shifts, δ, ppm (SSCC, *J*, Hz)*[2]				
	CH_3 in *t*-Bu (9H, s)	CH_3 in the aromatic unit	CH_2 (1H, dd and 1H, dd)	CH	H_{arom}
3a	1.08	2.39 (6H, 4,6-CH_3)	3.97 and 3.68 (^2J = 11.8, $^3J_1$ = 3.9*[3], $^3J_2$ = 8.5)	4.23 (dd)	6.72 (1H, s, H-5)
3b	1.07	2.35 (3H), 2.45 (3H)	3.96 and 3.71 (^2J = 11.8, $^3J_1$ = 4.5, $^3J_2$ = 7.1)	4.42 (dd)	6.78 (1H, s, H-5)
3c	1.10	—	3.97 and 3.58 (^2J = 11.5, $^3J_1$ = 2.4, $^3J_2$ = 9.3)	3.87 (dd)	9.14 (1H, d, *J* = 2.4, H-2), 8.55 (1H, d, *J* = 7.6, H-4), 8.32 (1H, d, *J* = 2.4, H-5), 8.19 (1H, d, *J* = 8.3, H-7), 7.81 (2H, m , H-3,6)
3d	1.12	—	4.08 and 3. 81 (^2J = 11.8, $^3J_1$ = 4.4, $^3J_2$ = 7.0)	4.32 (dd)	7.84 (1H, d, *J* = 8.1, H-4), 7.71 (1H, d, *J* = 7.9, H-7), 7.38 (1H, t, J_1 = 7.4, J_2 = 8.1, H-5), 7.26 (1H, t, J_1 = 7.4, J_2 = 7.9, H-6)
4a	1.05	2.75 (3H), 2.60 (3H)	5.09 and 4.82 (^2J = 13.8, $^3J_1$ = 9.2, $^3J_2$ = 8.5)	4.48 (dd)	7.68 (1H, s, H-6)
5a	1.06	2.80 (3H), 2.63 (3H)	5.18 and 4.92 (^2J = 13.8, $^3J_1$ = 9.2, $^3J_2$ = 8.5)	4.61 (dd)	7.73 (1H, s, H-6)
6a	1.17	—	5.61 and 4.92 (^2J = 13.8, $^3J_1$ = $^3J_2$ = 11.2)	3.86 (d)	9.66 (1H, d, *J* = 5.9, H-5), 9.29 (1H, d, *J* = 7.9, H-7), 8.18 (3H, m, H-8,9,10), 7. 90 (1H, t, J_1 = 7.9, J_2 = 5.9, H-6)
7a	1.16	—	5.77 and 4.81 (^2J = 11.8, $^3J_1$ = 9.8, $^3J_2$ = 8.7)	5.22 (t)	8.10 (1H, d, *J* = 8.1, H-5), 8.04 (1H, d, *J* = 8.1, H-8), 7.65 (1H, t, J_1 = 7.5, J_2 = 8.1, H-7), 7.44 (1H, t, J_1 = 7.5, J_2 = 8.1, H-6)
8	—*[4]	—	3.59 and 3.42 (^2J = 13.7, $^3J_1$ = 2.5, $^3J_2$ = 7.8)	2.69 (m)	8.43 (1H, d, *J* = 8.0, H-6), 8.21 (1H, d, *J* = 8.0, H-9), 7.72 (2H, m, H-7,8)

* Date for the spectra of the salts **4a-7a** and the corresponding perchlorates **4b-7b** are identical.

*[2] The ^{1}H NMR spectra were taken in $CDCl_3$ (compounds **3a-d**, **7a**) and DMSO-d_6 (compounds **4a-6a**, **8**).

*[3] Here and below $^3J_1$ refers to the low field signal.

*[4] For compound **8** these signals were at 1.91 (6H, s, 2CH_3-4) and 1.22 ppm (3H, d, *J* = 7.2 Hz, 3-CH_3).

TABLE 4. ^{13}C NMR Spectra of Compounds **3d**, **4a-7a***

Compound	Chemical shifts, δ, ppm*[2]					
	CH_3 in the aromatic unit	CH_3 in *t*-Bu	$\underline{C}(CH_3)_3$	CH_2	CH	C_{arom}
3d	–	27.92	35.70	45.68	61.71	120.74, 121.29, 124.19, 125.82, 133.48, 152.55, 166.75
4a	24.33, 20.30	26.36	33.57	55.69	54.60	118.64, 162.20, 168.71, 175.19
5a	21.17, 20.74	26.52	33.71	59.53	58.18	105.04, 113.01, 125.08, 157.70, 160.52, 161.67
6a	—	26.89	33.35	47.12	59.49	122.20, 126.60, 127.29, 129.22, 130.44, 132.45, 133.00, 148.37, 150.29
7a	—	26.86	34.47	53.37	67.97	115.92, 122.55, 124.48, 128.97, 135.07, 137.18, 180.43

* The spectra of compounds **4a-7a** and **4b-7b** were identical.
* ^{13}C NMR spectra were taken in $CDCl_3$ (compounds **3d** and **7a**) and in DMSO-d_6 (compounds **4a-6a**).

In the IR spectra of the β-chloro sulfides **3a-d** there are absorption bands ascribable to the vibrations of the hetaryl ring, ν, cm^{-1}: 1652, 1583, 1537, 1438, 1340, 1302, 1257 (**3a**); 1602, 1565, 1460, 1380, 1286 (**3b**); 1605, 1590, 1565, 1488, 1455, 1380, 1357, 1305, 987, 827 (**3c**); 1448, 1410, 1282, 1020, 732 (**3d**). The IR spectra of the condensed systems **4a-7a** and **8** contain absorption bands ascribable to vibrations of the aromatic rings, ν, cm^{-1}: 1634, 1537, 1463, 1375, 1282 (**4a**); 1642, 1575, 1480, 1390, 1255 (**5a**); 1655, 1577, 1552, 1382, 1300, 842 (**6a**); 1645, 1465, 1388, 1255, 775 (**7a** and **8**). The IR spectra of the salts **4b-7b** and **8** also contain broad intense band in the 1100 cm^{-1} corresponding to Cl–O vibrations.

Fig. 1 shows a spatial model of salt **5a** (Tables 5-7). The shortest intermolecular contact $Cl\cdots N_{(1)}$ is 3.240 Å.

Thus σ- and π-routes to ring formation have been revealed in the reaction of hetarenesulfenyl chlorides **2a-d** with alkene **1** in which the nitrogen atom of the thiohetaryl unit takes part.

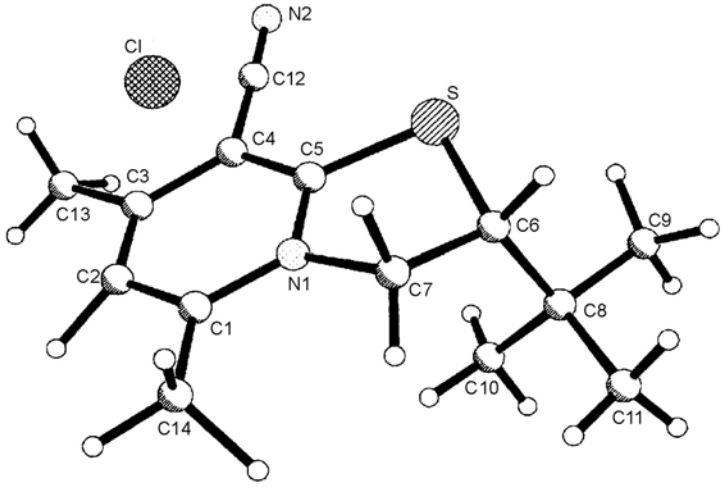

Fig. 1. Spatial model of compound **5a**.

TABLE 5. Bond Lengths (*d*) in Compound **5a**

Bond	*d*, Å	Bond	*d*, Å
S–$C_{(5)}$	1.729(4)	$C_{(3)}$–$C_{(4)}$	1.397(6)
S–$C_{(6)}$	1.843(4)	$C_{(3)}$–$C_{(13)}$	1.502(7)
$N_{(1)}$–$C_{(5)}$	1.342(5)	$C_{(4)}$–$C_{(5)}$	1.396(5)
$N_{(1)}$–$C_{(1)}$	1.360(5)	$C_{(4)}$–$C_{(12)}$	1.434(6)
$N_{(1)}$–$C_{(7)}$	1.486(5)	$C_{(6)}$–$C_{(7)}$	1.526(6)
$N_{(2)}$–$C_{(12)}$	1.135(5)	$C_{(6)}$–$C_{(8)}$	1.545(6)
$C_{(1)}$–$C_{(2)}$	1.378(6)	$C_{(8)}$–$C_{(10)}$	1.514(7)
$C_{(1)}$–$C_{(14)}$	1.478(7)	$C_{(8)}$–$C_{(9)}$	1.524(7)
$C_{(2)}$–$C_{(3)}$	1.381(6)	$C_{(8)}$–$C_{(11)}$	1.532(7)

TABLE 6. Bond angles (ω) in Compound **5a**

Angle	ω, deg.	Angle	ω, deg.
$C_{(5)}$–S–$C_{(6)}$	91.2(2)	$N_{(1)}$–$C_{(5)}$–S	114.2(3)
$C_{(5)}$–$N_{(1)}$–$C_{(1)}$	122.2(3)	$C_{(4)}$–$C_{(5)}$–S	125.7(3)
$C_{(5)}$–$N_{(1)}$–$C_{(7)}$	113.6(3)	$C_{(7)}$–$C_{(6)}$–$C_{(8)}$	116.7(3)
$C_{(1)}$–$N_{(1)}$–$C_{(7)}$	123.7(3)	$C_{(7)}$–$C_{(6)}$–S	103.6(3)
$N_{(1)}$–$C_{(1)}$–$C_{(2)}$	117.9(4)	$C_{(8)}$–$C_{(6)}$–S	113.3(3)
$N_{(1)}$–$C_{(1)}$–$C_{(14)}$	118.3(4)	$N_{(1)}$–$C_{(7)}$–$C_{(6)}$	107.5(3)
$C_{(2)}$–$C_{(1)}$–$C_{(14)}$	123.8(4)	$C_{(10)}$–$C_{(8)}$–$C_{(9)}$	110.1(5)
$C_{(1)}$–$C_{(2)}$–$C_{(3)}$	122.3(4)	$C_{(10)}$–$C_{(8)}$–$C_{(11)}$	110.3(5)
$C_{(2)}$–$C_{(3)}$–$C_{(4)}$	117.8(4)	$C_{(9)}$–$C_{(8)}$–$C_{(11)}$	107.8(4)
$C_{(2)}$–$C_{(3)}$–$C_{(13)}$	121.5(5)	$C_{(10)}$–$C_{(8)}$–$C_{(6)}$	112.3(4)
$C_{(5)}$–$C_{(4)}$–$C_{(3)}$	119.3(4)	$C_{(9)}$–$C_{(8)}$–$C_{(6)}$	108.6(4)
$C_{(5)}$–$C_{(4)}$–$C_{(12)}$	118.1(4)	$C_{(11)}$–$C_{(8)}$–$C_{(6)}$	107.5(4)
$C_{(3)}$–$C_{(4)}$–$C_{(12)}$	122.6(4)	$N_{(2)}$–$C_{(12)}$–$C_{(4)}$	177.9(4)
$N_{(1)}$–$C_{(5)}$–$C_{(4)}$	120.1(3)	$C_{(4)}$–$C_{(3)}$–$C_{(13)}$	120.7(5)

TABLE 7. Coordinates of Non-hydrogen Atoms (*x, y, z*) and Equivalent Isothermal Motion Parameters (U_{eq}) of Compound **5a**

Atom	$x \times 10^4$	$y \times 10^4$	$z \times 10^4$	$U_{eq} \times 10^3$, Å^2
Cl	6379(1)	4394(1)	5000	51(1)
S	4923(1)	7745(1)	4899(2)	49(1)
$N_{(1)}$	5753(3)	6570(2)	6697(3)	38(1)
$N_{(2)}$	7711(4)	8854(4)	3793(4)	66(1)
$C_{(1)}$	6537(3)	6064(3)	7438(4)	42(1)
$C_{(2)}$	7666(4)	6371(3)	7303(4)	49(1)
$C_{(3)}$	8024(3)	7126(4)	6413(4)	47(1)
$C_{(4)}$	7203(3)	7554(3)	5605(4)	42(1)
$C_{(5)}$	6055(3)	7272(3)	5782(4)	40(1)
$C_{(6)}$	3886(3)	7247(3)	6052(4)	42(1)
$C_{(7)}$	4515(3)	6285(4)	6705(4)	42(1)
$C_{(8)}$	3412(3)	8214(4)	6866(4)	46(1)
$C_{(9)}$	2890(6)	9122(5)	6042(5)	69(2)
$C_{(10)}$	4223(5)	8735(6)	7675(7)	66(1)
$C_{(11)}$	2451(5	7711(5)	7651(5)	61(1)
$C_{(12)}$	7485(4)	8295(4)	4607(4)	49(1)
$C_{(13)}$	9253(5)	7482(6)	6307(7)	67(2)
$C_{(14)}$	6133(4)	5205(4)	8328(5)	58(1)

EXPERIMENTAL

IR spectra of KBr disks or liquid films were recorded with a Specord M-80 machine. ^{1}H NMR spectra were recorded with a Bruker WM-250 (250 MHz) machine and ^{13}C NMR spectra with a Bruker AM-300 (75 MHz) machine.

X-ray Crystallographic Analysis of Compound 5a. Crystals of compound **5a**, grown from methylene chloride, are orthorhombic at 293 K with a = 11.698(2), b = 11.748(2), c = 10.914(4) Å; $\alpha = \beta = \gamma = 90°$, V = 1449(5) Å^3, d_{calc} = 1.252 g/cm^3, space group $Pca2_1$, Z = 4, $F(000)$ = 600. Analysis was carried out with an Enraf-Nonius CAD-4 automatic diffractometer (Mo$K\alpha$ radiation, $\theta/2\theta$ scanning, $2\theta_{max} = 50°$). The structure was solved by direct methods using the SHELXTL programme. In the calculations 896 reflexions with $I > 2\sigma(I)$. Refinement was carried out by full matrix least squares. The final divergense factor was R = 0.023.

Hetaryl 1-Chloro-3,3-dimethylbutyl-2 Sulfides 3a-d. A solution of alkene **1** (10 mmol) in chloroform (10 ml) was added with stirring to a solution of the sulfenyl chloride **2** (10 mmol) in chloroform (20 ml) at 20°C. After 15 min the solvent was removed in vacuum and products **3a-d** were obtained in quantitative yield. The β-chloro sulfide **3d** was recrystallized from a mixture of hexane and methylene chloride.

Intramolecular Heterocyclization of the β-Chloro Sulfides 3a-d. A. Synthesis of Salts **4a-7a**. A solution of a chloro sulfide **3** (10 mmol) in chloroform (60 ml) was kept at room temperature and monitored every 12-24 h (the ^{1}H NMR spectrum was taken of the residue after evaporation of 0.5 ml of solution). The solution, which contained only the products **4a-6a** or compounds **3d** and **7a**, was evaporated and the salts **4a-7a** were recrystallized from a mixture of hexane and methylene chloride.

B. Synthesis of Salts **4b-7b.** Cyclization of compounds **3a-d** was carried analogously to method A, but nitromethane (60 ml) containing lithium perchlorate (30 mmol) was used in place of chloroform. Methylene chloride (100 ml) was added to the residue after evaporation of the solvent and LiCl and $LiClO_4$ were filtered off. The filtrate was evaporated and salts **4b-7b** were isolated by fractional recrystallization from methylene chloride.

Cycloaddition of the Hetarenesulfenyl Chlorides 2a-d to the Alkene 1. Solutions of lithium perchlorate (10 mmol) in nitromethane (40 ml) and alkene **1** (10 mmol) in nitromethane (10 ml) were added with stirring to a solution of a sulfenyl chloride **2** (10 mmol) in nitromethane (10 ml). After 15 min methylene chloride (100 ml) was added to the reaction mixture, the precipitate of LiCl and $LiClO_4$ was filtered off and washed with methylene chloride on the filter. The filtrate was evaporated and the salts **4b-7b** and **8** were isolated from the residue by fractional crystallization from methylene chloride.

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