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Ring-opening Reactions of Heterocyclic Organometallics. VII.* The Regio- and Stereospecific Synthesis of Alkylselenovinyl Acetylenes

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Ring-opening of 3-selenienyllithium derivatives (*2*) with nonidentical substituents in the α-positions leads to enyneselenolates (*3*), which are alkylated by ethyl bromide to give ethylselenovinyl acetylenes (*4*) with *Z*-configuration and with the ethylseleno function attached to either of the terminal carbons of the vinylacetylenic moiety.

The 3-selenienyllithium derivatives (*2*) were prepared by halogen-metal exchange between the corresponding 3-bromoselenophenes (*1*) and ethyllithium. The synthesis of the starting materials is described.

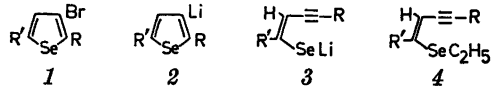
RING-OPENING REACTIONS

We have previously described the synthetic possibilities of the ring-opening reaction of some 3-thienyllithium derivatives with nonidentical substituents in the α-positions in order to prepare alkylthiovinyl acetylenes with controlled stereochemistry (*Z*-configuration), and with the alkylthio function attached to either of the terminal carbon atoms of the vinylacetylenic moiety.[1]

Since 3-selenienyllithium derivatives show a tendency to ring-open much faster than the corresponding 3-thienyllithium derivatives,[2-4] it could be expected that 3-selenienyllithium derivatives with non-identical substituents in the 2- and 5-positions would give alkylselenovinyl acetylenes in analogy with the 3-thienyllithium derivatives.

It should be mentioned that ethanethiolate adds to 2,4-heptadiyne in a non-specific manner to give a mixture of 2-ethylthio-2-hepten-4-yne and 3-ethylthio-3-hepten-5-yne,[1] both probably with *Z*-configuration (*cf.* Ref. 5). The use of ethaneselenolate instead of ethanethiolate would probably not give a significantly more specific reaction. Thus, the ring-openings of the lithium derivatives *2b* and *2a* could be expected to provide convenient routes to *4b* and *4a*, respectively.

Therefore *1b* and *1a*, the synthesis of which will be described below, were treated with ethyllithium followed by ethyl bromide in ether for 4 h at room temperature. After hydrolysis of the reaction mixture, *4b* and *4a* were isolated in 54 and 60 % yield, respectively. The crude products contained only minor amounts of by-products, which makes the reaction of synthetic value. However, upon the isolation of the compounds (distillation) much decomposition took place, explaining the relatively moderate yields.



1 *2* *3* *4*

Scheme 1. a, R′= C_2H_5, R=CH_3; b, R′= CH_3, R=C_2H_5.

The reaction path by which these compounds are formed is most likely initiated by a halogen-metal exchange between *1a* and *1b* with ethyllithium to give *2a* and *2b*, respectively. Then, *2a* and *2b* ring-open as described previously [1]

* Part VI, see Ref. 1.
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to form the enyne selenolates *3a* and *3b*, which eventually react with ethyl bromide at selenium (S_N2 reaction) to give *4a* and *4b*.

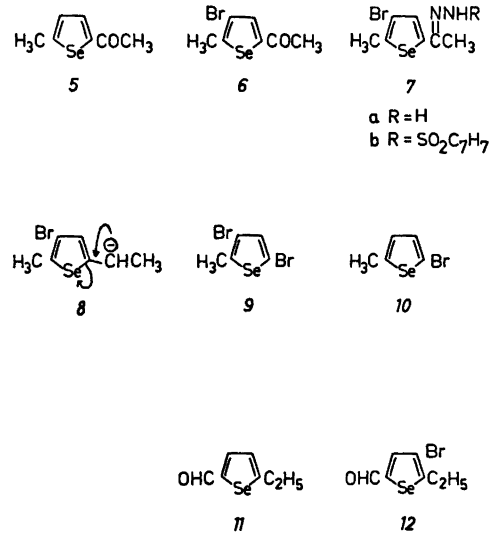
The structures of these compounds were deduced from IR, NMR and mass spectra as well as from elemental analyses (see experimental part). In the mass spectra of *4a* and *4b* the fragment at $m/e = 93$ was quite abundant (32 and 66 %, respectively).

Since the coupling constant between vinylic protons and the protons of the vinylic methyl or methylene groups (the allylic coupling constant) is of the same magnitude for both *cis* and *trans* arrangements,[6] it could not be determined whether *4a* and *4b* had the *Z*- or *E*-configuration with the aid of this tool. However, it has been shown by us that the products originating from the ring-opening of 3-thienyl- and 3-selenienyllithium had the *Z*-configuration.[4] Independently, Jakobsen showed that the ring-opening of 2-methyl-3-thienyllithium also gave a thioenyne with *Z*-configuration.[7] Thus it seems quite likely that the ring-opening takes place in a *trans* manner leading to thio- and selenoenynes with the heteroatom and the triple bond in a *cis* arrangement.

SYNTHESIS OF THE STARTING MATERIALS

The synthesis of *1b* was performed as follows. First, 2-ethylselenophene [18] was treated with butyllithium followed by dimethylformamide at −70 °C to give *11*. Bromination of *11* with the "swamping catalyst" method [9] (bromine in aluminium trichloride) gave *12*, which was subsequently reduced with the Wolff-Kishner method to give *1b*. This synthetic route is essentially analogous to that used in the thiophene series for the preparation of 3-bromo-2-ethyl-5-methylthiophene,[10] except that the formyl group was introduced by Vilsmeier formylation in the latter case.

Then it seemed natural to apply an analogous route to obtain *1a* as well. By the "swamping catalyst" method, *5* [11] was converted to *6* without difficulty. However, when the modified Wolff-Kishner reduction [8] was attempted on *6*, no reduction took place. A yellow precipitate was formed when hydrazine was heated together with *6* in ethylene glycol. The subsequent base treatment did not lead to nitrogen evolution, even at 160 − 180 °C. At this temperature red elemental selenium started to form, and therefore the experiment was stopped and a small amount of the yellow crystals was isolated. The most heavy fragment was around $m/e =$ 280, which indicated the presence of the hydrazone, *7a*. Despite several recrystallisations, the substance was not obtained pure as judged from its broad melting interval. Lithium aluminium hydride reduction of the tosyl hydrazone *7b* was also unsuccessful. Red selenium precipitated when the excess of lithium aluminium hydride was destroyed with moist ether. Since base treatment of hydrazones and tosyl hydrazones may form carbanions, it is suggested that an anion such as *8* is involved. We have shown in previous work that ring-opening from the side-chain is possible in the thiophene series [12] and *8* may also undergo ring-opening as indicated in the figure. It should be pointed out, however, that 2-acetylselenophene and *12* were successfully converted to 2-ethylselenephene and *1b*, respectively, with the Wolff-Kishner procedure as mentioned. Further work is necessary in order to reveal the causes of these results.



Consequently, another approach had to be used for the synthesis of *1a*. Starting from 2,5-dibromoselenophene, *10* was prepared in 67 % yield by halogen-metal exchange with butyllithium followed by dimethyl sulfate. Bromina-

tion of *10* with bromine in acetic acid gave a 53 % yield of *9*. This route was used as it was found that the reaction of 2-methylselenophene with two equivalents of bromine in acetic acid led to extensive tar formation. The decomposition was almost instantaneous when only a small amount of bromine had been added. The corresponding reaction with 2-methylthiophene gives 3,5-dibromo-2-methylthiophene in good yield.[13] It was also found that 2-methylselenophene could be brominated at room temperature in 55 % yield, when two equivalents of *N*-bromosuccinimide in acetic acid were used.[14] These experiments indicate that it was 2-methylselenophene or some intermediate that decomposed in the presence of strong acids (*e.g.* HBr) and thus illustrate the greater acid sensitivity of selenophenes compared to thiophenes.

When 2-methylselenophene was treated with bromine in carbon disulfide at −20 °C, a brown oil was obtained, which darkened rapidly. An exothermic reaction took place when potassium hydroxide pellets were added and a 41 % yield of *9* could be isolated after distillation (decomposition of 9 took place to a great extent during all attempts at distillation). Thus it seems probable that an addition product was initially formed between 2-methylselenophene and bromine and that the elimination of hydrogen bromide started the decomposition in the absence of base.

Halogen-metal exchange between *9* and butyllithium followed by reaction with diethyl sulfate gave the desired compound *1a*. Unfortunately it could not be obtained pure by distillation or by TLC. The diethyl sulfate, left from the reaction, showed an amazing tendency to adhere to *1a*. However, the preparation was considered pure enough (90 %, NMR) to be used in the ring-opening experiments.

EXPERIMENTAL

General remarks. See Ref. 1.

2-Ethyl-5-selenophenealdehyde (11). To 20.0 g (0.126 mol) of 2-ethylselenophene [18] in 100 ml of ether, 80 ml (0.13 mol) of 1.60 M butyllithium in hexane was added at such a rate that gentle reflux was maintained. After the addition, the reaction mixture was cooled to 0 °C and 9.5 g (0.13 mol) of DMF in 25 ml of ether was added. The mixture was allowed to reach room temperature and poured onto ice/conc. HCl. The aqueous layer was extracted with ether and the collected ethereal phases were washed with water, dried and evaporated, leaving 19.9 g of crude product. Distillation gave 15.0 g (64 %) of the title compound, b.p.$_{11}$ 121–123 °C. NMR (CCl_4): δ 7.75 (d, 1 H, 3-H), 7.05 (d, t, 1 H, 4-H), 2.93 (bq, 2 H, -CH_2-) and 1.33 (t, 3 H, CH_3), 9.58 (s, 1 H, CHO), $J_{3H,4H}$ 4.0 Hz, $J_{4H,5\text{-}CH_2-}$ = 1.1 Hz. [Found: C 44.80; H 4.52; Se 42.04. Calc. for C_7H_8OSe (187.10): C 44.94; H 4.31; Se 42.20].

3-Bromo-2-ethyl-5-selenophenealdehyde (12) was prepared in a way analogous to that used for *6* from 14.0 g (0.0748 mol) of *11*, 25.0 g (0.187 mol) of dry $AlCl_3$ and 13 g (0.081 mol) of bromine. After distillation, 12.6 g (63 %) of the title compound was obtained, b.p.$_{1.8}$ 113–115 °C. NMR (CCl_4): δ 7.77 (s, 1 H, 4-H), 2.84 (q, 2 H, -CH_2-) and 1.32 (t, 3 H, CH_3), 9.60 (s, 1 H, CHO), $J_{CH_2-CH_3}$ 7.0 Hz. [Found: C 31.61; H 2.65; Se 29.69. Calc. for C_7H_7BrOSe (266.00): C 31.61; H 2.65; Se 29.68].

3-Bromo-2-ethyl-5-methylselenophene (1b). A mixture of 10.0 g (0.0376 mol) of *12*, 6 ml of 99.5 % hydrazine hydrate and 30 ml of ethylene glycol was heated gradually to 140 °C, during which time water and hydrazine were distilled off. After cooling to room temperature, 6.0 g of KOH pellets was added, and the mixture was heated to 90–116 °C. The solution became red and was kept at 116 °C for 2.5 h, until nitrogen formation had subsided. After steam distillation, extraction of the distillate with ether, drying and evaporation of the solvent 6.0 g of crude *1b* remained. Distillation gave 5.0 g (53 %) of the pure title compound, b.p.$_{12}$ 99–102 °C. NMR (CCl_4): δ 6.65 (q, 1 H, 4-H), 2.46 (m, 3 H, 5-CH_3), 2.73 (q, 2 H, -CH_2-) and 1.22 (t, 3 H, CH_3), $J_{4H,5CH_3}$ 1.3 Hz, $J_{CH_2-CH_3}$ 7.0 Hz. [Found: C 33.34; H 3.69; Se 31.38. Calc. for C_7H_9BrSe (252.01): C 33.36; H 3.60; Se 31.33].

2-Acetyl-5-methylselenophene (5). A mixture of 25.0 g (0.172 mol) of 2-methylselenophene,[15] 40 g (0.39 mol) of acetic anhydride and 8 drops of perchloric acid (70 %) was refluxed for 1 h, cooled and poured into ice-water. The aqueous phase was extracted with ether and the ethereal portions were dried. Evaporation and distillation yielded 16.5 g (51 %) of the title compound, b.p.$_{13}$ 122–124 °C (lit.[11] b.p.$_{12}$ 114–116 °C). NMR (CCl_4): δ 7.61 (d, d, 1 H, 3-H), 6.90 (d, q, 1 H, 4-H), 2.55 (d, d, 3 H, 5-CH_3), 2.39 (s, 3 H, $COCH_3$), $J_{3H,4H}$ 3.6 Hz, $J_{4H,5CH_3}$ 1.0 Hz, $J_{3H,5CH_3}$ 0.4 Hz.

2-Acetyl-3-bromo-5-methylselenophene (6). To 33 g(0.25 mol) of aluminium trichloride, 18.7 g (0.100 mol) of *5* was added with stirring, cooling the reaction flask in an ice bath. The cooling bath was removed and after 30 min 19.2 g (0.120 mol) of bromine was added rapidly. The reaction mixture was stirred for 1 h, whereupon it was decomposed by treatment

with ice/conc. HCl. The aqueous phase was extracted several times with ether and the ethereal portions were washed with water to neutral reaction and dried. Evaporation and distillation gave the title compound, 18.4 g (69 %), b.p.$_{14}$ 108–111 °C. NMR (CCl_4): δ 7.60 (bs, 1 H, 4-H), 2.45 (bs, 2-CH_3; $COCH_3$). The integral of the methyl band represented 6 H. [Found: C 31.57; H 2.70; Se 29.67. Calc. for C_7H_7BrOSe (266.00): C 31.61; H 2.65; Se 29.69].

Attempted reduction of 5-acetyl-3-bromo-2-methylselenophene (6). (a) When the procedure used for the preparation of *1b* (see above) was applied to *6*, yellow crystals were formed but no trace of the title compound was found. When the reaction mixture was heated with potassium hydroxide gradually to 170–180 °C, red selenium was formed, but no nitrogen evolution was noticed. The reaction was stopped and the crystals were collected by suction and recrystallised from ethanol twice, but the melting interval was unchanged; 100–105 °C. IR: N-H 3320, 3180 cm^{-1}; $m/e = 280$; calc. for the hydrazone (*7a*) ($C_7H_9{}^{79}BrN_2{}^{80}Se$) = 280. NMR ($CCl_4$): δ 6.88 (s, 1 H, 4-H), 2.36 (s, 3 H, CH_3), 2.00 (s, 3 H, CH_3), 5.11 (2 H, NH_2).

(b) The tosylhydrazone *7b* was prepared according to a procedure described in Ref. 16, from 2.66 g (0.0100 mol) of *6* and 2.80 g (0.0150 mol) of tosylhydrazide in 50 ml of methanol; yield 3.4 g (78 %), m.p. 191–193 °C from methanol in the cold (–25 °C). [Found: C 38.4; H 3.54; N 6.78. Calc. for $C_{14}H_{15}BrN_2O_2SSe$ (434.23): C 38.72; H 3.48; N 6.45].

The tosylhydrazone, 3.0 g (0.0069 mol), was treated with 2.7 g (0.071 mol) of $LiAlH_4$ in 50 ml of refluxing THF for 18 h (*cf.* Ref. 16). Upon hydrolysis of the reaction mixture with moist ether, red selenium precipitated and 0.3 g of a brown, uncharacterised oil remained after work-up and evaporation.

2,5-Dibromoselenophene. To a solution of 50.0 g (0.382 mol) of selenophene in 50 ml of acetic acid, 126 g (0.788 mol) of bromine in 80 ml of acetic acid was added (ice cooling). After stirring overnight the reaction mixture was poured into water. The aqueous phase was neutralised with sodium hydrogen carbonate and extracted with ether several times. The ethereal portions were washed with water to neutral reaction and dried. Evaporation and distillation yielded 77.7 g (70 %) of the title compound, b.p.$_{13}$ 101–104 °C, n_D^{20} 1.6665 (lit.[17] b.p.$_{0.03}$ 42 °C, n_D^{20} 1.667).

2-Bromo-5-methylselenophene (10). To a solution of 28.9 g (0.100 mol) of 2,5-dibromoselenophene in 200 ml of ether, 65 ml (0.10 mol) of 1.60 M butyllithium in hexane was added at –50 °C, followed by 13.0 g (0.103 mol) of dimethyl sulfate in 50 ml of ether at –30 °C. The reaction mixture was allowed to reach room temperature and was kept there for 1 h, whereupon conc. ammonium hydroxide was added. Extraction with ether, drying, evaporation and distillation gave 15.0 g (67 %) of the title compound, b.p.$_{11}$ 79–80 °C. NMR (CCl_4): δ 6.90 (d, 1 H, 3-H), 6.52 (d, q, 1 H, 4-H), 2.46 (d, 3 H, 5-CH_3), $J_{3H,4H}$ 3.8 Hz, $J_{4H,5CH_3}$ 1.2 Hz [Found: C 26.75; H 2.28; Br 35.66; Se 35.20. Calc. for C_5H_5BrSe (223.96): C 26.82; H 2.25; Br 35.68; Se 35.26].

3,5-Dibromo-2-methylselenophene (9). (a) To a solution of 7.0 g (0.048 mol) of 2-methylselenophene[15] in 50 ml of carbon disulfide, 16 g (0.10 mol) of bromine in 20 ml of acetic acid was added at –20 °C. When the addition was complete, the mixture was stirred at room temperature overnight and then poured into water and extracted with ether. Drying and evaporation of the solvent gave 16.8 g of a brown fuming oil, which reacted with solid KOH (4 g) under heat evolution. After the base treatment, the residue was distilled, giving 6.0 g (41 %) of the title compound having the same physical data as described earlier.[14]

(b) To a solution of 11.2 g (0.0500 mol) of *10* in 50 ml of acetic acid, 8.0 g (0.050 mol) of bromine in 25 ml of acetic acid was added at room temperature. The mixture was stirred for 1.5 h and then poured into water. The aqueous phase was neutralised with dil. aq. NaOH and extracted with ether. The ethereal portions were dried and the solvent evaporated, giving 12.9 g of a crude product, which was distilled to yield 8.0 g (53 %) of the title compound.

3-Bromo-5-ethyl-2-methylselenophene (1a). A solution of 9.10 g (0.300 mol) of *9* in 150 ml of ether was cooled to –70 °C and 20 ml (0.30 mol) of 1.50 M butyllithium in hexane was added drop-wise, followed by 9.25 g (0.0600 mol) of diethyl sulfate in 25 ml of ether after 10 min. The reaction mixture was slowly heated and an exothermic reaction took place between +17 and +25 °C, after which it was left overnight. Conc. ammonium hydroxide was added and the mixture was stirred for 1 h. The ethereal layer was separated, washed with 2 N HCl, water and dried. Evaporation and distillation gave 2.8 g (37 %) of almost pure title compound, b.p.$_{12}$ 101–104 °C. GLC (column BDS 10 %, 150 °C) and NMR analysis showed that some diethyl sulfate (≈10 %) was present. Repeated distillation and preparative TLC (1 mm silica gel; hexane) did not remove this impurity nor did prolonged treatment with ammonium hydroxide. Combined GLC-MS showed the molecular fragment at m/e 252 (100 %) with the isotopic distribution typical for 1 Br and 1 Se. Calc. for $C_7H_9{}^{79}Br^{80}Se$ 252. NMR (CCl_4): δ 6.66 (bt, 1 H, 4-H), 2.40 (bs, 3 H, 2-CH_3), 2.79 (q, 2 H, -CH_2-) and 1.25 (t, 3 H, CH_3); $J_{CH_3-CH_2}$ 7.5 Hz. [Found: C 34.89; H 3.72; Se 30.66. Calc. for C_7H_9BrSe (252.00): C 33.66; H 3.60; Se 31.33].

(Z)-2-Ethylseleno-2-hepten-4-yne (4b). To 2.52 g (0.0100 mol) of *1b* in 50 ml of ether, 17 ml (0.010 mol) of 0.60 M ethereal ethyllithium was added followed by 5.45 g (0.0500 mol) of ethyl

bromide. GLC analysis (column BDS 10 %, 170 °C) of the washed and dried ethereal solution showed 98 % of *4b*. The crude product was distilled from paraffin oil (to prevent decomposition of the enyne to some extent), b.p.$_{12}$ 108–109 °C, 1.1 g (54 %). IR: C≡C 2220 cm^{-1}. Mass spectrum, *m/e* (%); selenium-containing fragments: 202 (100), 187 (8), 173 (12), 159 (27), 145 (8), 133 (7), 119 (2), 117 (4), 107 (33). Fragments not containing selenium: 93 (66), 92 (40), 91 (73), 79 (10), 78 (23), 77 (43), 67 (3), 66 (3), 65 (23), 64 (3), 63 (10), 62 (3), 55 (3), 53 (10), 52 (3), 51 (17), 50 (3), 44 (3), 41 (17), 40 (3), 39 (23). NMR (CCl_4): δ 2.13 (m, 3 H, 1-H), 5.58 (m, 1 H, 3-H), 2.35 (bq, 6-H), 1.67–0.92 (7-H), 2.78 (q, 2 H) and 1.67–0.92 (SeC_2H_5), $J_{6H,7H}$ 7.0 Hz. [Found: C 53.70; H 7.10; Se 39.25. Calc. for $C_9H_{14}Se$ (201.16): C 53.73; H 7.01; Se 39.25].

(Z)-3-Ethylseleno-3-hepten-5-yne (4a). As above, the title compound was prepared from 2.45 g (9.60 mmol) of *1a* in 50 ml of ether, 16 ml (9.6 mmol) of 0.60 M ethereal ethyllithium and 5.45 g (50.0 mmol) of ethyl bromide. GLC analysis (conditions as above) of the washed and dried ethereal solution showed about 90 % of *4a*, b.p.$_{12}$ 107–108 °C, 1.2 g (60 %). (The distillation was performed from paraffin oil.) IR: C≡C 2220 cm^{-1}. Mass spectrum *m/e*, (%); selenium-containing fragments: 202 (100), 187 (5), 173 (9), 159 (16), 145 (5), 133 (4), 131 (5), 119 (4), 117 (4), 107 (9). Fragments not containing selenium: 93 (32), 92 (40), 91 (100), 79 (12), 78 (24), 77 (80), 67 (4), 66 (4), 65 (28), 64 (4), 63 (12), 62 (4), 55 (4), 53 (16), 52 (4), 51 (16), 50 (4), 44 (4), 41 (20), 40 (4), 39 (28). NMR (CCl_4): δ 1.67–0.83 (1-H), 2.33 (q, 2-H), 5.67 (m, 4-H), 1.95 (m, 7-H), 2.77 (q) and 1.67–0.83 (SeC_2H_5), $J_{SeCH_2-CH_3} = J_{1H,2H} = 7.0$ Hz. [Found: C 53.67; H 6.89; Se 39.22. Calc. for $C_9H_{14}Se$ (201.16): C 53.73; H 7.01; Se 39.25].

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