# Fused polycyclic nitrogen-containing heterocycles
## 16.* Selenazolo[3,4-*a*]- and thiazolo[3,4-*a*]quinoxalin-4(5*H*)-ones

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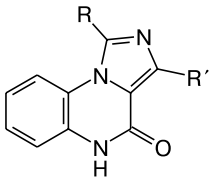
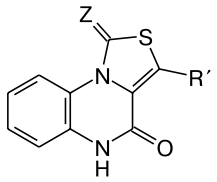
Upon treatment with acetic acid, 3-(2-phenyl-1-selenocyanatoethyl)-, 3-(3-phenyl-1-selenocyanatopropyl)quinoxalin-2(1*H*)-ones and their thio analogs undergo intramolecular condensation to form selenazolo- and thiazolo[3,4-*a*]quinoxalines.

**Key words:** selenazolo[3,4-*a*]quinoxalines, thiazolo[3,4-*a*]quinoxalines, intramolecular condensation, substituted quinoxalines, IR, NMR spectra.

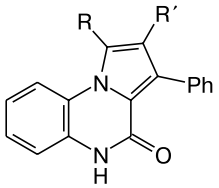
Azolo[*a*]quinoxalines and their 4,5-dihydro derivatives show various biological and pharmacological properties[2—6] and are of use in the synthesis of many biologically important compounds and drugs.[7—10] In this connection, numerous methods for the synthesis of imidazo[1,5-*a*]-,[11—15] thiazolo[3,4-*a*]-,[16—21] and especially pyrrolo[1,2-*a*]quinoxalines[22—26] have been elaborated. For example, a number of imidazo[1,5-*a*]- (**1**),[13—15] thiazolo[3,4-*a*]- (**2**),[20,21] and pyrrolo[1,2-*a*]quinoxalines (**3**)[26] were synthesized by the reaction of 3-(α-chlorobenzyl)quinoxalin-2(1*H*)-one with potassium cyanate and thiocyanate, thiourea, and compounds with active methylene group, which were used as synthetic equivalents of $N^{-}=C^{+}$, $S^{-}–C^{+}$, and $RC^{+(-)}=C^{-(+)}R'$ dipoles.



**1**



**2**



**3**

**1:** R = H, OH, SH, SAlk, Ar; R′ = Ph, $CO_2Et$
**2:** Z = O, S, NH, NAr, NAc, *N*-naphthyl, *N*-thiazolyl; R′ = Me, Ar
**3:** R = Me, Ph, $NH_2$, OEt; R′ = Ac, Bz, CN, $CONH_2$

In contrast to the diversity of azoloquinoxalines, their selenium analogs, *i.e.*, selenazolo[3,4-*a*]quinoxalines, which, in comparison with the other representatives of azolo[*a*]quinoxalines, are of much interest for their biological and physical and chemical properties, so far are not synthesized.

As it follows from the proposed by us methods of synthesis,[13,26,27] based on the retro-synthetic analysis of azolo[*a*]quinoxaline structures, a selenium analog of potassium thiocyanate, *i.e.*, potassium selenocyanate, could serve as the synthetic equivalent of the two-atomic fragment, the reaction of which with 3-(α-chlorophenylalkyl)quinoxalin-2(1*H*)-ones (**4a,b**) would lead to the key intermediates on the way to the desired tricyclic systems.

Nucleophilic substitution of chlorine atom in compounds **4a,b** (see Experimental) proceeds in DMF in the presence of the excess of KSeCN under argon atmosphere at room temperature for chlorophenylethylquinoxalinone **4a** and at 40 °C for chlorophenylpropylquinoxalinone **4b**. The subsequent aqueous treatment leads to the formation of light pink crystals of compounds **5a**,**b** (Scheme 1). In the IR spectra of the latter, a characteristic absorption band of SeCN group in the region 2150—2155 $cm^{-1}$ is presented, whereas in the $^1H$ NMR spectra, signals of the methyne protons are upfield shifted by ~0.5 ppm in comparison with the corresponding signals in the starting compounds **4a**,**b**, signals of the methyne protons in which are observed at 5.67 and 5.43 ppm, respectively (see Experimental).

Selenazolo-annulation of quinoxaline **5a** was carried out in boiling acetic acid for 15 h, whereas only 5 h were required for **5b** (Scheme 2). During this, the crystalline compounds with high melting points (292—294 °C (**6a**) and 305—308 °C (**6b**)) were formed, the latter were by 90 and 110 °C higher then the melting points of the corre-

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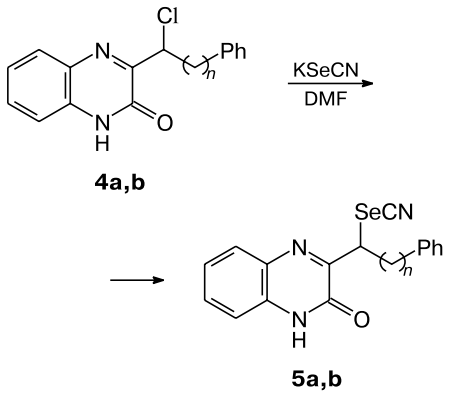
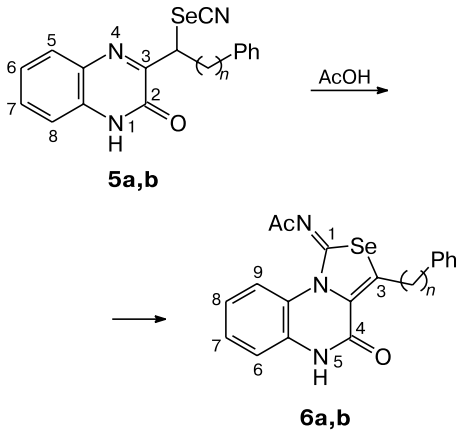
**Scheme 2**



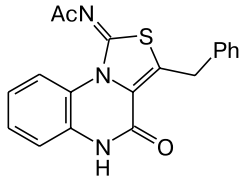

*n* = 1 (**a**), 2 (**b**)

sponding starting compounds, which is characteristic of the condensed quinoxaline systems.[20,26] The disappearance of the absorption band of SeCN group in the IR spectrum is also an evidence in favor of the formation of selenazolo[3,4-*a*]quinoxaline tricyclic system, as well as the changes in the $^1$H NMR spectra, where, along with the signals of the protons of the aromatic rings and carbamoyl group, the chemical shifts and multiplicity of signals of the protons at sp$^3$-carbon atoms are changed, namely, instead of signals of ABX-system of —CH—CH$_2$— group of compound **5a** and complicated multiplets of ABCDX-system of —CH—CH$_2$—CH$_2$— group of compound **5b**, a singlet of CH$_2$ group (4.8 ppm) of compound **6a** and two triplets of —CH$_2$—CH$_2$— group (3.0—3.7 ppm) of compound **6b** appear. In the $^1$H NMR spectrum, a doublet of H(9) proton of compounds **6a,b** is downfield shifted by 2.1 ppm in comparison with the signal of H(5) proton in the starting compounds **5a,b** and is observed at 9.9 ppm, which is diagnostical for azolo[*a*]-annulated quinoxaline systems.[27,28]

**Scheme 2**




In the $^1$H NMR spectrum of the sulfur analog of tricycle **6a**, *i.e.*, 1-acetylimino-3-benzylthiazolo[3,4-*a*]quinoxalin-4(5*H*)-one (**7**), purposely synthesized from the corresponding 3-(α-thiocyanatophenylethyl)quinoxalin-2(1*H*)-one, according to the procedure similar to that for the preparation of compound **6a**, the doublet of H(9) proton of thiazoloquinoxaline appears close to the region (9.92 ppm, $J$ = 8.4 Hz) for the selenium derivatives **6a,b**.




**7**

It should be noted that a general tendency of the downfield movement of chemical shifts of H(9) protons going from pyrrolo[1,2-*a*]quinoxalines[26] (8.15 ppm) through imidazo[1,5-*a*]-[13] (8.60 ppm) and thiazolo[3,4-*a*]quinoxalines[19—21] (9.42 ppm) to selenazolo[3,4-*a*]quinoxalines (≈ 10.0 ppm) is observed.

## Experimental

Melting points were determined on the Boetius heating table. IR spectra were recorded on a Bruker Vector-22 Fourier spectrometer in Nujol for all the compounds. Mass spectra of electron ionization were recorded on a ThermoQuest/Finnigan TRACE MS quadruple mass-spectrometer, insertion of the samples were carried out through the direct injection system with water cooling. $^1$H NMR spectra of compounds **4a,b**, **5a,b**, and **6b** were recorded on a Bruker MSL-400 spectrometer (400.13 MHz), of compounds **6a** and **7**, on a Bruker Avance-600 spectrometer (600.13 MHz) in DMSO-d$_6$ for all the compounds. Chemical shifts are given in δ scale with signals of residual protons in DMSO-d$_6$ ($\delta_H$ 2.54 ppm) as the internal standard.

**3-(1-Chloro-2-phenylethyl)quinoxalin-2(1*H*)-one (4a).** The product was obtained by the reaction of methyl 3-chloro-2-oxo-4-phenylbutanoate[29] with *o*-phenylenediamine in quantitative yield according to the procedure elaborated by us earlier,[30] m.p. 199—200 °C. Found (%): C, 67.31; H, 4.45; N, 9.97; Cl, 12.38. $C_{16}H_{13}ClN_2O$. Calculated (%): C, 67.49; H, 4.60; N, 9.84; Cl, 12.45. IR, ν/cm$^{-1}$: 503, 589, 698. 709, 755, 761, 907, 1031, 1432, 1482, 1498, 1577, 1599, 1609, 1661, 3065, 3157. $^1$H NMR, δ: 3.44 (dd, 1 H, C$\underline{H}_2$Ph, $J_{ab}$ = 13.93 Hz, $J_{ax}$ = 7.96 Hz); 3.69 (dd, 1 H, C$\underline{H}_2$Ph, $J_{ab}$ = 13.93 Hz, $J_{bx}$ = 6.97 Hz); 5.67 (dd, 1 H, CHCl, $J_{ax}$ = 7.96 Hz, $J_{bx}$ = 6.97 Hz); 7.20 (ddd, 1 H, H(6), $J$ = 7.29 Hz, $J$ = 6.31 Hz, $J$ = 1.99 Hz); 7.25—7.35 (m, 5 H, Ph); 7.37 (d, 1 H, H(8), $J$ = 7.96 Hz); 7.57 (ddd, 1 H, H(7), $J$ = 8.30 Hz, $J$ = 7.30 Hz, $J$ = 1.32 Hz); 7.83 (d, 1 H, H(5), $J$ = 8.63 Hz); 12.48 (br.s, 1 H, NH).

**3-(1-Chloro-3-phenylpropyl)quinoxalin-2(1*H*)-one (4b).** The product was obtained similarly[30] with the use of methyl 3-chloro-2-oxo-5-phenylpentanoate,[29] m.p. 201—203 °C. Found (%): C, 68.13; H, 4.93; N, 9.55; Cl, 11.75. $C_{17}H_{15}ClN_2O$. Calculated (%): C, 68.34; H, 5.06; N, 9.38; Cl, 11.87. IR, ν/cm$^{-1}$: 586, 701, 738, 751, 764, 906, 1289, 1429, 1560, 1608, 1677, 2720, 3021, 3067. $^1$H NMR, δ: 2.75—2.83, 2.88—2.94 (both m, 4 H each, $CH_2CH_2$); 5.40—5.45 (m, 1 H, CHCl); 7.20—7.40 (m, 7 H, Ph + H(6) and H(8)); 7.60 (dd, 1 H, H(7), $J$ = 7.96 Hz,

*J* = 7.32 Hz); 7.82 (d, 1 H, H(5), *J* = 7.96 Hz); 12.48 (br.s, 1 H, NH).

**3-(2-Phenyl-1-selenocyanatoethyl)quinoxalin-2(1*H*)-one (5a).** A solution of 3-(1-chloro-2-phenylethyl)quinoxalin-2(1*H*)-one (**4a**) (0.31 g, 1.1 mmol) and KSeCN (0.47 g, 3.3 mmol) in DMF (10 mL) was stirred for 8 h under argon, then, the reaction mixture was kept for 48 h and quenched with water. The crystals formed were filtered off, washed with water (3×10 mL), and dried in air. The yield was 0.37 g (94%), m.p. 202—204 °C. Found (%): C, 57.23; H, 3.53; N, 11.98. $C_{17}H_{13}N_3OSe$. Calculated (%): C, 57.64; H, 3.70; N, 11.86. IR, ν/cm$^{-1}$: 433, 468, 494, 592, 700, 754, 767, 931, 1155, 1608, 1668, 2151, 3063, 3329. $^1$H NMR, δ: 3.72 (dd, 1 H, C$\underline{H}_2$Ph, $J_{ab}$ = 13.70 Hz, $J_{ax}$ = 8.58 Hz); 3.81 (dd, 1 H, C$\underline{H}_2$Ph, $J_{ab}$ = 13.70 Hz, $J_{bx}$ = 5.82 Hz); 5.17 (dd, 1 H, CHSeCN, $J_{ax}$ = 8.58 Hz, $J_{bx}$ = 5.82 Hz); 7.24—7.44 (m, 7 H, Ph + H(6), H(8)); 7.50 (dd, 1 H, H(7), *J* = 7.56 Hz, *J* = 7.52 Hz); 7.83 (d, 1 H, H(5), *J* = 7.88 Hz); 12.46 (br.s, 1 H, NH).

**3-(3-Phenyl-1-selenocyanatopropyl)quinoxalin-2(1*H*)-one (5b).** Compound **5b** was obtained similarly to **5a** from 3-(1-chloro-3-phenylpropyl)quinoxalin-2(1*H*)-one (**4b**) (1.00 g, 3.3 mmol) and KSeCN (1.45 g, 9.9 mmol) at 40 °C. The yield was 1.00 g (81%), m.p. 196—197 °C. Found (%): C, 58.41; H, 3.95; N, 11.56. $C_{18}H_{15}N_3OSe$. Calculated (%): C, 58.70; H, 4.11; N, 11.41. IR, ν/cm$^{-1}$: 414, 487, 593, 752, 915, 945, 1348, 1496, 1553, 1607, 1666, 2154, 2720, 3087, 3220. $^1$H NMR, δ: 2.60—2.95 (m, 4 H, $CH_2CH_2$); 4.90—5.00 (m, 1 H, CHSeCN); 7.15—7.41 (m, 7 H, Ph + H(6), H(8)); 7.59 (dd, 1 H, H(7), *J* = 8.24 Hz, *J* = 6.84 Hz); 7.83 (d, 1 H, H(5), *J* = 8.24 Hz); 12.57 (br.s, 1 H, NH).

**1-Acetylimino-3-benzylselenazolo[3,4-*a*]quinoxalin-4(5*H*)-one (6a).** A solution of compound **5a** (0.2 g, 0.56 mmol) in acetic acid (10 mL) was refluxed for 15 h, the reaction mixture was kept for 12 h at room temperature, the crystals formed were filtered off and recrystallized from acetic acid. The yield was 0.092 g (41%), m.p. 292—294 °C. Found (%): C, 57.39; H, 3.67; N, 10.49. $C_{19}H_{15}N_3O_2Se$. Calculated (%): C, 57.58; H, 3.81; N, 10.60. IR, ν/cm$^{-1}$: 698, 761, 1230, 1272, 1304, 1328, 1400, 1499, 1575, 1606, 1672, 3036, 3127, 3185. $^1$H NMR, δ: 2.36 (s, 3 H, MeCO); 4.82 (s, 2 H, C$\underline{H}_2$Ph,); 7.17—7.42 (m, 8 H, Ph + H(6), H(7), H(8)); 9.95 (d, 1 H, H(9), *J* = 8.4 Hz); 11.42 (br.s, 1 H, NH). MS (EI, 70 eV), *m*/*z* ($I_{rel}$ (%)): 399 (12), 398 (15), 397 (60), 396 (8), 395 [M]$^+$ (32), 382 (12), 380 (12), 328 (40), 327 (25), 326 (22), 325 (18), 299 (24), 297 (10), 249 (26), 248 (38), 247 (100), 220 (14), 219 (76), 218 (21), 115 (13), 105 (11), 102 (18), 91 (21), 90 (17), 77 (12).

**1-Acetylimino-3-phenylethylselenazolo[3,4-*a*]quinoxalin-4(5*H*)-one (6b).** A solution of compound **5b** (0.2 g, 0.54 mmol) in acetic acid (10 mL) was refluxed for 5 h, the reaction mixture was kept for 12 h at room temperature, the crystals formed were filtered off and recrystallized from acetic acid. The yield was 0.103 g (46%), m.p. 305—307 °C. Found (%): C, 58.38; H, 3.93; N, 10.15. $C_{20}H_{17}N_3O_2Se$. Calculated (%): C, 58.54; H, 4.18; N, 10.24. IR, ν/cm$^{-1}$: 698, 736, 750, 1224, 1258, 1272, 1305, 1328, 1396, 1496, 1568, 1670, 2688, 2727, 3123, 3180. $^1$H NMR, δ: 2.49 (s, 3 H, MeCO); 3.03 (t, 2 H, C$\underline{H}_2$Ph, *J* = 7.66 Hz); 3.75 (t, 2 H, $CH_2$, *J* = 7.66 Hz); 7.20—7.36 (m, 8 H, Ph + H(6), H(7), H(8)); 9.98 (d, 1 H, H(9), *J* = 8.63 Hz); 11.28 (br.s, 1 H, NH). MS (EI, 70 eV), *m*/*z* ($I_{rel}$ (%)): 411 (0.17), 409 [M]$^+$ (0.11), 278 (1), 261 (1), 251 (4), 91 (100), 65 (27), 43 (41).

**3-(2-Phenyl-1-thiocyanatoethyl)quinoxalin-2(1*H*)-one.** A solution of 3-(1-chloro-2-phenylethyl)quinoxalin-2(1*H*)-one **4a** (0.30 g, 1.1 mmol) and KSCN (0.32 g, 3.3 mmol) in DMF (10 mL) was stirred for 7 days and quenched with water. The crystals formed were filtered off, washed with water (3×10 mL), and dried in air. The yield was 0.30 g (93%), m.p. 207—209 °C. Found (%): C, 66.21; H, 4.19; N, 13.56; S, 10.97. $C_{17}H_{13}N_3OS$. Calculated (%): C, 66.43; H, 4.26; N, 13.67; S, 10.43. IR, ν/cm$^{-1}$: 405, 471, 502, 592, 704, 764, 919, 953, 1030, 1461, 1484, 1497, 1605, 1664, 2156, 3087, 3168. $^1$H NMR, δ: 3.48 (dd, 1 H, C$\underline{H}_2$Ph, $J_{ab}$ = 13.89 Hz, $J_{ax}$ = 7.89 Hz); 3.71 (dd, 1 H, C$\underline{H}_2$Ph, $J_{ab}$ = 13.89 Hz, $J_{bx}$ = 6.86 Hz); 5.18 (dd, 1 H, CHSCN, $J_{ax}$ = 7.89 Hz, $J_{bx}$ = 6.86 Hz); 7.22—7.42 (m, 7 H, Ph + H(6), H(8)); 7.62 (dd, 1 H, H(7), *J* = 8.24 Hz, *J* = 7.52 Hz); 7.87 (d, 1 H, H(5), *J* = 8.24 Hz); 12.64 (br.s, 1 H, NH).

**1-Acetylimino-3-benzylthiazolo[3,4-*a*]quinoxalin-4(5*H*)-one (7).** A solution of 3-(2-phenyl-1-thiocyanatoethyl)quinoxalin-2(1*H*)-one (0.2 g, 0.65 mmol) in acetic acid (10 mL) was refluxed for 8.5 h, the reaction mixture was kept for 7 h at room temperature, the crystals formed were filtered off. The yield was 0.125 g (55%), m.p. 296—298 °C. Found (%): C, 65.10; H, 4.32; N, 11.90; S, 9.97. $C_{19}H_{15}N_3O_2S$. Calculated (%): C, 65.31; H, 4.33; N, 12.03; S, 9.18. IR, ν/cm$^{-1}$: 603, 657, 697, 762, 775, 791, 982, 1162, 1240, 1271, 1293, 1309, 1331, 1405, 1443, 1497,1508, 1582, 1611, 1671, 3030, 3127, 3184. $^1$H NMR, δ: 2.33 (s, 3H, $CH_3CO$); 4.77 (s, 2 H, $CH_2$); 7.20—7.40 (m, 8 H, Ph + H(6), H(7), H(8)); 9.92 (d, 1 H, H(9), *J* = 8.4 Hz); 11.46 (br.s, 1 H, NH).

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