# Aminomethylation of *N*-methylmorpholinium 6-amino-4-aryl-3,5-dicyano-1,4-dihydropyridine-2-selenolates as an approach to the preparation of 3,5,7,11-tetraazatricyclo[7.3.1.$0^{2,7}$]tridec-2-ene-8-selenone derivatives

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A reaction of *N*-methylmorpholinium 6-amino-4-aryl-3,5-dicyano-1,4-dihydropyridine-2-selenolates with primary amines and excess of formaldehyde leads to 3,5,7,11-tetraazatricyclo[7.3.1.$0^{2,7}$]tridec-2-ene-8-selenone derivatives. The same compounds were obtained by a multicomponent cascade cyclocondensation of benzaldehyde, cyanoselenoacetamide, primary amine, and excess of formaldehyde.

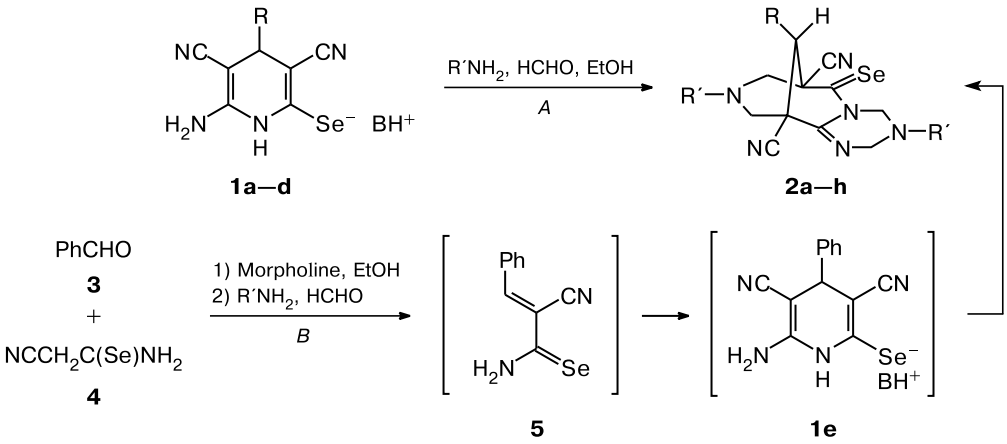
**Key words:** *N*-methylmorpholinium 6-amino-4-aryl-3,5-dicyano-1,4-dihydropyridine-2-selenolate, multicomponent cyclocondensation, cyanoselenoacetamide, Mannich reaction, 3,5,7,11-tetraazatricyclo[7.3.1.$0^{2,7}$]tridec-2-ene-8-selenone.

Pyridine-2(1*H*)-chalcogenones are an interesting class of heterocyclic compounds with a wide range of practically useful properties and a possibility of chemical conversions, which lead to various new heterocyclic systems.[1,2]

In continuation of our studies in the field of aminomethylation of such compounds,[3—18] we have found that treatment of *N*-methylmorpholinium 6-amino-4-aryl-3,5-dicyano-1,4-dihydropyridine-2-selenolates **1a—c** with primary amines in the presence of an excess of the 37% aqueous formaldehyde resulted in 3,5,7,11-tetraazatricyclo-[7.3.1.$0^{2,7}$]tridec-2-ene-8-selenone derivatives **2a—h** in 12—55% yields (Scheme 1, method *A*).

An alternative approach to the preparation of the target structures **2** consisted in the multicomponent cyclocondensation of benzaldehyde (**3**), cyanoselenoacetamide (**4**), primary amine, and formaldehyde. Thus, the reaction of benzaldehyde with cyanoselenoacetamide **4** in the presence of morpholine and subsequent treatment with primary

**Scheme 1**




**1:** R = 2-$FC_6H_4$ (**a**), 2-$MeOC_6H_4$ (**b**), 2-$EtOC_6H_4$ (**c**), Ph (**d**, **e**); B = *N*-methylmorpholine (**a—d**), morpholine (**e**)

| **2** | R | R´ | **2** | R | R´ | **2** | R | R´ | **2** | R | R´ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **a** | Ph | Ph | **c** | 2-$MeOC_6H_4$ | 4-$MeC_6H_4$ | **e** | 2-$MeOC_6H_4$ | $CH_2Ph$ | **g** | 2-$EtOC_6H_4$ | 4-$MeC_6H_4$ |
| **b** | Ph | Me | **d** | 2-$MeOC_6H_4$ | Ph | **f** | 2-$MeOC_6H_4$ | Me | **h** | 2-$EtOC_6H_4$ | $CH_2Ph$ |

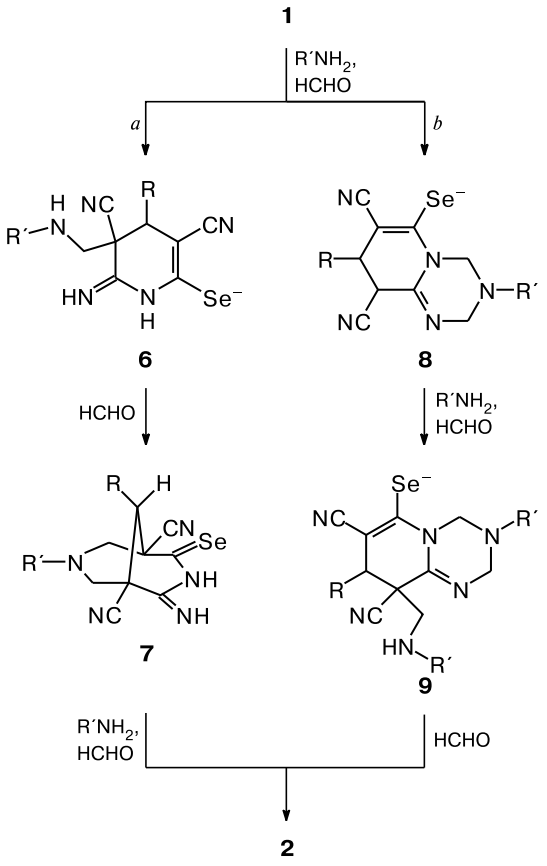
Published in Russian in *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 6, pp. 1401—1404, June, 2013.

amines and excess of 37% aqueous formaldehyde resulted in compounds **2a**,**b** in 14 and 39% yields. The reaction possibly proceeds through the consecutive formation of unsaturated selenoamide **5** and selenolate **1e**. The latter further undergoes a cascade aminomethylation to form 3,5,7,11-tetraazatricyclo[7.3.1.$0^{2,7}$]tridec-2-ene-8-selenones **2** (see Scheme 1, method *B*).

Earlier,[19] we have shown that the reaction of benzaldehyde with cyanoselenoacetamide, primary amines, and excess of formaldehyde catalyzed by *N*-methylmorpholine led to pyrimido[4,3-*b*][1,3,5]selenadiazine derivatives. Supposedly, the alternative direction of the reaction observed in this case is due to the higher bacisity of morpholine as compared to that of *N*-methylmorpholine. Apparently, 2-cyano-3-phenylprop-2-eneselenoamide **5** formed in the first step reacts *in situ* in the presence of morpholine with the second equivalent of cyanoselenoacetamide to form selenolate **1e**. Subsequent aminomethylation proceeds similarly to that in method *A*.

Two plausible pathways can be suggested for the aminomethylation of compounds **1** (Scheme 2, *a* and *b*). Pathway *a* supposes an initial formation of products of C-aminomethylation of **6**, which through the bispidine intermediate **7** and subsequent N,N´-diaminomethylation

**Scheme 2**




are converted to compounds **2**. According to pathway *b*, the initial aminomethylation takes place at the nitrogen atoms with the formation of pyridotriazines **8**, which in the course of subsequent C-aminomethylation through the intermediate **9** are converted to the final products **2**.

Both pathways for the formation of tetraazatricyclotridecene skeleton of compounds **2** can proceed simultaneously. Supposedly, the construction of the 3,7-diazabicyclo[3.3.1]nonane fragment begins from the C(5)-aminomethylation of dihydropyridine ring. As it was shown earlier[20—22] using related pyridine-2-thiolates as an example, the presence of an electron-withdrawing substituent at position C(5) was a determining factor for the reaction of C(3),C(5)-diaminomethylation to proceed. In the absence of an electron-withdrawing substituent, pyridine-2-thiolates play the role of typical S,N-binucleophiles and do not give the C-aminomethylation products.[8]

The structure of 3,5,7,11-tetraazatricyclo[7.3.1.$0^{2,7}$]-tridec-2-ene-8-selenones **2** was confirmed by spectroscopic studies. The IR spectra of the compounds synthesized exhibit absorption bands at 2248—2255 and 1646—1655 $cm^{-1}$, which indicate the presence of the unconjugated nitrile group and the C=N fragment, respectively. The $^1$H NMR spectra of compounds **2** are characterized by the presence of four sets of signals, which correspond to four methylene groups. The signals for the protons C(4)$H_2$ and C(6)$H_2$ of the tetrahydro-1,3,5-triazine ring were found as two pairs of doublets in the region δ 4.23—5.18 ($^2J$ = 17.0—17.4 Hz) and 5.07—6.12 ($^2J$ = 12.7—13.7 Hz), respectively. Protons C(10)$H_2$ and C(12)$H_2$ resonate at δ 2.89—4.29 as two pairs of doublets with $^2J$ = 10.5—11.8 Hz or as multiplets resulting from the partial overlap of the signals. The $^1$H NMR spectra of compounds **2** also exhibit signals for proton C(13)H as a singlet at δ 4.21—4.77 and three sets of signals for the substituents: one from C(13)Ar and two from the primary amine. The $^1$H NMR spectra of compounds **2** agree with the data obtained for the related 8-thioxo-3,5,7,11-tetraazatricyclo[7.3.1.$0^{2,7}$]tridec-2-enes,[4,5] whose structure is unambiguously confirmed by X-ray crystallography.[5]

In conclusion, *N*-methylmorpholinium 6-amino-4-aryl-3,5-dicyano-1,4-dihydropyridine-2-selenolates under the Mannich reaction conditions form 3,5,7,11-tetraazatricyclo[7.3.1.$0^{2,7}$]tridec-2-ene-8-selenones. Similar result was obtained by the morpholine-catalyzed multicomponent cascade cyclocondensation of benzaldehyde, cyanoselenoacetamide, primary amine, and formaldehyde.

## Experimental

$^1$H NMR spectra were recorded on a Bruker Avance II 400 spectrometer (399.97 MHz) in DMSO-$d_6$, using $Me_4Si$ as an internal standard. IR spectra were recorded on a IKS-29 spectrophotometer (in Nujol), elemental analysis was performed on

a Carlo Erba Strumentazione Elemental Analyzer 1106 instrument. The individuality of the samples obtained was controlled by TLC on Silufol UV-254 plates, using a mixture of acetone—hexane (1 : 1) as an eluent and the iodine vapors or a UV detector as a visualizing agent. Melting points of compounds were determined on a Kofler heating stage and were not corrected. The preparation and the properties of selenolates **1b** [23] and **1d** [24] were described earlier. Selenolates **1a**,**c** were synthesized according to the known procedure[25] by the reaction of aldehydes with 2 equiv. of cyanoselenoacetamide **4** in the presence of *N*-methylmorpholine. Cyanoselenoacetamide **4** was obtained by passing $H_2Se$ through the solution of malononitrile in diethyl ether.[26] All the syntheses were carried out under argon.

***N*-Methylmorpholinium 6-amino-3,5-dicyano-4-(2-fluorophenyl)-1,4-dihydropyridine-2-selenolate (1a).** The yield was 66%, m.p. 155—157 °C. Found (%): C, 54.21; H, 5.09; N, 13.31. $C_{13}H_8FN_4Se \cdot C_5H_{12}NO$. Calculated (%): C, 54.42; H, 5.05; N, 13.36. IR, ν/cm$^{-1}$: 3415, 3315, 3270 ($NH_2$, NH); 2175 (2 C≡N); 1650 (C=C). $^1$H NMR, δ: 2.35 (s, 3 H, NMe); 2.51—2.65 (m, 4 H, $N(CH_2)_2$); 3.59—3.71 (m, 4 H, $O(CH_2)_2$); 4.64 (s, 1 H, C(4)H); 5.84 (br.s, 2 H, $NH_2$); 7.05—7.31 (m, 4 H, $H_{Ar}$); 9.34 (s, 1 H, NH).

***N*-Methylmorpholinium 6-amino-3,5-dicyano-4-(2-ethoxyphenyl)-1,4-dihydropyridine-2-selenolate (1c).** The yield was 63%, m.p. 120—122 °C. Found (%): C, 53.73; H, 5.68; N, 15.47. $C_{15}H_{13}N_4OSe \cdot C_5H_{12}NO$. Calculated (%): C, 53.81; H, 5.60; N, 15.69. IR, ν/cm$^{-1}$: 3382, 3310 ($NH_2$, NH); 2190 (2 C≡N); 1620 (C=C). $^1$H NMR, δ: 1.44 (t, 3 H, $OCH_2C\underline{H}_3$, $^3J$ = 6.9 Hz); 2.26 (s, 3 H, NMe); 2.38—2.47 (m, 4 H, $N(CH_2)_2$); 3.56—3.61 (m, 4 H, $O(CH_2)_2$); 4.07 (q, 2 H, $OC\underline{H}_2CH_3$, $^3J$ = 6.9 Hz); 4.72 (s, 1 H, C(4)H); 5.69 (br.s, 2 H, $NH_2$); 6.88—7.49 (m, 4 H, $H_{Ar}$); 9.07 (s, 1 H, NH).

**Tetraazatricyclo[7.3.1.0$^{2,7}$]tridec-2-ene-8-selenones 2. Method *A*.** A mixture of the corresponding selenolate **1** (2.3 mmol), a primary amine (6 mmol), and excess of the 37% aqueous formaldehyde (54 mmol, 4.0 mL) in EtOH (40 mL) was stirred for 10 min under argon, then refluxed until the starting reagents were dissolved (~2—3 min), rapidly filtered through a paper filter, and allowed to stand for 24 h at room temperature under argon. A precipitate formed was filtered off, washed with EtOH and hexane.

**Method *B*.** A mixture of benzaldehyde **3** (0.14 mL, 1.36 mmol), freshly prepared cyanoselenoacetamide **4** (0.20 g, 1.36 mmol), and morpholine (1 drop) in ethanol (15 mL) was stirred under argon at 20 °C. After 10 min, the corresponding primary amine (2.9 mmol) and excess of 37% aq. formaldehyde (2.0 mL, 27 mmol) were added, and the mixture was stirred for another 10 min. Then, the solution obtained was refluxed for 2—3 min, filtered through a folded paper filter, and allowed to stand for 24 h at room temperature under argon. A precipitate formed was filtered off and washed with EtOH and hexane.

**5,11,13-Triphenyl-8-selenoxo-3,5,7,11-tetraazatricyclo-[7.3.1.0$^{2,7}$]tridec-2-ene-1,9-dicarbonitrile (2a).** The yield was 0.15 g (12%, method *A*), 0.10 g (14%, method *B*), m.p. 218—220 °C (DMF). Found (%): C, 64.86; H, 4.54; N, 15.51. $C_{29}H_{24}N_6Se$. Calculated (%): C, 65.04; H, 4.52; N, 15.69. IR, ν/cm$^{-1}$: 2250 (2 C≡N); 1655 (C=N). $^1$H NMR, δ: 3.68, 3.84, 4.07, 4.29 (all d, 1 H each, C(10)$H_2$, C(12)$H_2$, $^2J$ = 11.7 Hz); 4.45 (s, 1 H, C(13)H); 4.85, 5.18 (both d, 1 H each, C(4)$H_2$, $^2J$ = 17.1 Hz); 5.41, 6.07 (both d, 1 H each, C(6)$H_2$, $^2J$ = 13.7 Hz); 6.80—7.34 (m, 15 H, 3 Ph).

**5,11-Dimethyl-13-phenyl-8-selenoxo-3,5,7,11-tetraazatricyclo[7.3.1.0$^{2,7}$]tridec-2-ene-1,9-dicarbonitrile (2b).** The yield was 0.22 g (23%, method *A*), 0.22 g (39%, method *B*), m.p. 200—202 °C (EtOH). Found (%): C, 55.31; H, 4.94; N, 20.13. $C_{19}H_{20}N_6Se$. Calculated (%): C, 55.48; H, 4.90; N, 20.43. IR, ν/cm$^{-1}$: 2255 (2 C≡N); 1655 (C=N). $^1$H NMR, δ: 2.41, 2.49 (both s, 3 H each, 2 Me); 2.89, 3.09, 3.31, 3.35 (all d, 1 H each, C(10)$H_2$, C(12)$H_2$, $^2J$ = 10.8 Hz); 4.21 (s, 1 H, C(13)H); 4.23, 4.46 (both d, 1 H each, C(4)$H_2$, $^2J$ = 17.2 Hz); 5.07, 5.54 (both d, 1 H each, C(6)$H_2$, $^2J$ = 12.7 Hz); 7.25—7.44 (m, 5 H, Ph).

**13-(2-Methoxyphenyl)-5,11-di(4-methylphenyl)-8-selenoxo-3,5,7,11-tetraazatricyclo[7.3.1.0$^{2,7}$]tridec-2-ene-1,9-dicarbonitrile (2c).** The yield was 0.67 g (49%, method *A*). Spectroscopic characteristics of the sample obtained were identical to those given in the work.[17]

**13-(2-Methoxyphenyl)-5,11-diphenyl-8-selenoxo-3,5,7,11-tetraazatricyclo[7.3.1.0$^{2,7}$]tridec-2-ene-1,9-dicarbonitrile (2d).** The yield was 0.47 g (36%, method *A*), m.p. 212—214 °C (EtOH). Found (%): C, 63.49; H, 4.68; N, 14.69. $C_{30}H_{26}N_6OSe$. Calculated (%): C, 63.71; H, 4.63; N, 14.86. IR, ν/cm$^{-1}$: 2250 (2 C≡N); 1655 (C=N). $^1$H NMR, δ: 3.74, 4.26 (both d, 1 H each, C(10)$H_2$ or C(12)$H_2$, $^2J$ = 11.6 Hz); 3.85 (s, 3 H, OMe); 3.88, 4.06 (both d, 1 H each, C(12)$H_2$ or C(10)$H_2$, $^2J$ = 11.8 Hz); 4.77 (s, 1 H, C(13)H); 4.88, 5.17 (both d, 1 H each, C(4)$H_2$, $^2J$ = = 17.4 Hz); 5.41, 6.12 (both d, 1 H each, C(6)$H_2$, $^2J$ = 13.4 Hz); 6.58—7.33 (m, 14 H, $H_{Ar}$).

**5,11-Dibenzyl-13-(2-methoxyphenyl)-8-selenoxo-3,5,7,11-tetraazatricyclo[7.3.1.0$^{2,7}$]tridec-2-ene-1,9-dicarbonitrile (2e).** The yield was 0.69 g (51%, method *A*), m.p. 190—192 °C (EtOH). Found (%): C, 64.59; H, 5.12; N, 14.96. $C_{32}H_{30}N_6OSe$. Calculated (%): C, 64.75; H, 5.09; N, 14.16. IR, ν/cm$^{-1}$: 2252 (2 C≡N); 1650 (C=N). $^1$H NMR, δ: 2.94, 3.12, 3.52, 3.78 (all d, 1 H each, C(10)$H_2$, C(12)$H_2$, $^2J$ = 10.7 Hz); 3.79—3.84 (m, 4 H, overlap of two signals C$\underline{H}_2$Ph); 3.89 (s, 3 H, OMe); 4.28, 4.44 (both d, 1 H each, C(4)$H_2$, $^2J$ = 17.2 Hz); 4.49 (s, 1 H, C(13)H); 5.24, 5.47 (both d, 1 H each, C(6)$H_2$, $^2J$ = 12.7 Hz); 6.99—7.42 (m, 14 H, $H_{Ar}$).

**13-(2-Methoxyphenyl)-5,11-dimethyl-8-selenoxo-3,5,7,11-tetraazatricyclo[7.3.1.0$^{2,7}$]tridec-2-ene-1,9-dicarbonitrile (2f).** The yield was 0.56 g (55%, method *A*). Spectroscopic characteristics of the sample obtained were identical to those given in the work.[17]

**13-(2-Ethoxyphenyl)-5,11-di(4-methylphenyl)-8-selenoxo-3,5,7,11-tetraazatricyclo[7.3.1.0$^{2,7}$]tridec-2-ene-1,9-dicarbonitrile (2g).** The yield was 0.56 g (40%, method *A*), m.p. 191—193 °C (BuOH). Found (%): C, 65.00; H, 5.38; N, 13.67. $C_{33}H_{32}N_6OSe$. Calculated (%): C, 65.23; H, 5.31; N, 13.83. IR, ν/cm$^{-1}$: 2248 (2 C≡N); 1655 (C=N). $^1$H NMR, δ: 1.48 (t, 3 H, $OCH_2C\underline{H}_3$, $^3J$ = 6.9 Hz); 2.15, 2.25 (both s, 3 H each, 2 Me); 3.57 (d, 1 H, C(12)$H_2$ or C(10)$H_2$, $^2J$ = 11.7 Hz); 3.71, 3.93 (both d, 1 H each, C(10)$H_2$ or C(12)$H_2$, $^2J$ = 11.5 Hz); 4.07—4.16 (m, 3 H, overlap of two signals C(12)H or C(10)H and $OC\underline{H}_2CH_3$); 4.64 (s, 1 H, C(13)H); 4.86, 5.05 (both d, 1 H each, C(4)$H_2$, $^2J$ = 17.3 Hz); 5.66, 5.89 (both d, 1 H each, C(6)$H_2$, $^2J$ = 13.6 Hz); 6.61—7.30 (m, 12 H, $H_{Ar}$).

**5,11-Dibenzyl-13-(2-ethoxyphenyl)-8-selenoxo-3,5,7,11-tetraazatricyclo[7.3.1.0$^{2,7}$]tridec-2-ene-1,9-dicarbonitrile (2h).** The yield was 0.52 g (37%, method *A*), m.p. 204—206 °C (EtOH). Found (%): C, 65.04; H, 5.35; N, 13.55. $C_{33}H_{32}N_6OSe$. Calculated (%): C, 65.23; H, 5.31; N, 13.83. IR, ν/cm$^{-1}$: 2250 (2 C≡N); 1650 (C=N). $^1$H NMR, δ: 1.50 (t, 3 H, $OCH_2C\underline{H}_3$, $^3J$ = 6.9 Hz);

2.91, 3.52 (both d, 1 H each, C(12)$H_2$ or C(10)$H_2$, $^2J$ = 10.9 Hz); 3.11, 3.55 (both d, 1 H each, C(10)$H_2$ or C(12)$H_2$, $^2J$ = 10.5 Hz); 3.75—3.84 (m, 4 H, overlap of two signals C$\underline{H}_2$Ph); 4.05—4.18 (m, 2 H, OC$\underline{H}_2$$CH_3$); 4.28, 4.44 (both d, 1 H each, C(4)$H_2$, $^2J$ = = 17.0 Hz); 4.49 (s, 1 H, C(13)H); 5.26, 5.44 (both d, 1 H each, C(6)$H_2$, $^2J$ = 12.9 Hz); 7.01—7.33 (m, 14 H, $H_{Ar}$).

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*Received October 25, 2012;*
*in revised form February 5, 2013*