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MANNICH REACTION IN THE SYNTHESIS OF N,S-CONTAINING HETEROCYCLES. 15*. MULTICOMPONENT CASCADE SYNTHESIS OF 3,4,6a,7,8,9,10,10a-OCTAHYDRO-2*H*,6*H*-PYRIMIDO[4',5':4,5]PYRIDO[2,1-*b*][1,3,5]THIA(SELENA)-DIAZINE DERIVATIVES

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The reaction of (2E,4E)-2-cyano-5-phenylpenta-2,4-dienethio(seleno)amides with formaldehyde and primary aromatic amines gives 3,8,10-trisubstituted 6-phenyl-3,4,6a,7,8,9,10,10a-octahydro-2H,6H-pyrimido[4',5':4,5]pyrido[2,1-b][1,3,5]thia(selena)diazine-11-carbonitriles. The latter were also obtained by a multicomponent condensation of cinnamaldehyde, cyanothioacetamide, formaldehyde, and anilines in the presence of catalytic amounts of base. The structure of the 3,8,10-tri(4-methylphenyl)-6-phenyl-3,4,6a,7,8,9,10,10a-octahydro-2H,6H-pyrimido[4',5':4,5]pyrido[2,1-b][1,3,5]thiadiazine-11-carbonitrile was studied by X-ray structural analysis.

Keywords: pyrimido[4',5',:4.5]pyrido[2,1-*b*][1,3,5]thia(selena)diazines, cyanoselenoacetamide, cyanothioacetamide, aminomethylation, multicomponent condensation, Mannich reaction, X-ray structural analysis.

It has previously been shown that treatment of 3-aryl-2-cyanoprop-2-enethio(seleno)amides **1** with primary amines and HCHO [2-4], and also in the course of recyclization of 4*H*-thio(seleno)pyrans **2** under Mannich reaction conditions [4, 5], during the aminomethylation of tetrahydropyridine-2-thiolates **3** [6], and as a result of a multicomponent condensation of aldehydes, cyanothio(seleno)acetamides **4a**,**b**, primary amines and HCHO [4, 5, 7] the same products are formed, *viz*. the 3,4,7,8-tetrahydro-2*H*,6*H*-pyrimido[4,3-*b*]-[1,3,5]thia(selena)diazine-9-carbonitriles **5**.

The nitriles **5** are of both applied and theoretical interest in view of the broad spectrum of practically important properties of 1,3,5-thiadiazine derivatives [8-10] on the one hand, and the poorly studied reactivity and methods of preparation of compounds with a 1,3,5-thiadiazine (and especially a 1,3,5-selenadiazine) fragment [4] on the other.

In continuation of our work in this field, we have studied the reaction of the unsaturated thio(seleno)-amides **1**, *viz.* the (2*E*,4*E*)-2-cyano-5-phenylpenta-2,4-dienethio(seleno)amides **6a**,**b** with formaldehyde and primary aromatic amines. The cinnamamides **6a**,**b** are readily formed by the Michael reaction of

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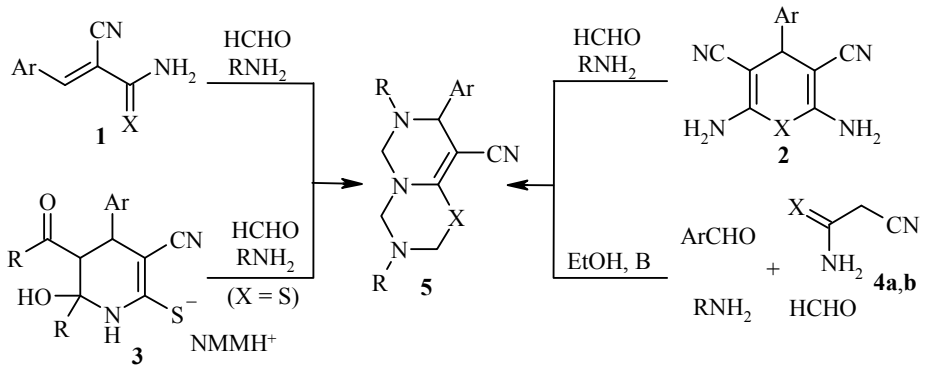
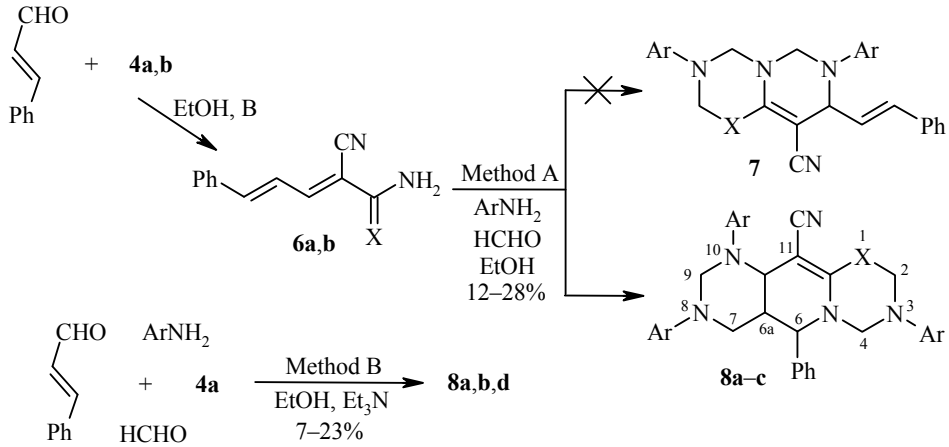


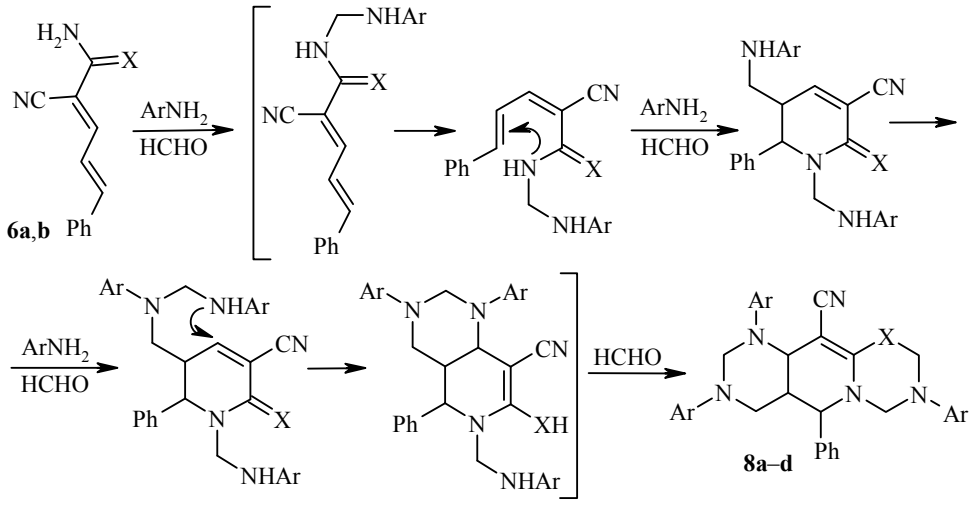
X = S, Se; $NMMH^+$ = *N*-methylmorpholinium; B = Et_3N or *N*-methylmorpholine

cinnamaldehyde and cyanothio(seleno)acetamides **4a**,**b**. It was found that the cinnamamides **6a**,**b** undergo the Mannich reaction with an excess of formalin and $ArNH_2$. Surprisingly, 3,4,6a,7,8,9,10,10a-octahydro-2*H*,6*H*-pyrimido[4',5':4,5]pyrido[2,1-*b*][1,3,5]thia(selena)diazine derivatives **8a-c** were obtained as the condensation products of cinnamamides **6a**,**b**, primary aromatic amines, and HCHO in the ratio of 1:3:4 instead of the expected pyrimido[4,3-*b*][1,3,5]thia(selena)diazines **7** (method A). Compounds **8a**,**b**,**d** were also synthesized in comparable yields by the alternative method B through condensation of cyanothioacetamide **4a** with cinnamaldehyde in the presence of catalytic amounts of Et_3N followed by boiling with an excess of HCHO and 3.1 equivalents of aniline. It was found that the multicomponent route B generally leads to the formation of less pure products. Several limitations determined by the structure of the amine component were also revealed. Hence we were unable to obtain the corresponding compound **8** in the case of *N*-acetyl-*para*-phenylenediamine and 4-aminobenzenesulfamide. In this case a tar was obtained, containing a large fraction of unidentified impurities according to HPLC-MS data.



4, **6 a** X = S, **b** X = Se; **8 a** X = S, Ar = 4-MeC_6H_4; **b** X = S, Ar = Ph; **c** X = Se, Ar = 4-MeC_6H_4; **d** X = S, Ar = 4-$MeOC_6H_4$; B = Et_3N or *N*-methylmorpholine

Clearly a key part of the synthesis of compounds **8** is the formation of the N(5)–C(6) bond, which is a variation of an intramolecular aza-Michael reaction. Similar *6-endo-trig* processes leading to formation of pyridine derivatives are known [11-14], but previously unreported for cinnamothio(seleno)amides. Clarification of mechanistic details and optimization of the cascade process conditions will be the subject of further investigation.



Compounds **8a-d** are colorless or variously yellow tinged crystals, insoluble in hot EtOH but soluble in acetone, DMSO and hot DMF. Stretching vibration absorptions corresponding to free N–H groups are absent in the IR spectra of compounds **8a-d**, but they show the presence of strong bands at 2174-2176 cm^{-1} (a conjugated C≡N group). The ^{1}H NMR spectra of compounds **8a-d** show signals for the protons of one phenyl group and three aryl substituents from the $ArNH_2$ as well as a complex set of signals for the 11 protons of the tricyclic ring.

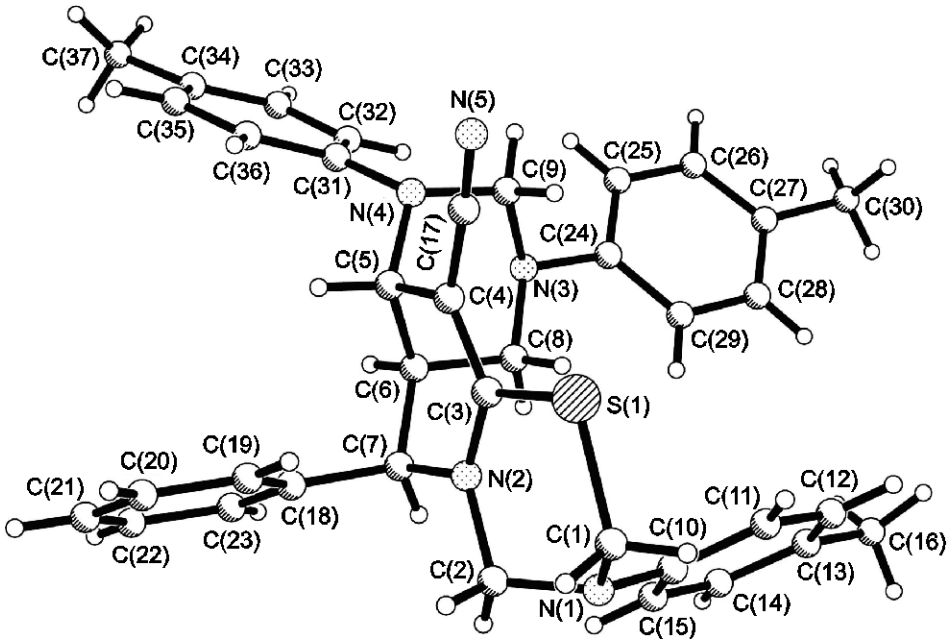


Fig. 1. Molecular structure of compound **8a** from X-ray analytical data.

The decisive answer to the question of the structure of compounds **8a-d** came from an X-ray structural investigation (Fig. 1) of the 3,8,10-tri(4-methylphenyl)-6-phenyl-3,4,6a,7,8,9,10,10a-octahydro-2*H*,6*H*-pyrimido-[4',5':4,5]pyrido[2,1-*b*][1,3,5]thiadiazine-11-carbonitrile **8a**. The tetrahydropyridine ring occurs in a "sofa" conformation with the atom C(6) deviating by 0.70 Å from the plane of the remaining atoms. The conformation of the 1,3,5-thiadiazine ring can be regarded as an asymmetric "half chair" and is characterized by a deviation of N(1) and C(2) atoms from the plane of the remaining ring atoms by 0.55 and -0.23 Å, respectively. The perhydropyrimidine ring has a "chair" conformation with absolute values of the endocyclic dihedral angles in

the range of 51.75(17)-59.66(17)º and with a *cis* type fusion to the tetrahydropyridine ring (dihedral angle H(5)–C(5)–C(6)–H(6) 52º). The nitrogen atoms N(1), N(3), and N(4) have a pyramidal configuration, the sums of the valence angles centered on these atoms being 344.5(4), 335.5(4), and 345.6(4)º, respectively. The orientation of the tolyl substituents relative to the idealized positions for the lone electron pairs (Lp) of these nitrogen atoms points to the presence of π-conjugation between them (torsion angles Lp(N1)–N(1)–C(10)–C(15) 76º, LpN(3)–N(3)–C(24)–C(25) 54º, and LpN(4)–N(4)–C(31)–C(36) 52º).

It should be noted that, in spite of several differences in the degree of the pyramidal nature of the N(1), N(3), and N(4) nitrogen atoms and the angles between the aromatic π-system of the substituent and the lone electron pairs of the nitrogen atoms, the lengths of the N(sp^3)–C(Ar) bonds are virtually the same (N(1)–C(10) 1.431(2), N(3)–C(24) 1.434(2), and N(4)–C(31) 1.428(2) Å). Steric hindrance arises because of this type of orientation of the benzene rings, and this is reflected in the appearance of shortened intramolecular contacts between the hydrogen atoms at the *ortho* position of the aromatic ring as well as and the methylene groups neighboring the nitrogen atom (H(11)···H(1a) 2.07 Å, H(15)···H(2b) 2.01 Å, H(29)···H(8a) 2.28 Å, H(29)···H(8b) 2.29 Å, H(25)···H(9b) 2.25 Å, H(32)···H(9b) 2.27 Å, and H(36)···H(5) 2.25 Å, while the sum of the van der Waals radii is 2.32 Å [15]). The N(3) tolyl group has an equatorial conformation, but the substituents at atoms N(1), N(4), and C(7) are axial (dihedral angles C(24)–N(3)–C(9)–N(4) 172.15(13)º, C(10)–N(1)–C(1)–S(1) 76.00(18)º, C(31)–N(4)–C(5)–C(6) 87.24(16)º, and C(2)–N(2)–C(7)–C(18) 77.34(18)º).

In summary, the reactions of (2*E*,4*E*)-2-cyano-5-phenylpenta-2,4-dienethio(seleno)amides with HCHO and primary aromatic amines give derivatives of the novel heterocyclic system – pyrimido[4',5':4,5]pyrido[2,1-*b*]-[1,3,5]thia(selena)diazine-11-carbonitrile. These same products are formed in the course of a cascade cyclocondensation of cyanothio(seleno)acetamide, cinnamaldehyde, primary amines, and HCHO. The structure of the compounds obtained was confirmed by the results of X-ray structural investigation of 3,8,10-tri-(4-methylphenyl)-6-phenyl-3,4,6a,7,8,9,10,10a-octahydro-2*H*,6*H*-pyrimido[4',5':4,5]pyrido[2,1-*b*][1,3,5]thiadi-azine-11-carbonitrile.

EXPERIMENTAL

IR spectra were recorded on an IKS-29 spectrophotometer in nujol mulls. The ^{1}H NMR spectra were recorded on a Varian Unity Plus instrument (400 MHz, compounds **6b**, **8c**,**d**) or on a Bruker DRX-500 instrument (500 MHz, compounds **8a**,**b**) using DMSO-d_6 with TMS as internal standard. Elemental analysis was carried out on a Carlo-Erba 1106 Elemental Analyzer with a measurement accuracy of ±0.25%. Melting points were determined on a Koffler hot stage apparatus and are not corrected. HPLC-MS analysis of compound **8d** was carried out on a Shimadzu LC-10AD liquid chromatograph using Shimadzu SP D-10A UV-Vis (254 nm) and Sedex 75 ELSD detectors together with a PE SCIEX API 150EX mass spectrometer. Monitoring of the purity of the compounds prepared was carried out by TLC on Silufol UV-254 plates with acetone–hexane (1:1) as eluent and visualized with iodine vapor and UV detection. The starting cyanothioacetamide (**4a**) [16] and cyanoselenoacetamide (**4b**) [17] were prepared by known methods.

(2*E*,4*E*)-2-Cyano-5-phenylpenta-2,4-dienethioamide (6a) was prepared by modification of method [18] without heating the reaction mixture. Brick-red, finely crystalline powder; mp 153-155ºC (mp 149ºC (EtOH) [18], 158-160ºC (C_6H_6) [19]). The spectroscopic parameters agreed with those given in the literature [18].

(2*E*,4*E*)-2-Cyano-5-phenylpenta-2,4-dieneselenoamide (6b). Cinnamaldehyde (0.86 ml, 6.84 mmol) and *N*-methylmorpholine (1 drop) were added to a suspension of the cyanoselenoacetamide (**4b**) (1.0 g, 6.8 mmol) in EtOH (4 ml). The mixture was stirred and slowly heated to about 50ºC under an argon stream until dissolution of the selenoamide **4b**. After about 5 min crystallization started. The mixture was stirred for 3 h at 20ºC under an argon stream, filtered, and washed with EtOH and ether. Yield 1.37 g (77%). Bordeaux colored powder; mp 123-125ºC (EtOH). The product was used in further reactions without additional purification. IR

spectrum, ν, cm^{-1}: 3330 (NH_2), 2224 (C≡N). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.13 (1H, dd, ^3J = 11.3, ^3J = 11.5, 4-CH); 7.46-7.60 (4H, m, 3-CH, H Ph); 7.71-7.73 (2H, m, H Ph); 8.01 (1H, d, ^3J = 11.3, 5-CH); 10.13 (1H, br. s) and 10.85 (1H, br. s, $CSeNH_2$). Found, %: C 55.46; H 3.96; N 10.61. $C_{12}H_{10}N_2Se$. Calculated, %: C 55.18; H 3.86; N 10.73.

Preparation of Pyrimido[4',5':4,5]pyrido[2,1-*b*][1,3,5]thia(selena)diazine-11-carbonitriles 8a-c from Cinnamamides 6a,b (General Method A). A suspension of cinnamamide **6a**,**b** (2.0 mmol) in EtOH (20 ml) was treated with a primary aromatic amine (7 mmol) and excess of 37% formaldehyde free of paraformaldehyde (2-3 ml, 26.6-40.0 mmol). The mixture was refluxed with stirring for 3-4 min, stirred for 2-3 h at 20ºC, and left overnight. The tarry residue was filtered off, washed several times with boiling EtOH, and recrystallized from a suitable solvent.

3,8,10-Tri(4-methylphenyl)-6-phenyl-3,4,6a,7,8,9,10,10a-octahydro-2*H*,6*H*-pyrimido[4',5':4,5]pyrido-[2,1-*b*][1,3,5]thiadiazine-11-carbonitrile (8a). Yield 0.33 g (28%). Light-yellow crystals; mp 201-203ºC (decomp., DMF). IR spectrum, ν, cm^{-1}: 2174 (C≡N). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.12 (3H, s, CH_3); 2.24 (3H, s, CH_3); 2.25 (3H, s, CH_3); 2.32-2.38 (1H, m, 6a-CH); 3.33-3.36 (1H, m) and 3.57 (1H, dd, ^2J = 8.9, ^3J = 3.6, 7-CH_2); 3.78 (1H, d, ^2J = 12.7, 9-CH_A); 3.93 (1H, d, ^3J = 4.9, 10a-CH); 4.64 (1H, br. s, 6-CH); 4.87-4.90 (2H, m, 4-CH_A, 9-CH_B); 5.02 (1H, d, ^2J = 13.2, 4-CH_B); 5.26 (1H, d, ^2J = 12.8) and 5.41 (1H, d, ^2J = 12.8, 2-CH_2); 6.68 (2H, d, ^3J = 8.3, H Ar); 6.85 (2H, d, ^3J = 8.3, H Ar); 6.90 (2H, d, ^3J = 8.3, H Ar); 7.08 (2H, d, ^3J = 8.3, H Ar); 7.13-7.17 (4H, m, H Ar); 7.25 (2H, d, ^3J = 7.8, H-2,6 Ph); 7.33 (1H, t, ^3J = 7.4, H-4 Ph); 7.41 (2H, dd, ^3J = 7.4, ^3J = 7.8, H-3,5 Ph). Found, %: C 75.96; H 6.40; N 12.09. $C_{37}H_{37}N_5S$. Calculated, %: C 76.12; H 6.39; N 12.00.

3,6,8,10-Tetraphenyl-3,4,6a,7,8,9,10,10a-octahydro-2*H*,6*H*-pyrimido[4',5':4,5]pyrido[2,1-*b*][1,3,5]thia-diazine-11-carbonitrile (8b). Yield 0.13 g (12%). Yellow-orange crystals; mp 189-192ºC (decomp., DMF). IR spectrum, ν, cm^{-1}: 2175 (C≡N). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.27-2.32 (1H, m, 6a-CH); 3.31-3.36 (1H, m) and 3.69 (1H, dd, ^2J = 12.0, ^3J = 3.7, 7-CH_2); 3.88 (1H, d, ^2J = 12.7, 9-CH_A); 4.04 (1H, d, ^3J = 4.9, 10a-CH); 4.69 (1H, br. s, 6-CH); 4.94 (1H, d, ^2J = 13.7, 4-CH_A); 5.07-5.10 (2H, two overlapping doublets: d, ^2J = 13.7, 4-CH_B and d, ^2J = 12.7, 9-CH_B); 5.32 (1H, d, ^2J = 12.7) and 5.45 (1H, d, ^2J = 12.8, 2-CH_2); 6.71 (1H, t, ^3J = 7.3, H Ph); 6.76 (2H, d, ^3J = 8.3, H Ph); 6.86 (1H, t, ^3J = 7.3, H Ph); 6.97-7.01 (3H, m, H Ph); 7.10 (2H, dd, ^3J = 7.3, ^3J = 7.8, H Ph); 7.25-7.35 (9H, m, H Ph); 7.40-7.43 (2H, m, H Ph). Found, %: C 75.16; H 5.85; N 13.05. $C_{34}H_{31}N_5S$. Calculated, %: C 75.39; H 5.77; N 12.93.

3,8,10-Tri(4-methylphenyl)-6-phenyl-3,4,6a,7,8,9,10,10a-octahydro-2*H*,6*H*-pyrimido[4',5':4,5]pyrido-[2,1-*b*][1,3,5]selenadiazine-11-carbonitrile (8c). Yield 0.32 g (25%). Light-brown, finely crystalline powder; decomp. temp. > 160ºC (DMF). IR spectrum, ν, cm^{-1}: 2176 (C≡N). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.15 (3H, s, CH_3); 2.22-2.32 (1H, m, 6a-CH); 2.26 (3H, s, CH_3); 2.27 (3H, s, CH_3); 3.03-3.06 (1H, m) and 3.35 (1H, br. d, ^2J = 8.8, 7-CH_2); 3.85 (1H, d, ^2J = 12.7) and 4.75 (1H, d, ^2J = 12.7, 9-CH_2); 3.90 (1H, d, ^3J = 2.4, H-10a); 4.53 (1H, br. s, H-6); 4.82 (1H, d, ^2J = 13.7) and 4.97 (1H, d, ^2J = 13.7, 4-CH_2); 5.21 (1H, d, ^2J = 10.8) and 5.46 (1H, d, ^2J = 10.8, 2-CH_2); 6.67 (2H, d, ^3J = 8.0, H Ar); 6.71 (2H, d, ^3J = 8.0, H Ar); 6.85 (2H, d, ^3J = 8.0, H Ar); 7.00 (2H, d, ^3J = 8.0, H Ar); 7.08-7.13 (4H, m, H Ar); 7.20 (2H, d, ^3J = 7.5, H-2,6 Ph); 7.28 (1H, t, ^3J = 7.3, H-4 Ph); 7.36 (2H, dd, ^3J = 7.3, ^3J = 7.5, H-3,5 Ph). Found, %: C 70.23; H 6.00; N 11.21. $C_{37}H_{37}N_5Se$. Calculated, %: C 70.46; H 5.91; N 11.10.

Preparation of Pyrimido[4',5':4,5]pyrido[2,1-*b*][1,3,5]thiadiazine-11-carbonitriles 8a,b,d by Multicomponent Condensation (General Method B). Et_3N (5 drops) was added to a solution of the cyanothioacetamide (**4a**) (0.5 g, 5 mmol) and cinnamaldehyde (0.63 ml, 5 mmol) in EtOH (10 ml) and stirred for 15 min. Then additional EtOH (10 ml), a primary aromatic amine (3.1 equiv., 15.5 mmol), and 37% formaldehyde free of paraformaldehyde (4.5 ml, 60 mmol) were added in succession. The solution obtained was refluxed for 2-3 min with vigorous stirring, stirred at 20ºC for 3-4 h, and left overnight. The tarry precipitate was separated, treated with EtOH, filtered, washed twice with boiling EtOH, and recrystallized.

Compound 8a. Yield 0.53 g (18%); mp 201-203ºC (decomp., DMF–EtOH). The spectral parameters were identical to those of a sample obtained using method A.

Compound 8b. Yield 0.19 g (7%); mp 188-190ºC (decomp., DMF). The spectral parameters were identical to those of a sample obtained using method A.

3,8,10-Tri(4-methoxyphenyl)-6-phenyl-3,4,6a,7,8,9,10,10a-octahydro-2*H*,6*H*-pyrimido-[4',5':4,5]-pyrido[2,1-*b*][1,3,5]thiadiazine-11-carbonitrile (8d). Yield 0.73 g (23%). Colorless, finely crystalline powder; mp 191-193ºC (decomp., EtOH–acetone 1:1). IR spectrum, ν, cm^{-1}: 2175 (C≡N). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.23-2.31 (1H, m, 6a-CH); 3.30-3.33 (1H, m) and 3.39 (1H, br. d, ^2J = 8.1, 7-CH_2); 3.61 (3H, s, OCH_3); 3.71-3.74 (7H, m, 2OCH_3, 9-CH_A); 3.79 (1H, d, ^3J = 2.7, 10a-CH); 4.54 (1H, br. s, 6-CH); 4.68 (1H, d, ^2J = 12.6, 9-CH_B); 4.84 (1H, d, ^2J = 13.2) and 4.99 (1H, d, ^2J = 13.2, 4-CH_2); 5.20 (1H, d, ^2J = 12.4) and 5.39 (1H, d, ^2J = 12.4, 2-CH_2); 6.70 (2H, d, ^3J = 8.9, H Ar); 6.80 (2H, d, ^3J = 8.9, H Ar); 6.83-6.86 (4H, m, H Ar); 6.90 (2H, d, ^3J = 8.6, H Ar); 7.19-7.23 (4H, m, H Ar); 7.29-7.32 (1H, m, H-4 Ph); 7.38 (2H, dd, ^3J = 7.0, ^3J = 7.5, H-3,5 Ph). Mass spectrum, *m/z* (I_{rel}, %): 632.3 $[M+H]^+$, 497.3 $[M+H\text{-}ArN{=}CH_2]^+$, 362.1 $[M+H\text{-}2ArN{=}CH_2]^+$, 228.4 $[M+H\text{-}3ArN{=}CH_2]^+$. Found, %: C 70.20; H 5.99; N 11.17. $C_{37}H_{37}N_5O_3S$. Calculated, %: C 70.34; H 5.90; N 11.08.

X-ray Structural Investigation of Compound 8a. Crystals of compound **8a** are monoclinic, $C_{37}H_{37}N_5S$, at 298 K: *a* 10.5638(7), *b* 9.2115(7), *c* 32.074(2) Å; β 92.996(6)º, *V* 3116.8(4) $Å^3$, *M*=583.78; *Z* 4; space group $P2_1/c$; d_{calc} 1.244 g/cm^3; μ(MoKα) 0.138 mm^{-1}; *F*(000) 1240. The unit cell parameters and intensities of 23684 reflections (9913 independent, R_{int} 0.025) were measured on an Xcalibur 3 automatic, four circle diffractometer (MoKα radiation, graphite monochromator, CCD detector, ω-scanning to $2\theta_{max}$ 63.6º). The structure was solved by the direct method using the SHELX-97 software package [20]. The position of the hydrogen atoms was calculated geometrically and refined using the "riding" model with $U_{iso} = nU_{eq}$ for the attached atom (n = 1.5 for methyl groups and n = 1.2 for remaining hydrogen atoms). The structure was refined in F^2 full-matrix least-squares analysis in the anisotropic approximation for non-hydrogen atoms to wR_2 0.151 for 9913 reflections (R_1 0.060 for 5024 reflections with $F > 4\sigma(F)$, *S* 1.02). The full crystallographic data has been placed at the Cambridge Crystallographic Data Center as deposit CCDC 874747.

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