ISSN 1070-3632, Russian Journal of General Chemistry, 2015, Vol. 85, No. 5, pp. 1069–1073. © Pleiades Publishing, Ltd., 2015.
Original Russian Text © V.D. Dyachenko, T.A. Ryl'skaya, I.V. Dyachenko, I.N. Kalashnik, A.V. Chernykh, 2015, published in Zhurnal Obshchei Khimii, 2015, Vol. 85, No. 5, pp. 771–775.

Three-Component Synthesis of Nicotinamide Derivatives Based on Cross-Recyclization of 2,6-Diamino-4-aryl-3,5-dicyano-4*H*-thiopyrans with Acetoacetanilide and Alkylating Reagents

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Received February 2, 2015

Abstract—Three-component reaction of 2,6-diamino-4-aryl-3,5-dicyano-4*H*-thiopyrans, acetoacetanilides, and alkylating reagents proceeds via cross-recyclization of thiopyrans to form 6-alkylthio-4-aryl-*N*-aryl-2-methyl-5-cyanonicotinamides, 3-imino-6-methyl-2-(4-methoxybenzoyl)-4-(3,4-dimethoxyphenyl)-*N*-phenyl-2,3-dihydrothieno[2,3-*b*]pyridine-5-carboxamide, and 3-amino-6-methyl-4-(3,4-dimethoxyphenyl)-2-(4-oxo-4*H*-pyrido[1,2-*a*]pyrimidin-2-yl)-*N*-phenylthieno[2,3-b]pyridine-5-carboxamide.

Keywords: 4*H*-thiopyran, cross-recyclization, acetoacetanilide, substituted nicotinamides, thieno[2,3-*b*]pyridine-5-carboxamide.

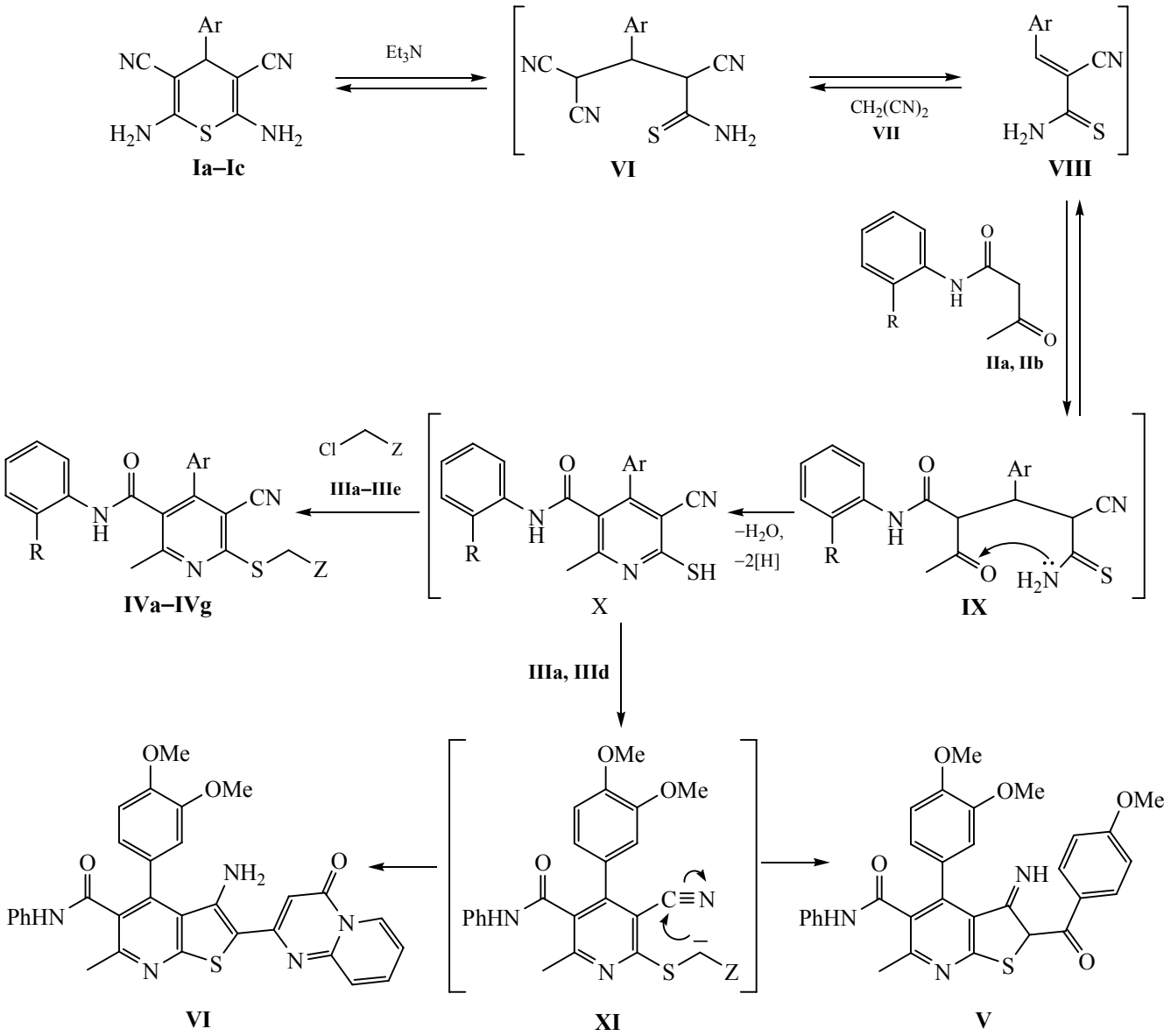
DOI: 10.1134/S1070363215050114

Search for biologically active nicotinamide derivatives is a promising to develop herbicides [1, 2] and drugs for Alzheimer's disease treatment [3, 4].

Studies on preparation of functionally substituted nicotinamide derivatives [5–8] showed that cross-recyclization of 2,6-diamino-4-aryl-3,5-dicyano-4*H*-thiopyrans **Ia**–**Ic** with acetoacetanilides **IIa** and **IIb** in refluxing ethanol in the presence of an equimolar quantity of triethylamine followed by addition of the alkylating reagent **IIIa**–**IIIe** afforded new nicotinamide derivatives such as 6-alkylthio-4,*N*-diaryl-2-methyl-5-cyanonicotinamides **IVa**–**IVg**, 3-imino-6-methyl-2-(4-methoxybenzoyl)-4-(3,4-dimethoxyphenyl)-*N*-phenyl-2,3-dihydrothieno[2,3-*b*]pyridine-5-carboxamide **V** and 3-amino-6-methyl-4-(3,4-dimethoxyphenyl)-2-(4-oxo-4*H*-pyrido[1,2-*a*]pyrimidin-2-yl)-*N*-phenylthieno-[2,3-*b*]pyridine-5-carboxamide **VI** (Scheme 1).

Apparently, the reaction pathway included opening of the thiopyran ring to form intermediate **VI** that was further transformed into arylmethylidenecyanothioacetamide **VIII** via malononitrile **VII** elimination [9–11]. The Michael addition of acetoacetanilide **II** to arylmethylidenecyanothioacetamide **VIII** led to formation of the corresponding labile adduct **IX**, which underwent intramolecular heterocyclization to give the substituted pyridine **X**. The latter was subject to regioselective alkylation at the sulfur atom to form the corresponding thioethers **IVa**–**IVg**, in accordance with fundamentals of 2-mercaptopyridine chemistry [12, 13]. At the same time, the cross-recyclization of thiopyran **Ia**, acetoacetanilide **IIa**, and 4-methoxyphenacylchloride **IIIa** yielded unknown 3-imino-6-methyl-2-(4-methoxybenzoyl)-4-(3,4-dimethoxyphenyl)-*N*-phenyl-2,3-dihydrothieno[2,3-*b*]pyridine-5-carboxamide **V** instead of the expected thioether **XI**, due to rapid intramolecular cyclization of intermediate **XI**. Noteworthily, formation of the substituted thieno[2,3-*b*]pyridines of that type generally does not stop at the stage of the imine **V** formation due to spontaneous transformation of the latter into the amine [14–16]. That reaction pathway was realized when 4-oxo-2-chloromethyl-4*H*-pyrido[1,2-*a*]pyrimidine **IIId** was used as an alkylating agent. As a result, 3-amino-6-methyl-4-(3,4-dimethoxyphenyl)-2-(4-oxo-4*H*-pyrido [1,2-*a*]pyrimidin-2-yl)-*N*-phenylthieno[2,3-*b*]pyridine-5-carboxamide **VI** was obtained.

Scheme 1.



I, Ar = Ph (**a**), 3-FC_6H_4 (**b**), 3,4-$(MeO)_2C_6H_3$ (**c**); **II**, R = H (**a**), Me (**b**); **III**, Z = 4-$MeOC_6H_4CO$ (**a**), 4-$MeOC_6H_4NHCO$ (**b**), 4-oxo-4,5,6,7-tetrahydro-3*H*-cyclopenta[4,5]thieno[2,3-*b*]pyrimidin-2-yl (**c**), 4-oxo-4*H*-pyrido[1,2-*a*]pyrimidin-2-yl (**d**), 2-phenyloxazol-4-yl (**e**); **IV**, R = Me, Ar = 3-FC_6H_4, Z = 4-$MeOC_6H_4CO$ (**a**), R = H, Ar = 3,4-$(MeO)_2C_6H_3$, Z = 4-$MeOC_6H_4NHCO$ (**b**); R = H, Ar = Ph, Z = 4-oxo-4,5,6,7-tetrahydro-3*H*-cyclopenta[4,5]thieno[2,3-*b*]pyrimidin-2-yl (**c**); R = Me, Ar = 3-FC_6H_4, Z = 4-oxo-4,5,6,7-tetrahydro-3*H*-cyclopenta[4,5]thieno[2,3-*b*]pyrimidin-2-yl (**d**); R = H, Ar = Ph, Z = 4-oxo-4*H*-pyrido[1,2-*a*]pyrimidin-2-yl (**e**); R = Me, Ar = 3-FC_6H_4, Z = 2-phenyloxazol-4-yl (**f**); R = H, Ar = 3,4-$(MeO)_2C_6H_3$, Z = 2-phenyloxazol-2-yl (**g**).

The IR and NMR spectroscopy data confirmed the structures of compounds **IVa**–**IVg**, **V**, and **VI**. For example, the IR spectra contained characteristic absorption bands of the conjugated cyano group (2212–2226 cm^{-1}), amide moiety (1675–1684 cm^{-1}), and carbonyl group (1704–1718 cm^{-1}) stretching. Existence of **V** in the imine form was concluded on the basis of the absence of the conjugated cyano group stretching band in the IR spectrum. At the same time the absorption band of the imino moiety stretching was observed at 1625 cm^{-1}, coinciding with the reference data [17]. In addition, ^1H NMR spectrum of compound

V contained the singlet signal of C^2H proton at 3.98 ppm along with the proton signals of thienopyridine fragment and imino moiety (6.76 ppm).

^{13}C NMR spectra of compounds **IVa–IVf** and **VI** contained signals of all structural fragments of the suggested structures at the expected positions.

EXPERIMENTAL

IR spectra of KBr pellets were recorded using an UR-20 spectrophotometer. ^1H and ^{13}C NMR spectra of solutions in DMSO-d_6 were registered with a Varian-Gemini spectrometer (500.13 and 125.75 MHz, respectively) relative to TMS as internal reference. Elemental analysis was performed using an EuroVector EA-3000 microanalyzer. GC–MS spectra were obtained with an Agilent 1100/DAD/HSD/VLG 119562 device. Melting points were determined with a Kofler bench. The reaction progress was monitored by TLC on Silufol UV-254 plates eluting with an acetone–hexane mixture (3 : 5) and developing with iodine vapor or UV irradiation.

2,6-Diamino-4-phenyl-3,5-dicyano-4*H*-thiopyran **Ia**, 2,6-diamino-4-(3-fluorophenyl)-3,5-dicyano-4*H*-thiopyran **Ib** and 2,6-diamino-4-(3,4-dimethoxyphenyl)-3,5-dicyano-4*H*-thiopyran **Ic** were obtained by the procedures described in Refs. [18–20], respectively.

6-Alkylthio-4,*N*-diaryl-2-methyl-5-cyanonicotinamides (IVa–IVg), 3-imino-6-methyl-2-(4-methoxybenzoyl)-4-(3,4-dimethoxyphenyl)-*N*-phenyl-2,3-dihydrothieno[2,3-*b*]pyridine-5-carboxamide (V), and 3-amino-6-methyl-4-(3,4-dimethoxyphenyl)-2-(4-oxo-4*H*-pyrido[1,2-*a*]pyrimidine-2-yl)-*N*-phenylthieno[2,3-*b*]pyridine-5-carboxamide (VI) (*general procedure*). A mixture of 10 mmol of thiopyran **I**, 10 mmol of acetoacetanilide **II**, and 1.4 mL (10 mmol) of triethylamine in 50 mL of ethanol was refluxed during 5 h. After cooling to 20°C, 5.6 mL (10 mmol) of 10% aqueous KOH and a solution of 10 mmol of alkyl chloride **III** in 25 mL of DMF were added to the reaction mixture upon stirring. The mixture was stirred during 2 h, incubated during 48 h, and then diluted with 25 mL of water. The precipitate was filtered off, washed with water, ethanol, and hexane, and dried.

2-Methyl-6-[(2-(4-methoxyphenyl)-2-oxoethyl)thio]-*N*-(*o*-tolyl)-4-(3-fluorophenyl)-5-cyanonicotinamide (IVa). Yield 4.5 g (85%), colorless crystals, mp 204–206°C (BuOH). IR spectrum, ν, cm^{-1}: 3338 (NH), 2226 (C≡N), 1718 (C=O), 1684 (CONH). ^1H NMR spectrum, δ, ppm: 1.87 s (3H, Me), 2.37 s (3H, Me), 3.88 s (3H, MeO), 4.91 s (2H, CH_2), 6.98 d (1H, H_{arom}, *J* 6.8 Hz), 7.06–7.21 m (5H, H_{arom}), 7.28–7.42 m (3H, H_{arom}), 7.57–7.62 m (1H, H_{arom}), 8.11 d (2H, H_{arom}, *J* 8.8 Hz), 9.85 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 12.38, 32.63, 37.69, 55.69, 103.32, 114.10 (2C), 114.77, 115.79, 115.97, 116.65, 116.82, 125.06, 126.06, 128.99, 129.22, 130.48, 130.75, 130.87, 130.94, 132.27, 135.07, 149.70, 158.14, 160.67, 160.83, 162.62, 163.47, 163.82, 191.62. Mass spectrum, *m/z* (I_{rel}, %): 526 (100) $[M + 1]^+$. Found, %: C 68.47; H 4.55; N 7.86. $C_{30}H_{24}FN_3O_3S$. Calculated, %: C 68.56; H 4.60; N 7.99. *M* 525.606.

2-Methyl-4-(3,4-dimethoxyphenyl)-6-[2-(4-methoxyphenylamino)-2-oxoethylthio]-*N*-phenyl-5-cyanonicotinamide (IVb). Yield 4.3 g (76%), colorless crystals, mp 208–210°C (BuOH). IR spectrum, ν, cm^{-1}: 3390 (NH), 2212 (C≡N), 1675 (CONH). ^1H NMR spectrum, δ, ppm: 2.52 s (3H, Me), 3.65 s (3H, MeO), 3.72 s (3H, MeO), 3.76 s (3H, MeO), 4.22 s (2H, CH_2), 6.89 d (2H, H_{arom}, *J* 7.9 Hz), 7.04–7.12 m (5H, H_{arom}), 7.27 s (1H, H_{arom}), 7.43 d (2H, H_{arom}, *J* 7.2 Hz), 7.51 d (2H, H_{arom}, *J* 7.8 Hz), 10.20 br.s (1H, NH), 10.45 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 22.83, 35.04, 55.22, 55.48, 55.53, 103.61, 111.56, 112.16, 114.00 (2C), 115.22, 119.46 (2C), 120.79 (2C), 121.49, 124.18, 126.14, 128.90 (2C), 129.15, 132.23, 138.32, 148.24, 149.86, 151.02, 155.39, 157.86, 161.35, 164.07, 165.38. Mass spectrum, *m/z* (I_{rel}, %): 569 (100) $[M + 1]^+$. Found, %: C 65.33; H 4.85; N 9.78. $C_{31}H_{28}N_4O_5S$. Calculated, %: C 65.48; H 4.96; N 9.85. *M* 568.657.

2-Methyl-6-[(4-oxo-4,5,6,7-tetrahydro-3*H*-cyclopenta[4,5]thieno[2,3-*d*]pyrimidin-2-yl)methylthio]-*N*,4-diphenyl-5-cyanonicotinamide (IVc). Yield 3.7 g (68%) colorless powder, mp 285–287°C (DMF). IR spectrum, ν, cm^{-1}: 3338, 3374 (NH), 2220 (C≡N), 1679 (CONH). ^1H NMR spectrum, δ, ppm: 2.36 t (2H, CH_2, *J* 6.7 Hz), 2.56 s (3H, Me), 2.83– 2.94 m (4H, $2CH_2$), 4.58 s (2H, SCH_2), 7.04 t (1H, H_{arom}, *J* 7.3 Hz), 7.24 t (2H, H_{arom}, *J* 7.6 Hz), 7.33 d (2H, H_{arom}, *J* 7.6 Hz), 7.45 br.s (5H, Ph), 10.36 br.s (1H, NH), 12.69 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 22.77, 27.49, 28.67, 29.03, 32.69, 103.63, 114.80, 118.60, 119.71 (2C), 124.25, 128.40, 128.50 (2C), 128.83, 129.31, 129.78, 133.97, 137.40, 138.06, 139.54, 144.25, 151.45, 153.39, 157.87, 158.25, 160.56, 163.63, 167.93, 186.62. Mass spectrum, *m/z* (I_{rel}, %): 548 (100) $[M - 1]^+$. Found, %: C 65.44; H 4.17; N 12.60. $C_{30}H_{23}N_5O_2S_2$. Calculated, %: C 65.56; H 4.22; N 12.74. *M* 549.679.

2-Methyl-6-[(4-oxo-4,5,6,7-tetrahydro-3*H*-cyclopenta[4,5]thieno[2,3-*d*]pyrimidin-2-yl)methylthio]-*N*-(*o*-tolyl)-4-(3-fluorophenyl)-5-cyanonicotinamide (IVd). Yield 4.2 g (72%), colorless crystals, mp 275–277°C (DMF). IR spectrum, ν, cm^{-1}: 3344, 3388 (NH), 2217 (C≡N), 1681 (CONH). ^{1}H NMR spectrum, δ, ppm: 1.89 s (3H, Me), 2.37 t (2H, CH_2, *J* 6.9 Hz), 2.63 s (3H, Me), 2.76–2.89 m (4H, $2CH_2$), 4.60 s (2H, SCH_2), 7.01–7.18 m (4H, H_{arom}), 7.26–7.42 m (3H, H_{arom}), 7.52–7.63 m (1H, H_{arom}), 9.97 br.s (1H, NH), 12.72 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 17.45, 22.83, 27.48, 28.67, 29.02, 32.69, 103.49, 114.63, 115.83, 116.64, 118.63, 125.09, 126.05, 129.36, 130.47, 130.83, 130.89, 132.33, 135.11, 136.14, 137.40, 139.54, 149.86, 153.35, 157.86, 158.43, 160.35, 160.66, 162.60, 163.79, 167.92. Mass spectrum, *m/z* (I_{rel}, %): 582 (100) $[M + 1]^+$. Found, %: C 63.94; H 4.12; N 13.95. $C_{31}H_{24}FN_5O_2S_2$. Calculated, %: C 64.01; H 4.16; N 12.04. *M* 581.697.

2-Methyl-6-[(4-oxo-4*H*-pyrido[1,2-*a*]pyrimidin-2-yl)methylthio]-*N*,4-diphenyl-5-cyanonicotinamide (IVe). Yield 4.1 g (81%), colorless crystals, mp 232–234°C (BuOH). IR spectrum, ν, cm^{-1}: 3352 (NH), 2218 (C≡N), 1682 (C=O), 1676 (CONH). ^{1}H NMR spectrum, δ, ppm: 2.62 s (3H, Me), 4.62 s (2H, SCH_2), 6.59 s (1H, C^3H, pyridopyrimidine), 7.04 t (1H, H_{arom}, *J* 7.2 Hz), 7.24 t (2H, H_{arom}, *J* 7.8 Hz), 7.32–7.39 m (3H, H_{arom}, pyridopyrimidine), 7.42–7.51 m (5H, Ph), 7.71 d (1H, pyridopyrimidine, *J* 8.8 Hz), 7.98 t (1H, pyridopyrimidine, *J* 7.2 Hz), 8.94 d (1H, pyridopyrimidine, *J* 6.7 Hz), 10.39 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 22.93, 35.28, 102.17, 103.80, 114.83, 116.48, 119.74 (2C), 124.24, 125.72, 127.11, 128.42 (2C), 128.48 (2C), 128.82, 129.27, 129.74, 134.05, 137.95, 138.10, 150.81, 151.51, 157.12, 158.23, 160.63, 163.24, 163.72, 186.53. Mass spectrum, *m/z* (I_{rel}, %): 504 (100) $[M + 1]^+$. Found, %: C 69.10; H 3.95; N 13.85. $C_{29}H_{21}N_5O_2S$. Calculated, %: C 69.17; H 4.20; N 13.91. *M* 503.587.

2-Methyl-*N*-(*o*-tolyl)-6-(2-phenyloxazol-4-ylmethylthio)-4-(3-fluorophenyl)-5-cyanonicotinamide (IVf). Yield 3.8 g (72%), colorless crystals, mp 233–235°C (BuOH). IR spectrum, ν, cm^{-1}: 3366 (NH), 2220 (C≡N), 1674 (CONH). ^{1}H NMR spectrum, δ, ppm: 1.92 s (3H, Me), 2.76 s (3H, Me), 4.60 s (2H, SCH_2), 7.03–7.19 m (4H, H_{arom}), 7.31–7.44 m (3H, H_{arom}), 7.52–7.63 m (4H, H_{arom}), 7.99 d (2H, H_{arom}, *J* 7.9 Hz), 8.20 s (1H, C^5H, oxazole), 9.87 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 17.41, 23.00, 25.43, 103.88, 114.65, 115.81, 116.00, 116.63, 116.80, 125.08, 125.99 (2C), 126.10 (2C), 126.74, 129.26 (2C), 130.51, 130.82, 132.28, 135.11, 137.56, 137.61 (2C), 149.87, 158.57, 160.54, 160.67, 160.75, 162.62, 163.93. Mass spectrum, *m/z* (I_{rel}, %): 535 (100) $[M + 1]^+$. Found, %: C 69.58; H 4.25; N 10.33. $C_{31}H_{23}N_4FO_2S$. Calculated, %: C 69.65; H 4.34; N 10.48. *M* 534.617.

2-Methyl-4-(3,4-dimethoxyphenyl)-*N*-phenyl-6-(2-phenyloxazol-4-ylmethylthio)-5-cyanonicotinamide (IVg). Yield 4.8 g (85%), colorless crystals, mp 202–204°C (DMF). IR spectrum, ν, cm^{-1}: 3355 (NH), 2219 (C≡N), 1676 (CONH). ^{1}H NMR spectrum, δ, ppm: 2.71 s (3H, Me), 3.66 s (3H, MeO), 3.77 s (3H, MeO), 4.54 s (2H, SCH_2), 6.86–7.09 m (4H, H_{arom}), 7.24 t (2H, H_{arom}, *J* 7.4 Hz), 7.44 d (1H, H_{arom}, *J* 7.8 Hz), 7.46–7.54 m (4H, H_{arom}), 7.97 d (2H, H_{arom}, *J* 8.1 Hz), 8.06 s (1H, C^5H, oxazole), 10.34 br.s (1H, NH). Mass spectrum, *m/z* (I_{rel}, %): 563 (100) $[M + 1]^+$. Found, %: C 68.22; H 4.54; N 9.85. $C_{32}H_{26}N_4O_4S$. Calculated, %: C 68.31; H 4.66; N 9.96. *M* 562.648.

3-Imino-6-methyl-2-(4-methoxybenzoyl)-4-(3,4-dimethoxyphenyl)-*N*-phenyl-2,3-dihydrothieno[2,3-*b*]pyridine-5-carboxamide (V). Yield 4.0 g (72%), yellow powder, mp 142–144°C (EtOH). IR spectrum, ν, cm^{-1}: 3387, 3339 (NH), 1693 (C=O), 1670 (CONH), 1625 (C= NH). ^{1}H NMR spectrum, δ, ppm: 2.68 s (3H, Me), 3.73 s (3H, MeO), 3.80 s (3H, MeO), 3.88 s (3H, MeO), 3.98 s (1H, C^2H), 6.76 br.s (1H, C= NH), 6.94–7.06 m (5H, H_{arom}), 7.09 s (1H, H_{arom}), 7.22 t (2H, H_{arom}, *J* 7.5 Hz), 7.42 d (2H, H_{arom}, *J* 8.0 Hz), 7.78 d (2H, H_{arom}, *J* 8.0 Hz), 10.25 br.s (1H, CONH). Mass spectrum, *m/z* (I_{rel}, %): 552 (100) $[M - 1]^+$. Found, %: C 67.16; H 4.80; N 7.43. $C_{31}H_{27}N_3O_5S$. Calculated, %: C 67.25; H 4.92; N 7.59. *M* 553.635.

3-Amino-6-methyl-4-(3,4-dimethoxyphenyl)-2-(4-oxo-4*H*-pyrido[1,2-*a*]pyrimidin-2-yl)-*N*-phenylthieno[2,3-*b*]pyridine-5-carboxamide (VI). Yield 4.2 g (75%), yellow needles, mp 273–275°C (DMF), mp 250°C. IR spectrum, ν, cm^{-1}: 3215, 3345, 3398 (NH, NH_2), 1689 (C=O), 1670 (CONH), 1647 [δ(NH_2)]. ^{1}H NMR spectrum, δ, ppm: 2.60 s (3H, Me), 3.71 s (3H, MeO), 3.79 s (3H, MeO), 6.16 s (1H, C^3H, pyridopyrimidine), 6.37 br.s (2H, NH_2), 6.95–7.14 m (3H, H_{arom}), 7.16 s (1H, H_{arom}), 7.22–7.31 m (3H, H_{arom}), 7.42 d (2H, H_{arom}, *J* 7.5 Hz), 7.75 d (1H, H_{arom}, *J* 8.8 Hz), 7.91 t (1H, H_{arom}, *J* 7.2 Hz), 8.90 d (1H, H_{arom}, *J* 6.3 Hz), 10.19 br.s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 22.46, 55.53, 55.64, 95.33, 101.94, 111.43, 112.51, 115.69, 119.46, 121.20, 121.51, 123.94, 125.28, 125.58, 127.06, 128.83, 130.78, 136.16, 138.54, 142.99,

144.14, 148.24, 149.18, 150.31, 154.22, 156.41, 158.74, 158.87, 162.37, 165.12, 186.58. Mass spectrum, *m/z* (I_{rel}, %): 564 (100) $[M + 1]^+$. Found, %: C 65.92; H 4.30; N 12.32. $C_{31}H_{25}N_5O_4S$. Calculated, %: C 66.06; H 4.47; N 12.43. *M* 563.641.

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