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Synthesis of Maleopimaric and Citraconopimaric Acids *N*-[3-(Pyrimidin-2-yl)aryl]amides

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Abstract—Acylation of 6-methyl-*N*-[4-(pyridin-3-yl)pyrimidin-2-yl]benzene-1,3-diamine, 4-methyl-*N*-[4-(pyridin-3-yl)pyrimidin-2-yl]benzene-1,3-diamine, and *N*-[4-(pyridin-3-yl)pyrimidin-2-yl]benzene-1,3-diamine with maleopimaric and citraconopimaric acid chlorides, with benzotriazolyl maleopimarate afforded *N*-[3-(pyrimidin-2-yl)aryl]amides of maleopimaric and citraconopimaric acids. By the reaction of substituted N-arylamides of maleopimaric acid with methanesulfonic acid biologically active methanesulfonates were obtained.

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Due to the high physiological activity the 2-aminopyrimidine derivatives are promising for the application as bioactive substances with a wide range of therapeutical action. They are the active substances of known drugs like fluorofur, acyclovir, trimethoprim, rosuvastatin [1]. Based on 2-arylaminopyrimidine derivatives antitumor drugs glivec (imatinib) and tasigna (nilotinib) has been developed that are now the most efficient drugs in the treatment of chronic myeloid leukemia [2, 3]. The high price of glivec and tasigna and the resistance to the medical treatment in some cases call to the search for and development of analogs of these drugs.

In the structure of the active substance of imatinib one of the main pharmacophoric fragments is the amide group binding *N*-(2-methyl-5-aminophenyl)-4-(pyridin-3-yl)pyrimidin-2-amine and the residue of the 4-(4-methylpiperazin-1-yl)methylbenzoic acid. We describe here the synthesis of new analogs of the active substance of imatinib containing instead of the benzylpiperazine fragment a residue of maleopimaric or citraconopimaric acid.

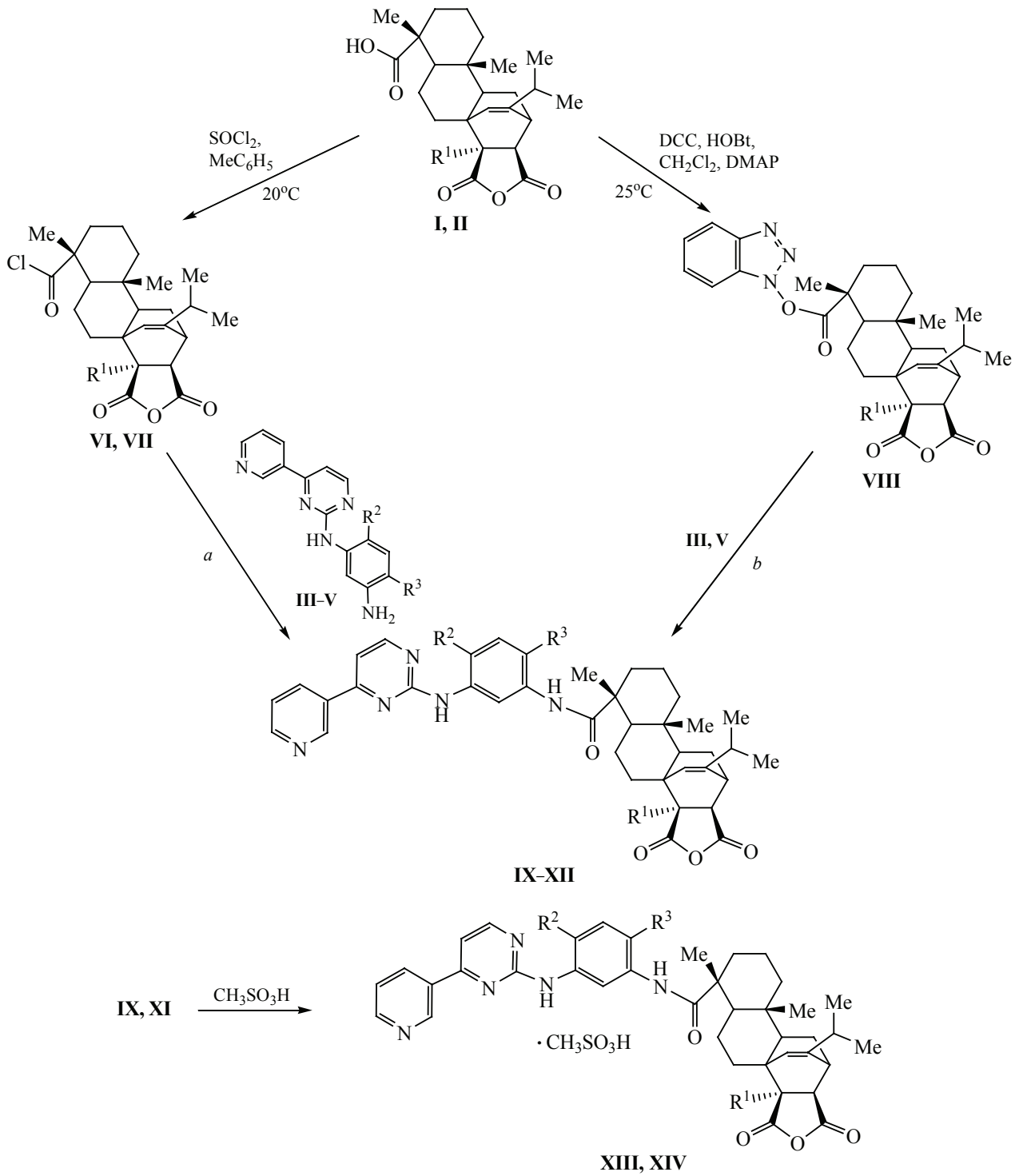
Methods were developed for the preparation of a number of maleopimaric acid derivatives at the carboxy and anhydride groups, in particular, of *N*-aryl- and *N*-alkylamides, imides, amidoimides [4–6]. Amides and imidoamides of the maleopimaric acid and also the acid itself exhibited pronounced hepatoprotective, immunostimulating action, fungicidal and other properties [5, 7–9]. Maleopimaric (**I**) and citraconopimaric (**II**) acids are obtained from wood chemical raw material, purified and described in [10–12]. The derivatives of citraconopimaric acid except for its methyl, allyl, and propargyl esters were not obtained before.

We for the first time brought into the synthesis of amides from the derivatives of maleopimaric (**I**) and citraconopimaric (**II**) acids 6-methyl-*N*-[4-(pyridin-3-yl)pyrimidin-2-yl]benzene-1,3-diamine (**III**), the key precursor of the imatinib substance, its isomer 4-methyl-*N*-[4-(pyridin-3-yl)pyrimidin-2-yl]benzene-1,3-diamine (**IV**), and the demethylated analog *N*-[4-(pyridin-3-yl) pyrimidin-2-yl]benzene-1,3-diamine (**V**).

The amides were synthesized by acylation of amines **III–V** with acid chlorides **VI, VII** of maleopimaric and citraconopimaric acids (procedure *a*) and with activated ester **VIII** of acid **I** and hydroxybenzotriazole (procedure *b*) [13, 14]. Acid chlorides **VI, VII** were obtained by treating acids **I, II** with excess thionyl chloride.

By procedure *a* compounds **III–V** were acylated with an excess of the corresponding acid chloride **VI, VII** in THF in the presence of triethylamine (Et_3N). Amides **IX–XII** of maleopimaric and citraconopimaric acids were obtained in 40–78% yields.

Scheme.



(*a*) Et_3N, THF, 45°C; (*b*) DMAP, CH_2Cl_2, 25°C; R^1 = H (**I, VI, VIII**), Me (**II, VII**); R^2 = Me, R^3 = H (**III**); R^2 = H, R^3 = Me (**IV**), $R^2 = R^3$ = H (**V**); $R^1 = R^3$ = H, R^2 = Me (**IX, XIII**); $R^1 = R^2$ = H, R^3 = Me (**X**); $R^1 = R^2 = R^3$ = H (**XI, XIV**); $R^1 = R^2$ = Me, R^3 = H (**XII**).

The acylation of amines **III–V** by method *b* was carried out in two steps. First from acid **I**, 1-hydroxybenzotriazole (HOBt), and dicyclohexylcarbodiimide (DCC) ester **VIII** was prepared, in the second stage the amine was added, and the reaction was performed in the presence of catalytic quantities of 2-dimethylaminopyridine (DMAP). The application of procedure *b* makes it possible to avoid heating of the reaction mixture, but it takes longer time than acylation with the acid chlorides.

The maximum yield (78%) was obtained from amine **V** lacking the methyl group in the aniline fragment of the molecule. The sufficiently high yield of amides **IX, XII** (48–58%) was obtained from amine **III**. The lower yield of amide **X** (40%) is evidently due to the steric hindrance from the *o*-methyl group in the molecule of amine **IV**.

At low temperature (20–65°C) the anhydride group of the maleopimaric and citraconopimaric acids is not involved into processes of chloroacylation, esterification, and aminolysis, and the reactions proceed selectively at the carboxy group.

Amides **IX–XII** were identified by spectral methods. The IR spectra of compounds **IX–XII** contain characteristic bands of the stretching vibrations of the C=O bonds of the amide (1680–1660) and anhydride (1840–1770 cm^{-1}) fragments, of the stretching (3440–3350) and bending (1640–1610 cm^{-1}) vibrations of the amino groups.

In the ^{13}C NMR spectra of amides **IX–XII** the proton signals of the *N*-aryl substituents (of pyridine, pyrimidine, benzene rings and amino groups), except for the proton signals of the methyl group (singlet at 2.27–2.33 ppm) are located in the region of the signals of aromatic protons (6.90–9.32 ppm). The characteristic proton signals of the methyl groups of the acid residue appear at 0.57–0.65, 0.96–1.00, 1.18–1.36 ppm, and of the vinyl proton at the bridging double bond, in the region 5.46–5.57 ppm.

In the ^{13}CNMR spectrum of citraconopimaric acid amide **XII** the signals of the aliphatic carbon atoms appear in the region 8.30–65.54 ppm, of aromatic carbon atoms, at 108.00–158.78 ppm. The signals of carbon atoms of the carbonyl groups are at 162.40, 172.41, 176.31 ppm.

In order to prepare pharmacologicaly active substances for the estimation of the physiologic action we obtained salts **XIII, XIV** of compounds **IX, XI** with methanesulfonic acid by the procedure of the synthesis of imatinib methanesulfonate [15].

In the IR spectra of methansulfonates **XIII, XIV** alongside the characteristic bands of initial amides a band of the stretching vibrations of the S=O group is present at 1150–1140 cm^{-1}. In the ^{1}H NMR spectra of salts compared to the spectra of amides a singlet appears of the protons of the methyl group of the methanesulfonate moiety at 2.60–2.65 ppm.

EXPERIMENTAL

IR spectra were recorded on a Fourier spectrometer Nicolet Protege-460 from pellets with KBr. ^{1}H NMR spectra were registered on a spectrometer Bruker Avance AC-500 (500 and 100 MHz) in $CDCl_3$ (for amides) and methanol (d_4) (for salts); internal reference TMS. TLC was carried out on Merk DC-Plasticfolien Kieselgel 60 F_{254} plates, eluents butanol–ethanol–NH_4OH, 8 : 1 : 1, and chloroform–methanol, 95 : 5. Melting points were determined on a Koeffler heating block.

Amines **III–V** were prepared by procedure [16].

Maleopimaric and citraconopimaric acid chlorides (VI, VII). To 3 mmol of acid **I, II** (for acid **I** in toluene) was added 3 ml (40 mmol) of thionyl chloride, the reaction mixture was stirred for 8–10 h, later it was thoroughly evaporated in a vacuum.

Maleopimaroyl chloride (VI). Yield 99%, colorless crystals, mp 190°C (decomp.) (cf. [17]). IR spectrum, ν, cm^{-1}: 1850, 1790 [$(C=O)_2O$], 1710 [(C=O)Cl], 1250 [$CH(CH_3)_2$]. Found, %: C 68.82; H 7.45; Cl 8.47. $C_{24}H_{31}ClO_4$. Calculated, %: C 68.80; H 7.46; Cl 8.46.

Citraconopimaroyl chloride (VII). Yield 99%, colorless crystals, mp 154–156°C. IR spectrum, ν, cm^{-1}: 1850, 1780 [$(C=O)_2O$], 1700 [(C=O)Cl], 1250 [$CH(CH_3)_2$]. ^{1}H NMR spectrum, δ, ppm: 0.63 s (3H, Me), 0.97 m (1H, CH), 1.00 d (6H, $\underline{Me}_2CH$, *J* 7.0 Hz), 1.28 s (3H, Me), 1.36 m (2H, CH_2), 1.45 s (3H, Me), 1.47−1.78 m (10H, CH_2), 2.25 sextet (1H, $Me_2\underline{CH}$), 2.34 m (1H, CH), 2.61 d (1H, CH, *J* 3.0 Hz), 3.05 s (1H, CH), 5.59 s (1H, CH=C). Found, %: C 69.34; H 7.65; Cl 8.17. $C_{25}H_{33}ClO_4$. Calculated, %: C 69.35; H 7.68; Cl 8.19.

Acylation of 2-arylaminopyrimidines III–V. *a*. To a solution of 0.01 mol of 2-arylaminopyrimidine **III–V** in 10 ml of THF was added 0.02 mol of triethylamine, the mixture was heated at 60–65°C for 30 min, then 0.013 mol of maleopimaroyl chloride (**VI**) or citraconopimaroyl chloride (**VII**) was added, the mixture was stirred at 60–65°C over 4–6 h. On the completion

of the reaction (TLC monitoring) with the solvent was evaporated in a vacuum, the residue was treated with 20% water solution of NaOH to pH 9. The reaction product was extracted into chloroform (3 × 40 ml), the extract was dried over $MgSO_4$, chloroform was removed in a vacuum. The obtained amide was recrystallized from a mixture ethyl acetate–ethyl ether.

b. A solution of 0.4 g (1 mmol) of maleopimaric acid (**I**) and 0.34 g (1.5 mmol) of DCC in 20 ml of dichloromethane was stirred at room temperature for 10 min, and 0.24 g (1.8 mmol) of 1-hydroxybenzotriazole was added. The reaction mixture was stirred at room temperature over 15–18 h, then the precipitate of dicyclohexylurea was filtered off and washed with dichloromethane (3 × 20 ml). To the combined filtrates was added 1 mmol of compound **III** or **V**, 0.05 g (catalytic amount) of DMAP, and the reaction mixture was stirred at 18–20°C over 36 h. On the completion of the reaction (TLC monitoring) the mixture was diluted with 10 ml of water, 5 ml of 25% NH_4OH, the reaction product was extracted over dichloromethane (3 × 20 ml), the extract was dried over Na_2SO_4 and evaporated in a vacuum. The solid residue of amide was recrystallized from a mixture chloroform–ethyl ether.

Maleopimaric acid *N*-{4-methyl-3-[4-(pyridin-3-yl)pyrimidin-2-ylamino]phenyl}amide (IX). Yield 58% (*a*), 56% (*b*), colorless crystals, mp 161–162°C. IR spectrum, cm^{-1}: 3440, 2870, 1795, 1670, 1610, 1580, 1450, 1320, 1275. ^1H NMR spectrum, δ, ppm: 0.59 s (3H, Me), 0.97 d (1H, CH, *J* 9.0 Hz), 1.00 m (6H, Me_2), 1.18 m (3H, Me), 1.25–1.89 m (8H, CH_2), 2.01 s (1H, CH), 2.05 s (1H, CH), 2.30 s (3H, Me), 2.51–3.10 m (7H, CH, CH_2), 5.54 s (1H, CH=C), 7.15–7.20 m ($3H_{arom}$, NH), 7.44 m ($2H_{arom}$), 7.58 d ($1H_{arom}$, *J* 7.4 Hz), 7.78 m ($1H_{arom}$), 8.40–8.46 m ($2H_{arom}$), 8.72 m ($1H_{arom}$), 9.32 s (1H, NH). Found, %: C 72.83; H 6.86; N 10.62. $C_{40}H_{45}N_5O_4$. Calculated, %: C 72.81; H 6.87; N 10.61.

Maleopimaric acid *N*-{2-methyl-5-[4-(pyridin-3-yl)pyrimidin-2-ylamino]phenyl}amide (X). Yield 40% (*a*), colorless crystals, mp 177–178°C. IR spectrum, cm^{-1}: 3440–3350, 2880, 1840, 1790, 1680, 1620, 1580, 1450, 1310, 1280. ^1H NMR spectrum, δ, ppm: 0.63 s (3H, Me), 0.98 d (3H, Me, *J* 3.5 Hz), 1.00 d (3H, Me, *J* 3.5 Hz), 1.32 s (3H, Me), 1.33–1.89 m (11H, CH, CH_2), 2.27 s (3H, Me), 2.53–3.18 m (7H, CH, CH_2), 5.52 s (1H, CH=C), 7.09–7.18 m ($4H_{arom}$), 7.42 m ($2H_{arom}$), 7.54 s (1H, NH), 8.45–8.51 m ($2H_{arom}$), 8.72 d ($1H_{arom}$, *J* 4.6 Hz), 9.25 s (1H, NH). Found, %: C 72.80; H 6.85; N 10.63. $C_{40}H_{45}N_5O_4$. Calculated, %: C 72.81; H 6.87; N 10.61.

Maleopimaric acid *N*-{3-[4-(Pyridin-3-yl)pyrimidin-2-yl-amino]phenyl}amide (XI). Yield 78% (*a*), 69% (*b*), colorless crystals, mp 186–187°C. IR spectrum, cm^{-1}: 3440–3350, 2880, 1840, 1785, 1670, 1640, 1570, 1320, 1270. ^1H NMR spectrum, δ, ppm: 0.57 s (3H, Me), 0.78 t (1H, CH, *J* 11.8 Hz), 0.96 d (3H, Me, *J* 1.9 Hz), 0.97 d (3H, Me, *J* 1.9 Hz), 1.15–1.27 m (4H, CH_2), 1.33 s (3H, Me), 1.39–1.55 m (6H, CH_2), 1.90 d (1H, CH, *J* 11.8 Hz), 2.20 m (3H, CH, CH_2), 2.32 m (1H, CH), 2.99 d.d (1H, CH, *J* 8.8, 3.1 Hz), 3.05 s (1H, CH), 5.46 s (1H, CH=C), 6.90 d ($1H_{arom}$, *J* 8.8 Hz), 7.19 d ($1H_{arom}$, *J* 5.2 Hz), 7.31 t ($1H_{arom}$, *J* 8.0 Hz), 7.43 d.d ($1H_{arom}$, *J* 7.9, 4.8 Hz), 7.70 s (1H, NH), 7.82 d ($1H_{arom}$, *J* 8.5 Hz), 8.29 s ($1H_{arom}$), 8.36 s ($1H_{arom}$), 8.43 d.t ($1H_{arom}$, *J* 8.0, 1.9 Hz), 8.54 d ($1H_{arom}$, *J* 5.1 Hz), 8.72 d.d ($1H_{arom}$, *J* 4.2, 1.6 Hz), 9.29 d (1H, NH, *J* 1.9 Hz). Found, %: C 72.52; H 6.72; N 10.83. $C_{39}H_{43}N_5O_4$. Calculated, %: C 72.53; H 6.71; N 10.84.

Citraconopimaric acid *N*-{4-methyl-3-[4-(pyridin-3-yl)pyrimidin-2-ylamino]phenyl}amide (XII). Yield 48% (*a*), colorless crystals, mp 176–178°C. IR spectrum, cm^{-1}: 3400–3350, 2980, 1850, 1790, 1740, 1660, 1580, 1430, 1310, 1250. ^1H NMR spectrum, δ, ppm: 0.65 s (3H, Me), 0.99 d (6H, Me_2, *J* 6.7 Hz), 1.20 m (2H, CH_2), 1.32 s (3H, Me), 1.36 s (3H, Me), 1.46–1.64 m (8H, CH_2), 1.70 m (1H, CH), 1.83 d (1H, CH, *J* 7.8 Hz), 2.04 s (1H, CH), 2.23 m (2H, CH_2), 2.33 s (3H, Me), 2.57 d (1H, CH, *J* 2.6 Hz), 3.02 s (1H, CH), 5.57 s (1H, CH=C), 7.10 s ($1H_{arom}$), 7.24 m ($3H_{arom}$), 7.43 d.d ($1H_{arom}$, *J* 7.8, 4.8 Hz), 7.53 s ($1H_{arom}$), 8.40 s (1H, NH), 8.49 d ($1H_{arom}$, *J* 7.8 Hz), 8.52 d ($1H_{arom}$, *J* 4.8 Hz), 8.72 d ($1H_{arom}$, *J* 4.8 Hz), 9.24 s (1H, NH). ^{13}C NMR spectrum, δ, ppm: 8.30, 13.89, 16.14, 17.34, 18.22, 19.58, 20.71, 27.18, 29.49, 32.29, 36.65, 37.59, 37.92, 41.87, 45.75, 46.55, 47.24, 49.31, 52.92, 54.59, 57.63, 60.07, 65.54, 108.00, 113.30, 115.39, 123.40, 124.25, 127.44, 130.41, 132.43, 134.58, 136.06, 137.40, 147.29, 148.22, 151.17, 158.78, 162.40, 172.41, 176.31. Found, %: C 73.10; H 7.04; N 10.38. $C_{41}H_{47}N_5O_4$. Calculated, %: C 73.08; H 7.03; N 10.39.

Methanesulfonates XIII, XIV. To a suspension of 0.4 mmol of amide **IX, XI** in 10 ml of ethanol was added dropwise 0.04 g (0.4 mmol) of methanesulfonic acid within 20 min. The reaction mixture was boiled for 40 min, then it was filtered at 65°C. The filtrate was evaporated to dryness, the residue was washed with ethyl ether (2 × 20 ml), dissolved at heating in 30 ml of ethanol, 5 ml of water was added, and the reaction mixture was kept at room temperature for 14–18 h. The precipitate was

filtered off and dried in a vacuum at 60°C till constant weight (~4 h).

Salt (XIII) of methanesulfonic acid and maleopimaric acid *N*-{4-methyl-3-[4-(pyridin-3-yl) pyrimidin-2-ylamino]phenyl}amide (IX). Yield 78%, yellow nonhygroscopic crystals, mp 175–178°C. IR spectrum, cm^{-1}: 3430–3300, 2945, 1785, 1650, 1575, 1440, 1260, 1150. ^1H NMR spectrum, δ, ppm: 0.63 s (3H, Me), 0.92 m (1H, CH), 1.02 m (6H, Me_2), 1.22 s (3H, Me), 1.24–1.90 m (8H, CH_2), 2.36 s (3H, Me), 2.60 m (2H, CH_2), 2.70 s (3H, Me), 2.74–3.27 m (7H, CH, CH_2), 5.41 s (1H, CH=C), 7.12 d ($2H_{arom}$, *J* 7.8 Hz), 7.25 m ($1H_{arom}$), 7.48 d ($1H_{arom}$), 7.83 s ($1H_{arom}$), 8.02 m ($1H_{arom}$), 8.59 m ($2H_{arom}$), 9.04 m ($1H_{arom}$). Found, %: C 64.93; H 6.38; N 9.19; S 4.01. $C_{41}H_{49}N_5O_7S$. Calculated, %: C 65.17; H 6.49; N 9.27; S 4.24.

Salt (XIV) of methanesulfonic acid and maleopimaric acid *N*-{3-[4-(pyridin-3-yl)pyrimidin-2-ylamino]-phenyl}amide (XI). Yield 98%, light-brown nonhygroscopic crystals, mp 195–202°C. IR spectrum, cm^{-1}: 3450, 3350, 2950, 1790, 1650, 1580, 1450, 1240, 1140. ^1H NMR spectrum, δ, ppm: 0.61 d (3H, Me, *J* 11.0 Hz), 0.93 t (3H, Me, *J* 2.0 Hz), 1.05 d (3H, Me, *J* 2.0 Hz), 1.10 m (2H, CH_2), 1.24 s (3H, Me), 1.28–1.83 m (10H, CH_2), 2.62 m (1H, CH), 2.66 s (3H, Me), 2.76 m, 2.95 m, 2.98 m, 3.88 m (5H, CH), 5.46 s (1H, CH=C), 7.01 d.d (1H, H_{arom}, *J* 8.0, 4.1 Hz), 7.26 m ($2H_{arom}$), 7.44 d ($1H_{arom}$, *J* 5.1 Hz), 8.08 d ($1H_{arom}$, *J* 1.8 Hz), 8.21 d ($1H_{arom}$, *J* 8.0 Hz), 8.56 s ($1H_{arom}$), 8.89 br.s ($1H_{arom}$), 9.32 d.d ($1H_{arom}$, *J* 8.0, 1.8 Hz), 9.51 s ($1H_{arom}$). Found, %: C 64.55; H 6.21; N 9.37; S 4.17. $C_{40}H_{47}N_5O_7S$. Calculated, %: C 64.78; H 6.34; N 9.45; S 4.32.

REFERENCES

1. Koroleva, E.V., Gusak, K.N. and Ignatovich, Zh.V., *Russ. Chem Rev,* 2010, vol.79, no. 8, pp. 655–681
2. Szakacs, Z., Beni, S., Vagra, Z., Orfi, L., Keri, G., and Noszal, B., *J. Med. Chem*., 2005, vol. 48, p. 249.
3. Huang, W. S. and Shakespeare, W.C., *Synthesis*, 2007, p. 2121.
4. Ramm, P.J. and Taylor, J.B., *J. Labelled Comp. Radiopharm*., 1976, vol. 12, p. 13; *Chem. Abstr.,* 1976, vol. 85, 94528h.
5. Delevalee, F., Deraedt, R., and Benzoni, J., Europe Patent 211774, 1989; *Chem. Abstr.,* 1987, vol. 106, 182679r.
6. Bei, M.P. and Yuvchenko, A.P., *Izv. Nation. Akad. Nauk Belarusi. Ser. Khim,* 2010, no. 1, p. 78.
7. Danswan, G.W., Ramm, P.J., Matharu, S., and Taylor, J.B., *Arz.-Forsch. Drug Res.,* 1976, vol. 26, p. 2190.
8. Svikle, D.Ya., Prikule, A.Ya., Shuster, Ya.Ya., and Veselov, I.A., *Khim.-Farm. Zh.,* 1978, vol. 12, no. 5, p. 70.
9. Glovsky, M.M., Becker, E.L., and Halbrook, N.J., *J. Immunology*, 1968, vol. 100, p. 979.
10. Bei, M.P., *Izv. Nation. Akad. Nauk Belarusi, Ser. Khim.*, 2006, no. 5, p. 13.
11. Bei, M.P. and Yuvchenko, A.P., Abstracts of Papers, *XX Mezhdunar. nauchn.-tekhn. konf. "Khimicheskie reaktivy reagenty i protsessy malotonnazhnoi khimii"* (20th Int Sci. Techn. Conf. "Chemicals amd Processes of Small-Scale Chemistry"), Minsk: Belorusskaya Nauka, 2008.
12. Bei, M.P. and Yuvchenko, A.P., Belarus Patent 13646, 2010.
13. *The Peptides*, Gross, E. and Meienhofer, I., Eds., New York: Academic, 1979-1981, vols. 1-3.
14. Buehler, C.A. and Pearson, D. E., *Survey of Organic Syntheses*, New York: Wiley, 1970, vol. 1. 1977, vol. 2.
15. Macdonald, P., Pierluigi, R., Jegorov, A., Glolito, A., Tentorio, D., and Canavesi, A., WO Inventor's Certificate 2008/057291.
16. Koroleva, E.V., Ignatovich, Zh.V., Gusak, K.N., and Kovalev, V.V., *Izv. Nation. Akad. Nauk Belarusi, Ser. Khim.,* 2010, no. 2, p. 84.
17. Shuller, W.H. and Lawrence, R.V., *J. Chem. Eng. Data*., 1967, vol. 12, p. 267.