SYNTHESIS AND CHARACTERIZATION OF SUBSTITUTED 1,2,3-TRIAZoles COMBINED WITH γ-BUTANOLIDE RING

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The synthesis of new 1,2,3-triazoles has been actualized by means of the reaction of dipolar 1,3-cycloaddition of organic azides to the γ-lactones containing terminal triple bond. The synthesized compounds previously were nondescript in literature. High yields of the final products ensured the optimal reaction conditions.

Keywords: 1,2,3-triazoles, butanolides, 1,3-cycloaddition, click-reaction.

Introduction. It is known that the main part of medicines used in practical medicine contain heterocyclic compounds as active aglycones. In series of these compounds, azoles and, particularly, 1,2,4-triazoles deserve special attention. They have been studied quite deeply, a wide spectrum of their physiological action has been revealed, and some derivatives of them are part of the medicinal preparations recommended for treatment of various diseases. In particular, the fungicides: difenolonazole and epoxiconazole used in practice, the antifungals fluconazole, itraconazole, voriconazole, etc. Derivatives of 1,2,3-triazole, the methods of obtaining of which do not differ in variety, and basically boil down to the method of dipolar 1,3-cycloaddition of terminal alkynes with organic azides (click-reactions) [1–5], studied relatively little, the analogue of Farnizol-a has been synthesized [6].

Biological studies showed that individual representatives of 1,2,3-triazoles exhibit antibioblenic [7], anticancer [8], antiplasmodial [9], antimicrobacterial [10] activities. It is also known that some representatives of 1,2,3-triazole-containing compounds can be used as bioluminescent substances [11], as well as phosphonic and flat chiral phosphonic ligands for Suzuki-Miyaura cross-coupling reactions [12, 13].

Materials and Methods. It is clear from the foregoing that studies in the field of 1,2,3-triazoles are urgent. The analysis of literary data confirms that different properties of active substances are provided by changing the nature of substituents in the triazole cycle and proceeding from this. In order to create new heterocyclic systems not described yet in literature, as well as for introducing

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such powerful pharmacophore group as the γ-lactonic ring into the molecule of 1,2,3-triazole group. We choose 2-propargyl-5,5-disubstituted-4-butanolides as initial compounds. The synthesis of them has been carried out according to the Scheme:

![Scheme 1](image)

For investigation of the behavior of compounds 1,2 under the conditions of click reactions, we actualized the synthesis of necessary organic azides by the known method [14–17]. The selection of bromides is made in such a way as to make it possible in the future to follow the influence of the nature of the mixers on the biological activity of the final products. One of the azides is synthesized on the basis of the natural product 2-hydroxy-3-(2-methylprop-1-en-1-yl) naphtha-1,4-dione (Lapachol), which is isolated from the Bignoniaceae tree.

\[
R'\cdot Br + NaN_3 \rightarrow R'\cdot N_3 + NaBr
\]

3. \(R' = o-N_2C_6H_4\) 4. \(R' = m-N_2C_6H_4\) 5. \(R' = p-N_2C_6H_4\) 6. \(R' = p-F-C_6H_4\) 7. \(R' = naphthalene-1,4-dione-2-y1\) 8. \(R' = 2,2-dimethyl-2,3-dihydropapt(1,2-b)furane-4,5-dione-3-y1\)

Scheme 2.

The interaction of compounds 1,2 with azides of different structures has been studied and it has been established that the reaction proceeds smoothly and chemoselectively in the presence of copper(I) iodide in tetrahydrofuran at 60–65°C. It is expedient to use secondary amines as a base, in particular, triethylamine. Yields in this case fluctuate within the range of 55–98%.

![Scheme 3](image)

Thus, a synthesis of new heterocyclic system, previously non described, has been realized, and synthesized compounds can provide practical interest.
Experimental Part. $^1$H and $^{13}$C NMR spectra were registered on spectrometer Varian Mercury-300 from solution in DMSO–CCl$_4$ (1 : 3) or on Bruker AVANCE 400 MHz spectrometer in CDCl$_3$. Chemical shifts (δ) are reported as quoted relative to the residual signals of chloroform-d (7.25 for $^1$H NMR and 77.0 for $^{13}$C NMR) or DMSO-d$_6$ (2.49 for $^1$H NMR and 39.5 for $^{13}$C NMR) as internal references. The purity of the obtained compounds was checked by TLC on Silufol UV-254 plates (development in iodine vapor) and by instrument of KW 254 nm / LW 366 nm. The mass spectra of compounds were recorded using a spectra SYSTEM P400(LC) and MSQ PLUS$^TM$ Single Quad(MS) (Thermo Scientific) spectrometers. Melting points were determined on Boetius micro-heating stage. Methods of the starting compounds 2-((propin-2-yl)-5,5-disubstituted hydrofuran-2-(3H)-ones (1,2) synthesis were described in [18].

1-Azido-2-nitrobenzene (3). 2 g (14.2 mmol) of 2-nitroaniline is dissolved in a 10% solution of hydrochloric acid with intensive stirring. The solution was cooled to 0°C and 1.13 g (16.33 mmol) of sodium nitrite was added. Stirring was continued for 0.5 h and 1.3 g of sodium azide dissolved in 20 mL of water was added. After 1 h stirring the product was extracted with 120 mL of ethyl acetate the organic layer is alternately washed with 100 mL of 10% hydrochloric acid solution, saturated solutions of NaHCO$_3$ and NaCl. The organic layer was dried over on MgSO$_4$. After distilling off the solvent, the residue is recrystallized. Yield 1.65 g (69%), m.p. 129–130°C (methanol) [14].

1-Azido-2-nitrobenzene (4). Was obtained similarly. Yield 59%, m.p. 53–54°C (ethanol: water – 1:4) [14].

1-Azidomethyl-4-nitrobenzene (5). The mixer, of 1.5 g (7 mmol) 4-nitrobenzyl bromine in 10% solution of hydrochloric acid, was cooled to 0°C and 0.6825 g (10.5 mmol) sodium azide in minimal volume of water was added. Stirring was continued about 16 h. The product was extracted with ethyl acetate. The organic layer was dried over on arid MgSO$_4$. After the solvent was distilled off, the residue was purified by the method of column chromatography (ethyl acetate : n-hexane – 4:1). Yield 0.36 g (74%) [15].

Azidomethyl-4-fluorobenzene (6). Was obtained similarly. Yield 70% [15].

2-Azidonaphthalene-1,4-dione (7). 1 g (4.2 mmol) of 2-bromo-1,4-naphthoquinone 0.546 g (8.4 mmol) of sodium azide dissolved in the minimum volume of water was added to heated to 50°C abs. ethanol. Stirring for 0.5 h and left in the cold for 12 h. The precipitated crystals are filtered. Yield 0.62 g (74%), m.p. 111–112°C [16].

3-Azido-2,2-dimethyl-2,3-dihydropyrazol(1,2-b)furan-4,5-dione (8). 1.1 g (4.8 mmol) of 2-hydroxy-3-(2-methyl-1-en-1-yl)-1,4-naphthoquinone, 30 mL of methylene chloride were added to the flask and stirred for 15 min in an ice bath. Added 2 mL of bromine and after 5 min removed the solvent from the dark red solution. After complete distillation, 30 mL of methylene chloride was added to continue distillation. The process is repeated until the unlevered solvent is distilled off. To the solid residue, 30 mL of methylene chloride and 0.624 g (9.6 mmol) of sodium azide are added. Stirred for 96 h and filtered. Yield 1.27 g (98%), m.p. 200–202°C [17].

5,5-Disubstituted-3-(1-(3-substituted)-1H-1,2,3-triazole-4-yl)methyl)dihydrofuran-2 (3H)-ones (9–19, General Procedure). 1.1 mmol of the compounds 3–8, 2 mL of tetrahydrofuran, 1.1 mmol of ethyl isopropylamine and 0.1 mmol of copper (I) iodide were placed in a flask. The mixture was stirred 30 min at room temperature and
added 1 mmol of the corresponding 1.2. The mixture was heated at 60–65°C for 5 h, cooled, then reaction mixture was poured into a 30 mL 0.1 M solution of hydrochloric acid. The product extracted with methylene chloride (3 × 100 ml), the organic layer was dried over arid MgSO₄. After distilling off the solvent, depending on the aggregate state, the product is purified by the method of recrystallization or chromatographic column.

5,5-Dimethyl-3-((1-(3-nitrophenyl)-1H-1,2,3-triazole-4-yl)methyl)dihydrofuran-2(3H)-one (9). Yield 47.4%, m.p. 95.2°C, R₆0.50 (ethyl acetate : n-hexane = 7 : 3).

1H NMR (500 MHz, CDCl₃). δ, ppm: 8.61 t (J=2.1 Hz, 1H arom); 8.29 ddd (J=8.2, 2.1, 0.9 Hz, 1H arom); 8.15 ddd (J=8.1, 2.1, 1.0 Hz, 1H arom); 8.02 s (1H, N–CH=); 7.74 t (J=8.2 Hz, 1H arom); 3.33–3.25 m (1H, CH₂ et, 1H, CHC=O); 3.16–3.09 m (1H, CH₂ et); 2.43–2.30 m (1H, CH₂ in cycle); 2.04–1.96 m (1H, CH₂ in cycle); 1.41 s (3H, Me); 1.40 s (3H, Me). 13C NMR (126 MHz, CDCl₃). δ: 177.7, 149.1, 146.0, 137.9, 131.1, 125.9, 123.3, 120.6, 115.4, 82.9, 41.0, 40.6, 28.9, 27.1, 26.0: IR, cm⁻¹: 573, 579, 603, 645, 668, 701, 737, 762, 803, 843, 872, 894, 925, 954, 997, 1043, 1079, 1094, 1128, 1182, 1240, 1265, 1318, 1349, 1375, 1389, 1411, 1442, 1456, 1495, 1531, 1596 (C=C in atom), 1619 (C=C in atom), 1755 (C=O), 2854 (C–H), 2925 (C–H), 2975 (C–H), 3103 (=CH), 3144 (=CH).

MS: found 317.08. C₁₅H₁₉N₂O₄. Calculated 317.12.

Found, %: C 56.91, H 5.09, N 17.75. C₁₅H₁₆N₄O₄. Calculated, %: C 56.96; H 5.10; N 17.71.

5,5-Dimethyl-3-((1-(2-nitrophenyl)-1H-1,2,3-triazole-4-yl)methyl)dihydrofuran-2(3H)-one (10). Yield 92%, m.p. 108.7°C, R₆0.51 (ethyl acetate : n-hexane = 7 : 3).

1H NMR (500 MHz, CDCl₃). δ, ppm: 8.08 dd (J=8.1, 1.4 Hz, 1H arom); 7.79 m (1H arom); 7.76 s (1H, N–CH=); 7.70 m (1H arom); 7.60 dd (J=7.9, 1.3 Hz, 1H arom); 3.38–3.23 m (1H, CH₂H et, 1H, CHC=O); 3.15 dd (J=14.2, 6.3 Hz, 1H, CH₂H et); 2.33 m (1H, CH₂ in cycle); 1.94 dd (J=15.7, 8.7 Hz, 1H, CH₂ in cycle); 1.41 s (3H, Me); 1.39 s (1H, Me). 13C NMR (126 MHz, CDCl₃). δ: 177.6, 144.9, 144.4, 133.7, 130.7, 130.2, 127.7, 125.5, 123.9, 82.7, 40.9, 40.0, 30.8, 28.6, 26.9, 25.7.


Found, %: C 56.92; H 5.09; N 17.70. C₁₅H₁₆N₄O₄. Calculated, %: C 56.96; H 5.10; N 17.71.

5,5-Dimethyl-3-((1-(4-nitrophenyl)-1H-1,2,3-triazole-4-yl)methyl)dihydrofuran-2(3H)-one (11). Yield 98%, m.p. 98.1°C, R₆0.51 (ethyl acetate : n-hexane = 4:1).

1H NMR (500 MHz, CDCl₃). δ, ppm: 8.23–8.19 m (2H arom); 7.46 s (1H, N–CH=); 7.40–7.35 m (2H arom); 5.65–5.56 m (2H, NCH₂); 3.23–3.15 m (1H, CH₂H et, 1H, CHC=O); 3.02 dd (J=14.5, 6.5 Hz, 1H, CH₂H et); 2.30 dd (J=12.8, 8.8 Hz, 1H, CH₂ in cycle); 1.91 t (J=12.1 Hz, 1H, CH₂ in cycle); 1.36 s (3H, Me); 1.34 s (3H, Me). 13C NMR (126 MHz, CDCl₃). δ: 177.8, 148.2, 145.6, 141.9, 128.6, 124.4, 122.8, 82.8, 53.2, 41.0, 40.4, 28.9, 27.2, 26.0.


Found, %: C 58.15; H 5.52; N 17.00. C₁₆H₁₈N₂O₄. Calculated, %: C 58.17; H 5.49; N 6.96.

3-((1-(4-Fluorobenzyl)-1H-1,2,3-triazole-4-yl)methyl)-5,5-dimethyl-2H-dihydrofuran-2(3H)-one (12). Yield 95%, m.p. 123–124°C, R₆0.51 (ethanol : benzene = 1:5).

1H NMR (300 MHz, DMSO-d₆). δ, ppm: 7.75 s (1H, CH in triazole); 7.31–7.38 m (2H, CH arom); 7.01–7.09 m (2H, CH arom); 5.50 s (2H, CH₂ in chain); 3.10–3.22 m
(1H, CH in cycle); 3.09 dd (1 H, \(J=14.40, 3.9 \text{ Hz}\), CH\(_2\) in chain); 2.81 dd (1 H, \(J=14.40, 7.9 \text{ Hz}\), CH\(_2\) in chain); 2.24 dd (1 H, \(J=12.4, 8.6 \text{ Hz}\), CH\(_2\) in cycle); 1.82 dd (1H, \(J=12.4, 11.3 \text{ Hz}\), CH\(_2\) in cycle); 1.36 s (3 H, CH\(_3\)); 1.35 s (3 H, CH\(_3\)). \(^{13}\)C NMR (75 MHz, DMSO-d\(_6\)). \(\delta\): 25.6, 26.1, 28.3, 38.7, 38.9, 39.7, 39.9, 51.8, 81.0, 114.8, 115.1 (114.9 d, \(J=21.4 \text{ Hz}\)), 121.8, 122.1, 129.5, 129.6 (129.56 d, \(J=8.2 \text{ Hz}\)), 131.5 d (\(J=3.2 \text{ Hz}\)), 161.72 d (\(J=246 \text{ Hz}\)).

Found, %: C 63.37, H 5.90, F 6.30, N 14.00. \(\text{C}_{16}\text{H}_{18}\text{FN}_{3}\text{O}_2\). Calculated, %: C 63.35, H 5.98, F 6.26, N 13.85.

\(-4\) 2-(4,5,5-Dimethyl-2-oxotetrahydrofuran-3-yl)methyl)-1H-1,2,3-triazole-4-yl)-2,3-dihydronaphthalene-1,4-dione (I3). Yield 83%, m.p. 182.6°C, Rf 0.83 (ethyl acetate : n-hexane = 1:1). \(^{1}\)H NMR (500 MHz, CDCl\(_3\)). \(\delta\), ppm: 8.52 s (1H, =CHO=); 8.24–8.20 m (1H arom); 7.89–7.82 m (2H arom); 7.74 s (1H, N–CH=); 3.38 dd (\(J=15.0, 4.7 \text{ Hz}\), 1H, CH\(_2\) et); 3.33–3.25 m (1H, CH=O=); 3.05 dd (\(J=14.9, 8.2 \text{ Hz}\), 1H, CH\(_2\) et); 2.36 dd (\(J=12.8, 8.9 \text{ Hz}\), 1H, CH\(_2\) in cycle); 1.98 t (\(J=12.2 \text{ Hz}\), 1H, CH\(_2\) in cycle); 1.44 s (3H, Me); 1.40 s (3H, Me). \(^{13}\)C NMR (126 MHz, CDCl\(_3\)). \(\delta\): 184.0, 179.5, 177.5, 145.8, 139.5, 135.2, 134.6, 131.6, 131.2, 127.5, 126.7, 126.7, 124.5, 82.8, 41.0, 40.9, 29.0, 27.2, 26.3.

MS: found 352.28. \(\text{C}_{16}\text{H}_{18}\text{FN}_{3}\text{O}_{2}^{+}\). Calculated 352.12.

Found, %: C 64.91, H 4.90, N 11.95. \(\text{C}_{10}\text{H}_{12}\text{N}_{3}\text{O}_4\). Calculated, %: C 64.95, H 4.88, N 11.96.

5-Methyl-3-((1-(3-nitrophenyl)-1H-1,2,3-triazole-4-yl)methyl)dihydrofuran-2(3H)-one (I4). Yield 78.7%, m.p. 126.8°C, Rf 0.47 (ethyl acetate : n-hexane = 4:1). \(^{1}\)H NMR (500 MHz, CDCl\(_3\)). \(\delta\), ppm: 8.61 t (\(J=2.0 \text{ Hz}\), 1H arom); 8.35–8.25 m (1H arom); 8.21–8.09 m (1H arom); 8.01 d (\(J=5.8 \text{ Hz}\), 1H, N–CH=); 7.74 t (\(J=8.2 \text{ Hz}\), 1H arom); 4.65–4.49 m (1H, OCH); 3.38–3.07 m (3H, CH\(_2\)H et), 2.59 ddd (\(J=13.2, 8.0, 5.3 \text{ Hz}\), 0.66H, CH\(_2\) in cycle), 2.39 dt (\(J=13.1, 8.3 \text{ Hz}\), 0.34H, CH\(_2\) in cycle); 2.16 m (0.22H, CH\(_2\) in cycle); 1.76 m (0.64H, CH\(_2\) in cycle); 1.38 m (3H, Me). \(^{13}\)C NMR (126 MHz, CDCl\(_3\)). \(\delta\): 178.4, 178.1, 149.1, 145.9, 137.9, 131.1, 125.9, 123.3, 120.6, 115.4, 75.7, 75.3, 41.8, 39.3, 36.4, 34.0, 31.1, 26.1, 25.7, 21.3, 20.9.

MS: found 303.06. \(\text{C}_{10}\text{H}_{12}\text{N}_{3}\text{O}_{4}^{+}\). Calculated 303.1.

Found, %: C 55.69; H 4.60; N 18.54. \(\text{C}_{10}\text{H}_{14}\text{N}_{4}\text{O}_4\). Calculated, %: C 55.63; H 4.67; N 18.53.

5-Methyl-3-(1-(2-nitrophenyl)-1H-1,2,3-triazole-4-yl)methyl)dihydrofuran-2(3H)-one (I5). Yield 95%, m.p. 101.7°C, Rf 0.47 (ethyl acetate : n-hexane = 1:1). \(^{1}\)H NMR (500 MHz, CDCl\(_3\)). \(\delta\), ppm: 8.09 dd (\(J=8.2, 1.4 \text{ Hz}\), 1H arom); 7.82–7.77 m (1H arom); 7.74 s (1H, N–CH=); 7.73–7.68 m (1H arom); 7.63–7.59 m (1H); 4.68–4.44 m (1H, OCH); 3.37–3.09 m (3H, CH\(_2\)H et); 2.41–2.30 m (0.66H, CH\(_2\) in cycle); 2.13 ddd (\(J=13.1, 9.1, 4.1 \text{ Hz}\), 0.34H CH\(_3\) in cycle); 2.00–1.91 m (0.36 H CH\(_2\) in cycle); 1.77–1.67 m (0.64 H CH\(_2\) in cycle); 1.41–1.36 m (3H, Me). \(^{13}\)C NMR (126 MHz, CDCl\(_3\)). \(\delta\): 178.2, 145.1, 134.0, 130.9, 130.5, 128.0, 125.8, 124.0, 123.9, 83.0, 75.7, 75.4, 42.0, 41.2, 40.2, 39.5, 36.1, 34.0, 28.9, 27.2, 26.2, 25.9, 25.6, 21.4, 20.9.

MS: Found 303.10. \(\text{C}_{10}\text{H}_{12}\text{N}_{3}\text{O}_{4}^{+}\). Calculated 303.15.

Found, %: C 55.60; H 4.65; N 18.56. \(\text{C}_{10}\text{H}_{14}\text{N}_{4}\text{O}_4 \). Calculated, %: C 55.63; H 4.67; N 18.53.

5-Methyl-3-((1-(4-nitrobenzyl)-1H-1,2,3-triazole-4-yl)methyl)dihydrofuran-2(3H)-one (I6). Yield 80%, m.p. 82.6°C, Rf 0.2 (ethyl acetate : n-hexane = 4:1). \(^{1}\)H NMR (500 MHz, CDCl\(_3\)). \(\delta\), ppm: 8.22 m (2H arom); 7.45 d (\(J=3.4 \text{ Hz}\), 1H, NCH);
7.38 dd (J=8.8, 2.2 Hz, 2H arom); 5.67–5.54 m (2H, NCH3); 4.55–4.44 m (1H, CHO); 3.23–3.08 m (3H, CH3CH2H et); 2.08 ddd (J=21.8, 13.0, 8.5 Hz, 0.46H, CH2 in cycle); 1.97–1.16 m (4H); 1.72–1.61 m (1.5H, CH2 in cycle); 1.34 dd (J=14.0, 7.5 Hz, 3H, Me).


3-{[(1-(4-Fluorobenzyl)-1H-1,2,3-triazole-4-yl)methyl]-5-methylidihydrofuran-2(3H)-one (17). Yield 90%, m.p. 102–103°C, Rf 0.45 (ethanol : benzene = 1 : 5).

1H NMR (300 MHz, DMSO-d6). δ, ppm: 7.74 s (1H, CH in trazole); 7.30–7.37 m (2H, CH arom); 7.02–7.08 m (2H, CH arom); 5.51 s (2H, CH2 in chain); 3.11–3.21 m (1H, CH in cycle); 3.08 dd (1H, J=14.0, 3.9 Hz, CH2 in chain); 2.80 dd (1H, J=14.40, 7.9 Hz, CH2 in chain); 2.23 dd (1H, J=12.4, 8.6 Hz, CH2 in cycle); 1.80 dd (1H, J=12.4, 11.3 Hz, CH2 in cycle); 1.35 s (3H, CH3).

13C NMR (75 MHz, DMSO-d6). δ: 25.6, 26.1, 38.7, 38.9, 39.7, 39.9, 51.8, 81.0, 114.8, 115.1 (114.9 d J=21.4 Hz), 121.8, 122.1, 129.5, 129.6 d (129.56 d, J=8.2 Hz), 131.5 d (J=3.2 Hz), 161.72 d (J=246 Hz).


3-{[(3,5-Dimethyl-2-oxotetrahydrofuran-3-yl)methyl]-1H-1,2,3-triazole-1-yl}-2,2-dimethyl-2,3-dihydropyridinophene [1,2-b]furan-4,5-dione (18). Yield 76%, m.p. 200.2°C, Rf 0.54 (ethylacetate : n-hexane = 4:1).

1H NMR (500 MHz, CDCl3). δ, ppm: 8.22–8.11 m (1H arom), 7.82–7.68 m (3H arom); 7.38 d (J=2.1 Hz, 1H arom); 5.92 d (J=16.3 Hz, 1H, CHN); 3.25–3.07 m (1H, CH3H et, 1H, CHC=O); 3.04–2.90 m (1H, CH3H et); 2.24 dd (J=13.1, 8.7, 6.7 Hz, 1H, CH2 in cycle); 2.28–2.20 m (1H, CH2 in cycle); 1.92–1.86 m (0.5H, CH2 in cycle); 1.82–1.76 m (0.5H, CH2 in cycle); 1.73 d (J=1.0 Hz, 3H, Me); 1.36–1.30 m (6H, Me); 1.15 d (J=10.4 Hz, 3H, Me).

13C NMR (126 MHz, CDCl3). δ: 180.2, 177.9, 177.8, 174.7, 171.3, 162.7, 144.5, 144.3, 135.0, 133.50, 133.4, 131.6, 130.1, 126.8, 126.7, 125.8, 125.7, 122.1, 121.9, 111.4, 111.3, 96.0, 95.9, 82.9, 67.0, 66.9, 41.2, 41.0, 40.5, 40.0, 36.6, 31.6, 28.9, 28.9, 27.9, 27.8, 27.2, 27.1, 26.3, 25.8, 21.2, 21.1.

MS: found 422.15. C23H26N4O4. Calculated 422.17.


[2,2-Dimethyl-3-[(5-methyl-2-oxotetrahydrofuran-3-yl)methyl]-1H-1,2,3-triazole-1-yl]-2,3-dihydronaphtho[1,2-b]furan-4,5-dione (19). Yield 62%, m.p. 186.6°C, Rf 0.51 (ethylacetate : n-hexane = 4:1).

1H NMR (500 MHz, CDCl3). δ, ppm: 22–8.13 m (1H arom), 7.83–7.67 m (3H arom); 7.37 d (J=2.5 Hz, 1H arom); 5.93 dd (J=15.1, 2.3 Hz, 1H, CHN); 4.55–4.37 m (1H, CHO); 3.27–2.87 m (3H, CH3H et, CHC=O); 2.48 td (J=12.9, 7.9 Hz, 0.74H, CH2 in cycle); 2.36–2.27 m (0.26H, CH2 in cycle); 2.24–2.17 m (0.26H, CH2 in cycle); 2.08–2.00 m (0.74H, CH2 in cycle); 1.74 d (J=2.3 Hz, 3H, Me); 1.36–1.29 m (3H, Me); 1.17 dd (J=10.0, 3.6 Hz, 3H, Me).

13C NMR (126 MHz, CDCl3). δ: 180.2, 180.1, 178.6, 178.3, 178.2, 174.7, 171.3, 144.5, 144.3, 144.3, 135.0, 133.5, 131.6, 130.2, 126.8, 125.8, 125.7, 121.9, 121.8, 111.4, 111.3, 96.0, 95.9, 77.4, 77.2, 76.9, 75.6, 75.5, 75.4, 67.0, 60.5, 42.0, 41.8, 39.6, 39.5, 36.4, 35.9, 34.3, 34.0, 31.1, 27.9, 27.8, 26.5, 26.3, 26.0, 25.6, 21.4, 21.3, 21.2, 21.0, 20.9, 14.3.
MS: found 408.16. C_{22}H_{23}N_{3}O_{5}^+. Calculated 408.15.

Found, %: C 64.80; H 5.25; N 10.40. C_{22}H_{23}N_{3}O_{5}. Calculated, %: C 64.86; H 5.20; N 10.31.

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