ISSN 1070-4280, Russian Journal of Organic Chemistry, 2020, Vol. 56, No. 7, pp. 1217–1221. © Pleiades Publishing, Ltd., 2020.
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Three-Component Reaction of Pyrrolediones, Malononitrile, and Acyclic Enols

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Received February 10, 2020; revised March 31, 2020; accepted April 3, 2020

Abstract—Three-component condensation of ethyl 1-R-2-phenyl-4,5-dihydro-4,5-dioxo-1*H*-pyrrole-3-carboxylates, malononitrile, and acyclic enols (acetylacetone, alkyl acetoacetates, and ethyl benzoylacetate) afforded substituted ethyl 9-amino-10-cyano-1-oxo-3-phenyl-8-oxa-2-azaspiro[4.5]deca-3,6,9-triene-4-carboxylates. The products are interesting from the viewpoint of medicinal chemistry.

Keywords: 1*H*-pyrrole-2,3-diones, malononitrile, 2-amino-4*H*-pyran-3-carbonitriles, 1,3-dicarbonyl compounds, enols, three-component reactions

DOI: 10.1134/S1070428020070155

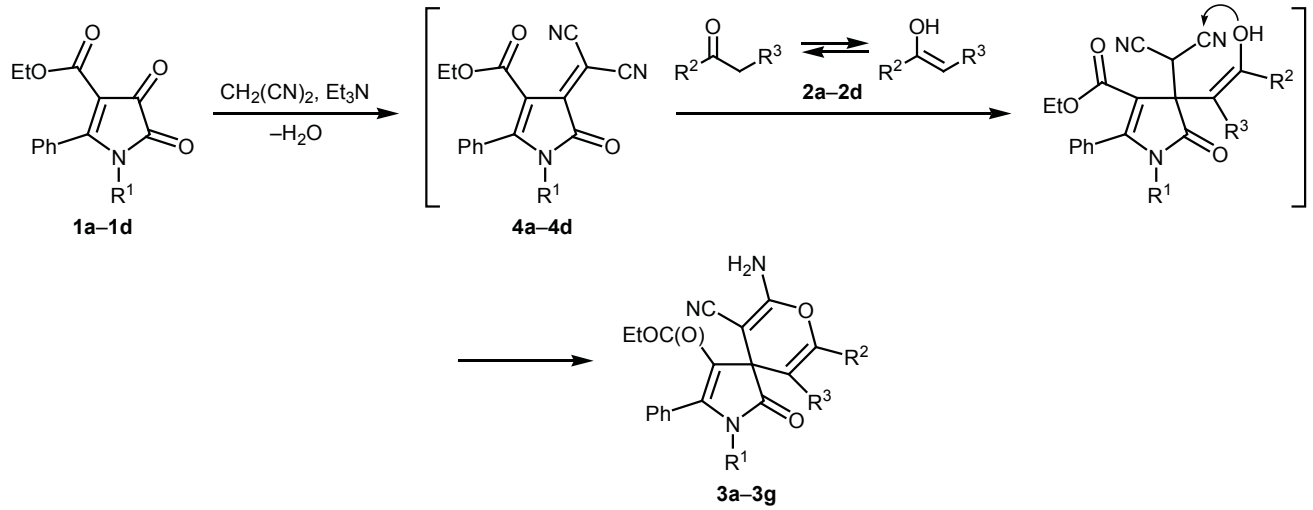
We previously studied three-component reactions of 1*H*-pyrrole-2,3-diones with malononitrile and six-membered carbo- and heterocyclic enols (5,5-dimethylcyclohexane-1,3-dione and 4-hydroxycoumarin), which led to the formation of spiro[chromene-4,3′-pyrroles] and spiro[pyrano[3,2-*c*]chromene-4,3′-pyrroles], respectively [1, 2]. Analogous reactions of 1*H*-pyrrole-2,3-diones with malononitrile and five-membered carbo- and heterocyclic enols [indan-1,3-dione, cyclopentane-1,3-dione, and furan-2,4(3*H*,5*H*)-dione] afforded spiro[indeno[1,2-*b*]pyran-4,3′-pyrroles], spiro[cyclopenta[*b*]pyran-4,3′-pyrroles], and spiro[furo[3,4-*b*]pyran-4,3′-pyrroles], respectively [3, 4]. Three-component reactions of 1*H*-pyrrole-2,3-diones with malononitrile and acyclic enols have not been reported so far.

By heating ethyl 1-R-2-phenyl-4,5-dihydro-4,5-dioxo-1*H*-pyrrole-3-carboxylates **1a**–**1d**, malononitrile, and acyclic enols **2a**–**2d** at a ratio of 1:1:4 in boiling anhydrous acetonitrile in the presence of triethylamine (20 mol %) for 19– 20 h (HPLC/MS monitoring) we obtained substituted 9-amino-10-cyano-1-oxo-3-phenyl-8-oxa-2-azaspiro[4.5]deca-3,6,9-triene-4-carboxylates **3a**–**3g** (Scheme 1). Unlike similar reactions with cyclic enols [1–4], the reaction with enols **2a**–**2d** required more severe conditions, i.e., prolonged heating and the use of excess enol (4 equiv).

Compounds **3a**–**3g** are high-melting colorless crystalline solids readily soluble in DMSO and acetone, poorly soluble in ethanol and toluene, and insoluble in alkanes and water. Their IR spectra showed N–H stretching band at 3175–3502 cm^{-1}), C≡N stretching band at 2185–2198 cm^{-1}, and carbonyl stretching bands in the region 1626–1758 cm^{-1}. The ^{1}H NMR spectra of **3a**–**3g** contained signals for aromatic protons, protons in the substituents R^1, R^2, R^3, ester ethoxy group (δ 0.78–0.90 t and 3.73–3.96 ppm, m) and amino group (δ 7.11–7.28 ppm, br.s). In the ^{13}C NMR spectra of **3a**–**3g** we observed signals of lactam (δ_C 178.1–179.0 ppm), ester (δ_C 161.3–165.0 ppm), and ketone (δ_C 197.1 ppm, **3b**) carbonyl carbon atoms, as well as a signal of the spiro carbon atom at δ_C 49.8– 50.7 ppm.

The structure of compound **3a** was confirmed by X-ray analysis. According to the X-ray diffraction data (Fig. 1), compound **3a** crystallized in the centrosymmetric space group belonging to the monoclinic crystal system. The pyran ring adopts a distorted *boat* conformation with the C^2 and O^4 atoms deviating from the $C^5C^6C^7C^8$ plane by 0.17 and 0.08 Å, respectively. The pyrrole ring is planar within 0.04 Å. The ethoxycarbonyl and methoxycarbonyl groups are turned through small angles relative to the pyrrole and pyran rings, respectively; the dihedral angles are $C^7C^8C^9O^5$ 7.5(4)° and $C^2C^8C^9O^6$ 3.5(3)°. Molecules **3a** in crystal are linked to form infinite two-dimensional networks through intermolecular hydrogen bonds N^3–$H^{3A}\cdots O^1$ [$-x$, $-0.5 + y$, $0.5 - z$] and N^3–$H^{3B}\cdots O^1$ [x, $0.5 - y$, $-0.5 + z$].

Scheme 1.



1, **4**, R^1 = $PhCH_2$ (**a**), C_6H_4Me-4 (**b**), Ph (**c**), Me (**d**); **2**, R^2 = Me, R^3 = COOMe (**a**), COMe (**b**), COOEt (**c**), R^2 = Ph, R^3 = COOEt (**d**); **3**, R^1 = $PhCH_2$, R^2 = Me, R^3 = COOMe (**a**), COMe (**b**), COOEt (**c**), R^1 = C_6H_4Me-4, R^2 = Me, R^3 = COOEt (**d**), R^1 = Ph, R^2 = Me, R^3 = COOEt (**e**), R^1 = R^2 = Me, R^3 = COOEt (**f**), R^1 = $PhCH_2$, R^2 = Ph, R^3 = COOEt (**g**).

Presumably, the formation of compounds **3a**–**3g** involves initial condensation of pyrroledione **1** through the ketone carbonyl group with malononitrile and subsequent addition of the β-CH and OH groups of the enol tautomer of **2** to the C^3 atom of the pyrrole ring in intermediate **4** and carbon atom of the cyano group, respectively. Analogous spiro heterocyclization was observed previously in the reaction of 2-(5-aryl-4-methyl-2-oxo-1,2-dihydro-3*H*-pyrrol-3-ylidene)malononitriles (analogs of **4**) with ethyl acetoacetate [5].

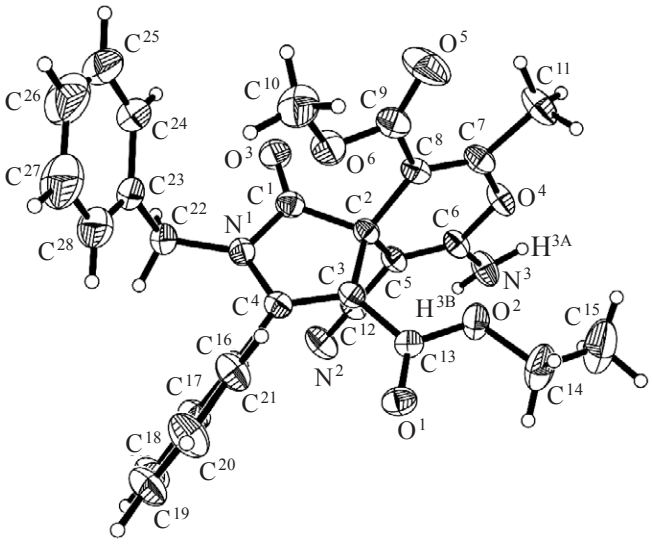


Fig. 1. Structure of the molecule of 4-ethyl 6-methyl 9-amino-2-benzyl-10-cyano-7-methyl-1-oxo-3-phenyl-8-oxa-2-aza-spiro[4.5]deca-3,6,9-triene-4,6-dicarboxylate (**3a**) according to the X-ray diffraction data. Non-hydrogen atoms are shown as thermal vibration ellipsoids with a probability of 30%.

The reaction described in this work is the first example of three-component spiro heterocyclization of substituted 1*H*-pyrrole-2,3-diones with malononitrile and acyclic enols with the formation of difficultly accessible 8-oxa-2-azaspiro[4.5]deca-3,6,9-triene system. Compounds containing a 2-amino-3-cyano-4*H*-pyran fragment are known to exhibit cytotoxicity [6] and antimicrobial activity [7–9].

EXPERIMENTAL

The IR spectra were recorded in mineral oil on a Perkin Elmer Spectrum Two spectrometer. The ^1H and ^{13}C NMR spectra were measured on a Bruker Avance III HD 400 spectrometer at 400 and 100 MHz, respectively, using DMSO-d_6 as solvent and hexamethyldisiloxane as internal standard. Elemental analysis was performed with a Vario Micro cube analyzer. The purity of the isolated compounds was checked by TLC on Silica gel 60 F_{254} plates (Merck) using toluene–ethyl acetate (3:1) as eluent; spots were visualized by treatment with iodine vapor and under UV light (λ 254 nm). The reaction conditions were optimized by HPLC/MS, and the mass spectra (positive electrospray ionization) were recorded with a Waters Acquity UPLC I-Class instrument; Acquity UPLC BEH C18 column (particle size 1.7 μm), eluent acetonitrile–water, flow rate 0.6 mL/min; PDA eλ UV detector and Xevo TQD mass-selective detector. Initial pyrroledione **1a**–**1d** were synthesized by reaction of the corresponding enamines with oxalyl chloride according to the procedure described in [10].

4-Ethyl 6-methyl 9-amino-2-benzyl-10-cyano-7-methyl-1-oxo-3-phenyl-8-oxa-2-azaspiro[4.5]deca-3,6,9-triene-4,6-dicarboxylate (3a). A solution of 335 mg (1.0 mmol) of pyrroledione **1a**, 66 mg (1.0 mmol) of malononitrile, 432 μL (4.0 mmol) of methyl 3-oxobutanoate, and 28 μL (0.2 mmol) of triethylamine in 10 mL of anhydrous acetonitrile was refluxed for 19 h. The mixture was cooled, and the precipitate was filtered off. Yield 338 mg (68%), colorless crystals, mp 206–208°C (from acetone). IR spectrum, ν, cm^{-1}: 3410, 3324, 3217, 3197 (NH_2), 2190 (CN), 1733, 1721, 1668, 1626 (C=O). ^1H NMR spectrum, δ, ppm: 0.84 t (3H, C**H**$_3$CH$_2$, J = 7.1 Hz), 2.33 s (3H, Me), 3.52 s (3H, OMe), 3.74–3.86 m (2H, CH$_3$C**H**$_2$), 4.43 d and 4.48 d (1H each, C**H**$_2$Ph, J = 15.9 Hz), 6.95–7.03 m (2H, H_{arom}), 7.08–7.13 m (2H, H_{arom}), 7.17 br.s (2H, NH_2), 7.18–7.22 m (3H, H_{arom}), 7.35–7.49 m (3H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 13.4 (**C**H$_3$CH$_2$), 18.8 (Me), 44.0 (**C**H$_2$Ph), 49.8 (C_{spiro}), 51.5 (OMe), 54.5, 58.7 (CH$_3$**C**H$_2$), 103.5, 112.6, 117.5, 127.1 (3C), 127.9 (2C), 128.0 (2C), 128.4 (2C), 129.4, 129.5, 136.3, 154.7, 159.3, 159.6, 161.3 (**C**OOEt), 165.0 (**C**OOMe), 179.0 (C^1=O). Mass spectrum: m/z 521.99 $[M + Na]^+$. Found, %: C 67.63; H 5.18; N 8.39. $C_{28}H_{25}N_3O_6$. Calculated, %: C 67.33; H 5.04; N 8.41. M + Na 522.16.

The X-ray diffraction data for compound **3a** were obtained with an Xcalibur Ruby single crystal diffractometer with a CCD detector according to standard procedure [Mo K_α radiation, 295(2) K, ω-scanning with a step of 1°]. A correction for absorption was applied empirically using SCALE3 ABSPACK algorithm [11]. Moniclinic crystal system, space group $P2_1/c$; $C_{28}H_{25}N_3O_6$, M 499.51; unit cell parameters: a = 14.912(3), b = 12.190(2), c = 14.942(3) Å; β = 108.92(2)°; V = 2569.4(10) Å^3; Z = 4; d_{calc} = 1.291 g/cm^3; μ = 0.092 mm^{-1}. The structure was solved using SHELXS [12] and was refined against F^2 by the full-matrix least-squares method in anisotropic approximation for all non-hydrogen atoms using SHELXL [13] with OLEX2 graphical interface [14]. Hydrogen atoms of the amino group were refined independently in isotropic approximation, and the positions of the other hydrogens were refined according to the riding model. Final divergence factors: R_1 = 0.0567 [3899 reflections with $I > 2\sigma(I)$], wR_2 = 0.1662 (all 6129 independent reflections); goodness of fit S = 1.047. The results were deposited to the Cambridge Crystallographic Data Centre (CCDC entry no. 1981651) and are available at www.ccdc.cam.ac.uk/data_request/cif.

Compounds **3b**–**3g** were synthesized in a similar way.

Ethyl 6-acetyl-9-amino-2-benzyl-10-cyano-7-methyl-1-oxo-3-phenyl-8-oxa-2-azaspiro[4.5]deca-3,6,9-triene-4-carboxylate (3b). Yield 183 mg (38%), colorless crystals, mp 168–169°C (from EtOH). IR spectrum, ν, cm^{-1}: 3390, 3293, 3181 (NH_2), 2188 (CN), 1723, 1698, 1682, 1651 (C=O). ^1H NMR spectrum, δ, ppm: 0.84 t (3H, C**H**$_3$CH$_2$, J = 7.1 Hz), 2.34 s (3H, Me), 2.35 s (3H Me), 3.73–3.85 m (2H, CH$_3$C**H**$_2$), 4.44 s (2H, C**H**$_2$Ph), 7.05–7.11 m (4H, H_{arom}), 7.12 br.s (2H, NH_2), 7.16–7.22 m (3H, H_{arom}), 7.30–7.42 m (3H, H_{arom}). ^{13}C NMR spectrum, δ, ppm: 13.5 (**C**H$_3$CH$_2$), 19.7 (Me), 31.3 (Me), 44.0 (**C**H$_2$Ph), 50.3 (C_{spiro}), 54.9, 58.7 (CH$_3$**C**H$_2$), 112.1, 114.7, 117.6, 126.6 (2C), 126.9, 127.8 (2C), 128.0 (2C), 128.3 (2C), 129.3, 129.7, 136.6, 154.7, 157.4, 159.9, 161.5 (**C**OOEt), 179.0 (C^1=O), 197.1 (**C**OMe). Mass spectrum: m/z 506.05 $[M + Na]^+$. Found, %: C 69.38; H 5.30; N 8.73. $C_{28}H_{25}N_3O_5$. Calculated, %: C 69.55; H 5.21; N 8.69. M + Na 506.17.

Diethyl 9-amino-2-benzyl-10-cyano-7-methyl-1-oxo-3-phenyl-8-oxa-2-azaspiro[4.5]deca-3,6,9-triene-4,6-dicarboxylate (3c). Yield 210 mg (41%), colorless crystals, mp 180–182°C (from EtOH). IR spectrum, ν, cm^{-1}: 3454, 3328, 3175 (NH_2), 2186 (CN), 1738, 1697, 1667, 1630 (C=O). ^1H NMR spectrum, δ, ppm: 0.85 t (3H, C**H**$_3$CH$_2$, J = 7.1 Hz), 1.11 t (3H, C**H**$_3$CH$_2$, J = 7.1 Hz), 2.33 s (3H, Me), 3.73–3.87 m (2H, CH$_3$C**H**$_2$), 3.97–4.18 m (2H, CH$_3$C**H**$_2$), 4.34 d and 4.54 d (1H each, C**H**$_2$Ph, J = 16.0 Hz), 6.99–7.25 m (9H, H_{arom}, NH_2), 7.33–7.49 m (3H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 13.4 (**C**H$_3$CH$_2$), 13.9 (**C**H$_3$CH$_2$), 18.9 (Me), 44.2 (**C**H$_2$Ph), 49.8 (C_{spiro}), 54.6, 58.7 (CH$_3$**C**H$_2$), 60.5 (CH$_3$**C**H$_2$), 103.7, 112.7, 117.6, 126.9 (2C), 127.1, 127.9 (2C), 128.0 (2C), 128.4 (2C), 129.5, 129.5, 136.4, 154.6, 159.1, 159.7, 161.3 and 164.6 (**C**OOEt), 179.0 (C^1=O). Mass spectrum: m/z 514.11 $[M + H]^+$. Found, %: C 67.99; H 5.47; N 8.27. $C_{29}H_{27}N_3O_6$. Calculated, %: C 67.83; H 5.30; N 8.18. M + H 514.20.

Diethyl 9-amino-10-cyano-7-methyl-2-(4-methylphenyl)-1-oxo-3-phenyl-8-oxa-2-azaspiro[4.5]deca-3,6,9-triene-4,6-dicarboxylate (3d). Yield 272 mg (53%), colorless crystals, mp 181–183°C (from EtOH). IR spectrum, ν, cm^{-1}: 3502, 3406, 3333, 3237, 3208 (NH_2), 2190 (CN), 1758, 1694, 1668, 1627 (C=O). ^1H NMR spectrum, δ, ppm: 0.90 t (3H, C**H**$_3$CH$_2$, J = 7.1 Hz), 1.21 t (3H, C**H**$_3$CH$_2$, J = 7.1 Hz), 2.22 s and 2.36 s (6H, Me), 3.77–3.96 m (2H, CH$_3$C**H**$_2$), 4.12–4.30 m (2H, CH$_3$C**H**$_2$), 6.81–6.92 m (2H, H_{arom}), 7.05–

7.18 m (4H, H_{arom}), 7.21 br.s (2H, NH_2), 7.25–7.35 m (3H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 13.5 ($\mathbf{C}H_3CH_2$), 14.1 ($\mathbf{C}H_3CH_2$), 18.9 (Me), 20.5 (Me), 50.0 (C_{spiro}), 54.8, 58.9 ($CH_3\mathbf{C}H_2$), 60.7 ($CH_3\mathbf{C}H_2$), 103.5, 112.7, 117.5, 127.6 (2C), 127.6 (2C), 129.0 (2C), 129.1, 129.3 (2C), 129.6, 131.5, 137.4, 153.7, 159.5, 159.7, 161.6 and 164.7 (**C**OOEt), 178.2 (C^1=O). Mass spectrum: *m*/*z* 514.02 $[M + H]^+$. Found, %: C 68.05; H 5.14; N 8.10. $C_{29}H_{27}N_3O_6$. Calculated, %: C 67.83; H 5.30; N 8.18. *M* + H 514.20.

Diethyl 9-amino-10-cyano-7-methyl-1-oxo-2,3-diphenyl-8-oxa-2-azaspiro[4.5]deca-3,6,9-triene-4,6-dicarboxylate (3e). Yield 264 mg (53%), colorless crystals, mp 185–187°C (from EtOH). IR spectrum, ν, cm^{-1}: 3435, 3328, 3265, 3222, 3193 (NH_2), 2190 (CN), 1743, 1716, 1673, 1637 (C=O). ^1H NMR spectrum, δ, ppm: 0.91 t (3H, $C\mathbf{H}_3CH_2$, *J* = 7.1 Hz), 1.22 t (3H, $C\mathbf{H}_3CH_2$, *J* = 7.1 Hz), 2.37 s (3H, Me), 3.81–3.94 m (2H, $CH_3C\mathbf{H}_2$), 4.15–4.28 m (2H, $CH_3C\mathbf{H}_2$), 6.98–7.02 m (2H, H_{arom}), 7.13–7.17 m (2H, H_{arom}), 7.20–7.37 m (8H, H_{arom}, NH_2). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 13.5 ($\mathbf{C}H_3CH_2$), 14.1 ($\mathbf{C}H_3CH_2$), 19.0 (Me), 50.2 (C_{spiro}), 54.8, 59.0 ($CH_3\mathbf{C}H_2$), 60.7 ($CH_3\mathbf{C}H_2$), 103.5, 112.9, 117.6, 127.6 (2C), 127.8 (2C), 127.9, 128.8 (2C), 129.0 (2C), 129.1, 129.5, 134.2, 153.6, 159.5, 159.8, 161.6 and 164.7 (**C**OOEt), 178.1 (C^1=O). Mass spectrum: *m*/*z* 500.11 $[M + H]^+$. Found, %: C 67.59; H 5.01; N 8.34. $C_{28}H_{25}N_3O_6$. Calculated, %: C 67.33; H 5.04; N 8.41. *M* + H 500.18

Diethyl 9-amino-10-cyano-2,7-dimethyl-1-oxo-3-phenyl-8-oxa-2-azaspiro[4.5]deca-3,6,9-triene-4,6-dicarboxylate (3f). Yield 223 mg (51%), colorless crystals, mp 218–219°C (from EtOH). IR spectrum, ν, cm^{-1}: 3427, 3285, 3266, 3176 (NH_2), 2185 (CN), 1716, 1697, 1678, 1661 (C=O). ^1H NMR spectrum, δ, ppm: 0.88 t (3H, $C\mathbf{H}_3CH_2$, *J* = 7.1 Hz), 1.14 t (3H, $C\mathbf{H}_3CH_2$, *J* = 7.1 Hz), 2.33 s (3H, CH_3), 2.74 s (3H, CH_3), 3.79–3.85 m (2H, $CH_3C\mathbf{H}_2$), 4.05–4.13 m (2H, $CH_3C\mathbf{H}_2$), 7.11 s (2H, NH_2), 7.29–7.34 m (2H, H_{arom}), 7.50–7.55 m (3H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 13.4 ($\mathbf{C}H_3CH_2$), 13.8 ($\mathbf{C}H_3CH_2$), 18.7, 27.5, 49.9 (C_{spiro}), 54.6, 58.7 ($CH_3\mathbf{C}H_2$), 60.4 ($CH_3\mathbf{C}H_2$), 103.4, 112.2, 117.3, 128.2 (2C), 128.4 (2C), 129.6 (2C), 154.6, 159.5, 159.5, 161.4, 164.5, 178.9 (C^1=O). Mass spectrum: *m*/*z* 437.92 $[M + H]^+$. Found, %: C 63.21; H 5.18; N 9.58. $C_{23}H_{23}N_3O_6$. Calculated, %: C 63.15; H 5.30; N 9.61. *M* + H 438.17.

Diethyl 9-amino-2-benzyl-10-cyano-1-oxo-3,7-diphenyl-8-oxa-2-azaspiro[4.5]deca-3,6,9-triene-4,6-dicarboxylate (3g). Yield 274 mg (48%), colorless crystals, mp 192–193°C (from EtOH). IR spectrum, ν, cm^{-1}: 3406, 3326, 3266, 3256, 3208 (NH_2), 2198 (CN), 1735, 1697, 1668, 1627 (C=O). ^1H NMR spectrum, δ, ppm: 0.78 t (3H, $C\mathbf{H}_3CH_2$, *J* = 7.1 Hz), 0.87 t (3H, $C\mathbf{H}_3CH_2$, *J* = 7.1 Hz), 3.80–3.90 m (4H, $CH_3C\mathbf{H}_2$), 4.41 d and 4.57 d (1H each, $PhC\mathbf{H}_2$, *J* = 16.1 Hz), 7.01–7.14 m (4H, H_{arom}), 7.17–7.24 m (3H, H_{arom}), 7.28 s (2H, NH_2), 7.34–7.52 (8H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 13.1 ($\mathbf{C}H_3CH_2$), 13.5 ($\mathbf{C}H_3CH_2$), 44.1 ($Ph\mathbf{C}H_2$), 50.7 (C_{spiro}), 54.3, 58.9 ($CH_3\mathbf{C}H_2$), 60.4 ($CH_3\mathbf{C}H_2$), 105.3, 112.3, 117.6, 126.7 (2C), 127.0, 127.9 (2C), 128.0 (2C), 128.1 (4C), 128.3 (2C), 129.3, 129.4, 130.0, 133.0, 136.2, 155.1, 156.5, 160.3, 161.3, 164.8, 178.5 (C^1=O). Mass spectrum: *m*/*z* 576.09 $[M + H]^+$. Found, %: C 71.20; H 5.15; N 7.24. $C_{34}H_{29}N_3O_6$. Calculated, %: C 70.94; H 5.08; N 7.30. *M* + H 576.21.

FUNDING

This study was performed under financial support by the Ministry of Science and Higher Education of the Russian Federation (project no. FCHF-2020-0008) and by the Government of Perm Krai.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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