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SYNTHESIS OF BRANCHED POLYNUCLEAR 1,3,4-OXADIAZOLES

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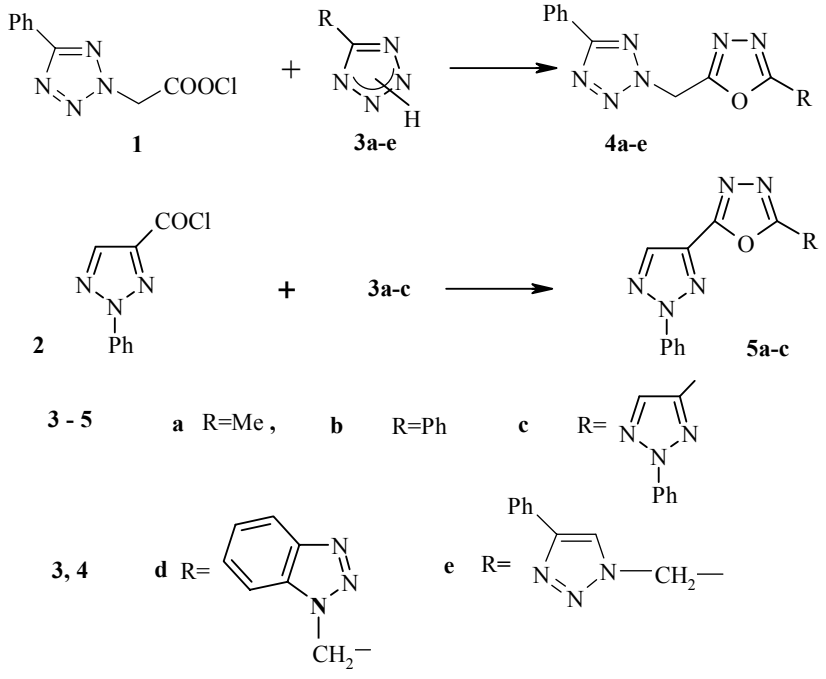
The reaction of 5-substituted mono- and polytetrazoles with heterocyclic acid chlorides and trifluoroacetic acid anhydride gave branched polynuclear 1,3,4-oxadiazole systems.

Keywords: trifluoroacetic acid anhydride, polynuclear 1,3,4-oxadiazoles, polynuclear tetrazoles, heterocyclic acid anhydrides, thermolysis of tetrazoles.

In searches for novel photosensitive [1] and biologically active materials [2-4] a broad series of 1,3,4-oxadiazoles with different substituents on the carbon atoms have been reported. The usual basis for their synthesis is mainly the dehydration of the corresponding acid hydrazides [5]. However, this reaction is done in relatively severe conditions and does not permit preparation of polynuclear systems with different proportions of oxadiazole rings and other heterocycles as substituents on the oxadiazoles. To a certain extent this restriction is



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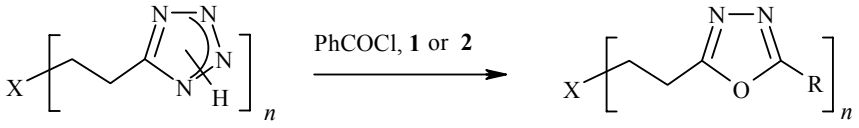
removed *via* thermolysis of tetrazoles in the presence of mono- and dicarboxylic acid chlorides [6, 7]. In this report we discuss the possible synthesis of mono- and polynuclear branched 1,3,4-oxadiazoles including those having 1,2,3-triazole and tetrazole components. With this in view the (5-phenyltetrazol-2-yl)acetic (**1**) and (2-phenyl-1,2,3-triazol-4-yl)carboxylic acid chlorides (**2**) were used in the reaction with 5-substituted tetrazoles. The reaction of these acid chlorides with tetrazoles **3a-e** occurs quite rapidly at 70-85ºC to give the corresponding tetrazole and triazole-substituted oxadiazoles **4, 5** in 60-70% yield.

The use of the heterocyclic acid chlorides makes possible the preparation of polynuclear systems with a different combination of triazole, tetrazole, and oxadiazole rings.



6a,b **7a–c**

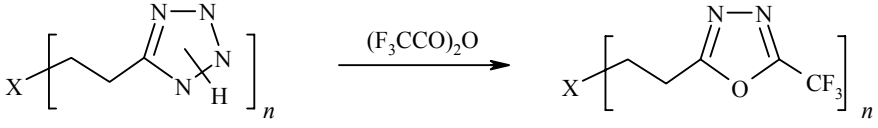
6, 7 a X = $>C(COOEt)_2$, $n = 2$; **b** X = $=CNO_2$, $n = 3$;

7 a,b R = Ph, **c** R = phenyltriazol-5-yl, X = $=CNO_2$, $n = 3$;

The successful use of acid chlorides **1** and **2** in the reaction with the monotetrazoles **3a-e** led us to expect them to be no less active in reaction with the polytetrazoles **6a-c**. However, not all of the polynuclear tetrazoles and the acid chlorides **1, 2** and also benzoyl chloride form the corresponding branched oxadiazoles. We were only able to separate and identify the products of reaction of compounds **6a** and **6b** with benzoyl chloride (**7a and 7b** respectively) and also compound **6b** with acid chloride **2** (compound **7c**) in 35-46% yields.

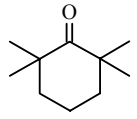
The thermolysis reaction of tetrazoles **6a-c** with acid chlorides **1** and **2** and benzoyl chloride at 90-100°C led to formation of tarry products which markedly hindered or made impossible the separation of pure compounds. Hence the diethyl bis[2-(tetrazol-5-yl)ethyl]malonate **6a** and acid chlorides **1** and **2** gave total tarring. The latter and the low yields of targeted products is evidently connected with their low stability in rather rigid conditions of both the polytetrazoles **6a-c** and the oxadiazoles **7a-c** formed. For this reason it was not possible to carry out a reaction of different acid chlorides with the polynuclear tetrazole **6c** (where X is a cyclohexane residue with unsubstituted atoms C-2 and C-6, $n = 4$).

On the other hand it was found that the polynuclear tetrazoles **6a-c** react with trifluoroacetic acid anhydride even at 10-15°C in low boiling solvents (methylene chloride, ether, benzene) without signs of tarring to give the corresponding trifluoromethyl-substituted branched polyoxadiazoles **8a-c** in high yields.



6a–c **8a–c**

6, 8 a X = $>C(COOEt)_2$, $n = 2$; **b** X = $=CNO_2$, $n = 3$;

c X =  ; $n = 4$

The composition and structure of the synthesized compounds **4-8** were confirmed by the results of elemental analysis and ^{13}C NMR spectroscopic data.

The ^{13}C NMR spectra of compounds **4, 5** show signals for the carbon atoms of the oxadiazole, tetrazole, and triazole rings in the regions 154-170, 150-160, and 130-145 ppm respectively. The chemical shifts of the carbon atoms signals for the phenyl substituents on different heterocycles are typical. Hence in the case of a benzene ring at atom N-2 of a triazole ring they are found at 117-131 ppm. More specifically, the C_o (double intensity) chemical shift occurs at 117-118 ppm and C_i at 130 ppm. The carbon atom signals for the benzene ring on atom C-5 of the tetrazole ring occur at 126-131 ppm and on atom C-2 of the oxadiazole ring at 122-132 ppm. In the latter case the high field shift of C_i (122 ppm) and low field shift of C_p (132 ppm) are typical. The observed chemical shift features allow an unambiguous assignment of the signals in the ^{13}C NMR spectra and to establish or confirm the structures of the compounds obtained. The ^{13}C NMR spectra of compounds **8a-c** show characteristic signals in the regions 160-173 and 155-159 ppm respectively for the atoms C-2 and C-5 of the 1,3,4-oxadiazole ring and also within the range 116-120 ppm assigned to the trifluoromethyl group. Additional confirmation of the positioning of the CF_3 group at position 5 of the oxadiazole ring is the equel splitting of the heterocycle C-5 atom signal as a quartet with the intensity ratio of the lines 1:3:3:1. The $^{13}C–^{19}F$ spin-spin splittings are 267-272 and 42-44 Hz respectively. Unfortunately a marked lowering of solubility was noted for these compounds with an increasing number of azole rings in the polynuclear oxadiazole molecules. The majority of the 1,3,4-oxadiazoles prepared have certain photoactivity as displayed by luminosity upon UV irradiation.

EXPERIMENTAL

^1H and ^{13}C NMR spectra were recorded on a Varian VXR-500S spectrometer (500 and 126 MHz respectively) using acetone-d_6, acetone, DMSO, or HMPA. In the case of acetone-d_6 the internal standard was HMDS but for the other solvents acetone-d_6 was added for "capture". Elemental analysis was performed on a FLASH EA 1112 Series CHN-analyzer. The tetrazoles **3c-e** were prepared by method [8] and the branched polynuclear tetrazoles **6a-c** as previously described [9, 10].

5-Phenyltetrazol-2-ylacetic Acid Chloride (1). A mixture of 5-phenyltetrazol-2-ylacetic acid (1.25 g, 6.1 mmol) and PCl_5 (1.5 g, 7.4 mmol) in benzene (10 ml) was heated to 60ºC and held until hydrogen chloride evolution had ceased and the starting acid had dissolved. After distillation of solvent and liquid products *in vacuo* on a water pump the dry residue was recrystallized from hexane to give colorless needle-like crystals of the acid chloride **1** (1.2 g, 88%) with mp 101°C (mp 103-104ºC [11]).

2-Phenyl-1,2,3-triazol-4-ylcarboxylic Acid Chloride (2). PCl_5 (0.7 g, 3.2 mmol) was added in small portions to 2-phenyl-1,2,3-triazol-4-ylcarboxylic acid (0.5 g, 2.6 mmol) in benzene (5 ml). The reaction product was held at room temperature until evolution of hydrogen chloride had ceased and the acid had dissolved completely. After distillation of solvent and liquid products the dry residue was recrystallized from hexane to give the acid chloride **2** (0.51 g, 94%) with mp 107-108°C.

2-Methyl-5-(5-phenyltetrazol-2-ylmethyl)-1,3,4-oxadiazole (4a). A mixture of the acid chloride **1** (1.15 g, 5.1 mmol) and 5-methyltetrazole **3a** (0.36 g, 4.3 mmol) in toluene (5 ml) was held at 85°C for 3 h until gas formation had ceased and a homogeneous mass had been formed. The reaction product was cooled and neutralized over 30 min with saturated Na_2CO_3 solution. The organic layer was separated and dried over $MgSO_4$. After removal of solvent the dry residue was recrystallized from ethanol to give the product **4a** (0.7 g, 67%) with mp 112-113°C. ^{13}C NMR spectrum (DMSO), δ, ppm: 8.22 (CH_3); 44.6 (CH_2); 124-128.8 (m, C_6H_5); 157.7 (C tetrazole); 162.6 (C-2 oxadiazole); 163.4 (C-5 oxadiazole). Found, %: C 54.21; H 3.85; N 34.22. $C_{11}H_{10}N_6O$. Calculated, %: C 54.55; H 4.13; N 34.71.

Compounds 4b,d, 5a-c and **7a-c** were prepared similarly.

5-Phenyl-2-(5-phenyltetrazol-2-ylmethyl)-1,3,4-oxadiazole (4b) is prepared from the acid chloride **1** (0.54 g, 2.5 mmol) and 5-phenyltetrazole **3b** (0.24 g, 1.6 mmol) in toluene (5 ml). Yield 0.35 g (71%); mp 158-160°C (a mixture of ethanol and DMF). ^{1}H NMR spectrum (acetone-d_6), δ, ppm: 6.40 (2H, s, CH_2); 7.52-7.59 (5H, m, H_{Ph} at tetrazole); 8.02-8.13 (5H, m, H_{Ph} at oxadiazole). ^{13}C NMR spectrum (HMPA), δ, ppm: 46.9 (CH_2); 122.83 (C_i Ph at oxadiazole); 125.94 (C_o Ph at tetrazole); 126.06 (C_m Ph at tetrazole); 126.63 (C_p Ph at tetrazole); 128 87 (C_o Ph at oxadiazole); 129.3 (C_m Ph at oxadiazole); 130.35 (C_i Ph at tetrazole); 132.02 (C_p Ph at oxadiazole); 160.2 (C tetrazole); 164.5 (C-2 oxadiazole); 164.8 (C-5 oxadiazole). Found, %: C 63.31; H 3.82; N 27.24. $C_{16}H_{12}N_6O$. Calculated, %: C 63.15; H 3.95; N 27.63.

2-(2-Phenyl-1,2,3-triazol-4-yl)-5-(5-phenytetrazol-2-ylmethyl)-1,3,4-oxadiazole (4c) is prepared from the acid chloride **1** (1 g, 4.5 mmol) and 5-(2-phenyl-1,2,3-triazol-4-yl)tetrazole **3c** (0.64 g, 3 mmol) in toluene (7 ml). Yield 0.8 g (72%); mp 142-143ºC (benzene). ^{13}C NMR spectrum (DMSO), δ, ppm: 46.3 (CH_2); 118.6 (C_o Ph at triazole); 125.7 (C_p Ph at triazole); 126.03 (C_m Ph at triazole); 130.5 (C_i Ph at triazole); 128.65 (C_p Ph at tetrazole); 128.9 (C_m Ph at tetrazole); 129.5 (C_o Ph at tetrazole); 133.8 (C_i Ph at tetrazole); 135.9 (C-4 triazole); 137.9 (C-5 triazole); 158.2 (C tetrazole); 159.9 (C-2 oxadiazole); 164.4 (C-5 oxadiazole). Found, %: C 58.95; H 3.67; N 33.85. $C_{18}H_{13}N_9O$. Calculated, %: C 58.55; H 3.50; N 33.96.

2-(Benzotriazol-1-ylmethyl)-5-(5-phenyltetrazol-2-ylmethyl)-1,3,4-oxadiazole (4d) is prepared from the acid chloride **1** (1 g, 4.5 mmol) and (N-methyltetrazol-5-yl)benzotriazole **3d** (0.9 g, 4.5 mmol) in toluene (7 ml). Yield 0.35 g (22%); mp 245-247°C (decomp., ethanol). ^{13}C NMR spectrum (HMPA), δ, ppm: 46.9 (CH_2 at tetrazole); 51.5 (CH_2 at benzotriazole); 109.6-132 (Ph at benzotriazole and at Ph tetrazole, assignments ambiguous); 142.6 (C-4 tetrazole); 143.9 (C-5 triazole); 160.9 (C tetrazole); 162.3 (C-2 oxadiazole); 162.6 (C-5 oxadiazole). Found, %: C 56.32; H 3.25; N 34.72. $C_{17}H_{13}N_9O$. Calculated, %: C 56.82; H 3.62; N 35.1.

2-(4-Phenyl-1,2,3-triazol-1-ylmethyl)-5-(5-phenyltetrazol-2-ylmethyl)-1,3,4-oxadiazole (4e) is prepared from the acid chloride **1** (0.43 g, 1.9 mmol) and tetrazole **3e** (0.37 g, 1.6 mmol) in toluene (5 ml). Yield 0.26 g (42%); mp 105°C (ethanol). ^{13}C NMR spectrum (DMSO), δ, ppm: 40.45 (CH_2 at triazole); 44.45 (CH_2 at tetrazole); 136.1 (C-4 triazole); 142.5 (C-5 triazole); 158.8 (C tetrazole); 160.5 (C-2 oxadiazole); 162.7 (C-5 oxadiazole); 120.3-130.9 (Ph at triazole and Ph at tetrazole, assignments ambiguous). Found, % C 59.41; H 3.78; N 32.47; $C_{19}H_{15}N_9O$. Calculated, %: C 59.22; H 3.9; N 32.73.

5-Methyl-2-(2-phenyl-1,2,3-triazol-4-yl)-1,3,4-oxadiazole (5a) is prepared from the acid chloride **2** (0.5 g, 2.4 mmol) and tetrazole **3a** (0.35 g, 4.1 mmol) in toluene (5 ml). Yield 0.6 g (67%); mp 132°C (ethanol). ^{13}C NMR spectrum (acetone), δ, ppm: 10 (CH_3); 119.2 (C_o Ph); 128.8 (C_p Ph); 129.8 (C_m Ph); 135.6 (C_i Ph); 135.7 (C-4 triazole); 139.5 (C-5 triazole); 157.9 (C-2 oxadiazole); 164.4 (C-5 oxadiazole). Found, %: C 58.23; H 3.54; N 30.42. $C_{11}H_9N_5O$. Calculated, %: C 58.15; H 3.96; N 30.84.

5-Phenyl-2-(2-phenyl-1,2,3-triazol-4-yl)-1,3,4-oxadiazole (5b) is prepared from the acid chloride **2** (0.54 g, 2.6 mmol) and tetrazole **3b** (0.25 g, 1.7 mmol) in toluene (10 ml). Yield 0.26 g (53%); mp 195°C (mixture of ethanol and DMF). ^{13}C NMR spectrum (HMPA), δ, ppm: 164.04 (C-5 oxadiazole); 157.1 (C-2 oxadiazole); 136.6 (C-5 triazole); 132.4 (C-4 triazole); 118.6 (C_o Ph at triazole); 126.5 (C_p Ph at triazole); 127.4 (C_m Ph at triazole); 135.2 (C_i Ph at triazole); 122.1 (C_o Ph at oxadiazole); 129.4 (C_p Ph at oxadiazole); 129.8 (C_m Ph oxadiazole). 138.4 (C_i Ph at oxadiazole). Found, %: C 66.12; H 3.85; N 24.05. $C_{16}H_{11}N_5O$. Calculated, %: C 66.44; H 3.81; N 24.22.

2,5-Bis(2-phenyl-1,2,3-triazol-4-yl)-1,3,4-oxadiazole (5c) is prepared from the acid chloride **2** (1 g, 4.8 mmol) and tetrazole **3c** (0.68 g, 3.2 mmol) in toluene (7 ml). Yield 0.93 g (84.5%); mp 217-219ºC (mixture of benzene and DMF). ^{13}C NMR spectrum HMPA), δ, ppm: 117.2 (C_o Ph); 127.5 (C_p Ph); 128.5 (C_m Ph); 135.5 (C_i Ph); 132.9 (C-4 triazole); 137.2 (C-5 triazole); 156.3 (C-2 oxadiazole); 162.6 (C-5 oxadiazole). Found, %: C 60.84; H 3.47; N 31.66. $C_{18}H_{12}N_8O$. Calculated, %: C 60.67; H 3.37; N 31.46.

Diethyl bis[2-(2-phenyl-1,3,4-oxadiazol-5-yl)ethyl]malonate (7a) is obtained from the bistetrazole **6a** (1 g, 2.8 mmol) and benzoyl chloride (1.2 g, 8.5 mmol) in toluene (10 ml). Yield 0.65 g (45%); mp 118-120°C (ethanol). ^{13}C NMR spectrum (acetone), δ, ppm: 13.6 (CH_3); 20.8 ($\underline{C}H_2C_{quat}$); 30.15 ($\underline{C}H_2$ at oxadiazole); 61.7

(CH_2O); 56.7 (C_{quat}); 124.4 (C_i Ph); 126.6 (C_o Ph); 129.3 (C_m Ph); 131.7 (C_p Ph); 164.6 (C-2 oxadiazole); 166.2 (C-5 oxadiazole); 170.3 (CO). Found, %: C 64.21; H 5.25; N 11.03. $C_{27}H_{28}N_4O_6$. Calculated, %: C 64.29; H 5.56; N 11.11.

Tris[2-(2-phenyl-1,3,4-oxadiazol-5-yl)ethyl]nitromethane (7b) is prepared from the tristetrazole **6b** (1 g, 2.9 mmol) and benzoyl chloride (1.8 g, 12.9 mmol) in toluene (10 ml). Yield 0.57 g (35%); mp 184-186°C (ethanol). ^{13}C NMR spectrum (HMPA), δ, ppm: 19.6 ($\underline{C}H_2$ at oxadiazole); 30.5 ($\underline{C}H_2CH_2$ at oxadiazole); 92.8 (CNO_2); 123.4 (C_i Ph); 125.8 (C_o Ph); 129 (C_m Ph); 131.4 (C_p Ph); 163.7 (C-2 oxadiazole); 165.3 (C-5 oxadiazole). Found, %: C 64.29; H 4.75; N 16.52. $C_{31}H_{27}N_7O_5$. Calculated, %: C 64.47; H 4.68; N 16.98.

Tris[2{2-(2-phenyl-1,2,3-triazol-4-yl)-1,3,4-oxadiazol-5-yl}ethyl]nitromethane (7c) is prepared from the tristetrazole **6b** (0.5 g, 1.4 mmol) and the acid chloride **2** (1.04 g, 5 mmol) in toluene (10 ml). Yield 0.5 g (46%); mp 223-225°C (decomp, mixture of ethanol and DMF). ^{13}C NMR spectrum (HMPA), δ, ppm: 19.6 ($\underline{C}H_2$ oxadiazole); 30.4 ($\underline{C}H_2CH_2$ at oxadiazole); 92.8 (C_{tert}); 118.2 (C_o Ph); 128.5 (C_p Ph); 129.7 (C_m Ph); 134.9 (C_i Ph); 135.8 (C-4 triazole); 138.5 (C-5 triazole); 157.1 (C-2 oxadiazole); 165.9 (C-5 oxadiazole). Found, %: C 56.84; H 3.65; N 28.44. $C_{37}H_{30}N_{16}O_5$. Calculated, %: C 57.07; H 3.86; N 28.79.

Diethyl Bis[2-(5-trifluromethyl-1,3,4-oxadiazol-2-yl)ethyl]malonate (8a). Trifluoroacetic acid anhydride (5.4 g, 25.6 mmol) was added dropwise at room temperature to the bistetrazole **6a** (3 g, 8.5 mmol) in methylene chloride (10 ml). After gas evolution had ceased the reaction product was neutralized with a saturated solution of Na_2CO_3. The aqueous layer was extracted with methylene chloride and the combined extract was dried over magnesium sulphate. Solvent was removed in air and the dry residue was recrystallized to give product **8a** (2.74 g, 87%) with mp 64-65°C (ethanol). ^1H NMR spectrum (acetone-d_6), δ, ppm (*J*, Hz): 2.04 (6H, t, J = 7, $2C\underline{H}_3CH_2$); 2.56 (4H, $2CH_2$ at oxadiazole); 3.15 (4H, $2C\underline{H}_2$ C_{quat}); 4.24 (4H, q, J = 7, $2CH_3C\underline{H}_2$). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 14.3 (CH_3); 21.6 (CH_2 at oxadiazole); 30.3 ($\underline{C}H_2CH_2$ at oxadiazole); 57.2 (C_{quat}); 62.6 (CH_2O); 116.6 (q, $J_{^{13}C^{19}F}$ = 270.2, CF_3); 156 (q, J = 43.6, C-5 oxadiazole); 169.9 (C-2 oxadiazole); 170.8 (C=O). Found, %: C 41.73; H 3.57; N 11.41. $C_{17}H_{18}F_6N_4O_6$. Calculated, %: C 41.80; H 3.69; N 11.48.

Tris[2-(5-trifluoromethyl-1,3,4-oxadiazol-2-yl)ethyl]nitromethane (8b) is prepared similarly to compound **8a** from the tristetrazole **6b** (0.5 g, 1.47 mmol) and trifluoroacetic acid anhydride (1.5 g, 7.2 mmol) in methylene chloride (10 ml). Yield 0.65 g (80%); mp 98-100°C (ethanol). ^{13}C NMR spectrum (acetone-d_6), δ, ppm (*J*, Hz): 20.1 (CH_2 at oxadiazole); 30.7 (C, $\underline{C}H_2CH_2$ at oxadiazole); 92.01 (CNO_2); 116.7 (q, $J_{^{13}C,^{19}F}$ = 267.7, CF_3); 155 (q, $J_{^{13}C,^{19}F}$ = 42.5, C-5 oxadiazole); 168.3 (C-2 oxadiazole). Found, %: C 34.97; H 2.24; N 17.58. $C_{16}H_{12}F_9N_7O_5$. Calculated, %: C 34.72; H 2.17; N 17.72.

2,2,6,6-Tetrakis[2-(5-trifluoromethyl-1,3,4-oxadiazol-2-yl)ethyl]cyclohexanone (8c) is prepared similarly to compound **8a** from the tetrakistetrazole (1.4 g, 2.9 mmol) and trifluoroacetic acid anhydride (3.6 g, 17.3 mmol) in methylene chloride (10 ml). Yield 1.17 g (54%) as colorless crystals; mp 75-77°C (ethanol). ^{13}C NMR spectrum (DMSO), δ, ppm (J, Hz): 19.3 (CH_2 at oxadiazole); 30.9 ($\underline{C}H_2CH_2$ at oxadiazole); 115.6 (q, $J_{^{13}C^{19}F}$ = 271.8, CF_3); 153.6 (q, $J_{^{13}C,^{19}F}$ = 43.7, C-5); 168.8 (C-5 oxadiazole); 215.2 (C=O); 15.4 (γ-CH_2 cyclohexane); 31.7 (β-CH_2 cyclohexane); 49.2 (α-CH_2 cyclohexane). Found, %: C 41.13; H 2.77; N 14.46. $C_{26}H_{22}F_{12}N_8O_5$. Calculated, %: C 41.38; H 2.92; N 14.85.

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