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SYNTHESIS OF SUBSTITUTED METHYL 2-OXO-4-ARYL-6-STYRYL-1,2,3,4-TETRAHYDROPYRIMIDINE-5-CARBOXYLATES

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Abstract. *The paper presents a study on the directed and previously unreported in the literature synthesis of methyl-substituted (E)-styrylpyrimidine-5-carboxylates, obtained by the reaction of dihydropyrimidine-2(1H)-ones with substituted aromatic aldehydes. The structures of the compounds obtained were confirmed by NMR spectroscopy and mass spectrometry*

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Introduction

Pyrimidines is a well-known class of heterocyclic compounds. They have attracted considerable attention due to their wide range of pharmaceutical and synthetic properties. They possess important therapeutic and pharmacological properties and are the most commonly used heterocycles in medicinal chemistry. Their derivatives are widely occurred in nature and have antimalarial [1–3], antibacterial [4, 5], antifungal [6, 7], anti-HIV [8], antiviral [9, 10], antitumour [11], and antiparasitic [12] activity.

According to research, derivatives of dihydropyrimidine-2(1*H*)-ones may also be used as antihypertensive agents [13] and α_1-adrenergic antagonists [14]. In this regard, the development of methods for synthesising new dihydropyrimidine-2(1*H*)-one derivatives and the investigation of their beneficial properties, with a view to producing safe and highly effective medicinal products, are the most crucial tasks in modern chemistry.

Nowadays, the development of promising pharmaceutical substrates is typically performed using *one-pot* synthesis methods. For instance, the Biginelli reaction [15] enables the synthesis of 3,4-dihydropyrimidine-2(1*H*)-ones via a three-component condensation. Furthermore, the introduction of a steric group into this class of heterocyclic compounds has

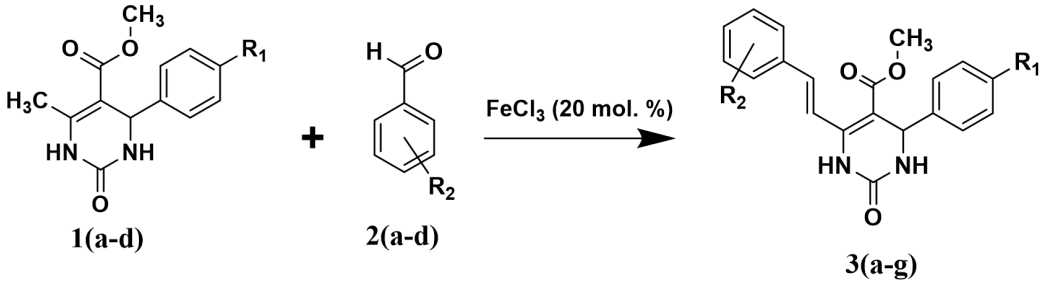
led to the discovery of a number of substances possessing biological properties that are beneficial to humans [16]. Latter can be achieved by vinylene-aldol condensation of the Biginelli products with aldehydes, using iron(III) as a catalyst [17].

Over the past few years, the use of iron-catalysed multi-component reactions has increased significantly. This is primarily due to their availability and the low cost of the catalyst. Such reactions are generally non-toxic, stable, and environmentally friendly [18, 19]. Applying these reactions to methyl (*E*)-styryl-pyrimidine-5-carboxylates makes it possible to expand the range of compounds of this class and obtain substances with specific desirable properties.

Main body

The starting compounds used were methyl-substituted 4-aryl-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylates **1(a–d)**, synthesised via a *one-pot* three-component condensation based on the classic Biginelli reaction [20].

Structures **1(a–d)** reacted with aromatic aldehydes **2(a–c)** in the presence of catalytic amounts of $FeCl_3 \cdot 6H_2O$ (20 mol.%). The reaction was conducted in acetonitrile under reflux for 24 hours. As a result, new methyl (*E*)-styryl-pyrimidine-5-carboxylates **3(a–g)** were obtained in yields of up to 49% (Scheme 1). Table 1 shows results for compounds **3(a–g)**.



1: R_1 = H (**a**), R_1 = Cl (**b**), R_1 = F (**c**), R_1 = Me (**d**); **2**: R_2 = 4-Cl (**a**), R_2 = 4-F (**b**), R_2 = 4,5-Cl (**c**);
3: R_1 = H, R_2 = 4-F (**a**); R_1 = Cl, R_2 = 4-Cl (**b**); R_1 = Cl, R_2 = 4-F (**c**); R_1 = F, R_2 = 4-F (**d**); R_1 = F, R_2 = 4,5-Cl (**e**); R_1 = Me, R_2 = 4-Cl (**f**); R_1 = Me, R_2 = 4-F (**g**).

Scheme 1

Table 1. Yield of products 3(a-g)

№	Coupling **3**	R_1, R_2	Yield, %
1	**a**	R_1 = H, R_2 = 4-F	33
2	**b**	R_1 = Cl, R_2 = 4-Cl	21
3	**c**	R_1 = Cl, R_2 = 4-F	27
4	**d**	R_1 = F, R_2 = 4-F	49
5	**e**	R_1 = F, R_2 = 4,5-Cl	41
6	**f**	R_1 = Me, R_2 = 4-Cl	28
7	**g**	R_1 = Me, R_2 = 4-F	15

The resulting compounds **3** were purified by recrystallisation from isopropyl alcohol. The structure of the resulting compounds was confirmed by a combination of spectroscopic analytical methods. In the IR spectra of compound **3**, absorption bands were observed for the NH approximately at 3226 cm^{-1}, the C=O – at 1687 cm^{-1}, the C=C – at 1635 cm^{-1}, C–O–CH_3 – at 1230 cm^{-1}, and benzene ring vibrations – at 1601 cm^{-1}. In the mass spectra obtained by electron impact for the three synthesised compounds **3**, a molecular ion was observed (Fig. 1).

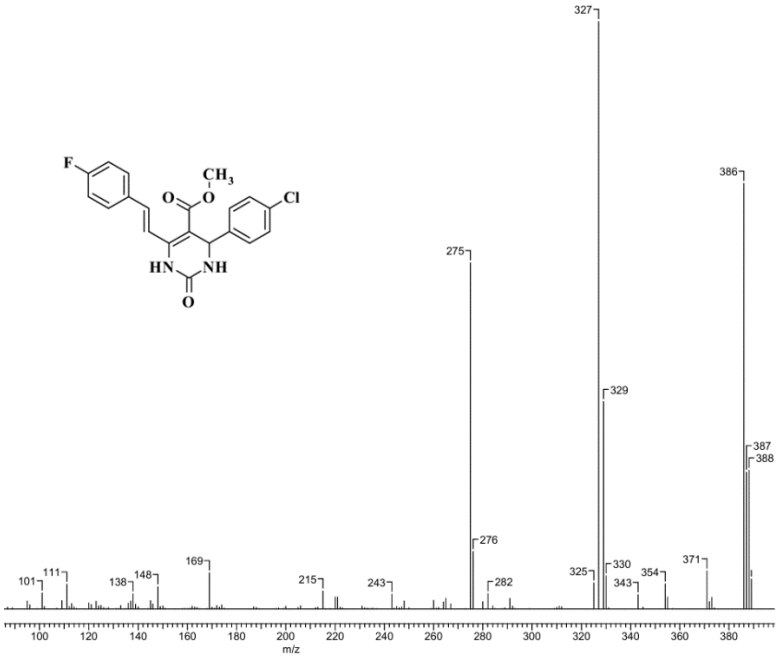


Fig. 1. Mass spectrum fragment of compound **3c**

In the ^{1}H NMR spectra of the synthesised compounds, characteristic doublets of *trans*-protons at the double bond are observed in the range 7.40-7.54 m and 7.84-7.91 m with a chemical shift of 16.7 Hz; a weakly split signal of the 1-NH proton in the region of 9.20–9.30 m and of the OMe group in the region approximately at 3.60 m. The complete assignment of the hydrogen signals of the **3d** product was made on the basis of data from two-dimensional NOESY correlation spectroscopy (Fig. 2).

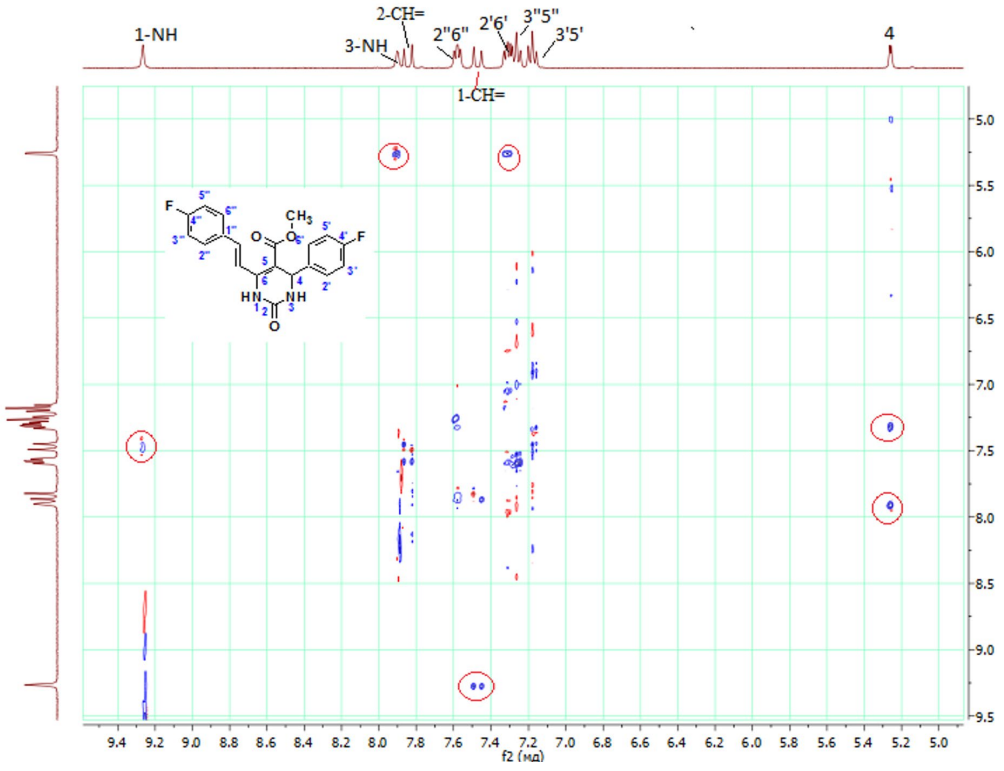


Fig. 2. NOESY spectrum fragment of compound **3d**

The NOESY spectrum shows symmetrical cross-peaks from the hydrogen atom of the 1-NH group, the proton of the double bond closest to the pyrimidine ring, the signals from the H-4 atom, the proton of the 3-NH group, and the *ortho*-protons of one of the aromatic substituents. In addition, weak cross-peaks from the second proton are observed at the double bond with the *ortho*-protons of the second aromatic ring. In the ^{13}C NMR spectra of the synthesised compounds, signals from all carbon atoms are observed.

Conclusions

A preparative method for the synthesis of new substituted (*E*)-styrylpyrimidine-5-carboxylates based on the aldol condensation of 4-aryl-6-methyl-2-oxo-1,2, 3,4-tetrahydropyrimidine-5-carboxylates with aromatic aldehydes, catalysed by ferric chloride has been developed. The structures of all the synthesised compounds have been confirmed by a combination of physicochemical analytical methods.

Experimental part

We recorded IR spectra in reflected light on a Spectrum Two PerkinElmer spectrometer at 700-4000 cm^{-1}. We recorded the ^{1}H and ^{13}C NMR spectra using a Varian Unity Plus 400 MHz instrument (at 400 MHz and 100 MHz respectively) in DMSO-d_6 solutions at 30 °C. The solvent signals were used as internal standards for the ^{1}H NMR (δH = 2.50 ppm) and ^{13}C NMR (δC = 39.50 ppm) spectra. The assignment of the proton signals in the **3d** bond was performed using two-dimensional NMR spectroscopy (^{1}H–^{1}H (NOESY)). We recorded mass spectra on a FINNIGAN MAT.INCOS 50 mass spectrometer at an ionisation voltage of 70 eV and an ionisation chamber temperature of 100-220 °C (IOC RAS, Moscow, Russia). We conducted elemental analysis in the analytical laboratory of INEOS RAS, Moscow, Russia, on a PerkinElmer 2400 unit. We determined the melting temperature using a Büchi M-560 melting and boiling point apparatus. We monitored the progress of the reaction by thin layer chromatography on Silufol 254 UV plates using hexane - ethyl acetate eluent.

The synthesis methods and physicochemical properties of compounds **1(a–d)** are described in [20].

Method for obtaining 3(a–g)

A mixture of dihydropyrimidine **1(a–d)** (1 mmol), a substituted aromatic aldehyde **2(a–d)** (2 mmol), iron(III) chloride hexahydrate (0.2 mmol) and acetonitrile (15 mL) was boiled under reflux for 24 h; after the reaction was complete, the mixture was diluted with water (50 mL), the precipitate was filtered off, washed with water, and dried in air. It was recrystallized in isopropanol.

3a Methyl (*E*)-6-(4-fluorostyryl)-2-oxo-4-phenyl-1,2,3,4-tetrahydropyrimidine-5-carboxylate: yield is 941 mg (33%), $T_{melt.}$ is 242–243 °C. IR spectrum, ν/cm^{-1}: 3234 (N-H), 2948 (νCH_3), 1685 (C=O), 1630 (C=C), 1595, 1582, 1504 (CC in Ph), 1228 (C–F), 1093 (ν C–O–CH_3), 975 (C–H in trans-CH=CH). NMR-spectrum ^{1}H (DMSO-d_6, δ, ppm, *J*/Hz): 3.62 (s, 3H, OMe), 5.26 (d, 1H, *J*=3.5, H-4), 7.21-7.31 (m, 5H, Ph), 7.35 (t, 2H, *J*=8.4, H-3",5"), 7.47 (d, 1H, *J*=16.7, C(a)–H in CH=CH), 7.58 (dd, 2H, *J*=8.4, 5.5, H-2",6"), 7.83 (s, 1H, 3-NH), 7.88 (d, 1H, *J*=16.7,

C(b)–H in CH=CH), 9.23 (s, 1H, 1-NH). Found (%): C, 68.17; H, 4.86; N, 7.95. $C_{20}H_{17}FN_2O_3$. Calculated (%): C, 68.26; H, 4.92; N, 8.02.

3b Methyl ($_E$)-4-(4-chlorophenyl)-6-(4-chlorostyril)-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate: yield is 586 mg (21%), T_{melt}. 236–237 °C. IR spectrum, ν/cm–*1*: 3226 (N-H), 2948 (ν CH^3), 1687 (C=O), 1635 (C=C), 1601 (CC in Ph), 1227, 1097 (ν C–O–CH_3), 976 (C–H in trans-CH=CH). Spectrum NMR ^{1}H (DMSO-d_6, δ, ppm, *J*/Hz): 3.61 (s, 3H, OMe), 5.25 (d, 1H, *J*=3.2, H-4), 7.30 (d, 2H, *J*=8.5, H-3',5'), 7.42 (d, 2H, *J*=8.5, H-2',6'), 7.46 (d, 1H, *J*=16.6, C(a)–H in CH=CH), 7.48 (d, 2H, *J*=8.5, H-3",5"), 7.54 (d, 2H, *J*=8.5, H-2",6"), 7.90 (d, 1H, *J*=16.6, C(b)–H in CH=CH), 7.93 (br.s., 1H, 3-NH), 9.31 (s, 1H, 1-NH). Spectrum NMR ^{13}C (DMSO-d_6, δ, ppm): 51.38, 53.23, 101.48, 120.20, 128.13 (2C), 128.58 (2C), 128.85 (2C), 129.04 (2C), 132.03, 133.55, 133.79, 134.80, 142.99, 144.97, 152.27, 165.47. Found (%): C, 59.57; H, 4.00; N, 6.95. $C_{20}H_{16}Cl_2N_2O_3$. Calculated (%): C, 59.51; H, 3.99; N, 7.03.

3c Methyl (*E*)-4-(4-chlorophenyl)-6-(4-fluorostyril)-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate: yield is 745 mg (27%), T_{melt}. 220–221 °C. IR spectrum, ν/cm^{-1}: 3226 (N-H), 2950 (CH3), 1688 (C=O), 1634 (C=C), 1226 (C–F), 976 (C–H in trans-CH=CH). Spectrum NMR ^{1}H (DMSO-d_6, δ, ppm, *J*/Hz): 3.61 (s, 3H, OMe), 5.26 (d, 1H, *J*=3.5, H-4), 7.22 (t, 2H, *J*=8.3, H-3",5"), 7.31 (dd, 2H, *J*=8.3, 5.4, H-2",6"), 7.47 (d, 1H, *J*=16.8, C(a)–H in CH=CH), 7.50 (d, 2H, *J*=8.5, H-3',5'), 7.58 (d, 2H, *J*=8.5, H-2',6'), 7.85 (d, 1 H, *J*=16.8, C(b)–H in CH=CH), 7.93 (s, 1H, 3-NH), 9.30 (br.s., 1H, 1-NH). Spectrum NMR ^{13}C (DMSO-d_6, δ, ppm): 51.37, 53.22, 101.15, 116.0 (d, 2C, J_{CF}=21.5), 119.35 (d, J_{CF}=2.9), 128.14 (2C), 128.59 (2C), 129.30 (d, 2C, J_{CF}=8.6), 132.03, 132.48 (d, J_{CF}=3.5), 134.00, 143.05, 145.16, 152.33, 162.52 (d, J_{CF}=247.2), 165.52. Found (%): C, 62.10; H, 4.17; N, 7.24. $C_{20}H_{16}ClFN_2O_3$. Calculated (%): C, 62.01; H, 4.19; N, 7.28.

3d Methyl (*E*)-4-(4-fluorophenyl)-6-(4-fluorostyryl)-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate: yield is 1.362 g (49%), T_{melt}. 223–224 °C. IR spectrum, ν/cm^{-1}: 3240 (N-H), 2953 (ν CH_3), 1680 (C=O), 1631 (C=C), 1606 (CC in Ph), 1224 (C–F), 1095 (ν C–O–CH_3), 972 (C–H in trans-CH=CH). Spectrum NMR ^{1}H (DMSO-d_6, δ, ppm, *J*/Hz): 3.62 (s, 3H, OMe), 5.26 (d, 1H, *J*=3.5, H-4), 7.18 (t, 2H, *J*=8.8, H-3',5'), 7.27 (t, 2H, *J*=8.8, H-3",5"),7.31 (dd, 2H, *J*=8.5, 5.5, H-2',6'), 7.47 (d, 1H, *J*=16.6, C(a)–H in CH=CH), 7.58 (dd, 2H, *J*=8.5, 5.5, H-2",6"), 7.84 (d, 1H, *J*=16.6, C(b)–H in CH=CH), 7.90 (br.s., 1H, 3-NH), 9.26 (br.s., 1H, 1-NH). Spectrum NMR ^{13}C (DMSO-d_6, δ, ppm): 51.33, 53.13, 101.47, 115.30 (d, 2C, J_{CF}=21.5), 115.97 (d, 2C, J_{CF}=21.5), 119.39 (d, J_{CF}=3.1), 128.19 (d, 2C, J_{CF}=8.6), 129.26 (d, 2C, J_{CF}=8.6), 132.48 (d, J_{CF}=3.1), 133.88, 140.36, 144.97, 152.34, 161.41 (d, $J_{CF.}$=246.5), 162.21 (d, J_{CF}=246.5), 165.55. Found (%): C, 64.86; H, 4.35; N, 7.56. $C_{20}H_{16}F_2N_2O_3$. Calculated (%): C, 64.79; H, 4.42; N, 7.62.

3e Methyl (*E*)-6-(3,4-dichlorostyryl)-4-(4-fluorophenyl)-2-hydroxy-1,2,3,4-tetrahydropyrimidine-5-carboxylate: yield is 1.306 g (41%), T_{melt}. 248–249 °C. IR spectrum, ν/cm^{-1}: 3238 (N-H), 2949 (νCH_3), 1669 (C=O), 1626 (C=C), 1604 (CC in Ph), 1218 (C–F), 1091 (ν C–O–CH_3), 975 (C–H in trans-CH=CH). Spectrum NMR ^{1}H (DMSO-d_6, δ, ppm, *J*/Hz): 3.61 (s, 3H, OMe), 5.26 (d, 1H, *J*=3.5, H-4), 7.18 (t, 2H, *J*=8.8, H-3',5'), 7.31 (dd, 2H, *J*=8.8, 5.0, H-2',6'), 7.40 (d, 1H, *J*=16.7, C(a)–H in CH=CH), 7.52 (dd, 1H, *J*=8.4, 2.0, H-6"), 7.69 (d, 1H, *J*=8.4, H-5"), 7.73 (d, 1H, *J*=2.0, H-2"), 7.89 (d, 1H, *J*=16.7, C(b)–H in CH=CH), 7.92 (br.s., 1H, 3-NH), 9.28 (br.s., 1H, 1-NH). Spectrum NMR ^{13}C (DMSO-d_6, δ, ppm): 51.43, 53.17, 102.23, 115.34 (d, 2C, J_{CF}=21.7), 121.88, 127.06, 128.22 (d, 2C, J_{CF}=8.3), 128.76, 131.17, 131.22, 131.70,

132.45, 136.79, 140.23 (d, J_{CF}=3.3), 144.52, 152.25, 161.44 (d, J_{CF}=243.8),165.45. Found (%): C, 57.03; H, 3.59; N, 6.65 $C_{20}H_{15}Cl_2FN_2O_3$. Calculated (%): C, 57.09; H, 3.52; N, 6.62.

3f Methyl (*E*)-6-(4-chlorostyril)-2-oxo-4-(*p*-tolyl)-1,2,3,4-tetrahydropyrimidine-5-carboxylate: yield is 811 mg (28%), Tmelt. 209–210 °C. IR spectrum, ν/cm^{-1}: 3221 (N-H), 2951 (ν CH_3), 1692 (C=O), 1631 (C=C), 1094 (ν C–O–CH_3), 974 (C–H in trans-CH=CH). Spectrum NMR ^{1}H (DMSO-d_6, δ, ppm, *J*/Hz): 2.24 (s, 3H, Me), 3.60 (s, 3H, OMe), 5.23 (d, 1H, *J*=3.4, H-4), 7.11 (d, 2H, *J*=8.5, H-3',5'), 7.16 (d, 2H, *J*=8.5, H-2',6'), 7.48 (d, 2H, *J*=8.2, H-3",5"), 7.54 (d, 1H, *J*=16.7, C(a)–H in CH=CH), 7.59 (d, 2H, *J*=8.2, H-2",6"), 7.70 (s, 1H, 3-NH), 7.91 (d, 1H, *J*=16.7, C(b)–H in CH=CH), 9.22 (s, 1H, 1-NH). Found (%): C, 65.88; H, 5.00; N, 7.32. $C_{21}H_{19}ClN_2O_3$. Calculated (%): C, 65.91; H, 4.99; N, 7.28.

3g Methyl (*E*)-6-(4-fluorostyryl)-2-oxo-4-(*p*-tolyl)-1,2,3,4-tetrahydropyrimidine-5-carboxylate: yield is 421 mg (15%), Tmelt. 220–221 °C. IR spectrum, ν/cm^{-1}: 3229 (N-H), 2951 (ν CH_3), 1687 (C=O), 1634 (C=C), 1598, 1586, 1507 (CC in Ph), 1227 (C–F), 1099 (ν C–O–CH_3), 974 (C–H in trans-CH=CH). Spectrum NMR ^{1}H (DMSO-d_6, δ, ppm, *J*/Hz): 2.26 (s, 3H, Me), 3.60 (s, 3H, OMe), 5.22 (d, 1H, *J*=3.4, H-4), 7.14 (d, 2H, *J*=8.4, H-3',5'), 7.17 (d, 2H, *J*=8.4, H-2',6'), 7.26 (t, 2H, *J*=8.6, H-3",5"), 7.46 (d, 1H, *J*=16.6, C(a)–H in CH=CH), 7.57 (dd, 2H, *J*=8.6, 5.6, H-2",6"), 7.84 (s, 1H, 3-NH), 7.85 (d, 1H, *J*=16.6, C(b)–H in CH=CH), 9.21 (s, 1H, 1-NH). Spectrum NMR ^{13}C (DMSO-d_6, δ, ppm): 20.63, 51.26, 53.48, 101.81, 115.85, 116.06, 126.09 (2C), 129.06 (2C), 129.17, 129.26, 132.52, 132.55, 133.63, 136.63, 141.17, 144.67, 152.49, 165.64. Mass-spectrum (EI, 70 eV), m/z (I_{rel}, %): 388 $[M]^+$(23), 386 $[M]^+$(72), 329 (35), 327 (100), 275 (59), 59(10). Found (%): C, 68.84; H, 5.23; N, 7.65. $C_{21}H_{19}FN_2O_3$. Calculated (%): C, 68.41; H, 5.29; N, 7.63.

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