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Regioselective Cycloaddition of *C*,*N*-Diarylnitrones to Arylallenes and of *N*-Aryl-*C*-carbamoylnitrones to Methyl Buta-2,3-dienoate

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Abstract—Cycloadditions of *C*,*N*-diarylnitrones to non-activated arylallenes and of *N*-aryl-*C*-carbamoylnitrones to methyl buta-2,3-dienoate regioselectively afforded mixtures of diastereoisomeric substituted 4-methylideneisoxazolidines.

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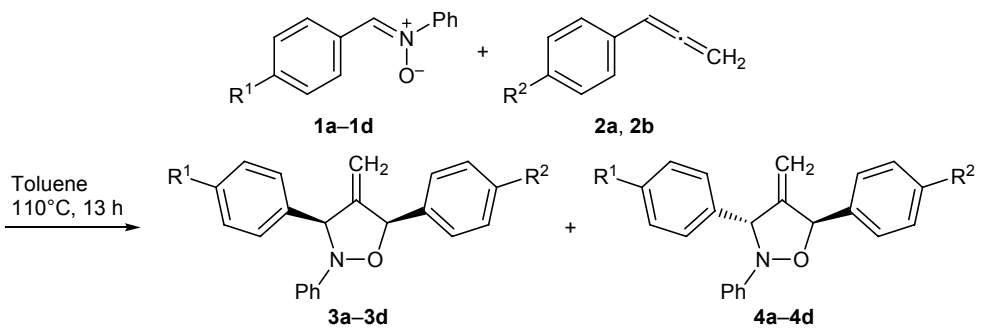
1,3-Dipolar cycloaddition reactions underlie one of the most important methods of synthesis of heterocyclic systems from simple starting compounds. Nitrones are among the most popular 1,3-dipoles due to their accessibility, biological activity, and versatile reactivity of cycloadducts derived therefrom [1–4]. Reactions of nitrones with alkenes and alkynes, leading to the formation of isoxazolidines and dihydroisoxazoles with high stereoselectivity, have been well documented, whereas only a few examples of their reactions with cumulenes have been reported [5, 6]. The direction of 1,3-dipolar cycloaddition of nitrones to allenes is determined by the nature of substituents in both reactants. It is known that allenes containing electron-withdrawing substituents react with arylnitrones in regioselective fashion to give 5-methylideneisoxazolidines [7–14]. The reactions of diphenylnitrone with 3-methylbuta-1,2-diene and 2-ethenylideneadamantane afforded mixtures of 4- and 5-methylideneisoxazolidines [15, 16]. Reactions of nitrones with allenes substituted by electron-donating groups have been poorly studied; however, the reaction of diphenylnitrone with methoxyallene was reported to produce 4-methylideneisoxazolidine [16]. We previously showed that *C*-carbamoylnitrones react with arylallenes to form 4-methylideneisoxazolidines with high regio- and stereoselectivity and that adducts derived from phenylallene and *N*-aryl-*C*,*C*-bis(methoxycarbonyl)nitrones are thermally unstable (they undergo rearrangement into substituted benzazepinones and pyrrolidinones) [17].

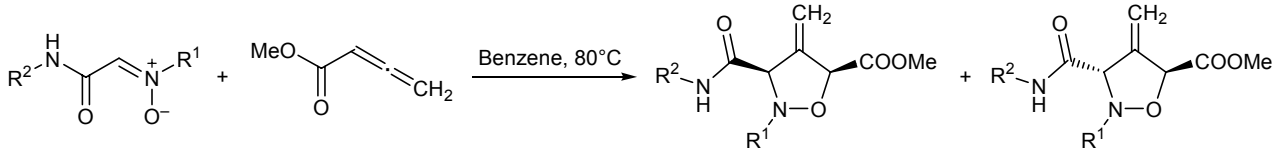
In this work we have found that *C*,*N*-diarylnitrones **1a**–**1d** react with arylallenes **2a** and **2b** on heating in toluene for 6–13 h to afford exclusively 4-methylideneisoxazolidines as mixtures of two diastereoisomers **3a**–**3d** and **4a**–**4d** at a ratio of 2:1 (according to the ^{1}H NMR data; Scheme 1). We failed to isolate diastereoisomers **3** and **4** in the pure state.

Scheme 1.



1, **3**, **4**, $R^1 = R^2 = H$ (**a**); $R^1 = MeO$, $R^2 = H$ (**b**); $R^1 = Br$, $R^2 = H$ (**c**); $R^1 = R^2 = Me$ (**d**); **2**, $R^2 = H$ (**a**), Me (**b**).

Scheme 2.



5a–5d **6** **7a–7d** **8a–8d**

$R^1 = R^2 = Ph$ (**a**); $R^1 = Ph$, $R^2 = 4\text{-}ClC_6H_4$ (**b**); $R^1 = 4\text{-}MeC_6H_4$, $R^2 = Ph$ (**c**); $R^1 = 4\text{-}MeC_6H_4$, $R^2 = 4\text{-}MeOC_6H_4$ (**d**).

The structure of **3a**–**3d** and **4a**–**4d** was determined on the basis of their spectral data. The ^{1}H NMR spectra of the major isomers contained signals from the $=CH_2$ protons at δ 4.85–5.05 ppm and from protons in position *3* and *5* of the isoxazolidine ring as broadened singlets in the regions δ 5.3–5.4 and 5.6–5.8 ppm, respectively. The minor isomers displayed two triplets at 4.75–4.85 ppm (J = 2.2–2.3 Hz) due to $=CH_2$ protons and signals at δ 4.85–4.95 (3-H) and 5.70–5.80 ppm (5-H, d, J = 1.8–2.0 Hz). Comparison of the chemical shifts and coupling constants with the data reported by us previously [18, 19] allowed us to presume *cis* arrangement of the aryl substituents on C^3 and C^5 in the major isomer and their *trans* orientation in the minor isomer. In the ^{13}C NMR spectra of **3a**–**3d** and **4a**–**4d**, the exocyclic methylene carbon atom resonated at δ_C 108–110 ppm, and signals from C^3 and C^5 were observed at δ_C 74–75 and 82–83 ppm.

In order to elucidate the regioselectivity of the cycloaddition of carbamoylnitrones to allenes containing electron-withdrawing groups, we examined the reaction of *C*-carbamoylnitrones **5a**–**5d** with methyl buta-2,3-dienoate (**6**). Compounds **5** and **6** failed to react with each other at room temperature; therefore, their mixtures were heated in benzene at 80°C for 7 h. In all cases, the products were equimolar mixtures of stereoisomeric 4-methylideneisoxazolidines **7a**–**7d** and **8a**–**8d**. We succeeded in separating isomers **7d** and **8d** by column chromatography, while the other products were characterized as stereoisomer mixtures.

The structure of **7a**–**7d** and **8a**–**8d** was determined by analysis of their NMR spectra. Compounds **7a**–**7d** showed in the ^{1}H NMR spectra two signals from the methylene protons at δ 4.68–4.70 and 5.15–5.25 ppm, signals from 3-H and 5-H were located at δ 5.50–5.60 and 5.70–5.75 ppm, respectively, and the NH proton gave a singlet at δ 8.85–9.05 ppm. The corresponding protons of isomers **8a**–**8d** resonated at δ 4.70–4.75, 5.15–5.25 ($=CH_2$), 5.5–5.6 (3-H), 5.8–5.85 (5-H), and 8.65–8.85 ppm (NH). The ^{13}C NMR spectra of **7a**–**7d** and **8a**–**8d** contained signals at δ_C 111–112 ($=CH_2$) and 71–73, 77–79 ppm (C^3, C^5). The elemental compositions of the products were confirmed by high-resolution ESI mass spectra. In the IR spectra of **7a**–**7d** and **8a**–**8d** we observed ester carbonyl absorption band at ~1750 cm^{-1} and N–H stretching vibration band in the region 3354–3336 cm^{-1}.

Thus, we have found that the cycloaddition of *C*,*N*-diarylnitrones to non-activated arylallenes and of *N*-aryl-*C*-carbamoylnitrones to methyl buta-2,3-dienoate regioselectively occurs at the substituted double bond of the allene with formation of 4-methylideneisoxazolidines as mixtures of two diastereoisomers.

EXPERIMENTAL

The mass spectra were recorded on a Bruker microTOF mass spectrometer (positive electrospray ionization). The IR spectra were measured on a Bruker Tensor 27 spectrometer from 2% solutions in carbon tetrachloride. The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker DPX-300 instrument at 300.13 and 75.47 MHz, respectively, using $CDCl_3$ as solvent. The progress of reactions and the purity of products were monitored by TLC on Silufol UV-254 plates.

The initial nitrones [18, 20] and arylallenes [21] were synthesized according to known procedures.

Reaction of diarylnitrones with arylallenes (*general procedure*). A mixture of 2 mmol of nitrone **1a**–**1d** and 2.6 mmol of allene **2a** or **2b** in toluene was heated for 6–13 h under reflux. The solvent was evaporated under reduced pressure, and the products were isolated by silica gel column chromatography using petroleum ether–ethyl acetate as eluent. The products were characterized as mixtures of diastereoisomers.

4-Methylidene-2,3,5-triphenylisoxazolidine (3a/4a). Yield 244 mg (39%), oily substance, R_f 0.80 (EtOAc–hexane, 1:5). IR spectrum, ν, cm^{-1}: 3067, 3033, 2927, 2857, 1599, 1490, 1454, 1283, 1257, 1078, 1030. ^{1}H NMR spectrum, δ, ppm: major isomer: 4.86 t and 5.03 t (1H each, $=CH_2$, J = 2.0 Hz), 5.37 br.s (1H, CH), 5.65 br.s (1H, CH), 6.99–7.65 (15H, H_{arom}); minor isomer: 4.77 t (1H, CH_2, J = 2.2 Hz), 4.82 t (1H,

CH_2, J = 2.3 Hz), 4.92–4.96 m (1H, CH), 5.75–5.79 m (1H, CH), 6.99–7.65 (15H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm (isomer mixture): 74.0 (CH), 74.8 (CH), 82.3 (CH), 82.7 (CH), 108.4 (CH_2), 109.0 (CH_2), 114.9 (CH), 117.5 (CH), 122.0 (CH), 123.1 (CH), 127.2 (CH), 127.6 (CH), 127.9 (CH), 128.0 (CH), 128.2 (CH), 128.5 (2CH), 128.6 (CH), 128.7 (CH), 128.8 (CH), 128.9 (CH), 129.0 (CH), 137.8 (C), 137.9 (C), 140.7 (C), 141.0 (C), 149.6 (C), 151.1 (C), 155.9 (C), 157.6 (C). Mass spectrum: m/z 314.1524 $[M + H]^+$. $C_{22}H_{19}NO$. Calculated: M + H 314.1545.

3-(4-Methoxyphenyl)-4-methylidene-2,5-diphenylisoxazolidine (3b/4b). Yield 400 mg (58%), oily substance, R_f 0.71 (EtOAc–hexane, 1:3). IR spectrum, ν, cm^{-1}: 3089, 3034, 2955, 2909, 2836, 1612, 1512, 1304, 1249, 1173, 1040. ^1H NMR spectrum, δ, ppm: major isomer: 3.85 s (3H, OCH_3), 4.84–4.88 m and 4.98–5.01 m (1H each, $=CH_2$), 5.31 br.s (1H, CH), 5.65 br.s (1H, CH), 6.95–7.55 m (14H, H_{arom}); minor isomer: 3.86 s (3H, OCH_3), 4.76 t and 4.80 t (1H each, $=CH_2$, J = 2.3 Hz), 4.85–4.88 m (1H, CH), 5.72–5.76 m (1H, CH), 6.95–7.55 (14H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm (isomer mixture): 55.3 ($2CH_3$), 73.6 (CH), 74.6 (CH), 82.2 (CH), 82.7 (CH), 108.2 (CH_2), 108.8 (CH_2), 114.1 (CH), 114.3 (CH), 115.0 (CH), 116.2 (CH), 117.7 (CH), 122.0 (CH), 123.2 (CH), 128.1 (CH), 128.4 (CH), 128.5 (2CH), 128.6 (CH), 128.7 (CH), 128.8 (CH), 128.9 (CH), 129.2 (CH), 132.5 (C), 133.0 (C), 138.1 (C), 144.7 (C), 149.7 (C), 151.1 (C), 156.1 (C), 157.9 (C), 159.1 (C), 159.4 (C). Mass spectrum: m/z 366.1462 $[M + Na]^+$. $C_{23}H_{21}NO_2$. Calculated: M + Na 366.1470.

3-(4-Bromophenyl)-4-methylidene-2,5-diphenylisoxazolidine (3c/4c). Yield 537 mg (68%), oily substance, R_f 0.53 (EtOAc–hexane, 1:3). IR spectrum, ν, cm^{-1}: 3068, 3034, 2868, 1714, 1671, 1598, 1489, 1454, 1407, 1294, 1250, 1073. ^1H NMR spectrum, δ, ppm: major isomer: 4.86–4.90 m (1H, $=CH_2$), 5.03 t (1H, $=CH_2$, J = 1.7 Hz), 5.32 br.s (1H, CH), 5.64 br.s (1H, CH), 7.01–7.60 (14H, H_{arom}); minor isomer: 4.79 t and 4.80 t (1H each, $=CH_2$, J = 2.3 Hz), 4.86–4.90 m (1H, CH), 5.71–5.75 m (1H, CH), 7.01–7.60 (14H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm (isomer mixture): 73.3 (CH), 74.1 (CH), 82.3 (CH), 82.7 (CH), 108.7 (CH_2), 109.3 (CH_2), 114.9 (CH), 116.0 (CH), 117.6 (CH), 121.6 (C), 122.0 (C), 122.2 (CH), 122.9 (CH), 123.3 (CH), 123.4 (CH), 127.5 (CH), 128.0 (CH), 128.1 (CH), 128.4 (CH), 128.5 (2CH), 128.6 (2CH), 128.7 (CH), 128.9 (2CH), 129.1 (CH), 129.3 (CH), 129.6 (CH), 131.9 (CH), 132.1 (CH), 136.1 (C), 137.6 (C), 139.6 (C), 140.0 (C), 149.3 (C), 150.7 (C), 155.4 (C), 157.2 (C). Mass spectrum: m/z 414.0456 $[M + Na]^+$. $C_{22}H_{18}BrNO$. Calculated: M + Na 414.0469.

4-Methylidene-3,5-bis(4-methylphenyl)-2-phenylisoxazolidine (3d/4d). Yield 300 mg (60%), oily substance, R_f 0.41 (EtOAc–hexane, 1:20). IR spectrum, ν, cm^{-1}: 3027, 2924, 2864, 1599, 1514, 1490, 1453, 1308, 1180, 1033. ^1H NMR spectrum, δ, ppm: major isomer: 2.41 s (6H, CH_3), 4.83 t (1H, $=CH_2$, J = 2.0 Hz), 4.97–5.01 m (1H, $=CH_2$), 5.31–5.35 m (1H, CH), 5.59–5.63 m (1H, CH), 6.98–7.52 m (13H, H_{arom}); minor isomer: 2.40 s (6H, CH_3), 4.75 t and 4.80 t (1H each, $=CH_2$, J = 2.3 Hz), 4.87–4.91 m (1H, CH), 5.71–5.75 m (1H, CH), 6.98–7.52 m (13H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm (isomer mixture): 21.1 ($2CH_3$), 21.2 ($2CH_3$), 73.9 (CH), 74.7 (CH), 82.3 (CH), 82.6 (CH), 108.0 (CH_2), 108.7 (CH_2), 114.8 (CH), 116.0 (CH), 117.5 (CH),121.8 (CH), 122.9 (CH), 127.1 (CH), 127.8 (CH), 128.3 (CH), 128.4 (CH), 128.5 (CH), 128.9 (CH), 129.2 (2CH), 129.3 (CH), 129.4 (CH), 129.6 (CH), 134.8 (C), 137.2 (C), 138.1 (C), 138.3 (C), 149.8 (C), 151.2 (C), 156.1 (C), 158.0 (C). Mass spectrum: m/z 364.1655 $[M + Na]^+$. $C_{24}H_{23}NO$. Calculated: M + Na 364.1677.

Reaction of carbamoylnitrones 5a–5d with methyl buta-2,3-dienoate (6) (*general procedure*). A mixture of 1 mmol of nitrone **5a**–**5d** and 2 mol of allene **6** in benzene was heated for 7 h under reflux. The solvent was removed under reduced pressure, and the residue was subjected to silica gel column chromatography using petroleum ether–ethyl acetate as eluent. We succeeded in isolating pure disatereoisomers **7d** and **8d**. In the other cases, the products were characterized as diastereoisomer mixtures.

Methyl 4-methylidene-2-phenyl-3-(phenylcarbamoyl)isoxazolidine-5-carboxylate (7a/8a). Yield 180 mg (54%), oily substance, R_f 0.45 (EtOAc–petroleum ether, 1:3). IR spectrum, ν cm^{-1}: 3347, 3064, 3033, 2955, 1749, 1699, 1602, 1525, 1493, 1444, 1313, 1278, 1200, 1176, 1090, 1020. ^1H NMR spectrum, δ, ppm: 3.72 s (3H, CH_3), 3.83 s (3H, CH_3), 4.71 br.s (1H, CH_2), 4.80 br.s (1H), 5.21 br.s (1H, CH_2), 5.22 br.s (1H, CH_2), 5.54 br.s (1H, CH), 5.55 br.s (1H, CH), 5.77 br.s (1H, CH), 7.09–7.22 m (8H, H_{arom}), 7.30–7.43 (8H, H_{arom}), 7.54–7.69 m (4H, H_{arom}), 8.80 s (1H, NH), 9.00 s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 52.6 (CH_3), 52.9 (CH_3), 70.8 (CH), 71.9 (CH), 77.2 (CH), 78.6 (CH), 111.5 (CH_2), 111.6 (CH_2), 115.4 (CH), 115.9 (CH), 119.8 (CH), 119.9 (CH), 123.7 (CH), 123.8 (CH), 124.5 (CH), 124.8 (CH), 128.9 (CH), 129.0 (CH),129.1 (CH), 129.4 (CH), 137.0 (C),

137.5 (C), 143.2 (C), 143.8 (C), 148.4 (C), 148.6 (C), 166.1 (CO), 166.3 (CO), 168.4 (CO), 168.7 (CO). Mass spectrum: *m*/*z* 361.1153 $[M + Na]^+$. $C_{19}H_{18}N_2O_4$. Calculated: *M* + Na 361.1164.

Methyl 3-(4-chlorophenylcarbamoyl)-4-methylidene-2-phenylisoxazolidine-5-carboxylate (7b/8b). Yield 225 mg (61%), oily substance, R_f 0.53 (EtOAc–petroleum ether, 1:3). IR spectrum, ν, cm^{-1}: 3336, 3004, 2955, 1749, 1701, 1597, 1520, 1493, 1455, 1401, 1306, 1283, 1239, 1203, 1175, 1093, 1014. ^1H NMR spectrum, δ, ppm: 3.71 s (3H, CH_3), 3.82 s (3H, CH_3), 4.72 br.s (1H, CH_2), 4.80 br.s (1H, CH_2), 5.20 br.s (1H, CH_2), 5.22 br.s (1H, CH_2), 5.54 br.s (1H, CH), 5.56 br.s (1H, CH), 5.75 br.s (1H), 5.83 br.s (1H), 7.10–7.15 (4H, H_{arom}), 7.15–7.20 (2H, H_{arom}), 7.30–7.39 (8H, H_{arom}), 7.52–7.62 (4H, H_{arom}), 8.82 s (1H, NH), 9.02 s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 52.6 (CH_3), 53.0 (CH_3), 70.4 (CH), 71.9 (CH), 77.2 (CH), 78.6 (CH), 111.7 (CH_2), 111.8 (CH_2), 115.3 (CH), 115.8 (CH), 121.0 (CH), 121.1 (CH), 123.8 (CH), 123.9 (CH), 129.0 (CH), 129.0 (CH), 129.0 (CH), 129.3 (CH), 129.5 (CH), 129.8 (CH), 135.6 (C), 136.0 (C), 143.0 (C), 143.6 (C), 148.3 (C), 148.5 (C), 166.2 (CO), 166.4 (CO), 168.4 (CO), 168.7 (CO). Mass spectrum: *m*/*z* 395.0796 $[M + Na]^+$. $C_{19}H_{17}ClN_2O_4$. Calculated: *M* + Na 395.0775.

Methyl 4-methylidene-2-(4-methylphenyl)-3-(phenylcarbamoyl)isoxazolidine-5-carboxylate (7c/8c). Yield 215 mg (62%), oily substance, R_f 0.29 (EtOAc–petroleum ether, 1:3). IR spectrum, ν, cm^{-1}: 3343, 3033, 2955, 1748, 1699, 1602, 1526, 1444, 1314, 1277, 1200, 1176, 1089. ^1H NMR spectrum, δ, ppm: 2.35 s (3H, CH_3), 2.36 s (3H, CH_3), 3.73 s (3H, CH_3), 3.81 s (3H, CH_3), 4.69 br.s (1H, CH_2), 4.75 br.s (1H, CH_2), 5.19 br.s (1H, CH_2), 5.20 br.s (1H, CH_2), 5.52 br.s (1H, CH), 5.53 br.s (1H, CH), 5.75 br.s (1H, CH), 5.81 br.s (1H, CH), 7.00–7.21 (6H, H_{arom}), 7.30–7.43 (10H, H_{arom}), 7.56–7.65 (2H, H_{arom}), 8.83 s (1H, NH), 9.01 s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 20.6 ($2CH_3$), 52.6 (CH_3), 52.9 (CH_3), 70.6 (CH), 72.0 (CH), 77.2 (CH), 78.5 (CH), 111.4 (CH_2), 111.6 (CH_2), 115.7 (CH), 116.5 (CH), 119.8 (CH), 119.9 (CH), 124.5 (CH), 124.7 (CH), 128.9 (CH), 129.0 (CH), 129.5 (CH), 129.8 (CH), 133.6 (2C), 137.1 (C), 137.5 (C), 143.5 (C), 143.0 (C), 146.0 (C), 146.1 (C), 166.1 (CO), 166.4 (CO), 168.4 (CO), 168.7 (CO). Mass spectrum: *m*/*z* 391.1073 $[M + Na]^+$. $C_{20}H_{20}N_2O_4$. Calculated: *M* + Na 391.1060.

Methyl 3-(4-methoxyphenylcarbamoyl)-4-methylidene-2-(4-methylphenyl)isoxazolidine-5-carboxylate (7d/8d). Yield 227 mg (60%), oily substance, R_f 0.38 (EtOAc–petroleum ether, 1:3). IR spectrum, ν, cm^{-1}: 3354, 2955, 2931, 2836, 1748, 1696, 1653, 1597, 1512, 1465, 1439, 1414, 1247, 1199, 1175, 1042, 1020.

Isomer **7d**. ^1H NMR spectrum, δ, ppm: 2.32 s (3H, CH_3), 3.70 s (3H, CH_3), 3.71 s (3H, CH_3), 4.69 br.s (1H, CH_2), 5.20 br.s (1H, CH_2), 5.51 br.s (1H, CH), 5.75 br.s (1H, CH), 6.89 d (2H, H_{arom}, *J* = 9.2 Hz), 7.04 d (2H, H_{arom}, *J* = 8.5 Hz), 7.12–7.21 (2H, H_{arom}), 7.52 d (2H, H_{arom}, *J* = 9.0 Hz), 8.91 s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 20.6 (CH_3), 52.9 (CH_3), 55.4 (CH_3), 71.9 (CH), 77.2 (CH), 111.5 (CH_2), 114.1 (2CH), 115.7 (CH), 121.4 (2CH), 129.8 (2CH), 130.6 (CH), 133.6 (2C), 144.0 (C), 146.0 (C), 156.5 (C), 166.1 (CO), 168.7 (CO).

Isomer **8d**. ^1H NMR spectrum, δ, ppm: 2.34 s (3H, CH_3), 3.73 s (3H, CH_3), 3.80 s (3H, CH_3), 4.74 t and 5.19 t (1H each, $=CH_2$, *J* = 1.8 Hz), 5.51 br.s (1H, CH), 5.81 br.s (1H, CH), 6.89 d (2H, H_{arom}, *J* = 9.0 Hz), 7.08–7.19 (4H, H_{arom}), 7.49 d (2H, H_{arom}, *J* = 9.0 Hz), 8.70 s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 20.6 (CH_3), 52.6 (CH_3), 55.4 (CH_3), 70.5 (CH), 78.4 (CH), 111.2 (CH_2), 114.2 (CH), 116.4 (CH), 121.5 (2CH), 129.5 (2CH), 130.2 (2CH), 133.6 (2C), 143.5 (C), 146.1 (C), 156.7 (C), 165.9 (CO), 168.5 (CO). Mass spectrum: *m*/*z* 405.1433 $[M + Na]^+$. $C_{21}H_{22}N_2O_5$. Calculated: *M* + Na 405.1426.

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