2-[(1,3-dihydro-2H-isoindol-2yl)methyl]melatonin – a novel MT2-selective melatonin receptor antagonist

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Experimental procedures and analytical data for compounds 4, 5a-b, 6a-e, 8, and 9

General Remarks. Melting points were determined using a capillary melting point apparatus (Gallenkamp, Sanyo) and are uncorrected. Column chromatography was carried out on silica gel 60 (0.063–0.200 mm) obtained from Merck. Bruker AV-400 spectrometers were used to obtain 1H NMR and 13C NMR spectra, respectively. Proton chemical shifts are referred to CHCl3 (7.24 ppm) and DMSO-d6 (2.55 ppm). Carbon chemical shifts are referred to CDCl3 (77.00 ppm) DMSO-d6 (39.50 ppm). The NMR resonances were assigned by means of HH-COSY, HMQC, and HMBC experiments. EI mass spectra were determined on a Finnigan MAT 90, and on a ESI-microTOF spectrometers. IR spectra, recorded as ATR, were obtained by using a Biorad PharmalyzIR FT-IR instrument. Elemental analyses were performed by the microanalytical section of the Institute of Inorganic Chemistry, University of Würzburg. All reactions were carried out under an argon atmosphere.
[2-(1,3-Dihydro-2H-isooindol-2-ylicarbonyl)-5-methoxy-1H-indol-3-yl]acetonitrile (8):

A solution of isoindoline\(^1\) (0.52 g, 4.34 mmol) in dry CH\(_2\)Cl\(_2\) (5 ml) was added to a stirred solution of 3-(cynomethyl)-5-methoxyindole-2-carboxylic acid 7\(^2\) (1.00 g, 4.34 mmol) and EDCI.HCl (1.30 g, 6.79 mmol) in dry CH\(_2\)Cl\(_2\) (10 ml) under inert atmosphere. The reaction mixture was stirred for 18 h at room temperature, extracted with 5M hydrochloric acid (3 x 5 ml), washed with water (2 x 10 ml) and dried (Na\(_2\)SO\(_4\)). The organic layer was evaporated in vacuo and the residue was subjected to silica gel chromatography (ethyl acetate/hexane, 1:1) to give a light brown solid (Found: C, 72.21; H, 5.17; N, 12.33. C\(_{20}\)H\(_{17}\)N\(_3\)O\(_2\) requires C, 72.49; H, 5.17; N, 12.68 %); mp 226 °C; \(\nu_{max}\)/cm\(^{-1}\): 3421, 3047, 2220, 1605, 1531, 750; \(\delta_H\) (400 MHz, DMSO-\(d_6\)) 3.86 (3H, s, OCH\(_3\)), 4.19 (2H, s, CH\(_2\)-CN), 4.99 (4H, s, H-1’, H-3’), 6.99 (1H, dd, J 8.8, 2.5, H-6), 7.27 (1H, d, J 2.5, H-4), 7.36-7.42 (4H, m, H-4’, H-5’, H-6’, H-7’), 7.44 (1H, d, J 8.8, H-7), 11.62 (1H, br., NH); \(\delta_C\) (100 MHz, DMSO-\(d_6\)) 12.8 (CH\(_2\)-CN), 52.0, 53.4 (C-1’, C-3’), 55.4 (OCH\(_3\)), 100.1 (C-4), 105.8 (CN), 113.2 (C-7), 114.5 (C-6), 118.9 (C-3), 122.8, 127.5 (CH\(_2\)ar), 126.5, 130.0, 130.7, 138.9, 139.3 (C\(_{ar}\)), 154.2 (C-5), 162.4 (O=C); m/z (EI): 331 (100%, M\(^+\)), 314 (15), 212 (40), 118 (81).

[2-(1,2,3,4-Tetrahydroisoquinolin-2-ylicarbonyl)-5-methoxy-1H-indol-3-yl]acetonitrile (9):

Tetrahydroisoquinoline (0.08 ml, 0.65 mmol) was added to a stirred solution of compound 7\(^2\) (150 mg, 0.65 mmol) and EDCI.HCl (190 mg, 0.98 mmol) in dry CH\(_2\)Cl\(_2\) (10 ml) under inert atmosphere. The reaction mixture was stirred for 18 h at room temperature, extracted with 5M hydrochloric acid (3 x 5 ml), washed with water (2 x 10 ml) and dried (Na\(_2\)SO\(_4\)). The organic layer was evaporated in vacuo and the residue was subjected to silica gel chromatography (ethyl acetate/hexane, 1:1) to give 140 mg (62 %) of 9 as an orange yellow solid (Found: C, 72.71; H, 5.50; N, 12.44. C\(_{21}\)H\(_{19}\)N\(_3\)O\(_2\) requires C, 73.03; H, 5.54; N, 12.17 %); mp 167-169 °C; \(\nu_{max}\)/cm\(^{-1}\): 3263, 2952, 2916, 2835, 1600, 1584, 1545, 1441, 1246, 1218; \(\delta_H\) (400 MHz, CDCl\(_3\)) 2.97 (2H, t, J 5.9, H-4’), 3.87 (2H, s, CH\(_2\)-CN), 3.88 (3H, s, OCH\(_3\)), 3.89 (2H, t, J 5.9, H-3’), 4.80 (2H, s, H-1’), 6.97 (1H, dd, J 8.9, 2.4, H-6), 7.08 (1H, d, J 2.4, H-4), 7.04-7.10 (1H, m, H-5’), 7.13-7.23 (3H, m, H-6’, H-7’, H-8’), 7.27 (1H, d, J 8.9, H-7), 8.65 (1H, br., NH); \(\delta_C\) (100 MHz, CDCl\(_3\)) 13.9 (CH\(_2\)-CN), 28.9 (C-4’), 43.3 (C-3’), 47.5 (C-1’), 55.8 (OCH\(_3\)), 100.0 (C-4), 105.0 (CN), 112.9 (C-7), 116.0 (C-6), 117.2 (C-3), 126.4, 126.7, 127.1, 128.9 (CH\(_{ar}\)), 126.8, 128.8, 130.7, 132.2, 133.9 (C\(_{ar}\)), 155.2 (C-5), 163.5 (O=C); m/z (EI): 345 (100%, M\(^+\)), 212 (35), 149 (71), 132 (64).
General procedure for the synthesis of amides 6a-e

A solution of 8 (0.25 g, 0.75 mmol) in dry THF (5 ml) was added dropwise to a stirred suspension of LiAlH₄ (0.29 g, 7.5 mmol) in dry diethyl ether (30 ml) at 0-5 °C. The reaction mixture was heated at 40 °C for 4 h. The reaction was quenched by a slow addition of saturated sodium sulfate solution at 0-5 °C. The formed precipitate was filtered off and washed with THF (10 ml). The combined filtrate and washings were dried (Na₂SO₄), filtered and evaporated under reduced pressure to give 0.24 g (100 %) of the primary amine 10 as a pale yellow viscous oil. δH (400 MHz, CDCl₃) 1.90 (2H, br. s, NH₂), 2.88 (2H, t, J 6.6, CH₂-CH₂-N), 2.96 (2H, t, J 6.6, CH₂-CH₂-N), 3.85 (3H, s, OCH₃), 3.94 (4H, s, H-1’, H-3’), 4.03 (2H, s, CH₂-N), 6.80 (1H, dd, J 8.8, 2.4, H-6), 7.01 (1H, d, J 2.4, H-4), 7.13-7.20 (5H, m, H-4’, H-5’, H-6’, H-7’, H-7), 8.59 (1H, br. s, NH); δc (100 MHz, CDCl₃) 27.9 (CH₂-CH₂-N), 42.5 (CH₂-CH₂-N), 50.7 (CH₂-N), 56.0 (OCH₃), 58.9 (C-1’, C-3’), 100.8 (C-4), 109.8 (C-3), 111.4 (C-7), 111.5 (C-6), 122.3, 126.9 (CHₙ), 128.6, 130.8, 134.0, 139.8 (Car), 153.9 (C-5).

For 6a, a stirred solution of crude 10 (0.1 g, 0.31 mmol) in dry CH₂Cl₂ (10 ml) was treated with triethylamine (0.16 ml, 1.09 mmol) and acetic anhydride (0.08 ml, 0.84 mmol) at 0-5°C. The reaction mixture was stirred at ambient temperature for 18 h. The solvent was evaporated under reduced pressure and the residue was subjected to silica gel chromatography to afford the amide 6a.

For 6b-e, the crude primary amine 10 (1 equiv) was dissolved in dry CH₂Cl₂ (5 ml) and was added dropwise to a stirred mixture of EDCI HCl (1.74 equiv) and the appropriate acid (1 equiv) in dry CH₂Cl₂ (5 ml) at -10 °C under inert atmosphere. The reaction mixture was further stirred at -10 °C for one hour and at room temperature for one hour. The solvent was evaporated under reduced pressure and the residue was subjected to silica gel chromatography to afford the respective amides 5b-e.

**N-2-{5-Methoxy-2-[1(3-dihydro-2H-isooindol-2-ylmethyl)-1H-indol-3-yl]ethyl}acetamide (6a):**

Compound 6a (0.074 g, 65 %) was obtained from 10 (0.1 g, 0.311 mmol) after purification by silica gel chromatography (chloroform/methanol/ammonia, 10:1:0.1) as a pale yellow solid (Found: C, 72.31; H, 6.49; N, 11.37. C₂₂H₂₅N₂O₂ requires C, 72.70; H, 6.93; N, 11.56 %); mp 213-215 °C; vₘₐₓ /cm⁻¹ 3226, 2925, 1633, 1555, 862, 745; δH (400 MHz, DMSO-d₆): 1.65 (3H, s, CH₃), 2.90 (2H, t, J = 6.9, CH₂-CH₂-N), 3.26-3.31 (2H, m, CH₂-CH₂-N), 3.81 (3H, s, OCH₃), 3.95 (4H, s, H-1’, H-3’), 4.00 (2H, s, CH₂-N), 6.74 (1H, dd, J = 8.8, 2.5, H-6), 7.07
N-2-{5-Methoxy-2-{(1,3-dihydro-2H-isooindol-2-ylmethyl)-1H-indol-3-yl}ethyl}propanamide (6b)

Compound 6b (0.072 g, 61 %) was obtained from 10 (0.1 g, 0.311 mmol) after purification by silica gel chromatography (chloroform/methanol/ammonia, 10:1:0.1) as a light beige solid (Found: C, 72.80; H, 7.00; N, 11.00. C_{24}H_{27}N_3O_2 requires C, 73.18; H, 7.21; N, 11.13 %); mp 178-180 °C; ν_{max} / cm^{-1} 3249, 2919, 1637, 862, 750; δ_{1H} (400 MHz, CDCl_3) 0.81 (3H, t, J = 7.6 Hz), 1.65 (2H, q, J = 7.6, CH₂-CH₂), 2.94 (2H, t, J = 6.3, CH₂-CH₂-N), 3.47-3.51 (2H, m, CH₂-CH₂-N), 3.83 (3H, s, OCH₃), 3.96 (4H, s, H-1´, H-3´), 3.97 (2H, s, CH₂-N), 6.81 (1H, br. s, NH), 6.83 (1H, dd, J = 8.8, 2.5, H-6), 6.97 (1H, d, J = 2.5, H-4), 7.14-7.28 (5H, m, H-4´, H-5´, H-6´, H-7´, H-7), 8.39 (1H, br. s, NH), δ_{13C} (100 MHz, CDCl₃) 20.3 (CH₂, 2H), 22.9 (CH₂-CH₂-N), 29.4 (CH₂-CH₂), 40.5 (CH₂-CH₂-N), 51.1 (CH₂-N), 56.3 (OCH₃), 59.4 (C-1´, C-3´), 100.7 (C-4), 110.7 (C-3), 111.9 (C-7), 112.6 (C-6), 122.7, 127.4 (CHₙ), 128.9, 130.8, 133.9, 139.8 (CHₙ), 154.5 (C-5), 174.6 (O=C); m/z (EI): 377 (M⁺, 5), 277 (19), 260 (26), 187 (38), 174 (71), 118 (100).

N-2-{5-Methoxy-2-{(1,3-dihydro-2H-isooindol-2-ylmethyl)-1H-indol-3-yl}ethyl}propanamide (6c)

Compound 6c (0.080 g, 66 %) was obtained from 10 (0.1 g, 0.311 mmol) after purification by silica gel chromatography (chloroform/methanol/ammonia, 10:1:0.1) as a light brown solid (Found: C, 73.75; H, 6.82; N, 10.70. C_{24}H_{27}N_3O_2 requires C, 74.01; H, 6.99; N, 10.79 %); mp 146-148 °C; ν_{max} / cm⁻¹ 3240, 2934, 1639, 921, 745; δ_{1H} (400 MHz, CDCl₃) 2.53 (2H, d, J = 7.1, O=C-CH₂), 2.94 (2H, t, J = 6.3, CH₂-CH₂-N), 3.47-3.51 (2H, m, CH₂-CH₂-N), 3.83 (3H, s, OCH₃), 3.97 (4H, s, H-1´, H-3´), 3.98 (2H, s, CH₂-N), 4.84-4.96 (2H, m, CH₂=CH), 5.64-5.75 (1H, m, CH₂=CH), 6.78 (1H, br. s, NH), 6.83 (1H, dd, J = 8.6, 2.3, H-6), 6.97 (1H, d, J = 2.3 Hz, H-4), 7.15-7.29 (5H, m, H-4´, H-5´, H-6´, H-7´, H-7), 8.48 (1H, br. s, NH); δ_{13C} (100 MHz, CDCl₃) δ 23.9 (CH₂-CH₂-N), 40.4 (CH₂-CH₂-N), 40.8 (O=C-CH₂), 50.8 (CH₂-N), 55.9 (OCH₃), 59.0 (C-1´, C-3´), 100.4 (C-4), 110.2 (C-3), 111.7 (C-7), 112.2 (C-6), 118.2
(CH₂=CH), 122.4, 127.2 (CH₃), 128.5, 130.6 (C₆), 131.7 (CH₂=CH), 133.5, 139.4 (C₈), 154.2 (C-5), 170.9 (O=C); m/z (EI): 389 (M⁺, 4), 272 (18), 187 (31), 174 (25), 118 (100).

N-2-{5-Methoxy-2-[(1,3-dihydro-2H-isindol-2-ylmethyl)-1H-indol-3-yl]ethyl}cyclopropanecarboxamide (6d)

Compound 6d (0.070 g, 58 %) was obtained from 10 (0.1 g, 0.311 mmol) after purification by silica gel chromatography (chloroform/methanol/ammonia, 10:1:0.1) as a light brown solid (Found: C, 73.65; H, 6.89; N, 10.65. C₂₅H₂₇N₃O₂ requires C, 74.01; H, 6.99; N, 10.79 %); mp 209-211 °C; v_max /cm⁻¹ 3204, 2934, 1638, 1064, 745; δ_H (400 MHz, CDCl₃) δ 0.17-0.22 (2H, m, cPr-Ch₂), 0.56-0.72 (3H, m, cPr-Ch₂, cPr-CH), 2.96 (2H, t, J = 6.3, CH₂-Ch₂-N), 3.48-3.52 (2H, m, CH₂-Ch₂-N), 3.83 (3H, s, OCH₃), 3.99 (2H, s, CH₂-N), 4.00 (4H, s, H-1', H-3'), 6.84 (1H, dd, J = 8.6, 2.5, H-6), 6.98 (1H, d, J = 2.5, H-4), 7.14-7.23 (6H, m, H-4', H-5', H-6', H-7', H-7, NH), 8.37 (1H, br. s, NH). δ_C (100 MHz, CDCl₃) δ 6.9 (2 x cPr-Ch₂), 14.2 (cPr-CH), 23.9 (CH₂-Ch₂-N), 40.7 (CH₂-Ch₂-N), 51.1 (CH₂-N), 56.2 (OCH₃), 59.2 (C-1', C-3'), 100.7 (C-4), 111.1 (C-3), 111.9 (C-7), 112.7 (C-6), 122.6, 127.4 (CH₃), 128.8, 130.8, 133.6, 139.7 (C₆), 154.5 (C-5), 174.2 (O=C). m/z (EI): = 389 (M⁺, 3), 272 (26), 187 (39), 174 (62), 118 (100).

N-2-{5-Methoxy-2-[(1,3-dihydro-2H-isindol-2-ylmethyl)-1H-indol-3-yl]ethyl}cyclobutanecarboxamide (6e):

Compound 6e (0.090 g, 71 %) was obtained from 10 (0.1 g, 0.311 mmol) after purification by silica gel chromatography (chloroform/methanol/ammonia, 10:1:0.1) as pale beige solid (Found: C, 74.05; H, 7.09; N, 10.32. C₂₅H₂₉N₃O₂ requires C, 74.41; H, 7.24; N, 10.41 %); mp 200-202 °C; v_max /cm⁻¹ 3245, 2931, 1642, 1031, 733; δ_H (400 MHz, CDCl₃) δ 1.45-1.61 (2H, m, cBu-Ch₂), 1.63-1.68 (2H, m, cBu-Ch₂), 2.03-2.13 (2H, m, cBu-Ch₂), 2.29-2.38 (1H, m, cBu-Ch₂), 2.95 (2H, t, J = 6.1, CH₂-Ch₂-N), 3.47-3.52 (2H, m, CH₂-Ch₂-N), 3.85 (3H, s, OCH₃), 3.98 (2H, s, CH₂-N), 3.99 (4H, s, H-1’, H-3’), 6.82 (1H, br. s, NH), 6.85 (1H, dd, J = 8.6, 2.3, H-6), 6.98 (1H, d, J = 2.3 Hz, H-4), 7.19-7.26 (5H, m, H-4’, H-5’, H-6’, H-7’, H-7), 8.48 (1H, br. s, NH). δ_C (100 MHz, CDCl₃) 18.2 (cBu-Ch₂), 23.7 (CH₂-Ch₂-N), 25.3 (2 x cBu-Ch₂, ), 39.6 (cBu-Ch₂, ), 40.3 (CH₂-Ch₂-N), 50.9 (CH₂-N), 56.1 (OCH₃), 59.2 (C-1’, C-3’), 100.6 (C-4), 110.7 (C-3), 111.9 (C-7), 112.4 (C-6), 122.5, 127.4 (CH₃), 128.7, 130.7, 133.6, 139.6 (C₆), 154.3 (C-5), 175.3 (O=C). m/z (EI): = 403 (M⁺, 3), 286 (29), 187 (57), 174 (63), 118 (100).
N-2-[(2,3,4-Tetrahydroisoquinolin-2-ylmethyl)-5-methoxy-1H-indol-3-yl]ethylacetamide (4):

A solution of 9 (0.13 g, 0.38 mmol) in dry THF (5 ml) was added dropwise to a stirred suspension of LiAlH₄ (0.15 g, 3.9 mmol) in dry diethyl ether (30 ml) at 0-5 °C. The reaction mixture was heated at 40 °C for 4 h. The reaction was quenched by a slow addition of saturated sodium sulfate solution at 0-5 °C. The formed precipitate was filtered off and washed with THF (10 ml). The combined filtrate and washings were dried (Na₂SO₄), filtered and evaporated under reduced pressure to give 126 mg (100 %) of the amine 11 as a yellow brown viscous oil. A stirred solution of crude amine 11 (126 mg, 0.38 mmol) in dry CH₂Cl₂ (10 ml) was treated with triethylamine (0.20 ml, 1.33 mmol) and acetic anhydride (0.10 ml, 0.95 mmol) at 0-5°C. The reaction mixture was stirred at ambient temperature for 18 h. The solvent was evaporated under reduced pressure and the residue was purified by silica gel chromatography (ethyl acetate) to give 58 g (41 %) of the amide 4 as a brown yellow solid (Found: C, 72.85; H, 7.12; N, 10.00. C₂₃H₂₇N₃O₂ requires C, 73.18; H, 7.21; N, 11.13 %); mp 47-49 °C; ν<sub>max</sub> /cm⁻¹: 3256, 3063, 2922, 2851, 1650, 1631, 1550, 1485, 1451, 1292, 1215; δ<sub>H</sub> (400 MHz, CDCl₃) 1.73 (3H, s, CH₃), 2.86 (2H, t, J 5.6, H-4ˊ), 2.89-2.98 (4H, m, CH₂-CH₂-N, H-3ˊ), 3.49 (2H, q, J 6.5, CH₂CH₂-N), 3.68 (2H, s, H-1ˊ), 3.82 (2H, s, CH₂-N), 3.84 (3H, s, OCH₃), 5.99 (1H, t, J 5.2, NH), 6.83 (1H, dd, J 8.9, 2.4, H-6), 6.92-6.97 (1H, m, H-5ˊ), 6.99 (1H, d, J 2.4, H-4), 7.06-7.18 (3H, m, H-6ˊ, H-7ˊ, H-8´), 7.21 (1H, d, J 8.9, H-7), 8.62 (1H, br., NH). δ<sub>C</sub> (100 MHz, CDCl₃) 23.0 (CH₃), 23.9 (CH₂-CH₂-N), 40.3 (CH₂-CH₂-N), 53.1 (CH₂-N), 56.0 (OCH₃), 28.6 (C-4´), 50.9 (C-3´), 63.7 (C-1´), 100.4 (C-4), 110.8 (C-3), 111.8 (C-7), 112.2 (C-6), 126.4, 126.7, 127.1, 128.9 (CH₃), 128.6, 130.7, 132.8, 133.6, 136.8 (CA), 154.2 (C-5), 170.3 (O=O); m/z (EI): 377 (M⁺, 3), 291 (23), 246 (25), 187 (31), 174 (74), 132 (100).

N-[2-(2-Indol-1-ylmethyl-5-methoxy-1H-indol-3-yl)-ethyl]acetamide (5a)

Pd/C 10% (32 mg) was added to a solution of 3² (230 mg, 0.633 mmol) in methanol (25 mL) and stirred for 10 min. The solvent was evaporated in vacuo and the residue was heated at 150 °C for 6 h. The reaction mixture was allowed to cool, chloroform (2 x 25 ml) was added, and the catalyst was removed by filtration (Celite®). The organic sovent was removed in vacuo and the residue purified by silica gel chromatography (chloroform/methanol/ammonia, 15:1:0.1) to give 124 mg 5a (54 %) as a yellow foam (C₂₃H₂₇N₃O₂Na⁺ required 384.16824, found 384.16825); ν<sub>max</sub> /cm⁻¹: 3277, 2922, 2852, 1648, 1540, 1486, 1459, 1363, 1297, 1216,
N-[2-(2-indol-1-ylmethyl-5-methoxy-1H-indol-3-yl)-ethyl)cyclobutane carboxamide (5b)

Pd/C 10% (9 mg) was added to a solution of N-(2-\{5-Methoxy-2-[(2,3-dihydro-1H-indol-1yl)methyl]-1H-indol-3-yl\}ethyl)cyclobutane carboxamide\(^2\) (70 mg, 0.174 mmol) in methanol (25 mL) and stirred for 10 min. The solvent was evaporated in vacuo and the mixture was heated at 150 °C for 3 h. The reaction mixture was allowed to cool, chloroform (2 x 7 ml) was added and the catalyst was removed by filtration (Celite®). The organic solvent was removed in vacuo and the residue was purified by silica gel chromatography (chloroform/methanol/ammonia, 15:1:0.1) to give 20 mg 5b (29 %) as a yellow viscous oil. (C\(_{25}\)H\(_{23}\)N\(_3\)O\(_2\)Na\(^+\) required 424.19956, found 424.19954) ν\(_{\text{max}}\) /cm\(^{-1}\): 3259, 2929, 2852, 1615, 1485, 1457, 1250, 1215, 1142, 1112, 1031, 799; δ\(_{\text{H}}\) (400 MHz, CDCl\(_3\)) 1.24 (2H, t, J 7.2, cBu-CH\(_2\)), 1.96-2.08 (3H, m, cBu-CH\(_2\), cBu-CH), 2.12-2.24 (2H, m, cBu-CH\(_2\)), 2.97 (2H, t, J 6.9, CH\(_2\)CH\(_2\)N), 3.45 (2H, qua, J 6.9, CH\(_2\)CH\(_2\)N), 3.83 (3H, s, OCH\(_3\)), 5.37 (2H, s, CH\(_2\)-N-1’), 5.38 (1H, br, NH), 6.52 (1H, d, J 2.5, H-4), 6.79 (1H, dd, J 8.7, 2.5, H-6), 6.97-7.21 (5H, m, H-7, H-2’, H-3’, H-4’, H-5’), 7.30 (1H, d, J 8.3, H-7’), 7.64 (1H, d, J 7.6, H-6’), 7.77 (1H, br, H-1); δ\(_{\text{C}}\) (100 MHz, CDCl\(_3\)) 18.0 (cBu-CH\(_2\)), 21.0 (cBu-CH), 24.4 (CH\(_2\)CH\(_2\)N), 25.3 (2C, cBu-CH\(_2\)), 40.0 (CH\(_2\)CH\(_2\)N), 41.9 (CH\(_2\)N-1’), 56.0 (OCH\(_3\)), 100.6 (C-3’), 102.4 (C-4), 109.2 (C-7’), 110.5 (C-3), 111.8 (C-7), 112.5 (C-6), 119.9 (C-4’), 121.2 (C-6’), 122.1 (C-5’), 127.6 (C-2’), 128.6 (C-3a’), 128.8 (C-3a), 130.7 (C-7a), 131.2 (C-2), 136.3 (C-7a’), 154.3 (C-5), 175.0 (CONH).

References