SUPPLEMENTARY MATERIAL

Synthesis of 3-benzoyl-4-benzylfurans structural related to furolignans

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Abstract

Furolignan-type natural products, possessing various important biological proprieties, have been synthesized from a commercially available furan. The elaborated synthetic strategy is based on an innovative Friedel-Crafts reaction starting from an alcohol or a carboxylic acid instead of a chloride analogues and triflic anhydride as promoter. Through this synthetic strategy, furolignans having two different aryl groups have been obtained. The products have been evaluated for their antimicrobial properties on Gram positive and Gram negative bacteria, in order to compare their biological activities with those of natural analogues.

Keywords: furolignan; furan; synthesis, Friedel-Crafts reaction, anhydride triflic, antimicrobial activity

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Experimental

General experimental procedures

All solvents and reagents were obtained from commercial supplier and used without further purification. The reactions involving air or moisture sensitive reagents were carried out under dry nitrogen atmosphere using dry solvents (Sigma-Aldrich 99.7%), stored over molecular sieves.

Thin layer chromatography (TLC) was performed on aluminium plates precoated with Merck Silica Gel 60 F_{254} as the adsorbent (0.25mm for analytical; 0.50, 1.0 and 2.0 mm, for preparative TLC). Spots were visualized by UV light and developed with 10% H_2SO_4 ethanolic solution.

Column chromatography was conducted on Silica Gel 0.06-0.20 mm (Merck Kieselgel).

Electron Ionization Mass spectra (EI-MS) measurements were performed with an Agilent 6850 GC (Milan, Italy) equipped with an HP-5MS capillary column and the Agilent 5973 Inert MS detector.

^1H NMR (400 MHz or 500 MHz) and ^13C NMR (100 MHz or 126 MHz) spectra were recorder on a Bruker DRX-400 or INOVA 500 spectrometers at room temperature. The carbon multiplicity was evidenced by DEPT experiments. The proton couplings were evidenced by ^1H-^1H COSY experiments.

Proton-detected heteronuclear correlations were measured using a gradient heteronuclear single-quantum coherence (HSQC), optimized for ^1J_{HC} = 155 Hz, a gradient heteronuclear multiple bond coherence (HMBC), optimized for ^3J_{HC} = 8 Hz.

Spectral data of synthetized compounds

Methyl 4-(hydroxymethyl) furan-3-carboxylate (1): ^1H NMR (400 MHz, CDCl_3): δ_H 7.99 (d, 1H, J = 1.6 Hz, H-2), 7.41 (d, 1H, J = 1.6 Hz, H-5), 4.64 (d, 2H, J = 6.8 Hz, -CH_2-), 3.89 (s, 3H, MeO); ^13C NMR (101 MHz, CDCl_3): δ_c 164.9 (COOMe), 149.3 (C-2), 141.1 (C-5), 125.2 (C-3), 117.8 (C-4), 55.2 (-CH_2-), 51.9 (MeO); EI-MS: m/z = 156.01 [C_7H_8O_4, calcd. 156.042, M^+].

Methyl 4-(4-methoxybenzyl) furan-3-carboxylate (3a): ^1H NMR (400 MHz, CDCl_3): δ_H 7.99 (s, 1H, H-2), 7.18 (d, 2H, J = 7.0 Hz, H-2’ and H-6’), 7.03 (s, 1H, H-5), 6.86 (d, 2H, J = 7.0 Hz, H-3’ and H-5’), 3.96 (s, 2H, -CH_2-), 3.82 and 3.81 (s, 6H, MeO); ^13C NMR (101 MHz, CDCl_3): δ_c 163.9 (COOMe), 158.0 (C-4’), 149.0 (C-2), 141.8 (C-5), 131.7 (C-1’), 129.7 (C-2’ and C-6’), 125.6 (C-3), 118.0 (C-4), 113.8 (C-3’ and C-5’), 55.2 (MeO), 51.3 (COOMe), 29.4 (-CH_2-); EI-MS: m/z = 246.06 [C_{14}H_{14}O_4, calcd. 246.089, M^+].
Methyl 4-(2-methoxybenzyl) furan-3-carboxylate (4a): $^1$H NMR (400 MHz, CDCl$_3$): $\delta$H 7.98 (s, 1H, H-2), 7.23 (t, 1H, $J = 7.8$, H-4'), 7.18 (d, 1H, $J = 7.0$ Hz, H-6'), 7.00 (s, 1H, H-5), 6.93-6.88 (m, 2H, H-3' and H-5'), 4.02 (s, 2H, -CH$_2$-), 3.84 and 3.83 (s, 6H, MeO); $^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$C 164.0 (COOMe), 157.3 (C-2'), 148.7 (C-2), 142.1 (C-5), 130.1 (C-4'), 128.2 (C-1'), 127.6 (C-6'), 124.4 (C-3), 120.4 (C-5'), 118.2 (C-4), 110.3 (C-3'), 55.3 (MeO), 51.2 (COOMe), 24.4 (-CH$_2$-); EI-MS: m/z = 246.04 [C$_{14}$H$_{14}$O$_4$, calcd. 246.089, M$^+$].

Methyl 4-(3,4-dimethoxybenzyl) furan-3-carboxylate (3b): $^1$H NMR (400 MHz, CDCl$_3$): $\delta$H 7.98 (s, 1H, Hz, H-2), 7.03 (s, 1H, H-5), 6.84-6.73 (m, 3H, H-2', H-5' and H-6'), 3.95 (s, 2H, -CH$_2$-), 3.87 (s, 3H, MeO), 3.86 (s, 3H, MeO), 3.81 (s, 3H, MeO); $^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$C 163.9 (COOMe), 149.0 (C-2), 148.8 and 147.5 (C-3' and C-4'), 141.8 (C-5), 132.2 (C-1'), 125.5 (C-3), 120.7 (C-6'), 117.9 (C-4), 112.1 (C-5'), 111.2 (C-2'), 55.9 (OMe), 55.8 (OMe), 51.3 (COOMe), 29.9 (-CH$_2$-); EI-MS: m/z = 276.11 [C$_{15}$H$_{16}$O$_5$, calcd. 276.099, M$^+$].

Methyl 4-(benzo[d][1,3] dioxol-5-ylmethyl)furan-3-carboxylate (3c): $^1$H NMR (400 MHz, CDCl$_3$): $\delta$H 8.00 (d, 1H, $J = 1.7$ Hz, H-2), 7.07 (d, 1H, $J = 1.7$ Hz, H-5), 6.85-6.68 (m, 3H, H-2', H-5' and H-6'), 5.95 (s, 2H, -OCH$_2$O-), 3.93 (s, 2H, -CH$_2$-), 3.82 (s, 3H, MeO); $^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$C 163.8 (COOMe), 149.1 (C-2), 147.6 and 145.9 (C-3' and C-4'), 141.8 (C-5), 133.5 (C-1'), 125.3 (C-3), 122.1 (C-6'), 117.9 (C-4), 109.2 (C-2'), 106.9 (C-5'), 100.8 (-OCH$_2$O-), 51.3 (COOMe), 29.9 (-CH$_2$-); EI-MS: m/z = 260.05 [C$_{14}$H$_{13}$O$_5$, calcd. 260.068, M$^+$].

Methyl 4-(2-hydroxybenzyl) furan-3-carboxylate (3d): $^1$H NMR (400 MHz, CDCl$_3$): $\delta$H 7.99 (d, 1H, $J = 1.7$ Hz, H-2), 7.13 (d, 2H, $J = 8.8$ Hz, H-2' and H-6'), 7.03 (br d, 1H, $J = 2.0$ Hz, H-5), 6.78 (d, 2H, $J = 8.8$ Hz, H-3' and H-5'), 3.94 (s, 2H, -CH$_2$-), 3.82 (OMe); $^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$C 163.9 (COOMe), 153.9 (C-4'), 149.1 (C-2), 141.8 (C-5), 131.9 (C-1'), 129.9 (C-2' and C-6'), 125.5 (C-3), 117.9 (C-4), 115.2 (C-3' and C-5'), 51.3 (OMe), 29.4 (-CH$_2$-); EI-MS: m/z = 230.02 [C$_{13}$H$_{12}$O$_4$, calcd. 230.073, M$^+$].

4-(4-Methoxybenzyl) furan-3-carboxylic acid (5a): $^1$H NMR (400 MHz, CDCl$_3$): $\delta$H 8.10 (s, 1H, H-2), 7.19 (d, 2H, $J = 7.8$ Hz, H-2' and H-6'), 7.03 (s, 1H, H-5), 6.87 (d, 2H, $J = 7.8$ Hz, H-3' and H-5'), 3.96 (s, 2H, -CH$_2$-), 3.82 (OMe); $^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$C 168.2 (COOMe), 155.0 (C-4'), 150.3 (C-2), 142.4 (C-5), 130.2 (C-2' and C-6'), 128.7 (C-1'), 125.6 (C-3), 117.3 (C-4), 113.8 (C-3' and C-5'), 55.3 (OMe), 29.3 (-CH$_2$-); EI-MS: m/z = 232.03 [C$_{13}$H$_{12}$O$_4$, calcd. 232.074, M$^+$].

4-(2-Methoxybenzyl) furan-3-carboxylic acid (6a): $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.90 (s, 1H, H-2), 7.20 (m, 1H, H-6'), 7.02 (s, 1H, H-5), 6.95-6.87 (m, 3H, H-3', H-4' and H-5'), 4.03 (s, 2H, -CH$_2$-), 3.85 (OMe); $^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$C 166.2 (COOMe), 156.5 (C-2'), 150.0 (C-2), 142.2 (C-5), 129.8 (C-6'), 127.7
(C-4’), 125.0 (C-3), 122.7 (C-1’), 121.1 (C-5’), 117.3 (C-4), 113.8 (C-3’), 55.3 (OMe), 27.2 (-CH₂-); EI-MS: m/z = 232.03 [C₁₃H₁₂O₄, calcd. 232.074, M⁺].

4-(3,4-Dimethoxybenzyl) furan-3-carboxylic acid (5b): ¹H NMR (400 MHz, CDCl₃): δH 8.11 (d, 1H, J= 1.7 Hz, H-2), 7.06 (d, 1H, J=1.7 Hz, H-5), 6.85-6.78 (m, 3H, H-2’, H-5’ and H-6’), 3.97 (br s, 2H, -CH₂-), 3.89 (s, 3H, OMe), 3.88 (s, 3H, OMe); ¹³C NMR (101 MHz, CDCl₃): δC 168.1 (COOMe), 150.4 (C-2), 148.8 (C-3’), 147.5 (C-4’), 142.2 (C-5), 132.0 (C-1’), 125.8 (C-3), 120.8 (C-6’), 117.3 (C-5), 112.2 (C-5’), 111.2 (C-2’), 55.9 (OMe), 55.8 (OMe), 29.8 (-CH₂-); EI-MS: m/z = 262.09 [C₁₄H₁₄O₅, calcd. 262.084, M⁺].

4-(Benzo[d][1,3]dioxol-5-ylmethyl)furan-3-carboxylic acid (5c): ¹H NMR (400 MHz, CDCl₃): δH 8.10 (d, 1H, J = 1.7 Hz, H-2), 7.09 (d, 1H, J = 1.7 Hz, H-5), 6.80-6.68 (m, 3H, H-2’, H-5’ and H-6’), 5.95 (s, 2H, -OCH₂O-), 3.94 (s, 2H, -CH₂-); ¹³C NMR (101 MHz, CDCl₃): δC 168.3 (COOMe), 150.5 (C-2), 147.6 and 145.9 (C-3’ and C-4’), 142.2 (C-5), 133.3 (C-3’), 125.6 (C-3), 121.5 (C-6’), 117.3 (C-4), 109.3 (C-2’), 108.2 (C-5’), 100.9 (-OCH₂O-), 29.7 (-CH₂-); EI-MS: m/z = 246.07 [C₁₃H₁₀O₅, calcd. 246.053, M⁺].

(3,4-Dimethoxyphenyl) (4-(4-methoxybenzyl)furan-3-yl) methanone (7a): ¹H NMR (400 MHz, CDCl₃): δH 7.77 (s, 1H, Hz, H-2), 7.54-7.43 (m, 2H, H-2” and H-6”’), 7.20 (d, 2H, J = 7.6 Hz, H-2 and H-3), 7.11 (s, 1H, H-5), 6.91 (d, 1H, J = 8.2 Hz, H-5”), 6.84 (d, 2H, J = 7.6 Hz, H-3’ and H-5’), 4.02 (s, 2H, -CH₂-), 3.97, 3.95, 3.79 (s, 9H, MeO); ¹³C NMR (101 MHz, CDCl₃): δC 189.2 (CO), 158.0 (C-4’), 153.0 (C-4”), 149.1 (C-3”), 148.8 (C-2), 141.9 (C-5), 132.2 (C-1”), 130.2 (C-1’), 129.8 (C-2” and C-6”), 126.6 (C-3), 125.8 (C-6”), 123.8 (C-4), 113.3 (C-3” and C-5”), 111.3 (C-2”), 109.9 (C-5”), 56.1 (X2 OMe), 56.0 (OMe), 29.5 (-CH₂-); EI-MS: m/z = 352.10 [C₂₁H₂₀O₅, calcd. 352.131, M⁺].

(3,4-Dimethoxyphenyl) (4-(2-methoxybenzyl)furan-3-yl) methanone (8a): ¹H NMR (400 MHz, CDCl₃): δH 7.75 (s, 1H, Hz, H-2), 7.55-7.47 (m, 2H, H-2” and H-6”’), 7.24-7.18 (m, 2H, H-4’ and H-6’), 7.11 (s, 1H, H-5), 6.91 (d, 1H, J = 8.2 Hz, H-5”), 6.89-6.87 (m, 2H, H-3’ and H-5’), 4.08 (s, 2H, -CH₂-), 3.98, 3.96, 3.82 (s, 9H, MeO); ¹³C NMR (101 MHz, CDCl₃): δC 189.2 (CO), 157.3 (C-2’), 153.0 (C-4”’), 148.8 (C-3”), 148.3 (C-2), 142.2 (C-5), 132.3 (C-1”), 130.2 (C-4”), 128.6 (C-3 and C-1’), 126.6 (C-6”), 125.3 (C-4), 123.8 (C-6’), 120.5 (C-2”), 111.2 (C-5”), 110.4 (C-5’), 109.9 (C-3’), 56.0 (OMe), 55.3 (OMe), 55.2 (OMe), 24.4 (-CH₂-); EI-MS: m/z = 352.10 [C₂₁H₂₀O₅, calcd. 352.131, M⁺].

(4-(3,4-Dimethoxybenzyl) furan-3-yl) (4-methoxyphenyl) methanone (7b): ¹H NMR (400 MHz, CDCl₃): δH 7.68 (s, 1H, H-2), 7.27 (obscured by solvent, 1H, H-5’ and H-6’), 7.10 (d, 2H, J = 8.8 Hz, H-2” and H-6”), 7.02 (s, 1H, H-5), 6.97 (brs, 1H, H-2’), 6.85 (d, 2H, H-3” and H-5”’), 4.31 (-CH₂-), 4.06 (OMe), 4.04 (OMe), 3.81 (OMe); ¹³C NMR (101 MHz, CDCl₃): δC 194.7 (CO), 163.0 (C-4”’), 158.2 (C-3’), 153.3 (C-4”), 149.1 (C-2), 136.1 (C-5), 134.5 (C-3), 132.4 (C-1’), 131.2 (C-1”), 129.4 (C-2” and C-6”), 119.2 (C-6’), 118.7 (C-4),
114.2 (C-3” and C-5”), 106.4 (C-5’), 103.0 (C-2’), 56.1 (OMe), 56.0 (OMe), 55.3 (OMe), 37.7 (-CH2-); EI-MS: m/z = 352.3 [C21H20O5, calcd. 352.131, M⁺].

(4-(Benzo[d][1,3]dioxol-5-ylmethyl)furan-3-yl)(4-methoxyphenyl)methanone (7c): ¹H NMR (400 MHz, CDCl3): δH 7.70 (s, 1H, H-2), 7.12 (d, 2H, J = 8.6 Hz, H-2” and H-6”), 7.02 (s, 1H, H-5), 6.95 (m, 2H, H-5’ and H-6’), 6.85 (m, 3H, H-2’, H-3” and H-5”), 6.11 (s, 2H, -OCH2O-), 4.29 (-CH2-), 3.80 (OMe); ¹³C NMR (101 MHz, CDCl3): δC 192.7 (CO), 157.9 (C-4” and C-3’), 157.3 (C-4’), 157.2 (C-2), 133.7 (C-5), 133.0 (C-3), 130.1 (C-1’), 129.9 (C-2” and C-6”), 129.7 (C-1”), 127.3 (C-6’), 120.4 (C-5”), 113.8 (C-3” and C-5”), 113.6 (C-2’), 110.4 (-OCH2O-), 55.2 (OMe), 40.1 (-CH2-); EI-MS: m/z = 336.0 [C20H16O5, calcd. 336.100, M⁺].

Microbroth dilution assay

Minimal inhibitory concentrations (MIC) of the tested compounds were determined in LB and TSB medium by the broth micro-dilution assay, according to the European Committee on Antimicrobial Susceptibility Testing. Bacterial suspensions were diluted to a final concentration of 1 × 10⁶ CFU ml⁻¹. The compounds were added to bacterial suspension in each well yielding a final cell concentration of 5 × 10⁵ CFU ml⁻¹ and a final compound concentration ranging from 2 μg ml⁻¹ to 128 μg ml⁻¹. Negative control wells were set to contain bacteria in LB or TSB plus the amount of DMSO used to dilute each compound. Positive controls included tobramycin and vancomycin (ranging from 0.25 μg ml⁻¹ to 4 μg ml⁻¹). Medium turbidity was measured by a microtiter plate reader (Tecan, Milan, Italy) at 595 nm. Absorbance was proportional to bacterial growth.

List of supporting information

Figure S1 ¹H NMR spectrum (400 MHz) of compound 1 in CDCl₃
Figure S2 ¹³C NMR spectrum (100 MHz) of compound 1 in CDCl₃
Figure S3: ¹H NMR spectrum (400 MHz) of compound 3a in CDCl₃
Figure S4: ¹³C NMR spectrum (100 MHz) of compound 3a in CDCl₃
Figure S5: ¹H NMR spectrum (400 MHz) of compound 4a in CDCl₃
Figure S6: ¹³C NMR spectrum (100 MHz) of compound 4a in CDCl₃
Figure S7: ¹H NMR spectrum (400 MHz) of compound 3b in CDCl₃
Figure S8: ¹³C NMR spectrum (100 MHz) of compound 3b in CDCl₃
Figure S9: $^1$H NMR spectrum (400 MHz) of compound 3c in CDCl$_3$
Figure S10: $^{13}$C NMR spectrum (100 MHz) of compound 3c in CDCl$_3$
Figure S11: $^1$H NMR spectrum (400 MHz) of compound 3d in CDCl$_3$
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Figure S13: $^1$H NMR spectrum (400 MHz) of compound 5a in CDCl$_3$
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Figure S15: $^1$H NMR spectrum (400 MHz) of compound 6a in CDCl$_3$
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Figure S17: $^1$H NMR spectrum (400 MHz) of compound 5b in CDCl$_3$
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Figure S19: $^1$H NMR spectrum (500 MHz) of compound 5c in CDCl$_3$
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Figure S24: $^{13}$C NMR spectrum (100 MHz) of compound 7b in CDCl$_3$
Figure S25: $^1$H NMR spectrum (500 MHz) of compound 7c in CDCl$_3$
Figure S26: $^{13}$C NMR spectrum (125 MHz) of compound 7c in CDCl$_3$
Figure S1: $^1$H NMR Spectrum of Methyl 4-(hydroxymethyl) furan-3-carboxylate (1)

Figure S2: $^{13}$C NMR Spectrum of Methyl 4-(hydroxymethyl) furan-3-carboxylate (1)
Figure S3: $^1$H NMR Spectrum of Methyl 4-(4-methoxybenzyl) furan-3-carboxylate (3a)

Figure S4: $^{13}$C NMR Spectrum of Methyl 4-(4-methoxybenzyl) furan-3-carboxylate (3a)
Figure S5: $^1$H NMR Spectrum of Methyl 4-(2-methoxybenzyl) furan-3-carboxylate (4a)

Figure S6: $^{13}$C NMR Spectrum of Methyl 4-(2-methoxybenzyl) furan-3-carboxylate (4a)
Figure S7: $^1$H NMR Spectrum of Methyl 4-(3,4-dimethoxybenzyl) furan-3-carboxylate (3b)

Figure S8: $^{13}$C NMR Spectrum of Methyl 4-(3,4-dimethoxybenzyl) furan-3-carboxylate (3b)
Figure S9: $^1$H NMR Spectrum of Methyl 4-(benzo[d][1,3] dioxol-5-ylmethyl)furan-3-carboxylate (3c)

Figure S10: $^{13}$C NMR Spectrum of Methyl 4-(benzo[d][1,3] dioxol-5-ylmethyl)furan-3-carboxylate (3c)
Figure S11: $^1$H NMR Spectrum of Methyl 4-(4-hydroxybenzyl) furan-3-carboxylate (3d)

Figure S12: $^{13}$C NMR Spectrum of Methyl 4-(4-hydroxybenzyl) furan-3-carboxylate (3d)
Figure S13: $^1$H NMR Spectrum of 4-(4-methoxybenzyl) furan-3-carboxylic acid (5a)

Figure S14: $^{13}$C NMR Spectrum of 4-(4-methoxybenzyl) furan-3-carboxylic acid (5a)
Figure S15: $^1$H NMR Spectrum of 4-(2-methoxybenzyl) furan-3-carboxylic acid (6a)

Figure S16: $^{13}$C NMR Spectrum of 4-(2-methoxybenzyl) furan-3-carboxylic acid (6a)
Figure S17: $^1$H NMR Spectrum of 4-(3,4-Dimethoxybenzyl) furan-3-carboxylic acid (5b)

Figure S18: $^{13}$C NMR Spectrum of 4-(3,4-Dimethoxybenzyl) furan-3-carboxylic acid (5b)
Figure S19: $^1$H NMR Spectrum of 4-(Benzo[d][1,3] dioxol-5-ylmethyl)furan-3-carboxylic acid (5c)

Figure S20: $^{13}$C NMR Spectrum of 4-(Benzo[d][1,3] dioxol-5-ylmethyl)furan-3-carboxylic acid (5c)
Figure S21: $^1$H NMR Spectrum of (3,4-dimethoxyphenyl) (4-(4-methoxybenzyl)furan-3-yl) methanone (7a) and (3,4-dimethoxyphenyl) (4-(2-methoxybenzyl)furan-3-yl) methanone (8a)
Figure S22: $^{13}$C NMR Spectrum of (3,4-dimethoxyphenyl) (4-(4-methoxybenzyl)furan-3-yl) methanone (7a) and (3,4-dimethoxyphenyl) (4-(2-methoxybenzyl)furan-3-yl) methanone (8a)
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Figure S24: $^{13}$C NMR Spectrum of (4-(3,4-dimethoxybenzyl) furan-3-yl) (4-methoxyphenyl) methanone (7b)
Figure S25: $^1$H NMR Spectrum of (4-(benzo[d][1,3]dioxol-5-ylmethyl)furan-3-yl)(4-methoxyphenyl)methanone (7c)
Figure S26: $^{13}$C NMR Spectrum of (4-(benzo[d][1,3]dioxol-5-ylmethyl)furan-3-yl)(4-methoxyphenyl)methanone (7c)