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SUPPLEMENTARY MATERIAL TO

Green and efficient synthesis of new *β*-amido-aroyl carbonyl derivatives catalyzed by choline chloride/urea as a deep eutectic solvent

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THE SPECTRAL AND ANALYTICAL DATA FOR THE NEW COMPOUNDS

N-*[1-(2-Hydroxy-4,4-dimethyl-6-oxocyclohex-1-enyl)-2-(4-nitrophenyl)-2--oxoethyl]-benzamide (**4a**)*. White powder; m.p.: 200 °C; Anal. calcd. for $C_{23}H_{22}N_2O_6$: C, 65.39; H, 5.25; N, 6.63 %. Found: C, 65.58; H, 5.41; N, 6.39 %; IR (KBr, ν_{max} / cm^{-1}): 3417 (OH), 3258 (NH), 1710 & 1610 (C=O); ^{1}H-NMR (250 MHz, $CDCl_3$, δ / ppm): 0.63 (3H, *s*, CH_3), 0.99 (3H, *s*, CH_3), 1.85–2.25 (2H, *m*, CH_2), 2.67 (2H, *s*, CH_2), 5.81 (1H, *s*, CH), 7.50–8.27 (10H, *m*, Ar & NH), 12.43(1H, broad, OH); ^{13}C-NMR (62.90 MHz, $CDCl_3$, δ / ppm): 26.08, 30.03, 31.05, 43.69, 49.44, 51.76, 114.12, 123.56, 127.47, 128.90, 131.50, 132.98, 140.33, 150.88, 163.15, 169.48, 177.85, 192.88, 196.34.

N-*[2-(4-Chlorophenyl)-1-(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-enyl)-2--oxoethyl]-benzamide (**4b**)*. White powder; m.p.: 205 °C; Anal. calcd. for $C_{23}H_{22}ClNO_4$: C, 67.07; H, 5.38; N, 3.40 %. Found: C, 67.31; H, 5.55; N, 3.25 %; IR (KBr, ν_{max} / cm^{-1}): 3444 (OH), 3308 (NH), 1686 & 1636 (C=O); ^{1}H-NMR (250 MHz, DMSO-d_6), δ / ppm): 1.11 (3H, *s*, CH_3), 1.25 (3H, *s*, CH_3), 2.47 (2H, *s*, CH_2), 2.76–2.80 (2H, *m*, CH_2), 6.55 (1 H, *s*, CH), 7.38–8.38 (10 H, *m*, Ar & NH), 12.23 (1H, broad, OH); ^{13}C-NMR (62.90 MHz, $CDCl_3$, δ / ppm): 26.08, 30.03, 31.87, 42.19, 50.66, 52.39, 107.81, 122.43, 126.64, 127.30, 128.70, 130.08, 131.85, 131.98 , 150.36, 166.33, 170.43, 193.75, 196.25.

N-[*2-(4-Bromophenyl)-1-(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-enyl)-2--oxoethyl]-benzamide (**4c**)*. White powder; m.p.: 225 °C, Anal. calcd. for $C_{23}H_{22}BrNO_4$: C, 60.54; H, 4.86; N, 3.07 %. Found: C, 60.41; H, 4.66; N, 3.41 %; IR (KBr, ν_{max} / cm^{-1}): 3416 (OH), 3311 (NH), 1701, 1636 (C=O); ^{1}H-NMR (250 MHz, DMSO-d_6, δ / ppm): 1.15 (3H, *s*, CH_3), 1.24 (3H, *s*, CH_3), 2.45 (2H,

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s, CH_2), 2.79 (2H, *s*, CH_2), 6.53 (1H, *s*, CH), 7.40–8.33 (10H, *m*, Ar & NH), 12.23 (1H, broad, OH); ^{13}C-NMR (62.90 MHz, $CDCl_3$, δ / ppm): 27.59, 28.46, 29.44, 43.25, 52.53, 60.25, 106.59, 126.51, 127.99, 128.84, 129.34, 130.41, 132.33, 133.56, 138.75, 165.45, 166.54, 192.13, 193.00.

N-*[2-(4-Bromophenyl)-1-(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-enyl)-2--oxoethyl]-acetamide(**4d**)*. White powder; m.p.: 210 °C; Anal. calcd. for $C_{18}H_{20}BrNO_4$: C, 54.84; H, 5.11; N, 3.55 %. Found: C, 54.92; H, 5.35; N, 3.72 %; IR (KBr, ν_{max} / cm^{-1}): 3412 (OH), 3322 (NH), 1698, 1621 (C=O); ^{1}H-NMR (250 MHz, $CDCl_3$: δ / ppm): 0.66 (3H, *s*, CH_3), 0.99 (3H, *s*, CH_3), 2.11(3H, *s*, CH_3), 2.29–2.44 (2H, *m*, CH_2), 2.79 (2H, *s*, CH_2), 5.53 (1H, *s*, CH), 6.59–7.65 (5H, *m*, Ar & NH), 12.09 (1H, broad, OH); ^{13}C-NMR (62.90 MHz, $CDCl_3$, δ / ppm): 22.48, 26.10, 30.06, 30.93, 43.61, 49.55, 51.16, 114.59, 126.64, 129.40, 131.69, 131.82, 172.92, 177.07, 193.75, 196.32.

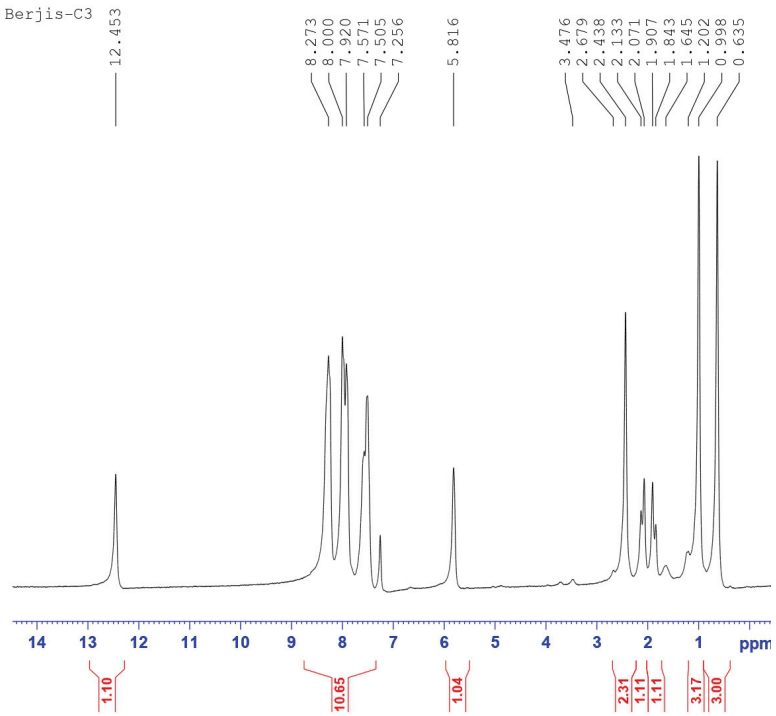
N-*[1-(2-Hydroxy-4,4-dimethyl-6-oxo cyclohex-1-enyl)-2-(4-nitrophenyl)-2--oxoethyl]-acetamide (**4e**)*.White powder; m.p.: 215 °C; Anal. calcd. for $C_{18}H_{20}N_2O_6$: C, 59.99; H, 5.59; N, 7.77 %. Found: C, 59.76; H, 5.68; N, 7.51 %; IR (KBr, ν_{max} / cm^{-1}): 3447 (OH), 3324 (NH), 1702, 1673 (2 C=O); ^{1}H-NMR (250 MHz, $CDCl_3$, δ / ppm): 0.60 (3H, *s*, CH_3), 0.99 (3H, *s*, CH_3), 2.15 (3H, *s*, CH_3), 2.40–248 (2H, *m*, CH_2), 2.85 (2H, *s*, CH_2), 5.58 (1H, *s*, CH), 7.90–8.25 (5H, *m*, Ar & NH), 12.18 (1H, broad, OH); ^{13}C-NMR (62.90 MHz, $CDCl_3$, δ / ppm): 22.49, 26.04, 30.01, 31.00, 42.69, 49.41, 51.52, 114.32, 123.52, 124.05, 125.39, 128.87, 173.11, 177.78, 193.75, 196.36.

N-*[2-(4-Bromophenyl)-1-(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-enyl)-2--oxoethyl]-propionamide (**4f**)*. White powder; m.p.: 210 °C; Anal. calcd. for $C_{19}H_{22}BrNO_4$: C, 55.89; H, 5.43; N, 3.43 %. Found: C, 55.75; H, 5.64; N, 3.57 %; IR (KBr, ν_{max} / cm^{-1}): 3433 (OH), 3307 (NH), 1695 & 1623 (2 C=O); ^{1}H-NMR (250 MHz, $CDCl_3$, δ / ppm): 0.66 (3H, *s*, CH_3), 0.98 (3H, *s*, CH_3), 1.18–2.24 (3H, *m,* CH_3), 1.83–2.04 (2H, *m*, CH_2), 2.36–2.45 (4H, *m*, CH_2), 5.53 (1H, *s*, CH), 7.42–7.73 (5H, *m*, Ar & NH), 12.24 (1H, broad, OH); ^{13}C-NMR (62.90 MHz, $CDCl_3$ δ /ppm): 9.39, 22.48, 26.10, 30.06, 30.93, 43.61, 49.55, 51.16, 114.59, 126.64, 129.40, 131.69, 131.82, 172.92, 177.07, 193.75, 196.32.

N-*[1-(6-Hydroxy-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydro-pyrimidin-5-yl)--2-oxo-2-*p*-tolyl-ethyl]-acetamide (**4g**)*. White powder; m.p.: 200–203 °C, Anal. calcd. for $C_{17}H_{19}N_3O_5$: C, 59.12; H, 5.55; N, 12.17 %. Found: C, 59.32; H, 5.81; N, 12.34 %; IR (KBr, ν_{max} / cm^{-1}): 3418 (OH, NH), 1677,1639(2 C=O). ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 2.02 (3H, *s*, CH_3), 3.13 (1H, *s*, CH_3), 3.30 (6H, *s*, 2N-CH_3), 3.33 (3H, *s*, CH_3), 5.62 (1H, *s*, CH), 7.32–8.19 (5H, *m*, Ar & NH), 12.24 (1H, broad, OH); ^{13}C-NMR (62.90 MHz, $CDCl_3$, δ / ppm): 22.48, 23.78, 28.05, 28.82, 49.05, 81.55, 128.05, 129.25, 135.42, 138.82, 160.24, 171.45, 177.32, 182.75, 187.32.

N-*[2-(4-Chlorophenyl)-1-(6-hydroxy-4-oxo-2-thioxo-1,2,3,4-tetrahydro-pyrimidin-5-yl)-2-oxoethyl]-propionamide* (***4h***). White powder, m.p.: 215–217 °C, Anal. calcd. for $C_{15}H_{14}ClN_3O_4S$: C, 48.98; H, 3.84 N, 11.42 %. Found: C, 48.76; H, 3.63 N, 11.61 %; IR (KBr, ν_{max} / cm^{-1}): 3386 (OH), 3203(NH), 1657 & 1630 (2 C=O); ^{1}H-NMR (250 MHz, $CDCl_3$, δ / ppm): 0.91 (3H, *m*, CH_3), 2.18–2.42 (2H, *m*, CH_2), 6.15 (1H, *s*, CH), 7.36–8.04 (5H, *m*, Ar & NH), 11.15 (1H, *s*, NH), 12.26 (1H, *s*, NH), 12.58 (1H, broad, OH);. ^{13}C-NMR (62.90 MHz, $CDCl_3$, δ / ppm): 11.61, 31.09, 51.05, 76.79, 129.08, 129.59, 131.44, 135.09, 164.27, 165.55, 172.55, 176.09, 186.09.

SELECTED ORIGINAL SPECTRA OF THE SYNTHESIZED B-AMIDO-AROYL CARBONYL DERIVATIVES



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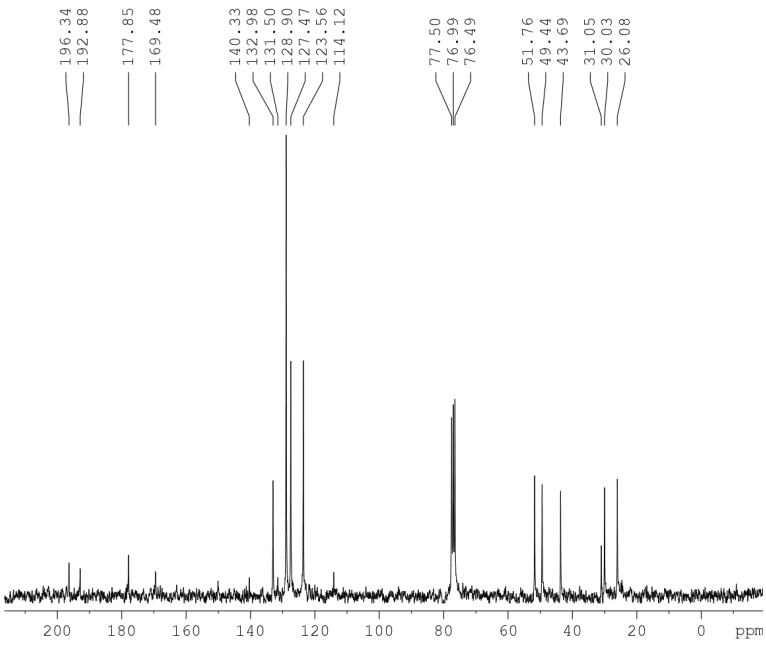
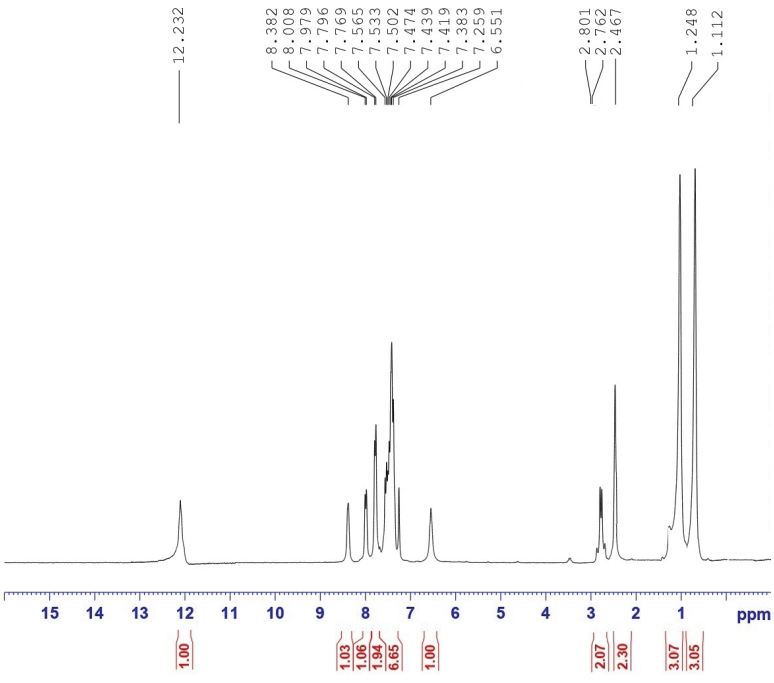


^{13}C-NMR-**4a**.



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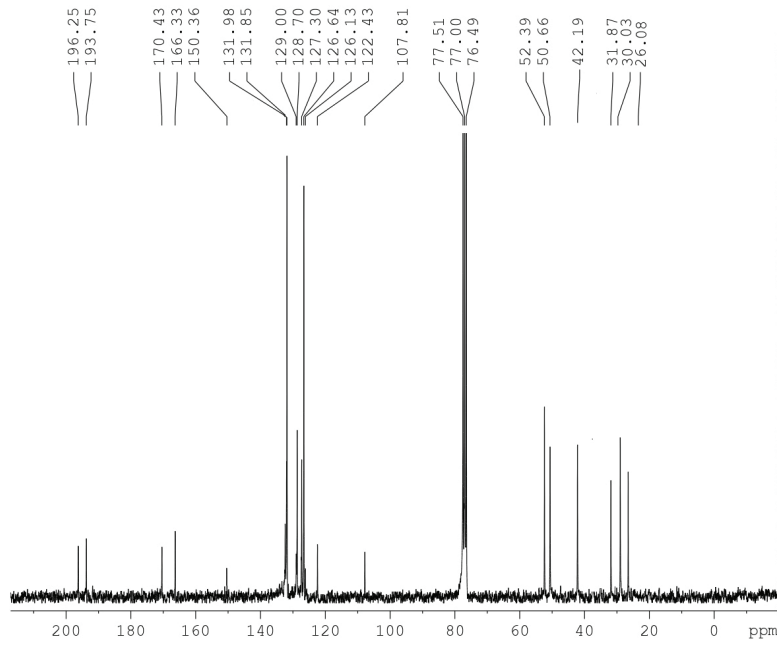
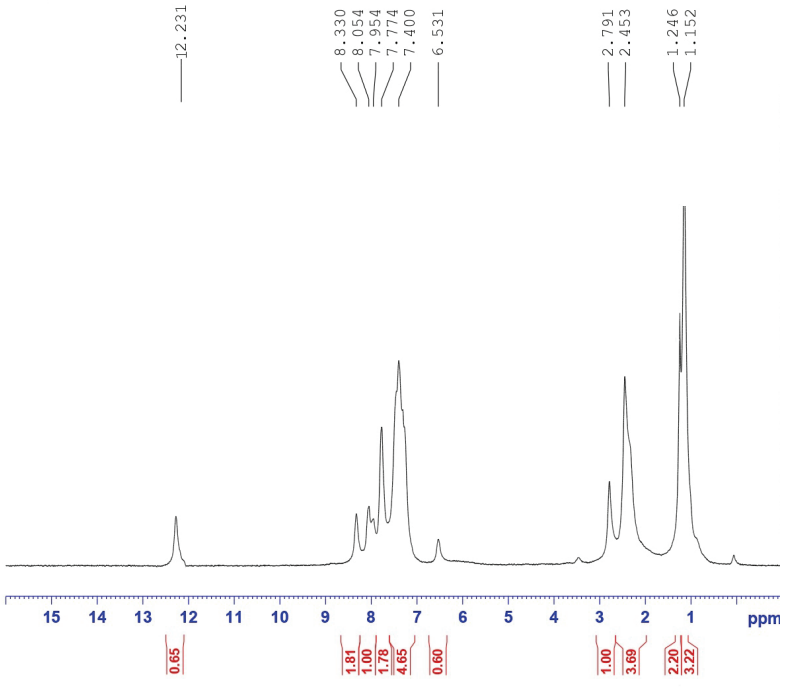


^{13}C-NMR-**4b**.



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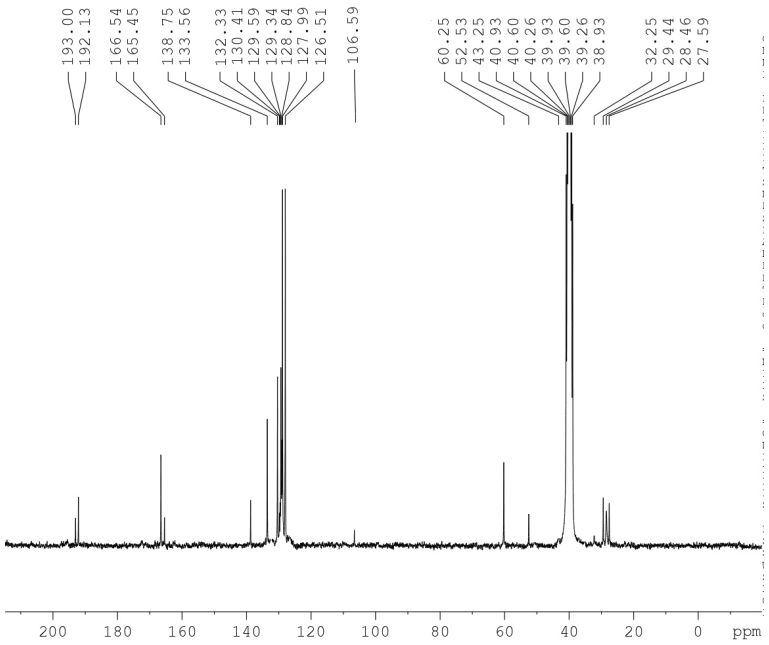

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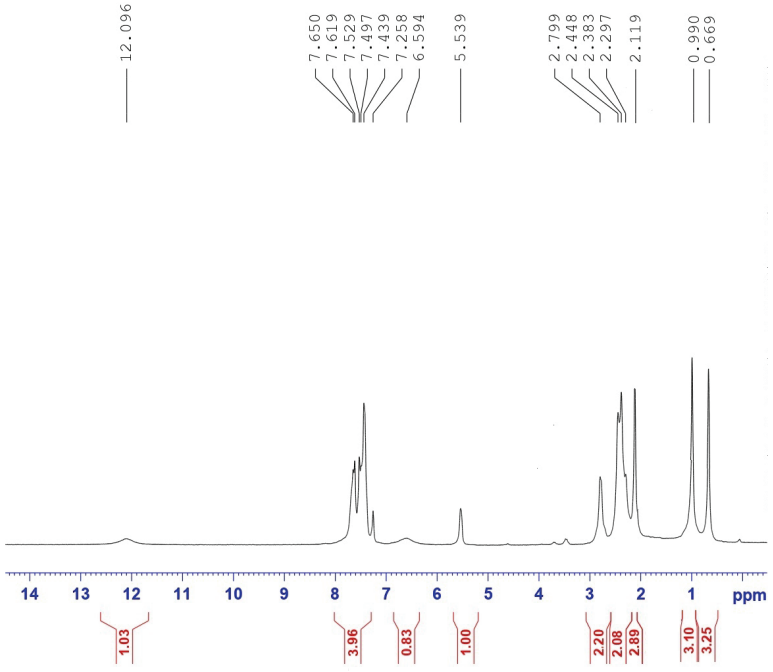

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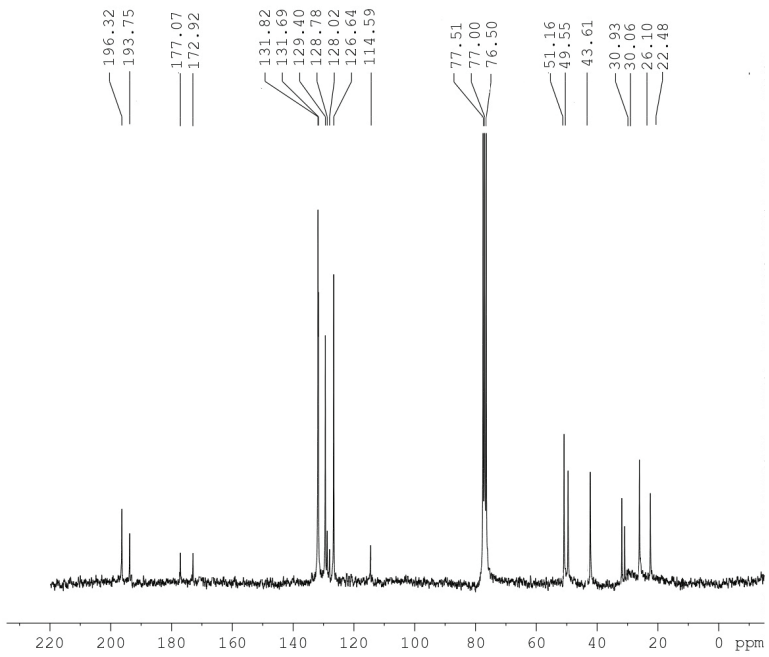

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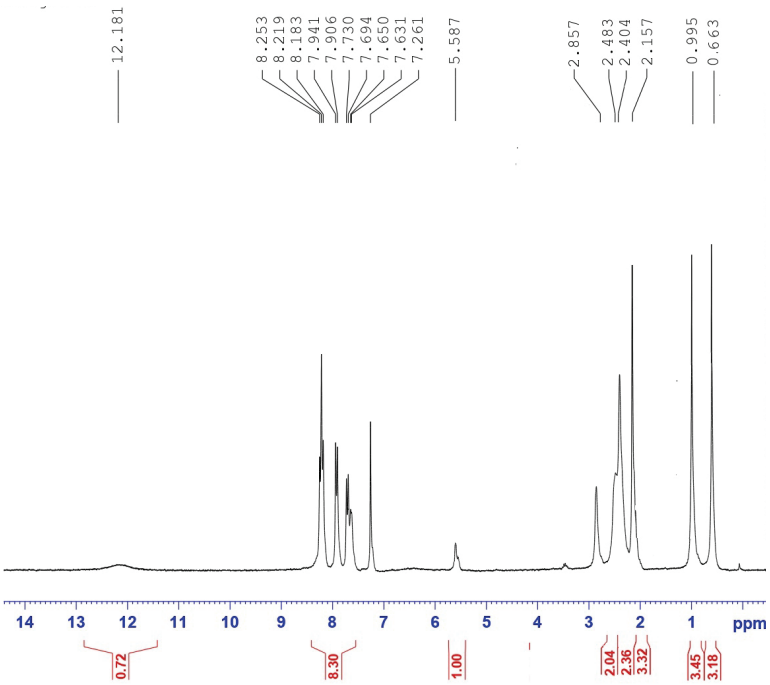

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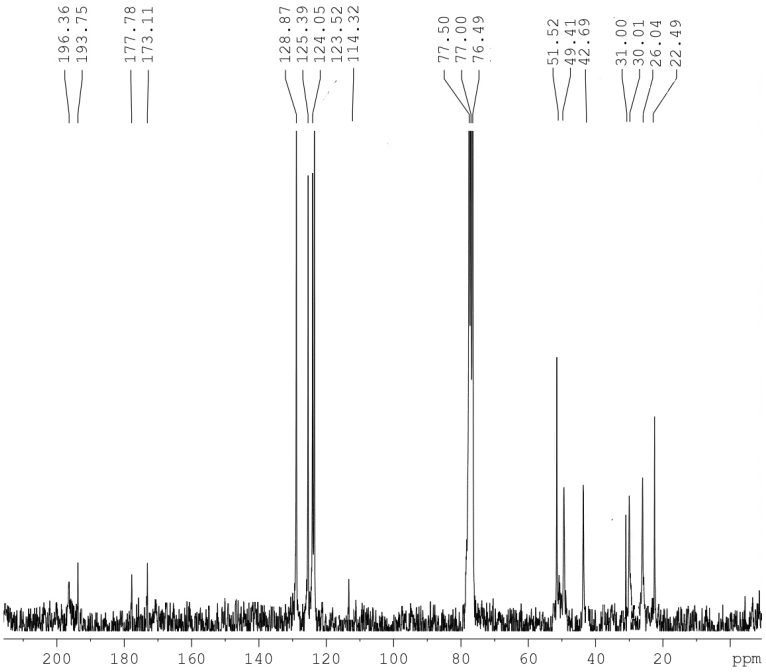

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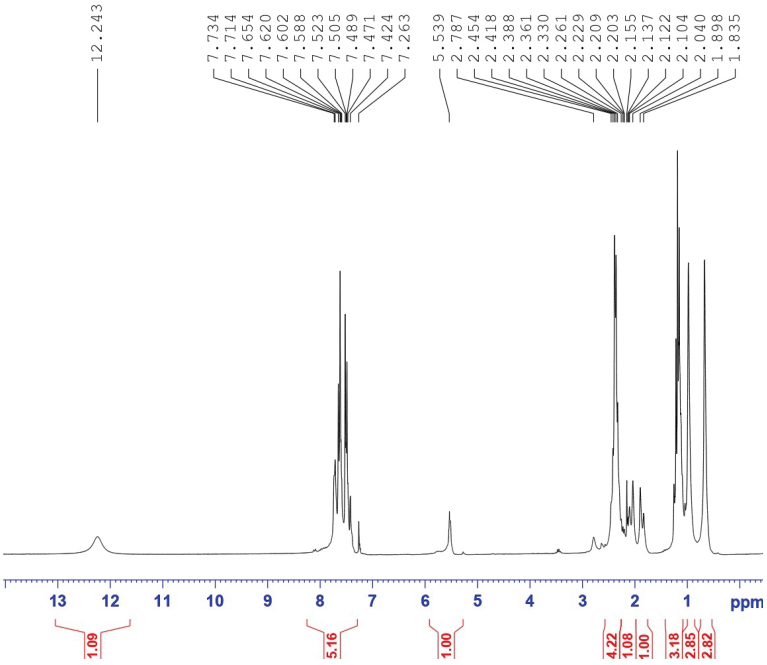

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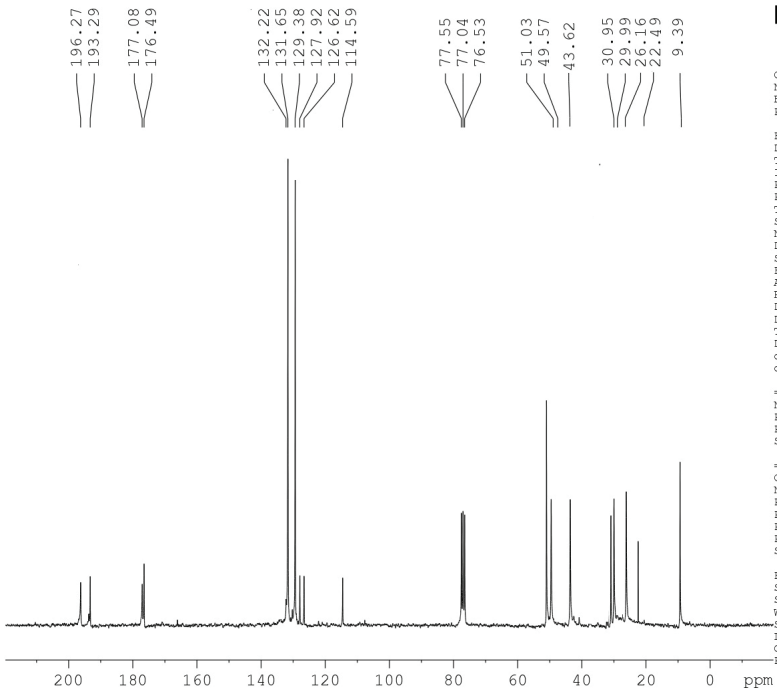

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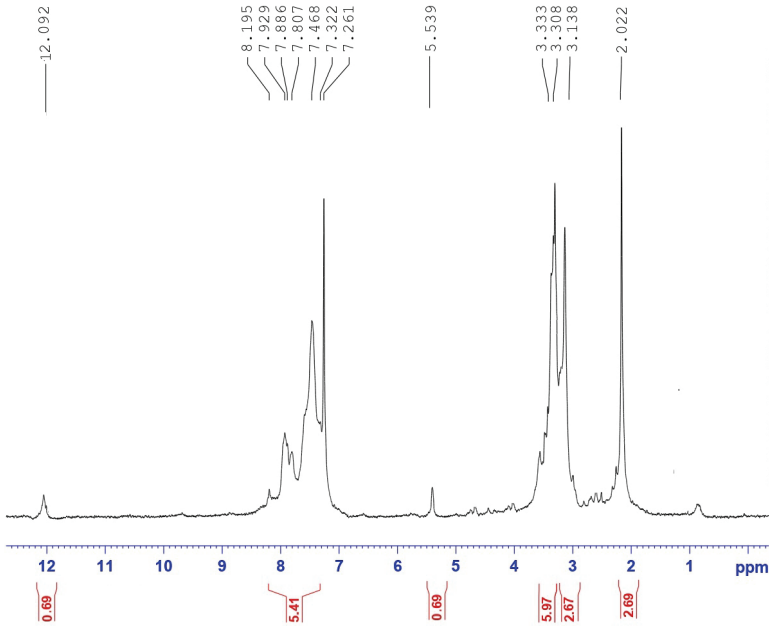

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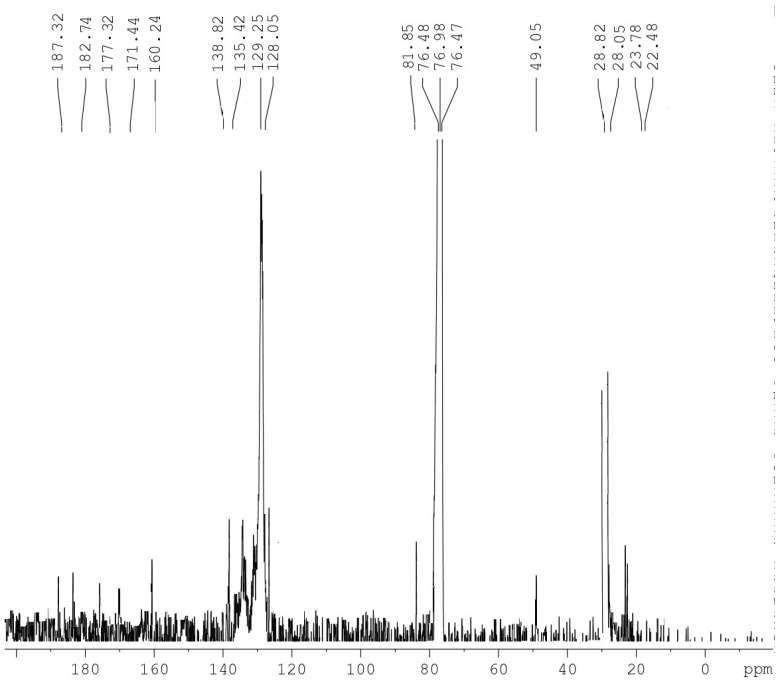

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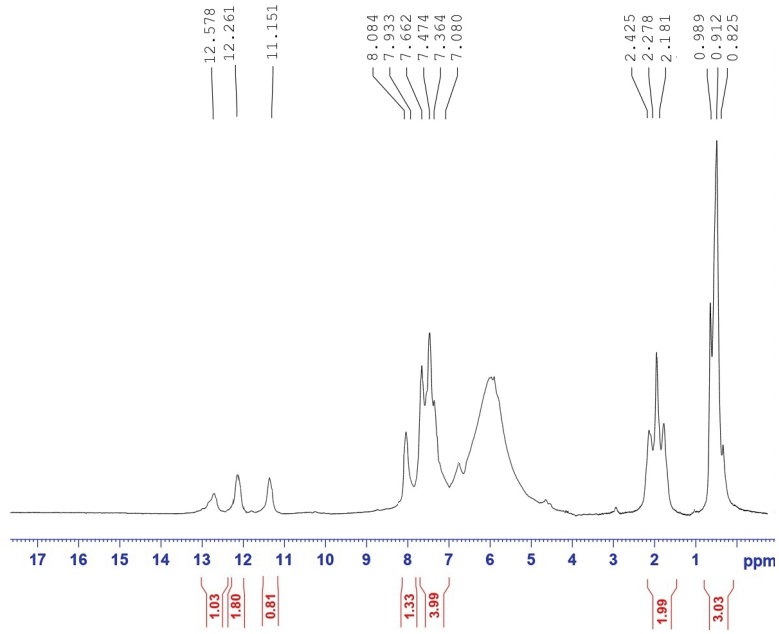
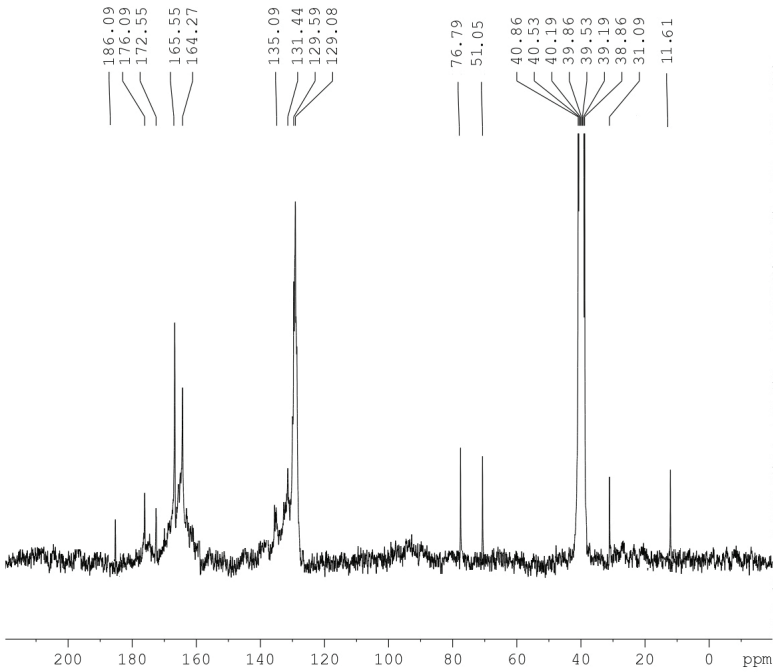


^{1}H-NMR-**4h**.



^{13}C-NMR-**4h**.