

Catalytic application of a novel melamine-naphthalene-1,3-disulfonic acid metal-organic frameworks in the synthesis of β -acetamido ketones

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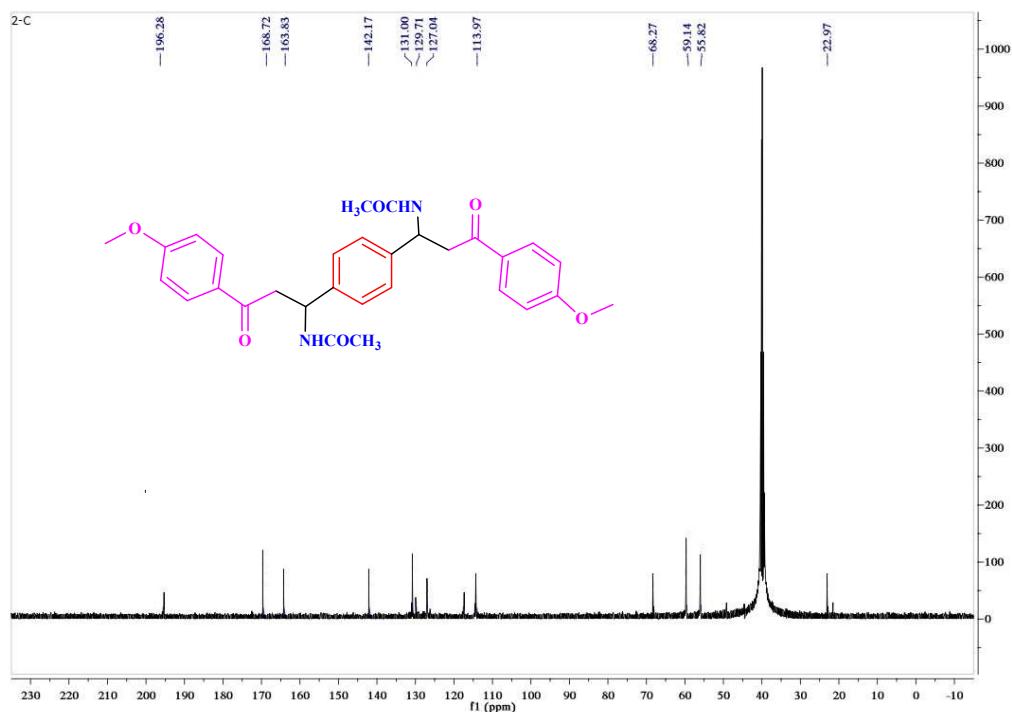
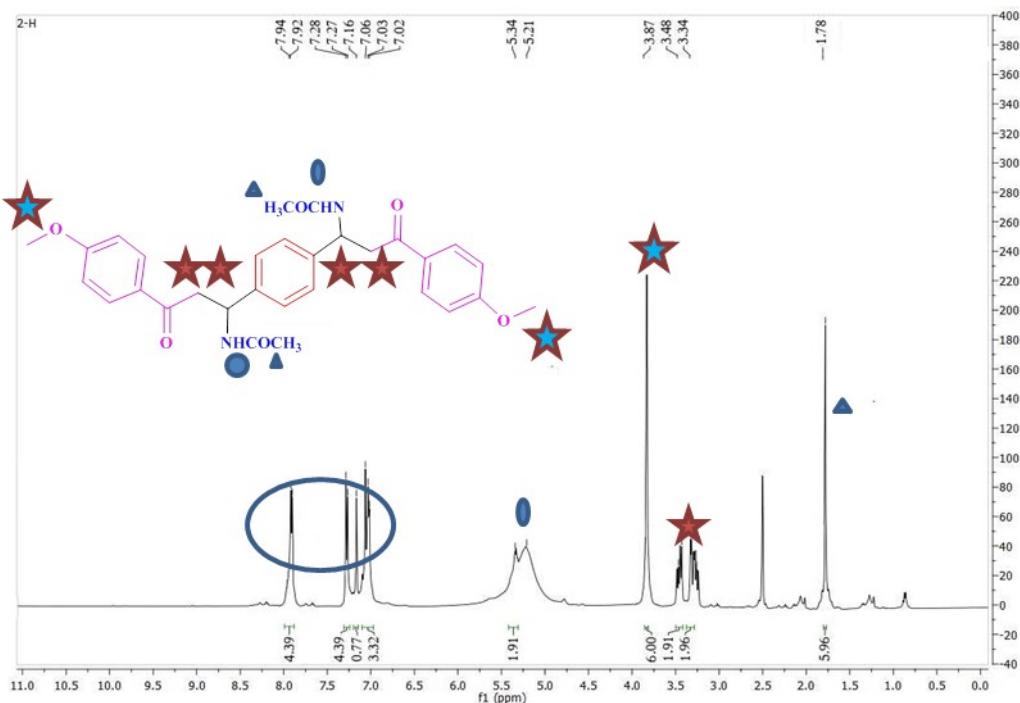
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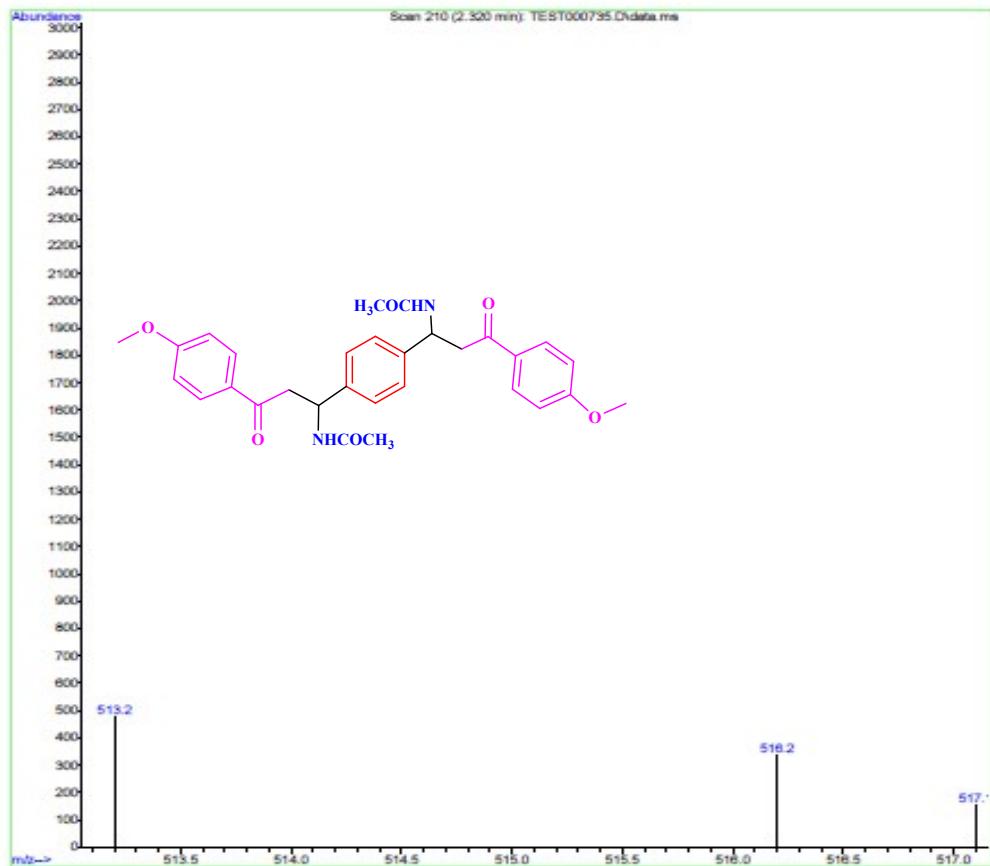
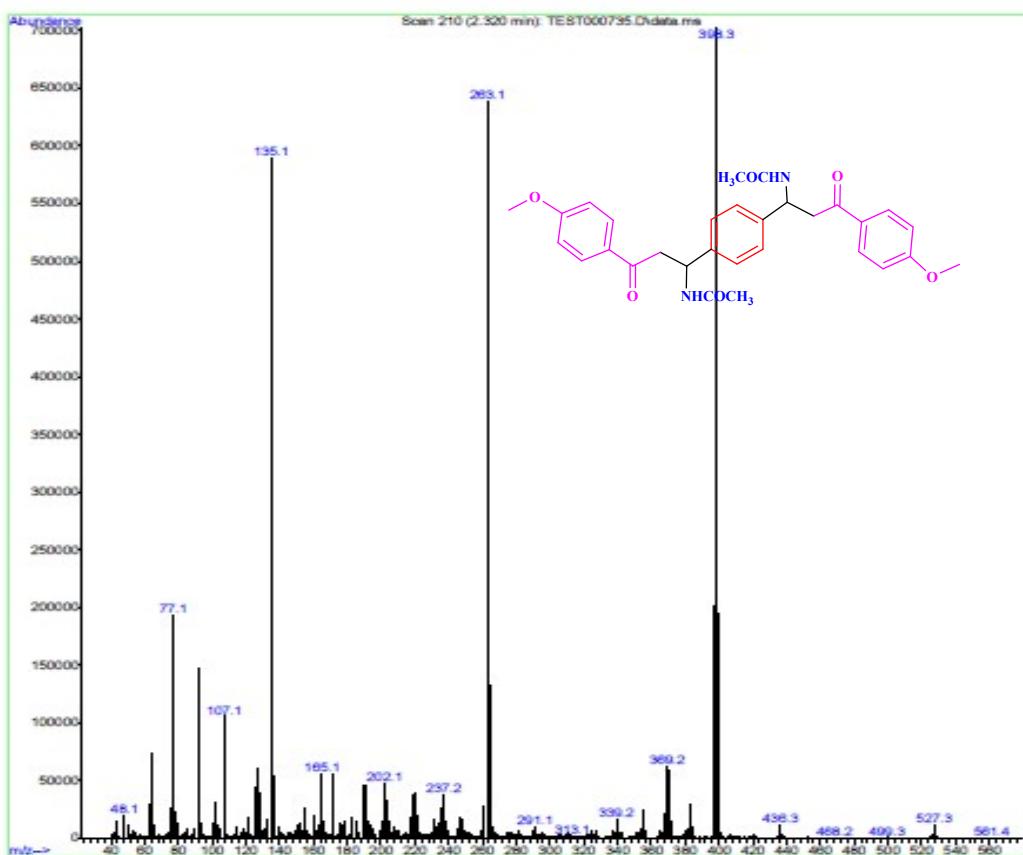
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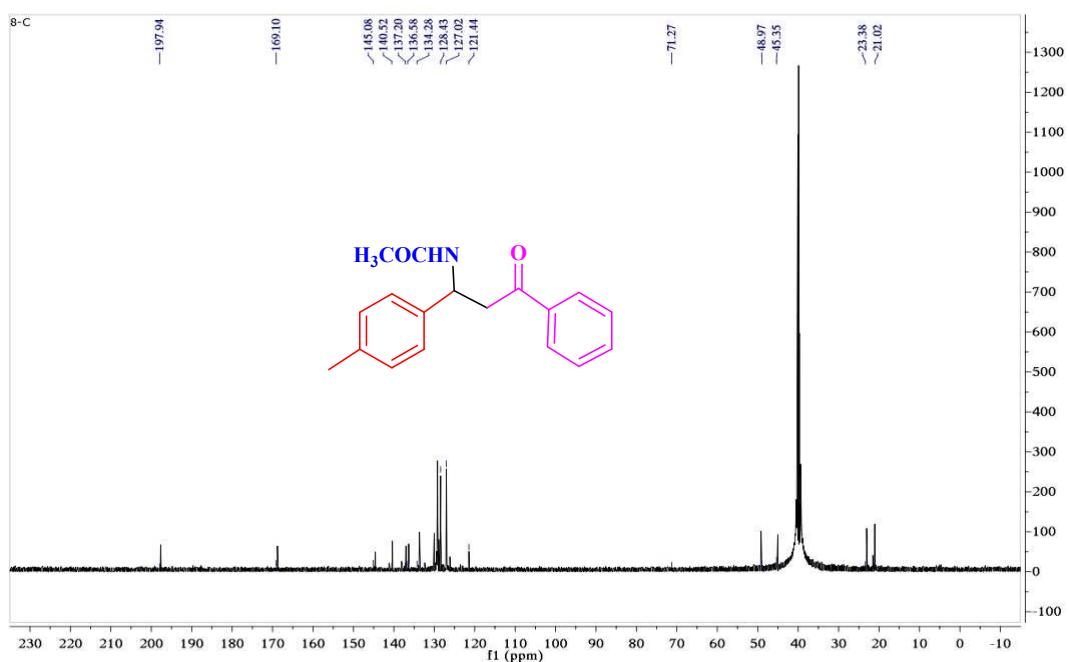
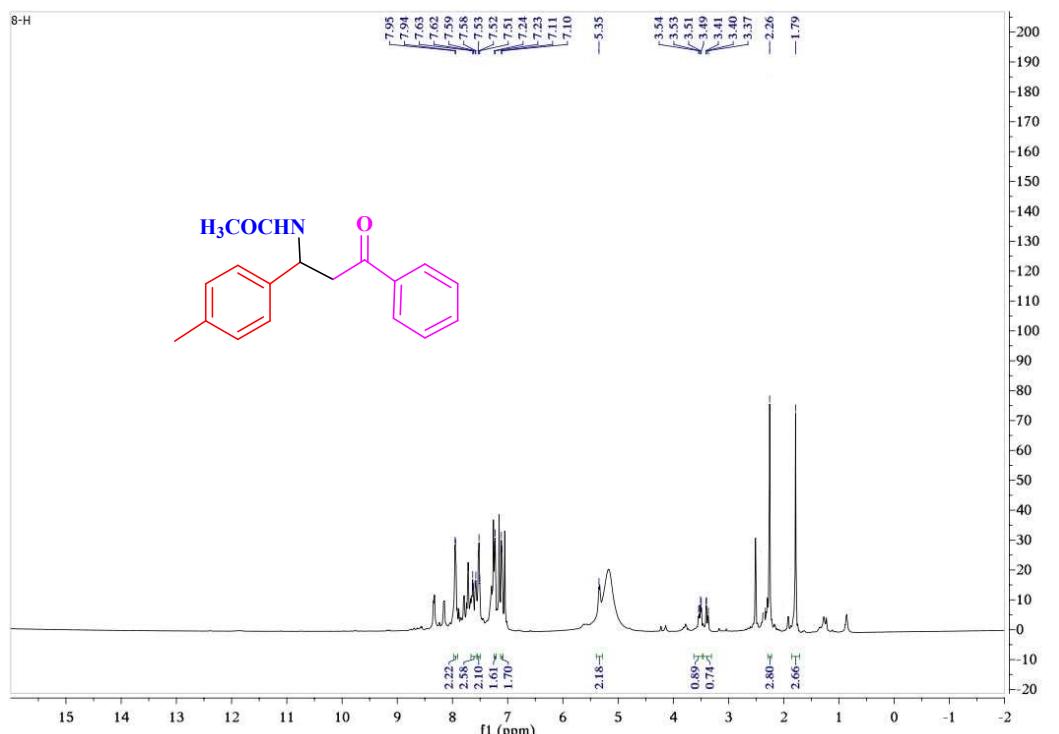
Spectral data:

***N,N'*-(1,4-Phenylenebis(3-(4-methoxyphenyl)-3-oxopropane-1,1-diyl))diacetamide:** Cream solid, m.p.: 255–260 °C, H NMR (500 MHz, DMSO-*d*6, TMS) δ 7.93 (2H, d, *J* = 8 Hz), 7.28 (2H, d, *J* = 9 Hz), 7.16 (1H, s), 7.03 (3H, d, *J* = 5.2 Hz), 5.34 (2H, s), 5.21 (NH, s), 3.87 (6H, s), 3.48 (m, 2H), 3.34 (m, 2H), 1.78 (6H, s) ppm; ¹³C NMR (126 MHz, DMSO-*d*6, TMS) δ 196.2, 168.7, 163.8, 142.1, 131.0, 129.7, 127.0, 113.9, 68.2, 59.1, 55.8, 22.9, MS m/z: 516.

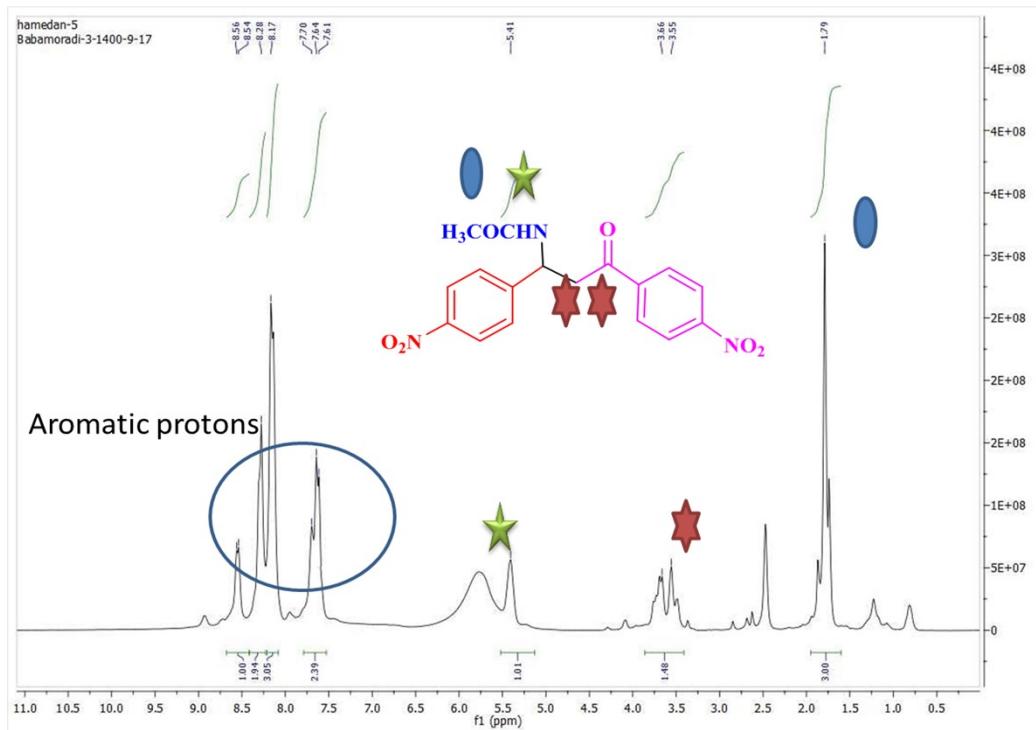


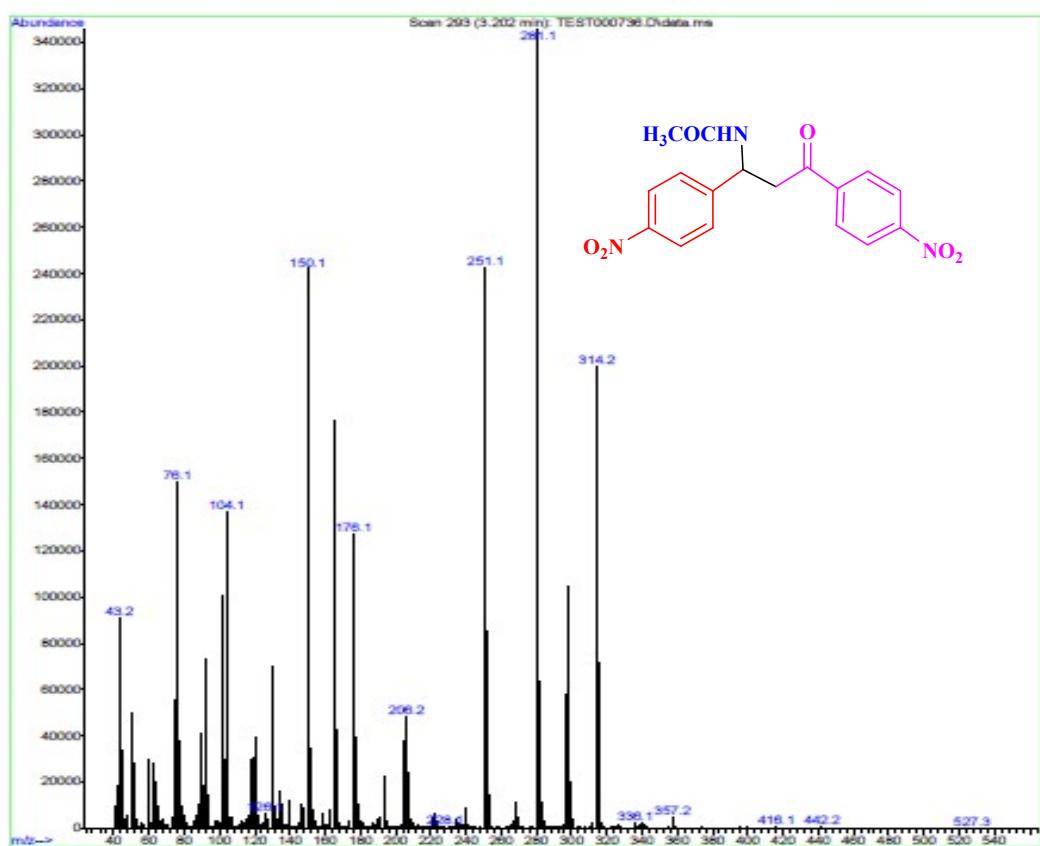
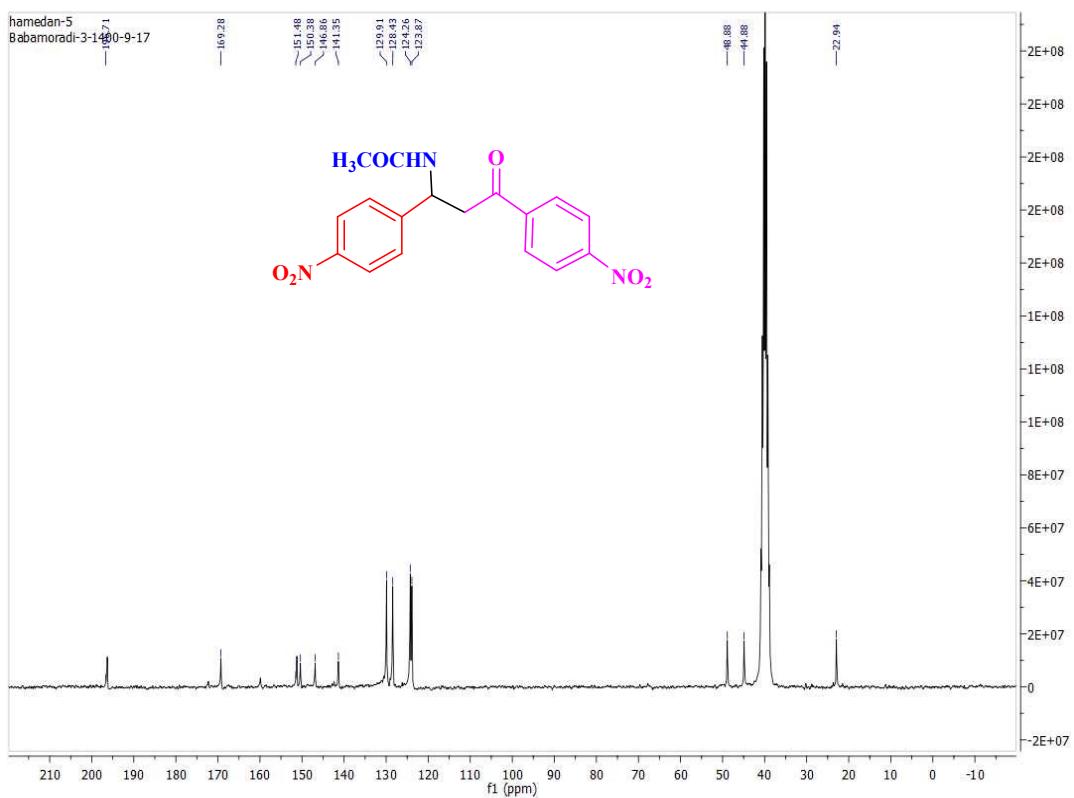


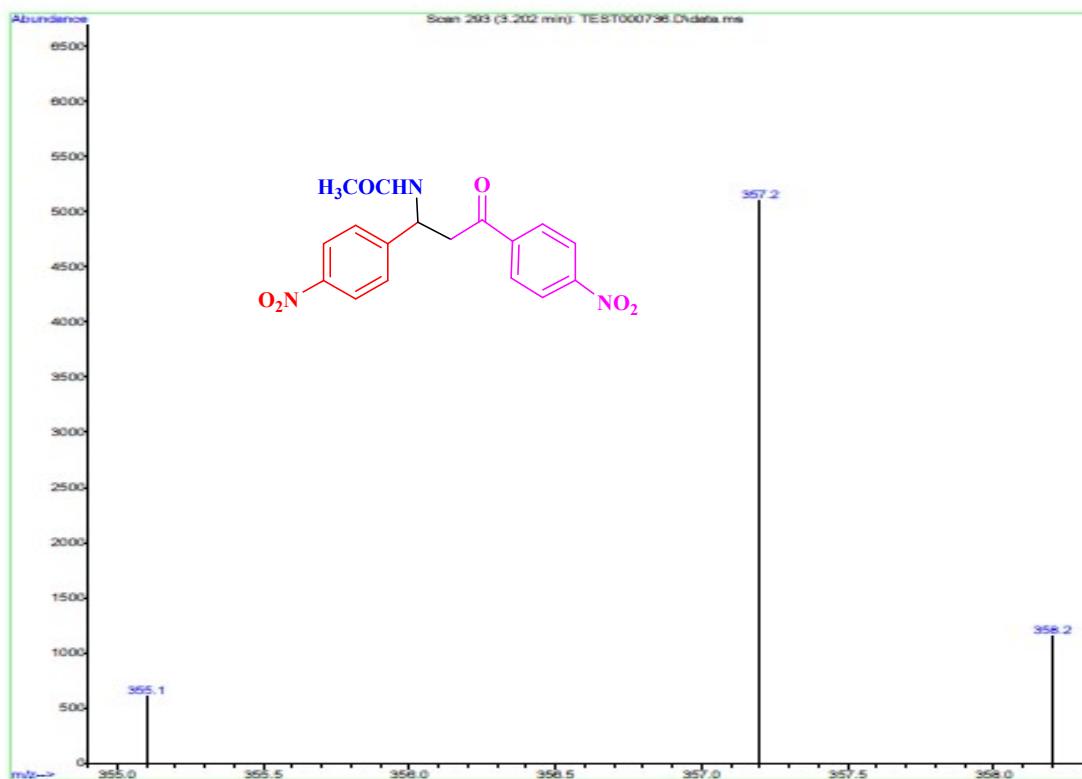
N-(3-Oxo-3-phenyl-1-(*p*-tolyl)propyl)acetamide: Cream solid, m.p.: 112-114 °C [1], 1H NMR (500 MHz, DMSO-*d*6, TMS) δ 7.95 (d, *J* = 7.2 Hz, 2H), 7.61 (dd, *J* = 11.0, 6.9 Hz, 2H), 7.53 (t, *J* = 7.0 Hz, 2H), 7.23 (d, *J* = 7.1 Hz, 2H), 7.11 (d, *J* = 7.1 Hz, 2H), 5.34 (d, *J* = 6.7 Hz, 1H), 3.52 (dd, *J* = 16.7, 7.6 Hz, 1H), 3.49 (dd, *J* = 16.7, 4.8 Hz, 1H), 2.26 (s, 3H), 1.79 (s, 3H) ppm; ¹³C NMR (126 MHz, DMSO-*d*6, TMS) 198.2, 169.1, 144.7, 140.2, 136.8, 135.9, 133.2, 130.0, 129.3, 129.2, 129.1, 128.8, 128.4, 127.0, 126.1, 121.4, 71.2, 48.9, 45.3, 23.3, 21.0.



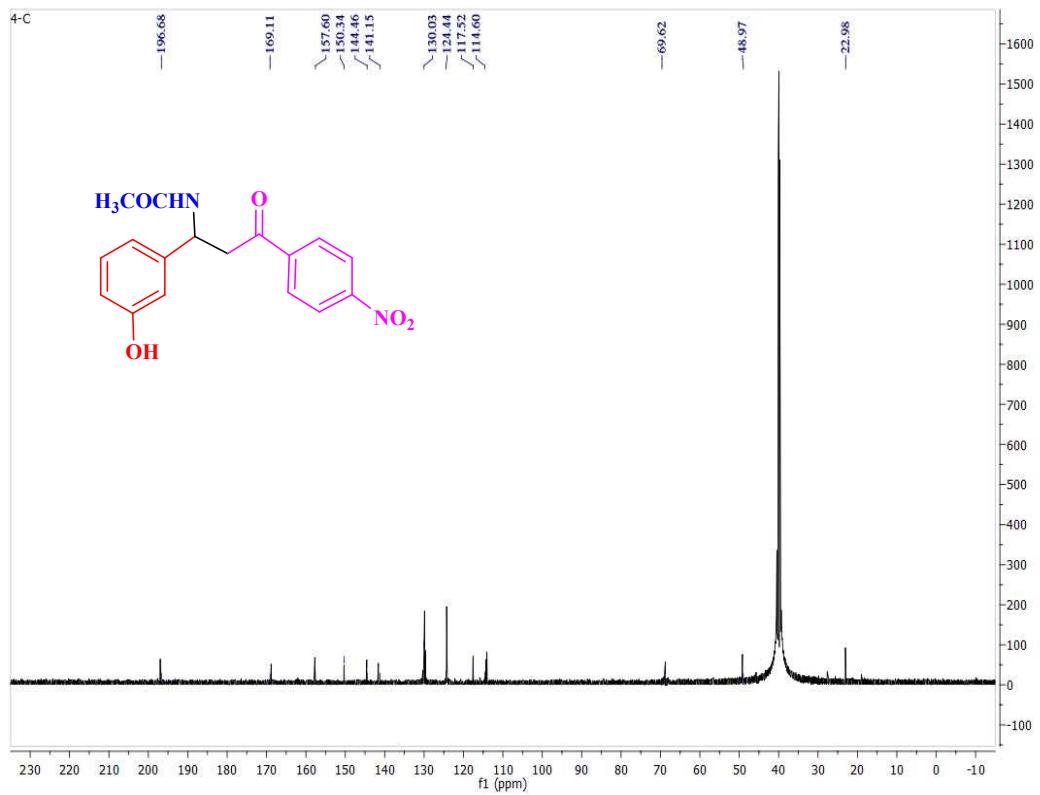
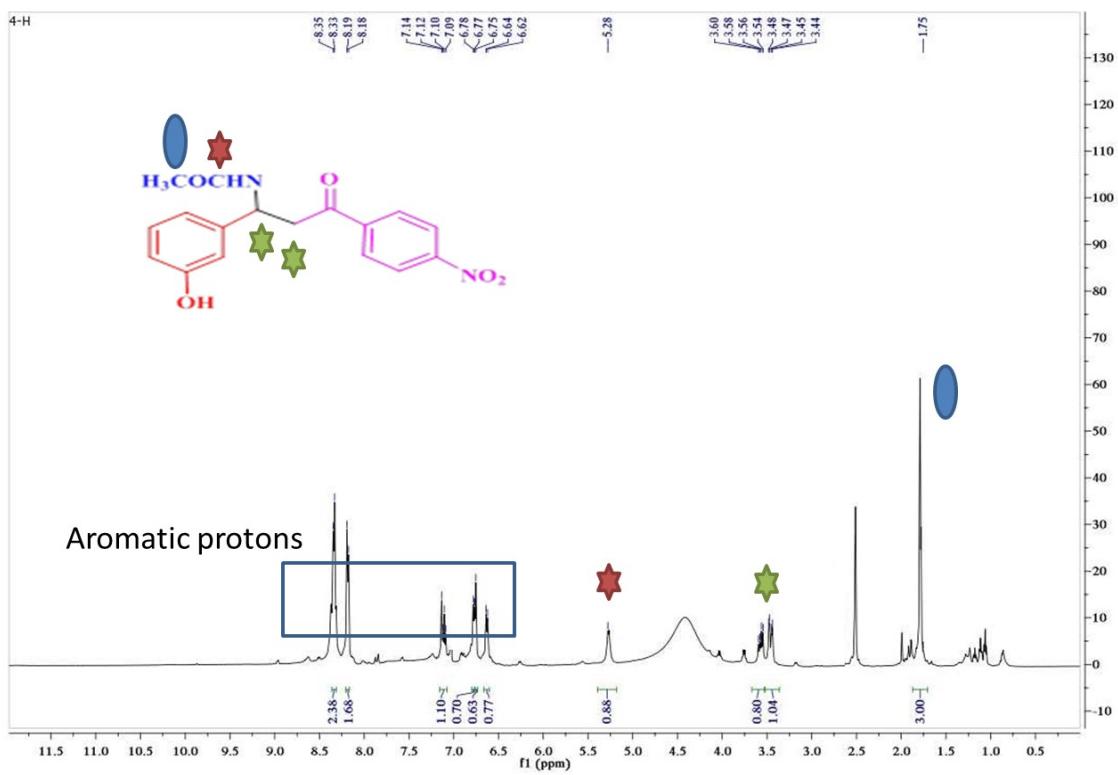
N-(1,3-Bis(4-nitrophenyl)-3-oxopropyl)acetamide: Cream solid, m.p.: 152–153 °C [2], ^1H NMR (250 MHz, $\text{DMSO}-d_6$, TMS) δ 8.55 (d, $J = 5.7$ Hz, 1H), 8.34–7.87 (m, 3H), 7.87–7.32 (m, 2H), 5.41 (s, 1H), 3.68 (d, $J = 7.5$ Hz, 1H), 3.52 (d, $J = 8.3$ Hz, 1H), 1.79 (s, 3H) ppm; ^{13}C NMR (63 MHz, DMSO) δ 196.3, 169.2, 152.9, 151.2, 150.8, 150.3, 146.8, 141.3, 129.9, 128.4, 124.2, 123.8, 48.8, 44.8, 22.9, MS m/z: 357.

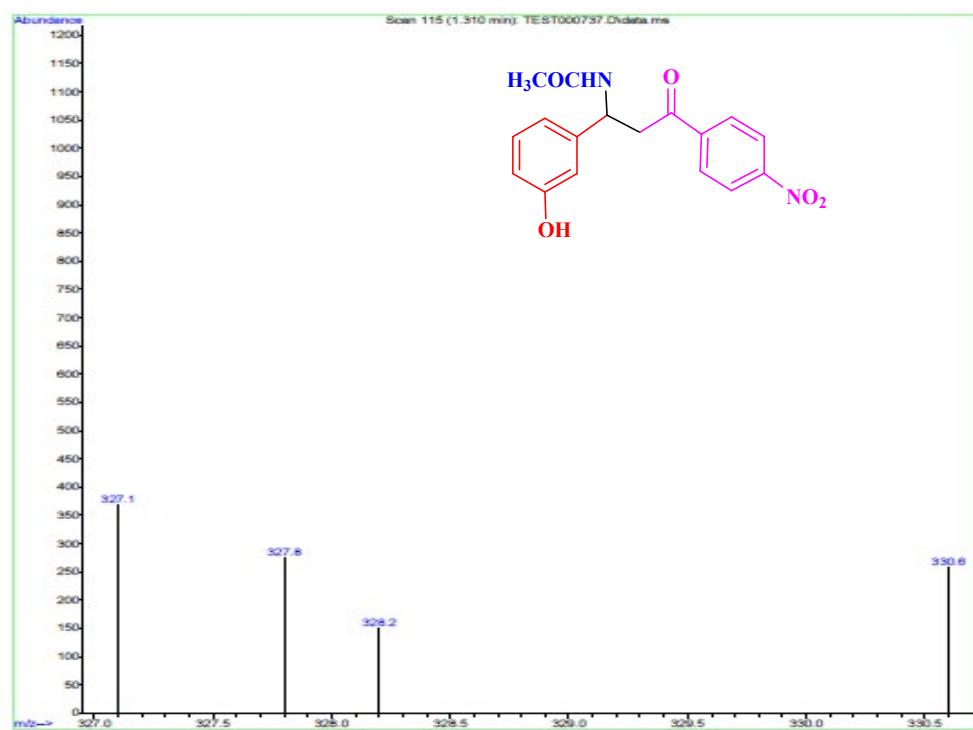
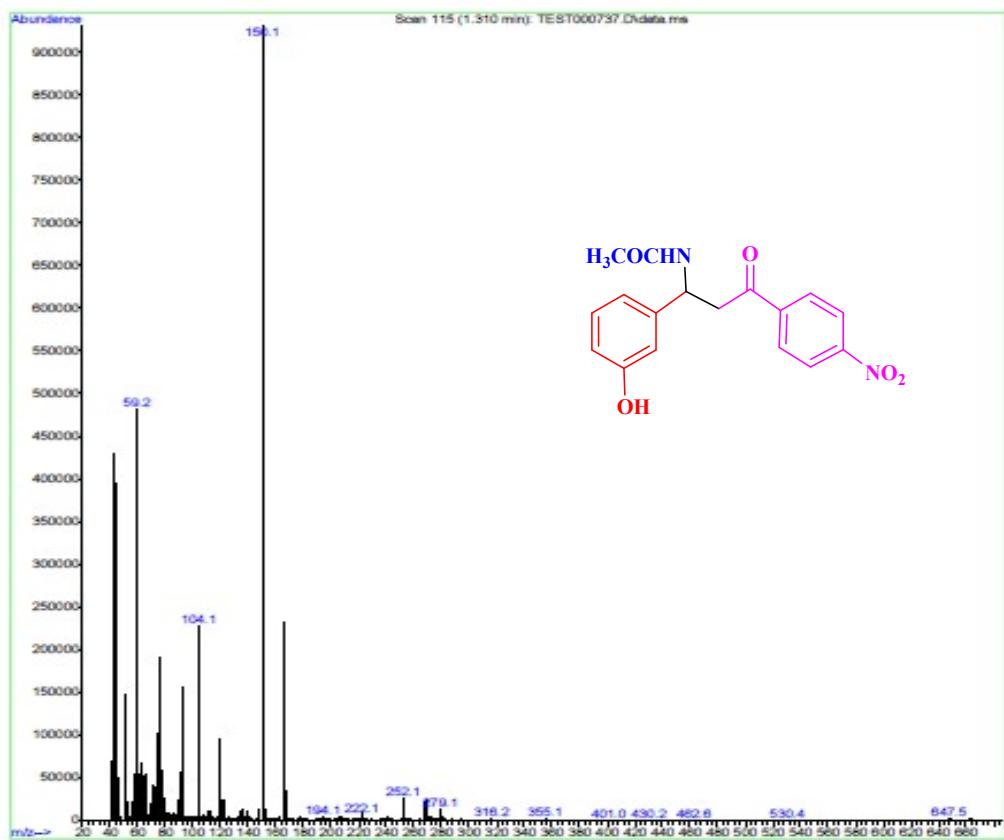




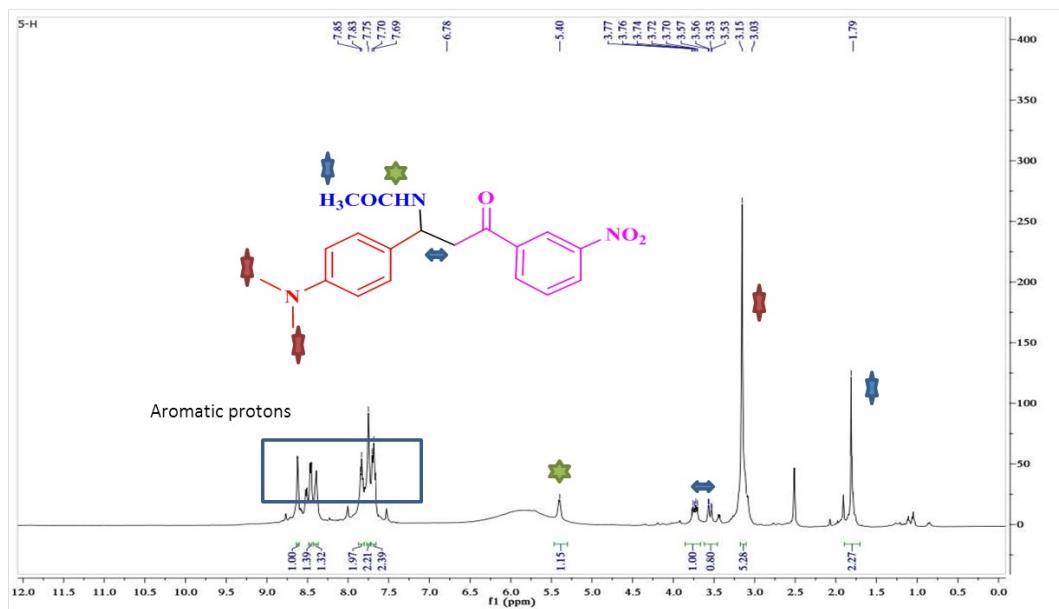


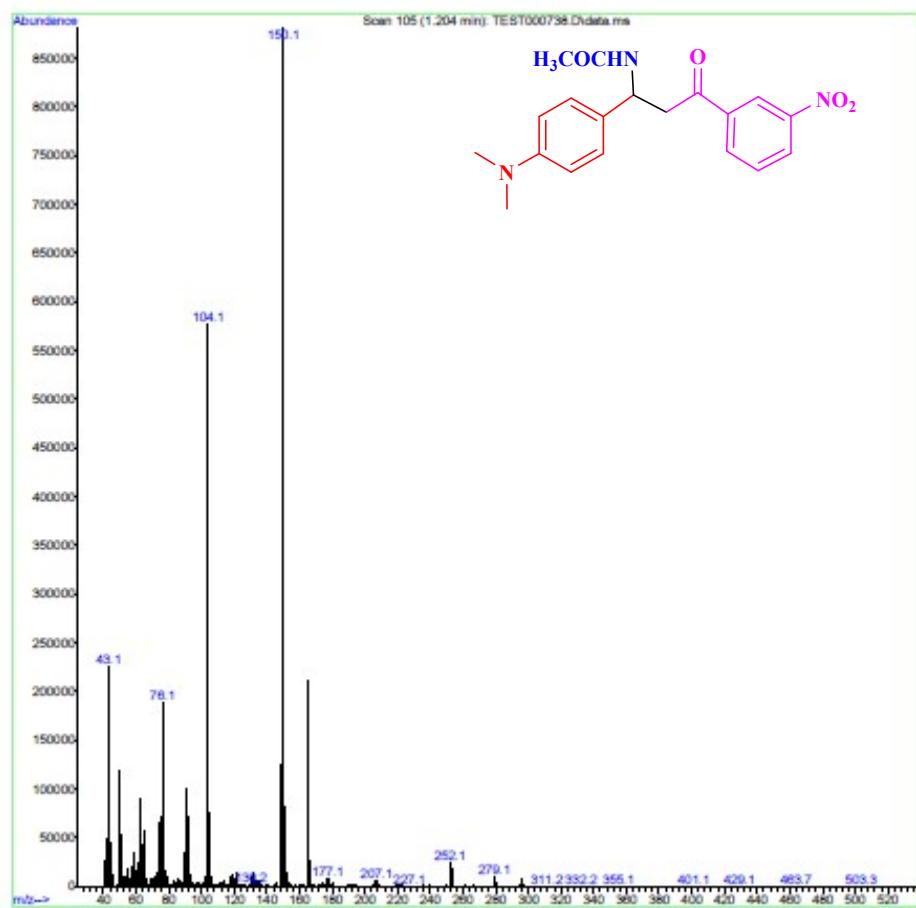
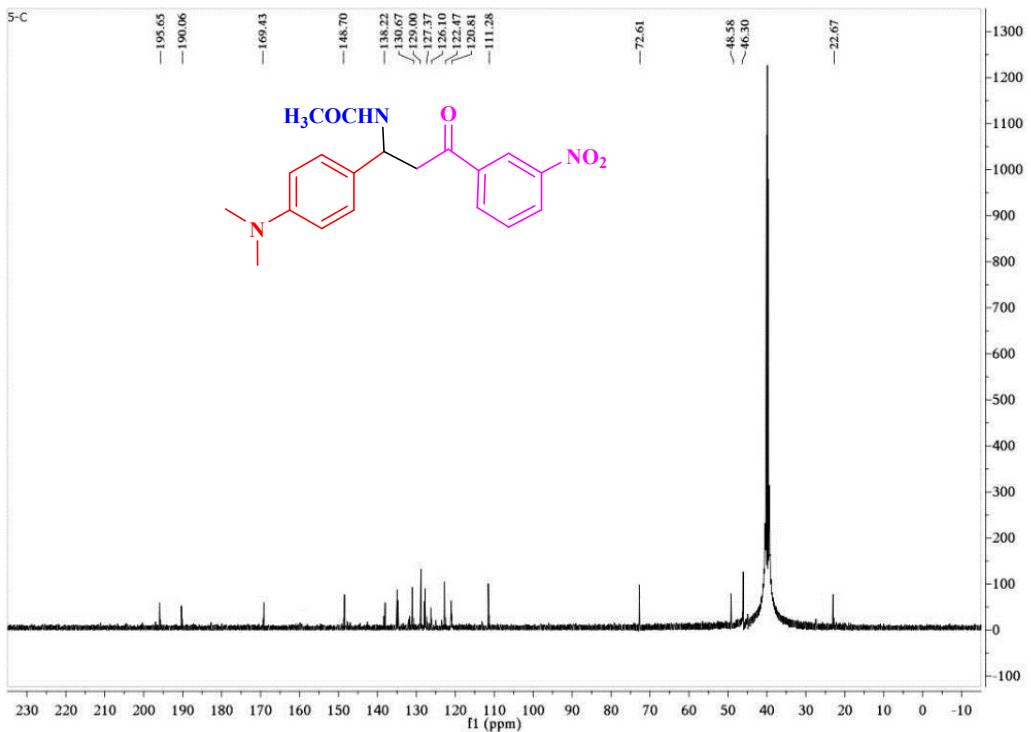
N-(1-(3-Hydroxyphenyl)-3-(4-nitrophenyl)-3-oxopropyl)acetamide: Cream solid, m.p.: 179-181 °C,
¹H NMR (500 MHz, DMSO-*d*6, TMS) δ ¹H NMR (500 MHz, dmso) δ 8.34 (d, *J* = 7.3 Hz, 2H), 8.18 (d, *J* = 8.3 Hz, 2H), 7.13 (dd, *J* = 15.3, 7.4 Hz, 1H), 6.77 (d, *J* = 7.3 Hz, 1H), 6.75 (s, 1H), 6.63 (d, *J* = 7.9 Hz, 1H), 5.28 (s, 1H), 3.57 (dd, *J* = 16.9, 8.6 Hz, 1H), 3.46 (dd, *J* = 14.4, 4.9 Hz, 1H), 1.75 (s, 3H) ppm; ¹³C NMR (126 MHz, DMSO-*d*6, TMS) δ 196.6, 169.1, 157.6, 150.3, 144.4, 141.1, 130.0, 124.4, 117.5, 114.6, 69.6, 48.9, 45.6, 22.9, MS m/z: 328.

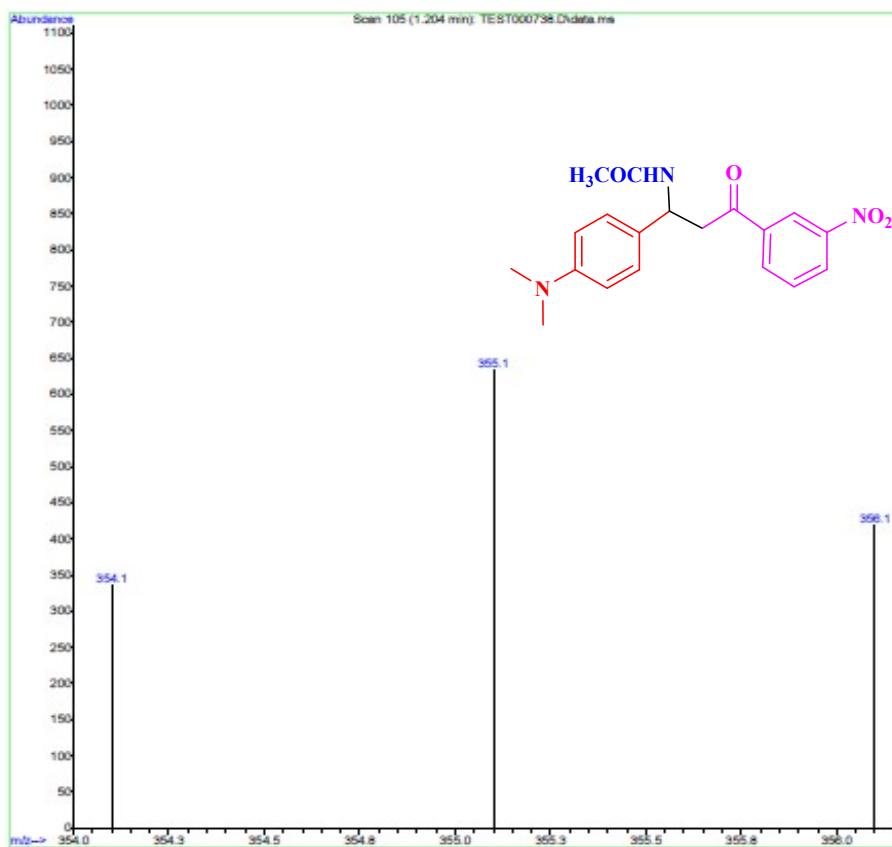




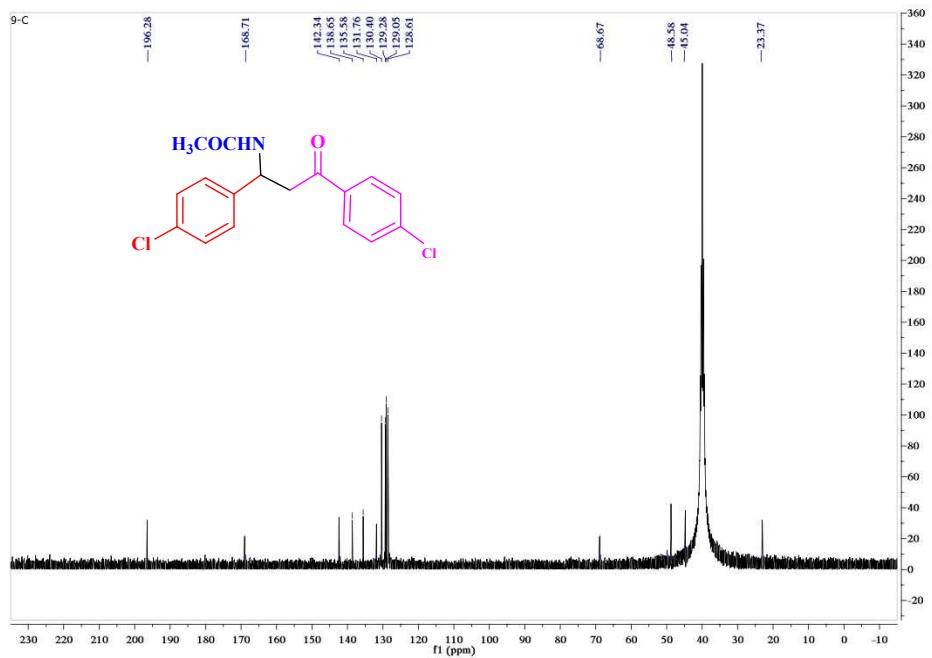
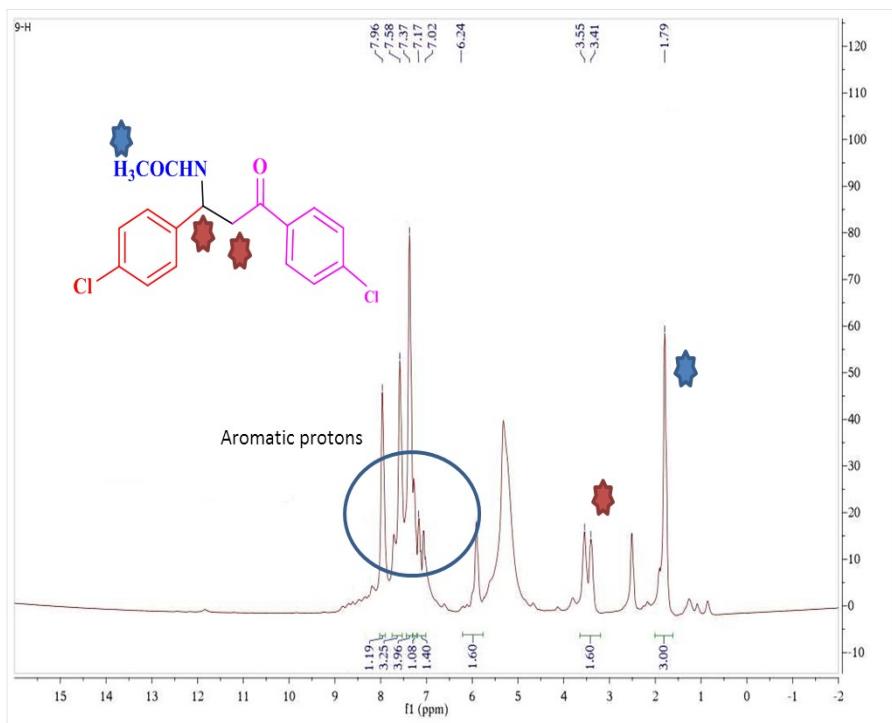
N-(1-(4-(Dimethylamino)phenyl)-3-(3-nitrophenyl)-3-oxopropyl)acetamide: Cream solid, m.p.: 190–192 °C, ^1H NMR (500 MHz, DMSO-*d*6, TMS) δ 8.62 (s, 1H), 8.46 (d, J = 7.4 Hz, 1H), 8.40 (d, J = 6.1 Hz, 1H), 7.84 (d, J = 7.0 Hz, 2H), 7.75 (s, 2H), 7.69 (d, J = 8.3 Hz, 2H), 5.40 (s, 1H), 3.87 – 3.77 (m, 1H), 3.55 (dd, J = 17.5, 4.3 Hz, 1H), 3.15 (s, 6H), 1.79 (s, 3H). ^{13}C NMR (126 MHz, DMSO-*d*6, TMS) δ 195.6, 190.0, 169.4, 148.7, 138.2, 130.6, 129.0, 127.3, 126.1, 122.4, 120.8, 111.2, 72.61, 48.5, 46.3, 22.6. MS m/z: 355.

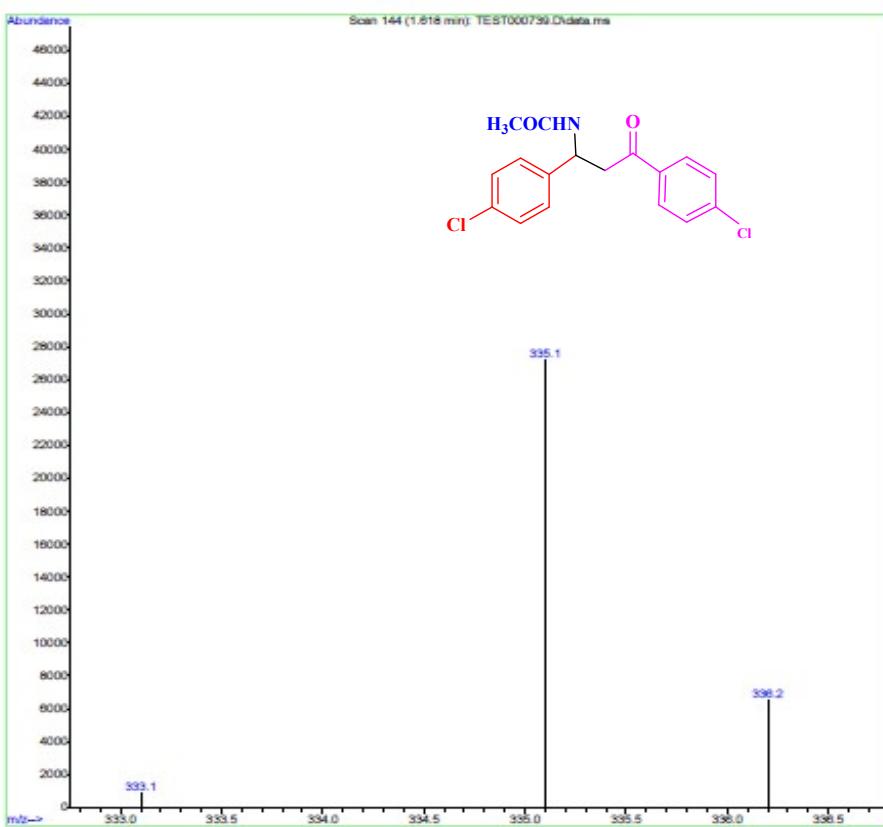
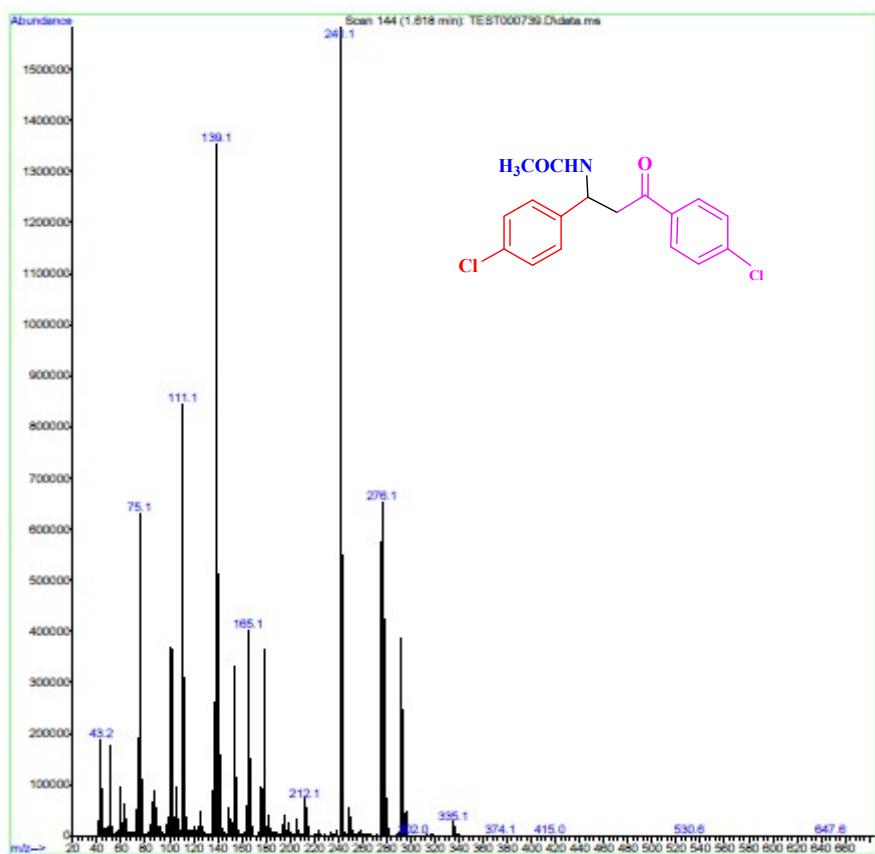




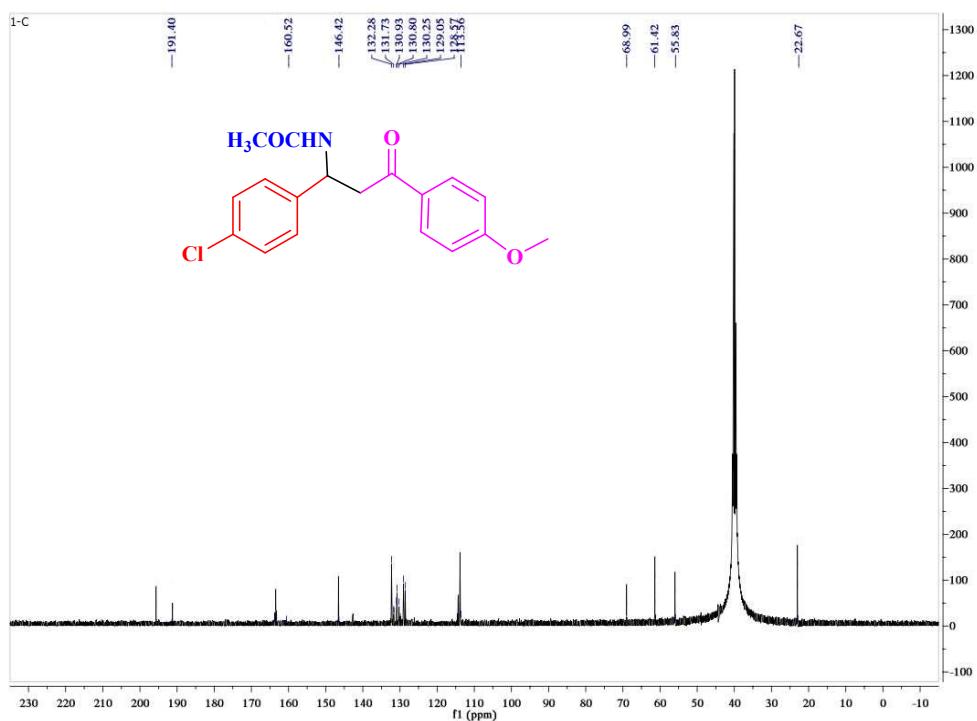
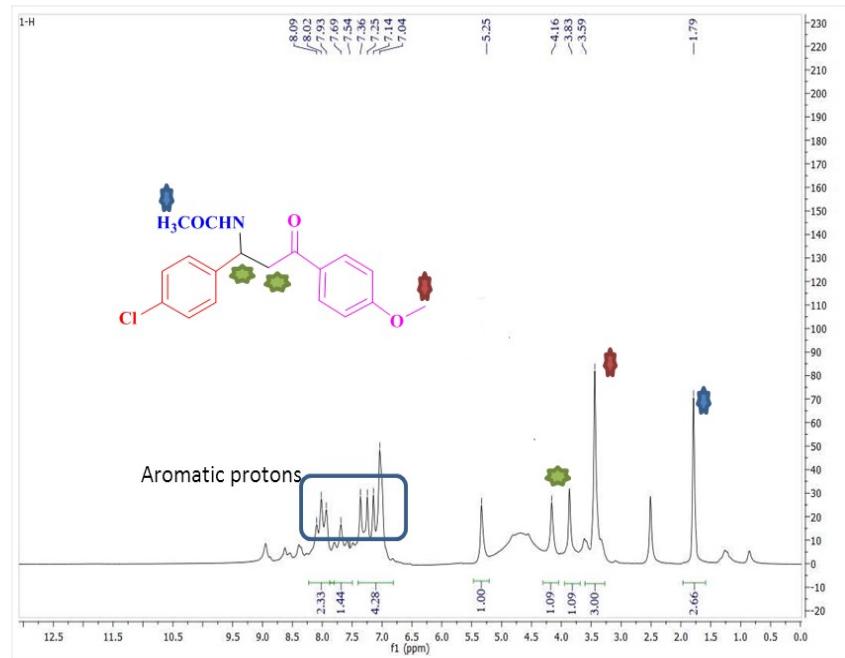


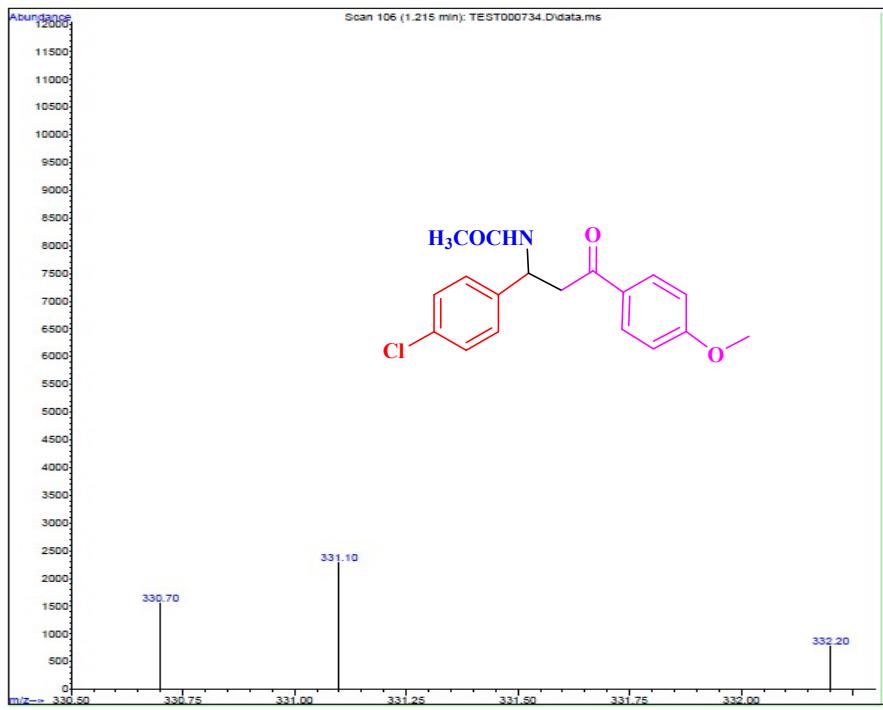
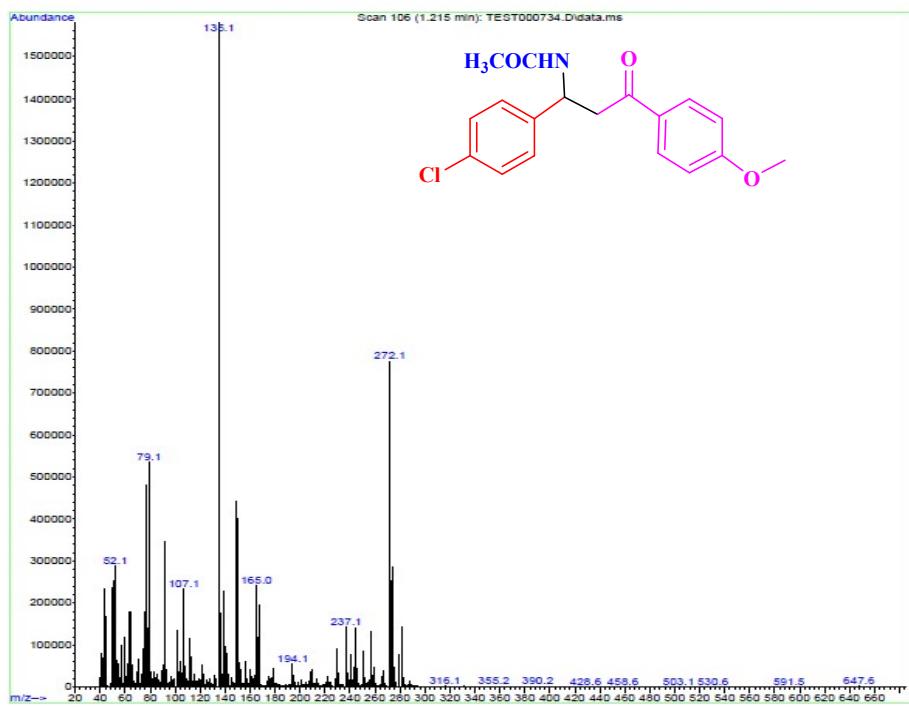
N-(1,3-Bis(4-chlorophenyl)-3-oxopropyl)acetamide: Cream solid, m.p.: 140-142 °C [3], ¹H NMR (500 MHz, DMSO-*d*6, TMS) δ 8.40 (s, 1H), 7.96 (s, 3H), 7.37 (s, 3H), 7.17 (s, 1H), 7.02 (m, 1H), 6.24 (s, 1H), 3.55 (s, 1H), 3.41(s, 1H), 1.79 (s, 3H), ¹³C NMR (126 MHz, DMSO-*d*6, TMS) δ 196.2, 168.7, 142.3, 138.6, 135.5, 131.7, 130.4, 129.2, 129.0, 128.6, 66.6, 48.5, 45.0, 23.3. MS m/z: 335.



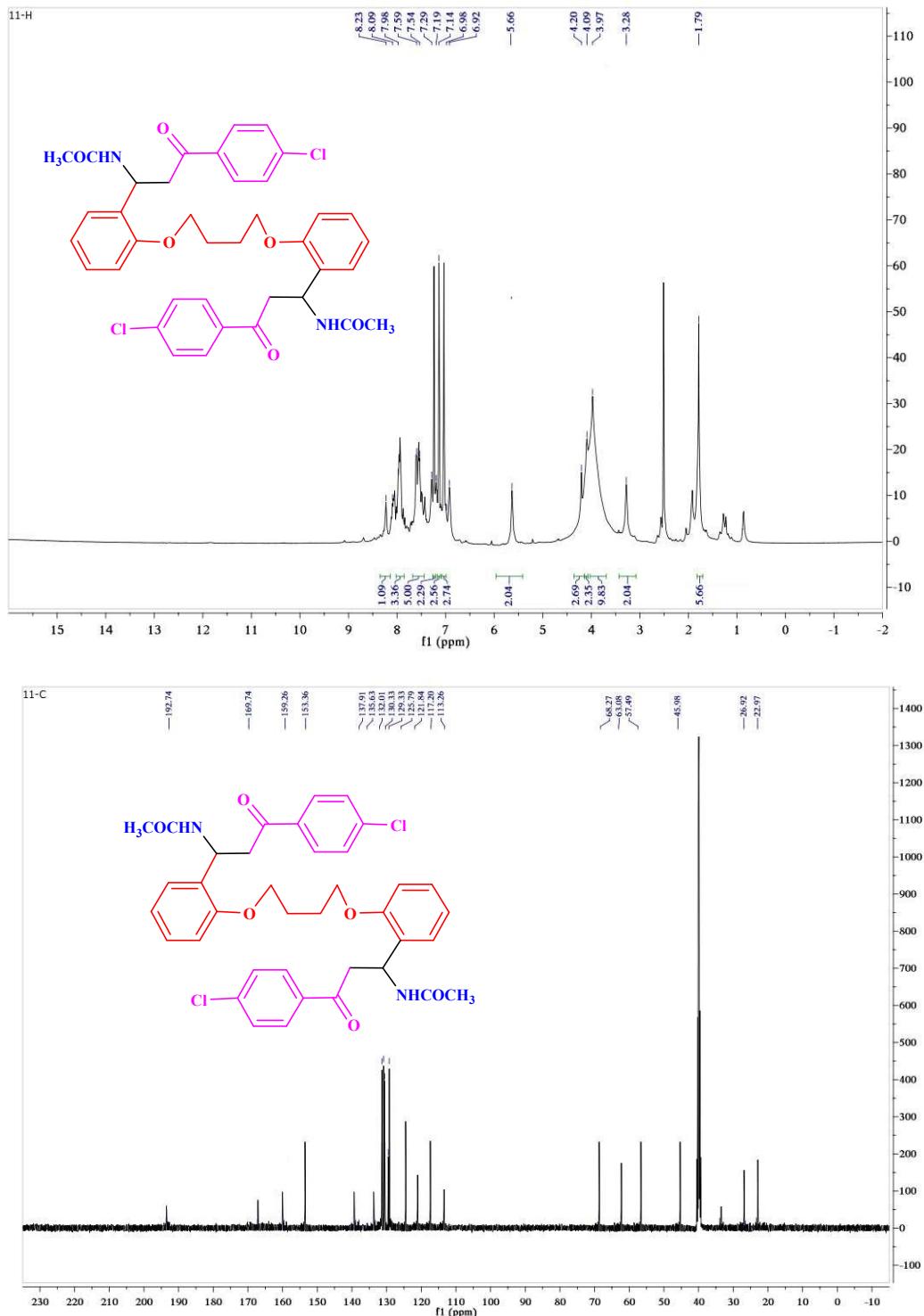


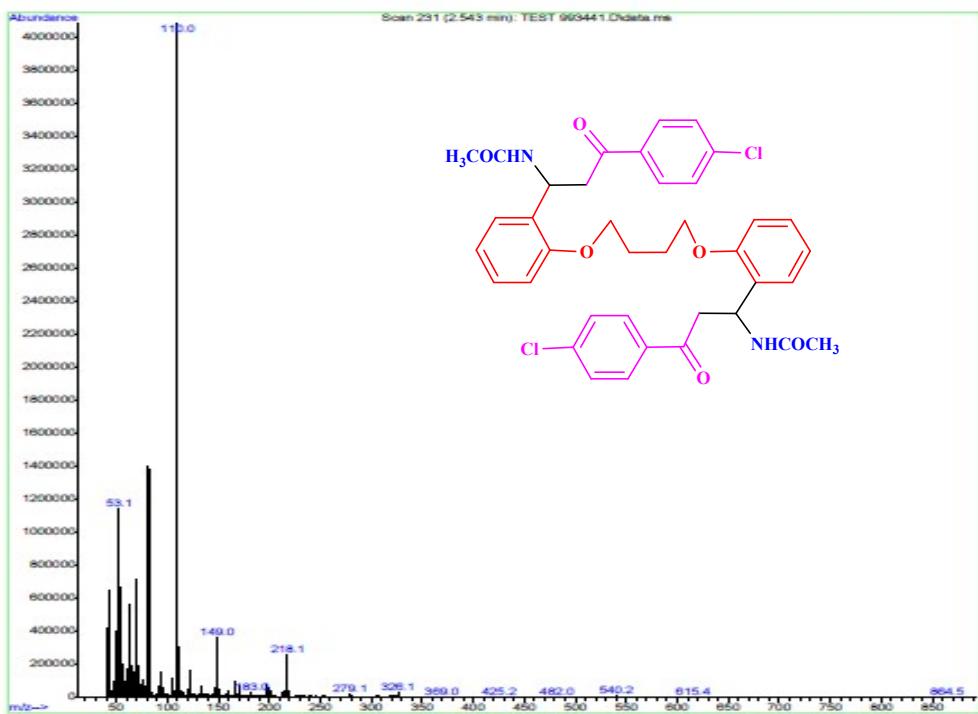
N-(1-(4-Chlorophenyl)-3-(4-methoxyphenyl)-3-oxopropyl)acetamide: Cream solid, m.p.: 59-61 °C [4], ^1H NMR (500 MHz, DMSO-*d*6, TMS) δ 8.09 (2H, dd, *J* = 12.9, 7.9 Hz), 7.93 (2H, dd, *J* = 13.9, 6.9 Hz), 7.36 (1H, s), 7.25 (2H, s), 7.14 (1H, s), 6.95-7.10 (m, 2H), 5.25 (1H, s), 4.16 (1H, s), 3.83 (1H, s), 3.59 (3H, s), 1.79 (3H, s). ^{13}C NMR (126 MHz, DMSO-*d*6, TMS) δ 191.4, 163.2, 160.5, 146.4, 132.2, 131.6, 130.8, 130.2, 129.9, 129.0, 128.5, 113.5, 68.9, 61.4, 55.8, 22.6, MS *m/z*: 331.



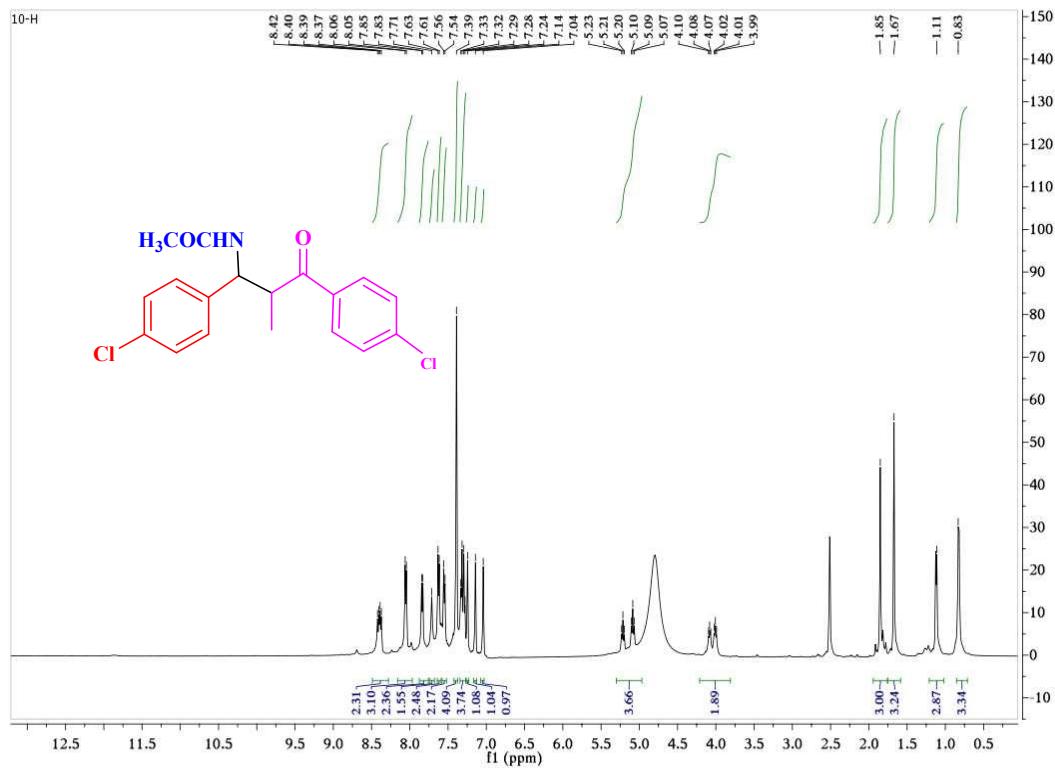


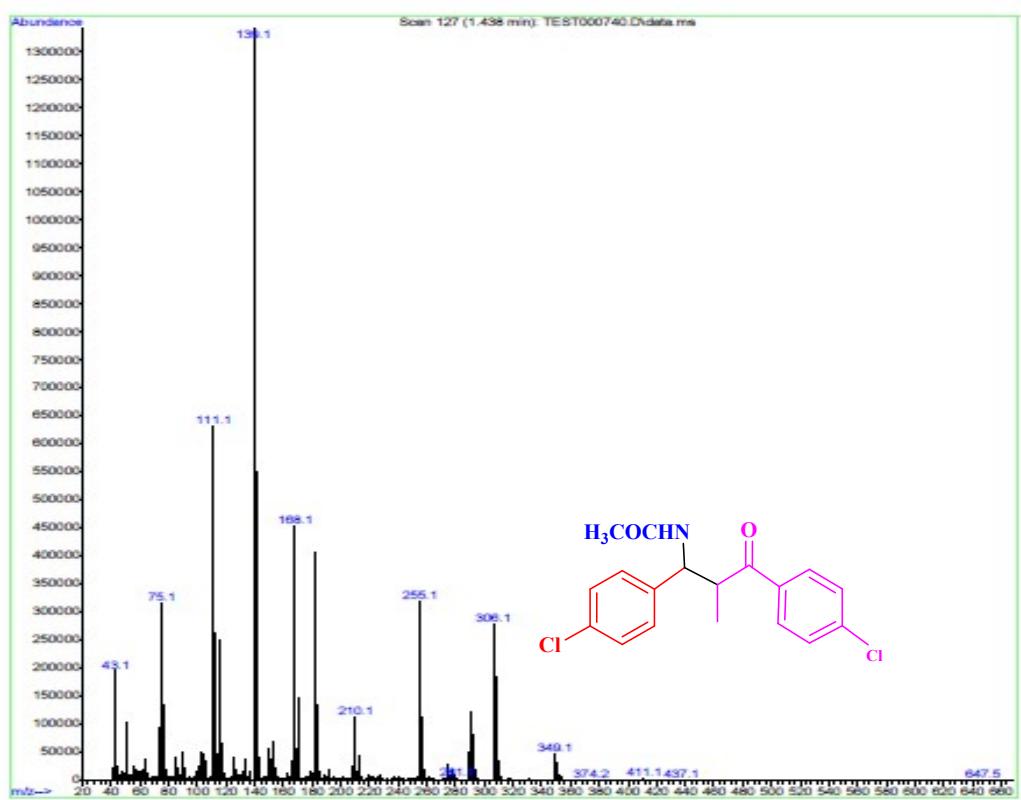
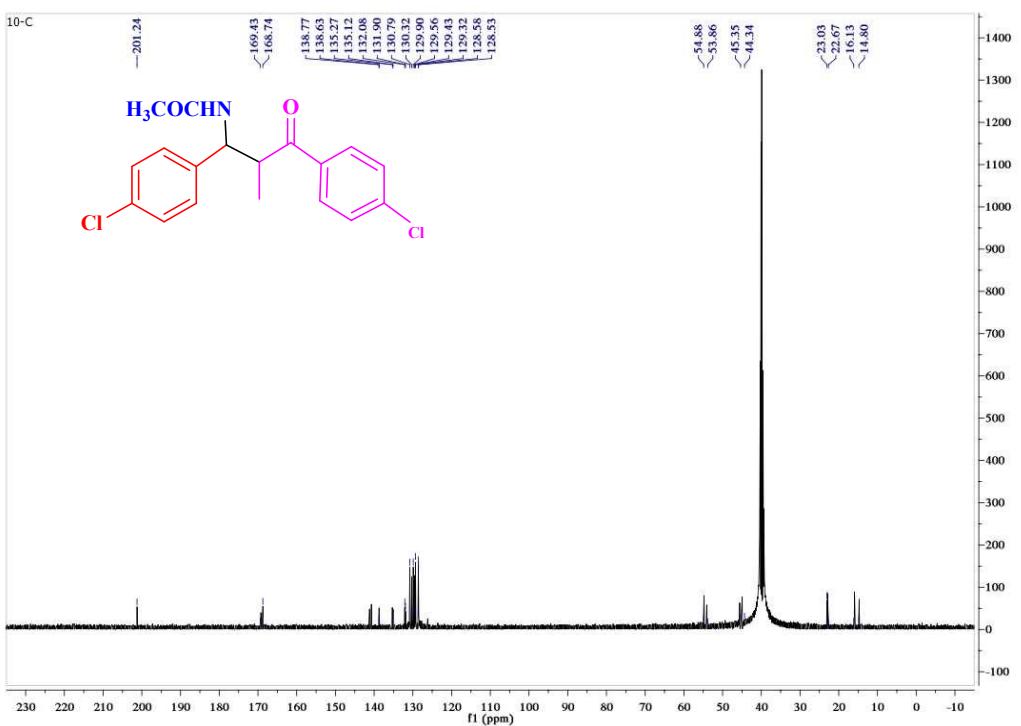
N,N'-(*((Butane-1,4-diylbis(oxo))bis(2,1-phenylene))bis(3-(4-chlorophenyl)-3-oxopropane-1,1-diyl*)diacetamide: Cream solid, m.p.:265-267 °C, ^1H NMR (500 MHz, DMSO-*d*6, TMS) δ 8.23 (s, 1H), 7.98 (s, 3H), 7.56 (d, J = 14.4 Hz, 3H), 7.22 (d, J = 13.9 Hz, 2H), 7.14 (s, 2H), 7.02 (d, J = 18.4 Hz, 3H), 5.66 (s, 2H), 4.20 (s, 2H), 4.09 (s, 2H), 3.97 (s, 9H), 3.28 (s, 2H), 1.79 (s, 6H). ^{13}C NMR (126 MHz, DMSO-*d*6, TMS) δ 192.7, 169.7, 159.2, 153.3, 132.0, 130.3, 129.3, 125.7, 121.8, 117.2, 113.3, 68.2, 63.0, 57.4, 45.9, 26.9, 22.9, MS m/z: 720.

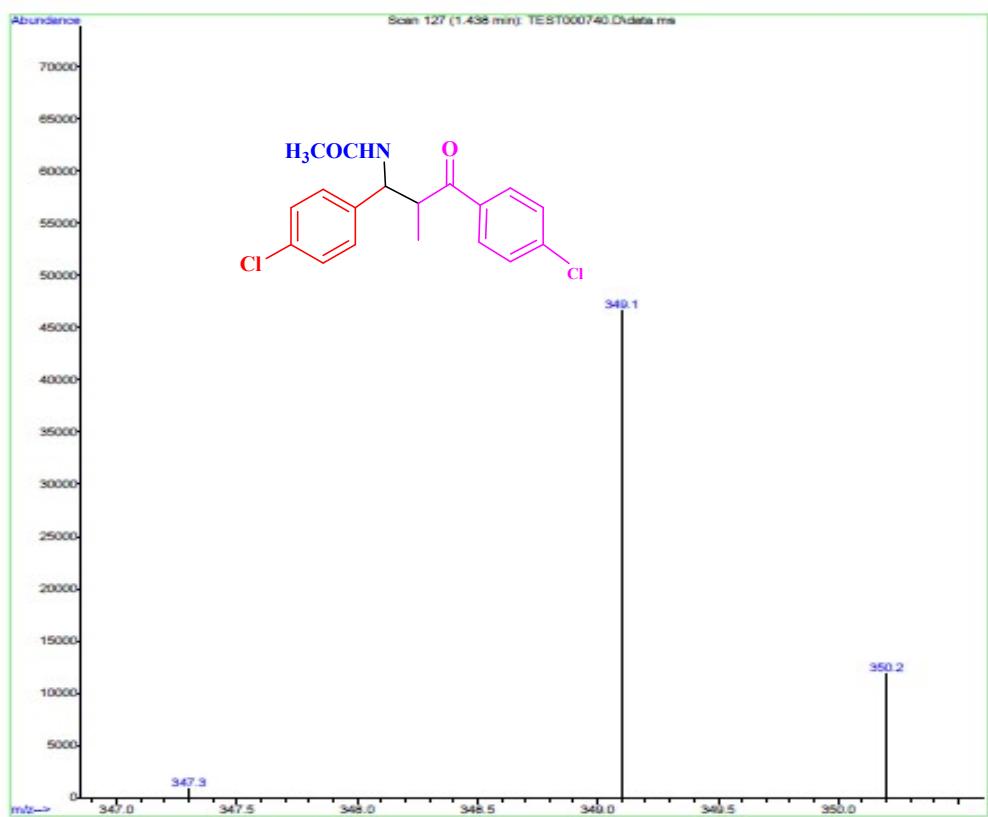




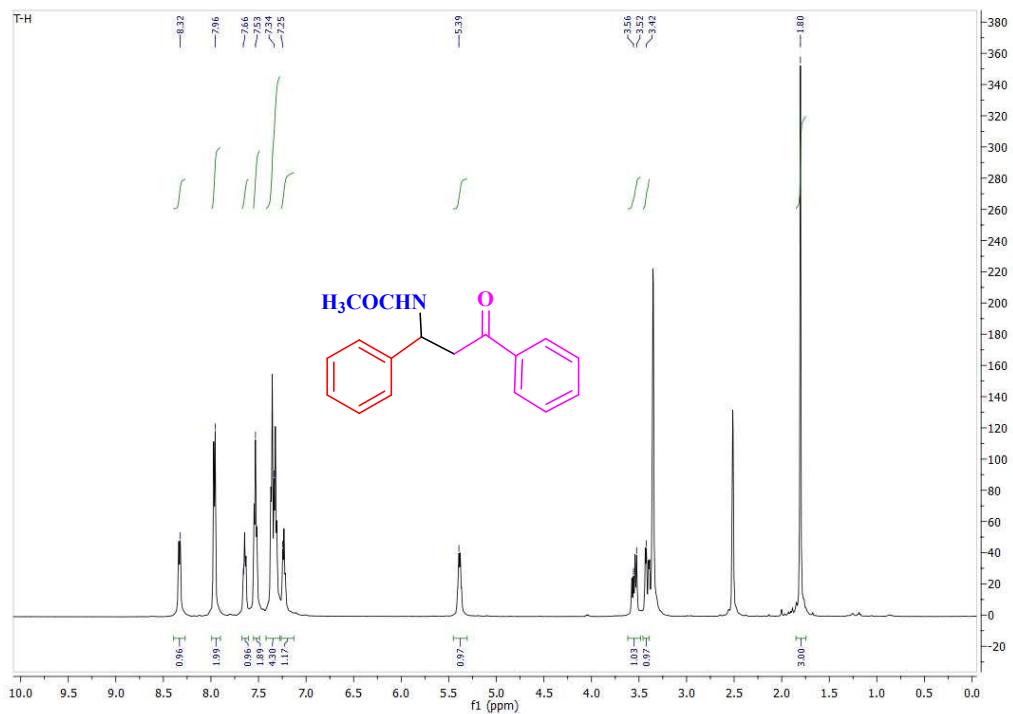
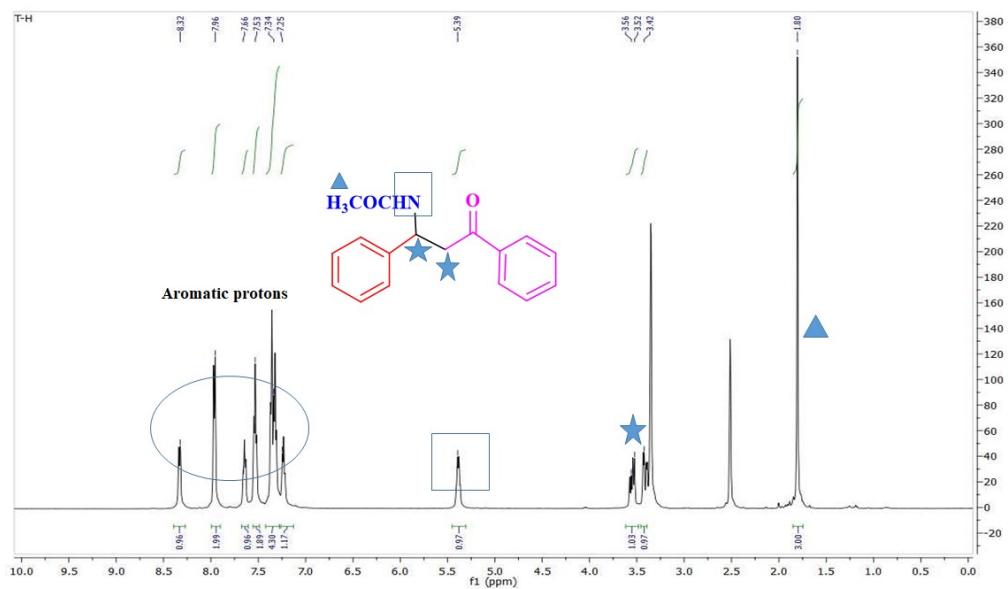
N-(1,3-Bis(4-chlorophenyl)-2-methyl-3-oxopropyl)acetamide: White solid (1:1 diastereomeric mixture); mp: 267–268; ¹H NMR (500 MHz, DMSO-*d*6, TMS) δ 8.40 (dd, *J* = 16.0, 8.9 Hz, 2H), 8.05 (d, *J* = 7.9 Hz, 3H), 7.84 (d, *J* = 7.9 Hz, 2H), 7.71 (s, 1H), 7.62 (d, *J* = 7.9 Hz, 2H), 7.55 (d, *J* = 7.8 Hz, 2H), 7.39 (s, 4H), 7.31 (dd, *J* = 19.4, 7.9 Hz, 3H), 7.24 (s, 1H), 7.14 (s, 1H), 7.04 (s, 1H), 5.15 (dt, *J* = 64.4, 8.8 Hz, 2H), 4.21 – 3.81 (m, 2H), 1.85 (s, 3H), 1.67 (s, 3H), 1.11 (s, 3H), 0.83 (s, 3H). ¹³C NMR (126 MHz, DMSO-*d*6, TMS) δ 201.2, 169.4, 168.7, 138.7, 135.1, 132.3 – 132.1, 131.9, 130.7, 130.3, 129.9, 129.6, 128.8, 128.5, 54.8, 53.8, 45.3, 44.3, 23.0, 22.6, 16.1, 14.8, MS *m/z*: 349.

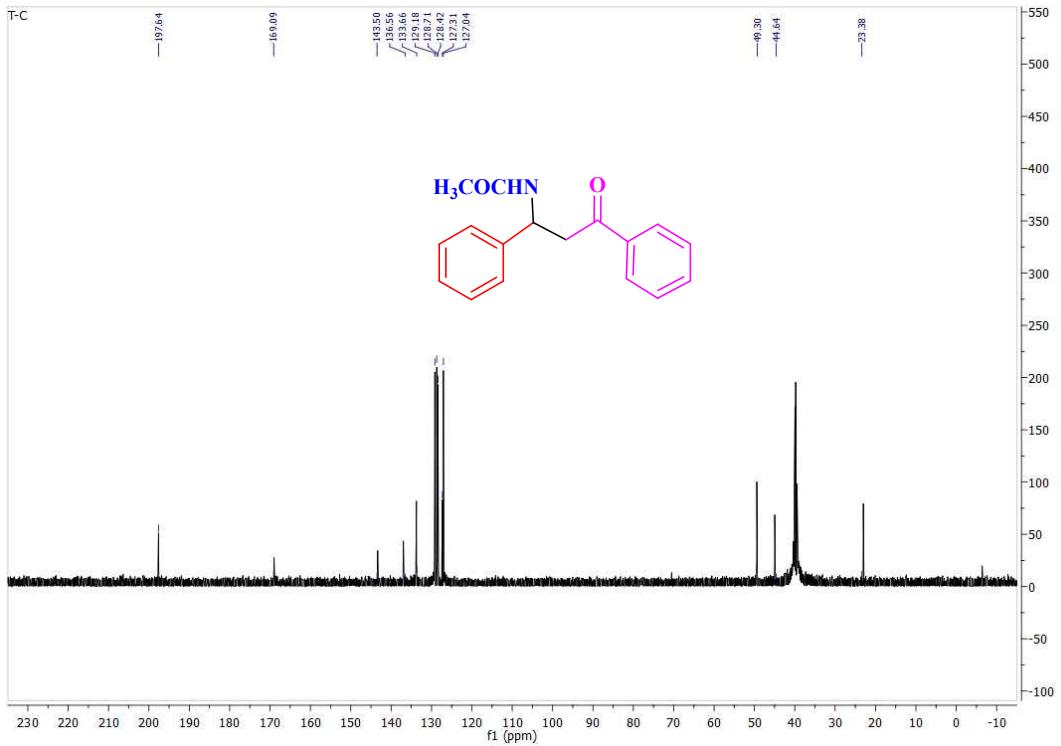




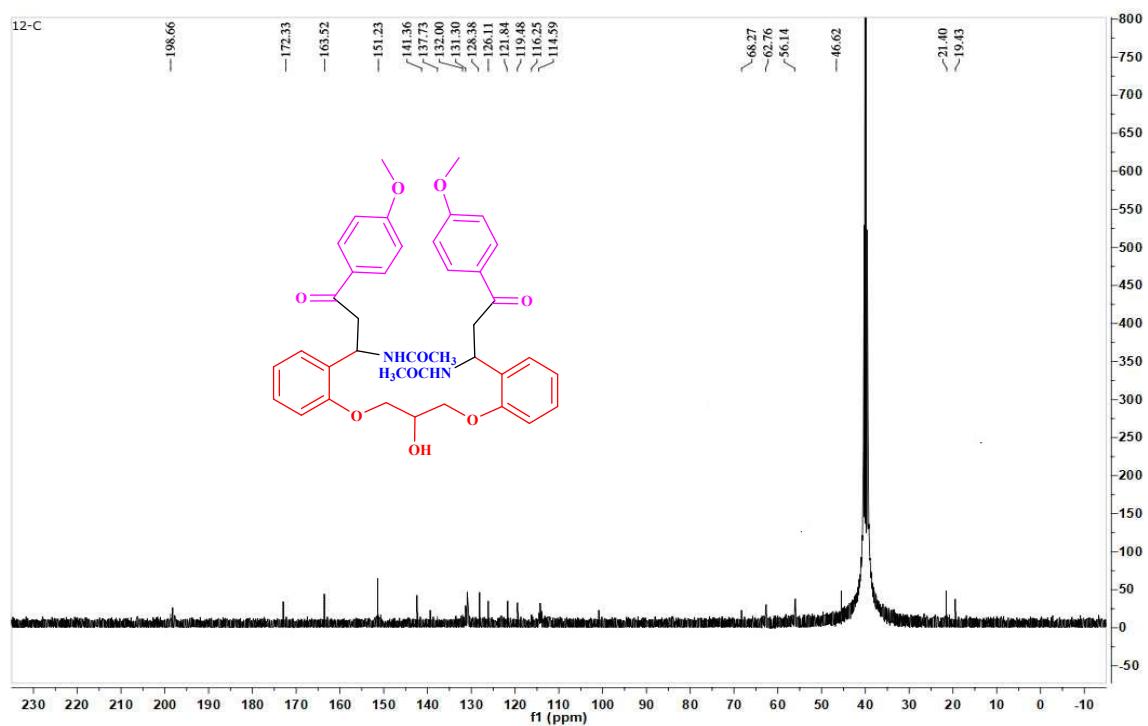
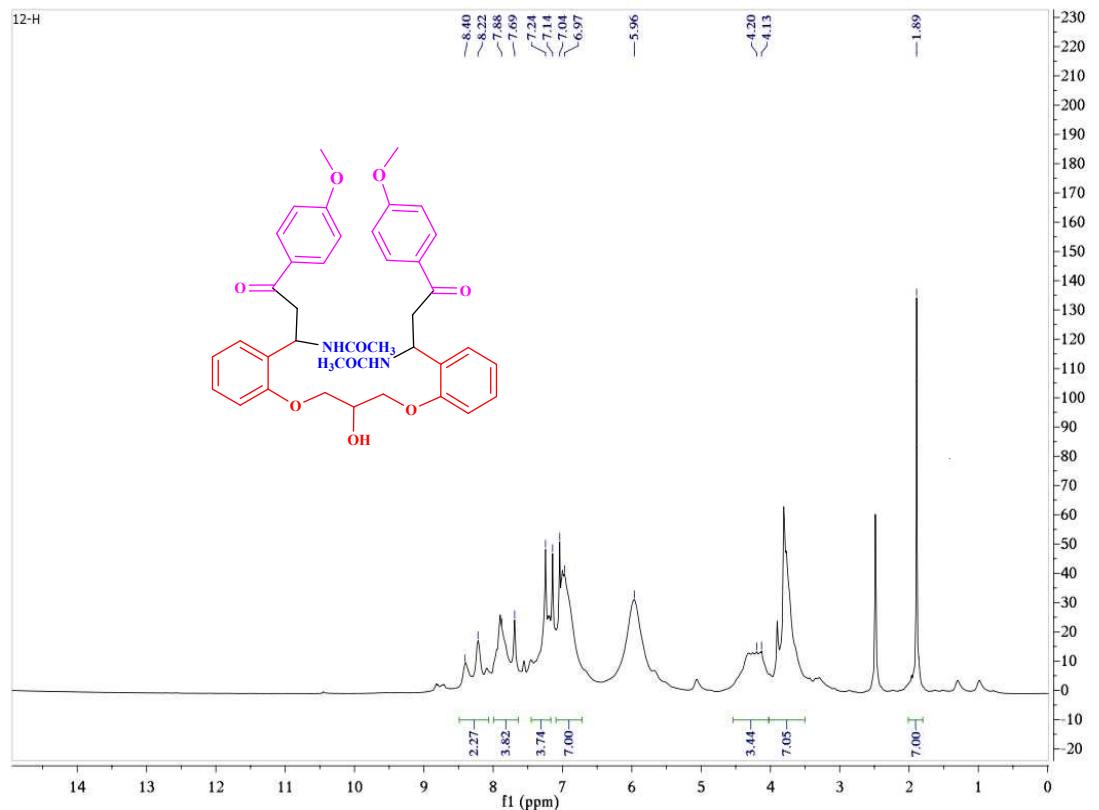


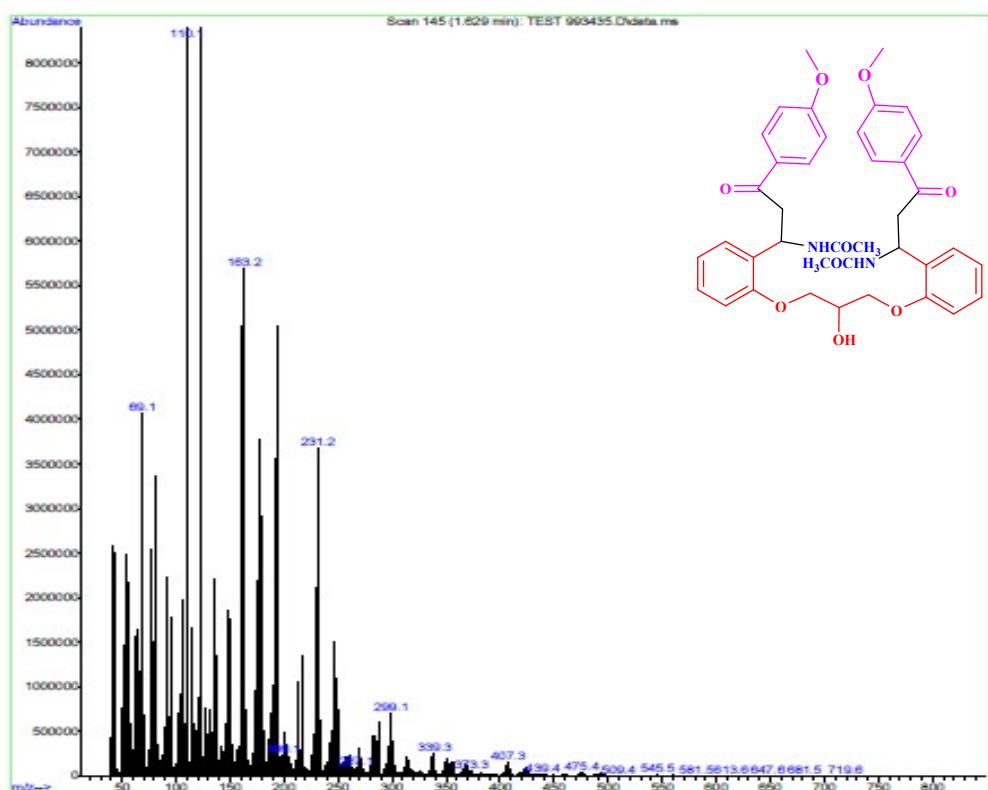
N-(3-Oxo-1,3-diphenylpropyl)acetamide: White solid, m.p. 103-105°C [1]; ¹H NMR (500 MHz, DMSO-*d*6, TMS) δ 8.32 (d, 1H, *J* = 8.0 Hz), 7.96 (d, 1H, *J* = 8.1 Hz) 7.66 (t, 2H, *J* = 7.8 Hz), 7.53 (t, 2H, *J* = 8.1 Hz), 7.34-7.25 (m, 4H), 7.23 (t, 1H, *J* = 7.4 Hz), 6.93 (br, 1H), 5.54 (m, 1H), 3.70 (dd, 1H, *J* = 16.8, 5.2 Hz), 3.41 (dd, 1H, *J* = 16.8, 6.0 Hz), 1.96 (s, 3H), ¹³C NMR (126 MHz, DMSO-*d*6, TMS) δ 197.5, 169.0, 143.5, 136.5, 133.6, 129.1, 128.7, 128.4, 127.3, 127.0, 49.3, 44.5, 23.3.





***N,N'*-(((2-Hydroxypropane-1,3-diyl)bis(oxy))bis(2,1-phenylene))bis(3-(4-methoxyphenyl)-3-oxopropane-1,1-diyl)diacetamide:** Cream solid, m.p.>300 °C, ^1H NMR (500 MHz, DMSO-d₆, TMS) δ : 8.40 (s, 2H), 8.22 (s, 1H), 7.88 (m, 2H), 7.69 (s, 1H), 7.24 (m, 2H), 7.14 (m, 2H), 7.04 (s, 1H), 6.97 (m, 6H), 4.13-4.48 (m, 3H), 3.90 (s, 1H), 3.80 (broad, 6H), 1.80 (s, 7H) ppm; 198.6, 172.3, 163.5, 151.2, 141.3, 137.7, 132.0, 131.3, 128.3, 126.1, 121.8, 119.4, 116.2, 114.5, 68.2, 62.7, 56.1, 46.6, 21.4, 19.4, MS m/z: 682.





References:

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