Supporting Information

for

Catalyst-free and solvent-free Michael addition of 1,3-dicarbonyl compounds to nitroalkenes by a grinding method

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General procedures and analytical data

Materials and general information

All reagents were obtained from commercial suppliers and were used without further purification unless otherwise noted. 1,3-cyclopentanedione was purified by flash column chromatography (silicagel, 300–400 mesh) prior to use. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker 400 MHz instrument by using $CDCl_3$ or DMSO-d_6 as a solvent with tetramethylsilane (TMS) as an internal standard at room temperature. Chemical shifts are given in δ relative to TMS; the coupling constants *J* are given in Hz. HPLC was carried out by using a Shimadzu organizer consisting of a LC-2010A HT Integrator and a UV–vis detector. C18 column (150 mm × 46 mm) was used in the HPLC experiments with MeOH/H_2O 30:70, 0.5 mL/min and λ_{max} = 265 nm. HRMS were performed on Bruker Daltonics Bio TOF mass spectrometer. Column chromatography was performed on EM silica gel (200–300 mesh).

General procedure for the preparation of nitroalkenes

Aldehyde (0.05 mol), $MeNO_2$ (0.05 mol), and MeOH (10–20 mL) were added to a round-bottom flask and then stirred vigorously. NaOH solution (10.5 M, 10 mL) was added dropwise in an ice bath; a large amount of white or yellow solid precipitated, and stirring was continued for 15 min. Distilled H_2O was added until the solution became clear, then the solution was added dropwise to concentrated HCl (30 mL), and a yellow solid precipitated. The yellow solid was filtered and washed with H_2O, then evaporated in a vacuum drying oven. After recrystallization (EtOH), yellow needle-like crystals were obtained.

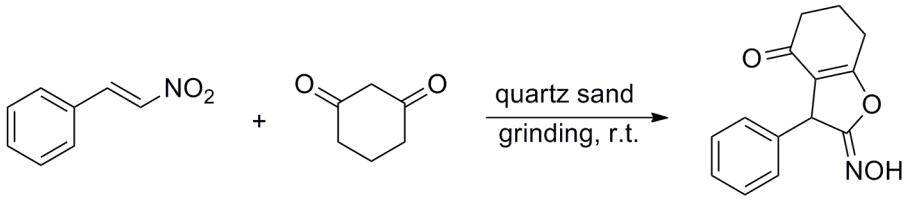
General procedure for the model reaction (optimizing conditions)

A quantity of β-nitrostyrene, 1,3-cyclopentanedione and grinding aid were added in a mortar, and after 10 min of continuous grinding, the mixture was left standing at rt for 10 min. Then, the reaction was quenched with MeOH. The reaction mixture was transferred to a centrifuge tube, shaken and centrifuged fully, and the yield was extracted by HPLC using the supernatant.

General procedure for the catalyst-free and solvent-free Michael addition

Nitroalkene (0.36 mmol), 1,3-dicarbonyl compound (0.30 mmol) and quartz sand (2.25 g) were added in a mortar, mixed thoroughly and ground well (intermittently) at rt, and then even placed overnight until reacted completely. The mixture was purified directly by flash column chromatography to give the product without any pretreatment. For the simpler purification procedure, the reaction mixture was transferred to a sand core funnel in which a thin layer of column-layer chromatographic silica gel was laid down in advance, and then the elution process was performed. First, redundant β-nitrostyrene was directly eluted with dichloromethane, and then the pure product was obtained by using dichloromethane/methanol 5:1 v/v as eluent.

No Michael product was detected in the reaction of β-nitrostyrene and cyclohexane-1,3-dione, and another product was only obtained by a tandem process (Scheme S1, 73% yield).

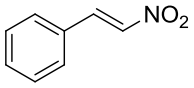
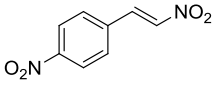
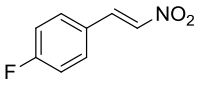
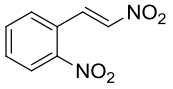
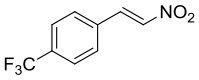
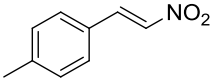
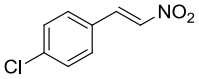
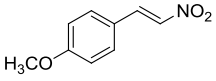
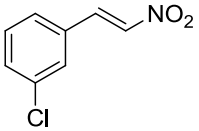
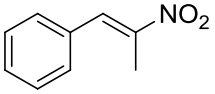
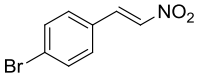
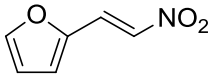
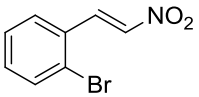


Scheme S1: The tandem reaction of β-nitrostyrene and cyclohexane-1,3-dione

Investigation of the reactivity of various nitroalkenes

The reactivity of various nitroalkenes with 1,3-cyclopentanedione was studied (Table S1). Compared with β-nitrostyrene (Table S1, entry 1), its derivatives showed lower reactivity (Table S1, entries 2–4,6–12) with the exception of 1-chloro-3-(2-nitrovinyl)benzene (Table S1, entry 5), and especially low in the cases of (2-nitroprop-1-en-1-yl)benzene (Table S1, entry 12) and 1-nitro-2-(2-nitrovinyl)benzene (Table S1, entry 9), where steric effects or electronic effects were probably the main reasons.

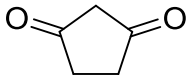
Table S1: The reactivity of different nitroalkenes with 1,3-cyclopentanedione.[a]

Entry	Acceptor	Conv.[%][b]	Entry	Acceptor	Conv.[%][b]
1		99	8		37
2		55	9		13
3		76	10		94
4		52	11		82
5		99	12		< 1
6		25	13		74
7		25			

[a]Conditions: acceptor (0.12 mmol), 1,3-cyclopentanedione (0.1 mmol), quartz sand (0.75 g), ground for 10 min and stood for 10 min. [b]Conv. referring to the percentage conversion of 1,3-cyclopentanedione as determined by HPLC.

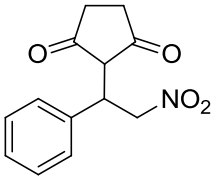
Analytical data of products

1,3-Cyclopentanedione (*material*)



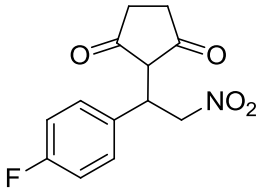
^{1}H NMR (400 MHz, DMSO-d_6) δ 12.07 (s, 1 H), 5.09 (s, 1 H), 2.38 (s, 4 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 105.46, 31.70.

2-(2-Nitro-1-phenylethyl)cyclopentane-1,3-dione (3a)



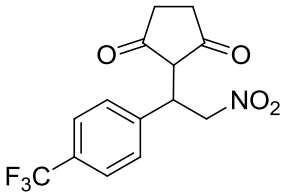
^{1}H NMR (400 MHz, DMSO-d_6) δ 12.31 (s, 1 H), 7.34–7.19 (m, 5 H), 5.23–5.12 (m, 2 H), 4.47 (t, J = 8.2 Hz, 1 H), 2.38 (s, 4 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 140.19, 128.99, 128.08, 127.42, 114.66, 77.05, 38.53, 30.61; HRMS–ESI (m/z): $[M + Na]^+$ calcd for $C_{13}H_{13}NO_4$, 270.0737; found, 270.0738.

2-(1-(4-Fluorophenyl)-2-nitroethyl)cyclopentane-1,3-dione (3b)



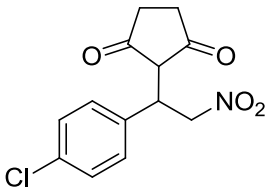
^{1}H NMR (400 MHz, DMSO-d_6) δ 7.38–7.34 (m, 2 H), 7.12 (t, J = 8.8 Hz, 2 H), 5.20–5.09 (m, 2 H), 4.46 (t, J = 8.2 Hz, 1 H), 2.38 (s, 4 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 162.83, 160.42, 136.34 (d, J = 3.1 Hz), 130.03 (d, J = 8.1 Hz), 115.83, 115.62, 114.45, 77.04, 37.80, 30.59; HRMS–ESI (m/z): $[M + H]^+$ calcd for $C_{13}H_{12}FNO_4$: 266.0823, found: 266.0834.

2-(2-Nitro-1-(4-(trifluoromethyl)phenyl)ethyl)cyclopentane-1,3-dione (3c)



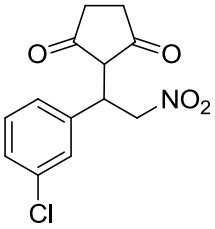
^{1}H NMR (400 MHz, DMSO-d_6) δ 7.67 (d, J = 8.2 Hz, 2 H), 7.56 (d, J = 8.2 Hz, 2 H), 5.24 (d, J = 8.2 Hz, 2 H), 4.57 (t, J = 8.1 Hz, 1 H), 2.39 (s, 4 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 144.92, 128.92, 128.29, 127.97, 126.02–125.86 (m), 123.31, 113.80, 76.41, 38.24, 30.64; HRMS–ESI (m/z): $[M + H]^+$ calcd for $C_{14}H_{12}F_3NO_4$, 316.0791; found, 316.0802.

2-(1-(4-Chlorophenyl)-2-nitroethyl)cyclopentane-1,3-dione (3d)



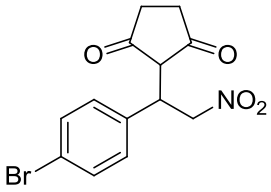
^{1}H NMR (400 MHz, DMSO-d_6) δ 7.35 (s, 4 H), 5.18–5.15 (m, 2 H), 4.46 (t, J = 8.2 Hz, 1 H), 2.39 (s, 4 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 139.14, 132.11, 129.97, 128.96, 114.19, 76.75, 37.88, 30.63; HRMS–ESI (m/z): $[M + Na]^+$ calcd for $C_{13}H_{12}ClNO_4$, 304.0347; found, 304.0356.

2-(1-(3-Chlorophenyl)-2-nitroethyl)cyclopentane-1,3-dione (3e)



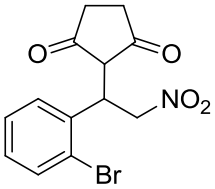
^{1}H NMR (400 MHz, DMSO-d_6) δ 7.39 (s, 1 H), 7.36–7.28 (m, 3 H), 5.20–5.17 (m, 2 H), 4.47 (t, *J* = 8.1 Hz, 1 H), 2.40 (s, 4 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 142.58, 133.53, 130.92, 127.89, 127.52, 126.89, 113.99, 76.59, 38.12, 30.66; HRMS–ESI (*m*/*z*): $[M + H]^+$ calcd for $C_{13}H_{12}ClNO_4$, 282.0528; found, 282.0533.

2-(1-(4-Bromophenyl)-2-nitroethyl)cyclopentane-1,3-dione (3f)



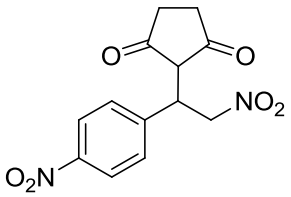
^{1}H NMR (400 MHz, DMSO-d_6) δ 7.49 (d, *J* = 8.4 Hz, 2 H), 7.29 (d, *J* = 8.4 Hz, 2 H), 5.19–5.15 (m, 2 H), 4.45 (t, *J* = 8.2 Hz, 1 H), 2.39 (s, 4 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 139.07, 131.39, 129.84, 120.11, 113.63, 76.16, 37.43, 30.14; HRMS–ESI (*m*/*z*): $[M + Na]^+$ calcd for $C_{13}H_{12}BrNO_4$, 347.9842; found, 347.9840.

2-(1-(2-Bromophenyl)-2-nitroethyl)cyclopentane-1,3-dione (3g)



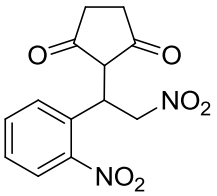
^{1}H NMR (400 MHz, DMSO-d_6) δ 7.60 (d, *J* = 8.0 Hz, 1 H), 7.54 (d, *J* = 7.8 Hz, 1 H), 7.34 (t, *J* = 7.5 Hz, 1 H), 7.18 (t, *J* = 7.6 Hz, 1 H), 5.23–5.16 (m, 1 H), 4.95–4.90 (m, 2 H), 2.41 (s, 4 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 138.61, 133.30, 130.20, 129.59, 128.47, 124.07, 113.11, 75.82, 38.12, 30.69; HRMS–ESI (*m*/*z*): $[M + H]^+$ calcd for $C_{13}H_{12}BrNO_4$, 326.0022; found, 326.0023.

2-(2-Nitro-1-(4-nitrophenyl)ethyl)cyclopentane-1,3-dione (3h)



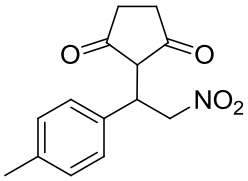
^{1}H NMR (400 MHz, DMSO-d_6) δ 8.17 (d, *J* = 8.7 Hz, 2 H), 7.61 (d, *J* = 8.7 Hz, 2 H), 5.31–5.20 (m, 2 H), 4.61 (t, *J* = 8.1 Hz, 1 H), 2.40 (s, 4 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 147.81, 146.98, 129.38, 124.19, 113.46, 76.10, 38.15, 30.66; HRMS–ESI (*m*/*z*): $[M + H]^+$ calcd for $C_{13}H_{12}N_2O_6$, 293.0768; found, 293.0774.

2-(2-Nitro-1-(2-nitrophenyl)ethyl)cyclopentane-1,3-dione (3i)



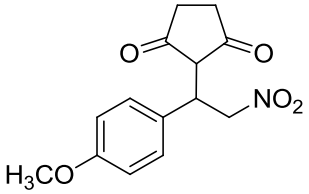
^{1}H NMR (400 MHz, DMSO-d_6) δ 7.86 (d, J = 8.1 Hz, 1 H), 7.75 (d, J = 7.7 Hz, 1 H), 7.67 (t, J = 7.5 Hz, 1 H), 7.49 (t, J = 7.6 Hz, 1 H), 5.38–5.33 (m, 1 H), 5.13–5.08 (m, 1 H), 4.97–4.93 (m, 1 H), 2.37 (s, 4 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 149.03, 133.21, 133.14, 130.09, 128.49, 124.16, 112.76, 75.24, 32.98, 30.10; HRMS–ESI (m/z): $[M + H]^+$ calcd for $C_{13}H_{12}N_2O_6$, 293.0768; found, 293.0779.

2-(2-Nitro-1-(p-tolyl)ethyl)cyclopentane-1,3-dione (3j)



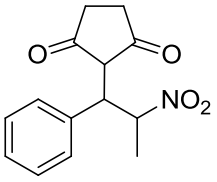
^{1}H NMR (400 MHz, DMSO-d_6) δ 6.97 (d, J = 7.9 Hz, 2 H), 6.85 (d, J = 7.9 Hz, 2 H), 4.95–4.85 (m, 2 H), 4.18 (t, J = 8.2 Hz, 1 H), 2.14 (s, 4 H), 2.00 (s, 3 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 137.16, 136.56, 129.52, 127.94, 114.80, 77.15, 38.18, 30.59, 21.02; HRMS–ESI (m/z): $[M + H]^+$ calcd for $C_{14}H_{15}NO_4$, 262.1074; found, 262.1085.

2-(1-(4-Methoxyphenyl)-2-nitroethyl)cyclopentane-1,3-dione (3k)



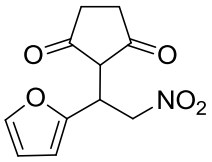
^{1}H NMR (400 MHz, DMSO-d_6) δ 7.25 (d, J = 8.5 Hz, 2 H), 6.85 (d, J = 8.5 Hz, 2 H), 5.17–5.06 (m, 2 H), 4.41 (t, J = 8.2 Hz, 1 H), 3.71 (s, 3 H), 2.38 (s, 4 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 158.67, 132.13, 129.17, 114.94, 114.35, 77.35, 55.47, 37.84, 30.60; HRMS–ESI (m/z): $[M + H]^+$ calcd for $C_{13}H_{15}NO_5$, 278.1023; found, 278.1032.

2-(2-Nitro-1-phenylpropyl)cyclopentane-1,3-dione (3l)



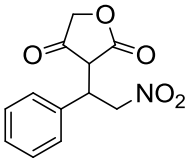
^{1}H NMR (400 MHz, DMSO-d_6) δ 7.43–7.18 (m, 5 H), 5.81–5.70 (m, 1 H), 4.11–4.04 (m, 1 H), 2.39 (s, 0.71 H), 2.32 (s, 3.17), 1.45 (d, J = 6.5 Hz, 0.56 H), 1.26 (d, J = 6.7 Hz, 2.49 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 140.61, 139.92, 129.14, 128.95, 128.87, 128.13, 127.57, 127.50, 115.35, 114.66, 85.34, 83.52, 45.59, 45.42, 30.48, 19.25, 19.05; HRMS–ESI (m/z): $[M + H]^+$ calcd for $C_{14}H_{15}NO_4$, 262.1074; found, 262.1081.

2-(1-(Furan-2-yl)-2-nitroethyl)cyclopentane-1,3-dione (3m)



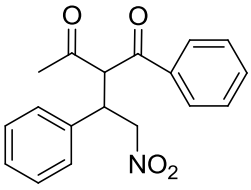
^{1}H NMR (400 MHz, DMSO-d_6) δ 7.52 (s, 1 H), 6.34 (t, J = 2 Hz, 1 H), 6.15 (d, J = 3.1 Hz, 1 H), 5.14–5.02 (m, 2 H), 4.61 (t, J = 7.9 Hz, 1 H), 2.41 (s, 4 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 151.81, 142.07, 111.52, 110.54, 106.34, 74.92, 31.74, 30.22; HRMS–ESI (m/z): $[M + H]^+$ calcd for $C_{11}H_{11}NO_5$, 238.0710; found, 238.0714.

3-(2-Nitro-1-phenylethyl)furan-2,4(3H,5H)-dione (3n)



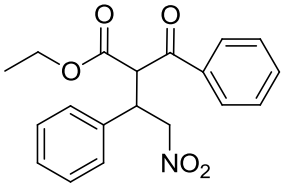
^{1}H NMR (400 MHz, DMSO-d_6) δ 7.38–7.24 (m, 5 H), 5.23 (dd, J = 13.3, 8.9 Hz, 1 H), 5.14 (dd, J = 13.3, 7.5 Hz, 1 H), 4.64 (s, 2 H), 4.51 (t, J = 8.2 Hz, 1 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 176.43, 174.33, 139.41, 129.14, 128.02, 127.75, 97.56, 76.84, 67.16, 38.13; HRMS–ESI (m/z): $[M + Na]^+$ calcd for $C_{12}H_{11}NO_5$, 272.0529; found, 272.0531.

2-(2-Nitro-1-phenylethyl)-1-phenylbutane-1,3-dione (3o)



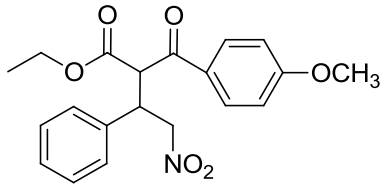
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03–7.14 (m, 10 H), 5.18 (t, J = 9.8 Hz, 1 H), 4.88–4.64 (m, 2 H), 4.56–4.40 (m, 1 H), 2.23 (s, 1.33 H), 1.94 (s, 1.48 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.67, 200.77, 194.06, 193.75, 136.53, 136.30, 136.17, 136.00, 134.49, 134.10, 129.28, 129.10, 129.03, 128.98, 128.56, 128.20, 128.15, 128.03, 78.18, 78.08, 65.36, 64.77, 43.44, 43.30, 29.78, 28.53; HRMS–ESI (m/z): $[M + Na]^+$ calcd for $C_{18}H_{17}NO_4$, 334.1050; found, 334.1064.

Ethyl 2-benzoyl-4-nitro-3-phenylbutanoate (3p)



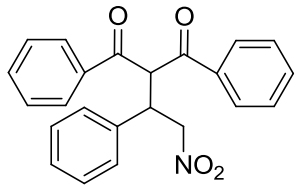
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06–7.16 (m, 10 H), 4.98–4.90 (m, 2 H), 4.82–4.73 (m, 1 H), 4.52–4.41 (m, 1 H), 4.17 (q, J = 7.2, 7.2 Hz, 1 H), 3.89–3.83 (m, 1 H), 1.17 (t, J = 7.1 Hz, 1.41 H), 0.89 (t, J = 7.1 Hz, 1.74 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.78, 192.74, 167.73, 166.99, 136.77, 136.27, 136.04, 135.85, 134.25, 133.84, 128.96, 128.93, 128.91, 128.75, 128.57, 128.36, 128.30, 128.13, 127.99, 78.00, 62.24, 61.96, 57.04, 56.38, 43.13, 43.11, 13.89, 13.59; HRMS–ESI (m/z): $[M + H]^+$ calcd for $C_{19}H_{19}NO_5$, 342.1336; found, 342.1339.

Ethyl 2-(4-methoxybenzoyl)-4-nitro-3-phenylbutanoate (3q)



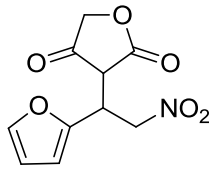
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.9 Hz, 1.11 H), 7.87 (d, *J* = 8.9 Hz, 0.83 H), 7.31–7.19 (m, 5H), 6.92 (dd, *J* = 26.4, 8.9 Hz, 2 H), 4.96–4.87 (m, 2 H), 4.82–4.70 (m, 1 H), 4.50–4.40 (m, 1 H), 4.17 (q, *J* = 6.0, 7.2 Hz, 1 H), 3.90–3.84 (m, 4 H), 1.18 (t, *J* = 7.1 Hz, 1.26 H), 0.90 (t, *J* = 7.1 Hz, 1.82 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.90, 190.84, 167.95, 167.21, 164.48, 164.15, 136.97, 136.41, 131.44, 131.11, 128.92, 128.89, 128.30, 128.05, 127.94, 114.10, 113.97, 78.11, 78.08, 62.14, 61.86, 56.68, 55.99, 55.61, 55.55, 43.14, 13.94, 13.63; HRMS–ESI (*m*/*z*): $[M + H]^+$ calcd for $C_{20}H_{21}NO_6$, 372.1442; found, 372.1448.

2-(2-Nitro-1-phenylethyl)-1,3-diphenylpropane-1,3-dione (3r)



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (dd, *J* = 8.4, 1.1 Hz, 2 H), 7.78 (dd, *J* = 8.3, 1.1 Hz, 2 H), 7.57–7.14 (m, 11 H), 5.84 (d, *J* = 8.0 Hz, 1 H), 5.00 (d, *J* = 6.9 Hz, 2 H), 4.63 (q, *J* = 7.2, 7.6 Hz, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.24, 193.62, 136.80, 136.22, 135.85, 134.06, 133.78, 128.97, 128.95, 128.83, 128.77, 128.60, 128.26, 128.15, 77.31, 59.89, 44.05; HRMS–ESI (*m*/*z*): $[M + H]^+$ calcd for $C_{23}H_{19}NO_4$, 374.1387; found, 374.1390.

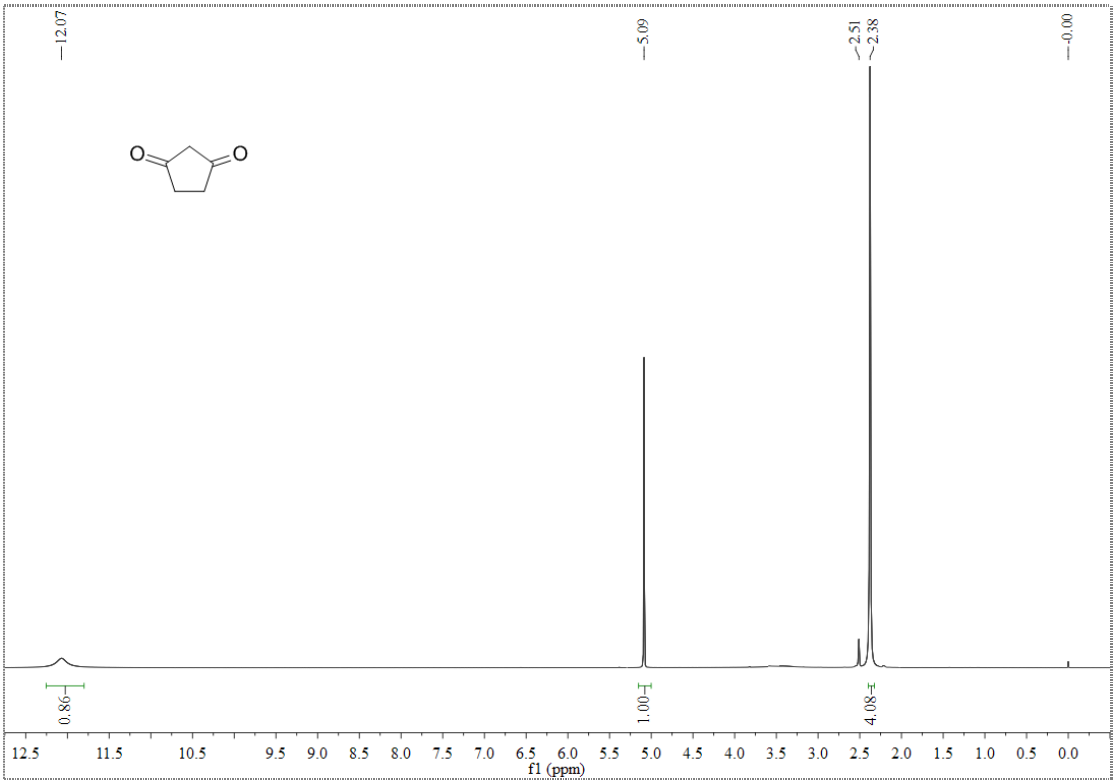
3-(1-(Furan-2-yl)-2-nitroethyl)furan-2,4(3H,5H)-dione (3s)

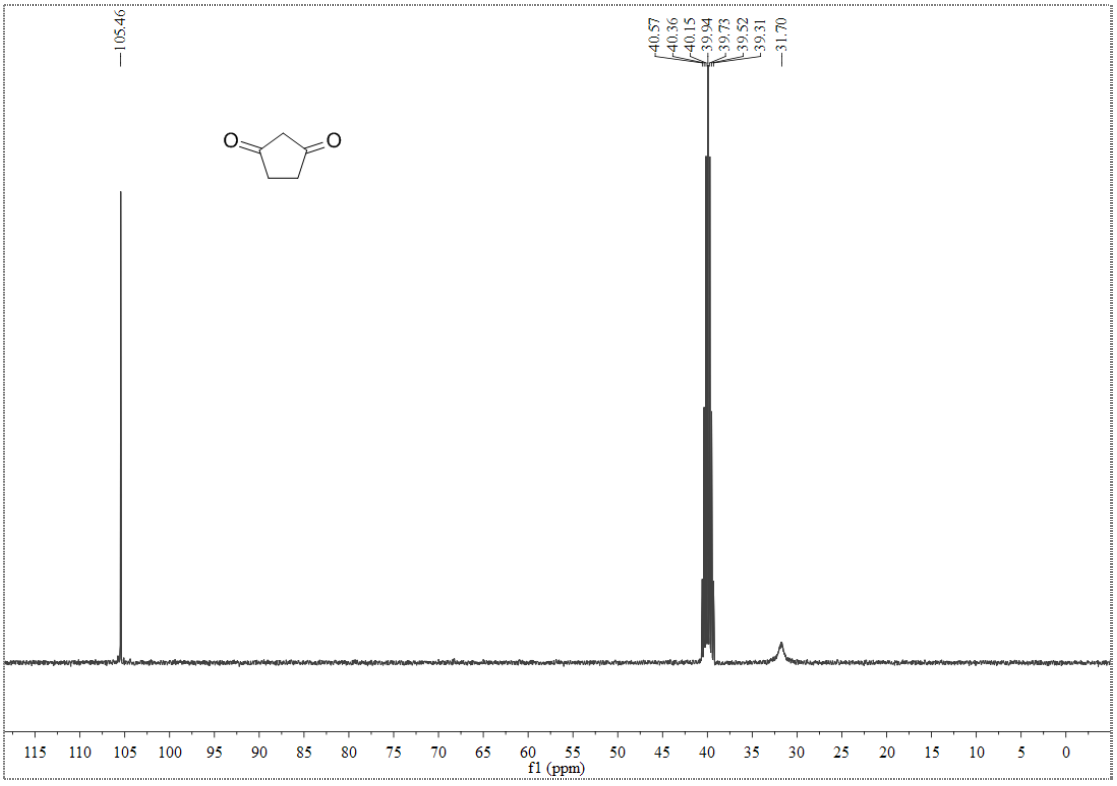


^{1}H NMR (400 MHz, DMSO-d_6) δ 7.56 (d, *J* = 1.3 Hz, 1 H), 6.38 (dd, *J* = 3.0, 2.0 Hz, 1 H), 6.23 (d, *J* = 3.3 Hz, 1 H), 5.14–5.06 (m, 2 H), 4.66–4.62 (m, 3 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 177.19, 173.81, 151.42, 142.85, 111.05, 107.10, 94.90, 75.07, 67.25, 31.98; HRMS–ESI (*m*/*z*): $[M + Na]^+$ calcd for $C_{10}H_9NO_6$, 262.0322; found, 262.0332.

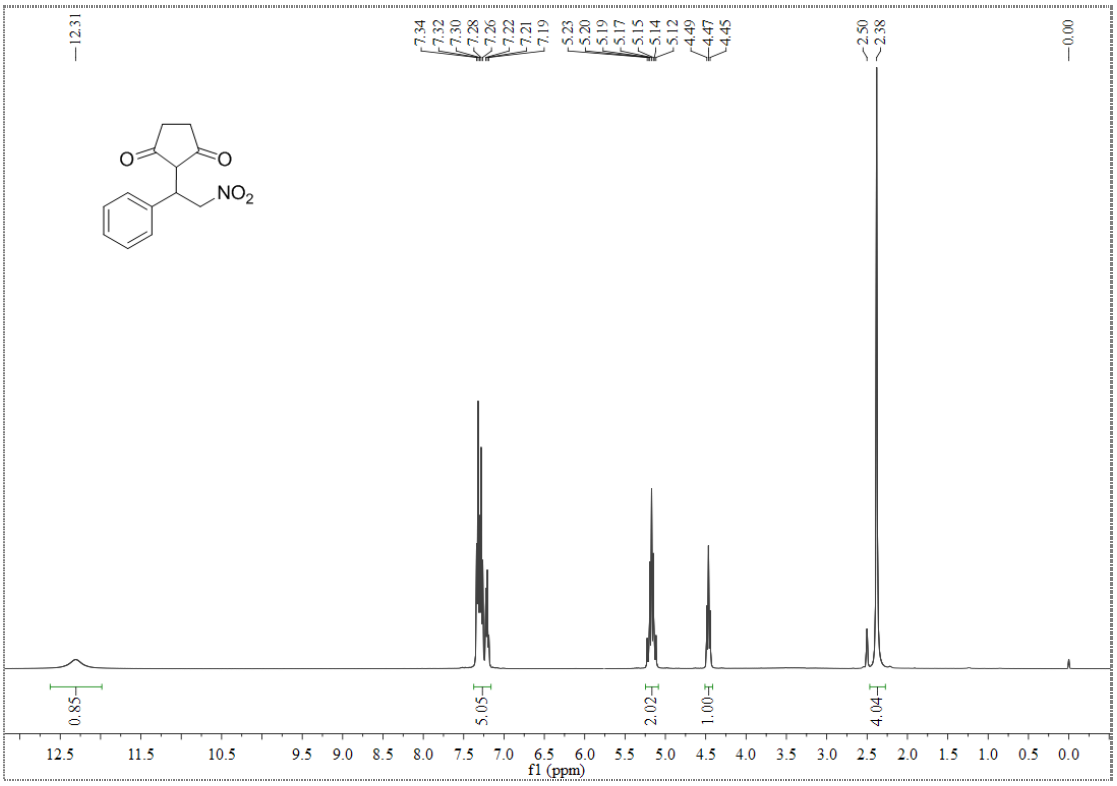
Spectra

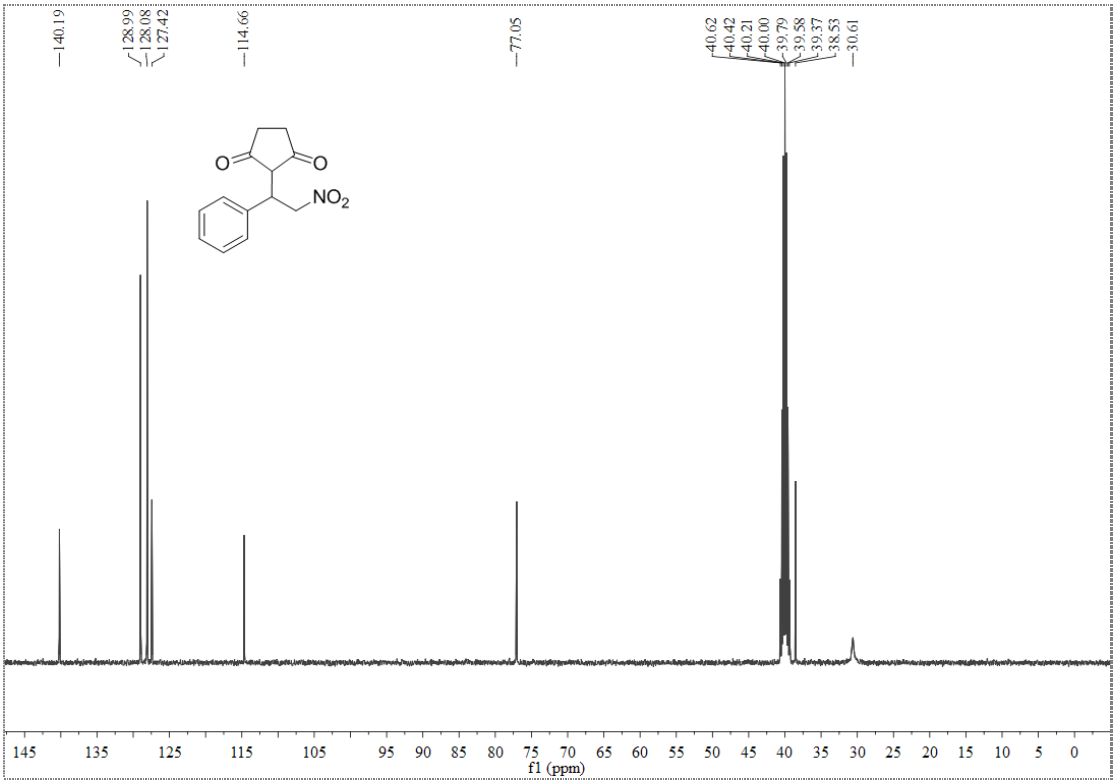
1,3-Cyclopentanedione (*material*)

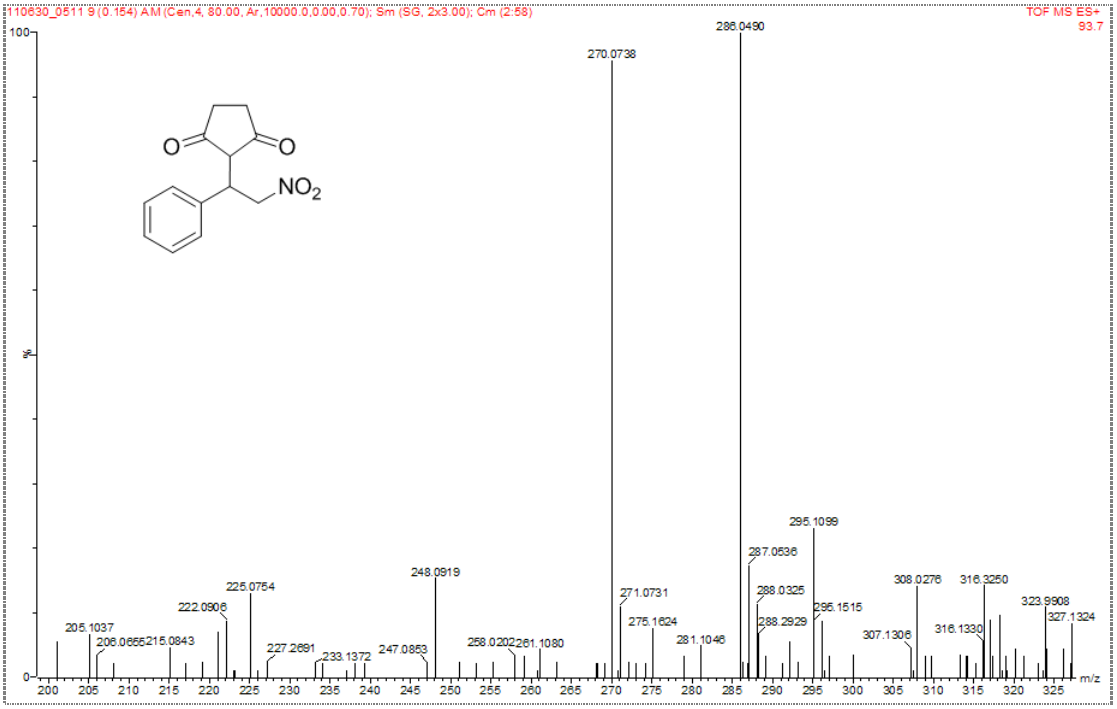




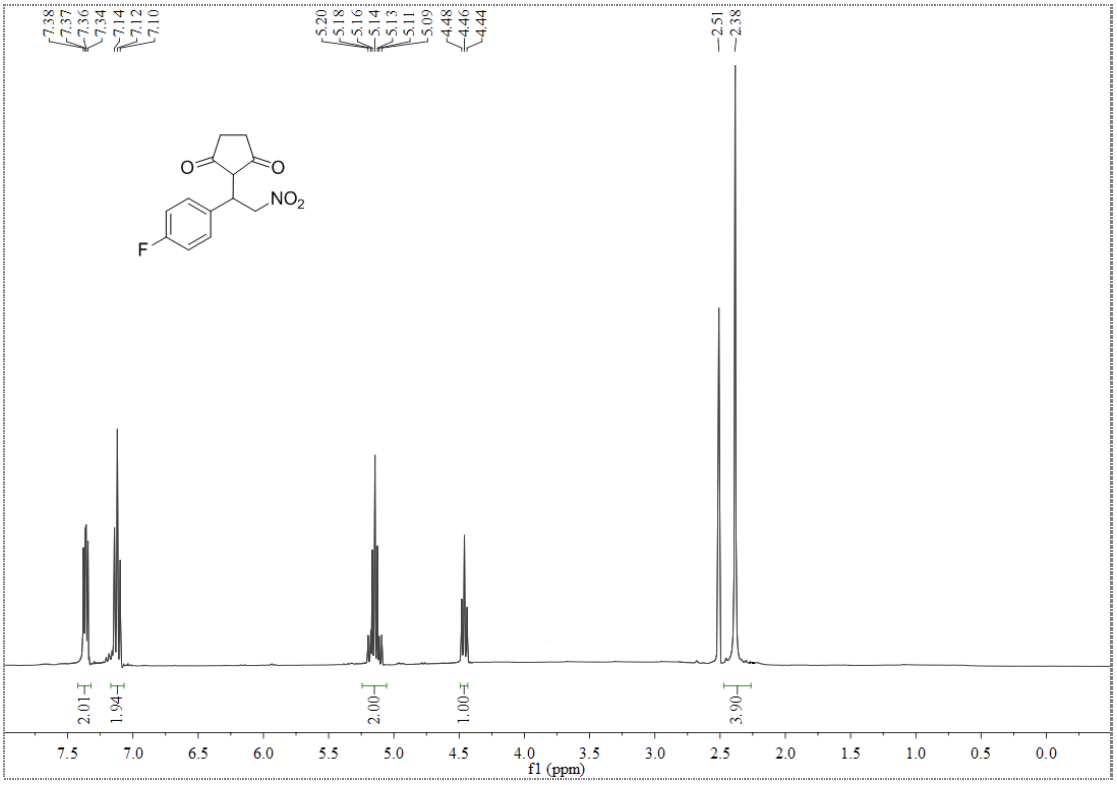
2-(2-Nitro-1-phenylethyl)cyclopentane-1,3-dione (3a)

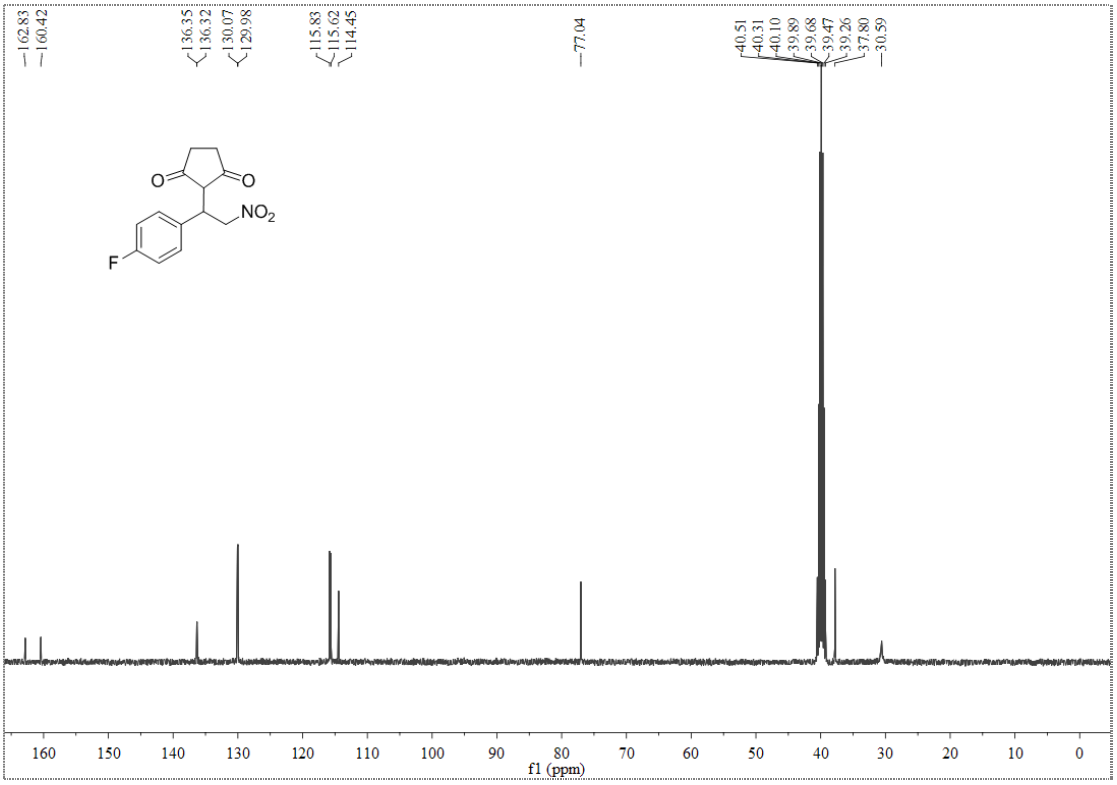


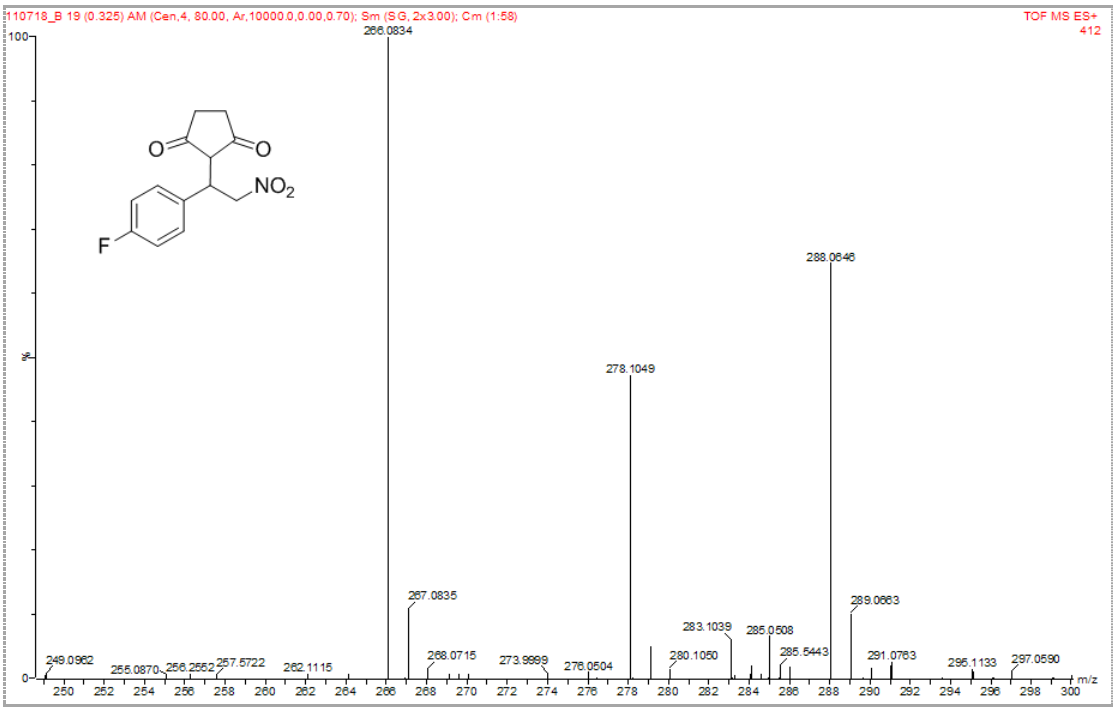




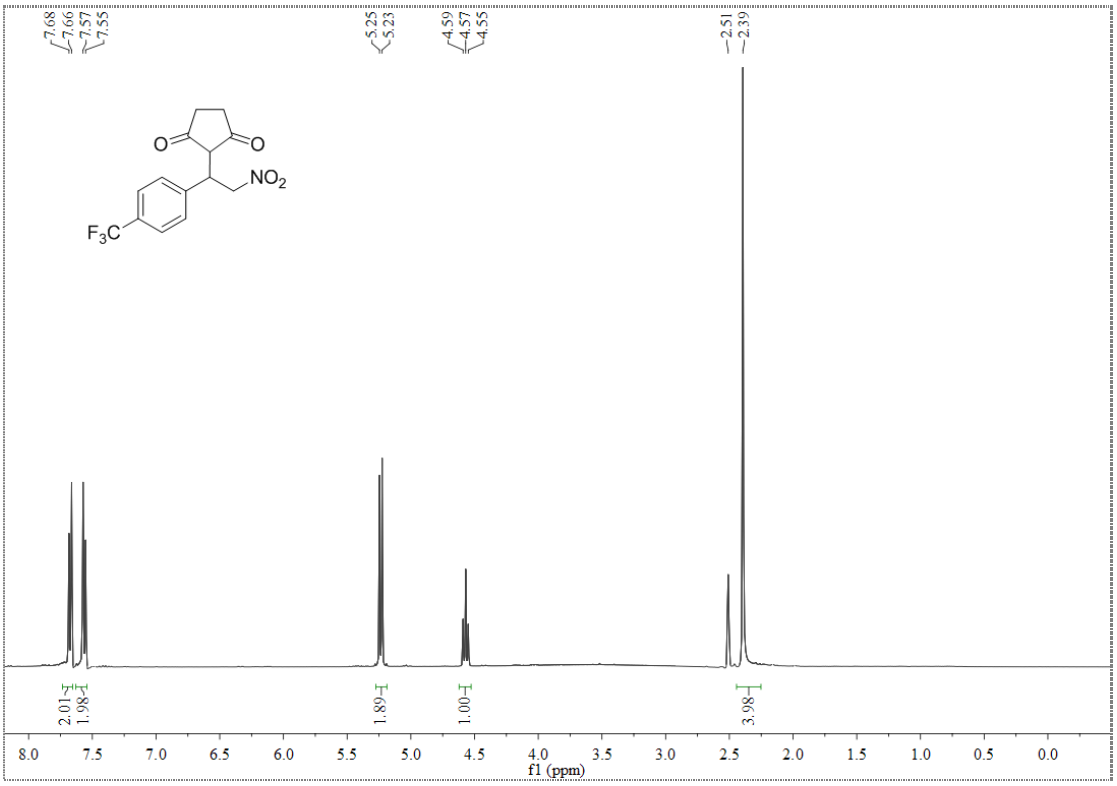
2-(1-(4-Fluorophenyl)-2-nitroethyl)cyclopentane-1,3-dione (3b)

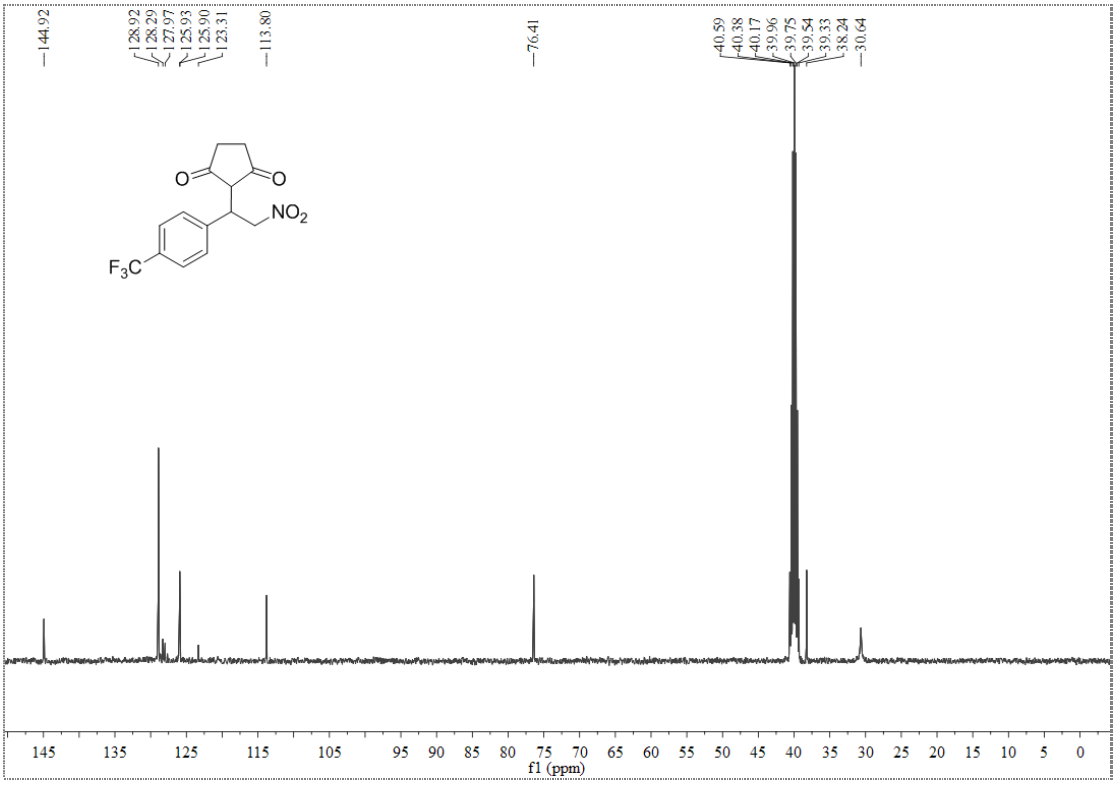


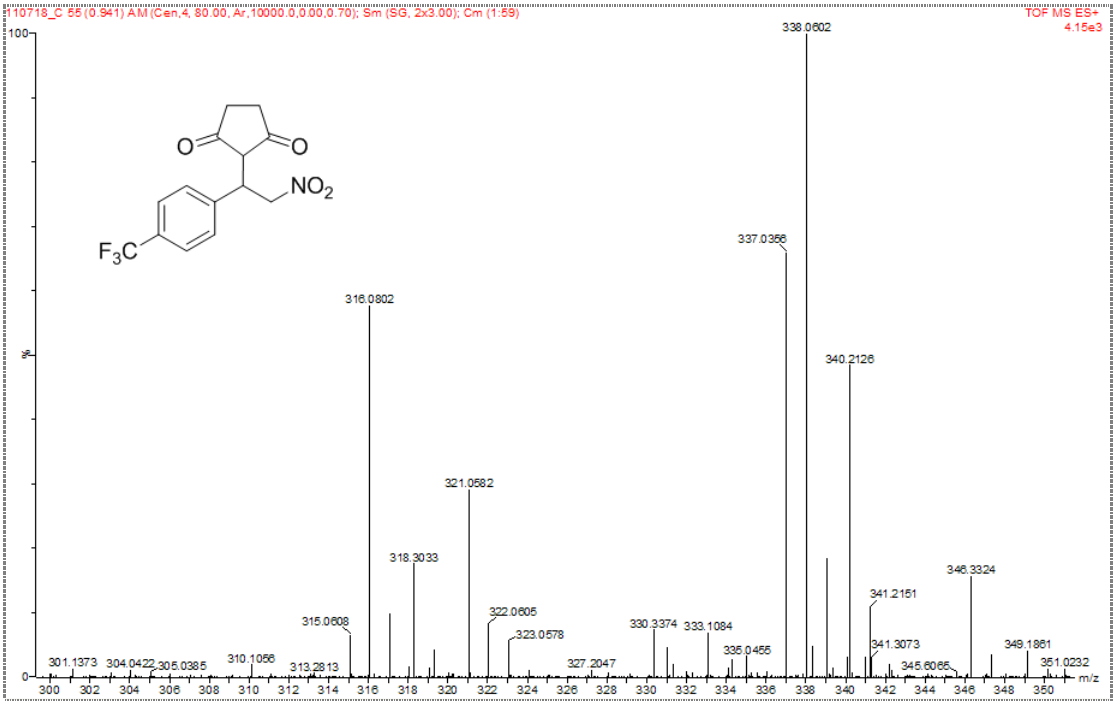

162.83
160.42
136.35
136.32
130.07
129.98
115.83
115.62
114.45
77.04
40.51
40.31
40.10
39.89
39.68
39.47
39.26
37.80
30.59
O
O
NO2
F
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


110718_B 19 (0.325) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.70); Sm (SG, 2x3.00); Cm (1:58)
TOF MS ES+
412
100
%
0
266.0834
288.0646
278.1049
267.0835
289.0663
283.1039
285.0508
285.5443
291.0763
295.1133
297.0590
249.0962
255.0870
256.2552
257.5722
262.1115
268.0715
273.9999
276.0504
280.1050
O
O
NO2
F
m/z

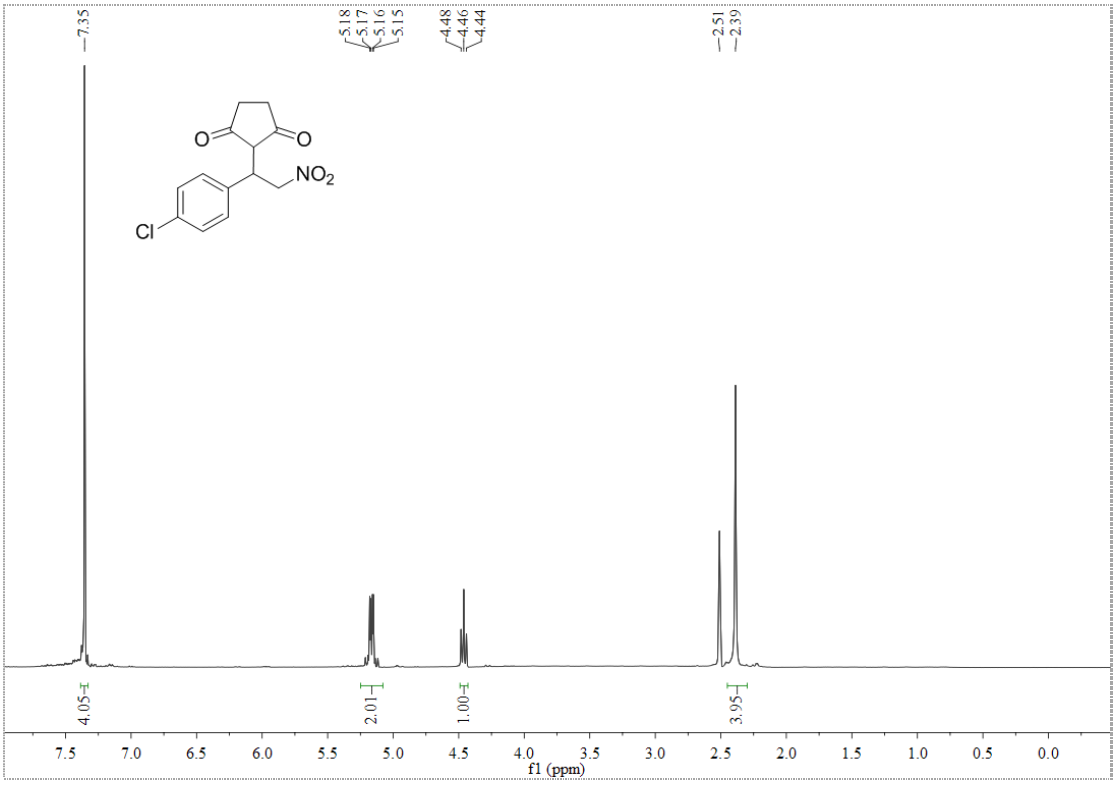
2-(2-Nitro-1-(4-(trifluoromethyl)phenyl)ethyl)cyclopentane-1,3-dione (3c)

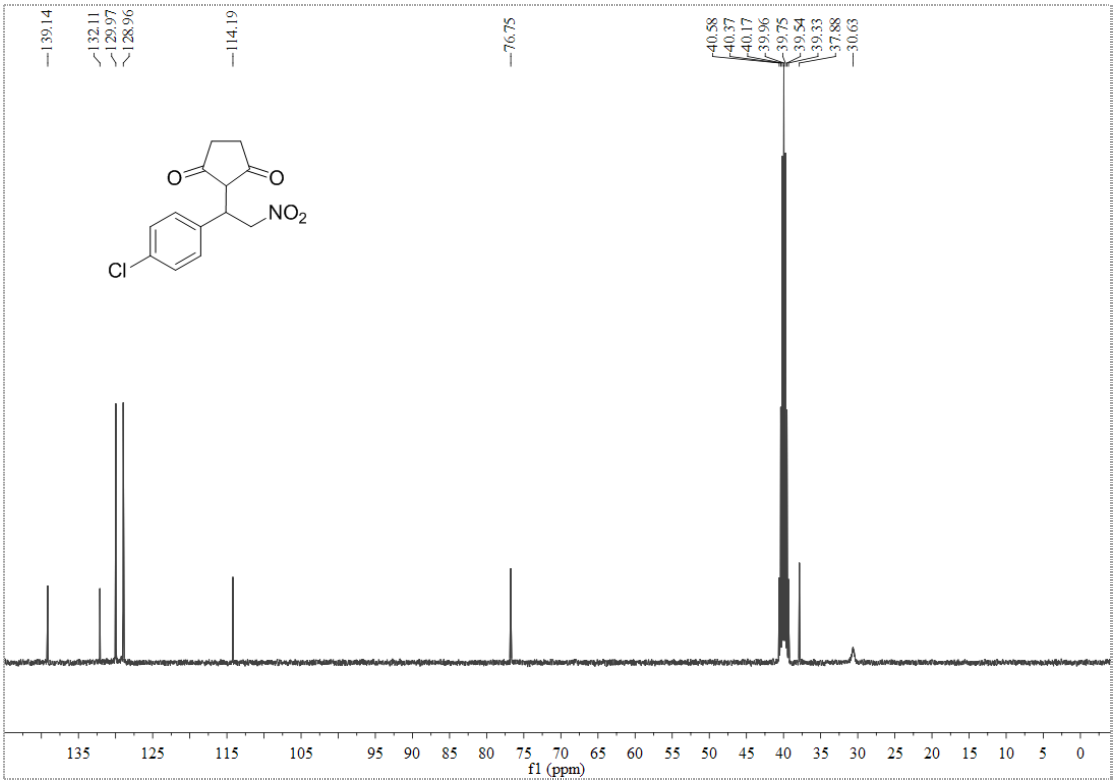


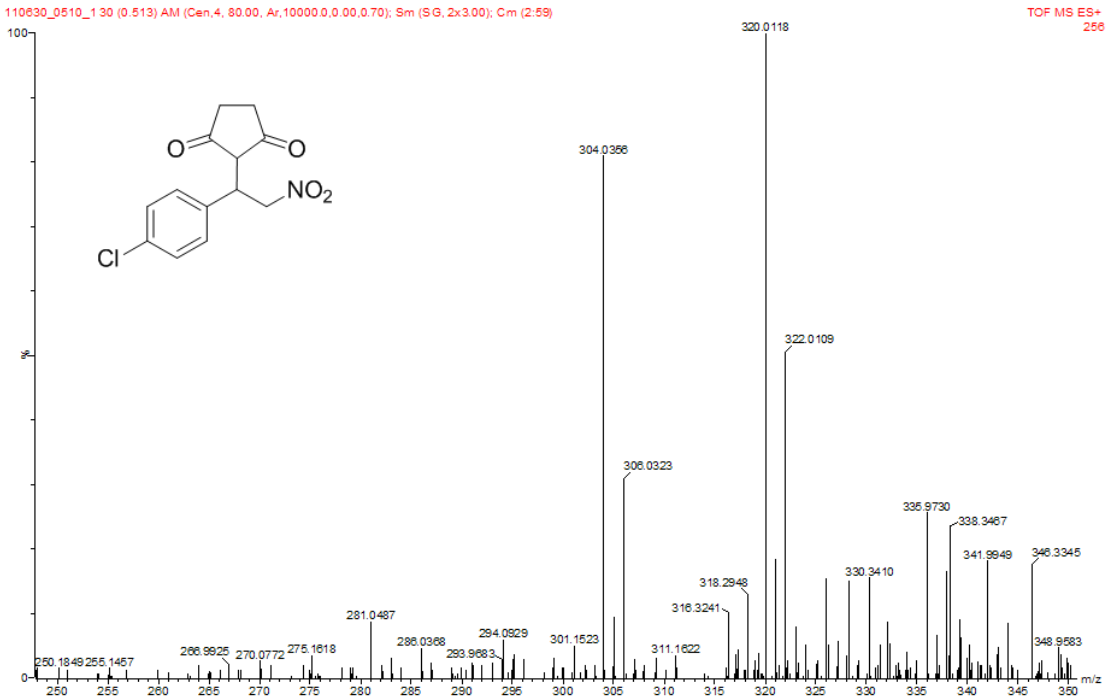




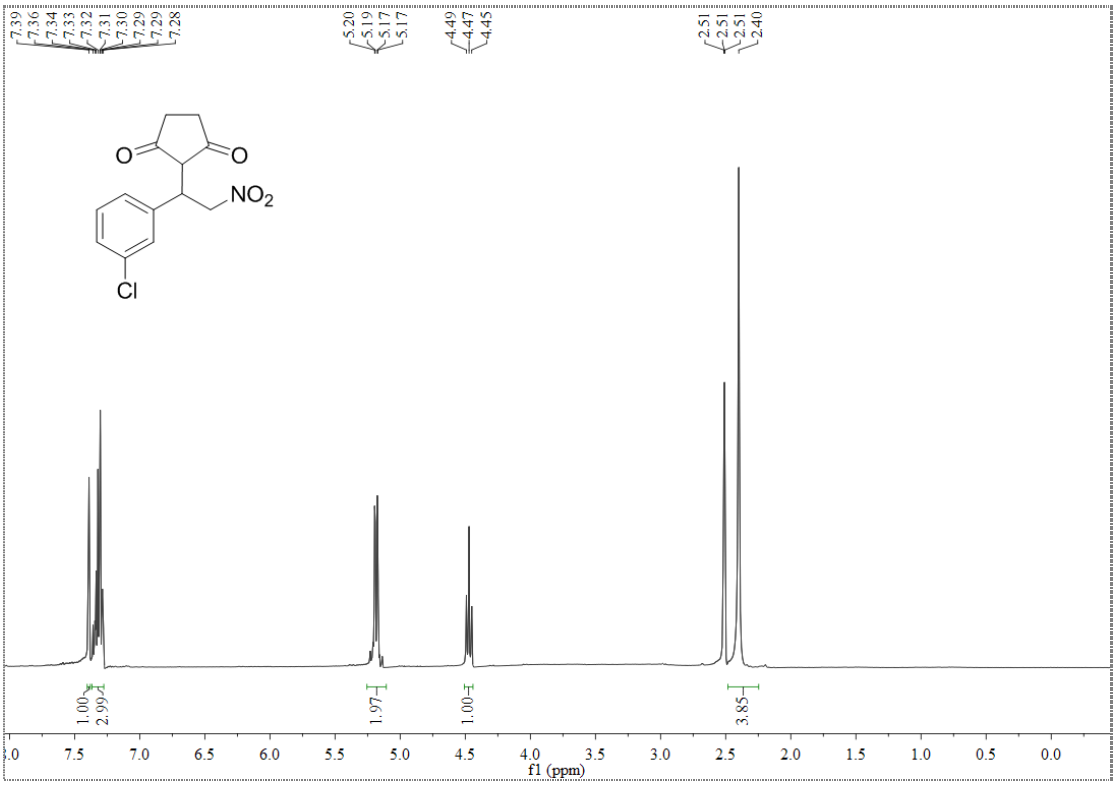
2-(1-(4-Chlorophenyl)-2-nitroethyl)cyclopentane-1,3-dione (3d)

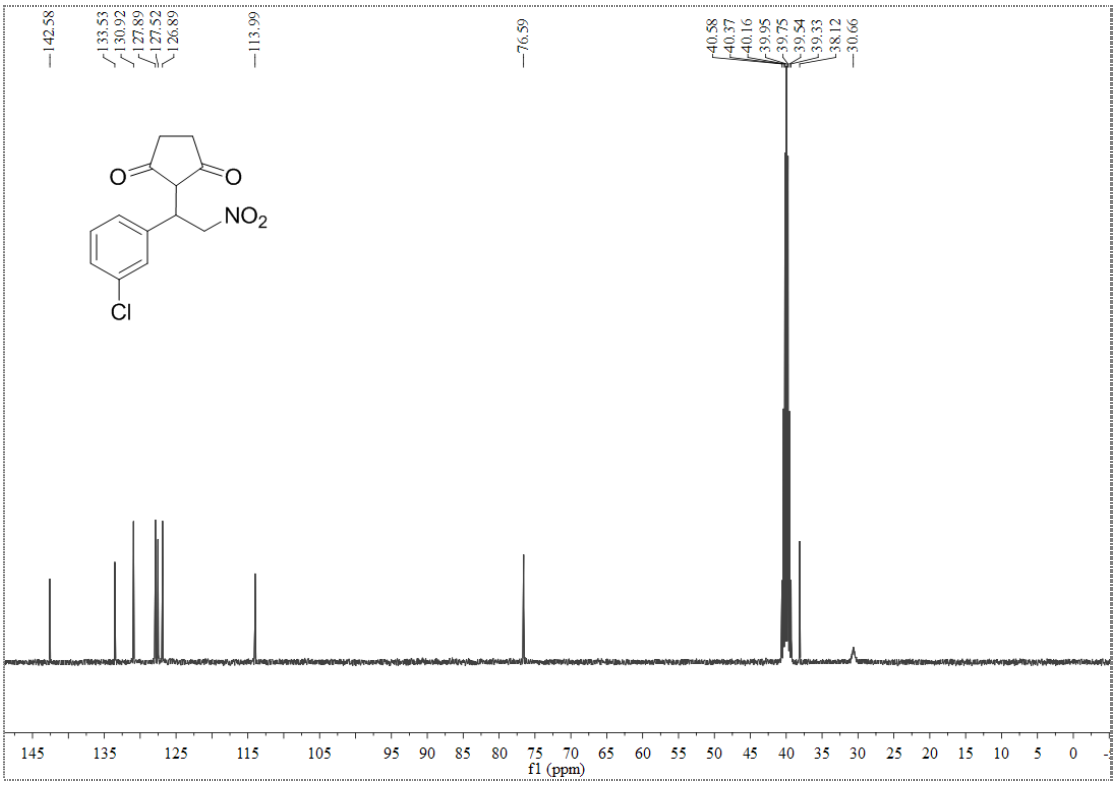


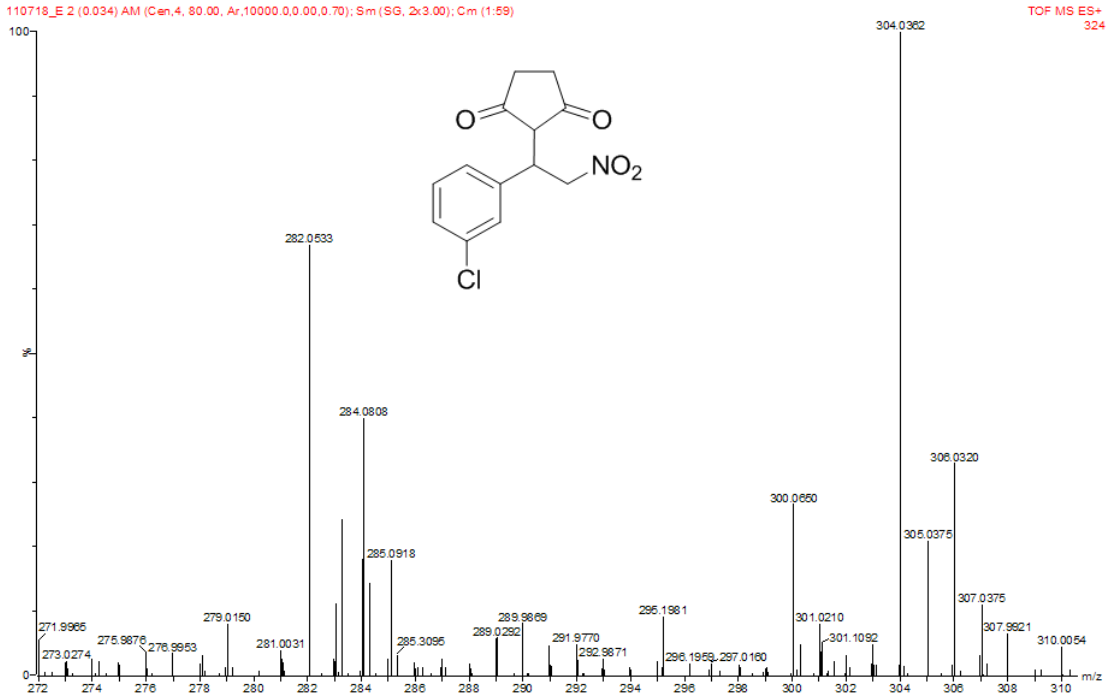

139.14
132.11
129.97
128.96
114.19
76.75
40.58
40.37
40.17
39.96
39.75
39.54
39.33
37.88
30.63
O
O
NO2
Cl
135 125 115 105 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)


110630_0510_1 30 (0.513) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.70); Sm (SG, 2x3.00); Cm (2:59)
TOF MS ES+
256
100
%
0
O
O
NO2
Cl
320.0118
304.0356
322.0109
306.0323
335.9730
338.3467
341.9949
346.3345
330.3410
318.2948
316.3241
281.0487
294.0929
286.0368
301.1523
293.9683
311.1622
275.1618
266.9925
270.0772
250.1849
255.1457
348.9583
250 255 260 265 270 275 280 285 290 295 300 305 310 315 320 325 330 335 340 345 350
m/z

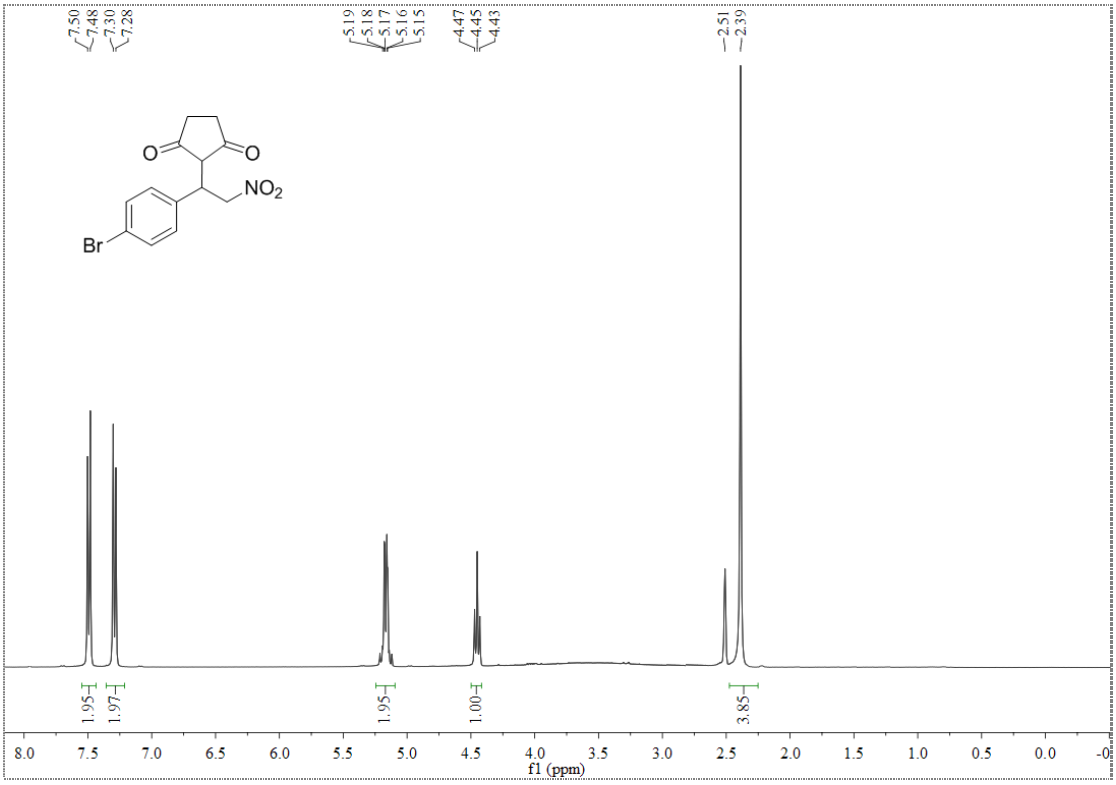
2-(1-(3-Chlorophenyl)-2-nitroethyl)cyclopentane-1,3-dione (3e)

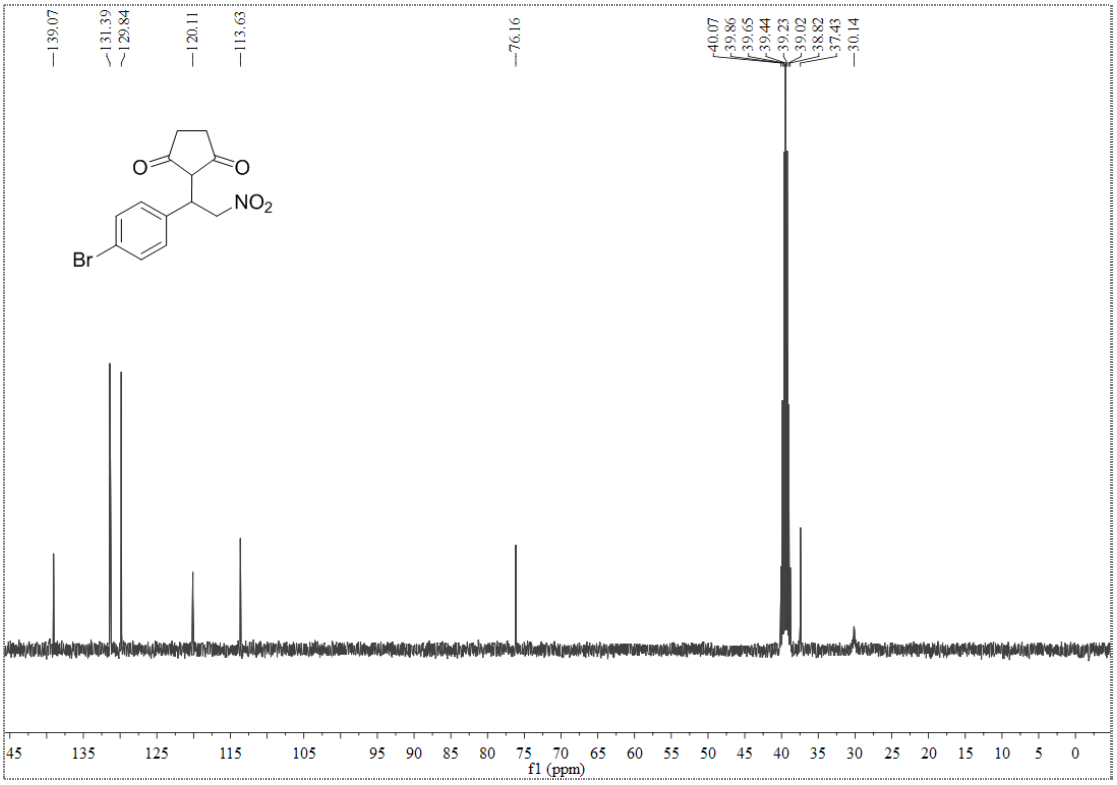


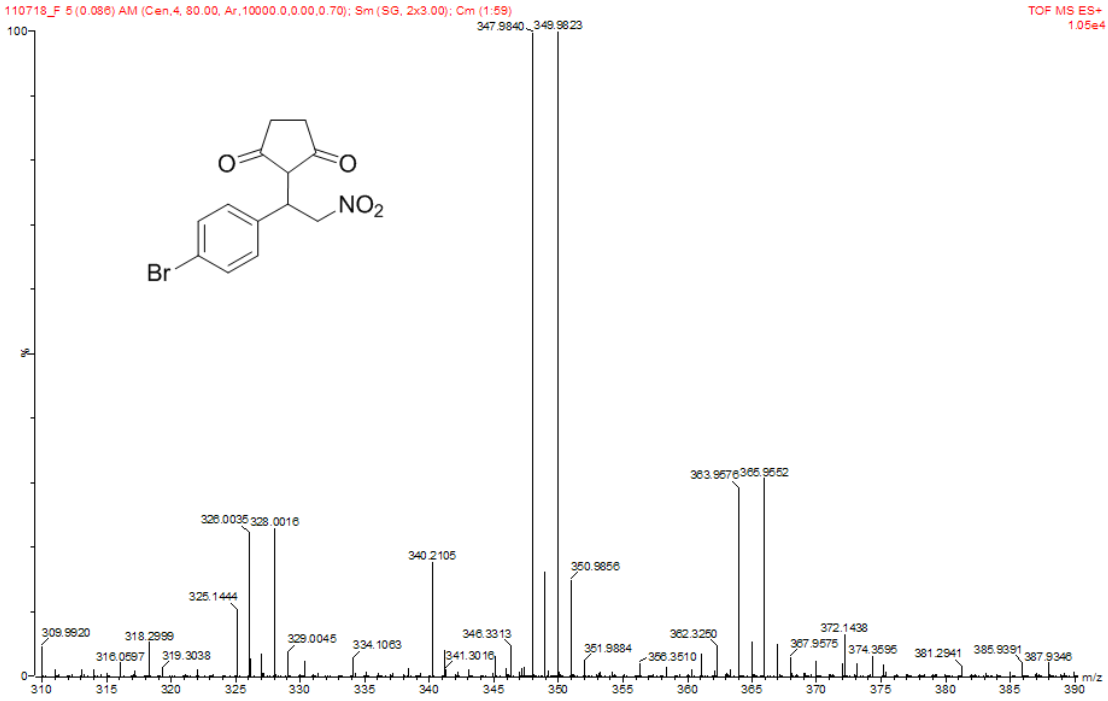




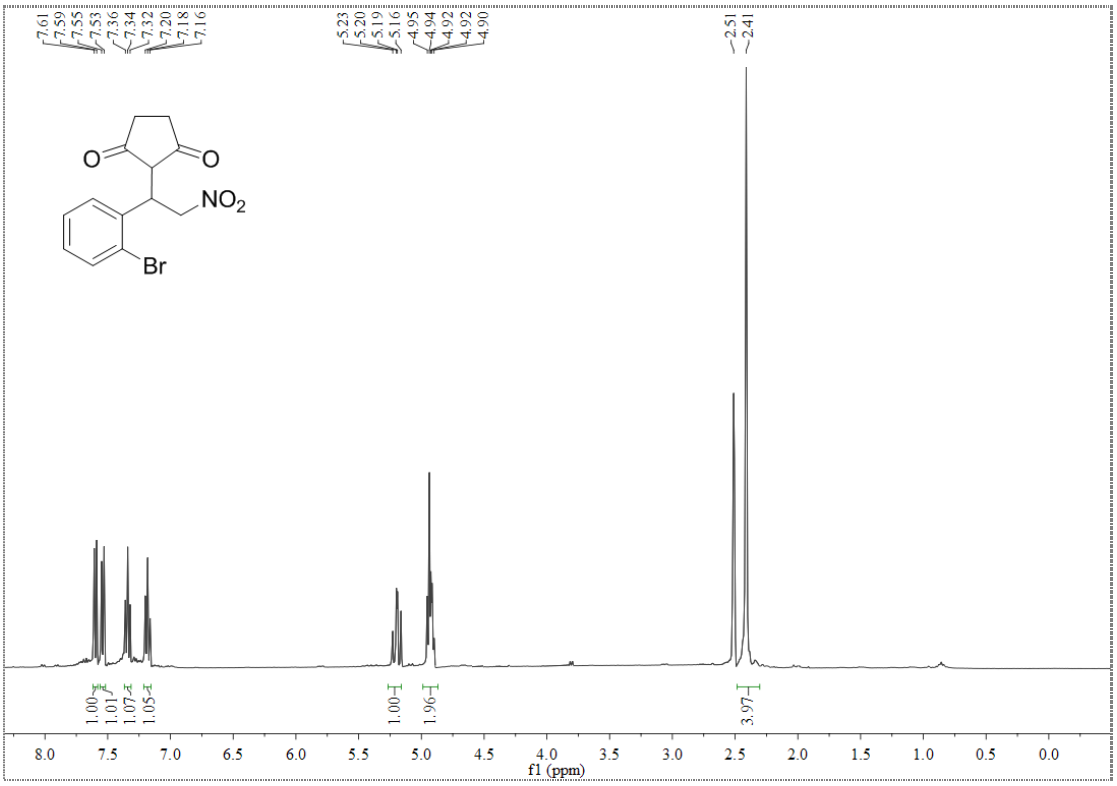
2-(1-(4-Bromophenyl)-2-nitroethyl)cyclopentane-1,3-dione (3f)

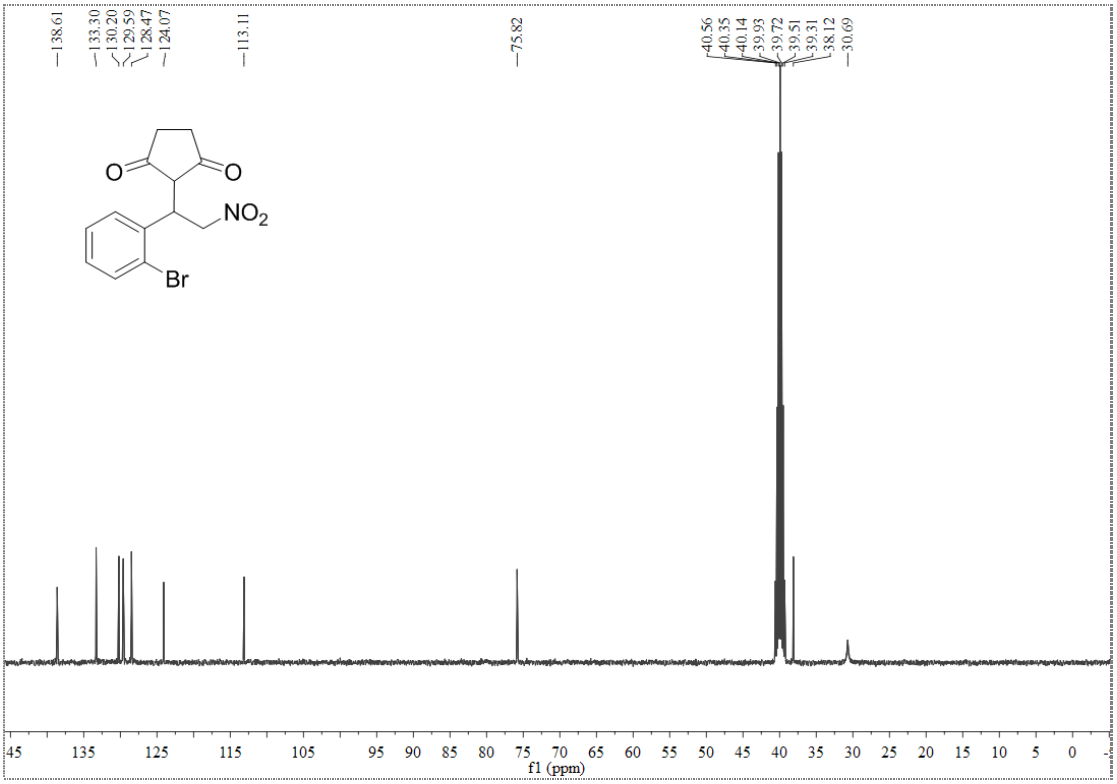


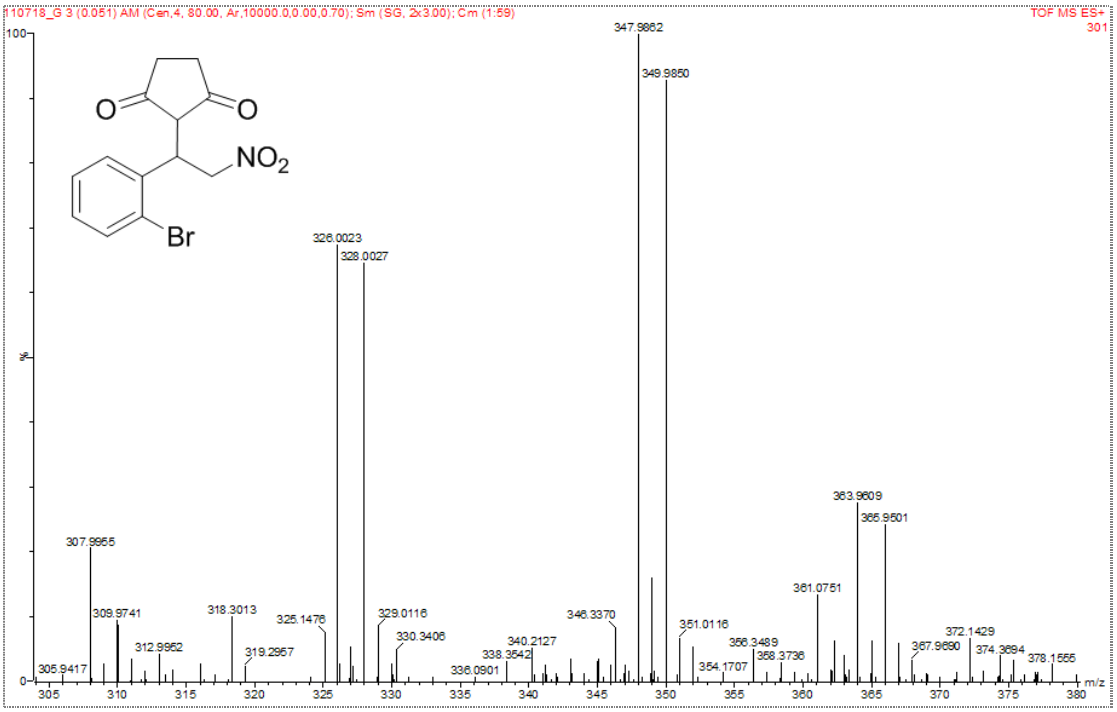

139.07
131.39
129.84
120.11
113.63
76.16
40.07
39.86
39.65
39.44
39.23
39.02
38.82
37.43
30.14
O
O
NO2
Br
f1 (ppm)


110718_F 5 (0.086) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.70); Sm (SG, 2x3.00); Cm (1:59)
TOF MS ES+
1.05e4
347.9840
349.9823
363.9576
365.9552
326.0035
328.0016
340.2105
350.9856
325.1444
309.9920
318.2999
316.0597
319.3038
329.0045
334.1063
346.3313
341.3016
351.9884
356.3510
362.3250
367.9575
372.1438
374.3595
381.2941
385.9391
387.9346
O
O
NO2
Br
m/z

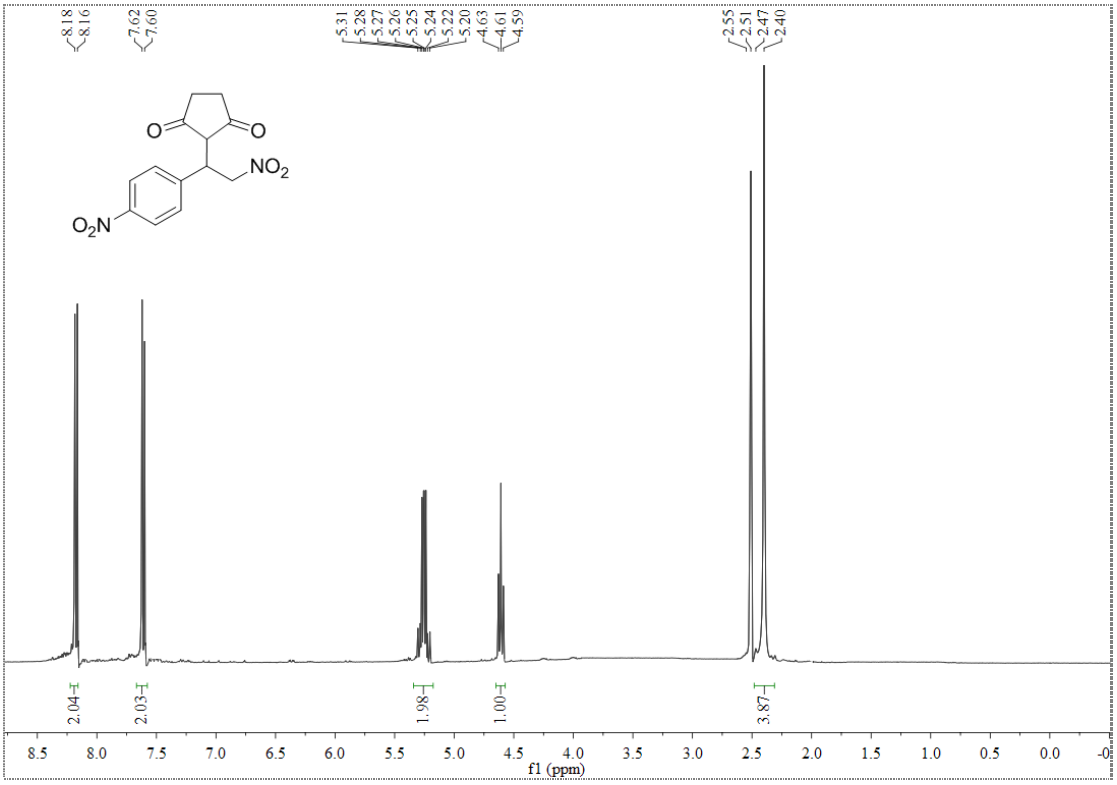
2-(1-(2-Bromophenyl)-2-nitroethyl)cyclopentane-1,3-dione (3g)

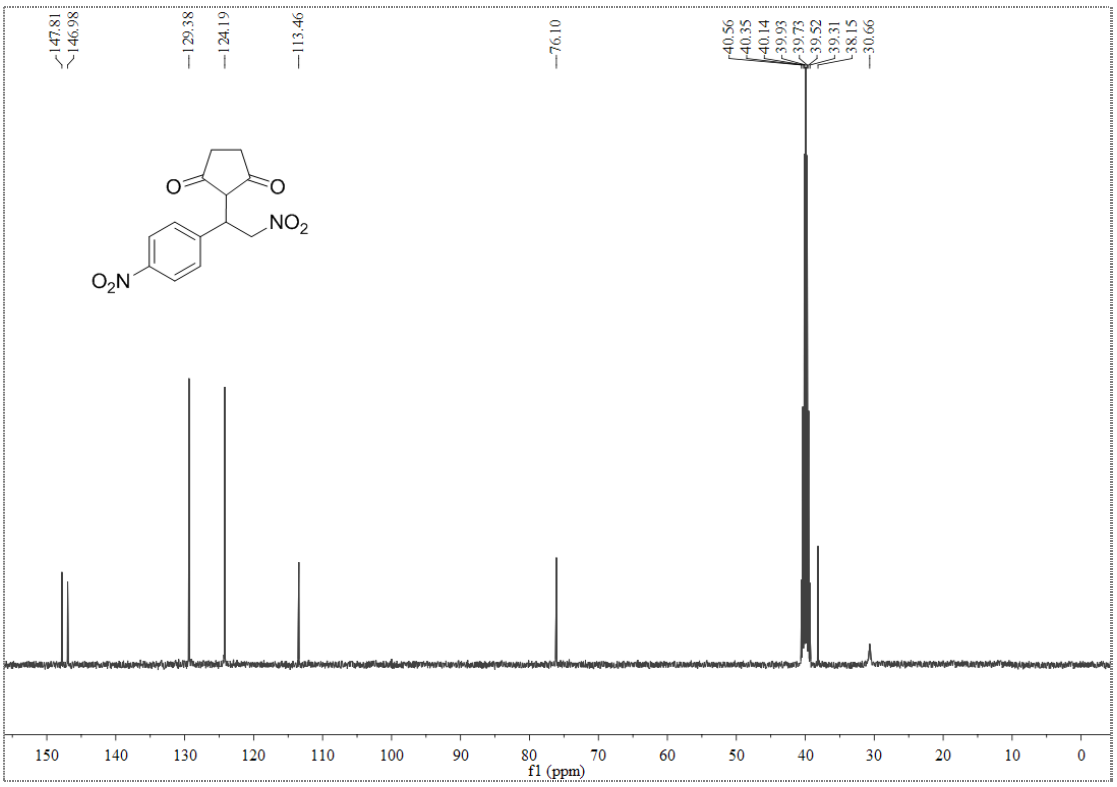


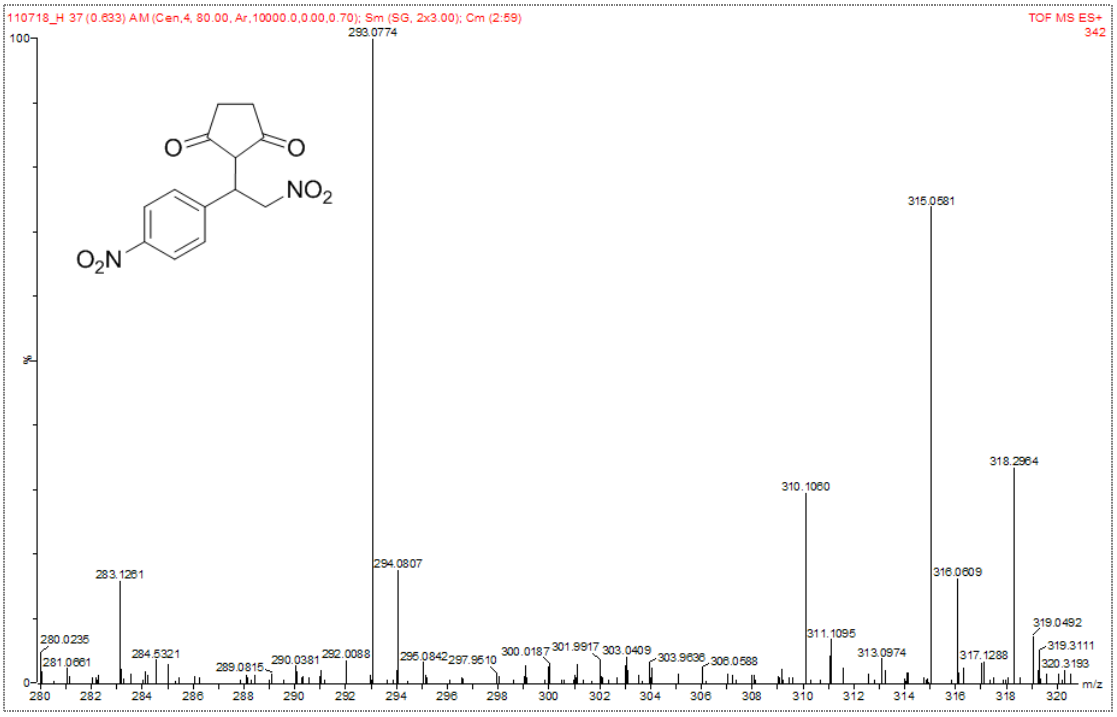




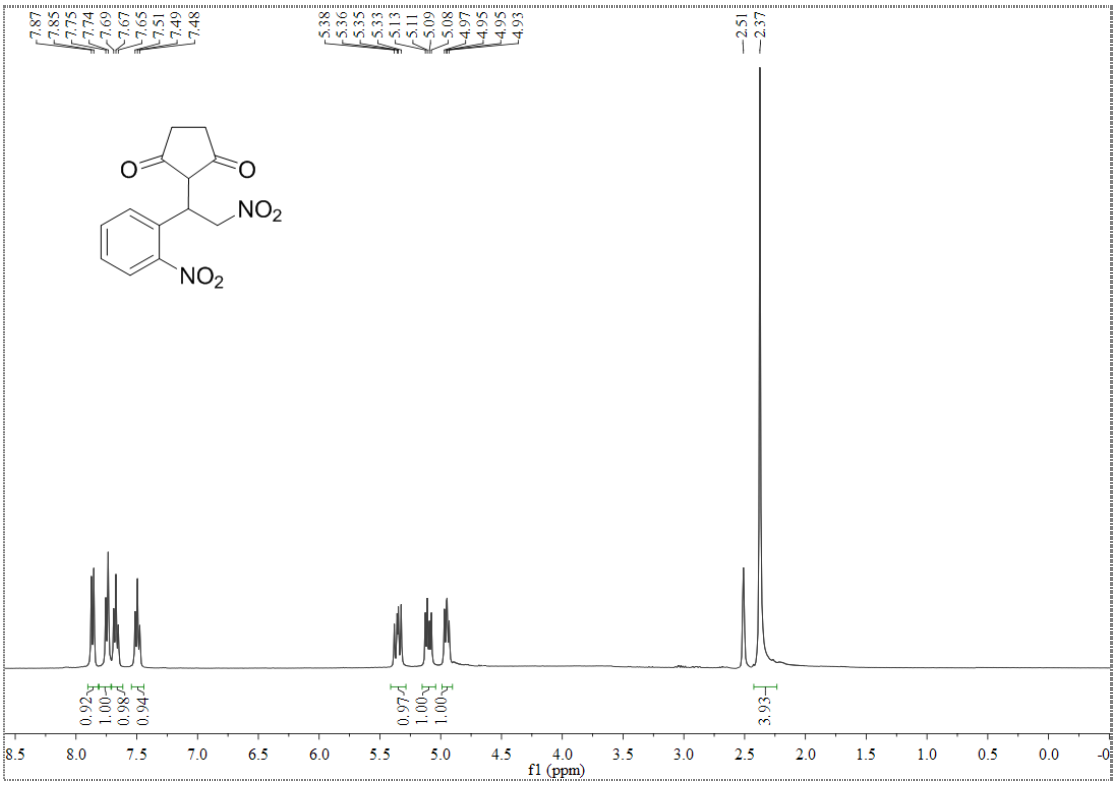
2-(2-Nitro-1-(4-nitrophenyl)ethyl)cyclopentane-1,3-dione (3h)

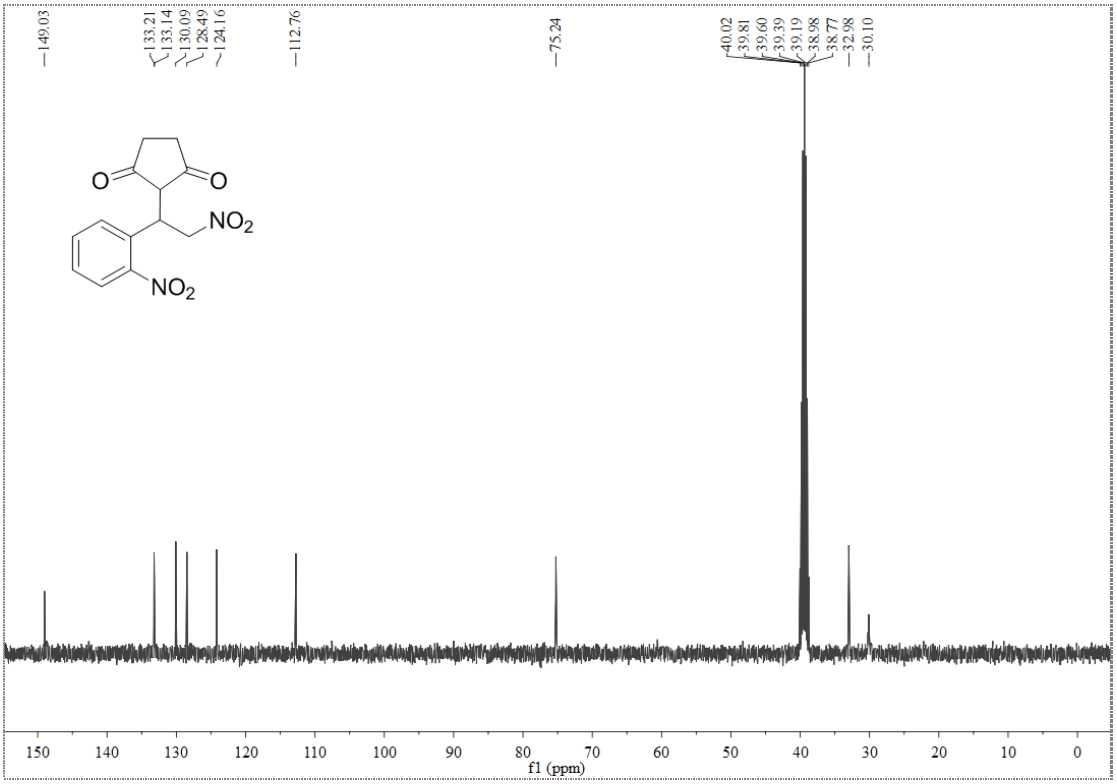


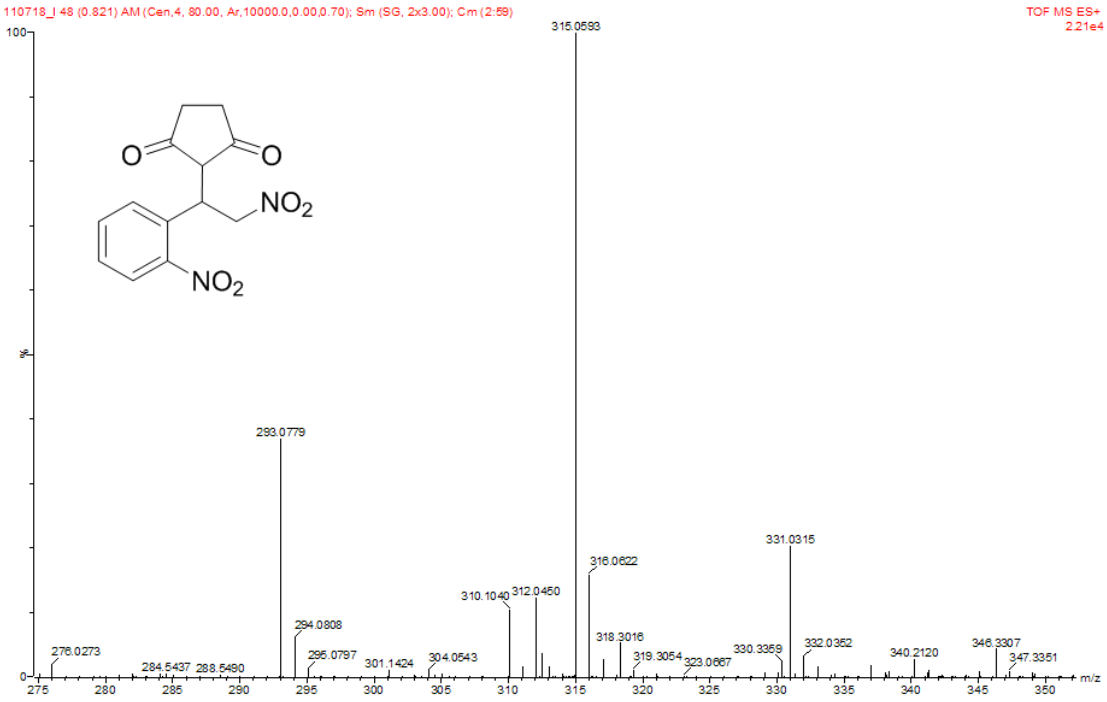

147.81
146.98
129.38
124.19
113.46
76.10
40.56
40.35
40.14
39.93
39.73
39.52
39.31
38.15
30.66
O
O
NO2
O2N
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


110718_H 37 (0.633) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.70); Sm (SG, 2x3.00); Cm (2:59)
TOF MS ES+
342
100
%
0
293.0774
315.0581
318.2964
310.1060
294.0807
316.0609
283.1261
319.0492
311.1095
319.3111
313.0974
320.3193
317.1288
280.0235
281.0661
284.5321
289.0815
290.0381
292.0088
295.0842
297.9510
300.0187
301.9917
303.0409
303.9636
306.0588
O
O
NO2
O2N
280
282
284
286
288
290
292
294
296
298
300
302
304
306
308
310
312
314
316
318
320
m/z

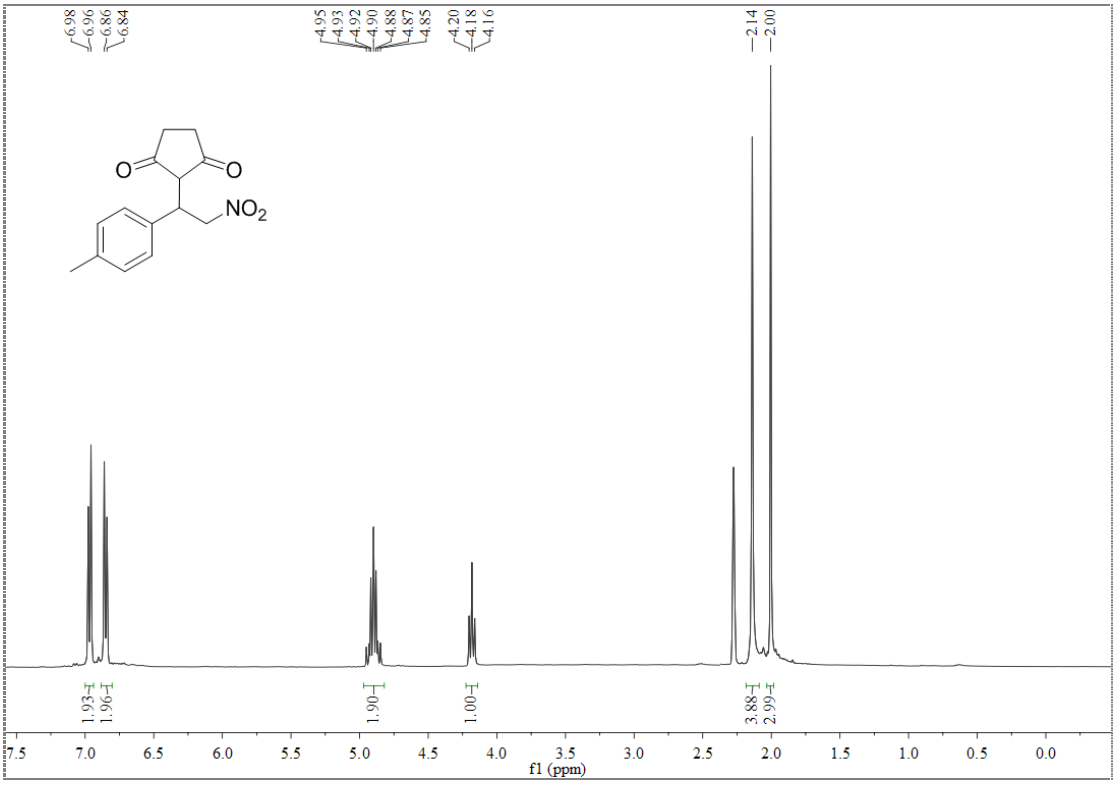
2-(2-Nitro-1-(2-nitrophenyl)ethyl)cyclopentane-1,3-dione (3i)

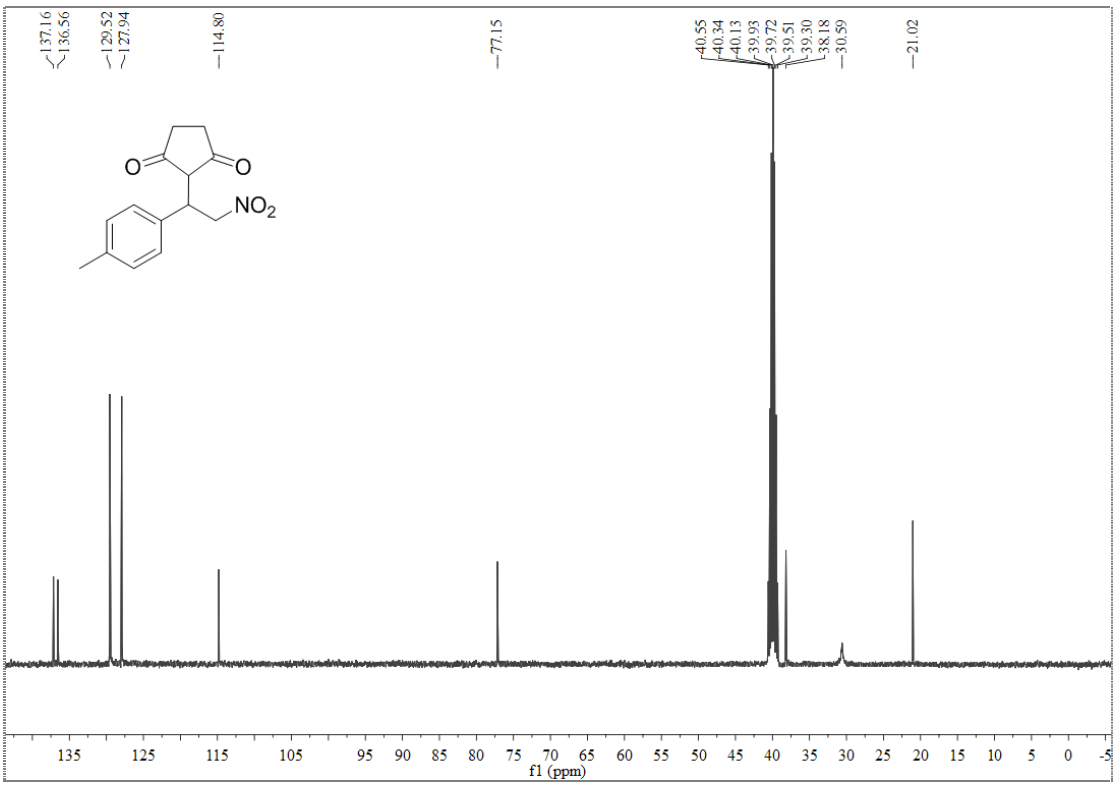


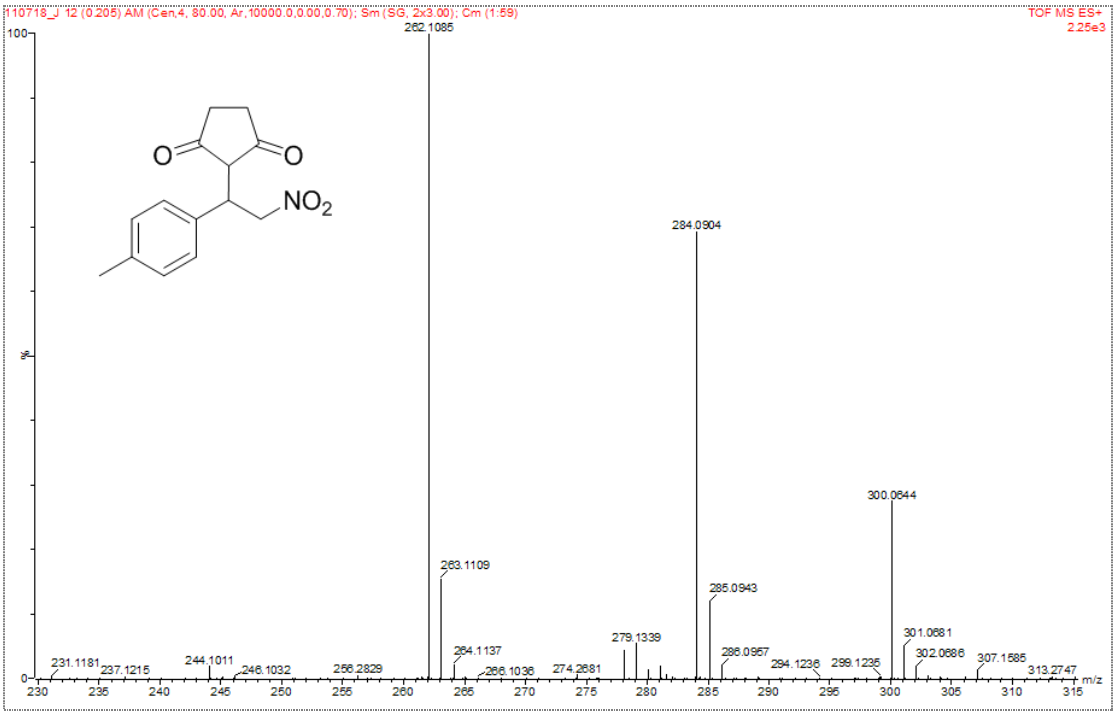




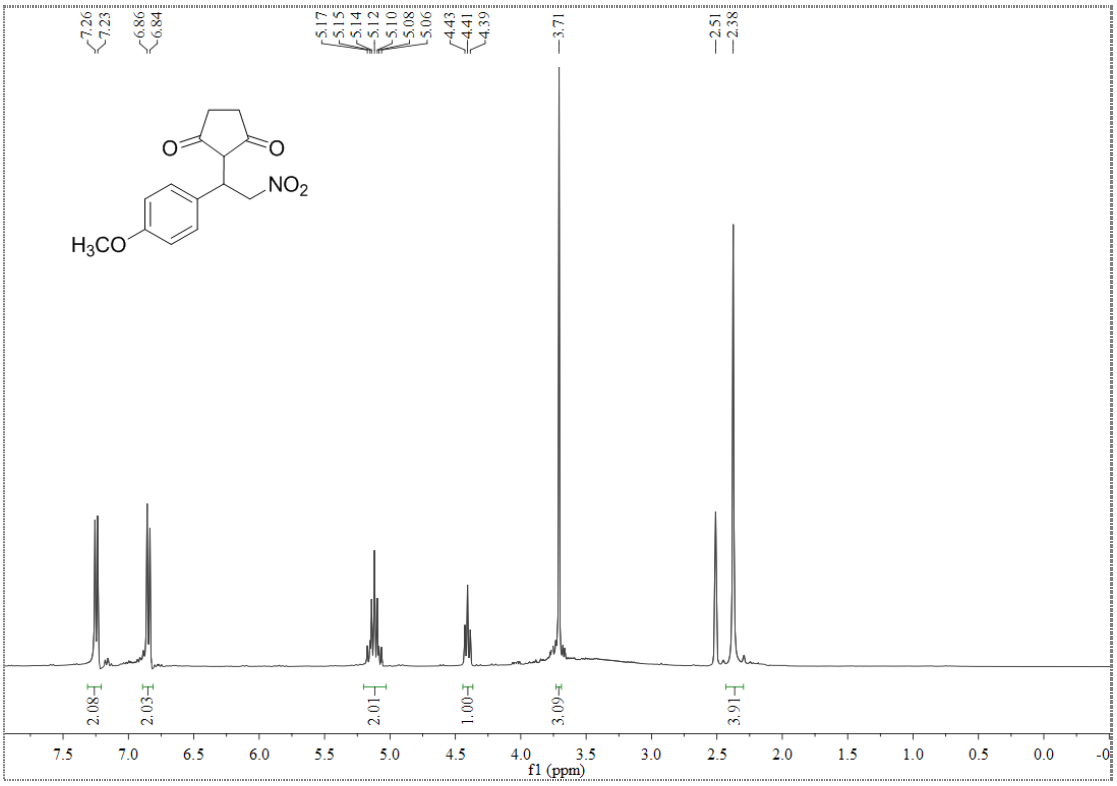
2-(2-Nitro-1-(p-tolyl)ethyl)cyclopentane-1,3-dione (3j)

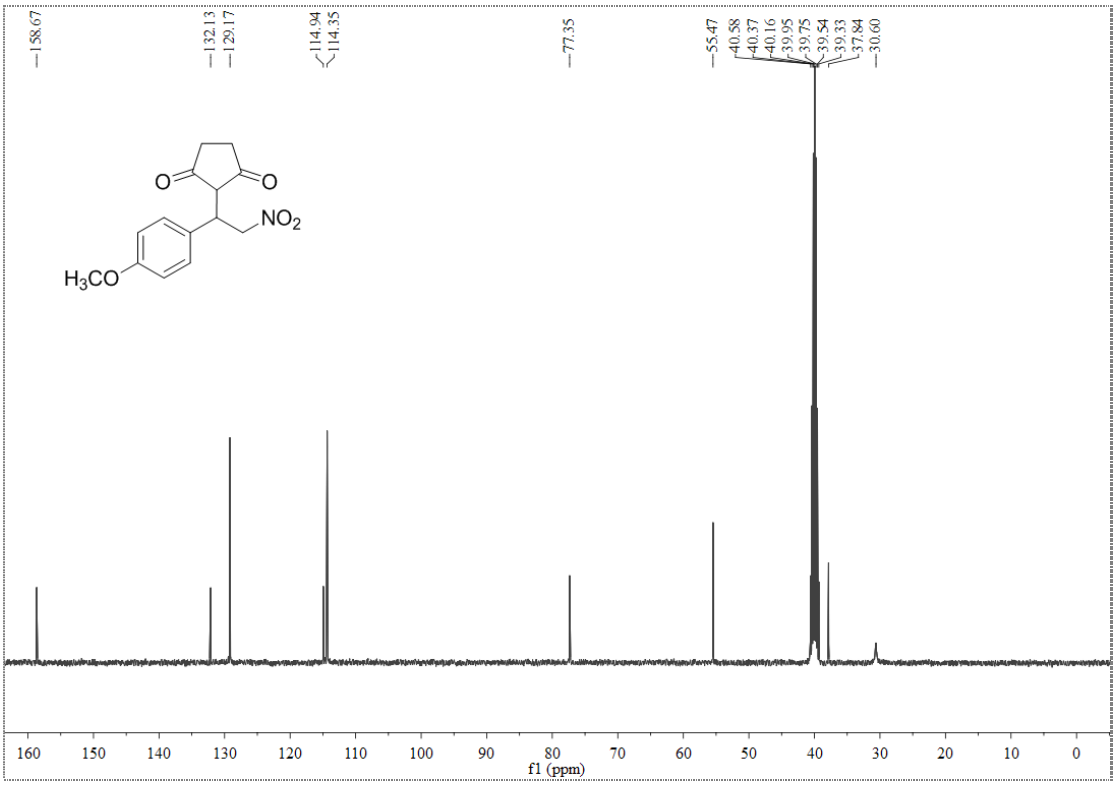


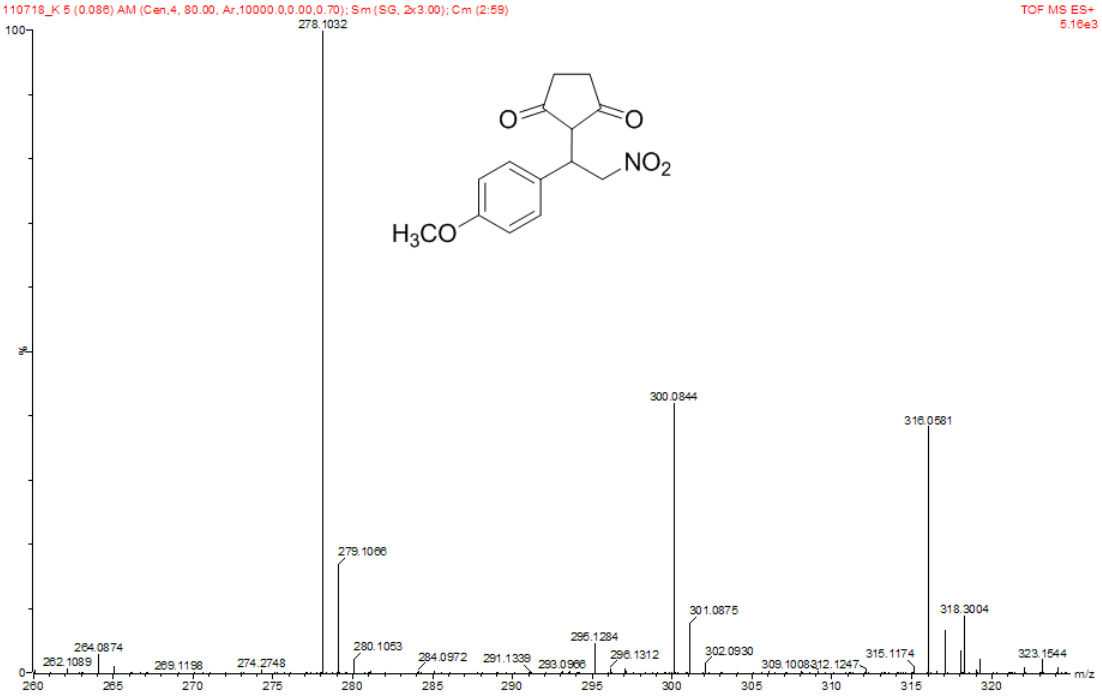

137.16
136.56
129.52
127.94
114.80
77.15
40.55
40.34
40.13
39.93
39.72
39.51
39.30
38.18
30.59
21.02
O
O
NO2
135
125
115
105
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
f1 (ppm)


110718_J 12 (0.205) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.70); Sm (SG, 2x3.00); Cm (1:59)
TOF MS ES+
2.25e3
100
%
0
262.1085
284.0904
300.0644
263.1109
285.0943
301.0681
279.1339
264.1137
286.0957
302.0686
307.1585
231.1181
237.1215
244.1011
246.1032
256.2829
266.1036
274.2681
294.1236
299.1235
313.2747
O
O
NO2
230
235
240
245
250
255
260
265
270
275
280
285
290
295
300
305
310
315
m/z

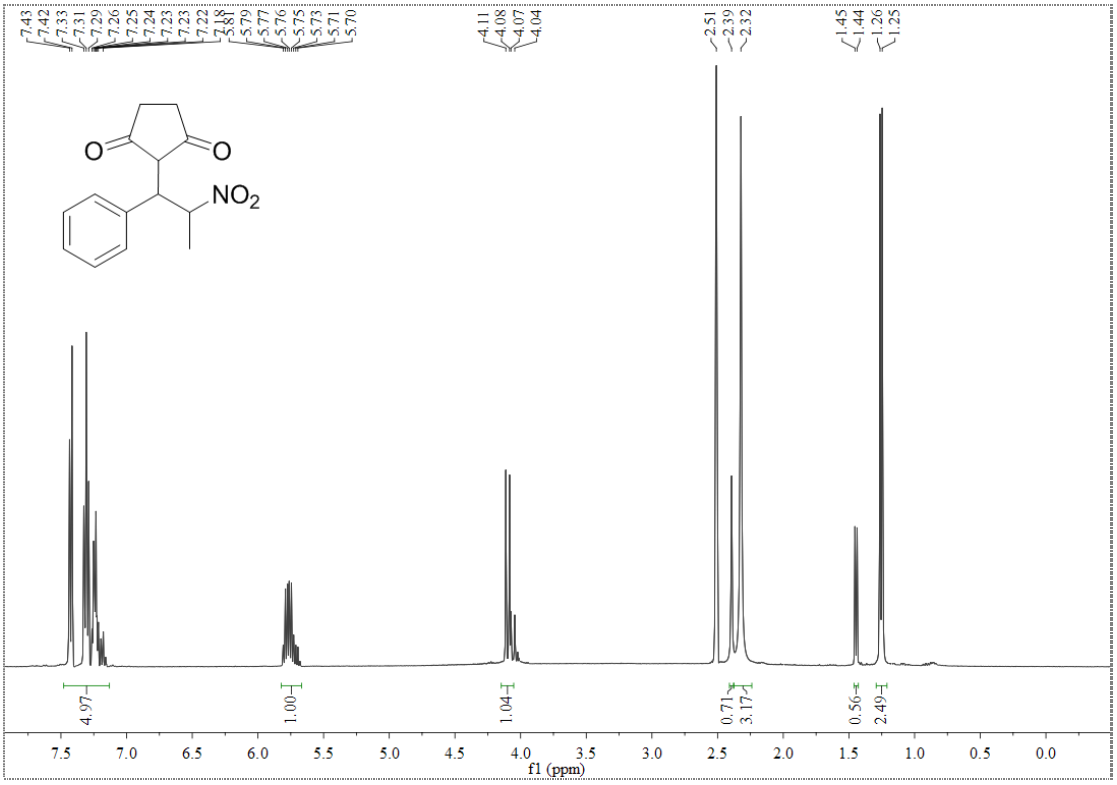
2-(1-(4-Methoxyphenyl)-2-nitroethyl)cyclopentane-1,3-dione (3k)

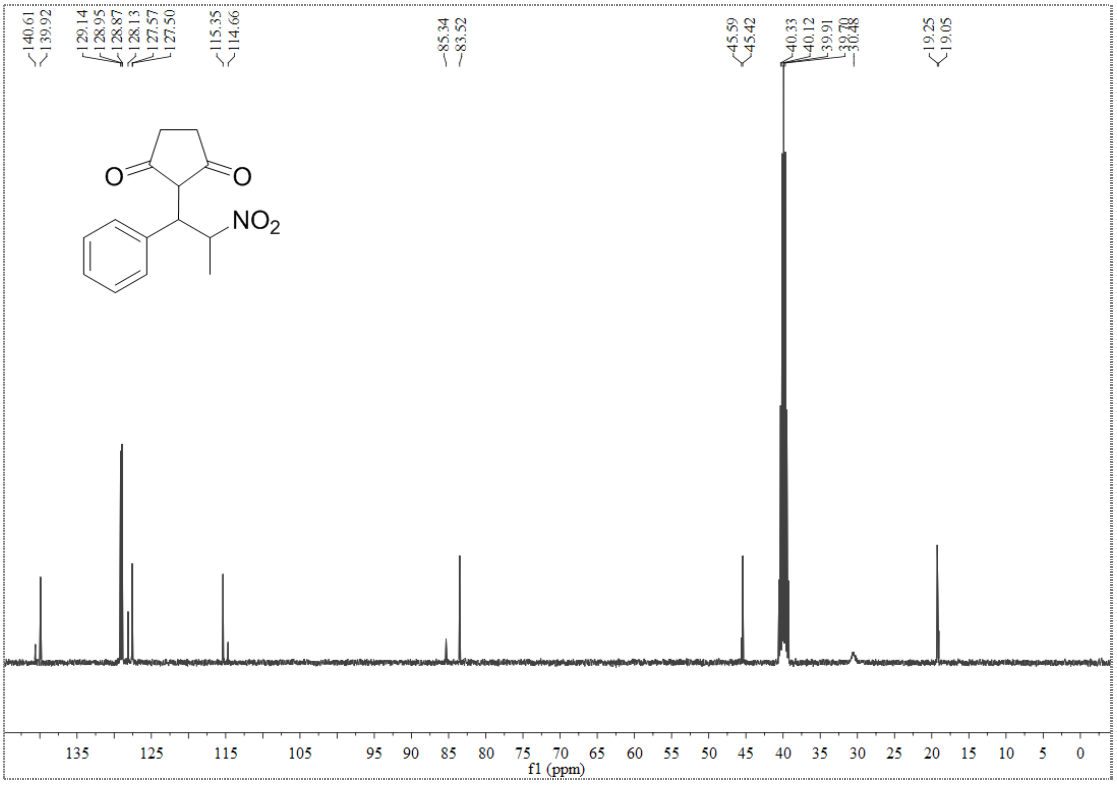


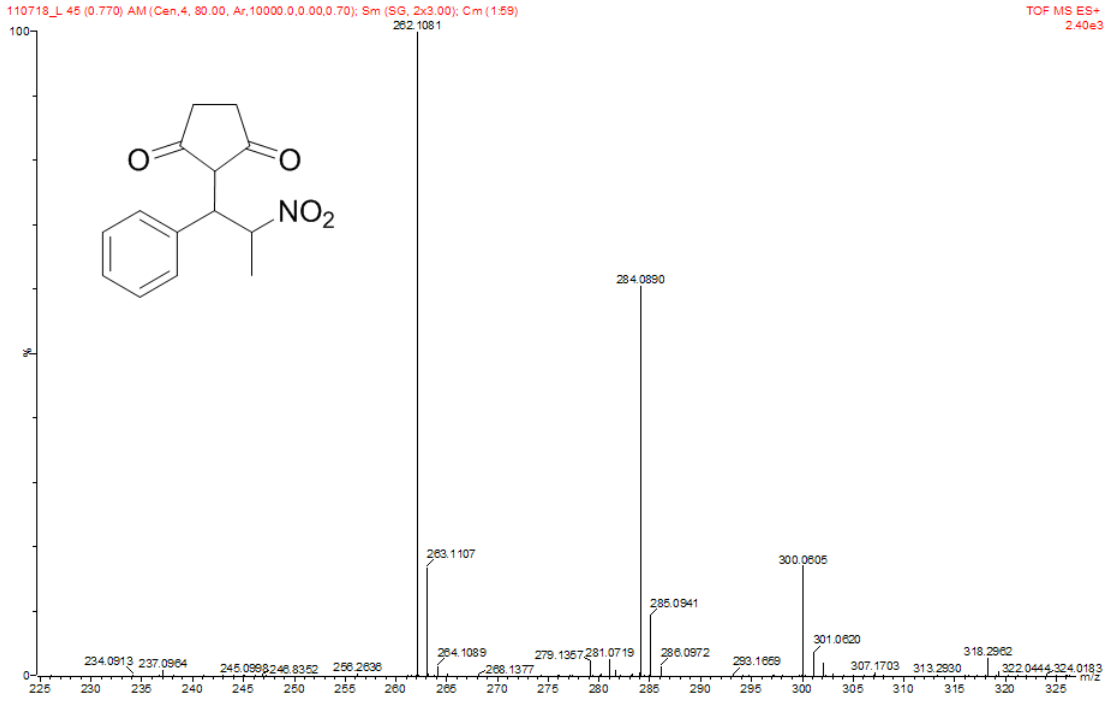




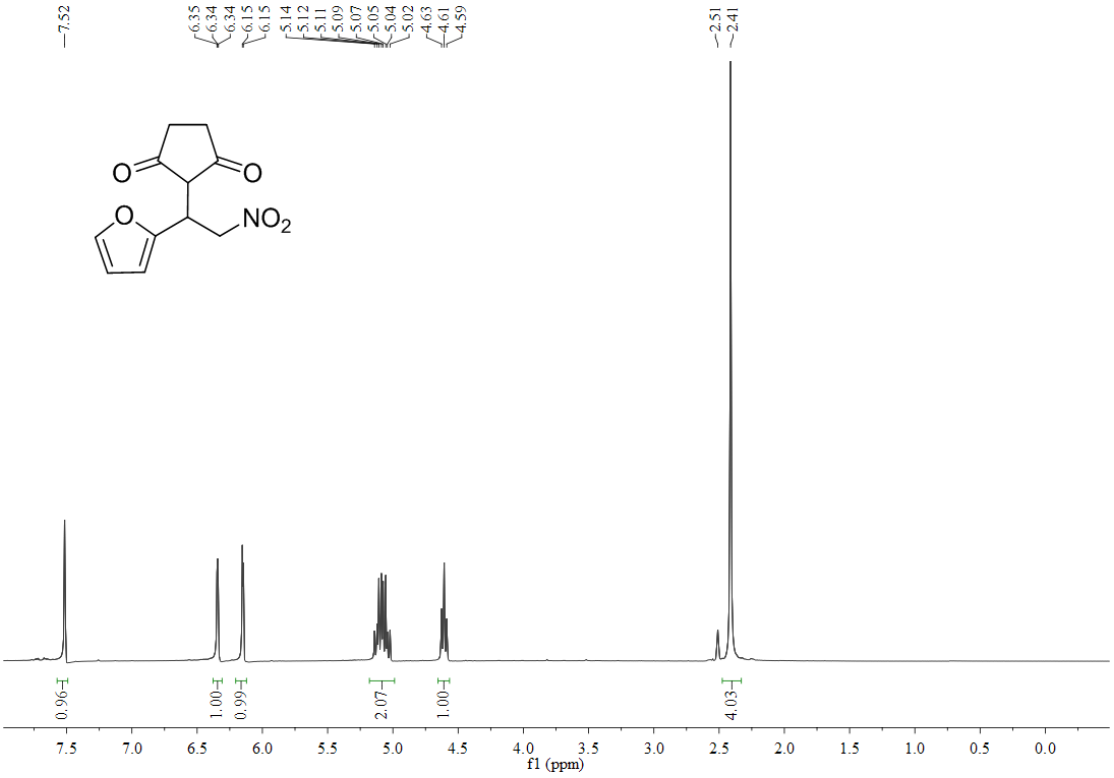
2-(2-Nitro-1-phenylpropyl)cyclopentane-1,3-dione (3l)

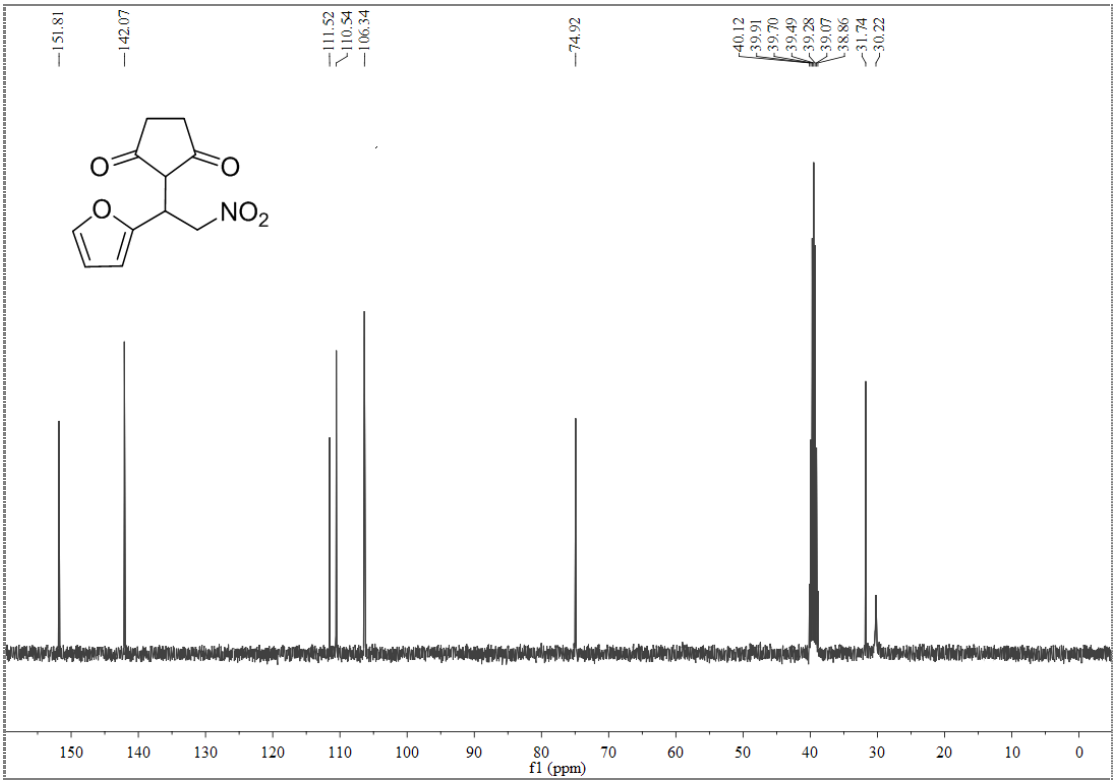


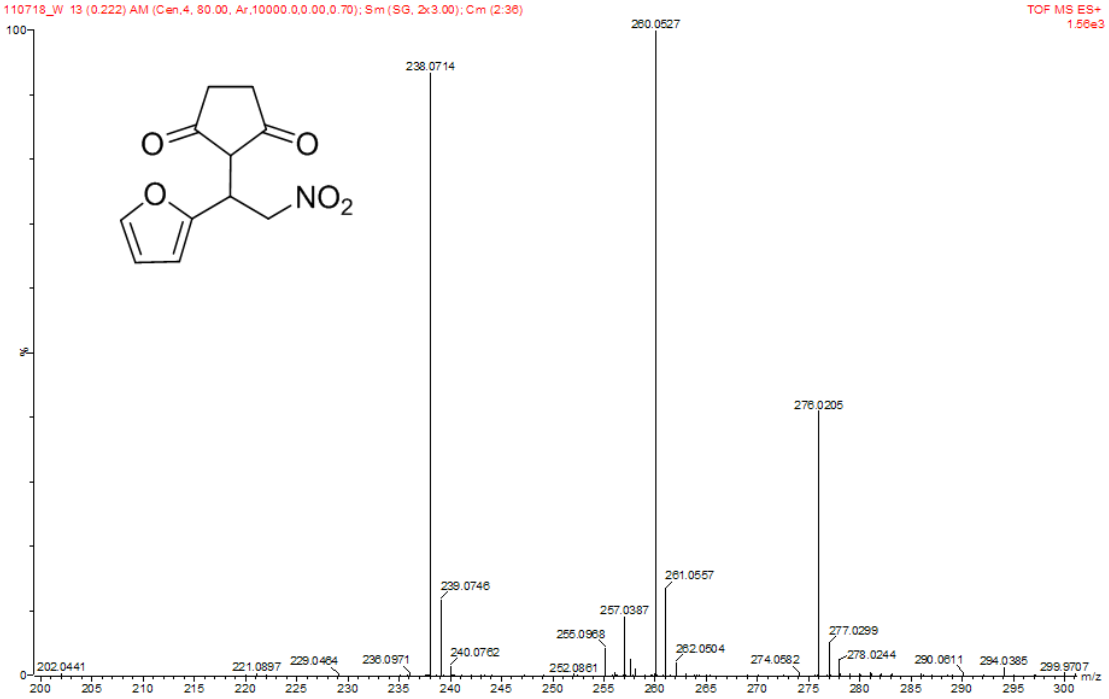

140.61
139.92
129.14
128.95
128.87
128.13
127.57
127.50
115.35
114.66
85.34
83.52
45.59
45.42
40.33
40.12
39.91
19.25
19.05
O
O
NO2
135
125
115
105
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
f1 (ppm)


110718_L 45 (0.770) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.70); Sm (SG, 2x3.00); Cm (1:59)
TOF MS ES+
2.40e3
100
%
0
262.1081
284.0890
263.1107
300.0605
285.0941
301.0620
318.2962
234.0913
237.0964
245.0998
246.8352
256.2636
264.1089
268.1377
279.1357
281.0719
286.0972
293.1669
307.1703
313.2930
322.0444
324.0183
O
O
NO2
225
230
235
240
245
250
255
260
265
270
275
280
285
290
295
300
305
310
315
320
325
m/z

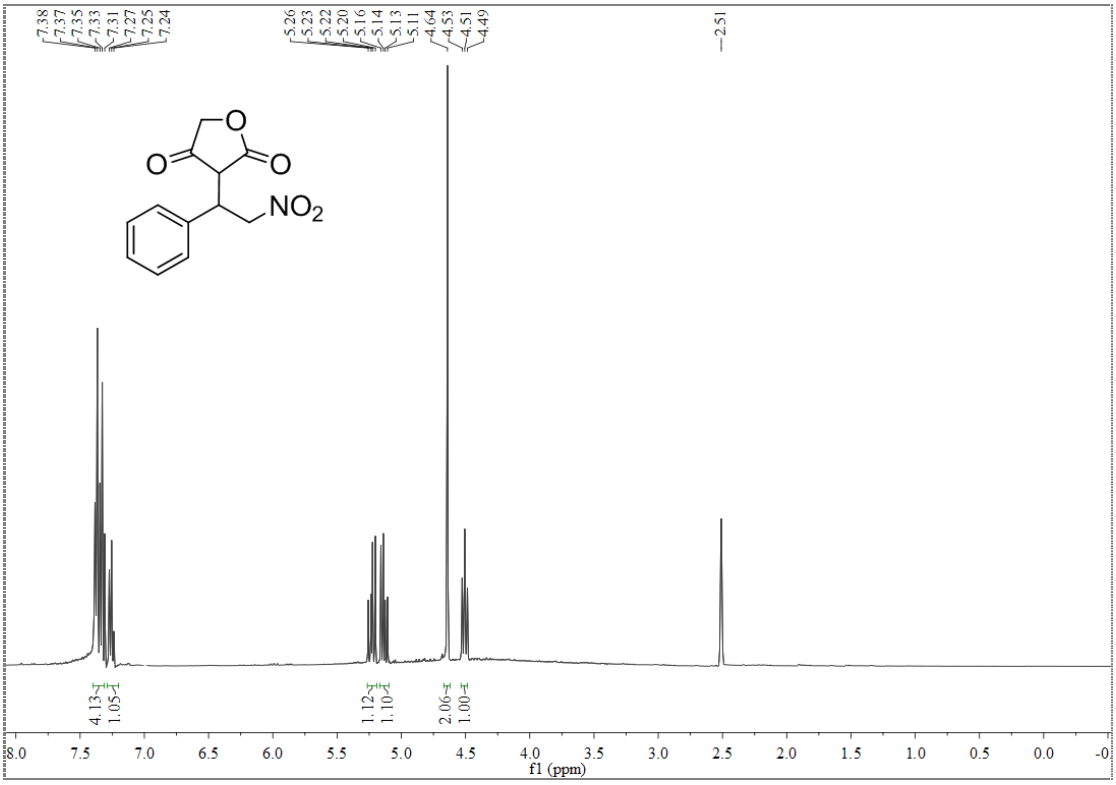
2-(1-(Furan-2-yl)-2-nitroethyl)cyclopentane-1,3-dione (3m)

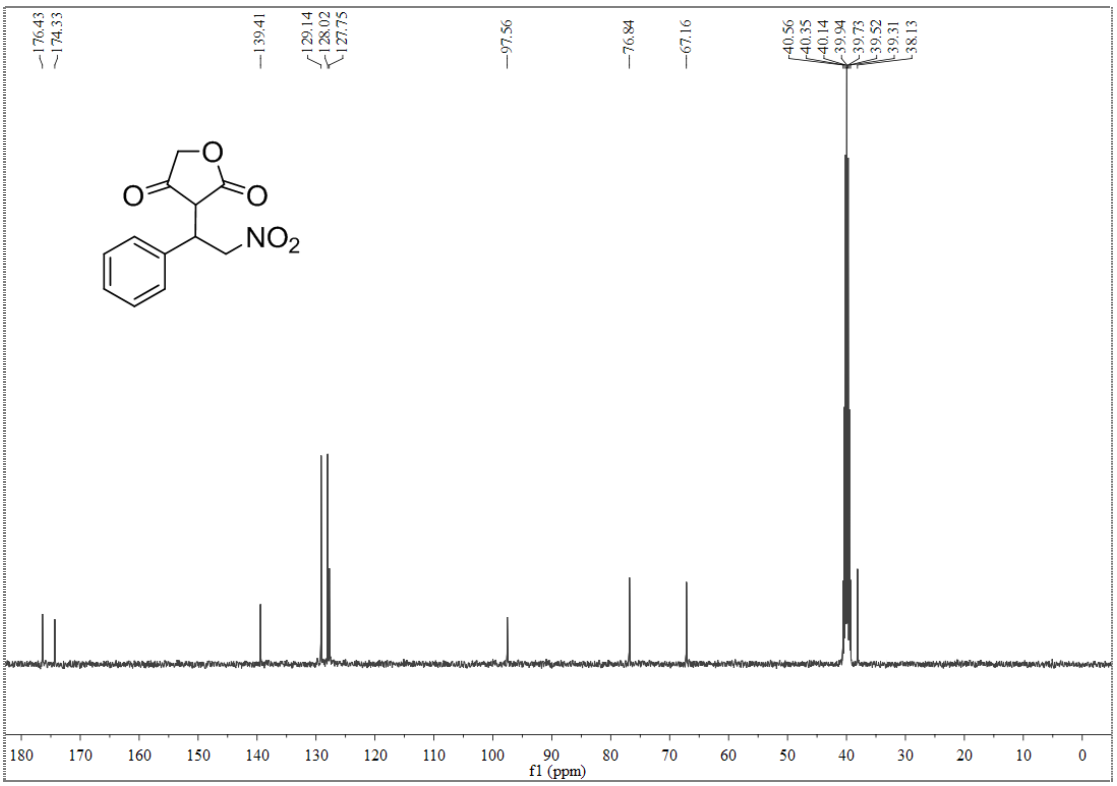


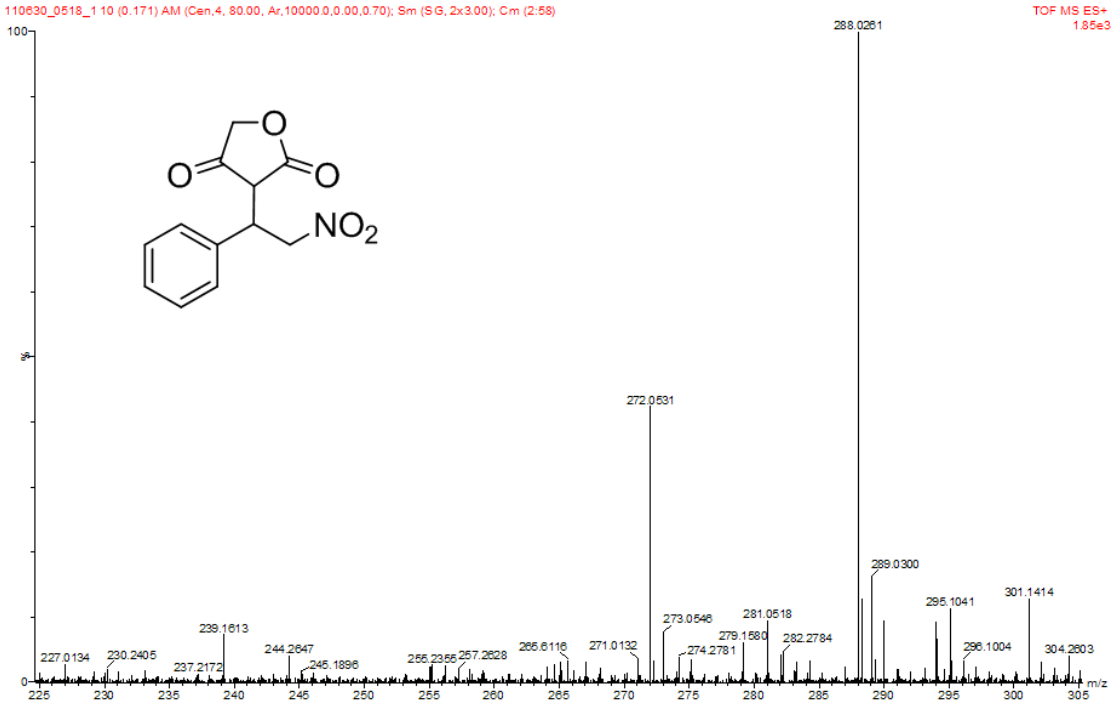




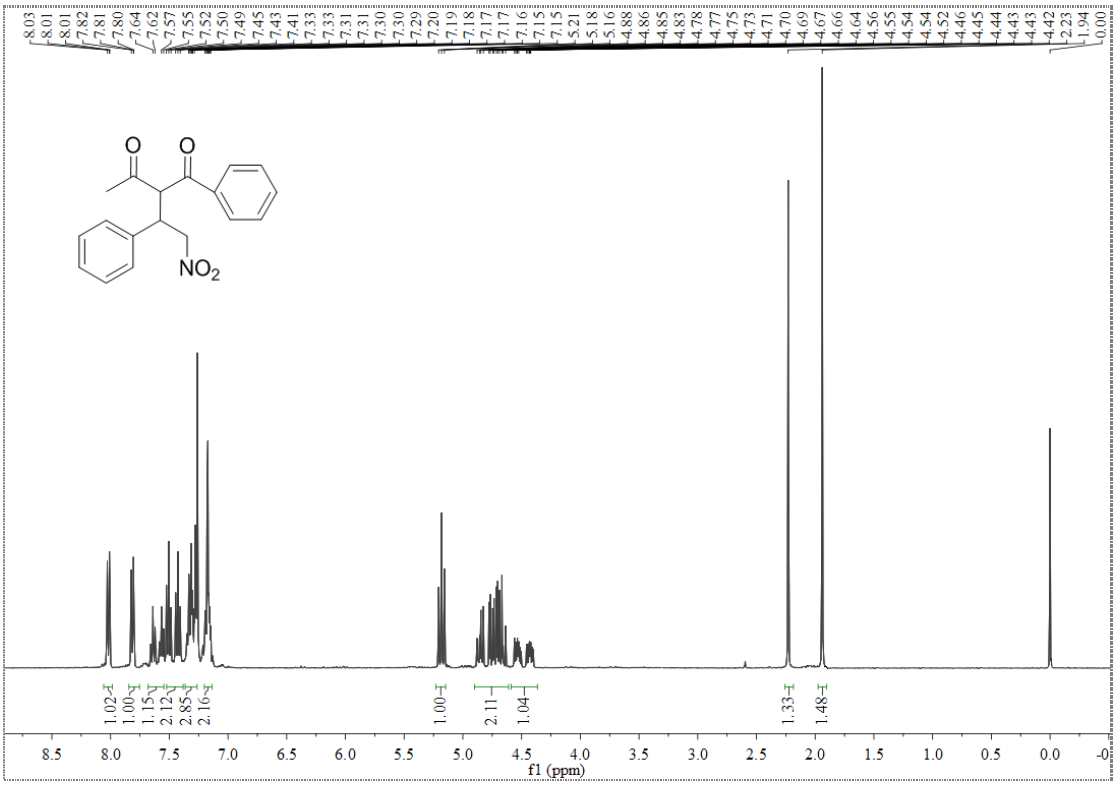
3-(2-Nitro-1-phenylethyl)furan-2,4(3H,5H)-dione (3n)

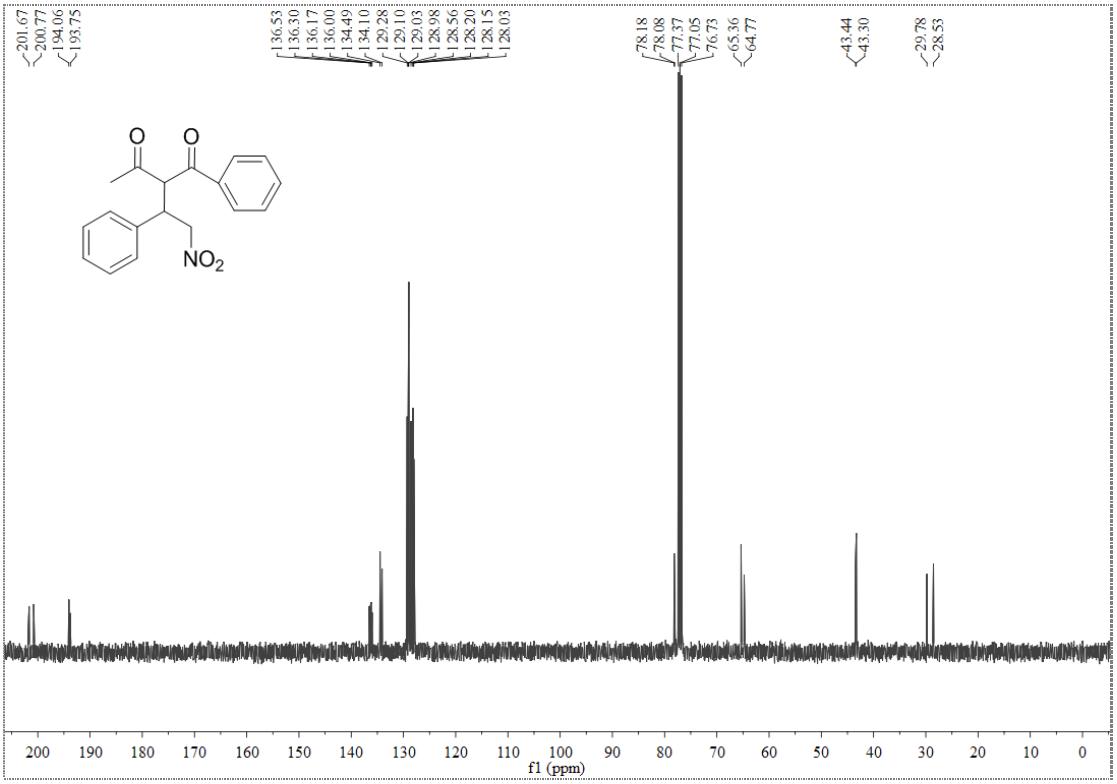


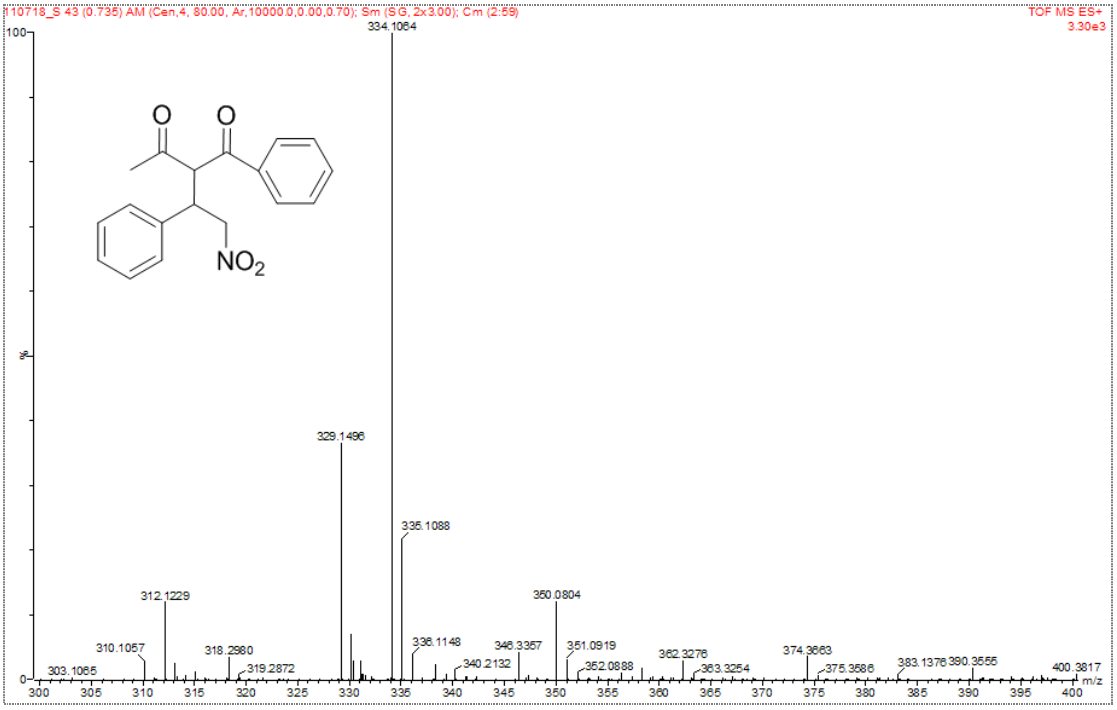

176.43
174.33
139.41
129.14
128.02
127.75
97.56
76.84
67.16
40.56
40.35
40.14
39.94
39.73
39.52
39.31
38.13
O
O
O
NO2
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


110630_0518_1 10 (0.171) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.70); Sm (SG, 2x3.00); Cm (2:58)
TOF MS ES+
1.85e3
100
%
0
O
O
O
NO2
288.0261
272.0531
289.0300
301.1414
295.1041
239.1613
273.0546
281.0518
279.1580
282.2784
244.2647
274.2781
296.1004
304.2603
227.0134
230.2405
237.2172
245.1896
255.2356
257.2628
265.6116
271.0132
225 230 235 240 245 250 255 260 265 270 275 280 285 290 295 300 305
m/z

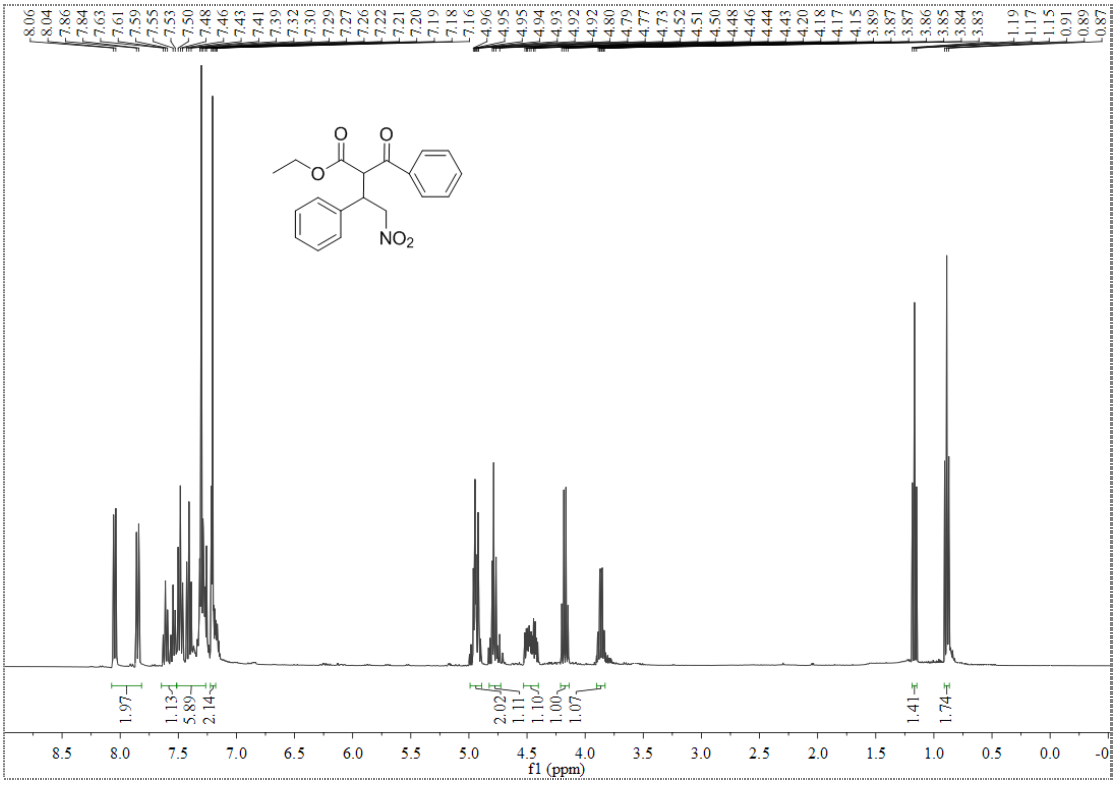
2-(2-Nitro-1-phenylethyl)-1-phenylbutane-1,3-dione (3o)

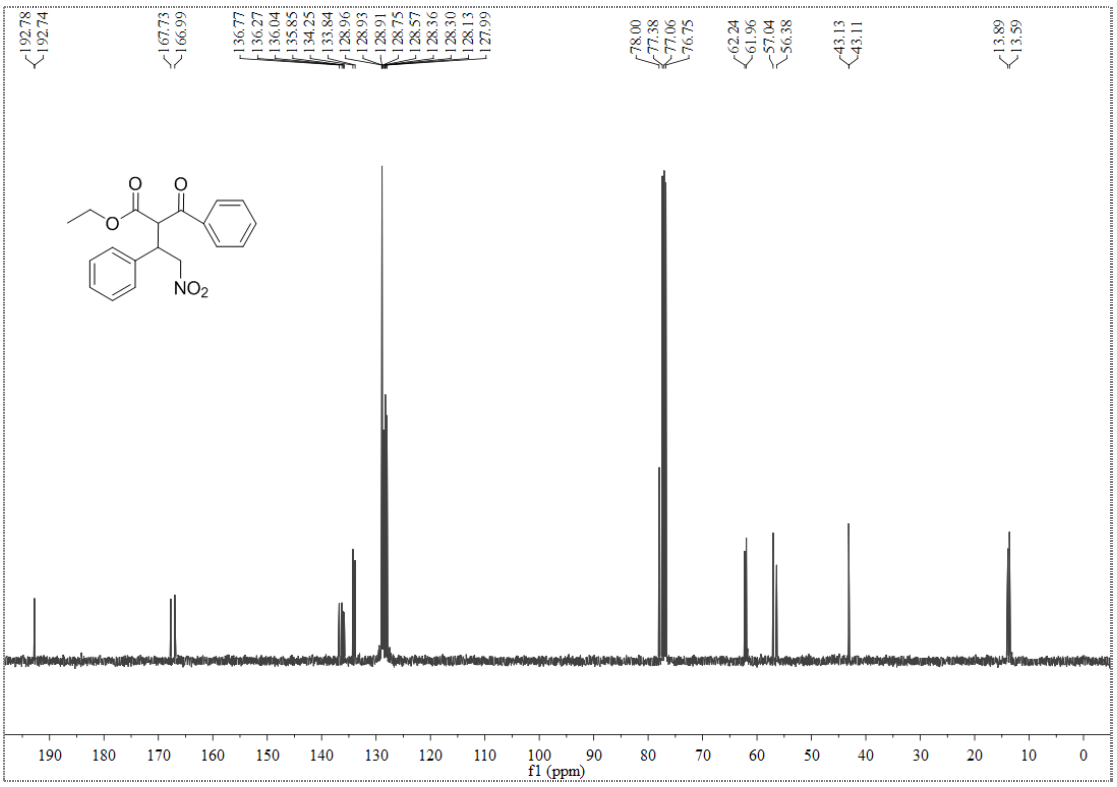


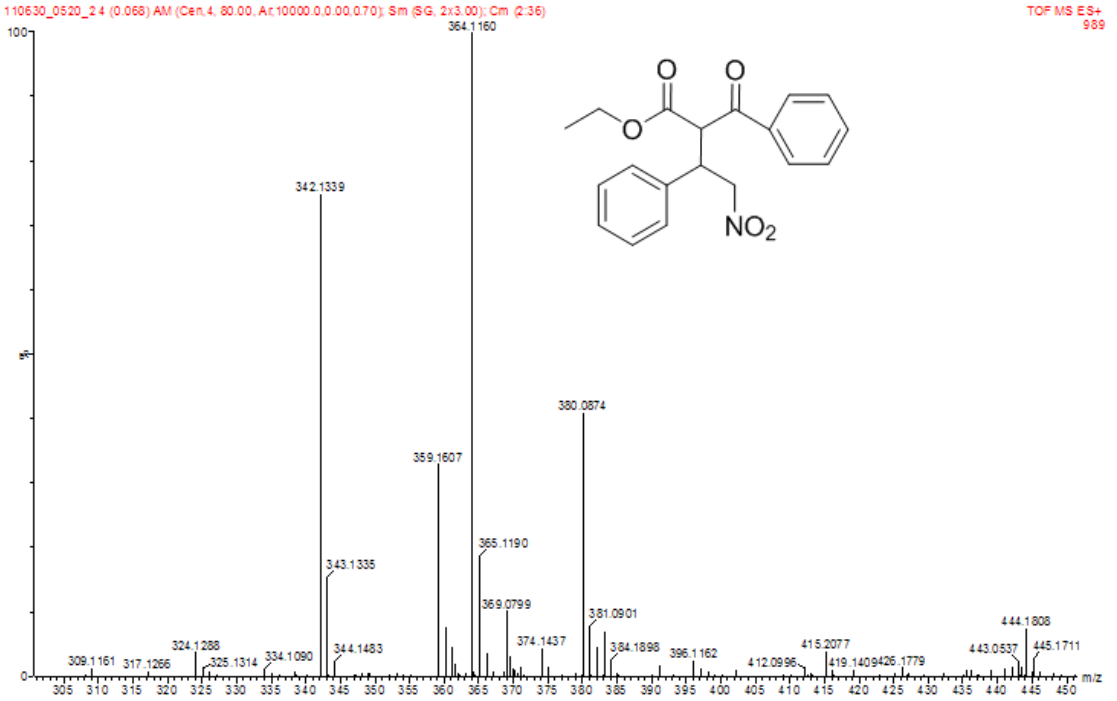




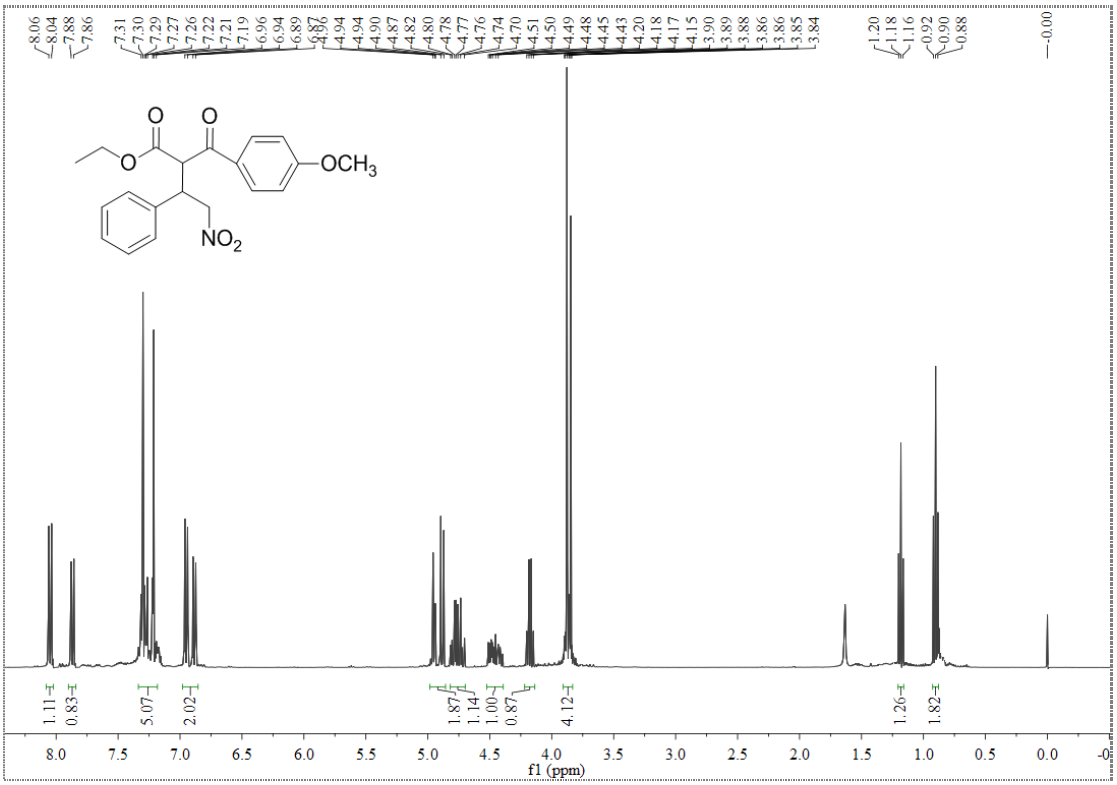
Ethyl 2-benzoyl-4-nitro-3-phenylbutanoate (3p)

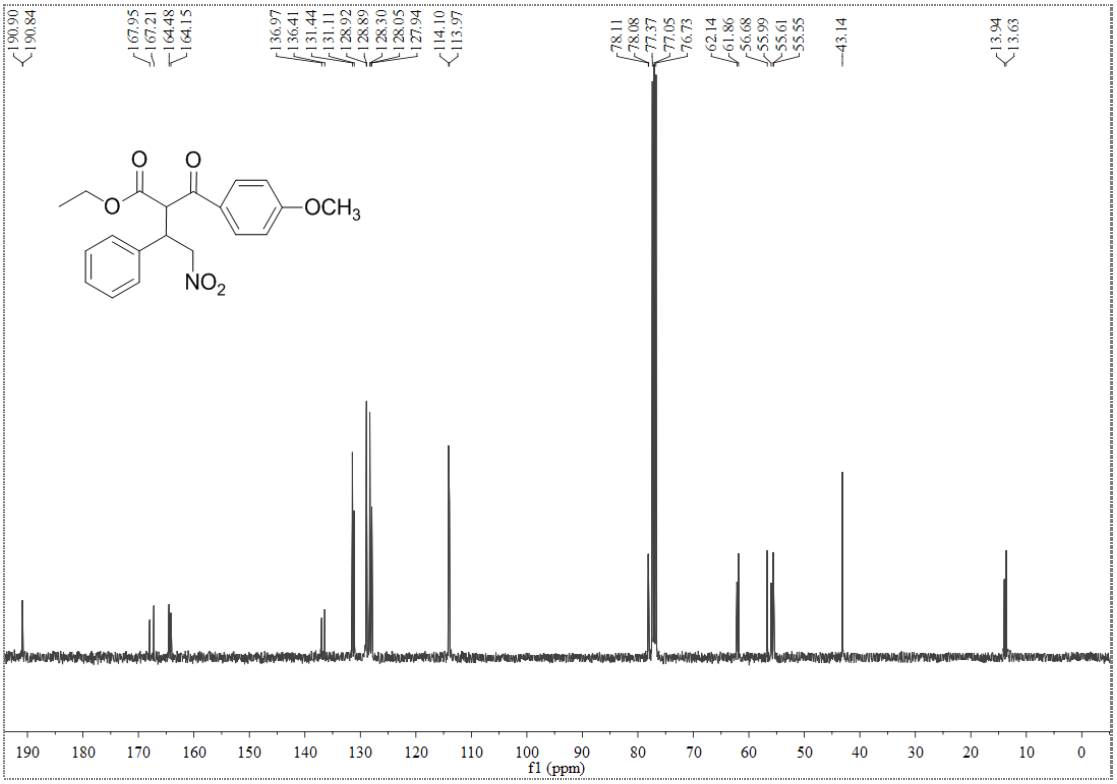


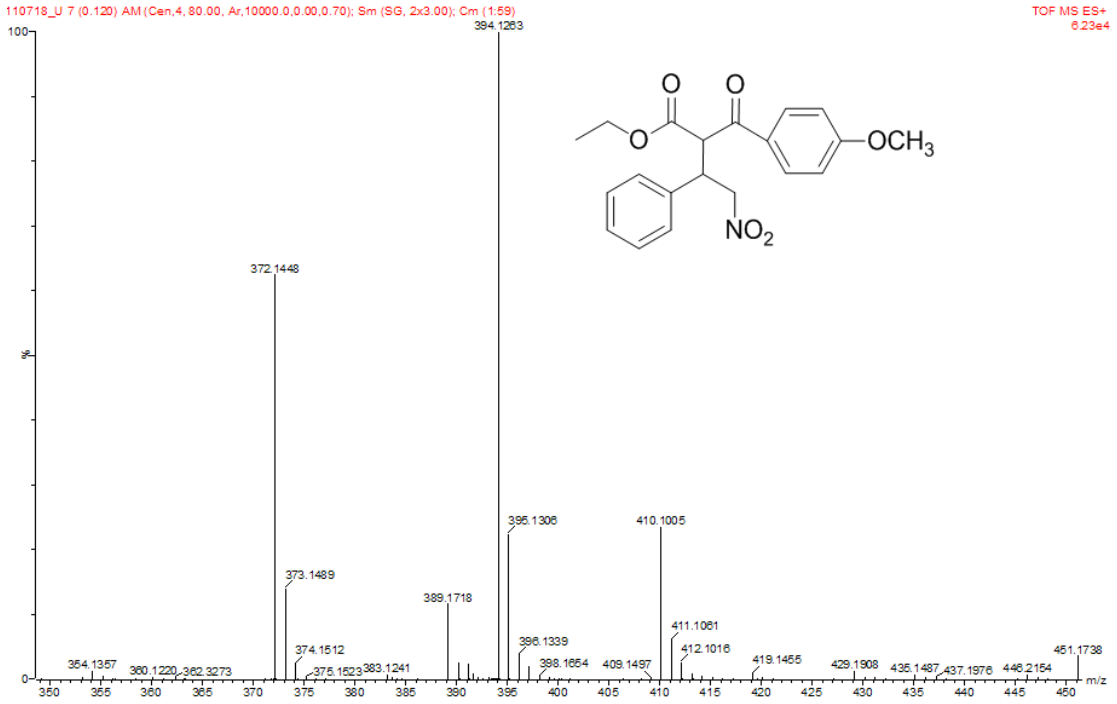

192.78
192.74
167.73
166.99
136.77
136.27
136.04
135.85
134.25
133.84
128.96
128.93
128.91
128.75
128.57
128.36
128.30
128.13
127.99
78.00
77.38
77.06
76.75
62.24
61.96
57.04
56.38
43.13
43.11
13.89
13.59
O
O
O
NO2
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


110630_0520_24 (0.068) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.70); Sm (SG, 2x3.00); Cm (2:36)
TOF MS ES+
989
100
%
0
364.1160
342.1339
380.0874
359.1607
365.1190
343.1335
369.0799
381.0901
374.1437
384.1898
396.1162
324.1288
309.1161
317.1266
325.1314
334.1090
344.1483
412.0996
415.2077
419.1409
426.1779
443.0537
444.1808
445.1711
O
O
O
NO2
305 310 315 320 325 330 335 340 345 350 355 360 365 370 375 380 385 390 395 400 405 410 415 420 425 430 435 440 445 450
m/z

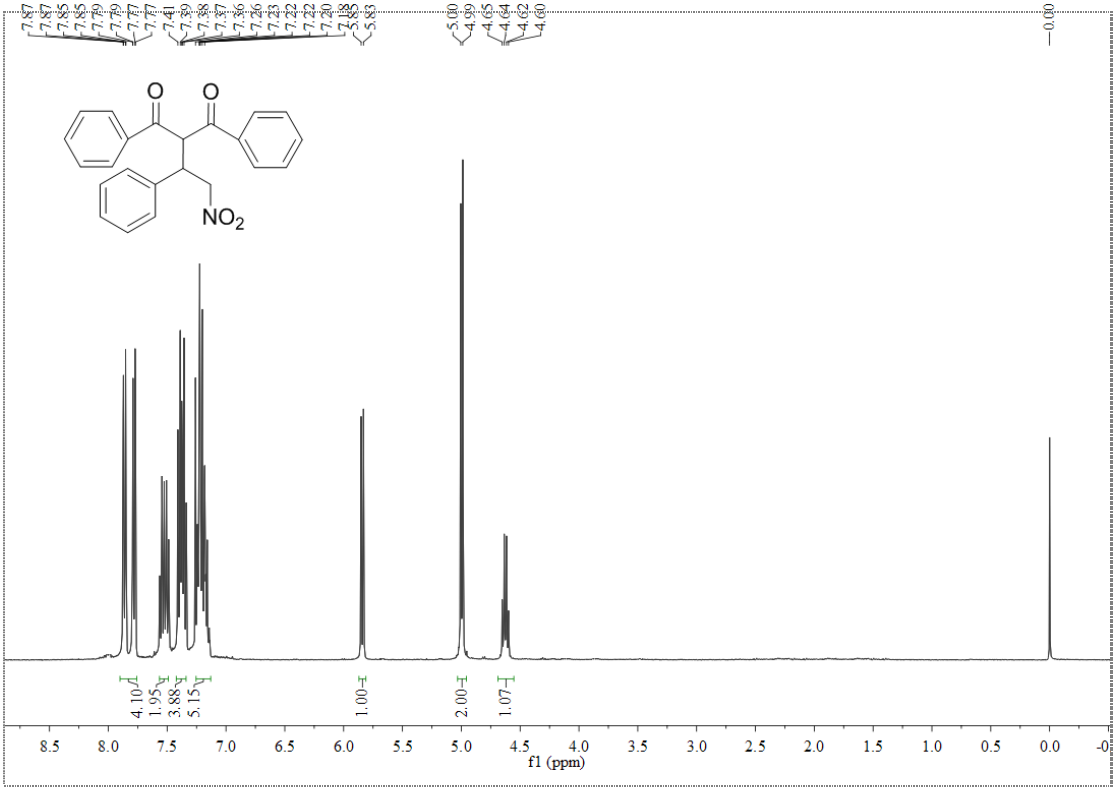
Ethyl 2-(4-methoxybenzoyl)-4-nitro-3-phenylbutanoate (3q)

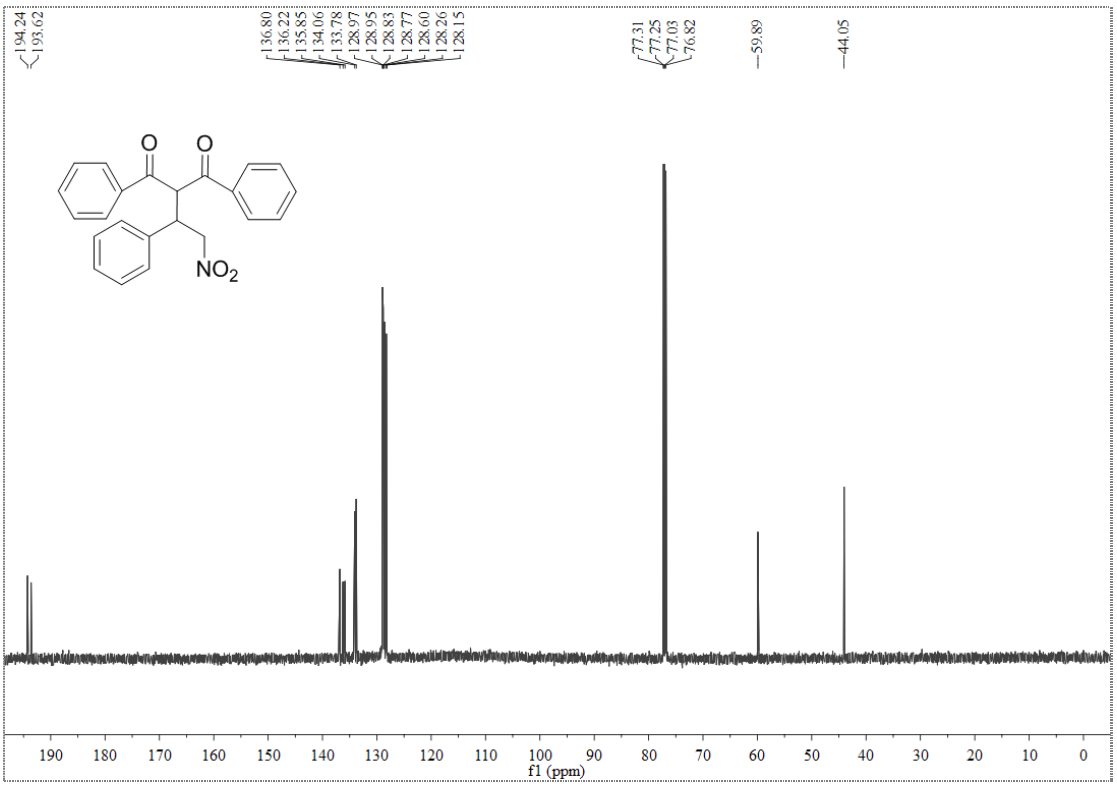


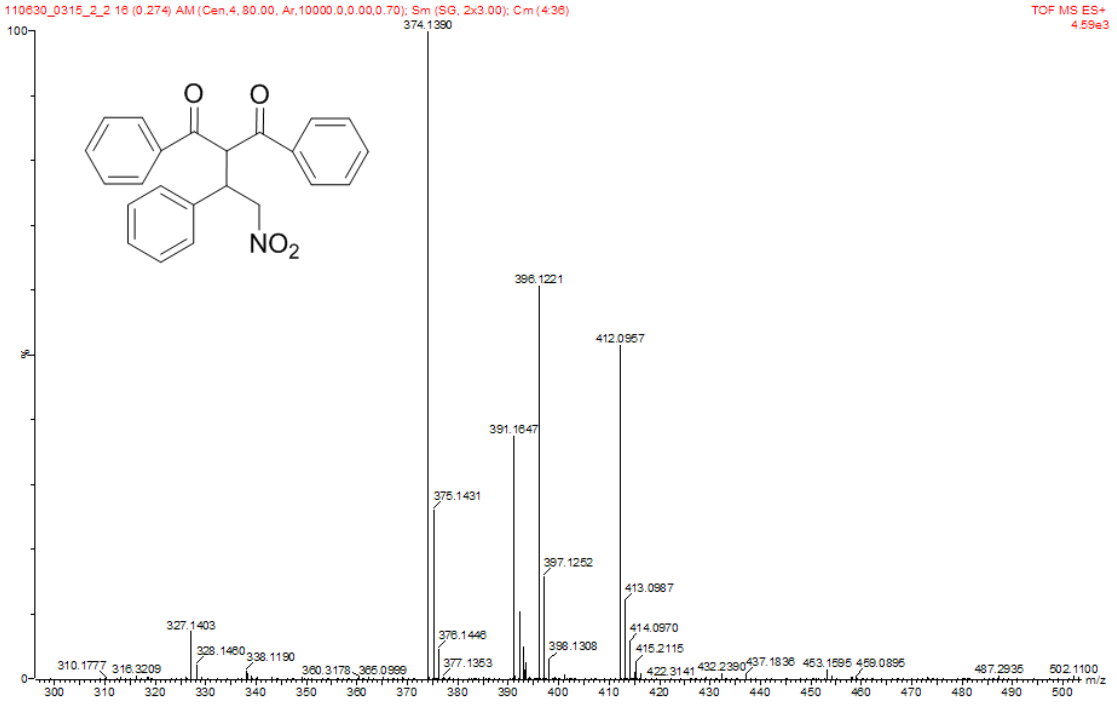




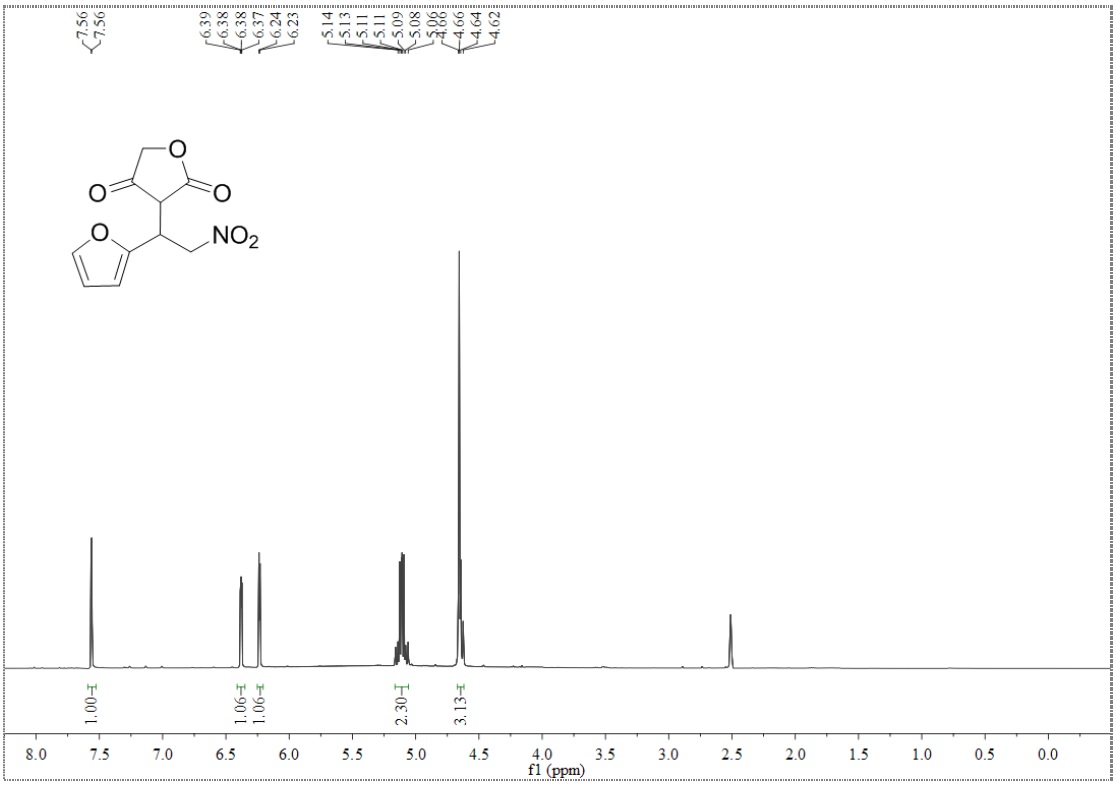
2-(2-Nitro-1-phenylethyl)-1,3-diphenylpropane-1,3-dione (3r)

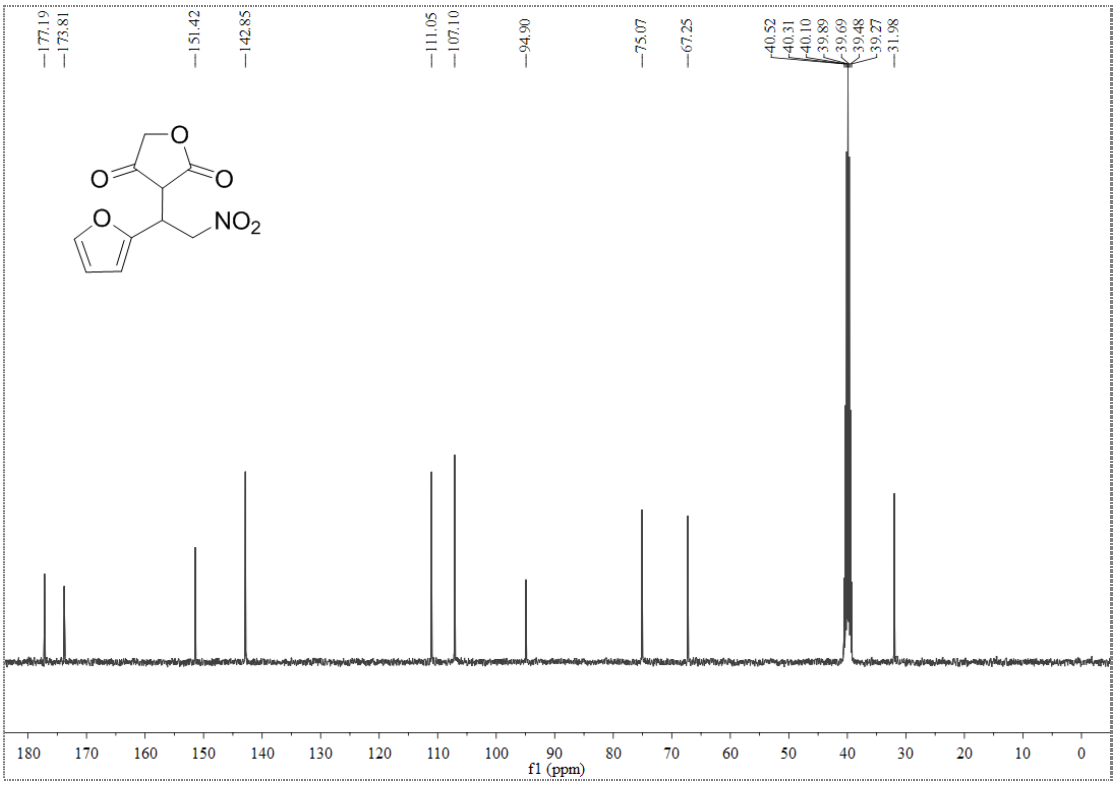


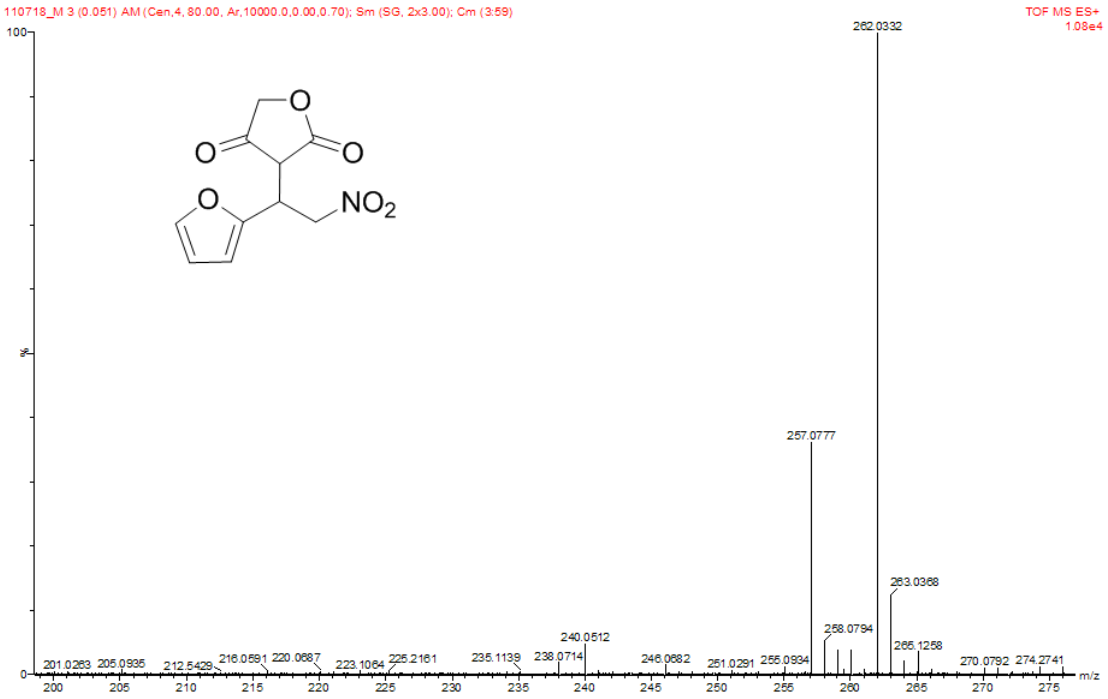

194.24
193.62
136.80
136.22
135.85
134.06
133.78
128.97
128.95
128.83
128.77
128.60
128.26
128.15
77.31
77.25
77.03
76.82
59.89
44.05
O
O
NO2
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


110630_0315_2_2 16 (0.274) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.70); Sm (SG, 2x3.00); Cm (4:36)
TOF MS ES+
4.59e3
100
%
0
374.1390
396.1221
412.0957
391.1647
375.1431
397.1252
413.0987
327.1403
376.1446
414.0970
328.1460
398.1308
338.1190
415.2115
310.1777
316.3209
360.3178
365.0999
377.1353
422.3141
432.2390
437.1836
453.1595
469.0895
487.2935
502.1100
m/z
300
310
320
330
340
350
360
370
380
390
400
410
420
430
440
450
460
470
480
490
500
O
O
NO2

3-(1-(Furan-2-yl)-2-nitroethyl)furan-2,4(3H,5H)-dione (3s)






110718_M 3 (0.051) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.70); Sm (SG, 2x3.00); Cm (3:59)
TOF MS ES+
1.08e4
100
%
0
O
O
O
O
NO2
262.0332
257.0777
263.0368
258.0794
265.1258
240.0512
201.0263
205.0935
212.5429
216.0591
220.0687
223.1064
225.2161
235.1139
238.0714
246.0682
251.0291
255.0934
270.0792
274.2741
200
205
210
215
220
225
230
235
240
245
250
255
260
265
270
275
m/z