

Three-Component Reaction between Substituted β -Nitrostyrenes, β -Dicarbonyl Compounds and Amines: Diversity-Oriented Synthesis of Novel β -Enaminones

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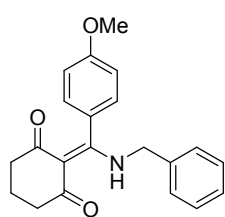
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Experimental section

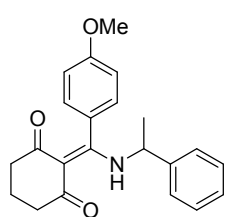
All melting points were determined in a Yanaco melting point apparatus and are uncorrected. IR spectra were recorded in a Nicolet FT-IR 5DX spectrometer. The ^1H NMR (600 MHz) and ^{13}C NMR (150 MHz) spectra were recorded in a Bruker AV-600 spectrometer with TMS as internal reference in CDCl_3 solutions. The J values are given in hertz. Only discrete or characteristic signals for the ^1H NMR are reported. The MS spectra were obtained on a ZAB-HS mass spectrometer with 70 eV. High-resolution ESI mass spectra were obtained on a UHR-TOF maXis (ESI) mass spectrometer. X-ray crystallographic analysis was performed with a SMART APEX-II diffractometer. Flash chromatography was performed on silica gel (230-400 mesh) eluting with ethyl acetate-hexanes mixture. All reactions were monitored by thin layer chromatography (TLC). All reagents and solvents were purchased from commercial sources and purified commonly before used.

General procedure for preparation of β -enaminones

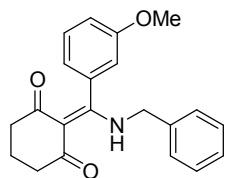
The standard procedure for the synthesis of β -enaminones via three-component reaction between substituted β -nitrostyrenes, β -dicarbonyl compounds and amines is as follows. To the mixture of substituted β -nitrostyrenes (3 mmol), β -dicarbonyl compound (3 mmol) and amine (3 mmol) in ethanol (15 mL) was added triethylamine (6mmol, 0.606g). The resulting mixture was stirred at 65 °C for 6-7 h, and the completion of reaction was confirmed by TLC (Hexanes/EtOAc, 5:1). Subsequently, the solvent was removed by reduced pressure, the residues was added with water (10 mL) and was extracted with dichloromethane (10 mL X 2). The organic phase was washed with water (10 mL) and brine (5 mL), and dried over anhydrate sodium sulfate. After removal of dichloromethane, the crude product was purified by flash chromatography (silica gel, EtOAc/hexanes, 1/8) to give the desirable products,

2-((Benzylamino)(4-methoxyphenyl)methylene)cyclohexane-1,3-dione(4a)

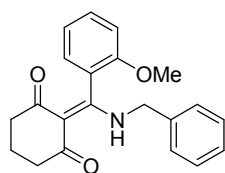
Mp 127.8-128.0 °C (MeOH/ CH₂Cl₂); ¹H-NMR (CDCl₃, 600 MHz) δ (ppm): 13.59 (s, 1H), 7.32 (t, *J* = 7.2 Hz, 2H), 7.28 (t, *J* = 7.2 Hz, 1H), 7.14 (d, *J* = 7.2 Hz, 2H), 7.07(d, *J* = 9.0 Hz, 2H), 6.95 (d, *J* = 8.4 Hz, 2H), 4.31 (d, *J* = 6.0 Hz, 2H), 3.84 (s, 3H), 2.57 (s, 2H), 2.36 (s, 2H), 1.95-1.91 (m, 2H); ¹³C-NMR (CDCl₃, 150 MHz) δ (ppm): 172.75(2C), 160.07, 136.40, 128.95(2C), 127.92,127.72(2C), 127.18(2C), 126.54, 114.12(2C), 109.24, 95.51, 55.27, 48.30, 39.22, 39.10, 19.45; IR (KBr, cm⁻¹): 3216, 2959, 1649, 1556, 1456, 1330, 1245, 836; MS(EI) (m/z): 336.46 [(M+1)⁺] (100%); HRESIMS calcd for C₂₁H₂₁NO₃ (M+H)⁺ 336.1600; found 336.1592.

2-((4-Methoxyphenyl)(1-phenylethylamino)methylene)cyclohexane-1,3-dione(4b)

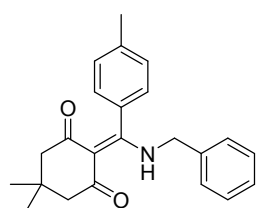
Liquid; ¹H-NMR (CDCl₃, 600 MHz) δ (ppm): 13.73 (s, 1H), 7.23 (t, *J* = 7.8 Hz, 2H), 7.18 (t, *J* = 7.2 Hz, 1H), 7.03 (d, *J* = 7.2 Hz, 2H), 6.88(d, *J* = 7.8 Hz, 2H), 6.83 (d, *J* = 8.4 Hz, 2H), 4.47-4.42 (m, 1H), 3.76 (s, 3H), 2.39 (t, *J* = 6.0 Hz, 4H), 1.87-1.83 (m, 2H), 1.41 (d, *J* = 7.2 Hz, 3H); ¹³C-NMR (CDCl₃, 150 MHz) δ (ppm): 166.30, 154.71, 137.02, 123.70, 123.67(2C), 122.43(2C), 121.50, 120.55(2C), 108.67(2C), 103.72, 49.99, 48.95, 33.69, 24.44, 18.78, 18.73, 14.20; IR (KBr, cm⁻¹): 3274, 2935, 2890, 1649, 1552, 1460, 1384, 1135, 852; MS(EI) (m/z): 350.52 [(M+1)⁺] (100%); HRESIMS calcd for C₂₂H₂₃NO₃ (M+H)⁺ 350.1756; found 350.1766.

2-((Benzylamino)(3-methoxyphenyl)methylene)cyclohexane-1,3-dione(4c)

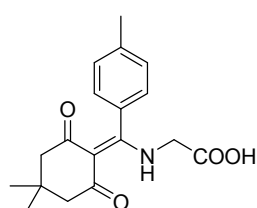
Mp 88.9-90.1 °C (MeOH/ CH₂Cl₂); ¹H-NMR (CDCl₃, 600 MHz) δ (ppm): 13.54 (s, 1H), 7.28 (d, *J* = 7.8 Hz, 1H), 7.25 (t, *J* = 8.4 Hz, 1H), 7.21 (d, *J* = 7.2 Hz, 2H), 7.06 (d, *J* = 7.2 Hz, 2H), 6.87 (dd, *J* = 8.4, 2.4 Hz, 1H), 6.65 (d, *J* = 7.8 Hz, 1H), 6.55 (t, *J* = 2.4 Hz, 1H), 4.19 (t, *J* = 6.0 Hz, 2H), 3.67 (s, 3H), 2.51 (s, 2H), 2.28 (s, 2H), 1.88-1.84 (m, 2H); ¹³C-NMR (CDCl₃, 150 MHz) δ (ppm): 172.31, 159.74, 136.40, 135.78, 129.90, 128.93, 127.93, 127.23, 118.15, 114.30, 111.63, 108.78, 55.17, 43.29, 19.45; IR (KBr, cm⁻¹): 3218, 2953, 1653, 1577, 1548, 1329, 1042, 834; MS(EI) (m/z): 336.48 [(M+1)⁺] (100%); HRESIMS calcd for C₂₁H₂₁NO₃ (M+H)⁺ 336.1600; found 336.1590.

2-((Benzylamino)(2-methoxyphenyl)methylene)cyclohexane-1,3-dione(4d)

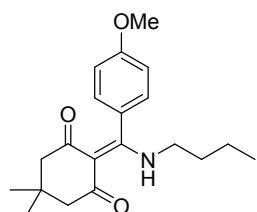
Mp 91.3-91.5 °C (MeOH/ CH₂Cl₂); ¹H-NMR (CDCl₃, 600 MHz) δ (ppm): 13.64 (s, 1H), 7.39 (t, *J* = 8.4 Hz, 1H), 7.30 (t, *J* = 7.2 Hz, 2H), 7.26 (t, *J* = 7.2 Hz, 1H), 7.14(d, *J* = 7.2 Hz, 2H), 6.99 (t, *J* = 7.2 Hz, 1H), 6.94 (d, *J* = 8.4 Hz, 2H), 4.29-4.19 (m, 2H), 3.74 (s, 3H), 2.58-2.55 (m, 2H), 2.35-2.32 (m, 2H), 1.94-1.89 (m, 2H); ¹³C-NMR (CDCl₃, 150 MHz) δ (ppm): 200.37, 195.34, 170.39, 155.52, 136.20, 130.34, 128.80, 127.84, 127.47, 126.76, 123.92, 120.95, 110.90, 109.09, 55.62, 48.27, 39.09, 38.65, 19.52; IR (KBr, cm⁻¹): 3211, 2944, 1647, 1578, 1504, 1423, 1248, 740; MS(EI) (m/z): 336.51 [(M+1)⁺] (35%); HRESIMS calcd for C₂₁H₂₁NO₃ (M+H)⁺ 336.1600; found 336.1589.

2-((Benzylamino)(p-tolyl)methylene)-5,5-dimethylcyclohexane-1,3-dione(4e)

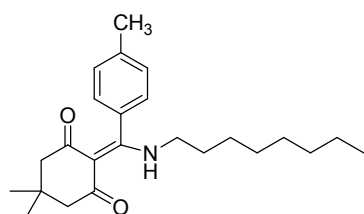
Mp 135.6-135.8 °C (MeOH/ CH₂Cl₂); ¹H-NMR (CDCl₃, 600 MHz) δ (ppm): 13.54 (s, 1H), 7.31 (t, *J* = 7.2 Hz, 2H), 7.27 (d, *J* = 7.2 Hz, 1H), 7.24 (d, *J* = 7.8 Hz, 2H), 7.14 (d, *J* = 7.2 Hz, 2H), 7.03 (d, *J* = 8.4 Hz, 2H), 4.27 (d, *J* = 6.0 Hz, 2H), 2.47 (s, 2H), 2.39 (s, 3H), 2.23 (s, 2H), 1.05 (s, 6H); ¹³C-NMR (CDCl₃, 150 MHz) δ (ppm): 199.72, 195.01, 172.40, 138.81, 136.36, 131.53, 129.39, 128.94, 127.92, 127.26, 125.99, 107.92, 52.02, 52.35, 48.27, 30.29, 28.44, 21.49; IR (KBr, cm⁻¹): 3157, 3057, 2956, 2868, 1654, 1552, 1448, 1412, 1242, 862; MS(EI) (*m/z*): 348.57 [(*M*+1)⁺] (100%); HRESIMS calcd for C₂₃H₂₅NO₂ (*M*+H)⁺ 348.1964; found 348.1951.

2-((4,4-Dimethyl-2,6-dioxocyclohexylidene)(p-tolyl)methylamino)acetic acid(4f)

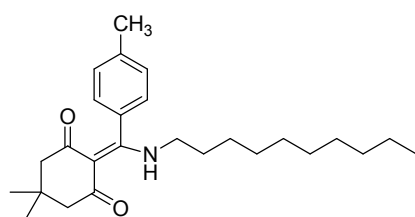
Mp 243.7-243.9 °C (MeOH/ CH₂Cl₂); ¹H-NMR (DMSO-*d*₆, 600 MHz) δ (ppm): 13.04 (t, *J* = 5.4 Hz, 1H), 7.21 (t, *J* = 7.8 Hz, 2H), 6.98 (d, *J* = 7.2 Hz, 2H), 3.78 (d, *J* = 5.4 Hz, 2H), 2.49-2.10 (m, 4H), 2.39 (s, 3H), 0.98 (s, 6H); ¹³C-NMR (DMSO-*d*₆, 150 MHz) δ (ppm): 176.09, 174.87(2C), 143.04, 136.94, 134.08(2C), 133.90, 131.33(2C), 112.72, 57.43, 50.91, 35.09(2C), 33.18(2C), 26.14; IR (KBr, cm⁻¹): 3427, 2963, 1730, 1615, 1541, 1304, 1229, 857; MS(EI) (*m/z*): 316.61 [(*M*+1)⁺] (100%); HRESIMS calcd for C₁₈H₂₁NO₄ (*M*+H)⁺ 316.1549; found 316.1548.

2-((butylamino)(4-methoxyphenyl)methylene)-5,5-dimethylcyclohexane-1,3-dione (4g)

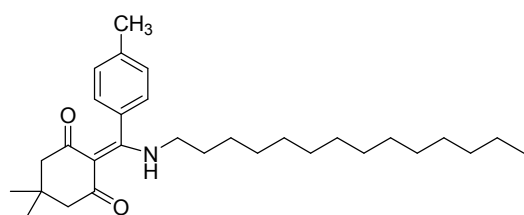
Mp 86.4-86.7 °C (MeOH/ CH₂Cl₂); ¹H-NMR (CDCl₃, 600 MHz) δ (ppm): 13.12 (s, 1H), 6.98 (t, *J* = 8.4 Hz, 2H), 6.89 (d, *J* = 7.8 Hz, 2H), 3.77 (s, 3H), 3.05-3.02 (m, 2H), 2.39 (s, 2H), 2.16 (s, 2H), 1.47-1.42 (m, 2H), 1.27-1.24 (m, 2H), 0.98 (s, 6H), 0.79 (t, *J* = 7.2 Hz, 3H); ¹³C-NMR (CDCl₃, 150 MHz) δ (ppm): 172.19(2C), 159.91, 128.02, 127.59(2C), 126.89(2C), 114.04, 107.71, 55.22, 53.02, 52.44, 44.22, 31.85, 30.25, 28.39(2C), 19.84, 13.52; IR (KBr, cm⁻¹): 3135, 2928, 1645, 1586, 1465, 1373, 1011, 834; MS(EI) (*m/z*): 330.95 [(*M*+1)⁺] (65%); HRESIMS calcd for C₂₀H₂₇NO₃ (*M*+H)⁺ 330.2069; found 330.2072.

5,5-Dimethyl-2-((octylamino)(p-tolyl)methylene)cyclohexane-1,3-dione(4h)

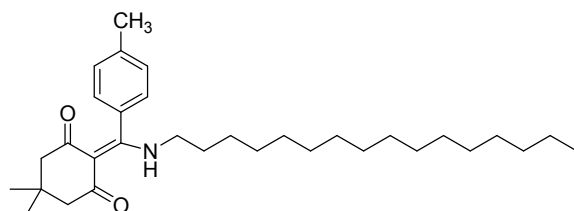
Liquid; ¹H-NMR (CDCl₃, 600 MHz) δ (ppm): 13.13 (s, 1H), 7.16 (t, *J* = 7.8 Hz, 2H), 6.93 (d, *J* = 8.4 Hz, 2H), 3.01-2.98 (m, 2H), 2.39 (s, 2H), 2.32 (s, 3H), 2.14 (s, 2H), 1.47-1.42 (m, 2H), 1.22-1.16 (m, 10H), 0.98 (s, 6H), 0.79 (t, *J* = 7.2 Hz, 3H); ¹³C-NMR (CDCl₃, 150 MHz) δ (ppm): 199.38, 194.35, 172.42, 138.53, 131.85, 129.26(2C), 125.83(2C), 107.56, 52.89, 52.32, 44.49, 31.72, 30.29, 29.80, 28.99(2C), 28.40(2C), 26.61, 22.58, 21.44, 14.07; IR (KBr, cm⁻¹): 3218, 2960, 2863, 1654, 1561, 1455, 1333, 1071, 862, 723; MS(EI) (*m/z*): 370.78 [(*M*+1)⁺] (25%); HRESIMS calcd for C₂₄H₃₅NO₂ (*M*+H)⁺ 370.2746; found 370.2742.

2-((Decylamino)(p-tolyl)methylene)-5,5-dimethylcyclohexane-1,3-dione(4i)

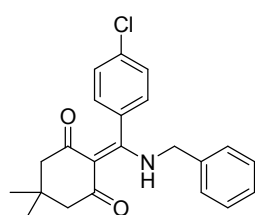
Liquid; $^1\text{H-NMR}$ (CDCl_3 , 600 MHz) δ (ppm): 13.14 (s, 1H), 7.16 (t, $J = 7.8$ Hz, 2H), 6.93 (d, $J = 7.8$ Hz, 2H), 3.00-2.97 (m, 2H), 2.38 (s, 2H), 2.31 (s, 3H), 2.14 (s, 2H), 1.47-1.42 (m, 2H), 1.21-1.14 (m, 14H), 0.97 (s, 6H), 0.79 (t, $J = 6.6$ Hz, 3H); $^{13}\text{C-NMR}$ (CDCl_3 , 150 MHz) δ (ppm): 172.38(2C), 138.45, 131.86, 129.37, 129.23(2C), 125.84(2C), 107.52, 52.47, 52.32, 44.44, 31.85, 30.25, 29.78, 29.68, 29.46, 29.32, 29.23, 29.02, 28.39, 26.59, 22.64, 21.43, 14.11; IR (KBr, cm^{-1}): 3438, 2974, 1655, 1561, 1457, 1381, 1332, 809, 764; MS(EI) (m/z): 398.67 $[(M+1)^+]$ (100%); HRESIMS calcd for $\text{C}_{26}\text{H}_{39}\text{NO}_2$ ($M+H$) $^+$ 398.3059; found 398.3062.

5,5-Dimethyl-2-((tetradecylamino)(p-tolyl)methylene)cyclohexane-1,3-dione(4j)

Liquid; $^1\text{H-NMR}$ (CDCl_3 , 600 MHz) δ (ppm): 13.13 (s, 1H), 7.16 (t, $J = 7.8$ Hz, 2H), 6.93 (d, $J = 8.4$ Hz, 2H), 3.01-2.97 (m, 2H), 2.39 (s, 2H), 2.32 (s, 3H), 2.14 (s, 2H), 1.47-1.42 (m, 2H), 1.21-1.14 (m, 22H), 0.98 (s, 6H), 0.81 (t, $J = 6.6$ Hz, 3H); $^{13}\text{C-NMR}$ (CDCl_3 , 150 MHz) δ (ppm): 172.43(2C), 138.54, 131.85, 129.78, 129.26(2C), 125.84(2C), 107.57, 52.91, 52.35, 44.49, 31.92, 30.29, 29.81, 29.65, 29.60, 29.53, 29.35, 29.05, 28.40, 26.62, 22.69, 21.44, 14.12; IR (KBr, cm^{-1}): 3108, 2922, 2880, 1653, 1559, 1458, 1333, 1021, 817; MS(EI) (m/z): 454.91 $[(M+1)^+]$ (100%); HRESIMS calcd for $\text{C}_{30}\text{H}_{47}\text{NO}_2$ ($M+H$) $^+$ 454.3685; found 454.3684.

2-((Hexadecylamino)(p-tolyl)methylene)-5,5-dimethylcyclohexane-1,3-dione(4k)

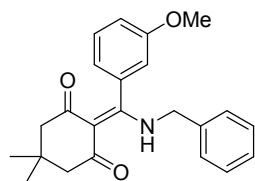
Mp 33.4-33.5 °C ($\text{MeOH}/\text{CH}_2\text{Cl}_2$); $^1\text{H-NMR}$ (CDCl_3 , 600 MHz) δ (ppm): 13.20 (s, 1H), 7.24 (t, $J = 7.8$ Hz, 2H), 7.01 (d, $J = 7.8$ Hz, 2H), 3.08-3.05 (m, 2H), 2.46 (s, 2H), 2.39 (s, 3H), 2.21 (s, 2H), 1.54-1.49 (m, 2H), 1.31-1.22 (m, 26H), 1.05 (s, 6H), 0.88 (t, $J = 7.2$ Hz, 3H); $^{13}\text{C-NMR}$ (CDCl_3 , 150 MHz) δ (ppm): 172.42, 138.55, 131.86, 129.27, 125.83, 107.57, 44.50, 31.93, 30.29, 29.81, 29.69, 29.60, 29.53, 29.36, 29.05, 28.40, 26.63, 22.69, 21.44, 14.12; IR (KBr, cm^{-1}): 3116, 2926, 2856, 1654, 1561, 1456, 1377, 1332, 1118, 817; MS(EI) (m/z): 482.80 $[(M+1)^+]$ (100%); HRESIMS calcd for $\text{C}_{32}\text{H}_{51}\text{NO}_2$ ($M+H$) $^+$ 482.3998; found 482.3992.

2-((Benzylamino)(4-chlorophenyl)methylene)-5,5-dimethylcyclohexane-1,3-dione(4l)

Mp 138.7-138.8 °C ($\text{MeOH}/\text{CH}_2\text{Cl}_2$); $^1\text{H-NMR}$ (CDCl_3 , 600 MHz) δ (ppm): 13.50 (s, 1H), 7.34 (t, $J = 8.4$ Hz, 2H), 7.27 (d, $J = 7.2$ Hz, 2H), 7.22 (d, $J = 7.2$ Hz, 1H), 7.05 (d, $J = 7.8$ Hz, 2H), 7.05 (d, $J = 8.4$ Hz, 2H), 4.18 (d, $J = 6.0$ Hz, 2H), 2.41 (s, 2H), 2.17 (s, 2H), 0.99 (s, 6H); $^{13}\text{C-NMR}$ (CDCl_3 , 150 MHz) δ (ppm): 199.98, 195.02, 170.91, 136.05, 134.96, 132.86, 129.05, 128.07, 127.51, 127.16, 107.71, 52.96, 52.31, 48.28, 30.31, 28.40; IR (KBr, cm^{-1}): 3209, 2912, 1643, 1578, 1548, 1485, 1084, 835; MS(EI)

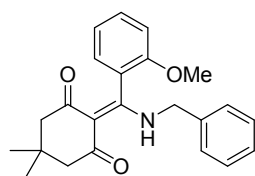
(m/z): 366.49 [(M-1)⁺] (100%), 368.52 [(M+1)⁺] (72%); HRESIMS calcd for C₂₂H₂₂ClNO₂ (M+H)⁺ 368.1417; found 368.1416.

2-((Benzylamino)(3-methoxyphenyl)methylene)-5,5-dimethylcyclohexane-1,3-dione(4m)



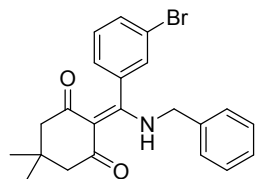
Mp 116.3-116.6 °C (MeOH/ CH₂Cl₂); ¹H-NMR (CDCl₃, 600 MHz) δ (ppm): 13.55 (s, 1H), 7.36 (t, *J* = 7.8 Hz, 1H), 7.34 (d, *J* = 4.2 Hz, 1H), 7.31 (d, *J* = 7.8 Hz, 1H), 7.28 (d, *J* = 7.2 Hz, 1H), 7.14 (d, *J* = 7.2 Hz, 2H), 6.95-6.93 (m, 1H), 6.72 (d, *J* = 7.2 Hz, 1H), 6.63-6.62 (m, 1H), 4.26 (d, *J* = 6.0 Hz, 2H), 3.74 (s, 3H), 2.48 (s, 2H), 2.25 (s, 2H), 1.06 (s, 6H); ¹³C-NMR (CDCl₃, 150 MHz) δ (ppm): 199.89, 194.81, 171.75, 159.75, 136.39, 135.73, 129.92, 128.93, 127.24, 118.20, 114.20, 111.69, 107.63, 55.16, 52.95, 52.53, 48.26, 30.32, 29.71, 28.49, 28.36; IR (KBr, cm⁻¹): 3121, 2961, 2865, 1651, 1561, 1494, 1437, 1249, 1043, 868; MS(EI) (m/z): 362.54 [(M-1)⁺] (100%), 364.28 [(M+1)⁺] (60%); HRESIMS calcd for C₂₃H₂₅NO₃ (M+H)⁺ 364.1913; found 364.1911.

2-((Benzylamino)(2-methoxyphenyl)methylene)-5,5-dimethylcyclohexane-1,3-dione(4n)



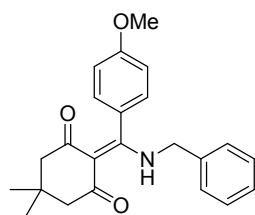
Mp 105.6-105.9 °C (MeOH/ CH₂Cl₂); ¹H-NMR (CDCl₃, 600 MHz) δ (ppm): 13.59 (s, 1H), 7.40-7.38 (m, 1H), 7.31 (d, *J* = 7.2 Hz, 2H), 7.27-7.25 (m, 1H), 7.15 (d, *J* = 7.2 Hz, 2H), 7.01 (d, *J* = 7.2 Hz, 1H), 6.97-6.94 (m, 2H), 4.29-4.20 (m, 2H), 3.73 (s, 3H), 2.46 (s, 2H), 2.22 (s, 2H), 1.06 (s, 3H), 1.04 (s, 3H); ¹³C-NMR (CDCl₃, 150 MHz) δ (ppm): 199.53, 194.82, 169.80, 155.45, 136.24, 130.32, 128.80, 127.83, 127.49, 126.90, 123.84, 120.96, 110.97, 107.98, 55.58, 52.76, 52.27, 48.24, 30.29, 28.46, 28.33; IR (KBr, cm⁻¹): 3159, 2956, 2890, 1645, 1576, 1544, 1499, 1333, 1105, 793; MS(EI) (m/z): 364.70 [(M+1)⁺] (90%); HRESIMS calcd for C₂₃H₂₅NO₃ (M+H)⁺ 364.1913; found 364.1917.

2-((Benzylamino)(3-bromophenyl)methylene)-5,5-dimethylcyclohexane-1,3-dione(4o)



Mp 71.3-71.6 °C (MeOH/ CH₂Cl₂); ¹H-NMR (CDCl₃, 600 MHz) δ (ppm): 13.49 (s, 1H), 7.33 (t, *J* = 7.8 Hz, 1H), 7.30 (d, *J* = 7.8 Hz, 1H), 7.28-7.25 (m, 2H), 7.23-7.21 (m, 1H), 7.05-7.03 (m, 3H), 6.94 (d, *J* = 7.8 Hz, 1H), 4.16 (d, *J* = 6.0 Hz, 2H), 2.41 (s, 2H), 2.17 (s, 2H), 0.99 (s, 6H); ¹³C-NMR (CDCl₃, 150 MHz) δ (ppm): 200.10, 194.94, 170.25, 136.15, 135.97, 134.77, 130.45, 129.00, 128.11, 127.26, 126.10, 124.23, 107.50, 52.88, 52.30, 48.39, 30.35, 28.43, 28.39; IR (KBr, cm⁻¹): 3111, 2960, 2875, 1651, 1560, 1493, 1447, 1259, 1043, 871; MS(EI) (m/z): 434.51 [(M+Na)⁺] (50%); HRESIMS calcd for C₂₂H₂₂BrNO₂ (M+H)⁺ 412.0912; found 412.0910.

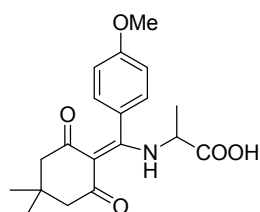
2-((Benzylamino)(4-methoxyphenyl)methylene)-5,5-dimethylcyclohexane-1,3-dione(4p)



Mp 88.9-89.1 °C (MeOH/ CH₂Cl₂); ¹H-NMR (CDCl₃, 600 MHz) δ (ppm): 13.46 (s, 1H), 7.25 (t, *J* = 7.2 Hz, 2H), 7.21 (d, *J* = 7.2 Hz, 1H), 7.07 (d, *J* = 7.2 Hz, 2H), 6.99 (d, *J* = 9.0 Hz, 2H), 6.88 (d, *J* = 8.4 Hz, 2H), 4.23 (d, *J* = 6.0 Hz, 2H), 3.76 (s, 3H), 2.40 (s, 2H), 2.17 (s, 2H), 0.98 (s, 6H); ¹³C-NMR (CDCl₃, 150 MHz) δ (ppm): 199.46, 195.23, 172.19, 160.06, 136.43, 128.94, 127.90, 127.73, 127.20, 126.51, 114.14, 108.07, 55.26,

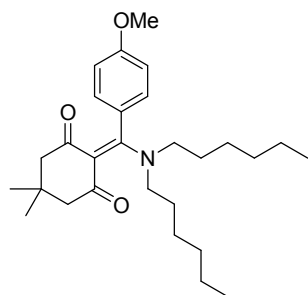
53.11, 52.30, 48.26, 30.28, 28.43; IR (KBr, cm^{-1}): 3381, 3051, 2959, 1656, 1611, 1554, 1465, 1249, 1027, 729; MS(EI) (m/z): 362.54 $[(M-1)^+]$ (100%), 364.48 $[(M+1)^+]$ (72%); HRESIMS calcd for $\text{C}_{23}\text{H}_{25}\text{NO}_3$ ($M+H$) $^+$ 364.1913; found 364.1912.

2-((4,4-Dimethyl-2,6-dioxocyclohexylidene)(4-methoxyphenyl)methylamino)propanoic acid(4q)



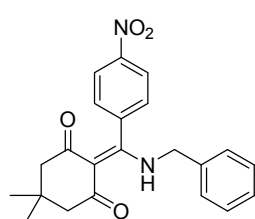
Mp 213.5-213.8 °C (MeOH/ CH_2Cl_2); $^1\text{H-NMR}$ ($\text{DMSO-}d_6$, 600 MHz) δ (ppm): 13.27 (d, J = 8.4 Hz, 1H), 13.21 (s, 1H), 7.10 (s, 1H), 7.01 (s, 1H), 6.96 (d, J = 7.8 Hz, 2H), 3.90-3.85 (m, 1H), 3.80 (s, 3H), 2.49-2.15 (m, 4H), 1.28 (d, J = 7.8 Hz, 3H), 0.98 (s, 6H); $^{13}\text{C-NMR}$ ($\text{DMSO-}d_6$, 150 MHz) δ (ppm): 199.20, 192.35, 172.39, 169.99, 159.31, 126.20, 113.70, 107.56, 55.07, 52.35, 52.10, 51.98, 29.84, 27.94, 19.13; IR (KBr, cm^{-1}): 3315, 2964, 1734, 1615, 1576, 1541, 1274, 1064, 835; MS(EI) (m/z): 346.72 $[(M+1)^+]$ (100%); HRESIMS calcd for $\text{C}_{19}\text{H}_{23}\text{NO}_5$ ($M+H$) $^+$ 346.1654; found 346.1640.

2-((Dihexylamino)(4-methoxyphenyl)methylene)-5,5-dimethylcyclohexane-1,3-dione(4r)



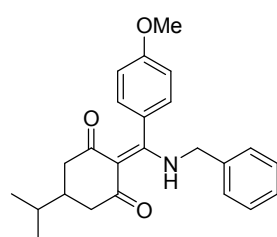
Liquid; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ (ppm): 7.05 (t, J = 8.4 Hz, 2H), 6.80 (d, J = 8.8 Hz, 2H), 3.76 (s, 3H), 3.72-3.63 (m, 2H), 2.64 (t, J = 7.6 Hz, 2H), 2.45-2.33 (m, 4H), 2.26 (s, 1H), 2.26 (s, 2H), 1.55-1.50 (m, 2H), 1.30-1.24 (m, 6H), 1.21 (s, 2H), 1.13 (s, 2H), 1.07 (s, 6H), 0.89-0.84 (m, 7H); $^{13}\text{C-NMR}$ (CDCl_3 , 150 MHz) δ (ppm): 194.83, 193.20, 157.62, 128.23, 126.26, 116.65, 112.36(2C), 60.39, 56.30, 54.15, 52.30, 48.85, 42.07, 39.95, 37.73, 32.84, 30.43, 28.20, 27.82, 27.60, 26.90, 21.49, 14.08, 12.95; IR (KBr, cm^{-1}): 3135, 3029, 2963, 1650, 1563, 1509, 1480, 1378, 1191, 1095, 831; MS(EI) (m/z): 440.01 $[(M-1)^+]$ (95%), 442.19 $[(M+1)^+]$ (62%); HRESIMS calcd for $\text{C}_{28}\text{H}_{43}\text{NO}_3$ ($M+H$) $^+$ 442.3321; found 442.3320.

2-((Benzylamino)(4-nitrophenyl)methylene)-5,5-dimethylcyclohexane-1,3-dione(4s)



Mp 173.4-173.7 °C (MeOH/ CH_2Cl_2); $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ (ppm): 13.59 (s, 1H), 8.28 (d, J = 8.4 Hz, 2H), 7.30 (t, J = 7.6 Hz, 3H), 7.26 (d, J = 8.0 Hz, 2H), 7.07 (d, J = 6.8 Hz, 2H), 4.19 (d, J = 8.4 Hz, 2H), 2.49 (s, 2H), 2.22 (s, 2H), 1.05 (s, 6H); $^{13}\text{C-NMR}$ (CDCl_3 , 150 MHz) δ (ppm): 200.37, 195.11, 169.56, 147.80, 141.11, 135.61, 129.15, 128.30, 127.14, 127.05, 124.09, 107.32, 52.72, 52.26, 48.40, 30.42, 28.38; IR (KBr, cm^{-1}): 3462, 2954, 1642, 1581, 1547, 1344, 850, 753; MS(EI) (m/z): 377.54 $[(M-1)^+]$ (100%), 379.51 $[(M+1)^+]$ (72%); HRESIMS calcd for $\text{C}_{22}\text{H}_{22}\text{N}_2\text{O}_4$ ($M+H$) $^+$ 379.1658; found 379.1660.

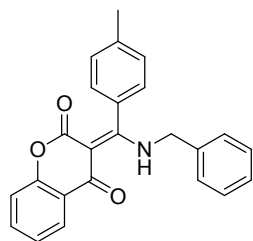
2-((Benzylamino)(4-chlorophenyl)methylene)-5-isopropylcyclohexane-1,3-dione(4t)



Liquid; $^1\text{H-NMR}$ (CDCl_3 , 600 MHz) δ (ppm): 13.56 (s, 1H), 7.33 (t, J = 7.6 Hz, 2H), 7.29 (d, J = 7.6 Hz, 1H), 7.14 (d, J = 7.8 Hz, 2H), 7.06 (s, 2H), 6.95 (d, J = 8.4 Hz, 2H), 4.32 (d, J = 6.0 Hz, 2H), 3.83 (s, 3H), 2.61-2.41 (m, 2H), 2.32-2.11 (m, 2H), 1.89-1.83 (m, 1H), 1.55-1.52 (m, 1H), 0.91 (d, J = 6.6 Hz, 6H); $^{13}\text{C-NMR}$ (CDCl_3 , 150 MHz) δ (ppm): 200.09, 195.37, 172.45, 160.05, 136.36, 128.88, 127.85, 127.72, 127.11,

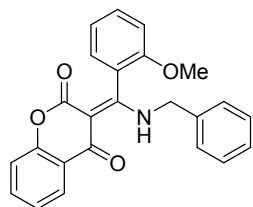
126.32, 114.07, 108.50, 55.20, 52.38, 52.02, 48.23, 37.56, 31.65, 19.40; IR (KBr, cm^{-1}): 3386, 3101, 2914, 2850, 1650, 1556, 1518, 1454, 1140, 877, 737; MS(EI) (m/z): 376.43 [(M-1)⁺] (100%), 378.38 [(M+1)⁺] (65%); HRESIMS calcd for $\text{C}_{24}\text{H}_{27}\text{NO}_3$ (M+H)⁺ 378.2069; found 378.2062.

(E)-3-((Benzylamino)(p-tolyl)methylene)chroman-2,4-dione(4u)



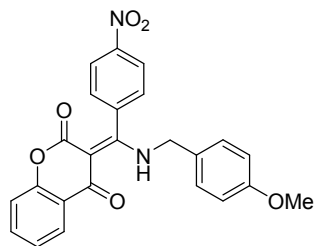
Mp 125.6-125.8 °C (MeOH/ CH_2Cl_2); $^1\text{H-NMR}$ (CDCl_3 , 600 MHz) δ (ppm): 14.30 (s, 1H), 7.93 (s, 1H), 7.38 (t, $J = 7.2$ Hz, 1H), 7.23 (t, $J = 7.2$ Hz, 2H), 7.18 (t, $J = 7.2$ Hz, 3H), 7.07 (t, $J = 7.8$ Hz, 3H), 7.03 (d, $J = 7.8$ Hz, 3H), 4.27 (d, $J = 6.0$ Hz, 2H), 2.31 (s, 3H); $^{13}\text{C-NMR}$ (CDCl_3 , 150 MHz) δ (ppm): 182.11, 182.08, 175.92, 161.46, 154.38, 139.69, 135.76, 134.05, 129.71, 129.11, 128.24, 127.38, 125.99, 123.56, 120.52, 120.45, 116.76, 97.26, 49.23, 21.64; IR (KBr, cm^{-1}): 3228, 2918, 1709, 1605, 1564, 1457, 1308, 1077, 760; MS(EI) (m/z): 368.44 [(M-1)⁺] (100%), 370.42 [(M+1)⁺] (38%); HRESIMS calcd for $\text{C}_{24}\text{H}_{19}\text{NO}_3$ (M+H)⁺ 370.1443; found 370.1440.

(E)-3-((Benzylamino)(2-methoxyphenyl)methylene)chroman-2,4-dione(4v)



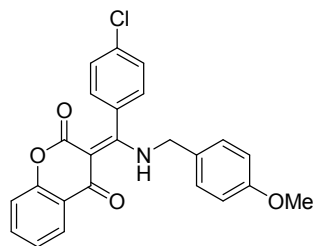
Liquid; $^1\text{H-NMR}$ (CDCl_3 , 600 MHz) δ (ppm): 14.40 (s, 1H), 7.98 (d, $J = 6.6$ Hz, 1H), 7.43-7.41 (m, 1H), 7.40-7.37 (m, 1H), 7.26 (t, $J = 7.2$ Hz, 2H), 7.23-7.20 (m, 1H), 7.14-7.11 (m, 3H), 7.06 (d, $J = 7.8$ Hz, 1H), 7.00-6.97 (m, 2H), 6.93 (d, $J = 8.4$ Hz, 1H), 4.36-4.26 (m, 2H), 3.69 (s, 3H); $^{13}\text{C-NMR}$ (CDCl_3 , 150 MHz) δ (ppm): 182.06, 173.66, 161.19, 155.44, 154.33, 135.57, 133.91, 131.20, 128.97, 128.15, 127.62, 126.84, 125.92, 123.39, 122.78, 121.12, 120.54, 116.77, 111.24, 97.52, 55.73, 49.31; IR (KBr, cm^{-1}): 3259, 2912, 2860, 1711, 1609, 1561, 1463, 1117, 756; MS(EI) (m/z): 384.46 [(M-1)⁺] (40%), 386.32 [(M+1)⁺] (32%); HRESIMS calcd for $\text{C}_{24}\text{H}_{19}\text{NO}_4$ (M+H)⁺ 386.1392; found 386.1389.

(E)-3-((Benzylamino)(4-nitrophenyl)methylene)chroman-2,4-dione(4w)



Mp 188.2-188.3 °C (MeOH/ CH_2Cl_2); $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ (ppm): 12.34 (s, 1H), 8.35 (d, $J = 8.4$ Hz, 2H), 8.05 (d, $J = 7.6$ Hz, 1H), 7.54 (t, $J = 8.0$ Hz, 1H), 7.41 (d, $J = 8.4$ Hz, 2H), 7.24 (t, $J = 7.2$ Hz, 1H), 7.15 (d, $J = 8.4$ Hz, 1H), 7.04 (d, $J = 8.4$ Hz, 2H), 6.86 (d, $J = 8.8$ Hz, 2H), 4.25 (d, $J = 6.0$ Hz, 2H), 3.79 (s, 3H); $^{13}\text{C-NMR}$ (CDCl_3 , 150 MHz) δ (ppm): 182.36, 172.85, 161.38, 159.70, 154.25, 148.29, 139.73, 134.52, 128.60, 127.19, 126.80, 125.99, 124.28, 123.79, 116.90, 114.62, 109.95, 96.87, 55.34, 48.97; IR (KBr, cm^{-1}): 3212, 3066, 2956, 1708, 1607, 1585, 1443, 1034, 855, 712; MS(EI) (m/z): 429.32 [(M-1)⁺] (100%), 431.36 [(M+1)⁺] (39%); HRESIMS calcd for $\text{C}_{24}\text{H}_{19}\text{N}_2\text{O}_6$ (M+H)⁺ 431.1243; found 431.1242.

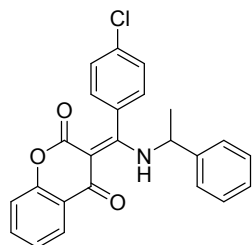
(E)-3-((4-Methoxybenzylamino)(4-chlorophenyl)methylene)chroman-2,4-dione(4x)



Mp 135.5-135.8 °C (MeOH/ CH_2Cl_2); $^1\text{H-NMR}$ (CDCl_3 , 600 MHz) δ (ppm): 14.28 (s, 1H), 7.98 (d, $J = 7.8$ Hz, 1H), 7.47 (t, $J = 7.2$ Hz, 1H), 7.42 (d, $J = 7.8$ Hz, 2H), 7.16 (t, $J = 7.2$ Hz, 1H), 7.12 (d, $J = 7.8$ Hz, 2H), 7.10 (d, $J = 8.4$ Hz, 1H), 7.01 (d, $J = 7.8$ Hz, 2H), 6.81 (d, $J = 8.4$ Hz, 2H), 4.24 (d, $J = 5.4$ Hz, 2H), 3.73 (s, 3H); $^{13}\text{C-NMR}$ (CDCl_3 , 150 MHz) δ (ppm): 182.23, 174.21, 159.60, 134.26, 129.44, 129.38, 128.76, 127.47, 127.26, 125.99, 123.62, 120.30, 116.88,

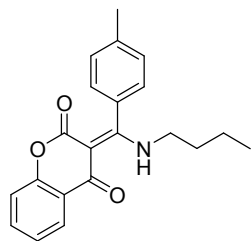
114.55, 55.36, 48.89; IR (KBr, cm^{-1}): 3379, 2952, 2860, 1711, 1608, 1563, 1464, 1179, 832; MS(EI) (m/z): 418.34 $[(M-1)^+]$ (100%), 420.50 $[(M+1)^+]$ (52%); HRESIMS calcd for $\text{C}_{24}\text{H}_{18}\text{ClNO}_4$ ($M+H$) $^+$ 420.1003; found 420.1001.

(E)-3-((4-Chlorophenyl)(1-phenylethylamino)methylene)chroman-2,4-dione(4y)



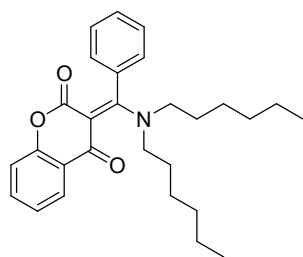
Mp 129.6-129.9 $^{\circ}\text{C}$ (MeOH/ CH_2Cl_2); $^1\text{H-NMR}$ (CDCl_3 , 600 MHz) δ (ppm): 14.66 (s, 1H), 8.10 (d, $J = 7.8$ Hz, 1H), 7.56-7.51 (m, 2H), 7.34 (t, $J = 7.2$ Hz, 3H), 7.31-7.27 (m, 3H), 7.17 (d, $J = 8.4$ Hz, 1H), 7.12 (d, $J = 7.8$ Hz, 2H), 6.82 (d, $J = 7.8$ Hz, 1H), 4.52 (q, $J = 7.2$ Hz, 1H), 1.61 (d, $J = 6.6$ Hz, 3H); $^{13}\text{C-NMR}$ (CDCl_3 , 150 MHz) δ (ppm): 182.35, 173.35, 161.30, 154.71, 141.40, 137.02, 135.04, 134.26, 131.92, 129.92, 129.16, 129.06, 128.11, 127.50, 127.15, 123.67, 120.32, 116.28, 96.90, 55.49, 23.95; IR (KBr, cm^{-1}): 3286, 2921, 1821, 1709, 1613, 1562, 1462, 1131, 836; MS(EI) (m/z): 402.35 $[(M-1)^+]$ (100%), 404.30 $[(M+1)^+]$ (47%); HRESIMS calcd for $\text{C}_{24}\text{H}_{18}\text{ClNO}_3$ ($M+H$) $^+$ 404.1053; found 404.1050.

(E)-3-((Butylamino)(p-tolyl)methylene)chroman-2,4-dione(4z)



Mp 115.7-115.9 $^{\circ}\text{C}$ (MeOH/ CH_2Cl_2); $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ (ppm): 14.05 (s, 1H), 8.08 (d, $J = 6.0$ Hz, 1H), 7.52 (t, $J = 7.2$ Hz, 1H), 7.31 (d, $J = 7.2$ Hz, 2H), 7.23 (t, $J = 6.0$ Hz, 1H), 7.17 (d, $J = 7.2$ Hz, 1H), 7.13 (d, $J = 7.8$ Hz, 2H), 3.23-3.20 (m, 2H), 2.44 (s, 3H), 1.62-1.58 (m, 2H), 1.39-1.36 (m, 2H), 0.89 (t, $J = 7.2$ Hz, 3H); $^{13}\text{C-NMR}$ (CDCl_3 , 150 MHz) δ (ppm): 190.94, 175.82, 161.53, 156.45, 154.31, 139.52, 133.86, 129.59, 125.80, 123.40, 120.28, 100.55, 116.81, 45.31, 31.79, 21.59, 19.87, 13.58; IR (KBr, cm^{-1}): 3244, 2958, 2881, 1723, 1602, 1567, 1461, 1454, 1108, 940, 759; MS(EI) (m/z): 334.45 $[(M-1)^+]$ (100%), 336.51 $[(M+1)^+]$ (38%); HRESIMS calcd for $\text{C}_{21}\text{H}_{21}\text{NO}_3$ ($M+H$) $^+$ 336.1600; found 336.1608.

(E)-3-((Dihexylamino)(phenyl)methylene)chroman-2,4-dione(4a')



Liquid; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ (ppm): 8.02 (d, $J = 8.0$ Hz, 1H), 7.51-7.46 (m, 2H), 7.44-7.42 (m, 4H), 7.18 (t, $J = 8.0$ Hz, 2H), 3.91 (t, $J = 7.2$ Hz, 2H), 3.74-3.61 (m, 2H), 1.82-1.75 (m, 2H), 1.65 (s, 2H), 1.35-1.33 (m, 2H), 1.28-1.26 (m, 4H), 1.20-1.13 (m, 4H), 1.09-1.04 (m, 2H), 0.84 (t, $J = 8.4$ Hz, 3H), 0.79 (t, $J = 7.2$ Hz, 3H); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ (ppm): 179.26, 174.72, 162.33, 154.35, 135.63, 132.77, 131.50, 129.48, 128.63, 126.21, 123.19, 121.40, 116.68, 100.11, 56.29, 53.25, 31.28, 30.89, 28.36, 28.20, 26.34, 25.80, 22.39, 22.24, 13.89, 13.85; IR (KBr, cm^{-1}): 3120, 2987, 2880, 1703, 1602, 1567, 1526, 1462, 1107, 873, 759; MS(EI) (m/z): 434.70 $[(M+1)^+]$ (100%); HRESIMS calcd for $\text{C}_{28}\text{H}_{35}\text{NO}_3$ ($M+H$) $^+$ 434.2695; found 434.2692.

X-ray Structure of 4f, 4l and 4x

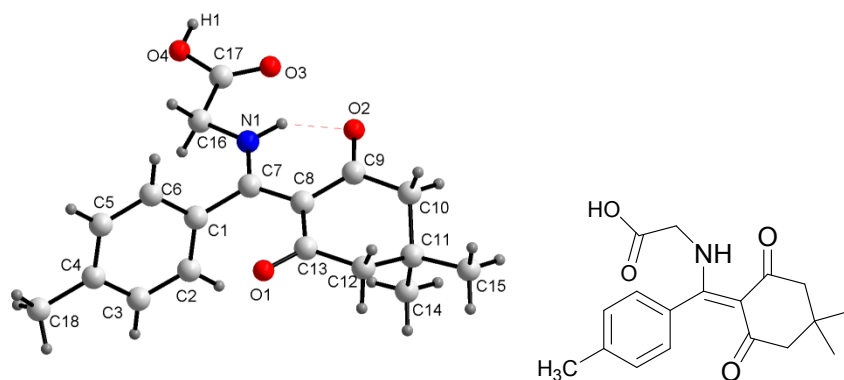


Figure 1 Molecular structure of β -enaminones 4f

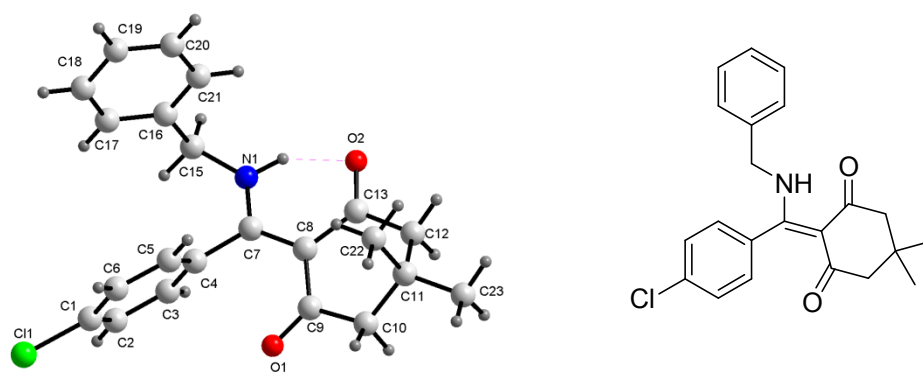


Figure 2 Molecular structure of β -enaminones 4l

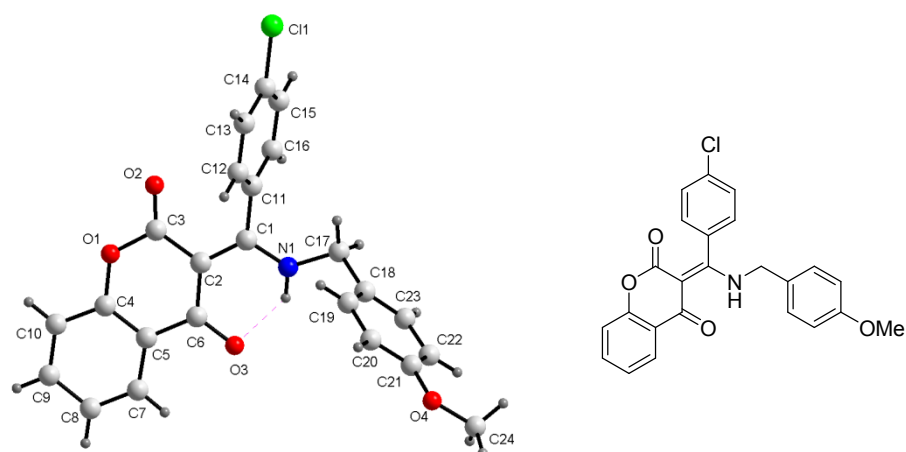


Figure 3 Molecular structure of β -enaminones 4x

Table 1 X-ray crystal data of **4f**, **4l** and **4x**

Phase	4l	4x	4f
Molecular formula	C ₂₂ H ₂₂ ClNO ₂	C ₂₄ H ₁₈ ClNO ₄	C ₁₈ H ₂₁ NO ₄
Formula weight	367.86	419.84	315.36
T/K	296(2)	296(2)	296(2)
Wavelength/nm	0.71073	0.71073	0.71073
Crystal system	Orthorhombic	Triclinic	Monoclinic
Space group	P2(1)2(1)2(1)	P -1	P2(1)/n
<i>a</i> / Å	7.5759(6)	10.6004(18)	10.0101(10)
<i>b</i> / Å	11.0478(10)	13.375(2)	10.7765(10)
<i>c</i> / Å	23.488(2)	16.069(2)	15.5711(14)
<i>A</i>	90	82.836(4)	90
<i>β</i> (°)	90	71.805(5)	93.900(3)
<i>Γ</i>	90	89.343(5)	90
<i>V</i> (Å ³)	1965.9(3)	2146.5(6)	1675.8(3)
<i>Z</i>	4	4	4
<i>F</i> (000)	776	872	672
<i>D</i> _{calc} (mg/m ³)	1.243	1.299	1.250
Absorption coefficient (mm ⁻¹)	0.209	0.208	0.088
θ range / (°)	1.73 – 27.50	1.91 - 25.00	2.30 – 27.53
Limiting indices	-9<= <i>h</i> <=9, 13<= <i>k</i> <=14, 26<= <i>l</i> <=30	- 12<= <i>h</i> <=12, 15<= <i>k</i> <=15, -19<= <i>l</i> <=17	- 13<= <i>h</i> <=10, 13<= <i>k</i> <=13, 19<= <i>l</i> <=20
Reflections collected/unique	19609 / 4520 [<i>R</i> (int) = 0.0459]	18337 / 7560 [<i>R</i> (int) = 0.0575]	20912 / 3851 [<i>R</i> (int) = 0.0357]
Completeness to theta	99.6 %	96.0 %	99.7 %
Data/restraints/parameters	4498/ 0 / 241	7256 / 0 / 543	3841/ 0 / 218
Refinement method	Full-matrix least-squares on <i>F</i> ²	Full-matrix least-squares on <i>F</i> ²	Full-matrix least-squares on <i>F</i> ²
Final <i>R</i> indices [<i>I</i> >2σ(<i>I</i>)]	<i>R</i> ₁ = 0.0577, <i>wR</i> ₂ = 0.1418	<i>R</i> ₁ = 0.0778, <i>wR</i> ₂ = 0.1928	<i>R</i> ₁ = 0.0551, <i>wR</i> ₂ = 0.1320
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0956, <i>wR</i> ₂ = 0.1606	<i>R</i> ₁ = 0.1751, <i>wR</i> ₂ = 0.2233	<i>R</i> ₁ = 0.0771, <i>wR</i> ₂ = 0.1459
Goodness-of-fit on	0.997	0.964	1.047

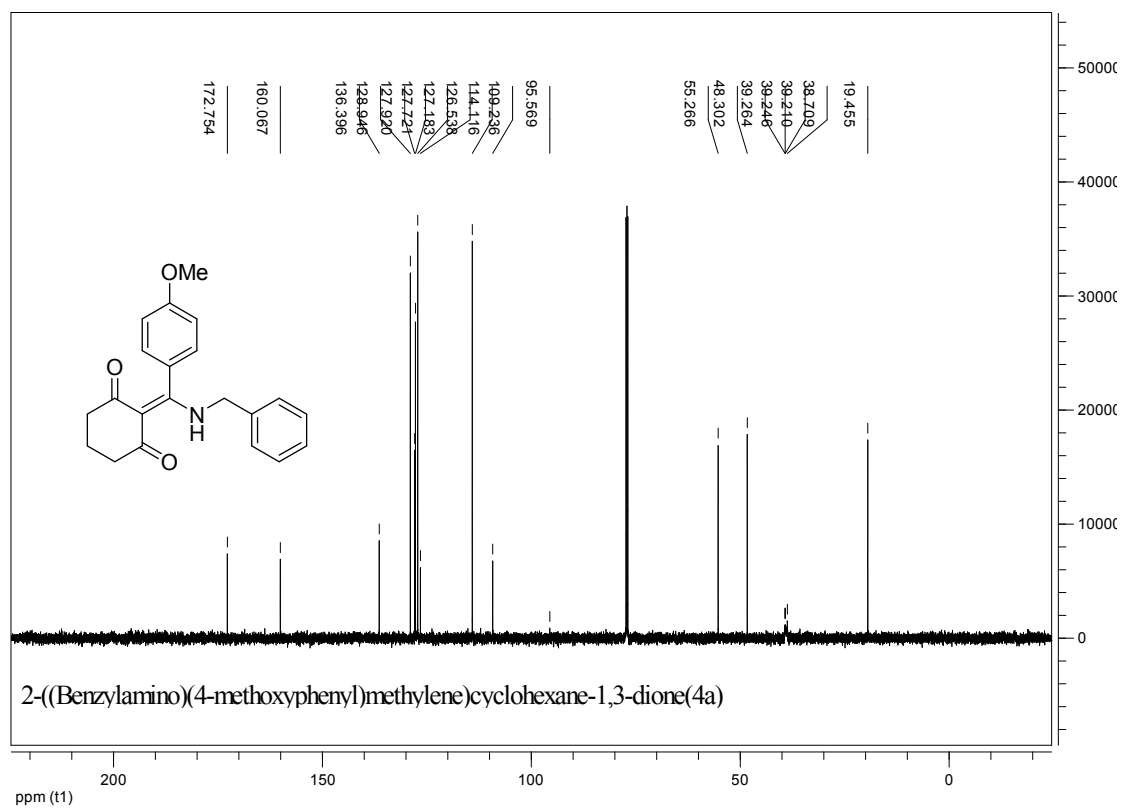
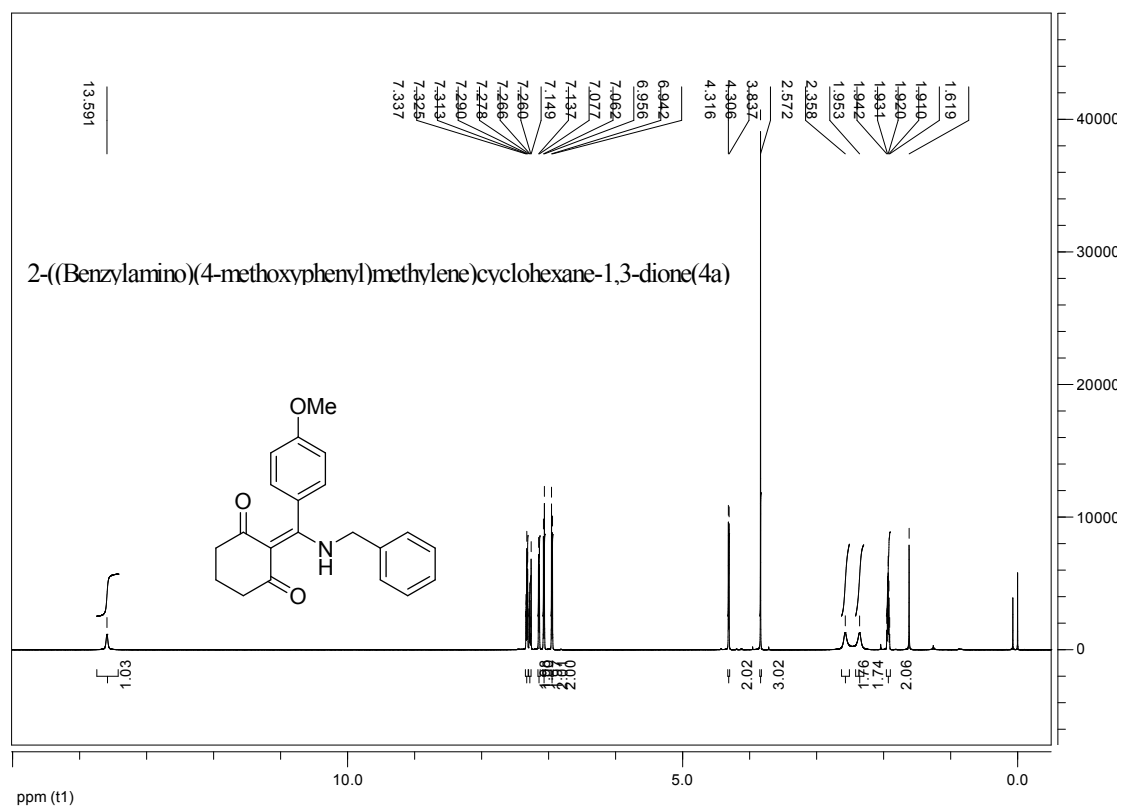
F^2

Largest diff. peak

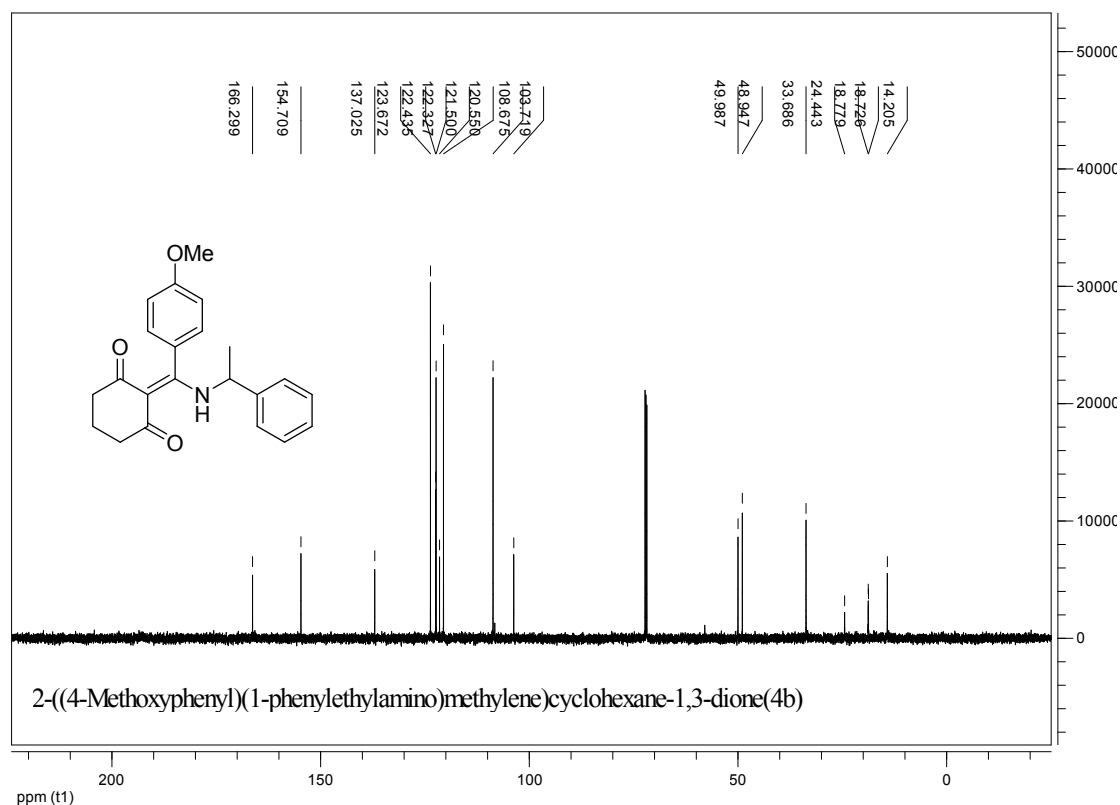
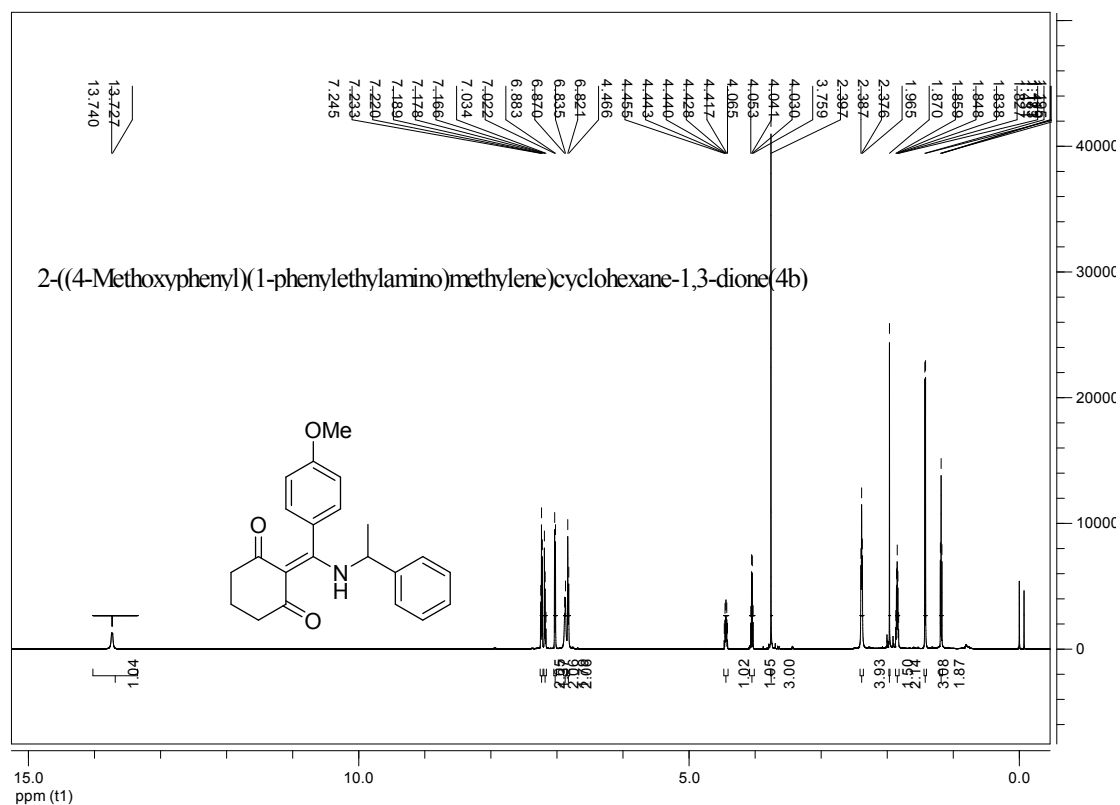
and hole $/(e \cdot nm^{-3}$	0.304 and -0.370	0.284 and -0.24	0.268 and -0.245
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$\times 10^{-3})$

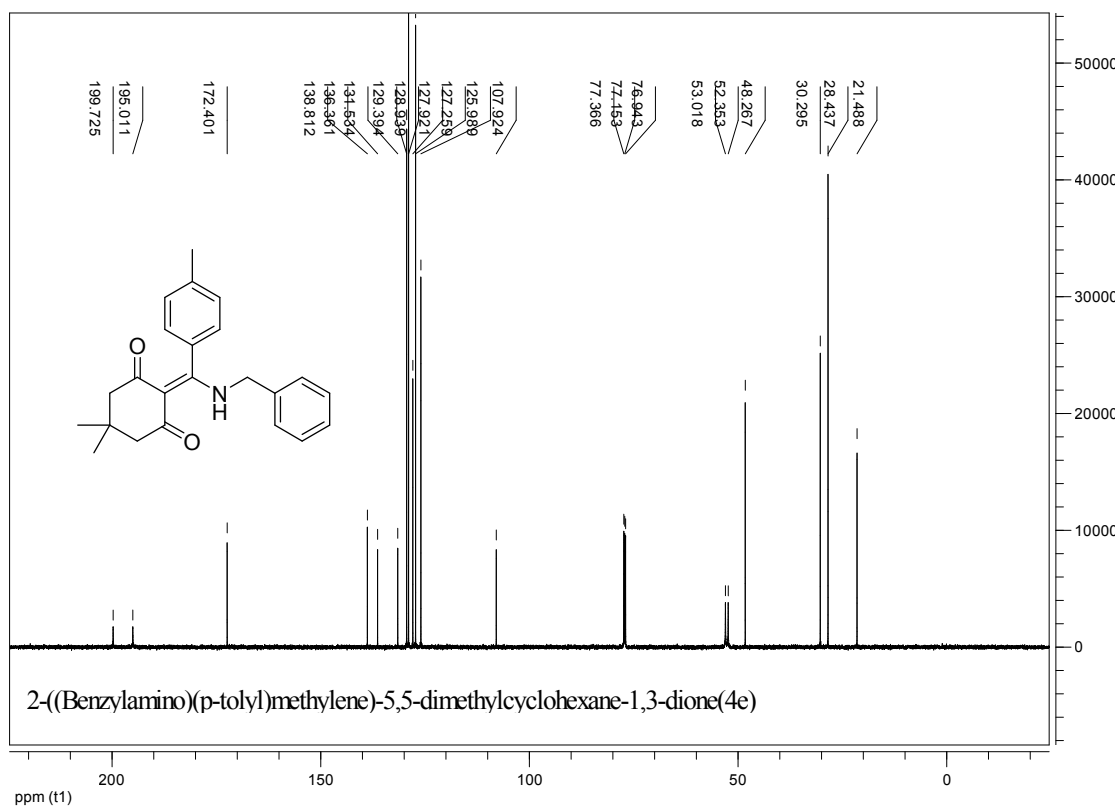
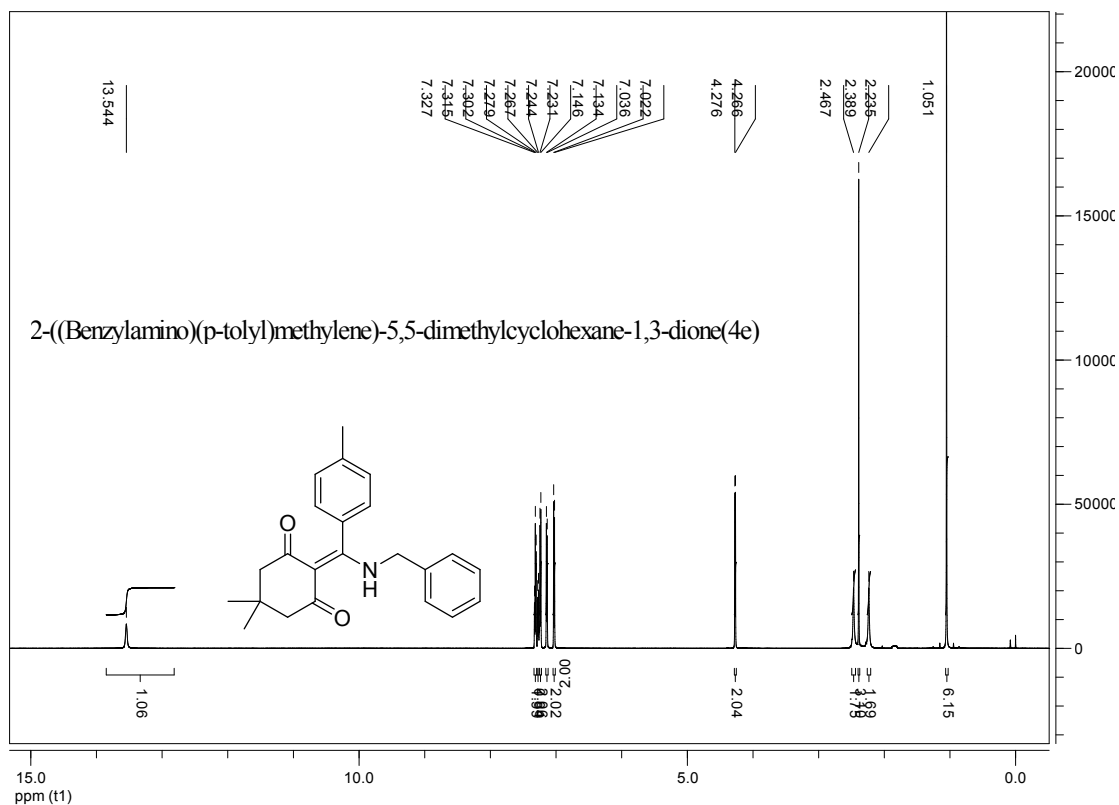
2-((Benzylamino)(4-methoxyphenyl)methylene)cyclohexane-1,3-dione(4a)



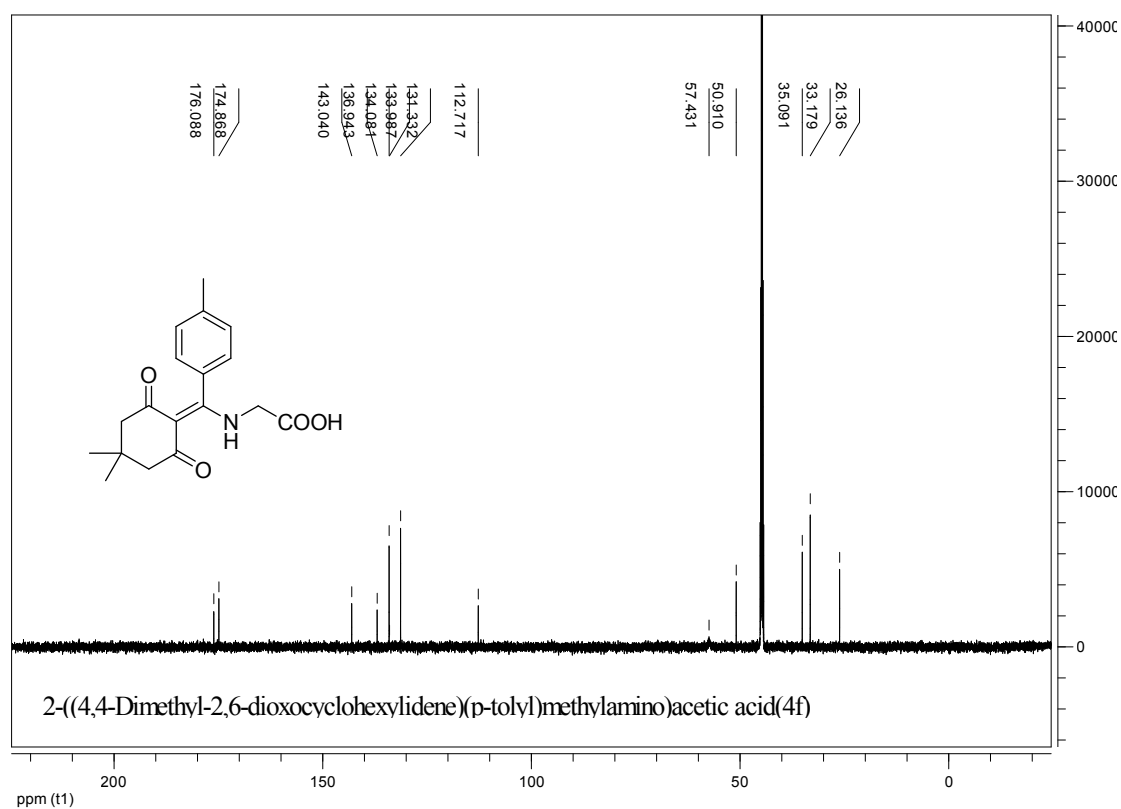
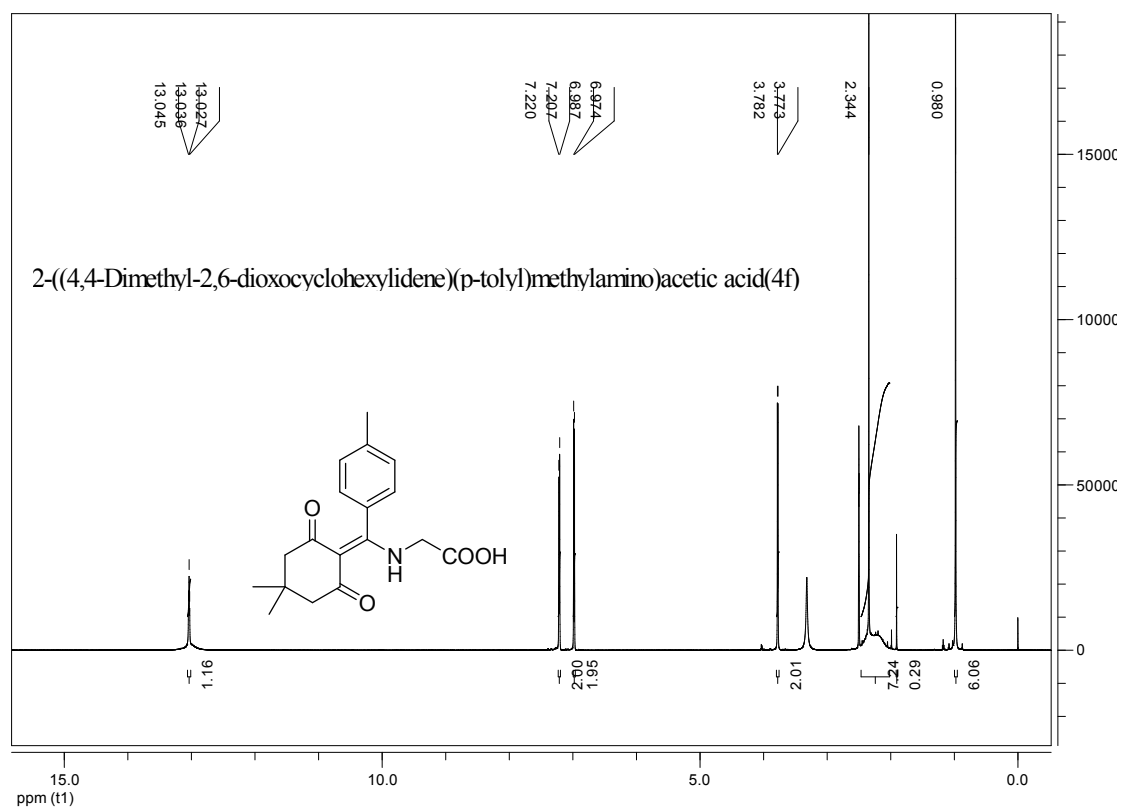
2-((4-Methoxyphenyl)(1-phenylethylamino)methylene)cyclohexane-1,3-dione(4b)



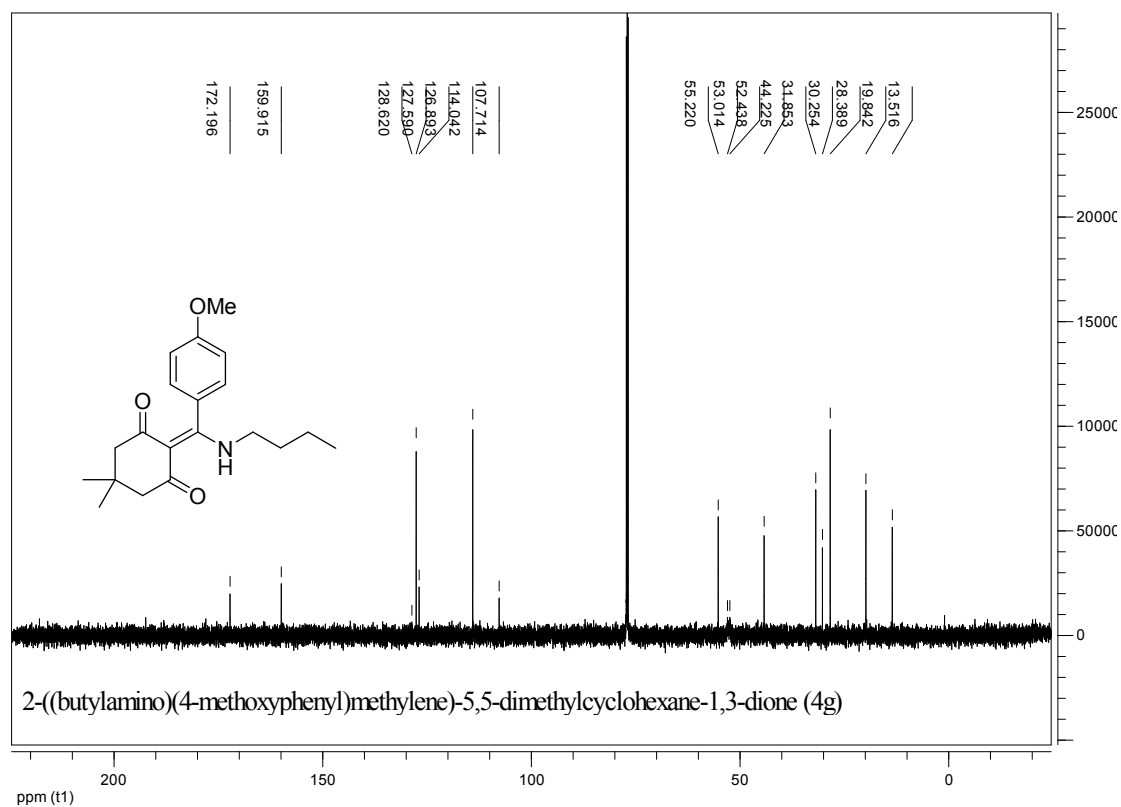
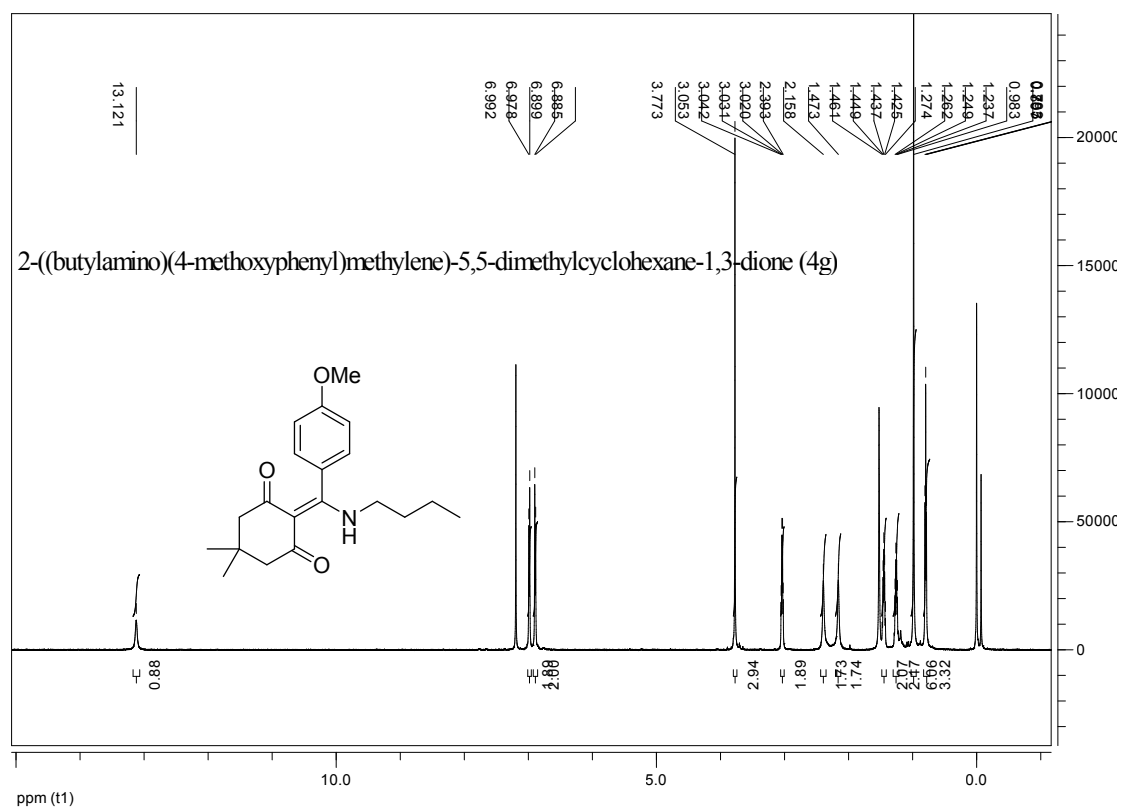
2-((Benzylamino)(3-methoxyphenyl)methylene)cyclohexane-1,3-dione(4c)



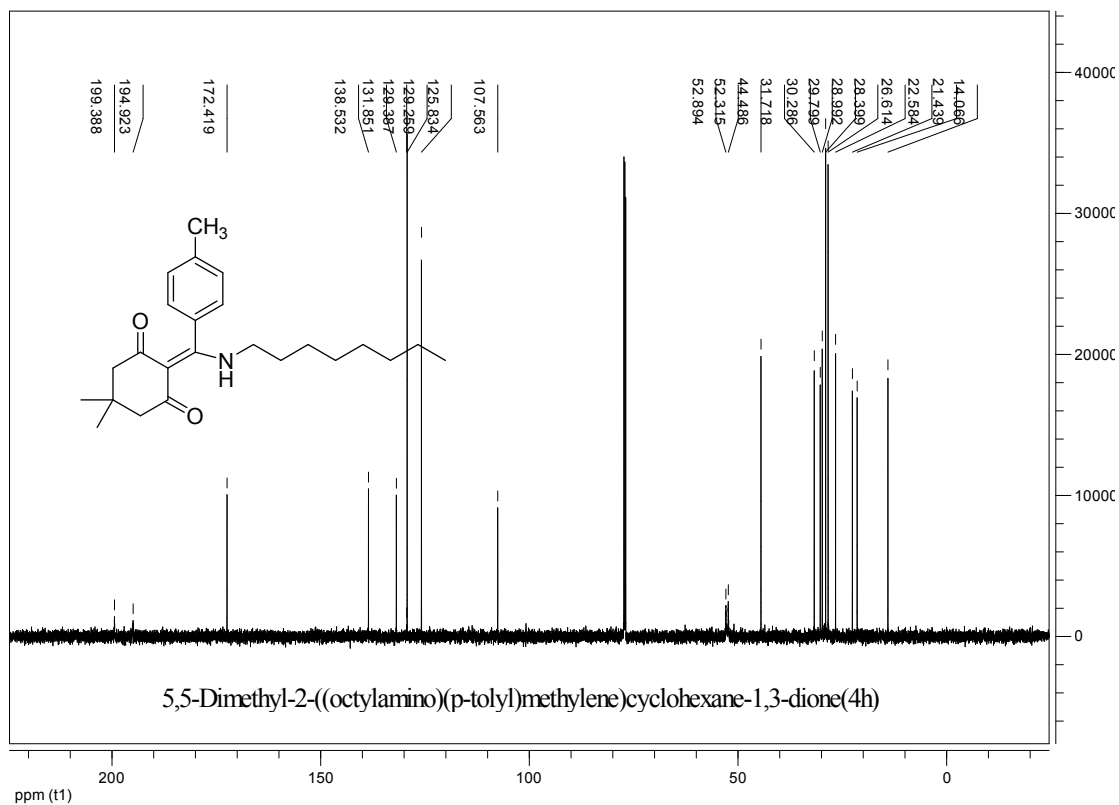
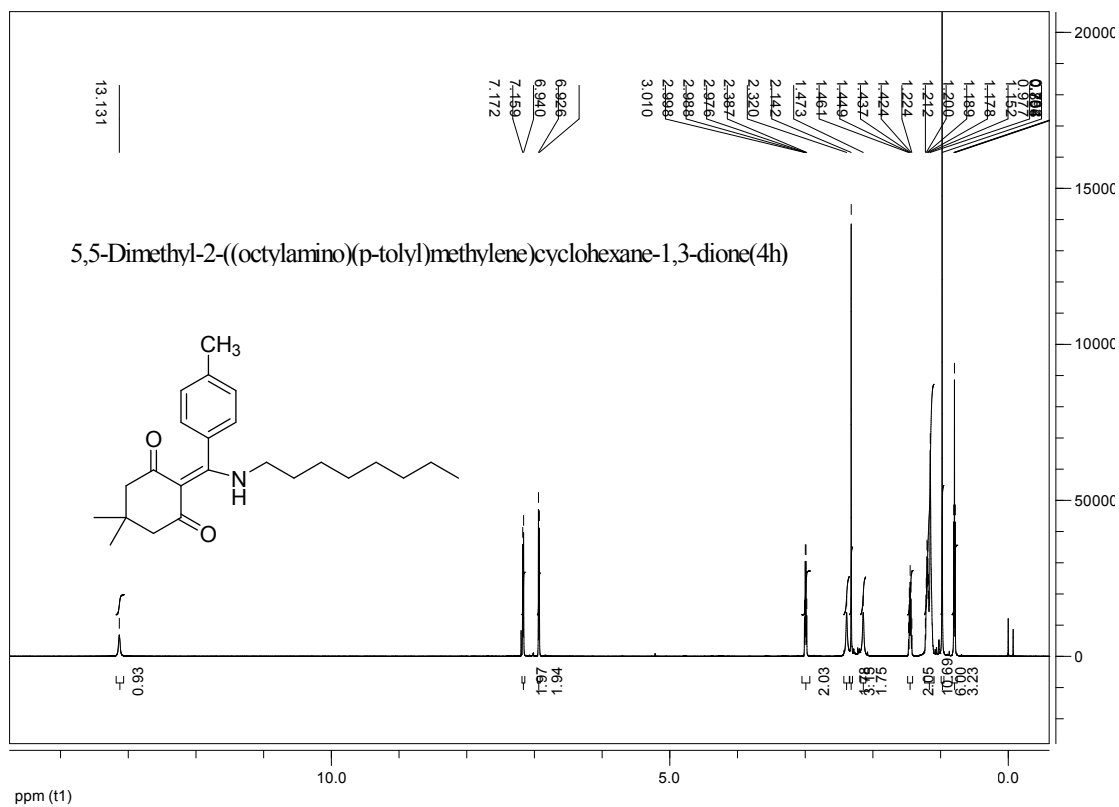
2-((4,4-Dimethyl-2,6-dioxocyclohexylidene)(p-tolyl)methylamino)acetic acid(4f)



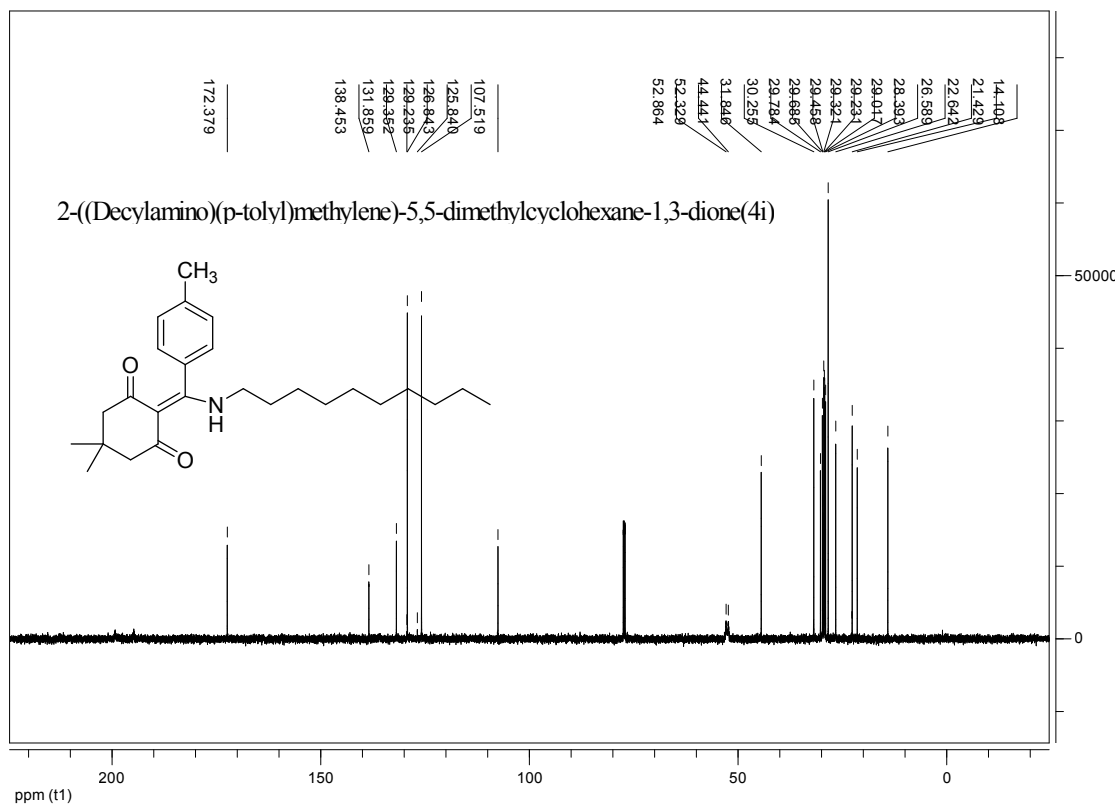
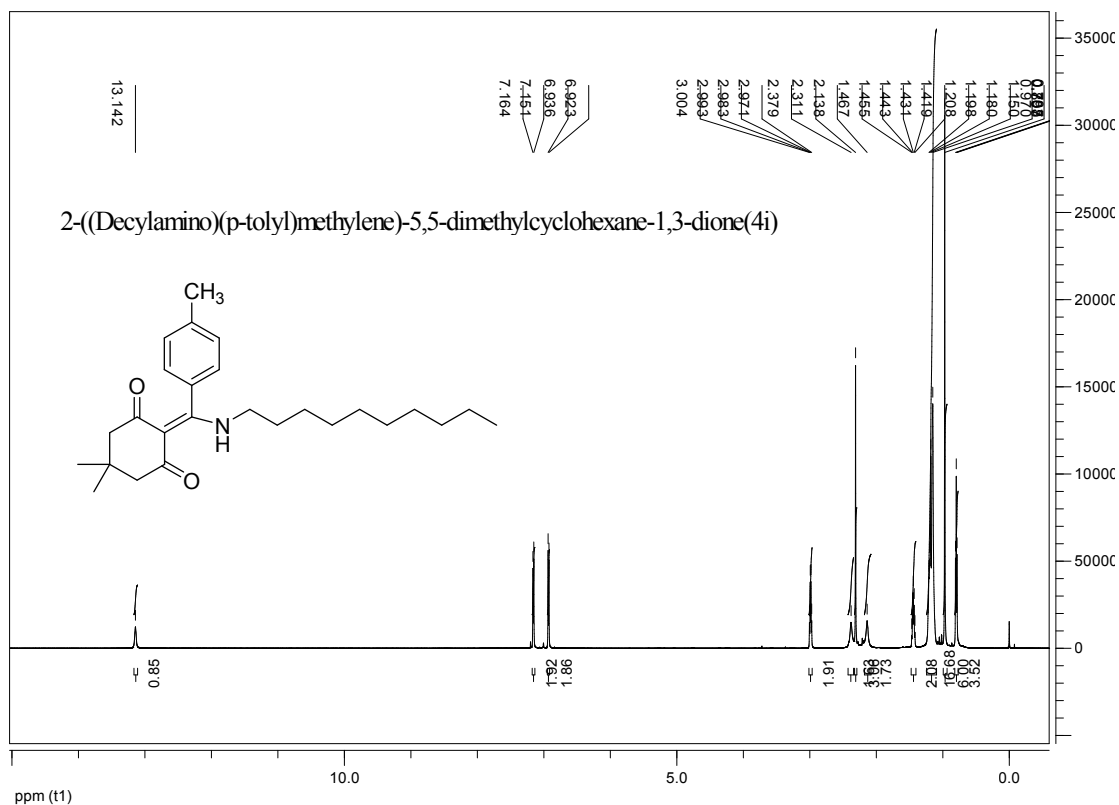
2-((butylamino)(4-methoxyphenyl)methylene)-5,5-dimethylcyclohexane-1,3-dione (4g)



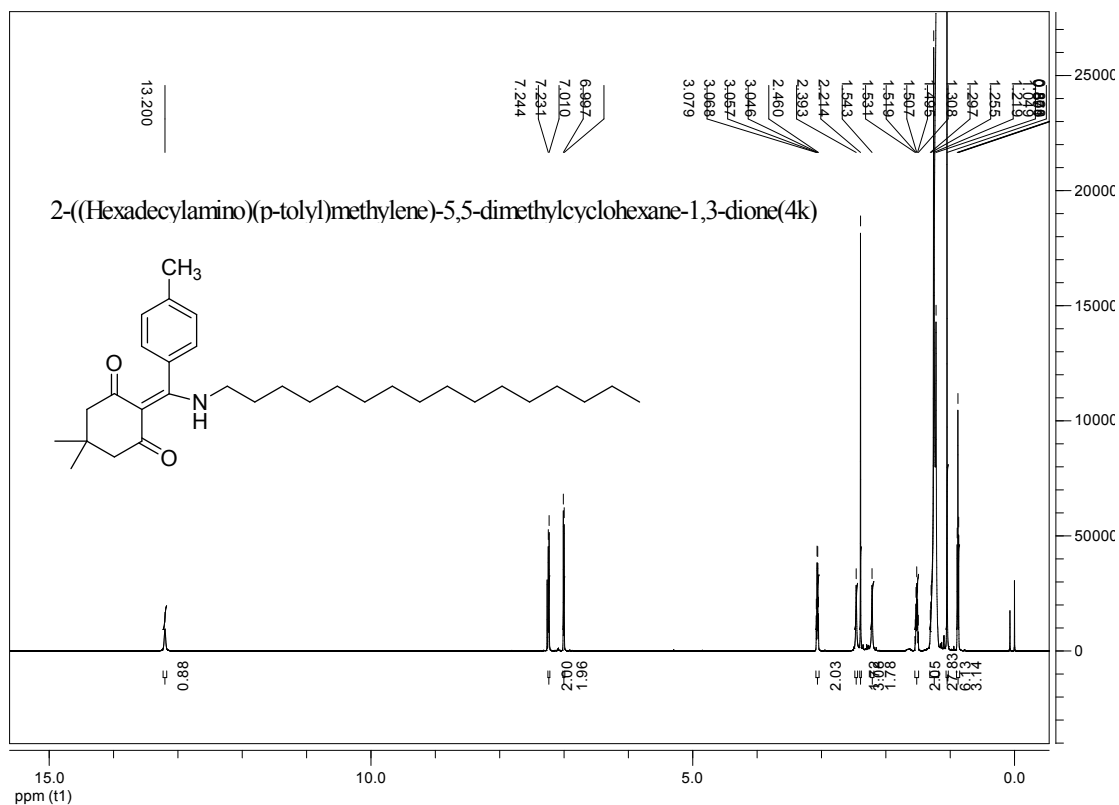
5,5-Dimethyl-2-((octylamino)(p-tolyl)methylene)cyclohexane-1,3-dione(4h)

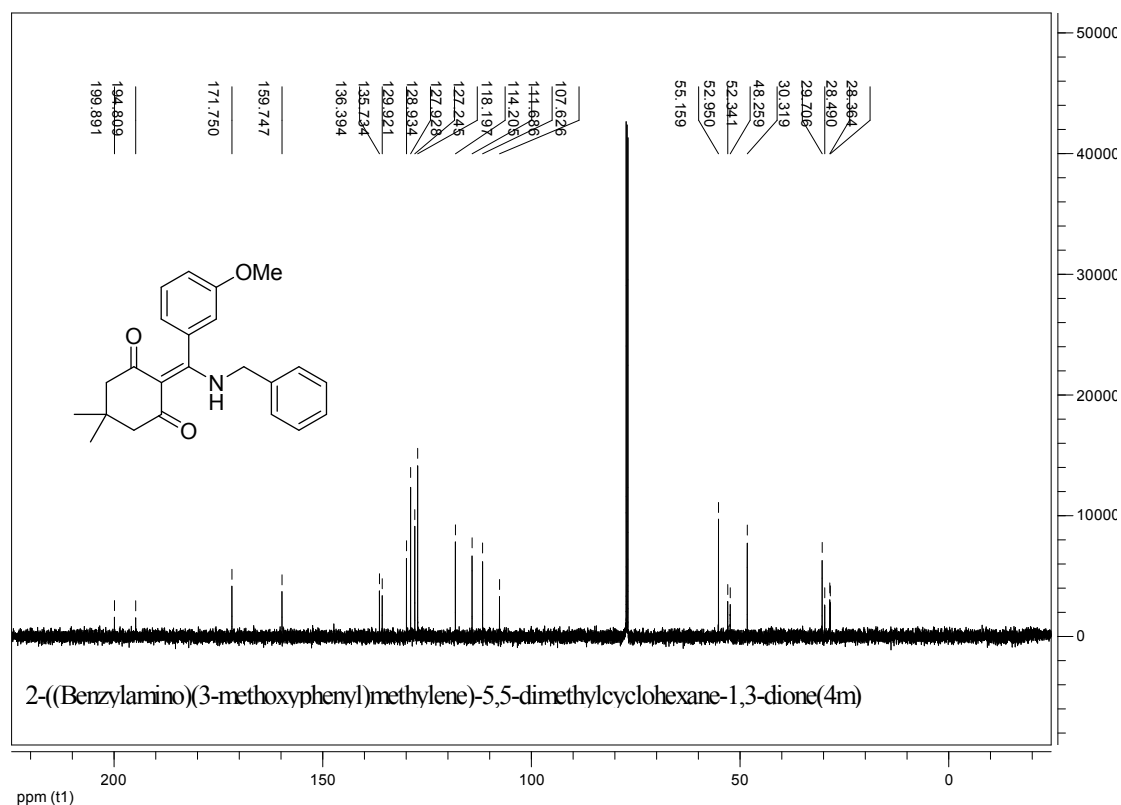
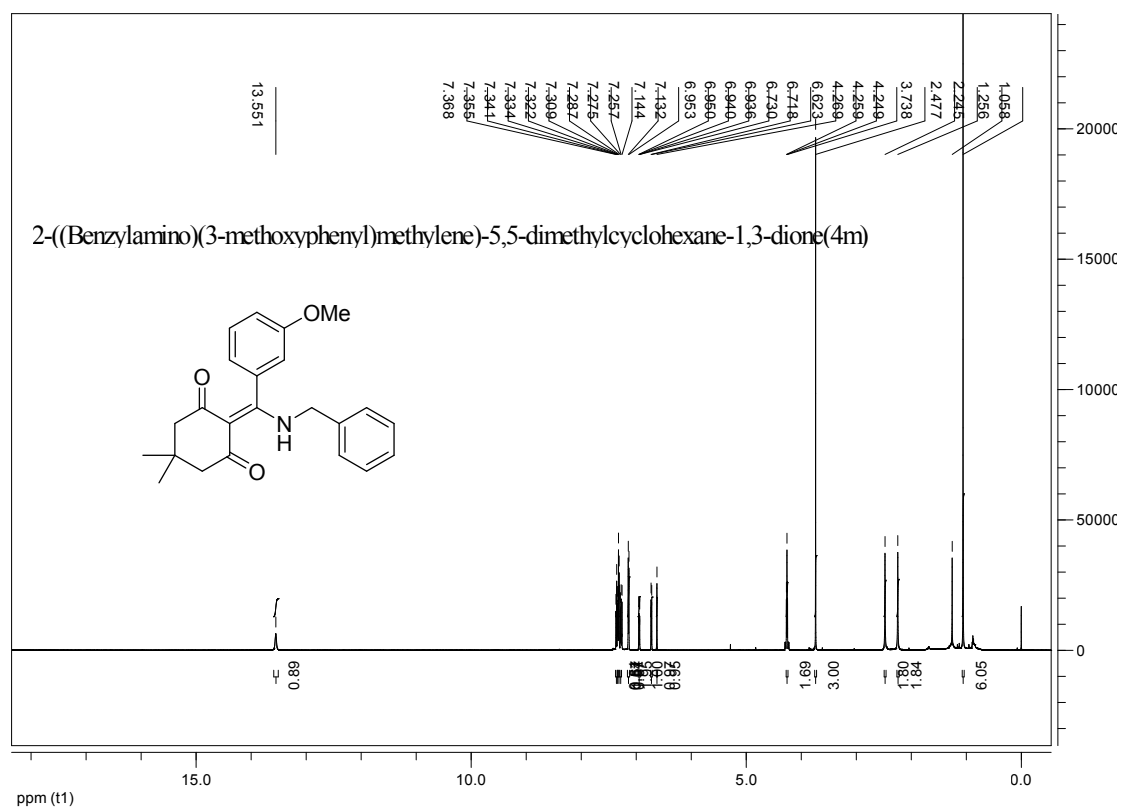


2-((Decylamino)(p-tolyl)methylene)-5,5-dimethylcyclohexane-1,3-dione(4i)

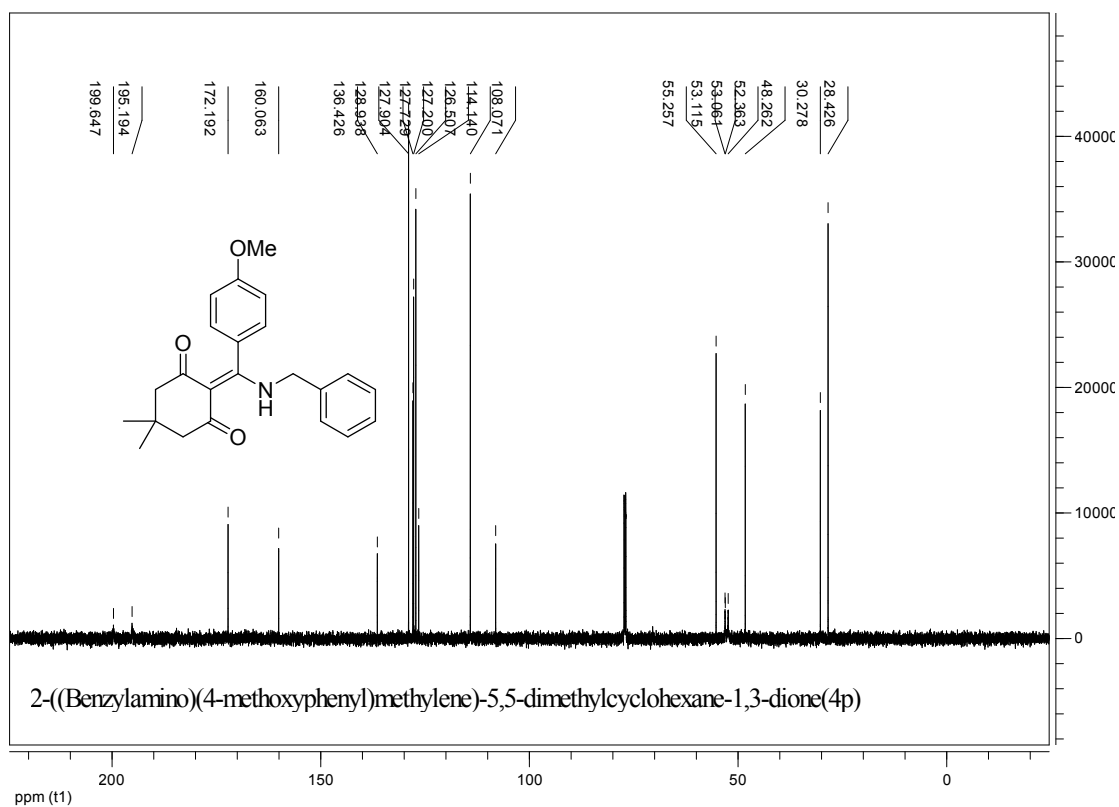
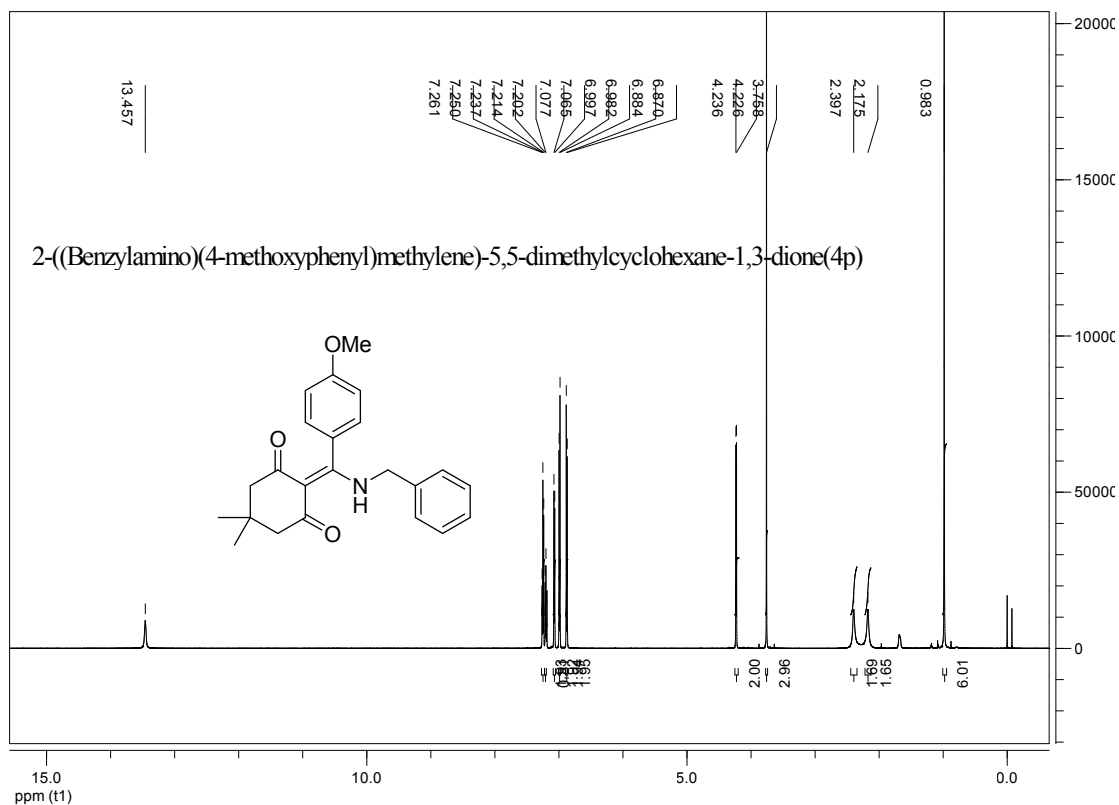


5,5-Dimethyl-2-((tetradecylamino)(p-tolyl)methylene)cyclohexane-1,3-dione(4j)

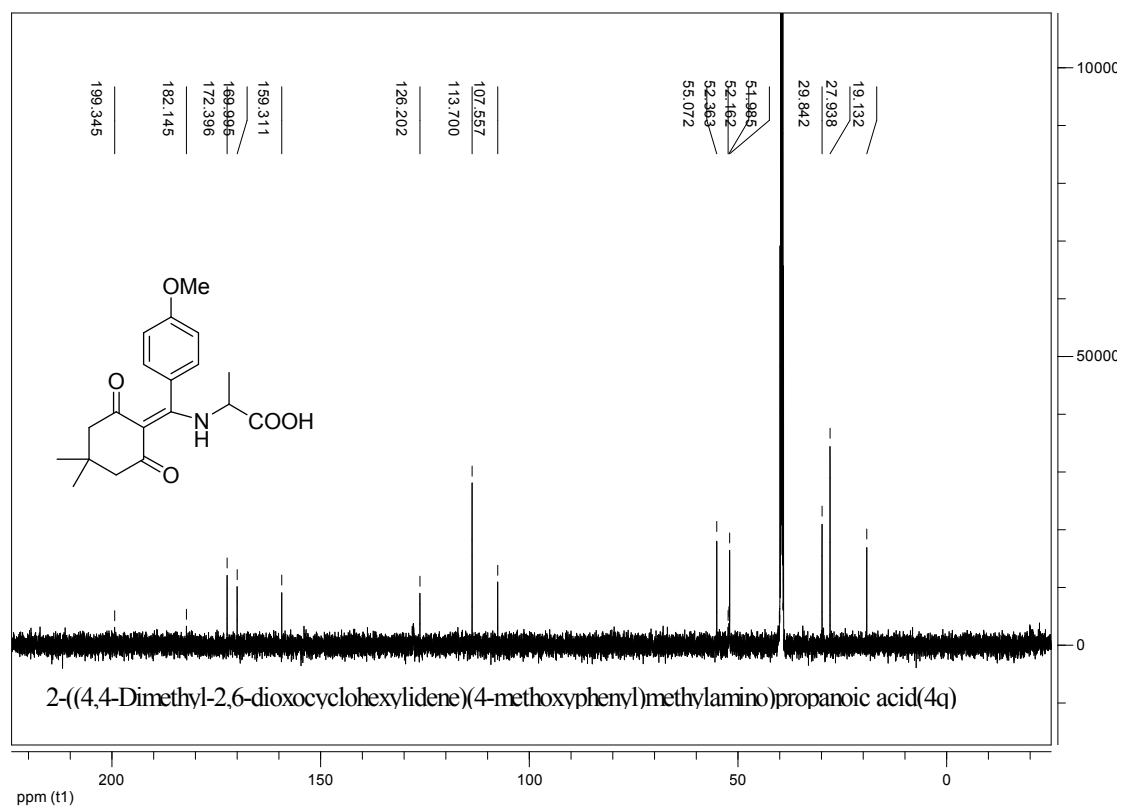
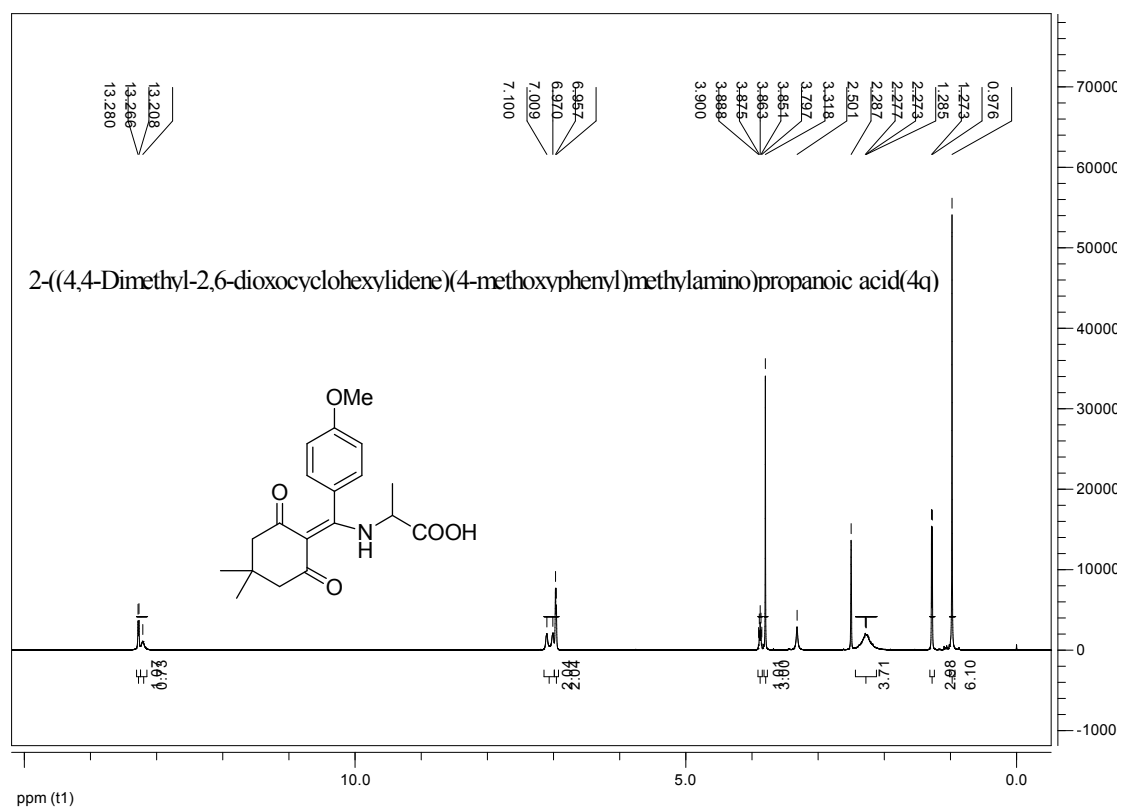




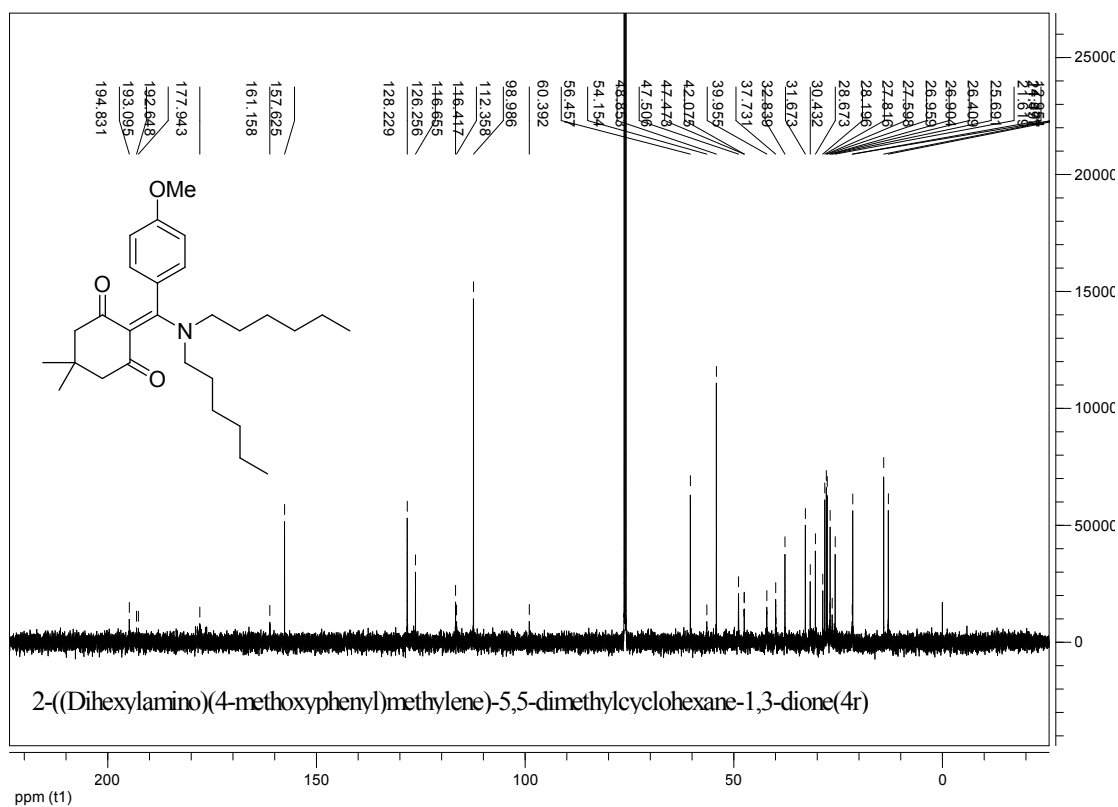
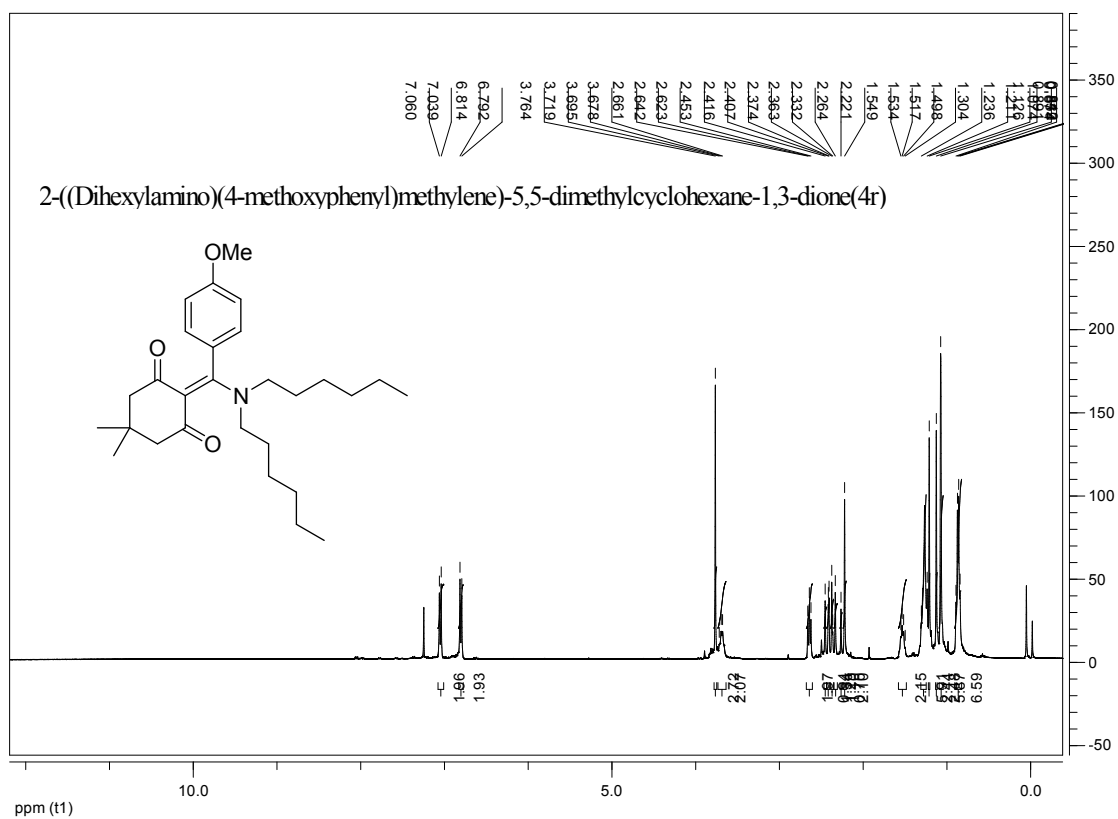
2-((Benzylamino)(2-methoxyphenyl)methylene)-5,5-dimethylcyclohexane-1,3-dione(4n)



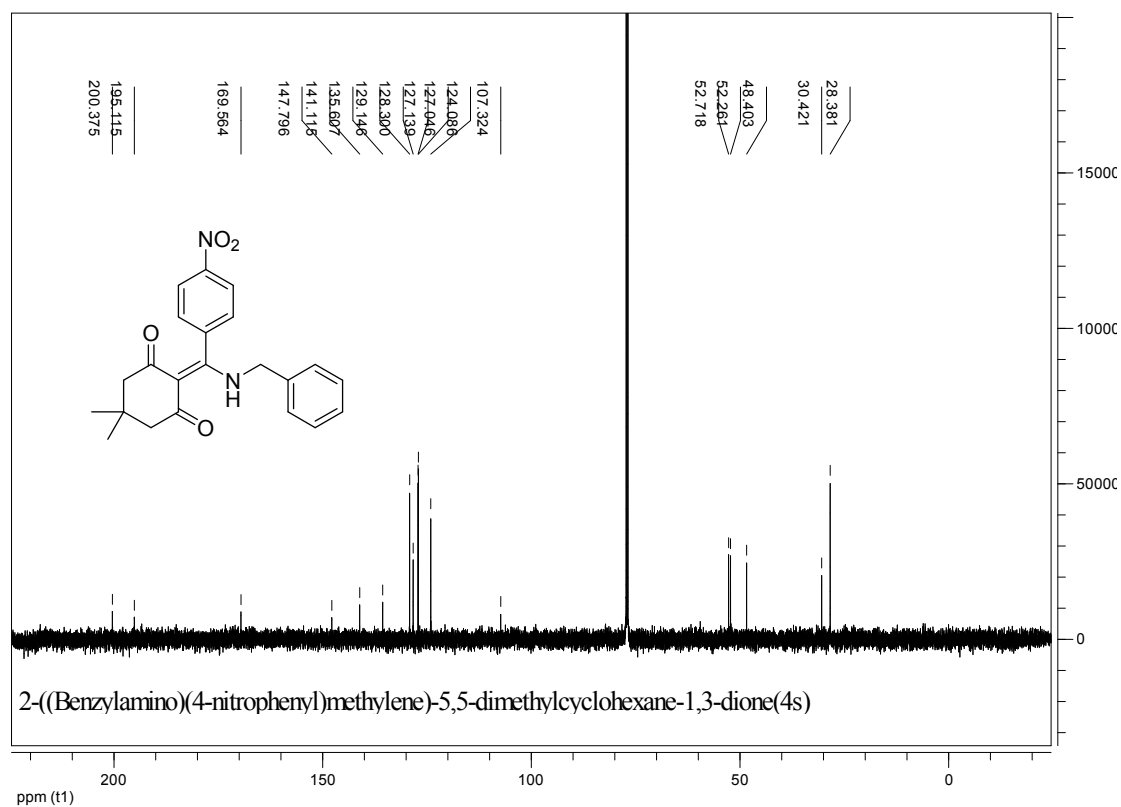
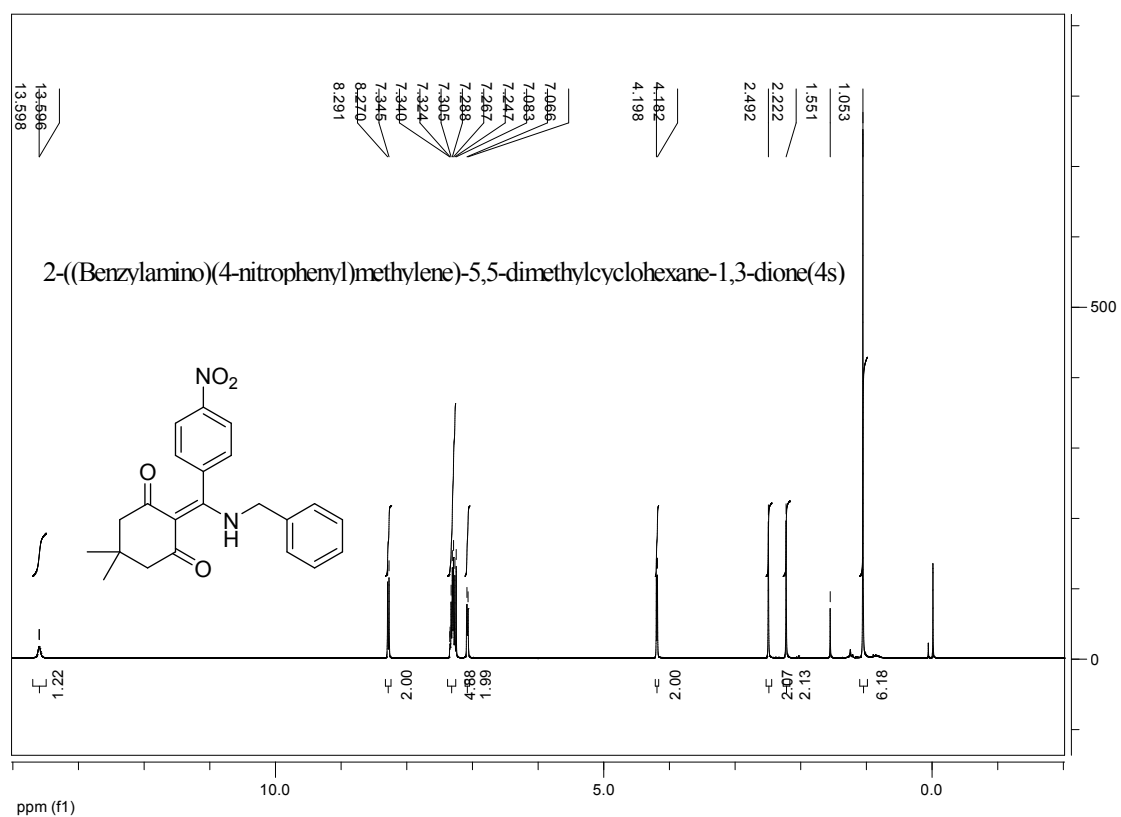
2-((4,4-Dimethyl-2,6-dioxocyclohexylidene)(4-methoxyphenyl)methylamino)propanoic acid(4q)



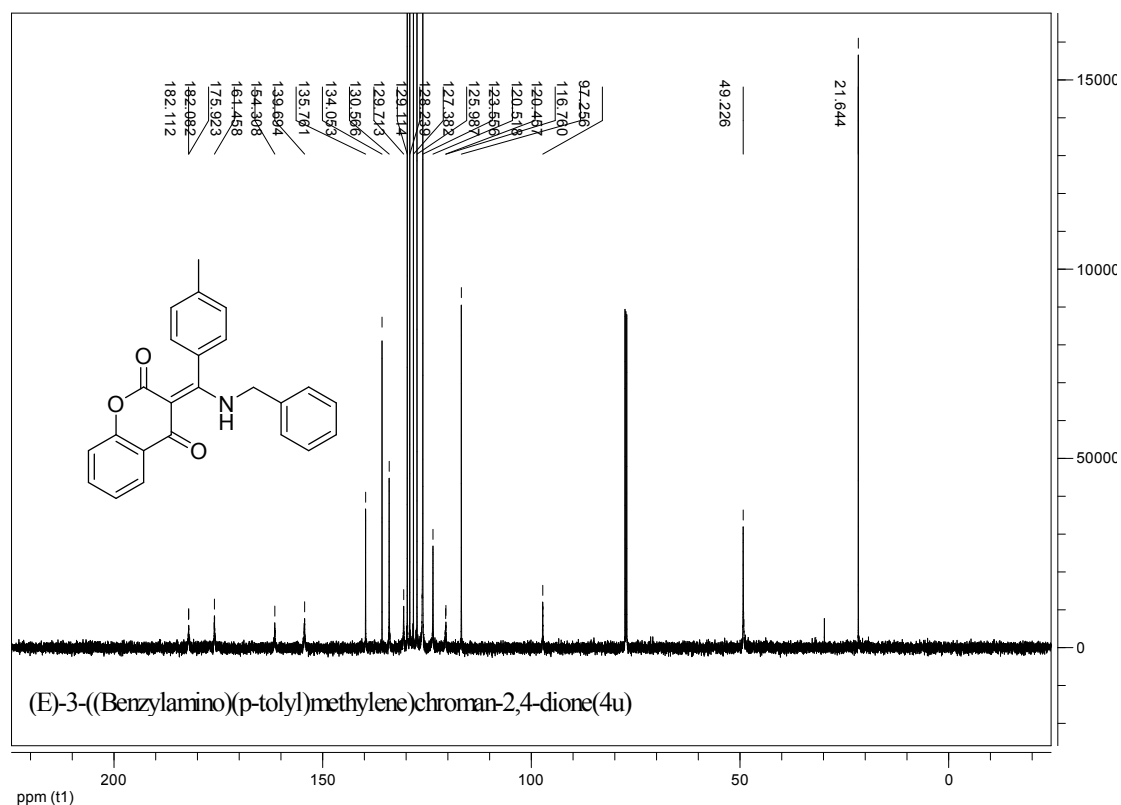
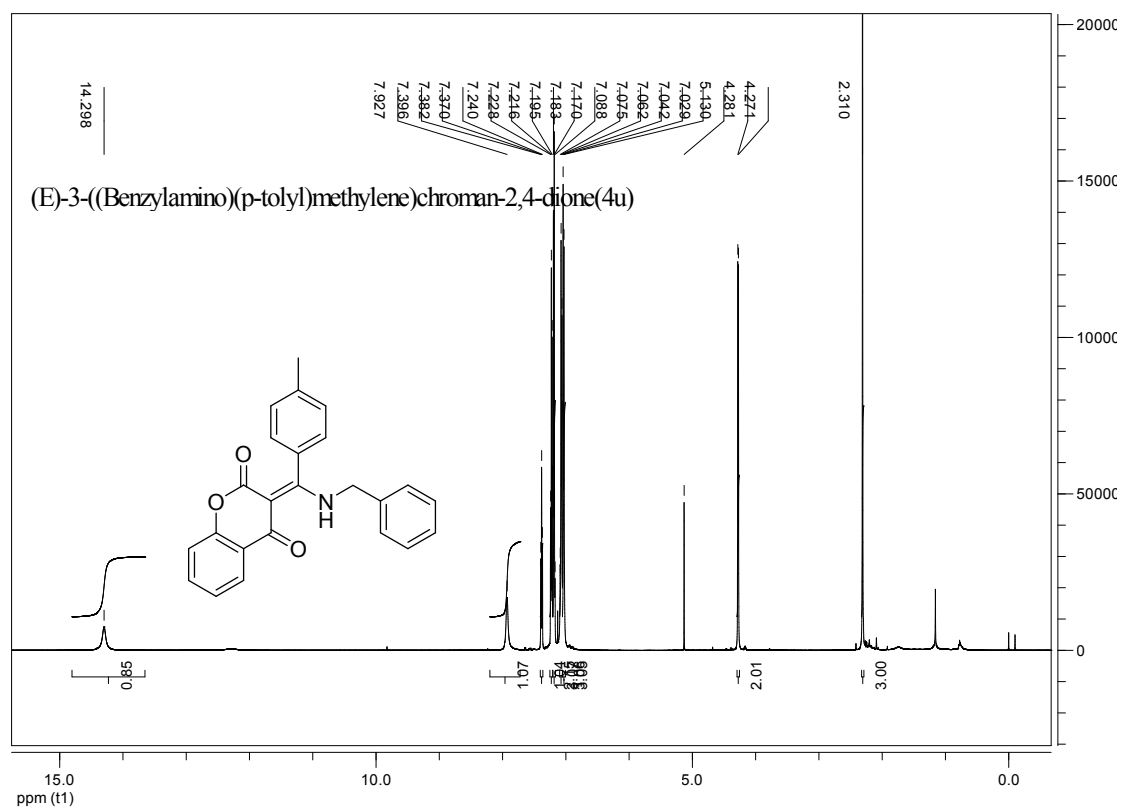
2-((Dihexylamino)(4-methoxyphenyl)methylene)-5,5-dimethylcyclohexane-1,3-dione(4r)



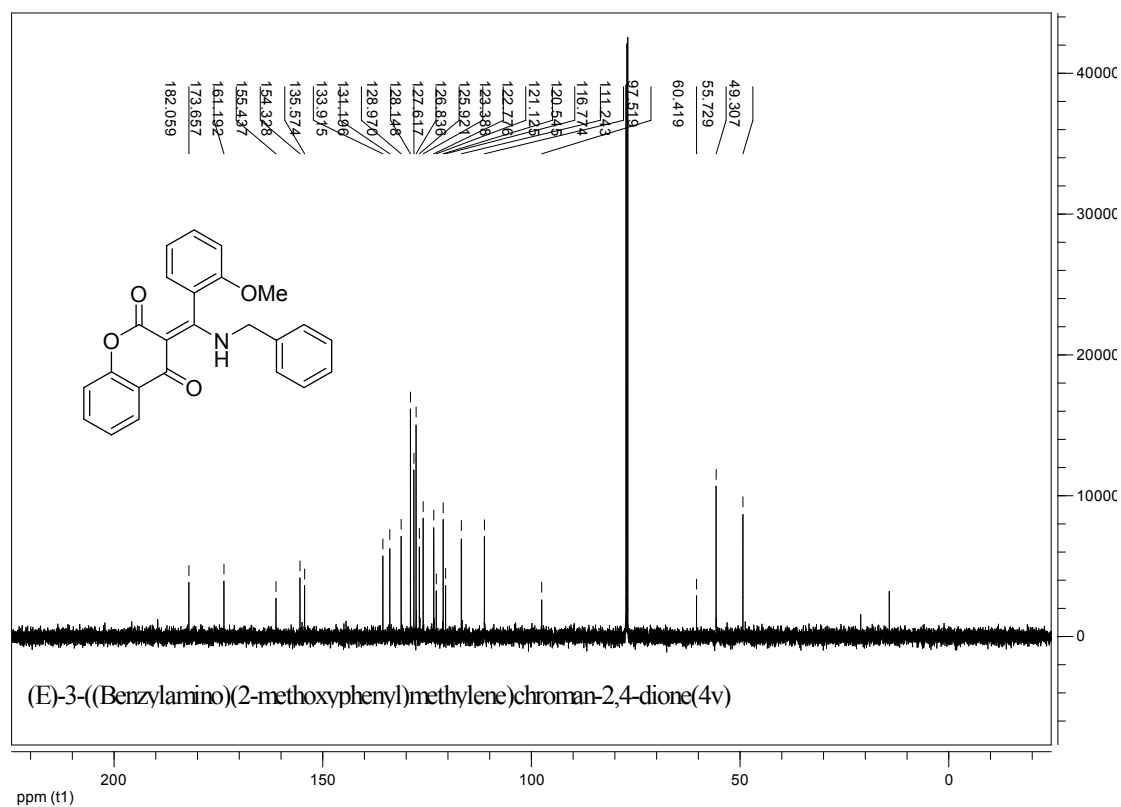
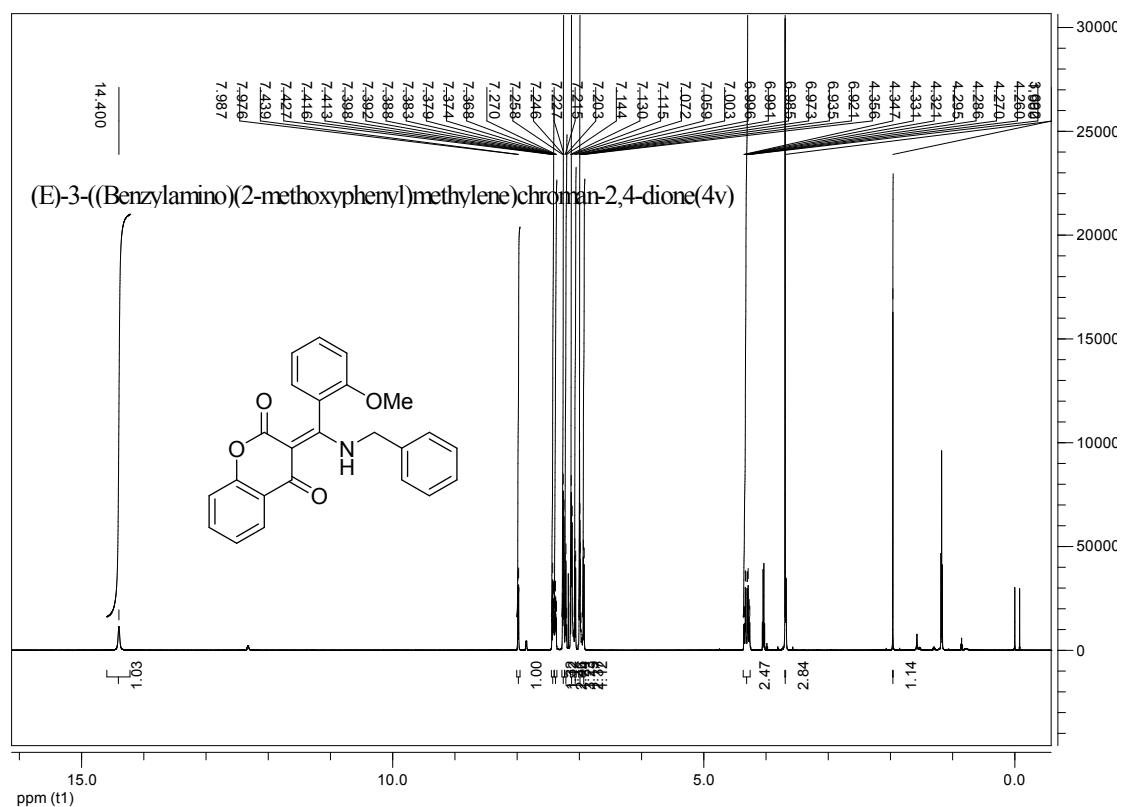
2-((Benzylamino)(4-nitrophenyl)methylene)-5,5-dimethylcyclohexane-1,3-dione(4s)



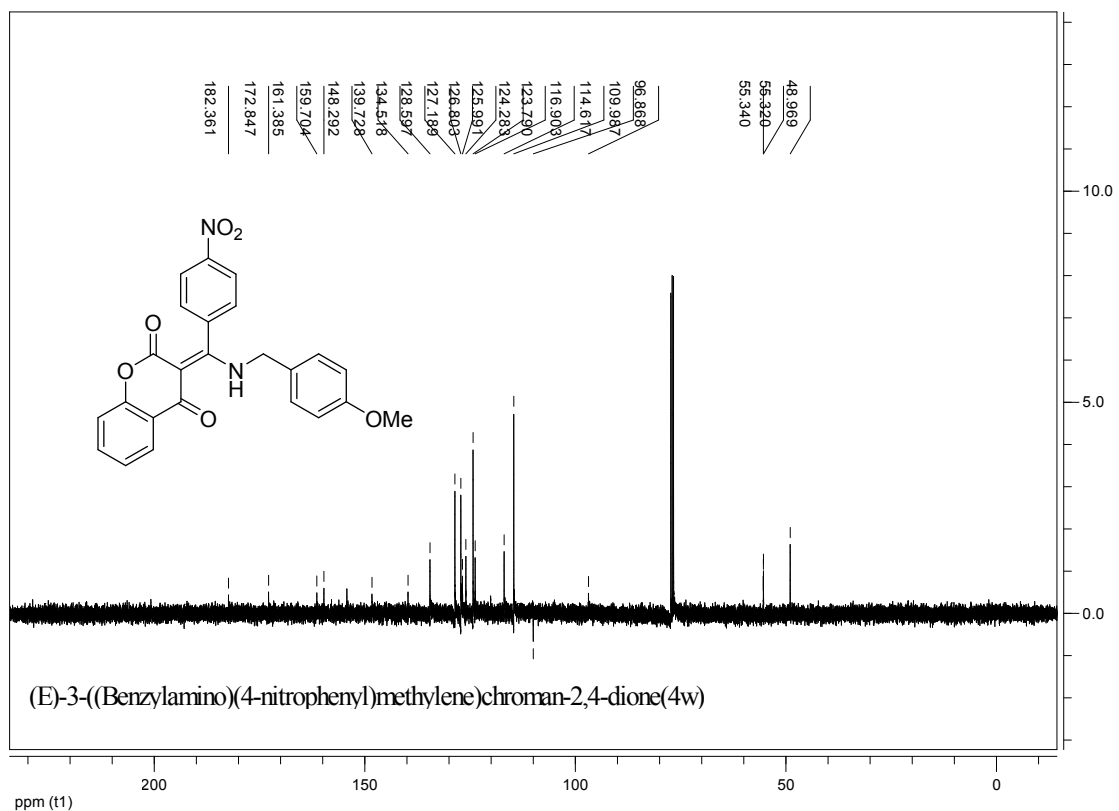
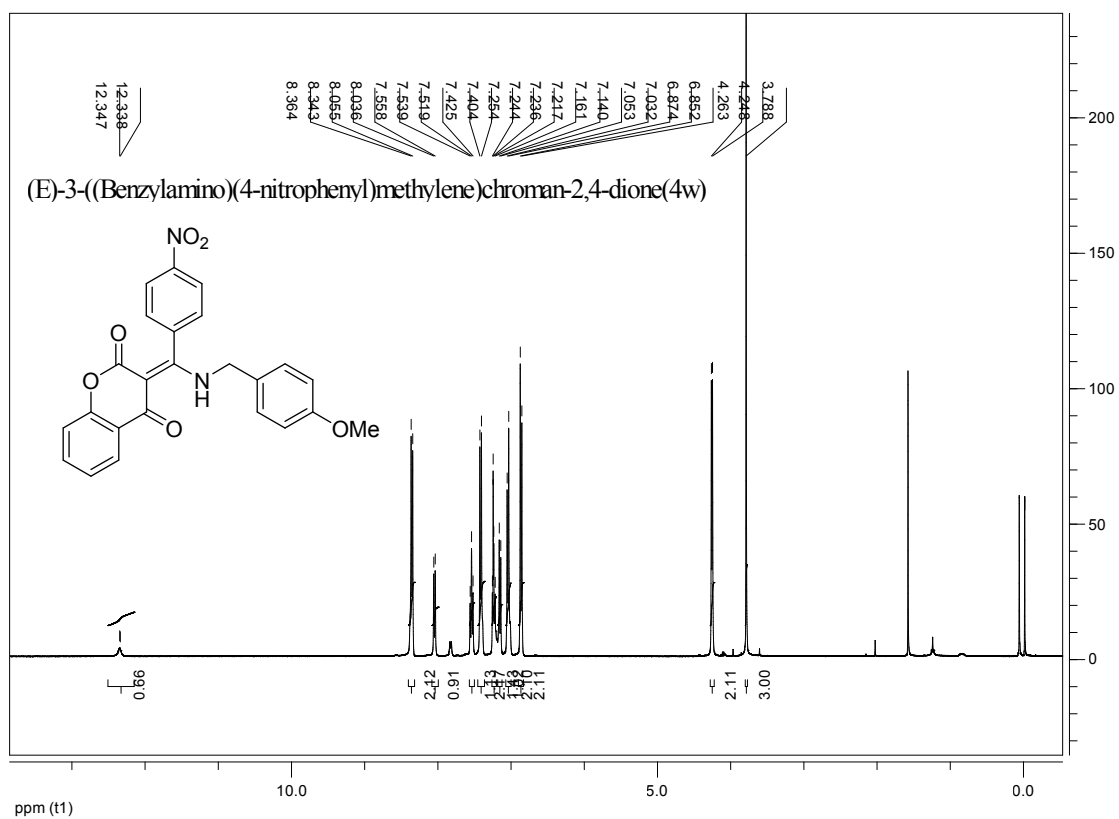
2-((Benzylamino)(4-chlorophenyl)methylene)-5-isopropylcyclohexane-1,3-dione(4t)



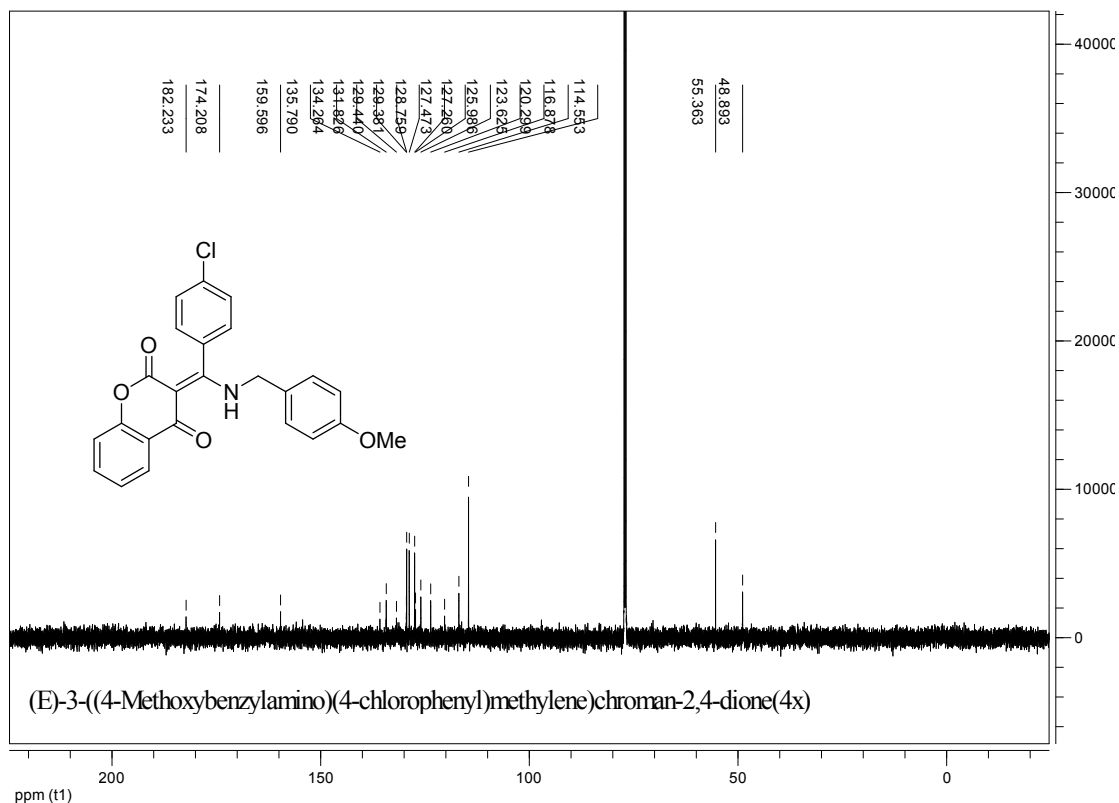
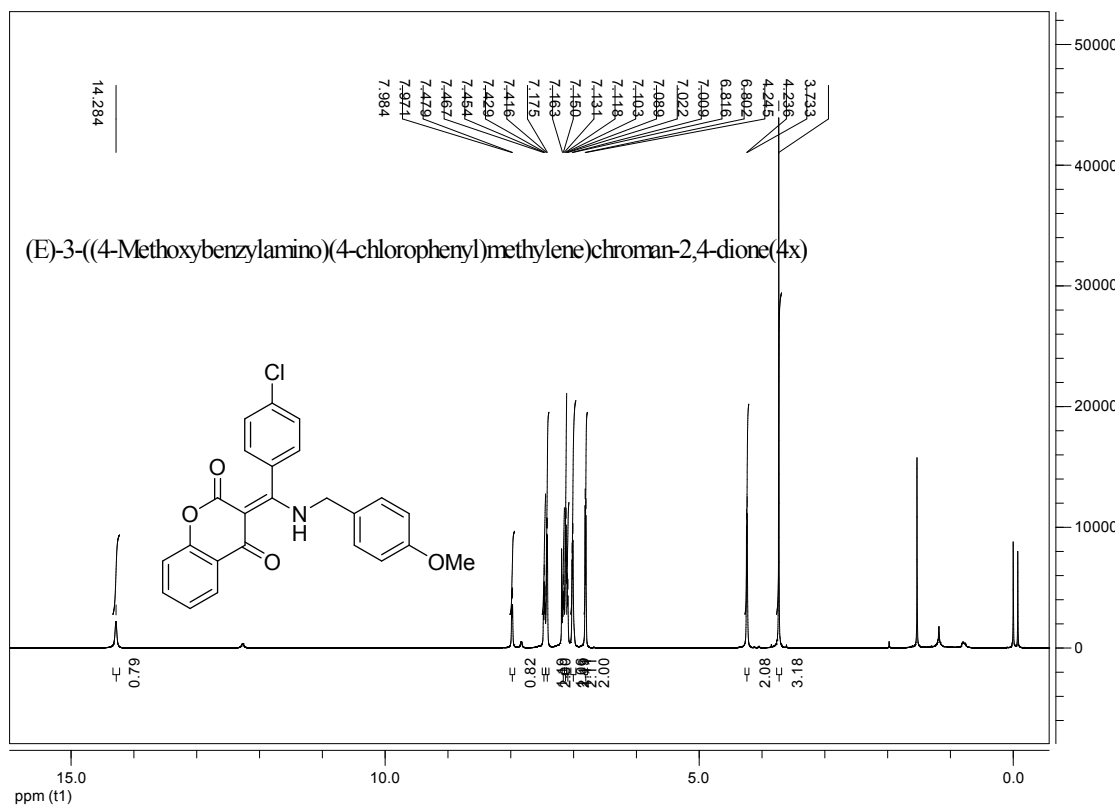
(E)-3-((Benzylamino)(2-methoxyphenyl)methylene)chroman-2,4-dione(4v)



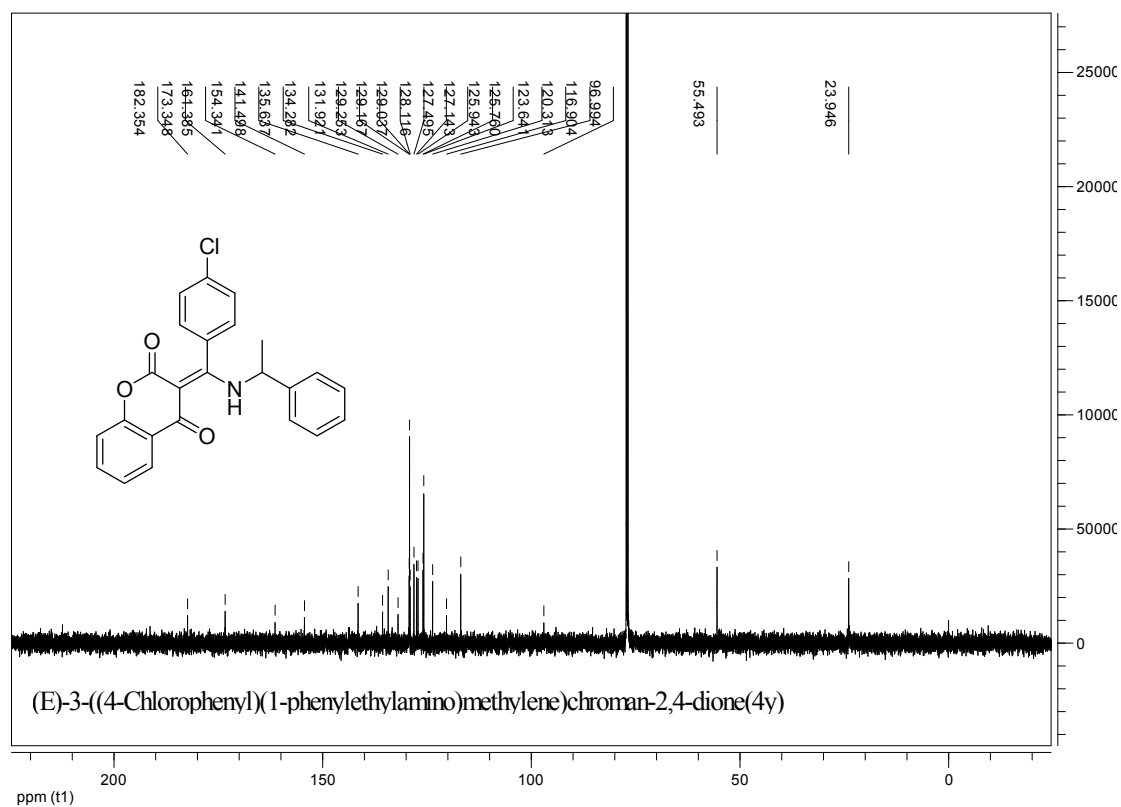
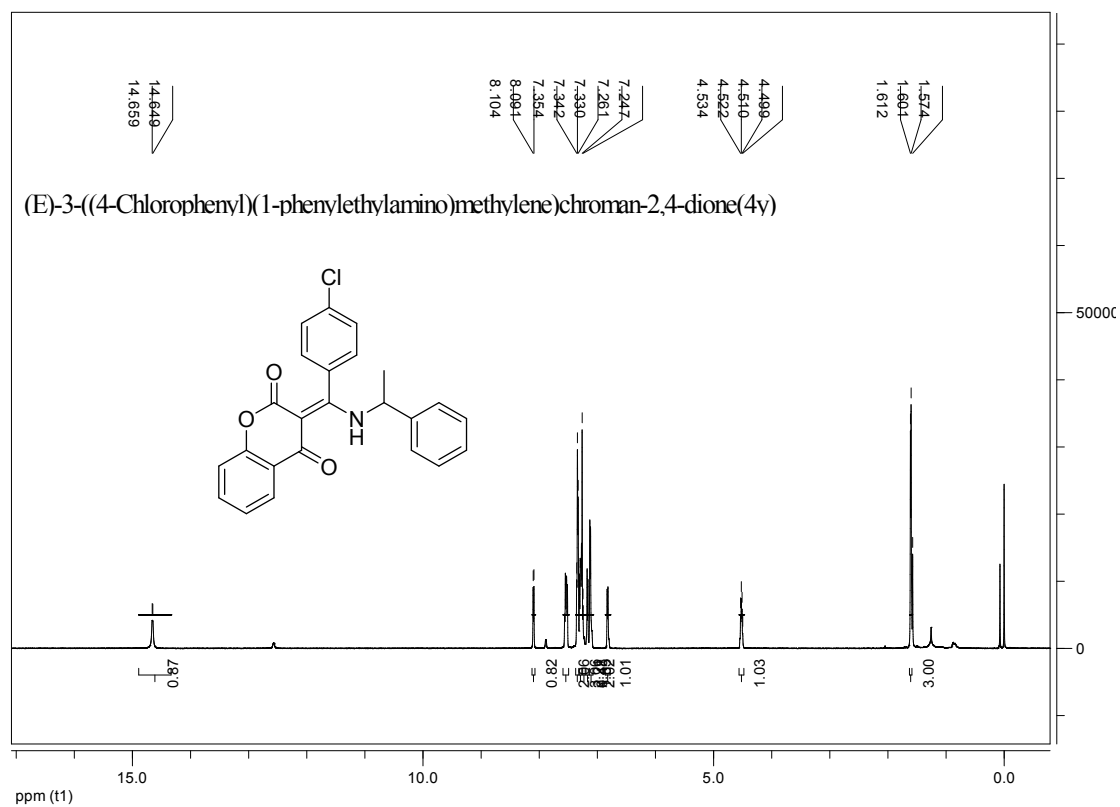
(E)-3-((Benzylamino)(4-nitrophenyl)methylene)chroman-2,4-dione(4w)



(E)-3-((4-Methoxybenzylamino)(4-chlorophenyl)methylene)chroman-2,4-dione(4x)



(E)-3-((4-Chlorophenyl)(1-phenylethylamino)methylene)chroman-2,4-dione(4y)



(E)-3-((Butylamino)(p-tolyl)methylene)chroman-2,4-dione(4z)

