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Supporting Information



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# Hydrogen-Borrowing and Interrupted-Hydrogen-Borrowing Reactions of Ketones and Methanol Catalyzed by Iridium**

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**Supporting Information**

# Contents



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## I. General Experimental

All solvents and reagents were used as commercially supplied without further purification unless otherwise stated. Anhydrous $CH_2Cl_2$, PhMe, and tetrahydrofuran (THF) were dried by filtration through an activated alumina purification column. Petrol refers to petroleum ether in the boiling range 40–60 $^oC$. Flash column chromatography (FCC) was performed using Merck Kieselgel 60 (40–63 μm). $^1H$ nuclear magnetic resonance spectra (NMR) were recorded on a Bruker DPX200 (200 MHz), Bruker AV400 (400 MHz) or Bruker AVII500 (500 MHz). $^{13}C$ NMR spectra were recorded on a Bruker AV400 (101 MHz) or AVII500 (126 MHz) as stated. $^{19}F$ NMR spectra are recorded on a Bruker AV400 (376 MHz) and are externally calibrated to $CFCl_3$. Chemical shifts are reported relative to residual solvent peaks. Coupling constants are quoted to the nearest 0.1 Hz for $^1H$ NMR and to the nearest 1 Hz for $^{13}C$ NMR. Mass spectra under the conditions of electrospray ionisation (ESI) were recorded on a Fisons Platform II. Mass spectra under the conditions of field ionisation (FI) were recorded on a Micromass LCT. Mass spectra under the conditions of chemical ionisation (CI) were recorded on a Fisons Autospec-oaTof. Fourier transform infrared spectra (FTIR) were recorded as evaporated films. Melting points (m.p) were obtained using a Leica VMTG heated-stage microscope and are uncorrected.

## II. General Procedure A (Ir-catalyzed alkylation)

Following literature procedure (Iuchi, Y.; Obora, Y.; Ishii, Y. *J. Am. Chem. Soc.* **2010**, *132*, 2536) with some modification where stated: Under an open atmosphere, $[Ir(cod)Cl]_2$, KOH and $PPh_3$ were added to a Biotage® microwave vial equipped with a stir bar, followed by methyl ketone and alcohol. The vial was sealed with a microwave vial cap (containing a Reseal™ septa) and degassed *via* a needle with a balloon of Ar. The mixture was stirred at 100 $^oC$ for the time stated. Purification (where necessary) by FCC afforded product.

## III. General Procedure B (Ir-catalyzed methylation)

Under an open atmosphere, $[Ir(cod)Cl]_2$, KOH and $PPh_3$ were added to a Biotage® microwave vial equipped with a stir bar, followed by "wet" methanol and starting ketone. The vial was sealed with a microwave vial cap (containing a Reseal™ septum) and a balloon of $O_2$ was inserted *via* a needle through the septum. The reaction was heated to 65 $^oC$ for the period of time stated. The reaction was quenched by diluting with diethyl ether and filtering through a pad of silica gel. Purification by FCC afforded product.

## IV. General Procedure C (Ir-catalyzed methylenation)

Under an open atmosphere, $[Ir(cod)Cl]_2$, KOH and cataCXium® A were added to a Biotage® microwave vial equipped with a stir bar, followed by "wet" methanol and starting ketone. The vial was sealed with a microwave vial cap (containing a Reseal™ septum) and degassed *via* a needle with a balloon of $O_2$. The reaction was heated to 65 $^oC$ for the period of time stated with the $O_2$ balloon attached. The reaction was quenched by diluting with diethyl ether and filtering through a pad of silica gel. Purification by FCC afforded product. (It is important to keep the balloon properly attached. If the

$O_2$ atmosphere is not sufficient, reaction mixture will turn brown from green and become less effective.)

## V. General Procedure D (Baeyer Villiger oxidation)

Under an open atmosphere, starting ketone, mCPBA, trifluoroacetic acid and $CH_2Cl_2$ were added to a Biotage® microwave vial equipped with a stir bar. The vial was sealed with a microwave vial cap (containing a Reseal™ septa) and stirred at rt for 48 h. The organic phase was washed with sat. aq. $NaHCO_3$ (3 x 10mL), extracted with $CH_2Cl_2$, dried over $Na_2SO_4$, filtered, and the solvent removed *in vacuo*. Purification by FCC afforded product.

## VI. General Procedure E (Pyridine formation using $NH_2OH{\cdot}HCl$)

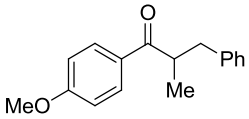
Under an open atmosphere, starting diketone (30 mg, 1 equiv.), hydroxylamine hydrochloride (3 equiv. ) were added to a Biotage® microwave vial equipped with a stir bar, followed by ethanol (0.15 mL). The vial was sealed with a microwave vial cap (containing a Reseal™ septum) and was heated to 80 ˚C for 24 h. The reaction was quenched by adding $NaHCO_3$ (sat.), extracted with $CH_2Cl_2$, then washed with brine, dried over $Na_2SO_4$, filtered, concentrated *in vacuo*. Purification by FCC afforded product.

## VII. General Procedure F (Pyridine formation using $NH_4OAc$)

Under an open atmosphere, starting diketone (30 mg, 1 equiv.), ammonium acetate (3 equiv. ), copper (II) acetate monohydrate (2.5 equiv.) were added to a Biotage® microwave vial equipped with a stir bar, followed by acetic acid (0.16 mL). The vial was sealed with a microwave vial cap (containing a Reseal™ septum), degassed with argon and heated to 120 ˚C for 24 h. The reaction was quenched by adding ammonia solution (28%-30%, 0.7 mL), extracted with EtOAc, then washed with brine, dried over $Na_2SO_4$, filtered, concentrated *in vacuo*. Purification by FCC afforded product.

## VIII. Compound Characterization

**(±)1-(4-Methoxyphenyl)-2-methyl-3-phenylpropan-1-one, 2**



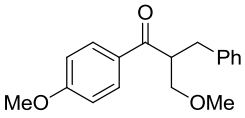

**Benzylation**: 4'-Methoxyacetophenone (300 mg, 2.00 mmol), $[Ir(cod)Cl]_2$ (13.4 mg, 0.0199 mmol), KOH (11.2 mg, 0.200 mmol), $PPh_3$ (21.0 mg, 0.0801 mmol), and benzyl alcohol (0.42 mL, 4.0 mmol) were subjected to general procedure A for 6 h. Purification by FCC (Petrol/$Et_2O$ 9:1) afforded 1-(4-methoxyphenyl)-3-phenylpropan-1-one (**1**, 418 mg, 1.74 mmol, 87 %) as a colourless solid. **m.p.** 95-97 $^oC$ (Lit: 96-98 $^oC$, Hajipour, A. R.; Ruoho, A. E.; Hajipour, A. R.; Khazdooz, L.; Zarei, A. *Synth.Commun.* **2009**, *39*, 2702); **$^1$H NMR** (400

MHz, $CDCl_3$) 7.94 (d, *J* 9.0 Hz, 2H), 7.16-7.33 (m, 5H), 6.92 (d, *J* 9.0 Hz, 2H), 3.86 (s, 3H), 3.05 (t, *J* 7.8 Hz, 2H), 2.25 (t, *J* 7.8 Hz, 2H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) 197.8, 163.4, 141.4, 130.3, 129.9, 128.5, 128.4, 126.0, 113.7, 55.4, 40.1, 30.3. All spectroscopic data were consistent with those previously reported: Hajipour, A. R.; Zarei, A.; Khazdooz, L.; Ruoho, A. E. *Synth. Comm.* **2009**, *39*, 2702.

**Methylation:** 1-(4-Methoxyphenyl)-3-phenylpropan-1-one (**1**, 72.0 mg, 0.300 mmol), $[Ir(cod)Cl]_2$ (2.0 mg, 0.0030 mmol), KOH (33.7 mg, 0.602 mmol), $PPh_3$ (3.2 mg, 0.012 mmol), MeOH (1.5 mL) were subjected to general procedure B for 48 h. Purification by FCC (Petrol/$Et_2O$ 10:1) afforded **2** (71.5 mg, 0.281 mmol, 94%) as a colorless oil.

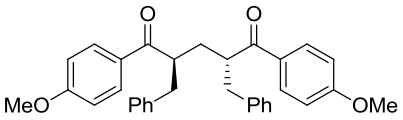
**One-pot dialkylation:** To a mixture of 4'-Methoxyacetophenone (150 mg, 1.00 mmol), [Ir (cod) $Cl]_2$ (6.7 mg, 0.010 mmol), KOH (5.6 mg, 0.10 mmol) and $PPh_3$ (10.5 mg, 0.0400 mmol) in a Biotage® microwave vial equipped with a stir bar was added benzyl alcohol (0.21 mL, 2.0 mmol). The vial was sealed with a microwave vial cap (containing a Reseal™ septa) and degassed *via* a needle with a balloon of Ar. The mixture was stirred at 100 $^{o}$C for 6 h before KOH (168 mg, 3.00 mmol), $PPh_3$ (10.5 mg, 0.0400 mmol) and MeOH (5 mL) were added, and the mixture was stirred under $O_2$ at 65 $^{0}$C for 48 h. Purification by FCC (Petrol/Et2O 10:1) afforded **2** (185 mg, 0.728 mmol, 73%) as a colorless oil. **$^{1}$H NMR** (400 MHz, $CDCl_3$) δ 7.91 (d, *J* 8.9 Hz, 2H), 7.13-7.28 (m, 5H), 6.91 (d, *J* 8.9 Hz, 2H), 3.84 (s, 3H), 3.65-3.74 (m, 1H), 3.15 (dd, *J* 6.7, 13.6 Hz, 1H), 2.68 (dd, *J* 7.9, 13.6 Hz, 1H), 1.19 (d, *J* 6.9 Hz, 3H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 202.3, 163.4, 140.2, 130.6, 129.3, 129.1, 128.4, 126.2, 113.8, 55.5, 42.4, 39.5, 17.6. Spectroscopic data are consistent with those previously reported: Samanta, S.; Mishra, B. K.; Pace, T. C. S.; Sathyamurthy, N.; Bohne, C.; Moorthy, J. N. *J. Org. Chem.* **2006**, *71*, 4453.

**(±)2-Benzyl-3-methoxy-1-(4-methoxyphenyl)propan-1-one, 4**

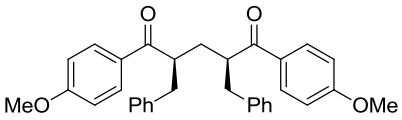



**Methylenation:** 1-(4-Methoxyphenyl)-3-phenylpropan-1-one (**1**, 72.0 mg, 0.300 mmol), $[Ir(cod)Cl]_2$ (4.0 mg, 0.0060 mmol), KOH (50.0mg, 0.893 mmol), cataCXium® A (8.4 mg, 0.024 mmol), MeOH (3.0 mL) were subjected to general procedure C for 48 h. Purification by FCC (Petrol/$Et_2O$ 8:2) afforded **4** (64.3 mg, 0.226 mmol, 75%) as a colorless oil. **IR $\nu_{max}$** ($cm^{-1}$) 2929, 1669, 1598, 1169, 842, 700; **$^{1}$H NMR** (500 MHz, $CDCl_3$) δ 7.89 (d, *J* 8.8 Hz, 2H), 7.25-7.15 (m, 5H), 6.89 (d, *J* 9.0 Hz, 2H), 3.98 (td, *J* 7.1, 1.9 Hz, 1H), 3.84 (s, 3H), 3.72 (dd, *J* 9.0, 7.4 Hz, 1H), 3.51 (dd, *J* 9.0, 5.4 Hz, 1H), 3.28 (s, 3H), 3.06 (dd, *J* 13.8, 7.5 Hz, 1H), 2.88 (dd, *J* 13.8, 6.7 Hz, 1H); **$^{13}$C NMR** (126 MHz, $CDCl_3$) δ 200.5, 163.5, 139.3, 130.7, 130.4, 129.0, 128.4, 126.3, 113.7, 73.7, 59.1, 55.4, 48.3, 35.5; **HRMS** ($ESI^+$) calculated for $[C_{18}H_{20}O_3+Na]^+$ 307.1305, found 307.1305, (Δ -0.3 ppm).

**(±)-(2*RR*, 4*RS*)-2,4-Dibenzyl-1,5-bis(4-methoxyphenyl)pentane-1,5-dione, 5**



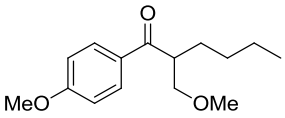

*anti*-XX




*syn*-XX

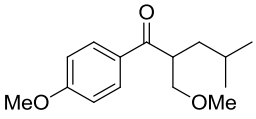
Under an open atmosphere, $[Ir(cod)Cl]_2$ (2.0 mg, 0.0030 mmol), $Cs_2CO_3$ (489 mg, 1.50 mmol) were added to a Biotage® microwave vial equipped with a stir bar, followed by "wet" methanol (0.75 mL) and 1-(4-methoxyphenyl)-3-phenylpropan-1-one (**1**, 72.1 mg, 0.300 mmol). The vial was sealed with a microwave vial cap (containing a Reseal™ septum) and a balloon of $O_2$ was inserted *via* a needle through the septum. The reaction was heated to 65 $^oC$ for 48 h. The reaction was quenched by dilution with diethyl ether and filtering, first through a cotton filter and then through a pad of silica gel. Purification by FCC (Petrol/$Et_2O$ 8:2) afforded **5** (61.0 mg, 83%) as a yellow oil. Dimers *anti*-**5** and *syn*-**5** were isolated as a 1:1 mixture of inseparable diastereoisomers. **IR $\nu_{max}$** ($cm^{-1}$) 2933, 2839, 1667, 1598, 1510, 1246, 1170, 1030, 836, 749, 700; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.88 (d, *J* 8.7, 4H), 7.50 (d, *J* 8.8, 4H), 7.21-7.07 (m, 16H), 6.99 (d, *J* 7.1, 4H), 6.92 (d, *J* 9.0, 4H), 6.61 (d, *J* 8.6, 4H), 3.86 (s, 6H), 3.74 (s, 6H), 3.76-3.71 (m, 2H), 3.67-3.60 (m, 2H), 3.06-3.02 (m, 2H), 3.01-2.97 (m, 2H), 2.74-2.69 (m, 2H), 2.67-2.62 (m, 2H), 2.41-2.35 (m, 1H), 2.12 (t, *J* 7.1, 2H), 1.75-1.69 (m, 1H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 202.4, 201.6, 163.6, 163.2, 139.6, 139.2, 130.8, 130.4, 130.4, 129.9, 129.0 (x2), 128.5, 128.4, 126.3, 126.7, 113.9, 113.5, 55.6, 55.3, 45.6, 45.2, 40.2, 38.5, 35.5, 34.8; **HRMS** ($ESI^+$) calculated for $[C_{33}H_{32}O_4+H]^+$ 493.2373, found 493.2367 (Δ –1.3 ppm).

**(±)2-(Methoxymethyl)-1-(4-methoxyphenyl)hexan-1-one, 7**



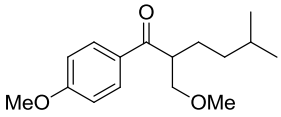

1-(4-Methoxyphenyl)hexan-1-one (**7a**, 61.8 mg, 0.300 mmol), $[Ir(cod)Cl]_2$ (4.0 mg, 0.0060 mmol), KOH (50.0mg, 0.893 mmol), cataCXium®A (8.4 mg, 0.024 mmol), MeOH (3.0 mL) were subjected to general procedure C for 48 h. Purification by FCC (Petrol/$Et_2O$ 8:2) afforded **7** (46.9 mg, 0.188 mmol, 63%) as a colorless oil. **IR $\nu_{max}$** ($cm^{-1}$) 2929, 1671, 1599, 1255, 1171, 843, 763; **$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.98 (d, *J* 8.8 Hz, 2H), 6.94 (d, *J* 8.8 Hz, 2H), 3.87 (s, 3H), 3.73-3.66 (m, 2H), 3.51-3.46 (m, 1H), 3.29 (s, 3H), 1.75-1.68 (m, 1H), 1.58-1.49 (m, 1H), 1.32-1.20 (m, 4H), 0.84 (t, *J* 7.0 Hz, 3H); **$^{13}$C NMR** (126 MHz, $CDCl_3$) δ 201.5, 163.5, 130.7, 130.7, 113.8, 74.4, 59.1, 55.4, 46.3, 29.7, 29.6, 22.9, 13.9; **HRMS** ($ESI^+$) calculated for $[C_{15}H_{22}O_3+Na]^+$ 273.1461, found 273.1458, (Δ 1.3 ppm).

**(±)2-(Methoxymethyl)-1-(4-methoxyphenyl)-4-methylpentan-1-one, 9**



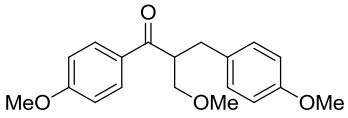
1-(4'-Methoxyphenyl)-4-methylpentan-1-one (**9a**, 61.8 mg, 0.300 mmol), $[Ir(cod)Cl]_2$ (4.0 mg, 0.0060 mmol), KOH (50.0mg, 0.893 mmol), cataCXium® A (8.4 mg, 0.024 mmol), MeOH (3.0 mL) were subjected to general procedure C for 48 h. Purification by FCC (Petrol/$Et_2O$ 8:2) afforded **9** (35.4 mg, 0.142 mmol, 47%) as a colorless oil. **IR $\nu_{max}$** ($cm^{-1}$) 2957, 1671, 1599, 1252, 1171, 842, 765; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.99 (d, *J* 9.0 Hz, 2H), 6.94 (d, *J* 9.0 Hz, 2H), 3.87 (s, 3H), 3.82-3.77 (m, 1H), 3.67 (t, *J* 8.5 Hz, 1H), 3.46 (dd, *J* 8.8, 5.2 Hz, 1H), 3.27 (s, 3H), 1.66 (ddd, *J* 13.5, 8.1, 6.6 Hz, 1H), 1.60-1.51 (m, 1H), 1.34 (ddd, *J* 13.4, 7.5, 5.8 Hz, 1H), 0.89 (d, *J* 6.5 Hz, 3H), 0.87 (d, *J* 6.5 Hz, 3H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 201,7, 163.5, 130.8, 130.6, 113.8, 75.0, 59.1, 55.4, 44.3, 39.0, 26.1, 23.0, 22.6; **HRMS** ($ESI^+$) calculated for $[C_{15}H_{22}O_3+Na]^+$ 273.1461, found 273.1464, (Δ -1.0 ppm).

**(±)2-(Methoxymethyl)-1-(4-methoxyphenyl)-5-methylhexan-1-one, 11**



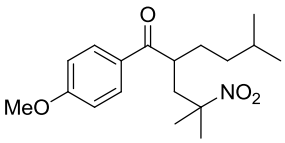

1-(4-Methoxyphenyl)-5-methylhexan-1-one (**11a**, 66.0 mg, 0.300 mmol), $Ir(cod)Cl]_2$ (4.0 mg, 0.0060 mmol), KOH (50.0mg, 0.893 mmol), cataCXium® (8.4 mg, 0.024 mmol), MeOH (3.0 mL) were subjected to general procedure C for 48 h. Purification by FCC (Petrol/$Et_2O$ 8:2) afforded **11** (52.2 mg, 0.200 mmol, 66%) as a colorless oil. **IR $\nu_{max}$** ($cm^{-1}$) 2955, 1671, 1599, 1255, 1170, 834, 760; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.90 (d, *J* 9.0 Hz, 2H), 6.87 (d, *J* 9.0 Hz, 2H), 3.79 (s, 3H), 3.65-3.55 (m, 2H), 3.44-3.38 (m, 1H), 3.21 (s, 3H), 1.68-1.55 (m, 1H), 1.52-1.37 (m, 2H), 1.09-1.03 (m, 2H), 0.76 (d, *J* 4.4 Hz, 3H), 0.74 (d, *J* 4.4 Hz, 3H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 201.5, 163.5, 130.7, 130.6, 113.7, 74.5, 59.1, 55.4, 46.5, 36.5, 28.2, 27.8, 22.4; **HRMS** ($ESI^+$) calculated for $[C_{16}H_{24}O_3+Na]^+$ 287.1618, found 287.1617, (Δ 0.4 ppm).

**(±)3-Methoxy-2-(4-methoxybenzyl)-1-(4-methoxyphenyl)propan-1-one, 13**



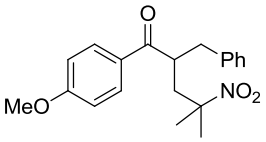

1,3-Bis(4-methoxyphenyl)propan-1-one (**13a**, 81.0 mg, 0.300 mmol), $[Ir(cod)Cl]_2$ (4.0 mg, 0.0060 mmol), KOH (50.0mg, 0.893 mmol), cataCXium® (8.4 mg, 0.024 mmol), MeOH (3.0 mL) were subjected to general procedure D for 48 h. Purification by FCC (Petrol/$Et_2O$ 7:3) afforded **13** (67.2 mg, 0.214 mmol, 71%) as a colorless oil. **IR $\nu_{max}$** ($cm^{-1}$) 2932, 2837, 1669, 1245, 840, 818; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.89 (d, *J* 8.8 Hz, 2H), 7.08 (d, *J* 8.6 Hz, 2H), 6.89 (d, *J* 9.0 Hz, 2H), 6.77 (d, *J* 8.6 Hz, 2H), 3.97-3.90 (m, 1H), 3.84 (s, 3H), 3.75 (s, 3H), 3.70 (dd, *J* 8.8, 7.6 Hz, 1H), 3.51-3.48 (m, 1H), 3.28 (s, 3H), 3.00 (dd, *J* 13.8, 7.5, 1H), 2.81 (dd, *J* 13.8, 6.7, 1H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 200.6, 163.4, 158.1, 131.3, 130.7, 130.5, 130.0, 113.8, 113.7, 73.6, 59.1, 55.4, 55.2, 48.5, 34.7; **HRMS** ($ESI^+$) calculated for $[C_{19}H_{22}O_4+H]^+$ 315.1591, found 315.1585, (Δ -1.8 ppm).

**(±)1-(4-Methoxyphenyl)-5-methyl-2-(2-methyl-2-nitropropyl)hexan-1-one, 14**



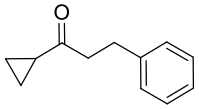

1-(4-Methoxyphenyl)-5-methylhexan-1-one (**11a**, 66.0 mg, 0.300 mmol). $[Ir(cod)Cl]_2$ (4.0 mg, 2 mol%), CataCXium A (8.6 mg, 8 mol%), KOH (50.0 mg, 0.900 mmol) and MeOH (3 mL) were subjected to general procedure C. After 48 h, SiliaMetS DMT (77.0 mg, 16 mol%) was added, and the resultant solution stirred at room temperature for 1 h open to the air. 2-Nitropropane (54 μL, 0.60 mmol), and KOH (34.0 mg, 0.600 mmol) were added, and the resultant solution stirred at 65 °C in a sealed tube under air for a further 14 h. The reaction was diluted with $Et_2O$, filtered for a short pad of silica, and concentrated *in vacuo*. Purification by FCC (Petrol/$Et_2O$ 20:1) afforded **14** (81.0 mg, 0.250 mmol, 84%) as a colourless oil. **IR $\nu_{max}$** ($cm^{-1}$) 2955, 1672, 1599, 1574, 1536, 1260, 1171; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.93 (d, *J* 9.0 Hz, 2H), 6.96 (d, *J* 9.0 Hz, 2H), 3.88 (s, 3H), 3.31-3.24 (m, 1H), 2.68 (dd, *J* 9.6, 14.8 Hz, 1H), 2.20 (dd, *J* 0.7, 14.7 Hz, 1H), 1.65-1.54 (m, 4H), 1.48-1.37 (m, 2H), 1.35 (s, 3H), 1.22-1.05 (m, 2H), 0.82 (d, *J* 6.6 Hz, 3H), 0.80 (d, *J* 6.6 Hz, 3H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 201.0, 163.7, 130.6, 129.3, 114.0, 88.4, 55.5, 41.6, 41.3, 35.8, 32.5, 28.2, 28.0, 24.5, 22.5, 22.2; **HRMS** ($ESI^+$) calculated for $[C_{18}H_{27}NO_4+H]^+$ 322.2013, found 322.2001, (Δ −2.0 ppm).

**(±)2-Benzyl-1-(4-methoxyphenyl)-4-methyl-4-nitropentan-1-one, 15**



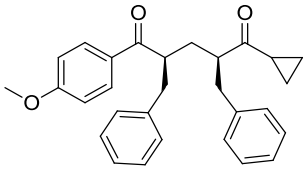

1-(4-Methoxyphenyl)-3-phenylpropan-1-one (**1**, 73.0 mg, 0.300 mmol). $[Ir(cod)Cl]_2$ (4.0 mg, 2 mol%), CataCXium A (8.6 mg, 8 mol%), KOH (50.0 mg, 0.900 mmol) and MeOH (3 mL) were subjected to general procedure C. After 48 h, SiliaMetS DMT (77.0 mg, 16 mol%) was added, and the resultant solution stirred at room temperature for 1 h open to the air. 2-Nitropropane (54 μL, 0.60 mmol), and KOH (34.0 mg, 0.60 mmol) were added, and the resultant solution stirred at 65 °C in a sealed tube under air for a further 14 h. The reaction was diluted with $Et_2O$, filtered for a short pad of silica, and concentrated *in vacuo*. Purification by FCC (Petrol/$Et_2O$20:1) afforded **15** (96.0 mg, 0.280 mmol, 94%) as a colourless oil. **IR $\nu_{max}$** ($cm^{-1}$) 2935, 1671, 1598, 1535, 1260, 1235, 1169; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.87 (d, *J* 8.9 Hz, 2H), 7.28-7.21 (m, 2H), 7.20-7.15 (m, 1H), 7.13-7.08 (m, 2H), 6.91 (d, *J* 8.9 Hz, 2H), 3.87 (s, 3H), 3.72-3.64 (m, 1H), 2.97 (dd, *J* 6.3, 13.8, 1H), 2.72-2.59 (m, 2H), 2.18 (dd, *J* 1.1, 14.7 Hz, 1H), 1.50 (s, 3H), 1.34 (s, 3H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 200.6, 163.7, 137.9, 130.6, 129.3, 129.0, 128.5, 126.7, 113.9, 88.0, 55.5, 43.4, 40.9, 40.5, 27.7, 24.9; **HRMS** ($ESI^+$) calculated for $[C_{20}H_{23}NO_4+Na]^+$ 364.1519 , found 364.1519, (Δ 0.1 ppm).

**1-Cyclopropyl-3-phenylpropan-1-one, 16a**

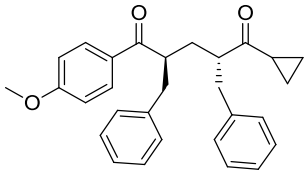


Cyclopropyl methyl ketone (0.40 mL, 4.00 mmol), $[Ir(cod)Cl]_2$ (26.8 mg, 0.0400 mmol), KOH (22.4 mg, 0.400 mmol), $PPh_3$ (42.0 mg, 0.160 mmol), and benzyl alcohol (2.1 mL, 20.0 mmol) were subjected to general procedure A for 4 h. Two of such reactions were combined and purified by FCC (Petrol/$Et_2O$ 10:1) afforded **16a** (1.30 g, 7.47 mmol, 93 %) as a colourless oil. **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.33-7.21 (m, 5H), 2.98-2.89 (m, 4H), 1.94 (m, 1H), 1.05 (m, 2H), 0.89 (m, 2H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 209.9, 141.2, 128.4, 128.3, 126.0, 44.9, 29.9, 20.5, 10.7. All spectroscopic data were consistent with those previously reported: Fernµndez, R.; Ferrete, A.; Llera, J. M.; Magriz, A.; Martín-Zamora, E.; Díez, E.; Lassaletta, J. M. *Chem. Eur. J.* **2004**, *10*, 737.

**(±)(2*RS*,4*SS*)-2,4-Dibenzyl-1-cyclopropyl-5-(4-methoxyphenyl)pentane-1,5-dione, 16**



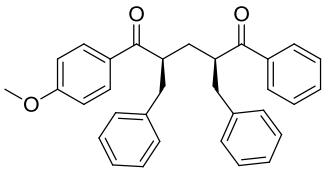
*Syn*-XX



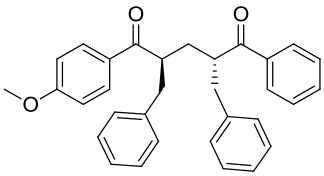
*Anti*-XX

Under an open atmosphere, $[Ir(cod)Cl]_2$ (13.3 mg, 0.0200 mmol), KOH (167 mg, 3 mmol) and cataCXium® (28.0 mg, 0.0800 mmol) were added to a Biotage® microwave vial equipped with a stir bar, followed by "wet" methanol (10 mL) and 1-(4-methoxyphenyl)-3-phenylpropan-1-one (**1**, 240.0 mg, 1 mmol). The vial was sealed with a microwave vial cap (containing a Reseal™ septum) and a balloon of $O_2$ was inserted via a needle through the septum. The reaction was heated to 65 ˚C for 48 h before being concentrated to 0.4 M (stream of Ar) and SiliaMetS DMT (257 mg, 0.16 mmol) was added. The mixture was stirred at rt open to air for 1 h before 1-cyclopropyl-3-phenylpropan-1-one (**16a**, 523 mg, 3 mmol) and KOH (167 mg, 3 mmol) were added. The vial was sealed and heated at 65 ˚C under $O_2$ for 21 h, and another 2 equiv. KOH (112.0 mg, 1 mmol) was added, heated for another 24 h. Crude mixture was diluted with $Et_2O$, filtered through $SiO_2$, concentrated *in vacuo*. Purification by FCC (Petrol/$Et_2O$ 8:2) afforded **16** as a light yellow oil (277 mg, 0.650 mmol, 65%). IR $\nu_{max}$ ($cm^{-1}$) 3062, 3027, 2934, 2841, 1598, 1259, 1169, 699; $^1$H NMR (500 MHz, $CDCl_3$) δ 7.89 (d, *J* = 8.8 Hz, 2 H), 7.84 (d, *J* = 9.0 Hz, 2 H), 7.30 - 7.10 (m, 18 H), 7.06 - 7.02 (m, 2 H), 6.92 (d, *J* = 8.8 Hz, 2 H), 6.88 (d, *J* = 8.8 Hz, 2 H), 3.86 - 3.82 (m, 3 H), 3.81 - 3.77 (m, 3 H), 3.77 - 3.64 (m, 2 H), 3.14 - 2.87 (m, 6 H), 2.80 - 2.69 (m, 3 H), 2.63 (dd, *J* = 6.7, 13.5 Hz, 1 H), 2.36 (td, *J* = 7.1, 14.1 Hz, 1 H), 2.14 - 2.05 (m, 1 H), 2.04 - 1.96 (m, 1 H), 1.94 - 1.84 (m, 1 H), 1.67 (td, *J* = 6.6, 13.8 Hz, 1 H), 1.57 - 1.48 (m, 1 H), 1.07 - 0.92 (m, 2 H), 0.91 - 0.79 (m, 3 H), 0.79 - 0.71 (m, 1 H), 0.65 - 0.51 (m, 2 H); $^{13}$C NMR (126 MHz, $CDCl_3$) δ 213.7, 213.1, 202.0, 201.4, 163.6, 163.5, 139.6, 139.4, 139.2, 138.9, 130.7, 130.6, 130.5, 129.8, 129.0, 129.0, 129.0, 128.5, 128.4, 128.4, 126.4, 126.3, 113.9, 113.8, 55.5, 55.4, 52.8, 52.6, 45.7, 45.3, 39.9, 39.3, 38.3, 38.1, 33.8, 33.7, 20.9, 20.7, 11.7, 11.5, 11.5, 11.5 (4 aromatic carbons were not observed due to overlapping); HRMS ($ESI^+$) calculated for $[C_{29}H_{30}O_3+H]^+$ 427.2268, found 427.2259 (Δ –2.1 ppm).

**(±)(2*SS*,4*RS*)-2,4-Dibenzyl-1-(4-methoxyphenyl)-5-phenylpentane-1,5-dione, 17**



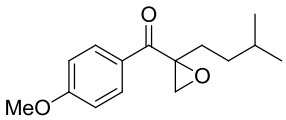
*Syn*-XX



*Anti*-XX

Under an open atmosphere, $[Ir(cod)Cl]_2$ (13.3 mg, 0.0200 mmol), KOH (167 mg, 3 mmol) and cataCXium® A (28.0 mg, 0.0800 mmol) were added to a Biotage® microwave vial equipped with a stir bar, followed by "wet" methanol (10 mL) and 1-(4-methoxyphenyl)-3-phenylpropan-1-one (**1**, 240.0 mg, 1 mmol). The vial was sealed with a microwave vial cap (containing a Reseal™ septum) and a balloon of $O_2$ was inserted via a needle through the septum. The reaction was heated to 65 ˚C for 48 h before concentrated to 0.4 M (stream of Ar) and SiliaMetS DMT (257 mg, 0.16 mmol) was added. The mixture was stirred at rt open to air for 1 h before 3-phenylpropiophenone (630 mg, 3 mmol) and KOH (167 mg, 3 mmol) were added. The vial was sealed and heated at 65 ˚C for 24 h under $O_2$ and another 1 equiv. KOH was added (56 mg, 1 mmol), heated for another 18 h. Crude mixture was diluted with $Et_2O$, filtered through $SiO_2$, concentrated *in vacuo*. Purification by FCC (Petrol/$Et_2O$ 8:2) afforded **17** as a light yellow oil (333 mg, 0.721 mmol, 72%). Dimers *anti*-**17** and *syn*-**17** were isolated as a 1:1 mixture of inseparable diastereoisomers. **IR $\nu_{max}$** ($cm^{-1}$) 3061, 3027, 2923, 2851, 2361, 2341, 1674, 1599, 1259, 1245, 1171, 699; **$^1$H NMR** (500 MHz, $CDCl_3$) δ 7.90-7.87 (m, 4H), 7.58-7.51 (m, 5H), 7.46-7.43 (m, 2H), 7.39-7.35 (m, 1H), 7.22-7.08 (m, 18H), 7.01-6.99 (m, 4H), 6.94-6.92 (m, 2H), 6.64-6.63 (m, 2H), 3.87 (s, 3H), 3.84-3.66 (m, 4H), 3.74 (s, 3H), 3.07-2.98 (m, 4H), 2.75-2.63 (m, 4H), 2.43-2.38 (m, 1H), 2.15-2.12 (m, 2H), 1.77-1.72 (m, 1H); **$^{13}$C NMR** (126 MHz, $CDCl_3$) δ 203.9, 203.2, 202.3, 201.5, 163.6, 163.3, 139.5, 139.3, 139.1, 138.8, 137.2, 136.8, 133.1, 132.7, 130.7, 130.4, 129.8, 129.0, 128.9, 128.9, 128.7, 128.5, 128.4, 128.4, 128.4, 128.4, 128.3, 128.0, 126.4, 126.3, 126.3, 113.9, 113.5, 55.5, 55.4, 46.0, 45.6, 45.5, 45.2, 39.9, 39.9, 38.5, 38.3, 34.9, 34.5 (3 carbon peaks are not observed due to overlapping); **HRMS** ($ESI^+$) calculated for $[C_{32}H_{30}O_3+H]^+$ 463.2268, found 463.2260 (Δ –1.6 ppm).

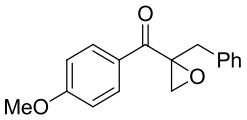
**(±) (2-Benzyloxiran-2-yl)(4-methoxyphenyl)methanone, 18**



1-(4-Methoxyphenyl)-5-methylhexan-1-one (**11a**, 66.0 mg, 0.300 mmol). $[Ir(cod)Cl]_2$ (4.0 mg, 2 mol%), CataCXium A (8.6 mg, 8 mol%), KOH (50.0 mg, 0.900 mmol) and MeOH (3 mL) were subjected to general procedure C. After 48 h, SiliaMetS DMT (77.0 mg, 16 mol%) was added, and the resultant solution stirred at room temperature for 1 h open to the air. *tert*-Butylhydroperoxide (410 μL, 3.00 mmol), and Triton B (1.62 mL, 3.00 mmol) were added, and the resultant solution stirred at rt for a further 15 h. The reaction was diluted with $Et_2O$, filtered for a short pad of silica, and concentrated *in vacuo*. Purification by FCC (Petrol/$Et_2O$

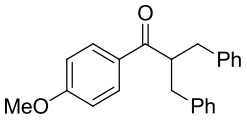
20:1) afforded **18** (67.0 mg, 0.270 mmol, 89%) as a colourless oil. **IR $\nu_{max}$** ($cm^{-1}$) 2956, 1667, 1599, 1257, 1165; **$^{1}$H NMR** (400 MHz, $CDCl_3$) δ 8.07 (d, *J* 9.0 Hz, 2H), 6.94 (d, *J* 9.0 Hz, 2H), 3.88 (s, 3H), 2.93 (d, *J* 5.0 Hz, 1H), 2.88 (d, *J* 5.0 Hz, 1H), 2.23 (ddd, *J* 5.1, 11.8, 14.0 Hz, 1H), 1.72 (ddd, *J* 5.1, 11.7, 14.1 Hz, 1H), 1.61-1.48 (m, 1H), 1.44-1.21 (m, 2H), 0.86 (d, *J* 6.6 Hz, 6H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 196.4, 163.8, 131.8, 127.6, 113.7, 63.6, 55.5, 50.9, 33.5, 31.3, 28.0, 22.3, 22.3; **m/z** (ESI+) 271.1 **HRMS** ($ESI^+$) calculated for $[C_{15}H_{20}O_3+H]^+$ 249.1485, found 249.1482, (Δ −1.1 ppm).

**(±) (2-Benzyloxiran-2-yl)(4-methoxyphenyl)methanone, 19**




1-(4-Methoxyphenyl)-3-phenylpropan-1-one (**1**, 73.0 mg, 0.300 mmol). $[Ir(cod)Cl]_2$ (4.0 mg, 2 mol%), CataCXium A (8.6 mg, 8 mol%), KOH (50.0 mg, 0.900 mmol) and MeOH (3 mL) were subjected to general procedure C. After 48 h, SiliaMetS DMT (77.0 mg, 16 mol%) was added, and the resultant solution stirred at room temperature for 1 h open to the air. *tert*-Butylhydroperoxide (410 μL, 3.00 mmol), and Triton B (1.62 mL, 3.00 mmol) were added, and the resultant solution stirred at rt for a further 15 h. The reaction was diluted with $Et_2O$, filtered for a short pad of silica, and concentrated *in vacuo*. Purification by FCC (Petrol/$Et_2O$ 20:1) afforded **19** (65.0 mg, 0.240 mmol, 81%) as a colourless oil. **IR $\nu_{max}$** ($cm^{-1}$) 3031, 2932, 1664, 1598, 1258, 1173; **$^{1}$H NMR** (400 MHz, $CDCl_3$) δ 7.87 (d, *J* 9.0 Hz, 2H), 7.21-7.10 (m, 5H), 6.79 (d, *J* 9.0 Hz, 2H), 3.76 (s, 3H), 3.52 (d, *J* 14.6 Hz, 1H), 3.00 (d, *J* 14.6 Hz, 1H), 2.77 (d, *J* 5.0 Hz, 1H), 2.73 (d, *J* 5.0 Hz, 1H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 196.3, 163.7, 135.3, 131.8, 129.8, 128.3, 127.5, 126.9, 113.6, 63.5, 55.4, 50.3, 38.7; **HRMS** ($ESI^+$) calculated for $[C_{17}H_{16}O_3+Na]^+$ 291.0992, found 291.0989 (Δ 1.1 ppm).

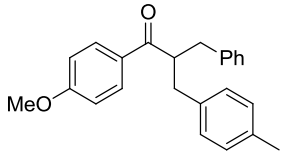
**2-Benzyl-1-(4-methoxyphenyl)-3-phenylpropan-1-one, 20**




1-(4-Methoxyphenyl)-3-phenylpropan-1-one (**1**, 73.0 mg, 0.300 mmol). $[Ir(cod)Cl]_2$ (4.0 mg, 2 mol%), CataCXium A (8.6 mg, 8 mol%), KOH (50.0 mg, 0.900 mmol) and MeOH (3 mL) were subjected to general procedure C. After 48 h, the reaction was concentrated to 0.25 M (1.2 mL) by bubbling argon through the solution. Subsequently, $[Rh(cod)Cl]_2$ (3.7 mg, 2.5 mol%), 1,5-cyclooctadiene (3.7 μL, 10 mol%), KOH (50.0 mg, 0.900 mmol) and 1,3,5-triphenylboroxine (47.0 mg, 0.450 mmol) were added. The vial was sealed with a microwave vial cap (containing a Reseal™ septum) and degassed with a ballon of Ar *via* a needle inserted through the septum. The balloon was them removed, and the reaction heated at 100 °C for 6 h. The reaction was diluted with $Et_2O$, filtered through a short plug of $SiO_2$, and concentrated *in vacuo*. Purification by FCC (PhMe) afforded **20** (72.0 mg, 0.220 mmol, 73%) as a colourless oil. **IR $\nu_{max}$** ($cm^{-1}$) 3027, 2932, 1669, 1599, 1261, 1238, 1170; **$^{1}$H NMR** (400

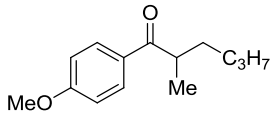
MHz, $CDCl_3$) δ 7.75 (d, *J* 9.0 Hz, 2H), 7.28-7.11 (m, 10H), 6.82 (d, *J* 9.0 Hz, 2H), 3.98 (tt, *J* 6.2 Hz, 7.9 Hz, 1H), 3.79 (s, 3H), 3.14 (dd, *J* 8.0, 13.8 Hz, 2H), 2.81 (dd, *J* 6.3, 13.8 Hz, 2H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 201.6, 163.2, 139.7, 130.4, 130.3, 129.0, 128.3, 126.2, 113.6, 55.4, 49.9, 38.3; **m/z** 363.1 **HRMS** ($ESI^+$) calculated for $[C_{23}H_{22}O_2+Na]^+$ 353.1512, found 353.1511, (Δ 0.3 ppm).

**2-Benzyl-3-(4-bromophenyl)-1-(4-methoxyphenyl)propan-1-one, 21**




1-(4-Methoxyphenyl)-3-phenylpropan-1-one (**1**, 73.0 mg, 0.300 mmol). $[Ir(cod)Cl]_2$ (4.0 mg, 2 mol%), CataCXium A (8.6 mg, 8 mol%), KOH (50.0 mg, 0.900 mmol) and MeOH (3 mL) were subjected to general procedure C. After 48 h, the reaction was concentrated to 0.25 M (1.2 mL) by bubbling argon through the solution. Subsequently, $[Rh(cod)Cl]_2$ (3.7 mg, 2.5 mol%), 1,5-cyclooctadiene (3.7 μL, 10 mol%), KOH (50.0 mg, 0.900 mmol) and 1 *p*-tolylboronic acid (61 mg, 1.50 mmol) were added. The vial was sealed with a microwave vial cap (containing a Reseal™ septum) and degassed with a ballon of Ar *via* a needle inserted through the septum. The balloon was them removed, and the reaction heated at 100 °C for 6 h. The reaction was diluted with $Et_2O$, filtered through a short plug of $SiO_2$, and concentrated *in vacuo*. Purification by FCC (PhMe) afforded **21** (65 mg, 0.19 mmol, 63%) **IR $\nu_{max}$** ($cm^{-1}$) 2906, 1689, 1598, 1510, 1237, 1169; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.65 (d, *J* 8.8 Hz, 2 H), 7.12 - 6.98 (m, 5 H), 6.96 - 6.88 (m, 4 H), 6.71 - 6.65 (m, 2 H), 3.85 ((quin, *J* 7.0 Hz, 1 H), 3.71 - 3.59 (m, 3 H), 3.00 (td, *J* 7.8, 13.8 Hz, 2 H), 2.67 (dt, *J* 6.2, 13.8 Hz, 2 H), 2.15 (s, 3 H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 201.6, 163.2, 139.7, 136.5, 135.6, 130.4, 130.3, 129.0, 129.0, 128.8, 128.3, 126.1, 113.6, 55.3, 50.0, 38.1, 37.8, 20.9; **m/z** 363.1 **HRMS** ($ESI^+$) calculated for $[C_{24}H_{24}O_2+Na]^+$ 367.1669, found 367.1661 , (Δ -2.1 ppm).

**(±)1-(4-Methoxyphenyl)-2-methylhexan-1-one, 22**

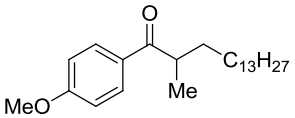



**Butylation**: 4'-Methoxyacetophenone (300 mg, 2.00 mmol), $[Ir(cod)Cl]_2$ (13.4 mg, 0.0199 mmol), KOH (11.2 mg, 0.200 mmol), $PPh_3$ (21.0 mg, 0.0801 mmol), and 1-butanol (0.92 mL, 10.0 mmol) were subjected to general procedure A for 6 h. Purification by FCC (Petrol/$Et_2O$ 10:1) afforded 1-(4-methoxyphenyl)hexan-1-one (**7a**, 366 mg, 1.78 mmol, 89 %) as a colourless solid. **m.p.** 32-33 $^o$C (Lit: 34-35 $^o$C, Manchand, P. S.; Schwartz, A.; Wolff, S.; Belica, P. S.; Madan, P.; Patel, P.; Saposnik, S. J. *Heterocycles* **1993**, *35*, 1351); **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.84 (d, *J* 8.0 Hz, 2H), 6.82 (d, *J* 8.0 Hz, 2H), 3.75 (s, 3H), 2.80 (m, 2H), 1.62 (m, 2H), 1.25 (m, 4H). 0.81 (m, 3H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 199.1, 163.3, 130.3, 130.2, 113.6, 55.4, 38.2, 31.6, 24.3, 22.6, 14.0. Spectroscopic data are consistent with those previously reported: Ruan, J.; Saidi, O.; Iggo, J. A.; Xiao, J. *J. Am. Chem. Soc.* **2008**, *130*, 10510.

**Methylation:** 1-(4-Methoxyphenyl)hexan-1-one **7a** (61.8 mg, 0.300 mmol), $[Ir(cod)Cl]_2$ (2.0 mg, 0.0030 mmol), KOH (33.7 mg, 0.602 mmol), $PPh_3$ (3.2 mg, 0.012 mmol), MeOH (1.5 mL) were subjected to general procedure B for 48 h. Purification by FCC (Petrol/$Et_2O$ 10:1) afforded **22** (62.3 mg, 0.283 mmol, 94%) as a colorless oil.

**One-pot dialkylation:** To a mixture of 4'-Methoxyacetophenone (150 mg, 1.00 mmol), [Ir (cod) $Cl]_2$ (6.7 mg, 0.010 mmol), KOH (5.6 mg, 0.10 mmol) and $PPh_3$ (10.5 mg, 0.0400 mmol) in a Biotage® microwave vial equipped with a stir bar was added 1-butanol (0.46 mL, 5.0 mmol). The vial was sealed with a microwave vial cap (containing a Reseal™ septa) and degassed *via* a needle with a balloon of Ar. The mixture was stirred at 100 $^oC$ for 6 h before KOH (224 mg, 4.00 mmol), $PPh_3$ (10.5 mg, 0.0400 mmol) and MeOH (5 mL) were added, and the mixture was stirred under $O_2$ at 65 $^0C$ for 72 h. Purification by FCC (Petrol/Et2O 10:1) afforded **22** (176 mg, 0.800 mmol, 80%) as a colorless oil. **IR** $\nu_{max}$ ($cm^{-1}$) 2932, 1672, 1599, 842, 734; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.95 (d, *J* 9.0 Hz, 2H), 6.94 (d, *J* 9.0 Hz, 2H), 3.86 (s, 3H), 3.42 (sxt, *J* 6.8 Hz, 1H), 1.78 (m, 1H), 1.43 (m, 1H), 1.29 (m, 4H), 1.17 (d, J 6.8 Hz, 3H), 0.86 (m, 3H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 203.2, 163.3, 130.5, 129.8, 113.7, 55.5, 40.1, 33.7, 29.7, 22.8, 17.4, 14.0; **HRMS** ($ESI^+$) calculated for $[C_{14}H_{20}O_2 + H]^+$ 221.1536, found 221.1539, (Δ -0.3 ppm).

**(±)1-(4-Methoxyphenyl)-2-methylhexadecan-1-one, 23**



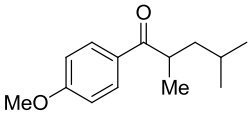

**First alkylation:** $[IrCl(cod)]_2$ (6.7 mg, 0.010 mmol), KOH (5.6 mg, 0.10 mmol), $PPh_3$ (10.5 mg, 0.040 mmol), 4-methoxyacetophenone (150 mg, 1.00 mmol), and 1-tetradecanol (1.07 g, 5.00 mmol) were subjected to general procedure A for 6 h. Purification by FCC (40:1 petrol/ether) afforded 1-(4-methoxyphenyl)hexadecan-1-one **(23a**, 272 mg, 0.79 mmol, 79%) as a colourless solid. **m.p.** 68-70 °C; **IR** $\nu_{max}$ ($cm^{-1}$) 3019, 2925, 2852, 1678, 1601, 1258, 1215, 1171; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.95 (d, *J* 9.0 Hz, 2H), 6.93 (d, *J* 9.0 Hz, 2H), 3.86 (s, 3H), 2.90 (t, *J* 7.5 Hz, 2H), 1.76-1.68 (m, 2H), 1.34-1.26 (m, 24 H), 0.88 (t, *J* 6.8 Hz, 3H); **$^{13}$C NMR** (101 MHz, CDCl3) δ 199.2, 163.2, 130.3, 130.2, 113.6, 55.4, 38.3, 31.9, 29.7-29.4, 24.6, 22.7, 14.1; **HRMS** ($ESI^+$) calculated for $[C_{23}H_{38}O_2+H]^+$ 347.2945, found: 347.2937 (Δ−2.29 ppm).

**Methylation**: 1-(4-Methoxyphenyl)hexadecan-1-one (**23a**, 104 mg, 0.300 mmol), $[Ir(cod)Cl]_2$ (2.0 mg, 0.0030 mmol), KOH (50.6 mg, 0.900 mmol), $PPh_3$ (3.2 mg, 0.012 mmol), MeOH (1.5 mL) were subjected to general procedure B for 48 h. Purification by FCC (Petrol/$Et_2O$ 20:1) afforded **23** (90 mg, 0.25 mmol, 83%) as a yellow solid.

**One-pot dialkylation:** To a mixture of 4'-Methoxyacetophenone (150 mg, 1.00 mmol), [Ir (cod) $Cl]_2$ (6.7 mg, 0.010 mmol), KOH (5.6 mg, 0.10 mmol) and $PPh_3$ (10.5 mg, 0.0400 mmol) in a Biotage® microwave vial equipped with a stir bar was added 1-tetradecanol (1.07 g, 5.00 mmol). The vial was sealed with a microwave vial cap (containing a Reseal™ septa) and degassed *via* a needle with a balloon of Ar. The mixture was stirred at 100 $^oC$ for 6 h before KOH (224 mg, 4.00 mmol), $PPh_3$ (10.5 mg, 0.0400 mmol) and MeOH (5 mL) were added, and the mixture was stirred under $O_2$ at 65 $^0C$ for 48 h. Purification by FCC (Petrol/Et2O 40:1) afforded **23** (251 mg, 0.63 mmol, 63%) as a yellow solid. **m.p.** 29-32 °C;

**IR** $\nu_{max}$ ($cm^{-1}$) 2923, 2852, 1675, 1600, 1575, 1509, 1461, 1308, 1257, 1233, 1170, 1034; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.95 (d, *J* 9.0 Hz, 2H), 6.94 (d, *J* 9.0 Hz, 2H), 3.87 (s, 3H), 3.46-3.38 (m, 1H), 1.81-1.73 (m, 1H), 1.44-1.40 (m, 1H), 1.30-1.24 (m, 24H), 1.18 (d, *J* 6.8 Hz, 3H), 0.88 (t, *J* 6.8 Hz, 3H); **$^{13}$C NMR** (101 MHz, CDCl3) δ 203.1, 163.2 , 130.5, 129.7, 113.7, 55.4, 40.1, 33.9, 31.9, 29.8, 29.7, 29.7, 29.7, 29.7, 29.7, 29.6, 29.5, 29.4, 27.4, 22.7, 17.4, 14.1; **HRMS** ($ESI^+$) calculated for $[C_{24}H_{40}O_2+H]^+$ 361.3101, found: 361.3092 (Δ 2.58 ppm).

**(±)1-(4-Methoxyphenyl)-2,4-dimethylpentan-1-one, 24**

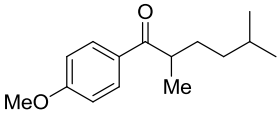



**Isobutylation:** $[IrCl(cod)]_2$ (13.4 mg, 0.020 mmol), KOH (22.4 mg, 0.40 mmol), $PPh_3$ (21.0 mg, 0.080 mmol), 4-methoxyacetophenone (300 mg, 2.0 mmol), and 2-methyl-1-propanol (0.93 mL, 10.0 mmol) were subjected to general procedure A for 6 h. Purification by FCC (9:1 petrol/ether) afforded 1-(4'-methoxyphenyl)-4-methylpentan-1-one (**9a**, 298 mg, 1.43 mmol, 72%) as a yellow oil. **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.87 (d, *J* 8.8 Hz, 2H), 6.85 (d, *J* 9.0 Hz, 2H), 3.79 (s, 3H), 2.83 (m, 2H), 1.56-1.47 (m, 3H), 0.87 (d, *J* 6.4 Hz, 6H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 199.4, 163.3, 130.3, 130.2, 113.7, 55.4, 36.3, 33.5, 27.9, 22.5. All spectroscopic data were consistent with those previously reported: Teague, S. J. *J. Org. Chem.*, **2008**, *73*, 9765.

**Methylation**: 1-(4'-methoxyphenyl)-4-methylpentan-1-one (**9a,** 62.4 mg, 0.300 mmol), $[Ir(cod)Cl]_2$ (2.0 mg, 0.0030 mmol), KOH (33.7 mg, 0.602 mmol), $PPh_3$ (3.2 mg, 0.012 mmol), MeOH (1.5 mL) were subjected to general procedure B for 48 h. Purification by FCC (Petrol/$Et_2O$ 10:1) afforded **24** (60.9 mg, 0.274 mmol, 91%) as a colorless oil.

**One-pot dialkylation:** To a mixture of 4'-Methoxyacetophenone (150 mg, 1.00 mmol), [Ir (cod) Cl]$_2$ (6.7 mg, 0.010 mmol), KOH (11.2 mg, 0.20 mmol) and $PPh_3$ (10.5 mg, 0.0400 mmol) in a Biotage® microwave vial equipped with a stir bar was added 2-methyl-1-propanol (0.46 mL, 5.0 mmol). The vial was sealed with a microwave vial cap (containing a Reseal™ septa) and degassed *via* a needle with a balloon of Ar. The mixture was stirred at 100 $^oC$ for 6 h before KOH (168 mg, 3.00 mmol), $PPh_3$ (10.5 mg, 0.0400 mmol) and MeOH (5 mL) were added, and the mixture was stirred under $O_2$ at 65 $^0C$ for 72 h. Purification by FCC (Petrol/Et2O 10:1) afforded **24** (150 mg, 0.676 mmol, 68%) as a colorless oil. **IR** $\nu_{max}$ ($cm^{-1}$) 2957, 1673, 1600, 1246, 892, 803; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.97 (m, 2H), 6.95 (m, 2H), 3.87 (s, 3H), 3.53 (sxt, *J* 6.9 Hz, 1H), 1.74-1.59 (m, 2H), 1.28(ddd, *J* 13.3, 7.2, 6.1 Hz, 1H), 1.16 (d, *J* 6.8 Hz, 3H), 0.92 (d, *J* 6.6 Hz, 3H), 0.89 (d, *J* 6.6 Hz, 3H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 203.2, 163.3, 130.5, 129.7, 113.8, 55.4, 43.0, 38.0, 25.9, 23.1, 22.4, 17.7. **HRMS** ($ESI^+$) calculated for $[C_{14}H_{20}O_2+H]^+$ 221.1536, found 221.1544, (Δ -3.5 ppm).

**(±)1-(4-Methoxyphenyl)-2,5-dimethylhexan-1-one, 25**

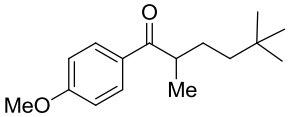


**First alkylation:** $[IrCl(cod)]_2$ (13.4 mg, 0.020 mmol), KOH (11.2 mg, 0.20 mmol), $PPh_3$ (21.0 mg, 0.080 mmol), 4-methoxyacetophenone (300 mg, 2.0 mmol), and 3-methyl-1-butanol (1.10 mL, 10.0 mmol) were subjected to general procedure A for 6 h. Purification by FCC (10:1 petrol/ether) afforded 1-(4-methoxyphenyl)-5-methylhexan-1-one (**11a**, 425 mg, 1.93 mmol, 97%) as a yellow oil. **IR** $\nu_{max}$ ($cm^{-1}$) 2954, 1675, 834, 807; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.91 (d, *J* 8.6 Hz, 2H), 6.89 (d, *J* 8.3 Hz, 2H), 3.82 (s, 3H), 2.86 (t, *J* 7.5 Hz, 2H), 1.69 (m, 2H), 1.56 (m, 1H), 1.23 (m, 2H), 0.87 (d, *J* 6.6 Hz, 6H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 199.2, 163.3, 130.3, 130.2, 113.7, 55.4, 38.7, 38.5, 28.0, 22.6, 22.5; **HRMS** ($ESI^+$) calculated for $[C_{14}H_{20}O_2+H]^+$ 221.1536, found 221.1540, (Δ -1.4 ppm).

**Methylation**: 1-(4-Methoxyphenyl)-5-methylhexan-1-one (**11a,** 66.0 mg, 0.300 mmol), $[Ir(cod)Cl]_2$ (2.0 mg, 0.0030 mmol), KOH (33.7 mg, 0.602 mmol), $PPh_3$ (3.2 mg, 0.012 mmol), MeOH (1.5 mL) were subjected to general procedure B for 72 h. Purification by FCC (Petrol/$Et_2O$ 10:1) afforded **25** (58.4 mg, 0.250 mmol, 83%) as a colorless oil.

**One-pot dialkylation:** To a mixture of 4'-Methoxyacetophenone (150 mg, 1.00 mmol), [Ir (cod) Cl]$_2$ (6.7 mg, 0.010 mmol), KOH (5.6 mg, 0.10 mmol) and $PPh_3$ (10.5 mg, 0.0400 mmol) in a Biotage® microwave vial equipped with a stir bar was added 3-methyl-1-butanol (0.55 mL, 5.0 mmol). The vial was sealed with a microwave vial cap (containing a Reseal™ septa) and degassed *via* a needle with a balloon of Ar. The mixture was stirred at 100 $^oC$ for 6 h before KOH (168 mg, 3.00 mmol), $PPh_3$ (10.5 mg, 0.0400 mmol) and MeOH (5 mL) were added, and the mixture was stirred under $O_2$ at 65 $^0C$ for 72 h. Purification by FCC (Petrol/$Et_2O$ 10:1) afforded (**25**,192 mg, 0.821 mmol, 82%) as a colorless oil. **IR** $\nu_{max}$ ($cm^{-1}$) 2956, 1673, 843, 762; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.95 (m, 2H), 6.94 (m, 2H), 3.86 (s, 3H), 3.39 (sxt, J 6.8 Hz, 1H), 1.78 (m, 1H), 1.45 (m, 2H), 1.20-1.14 (m, 2H), 1.18 (d, *J* 6.8, 3H), 0.85 (m, 6H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 203.2, 163.3, 130.5, 129.8, 113.7, 55.4, 40.4, 36.7, 31.8, 28.2, 22.6, 22.4, 17.5; **HRMS** ($ESI^+$) calculated for $[C_{15}H_{22}O_2+Na]^+$ 257.1512, found 257.1524, (Δ -4.5 ppm).

**1-(4-Methoxyphenyl)-2,5,5-trimethylhexan-1-one, 26**



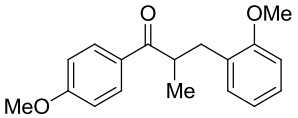

**First alkylation:** $[IrCl(cod)]_2$ (6.7 mg, 0.010 mmol), KOH (5.6 mg, 0.10 mmol), $PPh_3$ (10.5 mg, 0.040 mmol), 4-methoxyacetophenone (150 mg, 1.0 mmol), and 3,3-dimethylbutan-1-ol (0.63 mL, 5.00 mmol) were subjected to general procedure A for 6 h. Purification by FCC (40:1 petrol/ether) afforded 1-(4-methoxyphenyl)-5,5-dimethylhexan-1-one (**26a**, 234 mg, 1.00 mmol, quant.) as a yellow solid. **m.p.** 52-53 °C; **IR** $\nu_{max}$ ($cm^{-1}$) 3000, 2954, 2901, 2865, 1668, 1602, 1578, 1508, 1442, 1256, 1183, 1033; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.94 (d, *J* 9.0 Hz, 2H), 6.93 (d, *J* 9.0 Hz, 2H), 3.86 (s, 3H), 2.88 (t, *J* 7.4 Hz, 2H), 1.74-1.66 (m, 2H), 1.28-1.24 (m, 2H), 0.90 (s, 9H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 199.1, 163.3, 130.2, 130.2, 113.6, 55.4, 43.9, 39.0, 30.4, 29.3, 19.8; **HRMS** ($ESI^+$) calculated for $[C_{15}H_{22}O_2+H]^+$ 235.1693, found 235.1689, (Δ -1.58 ppm).

**Methylation**: 1-(4-methoxyphenyl)-5,5-dimethylhexan-1-one (**26a,** 70.0 mg, 0.300 mmol), $[Ir(cod)Cl]_2$ (2.0 mg, 0.0030 mmol), KOH (84.2 mg, 1.500 mmol), $PPh_3$ (3.2 mg, 0.012

mmol), MeOH (1.5 mL) were subjected to general procedure B for 48 h. Purification by FCC (Petrol/$Et_2O$ 40:1) afforded **26** (53 mg, 0.21 mmol, 70%) as a colorless oil.

**One-pot dialkylation:** To a mixture of 4'-Methoxyacetophenone (150 mg, 1.00 mmol), [Ir (cod) Cl]$_2$ (6.7 mg, 0.010 mmol), KOH (5.6 mg, 0.10 mmol) and $PPh_3$ (10.5 mg, 0.0400 mmol) in a Biotage® microwave vial equipped with a stir bar was added 3,3-dimethylbutan-1-ol (0.63 mL, 5.00 mmol). The vial was sealed with a microwave vial cap (containing a Reseal™ septa) and degassed *via* a needle with a balloon of Ar. The mixture was stirred at 100 $^oC$ for 6 h before KOH (336 mg, 6.00 mmol), $PPh_3$ (10.5 mg, 0.0400 mmol) and MeOH (5 mL) were added, and the mixture was stirred under $O_2$ at 65 $^0C$ for 48 h. Purification by FCC (Petrol/$Et_2O$ 40:1) afforded **26** (158 mg, 0.64 mmol, 64%) as a colorless oil. **IR** $\nu_{max}$ ($cm^{-1}$) 2953, 2867, 1673, 1599, 1575, 1510, 1462, 1419, 1393, 1308, 1253, 1224, 1168, 1032; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.95 (d, *J* 9.0 Hz, 2H), 6.94 (d, *J* 9.0 Hz, 2H), 3.87 (s, 3H), 3.39-3.30 (m, 1H), 1.82-1.73 (m, 1H), 1.44-1.35 (m, 1H), 1.26-1.11 (m, 5H), 0.85 (s, 9H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 203.1, 163.2, 130.4, 129.7, 113.7, 55.4, 41.7, 40.8, 30.2, 29.2, 28.8, 17.5; **HRMS** ($ESI^+$) calculated for $[C_{16}H_{24}O_2+H]^+$ 249.1849, found249.1845, (Δ -1.7 ppm).

**(±)3-(2-Methoxyphenyl)-1-(4-methoxyphenyl)-2-methylpropan-1-one, 28**



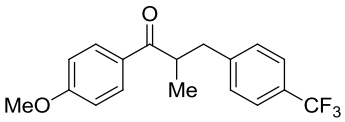

**First alkylation:** [IrCl(cod)]$_2$ (6.7 mg, 0.010 mmol), KOH (5.6 mg, 0.10 mmol), $PPh_3$ (10.5 mg, 0.040 mmol), 4-methoxyacetophenone (150 mg, 1.0 mmol), and 2-methoxybenzyl alcohol (0.27 mL, 2.00 mmol) were subjected to general procedure A for 6 h. Purification by FCC (9:1 petrol/ether) afforded 3-(2-methoxyphenyl)-1-(4-methoxyphenyl)propan-1-one (**28a**, 270 mg, 1.00 mmol, quant.) as a colorless oil. **$^1$H NMR** (400 MHz, $CDCl_3$) δ7.99 (d, *J* 9.0 Hz, 2H), 7.25-7.20 (m, 2H), 6.96-6.87 (m, 4H), 3.87 (s, 3H), 3.85 (s, 3H), 3.23 (t, *J* 7.7 Hz, 2H), 3.06 (t, *J* 7.7 Hz, 2H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 198.5, 163.2, 157.4, 130.3, 130.0, 130.0, 129.6, 127.4, 120.4, 113.6, 110.1, 55.3, 55.1, 38.5, 25.8. All spectroscopic data were consistent with those previously reported: Xu, Q.; Chen, J.; Tian, H.; Yuan, X.; Li, S.; Zhou, C.; Liu, J. *Angew. Chem. Int. Ed.* **2014**, *53*, 225.

**Methylation**: 3-(2-Methoxyphenyl)-1-(4-methoxyphenyl)propan-1-one (**28a,** 81 mg, 0.300 mmol), [Ir(cod)Cl]$_2$ (2.0 mg, 0.0030 mmol), KOH (84.2 mg, 1.500 mmol), $PPh_3$ (3.2 mg, 0.012 mmol), MeOH (1.5 mL) were subjected to general procedure B for 48 h. Purification by FCC (Petrol/$Et_2O$ 20:1) afforded **28** (65 mg, 0.23 mmol, 77%) as a yellow solid.

**One-pot dialkylation:** To a mixture of 4'-Methoxyacetophenone (150 mg, 1.00 mmol), [Ir (cod) Cl]$_2$ (6.7 mg, 0.010 mmol), KOH (5.6 mg, 0.10 mmol) and $PPh_3$ (10.5 mg, 0.0400 mmol) in a Biotage® microwave vial equipped with a stir bar was added 2-methoxybenzyl alcohol (0.27 mL, 2.00 mmol). The vial was sealed with a microwave vial cap (containing a Reseal™ septa) and degassed *via* a needle with a balloon of Ar. The mixture was stirred at 100 $^oC$ for 6 h before KOH (336 mg, 6.00 mmol), $PPh_3$ (10.5 mg, 0.0400 mmol) and MeOH (5 mL) were added, and the mixture was stirred under $O_2$ at 65 $^0C$ for 48 h. Purification by FCC (Petrol/$Et_2O$ 20:1) afforded **28** (210 mg, 0.74 mmol, 74%) as a yellow solid. **m.p.** 61–

63°C; **IR** $\nu_{max}$ ($cm^{-1}$) 2966, 2934, 2838, 1671, 1598, 1509, 1458, 1419, 1235, 1169, 1125, 1027; **$^1$H NMR** (400 MHz, $CDCl_3$) δ8.03 (d, *J* 9.0 Hz, 2H), 7.20 (td, *J* 7.7, 1.6 Hz, 1H), 7.13 (dd, 7.4, 1.6 Hz, 1H), 6.94 (d, *J* 9.0 Hz, 2H), 6.87 (m, 2H), 3.76 (s, 3H), 3.76 (s, 3H), 3.89-3.71 (m, 1H), 3.19 (dd, *J* 13.3, 5.4 Hz, 1H), 2.61 (dd, *J* 13.3, 8.7 Hz, 1H), 1.15 (d, *J* 6.8 Hz, 3H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 202.7, 163.2, 157.4, 130.6, 129.6, 128.2, 131.3, 127.5, 120.2, 113.5, 110.1, 55.4 , 55.1, 39.9, 35.2, 16.7; **HRMS** ($ESI^+$) calculated for $[C_{18}H_{20}O_3+H]^+$ 285.1485, found 285.1478, (Δ -2.5 ppm).

**(±)1-(4-methoxyphenyl)-2-methyl-3-(4-(trifluoromethyl)phenyl)propan-1-one, 29**

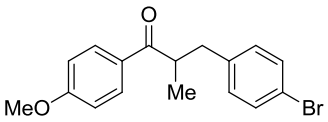



**First alkylation:** $[IrCl(cod)]_2$ (6.7 mg, 0.010 mmol), KOH (5.6 mg, 0.10 mmol), $PPh_3$ (10.5 mg, 0.040 mmol), 4-methoxyacetophenone (150 mg, 1.0 mmol), and 4-(trifluoromethyl)benzyl alcohol (0.68 mL, 5.00 mmol) were subjected to general procedure A for 6 h. Purification by FCC (9:1 petrol/ether) afforded 1-(4-methoxyphenyl)-3-(4-(trifluoromethyl)phenyl)propan-1-one (**29a**, 261 mg, 0.85 mmol, 85%) as a colorless solid. **m.p.** 56-61 °C; **IR** $\nu_{max}$ ($cm^{-1}$) 2938, 2844, 1676, 1601, 1325, 1169, 1117, 1068; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.95 (d, *J* 8.8 Hz, 2H), 7.55 (d, *J* 8.0 Hz, 2H), 7.37 (d, *J* 8.0 Hz, 2H), 6.94 (d, *J* 8.8 Hz, 2H), 3.86 (s, 3H), 3.28 (t, *J* 7.5 Hz, 2H), 3.12 (t, *J* 7.5 Hz, 2H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) 197.1, 163.5, 145.6, 130.2, 129.7, 128.8, 128.3 (q, *J* 32.3 Hz), 125.3 (q, *J* 3.7), 124.2 (q, *J* 271.8 Hz), 113.7, 55.4, 39.4, 29.9; **$^{19}$F NMR** (377 MHz, $CDCl_3$) δ −62.3; **HRMS** ($ESI^+$) calculated for $[C_{17}H_{15}O_2F_3+Na]^+$ 331.0916, found 331.0912, (Δ 1.2 ppm).

**Methylation**: 1-(4-Methoxyphenyl)-3-(4-(trifluoromethyl)phenyl)propan-1-one (**29a,** 93mg, 0.300 mmol), $[Ir(cod)Cl]_2$ (2.0 mg, 0.0030 mmol), KOH (50.0 mg, 0.900 mmol), $PPh_3$ (3.2 mg, 0.012 mmol), MeOH (1.5 mL) were subjected to general procedure B for 48 h. Purification by FCC (Petrol/$Et_2O$ 9:1) afforded **29** (82 mg, 0.25 mmol, 83%).) as a yellow oil.

**One-pot dialkylation:** To a mixture of 4'-Methoxyacetophenone (150 mg, 1.00 mmol), $[Ir(cod)Cl]_2$ (6.7 mg, 0.010 mmol), KOH (5.6 mg, 0.10 mmol) and $PPh_3$ (10.5 mg, 0.0400 mmol) in a Biotage® microwave vial equipped with a stir bar was added 4-(trifluoromethyl)benzyl alcohol (352 mg, 5.00 mmol). The vial was sealed with a microwave vial cap (containing a Reseal™ septa) and degassed *via* a needle with a balloon of Ar. The mixture was stirred at 100 $^oC$ for 6 h before KOH (224 mg, 4.00 mmol), $PPh_3$ (10.5 mg, 0.0400 mmol) and MeOH (5 mL) were added, and the mixture was stirred under $O_2$ at 65 $^0C$ for 48 h. Purification by FCC (Petrol/$Et_2O$ 20:1) afforded **29** (188 mg, 0.58 mmol, 58%) as a yellow oil. **IR** $\nu_{max}$ ($cm^{-1}$) 2970, 2936, 1672, 1599, 1323, 1260, 1235, 1164, 1114, 1066; **$^1$H NMR** (400 MHz, $CDCl_3$) δ7.91 (d, *J* 8.9 Hz, 2H), 7.51 (d, *J* 8.1 Hz, 2H), 7.31 (d, *J* 8.1 Hz, 2H), 6.93 (d, *J* 8.9 Hz, 2H), 3.86 (s, 3H), 3.78-3.68 (m, 1H), 3.23 (dd, *J* 13.7, 7.0 Hz, 1H), 2.77 (dd, *J* 13.7, 7.0 Hz, 1H), 1.22 (d, *J* 7.0 Hz, 3H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ; 201.5, 163.5, 144.3, 130.5, 129.3, 129.1, 128.4 (q, *J* 32.4 Hz), 124.1 (q, *J* 272.0 Hz), 125.2 (q, *J* 3.7 Hz), 113.8, 55.4, 42.0, 39.1, 17.9; **$^{19}$F NMR** (377 MHz, $CDCl_3$) δ −62.3; **HRMS** ($ESI^+$) calculated for $[C_{18}H_{17}O_2F_3+H]^+$ 323.1253, found 323.1247, (Δ -2.1 ppm).

**(±)3-(4-bromophenyl)-1-(4-methoxyphenyl)-2-methylpropan-1-one, 30**

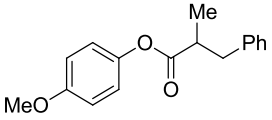



**First alkylation:** $[IrCl(cod)]_2$ (6.7 mg, 0.010 mmol), KOH (5.6 mg, 0.10 mmol), $PPh_3$ (10.5 mg, 0.040 mmol), 4-methoxyacetophenone (150 mg, 1.0 mmol), and 4-bromobenzyl alcohol (935 mg, 5.00 mmol) were subjected to general procedure A for 6 h. Purification by FCC (9:1 petrol/ether) afforded 3-(4-bromophenyl)-1-(4-methoxyphenyl)propan-1-one (**30a**, 273 mg, 0.86 mmol, 86%) as a colourless solid. **m.p.** 97-100 °C; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.93 (d, *J* 8.9 Hz, 2H), 7.41 (d, *J* 8.4 Hz, 2H), 7.13 (d, *J* 8.4 Hz, 2H), 6.93 (d, *J* 8.9 Hz, 2H), 3.86 (s, 3H), 3.23 (t, *J* 7.7 Hz, 2H), 3.01 (t, *J* 7.7 Hz, 2H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ197.3, 163.4, 140.4, 131.4, 130.2, 130.2, 129.8, 119.7, 113.7 , 55.4, 39.6, 29.6. All spectroscopic data were consistent with those previously reported: Xu, Q.; Chen, J.; Tian, H.; Yuan, X.; Li, S.; Zhou, C.; Liu, J. *Angew. Chem. Int. Ed.* **2014**, *53*, 225.

**Methylation**: 3-(4-Bromophenyl)-1-(4-methoxyphenyl)propan-1-one (**30a,** 96 mg, 0.300 mmol), $[Ir(cod)Cl]_2$ (2.0 mg, 0.0030 mmol), KOH (50.0 mg, 0.900 mmol), $PPh_3$ (3.2 mg, 0.012 mmol), MeOH (1.5 mL) were subjected to general procedure B for 48 h. Purification by FCC (Petrol/$Et_2O$ 9:1) afforded **30** (86 mg, 0.26 mmol, 87%) as a yellow oil.

**One-pot dialkylation:** To a mixture of 4'-Methoxyacetophenone (150 mg, 1.00 mmol), [Ir (cod) $Cl]_2$ (6.7 mg, 0.010 mmol), KOH (5.6 mg, 0.10 mmol) and $PPh_3$ (10.5 mg, 0.0400 mmol) in a Biotage® microwave vial equipped with a stir bar was 4-bromobenzyl alcohol (935 mg, 5.00 mmol). The vial was sealed with a microwave vial cap (containing a Reseal™ septa) and degassed *via* a needle with a balloon of Ar. The mixture was stirred at 100 $^o$C for 6 h before KOH (224 mg, 4.00 mmol), $PPh_3$ (10.5 mg, 0.0400 mmol) and MeOH (5 mL) were added, and the mixture was stirred under $O_2$ at 65 $^0$C for 48 h. Purification by FCC (Petrol/$Et_2O$ 20:1) afforded **30** (164 mg, 0.49 mmol, 62%) as a yellow oil. **IR** $\nu_{max}$ ($cm^{-1}$) 2968, 2932, 2839, 1671, 1598, 1574, 1509, 1488, 1458, 1259, 1169, 1030, 1011; **$^1$H NMR** (400 MHz, $CDCl_3$) δ7.91 (d, *J* 9.0 Hz, 2H), 7.37 (d, *J* 8.4 Hz, 2H), 7.07 (d, *J* 8.4 Hz, 2H), 6.92 (d, *J* 9.0 Hz, 2H), 3.86 (s, 3H), 3.72-3.63 (m, 1H), 3.11 (dd, *J* 13.7, 6.9 Hz, 1H), 2.66 (dd, *J* 13.7, 7.4 Hz, 1H), 1.19 (d, *J* 7.0 Hz, 3H); **$^{13}$C NMR** (101 MHz, $CDCl_3$) δ201.7, 163.4, 139.1, 131.3, 130.8, 130.5, 129.2, 119.9, 113.8, 55.4, 42.1, 38.7, 17.8; **HRMS** ($ESI^+$) calculated for $[C_{17}H_{17}O_2Br+H]^+$ 333.0485, found 333.0477, (Δ -2.5 ppm).

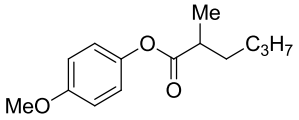
**(±)4-Methoxyphenyl 2-methyl-3-phenylpropanoate, 31**




1-(4-Methoxyphenyl)-2-methyl-3-phenylpropan-1-one(**27**, 30.0 mg, 0.120 mmol), mCPBA (80.0 mg, 0.480 mmol), trifluoroacetic acid (19 μL, 0.24 mmol) and $CH_2Cl_2$, (2.4 mL) were subjected to general procedure D. Purification by FCC (Petrol/$Et_2O$ 20:1) afforded **31** as a colourless oil (29.5 mg, 0.110 mmol, 92 %). **IR** $\nu_{max}$ ($cm^{-1}$) 3063, 3028, 2973, 2935, 2837,

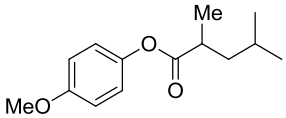
1751, 1606, 1597, 1505, 1455, 1248, 1192, 1137; **$^{1}$H NMR** (400 MHz, $CDCl_3$) δ 7.27-7.20 (m, 2H), 7.19-7.14 (m, 3H), 6.79-6.72 (m, 4H), 3.70 (s, 3H), 3.04 (dd, *J* 13.3, 7.6 Hz, 1H), 2.90 (sxt, *J* 7.0 Hz, 1H), 2.75 (dd, *J* 13.3, 7.2 Hz, 1H), 1.24 (d, *J* 6.9 Hz, 3H); **$^{13}$C NMR** (126 MHz, $CDCl_3$) δ 179.9, 157.1, 144.1, 139.0, 129.1, 128.4, 126.5, 122.2, 144.3, 55.5, 41.6, 39.8, 16.9; **HRMS** ($ESI^+$) calculated for $[C_{17}H_{18}O_3+Na]^+$ 293.1148, found 293.1149.

**(±)4-Methoxyphenyl 2-methylhexanoate, 32**



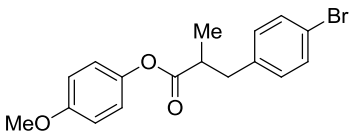

1-(4-Methoxyphenyl)-2-methylhexan-1-one (**22**, 50.0 mg, 0.227 mmol), mCPBA (157 mg, 0.909 mmol), trifluoroacetic acid (35 μL, 0.454 mmol) and $CH_2Cl_2$, (1.2 mL) were subjected to general procedure D. Purification by FCC (Petrol/$Et_2O$ 10:1) afforded **32** as a colourless oil (46.8 mg, 0.198 mmol, 87%). **IR $\nu_{max}$** ($cm^{-1}$) 2935, 1754, 1506, 1195, 819, 746; **$^{1}$H NMR** (400 MHz, $CDCl_3$) δ 6.99 (d, *J* 9.0 Hz, 2H), 6.89 (d, *J* 9.0 Hz, 2H), 3.81 (s, 3H), 2.67 (sxt, *J* 7.0 Hz, 1H), 1.85-1.75 (m, 1H), 1.61-1.50 (m, 1H), 1.48-1.30 (m, 4H), 1.29 (d, *J* 6.9 Hz, 3H), 0.94 (t, *J* 7.1 Hz, 3H); **$^{13}$C NMR** (126 MHz, $CDCl_3$) δ 175.8, 157.1, 144.4, 122.3, 114.4, 55.6, 39.6, 33.5, 29.4, 22.6, 17.1, 14.0; **HRMS** ($ESI^+$) calculated for $[C_{14}H_{20}O_3+Na]^+$ 259.1305, found 259.1316, (Δ -4.5 ppm).

**(±)4-Methoxyphenyl 2,4-dimethylpentanoate, 33**




1-(4-Methoxyphenyl)-2,4-dimethylpentan-1-one (**24**, 30.0 mg, 0.135 mmol), mCPBA (93.2 mg, 0.540 mmol), trifluoroacetic acid (21 μL, 0.270 mmol) and $CH_2Cl_2$, (2.4 mL) were subjected to general procedure D. Purification by FCC (Petrol/$Et_2O$ 10:1) afforded **33** as a colourless oil (28.5 mg, 0.120 mmol, 89%). **IR $\nu_{max}$** ($cm^{-1}$) 2957, 1752, 1505, 1194, 815, 757; **$^{1}$H NMR** (400 MHz, $CDCl_3$) δ 6.99 (d, *J* 9.0 Hz, 2H), 6.89 (d, *J* 9.0 Hz, 2H), 3.81 (s, 3H), 2.75 (sxt, *J* 7.1 Hz, 1H), 1.80-1.68 (m, 2H), 1.40-1.34 (m, 1H), 1.28 (d, *J* 7.1 Hz, 3H), 0.98 (d, *J* 6.5Hz, 3H), 0.95 (d, *J* 6.3 Hz, 3H) ; **$^{13}$C NMR** (126 MHz, $CDCl_3$) δ 176.0, 157.1, 144.3, 122.3, 114.4, 55.6, 43.0, 37.7, 26.0, 22.5, 22.5, 17.5; **HRMS** ($ESI^+$) calculated for $[C_{14}H_{20}O_3+Na]^+$ 259.1305, found 259.1314, (Δ -3.4 ppm).

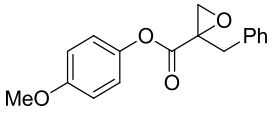
**(±)4-Methoxyphenyl 3-(4-bromophenyl)-2-methylpropanoate, 34**




3-(4-Bromophenyl)-1-(4-methoxyphenyl)-2-methylpropan-1-one (**30**, 30.0 mg, 0.0900 mmol), mCPBA (62.1 mg, 0.360 mmol), trifluoroacetic acid (14 μL, 0.180 mmol) and $CH_2Cl_2$, (2.4 mL) were subjected to general procedure D. Purification by FCC (toluene) afforded **34** as a

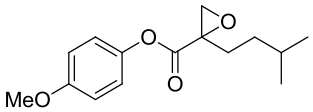
yellow oil (27.7 mg, 0.0794 mmol, 88%). **IR $\nu_{max}$** ($cm^{-1}$) 2934, 2836, 1751, 1505, 1489, 1460, 1248, 1192, 1140, 1103, 1011; **$^{1}$H NMR** (400 MHz, CDCl3) δ 7.45 (d, *J* 8.4 Hz, 2H), 7.13 (d, *J* 8.4 Hz, 2H), 6.90-6.84 (m, 4H), 3.80 (s, 3H), 3.09 (dd, *J* 13.4, 7.7 Hz, 1H), 3.00-2.91 (m, 1H), 2.79 (dd, *J* 13.4, 7.0 Hz, 1H), 1.32 (d, *J* 7.0 Hz, 3H); **$^{13}$C NMR** (101 MHz, CDCl3) δ 174.6, 157.2 , 144.0, 138.0, 131.5, 130.8, 122.4, 120.3, 114.4, 55.5, 41.4 , 39.1, 17.0; **HRMS** (ESI+) calculated for $[C_{17}H_{17}O_3Br+Na]^+$ 371.0253, found: 371.0249 (Δ −1.07 ppm).

**4-Methoxyphenyl 2-benzyloxirane-2-carboxylate, 35**



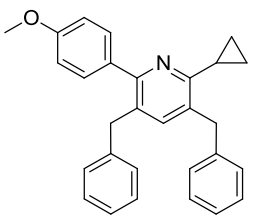

(2-Benzyloxiran-2-yl)(4-methoxyphenyl)methanone (**19**, 26.0 mg, 0.100 mmol), mCPBA (66.0 mg, 0.400 mmol), trifluoroacetic acid (14 μL, 0.20 mmol) and $CH_2Cl_2$ (2.0 mL) were subjected to general procedure D. Purification by FCC (Petrol/$Et_2O$ 20:1→10:1) afforded **35** (25.0 mg, 0.0880 mmol, 88 %) as a colourless oil. **IR $\nu_{max}$** ($cm^{-1}$) 2925, 1753, 1503, 1248, 1190, 1097; **$^{1}$H NMR** (400 MHz, $CDCl_3$) δ 7.38-7.24 (m, 5H), 6.91-6.84 (m, 4H), 3.79 (s, 3H), 3.51 (d, *J* 14.9 Hz, 1H), 3.24 (d, *J* 5.8 Hz, 1H), 3.22 (d, *J* 14.9 Hz, 1H), 2.86 (d, *J* 5.8 Hz, 1H); **$^{13}$C NMR** (126 MHz, $CDCl_3$) δ 169.1, 157.5, 143.7, 135.3, 129.8, 128.4, 127.0, 121.9, 114.5, 57.2, 55.5, 51.2, 36.9; **HRMS** ($ESI^+$) calculated for $[C_{17}H_{16}O_4+Na]^+$ 307.0941, found 307.0932, (Δ −2.8 ppm).

**4-Methoxyphenyl 2-isopentyloxirane-2-carboxylate, 36**



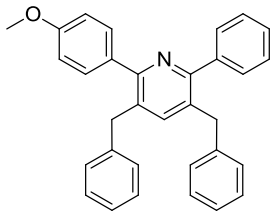

(2-Isopentyloxiran-2-yl)(4-methoxyphenyl)methanone (**18**, 22.0 mg, 0.090 mmol), mCPBA (61.0 mg, 0.360 mmol), trifluoroacetic acid (14 μL, 0.20 mmol) and $CH_2Cl_2$ (1.8 mL) were subjected to general procedure D. Purification by FCC (Petrol/$Et_2O$ 10:1) afforded **36** (17.5 mg, 0.0660 mmol, 74%) as a colourless oil. **IR $\nu_{max}$** ($cm^{-1}$) 2956, 1753, 1505, 1248, 1192; **$^{1}$H NMR** (400 MHz, $CDCl_3$) δ 7.02 (d, *J* 9.1 Hz, 2H), 6.90 (d, *J* 9.1 Hz, 2H), 3.81 (s, 3H), 3.23 (d, *J* 5.8 Hz, 1H), 2.91 (d, *J* 5.8 Hz, 1H), 2.19 (ddd, *J* 5.0, 11.6, 14.2 Hz, 1H), 1.79 ( ddd, *J* 5.2, 11.4, 14.2 Hz, 1H), 1.67-1.56 (m, 1H), 1.53-1.34 (m, 2H), 0.93 (d, *J* 6.7 Hz, 6H); **$^{13}$C NMR** (126 MHz, $CDCl_3$) δ 169.5, 157.4, 143.8, 122.0, 114.5, 57.3, 55.6, 52.0, 33.6, 29.2, 28.0, 22.4, 22.3; **m/z** (ESI+) 287.1 **HRMS** ($ESI^+$) calculated for $[C_{15}H_{20}O_4+Na]^+$ 287.1254 , found 287.1255 (Δ −1.9 ppm).

**3,5-Dibenzyl-2-cyclopropyl-6-(4-methoxyphenyl)pyridine, 37**



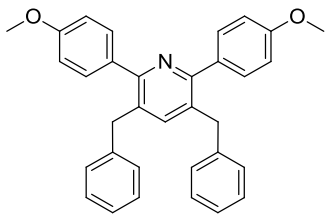
(±)(2*RS*,4*SS*)-2,4-Dibenzyl-1-cyclopropyl-5-(4-methoxyphenyl)pentane-1,5-dione (**16**, 30 mg, 0.0703 mmol), ammonium acetate (16.3 mg, 0.211 mmol), copper (II) acetate monohydrate (35.1 mg, 0.176 mmol) were subjected to general procedure F for 24 h. Purification by FCC (toluene/$Et_2O$ 20:1) afforded **37** as a yellow oil (27.0 mg, 0.0667 mmol, 95%). $\nu_{max}$ (thin film)/$cm^{-1}$ 3002, 1608, 1513, 1449, 1249, 1175, 840, 727, 698; $^1$H NMR (500MHz , $CDCl_3$) δ 7.36 - 7.30 (m, 2 H), 7.22 - 7.04 (m, 9 H), 6.92 (dd, *J* = 0.9, 7.8 Hz, 2 H), 6.85 - 6.79 (m, 2 H), 4.03 (s, 2 H), 3.91 (s, 2 H), 3.74 (s, 3 H), 2.04 - 1.91 (m, 1 H), 1.08 - 0.97 (m, 2 H), 0.82 - 0.68 (m, 2 H); $^{13}$C NMR (126MHz , $CDCl_3$) δ 159.3, 157.9, 155.5, 141.1, 140.1, 140.0, 133.4, 131.7, 130.5, 129.5, 128.7, 128.7, 128.5, 128.5, 126.2, 126.0, 113.4, 55.3, 38.2, 37.9, 13.7, 9.0; HRMS ($ESI^+$) calculated for $[C_{29}H_{27}NO+H]^+$ 406.2165, found 406.2162 (Δ –0.72 ppm).

**3,5-Dibenzyl-2-(4-methoxyphenyl)-6-phenylpyridine, 38**




(±)(2*SS*,4*RS*)-2,4-Dibenzyl-1-(4-methoxyphenyl)-5-phenylpentane-1,5-dione (**17**, 30 mg, 0.0649 mmol), hydroxylamine hydrochloride (13.6 mg, 0.0974 mmol) were subjected to general procedure E. Purification by FCC (Petrol/Et2O 8:2) afforded **38** as a colorless oil (26.9 mg, 0.0609 mmol, 94%). $\nu_{max}$ (thin film)/$cm^{-1}$ 3060, 3026, 2932, 2836, 1609, 1434, 1248, 1175, 728, 699; $^1$H NMR (500MHz , $CDCl_3$) δ 7.42 (d, *J* 7.6 Hz, 2 H), 7.38 (d, *J* 7.9 Hz, 2 H), 7.33 - 7.24 (m, 4 H), 7.15 (q, *J* 8.0 Hz, 4 H), 7.12 - 7.08 (m, 2 H), 6.93 (d, *J* 7.6 Hz, 2 H), 6.90 (d, *J* 7.4 Hz, 2 H), 6.83 (d, *J* 8.4 Hz, 2 H), 3.96 (s, 2 H), 3.93 (s, 2 H), 3.74 (s, 3 H); $^{13}$C NMR (126MHz , $CDCl_3$) δ 159.5, 156.5, 156.3, 141.2, 140.6, 140.2, 132.7, 140.5, 132.2, 132.0, 130.6, 129.3, 128.8, 128.8, 128.5, 128.5, 128.1, 127.9, 126.2, 126.2, 113.6, 55.4, 38.3, 38.2; HRMS ($ESI^+$) calculated for $[C_{32}H_{27}NO+H]^+$ 442.2165, found 442.2157 (Δ –1.97ppm).

**3,5-Dibenzyl-2,6-bis(4-methoxyphenyl)pyridine, 39**

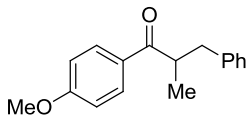


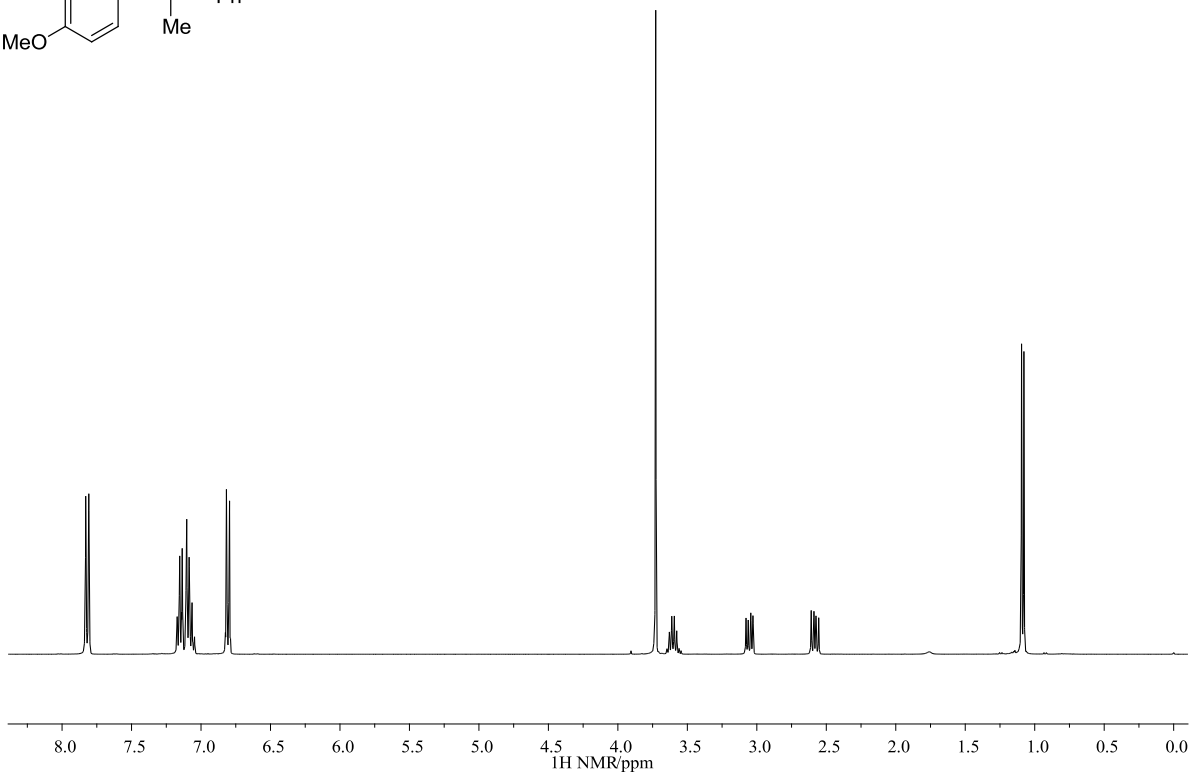

(±)(2*RS*,4*SS*)-2,4-Dibenzyl-1,5-bis(4-methoxyphenyl)pentane-1,5-dione (**5**, 30 mg, 0.0609 mmol), hydroxylamine hydrochloride (12.7 mg, 0.183 mmol) were subjected to general procedure E. Purification by FCC (toluene/Et2O 15:1) afforded **39** as an oily solid (23.2 mg,

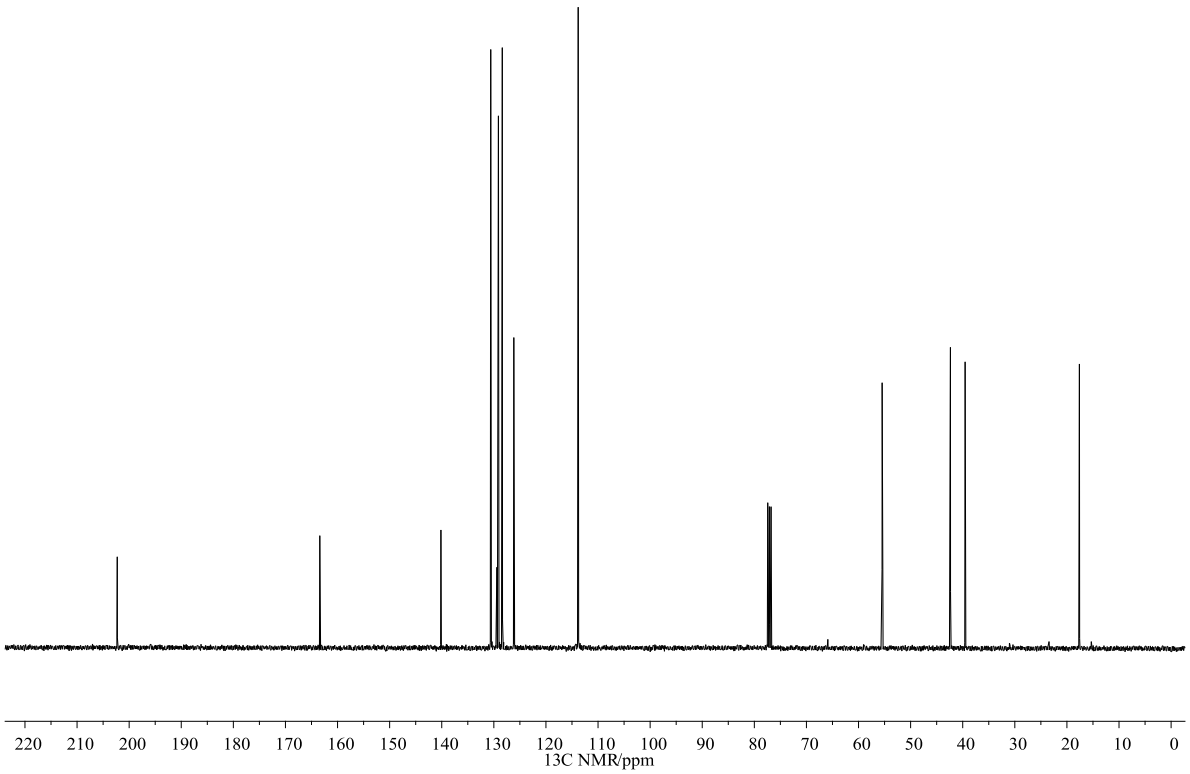
0.0492 mmol, 81%). $\nu_{max}$ (thin film)/cm-1 3015, 2970, 1609, 1511, 1434, 1294, 1175, 1031, 839; $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.48 (d, *J* 8.6 Hz, 4H), 7.34 (s, 1H), 7.25 (t, *J* 7.7 Hz, 4H), 7.18 (t, *J* 6.8 Hz, 2H), 7.02 (d, *J* 7.6, 4H), 6.93 (d, *J* 8.6, 4H), 4.04 (s, 4H), 3.83 (s, 6H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 159.6, 156.3, 141.4, 140.8, 132.9, 131.9, 130.7, 128.8, 128.6, 126.2, 113.6, 55.4, 38.4; HRMS ($ESI^+$) Calculated for $[C_{33}H_{29}NO_2+H]^+$ 472.2271, found 472.2264 (Δ –1.6 ppm).

## IX. Spectral Data

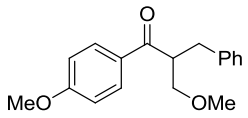
**(±)1-(4-Methoxyphenyl)-2-methyl-3-phenylpropan-1-one, 2**

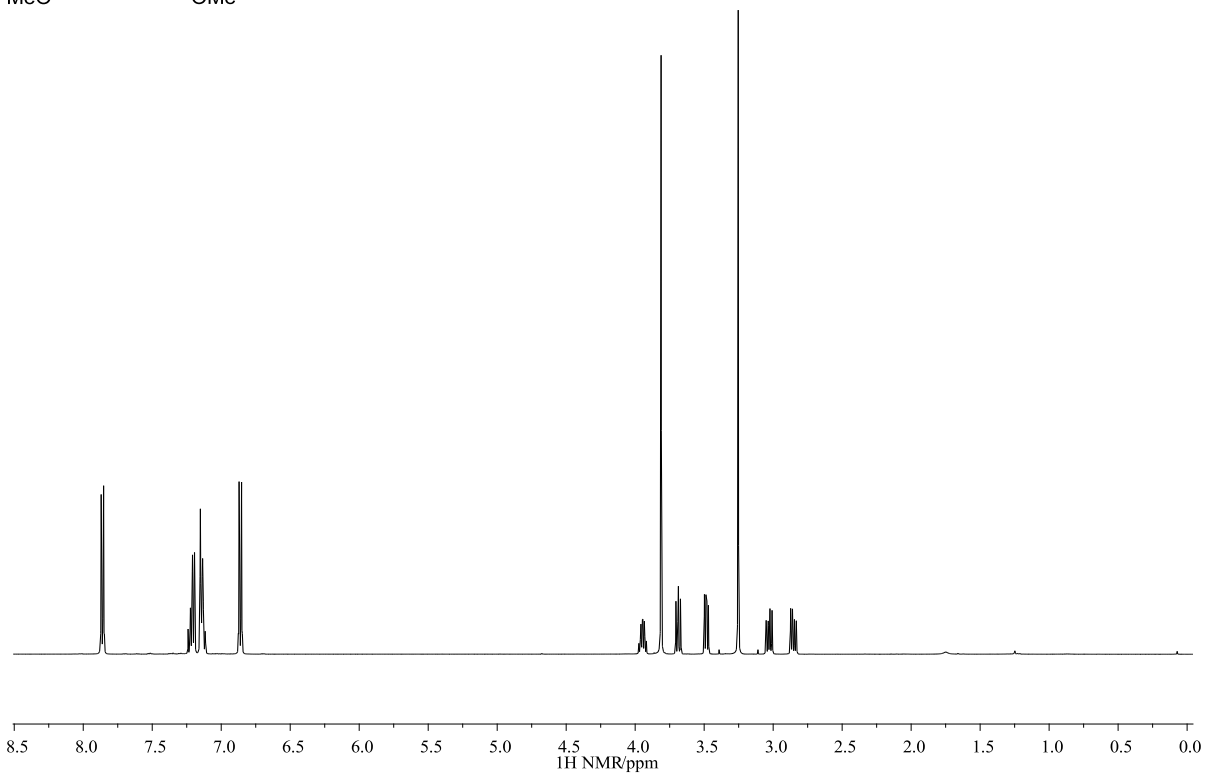


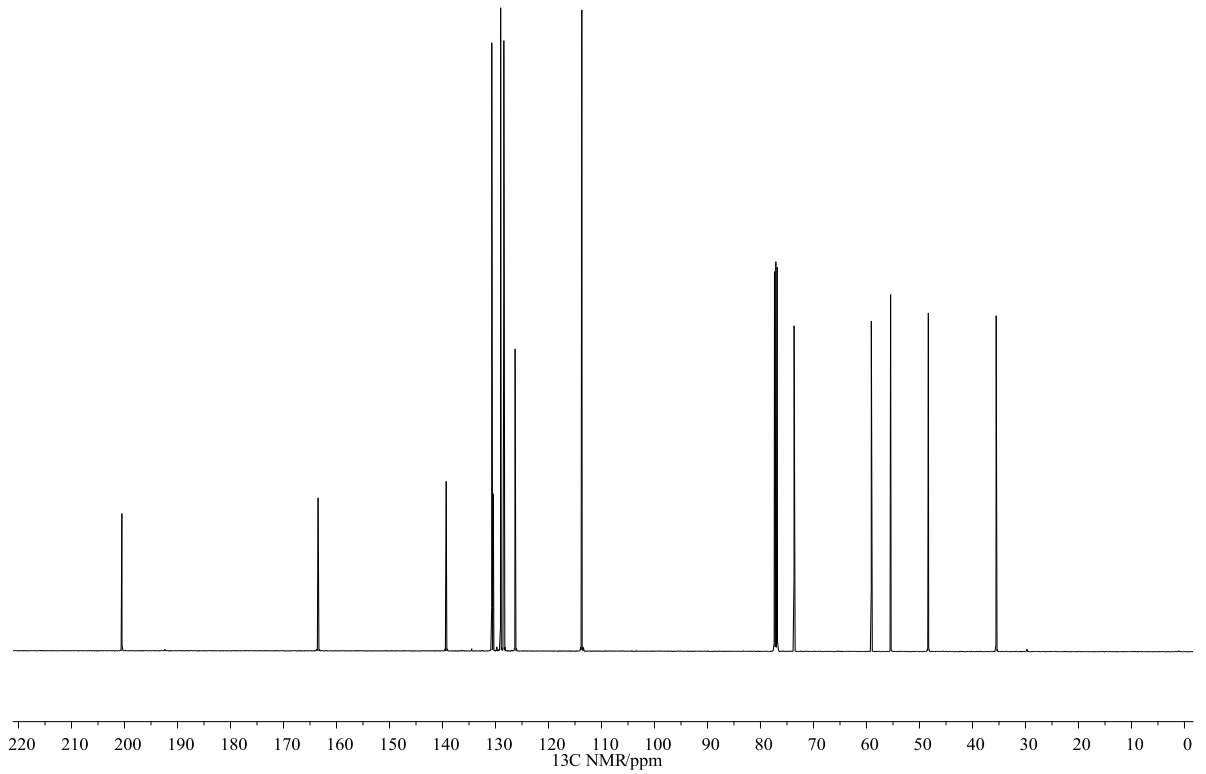



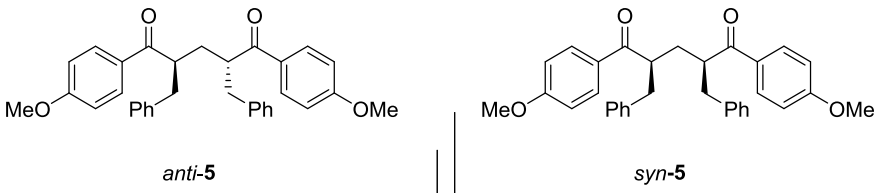
**(±)2-Benzyl-3-methoxy-1-(4-methoxyphenyl)propan-1-one, 4**



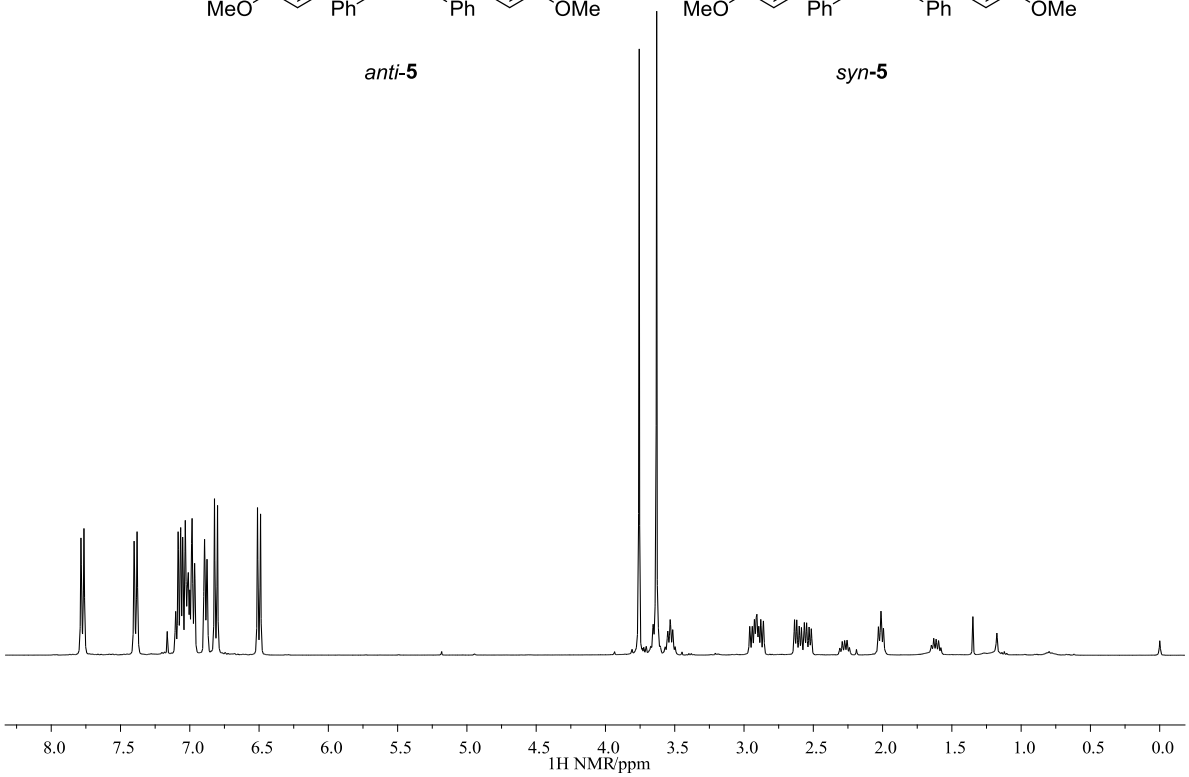
**(±)-(2RR, 4RS)-2,4-dibenzyl-1,5-bis(4-methoxyphenyl)pentane-1,5-dione, 5**

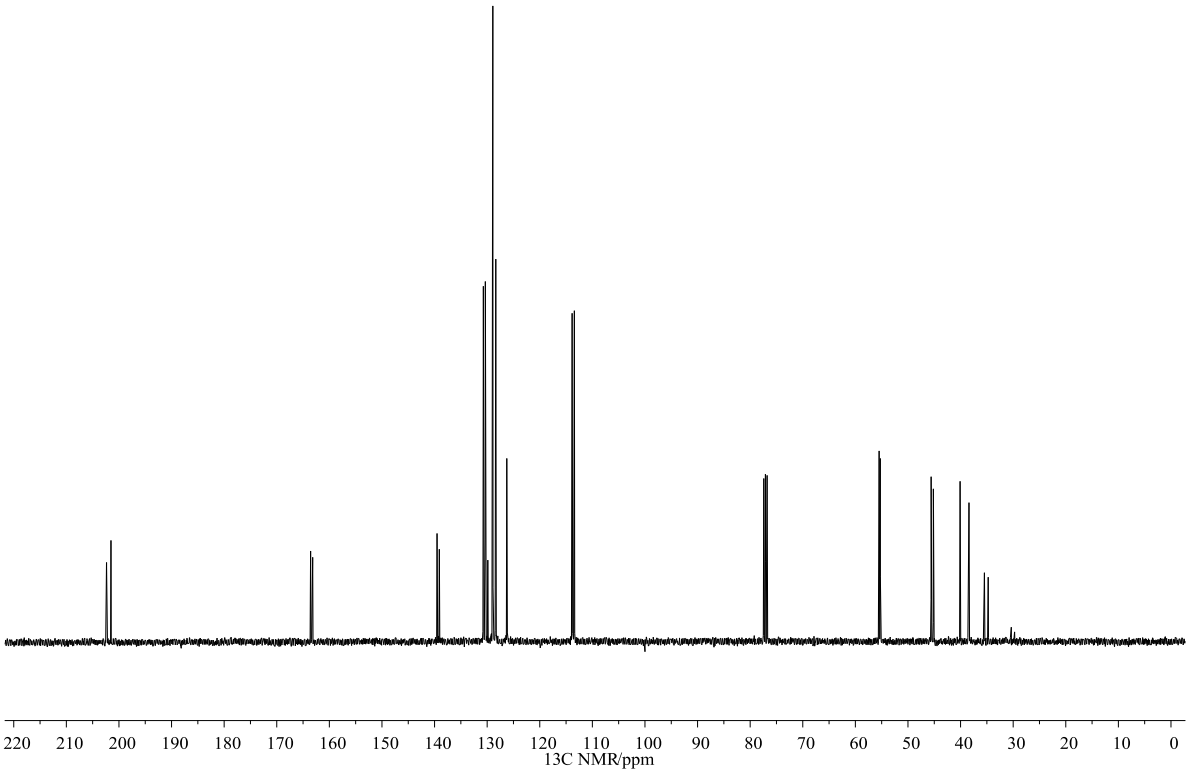



*anti*-**5**

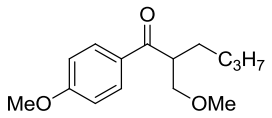


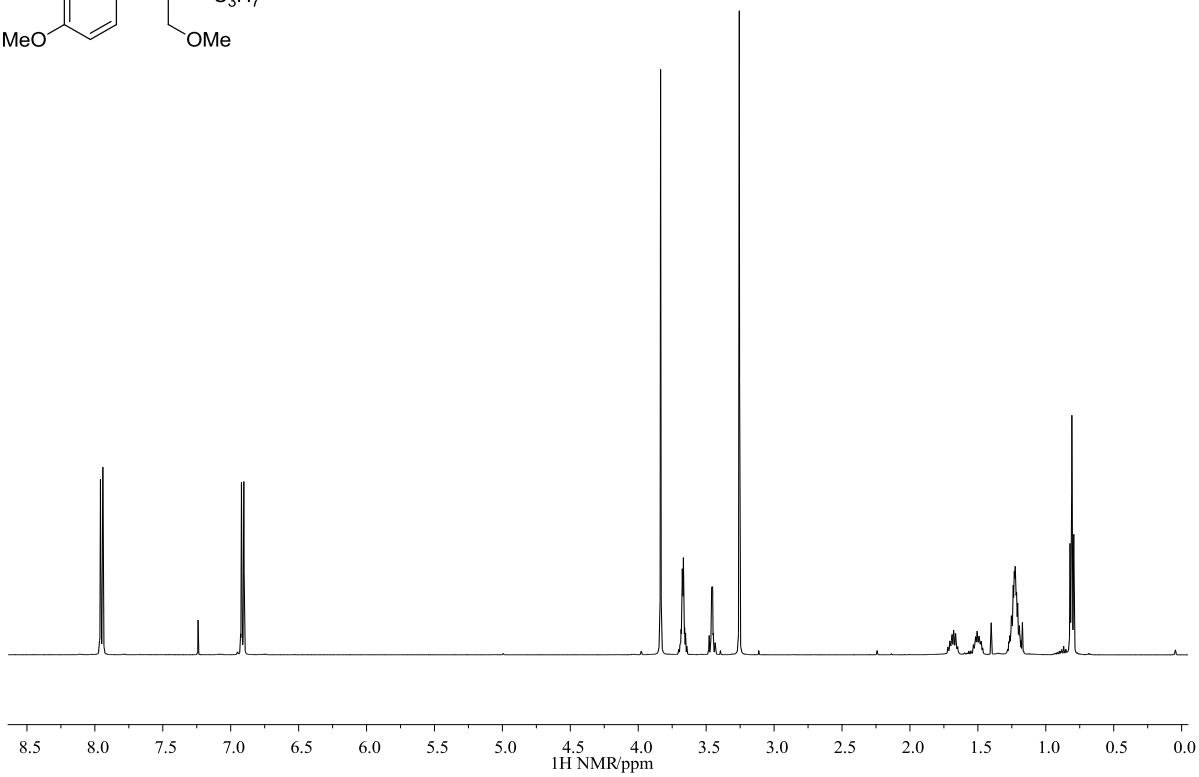

*syn*-**5**

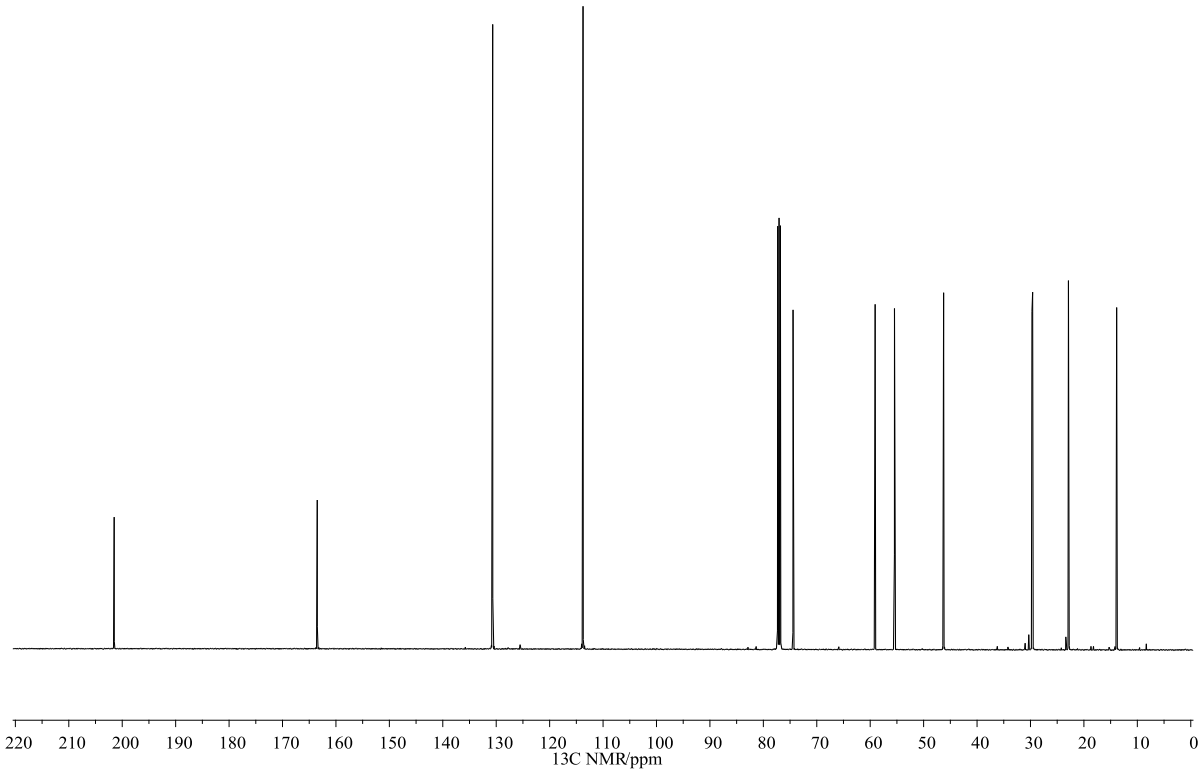


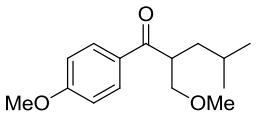
**(±)2-(Methoxymethyl)-1-(4-methoxyphenyl)hexan-1-one, 7**

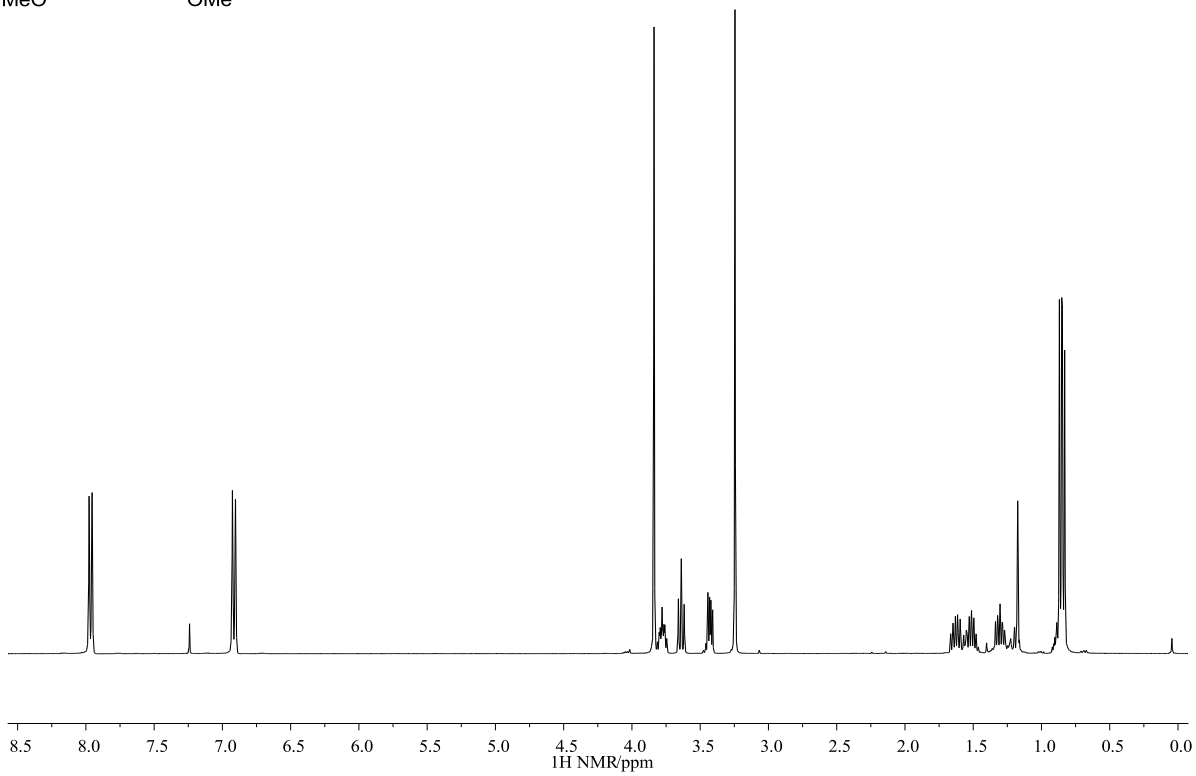


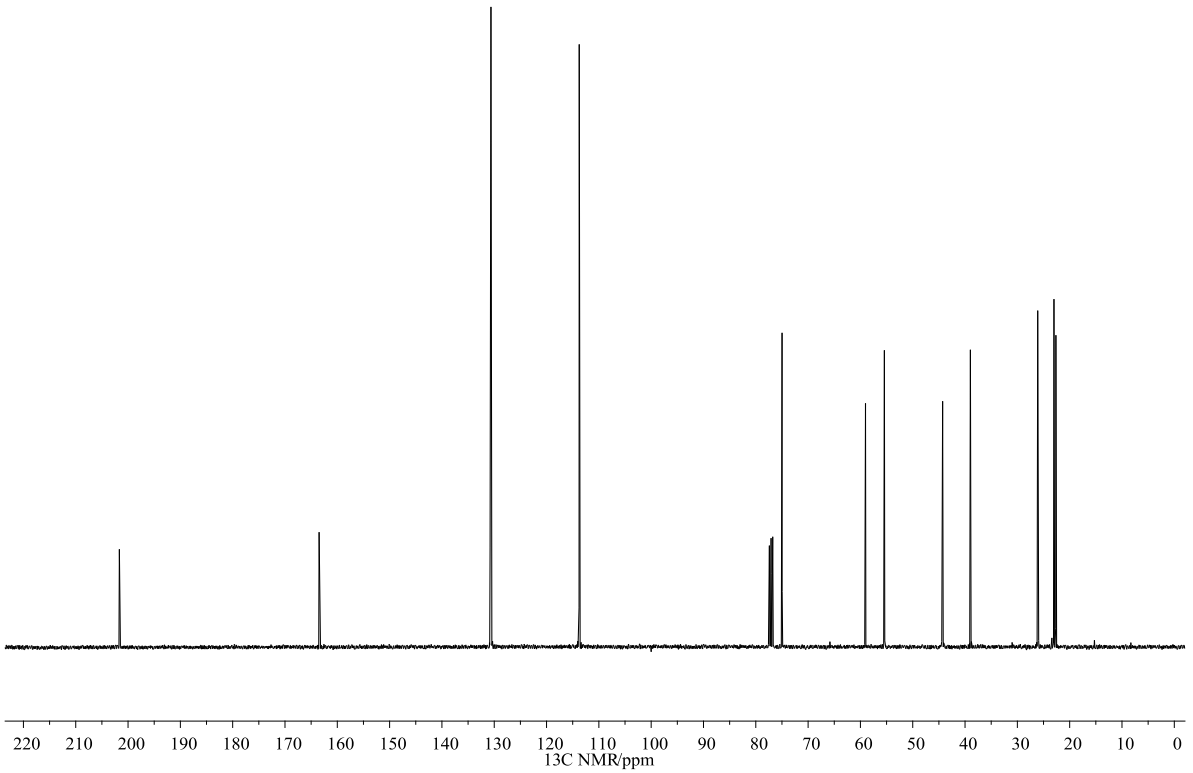



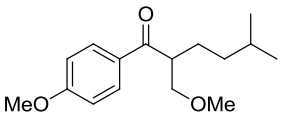
**(±)2-(Methoxymethyl)-1-(4-methoxyphenyl)-4-methylpentan-1-one, 9**

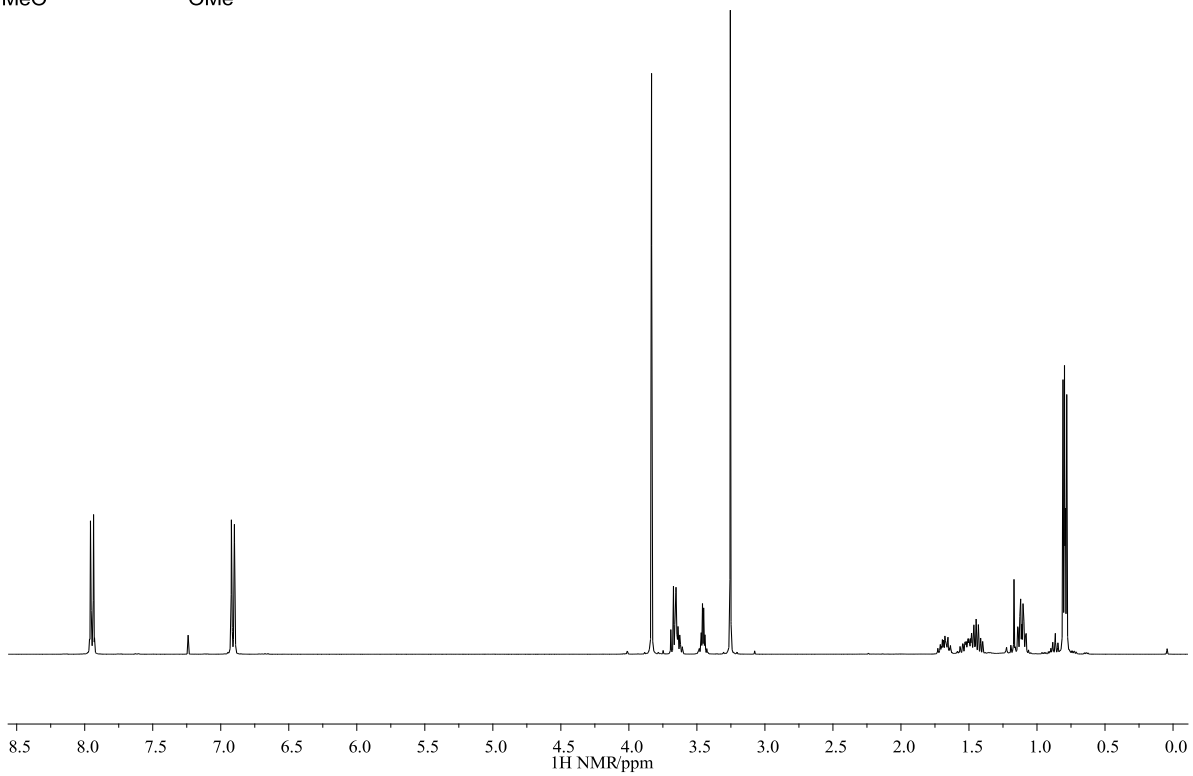


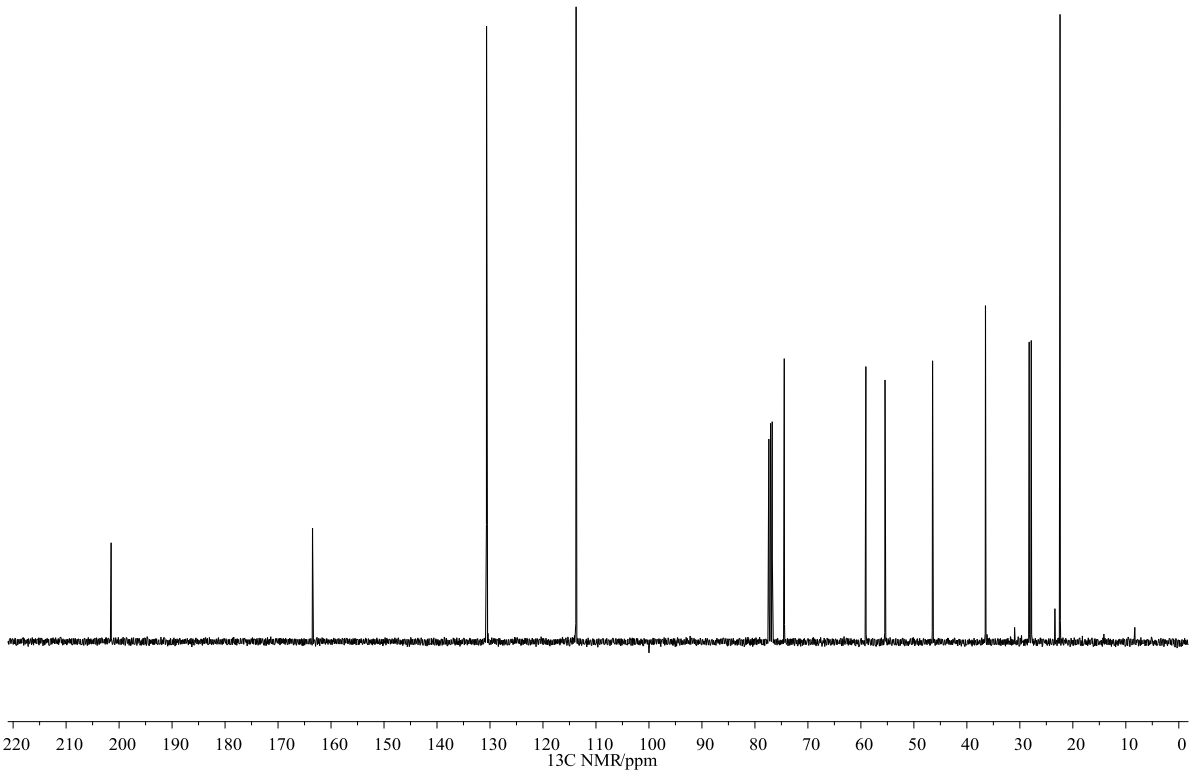



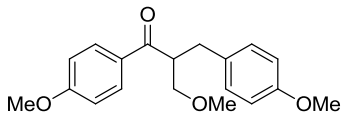
**(±)2-(Methoxymethyl)-1-(4-methoxyphenyl)-5-methylhexan-1-one, 11**

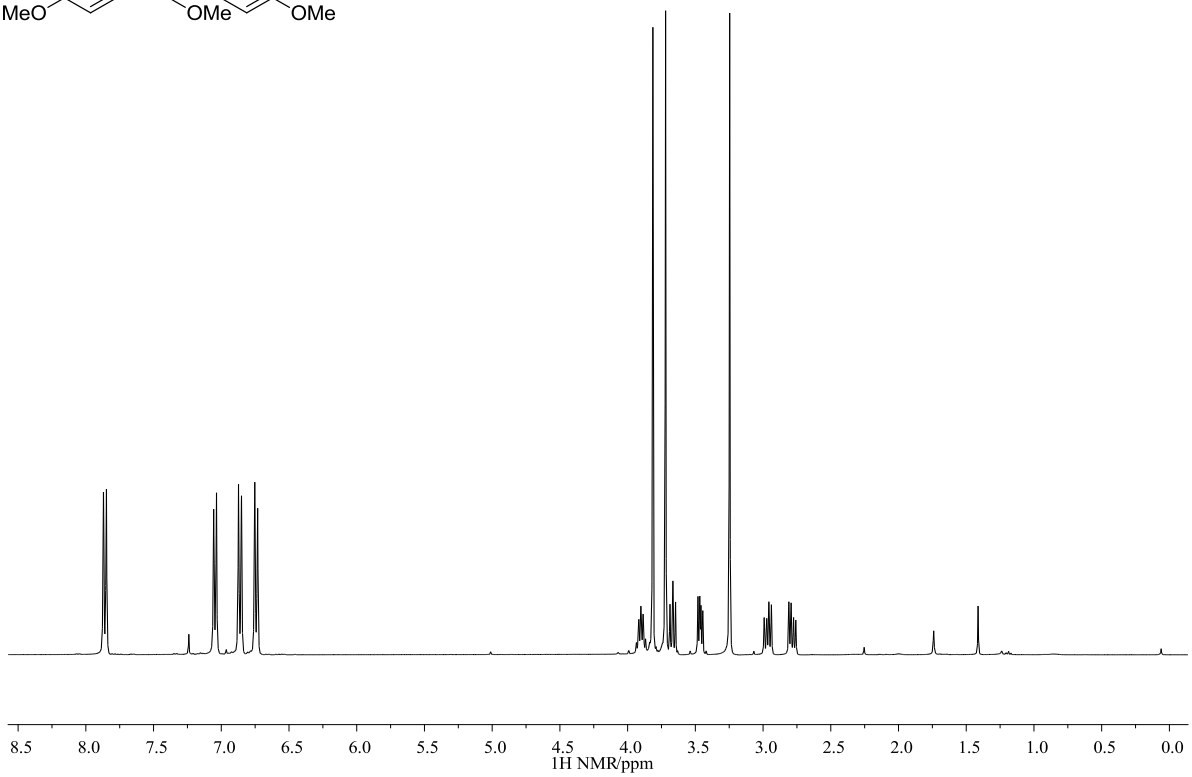


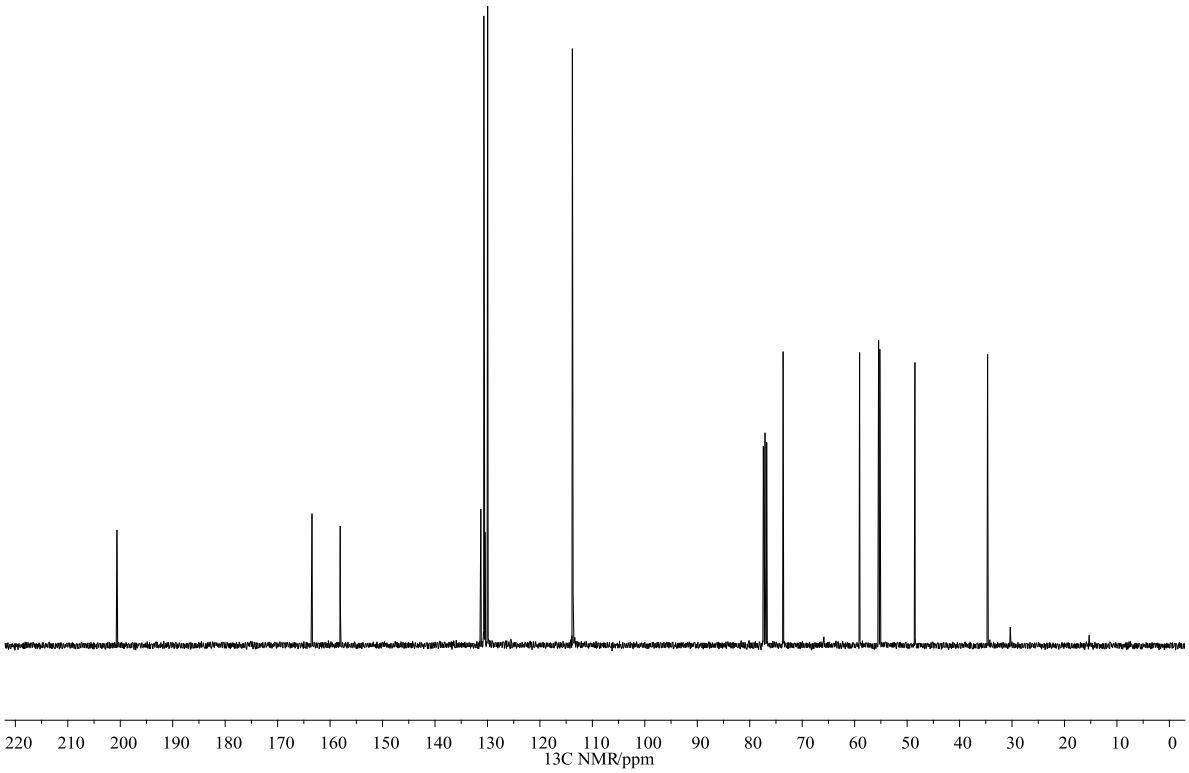



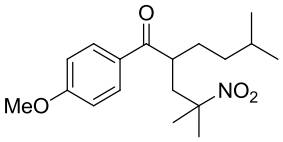
**(±)3-Methoxy-2-(4-methoxybenzyl)-1-(4-methoxyphenyl)propan-1-one, 13**

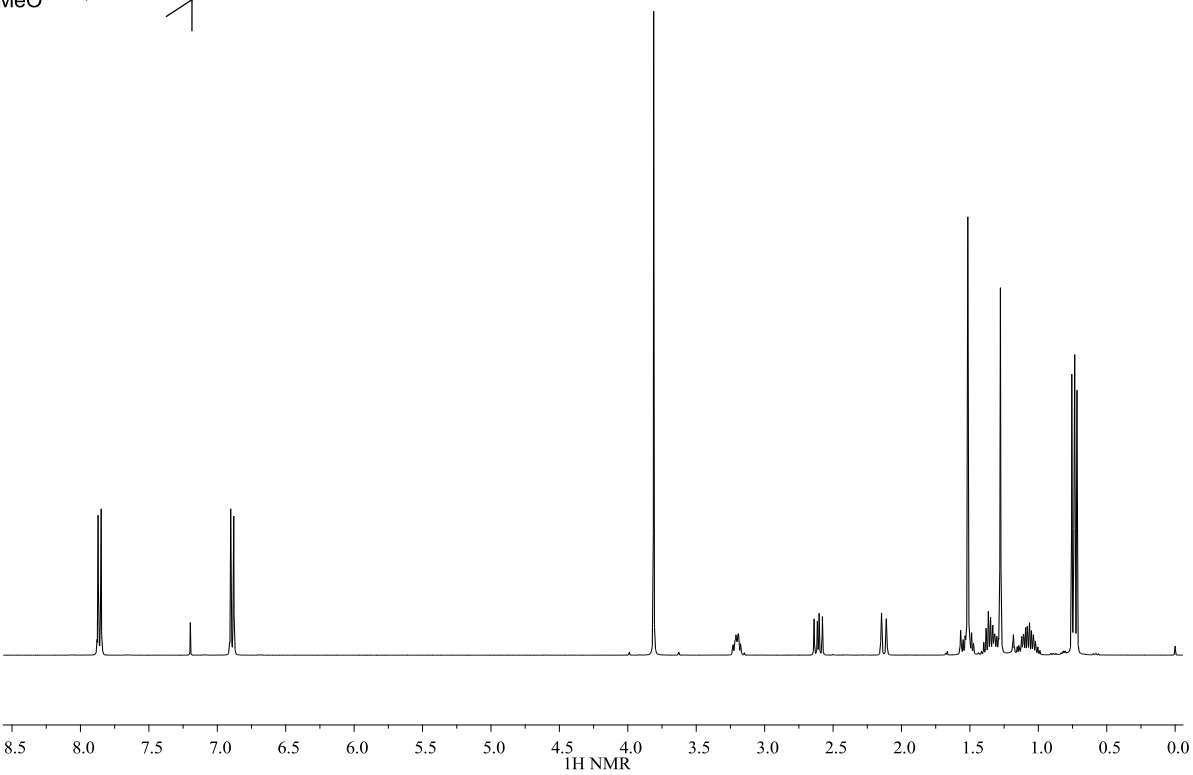


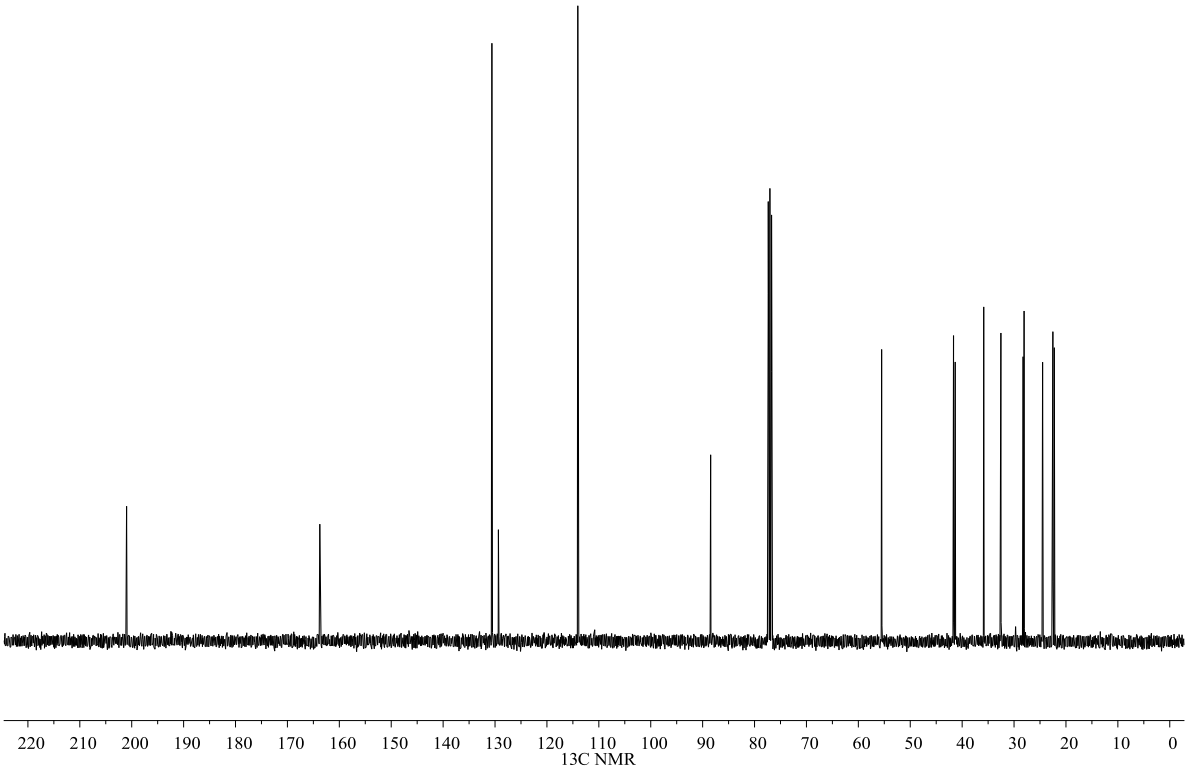



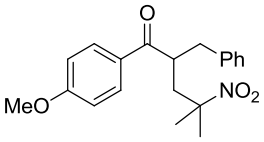
**(±)1-(4-Methoxyphenyl)-5-methyl-2-(2-methyl-2-nitropropyl)hexan-1-one, 14**

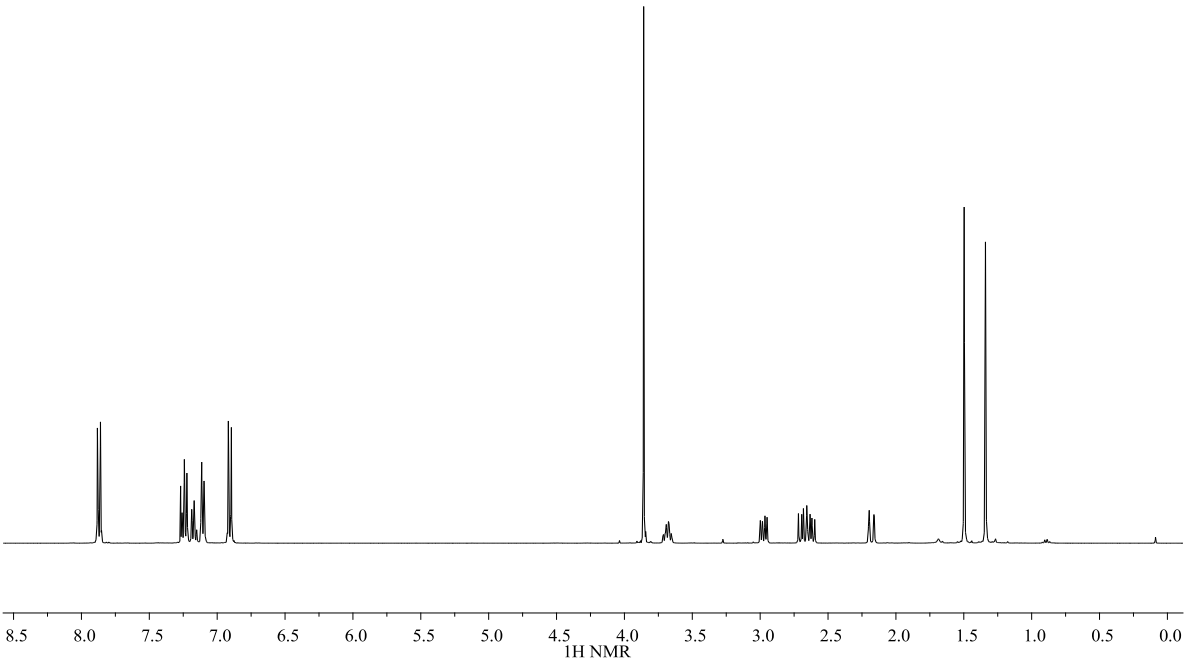


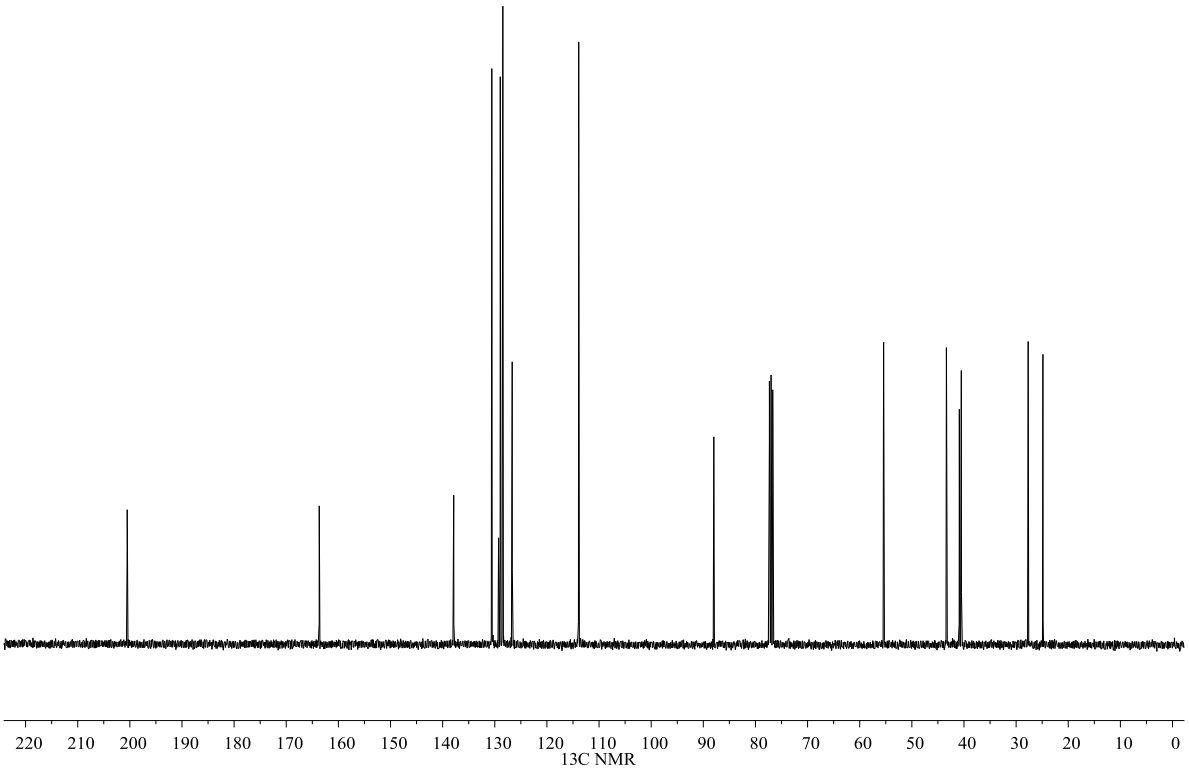



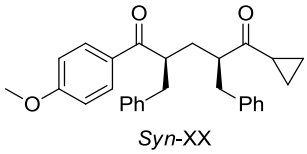
**(±)2-Benzyl-1-(4-methoxyphenyl)-4-methyl-4-nitropentan-1-one, 15**



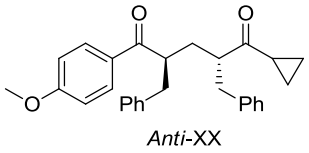



**(±)(2*RS*,4*SS*)-2,4-Dibenzyl-1-cyclopropyl-5-(4-methoxyphenyl)pentane-1,5-dione, 16**

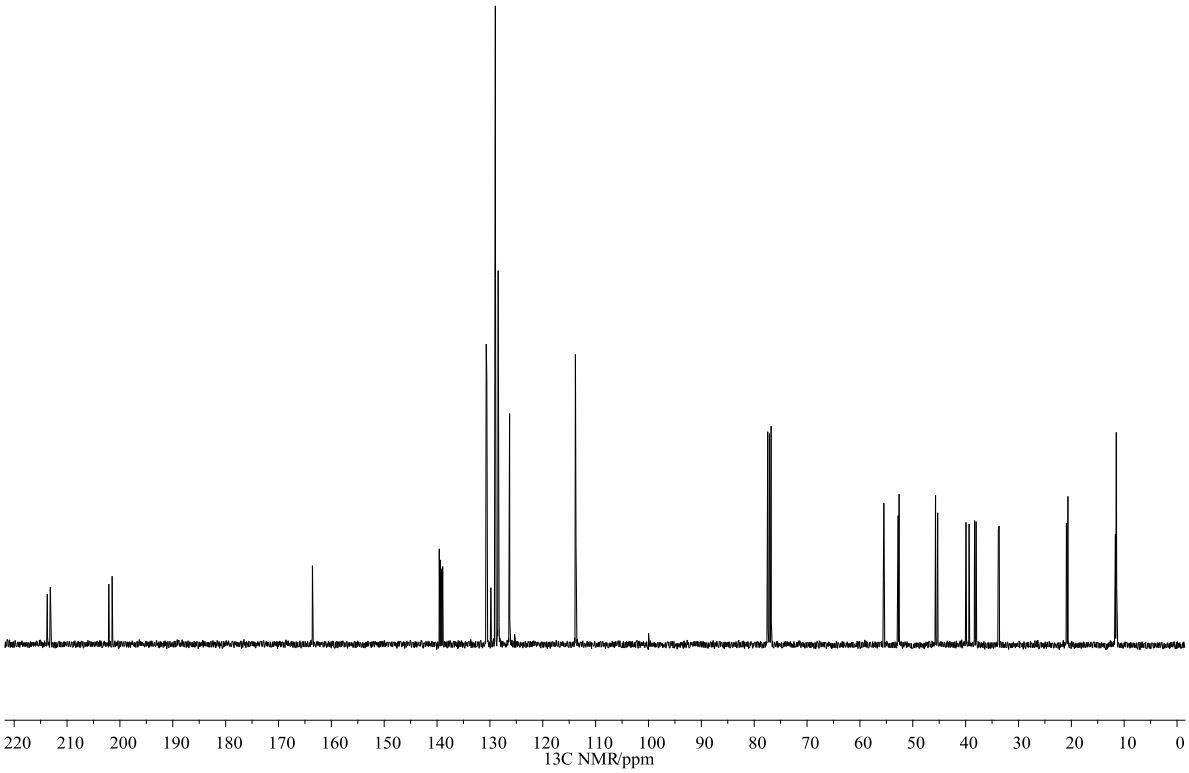



*Syn*-XX

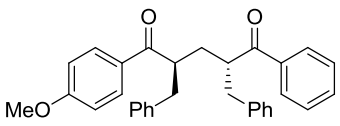



*Anti*-XX

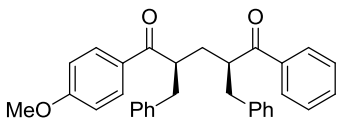


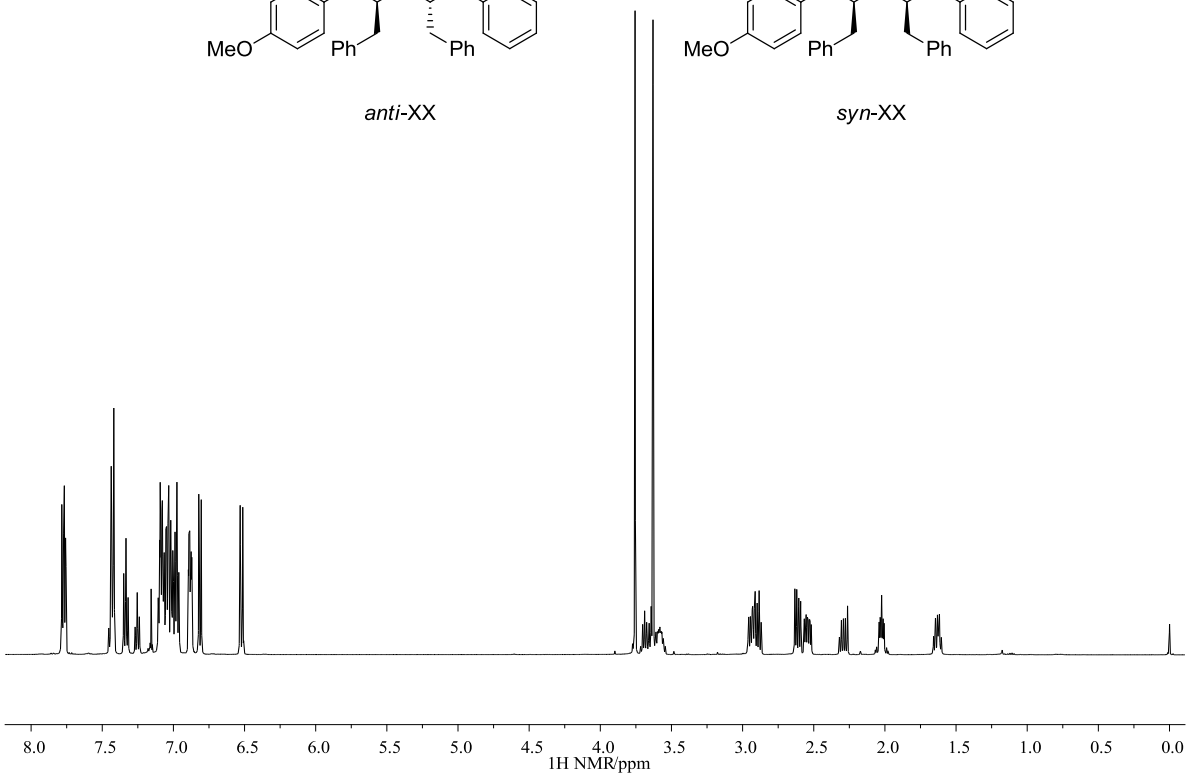
**(±) (2SS,4RS)-2,4-Dibenzyl-1-(4-methoxyphenyl)-5-phenylpentane-1,5-dione, 17**

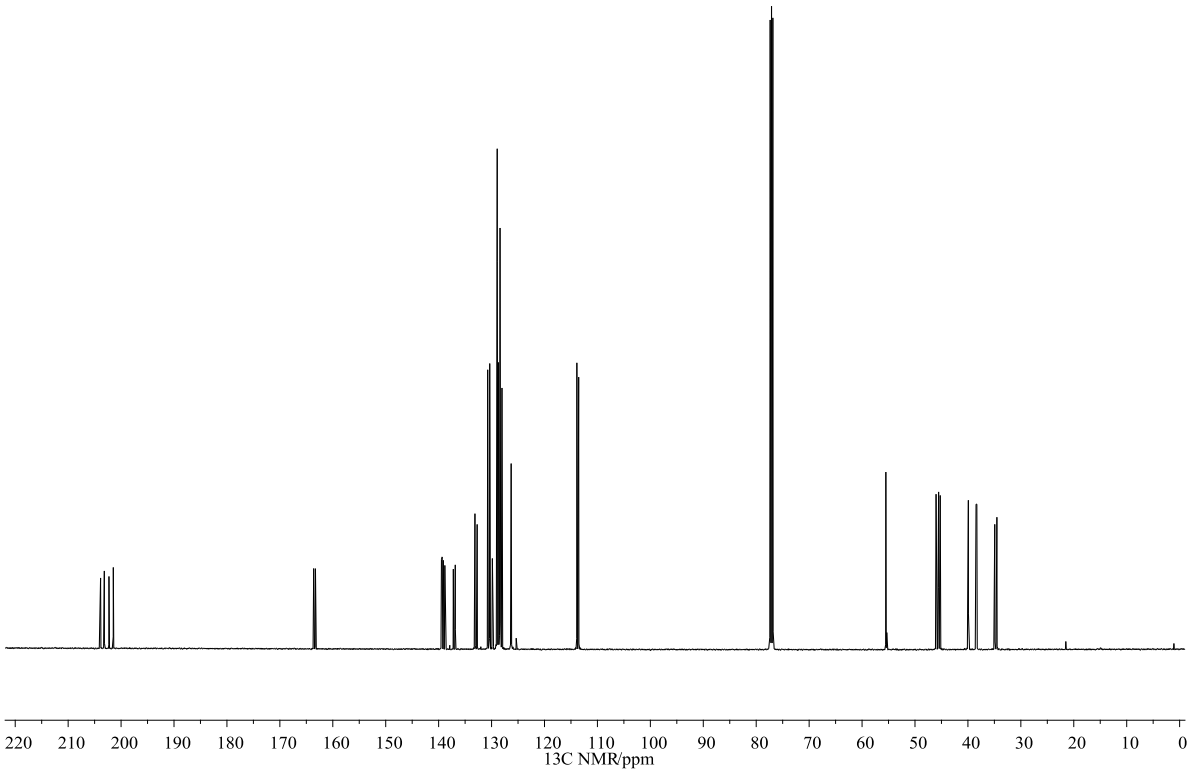



*anti*-XX

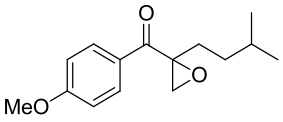


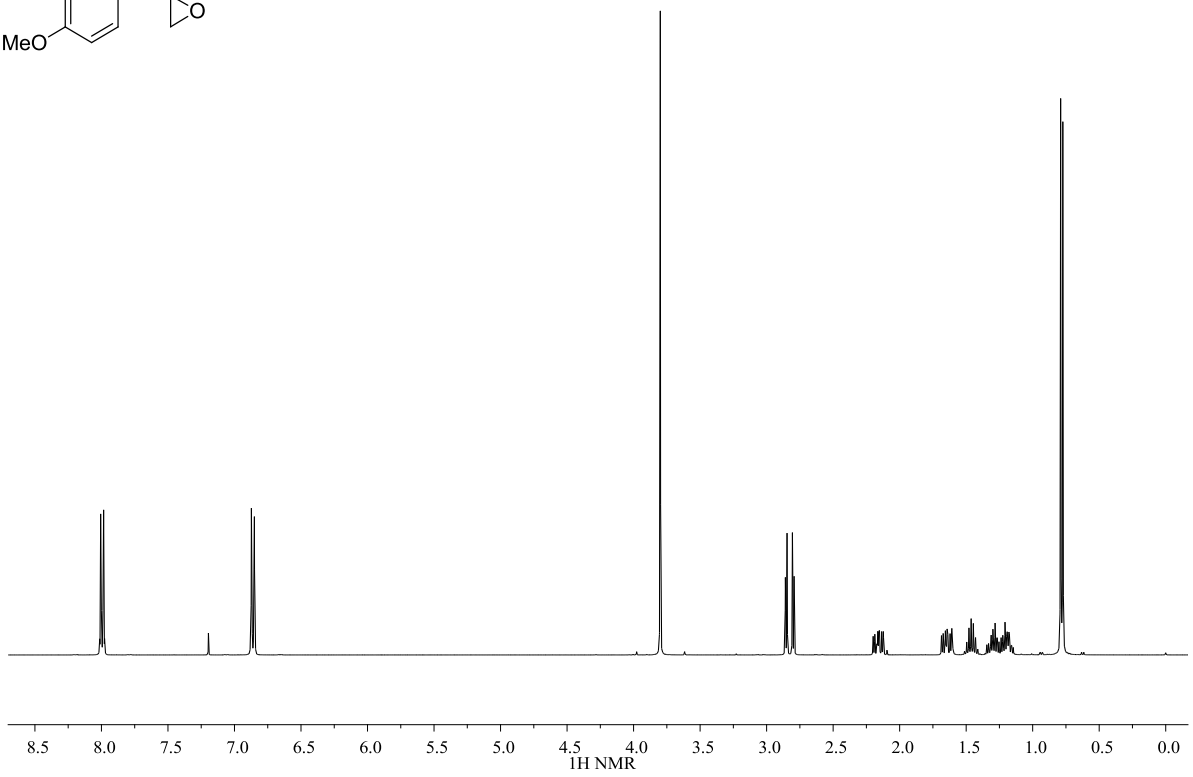

*syn*-XX



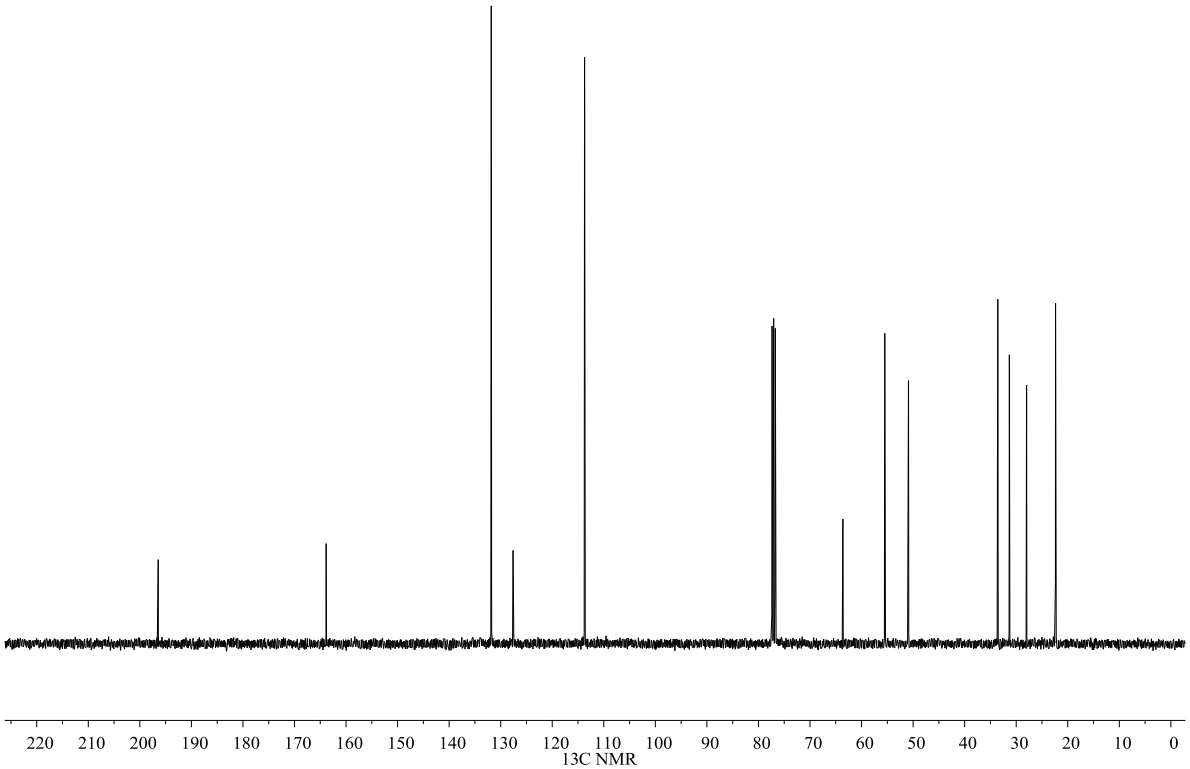



**(±) (2-Benzyloxiran-2-yl)(4-methoxyphenyl)methanone, 18**



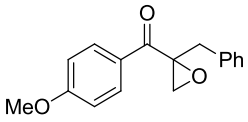



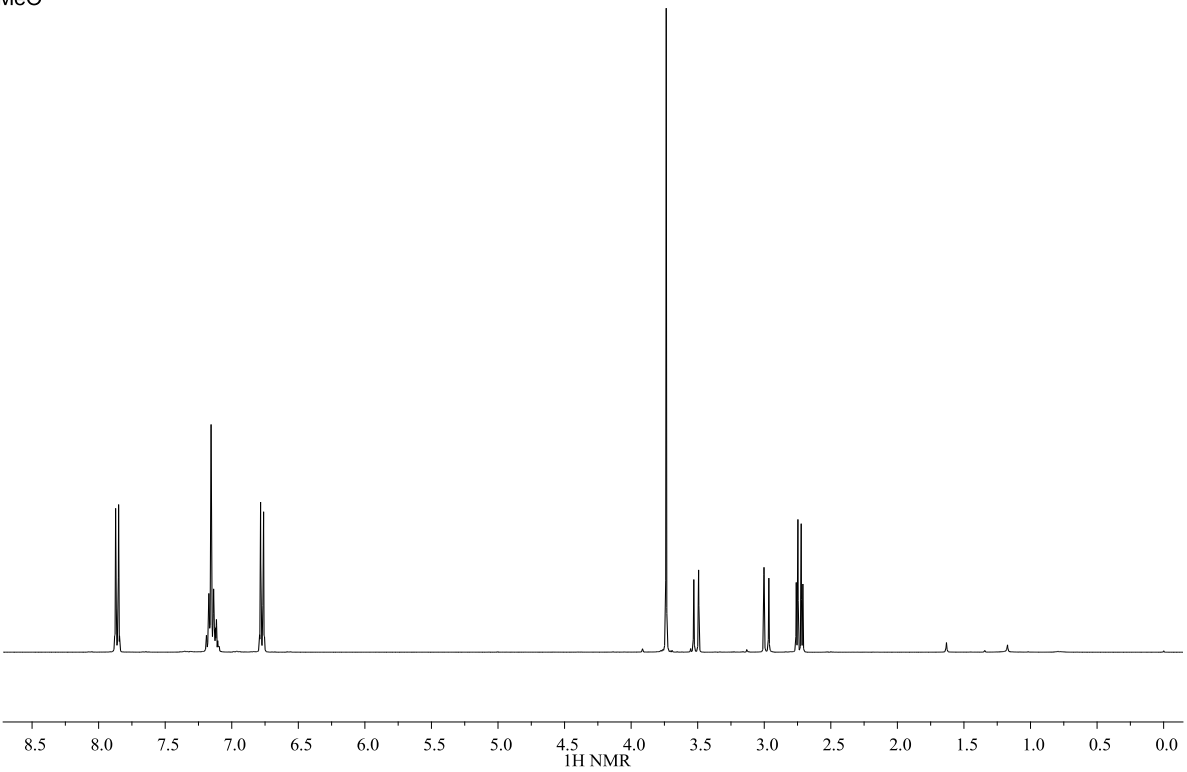

1H NMR

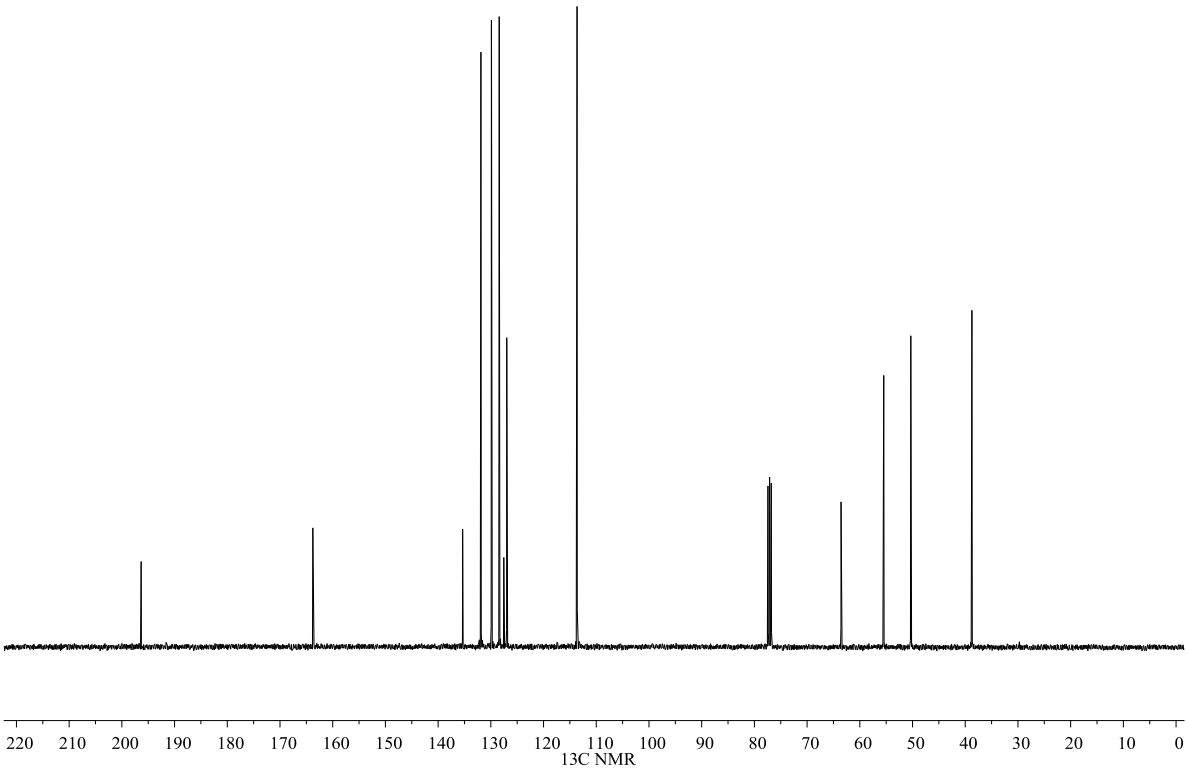



13C NMR

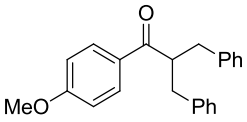
**(±) (2-Benzyloxiran-2-yl)(4-methoxyphenyl)methanone, 19**

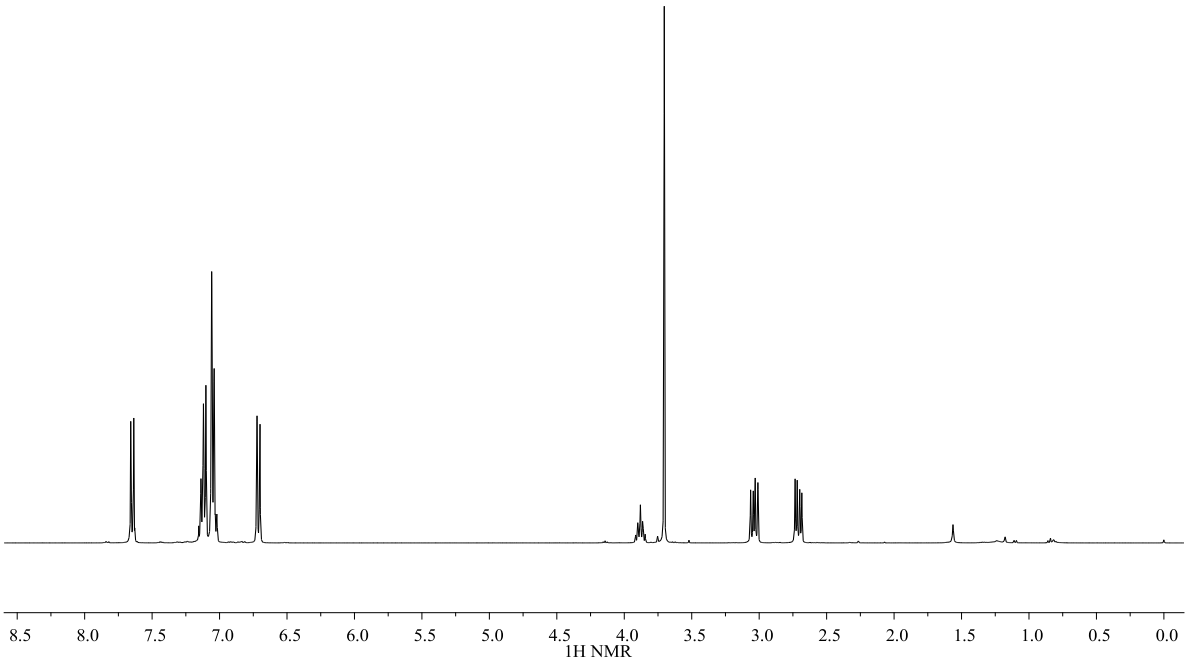


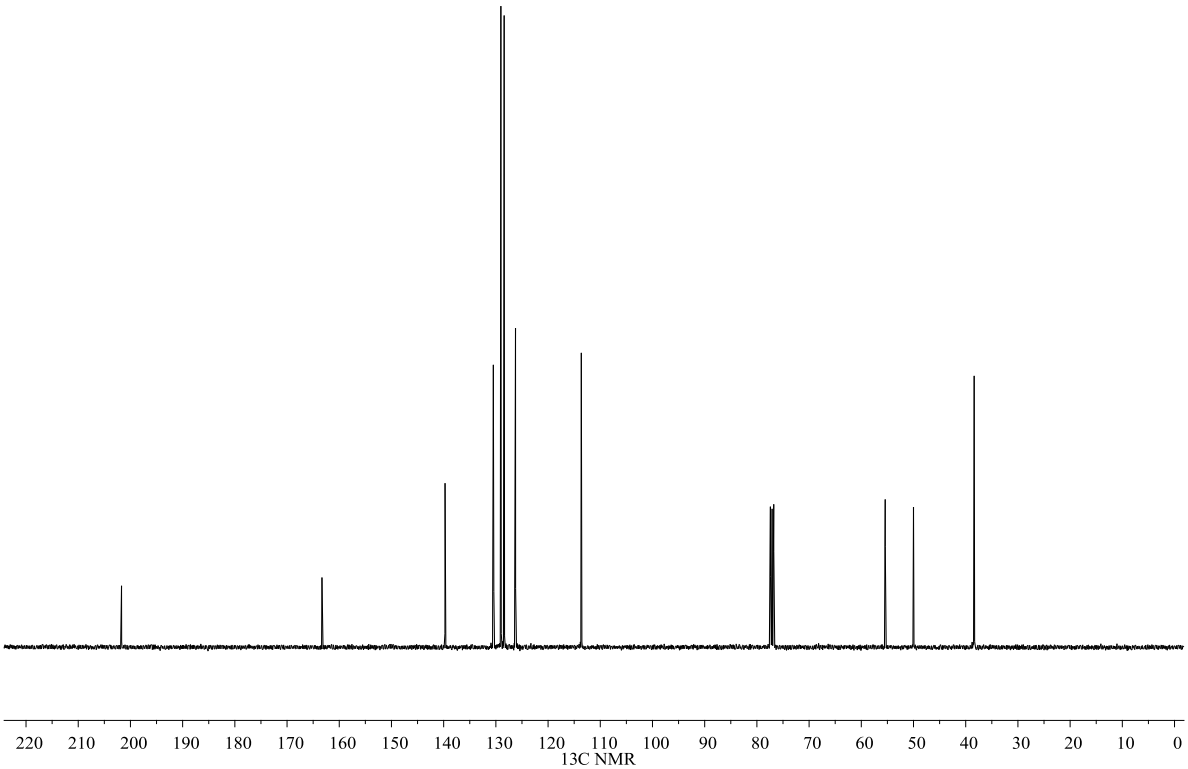



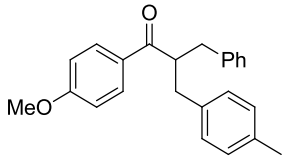
**2-Benzyl-1-(4-methoxyphenyl)-3-phenylpropan-1-one, 20**



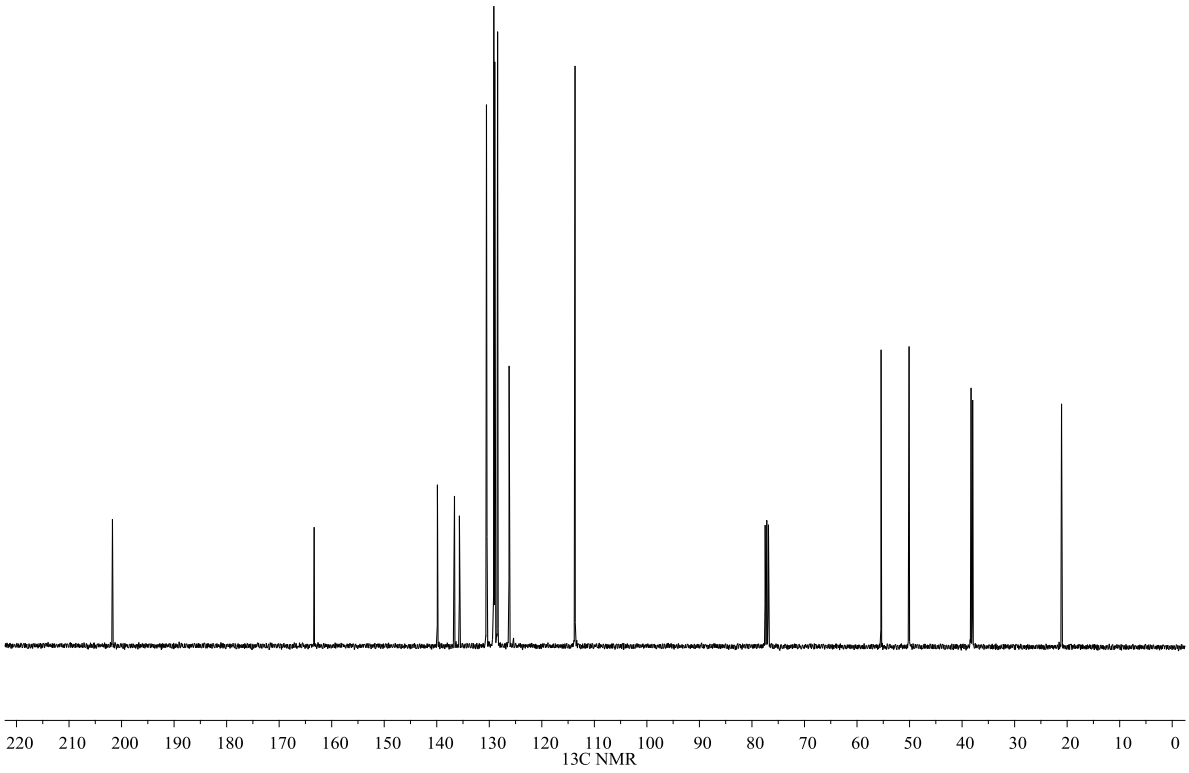



**2-Benzyl-1-(4-methoxyphenyl)-3-(p-tolyl)propan-1-one, 21**

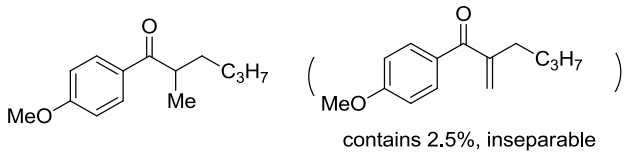






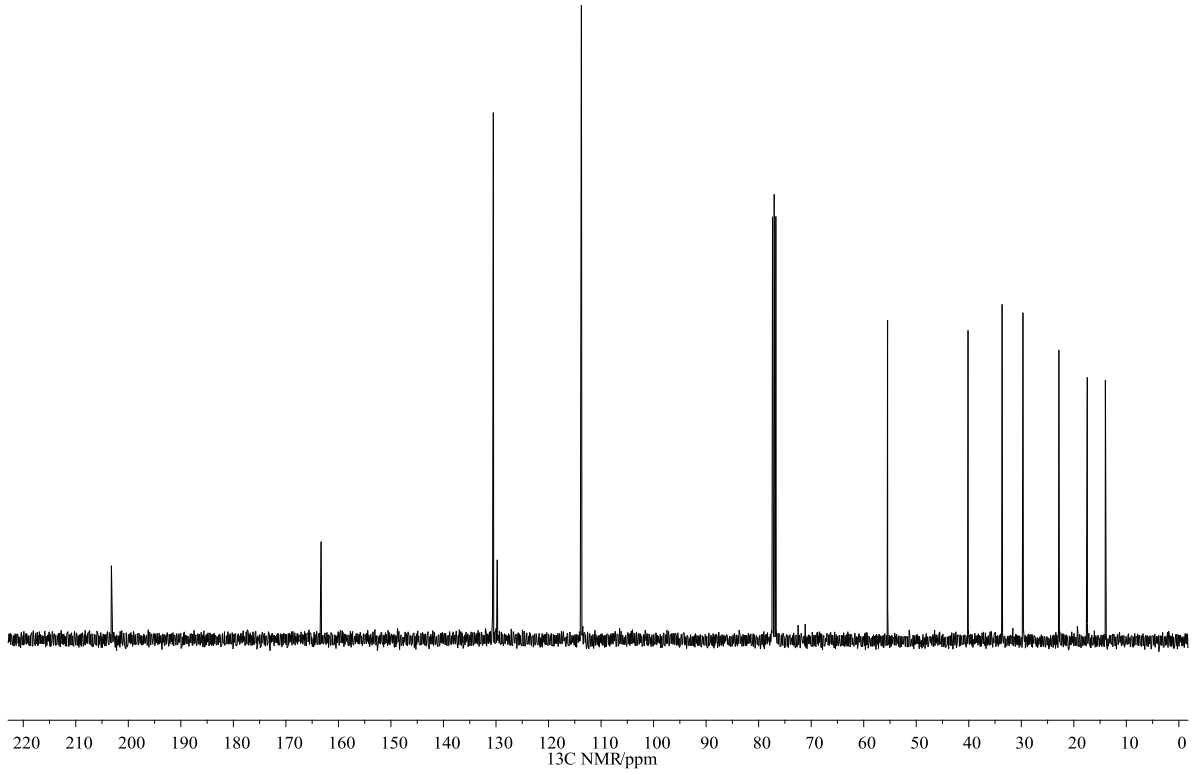


**(±)1-(4-Methoxyphenyl)-2-methylhexan-1-one, 22**

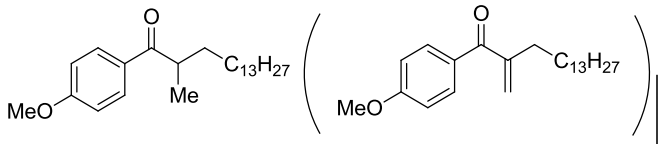



contains 2.5%, inseparable

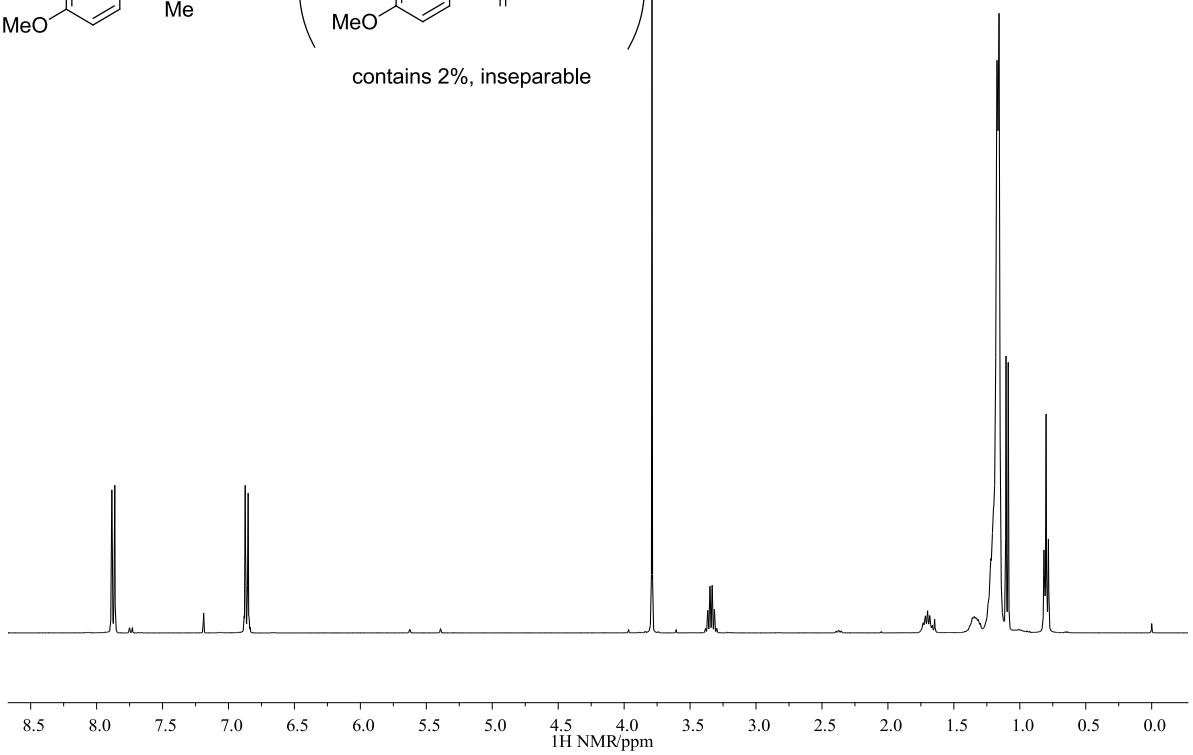


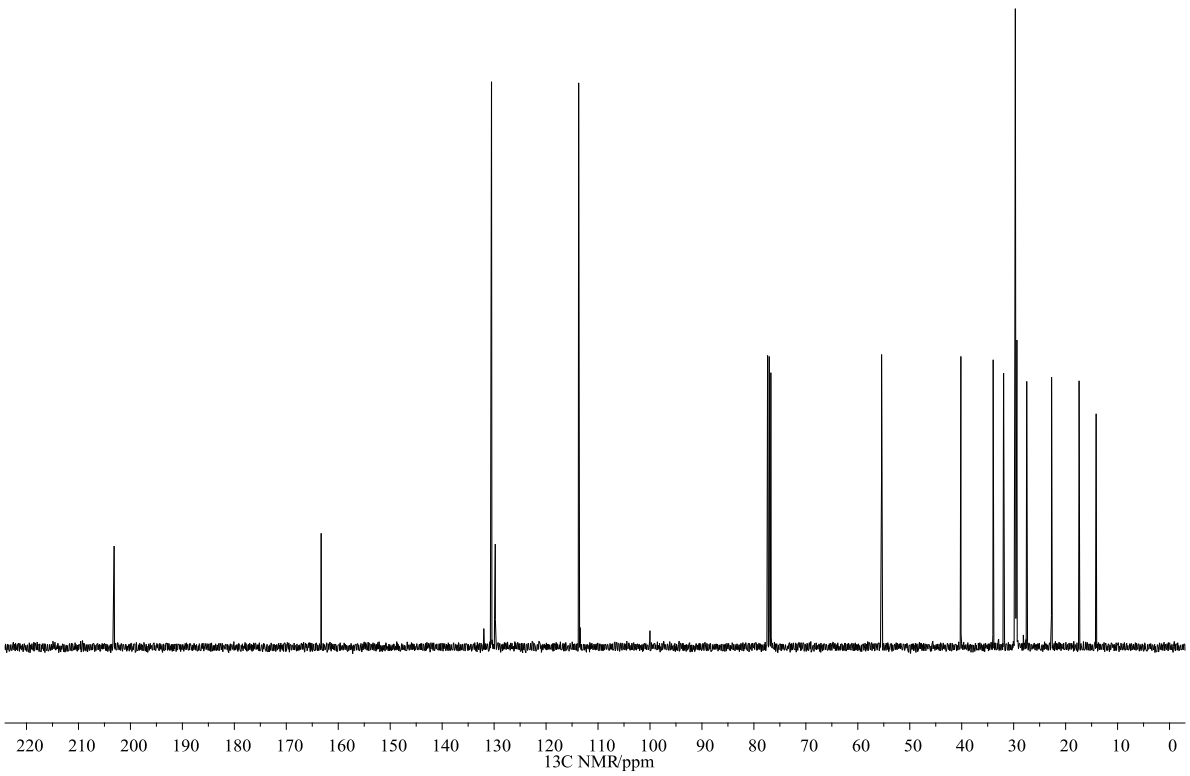



**(±)1-(4-Methoxyphenyl)-2-methylhexadecan-1-one, 23**

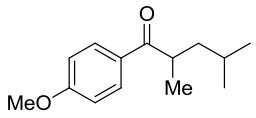


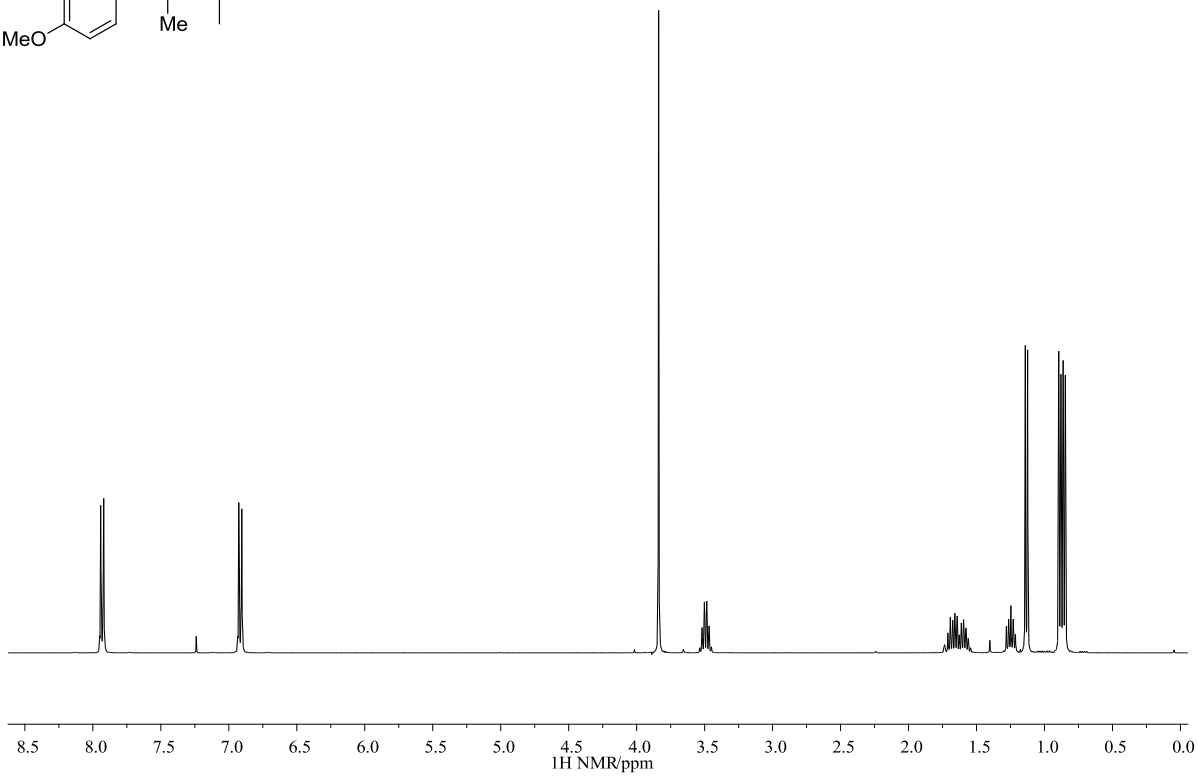

contains 2%, inseparable

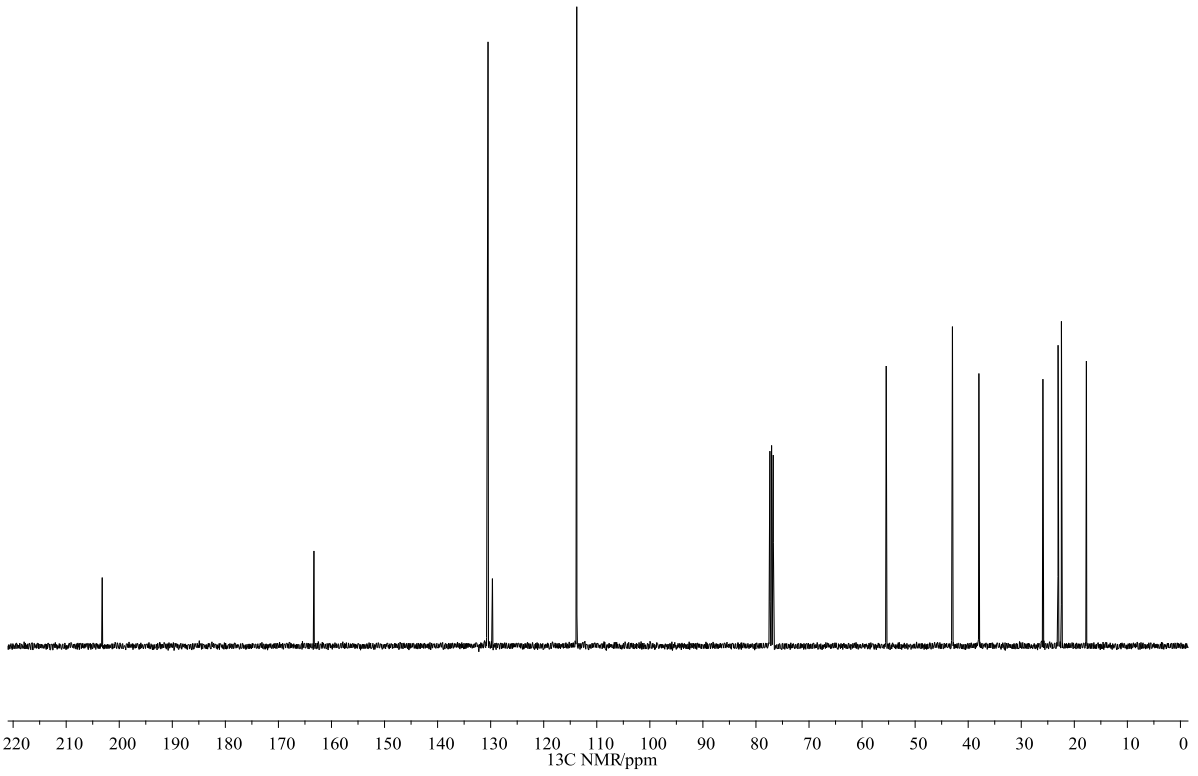


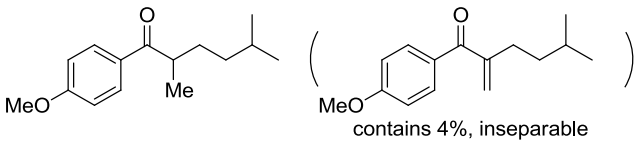
**(±)1-(4-methoxyphenyl)-2,4-dimethylpentan-1-one, 24**



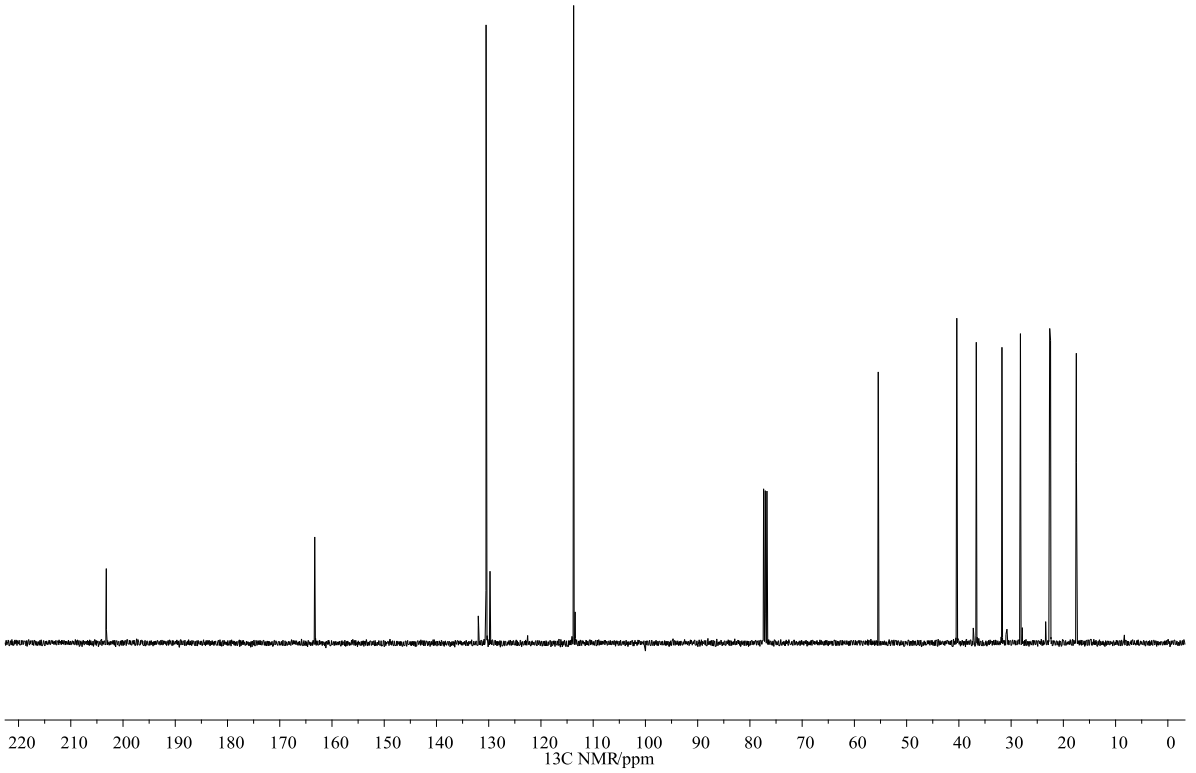



**(±)1-(4-Methoxyphenyl)-2,5-dimethylhexan-1-one, 25**

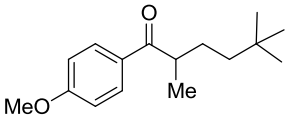


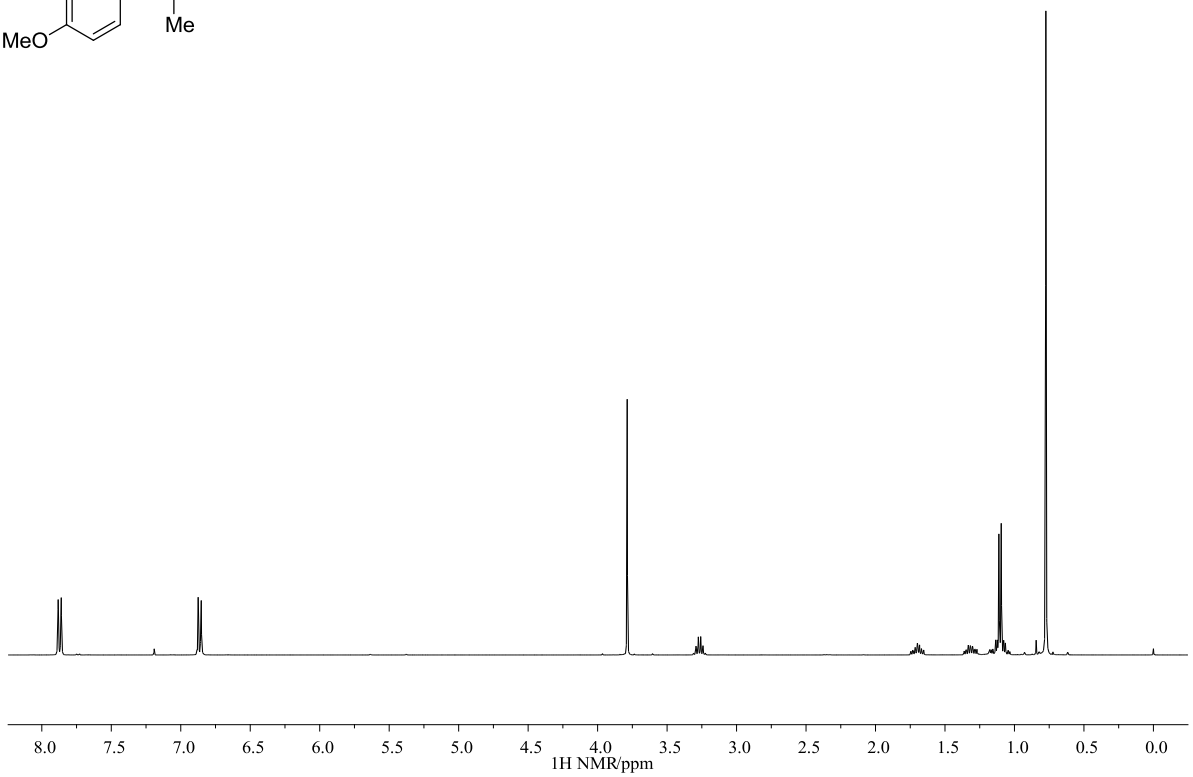

contains 4%, inseparable

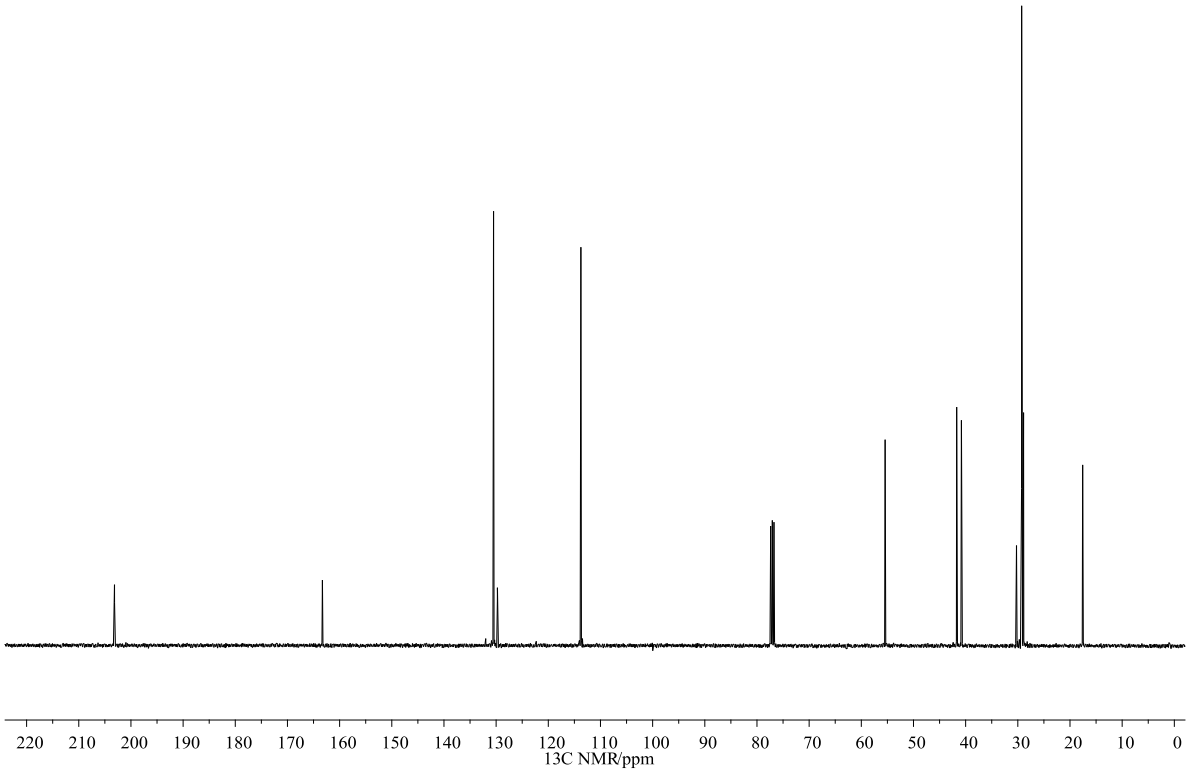


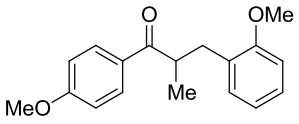
**1-(4-Methoxyphenyl)-2,5,5-trimethylhexan-1-one, 26**

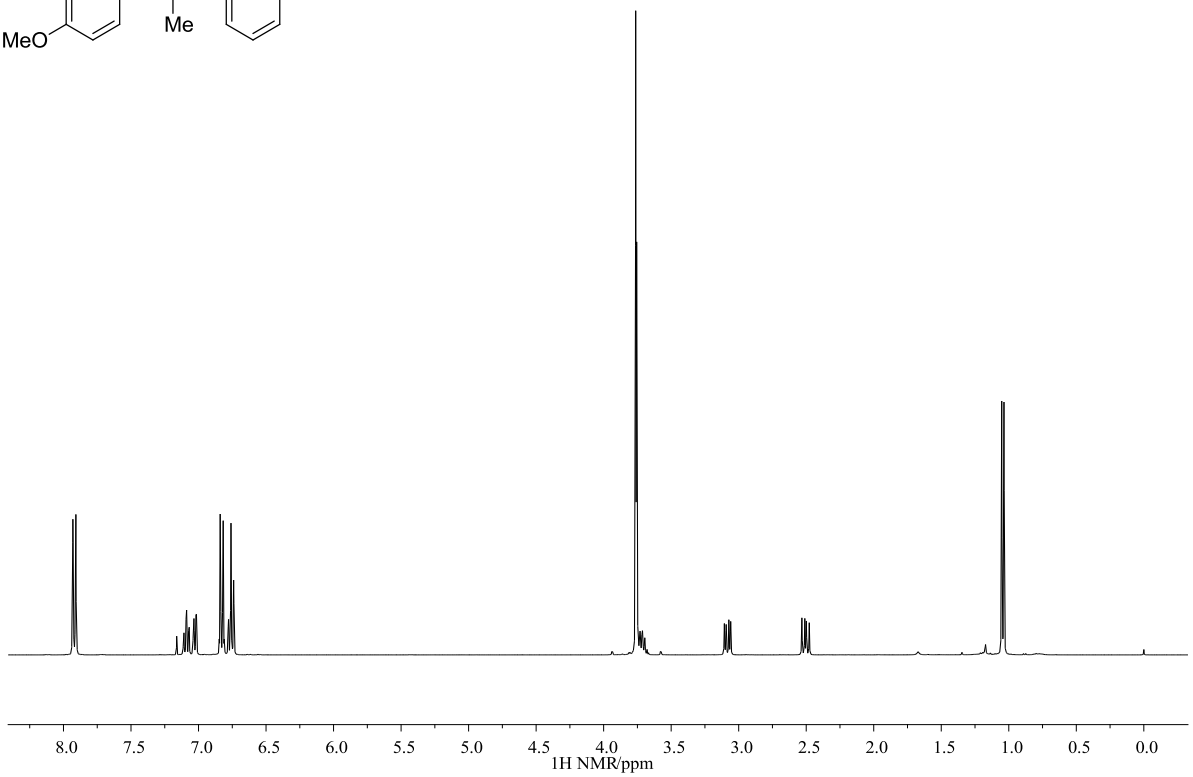


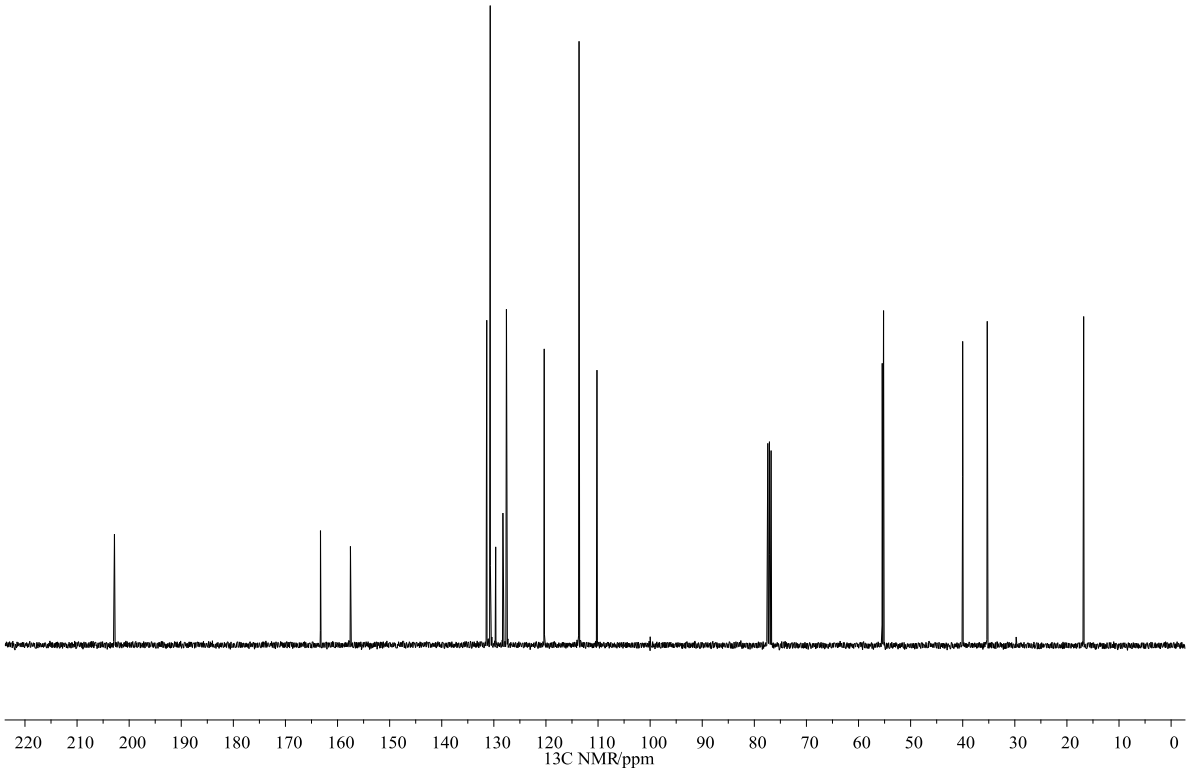



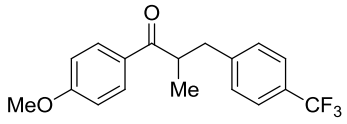
**(±)3-(2-methoxyphenyl)-1-(4-methoxyphenyl)-2-methylpropan-1-one, 28**

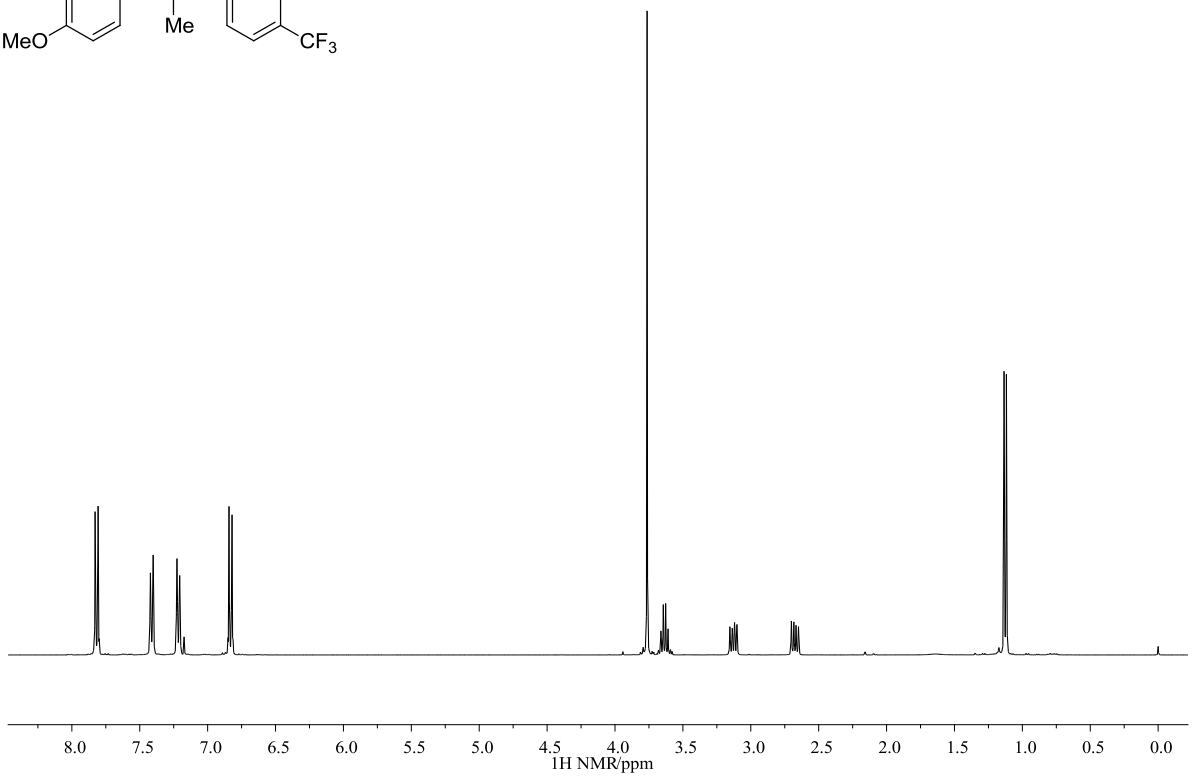


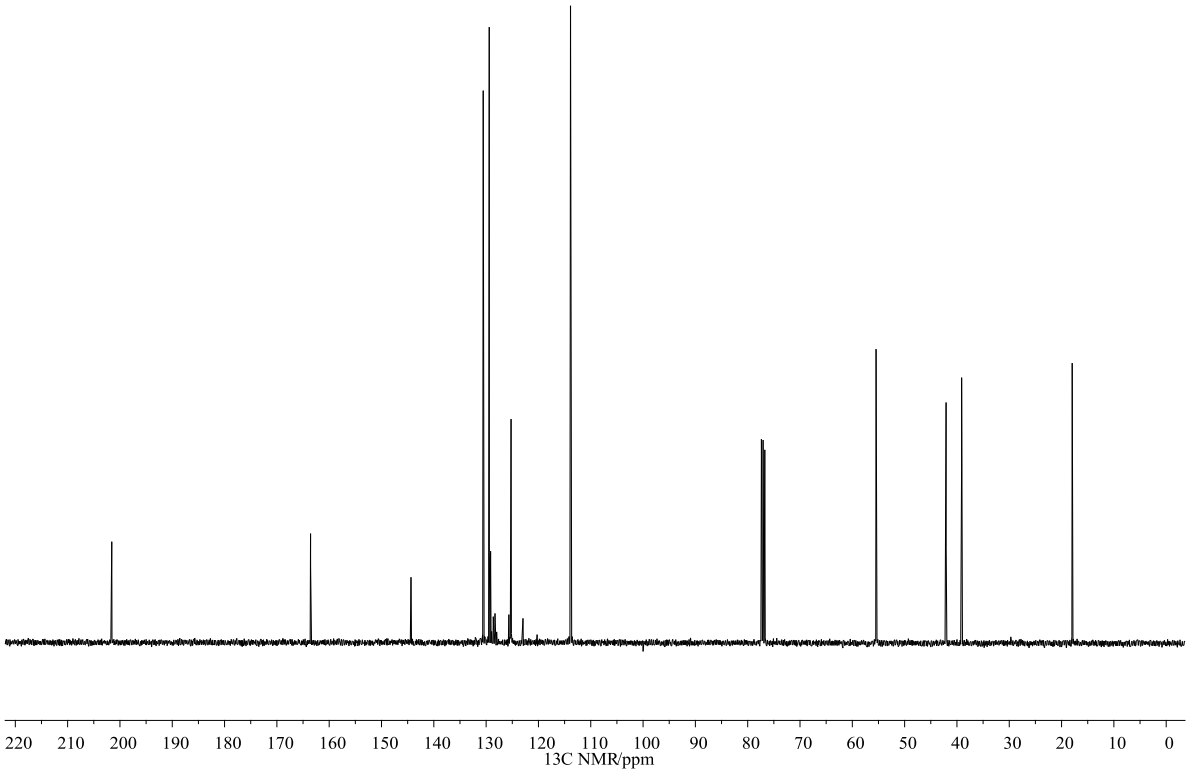



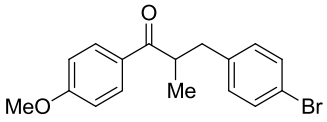
**(±)1-(4-methoxyphenyl)-2-methyl-3-(4-(trifluoromethyl)phenyl)propan-1-one, 29**

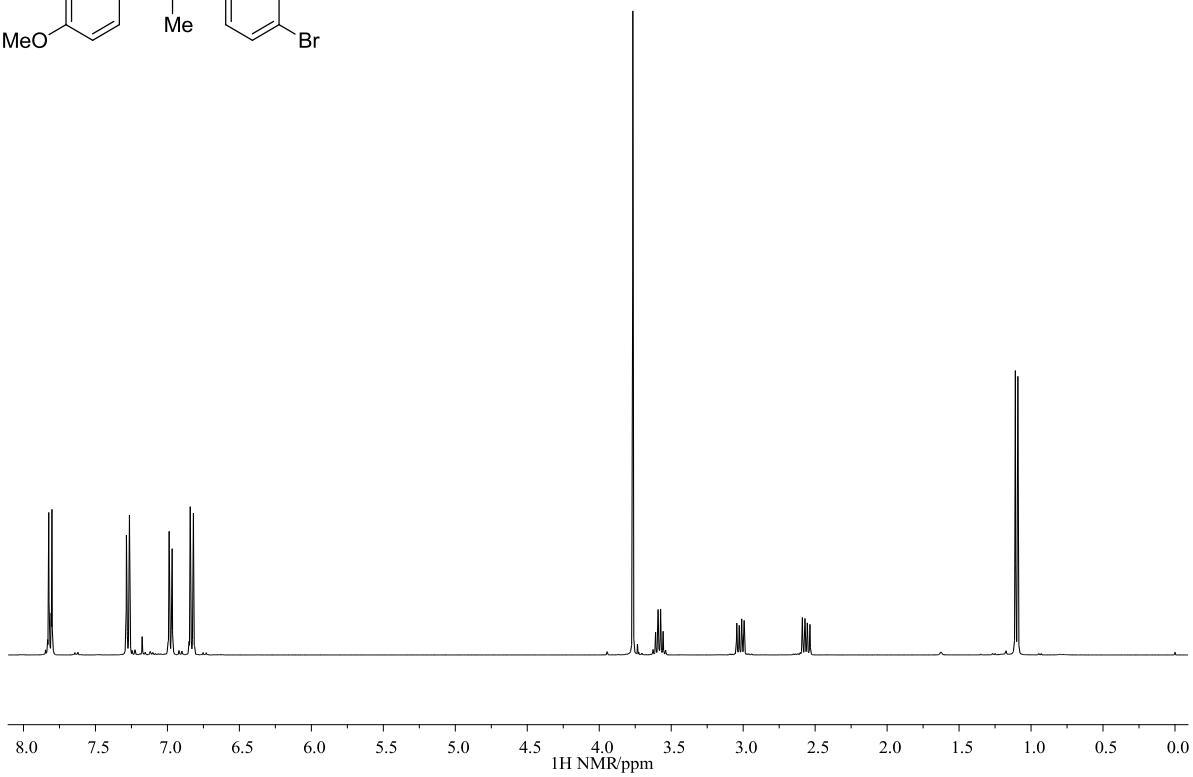


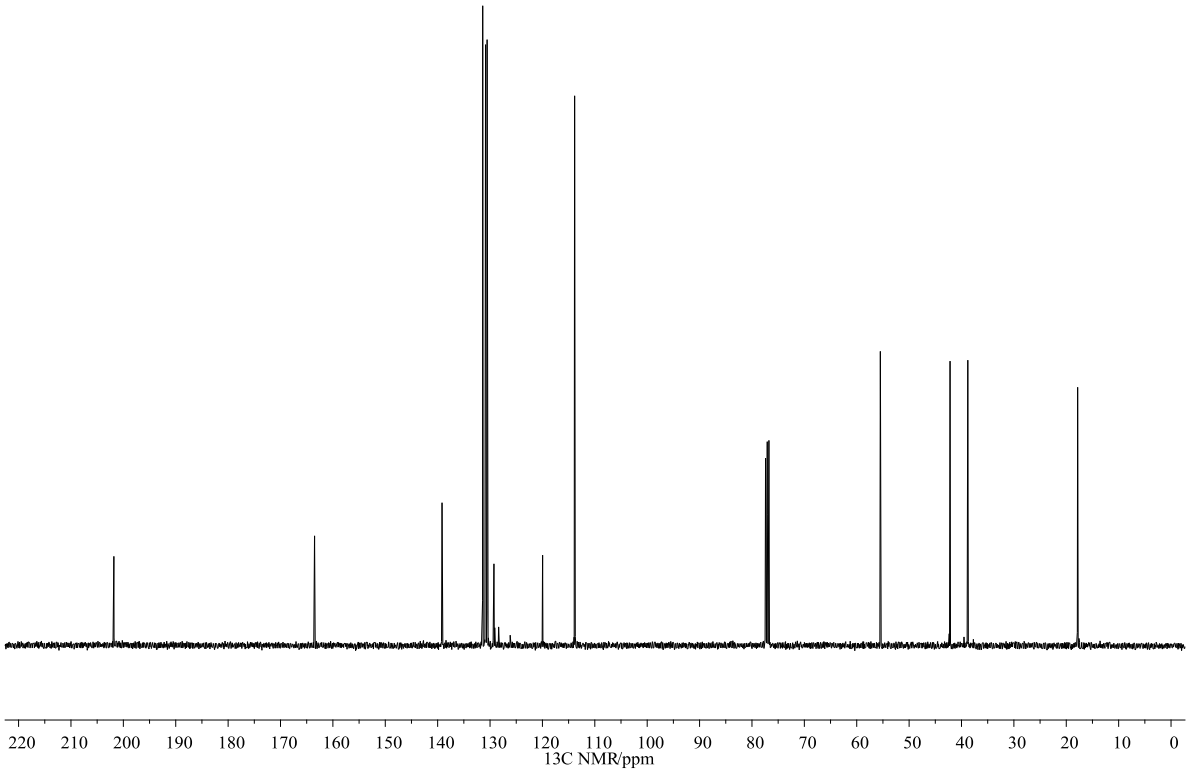



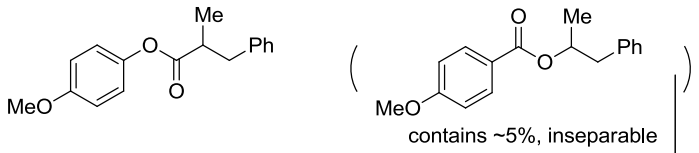
**(±)3-(4-bromophenyl)-1-(4-methoxyphenyl)-2-methylpropan-1-one, 30**

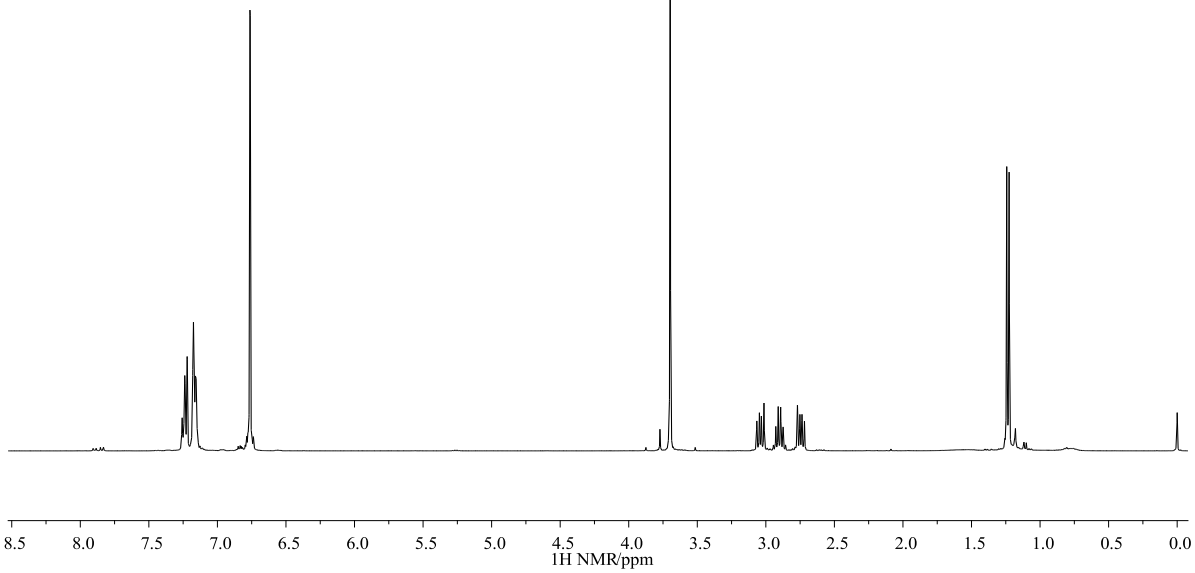


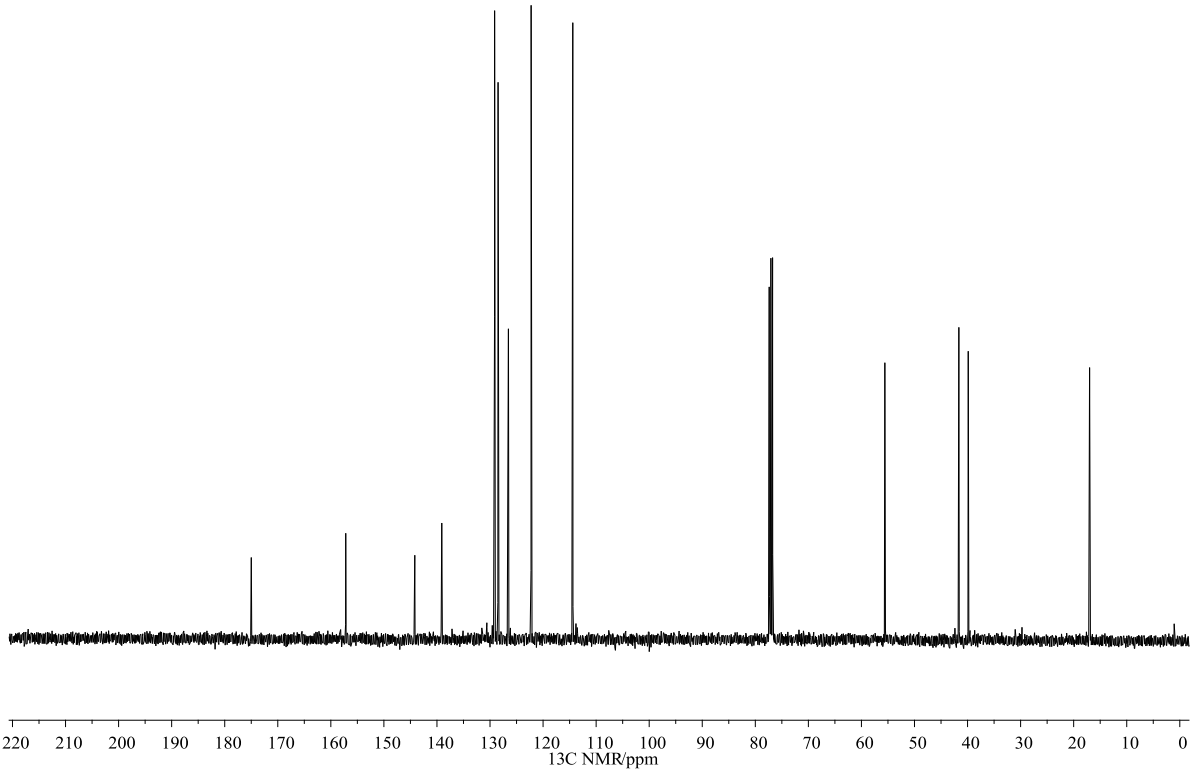



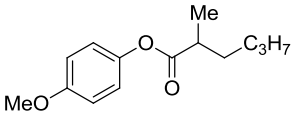
## (±)4-Methoxyphenyl 2-methyl-3-phenylpropanoate, 31

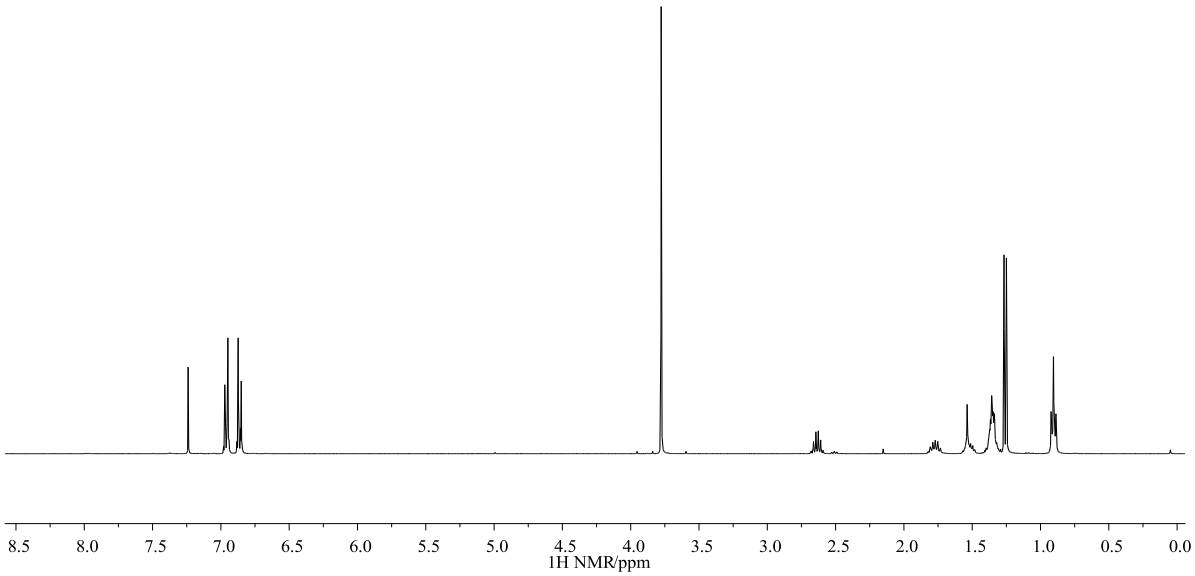


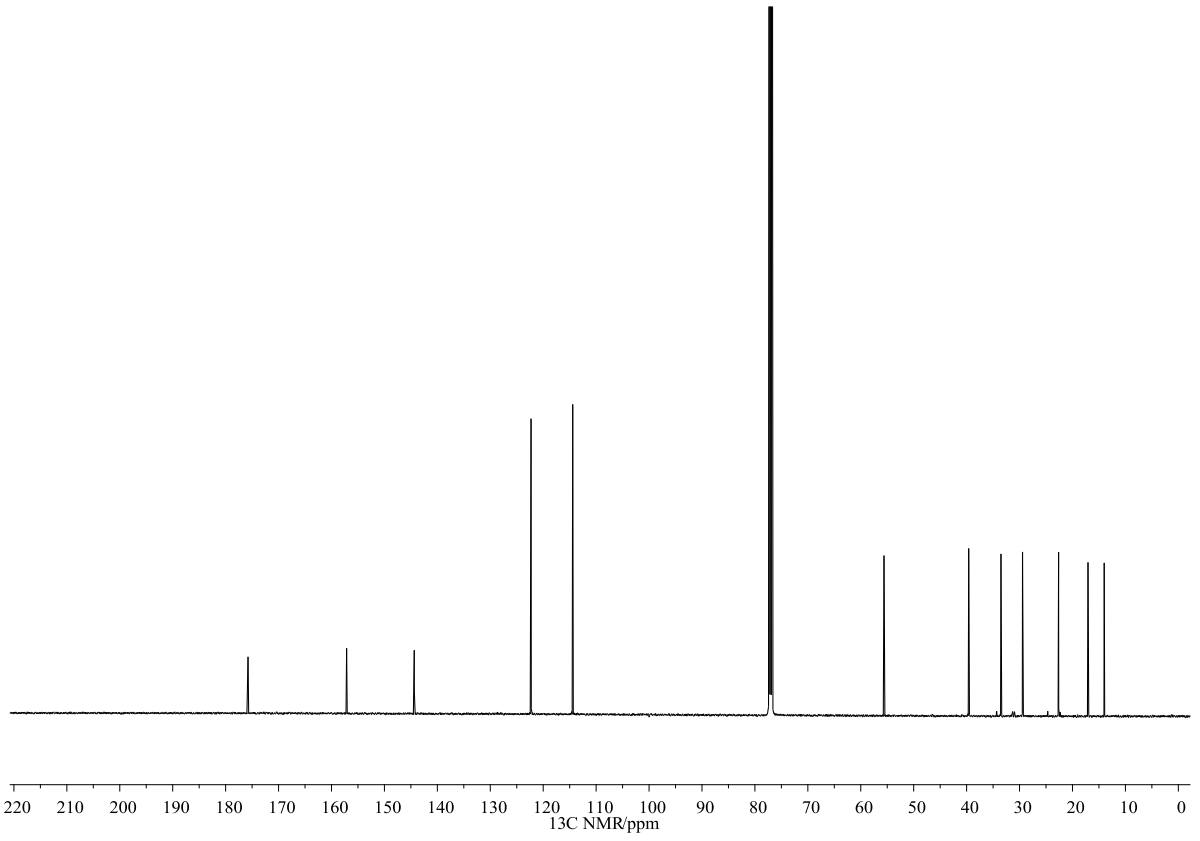



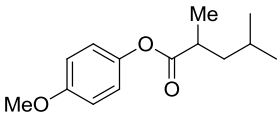
## (±)4-Methoxyphenyl 2-methylhexanoate, 32

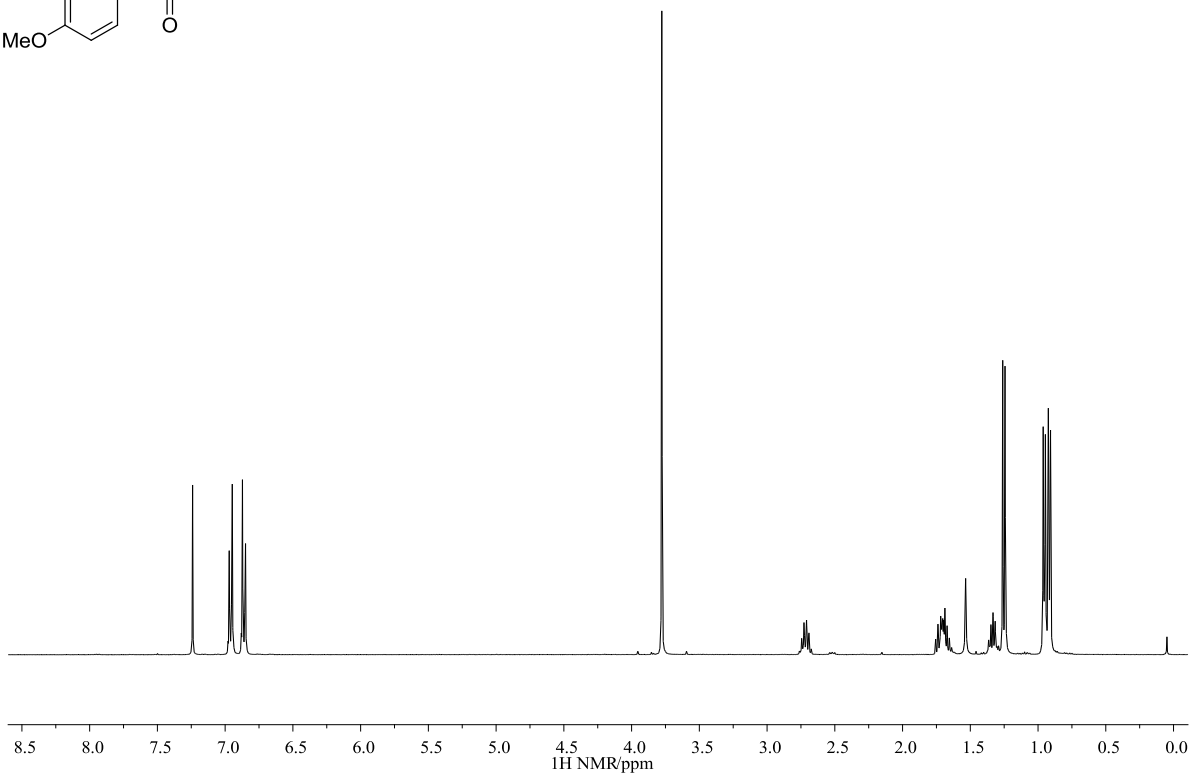


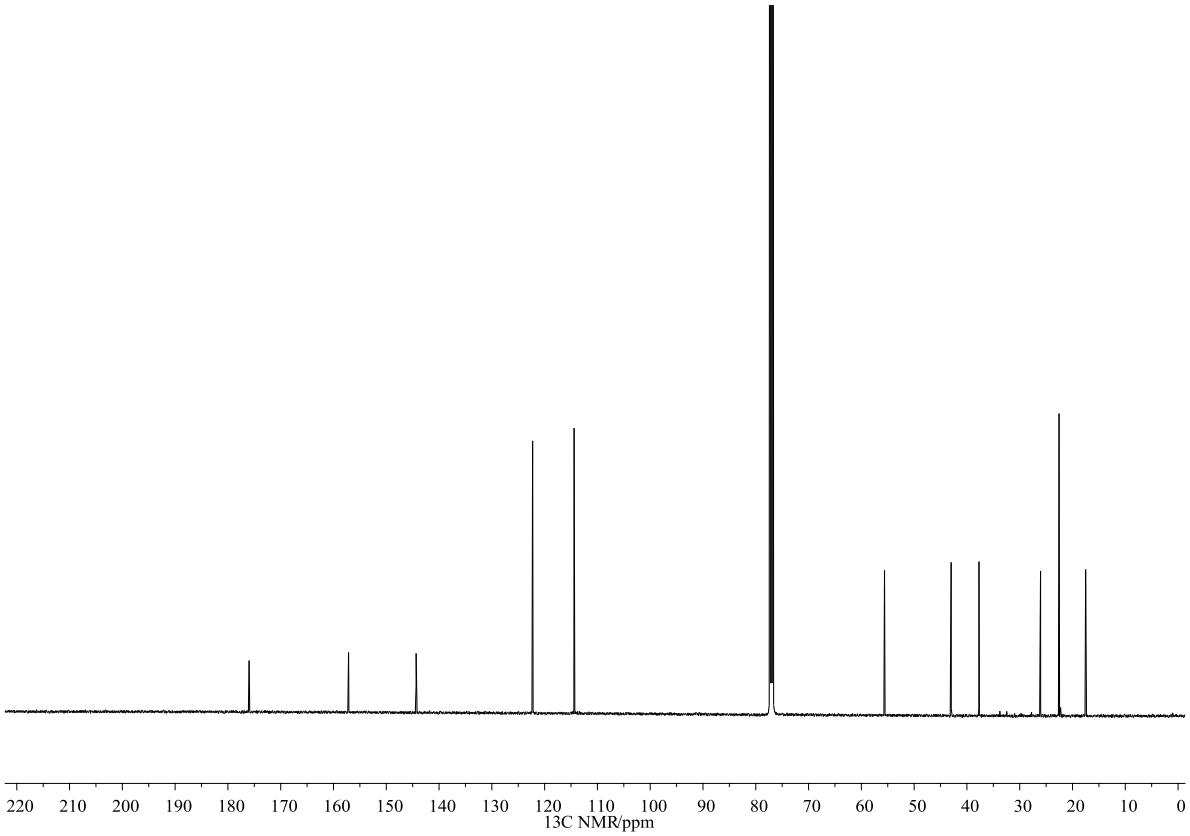



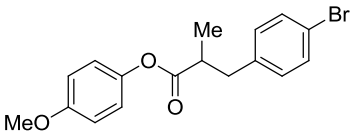
**(±)4-Methoxyphenyl 2,4-dimethylpentanoate, 33**

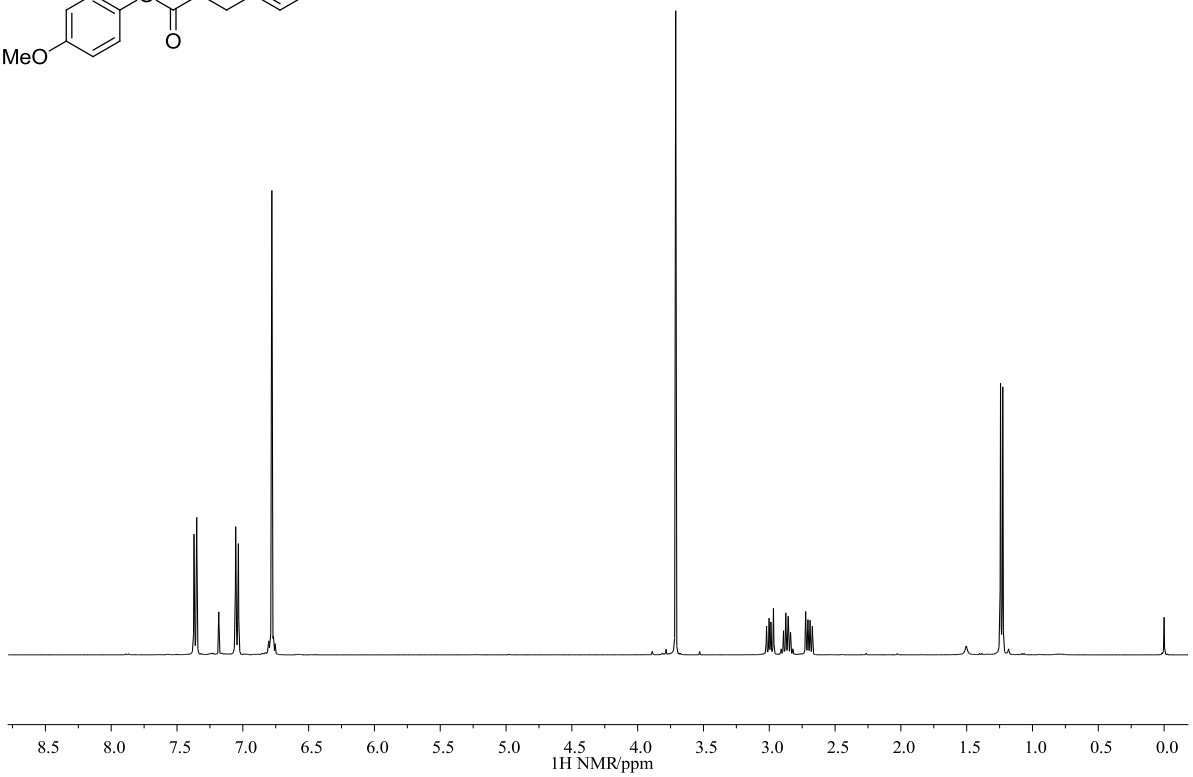


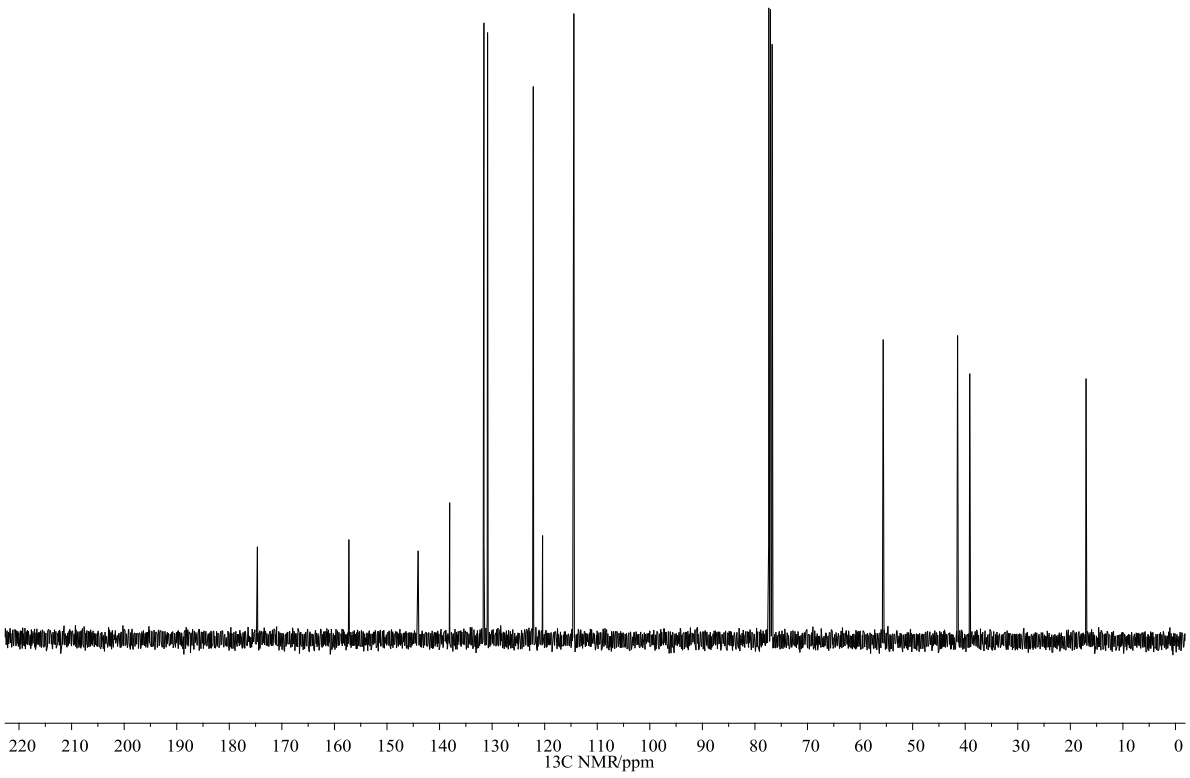



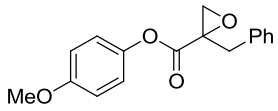
**(±)4-Methoxyphenyl 3-(4-bromophenyl)-2-methylpropanoate, 34**

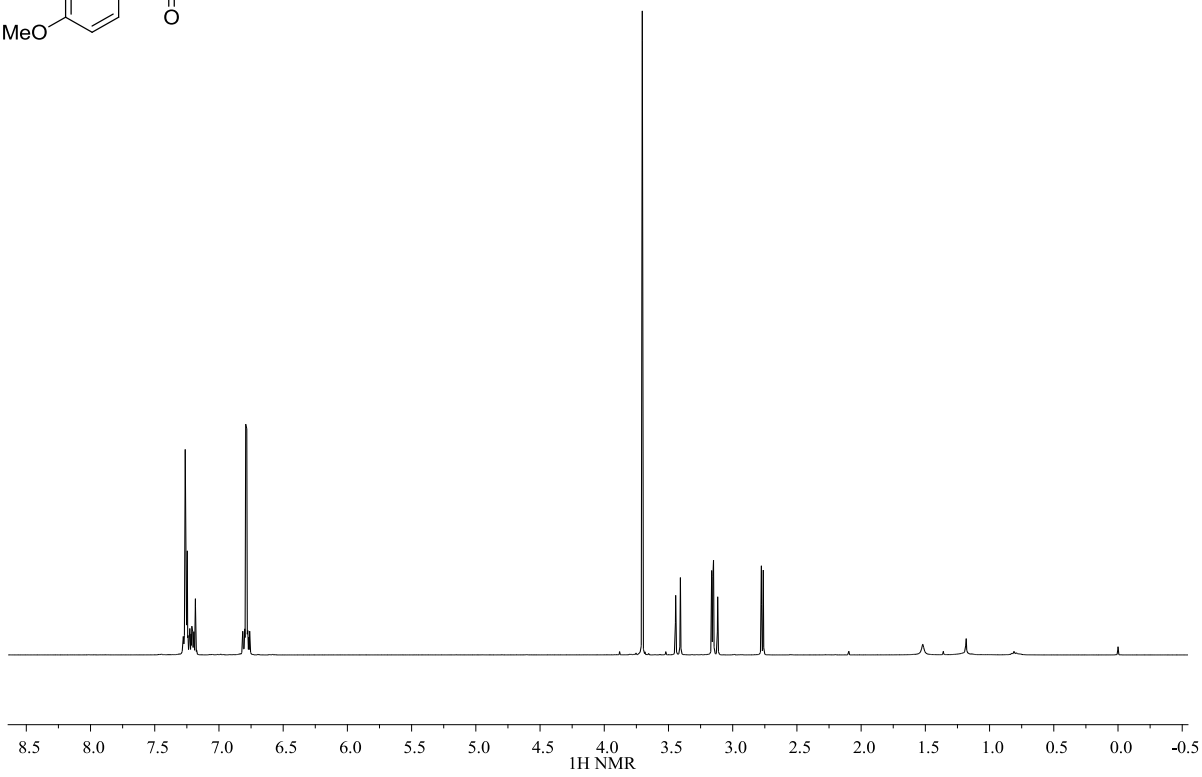


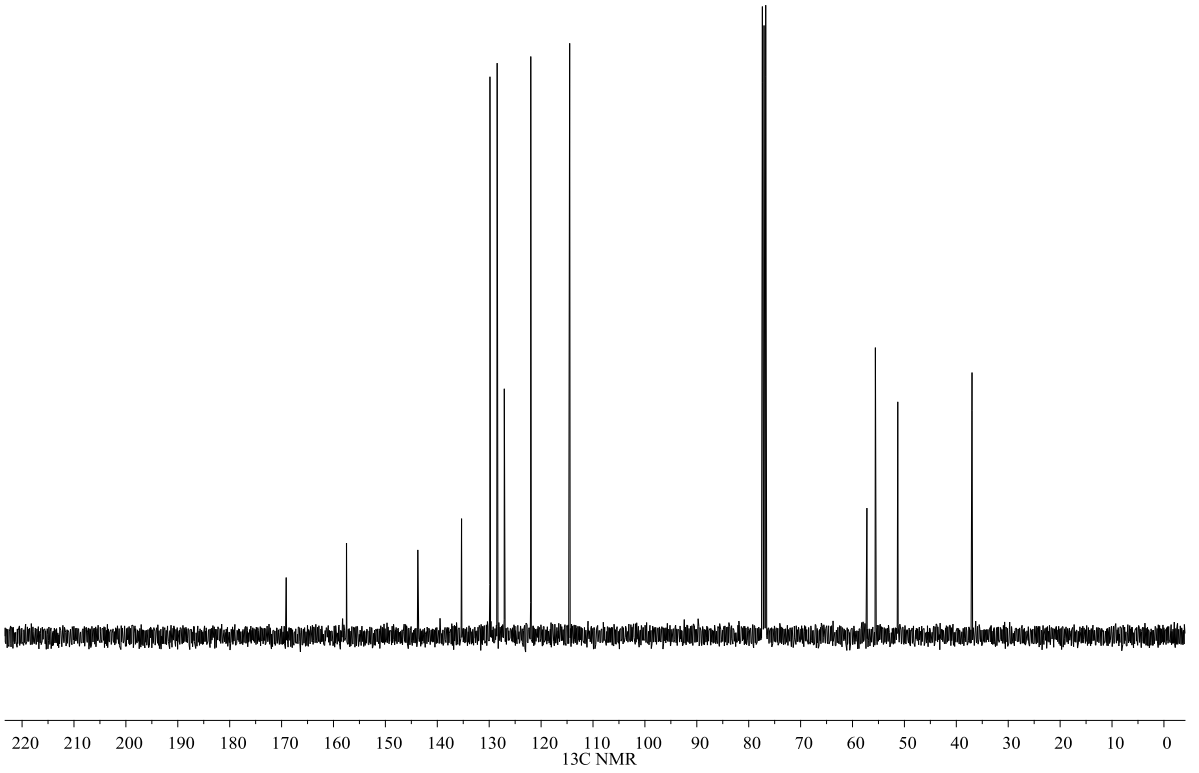



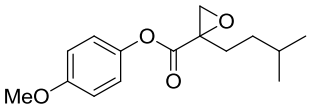
**4-Methoxyphenyl 2-benzyloxirane-2-carboxylate, 35**

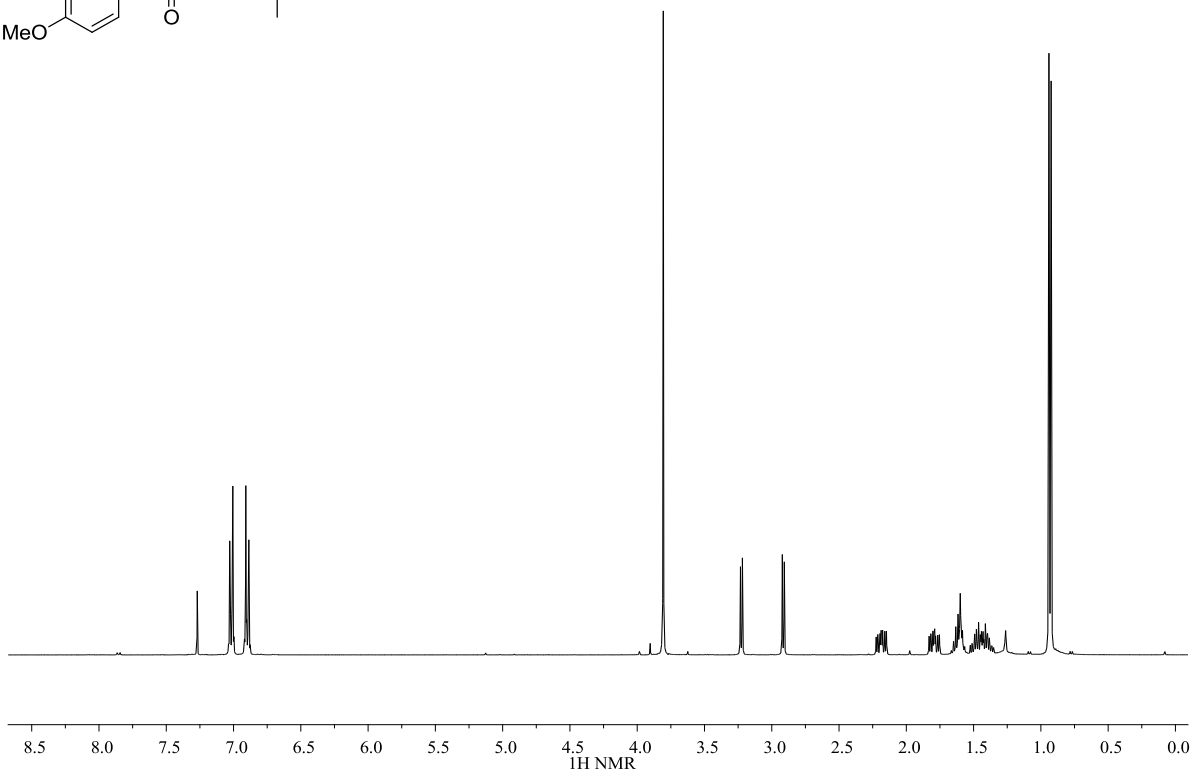


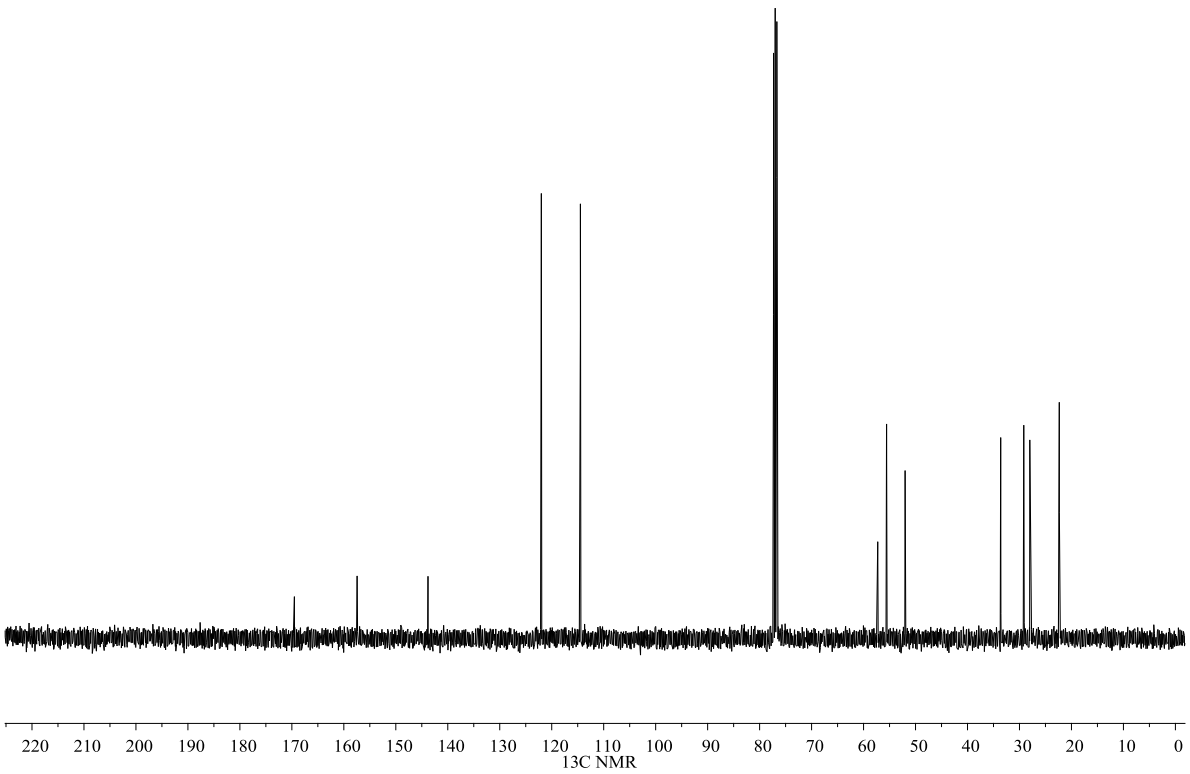



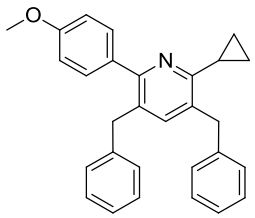
**4-Methoxyphenyl 2-isopentyloxirane-2-carboxylate, 36**



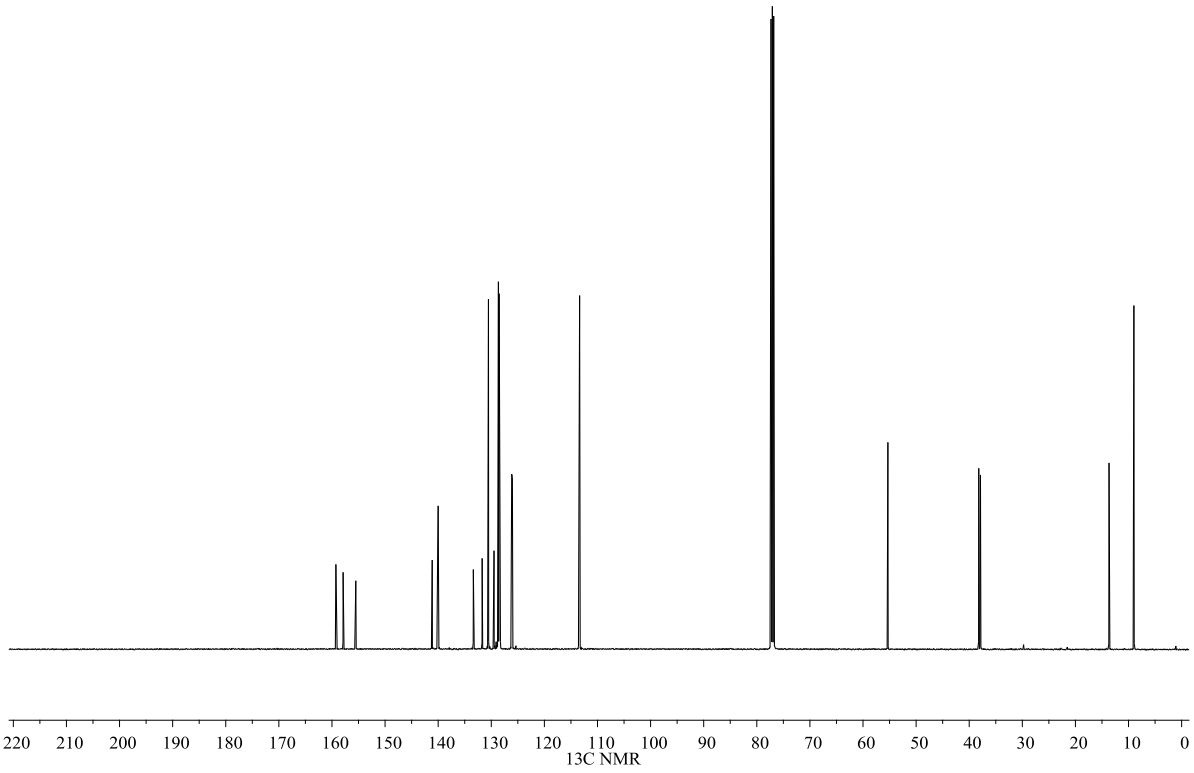



**3,5-Dibenzyl-2-cyclopropyl-6-(4-methoxyphenyl)pyridine, 37**

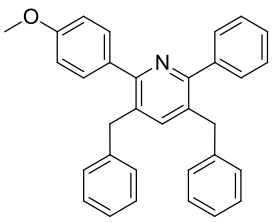


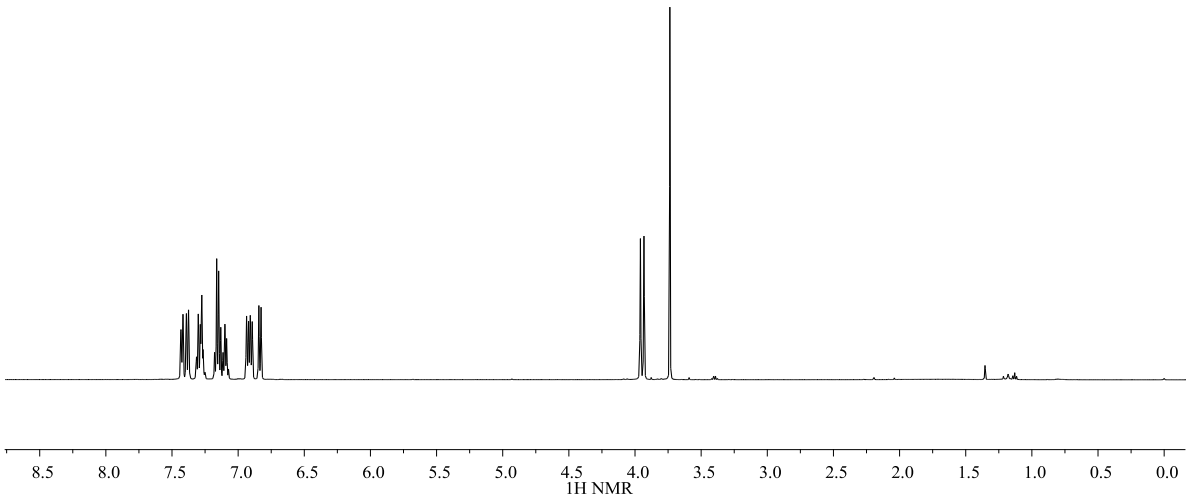


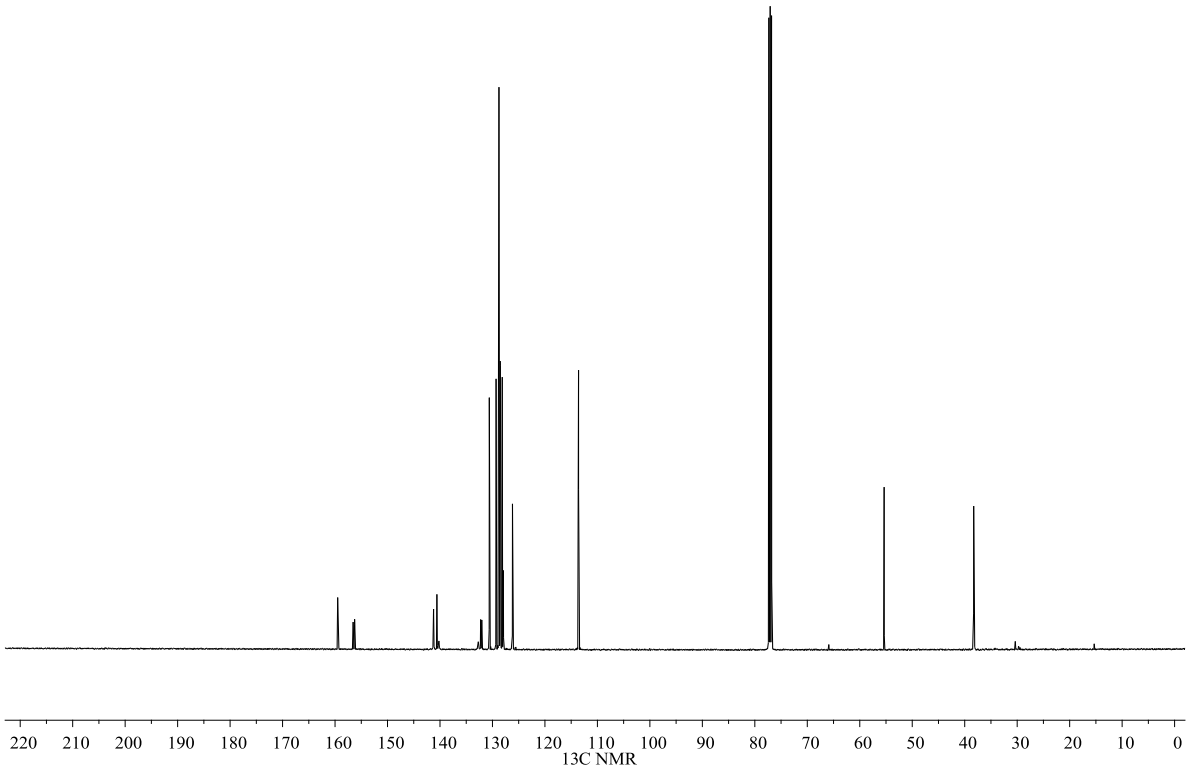




**3,5-Dibenzyl-2-(4-methoxyphenyl)-6-phenylpyridine, 38**



**3,5-Dibenzyl-2,6-bis(4-methoxyphenyl)pyridine, 39**