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Impregnated Palladium on Magnetite as Water Compatible Catalyst for the Cycloisomerization of Alkynoic Acid Derivatives

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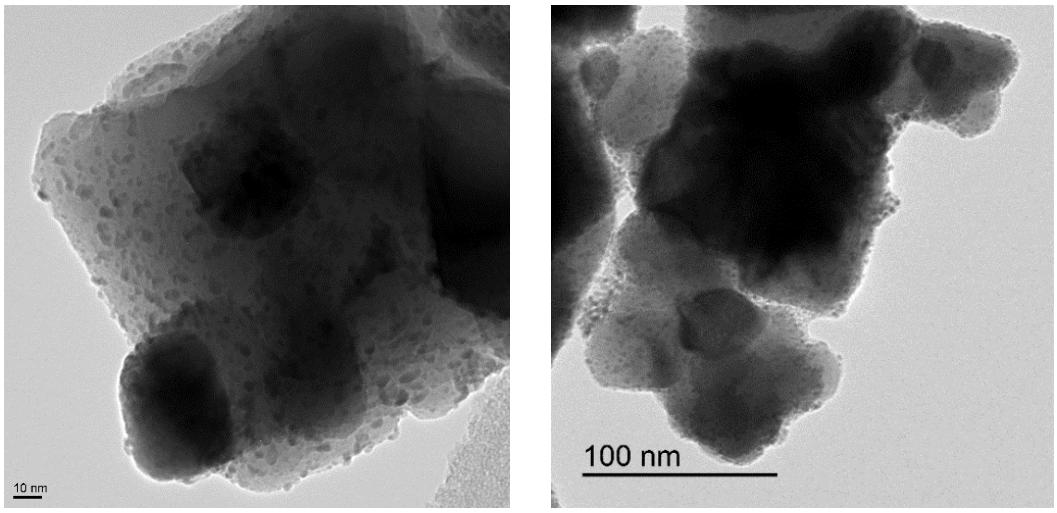
I. TEM images of the catalyst



A **B**

Image 1. TEM images of PdO-Fe_3O_4 catalyst before (**A**) and after (**B**) carrying out the reaction.

II. Kinetic Plots

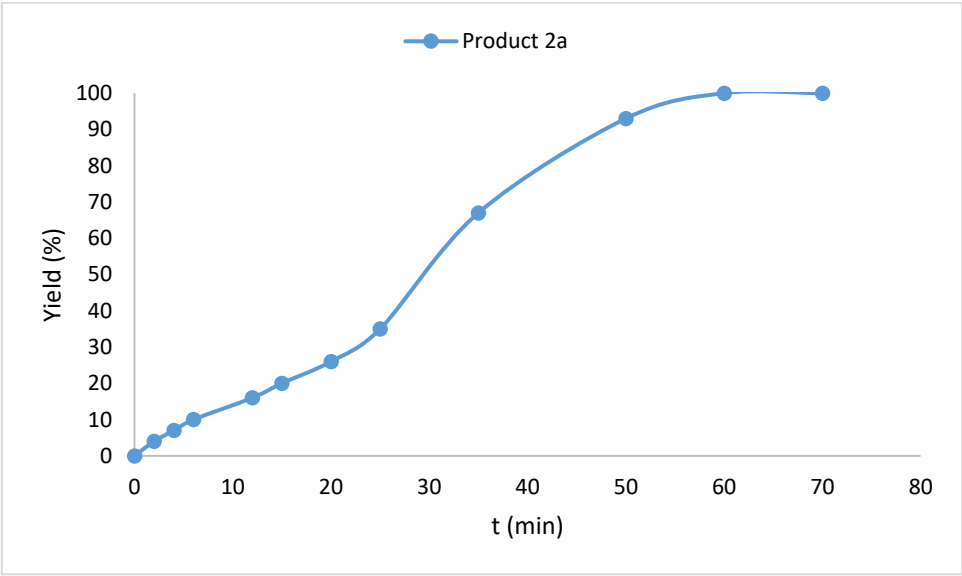


Figure S1. Plot time-yield product **2a** at 25 ^{o}C.

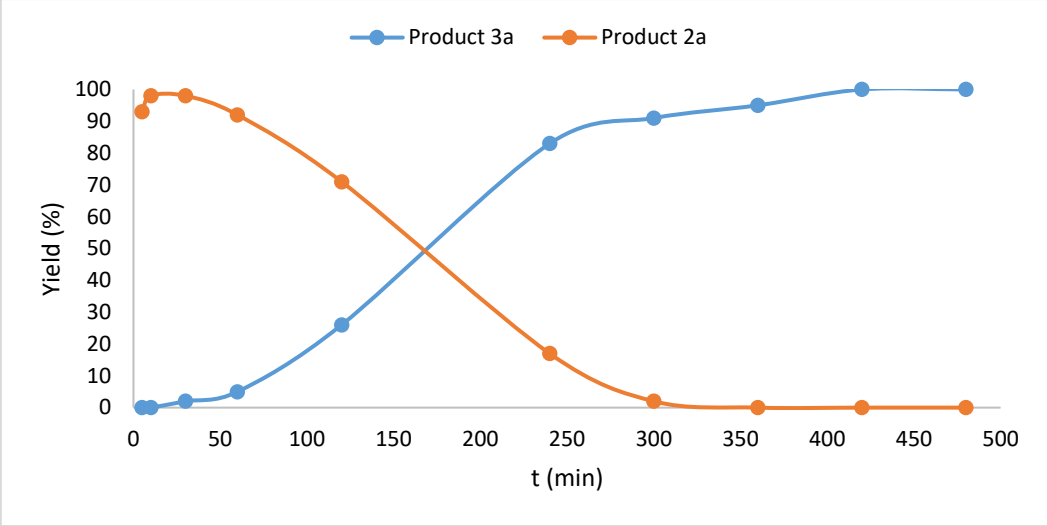
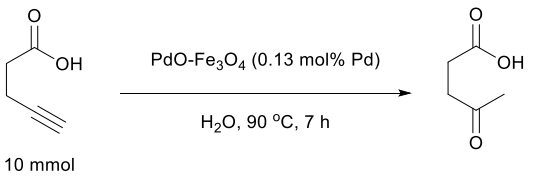


Figure S2. Plot time-yield product **2a** and **3a** at 90 °C.

III. Gram-scale reaction



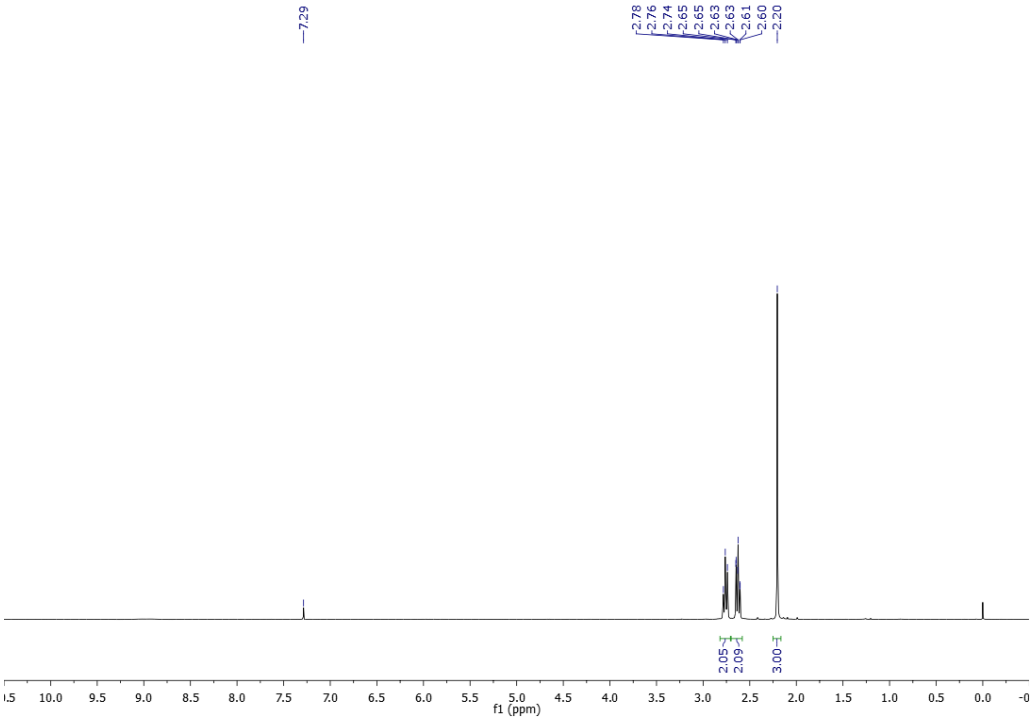


Figure S3. ^{1}H NMR gram-scale reaction crude **(**product **3a**).

IV. Characterization data

2,2-Dimethylpent-4-ynoic acid (**1b**).[1] Orange oil; t_R = 7.50 min; R_f = 0.33 (Hexane/AcOEt: 3/2); IR (ATR) *v* 2971, 2875, 1699, 1387, 1366, 1224, 1082, 969, 751 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 2.40 (d, *J* = 2.7 Hz, 2H, $H_2CC{\equiv}CH$), 1.97 (t, *J* = 2.7 Hz, 1H, C≡CH), 1.25 (s, 6H, CH_3) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 182.8, 89.5, 70.8, 41.0, 29.4, 24.8, 24.4 ppm; MS (EI, 70 eV): *m/z* 125 (M^+-1, 13%), 111 (100), 79 (27), 59 (30).

5-Phenylpent-4-ynoic acid (**1e**).[2] Brown solid; t_R = 12.10 min; R_f = 0.30 (Hexane/AcOEt: 1/1); m.p. 84-86 ºC; IR (ATR) *v* 2923, 1693, 1434, 1301, 1211, 1097, 917, 754, 690 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 7.35–7.30 (m, 2H, ArH), 7.25–7.20 (m, 3H, ArH), 2.70–2.60 (m, 4H, CH_2CH_2) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 177.8, 131.6, 128.2, 127.8, 123.4, 87.6, 81.3, 33.4, 15.1 ppm; MS (EI, 70 eV): *m/z* 174 (M^+, 47%), 173 (12), 147, (10), 146 (93), 131 (30), 129 (41), 128 (100), 127 (46), 117 (19), 116 (16), 114 (89), 89 (19), 78 (10), 77 (19), 51 (13).

***N*-tosylpent-4-ynamide** (**1f**).[3] White solid; t_R = 15.63 min; R_f = 0.36 (Hexane/AcOEt: 2/1); m.p. 119-123 ºC; IR (ATR) *v* 3272, 2360, 1727, 1434, 1413, 1330, 1160, 1120, 1079, 856, 817, 686, 665 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 8.75 (s, 1H, NH), 7.95 (d, *J* = 8.4 Hz, 2H, ArH), 7.35 (d, *J* = 8.4 Hz, 2H, ArH), 2.60–2.35 (m, 7H, $2CH_2$ and CH_3), 1.99 (t, *J* = 2.5 Hz, 1H, C≡CH) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 169.1, 145.5, 135.5, 129.8, 128.6, 81.9, 70.2, 35.2, 21.9, 13.8 ppm; MS (EI, 70 eV): *m/z* 251 (M^+, 7%), 187 (37), 186 (13), 155 (27), 92 (10), 91 (100), 65 (18).

5-Methylenedihydrofuran-2(3*H*)-one (**2a**).[4] Colourless oil; t_R = 5.25 min; R_f = 0.43 (Hexane/AcOEt: 4/1); IR (ATR) *v* 1702, 1398, 1366, 1162, 608 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 4.75 (dd, *J* = 4.6, 2.1 Hz, 1H, $C{=}CH_2$), 4.32 (dt, *J* = 2.1, 1.8 Hz, 1H, $C{=}CH_2$), 2.95–2.85 (m, 2H, CH_2CO_2H), 2.70–2.65 (m, 2H, $CH_2CH_2CO_2H$) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 175.0, 155.7, 88.9, 28.2, 25.2 ppm; MS (EI, 70 eV): *m/z* 98 (M^+, 100%), 70 (30), 56 (47), 55 (22).

4-Oxopentanoic acid (**3a**).[5] Colourless oil; t_R = 7.77 min; R_f = 0.36 (Hexane/AcOEt; 3/2); IR (ATR) *v* 2927, 1702, 1631, 1399, 1353, 1206, 1164 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 9.25 (s, 1H, COOH), 2.77 (t, *J* = 6.4 Hz, 2H, CH_2CO_2H), 2.64 (t, *J* = 6.4 Hz, 2H, $CH_2CH_2CO_2H$), 2.21 (s, 3H, $COCH_3$) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 206.8, 178.4, 37.8, 29.9, 27.9 ppm; MS (EI, 70 eV): *m/z* 116 (M^+, 46%), 101 (18), 99 (15), 73 (32), 56 (100), 55 (44).

3,3-Dimethyl-5-methylenedihydrofuran-2(3*H*)-one (**2b**).[6] Orange oil; t_R = 5.03 min R_f = 0.56 (Hexane/AcOEt: 3/2); IR (ATR) *v* 2969, 2932, 1797, 1705, 1703, 1672, 1080, 969 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 4.76 (s, 1H, C=CH_2), 4.33 (s, 1H, C=CH_2), 2.70 (s, 2H, CH_2C(CH_3)$_2$), 1.31 (s, 6H, CH_3CH_3CCO_2H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 180.3, 153.4, 89.4, 41.0, 40.4, 24.7 ppm; MS (EI, 70 eV): *m/z* 126 (M^+, 37%), 83 (37), 70 (16), 56 (100), 55 (16).

2,2-Dimethyl-4-oxopentanoic acid (**3b**).[7] Orange oil; R_f = 0.3 (Hexane/AcOEt: 4/1); IR (ATR) *v* 2975, 2875, 1784, 1705, 1475, 1361, 1135, 1055, 906 cm^{-1}; ^1H NMR (300 MHz, acetone-d_6) δ_H 2.78 (s, 2H, CH_2CO), 1.34 (s, 3H, $COCH_3$), 1.21 (s, 6H, CH_3CH_3CCO_2H) ppm; ^{13}C NMR (75 MHz, acetone-d_6) δ_C 210.1, 178.9, 53.1, 49.4, 40.2, 25.9 ppm; MS (EI, 70 eV): *m/z* 126 (M^+- H_2O, 10%), 87 (12), 85 (24), 59 (24), 58 (15), 56 (29), 55 (11), 43 (100), 41 (16).

5-Oxohexanoic acid (**3c**).[4] Orange oil; t_R = 8.52 min; R_f = 0.27 (Hexane/AcOEt; 3/2); IR (ATR) *v* 3160, 2962, 1703, 1409, 1363, 1155 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 9.82 (s, 1H, CO_2H), 2.55 (t, *J* = 7.2 Hz, 2H, CH_2CO), 2.40 (t, *J* = 7.2 Hz, 2H, CH_2CO_2H), 2.16 (s, 3H, $COCH_3$), 1.90 (q, *J* = 7.2 Hz, 2H, C$H_2CH_2CO_2$H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 208.4, 179.3, 42.4, 33.0, 30.0, 18.6 ppm; MS (EI, 70 eV): *m/z* 130 (M^+, 2%), 115 (10), 113 (10), 112 (100), 87 (32), 84 (29), 71 (18), 70 (55), 60 (29), 58 (21), 55 (27).

(*Z*)-5-benzylidenedihydrofuran-2(3*H*)-one (**2e**).[8] Pale yellow oil; t_R = 12.65 min; R_f = 0.60 (Hexane/AcOEt: 1/1); IR (ATR) *v* 2925, 1798, 1681, 1493, 1448, 1225, 1776, 1097, 939, 693 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 7.55 (d, *J* = 7.4 Hz, 2H, ArH), 7.32 (t, *J* = 7.4 Hz, 2H, ArH), 7.20 (t, *J* = 7.4 Hz, 1H, ArH), 5.55 (br s, 1H, C=CH), 3.10–2.95 (m, 2H, CH_2CO), 2.75–2.65 (m, 2H, CH_2CH_2CO) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 175.1, 148.3, 134.1, 128.7, 128.5, 127.0, 105.2, 27.2, 26.6 ppm; MS (EI, 70 eV): *m/z* 174 (M^+, 100%), 146 (22), 145 (32), 131 (12), 118 (43), 117 (12), 91 (25), 90 (53), 89 (30), 63 (11).

4-Oxo-5-phenylpentanoic acid (**3e**).[9] Orange oil; t_R = 12.84 min; R_f = 0.33 (Hexane/AcOEt: 3/2); IR (ATR) *v* 3029, 2920, 1703, 1596, 1399, 1158, 741, 698 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 7.40–7.20 (m, 5H, ArH), 3.74 (s, 2H, CH_2Ph), 2.75 (t, *J* = 6.2 Hz, 2H, H_2CCO), 2.61 (t, *J* = 6.2 Hz, 2H, H_2CCOOH) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 206.5, 178.1, 134.0, 129.6, 128.9, 127.3, 50.2, 36.3, 27.9 ppm; MS (EI, 70 eV): *m/z* 192 (M^+, 9%), 101 (98), 92 (92), 91 (100), 89 (12), 73 (30), 65 (32), 63 (10), 55 (31).

5-Methylene-1-tosylpyrrolidin-2-one (**2f**).[10] White solid; t_R = 16.04 min R_f = 0.40 (Hexane/AcOEt: 3/2); IR (ATR) *v* 2918, 2850, 1708, 1646, 1353, 1160, 1132, 1085 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 7.92 (d, *J* = 8.4 Hz, 2H, ArH), 7.32 (d, *J* = 8.4 Hz, 2H, ArH), 5.27 (s, 1H, C=CH), 5.13 (s, 1H, C=CH), 2.55–2.45 (t, *J* = 7.0 Hz, 2H, H_2CCO), 2.43 (s, 3H), 2.38 (t, *J* = 7.0 Hz, 2H, H_2CC).ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 170.8, 145.0, 139.0, 136.6, 129.5, 128.6, 111.3, 33.8, 30.6, 21.8 ppm; MS (EI, 70 eV): *m/z* 236 (M^+-CH_3, 2%), 201 (25), 200 (31), 172 (19), 155 (11), 92 (10), 91 (100), 89 (10), 65 (37), 55 (18).

4-Oxo-*N*-tosylpentanamide (**3f**). Orange solid; t_R = 16.29 min; R_f = 0.20 (Hexane/AcOEt: 1/1); m.p. 148-151 ºC; IR (ATR) *v* 3129, 2917, 1699, 1599, 1452, 1380, 1336, 1166, 1130, 1087, 862, 813, 659 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 8.87 (s, 1H, CONH), 7.92 (d, *J* = 8.2 Hz, 2H, ArH), 7.33 (d, *J* = 8.2 Hz, 2H, ArH), 2.75 (t, *J* = 6.1 Hz, 2H, CH_2CO), 2.51 (t, *J* = 6.1 Hz, 2H, CH_2CONH), 2.43 (s, 3H, $ArCH_3$), 2.15 (s, 3H, $COCH_3$) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 207.7, 170.4, 145.2, 135.8, 129.8, 128.5, 37.6, 30.2, 30.0, 21.9 ppm; MS (EI, 70 eV): *m/z* 155 (M^+-$C_5H_8O_2N$, 20%), 108 (100), 107 (14), 91 (54), 65 (15), 43 (29). HRMS calcd. (%) for $C_{12}H_{15}NO_4S$-$C_5H_6O_2$: 171.0354; found: 171.0352.

Methyl 5-methylene-2-oxo-1-tosylpyrrolidine-3-carboxylate (**2g**).[3] Yellow oil; t_R = 15.45 min; R_f = 0.33 (Hexane/AcOEt: 2/1); IR (ATR) *v* 3252, 2957, 2922, 2854, 1734, 1715, 1437, 1348, 1274, 1265, 1168, 1085, 813, 662 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 7.94 (d, *J* = 8.4 Hz, 2H, ArH), 7.36 (d, *J* = 8.4 Hz, 2H, ArH), 5.55 (dd, *J* = 3.8, 2.0 Hz, 1H, $C=CH_2$), 4.65 (dd, *J* = 3.8, 2.0 Hz, 1H, $C=CH_2$), 3.73 (s, 3H, OCH_3), 3.53 (dd, *J* = 9.9, 7.6 Hz, 1H, HCCON), 3.15–2.85 (m, 2H, H_2C-$C=CH_2$), 2.46 (s, 3H, $ArCH_3$) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 168.5, 167.8, 145.8, 138.5, 134.8, 129.7, 128.2, 95.2, 53.1, 47.2, 30.0, 21.7 ppm; MS (EI, 70 eV): *m/z* 251 (M^++1-$C_2H_3O_2$, 5%), 187 (33), 186 (13), 155 (25), 92 (10), 91 (100), 65 (17).

Methyl 4-oxo-2-(tosylcarbamoyl)pentanoate (**3g**). Orange oil; t_R = 12.58 min; R_f = 0.20 (Hexane/AcOEt: 3/2); IR (ATR) *v* 3248, 1743, 1713, 1596, 1436, 1347, 1166, 1084, 813, 660 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 9.98 (s, 1H, NH), 7.92 (d, *J* = 8.3 Hz, 2H, ArH), 7.32 (d, *J* = 8.3 Hz, 2H, ArH), 3.74 (t, *J* = 6.1 Hz, 1H, COCHCO), 3.68 (s, 3H, H_3CO), 3.10–3.05 (m, 2H, H_2CCO), 2.43 (s, 3H, $ArCH_3$), 2.14 (s, 3H, $COCH_3$) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 205.8, 169.3, 165.8, 145.2, 135.5, 129.7, 128.6, 53.4, 47.5, 41.1, 29.7, 21.8 ppm; MS (EI, 70 eV): *m/z* 155 (M^+-$C_7H_{10}O_4N$, 30%), 125 (10), 108 (100), 91 (61), 87 (15), 65 (14). HRMS calcd. (%) for $C_{14}H_{17}NO_6S$-$C_7H_8O_2$: 171.0354; found: 171.0353.

Methyl 3-allyl-5-methylene-2-oxo-1-tosylpyrrolidine-3-carboxylate (**2h**).[3] Yellow oil ; t_R = 17.38 min; R_f = 0.53 (Hexane/AcOEt: 2/1); IR (ATR) *v* 2958, 1759, 1736, 1655, 1435, 1368, 1258, 1172, 1087, 813, 669 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 7.92 (d, *J* = 8.4 Hz, 2H, ArH), 7.34 (d, *J* = 8.4 Hz, 2H, ArH), 5.55–5.40 (m, 2H, H_2C=CH), 5.05–5.00 (m, 2H, H_2C=C), 4.60 (d, *J* = 1.7 Hz, 1H, H_2C=C*H*), 3.60 (s, 3H, OCH_3), 3.04 (dt, *J* = 15.9, 1.5 Hz, 1H, H_2C-C=CH_2), 2.71 (dt, *J* = 15.9, 1.5 Hz, 1H, H_2C-C=CH_2), 2.65–2.50 (m, 2H, H_2C-CH=CH_2), 2.45 (s, 3H, $ArCH_3$) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 171.2, 169.8, 145.8, 138.3, 131.0, 129.6, 128.2, 126.5, 120.8, 94.9, 54.9, 53.1, 37.9, 21.8, 16.9 ppm; MS (EI, 70 eV): *m/z* 349 (M^+, 4%), 308 (46), 155 (64), 139 (10), 108 (25), 95 (18), 91 (100), 67 (10), 65 (21), 43 (18).

Ethyl 3-methyl-5-methylene-2-oxo-1-tosylpyrrolidine-3-carboxylate (**2i**).[3] Yellow oil ; t_R = 16.64 min; R_f = 0.46 (Hexane/AcOEt: 2/1); IR (ATR) *v* 2983, 2930, 2850, 1757, 1733, 1655, 1369, 1173, 1085, 1041, 813, 665 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 7.94 (d, *J* = 8.4 Hz, 2H, ArH), 7.34 (d, *J* = 8.4 Hz, 2H, ArH), 5.54 (dd, *J* = 3.6, 1.9 Hz, 1H, C=CH_2), 4.60 (dd, *J* = 3.6, 1.9 Hz, 1H, C=CH_2), 4.10–3.90 (m, 2H,OC$H_2$$CH_3$), 3.07 (d, *J* = 15.5 Hz, 1H, H_2C-C=CH_2), 2.56 (d, *J* = 15.5 Hz, 1H, H_2C-C=CH_2), 2.44 (s, 3H, $ArCH_3$), 1.38 (s, 3H, CCH_3), 1.05 (t, *J* = 7.1 Hz, 3H, OCH_2CH_3) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 172.5, 170.2, 145.8, 138.3, 135.0, 129.7, 128.3, 95.0, 62.2, 51.4, 39.4, 21.9, 19.8, 13.9 ppm; MS (EI, 70 eV): *m/z* 337 (M^+, 4%), 264 (100), 200 (41), 155 (49), 91 (97), 65 (17).

Ethyl (*Z*)-3-methyl-2-oxo-5-propylidene-1-tosylpyrrolidine-3-carboxylate (**2j**).[11] Pale yellow oil ; t_R = 17.36 min; R_f = 0.5 (Hexane/AcOEt: 2/1); IR (ATR) *v* 2977, 2935, 1754, 1734, 1691, 1363, 1165, 812, 664, 539 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 7.90 (d, *J* = 8.2 Hz, 2H, ArH), 7.32 (d, *J* = 8.2 Hz, 2H, ArH), 5.19 (t, *J* = 7.2 Hz, 1H, C=CHEt), 4.05–3.95 (m, 2H, COC$H_2$$CH_3$), 3.06 (dd, *J* = 13.2, 1.1 Hz, 1H, H_2CC=CHEt), 2.50–2.25 (m, 6H, $ArCH_3$ and H_2CC=CHEt), 1.31 (s, 3H, CCH_3), 1.07 (m, 6H, $COCH_2CH_3$ and $CHCH_2CH_3$).ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 174.4, 170.4, 145.4, 135.9, 129.9, 128.8, 121.9, 62.1, 53.5, 43.7, 23.3, 21.9, 19.2, 14.3, 14.0 ppm; MS (EI, 70 eV): *m/z* 365 (M^+, 2%), 292 (51), 210 (13), 155 (32), 136 (39), 92 (12), 91 (100), 65 (26).

V. References

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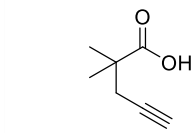
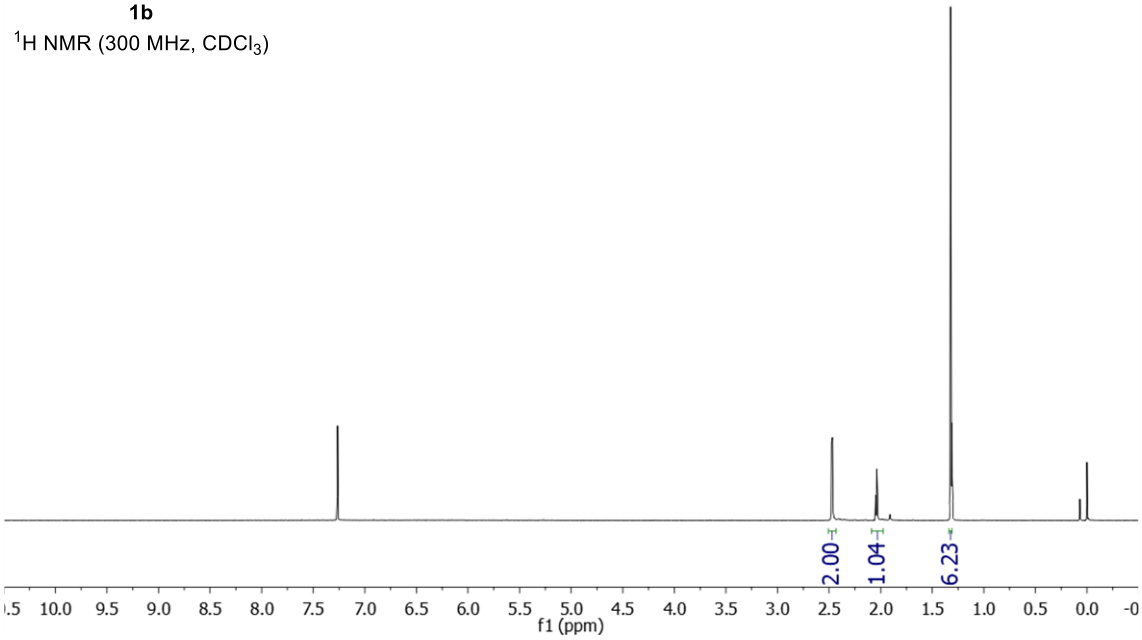
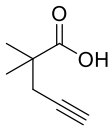
VI. Copies of ^{1}H and ^{13}C NMR



1b

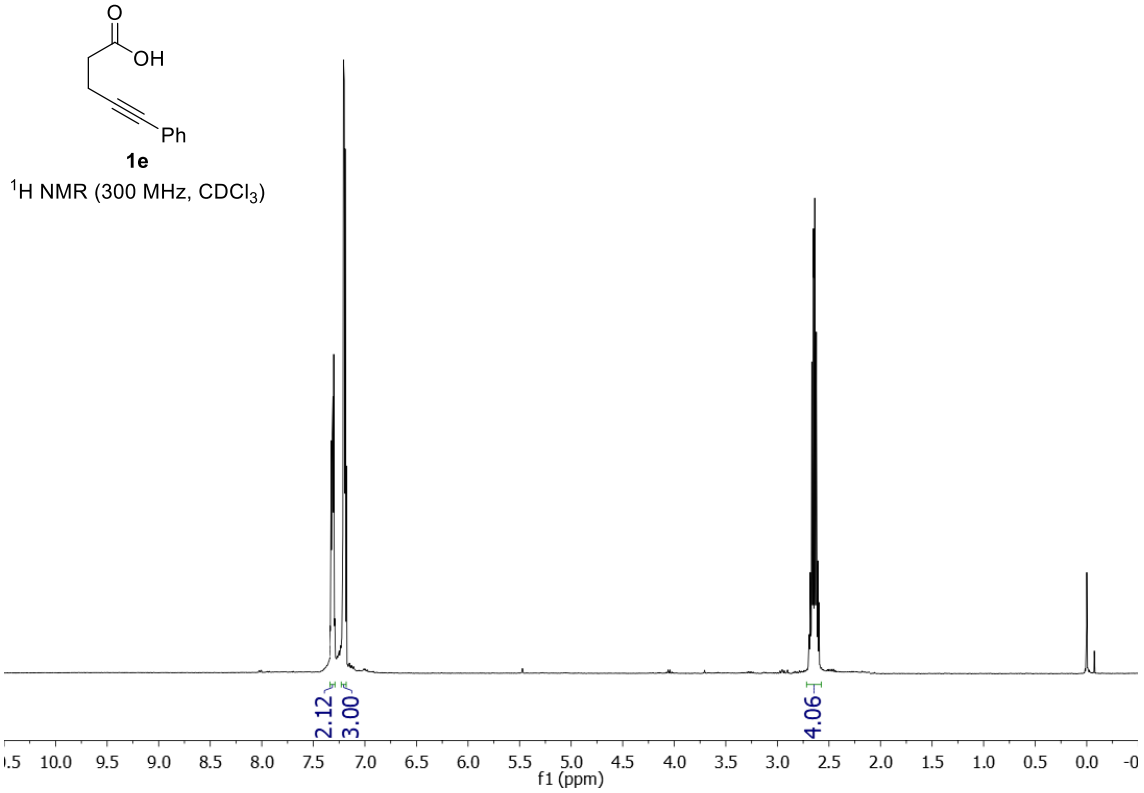
^{1}H NMR (300 MHz, $CDCl_3$)

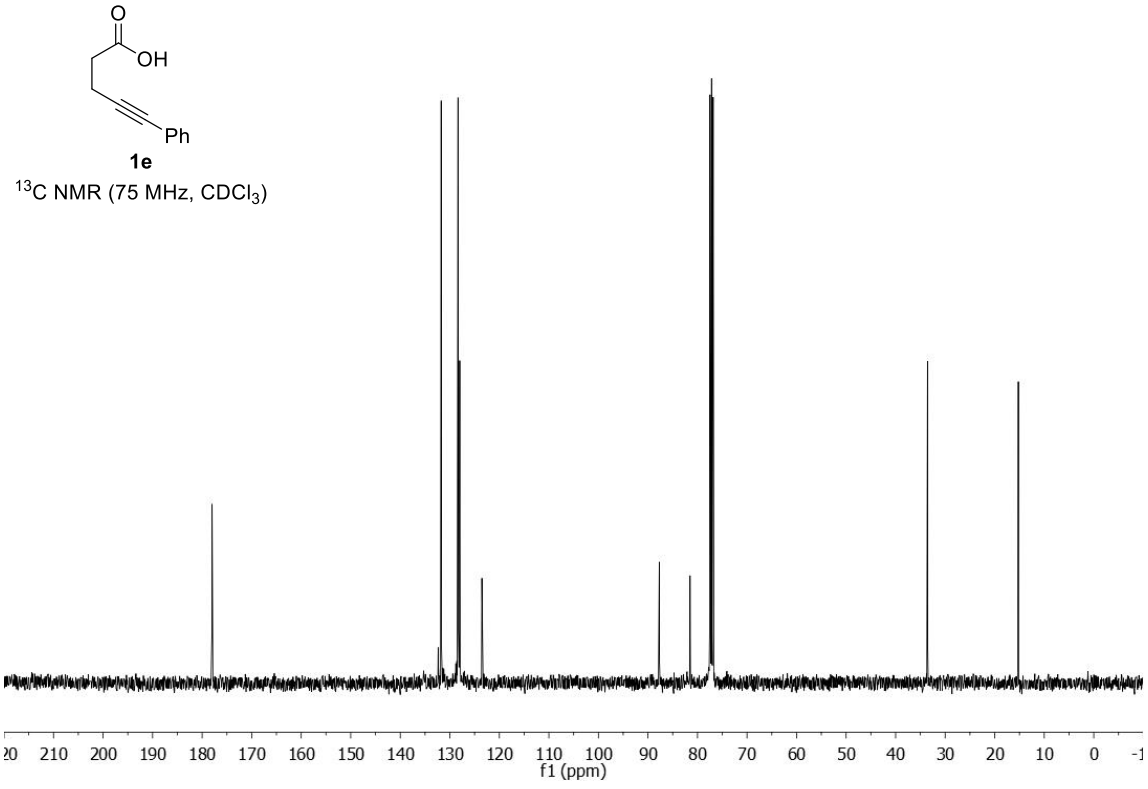


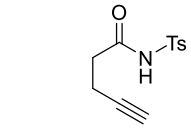


1b

^{13}C NMR (75 MHz, $CDCl_3$)

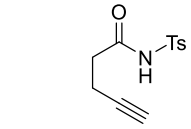

O
OH
Ph
1e
^{1}H NMR (300 MHz, CDCl$_3$)
2.12
3.00
4.06
f1 (ppm)


O
OH
Ph
1e
^{13}C NMR (75 MHz, CDCl$_3$)
f1 (ppm)



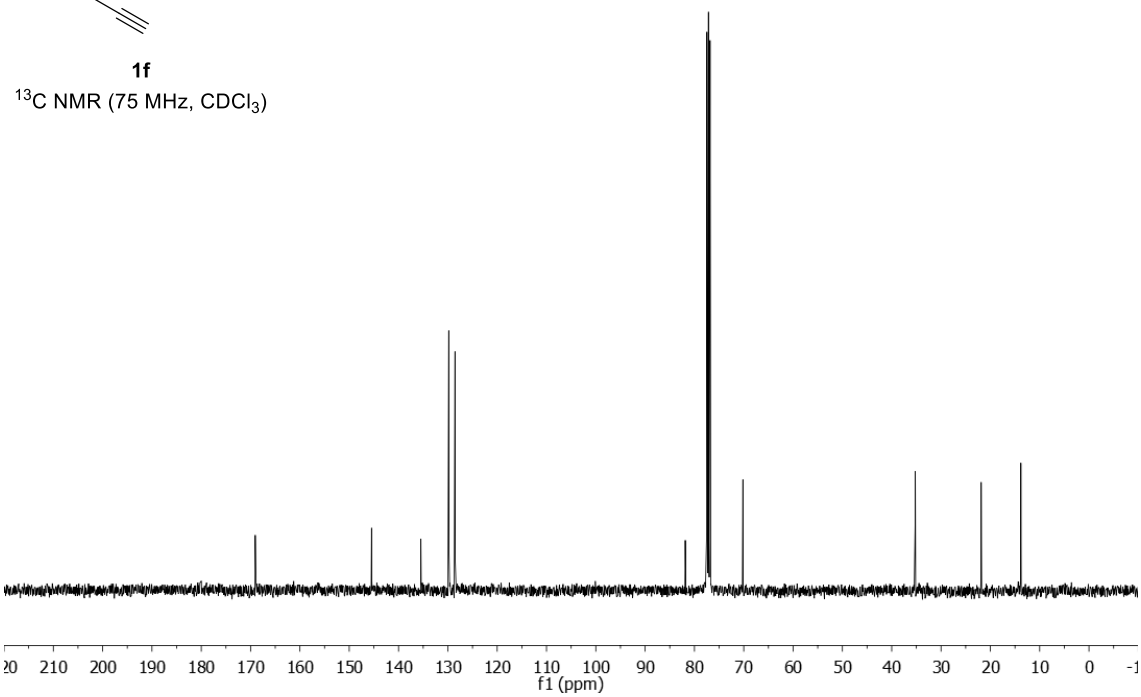
1f

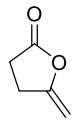
^{1}H NMR (300 MHz, $CDCl_3$)



1f

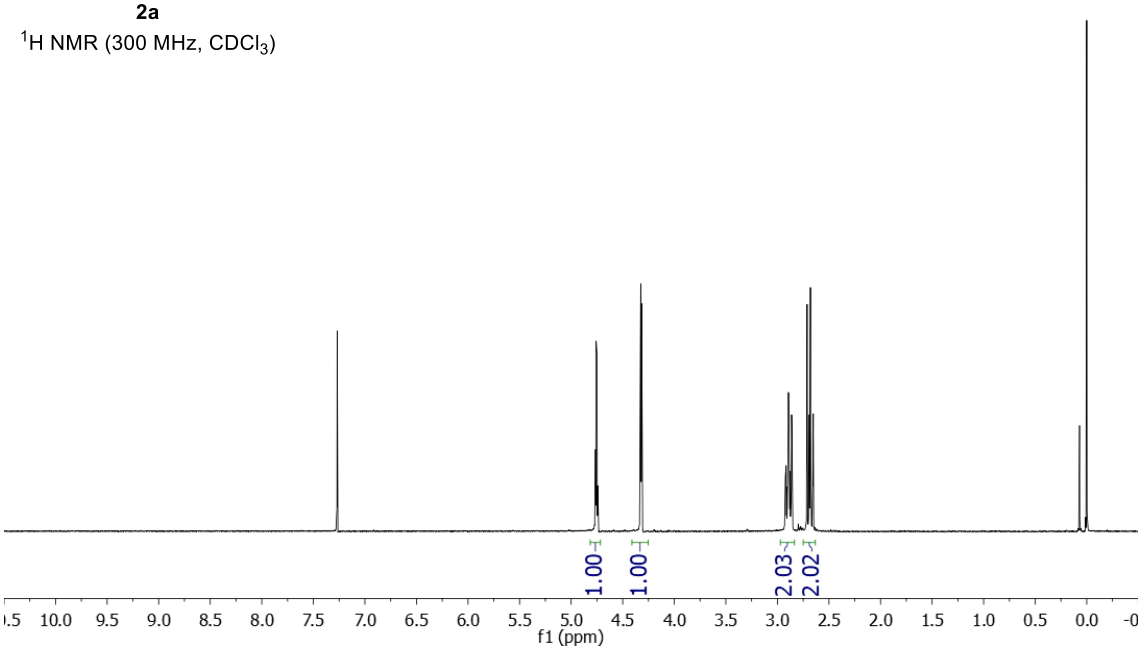
^{13}C NMR (75 MHz, $CDCl_3$)

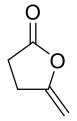




2a

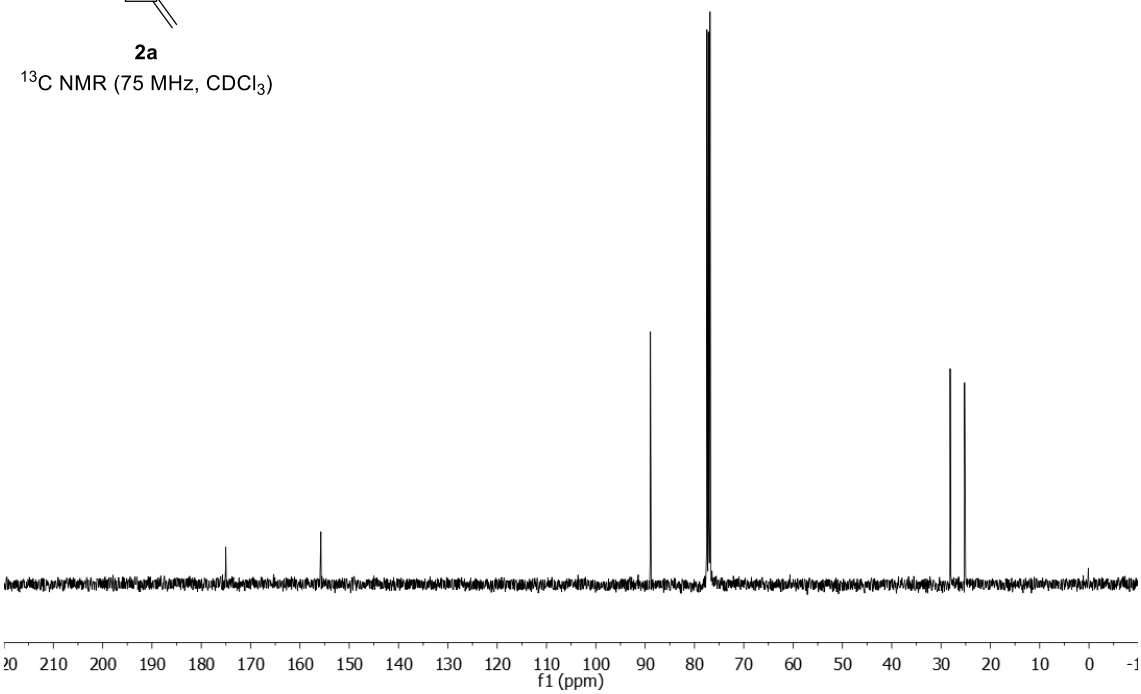
^{1}H NMR (300 MHz, $CDCl_3$)

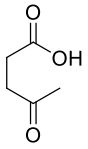




2a

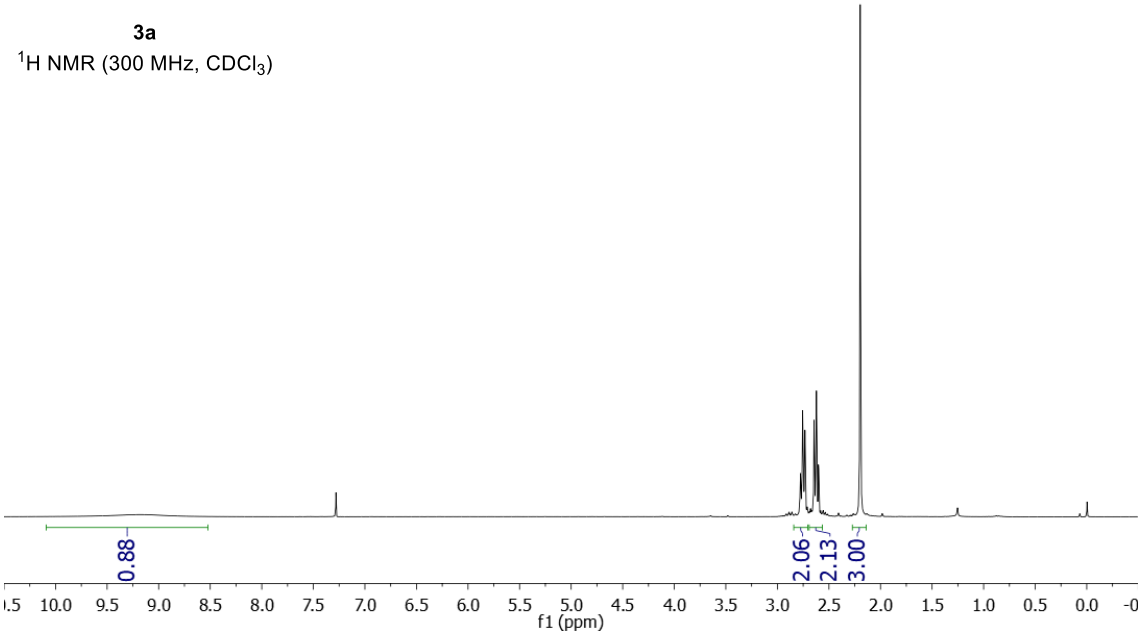
^{13}C NMR (75 MHz, $CDCl_3$)

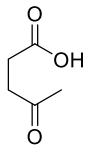




3a

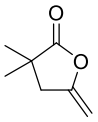
^{1}H NMR (300 MHz, $CDCl_3$)





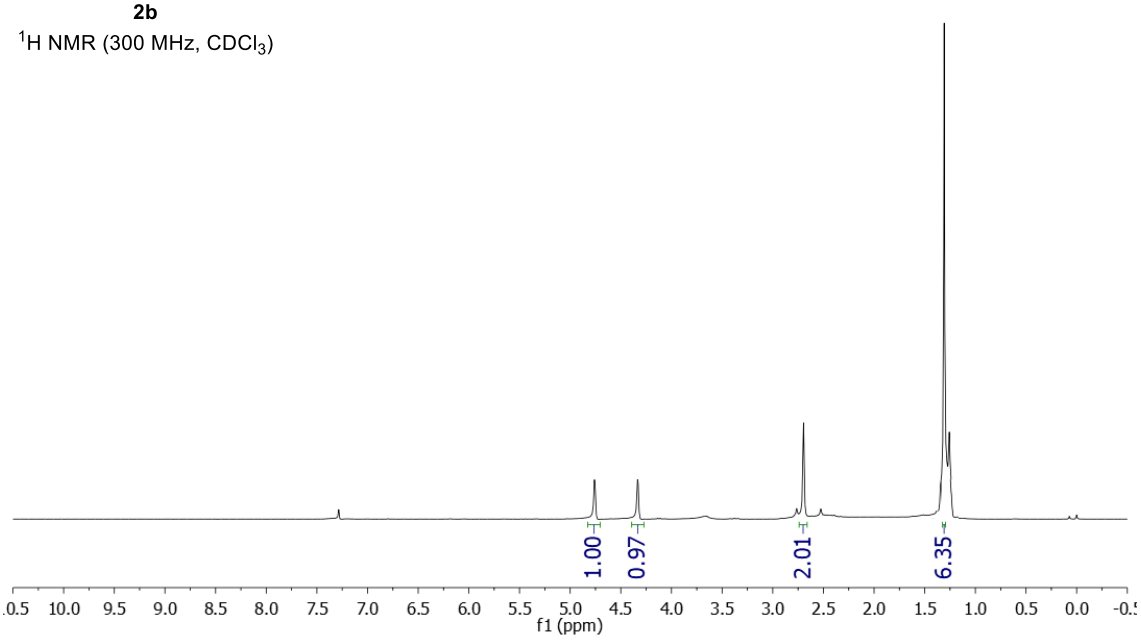
3a

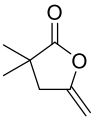
^{13}C NMR (75 MHz, $CDCl_3$)



2b

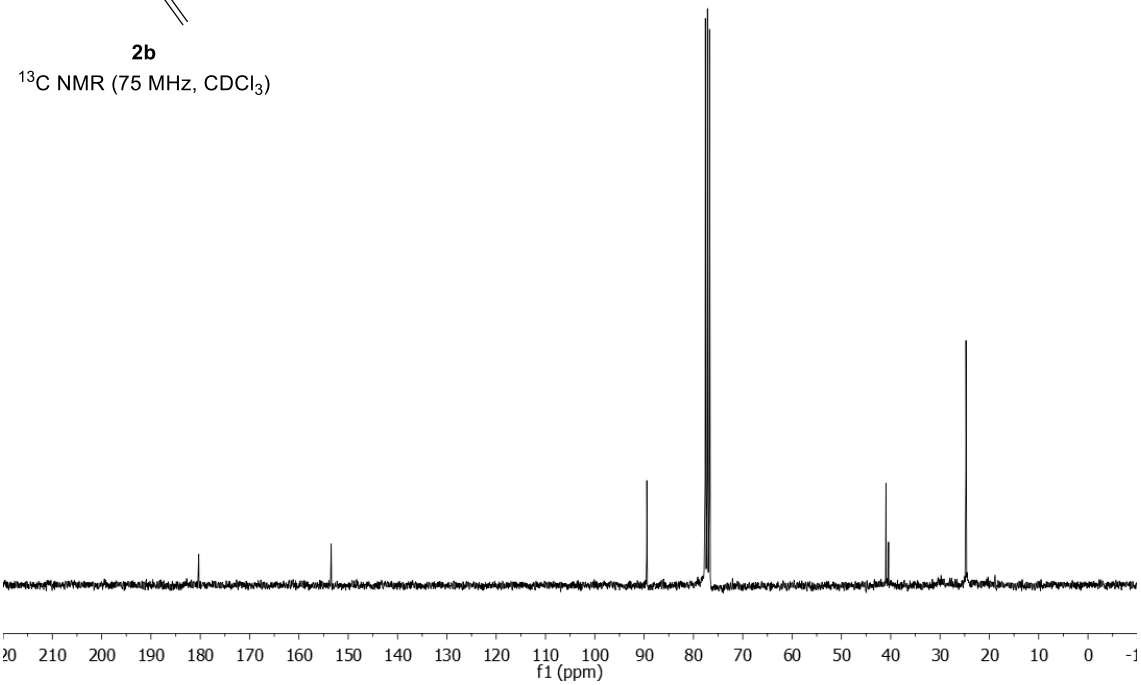
^{1}H NMR (300 MHz, $CDCl_3$)

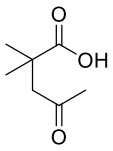




2b

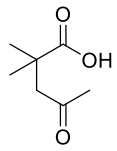
^{13}C NMR (75 MHz, $CDCl_3$)





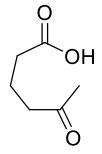
3b

^{1}H NMR (300 MHz, acetone-d_6)



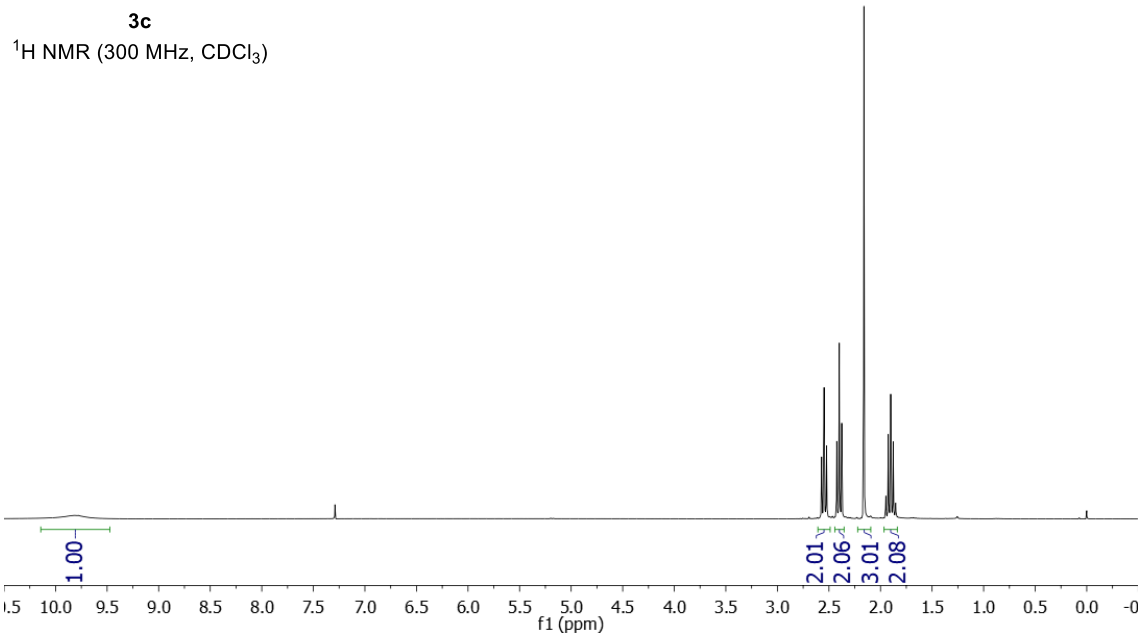
3b

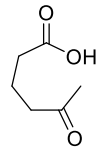
^{13}C NMR (75 MHz, acetone-d_6)



3c

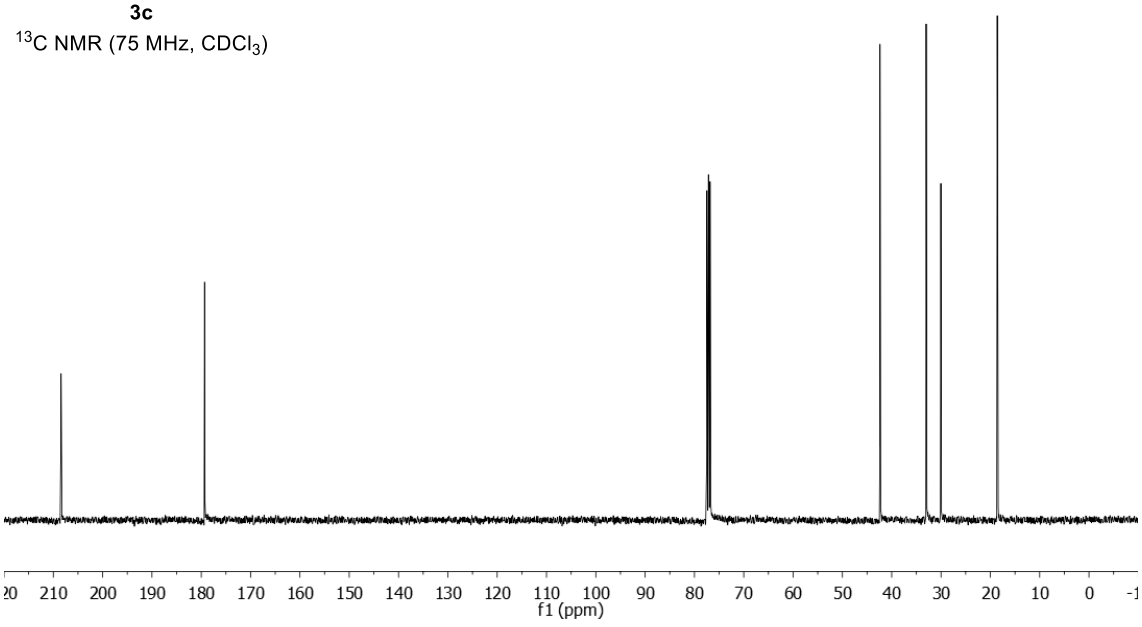
^{1}H NMR (300 MHz, $CDCl_3$)

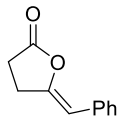




3c

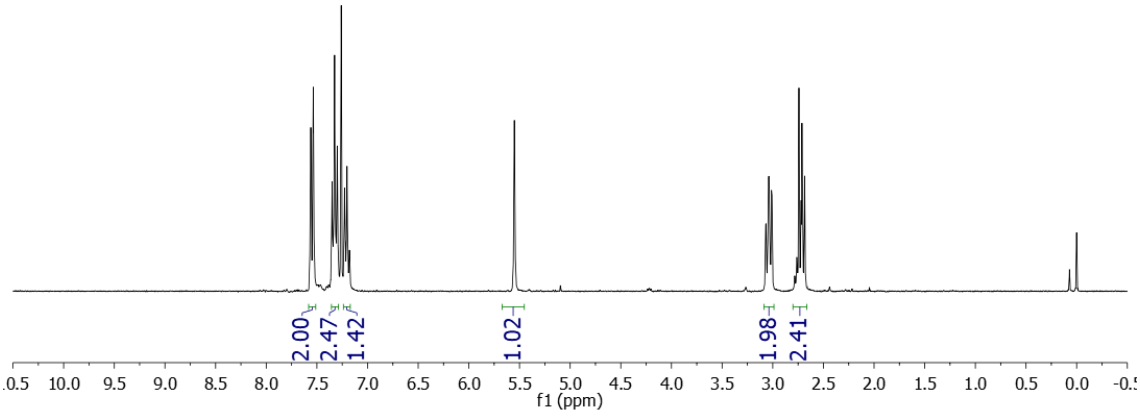
^{13}C NMR (75 MHz, $CDCl_3$)

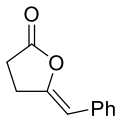




2e

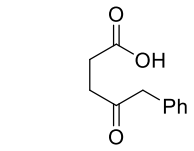
^{1}H NMR (300 MHz, $CDCl_3$)





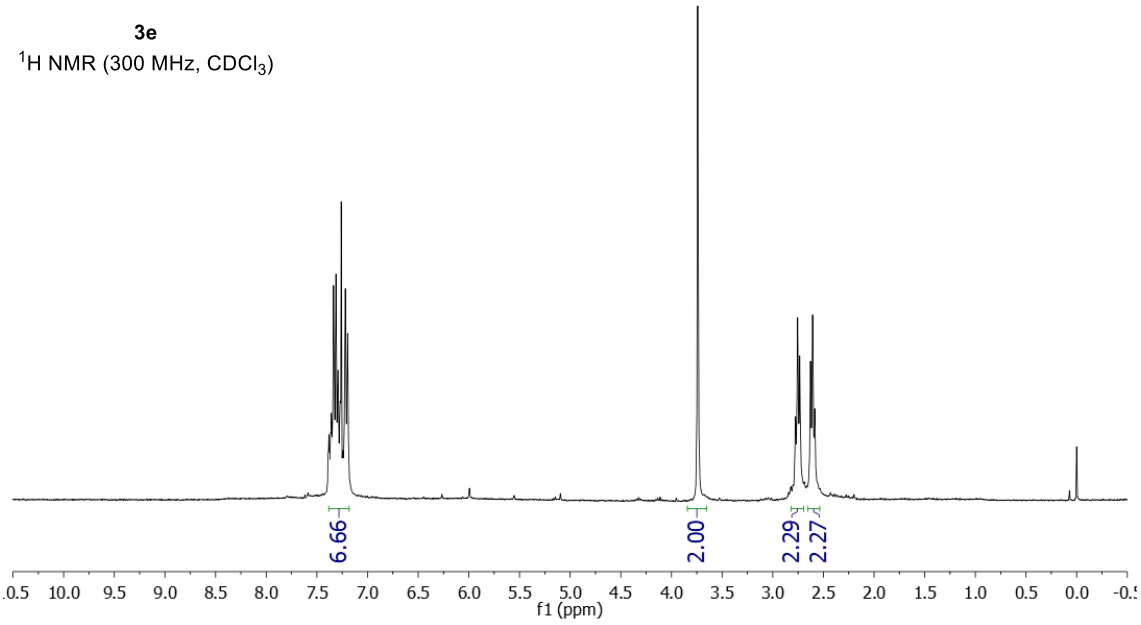
2e

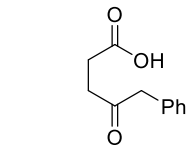
^{13}C NMR (75 MHz, $CDCl_3$)



3e

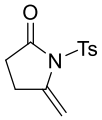
^{1}H NMR (300 MHz, $CDCl_3$)





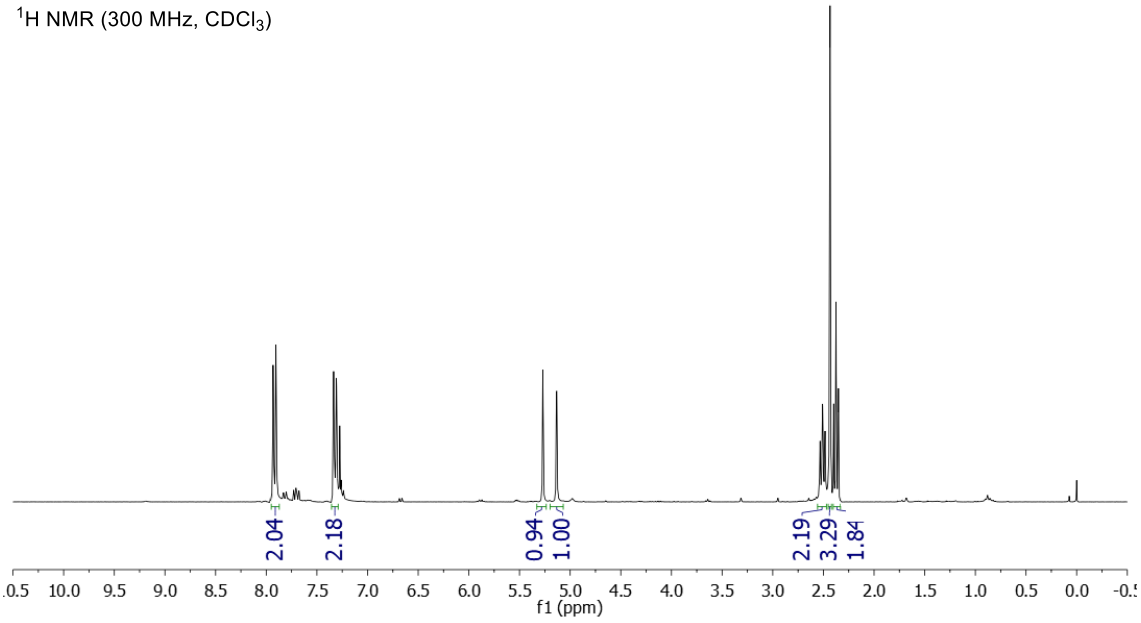
3e

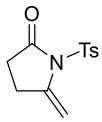
^{13}C NMR (75 MHz, $CDCl_3$)



2f

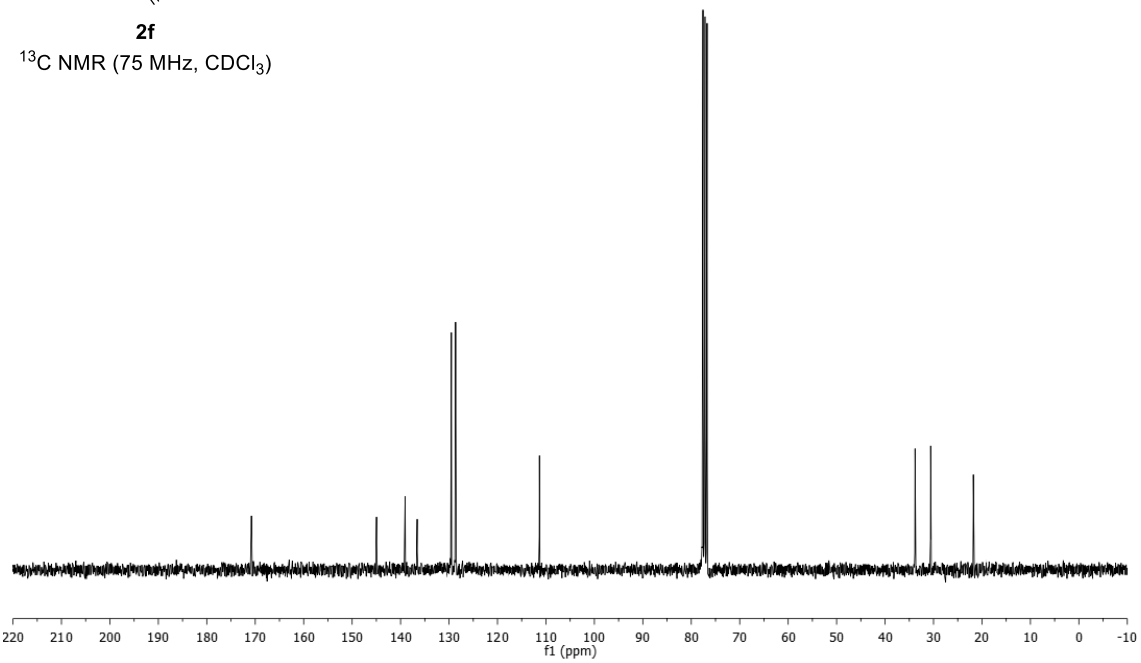
^{1}H NMR (300 MHz, $CDCl_3$)

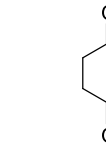




2f

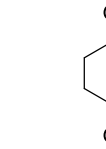
^{13}C NMR (75 MHz, $CDCl_3$)





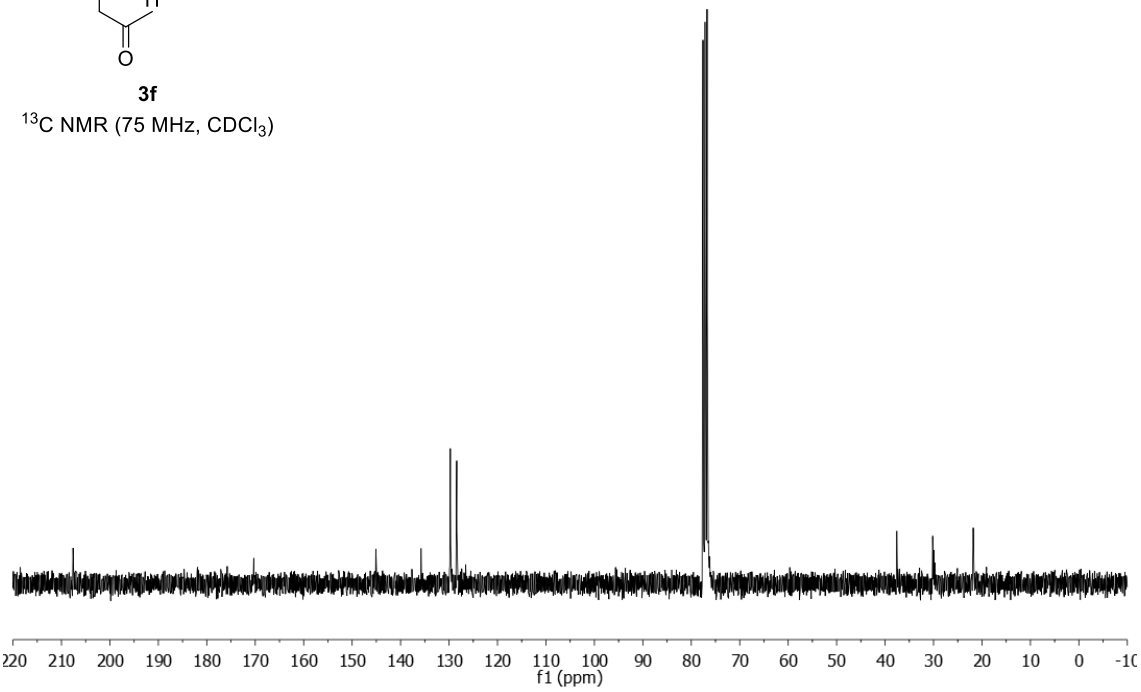
3f

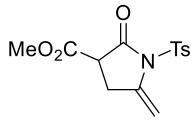
^{1}H NMR (300 MHz, $CDCl_3$)



3f

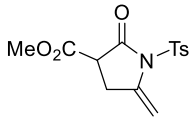
^{13}C NMR (75 MHz, $CDCl_3$)





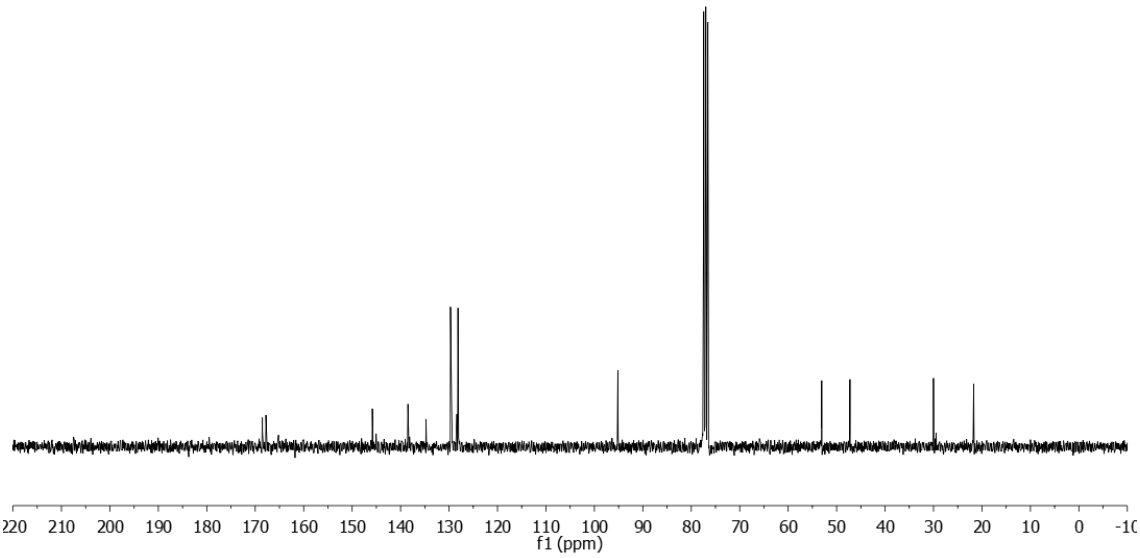
2g

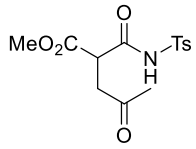
^{1}H NMR (300 MHz, $CDCl_3$)



2g

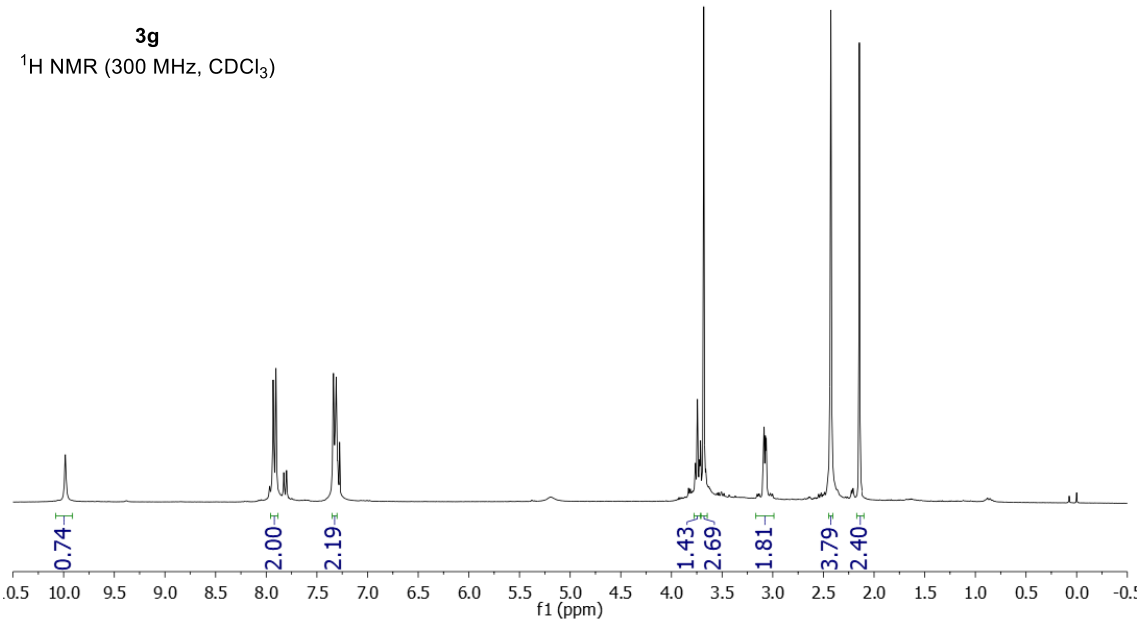
^{13}C NMR (75 MHz, $CDCl_3$)

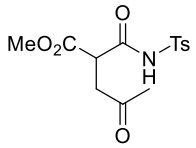




3g

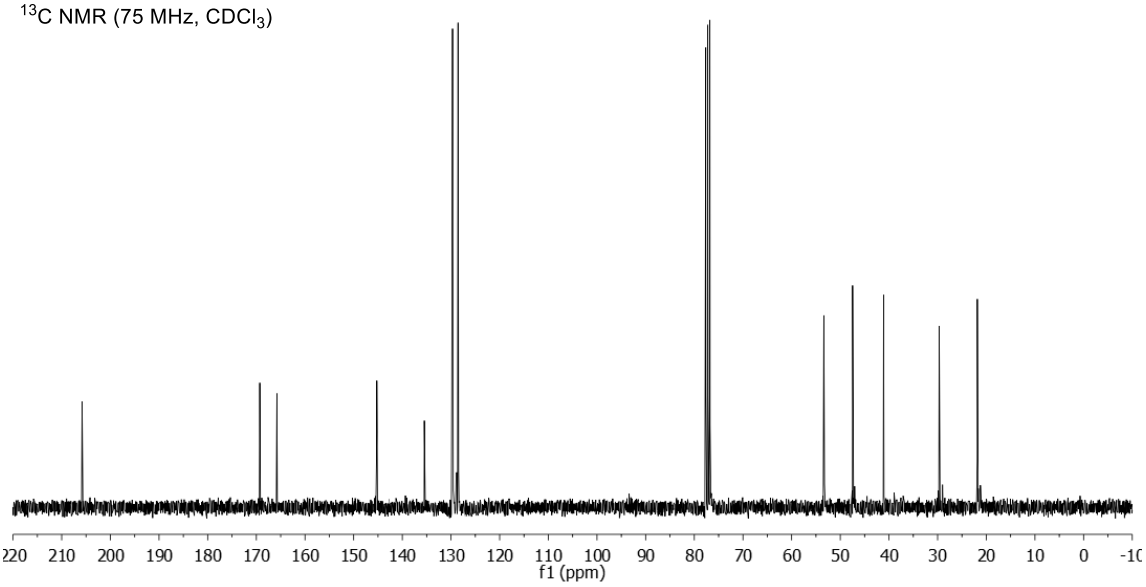
^{1}H NMR (300 MHz, $CDCl_3$)

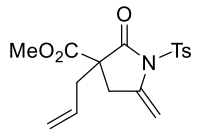




3g

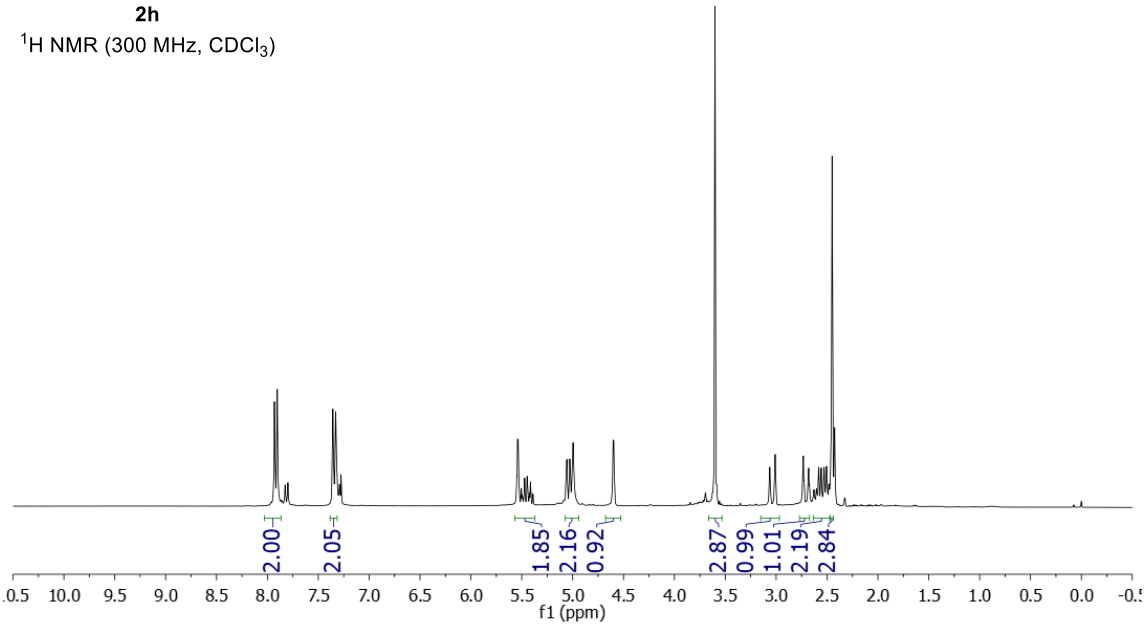
^{13}C NMR (75 MHz, $CDCl_3$)

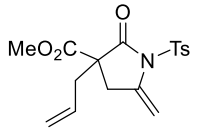




2h

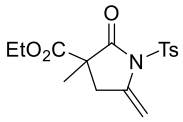
^{1}H NMR (300 MHz, $CDCl_3$)





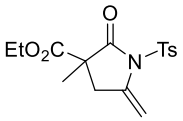
2h

^{13}C NMR (75 MHz, $CDCl_3$)



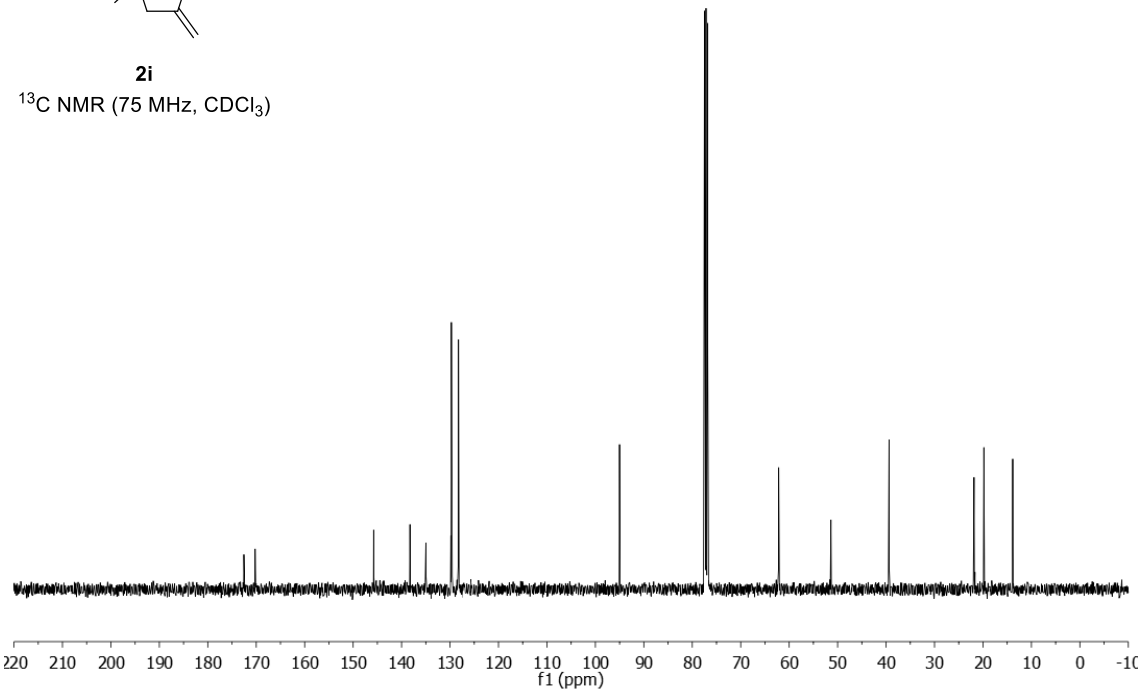
2i

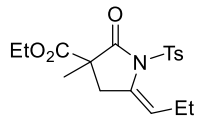
^{1}H NMR (300 MHz, $CDCl_3$)



2i

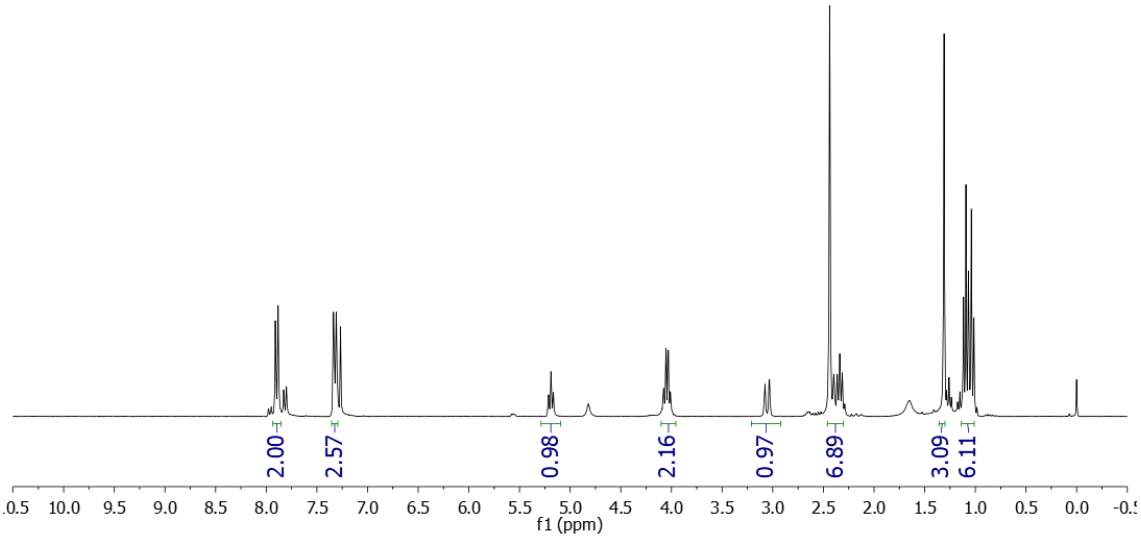
^{13}C NMR (75 MHz, $CDCl_3$)

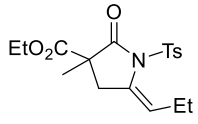




2j

^{1}H NMR (300 MHz, $CDCl_3$)





2j

^{13}C NMR (75 MHz, $CDCl_3$)