

Supporting Information

DAST-mediated ring-opening of cyclopropyl silyl ethers in nitriles: Facile synthesis of allylic amides *via* Ritter-type process

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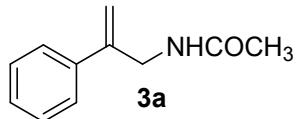
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General

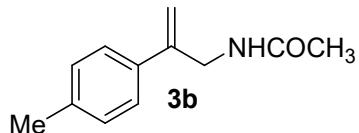
All reagents were purchased from Nacalai Tesque, Wako Pure Chemicals Industries, Kanto Kagaku, Kishida Reagents Chemical Co., Tokyo Chemical Industry, or Aldrich, and used without further purification. Melting Points (MPs) were measured with a Yanaco micro melting point apparatus (MP-J3) and are uncorrected. NMR spectra were recorded with a JEOL JNM-EX400 spectrometer (¹H-NMR 400 MHz, ¹³C-NMR 100 MHz, ¹⁹F-NMR 376 MHz) and a JEOL ECA600 spectrometer (¹H-NMR 600 MHz, ¹³C-NMR 151 MHz). ¹H-NMR spectra are reported as follows: chemical shift in ppm relative to the chemical shift of CHCl₃ at 7.26 ppm, integration, multiplicities (s = singlet, d = doublet, t = triplet, q = quartet, sep = septet, m = multiplet), and coupling constants (Hz). ¹³C-NMR spectra reported in ppm relative to the central line of triplet for CDCl₃ at 77 ppm. ¹⁹F-NMR spectra reported in ppm relative to the central line of singlet for CF₃CO₂H at 0 ppm as an external standard. The IR spectra were recorded using a Jasco IR-8300 FT-IR spectrophotometer and a METTLER React-IR45m. The mass spectra were recorded on a Shimadzu GCMS-QP1100EX spectrometer (EI), JEOL JMS-T100LC spectromete (ESI) or JEOL JMS-700 (FAB).

***N*-(2-phenylallyl) acetamido (3a)^[1]**



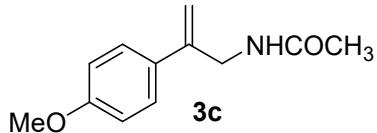
Brown oil; IR (neat) 3286, 3077, 1654, 1550, 903, 708 cm⁻¹; ¹H-NMR (400MHz, CDCl₃) δ 1.92 (3H, s), 4.28–4.26 (2H, d, *J* = 5.6 Hz), 5.20 (1H, s), 5.43 (1H, s), 5.93 (1H, br), 7.24–7.33 (3H, m), 7.37–7.40 (2H, m); ¹³C-NMR (100 MHz, CDCl₃) δ 23.1, 43.1, 113.7, 125.9, 127.9, 128.4, 138.2, 144.1, 177.0.

***N*-(2-(4-methylphenyl)-2-propen-1-yl) acetamide (3b)^[2]**



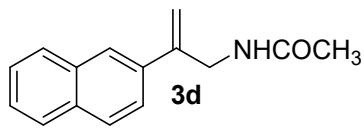
Pale yellow crystal (MP 93 °C); IR (neat) 3290, 3089, 2924, 2856, 1638, 1551, 893 cm⁻¹; ¹H-NMR (400MHz, CDCl₃) δ 1.93 (3H, s), 2.32 (3H, s), 4.26–4.28 (2H, d, *J* = 6.0 Hz), 5.16 (1H, s), 5.41 (1H, s), 5.70 (1H, br), 7.11–7.13 (2H, d, *J* = 7.8 Hz), 7.26–7.31 (2H, d, *J* = 7.8 Hz); ¹³C-NMR (100 MHz, CDCl₃) δ 21.0, 23.1, 43.3, 113.1, 125.8, 129.2, 135.3, 137.9, 143.9, 169.9; MS (EI) (m/z) 189 [M]⁺; HRMS (ESI) calcd for C₁₂H₁₅NONa [M+Na]⁺ 212.1046, found 212.1046.

***N*-(2-(4-methoxyphenyl)-2-propen-1-yl) acetamide (3c)^[2]**



White solid (MP 98 °C); ¹H-NMR (400MHz, CDCl₃) δ 1.92 (3H, s), 3.78 (3H, s), 4.25 (2H, d, *J* = 5.6 Hz), 5.11 (1H, d, *J* = 1.2 Hz), 5.35 (1H, s), 5.68 (1H, br), 6.84(2H, d, *J* = 8.8 Hz), 7.34 (2H, d, *J* = 8.8 Hz); ¹³C-NMR (100MHz, CDCl₃) δ 23.17, 43.35, 55.21, 112.30, 113.83, 127.13, 130.54, 143.41, 159.47, 169.87.

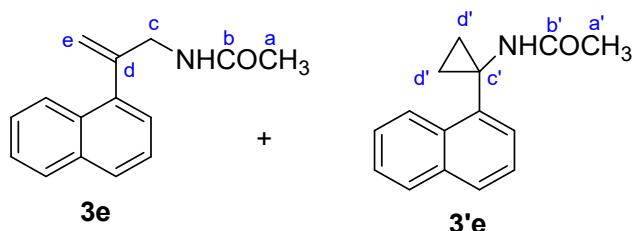
***N*-(2-Naphthalen-2-ylallyl) acetamide (3d)**



Pale yellow solid (MP 104 °C); IR (CHCl₃) 3271, 3085, 1642, 1568, 1430, 892, 745 cm⁻¹; ¹H-NMR (400 MHz, CDCl₃) δ 1.96 (3H, s), 4.44 (2H, d, *J* = 5.2 Hz), 5.35 (1H, s), 5.62 (1H, s), 5.72 (1H, br), 7.58 (1H, dd, *J* = 8.2 Hz, 1.8 Hz), 7.45 – 7.50 (2H, m), 7.80 – 7.86 (4H, m); ¹³C-NMR (100 MHz, CDCl₃) δ 23.2, 43.4, 114.6, 124.1, 124.6, 126.2, 126.2, 126.4, 127.5, 128.2, 128.3, 133.0, 133.3,

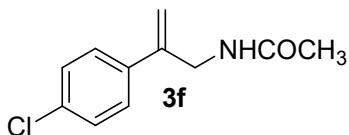
135.3, 170.0; MS (EI) (*m/z*) 225 [M]⁺; HRMS (ESI) calcd for C₁₅H₁₅NONa [M+Na]⁺ 248.1046, found 248.1047.

***N*-(2-(1-naphthyl)-2-propen-1-yl) acetamide (3e) and *N*-(1-(1-naphthyl) cyclopropyl)acetamide (3'e) as inseparable mixtures**



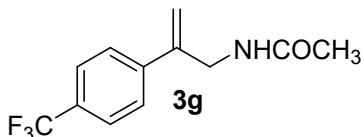
Brown solid; IR (neat) 3422, 3284, 3056, 3008, 2925, 1650, 1544, 912 cm⁻¹; ¹H-NMR (400 MHz, CDCl₃) δ 1.26–1.27 (2.6H, m), 1.38–1.43 (2.5H, m), 1.74 (3H, s), 1.94 (3.4H, s), 4.21–4.23 (1.7H, d, *J* = 6.0 Hz), 4.41–4.43 (0.6H, d, *J* = 6.0 Hz), 5.20–5.21 (0.8H, d, *J* = 1.2 Hz), 5.53–5.55 (0.8H, m), 5.92–5.96 (1H, m), 6.63–6.66 (1H, m), 7.26–7.30 (0.8H, m), 7.36–7.57 (7.5H, m), 7.73–7.86 (6.5H, m), 8.03–8.07 (0.9H, m), 8.41–8.43 (1H, d, *J* = 8.0 Hz); Selected ¹³C-NMR (100 MHz, CDCl₃) δ 14.7 (C^{d'}), 23.1 (C^a or C^{a'}), 23.5 (C^a or C^{a'}), 34.3 (C^{c'}), 45.5 (C^c), 115.8 (C^e), 145.1 (C^d), 170.0 (C^b or C^{b'}), 170.1 (C^b or C^{b'}); MS (EI) (*m/z*) : 225 [M]⁺; HRMS (ESI) calcd for, C₁₅H₁₅NONa [(M+Na)⁺], 248.1046, found : 248.1045.

***N*-[2-(4-chlorophenyl)-2-propen-1-yl] acetamide (3f)^[3]**



Pale yellow solid (MP 98.3 °C); IR (400 MHz, CHCl₃) 3276, 3087, 1635, 1556, 1497, 1372, 1285, 1230, 1100, 1017, 903, 833, 730 cm⁻¹; ¹H-NMR (100 MHz, CDCl₃) δ 1.97 (3H, s), 4.32 (2H, dd, *J* = 12.0, 5.6 Hz), 5.25 (1H, s), 5.46 (1H, s), 5.51 (1H, br), 7.32 (2H, d, *J* = 8.6 Hz), 7.37 (2H, d, *J* = 8.6 Hz); ¹³C-NMR (CDCl₃) δ 23.0, 42.9, 114.3, 127.2, 128.5, 133.7, 136.4, 143.0, 169.6; HRMS (ESI) calcd for C₁₁H₁₂ClNONa [M+Na]⁺ 232.0500, found 232.0498.

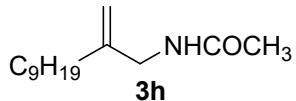
***N*-[2-[4-(trifluoromethyl)phenyl]-2-propen-1-yl] acetamide (3g)^[2]**



White solid (MP 121.7 °C); IR (CHCl₃) 3273, 3080, 1639, 1552, 1324, 1151, 1112, 1061, 845 cm⁻¹; ¹H-NMR (400 MHz, CDCl₃) δ 1.97 (3H, s), 4.35 (2H, d, *J* = 6.0 Hz), 5.35 (1H, s), 5.54 (1H, s), 5.67

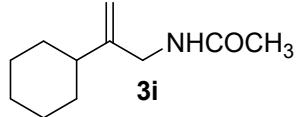
(1H, br), 7.53 (2H, d, $J = 8.2$ Hz), 7.60 (2H, d, $J = 8.2$ Hz); ^{19}F -NMR (CDCl_3) δ 63.09 (s); ^{13}C -NMR (100 MHz, CDCl_3) δ 23.2, 43.0, 115.9, 124.0 (q, $J_{\text{C}-\text{F}} = 272.0$ Hz), 125.5 (q, $J = 3.8$ Hz), 129.5 (q, $J = 32.6$ Hz), 141.8, 143.4, 169.9; MS (EI) (m/z) 243 [M] $^+$; HRMS (ESI) calcd for $\text{C}_{12}\text{H}_{17}\text{F}_3\text{NONa}$ [M+Na] $^+$ 266.0763, found 266.0761.

N-(2-methylene-1-undecyl) acetamide (3h)



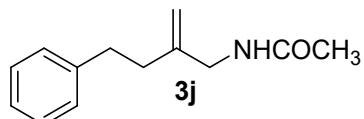
White solid (MP 37 °C); IR (neat) 3284, 3084, 2925, 2859, 1647, 1552, 896 cm^{-1} ; ^1H -NMR (400 MHz, CDCl_3) δ 0.80–0.83 (3H, t, $J = 6.4$ Hz), 1.11–1.25 (12H, m), 1.36–1.39 (2H, m), 1.92–1.97 (5H, m), 3.73–3.75 (2H, d, $J = 5.6$ Hz), 4.77–4.79 (2H, d, $J = 10.4$ Hz), 6.02 (1H, br); ^{13}C -NMR (100 MHz, CDCl_3) δ 13.7, 22.6, 23.0, 27.5, 29.2, 29.2, 29.4, 29.5, 31.8, 34.0, 43.9, 109.6, 146.1, 170.0; MS (EI) (m/z) 225 [M] $^+$; HRMS (ESI) calcd for $\text{C}_{14}\text{H}_{27}\text{NONa}$ [M+Na] $^+$ 248.1985, found 248.1983.

N-(2-cyclohexylallyl) acetamide (3i)



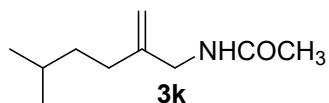
Yellow solid (MP 60 °C); IR (CHCl_3) 3245, 3079, 2921, 1639, 1544, 750 cm^{-1} ; ^1H -NMR (400 MHz, CDCl_3) δ 1.08–1.88 (11H, m), 1.99 (3H, s), 3.83 (2H, d, $J = 6.0$ Hz), 4.81 (1H, d, $J = 0.8$ Hz), 4.83 (1H, d, $J = 0.8$ Hz); ^{13}C -NMR (100 MHz, CDCl_3) δ 23.3, 26.2, 26.6, 32.2, 42.5, 43.1, 108.3, 151.4, 169.8; MS (EI) (m/z) 181 [M] $^+$; HRMS (ESI) calcd for $\text{C}_{11}\text{H}_{19}\text{NONa}$ [M+Na] $^+$ 204.1359, found 204.1359.

N-(2-phenethylallyl) acetamide (3j)^[4]



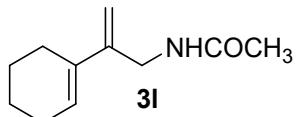
Yellow solid (MP 50 °C); IR (neat) 3280, 3075, 2922, 1647, 1552, 1285, 746, 699 cm^{-1} ; ^1H -NMR (400 MHz, CDCl_3) δ 1.97 (3H, s), 2.32 (2H, t, $J = 8.0$ Hz), 2.76 (2H, t, $J = 8.0$ Hz), 3.84 (2H, d, $J = 6.0$ Hz), 4.88–4.90 (2H, m), 5.47 (1H, br), 7.15–7.28 (5H, m); ^{13}C -NMR (100 MHz, CDCl_3) δ 23.3, 34.2, 35.7, 44.2, 110.9, 125.9, 128.4, 128.4, 141.6, 145.4, 169.9; MS (EI) (m/z) 203 [M] $^+$; HRMS (ESI) calcd for $\text{C}_{13}\text{H}_{17}\text{NONa}$ [M+Na] $^+$ 226.1202, found 226.1201.

N-[2-(3-methylbutyl)allyl] acetamide (3k)



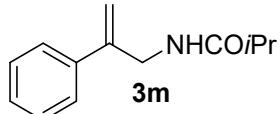
Yellow oil; IR (neet) 3276, 3080, 2950, 1651, 1549, 892, 754 cm⁻¹; ¹H-NMR (400 MHz, CDCl₃) δ 0.87 (6H, d, *J* = 7.2 Hz), 1.28 – 1.34 (2H, m), 1.47 – 1.57 (1H, m), 1.96 – 2.02 (5H, m), 3.81 (2H, s), 4.83 (2H, s), 5.50 (1H, br); ¹³C-NMR (100 MHz, CDCl₃) δ 22.6, 23.4, 27.8, 32.1, 36.1, 44.2, 109.8, 146.6, 170.0; MS (EI) (m/z) 169 [M]⁺; HRMS (ESI) calcd for C₁₀H₁₉NONa [M+Na]⁺ 192.1359, found 192.1359.

N-(2-cyclohex-1-enyl-allyl)acetamide (3l)



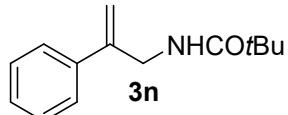
Brown oil; ¹H-NMR (400 MHz, CDCl₃) δ 2.00 (3H, s), 4.07 (2H, d, *J* = 5.6 Hz), 4.99 (1H, s), 5.10 (1H, s), 5.57 (1H, br), 5.91 – 5.92 (1H, m); ¹³C-NMR (100 MHz, CDCl₃) δ 22.0, 22.7, 23.3, 25.8 x 2, 42.5, 111.0, 125.7, 134.0, 144.1, 169.7; MS (EI) (m/z) 181 [M]⁺; HRMS (ESI) calcd for C₁₁H₁₇NONa [M+Na]⁺ 202.1202, found 202.1202.

N-(2-phenylallyl) isobutyramide (3m)



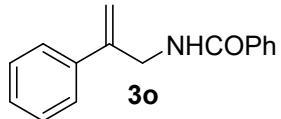
Brown solid (MP 44 °C); IR (CHCl₃) 3297, 3081, 2968, 2931, 1646, 1543, 1232, 697 cm⁻¹; ¹H-NMR (400 MHz, CDCl₃) δ 1.09 (6H, d, *J* = 6.8 Hz), 2.30 (1H, sep, *J* = 6.8 Hz), 4.32 (2H, d, *J* = 5.6 Hz), 5.21 (1H, s), 5.45 (1H, s), 5.52 (1H, br), 7.26 – 7.35 (3H, m), 7.39 – 7.42 (2H, m); ¹³C-NMR (100 MHz, CDCl₃) δ 19.5, 35.6, 43.0, 113.6, 126.1, 128.0, 128.5, 138.3, 144.5, 176.7; MS (EI) (m/z) 203 [M]⁺; HRMS (ESI) calcd for C₁₃H₁₇NONa [M+Na]⁺ 226.1202, found 226.1206.

2,2-dimethyl-N-(2-phenylallyl) propionamide (3n)^[5]



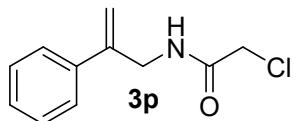
Pale yellow solid (MP 108 °C, lit.^[5] 102-104 °C); ¹H-NMR (400 MHz, CDCl₃) δ 1.14 (9H, s), 4.32 (2H, d, *J* = 5.6 Hz), 5.20 (1H, d, *J* = 0.8 Hz), 5.44 (1H, s), 5.72 (1H, br), 7.27 – 7.42 (5H, m); ¹³C-NMR (100 MHz, CDCl₃) δ 27.4, 38.6, 43.1, 113.3, 126.1, 127.9, 128.4, 138.4, 144.8, 178.1.

N-(2-phenylallyl) benzamide (3o)^[6]



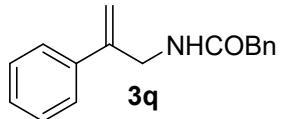
Pale yellow solid (MP 123 °C, lit. [6] 123-125 °C); ¹H-NMR (400 MHz, CDCl₃) δ 4.53 (2H, d, *J* = 5.6 Hz), 5.31 (1H, s), 5.52 (1H, s), 6.27 (1H, br), 7.26 – 7.49 (8H, m), 7.70 – 7.78 (2H, m); ¹³C-NMR (100 MHz, CDCl₃) δ 43.7, 114.1, 126.1, 126.9, 128.1, 128.5, 128.6, 131.5, 134.4, 138.2, 144.2, 137.3.

2-Chloro-N-(2-phenylallyl) acetamide (3p)



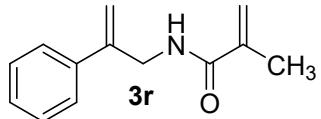
White solid (MP 44-45 °C); IR (KBr) 3309, 3067, 2890, 1656, 1551, 1415, 1224, 914 cm⁻¹; ¹H-NMR (600 MHz, CDCl₃) δ 4.05 (2H, s), 4.38 (2H, d, *J* = 5.5 Hz), 5.26 (1H, s), 5.50 (1H, s), 6.69 (1H, br), 7.32 (1H, tt, *J* = 7.2, 1.7 Hz), 7.35-7.37 (2H, m), 7.41-7.43 (2H, m); ¹³C-NMR (151 MHz, CDCl₃) δ 42.6, 43.3, 114.0, 126.0, 128.2, 128.6, 138.0, 143.5, 165.7; HRMS (ESI) calcd for C₁₁H₁₂ClN₁O₁Na [M+Na]⁺ 232.0500, found 232.0503.

2-Phenyl-N-(2-phenylallyl) acetamide (3q)^[7]



White solid (MP 68-69 °C); IR (KBr) 3324, 3087, 3031, 2931, 1639, 1541, 1496, 906 cm⁻¹; ¹H-NMR (600 MHz, CDCl₃) δ 3.55 (2H, s), 4.29 (2H, d, *J* = 5.5 Hz), 5.08 (1H, s), 5.36 (1H, s), 5.51 (1H, br), 7.12-7.14 (2H, m), 7.26-7.35 (8H, m); ¹³C-NMR (151 MHz, CDCl₃) δ 43.1, 43.7, 113.4, 126.0, 127.3, 128.0, 128.4, 128.9, 129.3, 134.6, 138.3, 144.3, 170.7; HRMS (ESI) calcd for C₁₇H₁₇NONa [M+Na]⁺ 274.1203, found 274.1204.

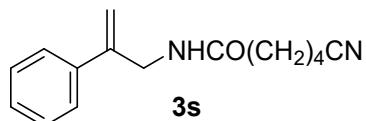
N-(2-phenylallyl) methacrylamide (3r)



White solid (MP 52-53 °C); IR (KBr) 3309, 3080, 3057, 2924, 1656, 1614, 1269, 905 cm⁻¹; ¹H-NMR (600 MHz, CDCl₃) δ 1.92 (3H, s), 4.39 (2H, d, *J* = 5.5 Hz), 5.25 (1H, s), 5.29 (1H, s), 5.48 (1H, s), 5.61 (1H, s), 5.93 (1H, br), 7.29-7.31 (1H, m), 7.33-7.36 (2H, m), 7.42-7.44 (2H, m); ¹³C-NMR (151

MHz, CDCl₃) δ 18.6, 43.2, 113.8, 119.5, 126.0, 128.0, 128.5, 138.3, 139.9, 144.2, 168.2; HRMS (ESI) calcd for C₁₅H₁₈N₂ONa [M+Na]⁺ 265.1311, found 265.1307.

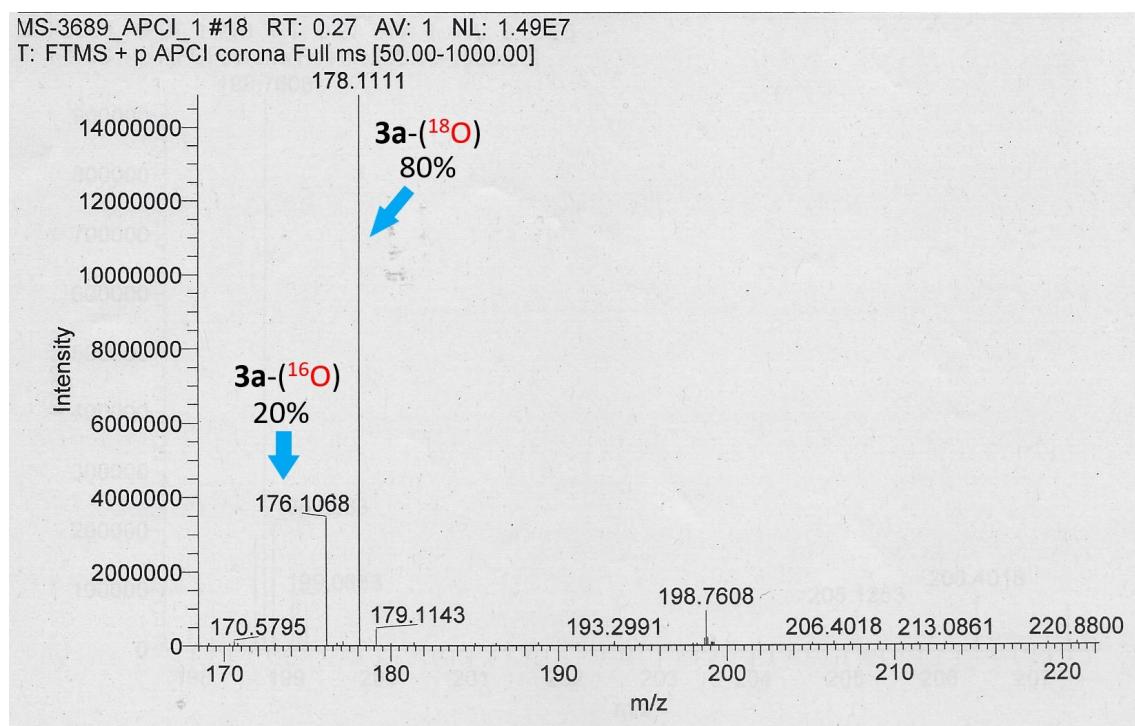
5-Cyano-pentanoic acid (2-phenylallyl) amide (3s)



Yellow oil; IR (CHCl₃) 3294, 3074, 2938, 2872, 2250, 1650, 1544, 1426, 1247, 906, 778, 705 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 1.56-1.63 (2H, m), 1.70-1.78 (2H, m), 2.18 (2H, t, *J* = 7.0 Hz), 2.29 (2H, t, *J* = 7.0 Hz), 4.3 (2H, d, *J* = 6.0 Hz), 5.23 (1H, d, *J* = 0.8 Hz), 5.46 (1H, s), 5.56 (1H, br), 7.28-7.37 (3H, m), 7.40-7.43 (2H, m); ¹³C NMR (CDCl₃) δ 16.91, 24.51, 24.67, 35.35, 43.24, 114.19, 119.42, 126.11, 128.12, 128.53, 138.16, 144.34, 171.55; HRMS (ESI) calcd for

O-isotopic labeled mechanistic studies

When the O-isotopic labeled H₂O (97 atom% ¹⁸O) was used for the reaction work-up, scrambling of ¹⁶O and ¹⁸O in the amide group was observed and the ratio between them (20/80) was determined *via* HRMS

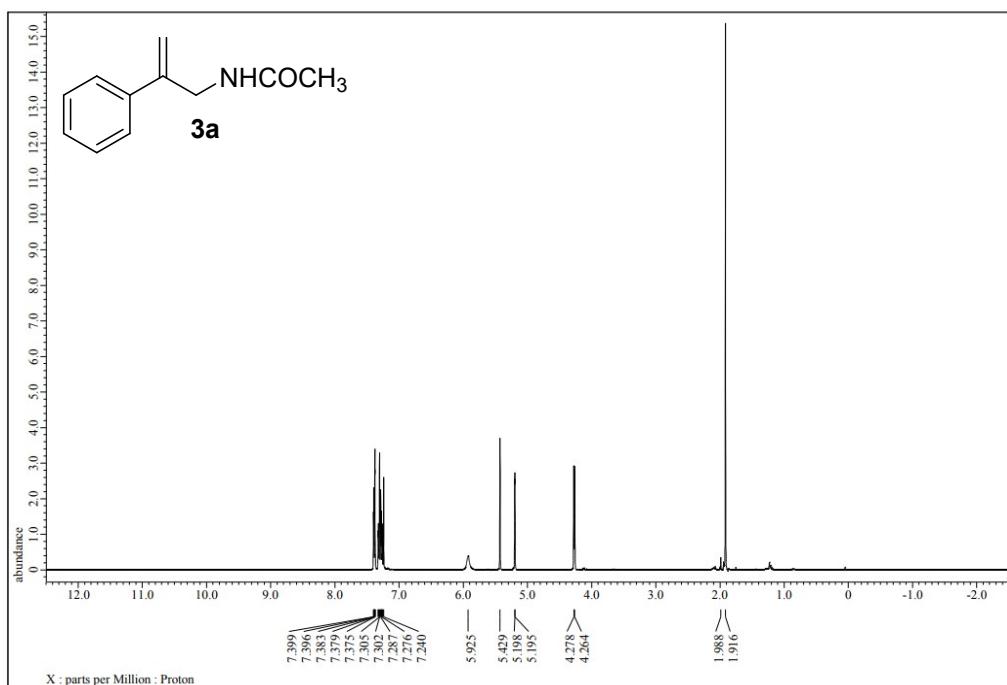


References

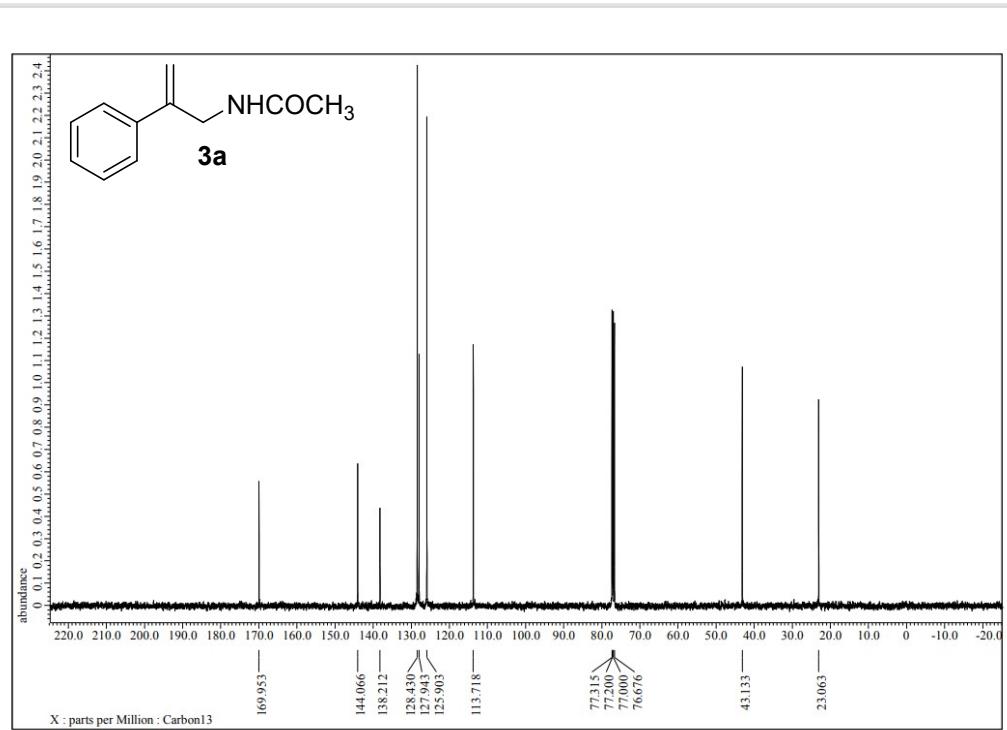
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NMR chart

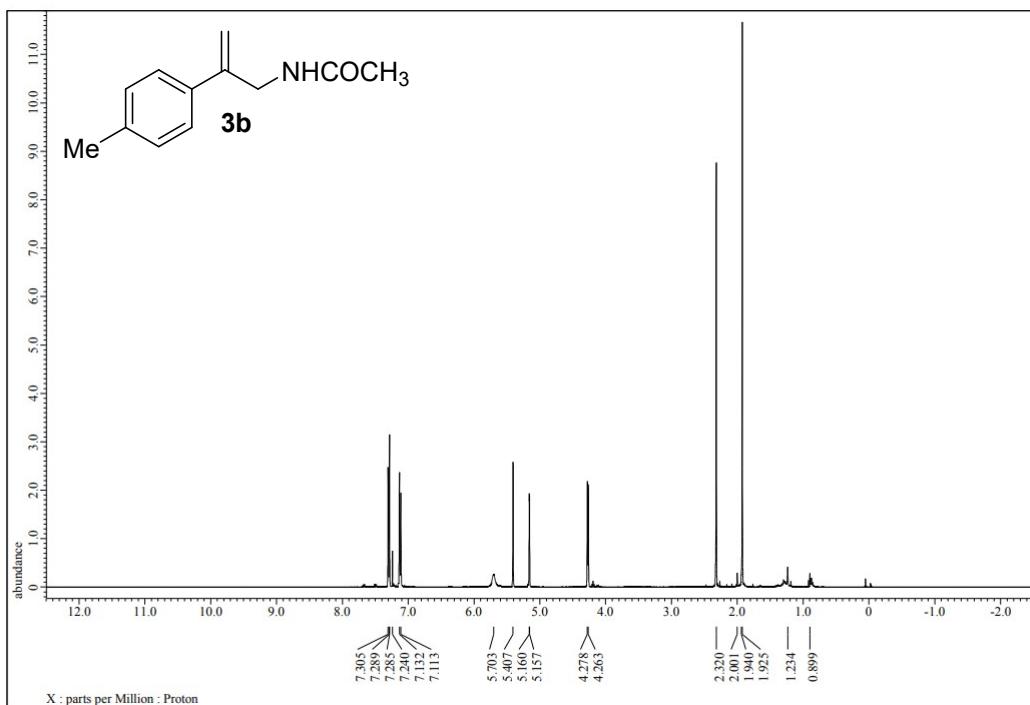
^1H -NMR (400 MHz, CDCl_3) chart of **3a**



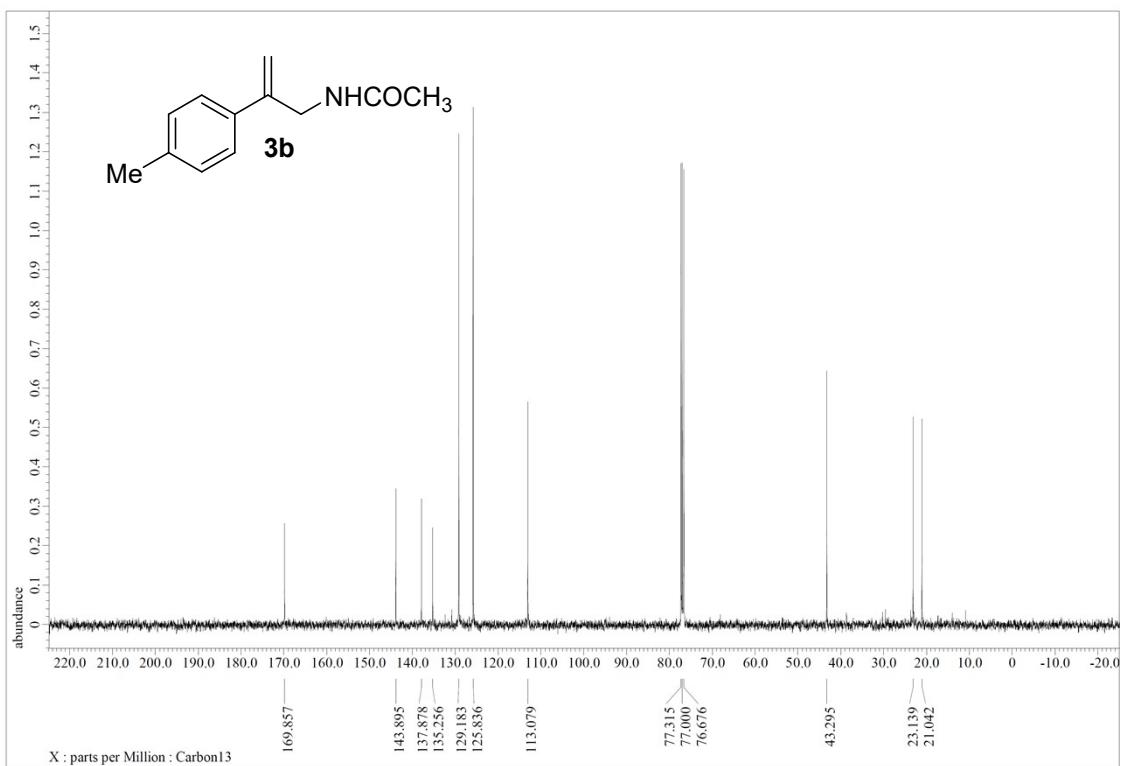
^{13}C -NMR (100 MHz, CDCl_3) chart of **3a**



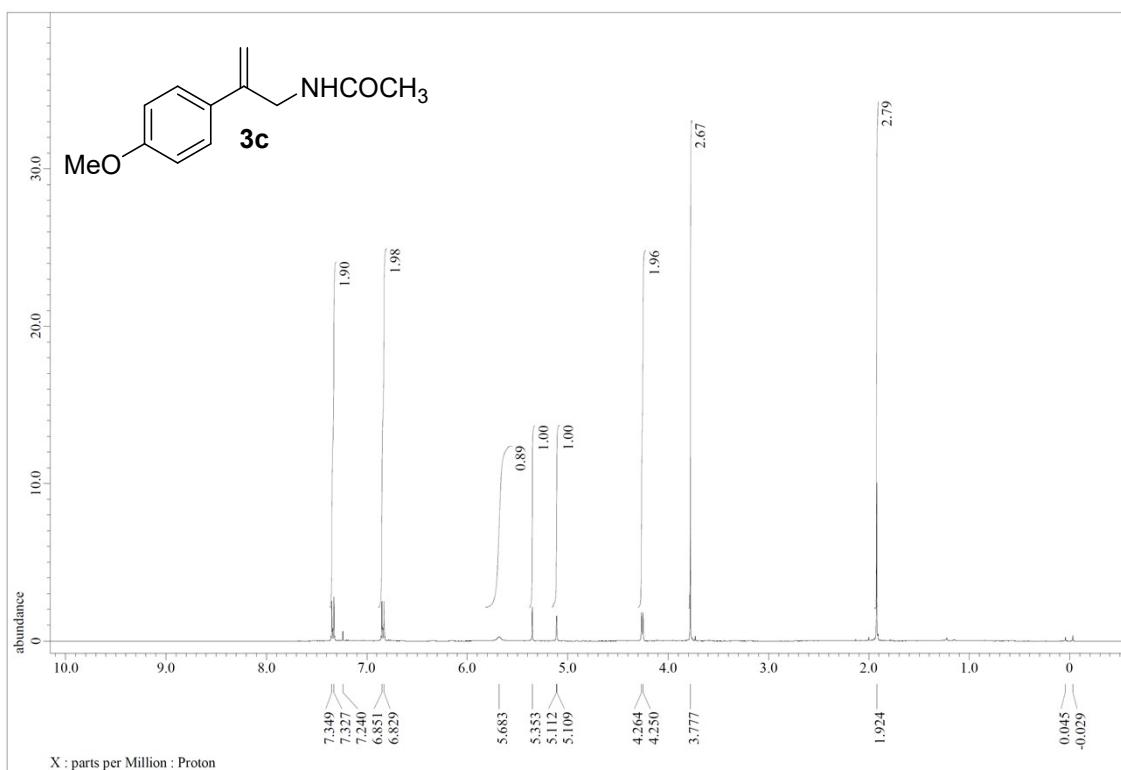
¹H-NMR (400 MHz, CDCl₃) chart of **3b**



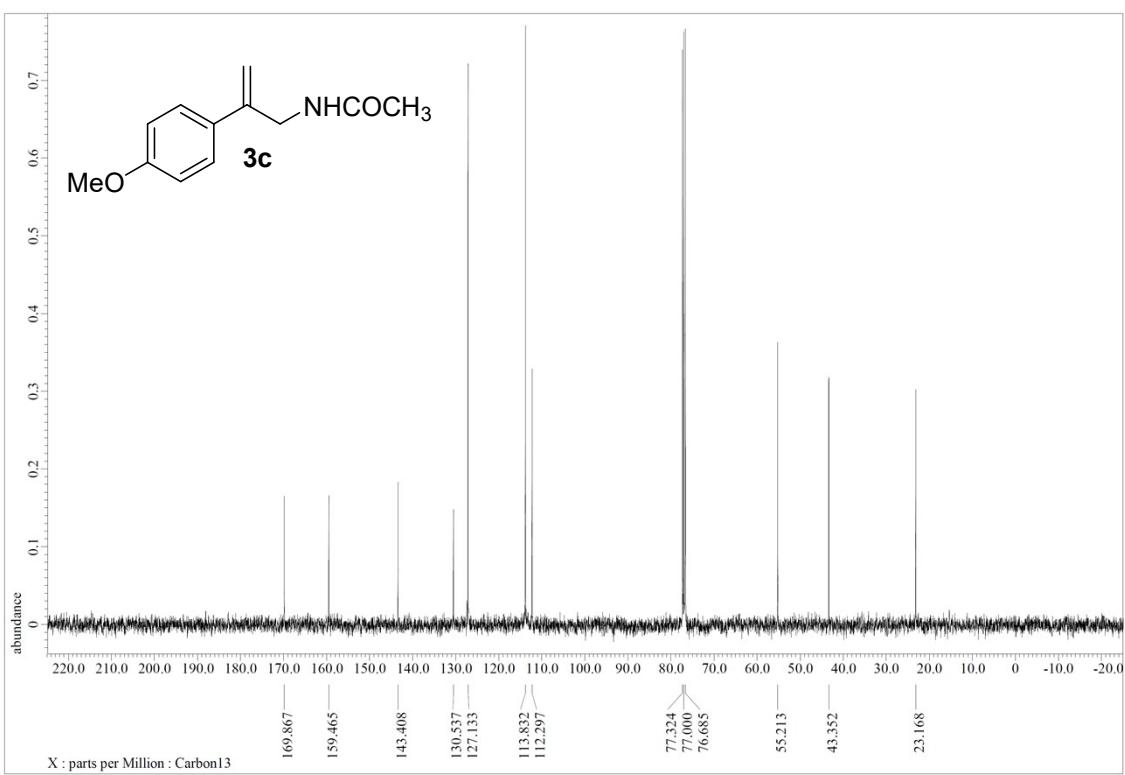
¹³C-NMR (100 MHz, CDCl₃) chart of **3b**



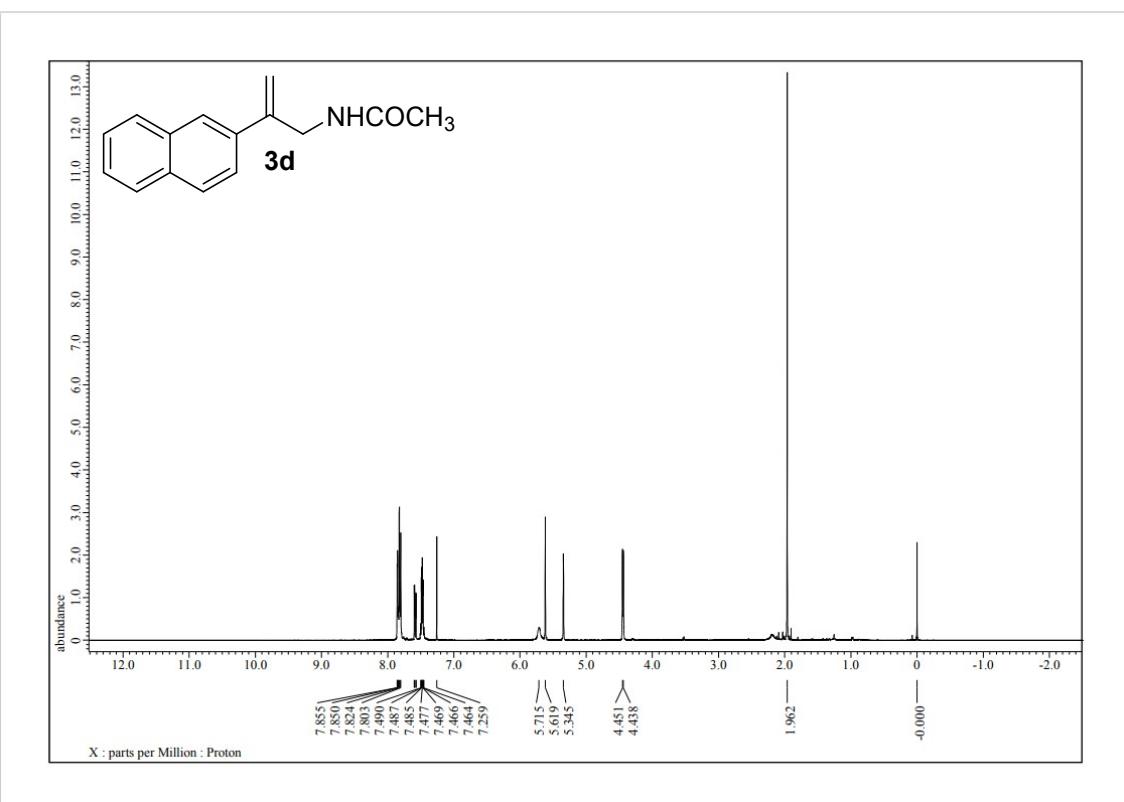
¹H-NMR (400 MHz, CDCl₃) chart of 3c



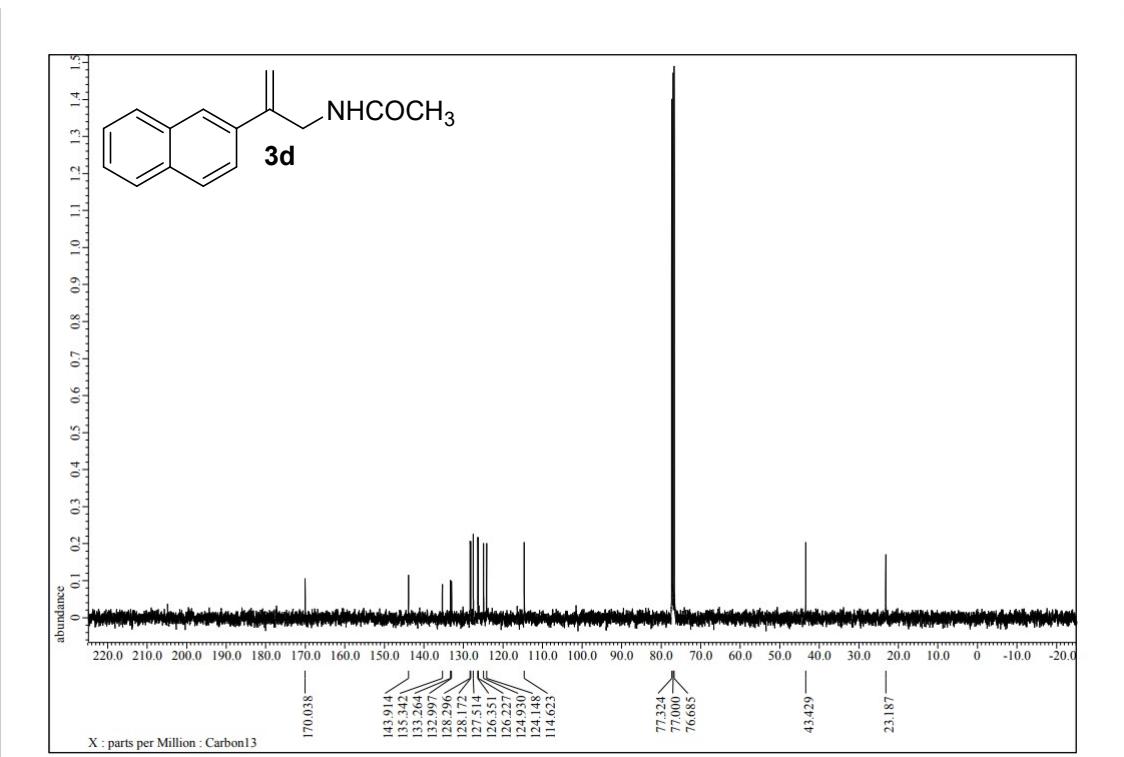
¹³C-NMR (100 MHz, CDCl₃) chart of 3c



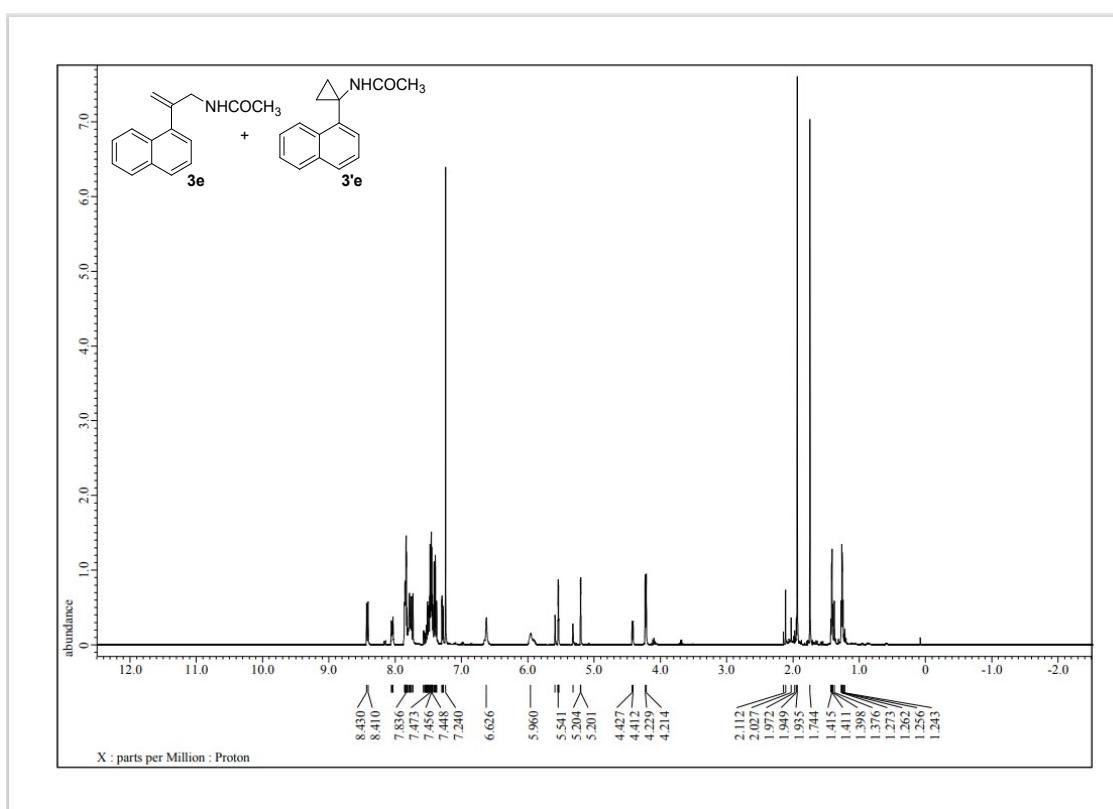
¹H-NMR (400 MHz, CDCl₃) chart of **3d**



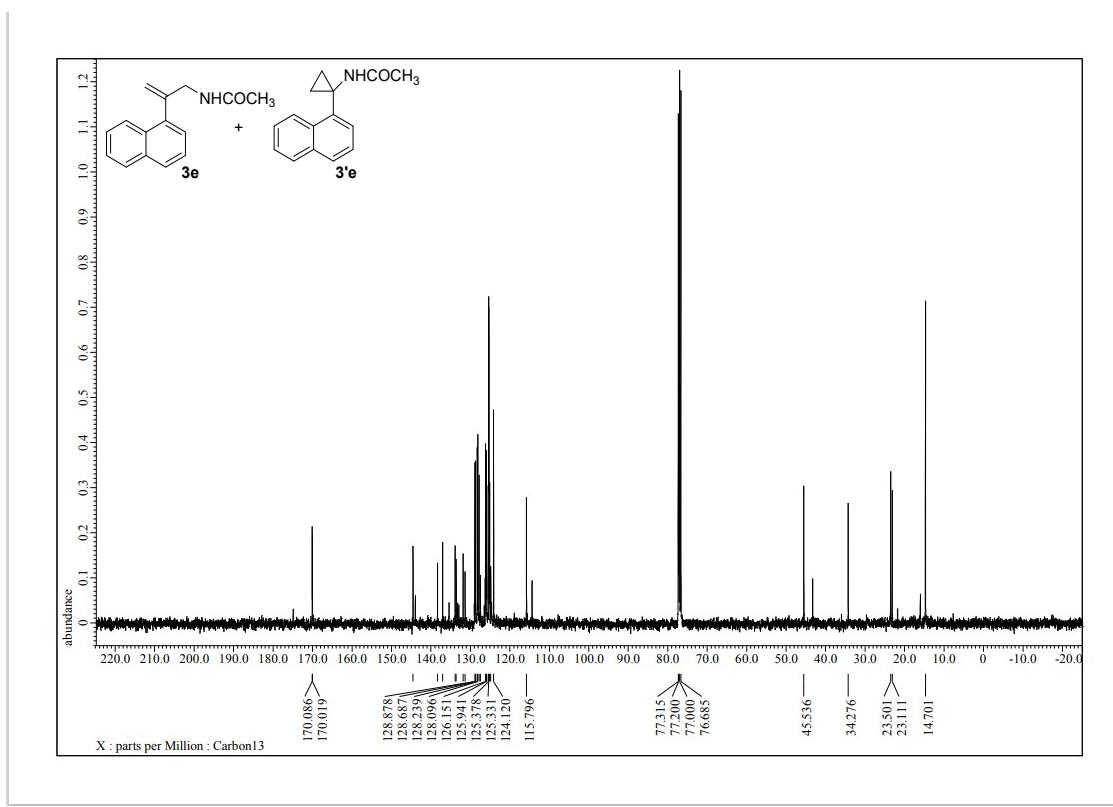
¹³C-NMR (100 MHz, CDCl₃) chart of **3d**



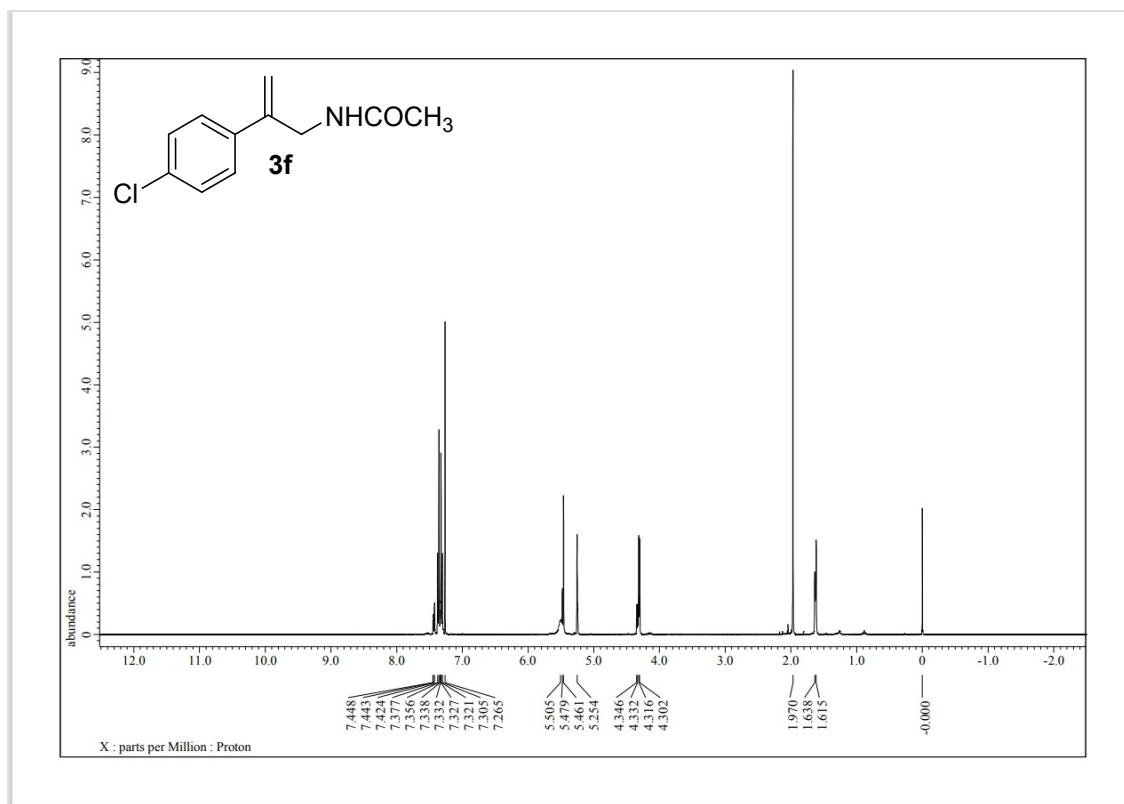
¹H-NMR (400 MHz, CDCl₃) chart of **3e** and **3'e** as inseparable mixtures



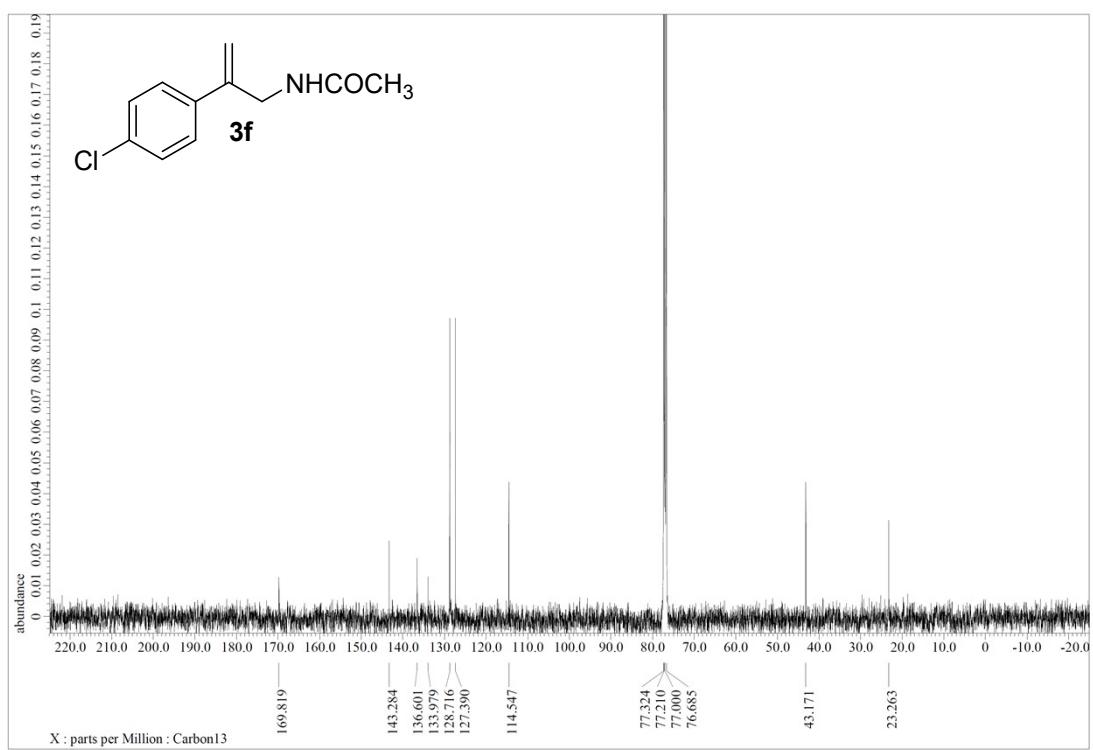
¹³C-NMR (100 MHz, CDCl₃) chart of **3e** and **3'e** as inseparable mixtures



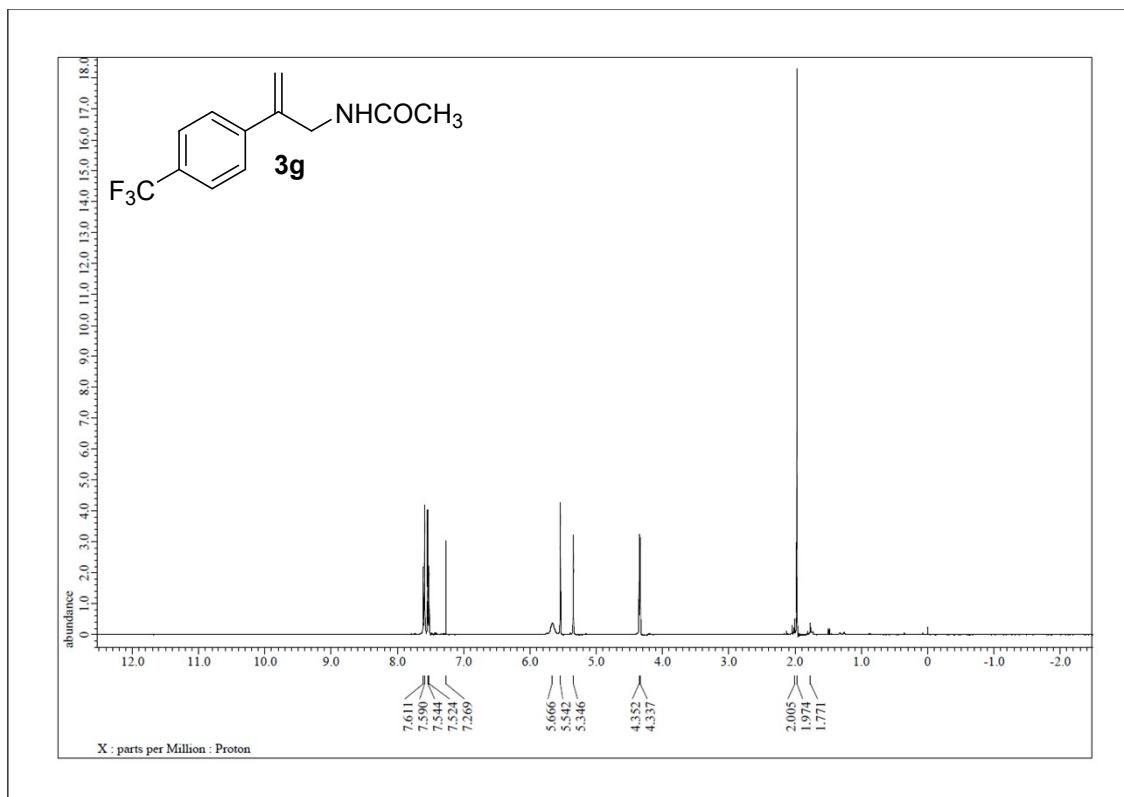
¹H-NMR (400 MHz, CDCl₃) chart of **3f**



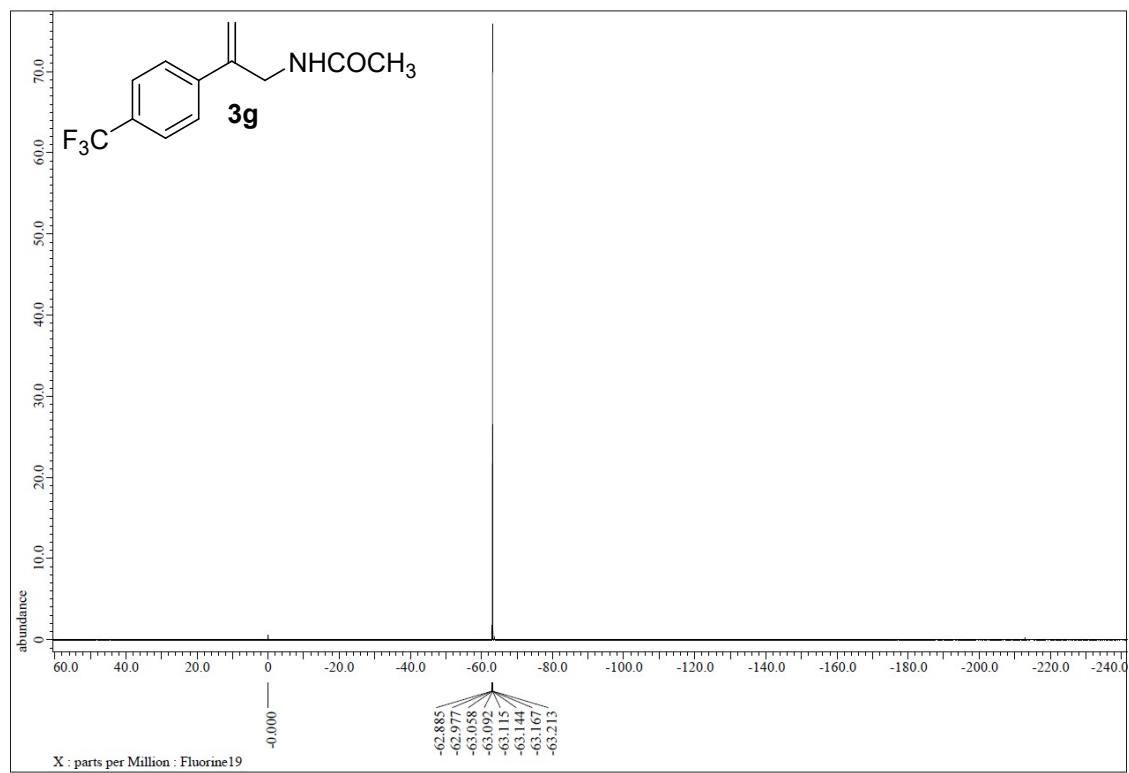
¹³C-NMR (100 MHz, CDCl₃) chart of **3f**



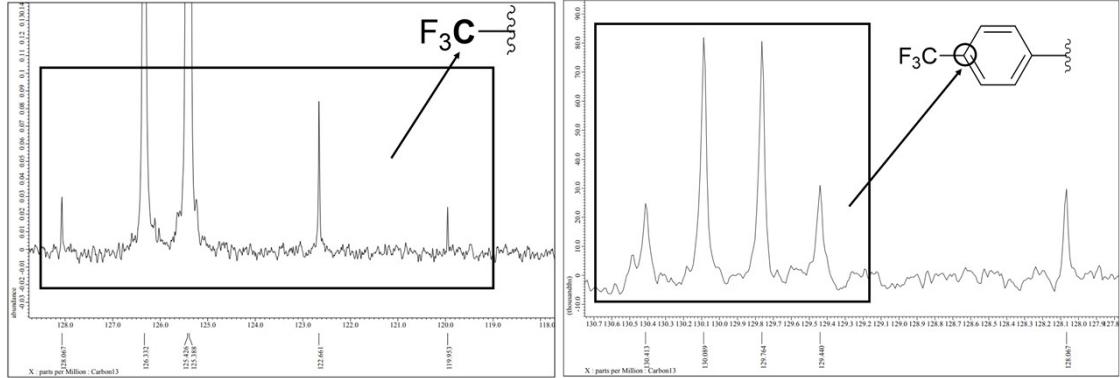
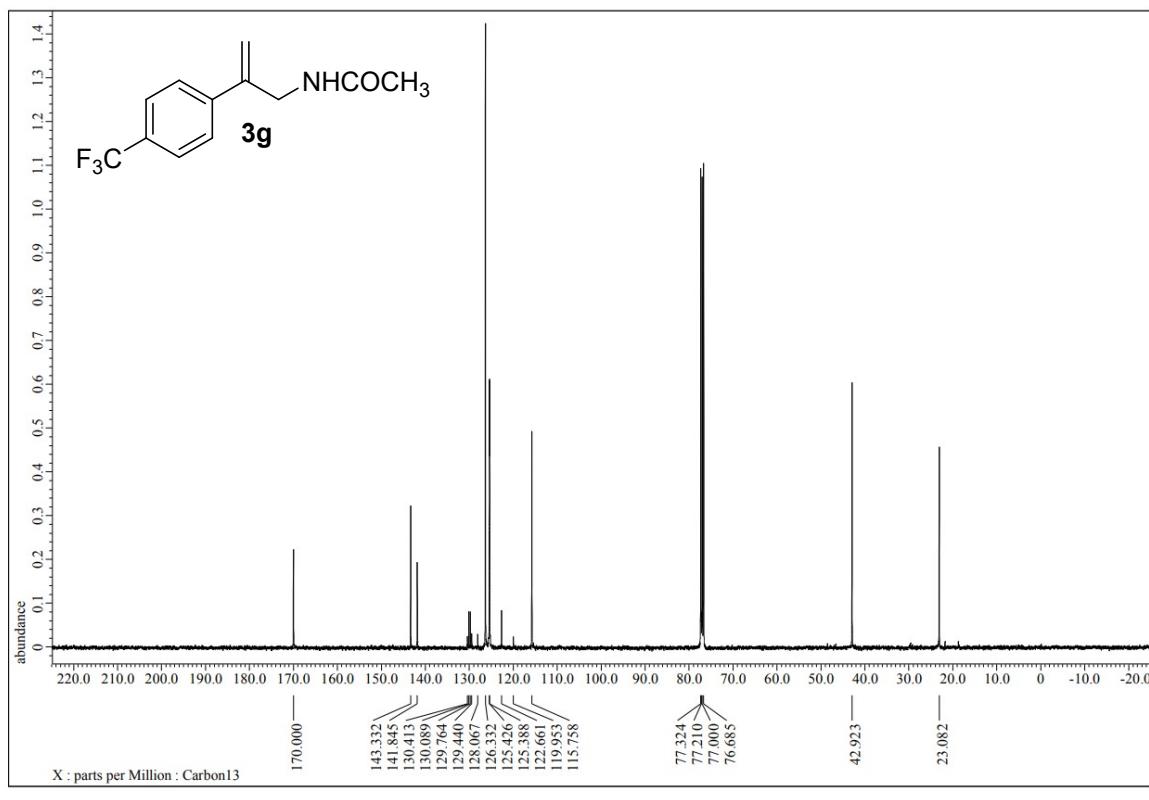
¹H-NMR (400 MHz, CDCl₃) chart of **3g**



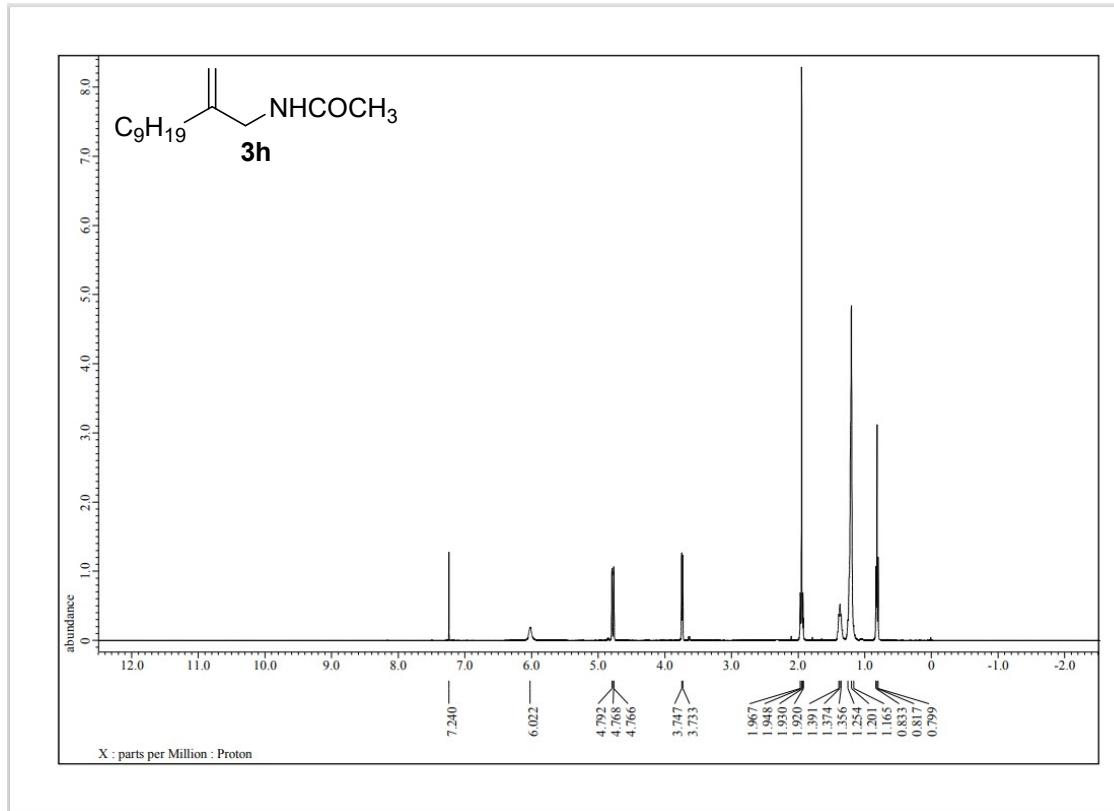
¹⁹F-NMR (376 MHz, CDCl₃) chart of **3g**



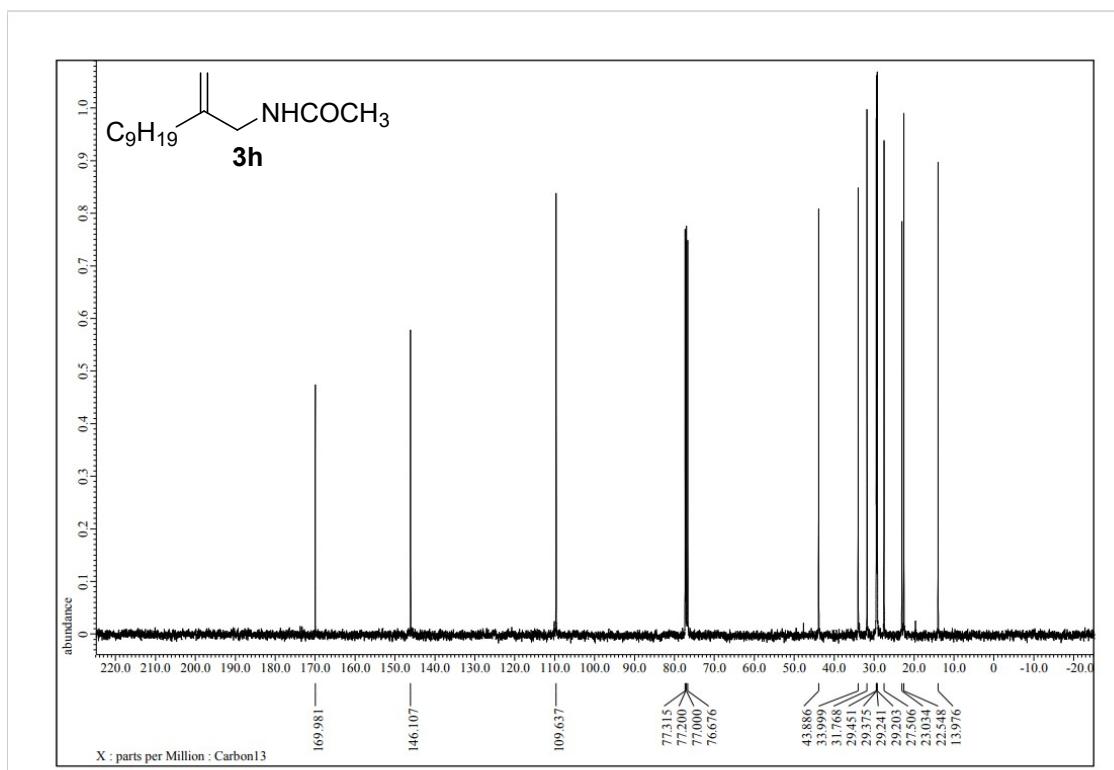
¹³C-NMR (100 MHz, CDCl₃) chart of **3g**



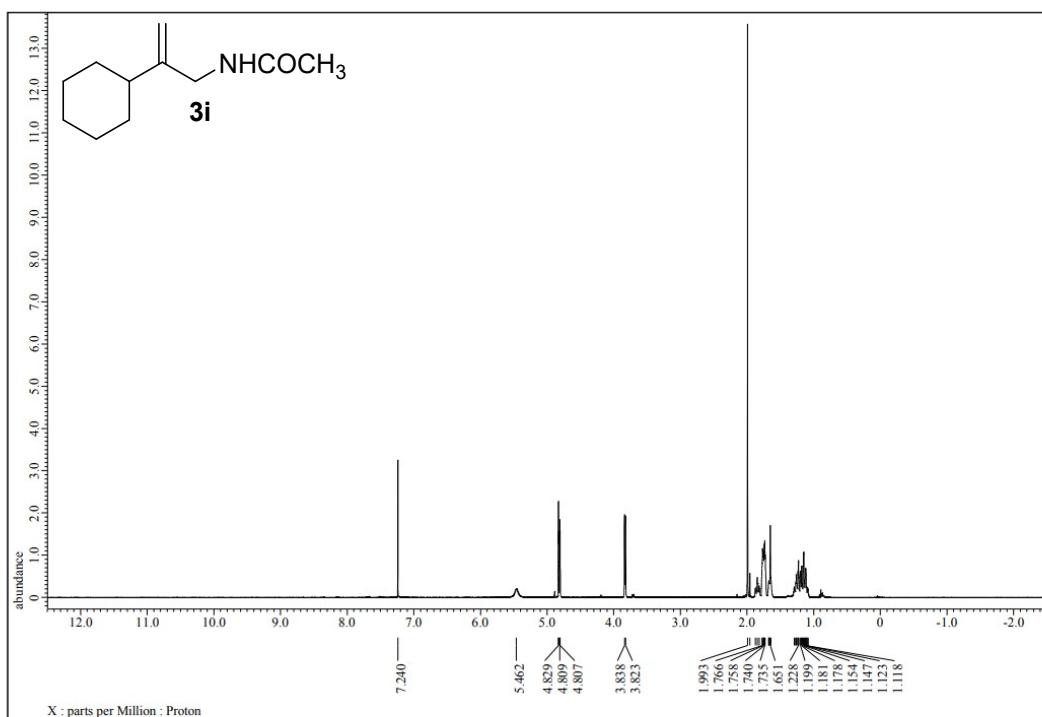
¹H-NMR (400 MHz, CDCl₃) chart of **3h**



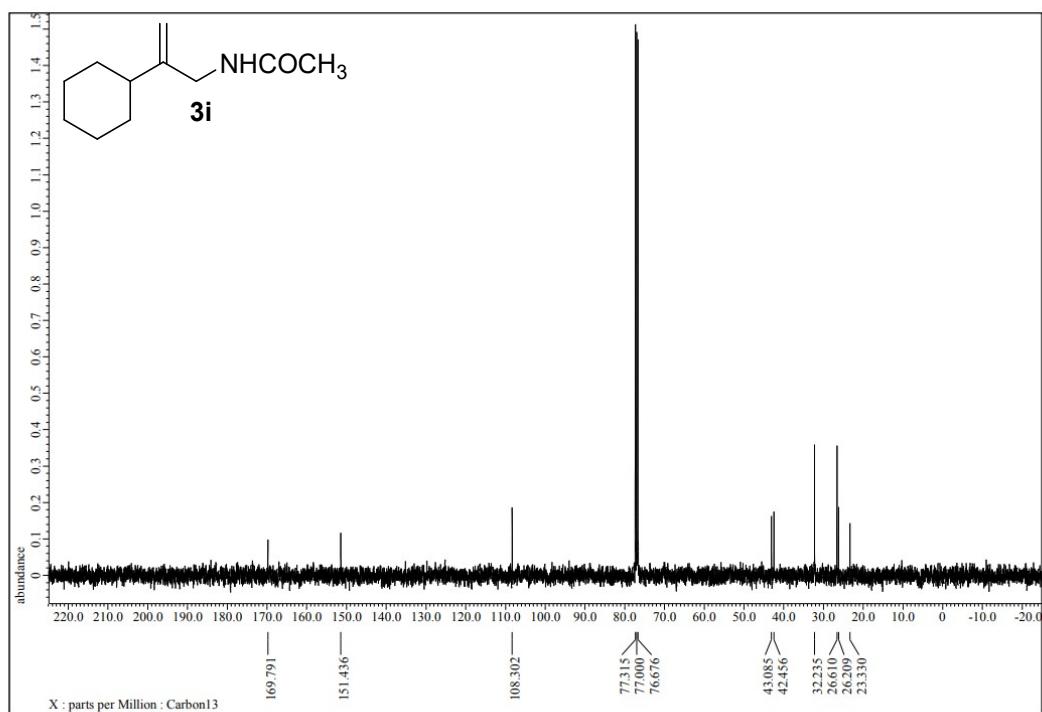
¹³C-NMR (100 MHz, CDCl₃) chart of **3h**



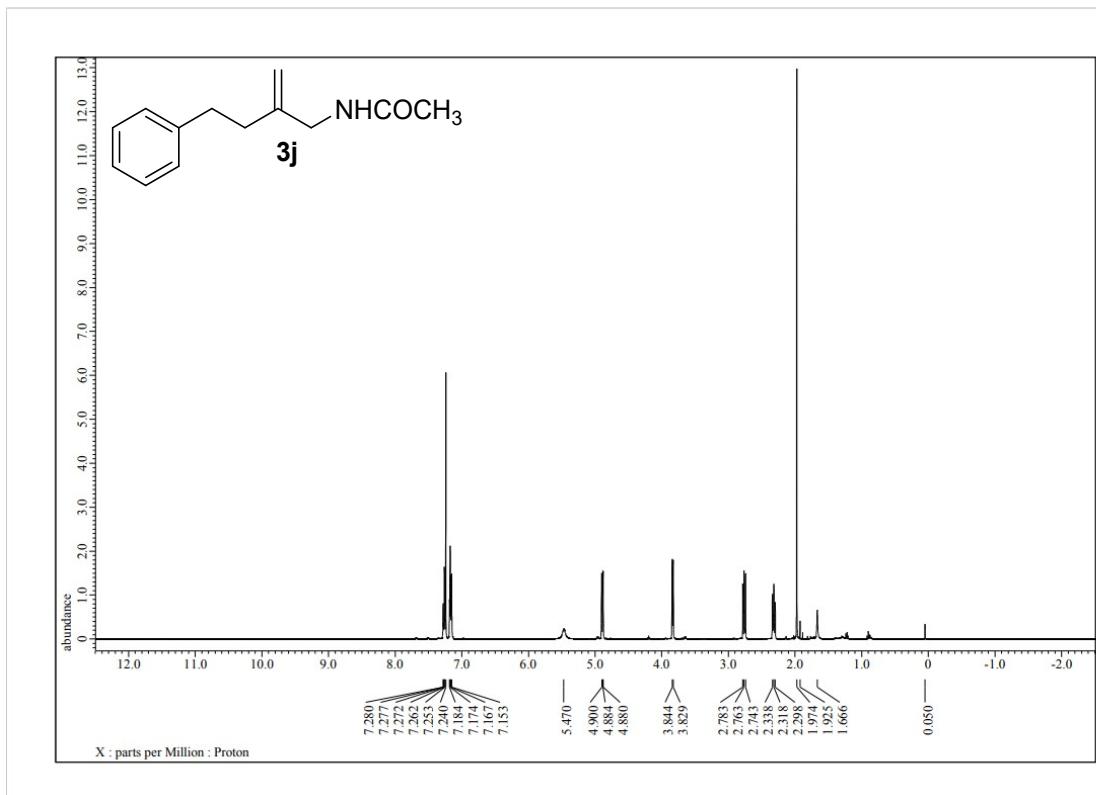
¹H-NMR (400 MHz, CDCl₃) chart of **3i**



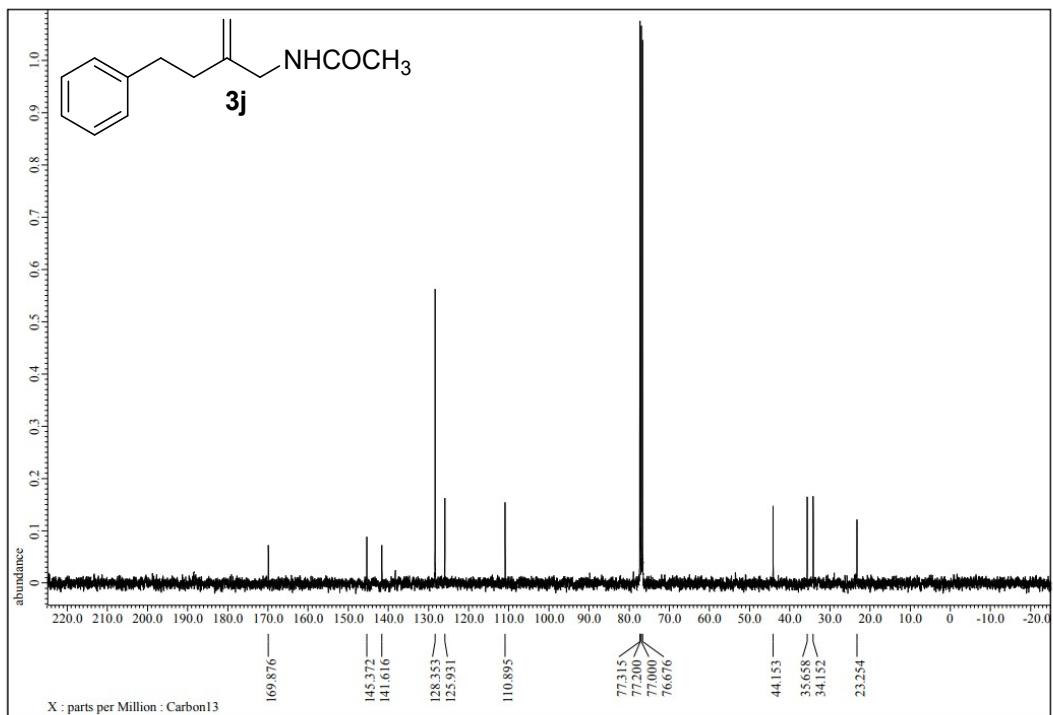
¹³C-NMR (100 MHz, CDCl₃) chart of **3i**



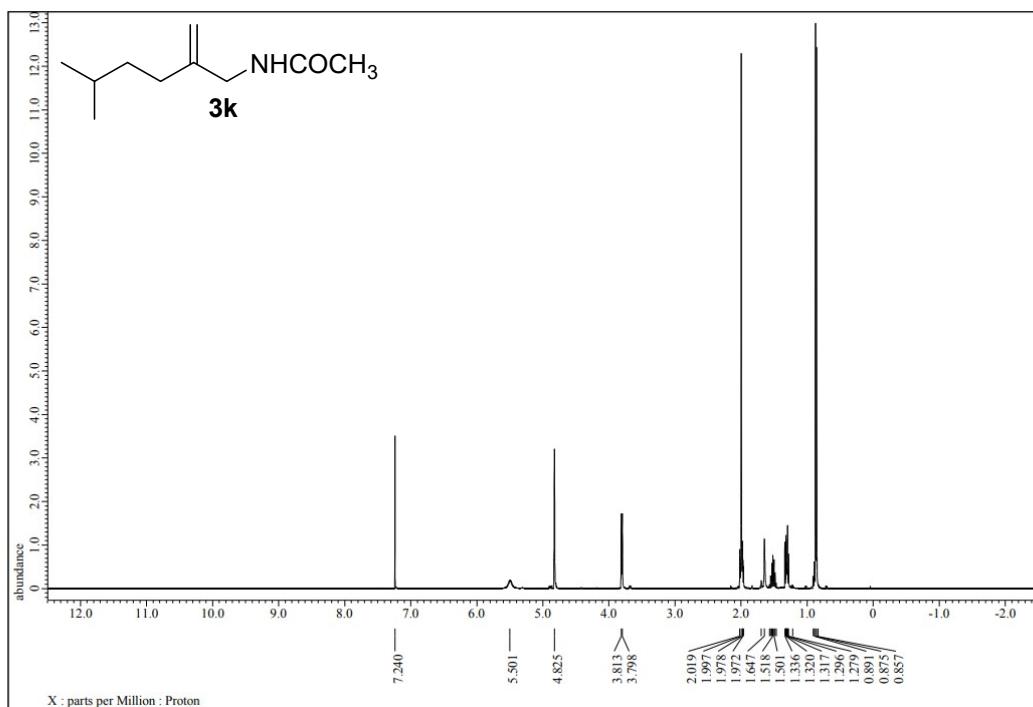
¹H-NMR (400 MHz, CDCl₃) chart of **3j**



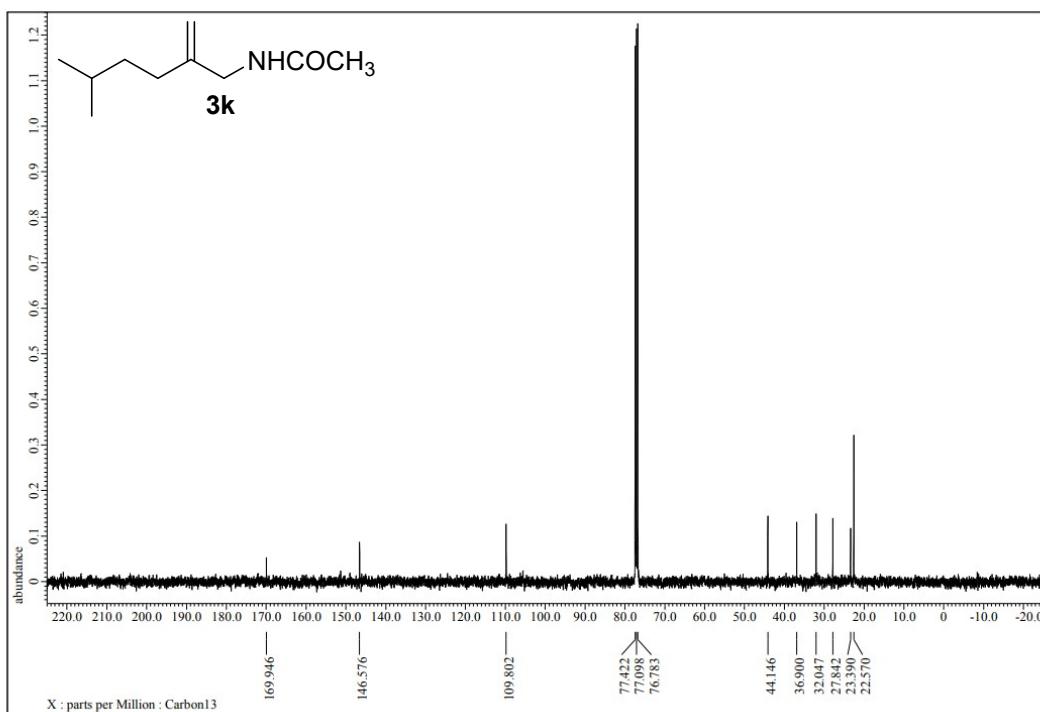
¹³C-NMR (100 MHz, CDCl₃) chart of **3j**



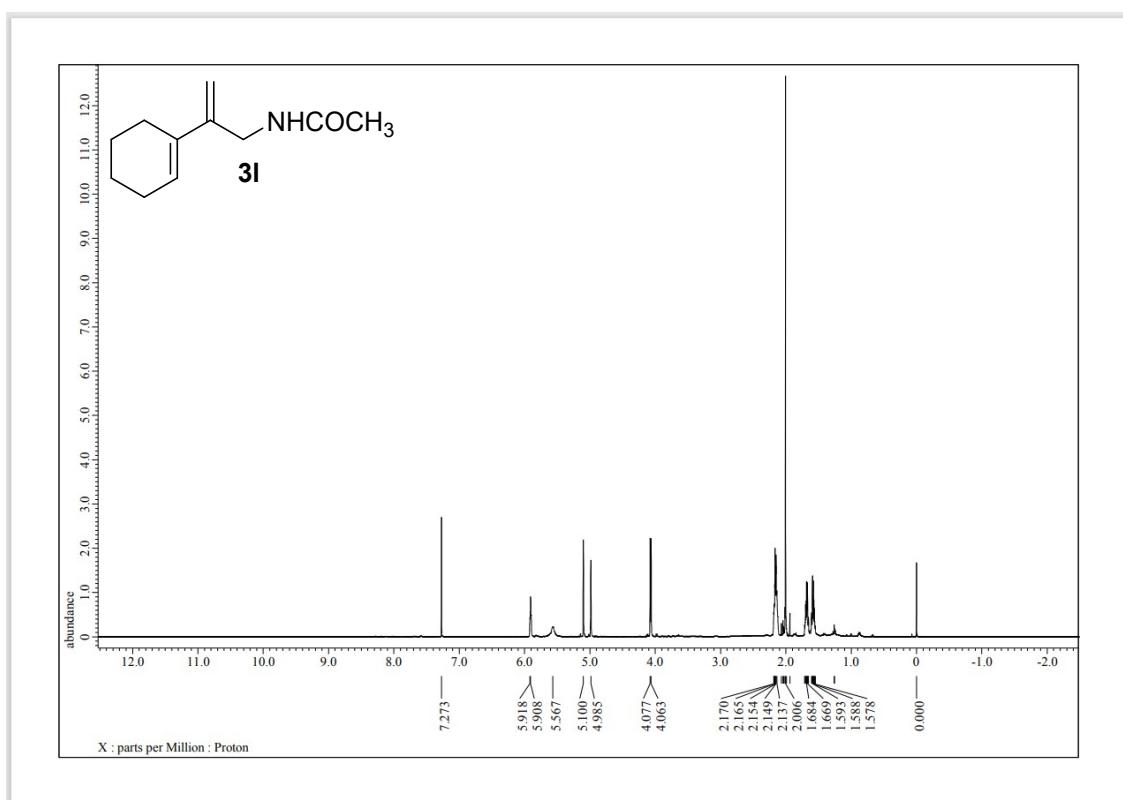
¹H-NMR (400 MHz, CDCl₃) chart of **3k**



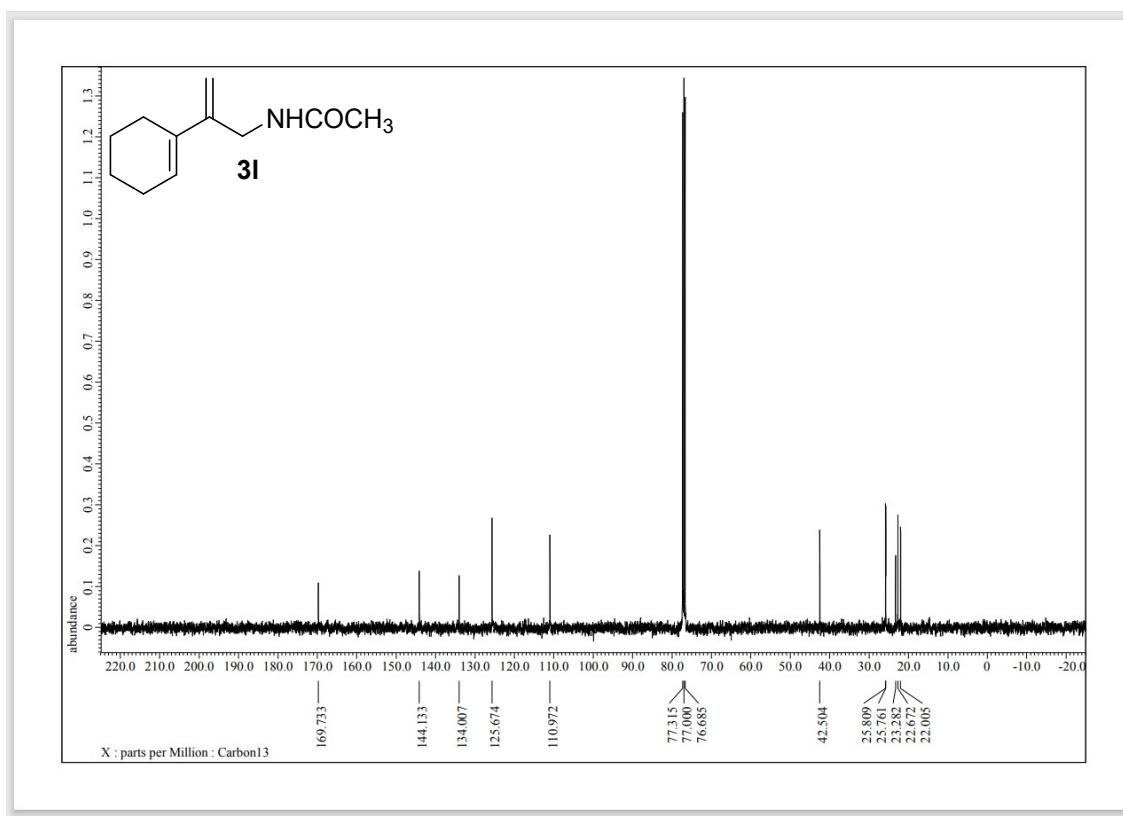
¹³C-NMR (100 MHz, CDCl₃) chart of **3k**



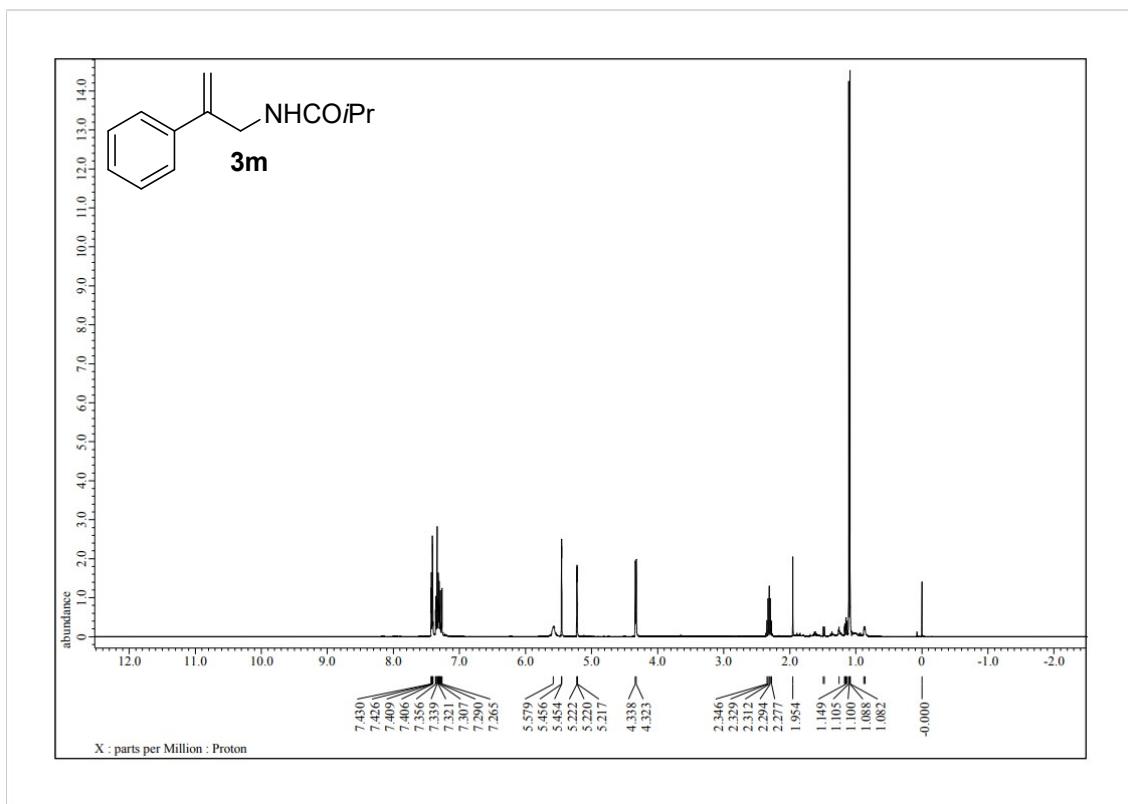
¹H-NMR (400 MHz, CDCl₃) chart of **3l**



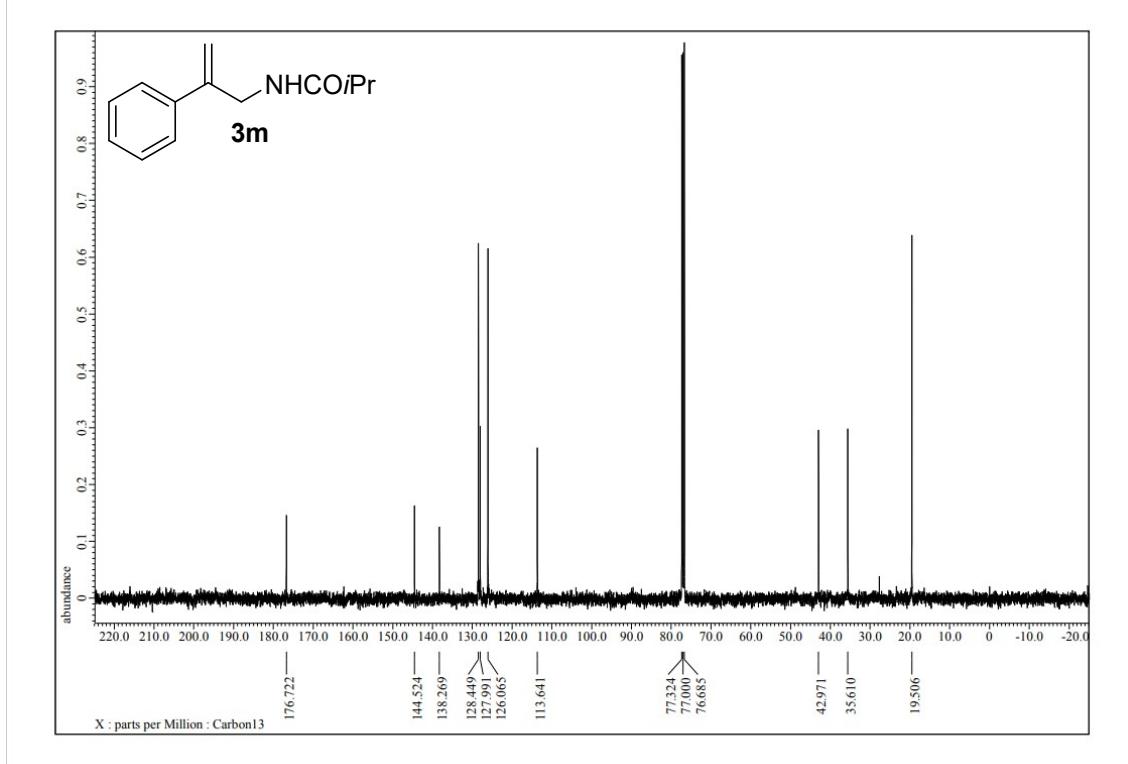
¹³C-NMR (100 MHz, CDCl₃) chart of **3l**



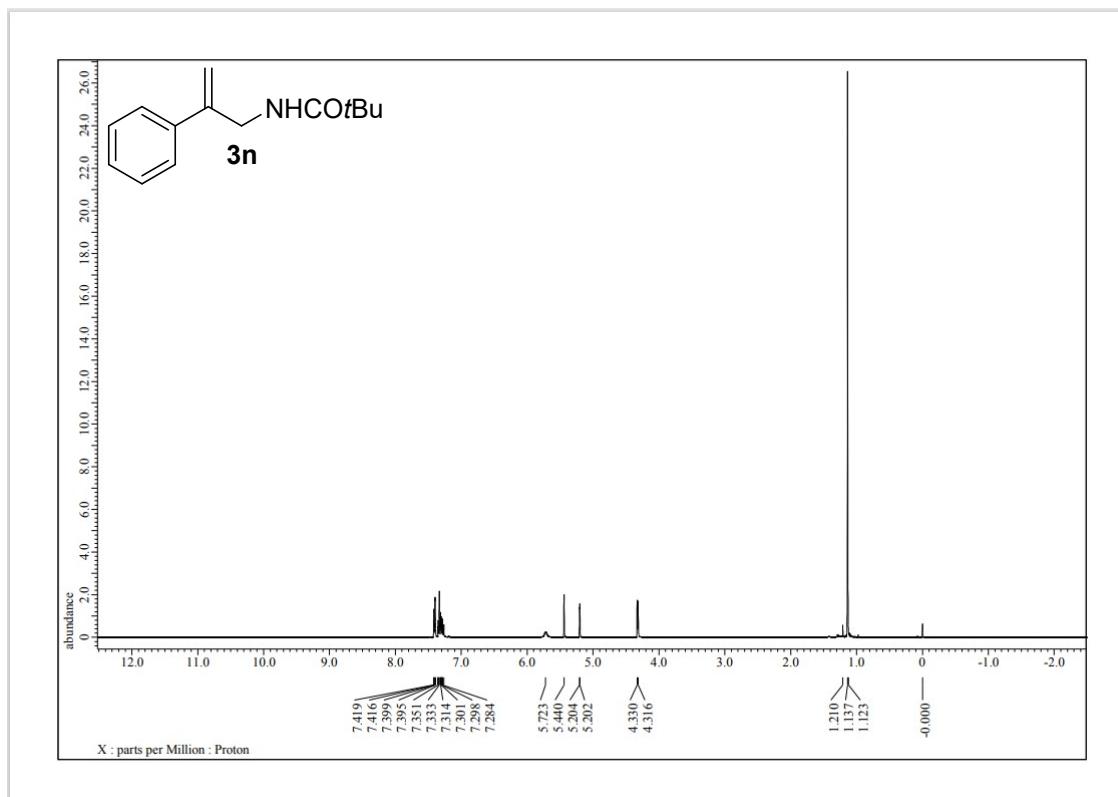
¹H-NMR (400 MHz, CDCl₃) chart of **3m**



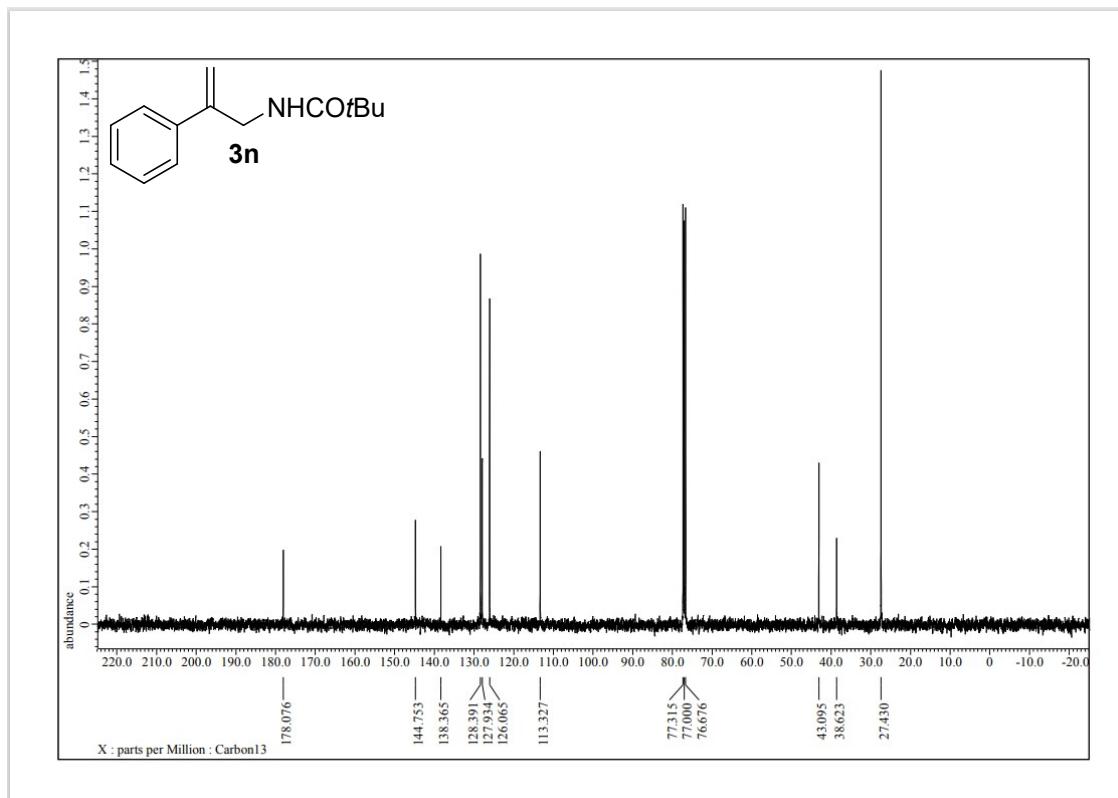
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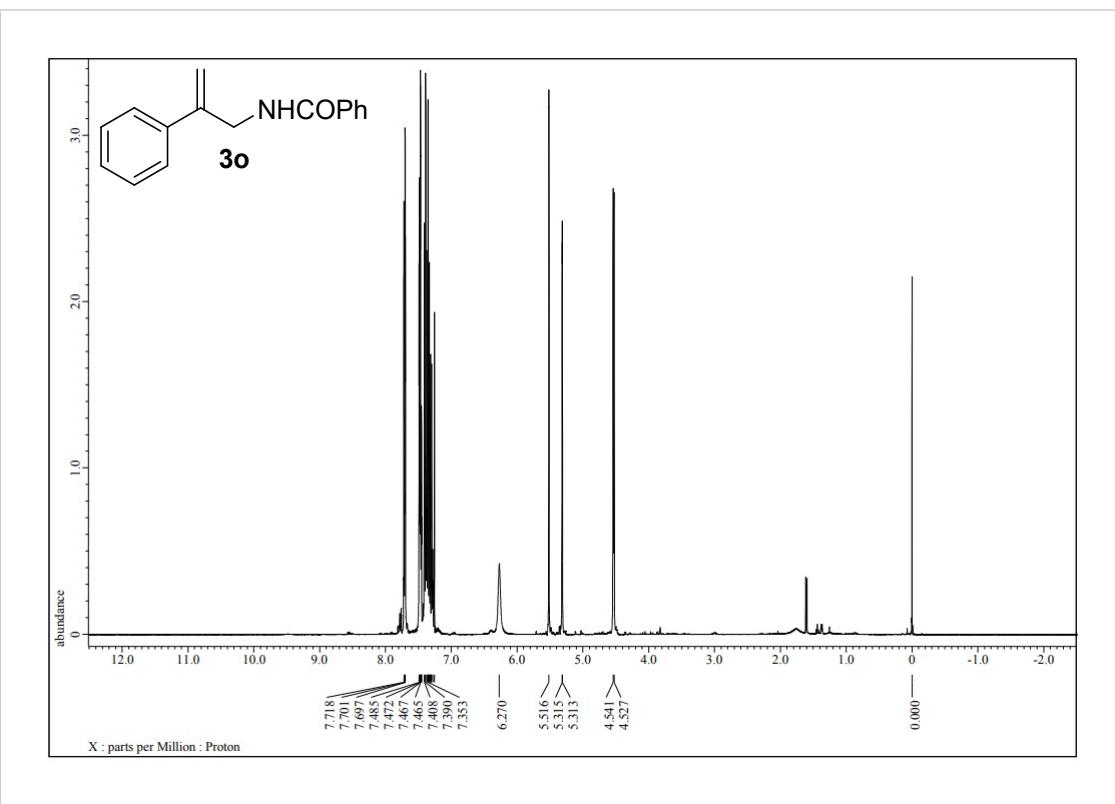
¹H-NMR (400 MHz, CDCl₃) chart of **3n**



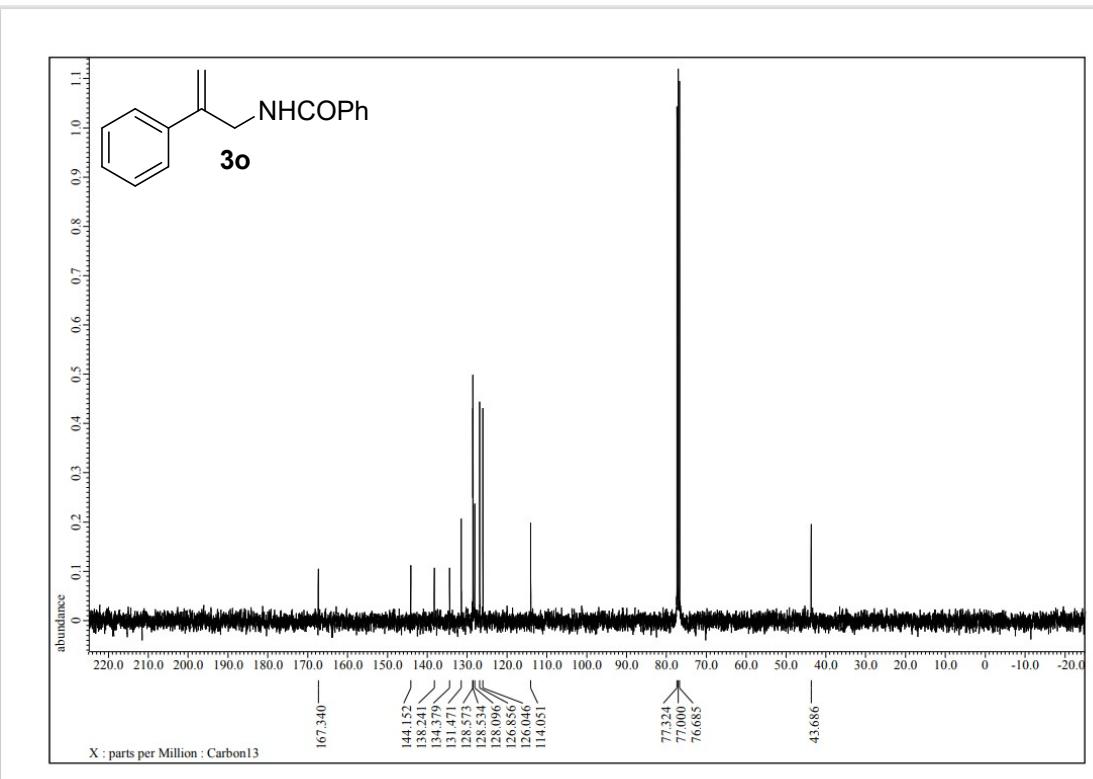
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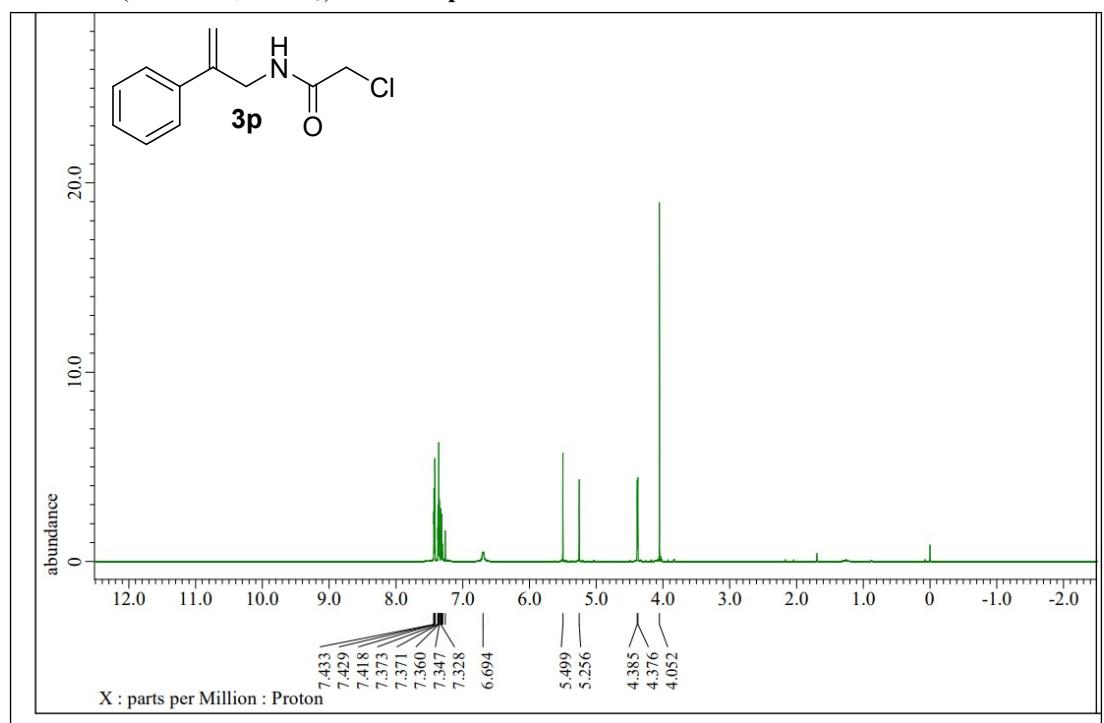
¹H-NMR (400 MHz, CDCl₃) chart of **3o**



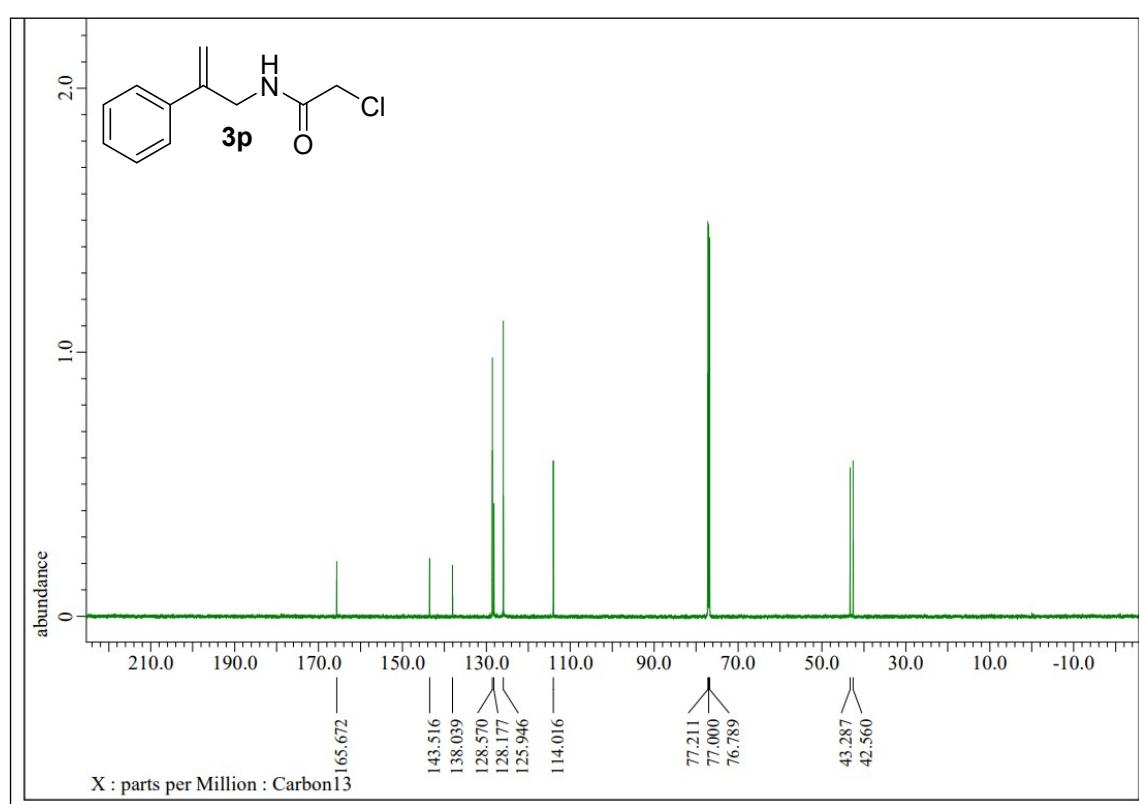
¹³C-NMR (100 MHz, CDCl₃) chart of **3o**



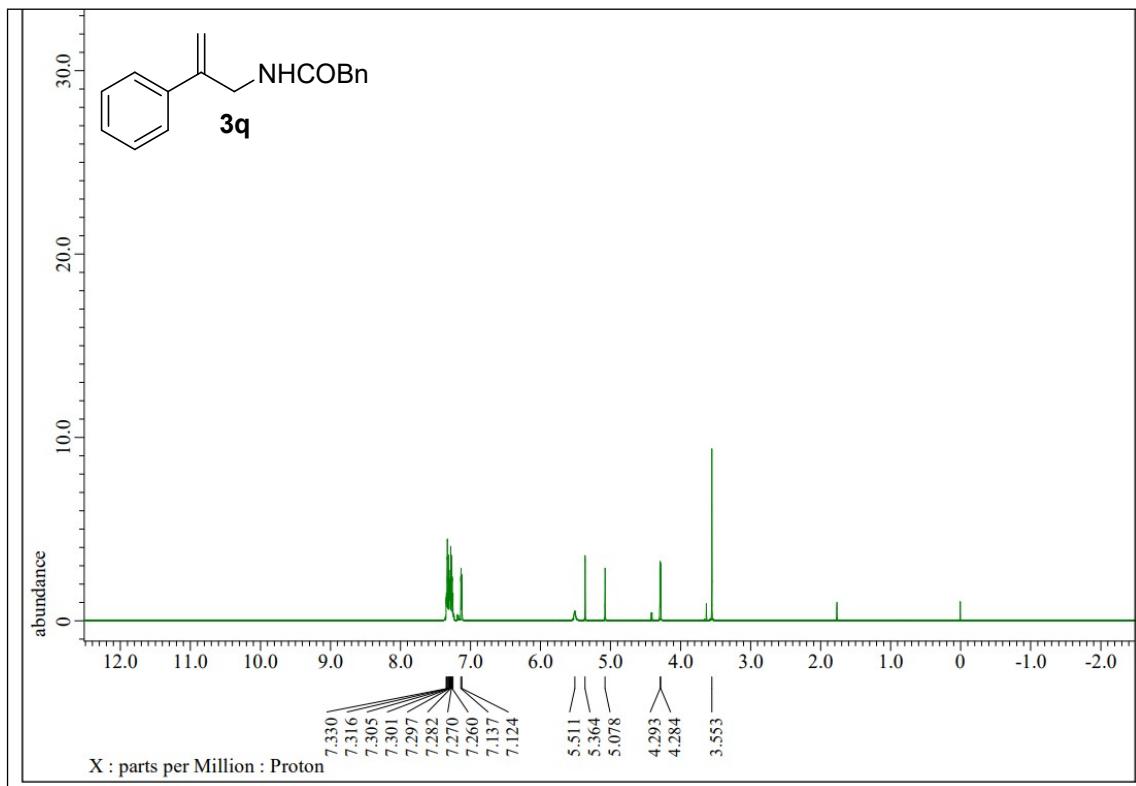
¹H-NMR (600 MHz, CDCl₃) chart of 3p



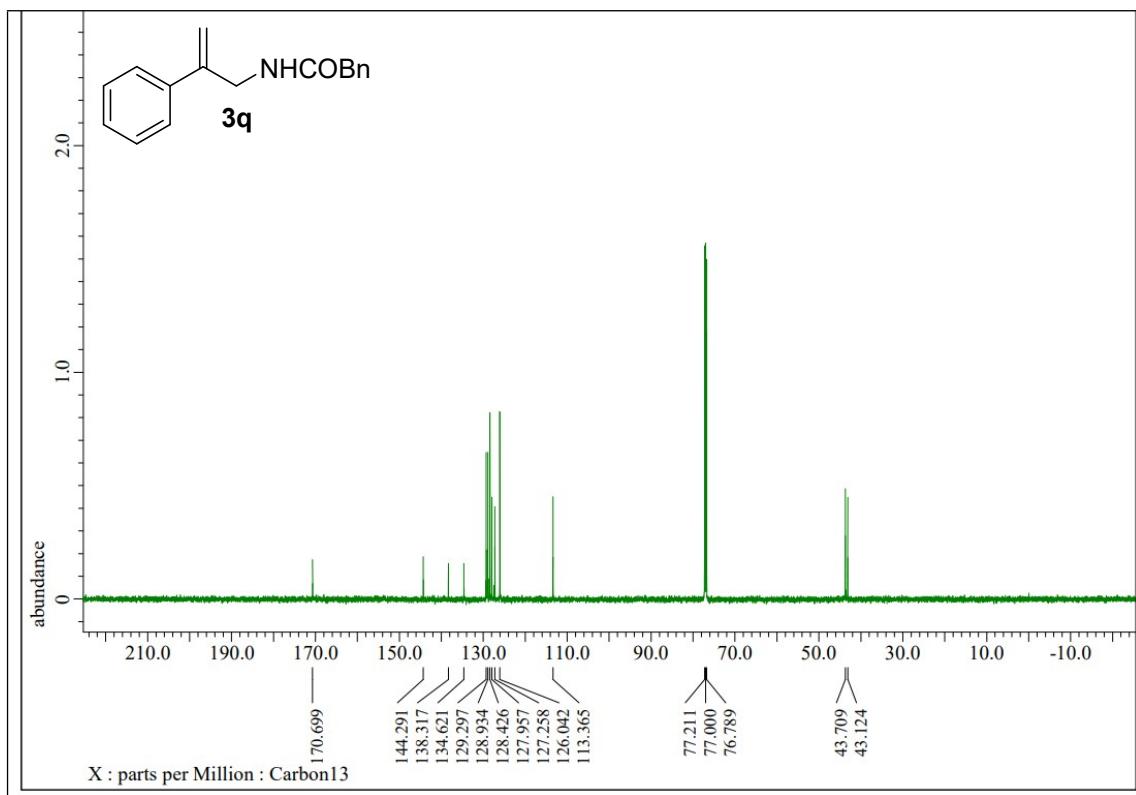
¹³C-NMR (151 MHz, CDCl₃) chart of 3p



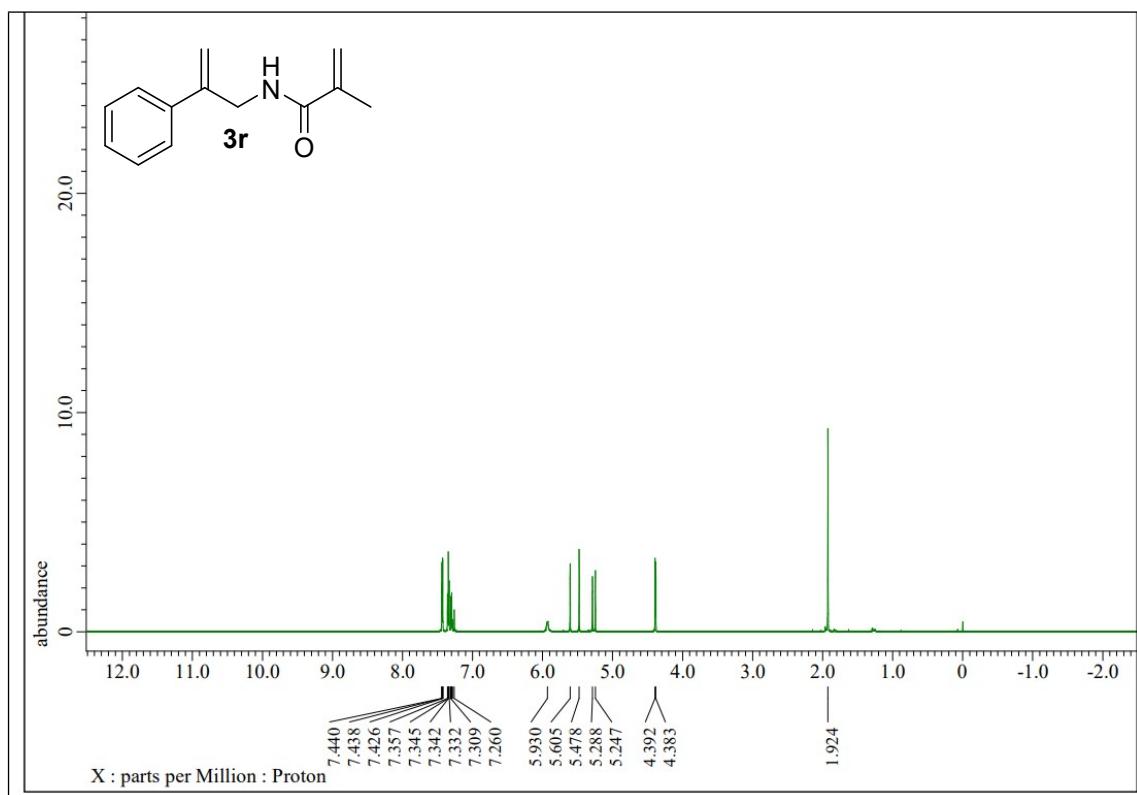
¹H-NMR (600 MHz, CDCl₃) chart of **3q**



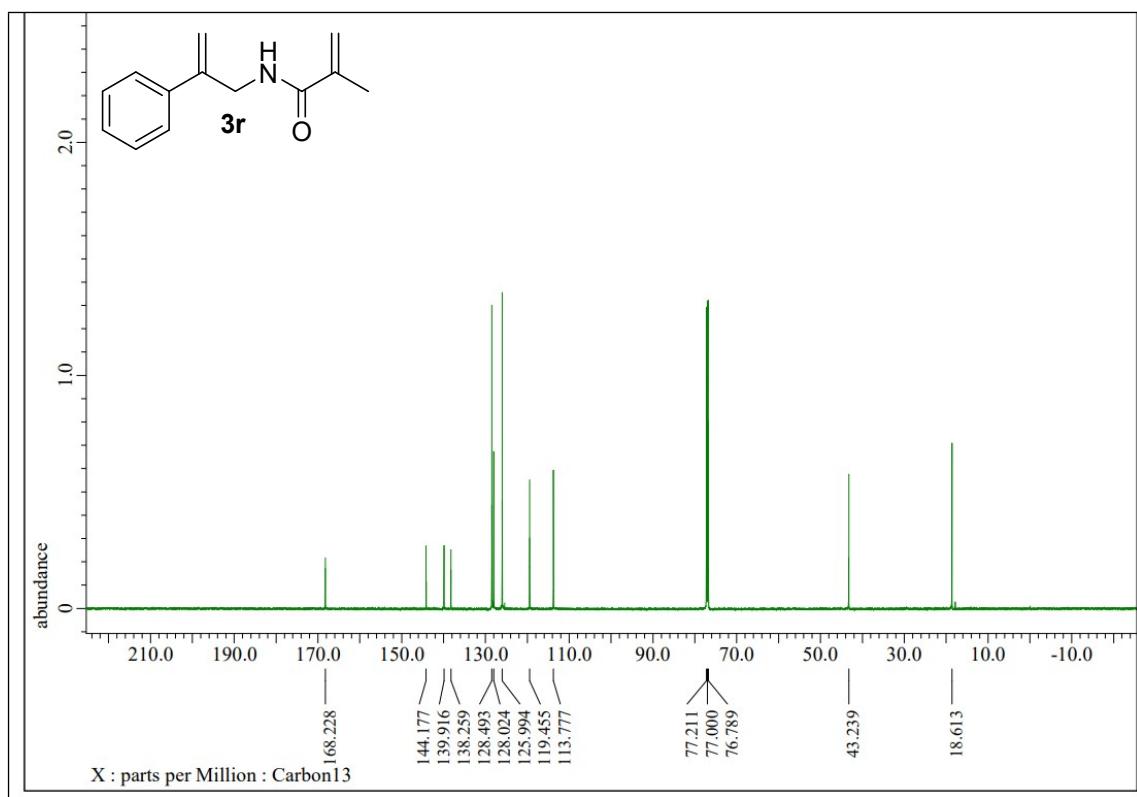
¹³C-NMR (151 MHz, CDCl₃) chart of **3q**



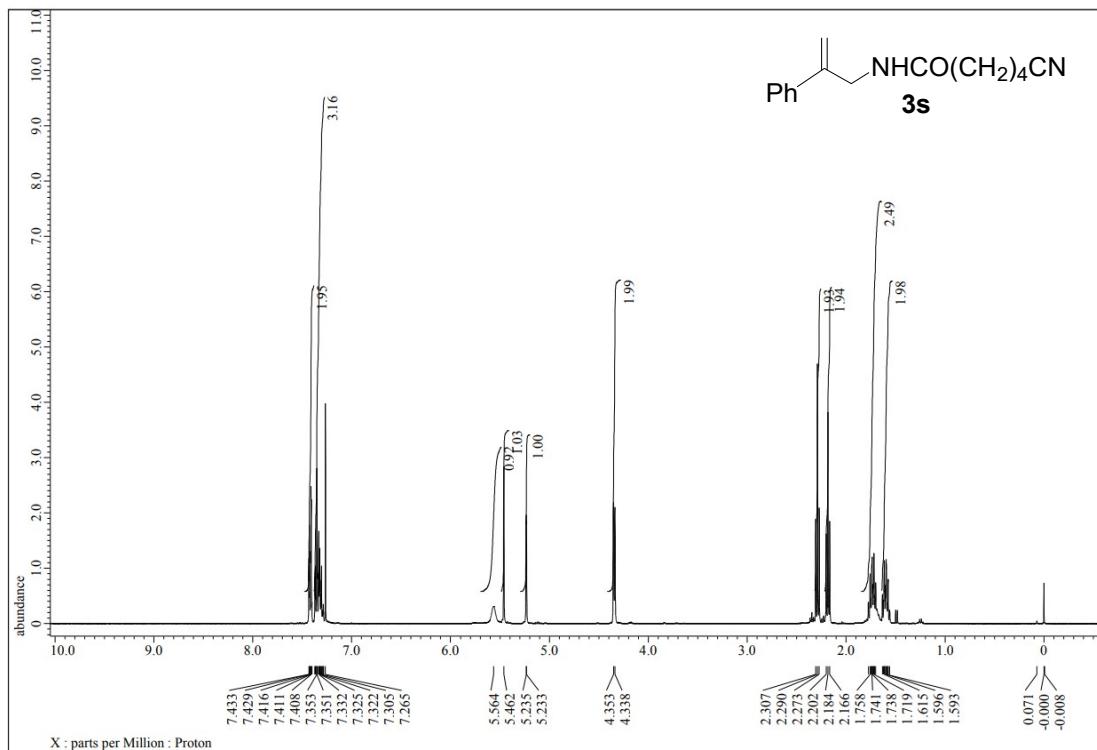
¹H-NMR (600 MHz, CDCl₃) chart of **3r**



¹³C-NMR (151 MHz, CDCl₃) chart of **3r**



¹H-NMR (400 MHz, CDCl₃) chart of **3s**



¹³C-NMR (100 MHz, CDCl₃) chart of **3s**

