



Supporting Information

for

Cross metathesis-mediated synthesis of hydroxamic acid derivatives

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Analytical data of all new compounds as well as copies of their ^1H and ^{13}C NMR spectra

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General information

General: Column chromatography was performed on silica gel, Merck grade 230–400 mesh and neutral alumina. Reactions were monitored by thin-layer chromatography; TLC plates were visualized with UV light, in an iodine chamber, or with vaniline solution, unless noted otherwise. Melting points were recorded in open capillaries and are uncorrected. IR spectra were recorded using KBr disks, chloroform solution or neat. Chemical shifts (δ) are given from TMS (0 ppm) as internal standard for ^1H NMR and $^{13}\text{CDCl}_3$ (77.0 ppm) for ^{13}C NMR. The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = double doublet, ddd = doublet of double doublet, dt = doublet of triplet, br = broad, etc. HRMS data were recorded on a Waters XEVO G2 QTOF instrument purchased through DST-PURSE Grant.

Dichloromethane, dimethyl sulfoxide were distilled over calcium hydride under an inert atmosphere. THF, toluene, benzene and ether were freshly distilled under argon from a purple solution of sodium benzophenone ketyl. Unless stated otherwise, all reagents were purchased from commercial sources and used without additional purification.

(E)-N-(Benzyloxy)tridec-2-enamide (6b)

Eluent: hexane:ethyl acetate (60:40)

Yield: 85%. Colorless viscous liquid.

IR (neat): 3429, 3200, 2924, 2853, 1666, 1634 cm^{-1} .

^1H NMR (400 MHz, DMSO- d_6): δ 11.11 (1H, s, NH), 7.62-7.40 (5H, s, ArH), 6.79-6.71 (1H, m, C3-H), 5.76 (1H, d, J = 14.8, C2-H), 4.86 (2H, s, OCH₂), 2.17 (2H, m, C4-H), 1.42 (3H, brs, CH₂), 1.29 (13H, s, CH₂), 0.90 (3H, t, J = 3.6Hz, C13-H₃).

^{13}C NMR (100 MHz, DMSO- d_6): δ 163.3 (CO), 144.2 (C3), 136.5 (ArC), 129.2 (ArCH), 128.8(ArCH), 128.7(ArCH), 121.3(C2), 77.4 (OCH₂), 31.8(C4), 31.7(C5), 29.5 (CH₂), 29.3(CH₂), 29.2(CH₂), 29.0(CH₂), 28.2(CH₂), 22.6(CH₂), 14.4 (C13).

HRMS (TOF MS ES⁺): m/z [M + Na]⁺ calcd. for C₂₀H₃₁NNaO₂ 340.2252; found 340.2243

(E)-N-(Benzyloxy)-5-bromopent-2-enamide (6c):

Eluent: hexane:ethylacetate (65:35).

Yield: 72%. Colorless viscous liquid.

IR (neat): 3201, 3031, 2963, 2887, 1958, 1667, 1635 cm^{-1} .

NMR (400 MHz, DMSO- d_6): δ 11.27 (1H, s, NH), 7.45-7.39 (5H, m, ArH), 6.77-6.69 (1H, m, C3-H), 5.88 (1H, d, J = 15.2Hz, C2-H), 4.89 (2H, s, OCH₂), 3.66 (2H, t, J = 6.8 Hz, CH₂Br), 3.46 (s, H₂O from DMSO- d_6), 2.77 (2H, q, J = 6.4Hz, C4-H₂).

^{13}C NMR (100 MHz, DMSO- d_6): δ 162.8(CO), 140.9(C3), 136.4(ArC), 129.2(ArCH), 128.8(ArCH), 128.8(ArCH), 123.4(C2), 77.4 (OCH₂), 34.9 (C5), 33.0 (C4).

Anal calcd for C₁₂H₁₄BrNO₂ C, 50.72; H, 4.97; N, 4.93; observed, C, 50.60; H, 4.78; N, 4.71.

(E)-N-(Benzyloxy)-4-phenylbut-2-enamide (6d):

Eluent: hexane:ethylacetate (60:40).

Yield: 77%. Colorless viscous liquid.

IR (neat): 3124, 3028, 2930, 1670, 1642 cm^{-1} .

^1H NMR (400 MHz, DMSO-d_6): δ 11.21 (1H, s, NH), 7.42-7.36 (8H, m, ArH), 7.30-7.24 (2H, m, ArH), 6.88 (1H, m, C3-H), 5.74 (1H, d, $J = 15.6\text{Hz}$, C2-H), 4.85 (2H, s, OCH_2), 3.53 (2H, d, $J = 6.8\text{ Hz}$, ArCH_2).

^{13}C NMR (100 MHz, DMSO-d_6): δ 163.1 (CO), 143.1(C3), 138.9 (ArC), 136.5 (ArC), 129.2 (ArCH), 129.0 (ArCH), 128.8(ArCH), 126.8(ArCH), 122.0 (C2), 77.4 (OCH_2) , 37.9 (C4).

HRMS (TOF MS ES^+): m/z $[\text{M} + \text{Na}]^+$ calcd. for $\text{C}_{17}\text{H}_{17}\text{NNaO}_2$ 290.1157; found 290.1154

(E)-N-(Benzyloxy)-4-(2-methoxyphenyl)but-2-enamide (6e):

Eluent: hexane:ethyl acetate (50:50)

Yield: 72%. Colorless viscous liquid.

IR (neat): 3153, 3028, 2960, 2838, 1805, 1667, 1634 cm^{-1} .

^1H NMR (400 MHz, DMSO-d_6): δ 11.13 (1H, s, NH), 7.37 (5H, brs, ArH), 7.23 (1H, t, $J = 7.6\text{Hz}$, ArH), 7.11 (1H, d, $J = 7.2\text{Hz}$, ArH), 6.98 (1H, d, $J = 8.4\text{Hz}$, ArH), 6.90-6.79 (2H, m, ArH+C3-H), 5.65 (1H, d, $J = 15.6\text{Hz}$, C2-H), 4.80 (2H, s, OCH_2), 3.78 (3H, s, OCH_3), 3.41 (H_2O from DMSO-d_6).

^{13}C NMR (100 MHz, DMSO-d_6): δ 163.2 (CO), 157.3 (O-ArC), 142.5(C3), 136.5(ArC), 130.4(ArC), 129.2(ArCH), 128.8 (ArCH, two signals), 128.5(ArCH), 126.7(ArCH), 121.6 (ArCH), 120.9 (C2), 111.3(ArCH), 77.4 (OCH_2), 55.8(OCH_3), 32.6 (C4).

HRMS (TOF MS ES⁺): m/z [M + Na]⁺ calcd. for C₁₈H₁₉NNaO₃ 320.1263; found 320.1269.

(E)-N-(Benzyloxy)-4-(2-methoxy-9-methyl-9H-carbazol-1-yl)but-2-enamide (6f):

Eluent: hexane:ethylacetate (50:50).

Yield: 78%. Light yellow solid.

M.p-160-161°C.

IR (neat): 3232, 2997, 2933, 1661, 1634 cm⁻¹.

¹H NMR (400 MHz, DMSO-d₆): δ 11.01 (1H, s, NH), 8.01 (2H, d, J = 7.6Hz, ArH), 7.48 (1H, d, J = 8Hz, ArH), 7.38-7.31 (6H, m, ArH), 7.17-7.07 (2H, m, ArH+C3-H), 6.98 (1H, d, J = 8Hz, ArH), 5.41 (1H, d, J = 15.6 Hz, C2-H), 4.73 (2H, s, OCH₂), 4.02 (2H, brs, ArCH₂), 3.91 (3H, s, OMe), 3.85 (3H, s, NMe).

¹³C NMR (100 MHz, DMSO-d₆): δ 163.7 (CO), 156.8 (OArC), 145.0 (ArC), 142.1 (C3), 140.4 (ArC), 135.8 (ArC), 129.2 (ArCH), 128.9 (ArCH), 128.8 (ArCH), 125.4 (ArCH), 122.5(ArC), 120.9 (ArC), 119.7 (ArCH), 119.6 (ArCH), 119.4 (ArCH), 118.1, 109.3(ArCH), 108.2 (ArC), 104.9(ArCH), 77.4 (OCH₂), 57.0 (OCH₃), 32.0 (NCH₃), 27.0 (C4).

HRMS (TOF MS ES⁺): m/z [M + Na]⁺ calcd for C₂₅H₂₄N₂NaO₃ 423.1685; found 423.1678

(E)-N-(Benzyloxy)-3-(4-methoxyphenyl)acrylamide (6g):

Eluent: hexane: ethyl acetate (50:50).

Yield: 57%. Colourless viscous liquid.

IR (neat): 3135, 2957, 2927, 2854, 2046, 1882, 1665, 1647 cm^{-1} .

^1H NMR (400 MHz, DMSO-d_6): δ 11.25 (1H, s, NH), 7.58-7.39 (7H, m, ArH), 7.02 (2H, d, J = 8.8 Hz, Ar H), 6.34 (1H, d, J = 15.6 Hz, C3-H), 4.91 (2H, s, OCH_2), 3.82 (3H, s, OCH_3), 3.43 (H₂O from DMSO-d_6).

^{13}C NMR (100 MHz, DMSO-d_6): δ 163.9 (CO), 161.0 (OArC), 139.8 (C3), 136.5 (ArC), 129.8 (ArCH), 129.3 (ArCH), 128.8 (ArCH, two signals), 127.6 (ArCH), 116.4 (C2), 114.8 (ArCH), 77.5 (OCH_2), 55.7 (OCH_3).

HRMS (TOF MS ES⁺): m/z $[\text{M} + \text{Na}]^+$ calcd. for $\text{C}_{17}\text{H}_{17}\text{NNaO}_3$ 306.1106; found 306.1115

(E)-Benzyl 6-(benzyloxyamino)-6-oxohex-4-enoate (6h):

Eluent: hexane:ethylacetate (60:40).

Yield: 79%. Colorless viscous liquid.

IR (neat): 3189, 3064, 3032, 2960, 1735, 1671, 1638 cm^{-1} .

^1H NMR (400 MHz, DMSO-d_6): δ 11.09 (1H, s, NH), 7.34-7.24 (10H, m, ArH), 6.65 (1H, dt, J = 15.6, 6.8 Hz, C4-H), 5.69 (1H, d, J = 15.6 Hz, C5-H), 5.03 (2H, s, $\text{CO}_2\text{CH}_2\text{Ph}$), 4.75 (2H, s, OCH_2Ph), 2.48-2.43 (3H, m, C2-H+residual DMSO), 2.35 (2H, q, J = 6.8 Hz, C3-H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 172.4 (CO_2Bn), 163.0 (CONHOBn), 142.4 (C4), 136.6 (ArC), 136.5 (ArC), 129.2 (ArCH), 128.9 (ArCH), 128.8 (ArCH), 128.7 (ArCH), 128.5 (ArCH), 128.4 (ArCH), 122.0 (C5), 77.4 ($\text{NHO-CH}_2\text{Ph}$), 66.0 ($\text{COO-CH}_2\text{Ph}$), 32.5 (C2), 27.2 (C3).

HRMS (TOF MS ES⁺): m/z $[\text{M} + \text{Na}]^+$ calcd. for $\text{C}_{20}\text{H}_{21}\text{NNaO}_4$ 362.1368; found 362.1360

(E)-Benzyl 7-(benzyloxyamino)-7-oxohept-5-enoate (6i):

Eluent: hexane:ethyl acetate (60:40).

Yield: 73%. Colorless viscous liquid.

IR (neat): 3381, 3196, 3032, 2928, 1732, 1670, 1641 cm^{-1} .

^1H NMR (400 MHz, DMSO-d_6): δ 11.13 (1H, s, NH), 7.39-7.26 (10H, m, ArH), 6.73-6.08 (1H, m, C5-H), 5.74 (1H, d, $J = 15.6\text{Hz}$, C6-H), 5.09 (2H, s, $\text{CO}_2\text{CH}_2\text{Ph}$), 4.82 (2H, s, $\text{CONHOCH}_2\text{Ph}$), 2.37 (2H, t, $J = 7.6\text{Hz}$, C2-H), 2.16 (2H, q, $J = 6.4\text{Hz}$, C4-H), 1.71-1.65 (2H, m, C3-H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 172.9 (C1), 163.2 (C7), 143.3 (C5), 136.7(ArC), 136.5(ArC), 129.2 (ArCH), 128.9 (ArCH), 128.8 (ArCH), 128.7 (ArCH), 128.5 (ArCH), 128.4 (ArCH), 121.8 (C6), 77.4($\text{CONHOCH}_2\text{Ph}$), 65.9 ($\text{CO}_2\text{CH}_2\text{Ph}$), 33.3(C2), 31.1(C4), 23.5(C3).

HRMS (TOF MS ES⁺): m/z $[\text{M} + \text{Na}]^+$ calcd. for $\text{C}_{21}\text{H}_{23}\text{NNaO}_4$ 376.1525; found 376.1519.

(E)-Benzyl 8-(benzyloxyamino)-8-oxooct-6-enoate (6j):

Eluent: hexane:ethylacetate (60:40).

Yield: 70%. Colorless viscous liquid.

IR (neat): 3186, 3032, 2933, 1732, 1667, 1533 cm^{-1} .

^1H NMR (400 MHz, DMSO-d_6): δ 11.16 (1H, s, NH), 7.43-7.35 (10H, m, ArH), 6.77-6.69 (1H, m, C6-H), 5.76 (1H, d, $J=15.2\text{Hz}$, C7-H), 5.12 (2H, s, $\text{CO}_2\text{CH}_2\text{Ph}$), 4.86 (2H, s,

CONHOCH₂Ph), 2.42 (2H, t, $J = 7.6\text{Hz}$, C2-H), 2.17 (2H, q, $J = 6\text{Hz}$, C5-H), 1.62-1.54 (2H, m, C4-H), 1.46-1.39 (2H, m, C3-H).

¹³C NMR (100 MHz, DMSO-d₆): δ 173.1 (C1), 163.3 (C8), 143.9 (C6), 136.7(ArC), 136.5(ArC), 129.2 (ArCH), 128.9 (ArCH), 128.8 (ArCH), 128.7(ArCH), 128.4(ArCH), 128.4(ArCH), 121.4 (C7), 77.4 (CONHOCH₂Ph), 65.8 (CO₂CH₂Ph), 33.7(C2), 31.4(C5), 27.5(C3), 24.4(C4).

HRMS (TOF MS ES⁺): m/z [M + Na]⁺ calcd. for C₂₂H₂₅NNaO₄ 390.1681; found 390.1684

(*S,E*)-Methyl 7-(benzyloxyamino)-2-((*tert*-butoxycarbonyl)amino)-7-oxohept-5-enoate (6k) :

Eluent: hexane : ethyl acetate (60:40).

Yield: 78%. Colorless viscous liquid.

$[\alpha]_D^{25} = +20.4$ (c 1.1, CHCl₃).

IR (neat): 3366, 3237, 2977, 2953, 2869, 1726, 1714, 1654 cm⁻¹.

¹H NMR (400 MHz, DMSO-d₆): δ 11.23 (1H, brs, NH), 7.45-7.41 (6H, m, ArH+NHBOC), 6.70 (1H, m, C5-H), 5.82 (1H, d, $J = 15.6\text{Hz}$, C6-H), 4.87 (2H, s, OCH₂Ph), 4.14 (1H, m, C2-H), 3.68 (3H, s, CO₂Me), 3.42 (H₂O from DMSO-d₆), 2.56-2.47 (4H, m, merged, C3-H+C4-H) 1.44 (9H, s, O-CMe₃).

¹³C NMR (100 MHz, CDCl₃): δ 172.6 (C1), 162.8 (C7), 155.9 (COOCMe₃), 139.5 (C5), 136.4 (ArC), 129.2 (ArCH), 128.7 (ArCH, two signals), 123.8 (C6), 78.9 (OCMe₃), 77.4 (CONHOCH₂Ph), 53.3 (CO₂Me), 52.3(C2), 33.6(C4), 28.3(OCMe₃ + C-3, two signals).

HRMS (TOF MS ES⁺): m/z [M + Na]⁺ calcd. for C₂₀H₂₈N₂NaO₆ 415.1845; found 415.1857.

(S,E)-Methyl 8-(benzyloxyamino)-2-((tert-butoxycarbonyl)amino)-8-oxooct-6-enoate (6l):

Eluent-hexane : ethyl acetate (60:40).

Yield: 75%. Colorless viscous liquid.

$[\alpha]_D^{25} = +11.0$ (c 0.2, CHCl₃).

IR (neat): 3447, 3267, 2977, 2753, 2869, 1757, 1714, 1644 cm⁻¹.

¹H NMR (400 MHz, CDCl₃): δ 8.76 (1H, brs, NHOBn), 7.31-7.28 (5H, m, ArH), 6.85-6.78 (1H, m, C6-H), 5.63 (1H, brs, NHBoc), 5.05 (1H, m, C7-H), 4.83 (2H, s, OCH₂Ph), 4.23-4.18 (1H, m, C2-H), 3.65 (3H, s, CO₂Me), 2.12 (2H, s, C5-H), 1.70 (1H, brs, C3-H_aH_b), 1.56-1.49 (1H, m, C3-H_aH_b), 1.46-1.39 (2H, m, C4-H), 1.36 (9H, s, CO₂CMe₃).

¹³C NMR (100 MHz, CDCl₃): δ 173.2 (C1), 164.6 (C8), 155.4 (COOCMe₃), 145.2 (C6), 135.5 (ArC), 129.2 (ArCH), 128.8 (ArCH), 128.6 (ArCH), 120.2 (C7), 80.0 (OCMe₃), 78.3 (CONHOCH₂Ph), 53.1 (CO₂Me), 52.3 (C2), 32.2 (C5), 31.6 (C3), 28.3 (OCMe₃), 23.8 (C4).

HRMS (TOF MS ES⁺): m/z [M + Na]⁺ calcd. for C₂₁H₃₀N₂NaO₆ 429.2002; found 429.2009

HRMS (TOF MS ES⁺): m/z [M + Na]⁺ calcd. for C₂₁H₂₃NNaO₄ 376.1525; found 376.1519.

(E)-Benzyl 8-(benzyloxyamino)-8-oxooct-6-enoate (6j):

Eluent: hexane:ethylacetate (60:40).

Yield: 70%. Colorless viscous liquid.

IR (neat): 3186, 3032, 2933, 1732, 1667, 1533 cm⁻¹.

^1H NMR (400 MHz, DMSO- d_6): δ 11.16 (1H, s, NH), 7.43-7.35 (10H, m, ArH), 6.77-6.69 (1H, m, C6-H), 5.76 (1H, d, $J=15.2\text{Hz}$, C7-H), 5.12 (2H, s, $\text{CO}_2\text{CH}_2\text{Ph}$), 4.86 (2H, s, $\text{CONHOCH}_2\text{Ph}$), 2.42 (2H, t, $J = 7.6\text{Hz}$, C2-H), 2.17 (2H, q, $J = 6\text{Hz}$, C5-H), 1.62-1.54 (2H, m, C4-H), 1.46-1.39 (2H, m, C3-H).

^{13}C NMR (100 MHz, DMSO- d_6): δ 173.1 (C1), 163.3 (C8), 143.9 (C6), 136.7(ArC), 136.5(ArC), 129.2 (ArCH), 128.9 (ArCH), 128.8 (ArCH), 128.7(ArCH), 128.4(ArCH), 128.4(ArCH), 121.4 (C7), 77.4 ($\text{CONHOCH}_2\text{Ph}$), 65.8 ($\text{CO}_2\text{CH}_2\text{Ph}$), 33.7(C2), 31.4(C5), 27.5(C3), 24.4(C4).

HRMS (TOF MS ES $^+$): m/z $[\text{M} + \text{Na}]^+$ calcd. for $\text{C}_{22}\text{H}_{25}\text{NNaO}_4$ 390.1681; found 390.1684

(*S,E*)-Methyl 7-(benzyloxyamino)-2-((*tert*-butoxycarbonyl)amino)-7-oxohept-5-enoate (6k):

Eluent: hexane : ethyl acetate (60:40).

Yield: 78%. Colorless viscous liquid.

$[\alpha]_{\text{D}}^{25} = +20.4$ (c 1.1, CHCl_3).

IR (neat): 3366, 3237, 2977, 2953, 2869, 1726, 1714, 1654 cm^{-1} .

^1H NMR (400 MHz, DMSO- d_6): δ 11.23 (1H, brs, NH), 7.45-7.41 (6H, m, ArH+NHBoc), 6.70 (1H, m, C5-H), 5.82 (1H, d, $J = 15.6\text{Hz}$, C6-H), 4.87 (2H, s, OCH_2Ph), 4.14 (1H, m, C2-H), 3.68 (3H, s, CO_2Me), 3.42 (H_2O from DMSO- d_6), 2.56-2.47 (4H, m, merged, C3-H+C4-H) 1.44 (9H, s, O-CMe $_3$).

^{13}C NMR (100 MHz, DMSO- d_6): δ 172.6 (C1), 162.8 (C7), 155.9 (COOCMe_3), 139.5 (C5), 136.4 (ArC), 129.2 (ArCH), 128.7 (ArCH, two signals), 123.8 (C6), 78.9 (OCMe_3), 77.4 ($\text{CONHOCH}_2\text{Ph}$), 53.3 (CO_2Me), 52.3(C2), 33.6(C4), 28.3(OCMe_3 + C-3, two signals).

HRMS (TOF MS ES $^+$): m/z $[\text{M} + \text{Na}]^+$ calcd. for $\text{C}_{20}\text{H}_{28}\text{N}_2\text{NaO}_6$ 415.1845; found 415.1857.

(S,E)-Methyl 8-(benzyloxyamino)-2-((tert-butoxycarbonyl)amino)-8-oxooct-6-enoate (6l):

Eluent- hexane : ethyl acetate (60:40).

Yield: 75%. Colorless viscous liquid.

$[\alpha]_D^{25} = +11.0$ (c 0.2, CHCl_3).

IR (neat): 3447, 3267, 2977, 2753, 2869, 1757, 1714, 1644 cm^{-1} .

^1H NMR (400 MHz, CDCl_3): δ 8.76 (1H, brs, NHOBn), 7.31-7.28 (5H, m, ArH), 6.85-6.78 (1H, m, C6-H), 5.63 (1H, brs, NHBoc), 5.05 (1H, m, C7-H), 4.83 (2H, s, OCH_2Ph), 4.23-4.18 (1H, m, C2-H), 3.65 (3H, s, CO_2Me), 2.12 (2H, s, C5-H), 1.70 (1H, brs, C3- H_aH_b), 1.56-1.49 (1H, m, C3- H_aH_b), 1.46-1.39 (2H, m, C4-H), 1.36 (9H, s, CO_2CMe_3).

^{13}C NMR (100 MHz, CDCl_3): δ 173.2 (C1), 164.6 (C8), 155.4(COOCMe_3), 145.2 (C6), 135.5(ArC), 129.2(ArCH), 128.8(ArCH), 128.6(ArCH), 120.2 (C7), 80.0 (OCMe_3), 78.3($\text{CONHOCH}_2\text{Ph}$), 53.1(CO_2Me), 52.3(C2), 32.2 (C5), 31.6 (C3), 28.3 (OCMe_3), 23.8 (C4).

HRMS (TOF MS ES $^+$): m/z $[\text{M} + \text{Na}]^+$ calcd. for $\text{C}_{21}\text{H}_{30}\text{N}_2\text{NaO}_6$ 429.2002; found 429.2009

N-Hydroxytridecanamide (7b):

Eluent: CHCl_3 : MeOH (97:3).

Yield: 83%. Colorless solid.

M.p 94°C.

IR (neat): 3259, 3056, 2914, 2847, 1662, 1625 cm^{-1} .

^1H NMR (400 MHz, DMSO- d_6): δ 10.41 (1H, s, NH), 8.74 (1H, s, OH), 3.97 (H₂O from DMSO- d_6), 2.57 (residual DMSO), 1.98 (2H, t, $J = 7.2\text{Hz}$, C2-H), 1.53-1.50 (2H, m, C3-H), 1.29 (18H, brs, C4-C12 9 x CH₂), 0.91 (3H, t, $J = 7.2\text{Hz}$, C13-H).

^{13}C NMR (100 MHz, DMSO- d_6): δ 169.7(C1), 32.7(C2), 31.7(CH₂), 29.5(2 x CH₂), 29.4 (2x CH₂), 29.2 (CH₂), 29.0 (2 x CH₂), 25.6(CH₂), 22.5(CH₂), 14.4(C13).

HRMS (TOF MS ES⁺): m/z [M + Na]⁺ calcd. for C₁₃H₂₇NNaO₂ 252.1939 found 252.1926.

5-Bromo-N-hydroxypentanamide (7c):

Eluent: CHCl₃: MeOH (97:3).

Yield: 70%. Colorless viscous liquid.

IR (neat): 3359, 3158, 2856, 1667, 1624 cm^{-1}

^1H NMR (400 MHz, in CDCl₃+ DMSO- d_6): δ 10.45 (1H, s, NH), 3.55 (H₂O from DMSO- d_6 + merged 2H, C5-H), 2.57 (residual DMSO), 2.04 (2H, t, $J = 7.2\text{Hz}$, C2-H), 1.84 (2H, t, $J = 7.2\text{Hz}$, C4-H), 1.77-1.66 (2H, m, C3-H).

^{13}C NMR (100 MHz, in CDCl₃+ DMSO- d_6): δ 168.7(C1), 34.1(C5), 31.6(C4), 31.2(C2), 23.7(C3).

Anal calcd. for C₅H₁₀BrNO₂ C, 30.63; H, 5.14; N, 7.14. found: C, 30.86; H, 5.40; N, 6.87

***N*-Hydroxy-4-phenylbutanamide (7d):**

Eluent: CHCl₃:MeOH (99:1).

Yield: 89%. Colorless solid.

M.p-52°C.

IR (neat): 3413, 3062, 3026, 2925, 2871, 1705, 1692, 1653 cm⁻¹.

¹H NMR (400 MHz, DMSO-d₆): δ 7.35-7.32 (2H, m, ArH), 7.25-7.21 (3H, m), 3.42 (H₂O from DMSO-d₆), 2.65-2.59 (2H, m, C4-H), 2.26 (2H, t, *J* = 7.6Hz, C2-H), 1.84 (2H, quin, *J* = 7.6Hz, C3-H).

¹³C NMR (100 MHz, DMSO-d₆): δ 169.4 (C1), 142.1 (ArC), 128.8 (ArCH, two signals), 126.3 (ArCH), 35.0 (C4), 32.2 (C2), 27.4 (C3).

HRMS (TOF MS ES⁺): *m/z* [M + Na]⁺ calcd. for C₁₀H₁₃NNaO₂ 202.0844; found 202.0842.

***N*-Hydroxy-4-(2-methoxyphenyl)butanamide (7e):**

Eluent: CHCl₃:MeOH (97:3).

Yield: 85%. Colorless viscous liquid.

IR (neat): 3218, 3003, 2925, 1783, 1646, 1601cm⁻¹.

¹H NMR (400 MHz, CDCl₃+ DMSO-d₆): δ 10.39 (1H, s, NH), 8.71 (1H, s, OH), 7.16-7.083 (2H, m, ArH), 6.92-6.84 (3H, m, ArH), 3.76 (3H, s, OMe), 3.58 (H₂O from DMSO-d₆), 2.52 (C4-H+residual DMSO), 1.97 (2H, t, *J* = 6.8Hz, C2-H), 1.76-1.72 (2H, m, C3-H).

^{13}H NMR (100 MHz, in $\text{CDCl}_3 + \text{DMSO-d}_6$): δ 169.7 (C1), 157.4 (ArC-OCH_3), 129.9 (ArCH), 127.4 (ArC), 120.5 (ArCH), 110.7 (ArCH), 55.4 (OCH_3), 32.6 (C4), 29.6 (C2), 25.9 (C3).

HRMS (TOF MS ES⁺): m/z $[\text{M} + \text{Na}]^+$ calcd. for $\text{C}_{11}\text{H}_{15}\text{NNaO}_3$ 232.0949; found 232.0945.

***N*-Hydroxy-4-(2-methoxy-9-methyl-9*H*-carbazol-1-yl)butanamide (7f):**

Eluent: CHCl_3 :MeOH (97:3).

Yield: 80%. Light yellow solid.

M.p-142-143 °C.

IR (neat): 3267, 3028, 2965, 2933, 1783, 1661, 1634 cm^{-1} .

^1H NMR (400 MHz, DMSO-d_6): δ 10.39 (1H, s, NH), 8.70 (1H, s, OH), 7.94 (1H, d, $J = 7.6$ Hz, ArH), 7.88 (1H, d, $J = 8.4$ Hz, ArH), 7.46 (1H, d, $J = 8.0$ Hz, ArH), 7.30 (1H, t, $J = 7.6$ Hz, ArH), 7.07 (1H, t, $J = 7.6$ Hz, ArH), 6.87 (1H, d, $J = 8.8$ Hz, ArH), 3.94 (3H, s, OCH_3), 3.81 (3H, s, NCH_3), 3.39 (H_2O from DMSO-d_6), 3.01 (2H, t, $J = 8.0$ Hz, C4-H), 2.44 (residual DMSO), 2.06 (2H, t, $J = 7.2$ Hz, C2-H), 1.79-1.71 (2H, m, C3-H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 169.5(C1), 156.8 (ArC-OCH_3), 142.4 (ArCH), 140.2(ArCH), 125.1(ArCH), 122.7(ArC), 119.4(ArCH), 119.3(ArCH), 118.7(ArC), 118.2(ArC), 112.8(ArC), 109.4(ArCH), 104.8(ArCH), 56.9(OCH_3), 32.8(NCH_3), 32.5(C2), 27.5 (C3), 24.0 (C4).

HRMS (TOF MS ES⁺): m/z $[\text{M} + \text{Na}]^+$ calcd. for $\text{C}_{18}\text{H}_{20}\text{N}_2\text{NaO}_3$ 335.1372; found 335.1374.

***N*-Hydroxy-3-(4-methoxyphenyl)propanamide (7g):**

Eluent: CHCl_3 :MeOH (99:1).

Yield: 83%. Colorless viscous liquid.

IR (neat): 3278, 3033, 2958, 2925, 2853, 2044, 1885, 1859, 1791, 1663, 1612 cm^{-1} .

^1H NMR (400 MHz, DMSO-d_6): δ 10.37 (1H, s, NH), 8.72 (1H, s, OH), 7.18-7.09 (2H, m, ArH), 6.82 (2H, d, J = 8Hz, ArH), 3.70 (3H, s, OCH_3), 3.41 (H_2O from DMSO-d_6), 2.73 (2H, t, J = 7.6Hz, C3-H), 2.50 (residual DMSO), 2.20 (2H, t, J = 7.2Hz, C2-H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 168.8(C1), 158.0(ArC-OCH_3), 133.3(ArC), 129.8(ArCH), 114.1(ArCH), 55.4(OCH_3), 34.7(C3), 30.4(C2).

HRMS (TOF MS ES⁺): m/z $[\text{M} + \text{Na}]^+$ calcd. for $\text{C}_{10}\text{H}_{13}\text{NNaO}_3$ 218.0793; found 218.0788.

6-(Hydroxyamino)-6-oxohexanoic acid (7h):

Eluent: CHCl_3 :MeOH (95:5).

Yield: 81%. Colorless viscous liquid.

IR (neat): 3202, 2933, 1710, 1656, 1461 cm^{-1}

^1H NMR (400 MHz, DMSO-d_6): δ 10.40 (1H, brs, NH), 3.41 (H_2O from DMSO-d_6), 3.17 (solvent impurity), 2.50 (residual DMSO), 2.17 (2H, t, J = 6.4 Hz, C2-H), 1.94 (2H, t, J = 6.4 Hz, C5-H), 1.47 (4H, brs, C3-H+C4-H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 175.3 (C1), 169.8(C6), 34.1(C2), 32.4(C5), 25.1(C4), 24.8(C3).

HRMS (TOF MS ES⁺): m/z $[\text{M} + \text{Na}]^+$ calcd. for $\text{C}_6\text{H}_{11}\text{NNaO}_4$ 184.0586; found 184.0598

7-(Hydroxyamino)-7-oxoheptanoic acid (7i):

Eluent: CHCl₃:MeOH (95:5).

Yield: 78%. Colorless viscous liquid.

IR (neat): 3419, 2925, 2855, 2256, 2127, 1651cm⁻¹

¹H NMR (400 MHz, DMSO-d₆): δ 10.42 (1H, s, NH), 3.54 (H₂O from DMSO-d₆), 2.59 (residual DMSO), 2.27 (2H, t, *J* = 7.2Hz, C2-H), 2.02 (2H, t, *J* = 7.2Hz, C6-H), 1.60-1.53 (4H, m, C3-H+C5-H), 1.35-1.32 (2H, m, C4-H).

¹³C NMR (100 MHz, DMSO-d₆): δ 174.9(C1), 169.6(C7), 34.0(C2), 32.6(C6), 28.4(C4), 25.3(C5), 24.7(C3).

HRMS (TOF MS ES⁺): *m/z* [M + Na]⁺ calcd. for C₇H₁₃NNaO₄ 198.0742; found 198.0748.

8-(Hydroxyamino)-8-oxooctanoic acid (7j):

Eluent: CHCl₃:MeOH (95:5).

Yield: 75%. Colorless viscous liquid.

IR (neat): 3414, 2922, 2855, 1637 cm⁻¹.

¹H NMR (400 MHz, DMSO-d₆): δ 12.06 (1H, brs, COOH), 10.40 (1H, s, NH), 8.74 (1H, brs, OH), 3.43 (H₂O from DMSO-d₆), 2.57 (residual DMSO), 2.25 (2H, t, *J* = 6.8Hz, C2-H), 1.99 (2H, t, *J* = 7.2Hz, C7-H), 1.53 (4H, brs, C3-H+C-6H), 1.30 (4H, brs, C4-H+C5-H).

¹³C NMR (100 MHz, DMSO-d₆): δ 175.0(C1), 169.6(C8), 34.1(C2), 32.7(C7), 28.7(two signals, C4+C5), 25.4(C6), 24.9(C3).

HRMS (TOF MS ES⁺): *m/z* [M + Na]⁺ calcd. for C₈H₁₅NNaO₄ 212.0899; found 212.0892.

(S)-Methyl 2-((*tert*-butoxycarbonyl)amino)-7-(hydroxyamino)-7-oxoheptanoate (7k) :

Eluent: CHCl₃:MeOH (97:3).

Yield: 86%. Colorless viscous liquid.

$[\alpha]_D^{25} = +4.2$ (c 2.5, CHCl₃).

IR (neat): 3414, 3320, 2927, 2855, 1744, 1694 cm⁻¹.

¹H NMR (400 MHz, DMSO-d₆): δ 10.35 (1H, s, NH), 8.69 (1H, brs, OH), 7.25 (1H, d, *J* = 8Hz, NHBoc), 3.92 (1H, m, C2-H), 3.62 (3H, s, COOCH₃), 3.61 (H₂O from DMSO-d₆), 2.50 (residual DMSO), 1.95 (2H, m, C6-H), 1.62-1.52 (6H, m, C3-H+C4-H+C5-H), 1.38 (9H, s, O-CMe₃).

¹³C NMR (100 MHz, CDCl₃): δ 173.5 (C1), 169.1(C7) 156.0 (COOCMe₃), 78.7(OCMe₃), 53.7 (CO₂Me), 52.4(C2), 32.1(C6), 30.6(C3), 28.6(OCMe₃ + C-3, two signals), 22.2(C4).

HRMS (TOF MS ES⁺): *m/z* [M + Na]⁺ calcd. for C₁₃H₂₄N₂NaO₆ 327.1532; found 327.1539.

(S)-Methyl 2-((*tert*-butoxycarbonyl)amino)-8-(hydroxyamino)-8-oxooctanoate (7l):

Eluent: CHCl₃:MeOH (97:3).

Yield: 84%. Colorless viscous liquid.

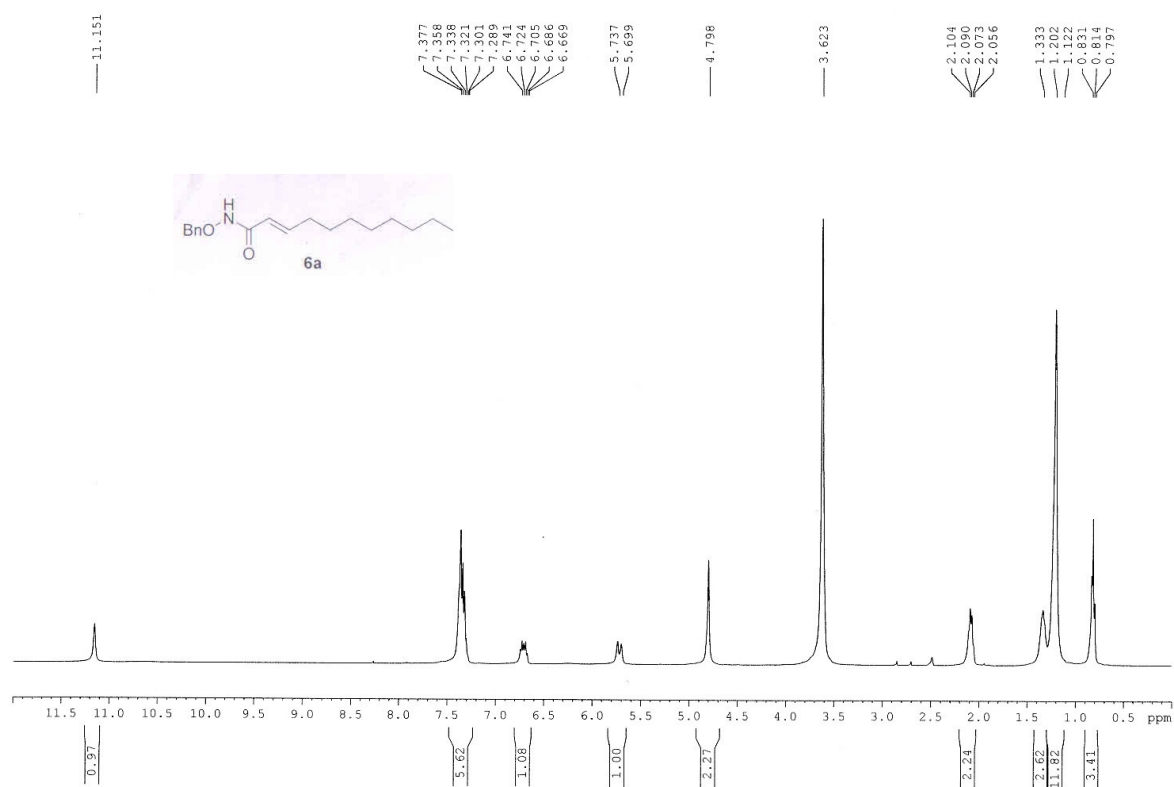
$[\alpha]_D^{25} = + 3.84$ (c 1.25, CHCl₃).

IR (neat): 3429, 3289, 2947, 1744, 1694, 1533 cm⁻¹.

¹H NMR (400 MHz, CDCl₃): δ 5.19 (1H, brs, NHBoc), 4.18 (1H, s, C2-H), 3.67 (3H, s, CO₂Me), 2.09 (2H, m, C7-H), 1.68-1.49 (4H, m, C3-H+C6-H), 1.36 (9H, s, CO₂CMe₃), 1.26-1.21 (4H, m, C4-H+C5-H).

^{13}C NMR (100 MHz, CDCl_3): δ 173.5(C1), 155.8(COO CMe_3), 80.2 (OC Me_3), 53.2(CO_2 Me), 52.3(C2), 32.4(C7), 29.7(C3), 28.3(OC Me_3), 28.1(C5), 24.9(C4), 24.7(C6).

HRMS (TOF MS ES⁺): m/z $[\text{M} + \text{Na}]^+$ calcd. for $\text{C}_{14}\text{H}_{26}\text{N}_2\text{NaO}_6$ 341.1688; found 341.1689



¹H NMR Spectrum of compound **6a** in DMSO-*d*₆

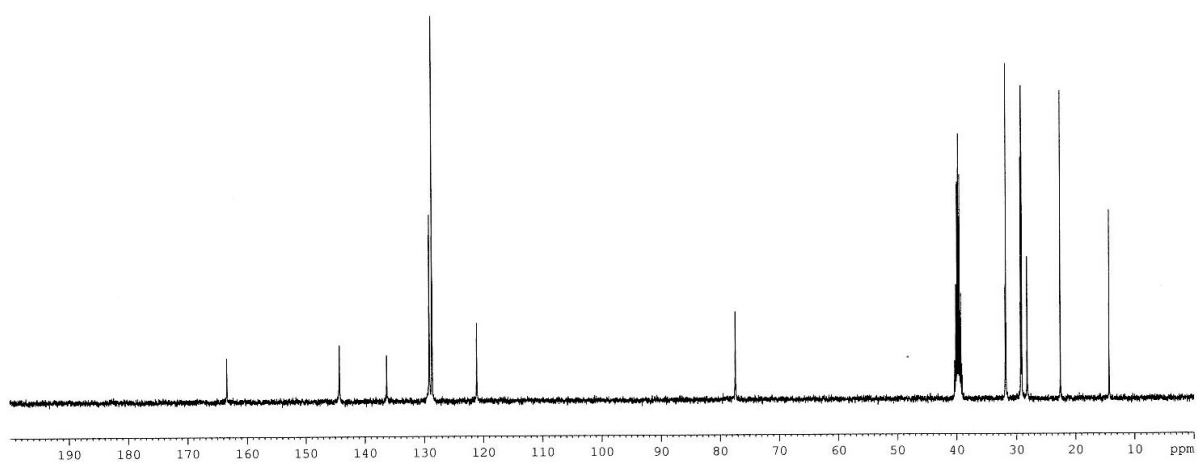
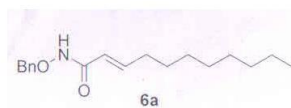
SS-2-94

01.10.2018

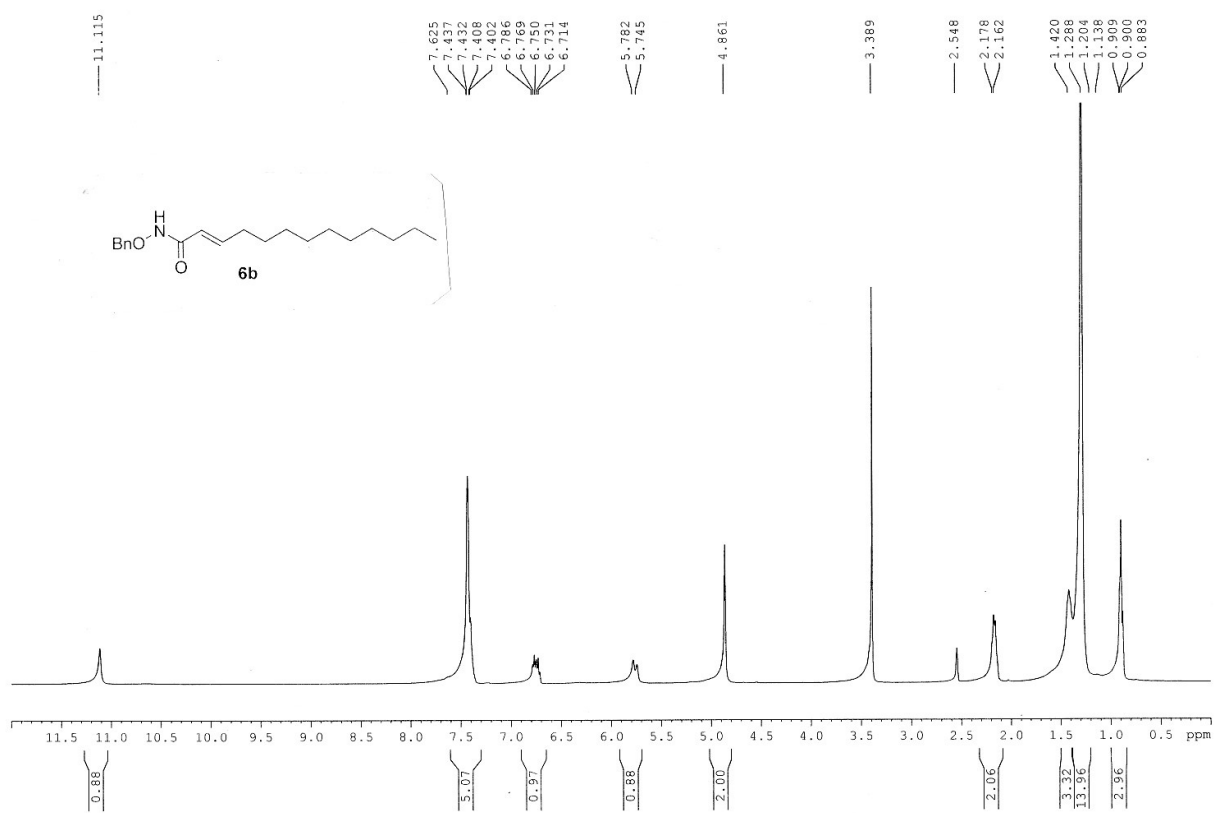
163.42
144.31
136.36
129.17
128.70
121.15

77.44

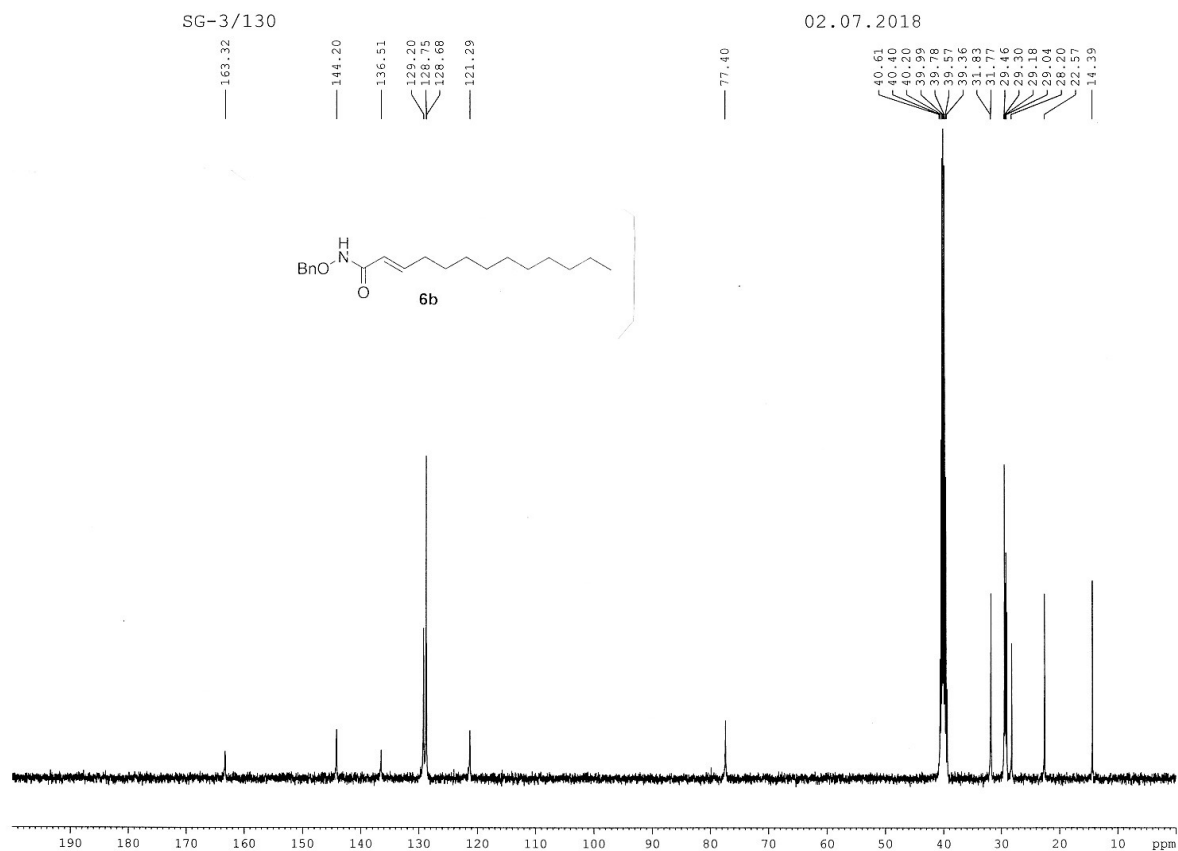
40.35
40.14
39.93
39.73
39.51
39.30
39.10
31.86
31.76
29.29
29.13
28.91
22.57
14.31



^{13}C NMR Spectrum of compound **6a** in $\text{DMSO}-d_6$



¹H NMR Spectrum of compound **6b** in DMSO-*d*₆

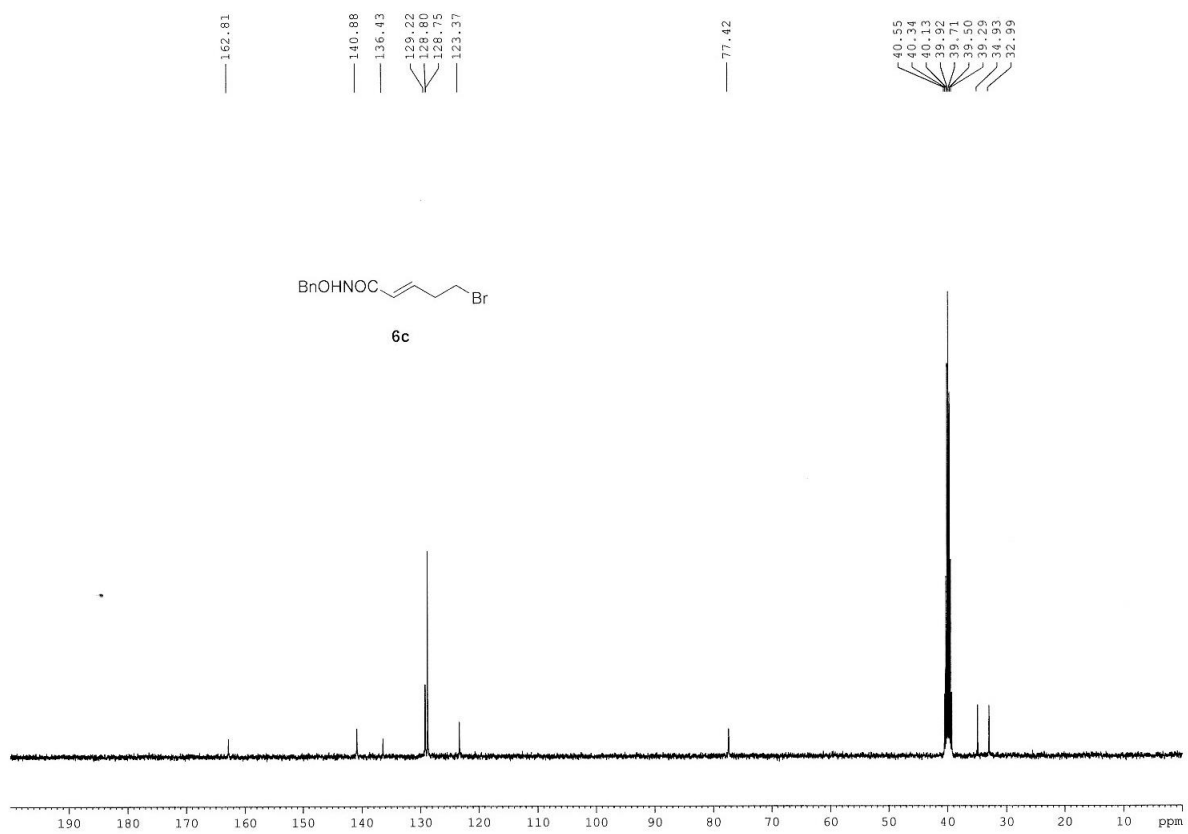


^{13}C NMR Spectrum of compound **6b** in $\text{DMSO-}d_6$

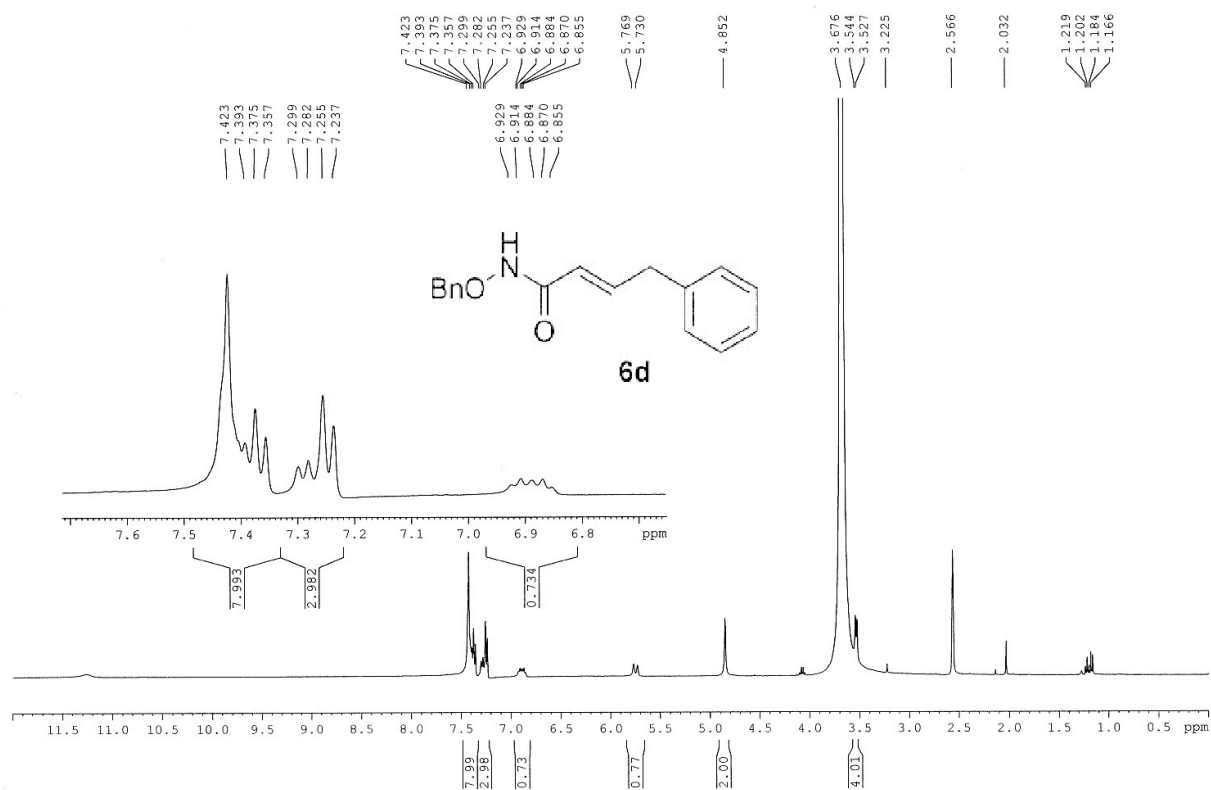


SS-2-30

01.10.2018



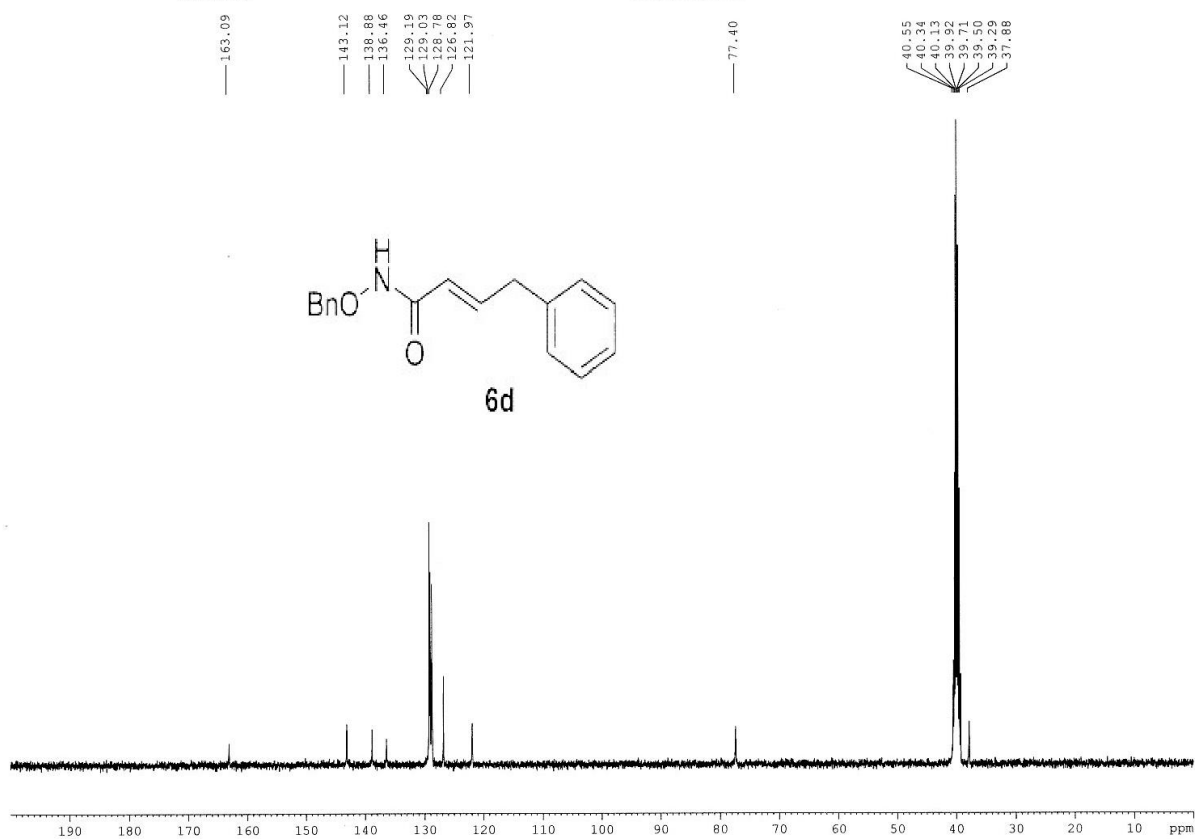
¹³C NMR Spectrum of compound **6c** in DMSO-*d*₆



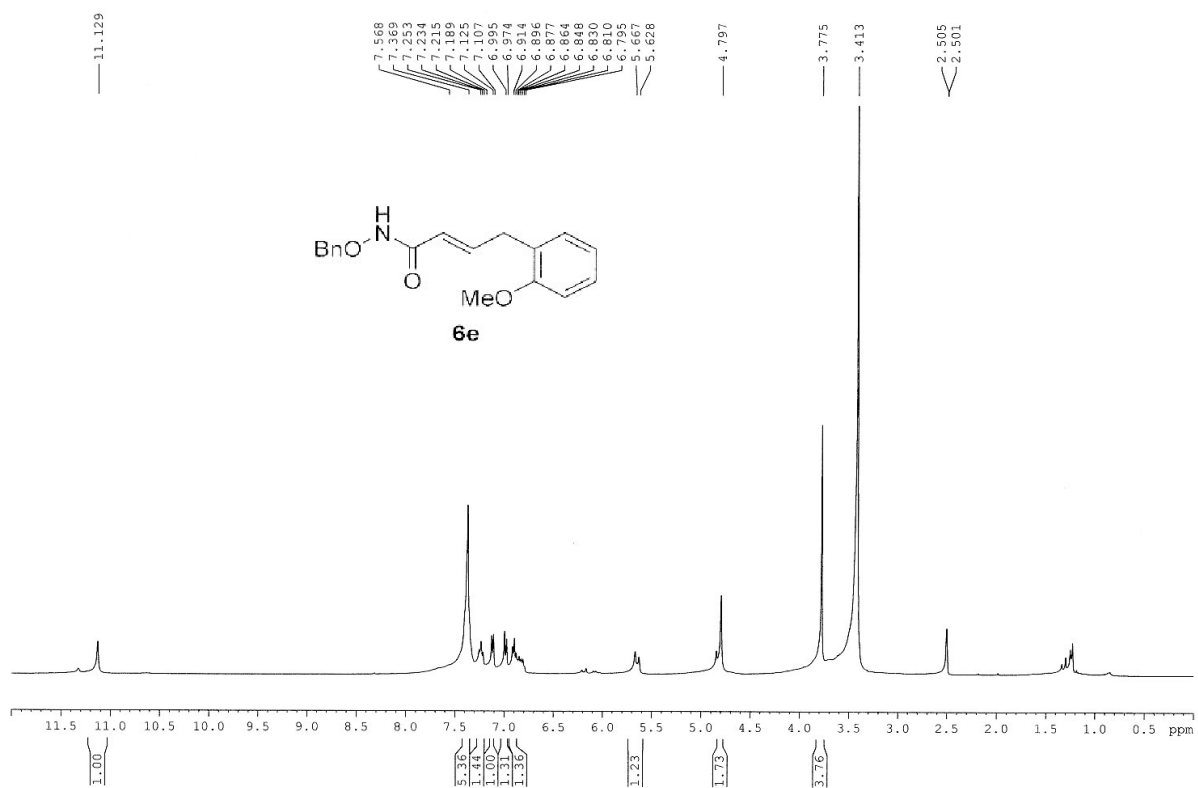
¹H NMR Spectrum of compound **6d** in DMSO-d₆

SGALCM

01.10.2018



¹³C NMR Spectrum of compound **6d** in DMSO-*d*₆



¹H NMR Spectrum of compound **6e** in DMSO-*d*₆

SS-2-54D

24.09.2018

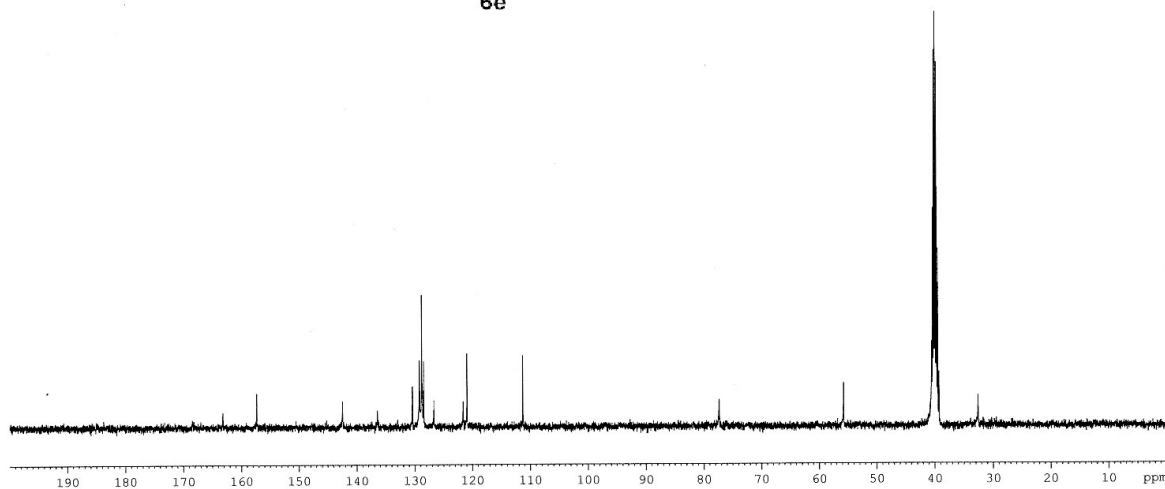
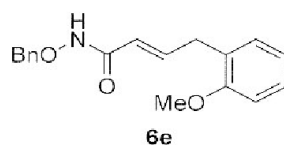
163.22
157.34

142.49
136.48
130.12
128.78
128.45
126.70
121.61
120.93
111.30

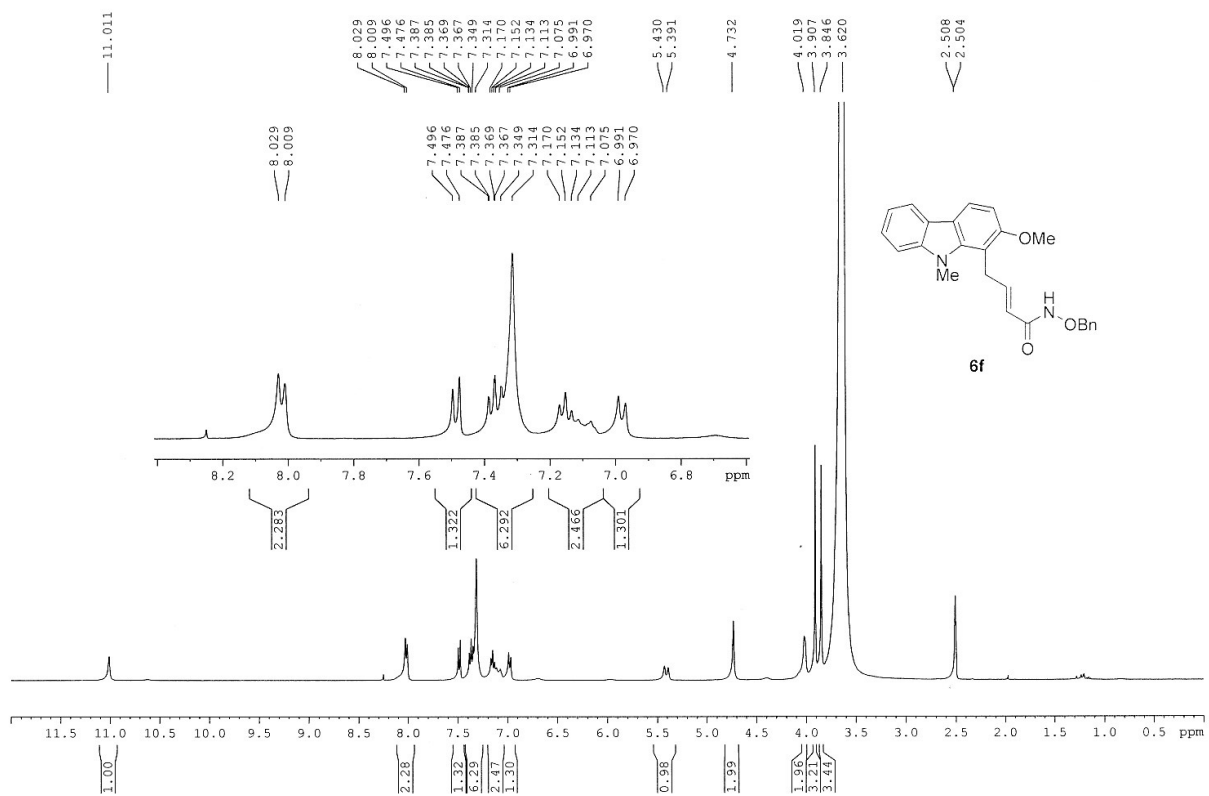
77.36

55.79

40.54
40.33
40.12
39.11
39.71
39.50
39.29
32.60



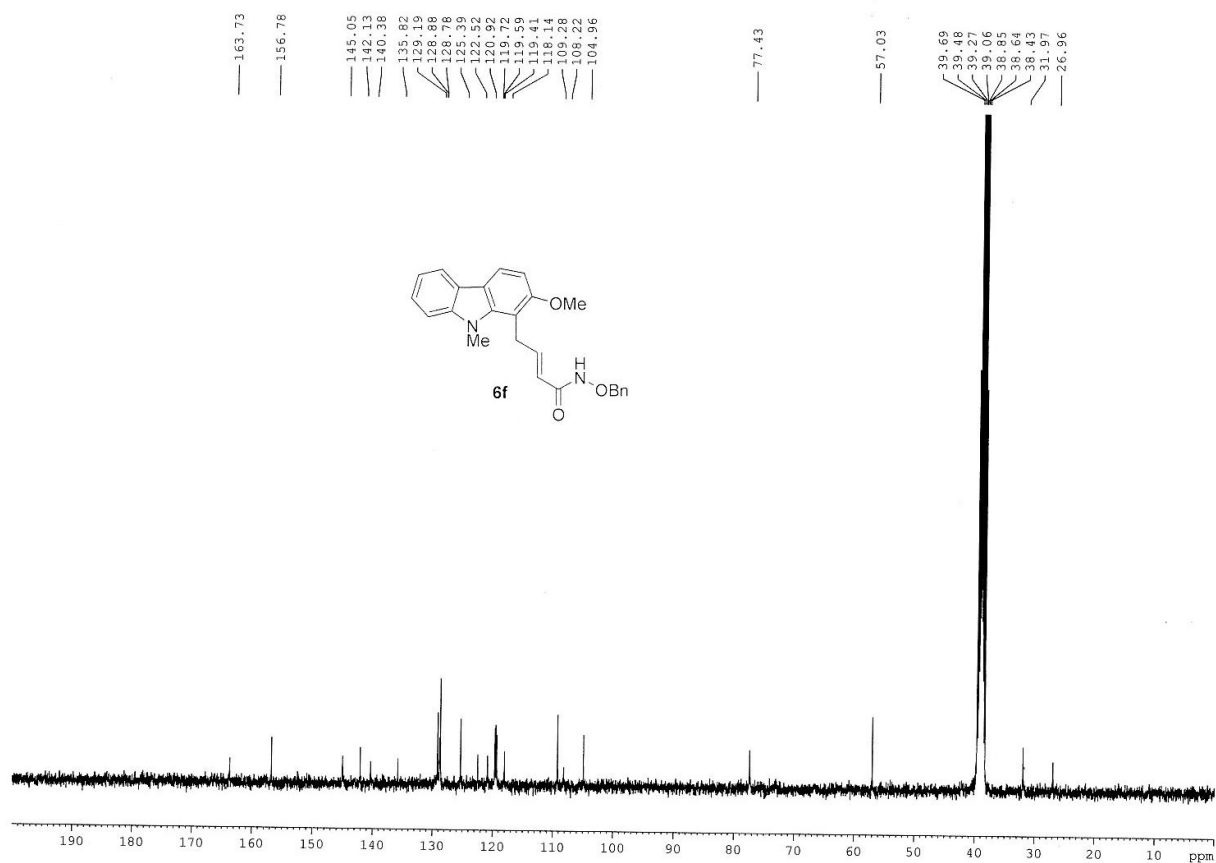
^{13}C NMR Spectrum of compound **6e** in $\text{DMSO-}d_6$



¹H NMR Spectrum of compound **6f** in DMSO-*d*₆

SG-CR-CM

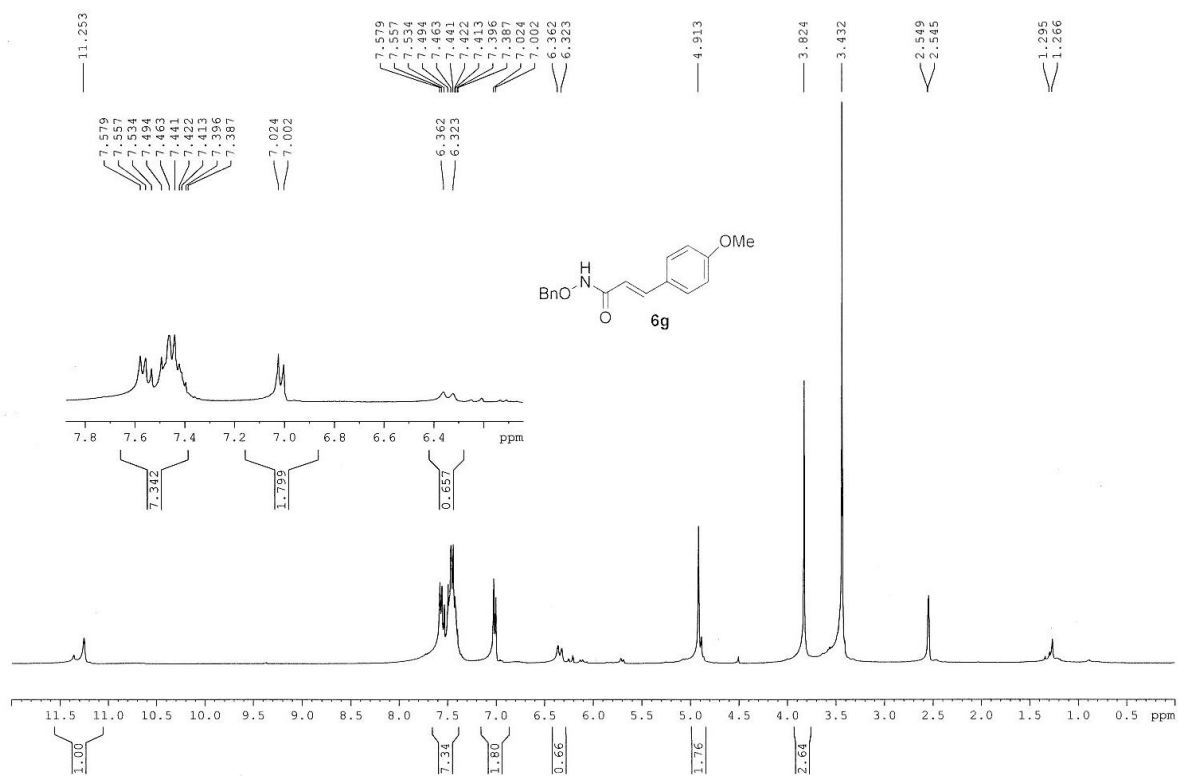
04.10.2018



¹³C NMR Spectrum of compound **6f** in DMSO-*d*₆

SS-1-41

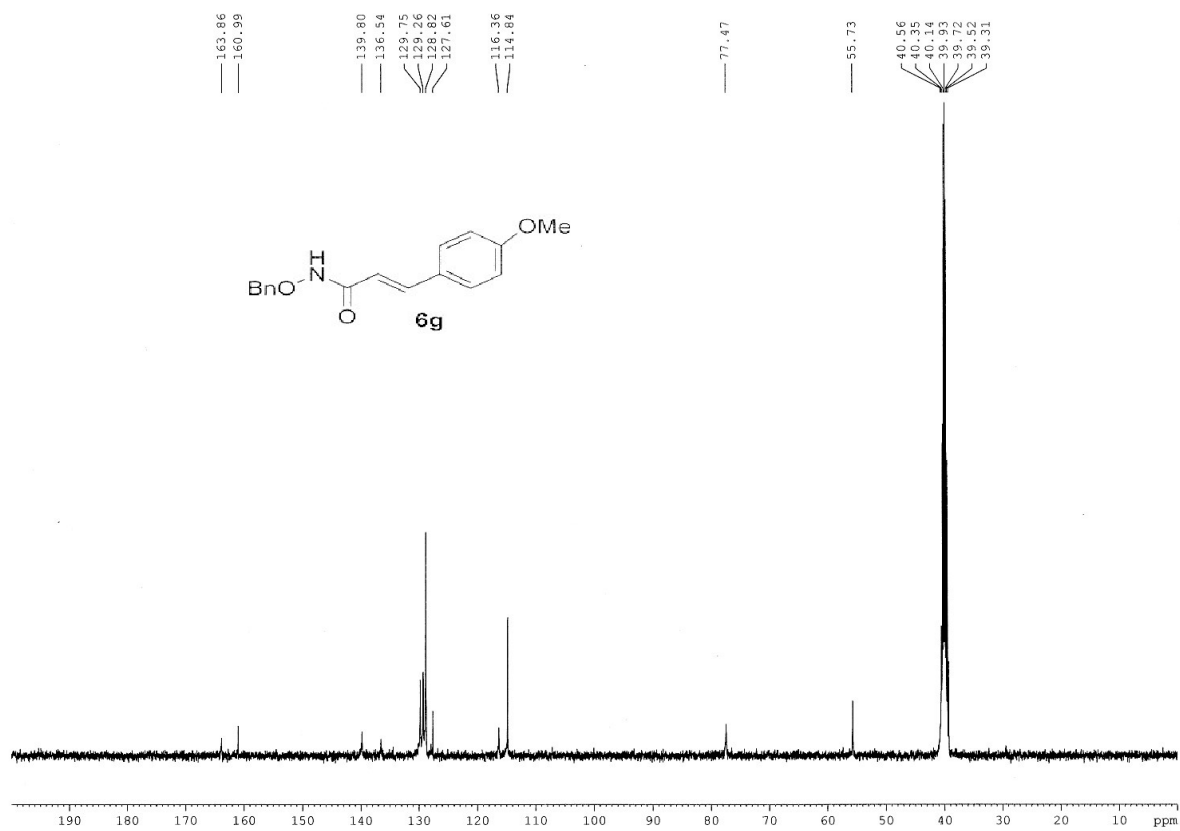
24.09.2018



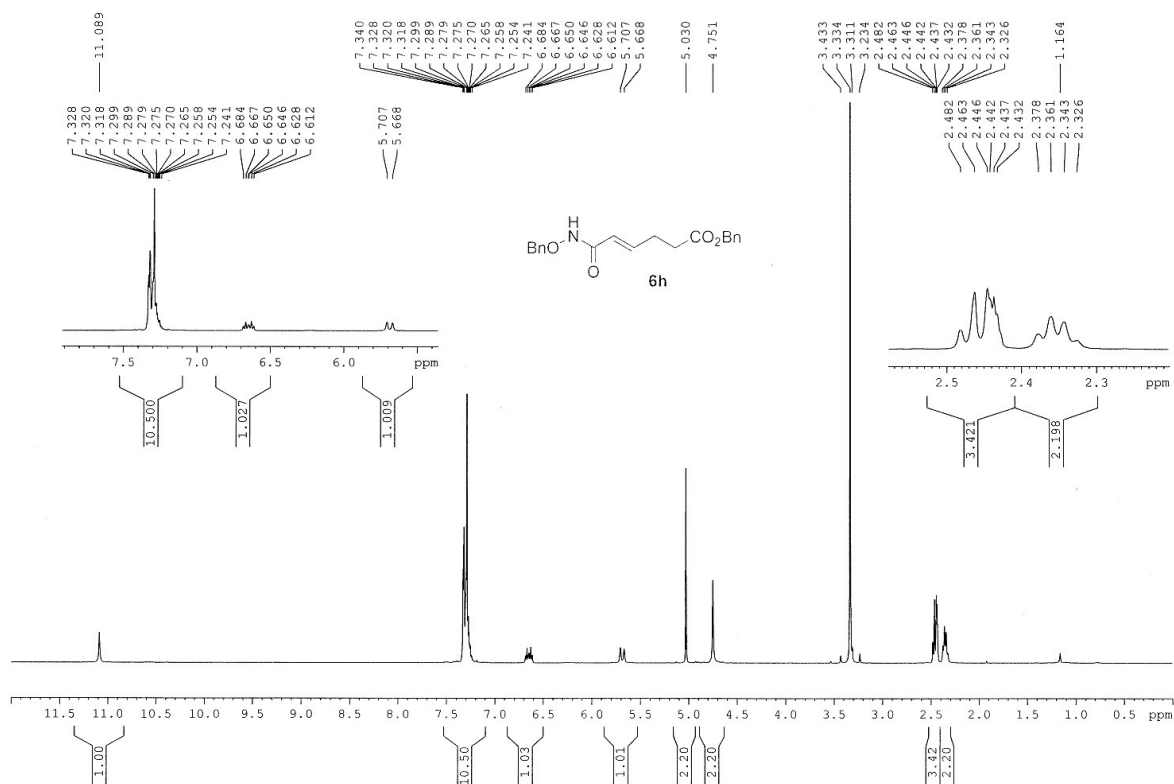
¹H NMR Spectrum of compound **6g** in DMSO-*d*₆

SS-1-41

24.09.2018

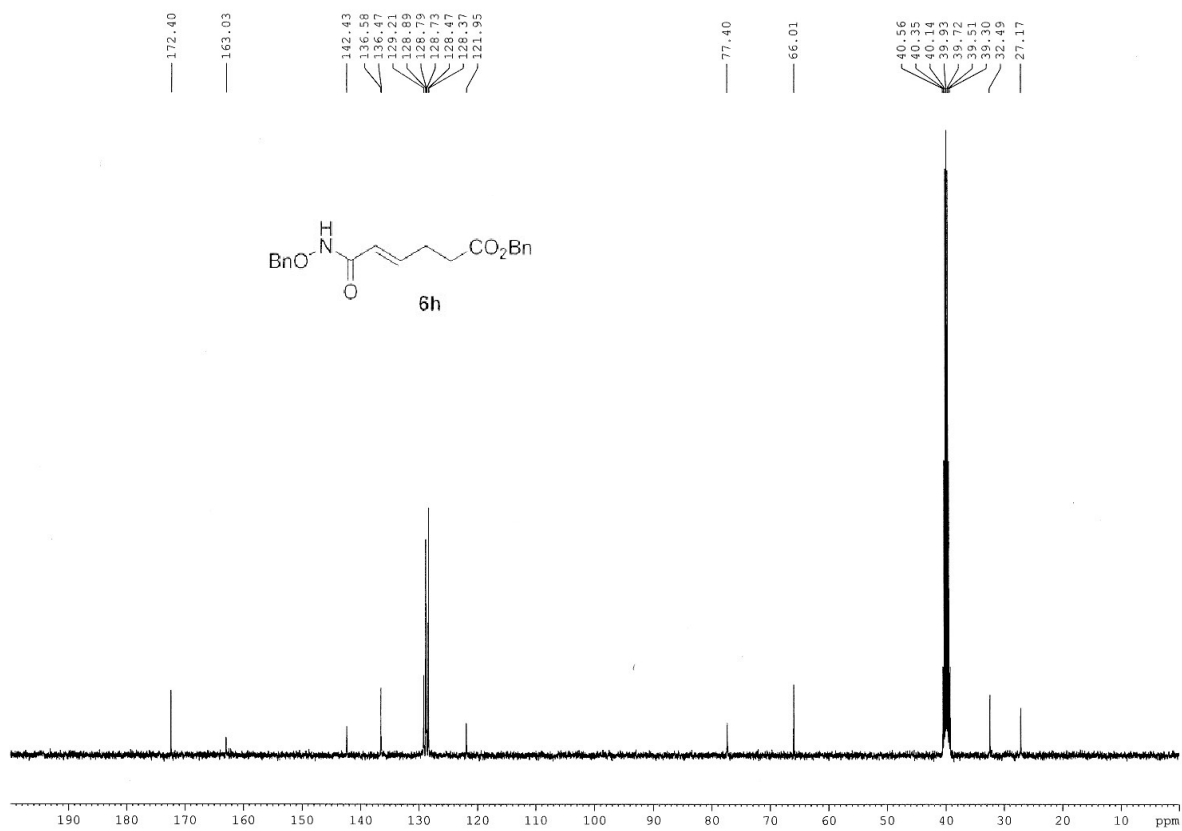


¹³C NMR Spectrum of compound **6g** in DMSO-*d*₆

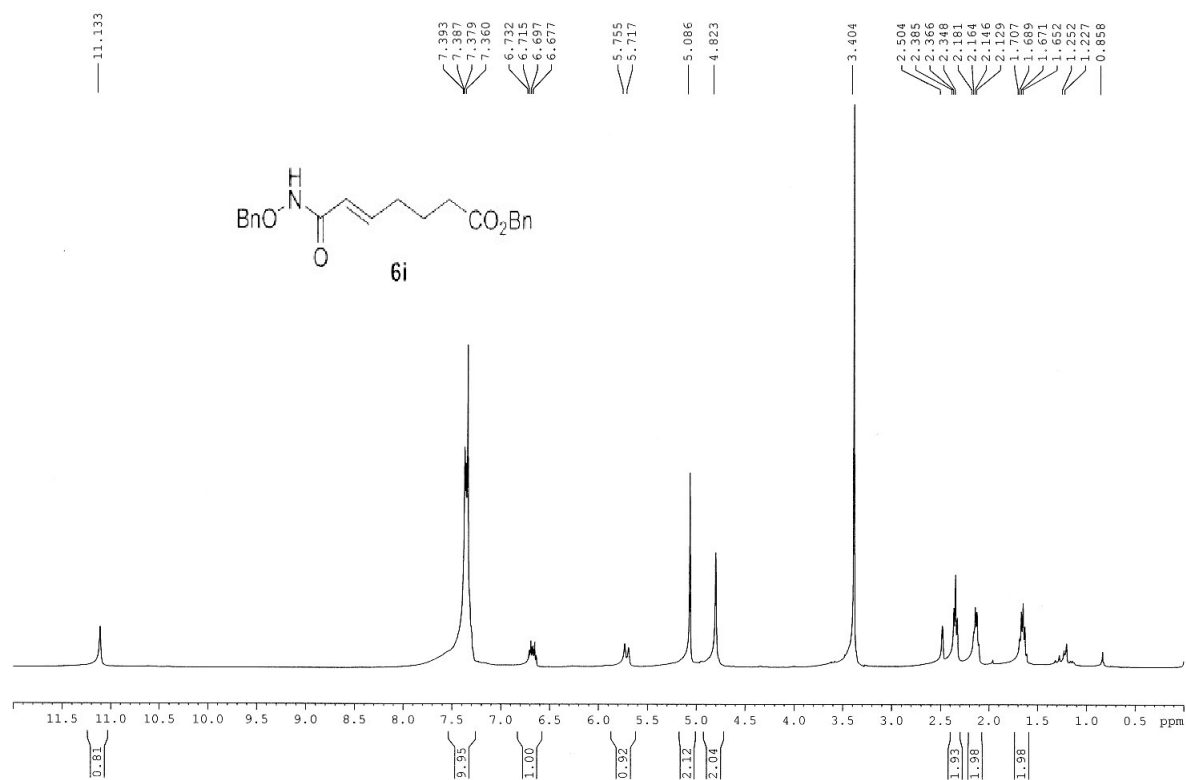


SS-2-27D

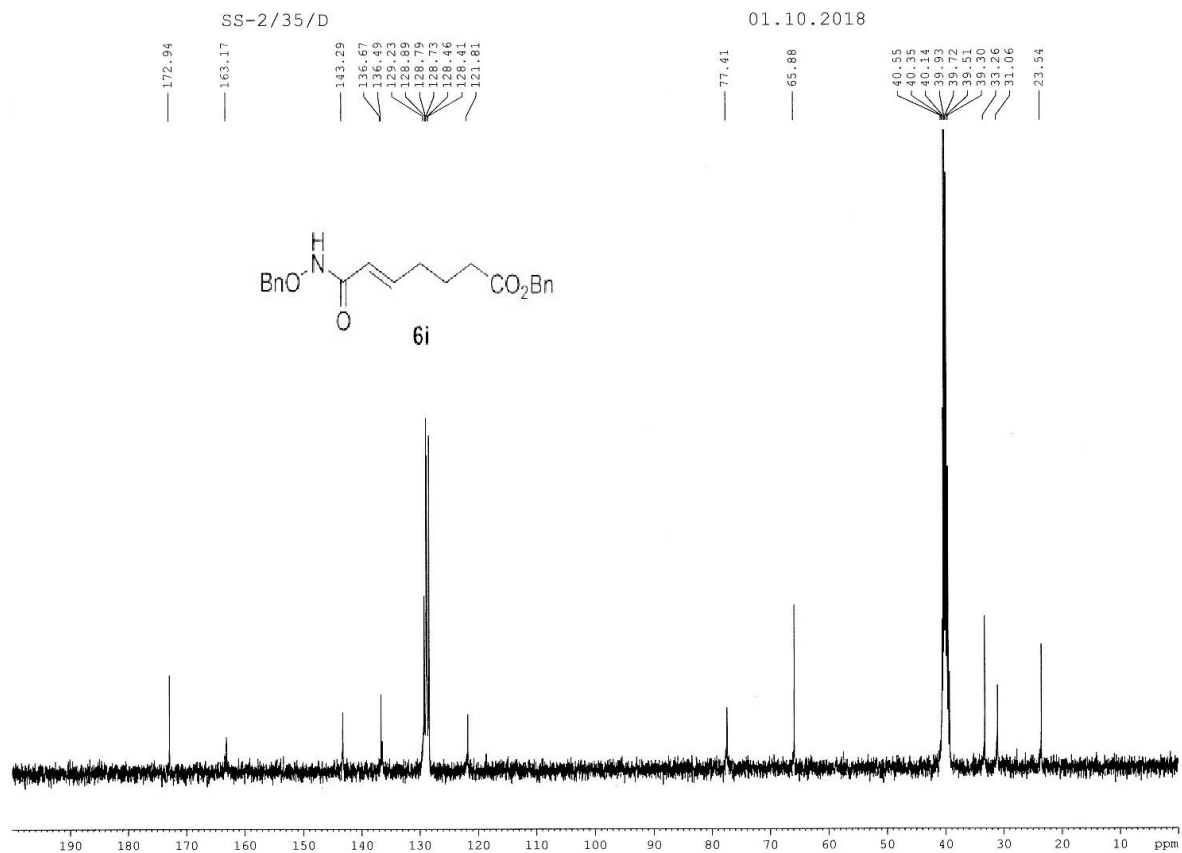
26.09.2018



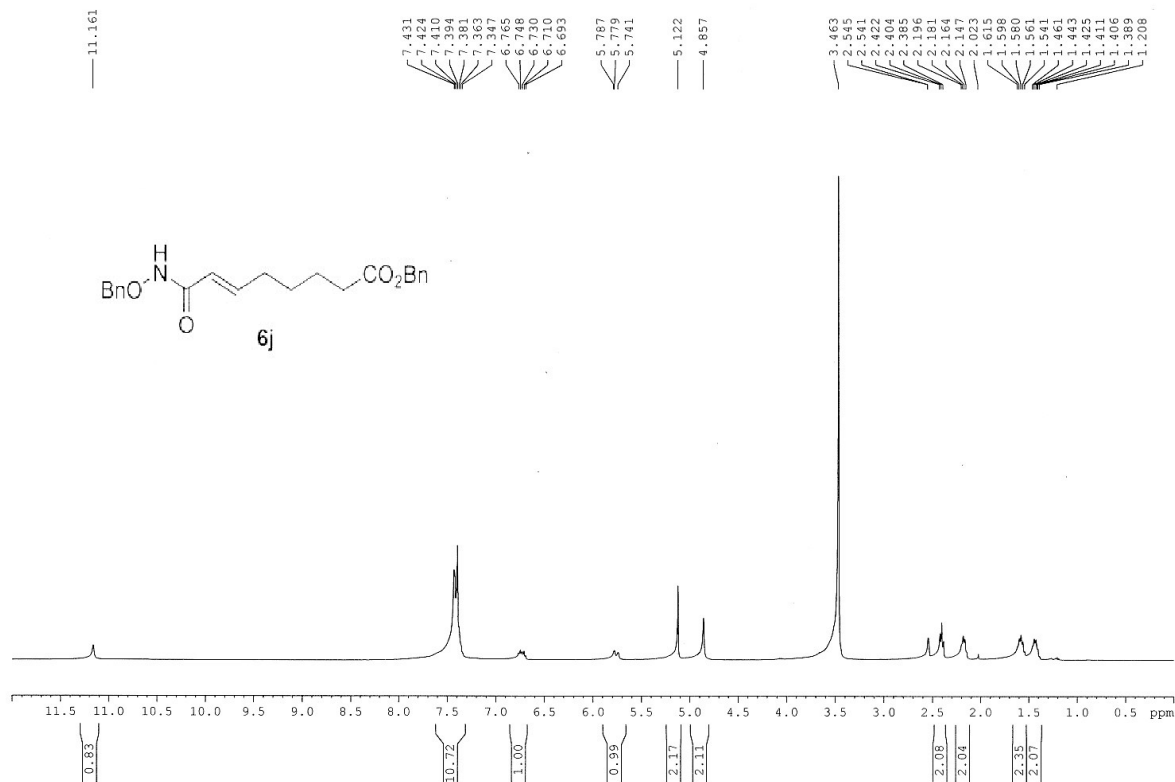
^{13}C NMR Spectrum of compound **6h** in DMSO- d_6



¹H NMR Spectrum of compound **6i** in DMSO-*d*₆

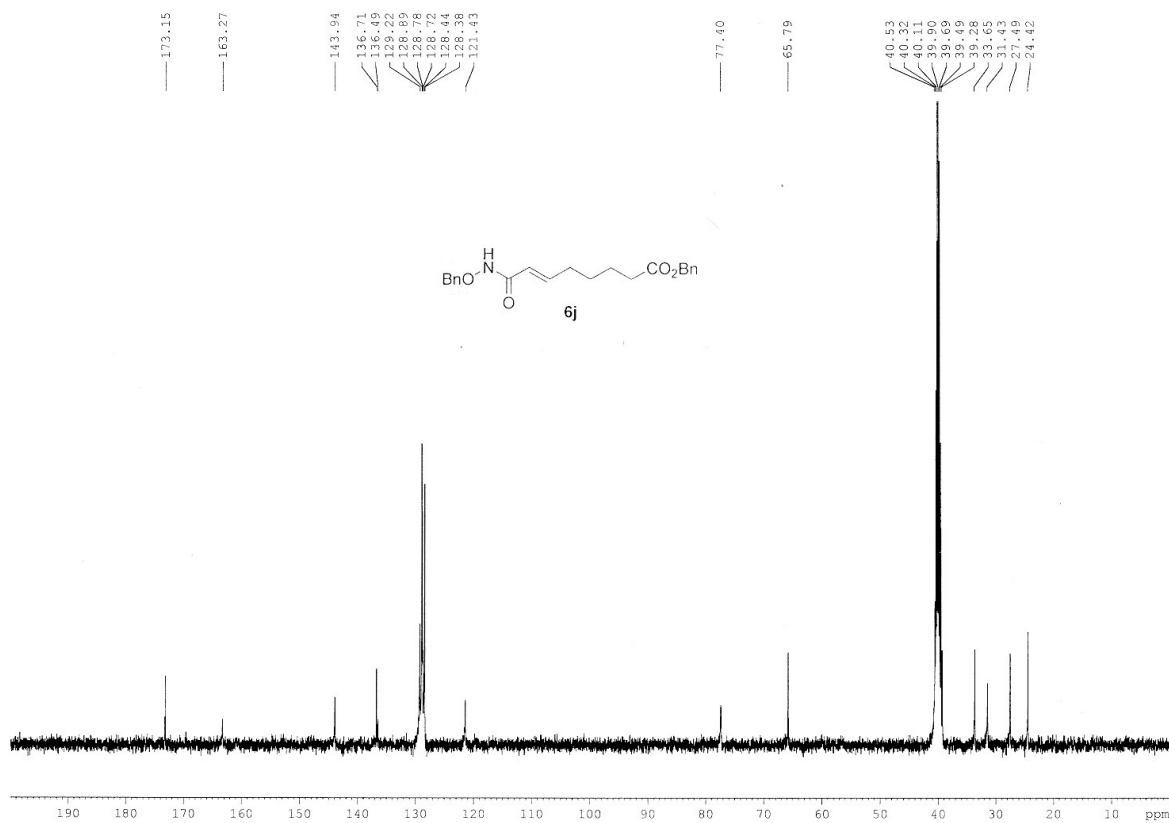


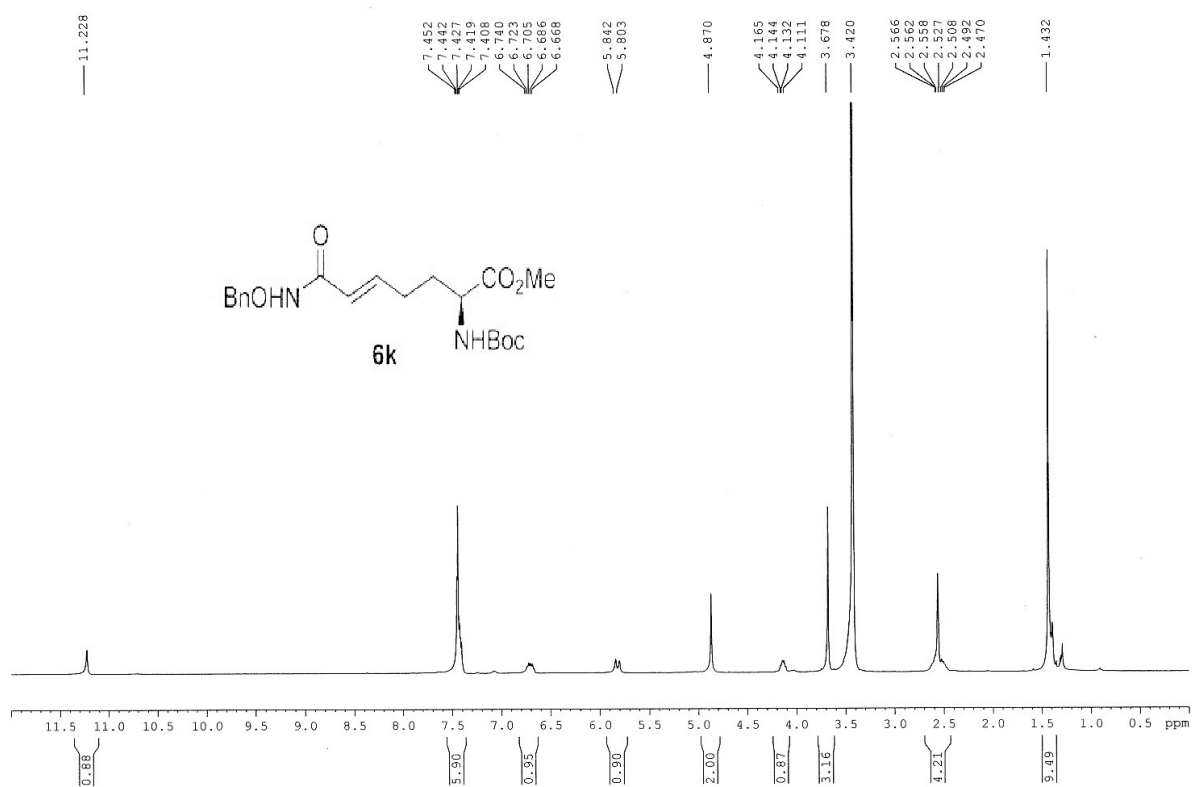
^{13}C NMR Spectrum of compound **6i** in $\text{DMSO-}d_6$



¹H NMR Spectrum of compound **6j** in DMSO-*d*₆

15.10.2015

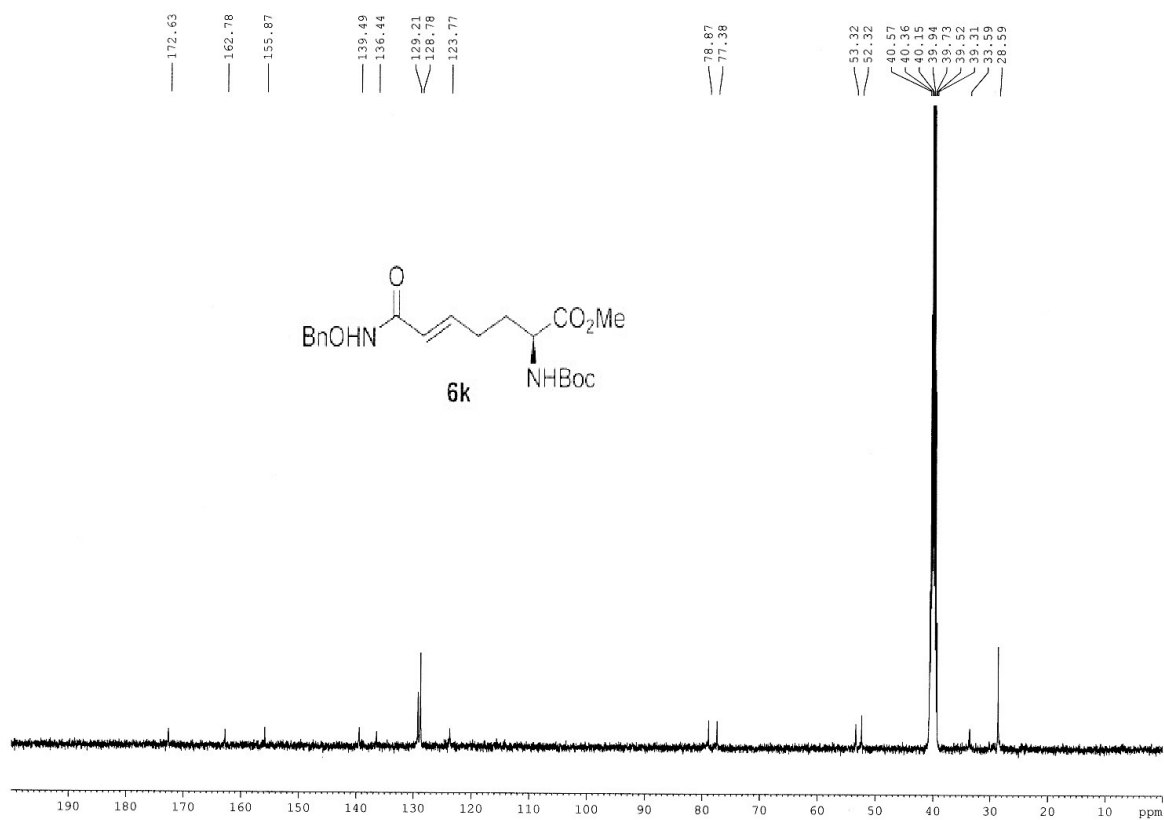
 ^{13}C NMR Spectrum of compound **6j** in DMSO- d_6



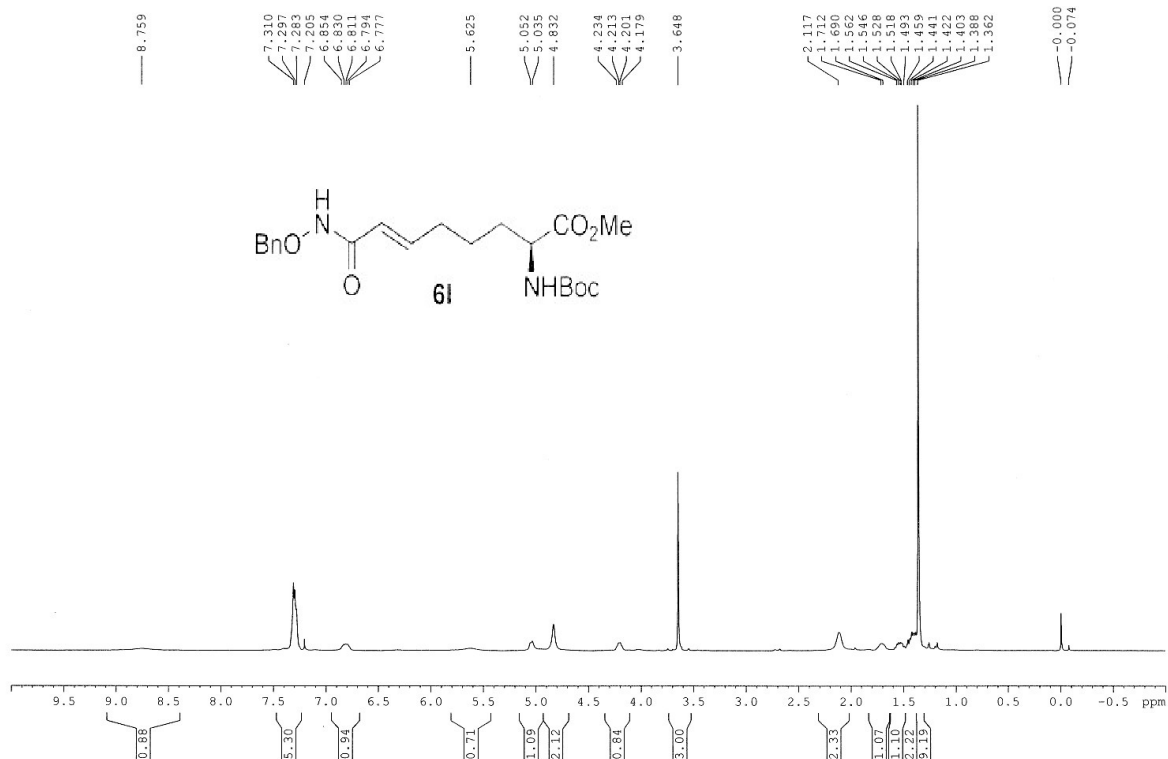
¹H NMR Spectrum of compound **6k** in DMSO-*d*₆

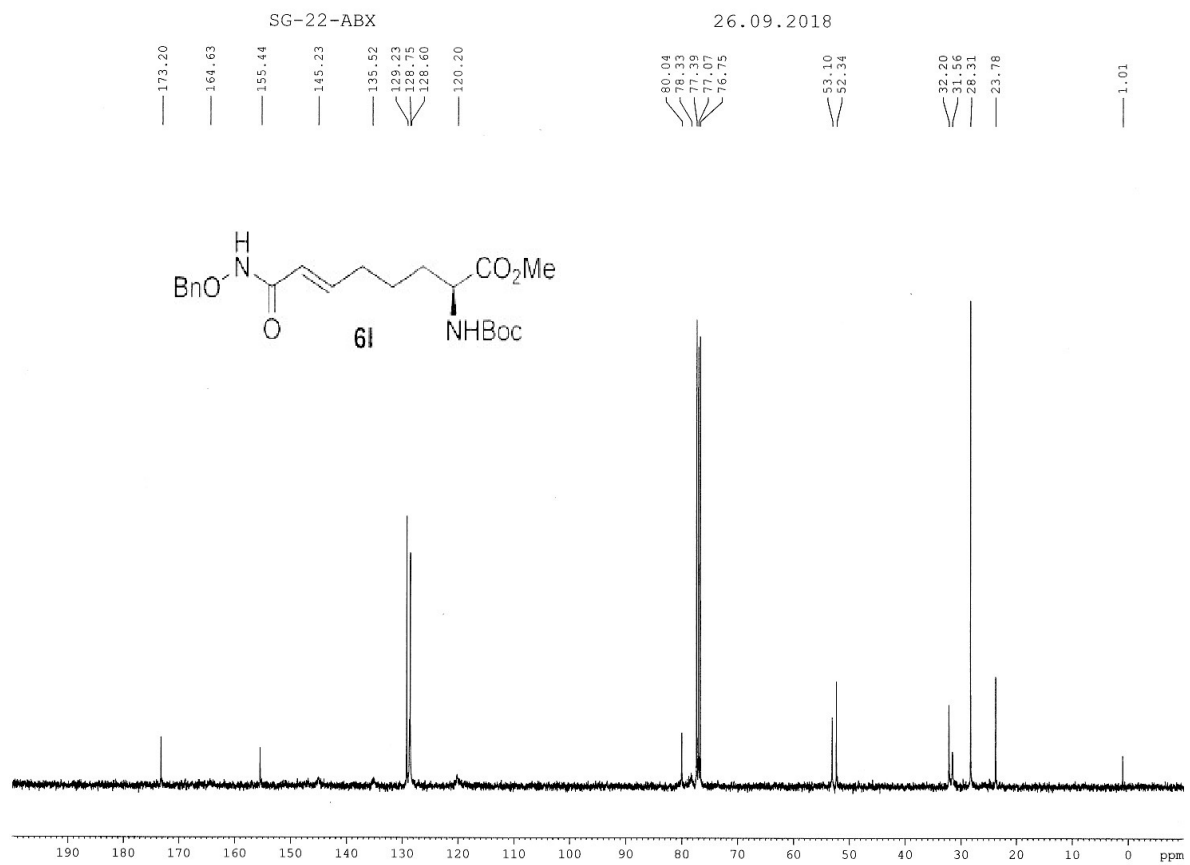
SG-ASP-OBn

25.09.2018

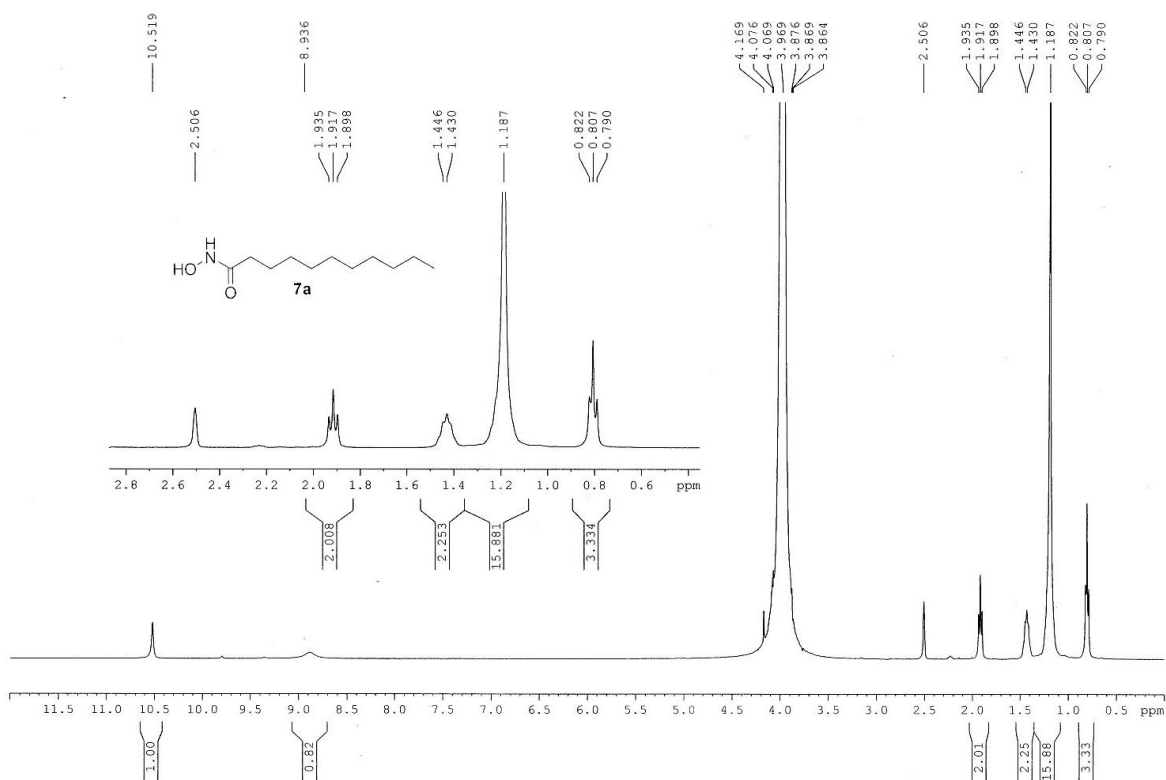


¹³C NMR Spectrum of compound **6k** in DMSO-*d*₆

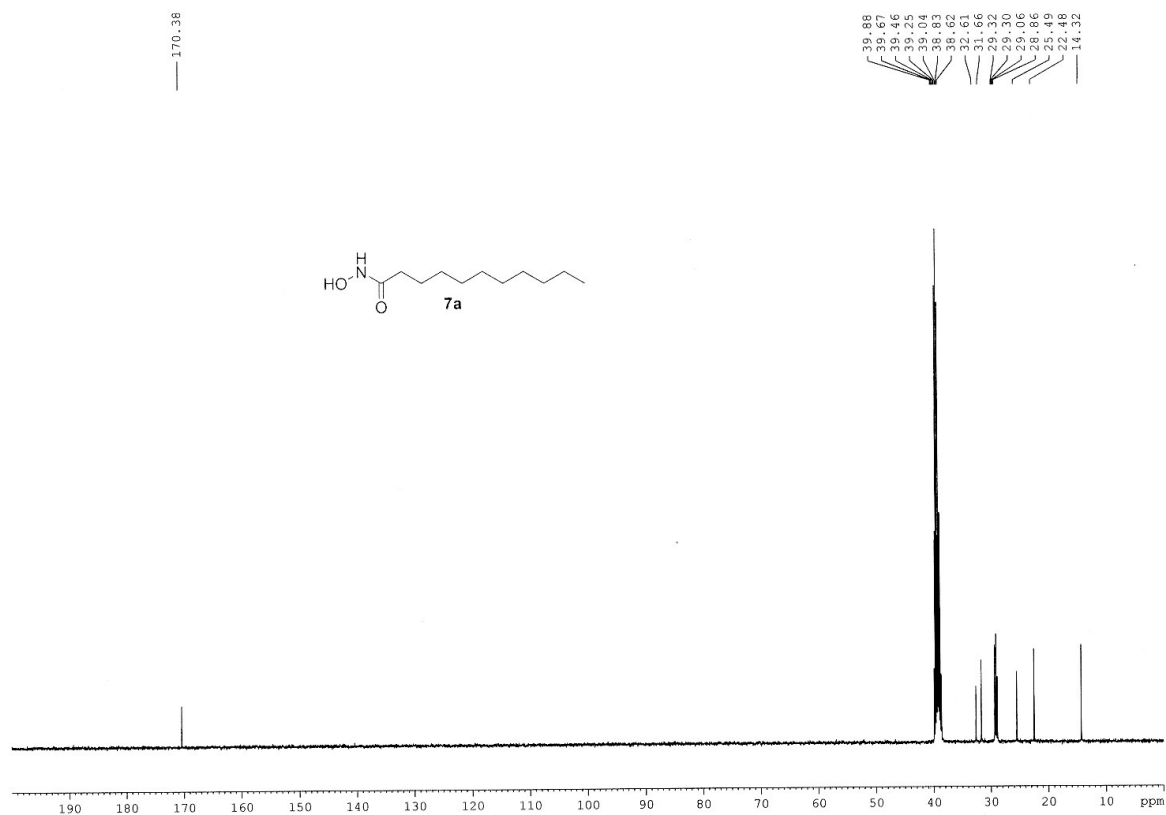
 ^1H NMR Spectrum of compound **6l** in DMSO- d_6



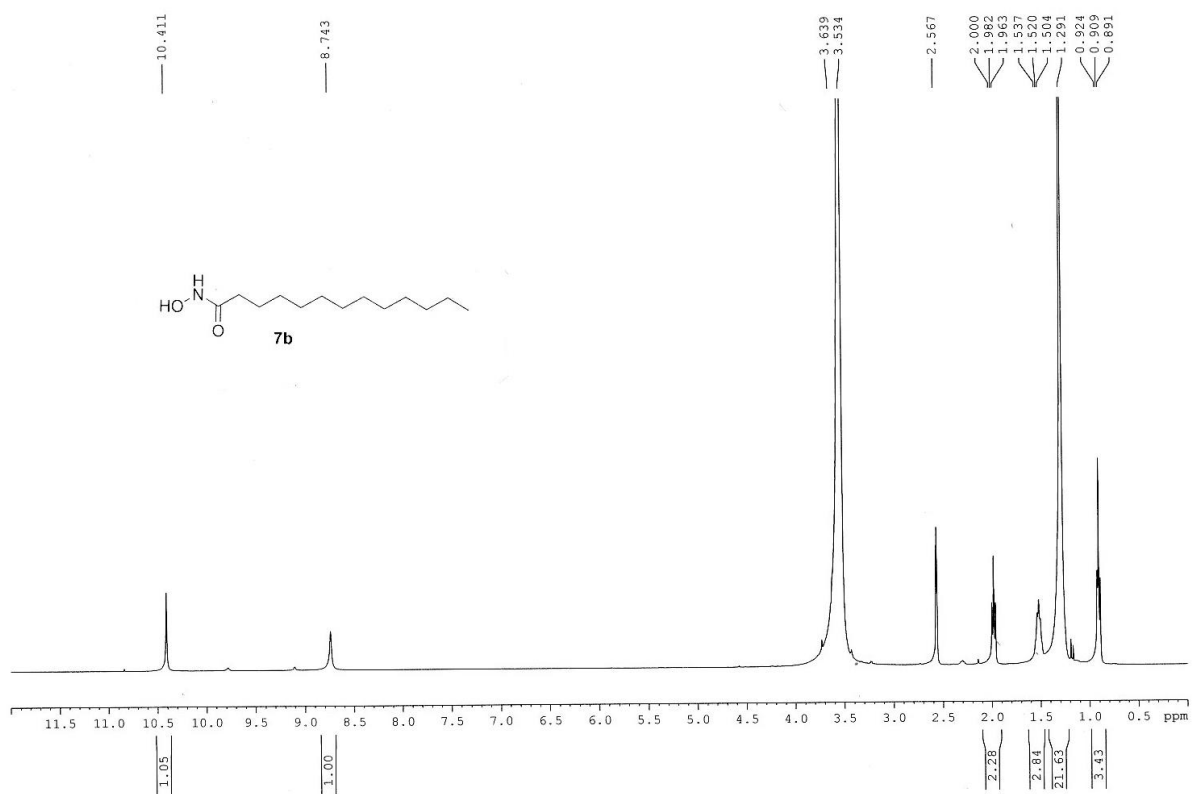
^{13}C NMR Spectrum of compound **6I** in $\text{DMSO-}d_6$



¹H NMR Spectrum of compound **7a** in DMSO-d₆



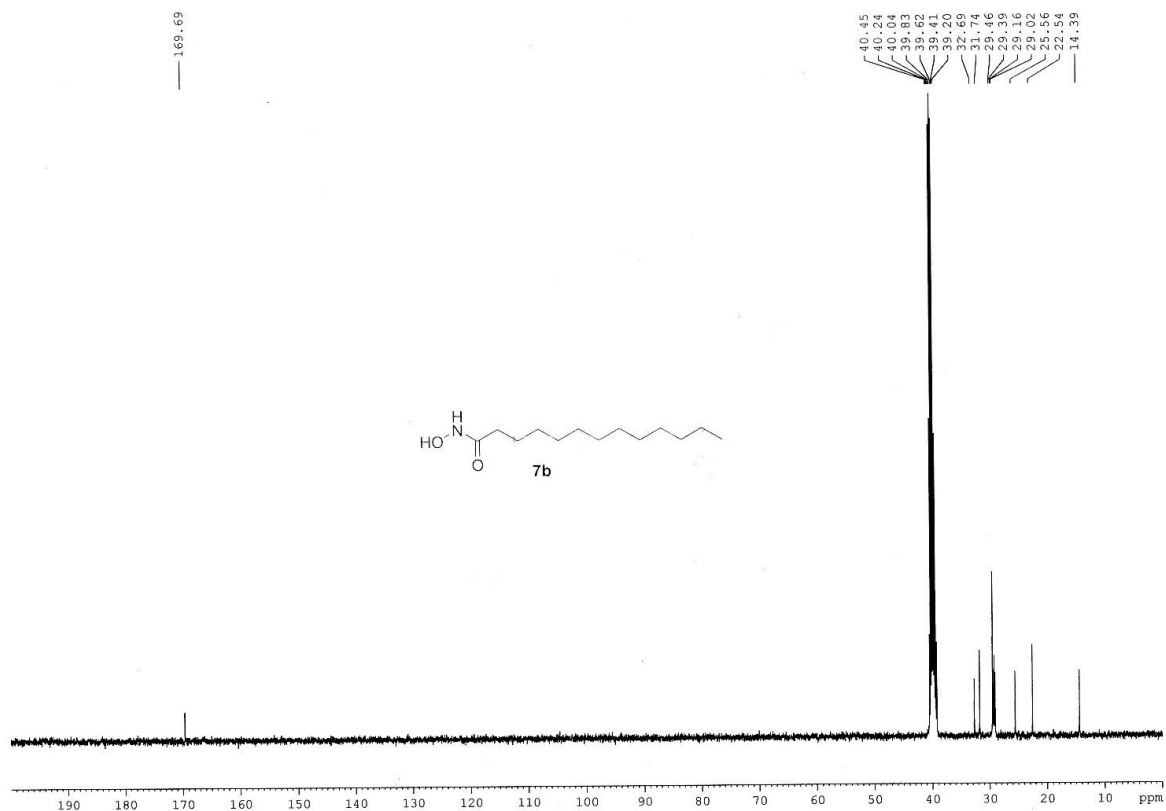
¹³C NMR Spectrum of compound **7a** in DMSO-*d*₆



¹H NMR Spectrum of compound **7b** in DMSO-*d*₆

SG-3/137

11.07.2018

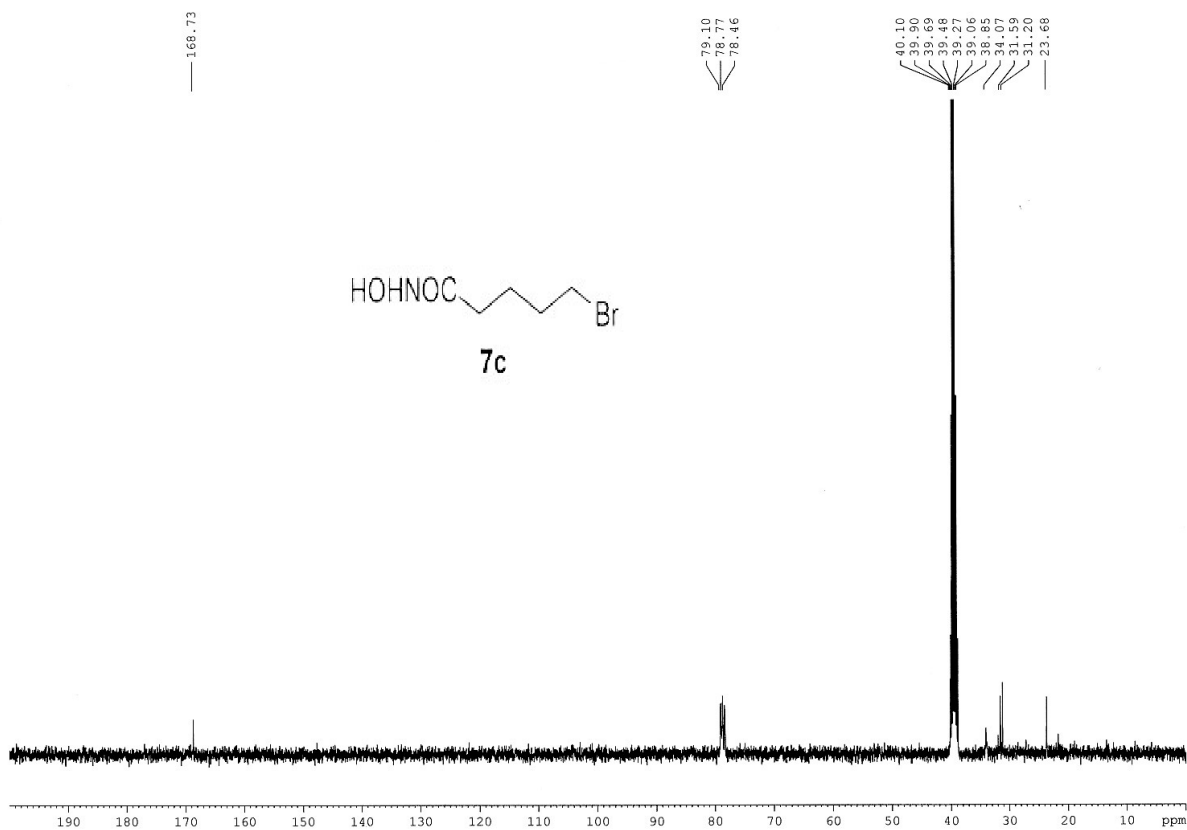


¹³C NMR Spectrum of compound **7b** in DMSO-*d*₆

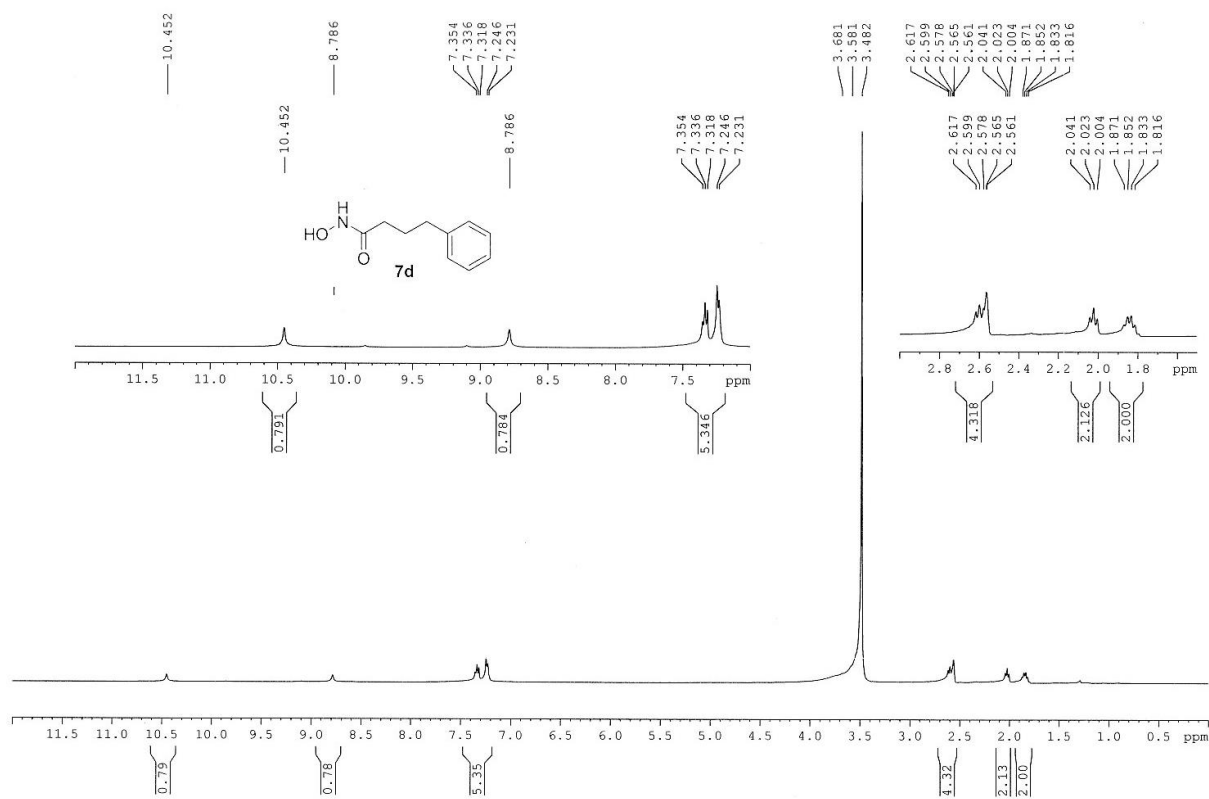


SS-2-31DR

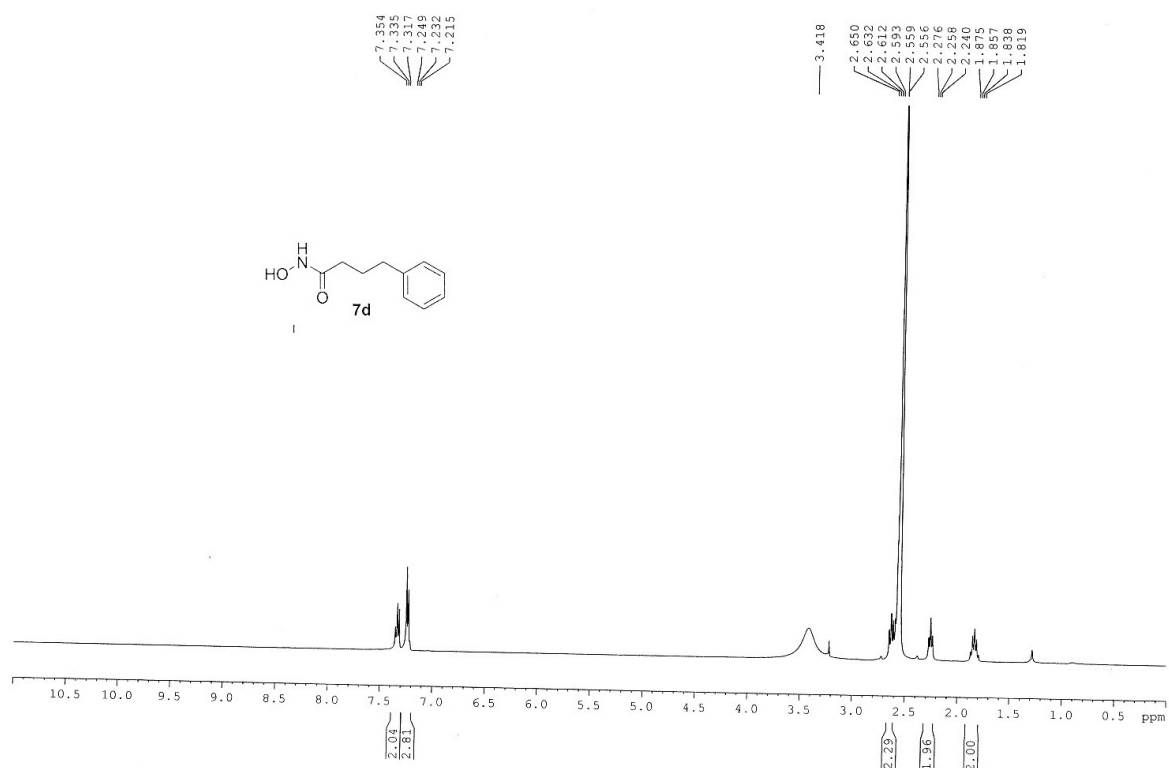
11.12.2015



¹³C NMR Spectrum of compound 7c in DMSO-*d*₆



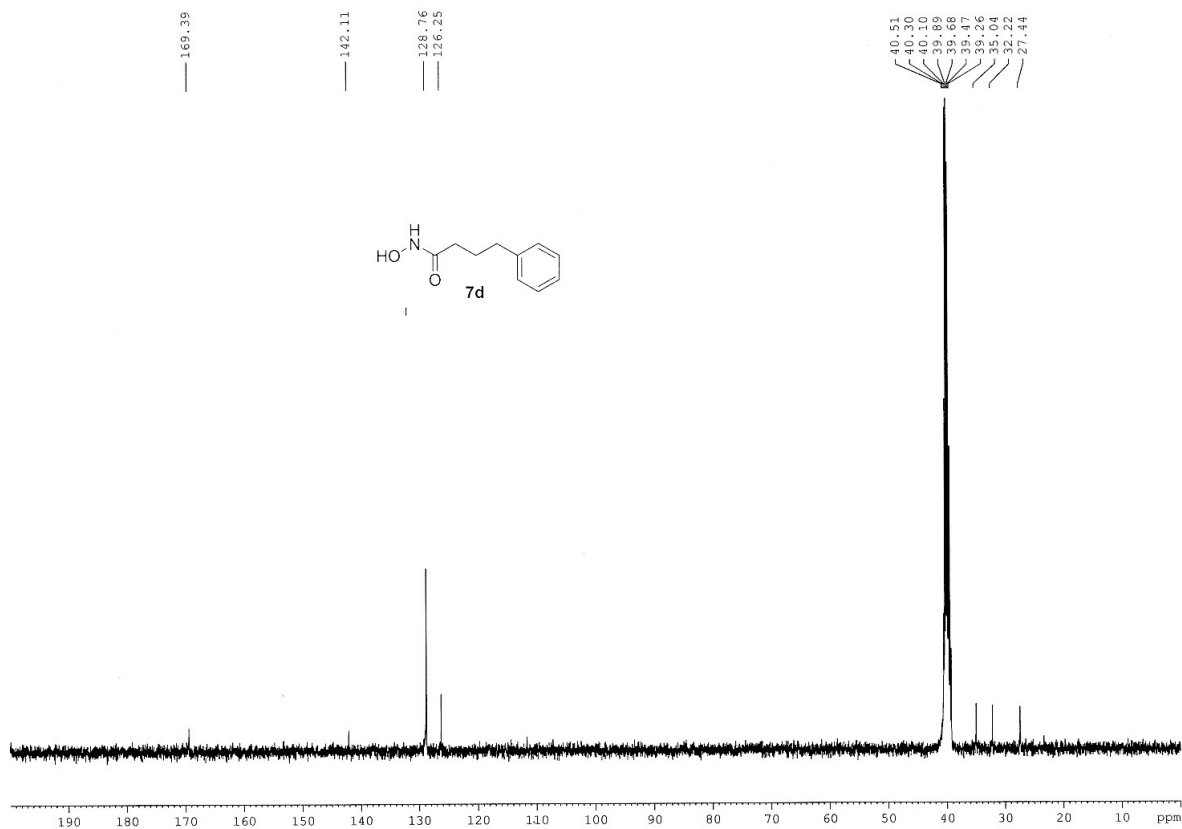
¹H NMR Spectrum of compound **7d** in DMSO-*d*₆



¹H NMR Spectrum of compound **7d** in dry DMSO-*d*₆

SG-10 (1)

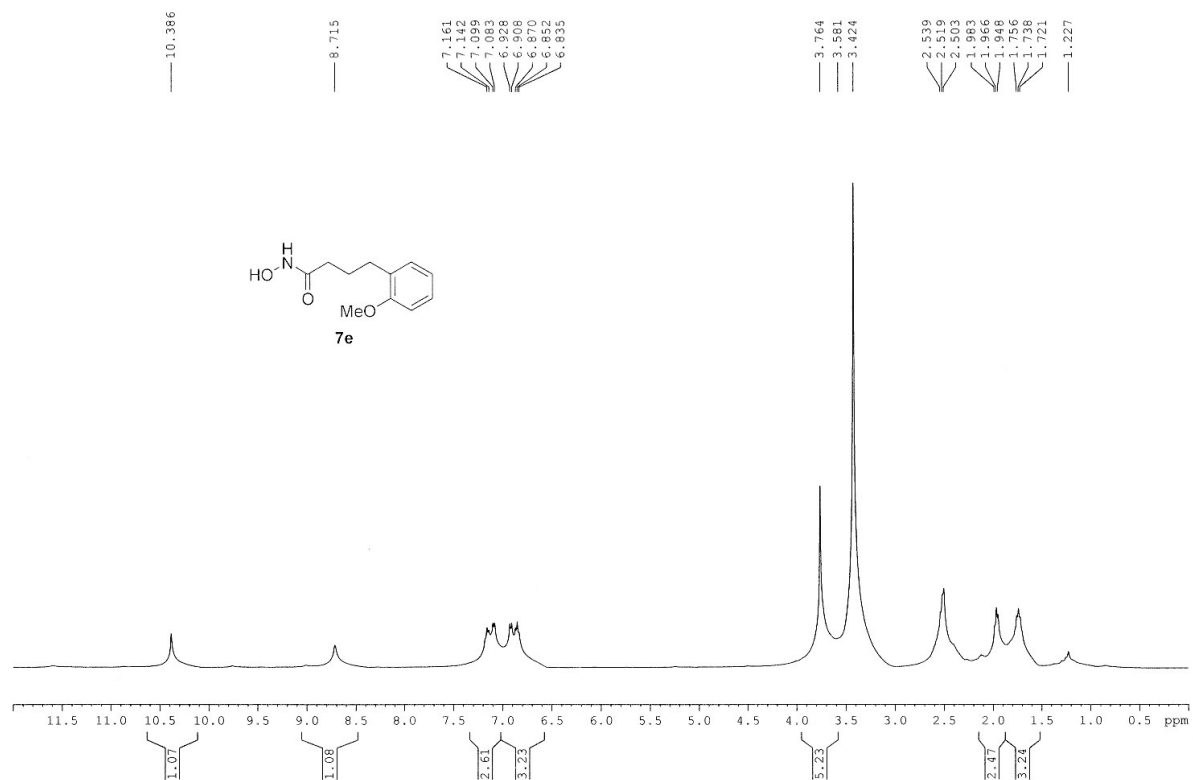
24.09.2018



¹³C NMR Spectrum of compound **7d** in DMSO-*d*₆

SS-2-55D

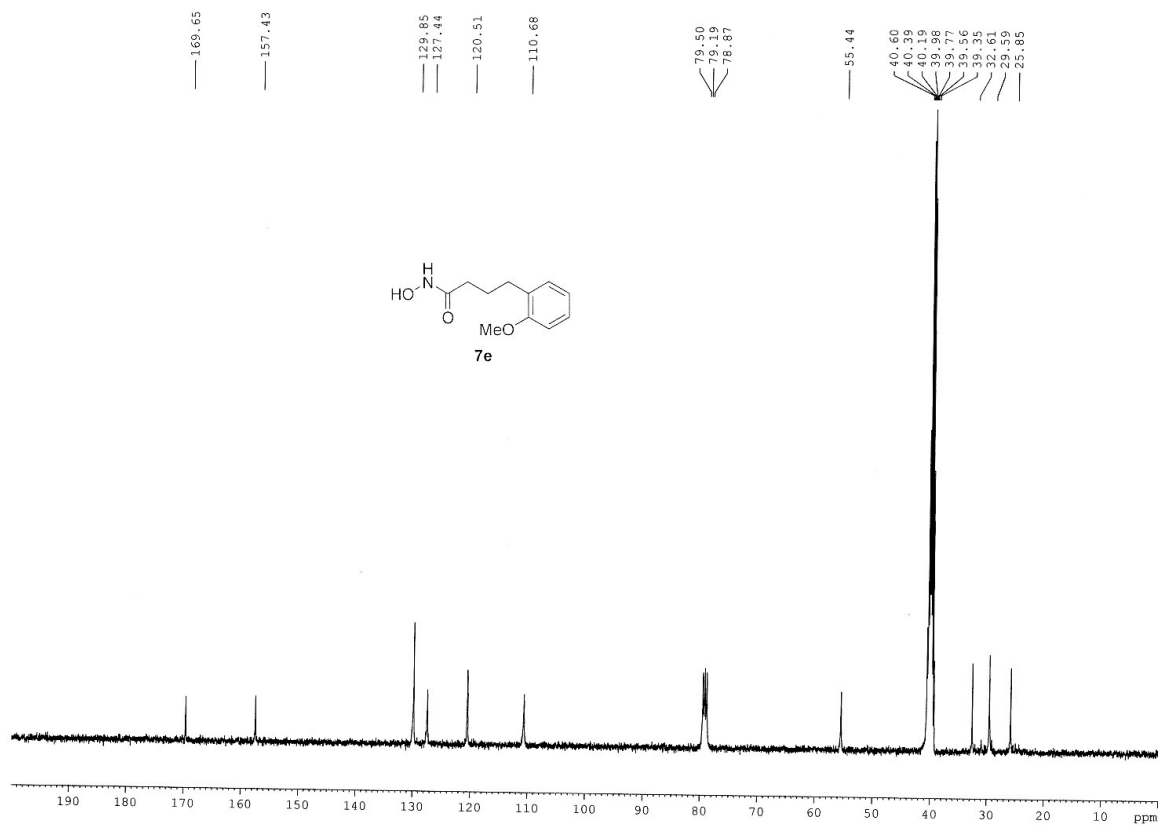
26.09.2018



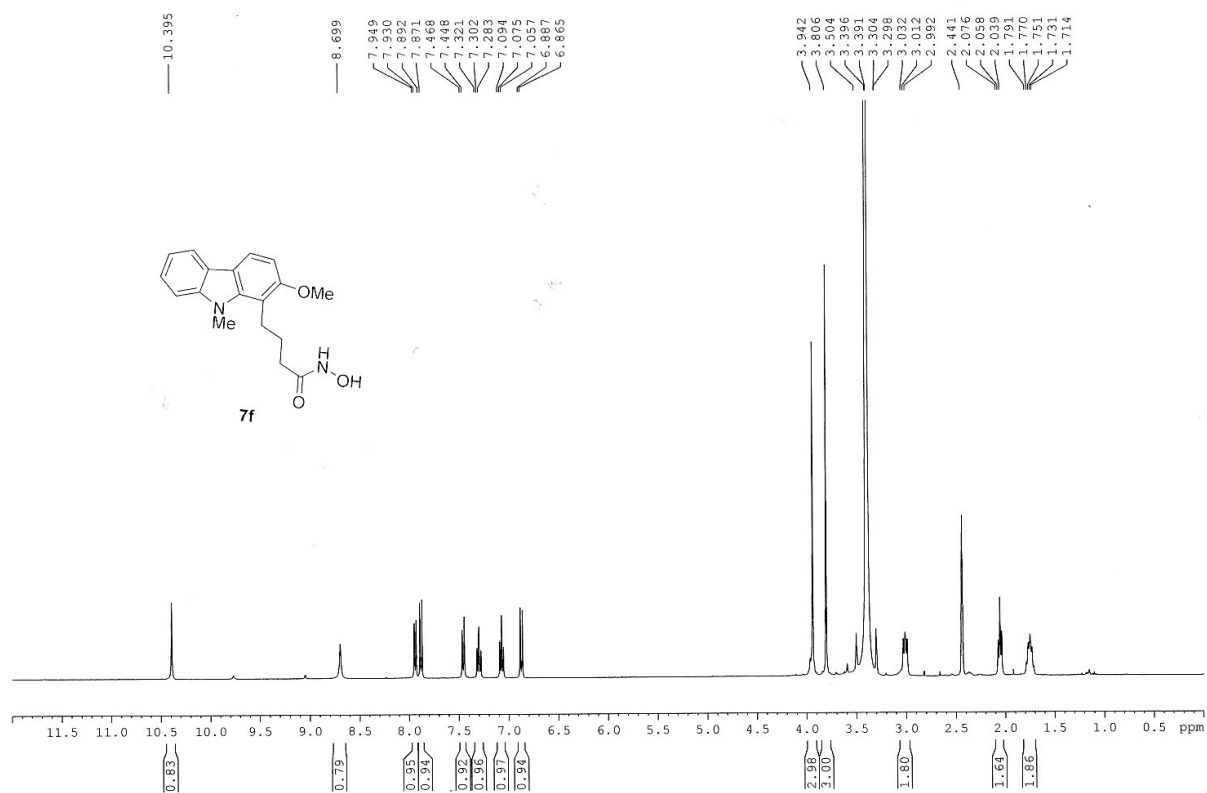
¹H NMR Spectrum of compound **7e** in DMSO-*d*₆

SS-2-55D

26.09.2018



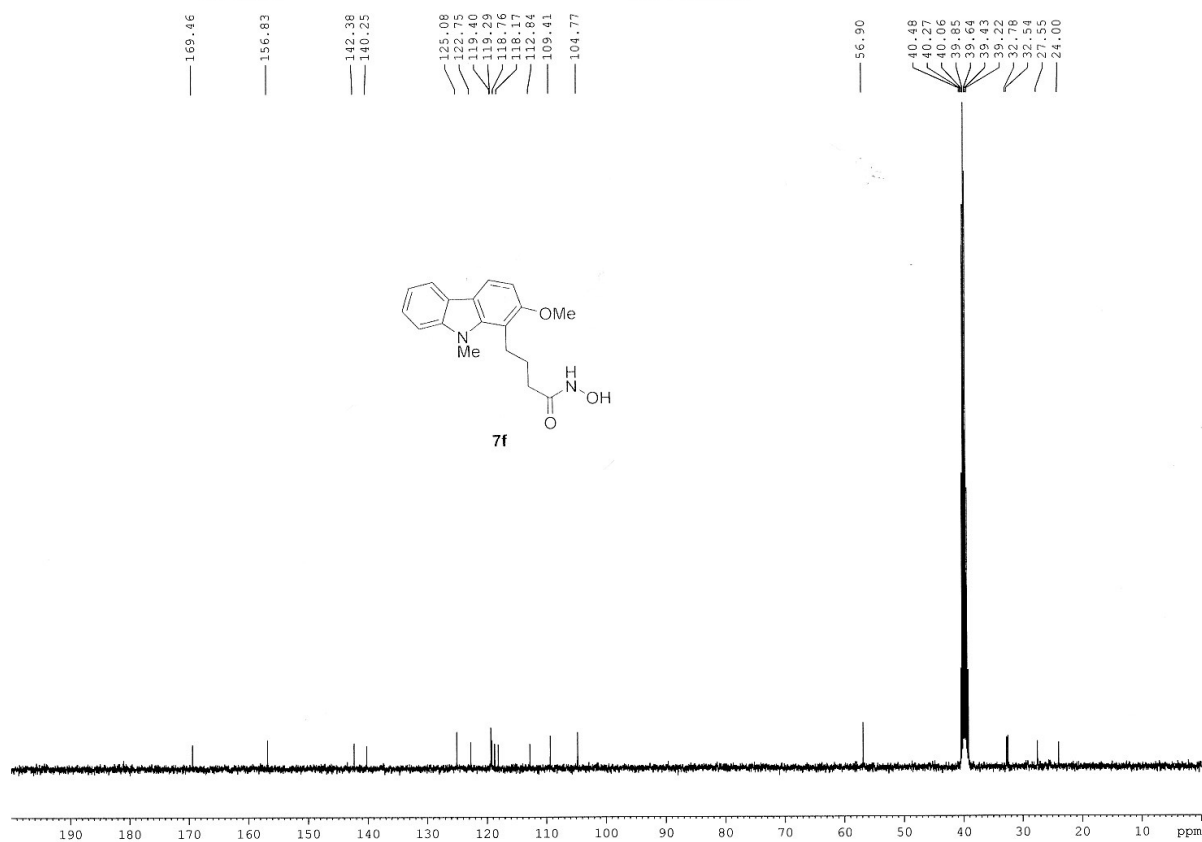
¹³C NMR Spectrum of compound **7e** in DMSO-*d*₆



¹H NMR Spectrum of compound **7f** in DMSO-*d*₆

SG-2/82

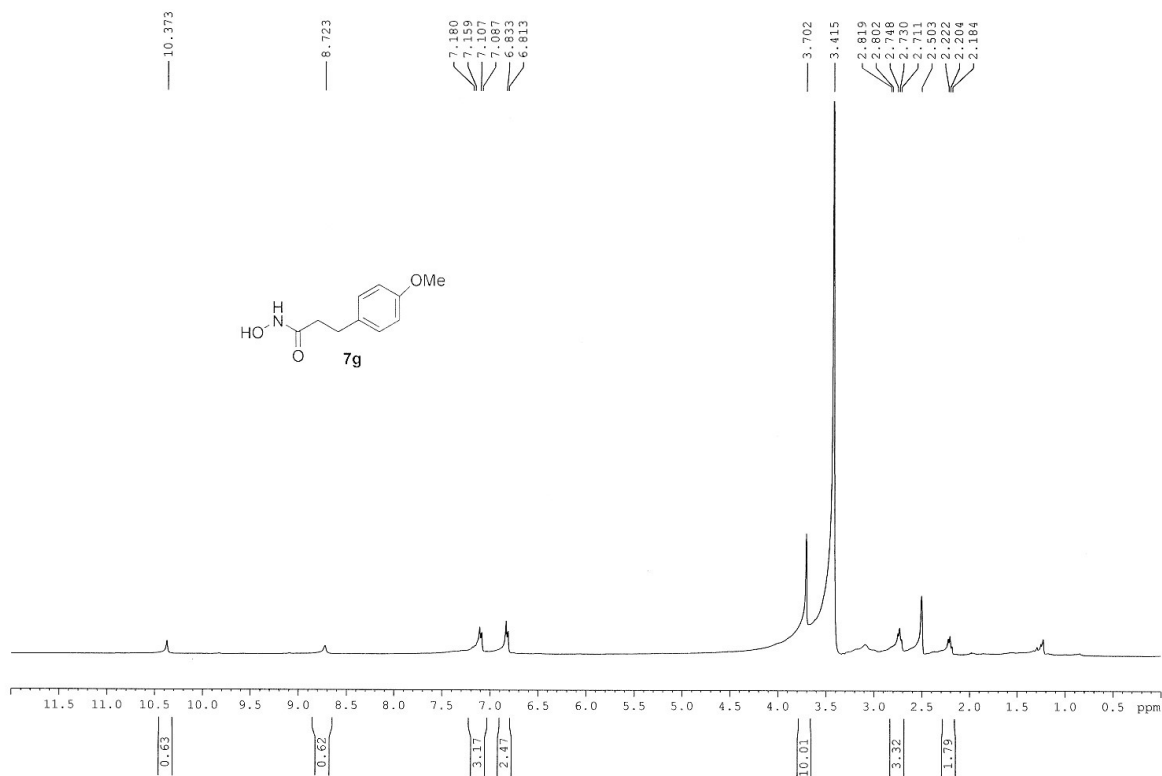
22.06.2016



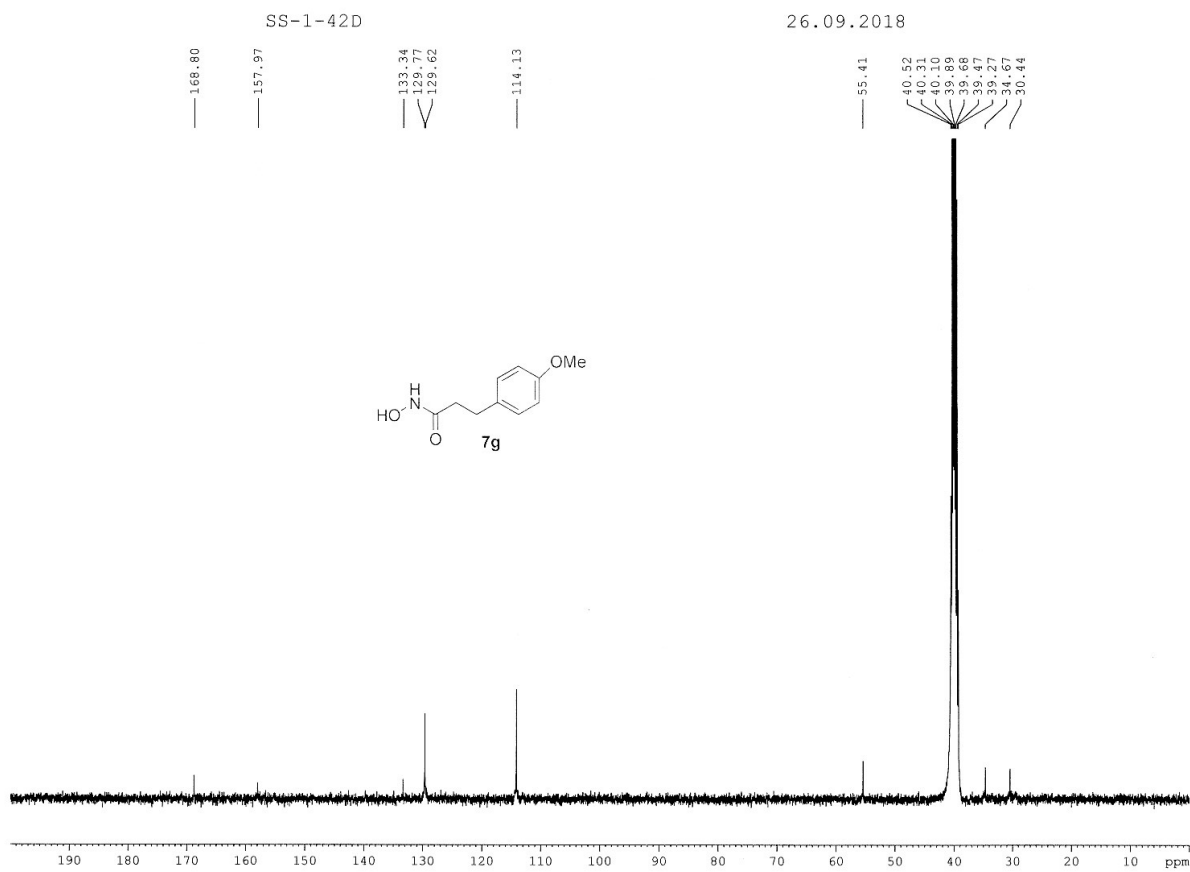
^{13}C NMR Spectrum of compound **7f** in $\text{DMSO}-d_6$

SS-1-42D

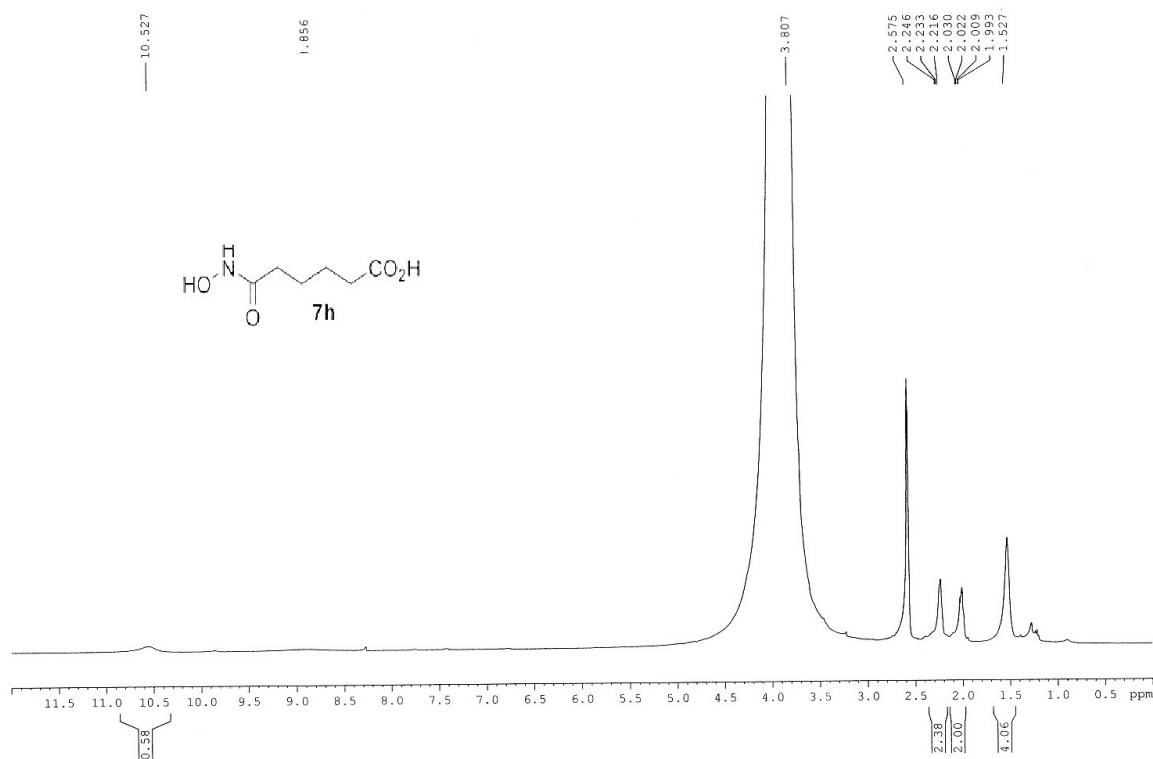
26.09.2018



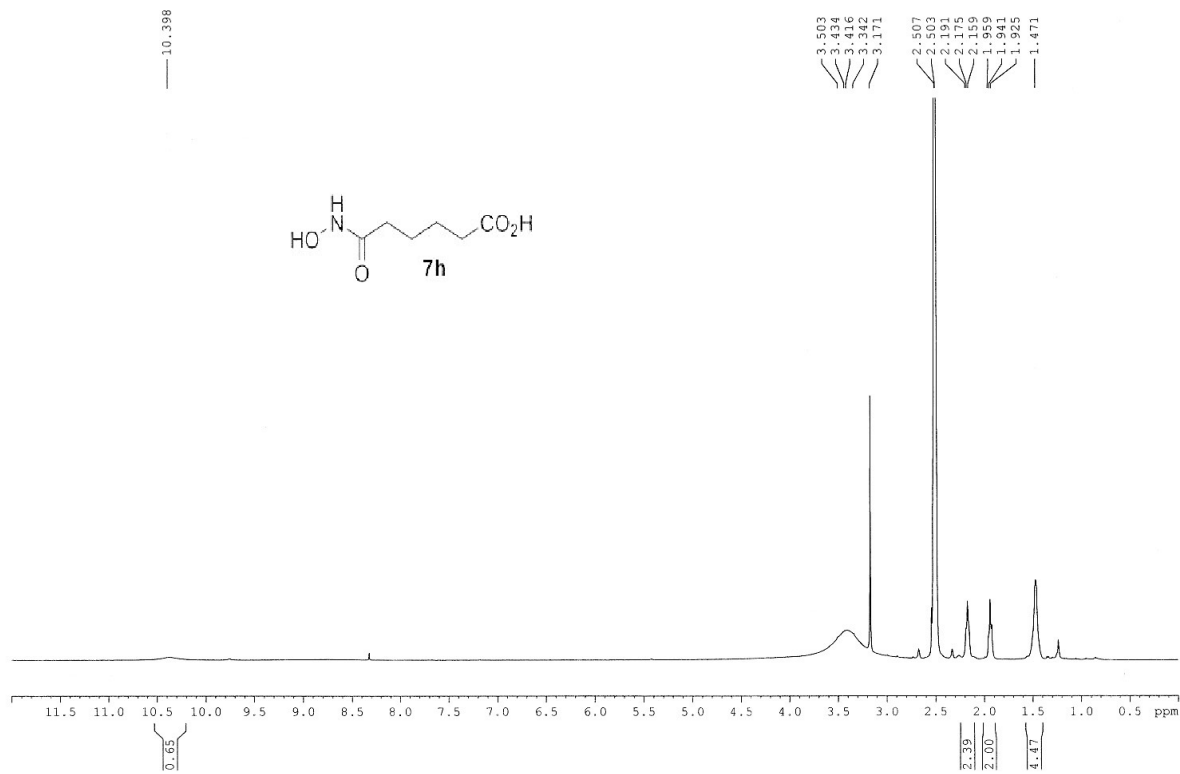
¹H NMR Spectrum of compound **7g** in DMSO-*d*₆



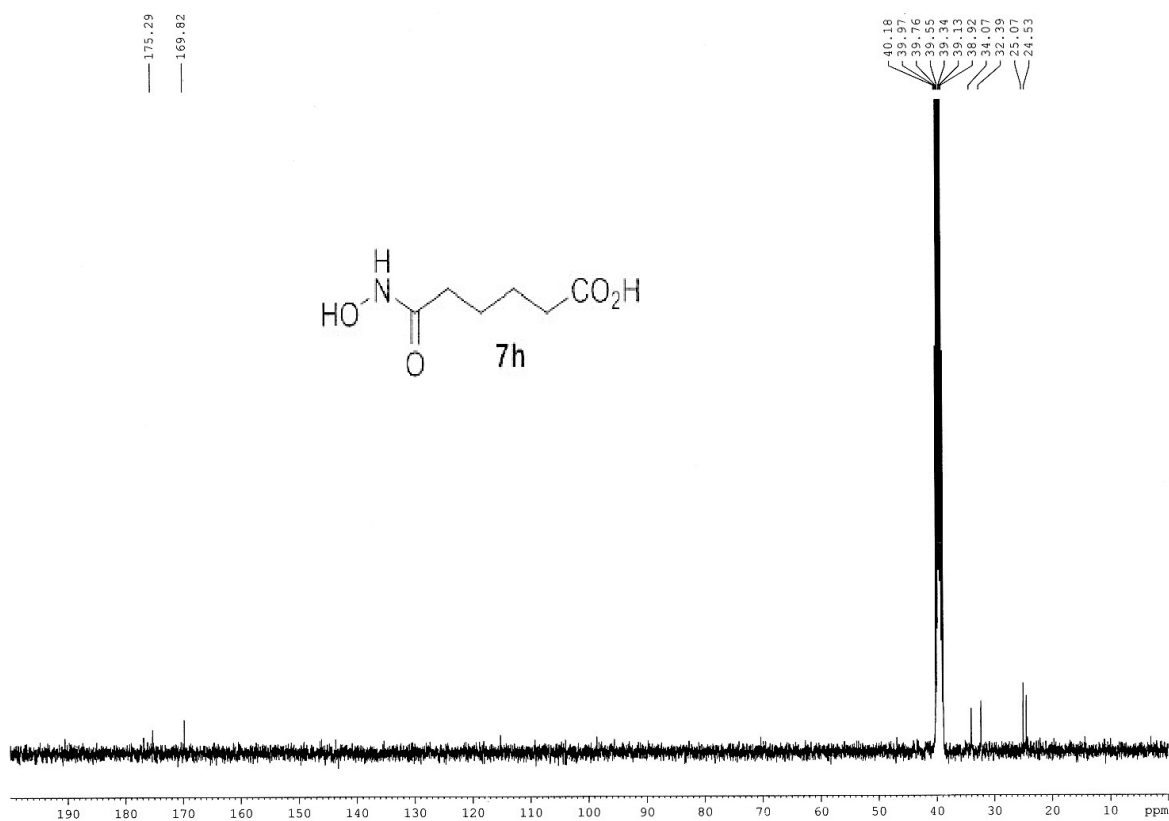
^{13}C NMR Spectrum of compound **7g** in $\text{DMSO-}d_6$



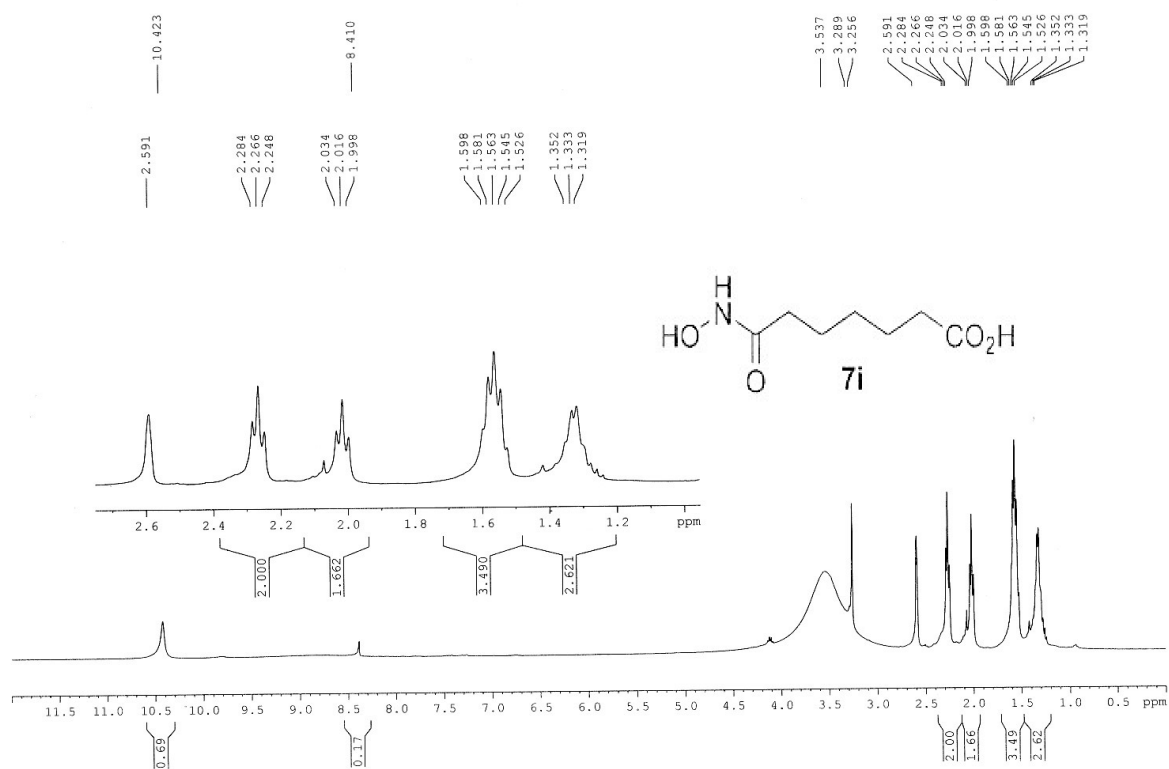
¹H NMR Spectrum of compound **7h** in DMSO-*d*₆



¹H NMR Spectrum of compound **7h** in dry DMSO-*d*₆



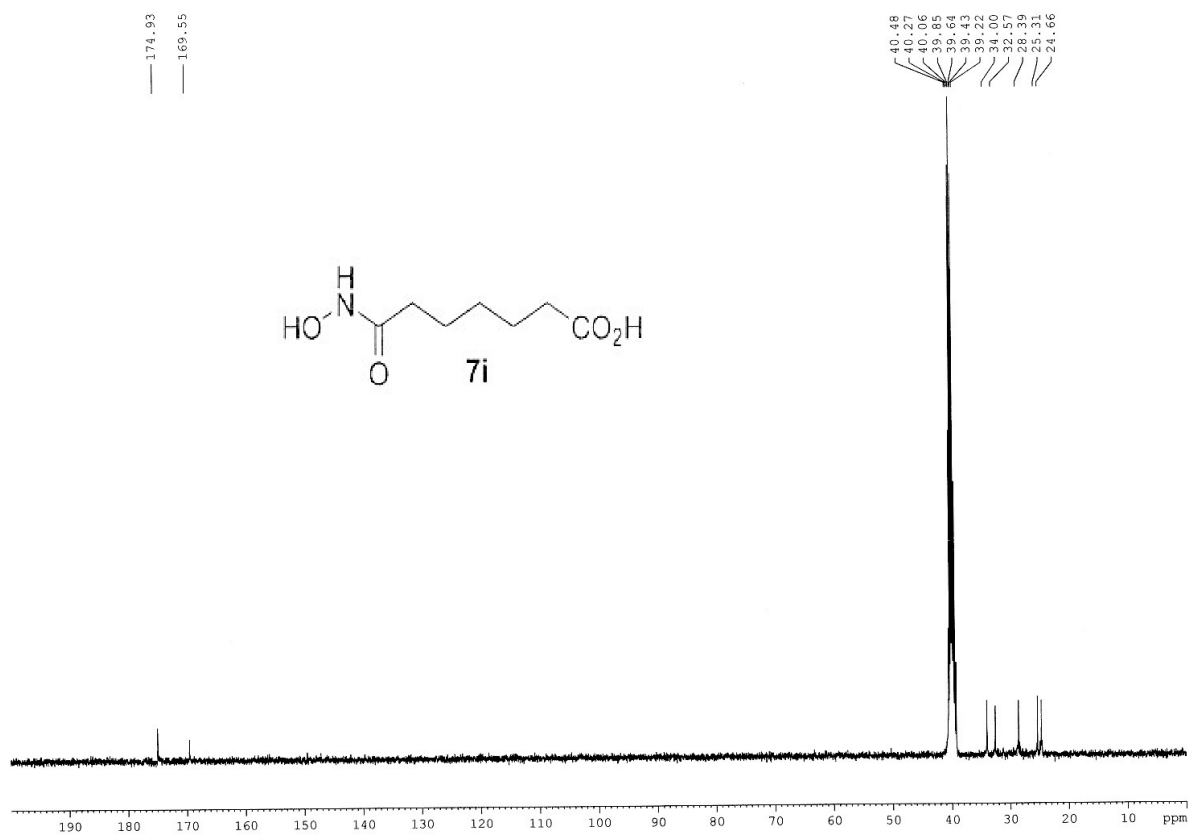
¹³C NMR Spectrum of compound **7h** in DMSO-*d*₆



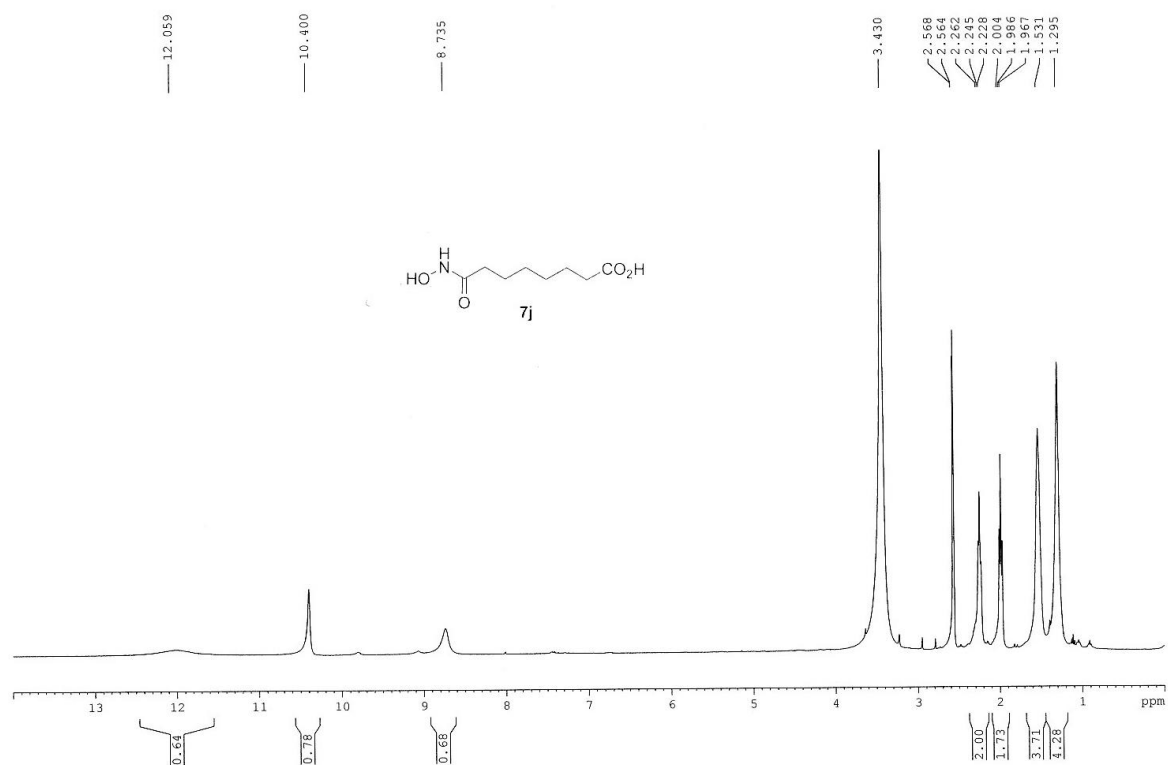
¹H NMR Spectrum of compound **7i** in DMSO-*d*₆

SS-2-36D

24.09.2018



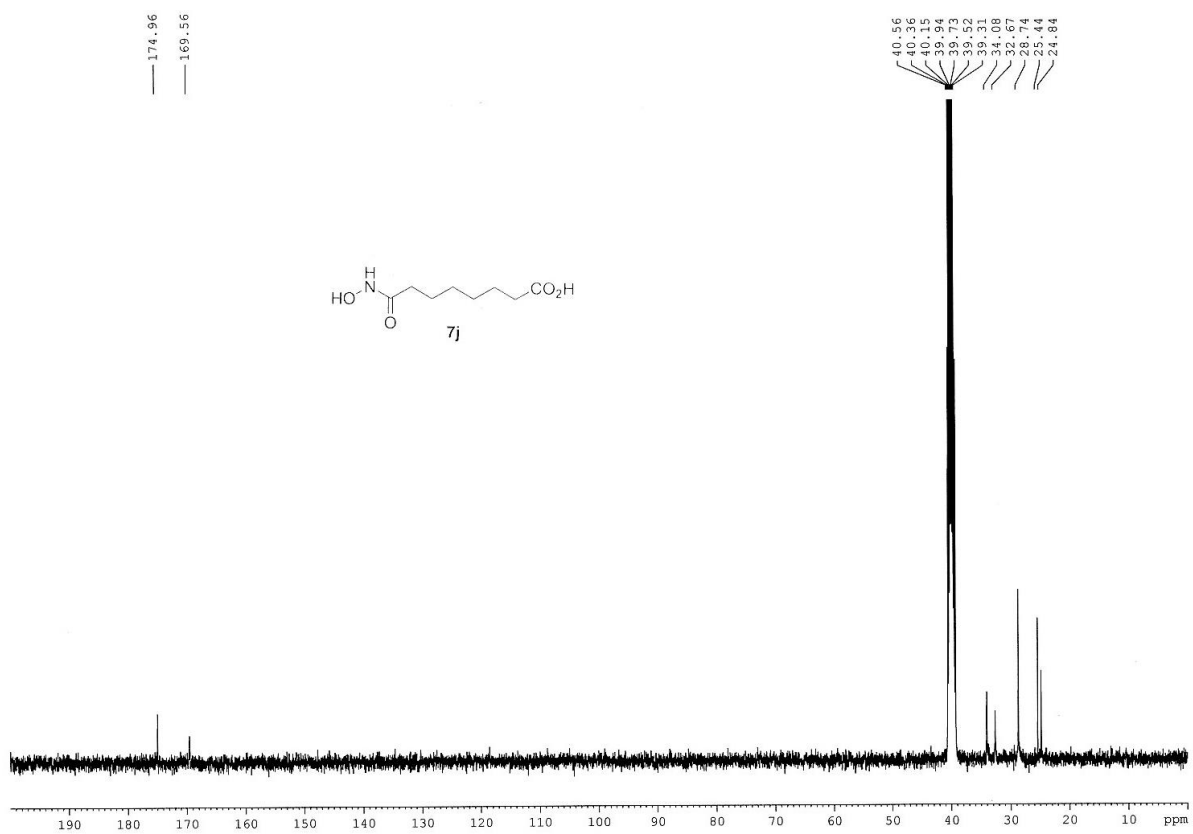
¹³C NMR Spectrum of compound **7i** in DMSO-*d*₆



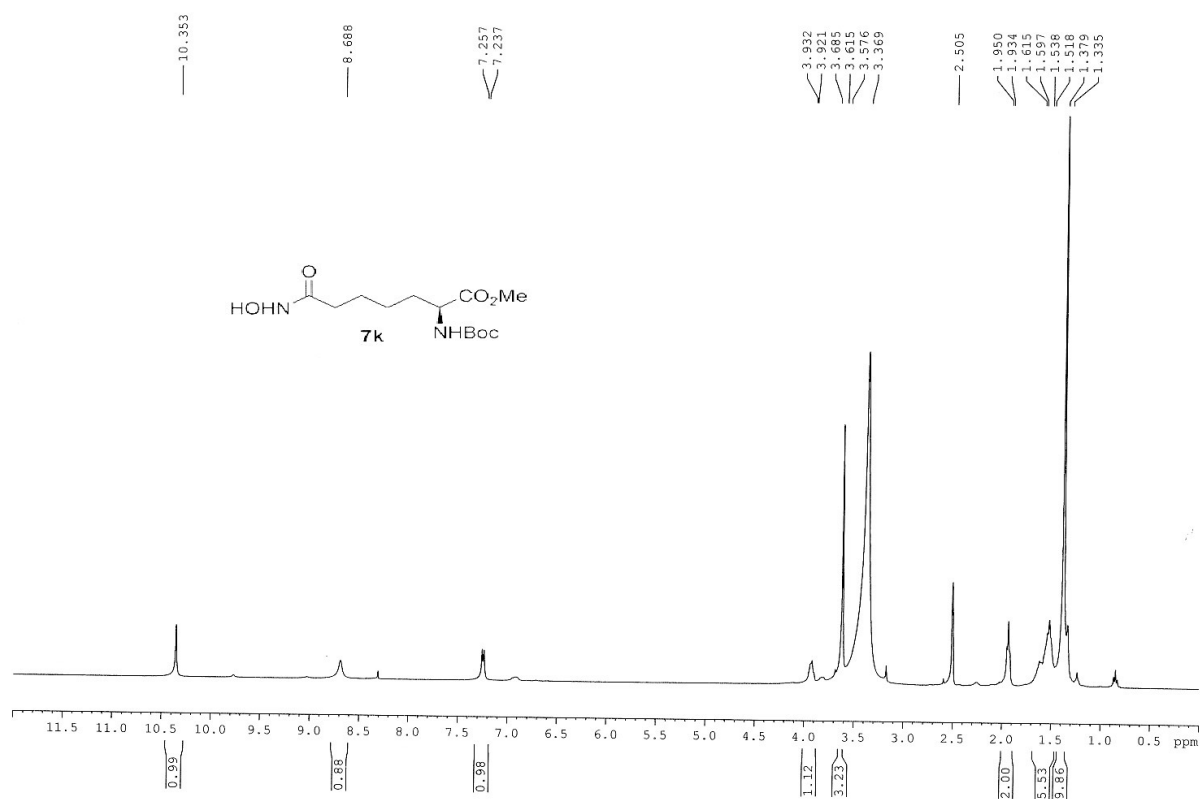
¹H NMR Spectrum of compound **7j** in DMSO-*d*₆

SG-3/139

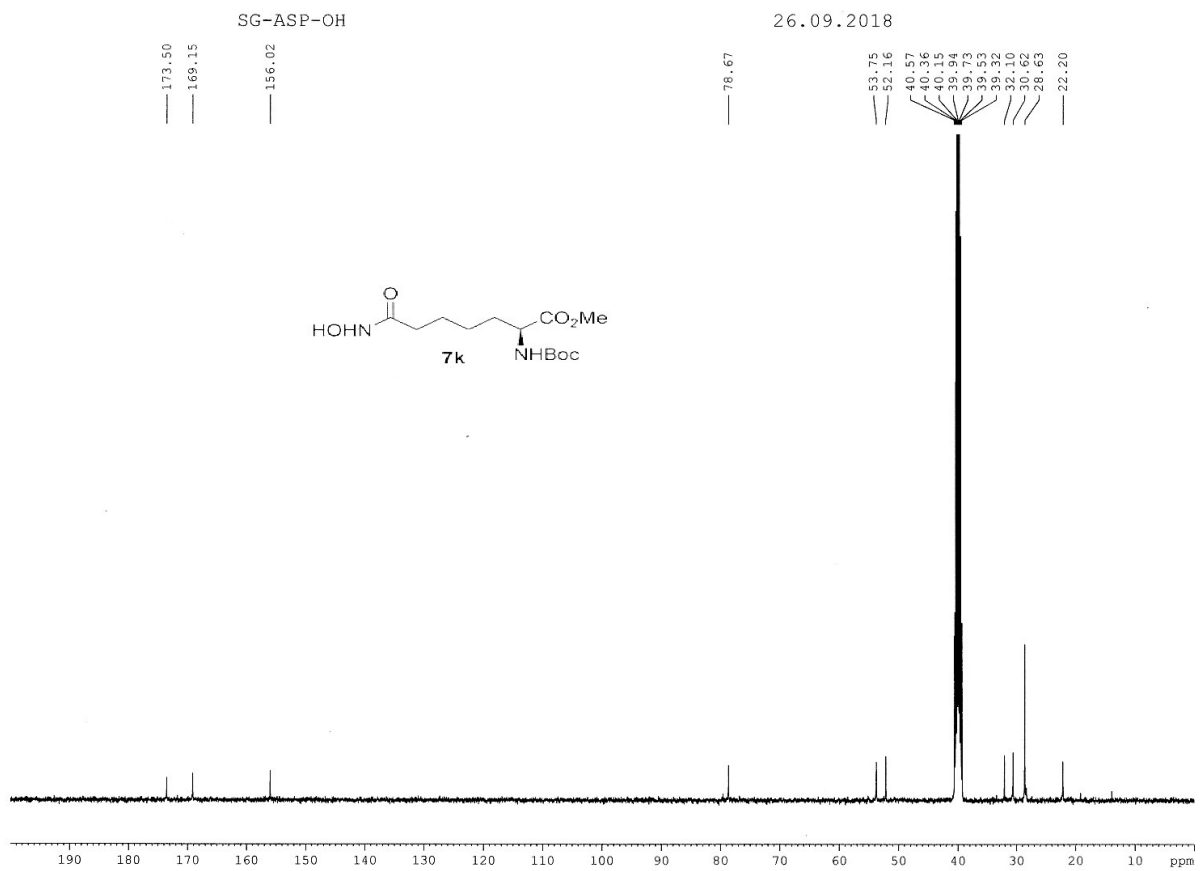
18.07.2018



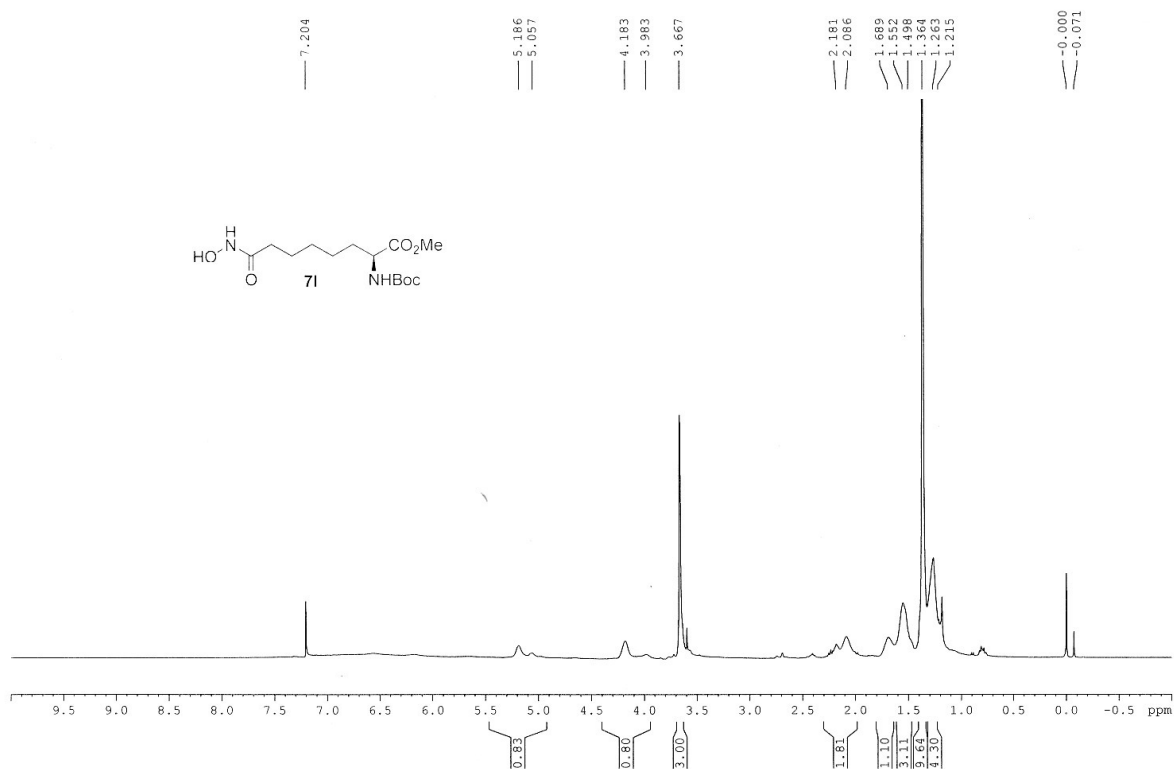
¹³C NMR Spectrum of compound **7j** in DMSO-*d*₆



¹H NMR Spectrum of compound **7k** in DMSO-*d*₆



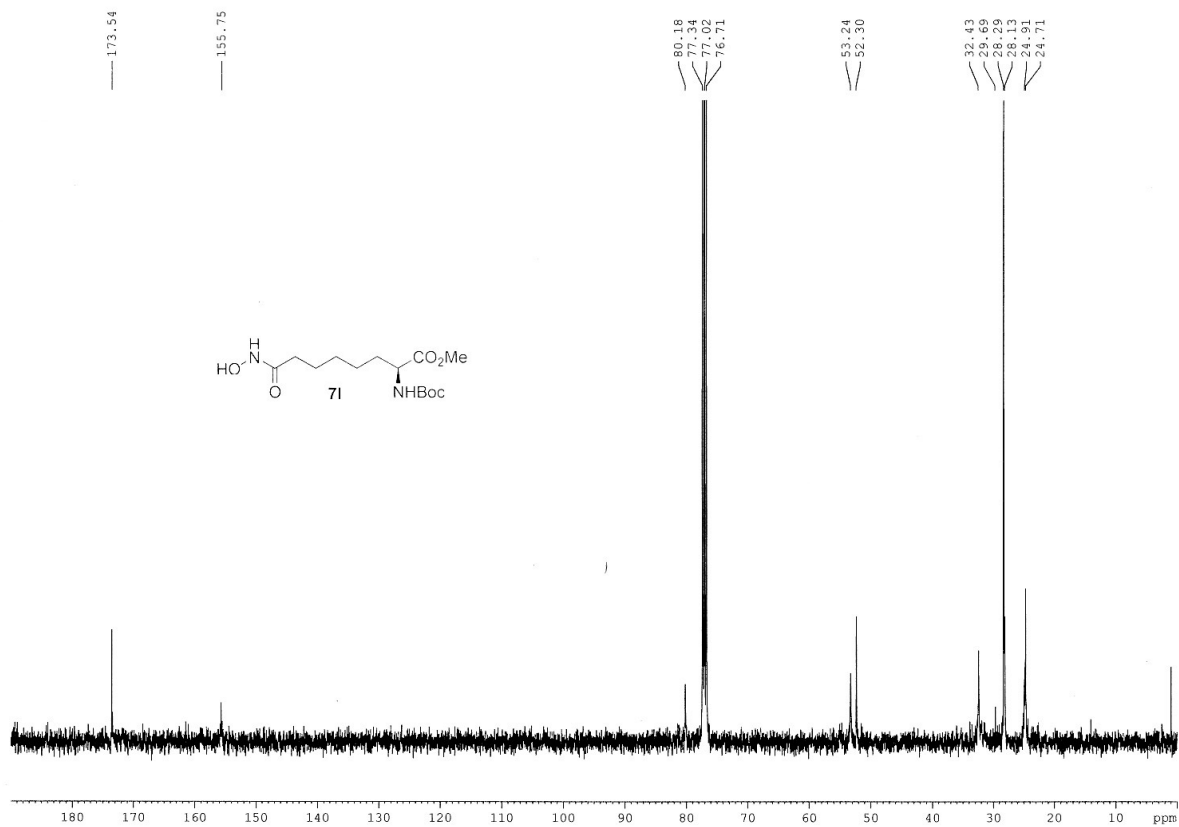
¹³C NMR Spectrum of compound **7k** in DMSO-*d*₆



¹H NMR Spectrum of compound **71** in CDCl₃

JPM-SS-1-23AHX

05.06.2015

 ^{13}C NMR Spectrum of compound **7I** in CDCl_3