Facile Construction of Highly Functionalized 2-Pyrrolines via $FeCl_3$-Catalyzed Reaction of Aziridines with Arylalkynes

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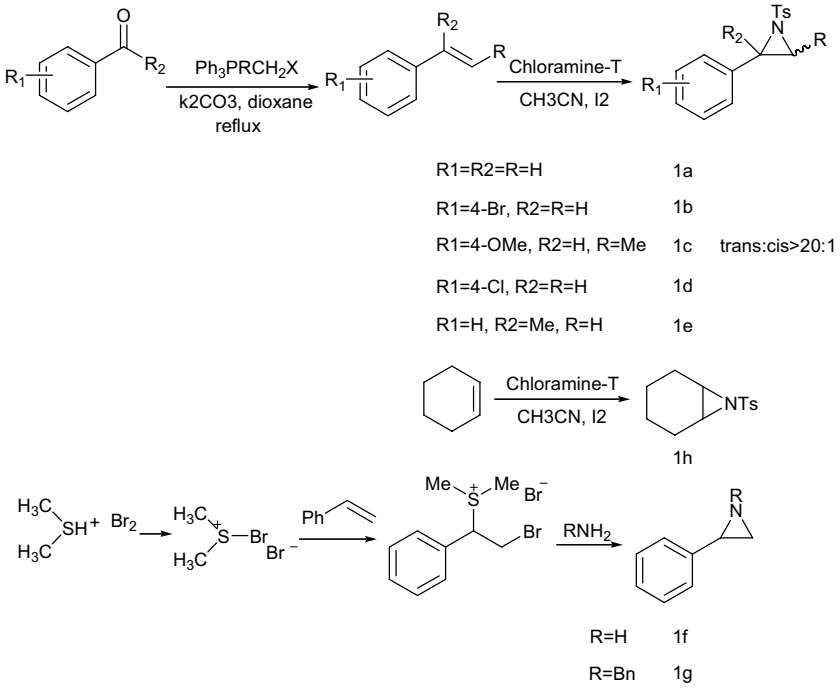
Contents:

General Remarks: All reactions were carried out in oven-dried flask sealed with rubber septa under a positive pressure of dry nitrogen unless otherwise indicated. Reactions were run using Teflon^{TM}-coated magnetic stir bars. Aziridines **1a-1e, 1h** were prepared following the procedure of M. Komatsu as steps in Scheme SI-1.[1] Aziridines **1f** and **1g** were prepared following the procedure of L. –N. He.[2] ^{1}H NMR and ^{13}C NMR were recorded on a Bruker AC-300 FT (^{1}H: 300 MHz, ^{13}C: 75 MHz) using TMS as internal reference. The chemical shifts (δ) and coupling constants (J) were expressed in ppm and Hz respectively. Infrared samples were recorded on a Perkin-Elmer 2000 FTIR spectrometer. HRMS were recorded on the TOF-HRMS–EI at the Instruments' Center for Physical Science, University of Science & Technology of China. Nitromethane, 1,2-dichloroethane and dichloromethane were distilled from CaH_2 and stored over 4 Å molsieves in screw-cap flasks. All commercially available reagents were used as received.

1. Synthesis of substrates 1a-1h

1.1 Synthesis of substrates 1a-1h[1, 2] (Scheme SI-1).

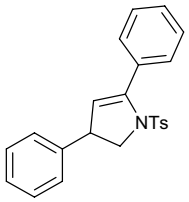


Scheme SI-1. synthesis of **1a-1h**.

1. T. Ando, D. Kano, S. Minakata, I. Ryu and M. Komatsu, *Tetrahedron.,* 1998, **54**, 13485.
2. Y. Du, Y. Wu, A. –H. Liu and L. –N. He, *J. Org. Chem.*, 2008, **73**, 4709.

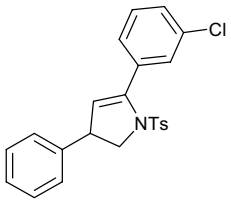
2. Characterization data of all products

3,5-diphenyl-1-tosyl-2,3-dihydro-1H-pyrrole (3a)



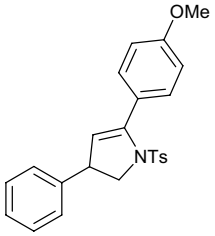
The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.63-7.60 (m, 2 H), 7.50 (d, J = 8.1 Hz, 2 H), 7.40-7.38 (m, 3 H), 7.23 (d, J = 8.1 Hz, 2 H), 7.17-7.15 (m, 3 H), 6.86-6.83 (m, 2 H), 5.43 (d, J = 2.7 Hz, 1 H), 4.45 (dd, J = 12.3, 9.9 Hz, 1 H), 3.83 (dd, J = 12.3, 8.1 Hz, 1 H), 3.71-3.64 (m, 1 H), 2.44 (s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 145.9, 144.0, 142.5, 133.5, 132.8, 129.6, 129.0, 128.6, 128.2, 127.9, 127.2, 126.9, 120.1, 59.8, 46.4, 21.7; IR (liquid film, cm^{-1}): ν = 3063, 3030, 1598, 1493, 1354, 1165, 1091, 1028, 910, 698; HRMS(EI-TOF) calc. $C_{23}H_{21}NO_2S$ (M^+): 375.1293. Found: 375.1290.

5-(3-chlorophenyl)-3-phenyl-1-tosyl-2,3-dihydro-1H-pyrrole (3b)



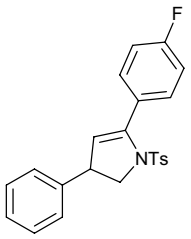
The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.54-7.48 (m, 3 H), 7.37-7.31 (m, 3 H), 7.29-7.23 (m, 3 H), 7.20-7.16 (m , 2 H), 6.85-6.83 (m, 2 H), 5.47 (d, J = 2.7 Hz , 1 H), 4.44 (dd, J = 12.3, 9.6 Hz, 1 H), 3.83 (dd, J = 12.3, 8.4 Hz, 1 H), 3.70-3.61 (m, 1 H), 2.45 (s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 144.7, 144.1, 142.1, 134.5, 133.8, 133.5, 129.6, 129.2, 129.0, 128.7, 128.1, 127.9, 127.2, 127.0, 126.4, 121.1, 59.6, 46.4, 21.6; IR (liquid film, cm^{-1}): ν = 3064, 3029, 1596, 1494, 1355, 1166, 1090, 1022, 910, 699; HRMS(EI-TOF) calc. $C_{23}H_{20}ClNO_2S$ (M^+): 409.0903. Found: 409.0906.

5-(4-methoxyphenyl)-3-phenyl-1-tosyl-2,3-dihydro-1H-pyrrole (3c)



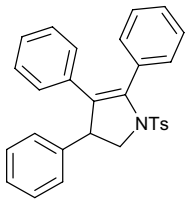
The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.54 (d, J = 8.7 Hz, 2 H), 7.47 (d, J = 8.1 Hz, 2 H), 7.22-7.13 (m, 5 H), 6.91 (d, J = 8.7 Hz, 2 H), 6.85-6.80 (m, 2 H), 5.32 (d, J = 2.4 Hz, 1 H), 4.42 (dd, J = 12.3, 9.6 Hz, 1 H), 3.82 (s, 3 H), 3.81-3.77 (m, 1 H), 3.68-3.62 (m, 1 H), 2.41 (s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 160.2, 145.6, 143.9, 142.7, 133.7, 129.8, 129.6, 129.5, 128.6, 128.1, 127.2, 126.8, 118.4, 113.4, 59.8, 55.3, 46.2, 21.7; IR (liquid film, cm^{-1}): ν = 3062, 3030, 1606, 1510, 1353, 1165, 1090, 1030, 911, 701; HRMS(EI-TOF) calc. $C_{24}H_{23}NO_3S$ (M^+): 405.1399. Found: 405.1397.

5-(4-fluorophenyl)-3-phenyl-1-tosyl-2,3-dihydro-1H-pyrrole (3d)



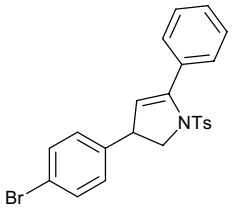
The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.58-7.56 (m, 2 H), 7.47 (d, J = 8.1 Hz, 2 H), 7.25-7.14 (m, 5 H), 7.09-7.04 (m, 2 H), 6.85-6.82 (m, 2 H), 5.38 (d, J = 2.4 Hz, 1 H), 4.43 (dd, J = 12.6, 9.9 Hz, 1 H), 3.82 (dd, J = 12.3, 8.1 Hz, 1 H), 3.71-3.67 (m, 1 H), 2.43 (s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 164.8, 161.5, 144.9, 144.1, 142.3, 133.5, 130.0, 129.9, 129.6, 129.3, 129.0, 128.6, 128.1, 127.2, 126.9, 119.8, 115.1, 114.8, 59.7, 49.3, 21.6; IR (liquid film, cm^{-1}): ν = 3063, 3029, 1600, 1507, 1355, 1167, 1089, 1015, 910, 701; HRMS(EI-TOF) calc. $C_{23}H_{20}FNO_2S$ (M^+): 393.1199. Found: 393.1197.

3,4,5-triphenyl-1-tosyl-2,3-dihydro-1H-pyrrole (3e)



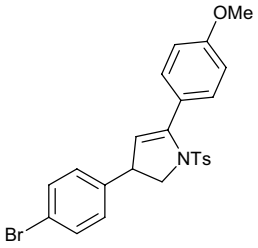
The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.48 (d, J = 8.4 Hz, 2 H), 7.39-7.36 (m, 2 H), 7.33-7.20 (m, 5 H), 7.11-7.09 (m, 3 H), 6.94-6.89 (m, 5 H), 6.65-6.62 (m, 2 H), 4.49 (dd, J = 11.7, 9.6 Hz, 1 H), 4.07 (dd, J = 9.6, 6.9 Hz, 1 H), 3.71-3.64 (dd, J = 11.7, 6.9 Hz, 1 H), 2.45 (s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 143.8, 142.3, 139.9, 135.1, 134.6, 132.1, 130.7, 129.9, 129.5, 128.8, 128.7, 128.2, 128.0, 127.9, 127.8, 127.7, 126.8, 126.5, 58.6, 50.9, 21.7; IR (liquid film, cm^{-1}): ν = 3060, 3027, 1598, 1493, 1358, 1166, 1090, 1029, 910, 697; HRMS(EI-TOF) calc. $C_{29}H_{25}NO_2S$ (M^+): 451.1606. Found: 451.1603.

3-(4-bromophenyl)-5-phenyl-1-tosyl-2,3-dihydro-1H-pyrrole (3h)



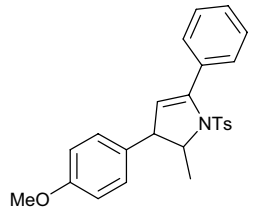
The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.63-7.56 (m, 2 H), 7.44-7.38 (m, 5 H), 7.26-7.17 (m, 4 H), 6.71-6.68 (m, 3 H), 5.38 (d, J = 2.4 Hz, 1 H), 4.45 (dd, J = 11.4, 9.9 Hz, 1 H), 3.83 (dd, J = 11.7, 6.9 Hz, 1 H), 3.71-3.67 (m, 1 H), 2.43 (s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 146.5, 144.1, 141.7, 133.5, 132.5, 131.6, 129.5, 129.2, 128.9, 128.2, 128.1, 127.9, 120.6, 118.8, 59.5, 45.5, 21.7; IR (liquid film, cm^{-1}): ν = 3063, 3025, 1597, 1487, 1356, 1166, 1072, 1010, 917, 687; HRMS(EI-TOF) calc. $C_{23}H_{20}BrNO_2S$ (M^+): 453.0398. Found: 453.0395.

3-(4-bromophenyl)-5-(4-methoxyphenyl)-1-tosyl-2,3-dihydro-1H-pyrrole (3i)



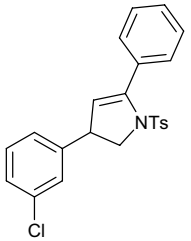
The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.54 (d, J = 8.4 Hz, 2 H), 7.42 (d, J = 8.1 Hz, 2 H), 7.23 (d, J = 7.5 Hz, 2 H), 7.17 (d, J = 8.1 Hz, 2 H), 6.92 (d, J = 8.7 Hz, 2 H), 6.69 (d, J = 8.4 Hz, 2 H), 5.29 (d, J = 2.4 Hz, 1 H), 4.42 (dd, J = 12.6, 9.9 Hz, 1 H), 3.83 (dd, J = 12.6, 7.5 Hz, 1 H), 3.66-3.63 (m, 1 H), 2.43 (s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 160.4, 146.2, 144.1, 142.0, 133.6, 131.6, 129.7, 129.5, 128.9, 128.1, 125.0, 120.5, 117.2, 113.4, 59.6, 55.4, 45.4, 21.7; IR (liquid film, cm^{-1}): v = 3063, 3028, 1606, 1509, 1354, 1165, 1089, 1010, 909, 811, 678; HRMS(EI-TOF) calc. $C_{24}H_{22}BrNO_3S$ (M^+): 483.0504. Found: 483.0504.

3-(4-methoxyphenyl)-2-methyl-5-phenyl-1-tosyl-2,3-dihydro-1H-pyrrole (syn: anti = 15:2) (3j)



The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.63-7.61 (m, 1 H), 7.42-7.39 (m, 3 H), 7.34-7.26 (m, 2 H), 7.12 (d, J = 8.1 Hz, 2 H), 6.55 (d, J = 4.5 Hz, 2 H), 6.37 (d, J = 8.7 Hz, 2 H), 5.21 (d, J = 3.3 Hz, 1 H), 3.96 (dd, J = 6.3, 3.6 Hz, 1 H), 3.77 (s, 3 H), 3.39 (dd, J = 3.6, 3.3 Hz, 1 H), 2.43 (s, 3 H), 1.62 (d, J = 6.3 Hz, 1 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 158.3, 144.5, 143.6, 135.3, 133.7, 133.3, 130.5, 129.9, 129.5, 128.5, 128.3, 127.9, 116.5, 113.8, 67.7, 55.3, 53.7, 24.4, 21.7; IR (liquid film, cm^{-1}): v = 3063, 3027, 1609, 1511, 1352, 1166, 1091, 1031, 917, 813, 693; HRMS(EI-TOF) calc. $C_{25}H_{25}NO_3S$ (M^+): 419.1555. Found: 419.1552.

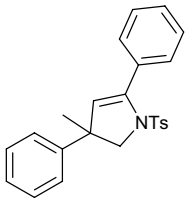
3-(3-chlorophenyl)-5-phenyl-1-tosyl-2,3-dihydro-1H-pyrrole (3k)



The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.64-7.61 (m, 2 H), 7.48-7.40 (m, 5 H), 7.26-7.20 (m, 2 H), 7.14-7.11 (m, 2 H), 6.80-6.77 (m, 2 H), 5.38 (d, J = 2.7 Hz, 1 H), 4.46 (dd, J = 12.6, 9.9 Hz, 1 H), 3.84 (dd, J = 12.6, 7.5 Hz, 1 H), 3.75-3.71 (m, 1 H), 2.44 (s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 146.6, 144.8, 144.2, 134.5, 133.3, 132.5, 129.9, 129.5, 129.2, 128.2, 128.1, 127.9, 127.3, 127.0, 125.5, 118.5, 59.3, 45.7, 21.7; IR (liquid film, cm^{-1}): v = 3063, 3030, 1596, 1492, 1356, 1166, 1089, 1026, 917, 693; HRMS(EI-TOF) calc.

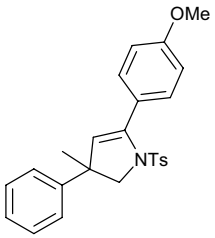
$C_{23}H_{20}ClNO_2S$ (M^+): 409.0903. Found: 409.0907.

3-methyl-3,5-diphenyl-1-tosyl-2,3-dihydro-1H-pyrrole (3l)



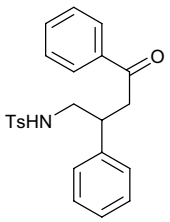
The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.62-7.59 (m, 2 H), 7.39-7.30 (m, 5 H), 7.14-7.12 (m, 3 H), 7.04 (d, J = 8.1 Hz, 2 H), 6.97-6.94 (m, 2 H), 5.40 (s, 1 H), 4.17 (d, J = 12.0 Hz, 1 H), 4.06 (d, J = 12.0 Hz, 1 H), 2.34 (s, 3 H), 1.24 (s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 147.7, 143.8, 143.6, 133.5, 132.8, 129.3, 128.9, 128.5, 128.4, 128.0, 127.8, 126.0, 125.6, 123.7, 65.7, 48.3, 29.0, 21.6; IR (liquid film, cm^{-1}): ν = 3059, 3027, 1598, 1493, 1446, 1354, 1166, 1090, 1028, 912, 759, 699; HRMS(EI-TOF) calc. $C_{24}H_{23}NO_2S$ (M^+): 389.1449. Found: 389.1447.

5-(4-methoxyphenyl)-3-methyl-3-phenyl-1-tosyl-2,3-dihydro-1H-pyrrole (3m)



The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.54(d, J = 8.4 Hz, 2 H), 7.29 (d, J = 8.1 Hz, 2 H), 7.13-7.10 (m, 3 H), 7.02 (d, J = 8.1 Hz, 2 H), 6.95-6.90 (m, 4 H), 5.31 (s, 1 H), 4.17 (d, J = 12.3 Hz, 1 H), 4.04 (d, J = 12.3 Hz, 1 H), 3.83 (s, 3 H), 2.32 (s, 3 H), 1.21 (s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 160.2, 147.9, 143.52, 143.48, 133.5, 129.9, 129.3, 128.4, 127.9, 125.9, 125.6, 125.1, 122.3, 113.3, 65.7, 55.3, 48.2, 29.2, 21.6; IR (liquid film, cm^{-1}): ν = 3059, 3024, 1607, 1509, 1461, 1355, 1251, 1166, 1090, 1031, 908, 759, 699; HRMS(EI-TOF) calc. $C_{25}H_{25}NO_3S$ (M^+): 419.1555. Found: 419.1557.

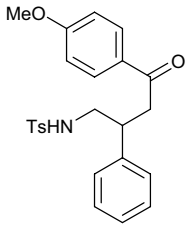
4-methyl-N-(4-oxo-2,4-diphenylbutyl)benzenesulfonamide (4a)



The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.65 (d, J = 8.1 Hz, 2 H), 7.51 (d, J = 7.2 Hz, 2 H), 7.56-7.47 (m, 1 H), 7.42-7.37 (m, 2 H), 7.26-7.18 (m, 5 H), 7.12 (d, J = 6.9 Hz, 2 H), 4.80 (t, J = 6.0 Hz, 1 H), 3.53-3.49 (m, 1 H), 3.37-3.26 (m, 3 H), 3.20-3.12 (m, 1 H), 2.36 (s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 198.3, 143.4, 141.2, 136.9, 136.8, 133.4, 129.7, 128.9, 128.6, 128.1, 127.7, 127.3, 127.1, 48.2, 42.1, 40.3, 21.5; IR (liquid film, cm^{-1}): ν = 3307, 2959, 1678, 1596, 1463, 1381, 1215, 1154, 927, 812, 761, 669;

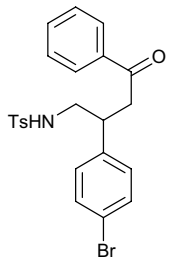
HRMS(EI-TOF) calc. $C_{23}H_{23}NO_3S$ (M^+): 393.1399. Found: 393.1396.

N-(4-(4-methoxyphenyl)-4-oxo-2-phenylbutyl)-4-methylbenzenesulfonamide (4b)



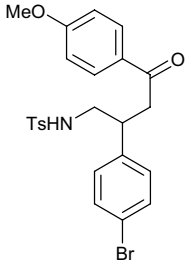
The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.83 (d, J = 9.0 Hz, 2 H), 7.65 (d, J = 8.1 Hz, 2 H), 7.25-7.11 (m, 7 H), 6.85 (d, J = 8.7 Hz, 2 H), 5.01 (t, J = 6.0 Hz, 1 H), 3.80 (s, 3 H), 3.52-3.47 (m, 1 H), 3.30-3.24 (m, 3 H), 3.21-3.13 (m, 1 H), 2.36 (s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 197.0, 163.6, 143.3, 141.4, 136.9, 130.5, 129.8, 129.7, 128.9, 127.7, 127.2, 127.1, 113.8, 55.5, 48.3, 41.8, 40.5, 21.5; IR (liquid film, cm^{-1}): v = 3279, 2934, 1673, 1599, 1454, 1327, 1212, 1160, 910, 814, 760, 664; HRMS(EI-TOF) calc. $C_{24}H_{25}NO_4S$ (M^+): 423.1504. Found: 423.1505.

N-(2-(4-bromophenyl)-4-oxo-4-phenylbutyl)-4-methylbenzenesulfonamide (4c)



The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.85 (d, J = 7.5 Hz, 2 H), 7.64 (d, J = 8.1 Hz, 2 H), 7.56-7.51 (m, 1 H), 7.44-7.32 (m, 4 H), 7.26-7.21 (m, 2 H), 7.01 (d, J = 8.4 Hz, 2 H), 4.86 (t, J = 6.3 Hz, 1 H), 3.51-3.47 (m, 1 H), 3.36-3.21 (m, 3 H), 3.18-3.12 (m, 1 H), 2.39 (s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 197.9, 143.5, 140.3，136.8, 136.6, 133.4, 131.9, 129.8, 129.5, 128.7, 128.1, 127.0, 121.0, 48.0, 41.8, 39.9, 21.5; IR (liquid film, cm^{-1}): v = 3283, 2924, 1684, 1597, 1448, 1411, 1208, 1158, 907, 814, 752, 664; HRMS(EI-TOF) calc. $C_{23}H_{22}BrNO_3S$ (M^+): 471.0504. Found: 471.0507.

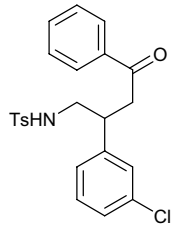
N-(2-(4-bromophenyl)-4-(4-methoxyphenyl)-4-oxobutyl)-4-methylbenzenesulfonamide (4d)



The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.83 (d, J = 9.0 Hz, 2 H), 7.63 (d, J = 8.4 Hz, 2 H), 7.32 (d, J = 8.4 Hz, 2 H), 7.22 (d, J = 8.1 Hz, 2 H), 7.00 (d, J = 8.4 Hz, 2 H), 6.87 (d, J = 9.0 Hz, 2 H), 5.05 (t, J = 6.3 Hz, 1 H), 3.83 (s, 3 H), 3.50-3.45 (m, 1 H),

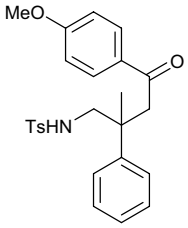
3.35-3.21 (m, 3 H), 3.18-3.11 (m, 1 H), 2.38 (s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 196.5, 163.7, 143.4, 140.5, 136.9, 131.8, 130.4, 129.7, 129.5, 127.0, 120.9, 113.8, 55.5, 48.1, 41.5, 40.1, 21.5; IR (liquid film, cm^{-1}): ν = 3279, 2934, 1673, 1599, 1489, 1420, 1261, 1159, 909, 814, 732, 665; HRMS(EI-TOF) calc. $C_{24}H_{24}BrNO_4S$ (M^+): 501.0609. Found: 501.0611.

N-(2-(3-chlorophenyl)-4-oxo-4-phenylbutyl)-4-methylbenzenesulfonamide (4e)



The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.87 (d, J = 7.2 Hz, 2 H), 7.65 (d, J = 8.4 Hz, 2 H), 7.58-7.53 (m, 1 H), 7.45-7.40 (m, 2 H), 7.26-7.18 (m, 4 H), 7.08-7.03 (m, 2 H), 4.70 (t, J = 6.0 Hz, 1 H), 3.52-3.48 (m, 1 H), 3.37-3.26 (m, 3 H), 3.22-3.14 (m, 1 H), 2.39(s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 197.8, 143.5, 143.4, 136.8, 136.6, 134.7, 133.4, 130.2, 129.8, 128.7, 128.1, 127.8, 127.5, 127.0, 126.0, 48.0, 41.8, 40.1, 21.5; IR (liquid film, cm^{-1}): ν = 3283, 2924, 1684, 1597, 1448, 1327, 1208, 1158, 908, 814, 754, 663; HRMS(EI-TOF) calc. $C_{23}H_{22}ClNO_3S$ (M^+): 427.1009. Found: 427.1005.

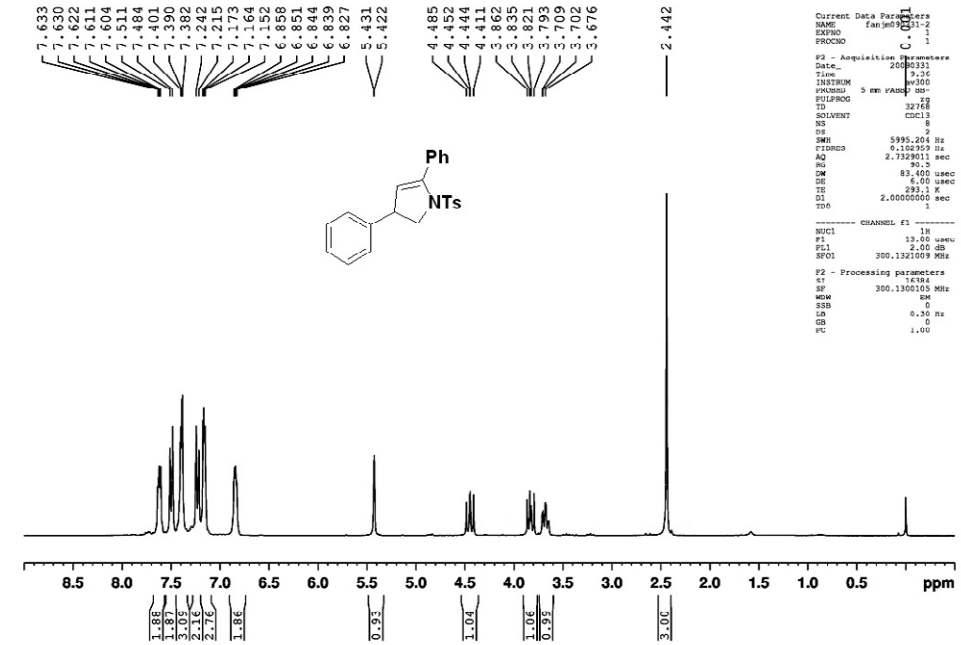
N-(4-(4-methoxyphenyl)-2-methyl-4-oxo-2-phenylbutyl)-4-methylbenzenesulfonamide (4f)

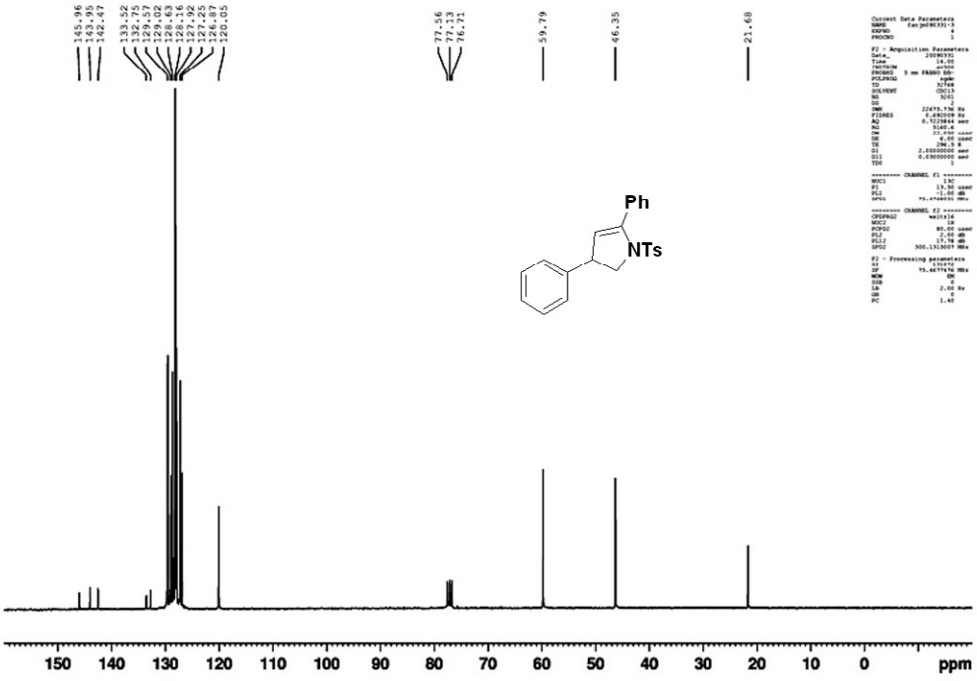


The title compound was a light yellow oil. ^{1}H-NMR ($CDCl_3$, 300 MHz, ppm): δ = 7.78 (d, J = 9.0 Hz, 2 H), 7.68 (d, J = 8.4 Hz, 2 H), 7.26-7.15 (m, 7 H), 6.84 (d, J = 9.0 Hz, 2 H), 4.98 (t, J = 6.9 Hz, 1 H), 3.82 (s, 3 H), 3.44-3.15 (m, 4 H), 2.39 (s, 3 H), 1.50 (s, 3 H); ^{13}C-NMR ($CDCl_3$, 75 MHz, ppm): δ = 197.2, 163.6, 144.4, 143.3, 137.1, 130.7, 130.5, 129.7, 128.7, 127.1, 126.7, 125.9, 113.7, 55.5, 53.2, 46.0, 41.3, 24.1, 21.5; IR (liquid film, cm^{-1}): ν = 3283, 2935, 1680, 1600, 1445, 1330, 1230, 1161, 911, 815, 760, 664; HRMS(EI-TOF) calc. $C_{25}H_{27}NO_4S$ (M^+): 437.1661. Found: 437.1660.

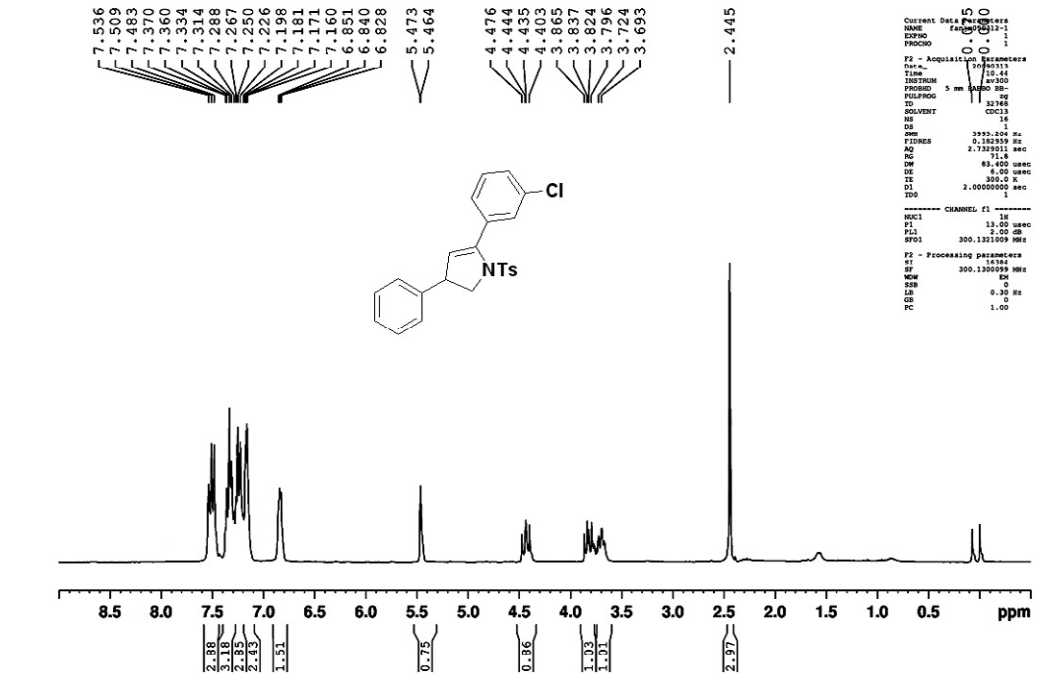
3 NMR Spectra of all products

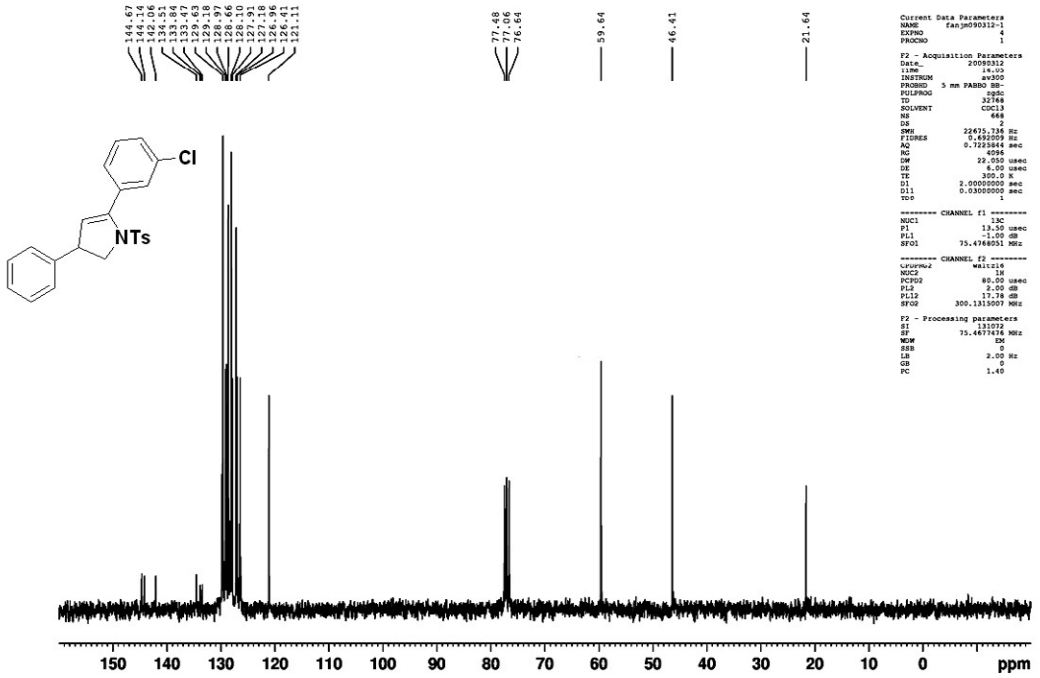
3a



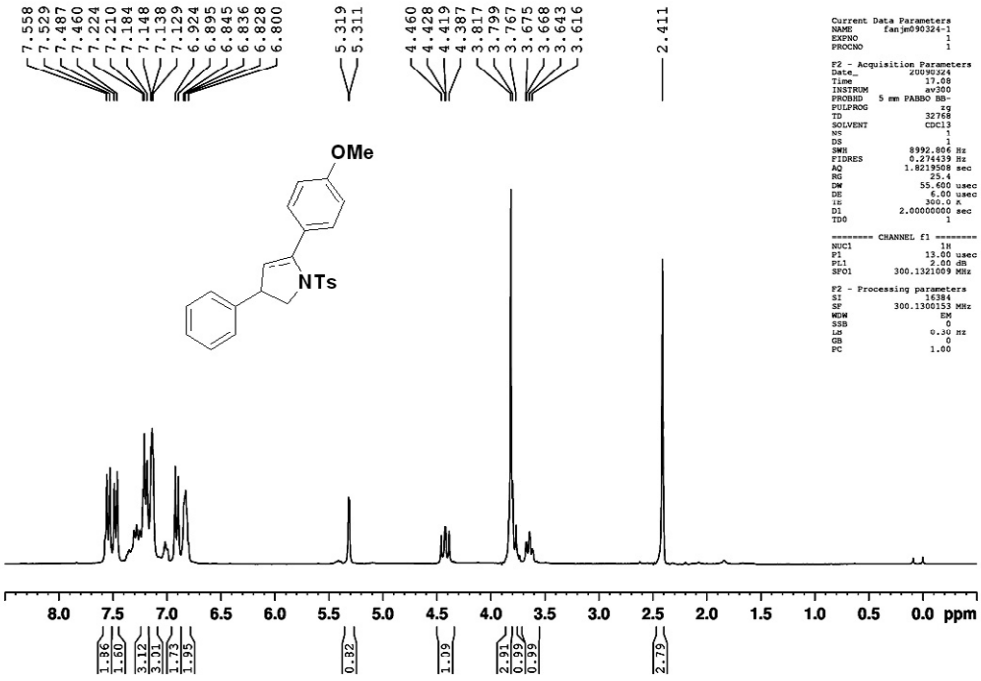


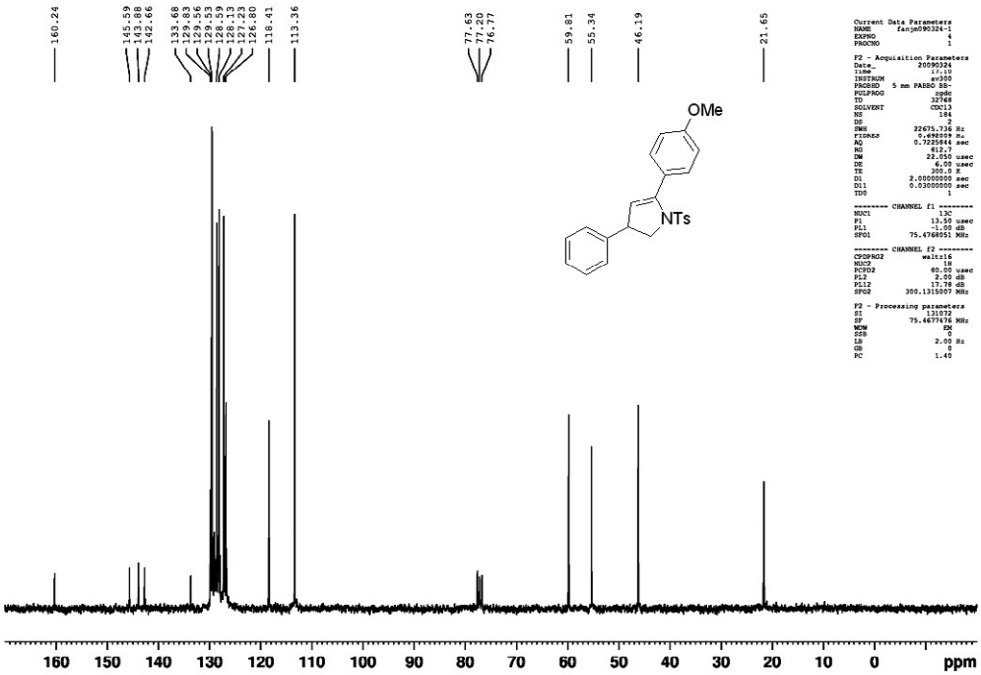
3b


7.536
7.509
7.483
7.370
7.360
7.334
7.314
7.288
7.267
7.250
7.226
7.198
7.181
7.171
7.160
6.851
6.840
6.828
5.473
5.464
4.476
4.444
4.435
4.403
3.865
3.837
3.824
3.796
3.724
3.693
2.445
Cl
NTs
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.88
3.18
2.85
2.43
1.51
0.75
0.86
1.03
1.01
2.97

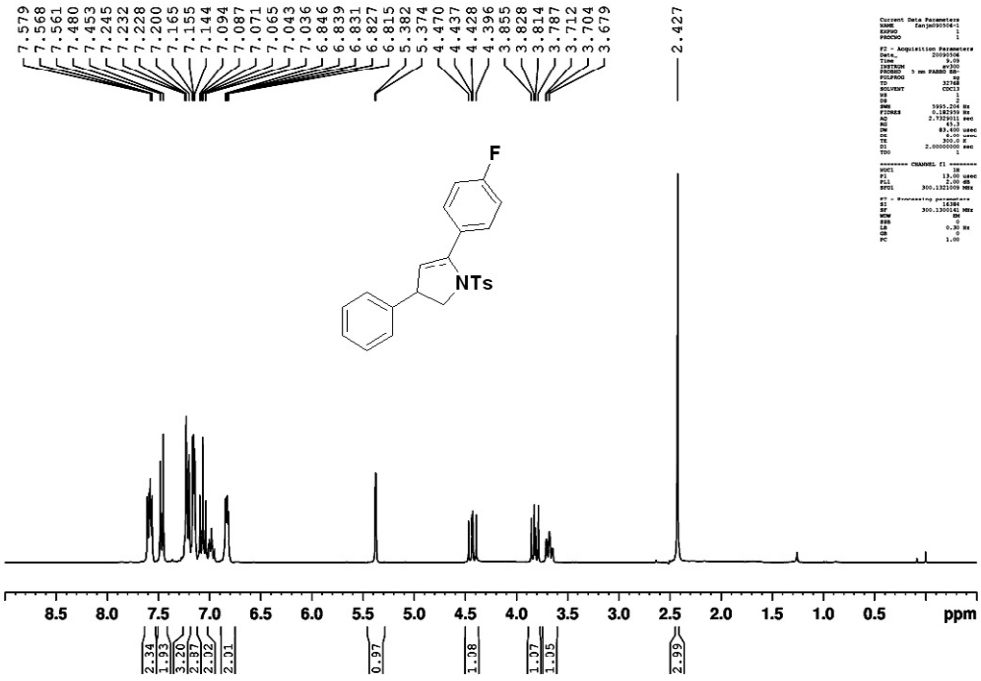

144.67
144.14
142.06
134.51
133.84
133.37
129.63
129.18
128.97
128.66
128.00
127.91
127.18
126.96
126.41
126.11
77.48
77.06
76.64
59.64
46.41
21.64
Cl
NTs
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

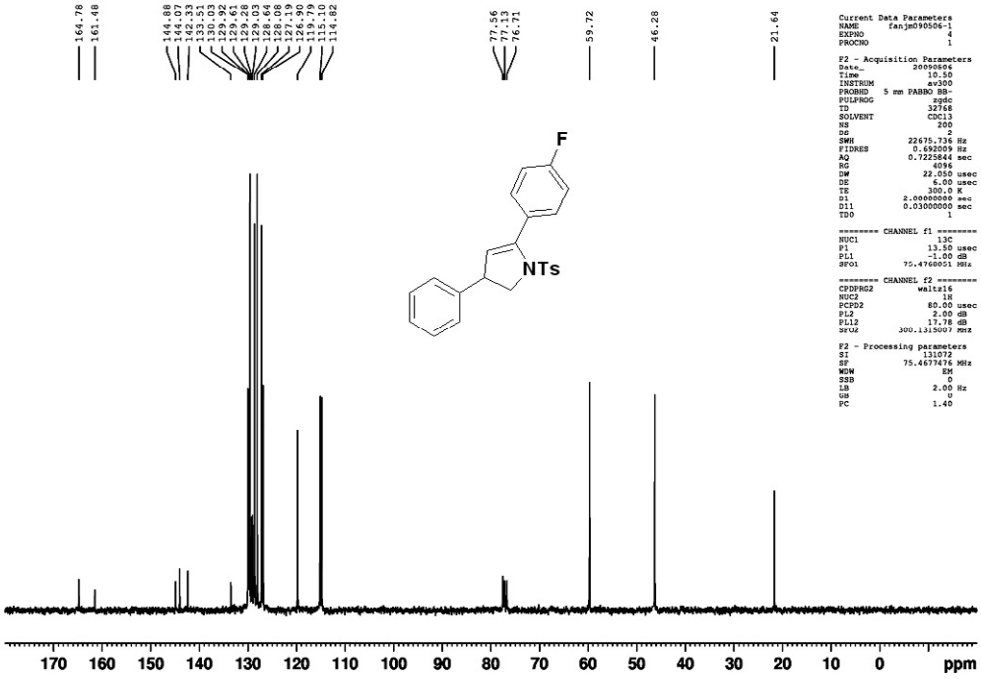
3c


7.558
7.529
7.487
7.460
7.224
7.210
7.184
7.148
7.138
7.129
6.924
6.895
6.845
6.836
6.828
6.800
5.319
5.311
4.460
4.428
4.419
4.387
3.817
3.799
3.767
3.675
3.668
3.643
3.616
2.411
OMe
NTs
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.36
1.60
3.12
3.01
1.73
1.95
0.82
1.09
2.91
0.99
0.99
2.79

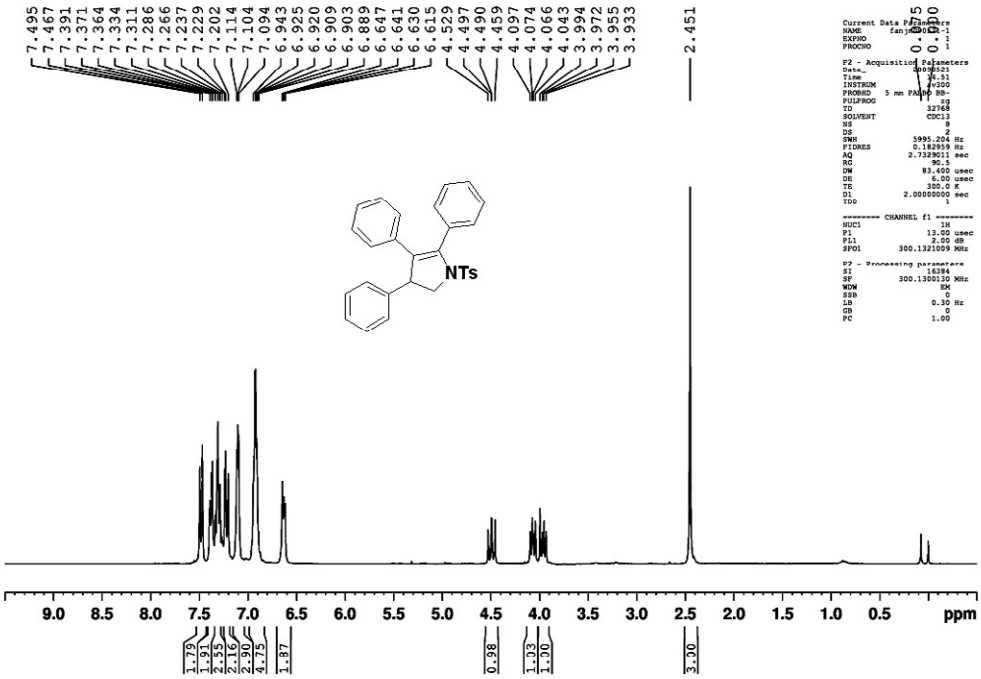

160.24
145.59
143.88
142.66
133.68
129.83
129.56
129.53
128.59
128.13
127.23
126.80
118.41
113.36
77.63
77.20
76.77
59.81
55.34
46.19
21.65
OMe
NTs
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

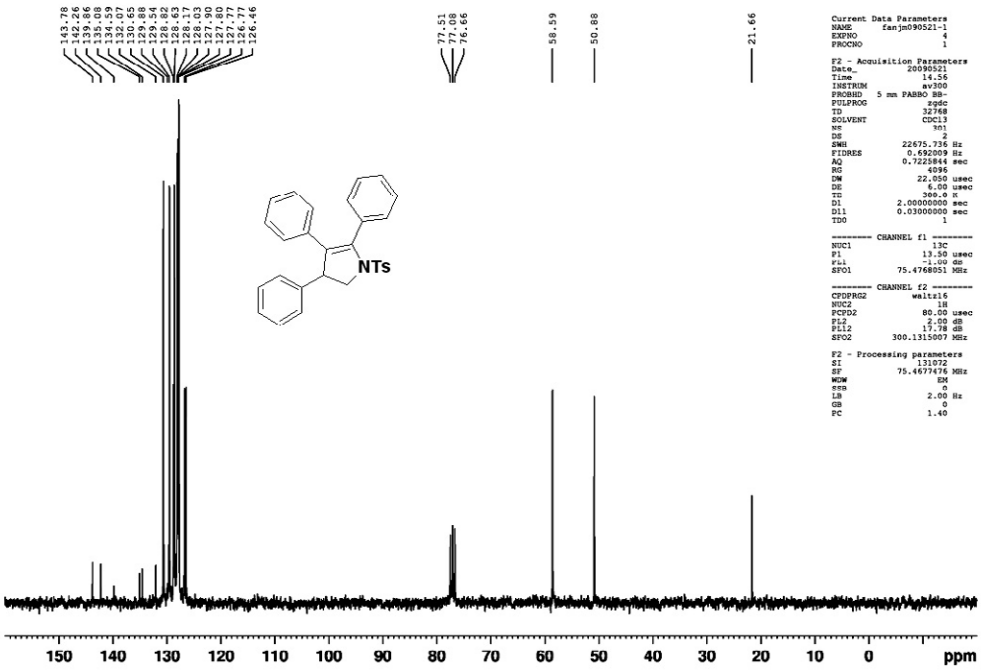
3d


7.579
7.568
7.561
7.480
7.453
7.245
7.232
7.228
7.200
7.165
7.155
7.144
7.094
7.087
7.071
7.065
7.043
7.036
6.846
6.839
6.831
6.827
6.815
5.382
5.374
4.470
4.437
4.428
4.396
3.855
3.828
3.814
3.787
3.712
3.704
3.679
2.427
F
NTs
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.34
1.93
3.20
2.87
2.02
2.01
0.97
1.08
1.07
1.05
2.99

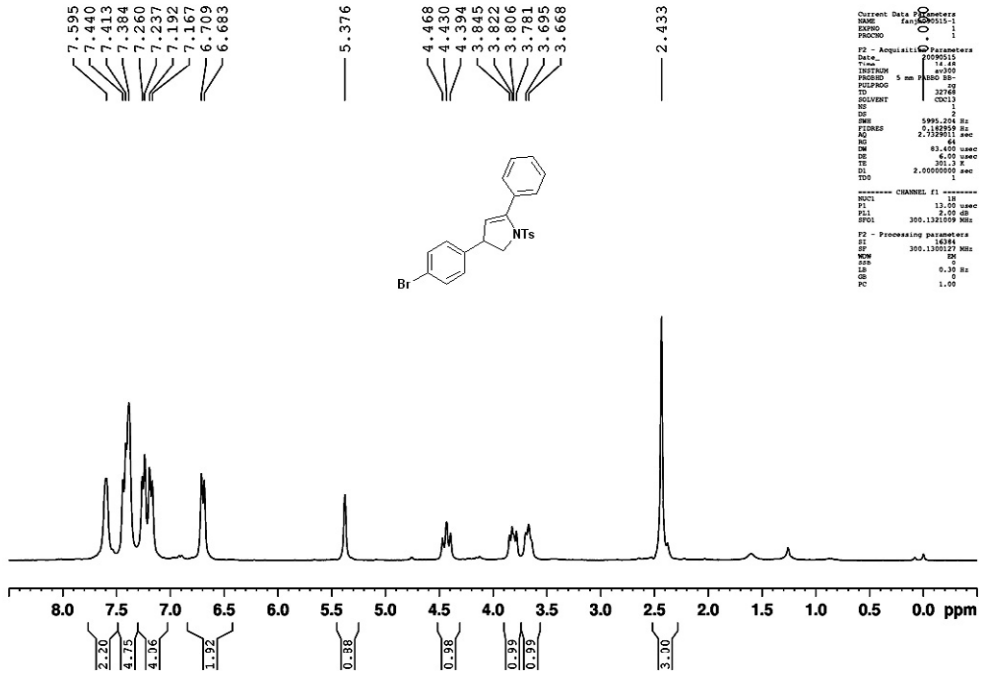

164.78
161.48
144.88
144.07
142.33
133.51
130.03
129.92
129.61
129.28
129.23
128.64
128.08
127.19
126.90
126.79
115.10
114.82
77.56
77.13
76.71
59.72
46.28
21.64
F
NTs
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

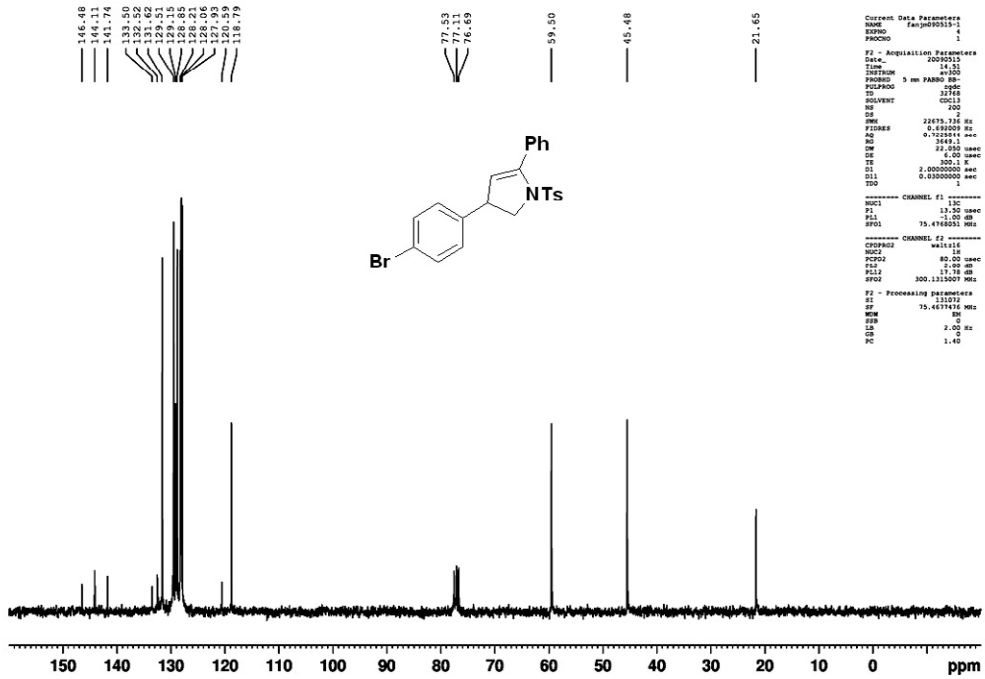
3e


7.495
7.467
7.391
7.371
7.364
7.334
7.311
7.286
7.266
7.237
7.229
7.202
7.114
7.104
7.094
6.943
6.925
6.920
6.909
6.903
6.889
6.647
6.641
6.630
6.615
4.529
4.497
4.490
4.459
4.097
4.074
4.066
4.043
3.994
3.972
3.955
3.933
2.451
NTs
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.79
1.91
2.55
2.16
2.90
4.75
1.87
0.98
1.03
1.00
3.00

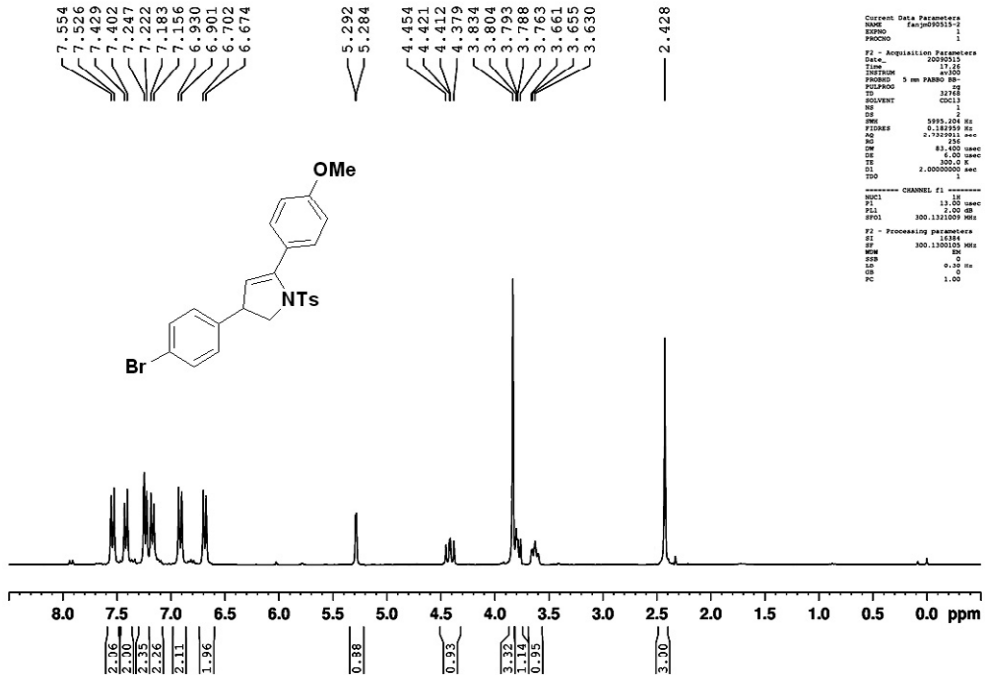

143.78
139.86
134.86
135.08
134.59
132.07
130.65
129.88
129.54
128.82
128.63
128.17
128.03
127.90
127.80
127.77
127.77
126.46
77.51
77.08
76.66
58.59
50.88
21.66
NTs
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

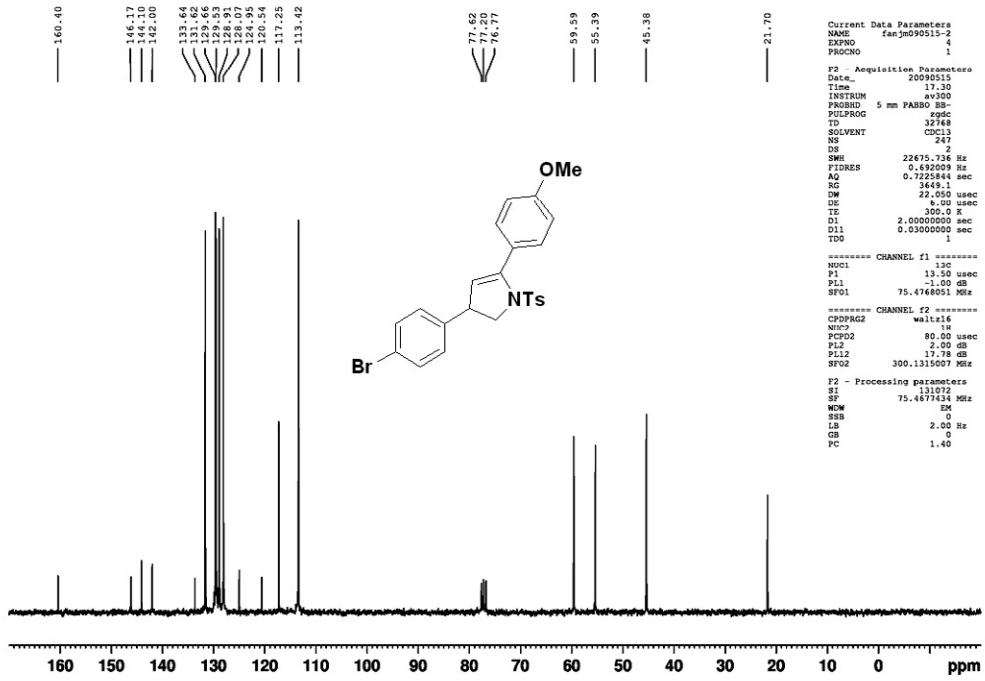
3h


7.595
7.440
7.413
7.384
7.260
7.237
7.192
7.167
6.709
6.683
5.376
4.468
4.430
4.394
3.845
3.822
3.806
3.781
3.695
3.668
2.433
NTs
Br
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.20
4.75
4.36
1.92
0.88
0.98
0.99
0.99
3.00

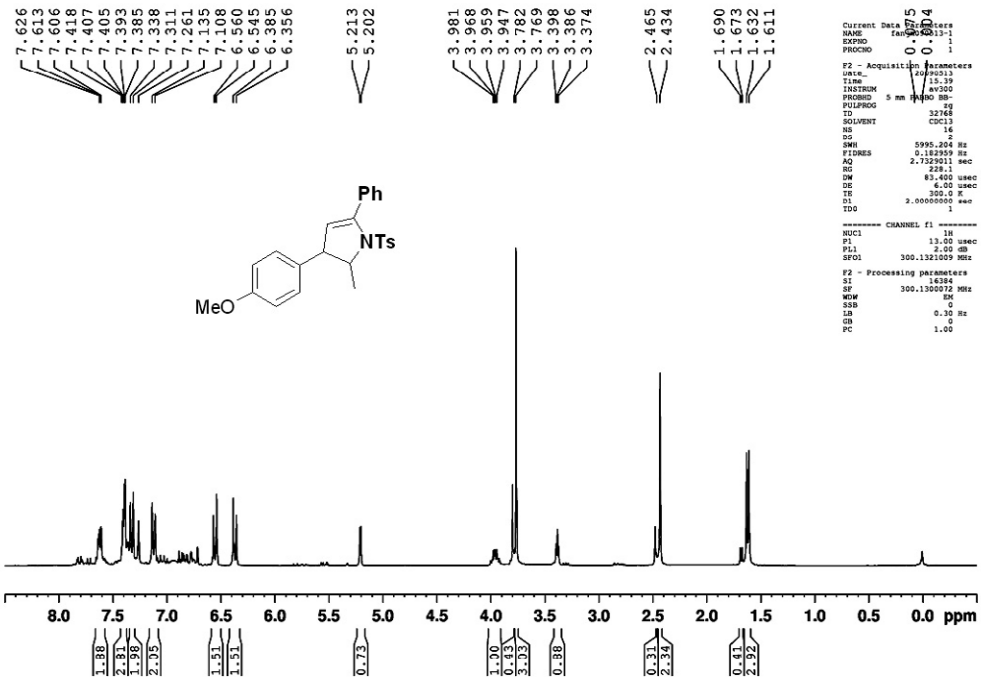

146.48
144.11
141.74
133.50
132.52
131.28
129.51
129.15
128.85
128.21
126.86
127.93
120.59
118.79
77.53
77.11
76.69
59.50
45.48
21.65
Ph
NTs
Br
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

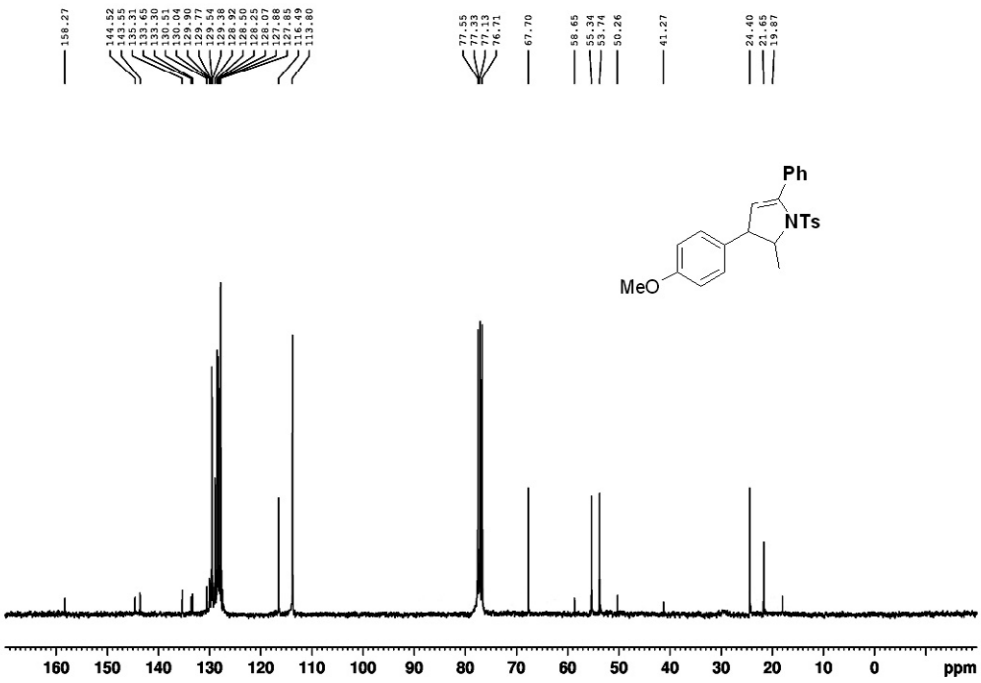
3i


7.554
7.526
7.429
7.402
7.247
7.222
7.183
7.156
6.930
6.901
6.702
6.674
5.292
5.284
4.454
4.421
4.412
4.379
3.834
3.804
3.793
3.788
3.763
3.661
3.655
3.630
2.428
OMe
NTs
Br
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.06
2.00
2.35
2.26
2.11
1.96
0.88
0.93
3.32
1.14
0.95
3.00

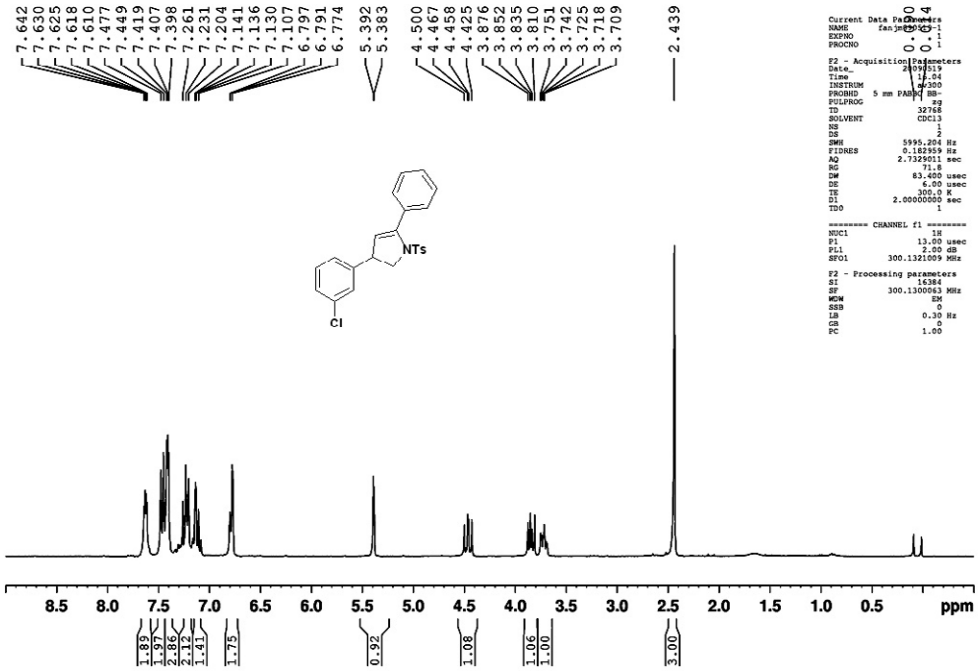

160.40
146.17
144.10
142.00
133.64
131.62
129.66
129.53
128.91
128.07
124.95
120.54
117.25
113.42
77.62
77.20
76.77
59.59
55.39
45.38
21.70
OMe
NTs
Br
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

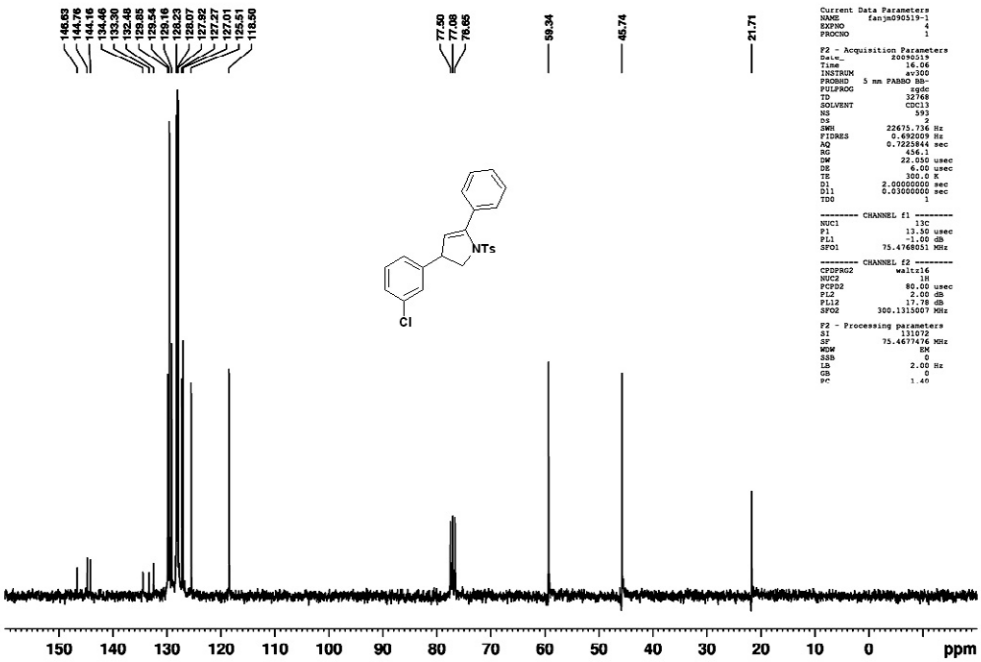
3j


7.626
7.613
7.606
7.418
7.407
7.405
7.393
7.385
7.338
7.311
7.261
7.135
7.108
6.560
6.545
6.385
6.356
5.213
5.202
3.981
3.968
3.959
3.947
3.782
3.769
3.398
3.386
3.374
2.465
2.434
1.690
1.673
1.632
1.611
Ph
NTs
MeO
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.88
2.81
1.98
2.05
1.51
1.51
0.73
1.00
1.43
3.03
0.38
0.31
2.34
0.41
2.92

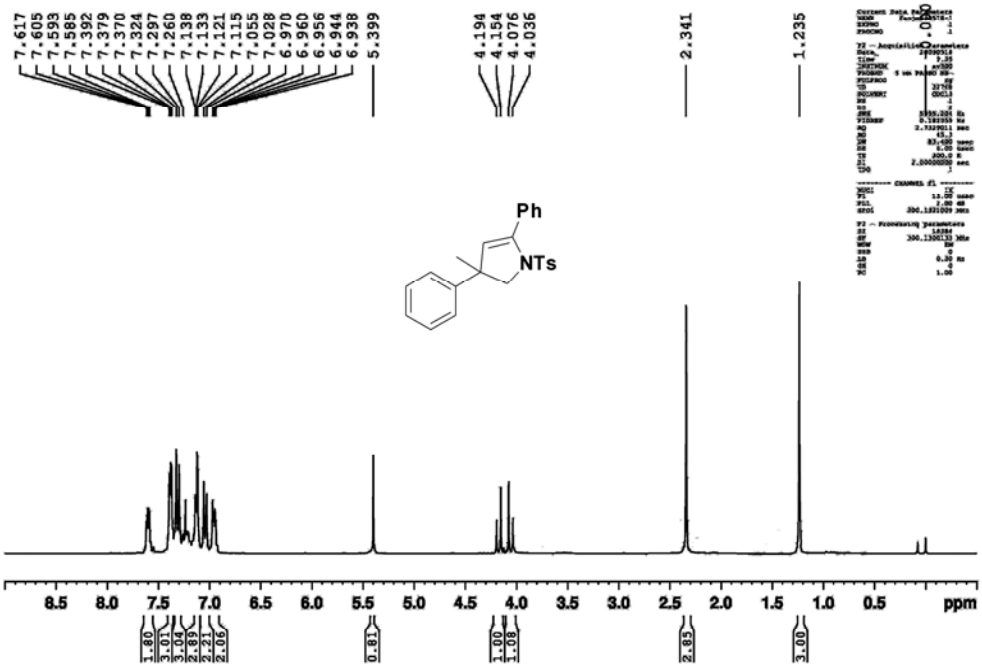

158.27
144.52
143.55
135.31
134.46
133.30
130.51
130.04
129.90
129.77
129.54
129.38
128.92
128.50
128.25
128.07
127.88
127.69
127.49
113.80
77.55
77.33
77.13
76.71
67.70
58.65
55.34
53.74
50.26
41.27
24.40
21.65
19.87
Ph
NTs
MeO
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

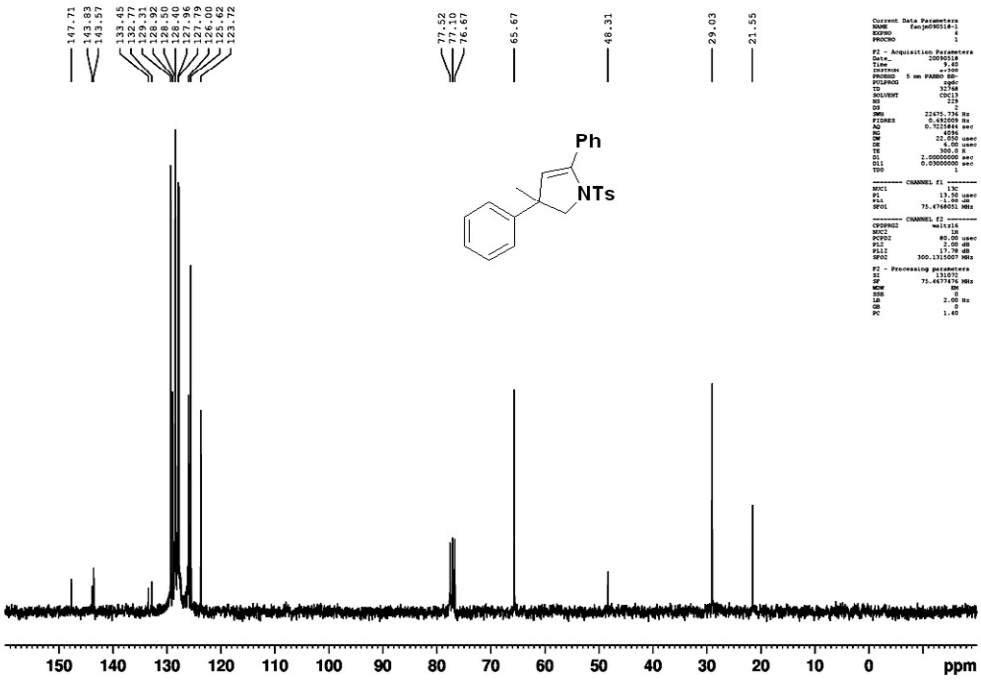
3k


7.642
7.630
7.625
7.618
7.610
7.477
7.449
7.419
7.407
7.398
7.261
7.231
7.204
7.141
7.136
7.130
7.107
6.797
6.791
6.774
5.392
5.383
4.500
4.467
4.458
4.425
3.876
3.852
3.835
3.810
3.751
3.742
3.725
3.718
3.709
2.439
NTs
Cl
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.89
1.97
2.86
2.12
1.41
1.75
0.92
1.08
1.06
1.00
3.00

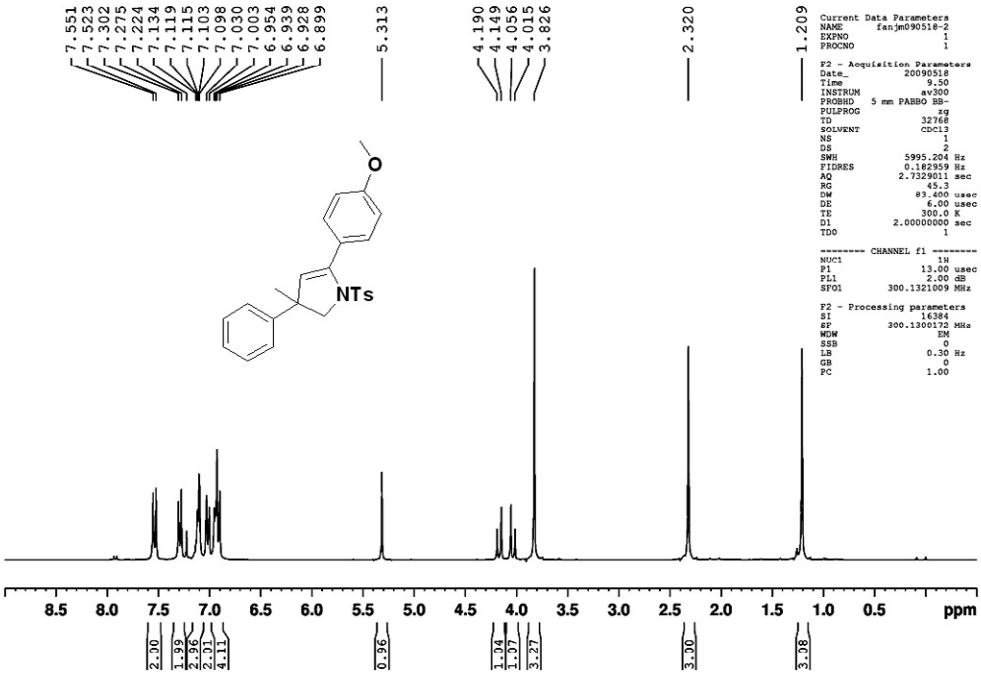

146.63
144.76
144.16
134.46
133.30
132.48
129.85
129.54
129.16
128.23
128.07
127.92
127.77
127.01
125.51
118.50
77.50
77.08
76.65
59.34
45.74
21.71
NTs
Cl
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

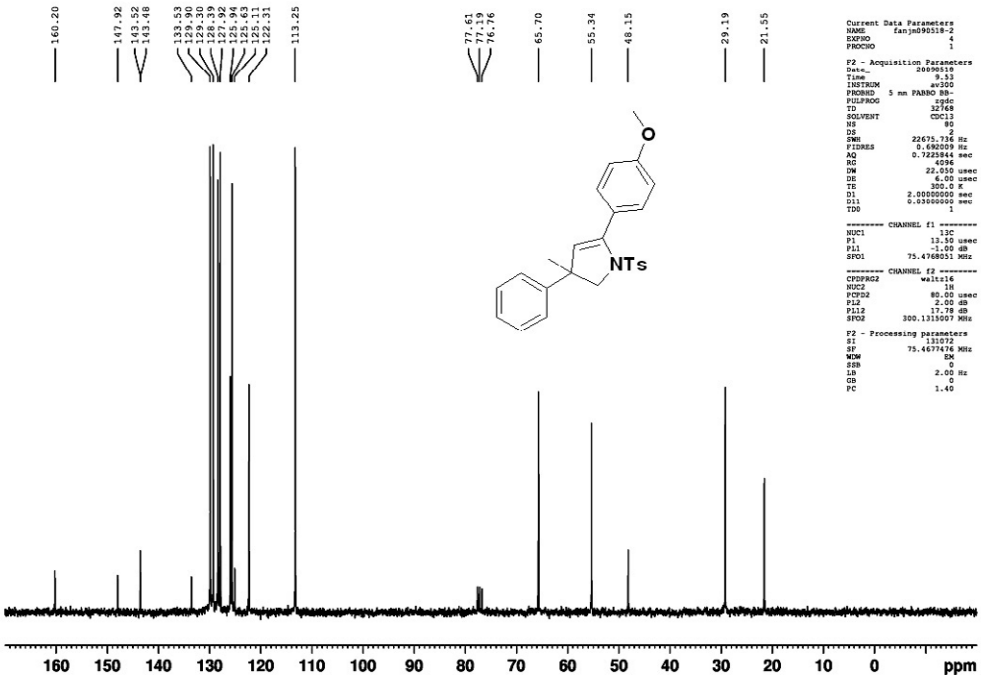
3l


7.617
7.605
7.593
7.585
7.392
7.379
7.370
7.324
7.297
7.260
7.138
7.133
7.121
7.115
7.055
7.028
6.970
6.960
6.956
6.944
6.938
5.399
4.194
4.154
4.076
4.036
2.341
1.235
Ph
NTs
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.80
3.01
3.04
2.89
2.21
2.06
0.81
1.00
1.08
2.85
3.00

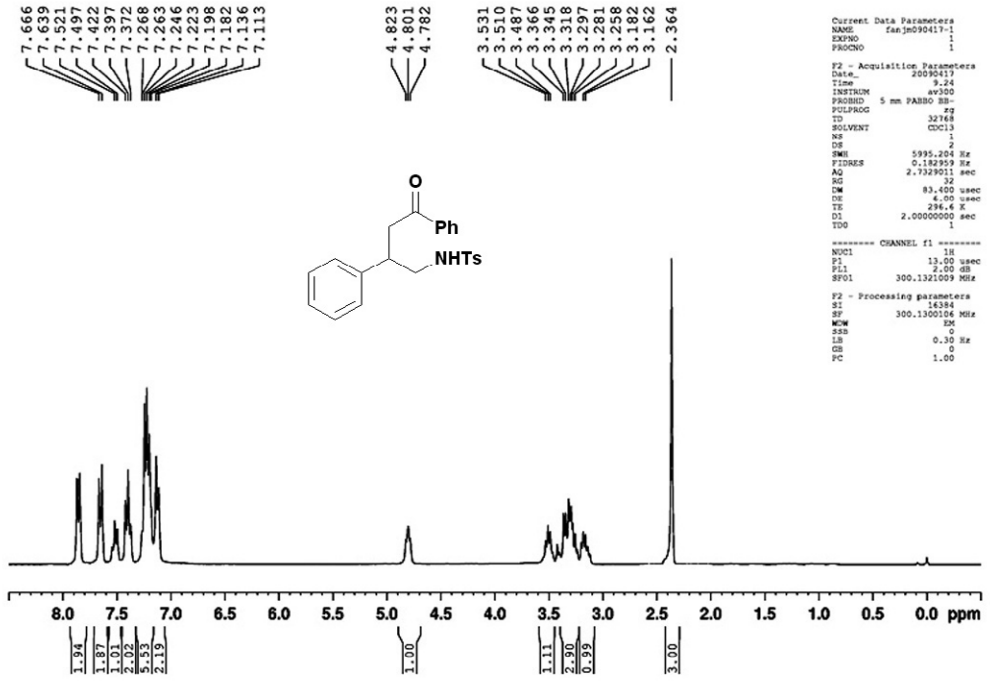

147.71
143.83
143.57
133.45
129.77
129.31
128.92
128.50
128.40
128.06
127.79
126.00
125.62
123.72
77.52
77.10
76.67
65.67
48.31
29.03
21.55
Ph
NTs
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

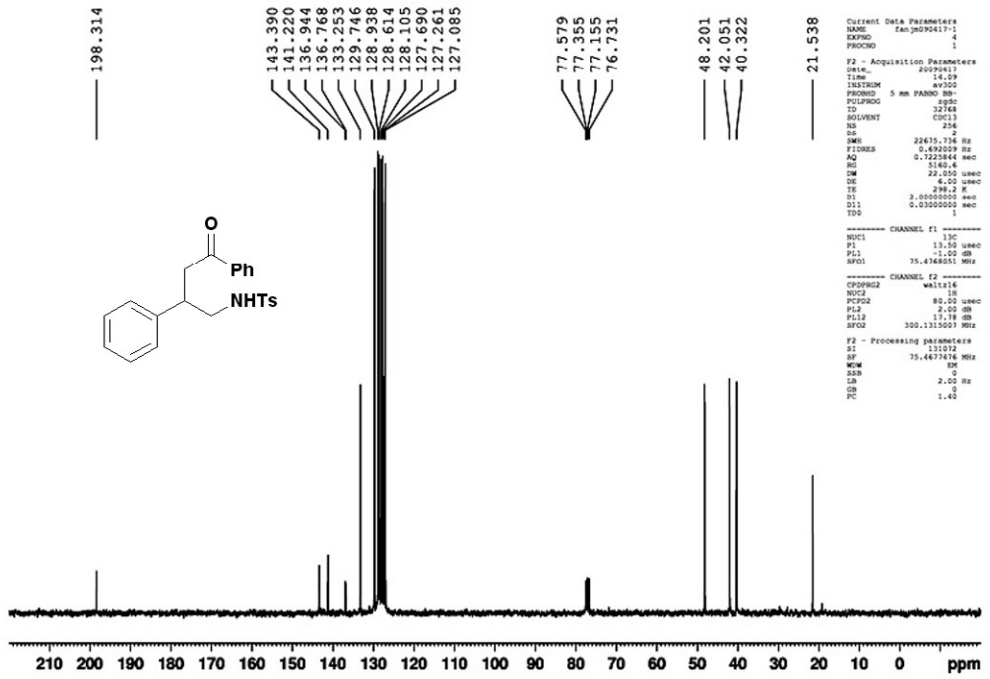
3m


7.551
7.523
7.302
7.275
7.224
7.134
7.119
7.115
7.103
7.098
7.030
7.003
6.954
6.939
6.928
6.899
5.313
4.190
4.149
4.056
4.015
3.826
2.320
1.209
O
NTs
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.00
1.99
2.96
2.01
4.11
0.96
1.94
1.07
3.27
3.00
3.08

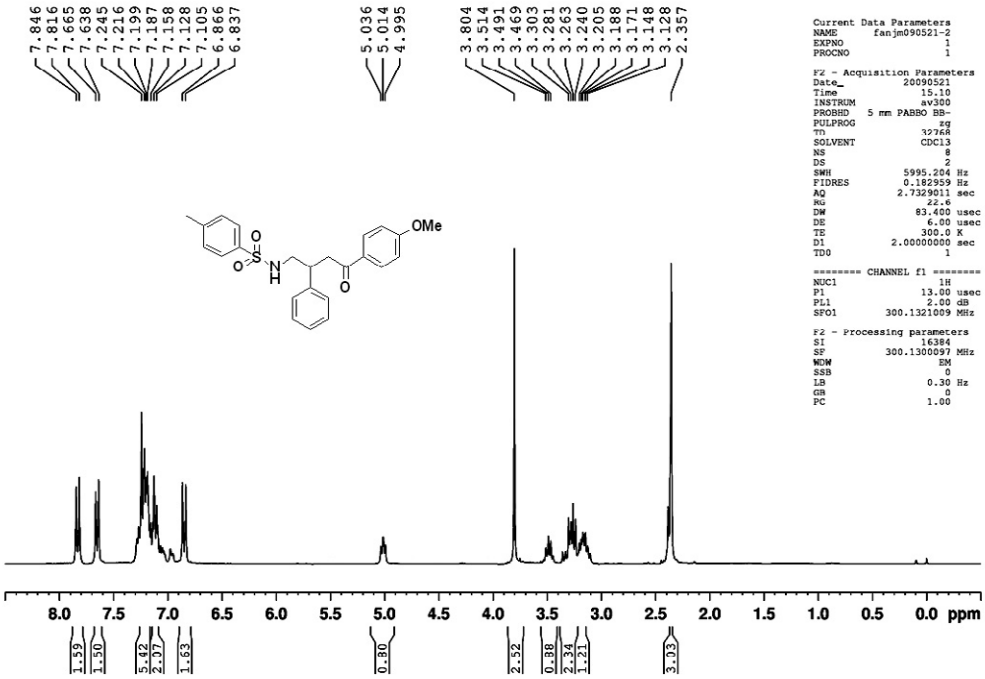

160.20
147.92
143.52
143.48
133.53
129.90
129.30
128.39
127.92
125.94
125.63
125.11
122.31
113.25
77.61
77.19
76.76
65.70
55.34
48.15
29.19
21.55
O
NTs
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

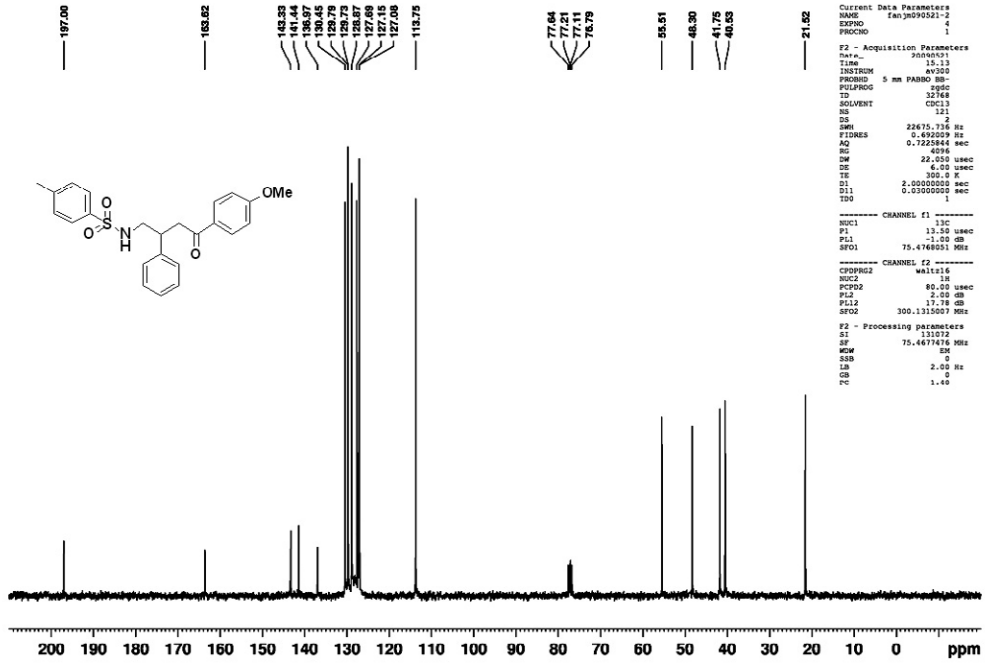
4a


7.666
7.639
7.521
7.497
7.422
7.397
7.372
7.268
7.263
7.246
7.223
7.198
7.182
7.136
7.113
4.823
4.801
4.782
3.531
3.510
3.487
3.366
3.345
3.318
3.297
3.281
3.258
3.182
3.162
2.364
O
Ph
NHTs
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.94
1.87
1.01
2.02
5.53
2.19
1.00
1.11
2.90
0.99
3.00

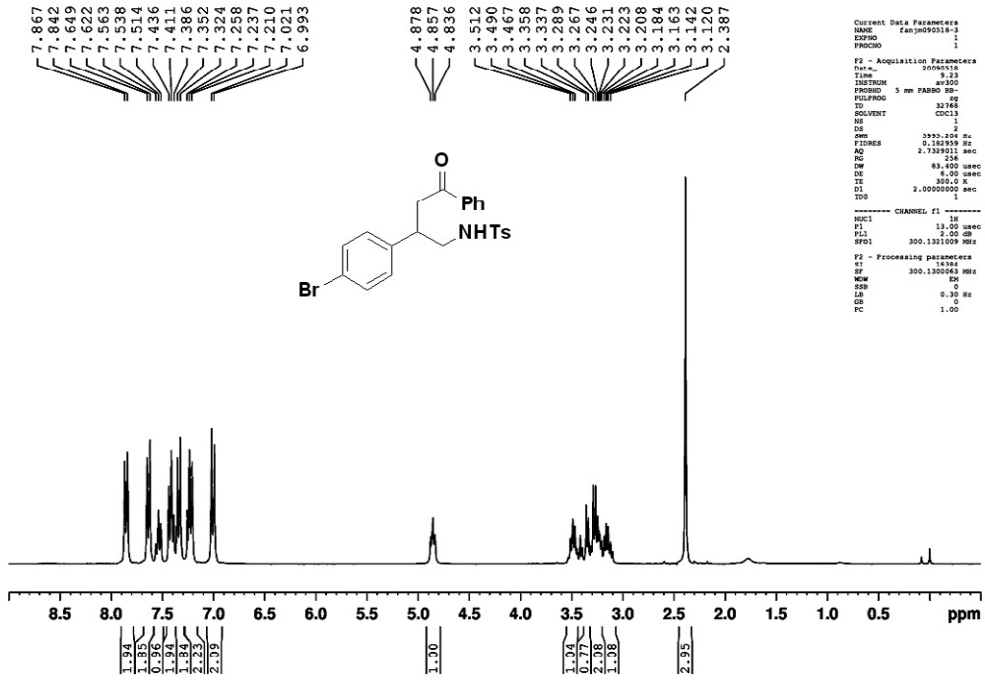

198.314
143.390
141.220
136.944
136.768
133.253
129.746
128.938
128.614
128.105
127.690
127.261
127.085
77.579
77.355
77.155
76.731
48.201
42.051
40.322
21.538
O
Ph
NHTs
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

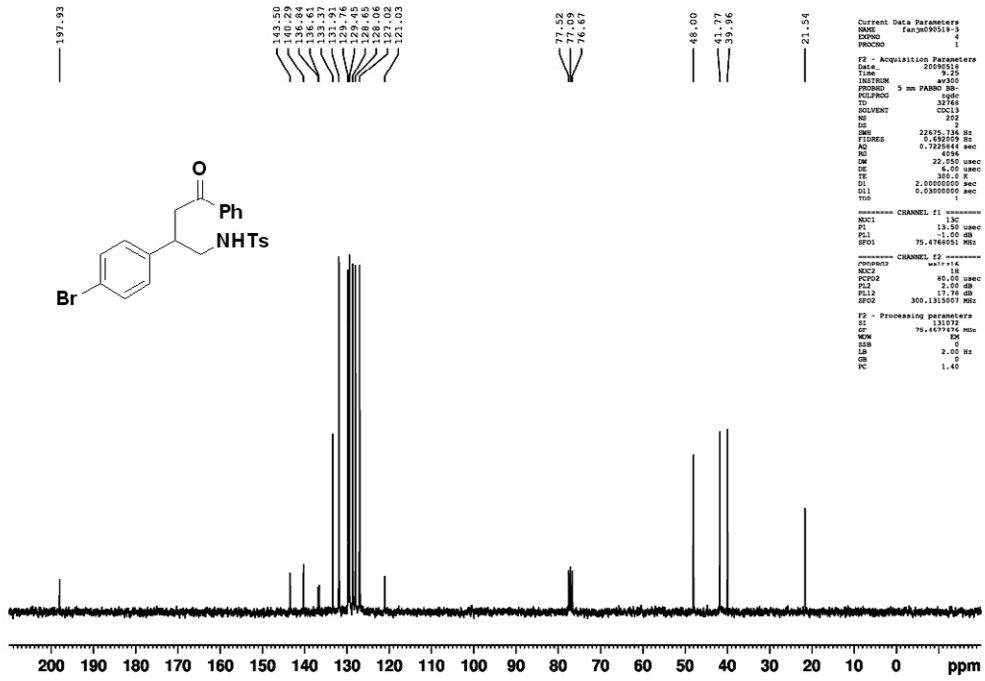
4b


7.846
7.816
7.665
7.638
7.245
7.216
7.199
7.187
7.158
7.128
7.105
6.866
6.837
5.036
5.014
4.995
3.804
3.514
3.491
3.469
3.303
3.281
3.263
3.240
3.205
3.188
3.171
3.148
3.128
2.357
OMe
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.59
1.50
5.42
2.07
1.63
0.80
2.52
0.88
2.34
1.21
3.03

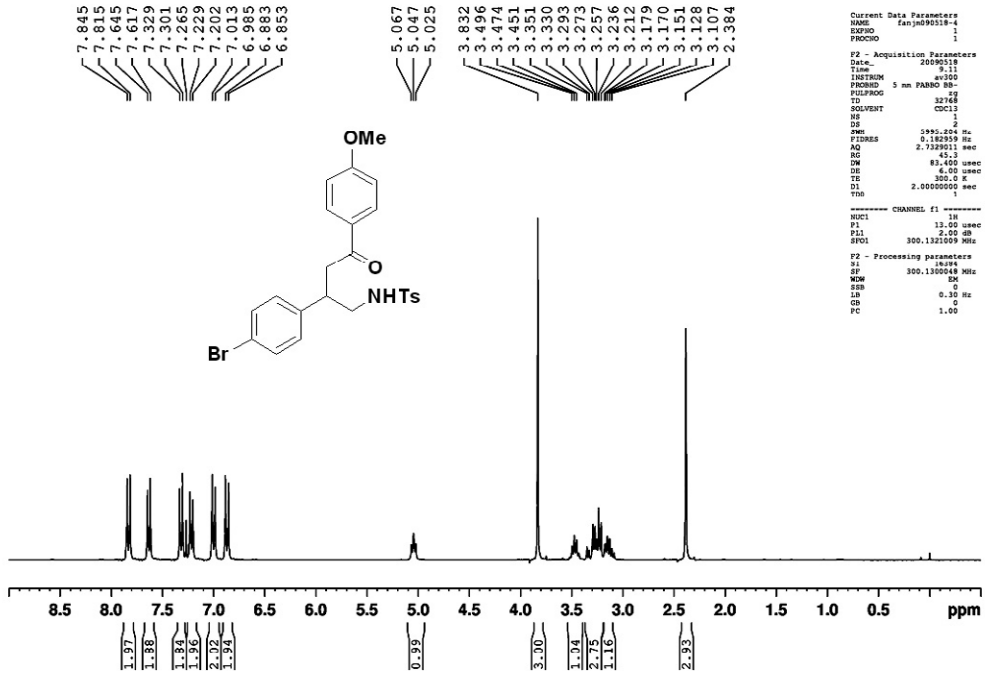

197.00
163.62
143.33
141.44
136.97
130.45
129.79
129.73
128.67
127.69
127.15
127.08
113.75
77.64
77.21
77.11
76.79
55.51
48.30
41.75
40.83
21.62
OMe
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

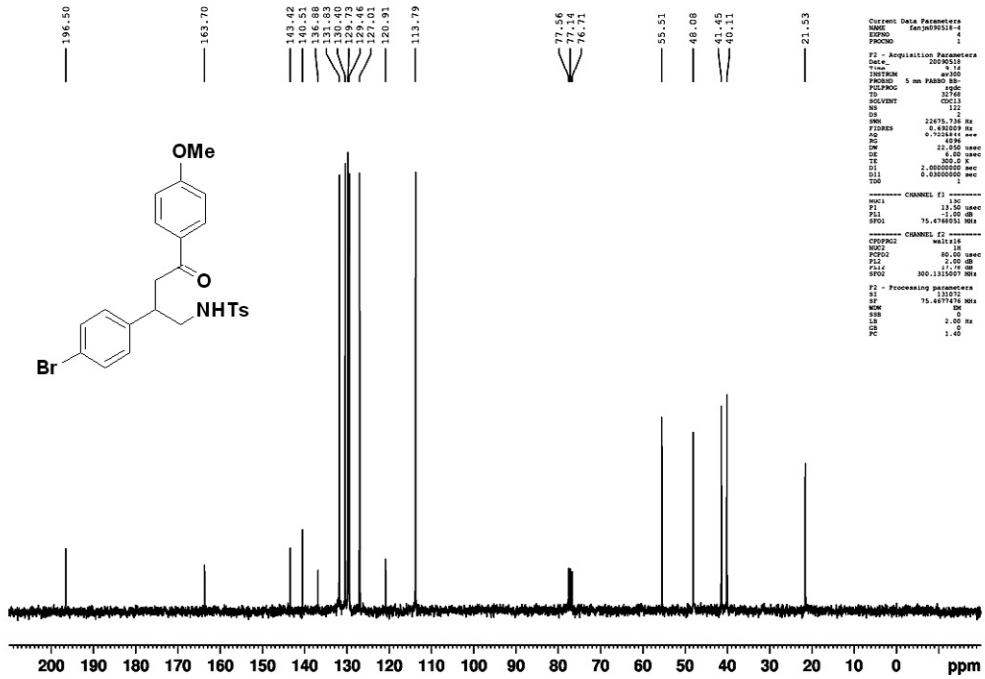
4c


7.867
7.842
7.649
7.622
7.563
7.538
7.514
7.436
7.411
7.386
7.352
7.324
7.258
7.237
7.210
7.021
6.993
4.878
4.857
4.836
3.512
3.490
3.467
3.358
3.337
3.289
3.267
3.246
3.231
3.223
3.208
3.184
3.163
3.142
3.120
2.387
O
Ph
NHTs
Br
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.94
1.85
0.96
1.94
1.84
2.23
2.09
1.00
1.04
0.77
2.08
1.08
2.95

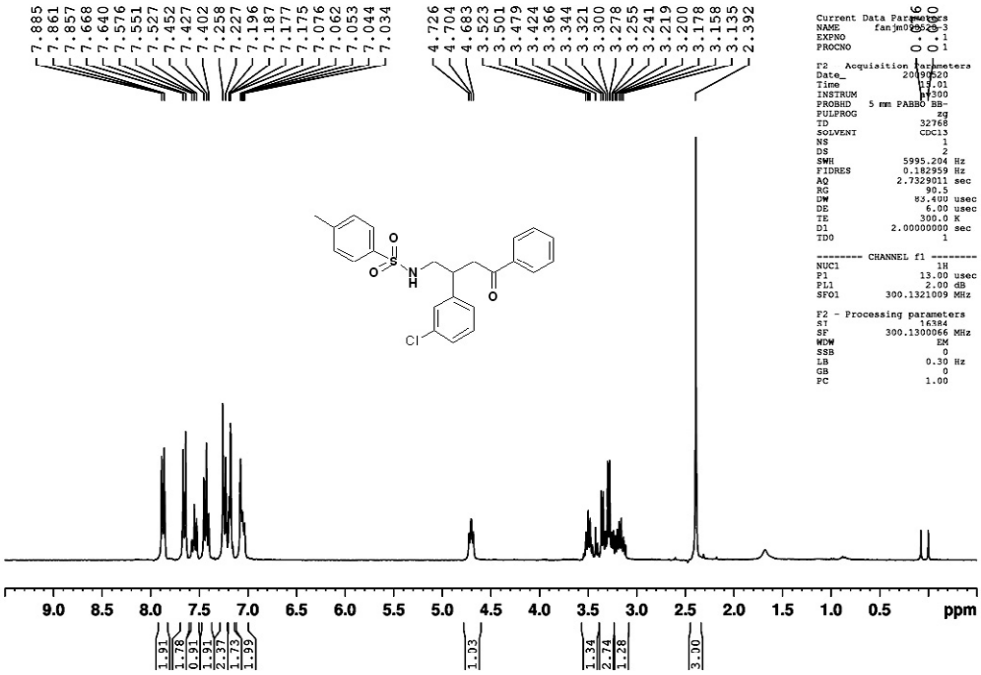

197.93
143.50
140.39
136.84
136.61
133.37
131.91
131.76
129.45
128.65
128.06
127.02
121.03
77.52
77.09
76.67
48.00
41.77
39.96
21.54
O
Ph
NHTs
Br
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

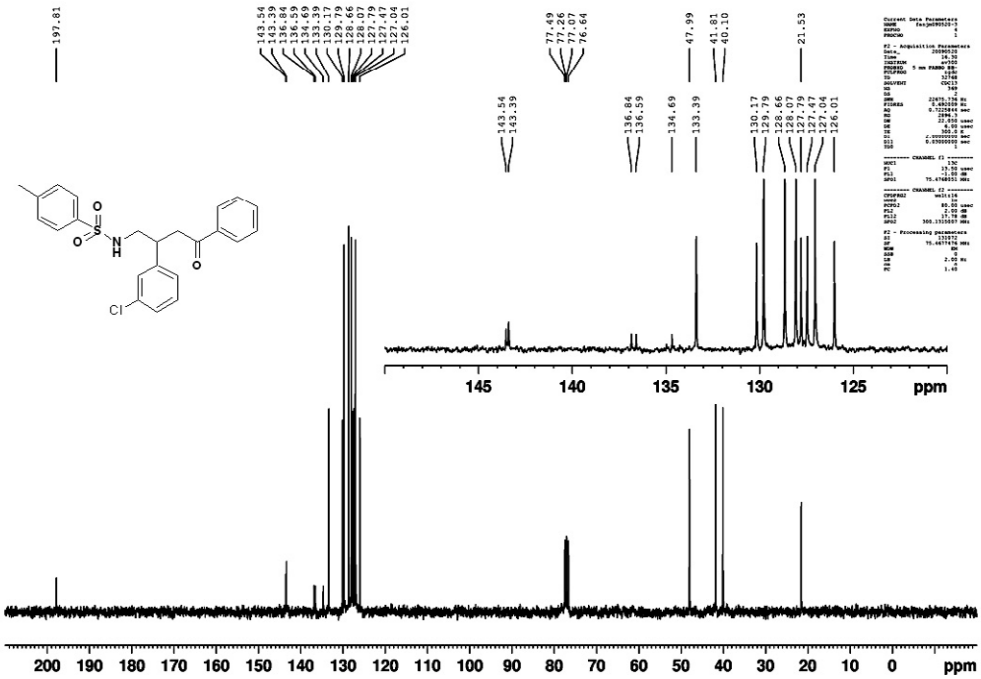
4d


7.845
7.815
7.645
7.617
7.329
7.301
7.265
7.229
7.202
7.013
6.985
6.883
6.853
5.067
5.047
5.025
3.832
3.496
3.474
3.451
3.351
3.330
3.293
3.273
3.257
3.236
3.212
3.179
3.170
3.151
3.128
3.107
2.384
OMe
O
NHTs
Br
1.97
1.88
1.84
1.96
2.02
1.94
0.99
3.00
1.04
2.75
1.16
2.93
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

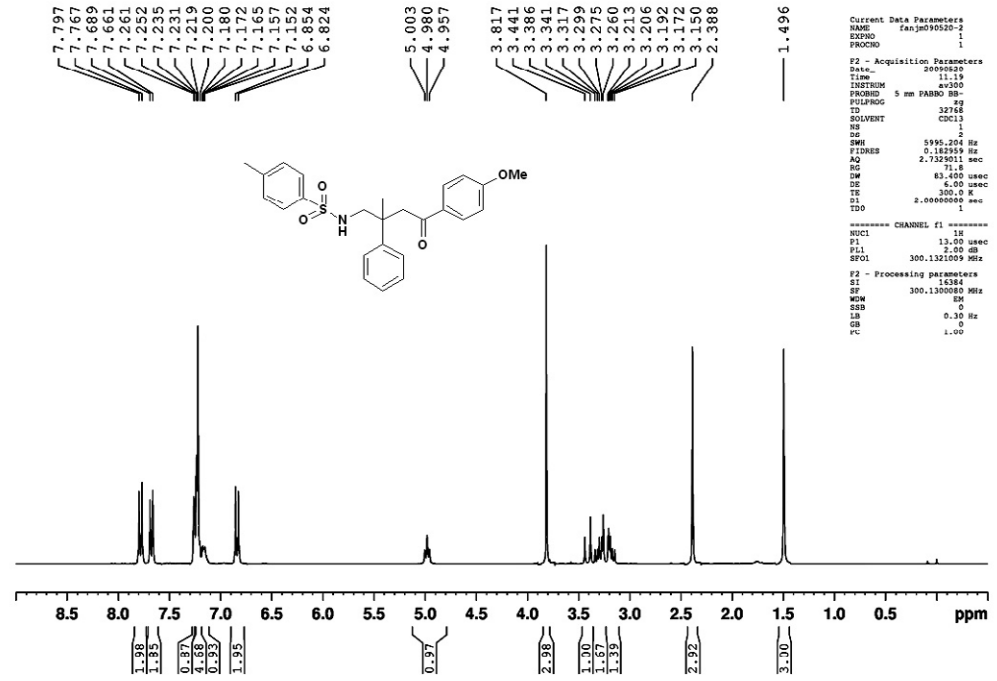

196.50
163.70
143.42
140.51
136.88
131.83
130.40
129.73
129.46
127.01
120.91
113.79
77.56
77.14
76.71
55.51
48.08
41.45
40.11
21.53
OMe
O
NHTs
Br
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

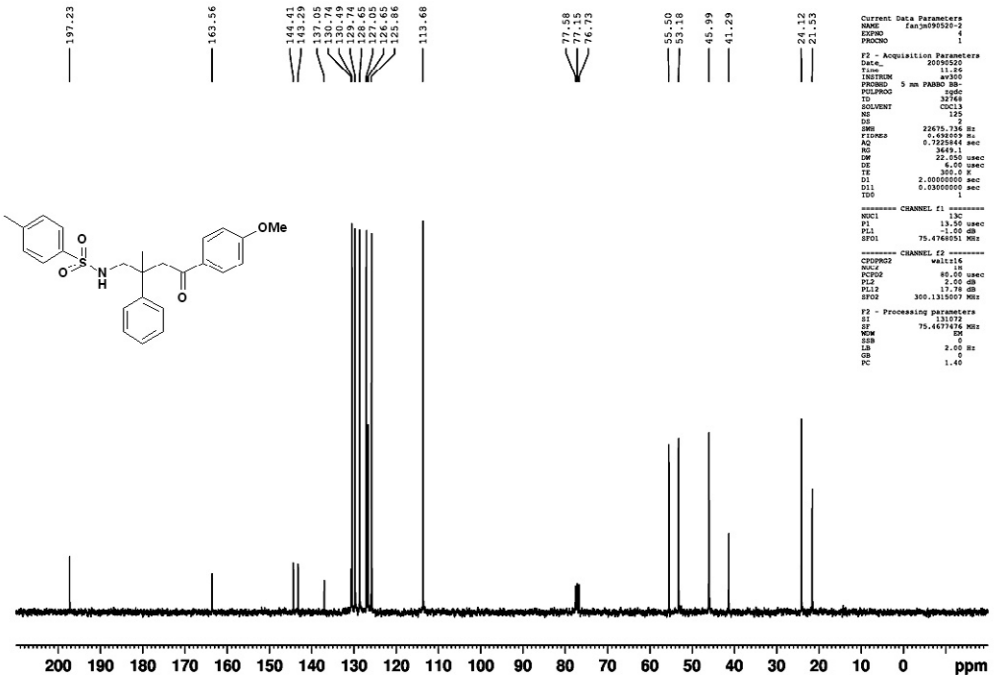
4e


7.885
7.861
7.857
7.668
7.640
7.576
7.551
7.527
7.452
7.427
7.402
7.258
7.227
7.196
7.187
7.177
7.175
7.076
7.062
7.053
7.044
7.034
4.726
4.704
4.683
3.523
3.501
3.479
3.424
3.366
3.344
3.321
3.300
3.278
3.255
3.241
3.219
3.200
3.178
3.158
3.135
2.392
Cl
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.91
1.78
0.91
1.91
2.37
1.73
1.99
1.03
1.34
2.74
1.28
3.00


197.81
143.54
143.39
136.84
136.59
134.69
133.39
130.17
129.79
128.66
128.07
127.79
127.47
127.04
126.01
77.49
77.26
77.07
76.64
47.99
41.81
40.10
21.53
Cl
145 140 135 130 125 ppm
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

4f


7.797
7.767
7.689
7.661
7.261
7.252
7.235
7.231
7.219
7.200
7.180
7.172
7.165
7.157
7.152
6.854
6.824
5.003
4.980
4.957
3.817
3.441
3.386
3.341
3.317
3.299
3.275
3.260
3.213
3.206
3.192
3.172
3.150
2.388
1.496
OMe
O
S
N
H
O
O
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.98
1.85
0.87
4.68
0.93
1.95
0.97
2.98
1.00
1.67
1.39
2.92
3.00


197.23
163.56
144.41
143.29
137.05
130.74
130.49
128.96
128.65
127.05
126.65
125.86
113.68
77.58
77.15
76.73
55.50
53.18
45.99
41.29
24.12
21.53
OMe
O
S
N
H
O
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm