

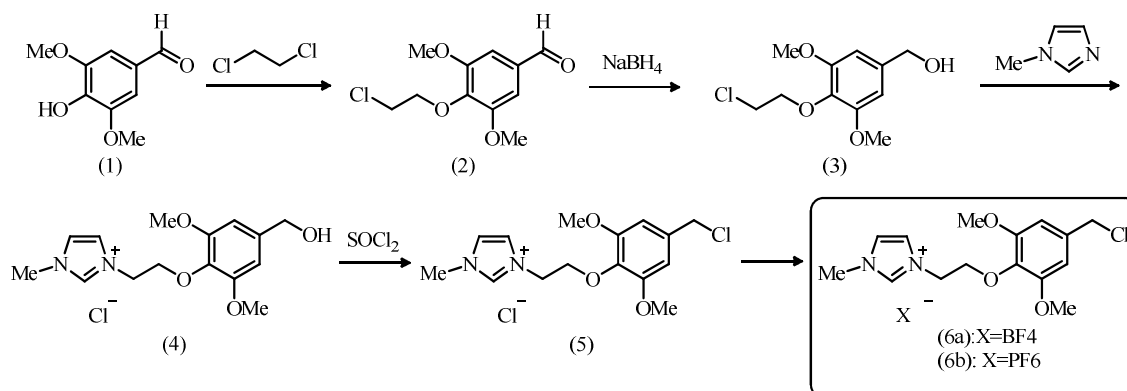
Supplementary Experimental Information

Ionic Liquid Phase Synthesis of 1,5-Disubstited 1,2,3-Triazole

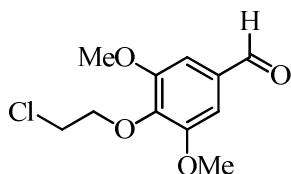
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259-1292 Japan

All reagents and chemicals received from commercial suppliers were of reagent grade and were used unmodified. NMR spectra were performed on Bruker Advance DRX 500 (^1H : 500 MHz, ^{13}C : 125 MHz) spectrometers. Deuterated solvents used are indicated in each case. Chemical shifts (δ) are expressed in ppm and refer to the residual peak of the solvent or TMS as an internal standard; coupling constants (J) are in Hz. The mass analyses were performed using a JEOL AccuTOF LC-plus JMS-T100LP spectrometer. The courses of the reactions were monitored using TLC aluminum sheets with silica gel 60 F₂₅₄ (Merck). The column chromatography was performed using silica gel 60 (Kanto Chemical).



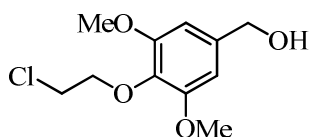
4-(2-chloroethoxy)-3,5-dimethoxybenzaldehyde (2)



The soln of syringaldehyde (3.00g, 16.5mmol) in DMF (90mL) was added to Potassium carbonate (11.41g, 82.5mmol) and Dichloroethane (32.68g, 330.2mmol). The resulting mixture was stirred at 65°C for 20h and then solvent was removed under vacuum and mixture extracted with ethyl acetate, and organic layer was washed with H₂O, dried (MgSO₄) and evaporated.

Yield: 3.92g, (16.0mmol 97%); white solid; mp 69-71°C. Rf = 0.75 (n-hexane/ethyl acetate 1:1)
¹H NMR (500 MHz, CDCl₃): δ= 3.79 (t, J=6.5 Hz, 2 H), 3.94 (s, 6 H), 4.32 (t, J=6.5 Hz, 2 H), 7.13 (s, 2 H), 9.88 (s, 1 H).
¹³C NMR (125 MHz, CDCl₃): δ=42.3, 56.3 (2 c), 72.8, 106.6 (2 c), 132.2, 141.9, 153.7 (2 c), 191.0.
HRMS (APCI): *m/z* [M+H]⁺ calcd for C₁₁H₁₃ClO₄: 245.0581; found: 245.0625.

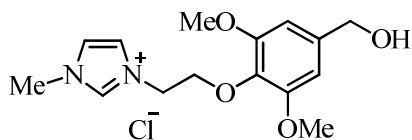
(4-(2-chloroethoxy)-3,5-dimethoxyphenyl)methanol (3)



The soln of 4-(2-Chloroethoxy)-3,5-dimethoxybenzaldehyde (2) (3.87g, 15.6mmol) in MeOH (38mL) was added to sodium borohydride (0.71g, 18.8mmol) at 0°C. The resulting mixture was stirred at 0°C for 30min and then mixture was quenched with 5%-HCl and solvent was removed under vacuum. The mixture extracted with ethyl acetate, and organic layer was washed with H₂O, dried (MgSO₄) and evaporated. The product was isolated by silica gel column chromatography (ethyl acetate - hexane).

Yield: 3.62g, (14.7mmol 94%); colorless oil. Rf = 0.50 (n-hexane/ethyl acetate 1:1)
¹H NMR (500 MHz, CDCl₃): δ=1.74 (t, J=6.0 Hz, 1 H), 3.77 (t, J=6.8 Hz, 2 H), 3.86 (s, 6 H), 4.20 (t, J=6.8 Hz, 2 H), 4.64 (d, J=6.0 Hz, 2 H), 6.60 (s, 2 H).
¹³C NMR (125 MHz, CDCl₃): δ=42.2, 56.1 (2 c), 65.2, 72.8, 103.7 (2 c), 135.5, 137.3, 153.2 (2 c).
HRMS (APCI): *m/z* [M+H]⁺ calcd for C₁₁H₁₅ClO₄: 247.0737; found:247.0783.

3-(2-(4-(hydroxymethyl)-2,6-dimethoxyphenoxy)ethyl)-1-methyl-imidazolium chloride (4)



The soln of (4-(2-chloroethoxy)-3,5-dimethoxyphenyl)methanol(3) (3.55g, 14.4mmol) and 1-Methylimidazole (1.42g, 17.3mmol) in toluene (10mL) was stirred at 120°C for 15h. and then solvent was removed under vacuum. The product was isolated by silica gel column chromatography (CHCl₃ - MeOH).

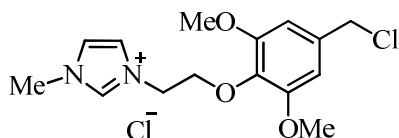
Yield: 4.79g, (14.6mmol quant.); yellow oil. Rf = 0.35 (chloroform/ methanol 95:5)

¹H NMR (500 MHz, DMSO): δ=3.69 (s, 6 H), 3.93 (s, 3 H), 4.16 (t, *J*=4.8 Hz, 2 H), 4.42 (d, *J*=5.5 Hz, 2 H), 4.45 (t, *J*=4.8 Hz, 2 H), 5.23 (t, *J*=5.5 Hz, 1 H), 6.62 (s, 2 H), 7.75 (m, 1 H), 7.83 (m, 1 H), 9.20 (s, 1 H).

¹³C NMR (125 MHz, DMSO): δ=36.2, 49.0, 49.8, 56.2 (2 c), 63.3, 71.0, 103.6 (2 c), 123.3, 123.6, 134.4, 137.7, 139.6, 152.9

HRMS (APCI): *m/z* [M-Cl]⁺ calcd for C₁₅H₂₁ClN₂O₄: 293.1501; found: 293.1519.

3-(2-(4-(chloromethyl)-2,6-dimethoxyphenoxy)ethyl)-1-methyl-imidazolium chloride (5)



The soln of 3-(2-(4-(hydroxymethyl)-2,6-dimethoxyphenoxy)ethyl)-1-methyl-imidazolium chloride (4) (4.50g, 13.6mmol) in CHCl₃ (10mL) was added to thionyl chloride (1.79g, 15.1mmol) at 0°C. The resulting mixture was stirred at r.t. for 2h and then solvent was removed under vacuum. The product was isolated by silica gel column chromatography (CHCl₃ - MeOH).

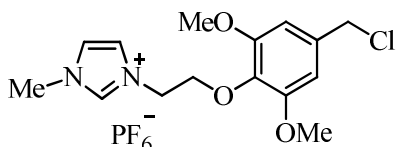
Yield: 4.7g, (13.5mmol 99%); colorless oil. Rf = 0.35 (chloroform/ methanol 95:5)

¹H NMR (500 MHz, DMSO): δ=3.72 (s, 6 H), 3.93 (s, 3 H), 4.19 (t, *J*=4.8 Hz, 2 H), 4.47 (t, *J*=4.8 Hz, 2 H), 4.69 (s, 2 H), 6.77 (s, 2 H), 7.76 (t, *J*=1.7 Hz, 1 H), 7.84 (t, *J*=1.7 Hz, 1 H), 9.22 (s, 1 H).

¹³C NMR (125 MHz, DMSO): δ=35.8, 46.6, 49.3, 56.0 (2 c), 70.7, 79.8, 106.1 (2 c), 122.8, 123.2, 133.8, 135.4, 137.3, 152.6.

HRMS (APCI): *m/z* [M-Cl]⁺ calcd for C₁₅H₂₀Cl₂N₂O₃: 311.1163; found: 311.1156

3-(2-(4-(chloromethyl)-2,6-dimethoxyphenoxy)ethyl)-1-methyl-imidazolium hexafluorophosphate (6b)

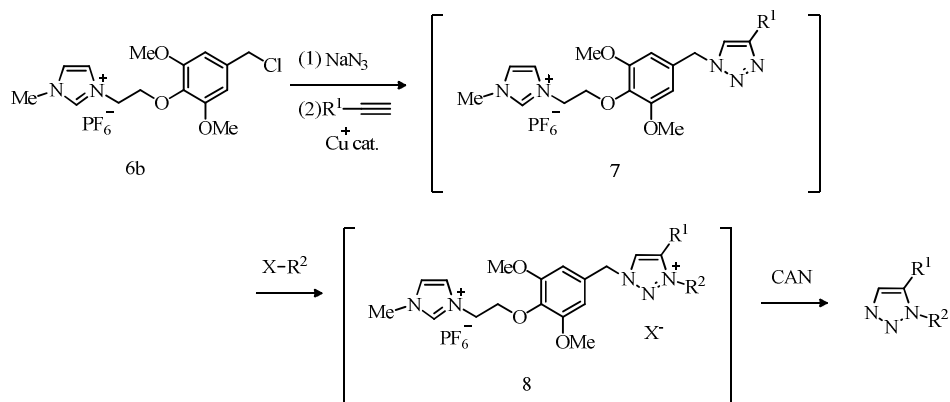


The soln of 3-(2-(4-(chloromethyl)-2,6-dimethoxyphenoxy)ethyl)-1-methyl-imidazolium chloride (5) (4.70g, 13.5mmol) and Potassium hexafluorophosphate (2.77g, 15.1mmol) in CHCl₃ (10mL) was

stirred at r.t. for 17h and then mixture was filtered out and solvent removed under vacuum. The product was isolated by silica gel column chromatography (CHCl₃ - MeOH).

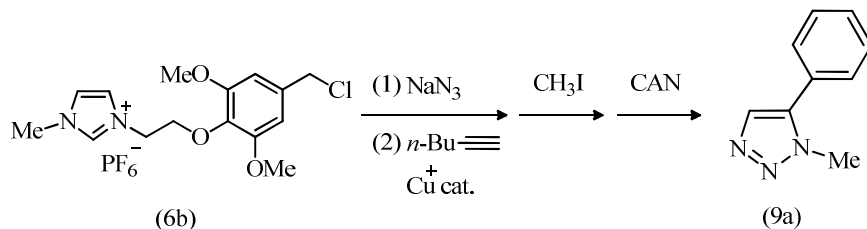
Yield: 3.83g, (8.3mmol 61%); white solid; mp 115-117°C. R_f = 0.55 (chloroform/ methanol 85:15)
¹H NMR (500 MHz, DMSO): δ=3.72 (s, 6 H), 3.92 (s, 3 H), 4.20 (t, *J*=4.8 Hz, 2 H), 4.46 (t, *J*=4.8 Hz, 2 H), 4.69 (s, 2 H), 6.77 (s, 2 H), 7.73 (t, *J*=1.8 Hz, 1 H), 7.82 (t, *J*=1.8 Hz, 1 H), 9.16 (s, 1 H).
¹³C NMR (125 MHz, DMSO): δ=35.7, 46.5, 49.3(2c), 55.9, 70.5, 106.1, 122.8(2 c), 123.2(2 c), 133.8, 135.4, 137.2, 152.6
 HRMS (APCI): *m/z* [M-PF₆]⁺ calcd for C₁₅H₂₀ClF₆N₂O₃P: 311.1163; found: 311.1176.

General Procedure for ionic liquid phase synthesis of 1,5- disubstitution -1*H*-1,2,3-triazole



The soln of ionic liquid bound dimethoxy benzyl chloride 3-(2-(4-(chloromethyl)-2,6-dimethoxyphenoxy)ethyl)-1-methyl-imidazolium hexafluorophosphate (**6b**) (1.0 equiv.) in acetone (3mL) was added to sodium azide (1.1 equiv.) in water (1mL). The mixture was stirred at r.t. for 16h and resulting mixture was added to terminal alkyne (1.5 equiv.), copper (II) sulfate (0.5 equiv.) and ascorbic acid (0.5 equiv.) in water (3mL) at r.t.. The mixture was stirred at 60°C for 24h and then acetone was removed under vacuum. After purification by washings with diethyl ether, the ionic liquid bound triazole **7** was treated with alkyl halide (10 equiv.) at 80°C for 24 h in pressure vessel and then the reaction mixture was washings with diethyl ether. Ionic liquid bound triazolium salt **8** was added to Ceric ammonium nitrate (CAN) (2.0 equiv.) and DMF (5mL). The mixture was stirred at 120°C for 24h and then solvent was removed under vacuum and extracted with CHCl₃. The product was isolated by silica gel column chromatography (ethyl acetate - hexane).

1-Methyl-5-phenyl-1*H*-1,2,3-triazole (9a)



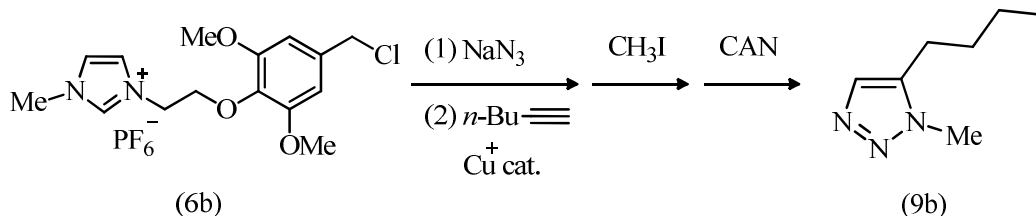
Yield: 154mg, (0.97mmol 72%); yellow oil. Rf = 0.34 (n-hexane/ethyl acetate 1:1)

$^1\text{H NMR}$ (500 MHz, CDCl_3): δ =4.09 (s, 3 H), 7.41-7.44 (m, 2 H), 7.47-7.53 (m, 3 H), 7.73 (s, 1 H).

$^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ =34.9, 126.1, 127.8(2 c), 128.5(2 c), 128.8, 132.0, 137.4.

HRMS (APCI): m/z $[\text{M} + \text{H}]^+$ calcd for $\text{C}_9\text{H}_{10}\text{N}_3$: 160.0875; found: 160.0859.

5-Butyl-1-methyl-1H-1,2,3-triazole (9b)



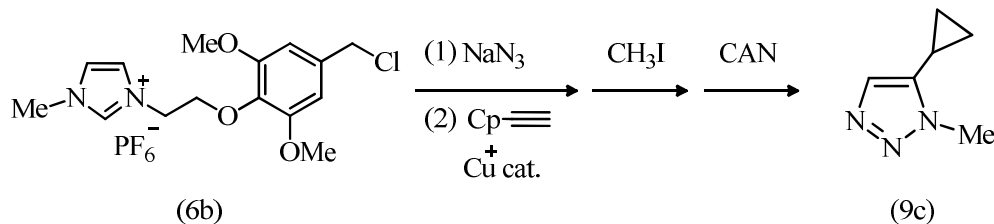
Yield: 127mg, (0.91mmol 78%); colorless oil. Rf = 0.62 (n-hexane/ethyl acetate 1:1)

$^1\text{H NMR}$ (500 MHz, CDCl_3): δ =0.96 (m, 3 H), 1.38-1.46 (m, 2 H), 1.61-1.68 (m, 2 H), 2.62 (m, 2 H), 3.96 (s, 3 H), 7.45 (s, 1 H).

$^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ =13.3, 21.8, 22.3, 29.6, 33.8, 131.5, 137.1.

HRMS (APCI): m/z $[\text{M} + \text{H}]^+$ calcd for $\text{C}_7\text{H}_{14}\text{N}_3$: 140.1188; found: 140.1198.

5-Cyclopropyl-1-methyl-1H-1,2,3-triazole (9c)



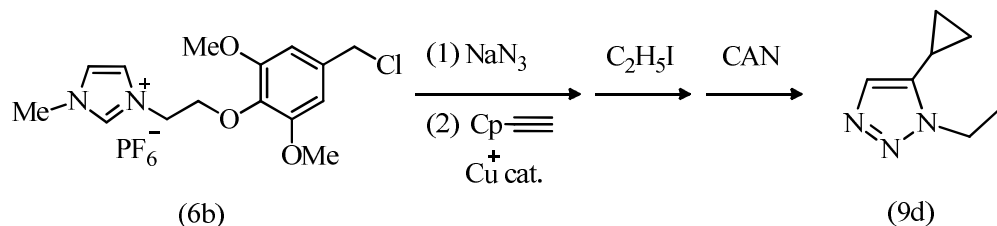
Yield: 118mg, (0.96mmol 81%); colorless oil. Rf = 0.30 (n-hexane/ethyl acetate 1:1)

$^1\text{H NMR}$ (500 MHz, CDCl_3): δ =0.68-0.72 (m, 2 H), 1.03-1.08 (m, 2 H), 1.67-1.73 (m, 1 H), 4.06 (s, 3 H), 7.27 (s, 1 H).

$^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ =3.3, 6.3 (2c), 34.1, 130.2, 139.5.

HRMS (APCI): m/z $[M + H]^+$ calcd for $C_6H_{10}N_3$: 124.0875; found: 124.0902.

5-Cyclopropyl-1-ethyl-1*H*-1,2,3-triazole (9d)



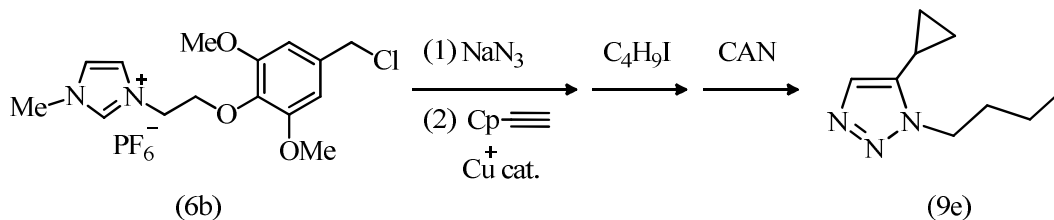
Yield: 125mg, (0.91mmol 71%); colorless oil. R_f = 0.43 (n-hexane/ethyl acetate 1:1)

¹H NMR (500 MHz, CDCl₃): δ=0.70-0.73 (m, 2 H), 1.04-1.08 (m, 2 H), 1.55 (t, $J = 7.3\text{Hz}$, 3 H), 1.68-1.73 (m, 1 H), 4.43 (q, $J = 7.3\text{Hz}$, 2 H), 7.26 (s, 1 H).

¹³C NMR (125 MHz, CDCl₃): δ=3.8, 6.9 (2 c), 15.3, 43.0, 130.7, 139.0.

HRMS (APCI): m/z $[M + H]^+$ calcd for $C_7H_{11}N_3$: 138.1031; found:138.1057.

5-Cyclopropyl-1-(*n*-butyl)-1*H*-1,2,3-triazole (9e)



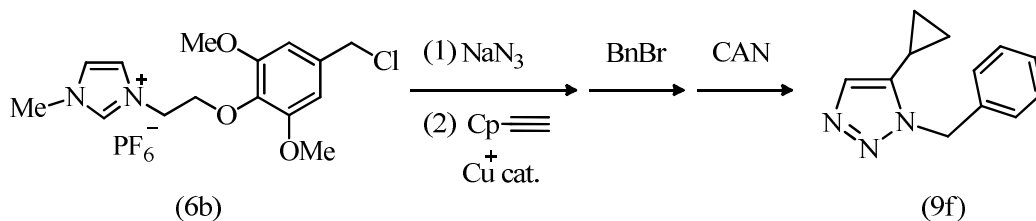
Yield: 141mg, (0.85mmol 66%); colorless oil. R_f = 0.50 (n-hexane/ethyl acetate 1:1)

¹H NMR (500 MHz, CDCl₃): δ=0.69-0.73 (m, 2 H), 0.97 (m, 3 H), 1.04-1.08 (m, 2 H), 1.35-1.43 (m, 2 H), 1.69-1.75 (m, 1 H), 1.88-1.95 (m, 2 H), 4.37 (m, 2 H), 7.24 (s, 1 H).

¹³C NMR (125 MHz, CDCl₃): δ=3.8, 6.9 (2 c), 13.5, 19.8, 31.8, 47.5, 130.3, 139.2.

HRMS (APCI): m/z $[M + H]^+$ calcd for $C_9H_{15}N_3$: 166.1344; found:166.1328.

1-Benzyl-5-cyclopropyl-1*H*-1,2,3-triazole (9f)



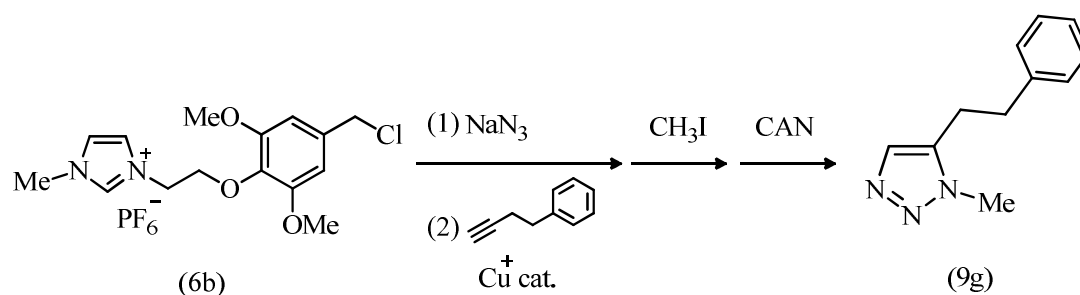
Yield: 176mg, (0.88mmol 70%); colorless oil. Rf = 0.45 (n-hexane/ethyl acetate 1:1)

^1H NMR (500 MHz, CDCl_3): δ =0.57-0.61 (m, 2 H), 0.89-0.94 (m, 2 H), 1.51-1.57 (m, 1 H), 5.59 (s, 2 H), 7.19-7.22 (m, 2 H), 7.28-7.35 (m, 4 H).

^{13}C NMR (125 MHz, CDCl_3): δ =3.9, 6.9 (2 c), 51.8, 127.4 (2 c), 128.3, 128.9 (2 c), 131.2, 135.2, 139.7.

HRMS (APCI): m/z $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{12}\text{H}_{13}\text{N}_3$: 200.1188; found: 200.1202.

1-Methyl-5-(phenylethyl)-1H-1,2,3-triazole (9g)



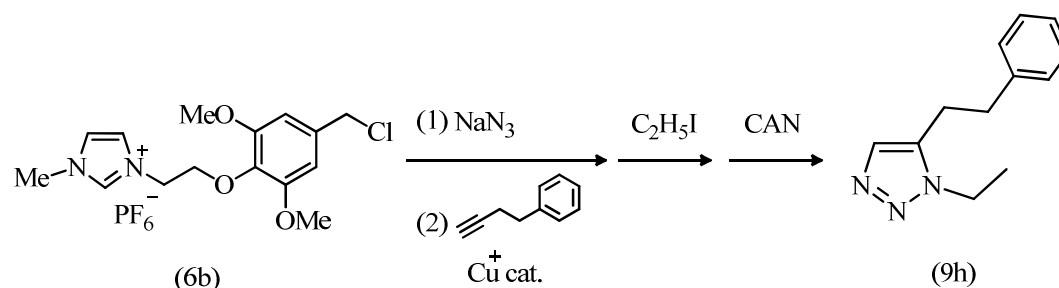
Yield: 191mg, (1.02mmol 88%); white solid; mp 61-62°C. Rf = 0.50 (n-hexane/ethyl acetate 1:1)

^1H NMR (500 MHz, CDCl_3): δ =2.96 (m, 4 H), 3.76 (s, 3 H), 7.11-7.14 (m, 2 H), 7.23-7.26 (m, 1 H), 7.28-7.32 (m, 2 H), 7.45 (s, 1 H).

^{13}C NMR (125 MHz, CDCl_3): δ =24.7, 33.7, 34.2, 126.3, 128.0(2 c), 128.3(2 c), 131.9, 136.3, 139.5.

HRMS (APCI): m/z $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{11}\text{H}_{13}\text{N}_3$: 188.1188; found: 188.1114.

1-Ethyl-5-Phenylethyl-1H-1,2,3-triazole (9h)



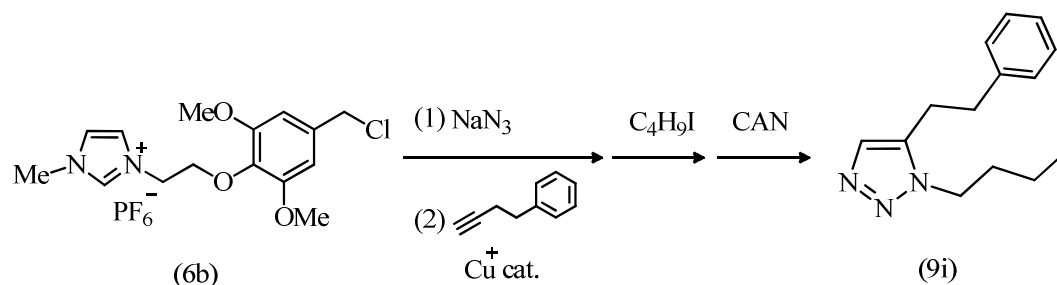
Yield: 197mg, (0.98mmol 78%); colorless oil. Rf = 0.53 (n-hexane/ethyl acetate 1:1)

^1H NMR (500 MHz, CDCl_3): δ =1.44 (t, J =7.3Hz, 3 H), 2.93-3.00 (m, 4 H), 4.17 (q, J =7.3Hz, 2 H), 7.14-7.17 (m, 2 H), 7.22-7.26 (m, 1 H), 7.29-7.33(m, 2 H), 7.47 (s, 1 H).

^{13}C NMR (125 MHz, CDCl_3): δ = 15.3, 25.1, 34.7, 42.6, 126.7, 128.3 (2 c), 128.7 (2 c), 132.3, 135.8, 139.8.

HRMS (APCI): m/z $[M + H]^+$ calcd for $C_{12}H_{15}N_3$: 202.1344; found:202.1369.

1-Butyl-5-phenylethyl-1H-1,2,3-triazole (9i)



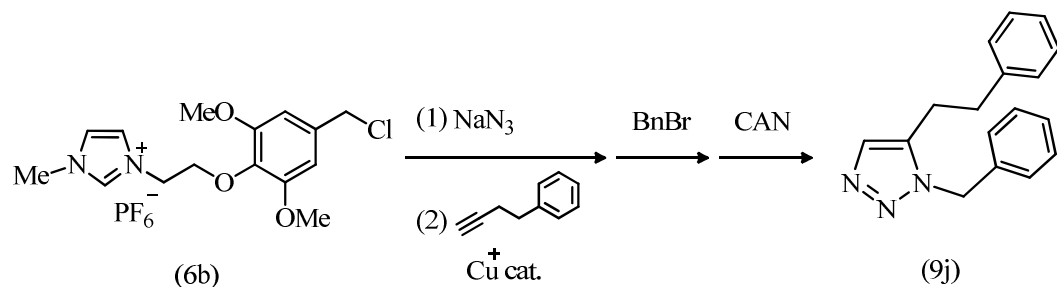
Yield: 210mg, (0.92mmol 71%); colorless oil. Rf = 0.53 (n-hexane/ethyl acetate 1:1)

^1H NMR (500 MHz, CDCl_3): δ = 0.93 (m, 3 H), 1.29-1.34 (m, 2 H), 1.74-1.80 (m, 2 H), 2.92-2.99 (m, 4 H), 4.10 (m, 2 H), 7.15-7.17 (m, 2 H), 7.22-7.26 (m, 1 H), 7.29-7.33 (m, 2 H), 7.47 (s, 1 H).

^{13}C NMR (125 MHz, CDCl_3): δ = 13.5, 19.8, 25.2, 32.0, 34.7, 47.4, 126.7, 128.3 (2 c), 128.7 (2 c), 132.1, 136.1, 139.9.

HRMS (APCI): m/z $[M + H]^+$ calcd for $C_{14}H_{19}N_3$: 230.1657; found:230.1666.

1-Benzyl-5-phenylethyl-1H-1,2,3-triazole (9j)



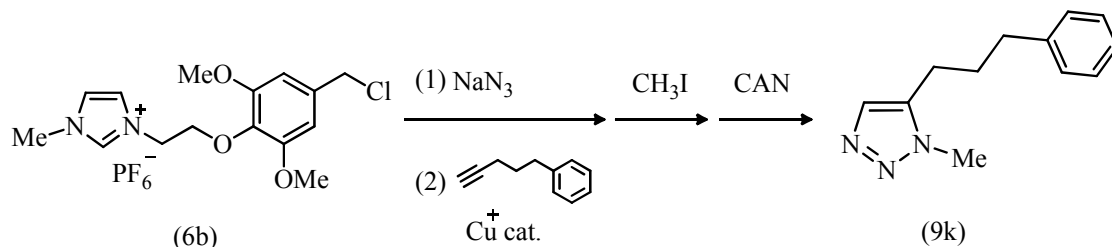
Yield: 180mg, (0.68mmol 54%); white solid; mp 70-72°C. Rf = 0.65 (n-hexane/ethyl acetate 1:1)

^1H NMR (500 MHz, CDCl_3): δ = 2.76-2.83 (m, 4 H), 5.36 (s, 2 H), 7.02-7.03 (m, 2 H), 7.10-7.12 (m, 2 H), 7.19-7.24 (m, 2 H), 7.25-7.29 (m, 2 H), 7.30-7.36 (m, 2 H), 7.48 (s, 1 H).

^{13}C NMR (125 MHz, CDCl_3): δ = 25.2, 34.6, 51.7, 126.8, 127.2 (2 c), 128.4 (2 c), 128.5, 128.8 (2 c), 129.1 (2 c), 133.1, 135.1, 136.6, 139.9.

HRMS (APCI): m/z $[M + H]^+$ calcd for $C_{17}H_{17}N_3$: 141.0980; found:142.0999.

1-Methyl-5-(phenylpropyl)-1H-1,2,3-triazole (9k)



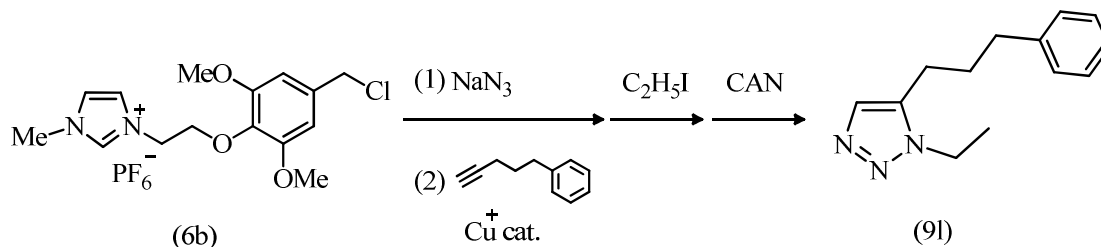
Yield: 197mg, (0.98mmol 83%); colorless oil. Rf = 0.50 (n-hexane/ethyl acetate 1:1)

$^1\text{H NMR}$ (500 MHz, CDCl_3): δ =2.01 (m, 2 H), 2.63 (t, J =7.5Hz, 2 H), 2.72 (t, J =7.5Hz, 2 H), 3.90 (s, 3 H), 7.16-7.18 (m, 2 H), 7.21-7.24 (m, 1 H), 7.29-7.33 (m, 2 H), 7.47 (s, 1 H).

$^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ =22.0, 29.0, 33.8, 34.7, 125.9, 128.1 (2 c), 128.2 (2 c), 131.7, 136.7, 140.6.

HRMS (APCI): m/z $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{12}\text{H}_{15}\text{N}_3$: 202.1344; found: 202.1363.

5-Phenylpropyl-1-ethyl-1H-1,2,3-triazole (9l)



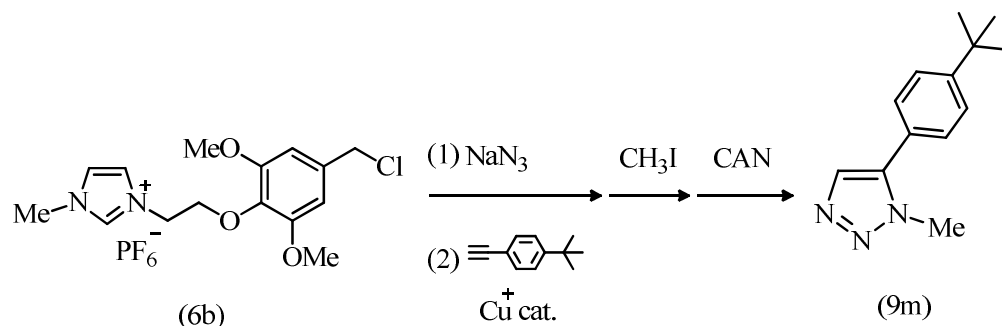
Yield: 190mg, (0.88mmol 70%); yellow oil. Rf = 0.50 (n-hexane/ethyl acetate 1:1)

$^1\text{H NMR}$ (500 MHz, CDCl_3): δ =1.45 (t, J =7.3Hz, 3 H), 1.97-2.04 (m, 2 H), 2.62 (m, 2 H), 2.71(m, 2H), 4.22 (q, J =7.3Hz, 2 H), 7.17 (d, J =7.5Hz, 2 H), 7.21 (t, J =7.5Hz, 1 H), 7.30 (t, J =7.5Hz, 2 H), 7.45 (s, 1 H).

$^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ =15.3, 22.3, 29.6, 35.0, 42.6, 126.2, 128.4 (2 c), 128.5 (2 c), 132.1, 136.2, 140.9.

HRMS (APCI): m/z $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{13}\text{H}_{17}\text{N}_3$: 216.1501; found:216.1483.

5-(4-*t*-Butyl-phenyl)-1-methyl-1H-1,2,3-triazole (9m)



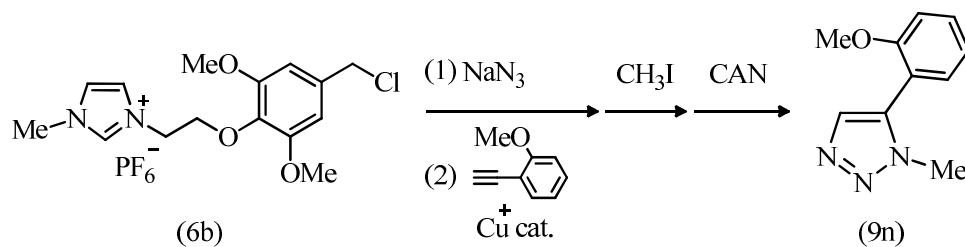
Yield: 142mg, (0.66mmol 56%); colorless oil. Rf = 0.33 (n-hexane/ethyl acetate 7:3)

¹H NMR (500 MHz, CDCl₃): δ=1.37 (s, 9 H), 4.08 (s, 3 H), 7.36 (d, *J*= 8.5 Hz, 2 H), 7.52 (d, *J*=8.5 Hz, 2 H), 7.71 (s, 1 H).

¹³C NMR (125 MHz, CDCl₃): δ=31.0(3 c), 34.5, 35.3, 123.7, 125.8(2 c), 128.0(2 c), 132.4, 137.8, 152.4.

HRMS (APCI): *m/z* [M + H]⁺ calcd for C₁₃H₁₇N₃: 216.1501; found: 216.1505.

5-(2-Methoxy-phenyl)-1-methyl-1H-1,2,3-triazole (9n)



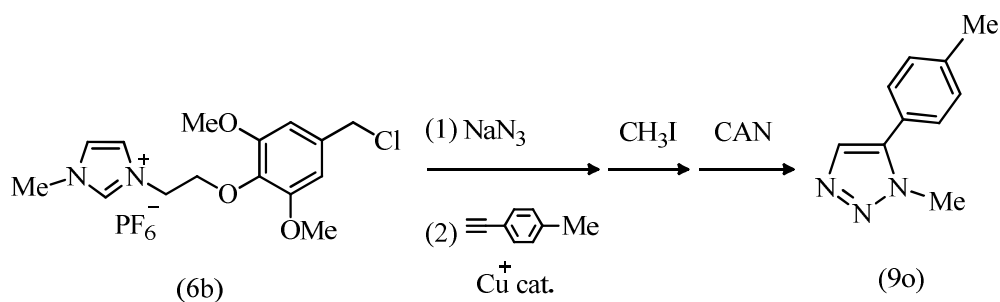
Yield: 175mg, (0.92mmol 77%); colorless oil. Rf = 0.38 (n-hexane/ethyl acetate 1:1)

¹H NMR (500 MHz, CDCl₃): δ=3.83 (s, 3 H), 3.93 (s, 3 H), 7.03-7.47 (m, 4 H), 7.66 (s, 1 H).

¹³C NMR (125 MHz, CDCl₃): δ=35.0, 55.2, 111.0, 115.3, 120.5, 130.9, 131.2, 133.3, 135.0, 156.6.

HRMS (APCI): *m/z* [M + H]⁺ calcd for C₁₀H₁₁N₃O: 190.0980; found: 190.0926.

5-(4-Methyl-phenyl)-1-methyl-1H-1,2,3-triazole (9o)



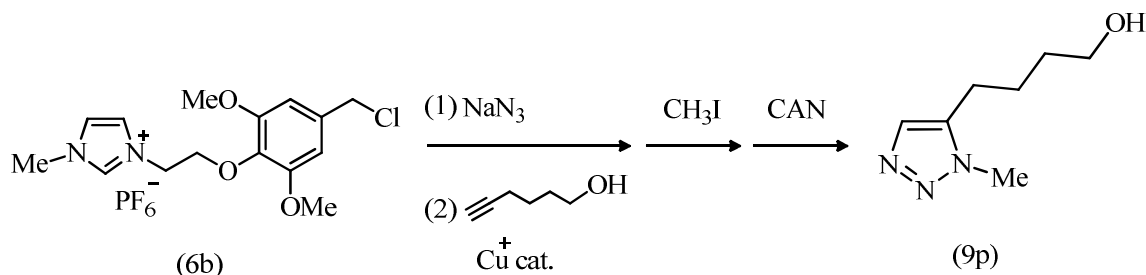
Yield: 73mg, (0.42mmol 35%); yellow oil Rf = 0.43 (n-hexane/ethyl acetate 1:1).

$^1\text{H NMR}$ (500 MHz, CDCl_3): δ =2.43 (s, 3 H), 4.07 (s, 3 H), 7.31 (s, 4 H), 7.70 (s, 1 H).

$^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ =21.3, 35.4, 123.8, 128.3 (2 c), 129.7 (2 c), 132.6, 138.1, 139.6.

HRMS (APCI): m/z $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{10}\text{H}_{11}\text{N}_3$: 174.1031; found: 174.0999.

5-(Butyl-4-ol)-1-methyl-1H-1,2,3-triazole (9p)



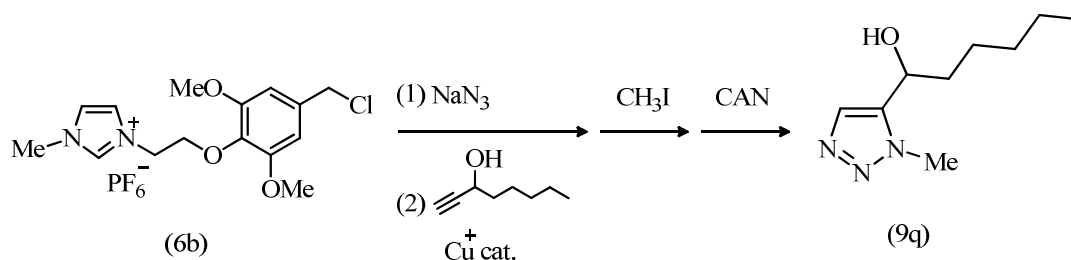
Yield: 73mg, (0.47mmol 39%); yellow oil. Rf = 0.25 (chloroform/ methanol 9:1)

$^1\text{H NMR}$ (500 MHz, CDCl_3): δ =1.66 (m, 2 H), 1.78 (s, 2 H), 2.68 (m, 2 H), 3.70 (m, 2 H), 3.96 (s, 3 H), 7.44 (s, 1 H).

$^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ =22.8, 24.3, 31.8, 34.2, 61.5, 131.8, 137.5.

HRMS (APCI): m/z $[\text{M} + \text{H}]^+$ calcd for $\text{C}_7\text{H}_{13}\text{N}_3\text{O}$: 156.1137; found: 156.1130.

5-(Hexyl-1-ol)-1-methyl-1H-1,2,3-triazole (9q)



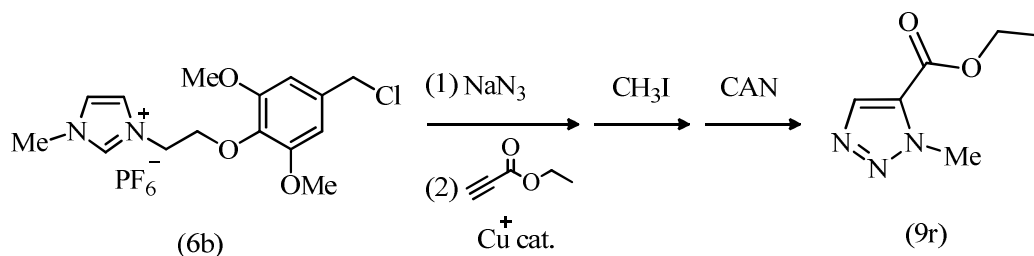
Yield: 176mg, (0.96mmol 80%); yellow oil. R_f = 0.45 (chloroform/ methanol 9:1)

¹H NMR (500 MHz, CDCl₃): δ=0.90 (m, 3 H), 1.34 (m, 6 H), 1.78-1.95 (m, 3 H), 4.09 (s, 3 H), 4.79(m, 1 H), 7.47(s, 1 H).

¹³C NMR (125 MHz, CDCl₃): δ=13.9, 22.5, 25.3, 31.4, 35.3, 36.0, 63.9, 131.2, 140.2.

HRMS (APCI): *m/z* [M + H]⁺ calcd for C₉H₁₇N₃O: 184.1450; found: 184.1403.

5-(Ethylcarboxylate)-1-methyl-1H-1,2,3-triazole (9r)



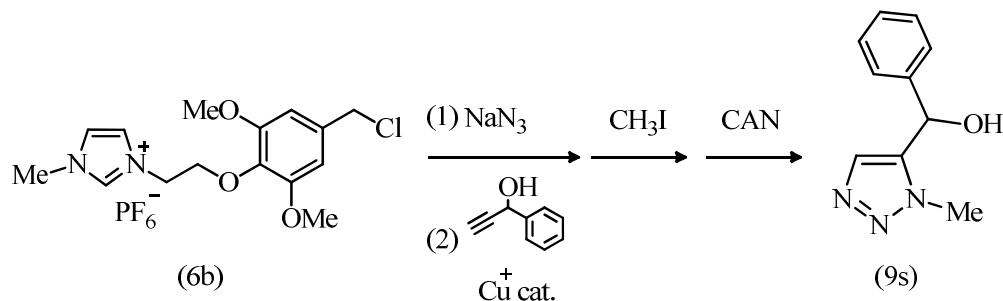
Yield: 40mg, (0.26mmol 20%); colorless oil. R_f = 0.65 (n-hexane/ethyl acetate 1:1)

¹H NMR (500 MHz, CDCl₃): δ=1.41 (t, *J*=7.0Hz, 3 H), 4.34 (s, 3 H), 4.40 (q, *J*=7.0Hz, 2 H), 8.12 (s, 1 H).

¹³C NMR (125 MHz, CDCl₃): δ=14.2, 37.5, 61.8, 128.5, 137.8, 158.6.

HRMS (APCI): *m/z* [M + H]⁺ calcd for C₆H₉N₃O₂: 156.0773; found: 156.0764.

5-[1-(Phenyl)-methyl-1-ol]-1-methyl-1H-1,2,3-triazole (9s)



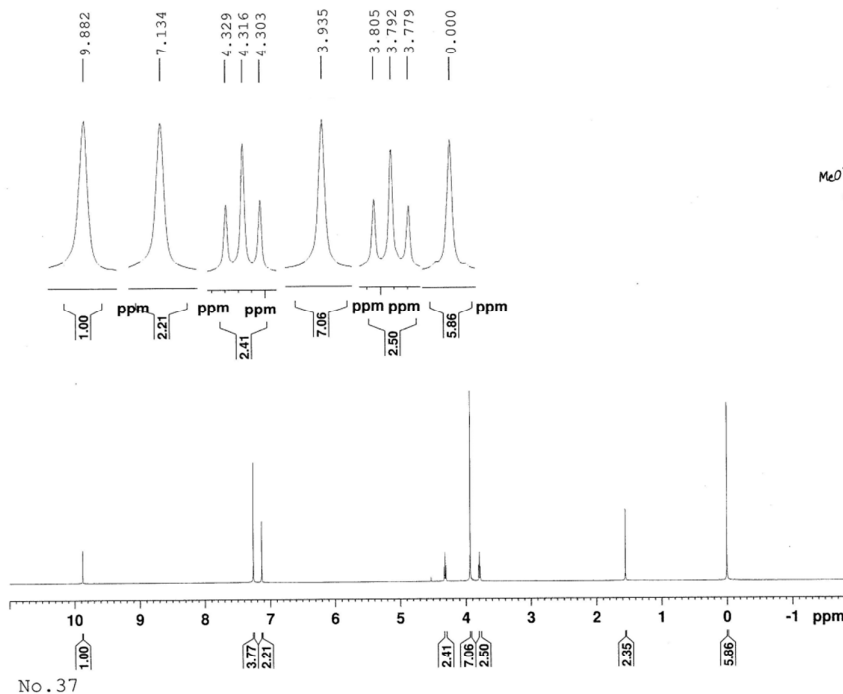
Yield: 53mg, (0.28mmol 23%); yellow oil. R_f = 0.30 (n-hexane/ethyl acetate 1:1)

^1H NMR (500 MHz, CDCl_3): δ =3.93 (s, 3 H), 5.96 (s, 1 H), 7.36(m, 6 H).

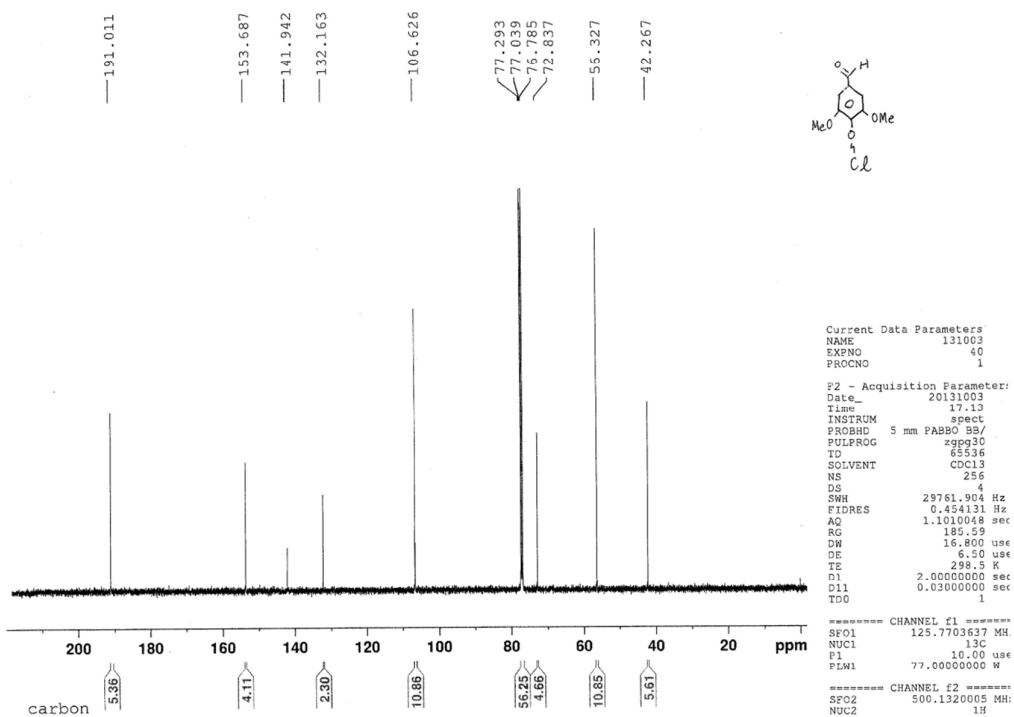
^{13}C NMR (125 MHz, CDCl_3): δ =35.6, 66.5, 126.4, 128.5(2 c), 128.8(2 c), 133.0, 139.6, 140.0.HRMS (APCI): m/z $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{10}\text{H}_{11}\text{N}_3\text{O}$: 190.0980; found: 190.0921.

Scanned NMR Spectra

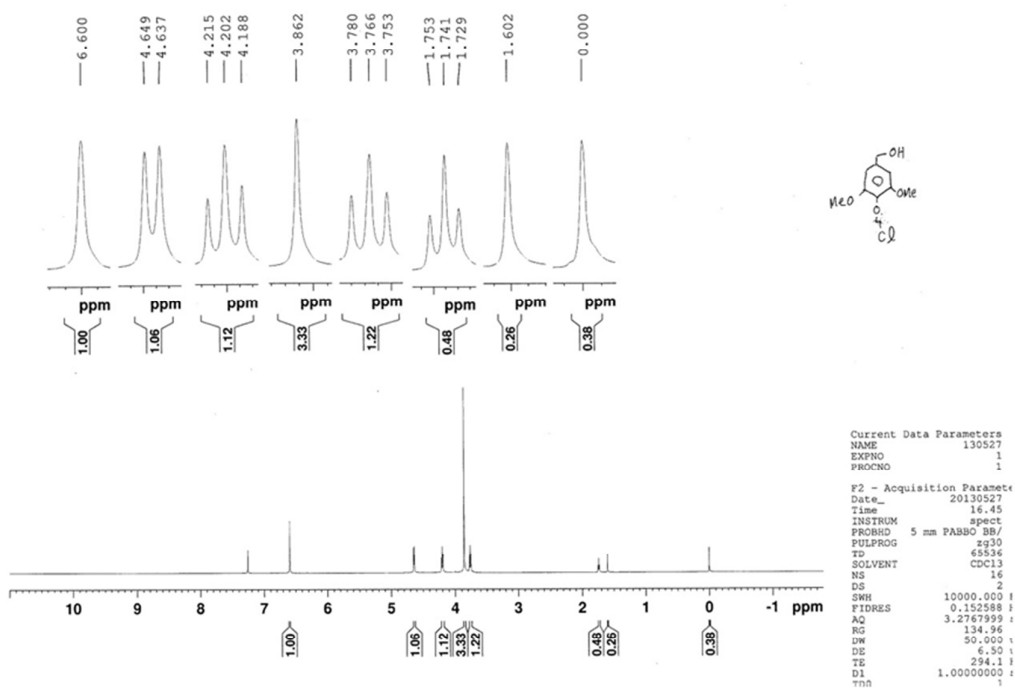
¹H NMR of 4-(2-chloroethoxy)-3,5-dimethoxybenzaldehyde (2)



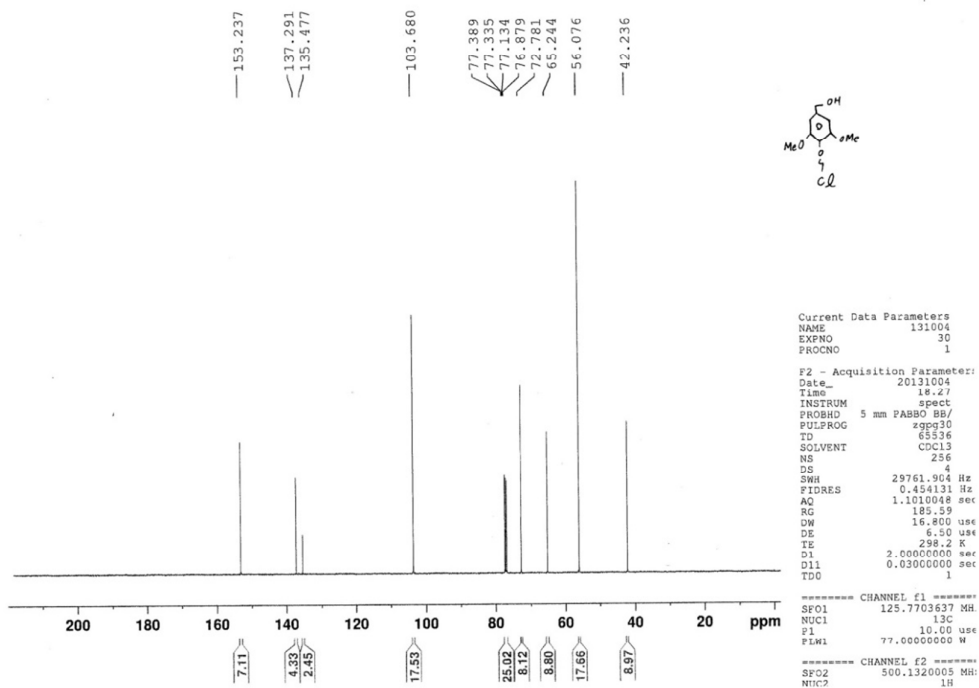
¹³C NMR of 4-(2-chloroethoxy)-3,5-dimethoxybenzaldehyde (2)



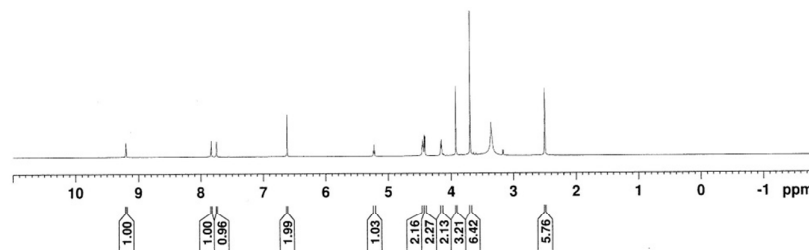
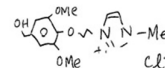
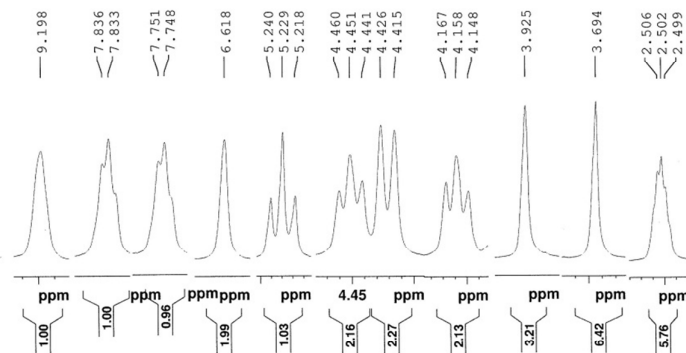
¹H NMR of (4-(2-chloroethoxy)-3,5-dimethoxyphenyl)methanol (3)



¹³C NMR of (4-(2-chloroethoxy)-3,5-dimethoxyphenyl)methanol (3)

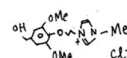
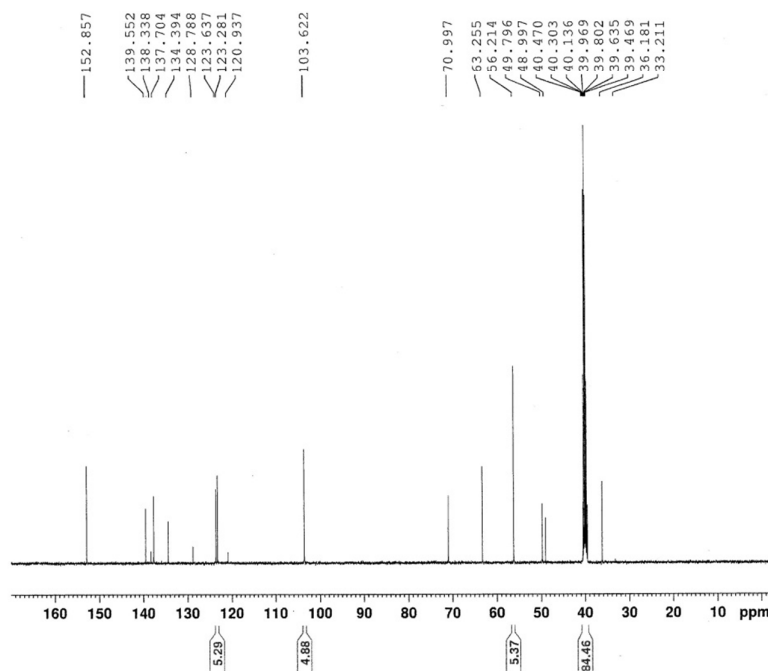


¹H NMR of 3-(2-(4-(hydroxymethyl)-2,6-dimethoxyphenoxy)ethyl)-1-methyl-imidazolium chloride (4)



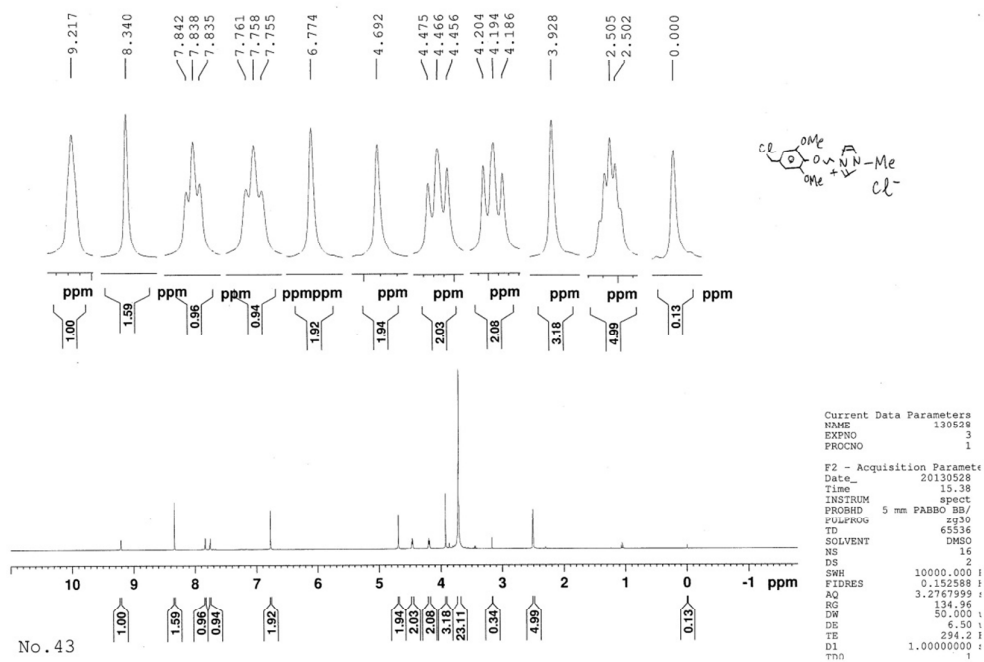
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 DS 2
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 FIDRES 0.152588 Hz
 AQ 3.2767999 sec
 RG 119.97
 DW 50.000 usec
 DE 6.50 usec
 TE 296.2 K
 D1 1.00000000 sec
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¹³C NMR of 3-(2-(4-(hydroxymethyl)-2,6-dimethoxyphenoxy)ethyl)-1-methyl-imidazolium chloride (4)

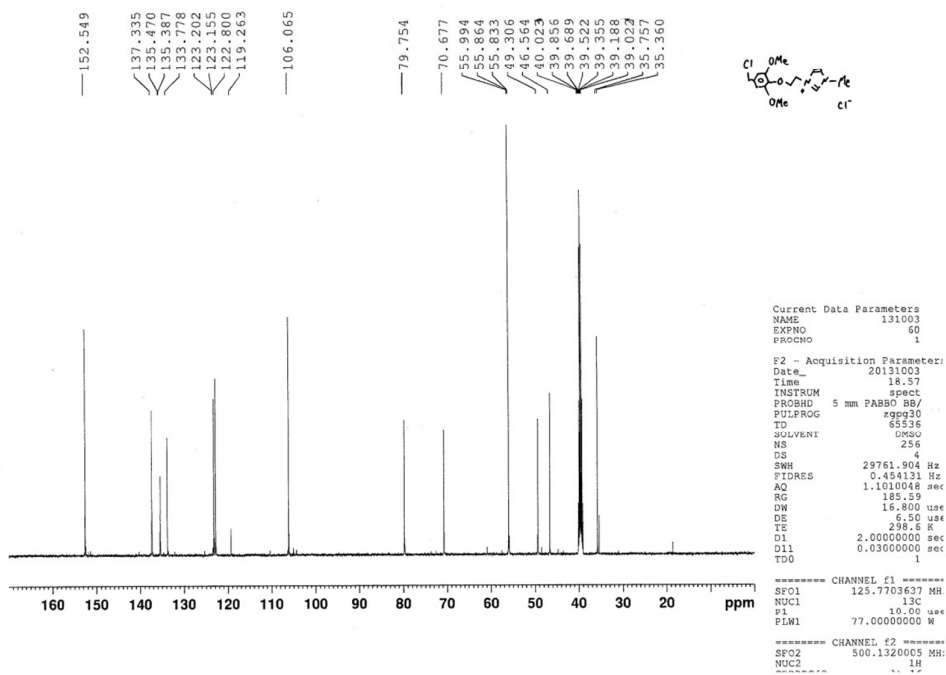


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 PROCNO 1
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 DS 4
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 FIDRES 0.454131 Hz
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 D11 0.03000000 sec

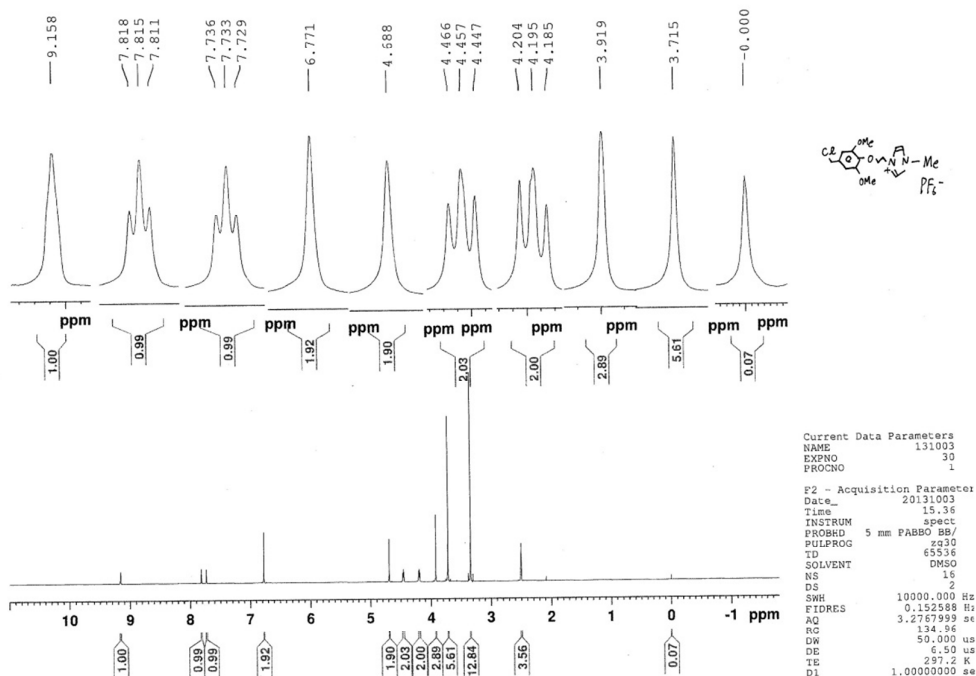
¹H NMR of 3-(2-(4-(chloromethyl)-2,6-dimethoxyphenoxy)ethyl)-1-methyl-imidazolium chloride (5)



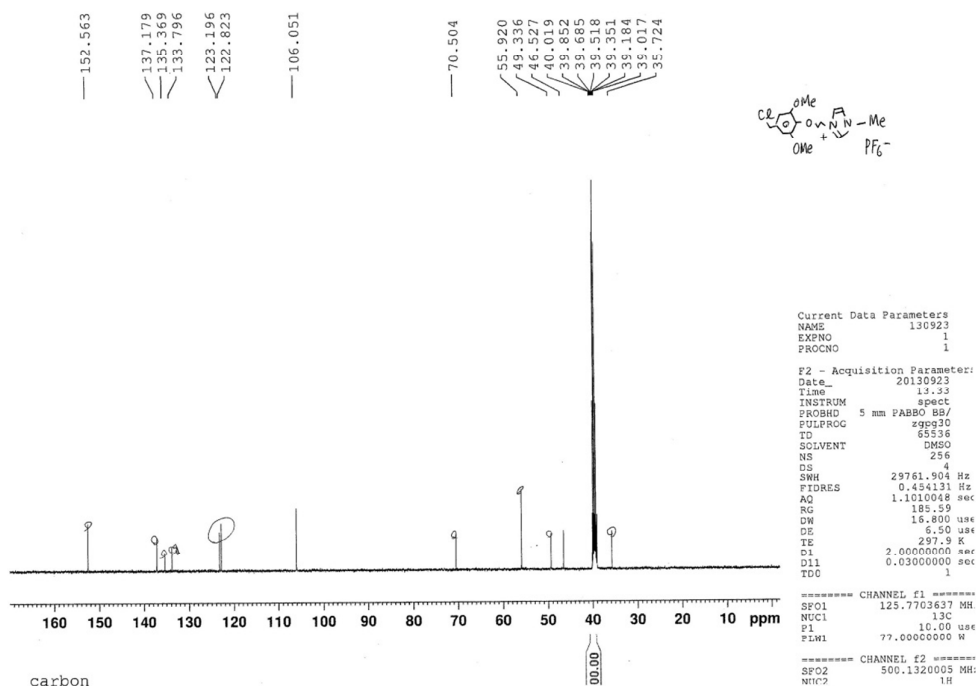
¹³C NMR of 3-(2-(4-(chloromethyl)-2,6-dimethoxyphenoxy)ethyl)-1-methyl-imidazolium chloride (5)



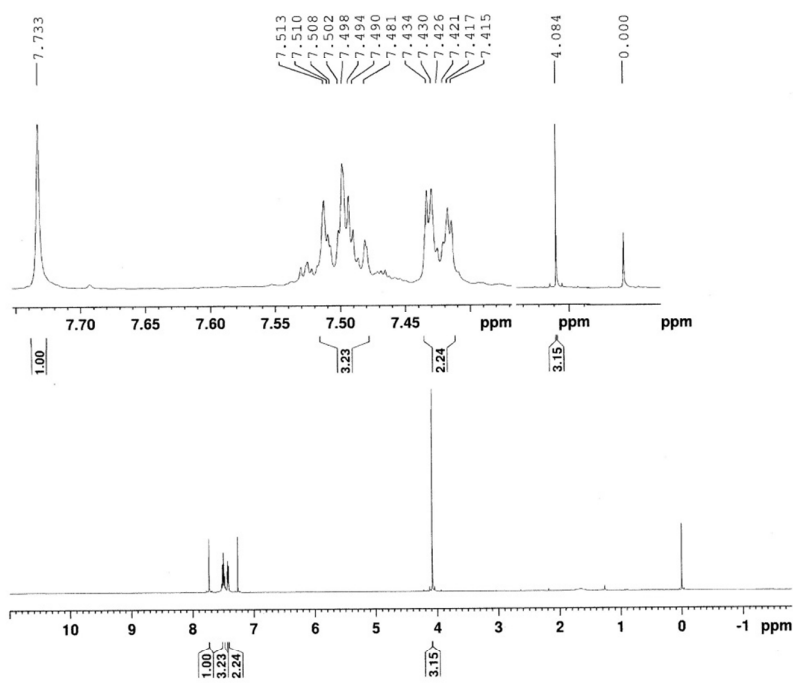
¹H NMR of 3-(2-(4-(chloromethyl)-2,6-dimethoxyphenoxy)ethyl)-1-methyl-imidazolium
hexafluorophosphate (6b)



¹³C NMR of 3-(2-(4-(chloromethyl)-2,6-dimethoxyphenoxy)ethyl)-1-methyl-imidazolium
hexafluorophosphate (6b)



¹H NMR of 1-Methyl-5-phenyl-1H-1,2,3-triazole (9a)



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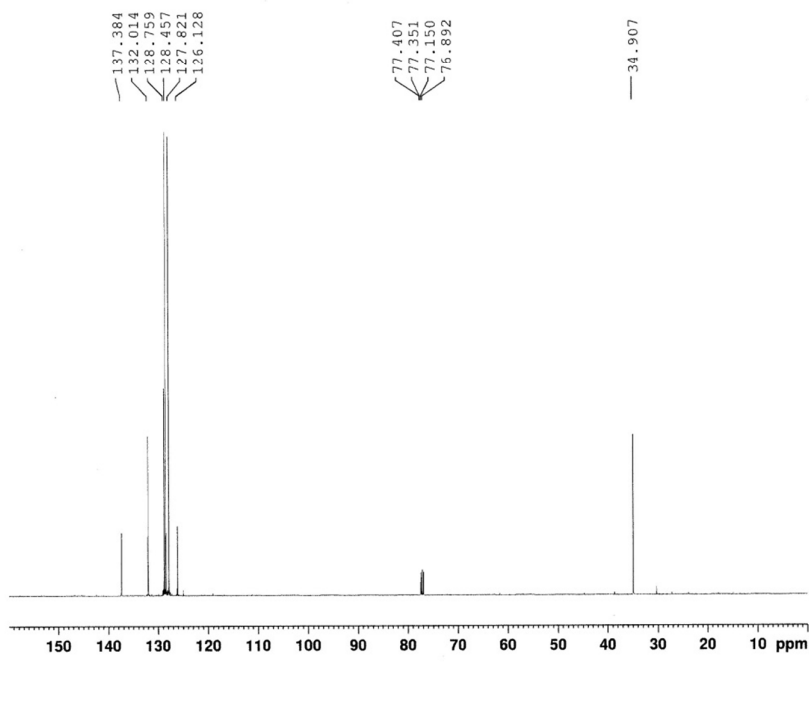
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AQ            3.2767989 sec
RG            185.59
DE            6.50 usec
TE            296.5 K
D1            1.00000000 sec
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NUC1           1H
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F2 - Processing parameters
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¹³C NMR of 1-Methyl-5-phenyl-1H-1,2,3-triazole (9a)



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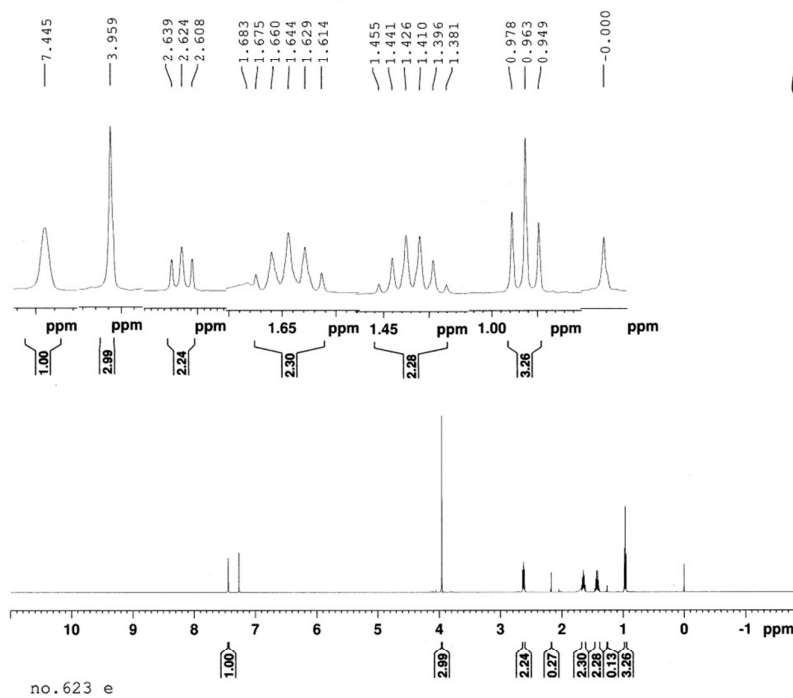
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RG            185.59
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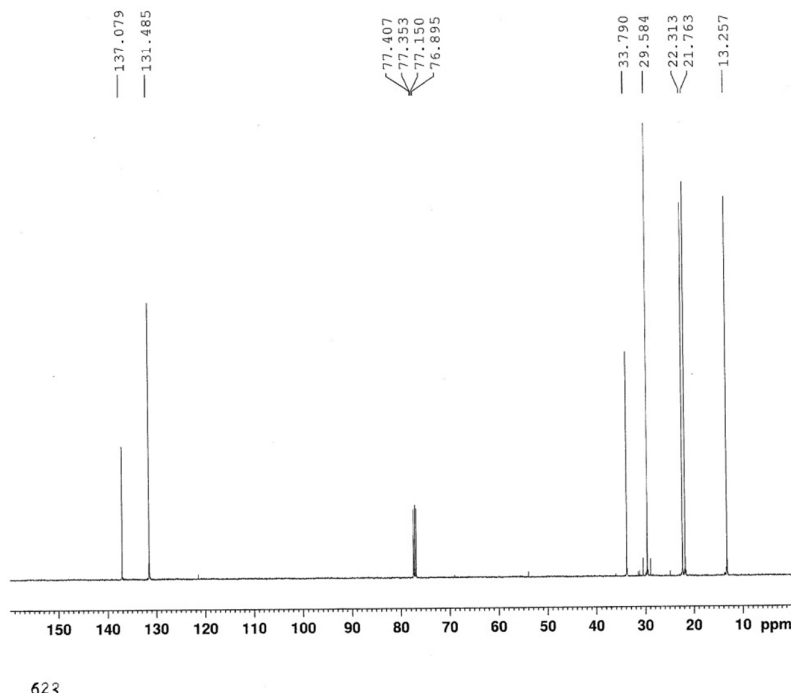
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NUC2           1H
  
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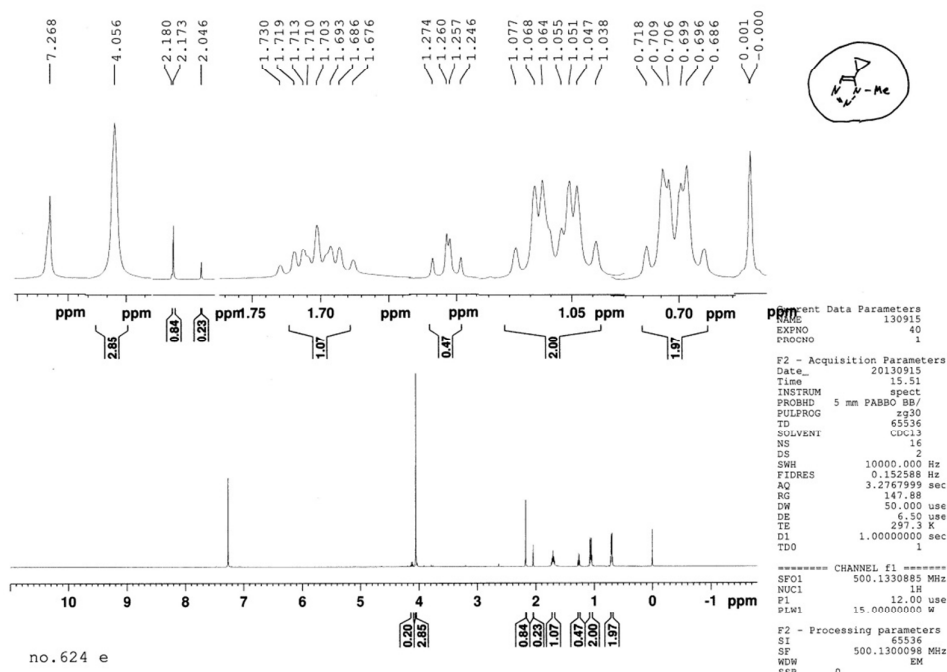
¹H NMR of **5-Butyl-1-methyl-1H-1,2,3-triazole (9b)**



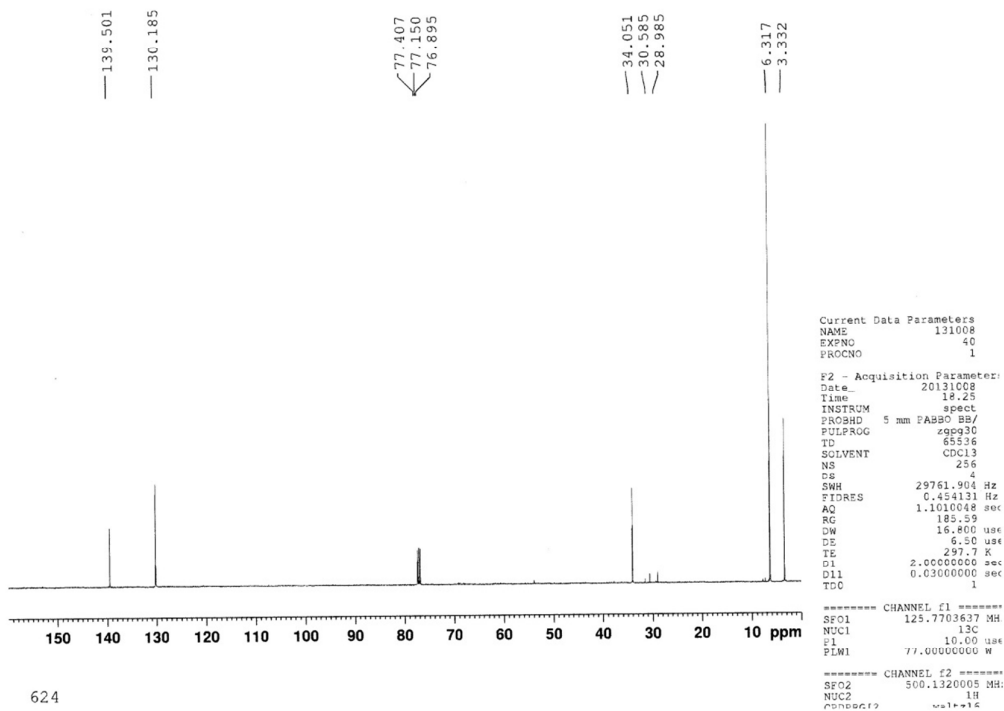
¹³C NMR of **5-Butyl-1-methyl-1H-1,2,3-triazole (9b)**



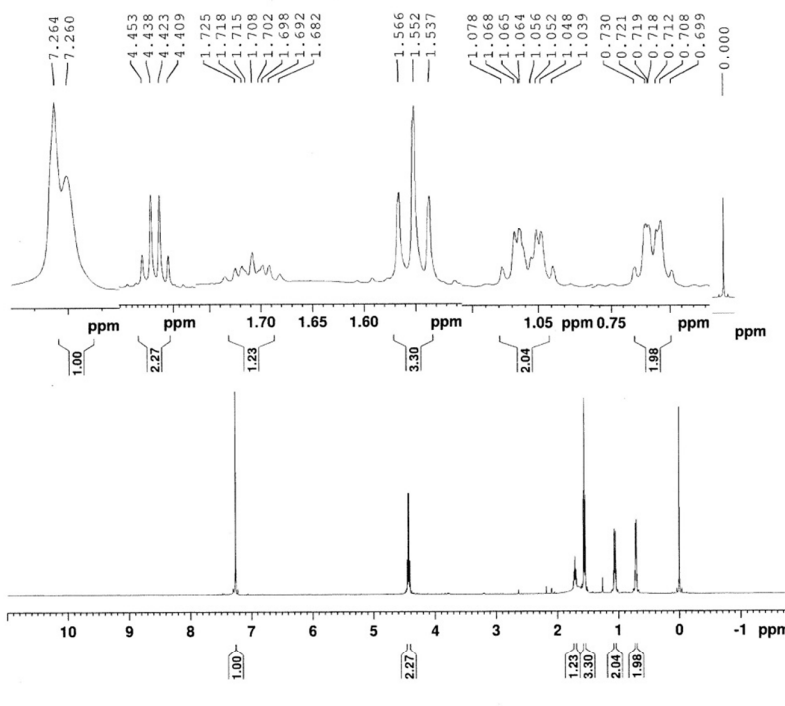
¹H NMR of 5-Cyclopropyl-1-methyl-1H-1,2,3-triazole (9c)



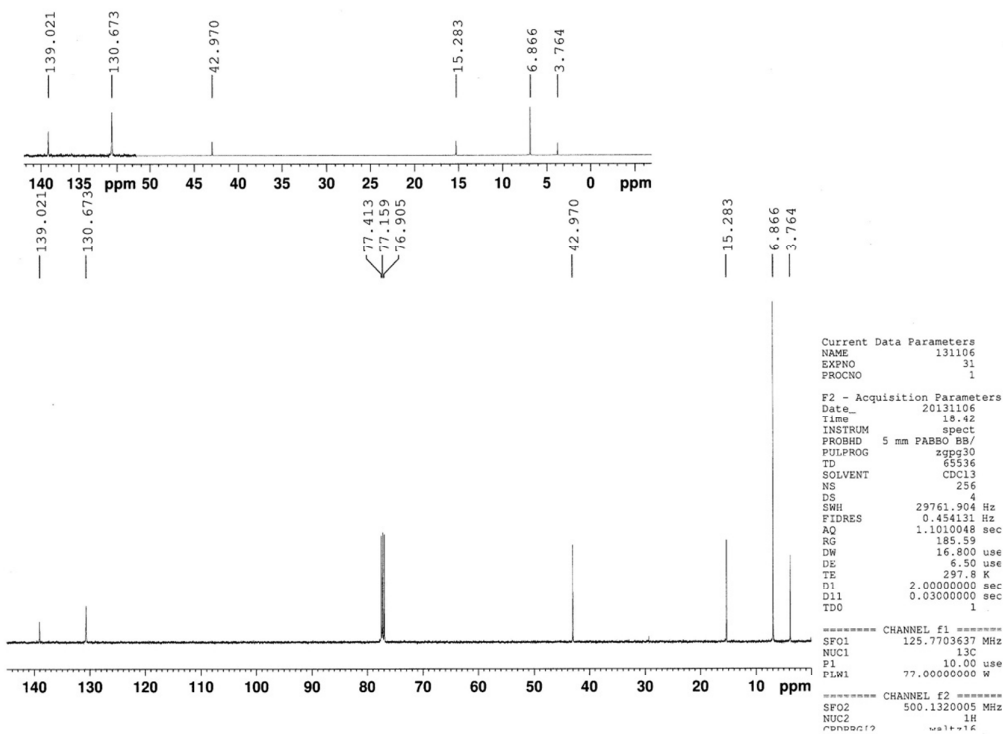
¹³C NMR of 5-Cyclopropyl-1-methyl-1H-1,2,3-triazole (9c)



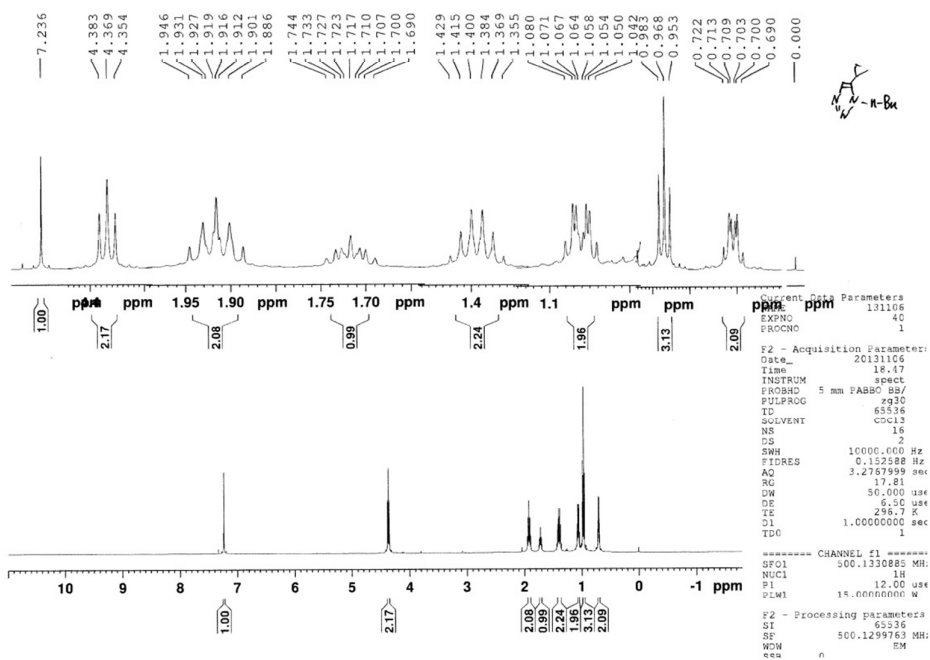
¹H NMR of 5-Cyclpropyl-1-ethyl-1H-1,2,3-triazole (9d)



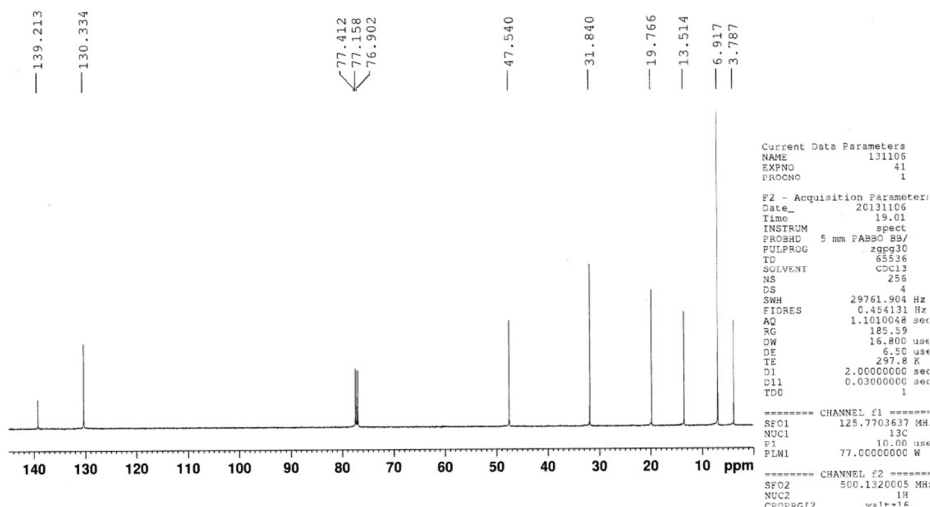
¹³C NMR of 5-Cyclpropyl-1-ethyl-1H-1,2,3-triazole (9d)



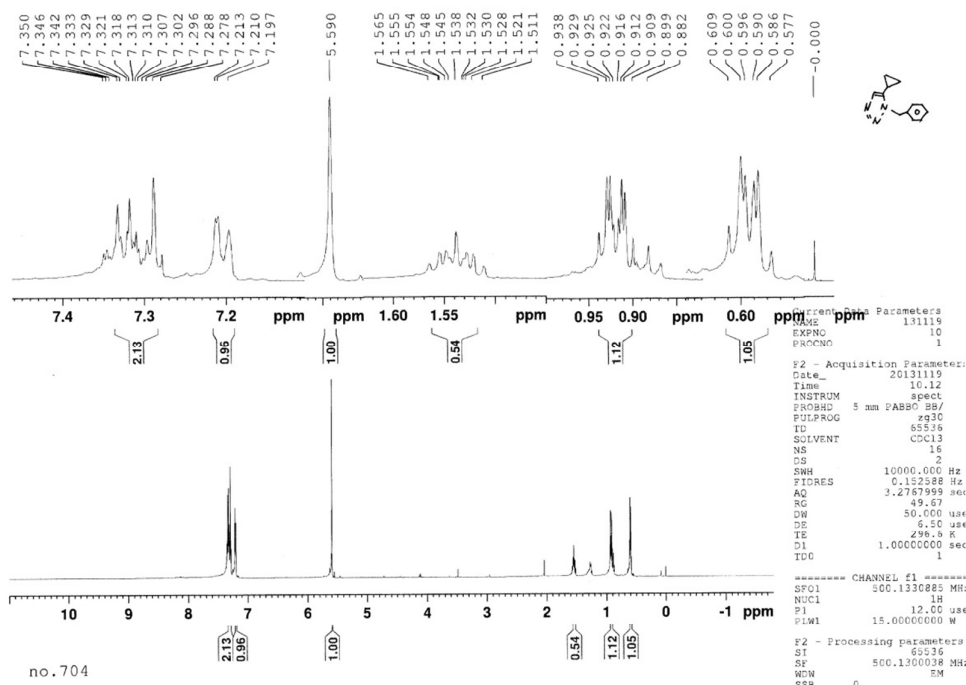
¹H NMR of **5-Cyclopropyl-1-(n-butyl)-1H-1,2,3-triazole (9c)**



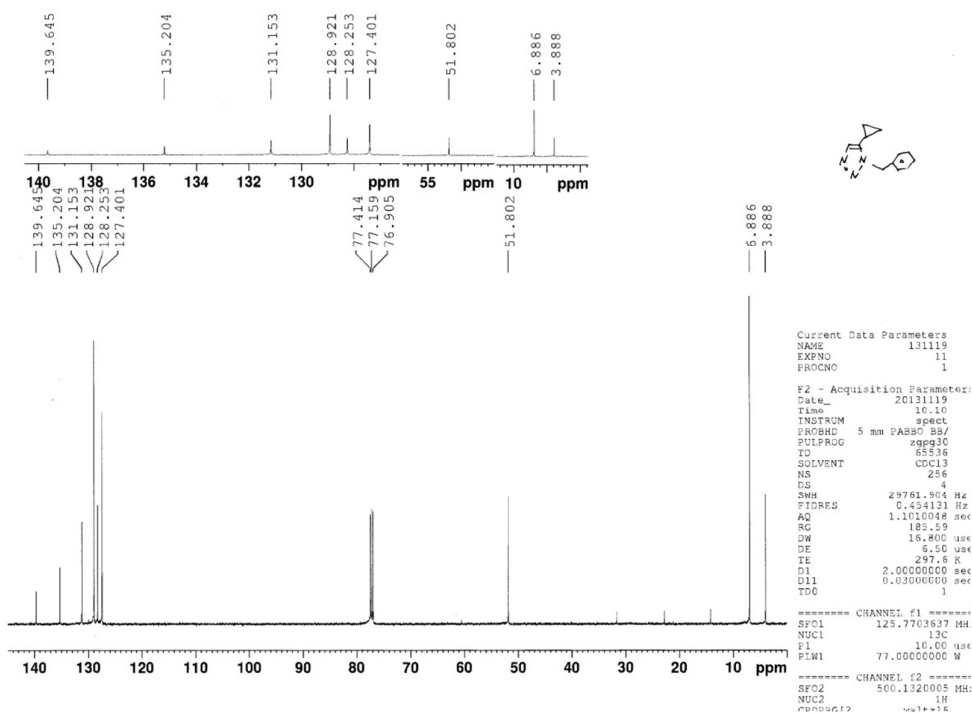
¹³C NMR of **5-Cyclopropyl-1-(n-butyl)-1H-1,2,3-triazole (9c)**



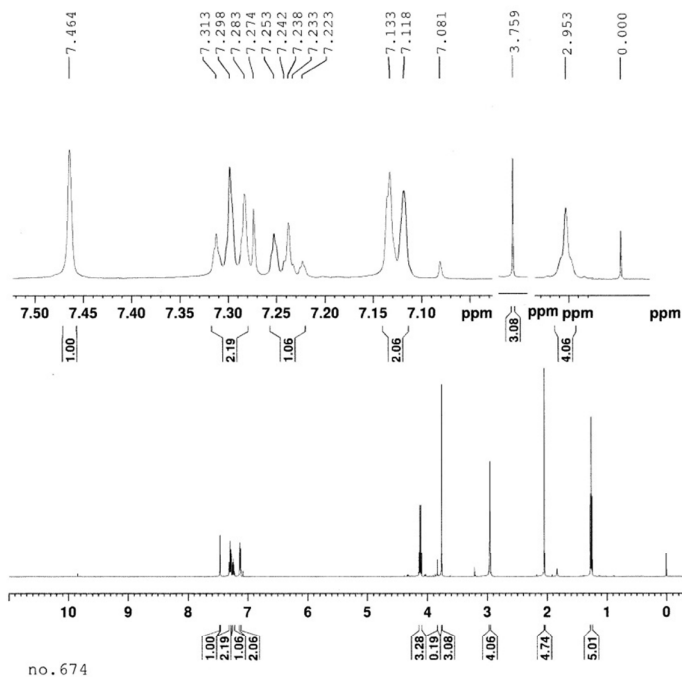
¹H NMR of **1-Benzyl-5-cyclopropyl-1H-1,2,3-triazole (9f)**



¹³C NMR of **1-Benzyl-5-cyclopropyl-1H-1,2,3-triazole (9f)**



¹H NMR of 1-Methyl-5-(phenylethyl)-1H-1,2,3-triazole (9g)



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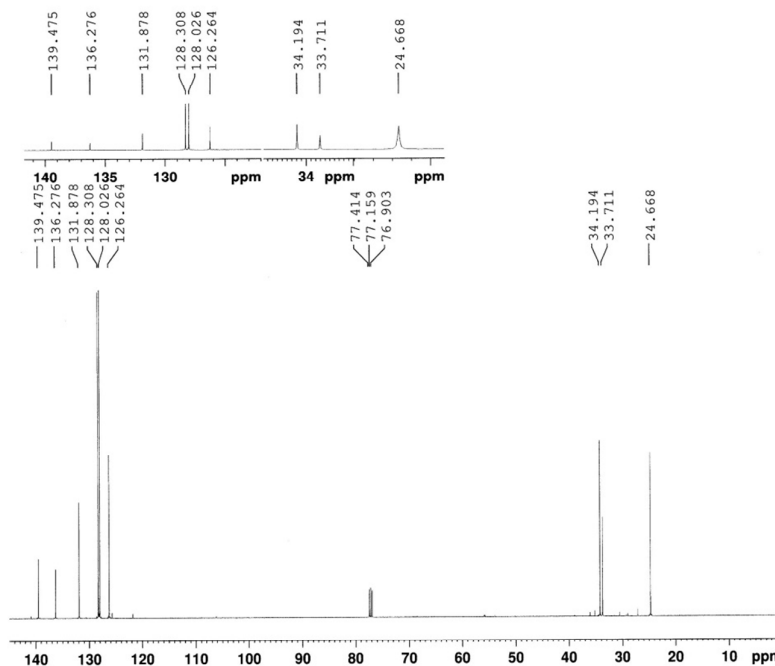
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RG         69.85
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D1         1.00000000 sec
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----- CHANNEL f1 -----
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NUC1       1H
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F2 - Processing parameters
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SF         500.1320069 MHz
WDW        EM
ca
    
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¹³C NMR of 1-Methyl-5-(phenylethyl)-1H-1,2,3-triazole (9g)



CN1C=NC=C1C2=CC=CC=C2C

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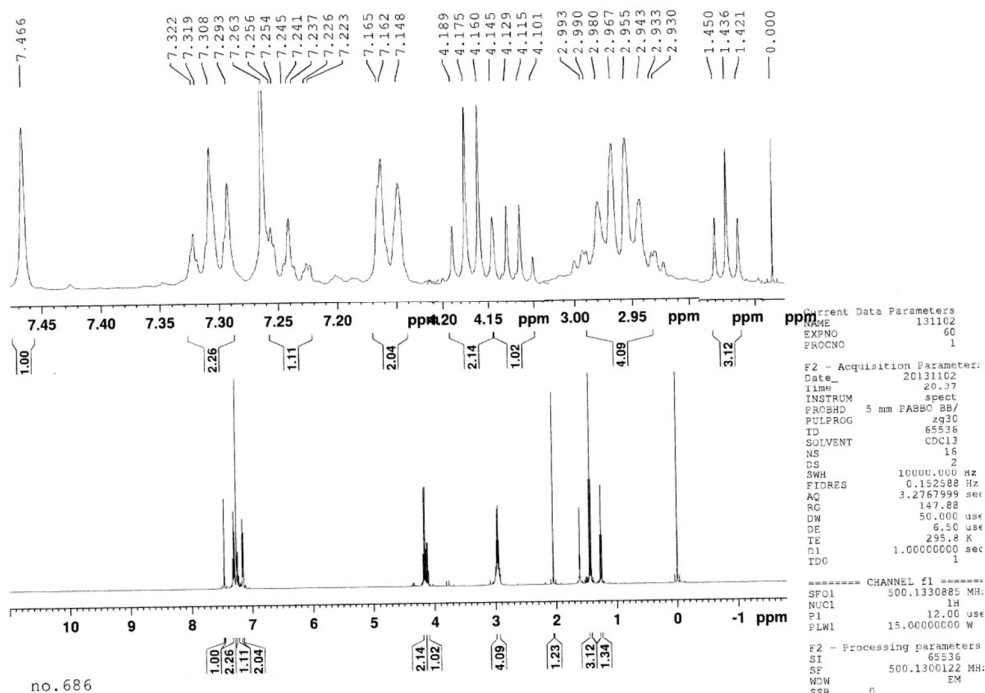
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FIDRES     0.454131 Hz
AQ         1.11010048 sec
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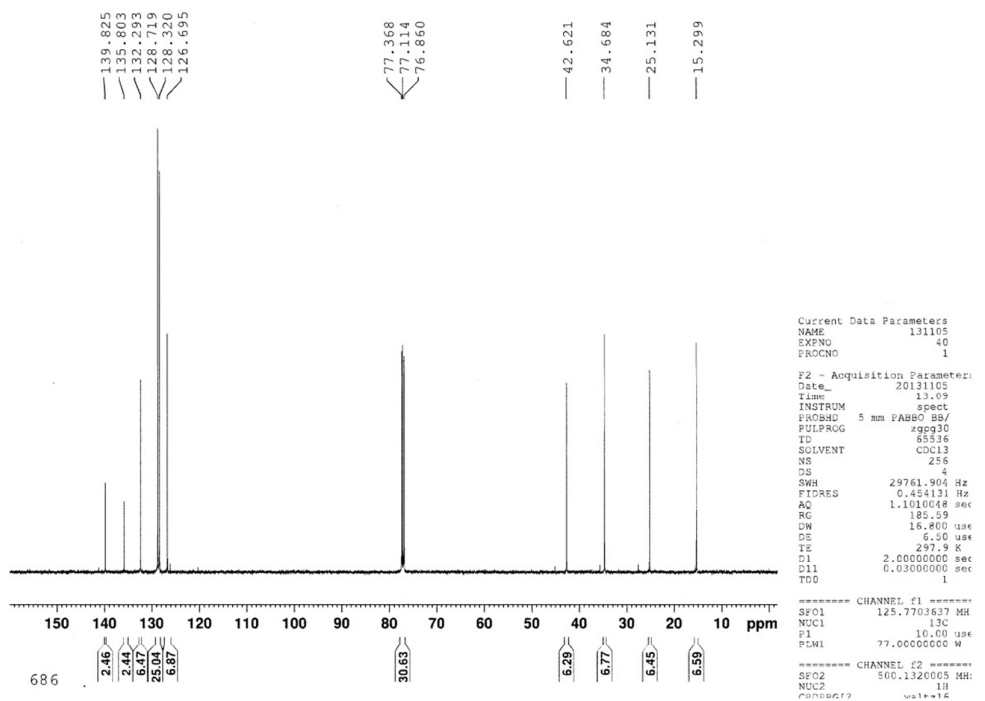
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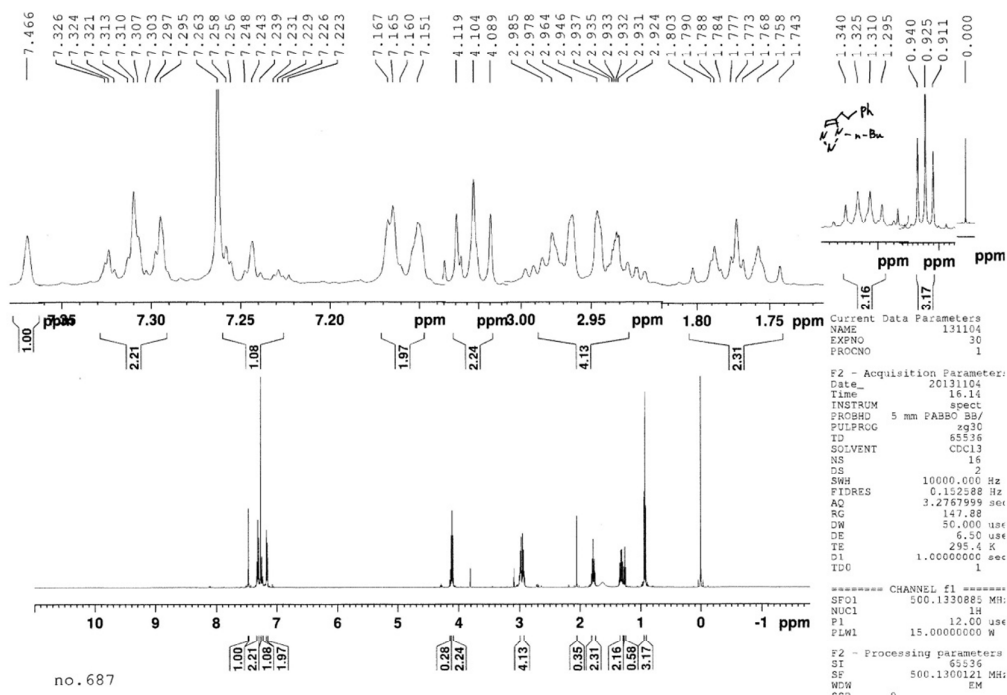
¹H NMR of 1-Ethyl-5-Phenylethyl-1H-1,2,3-triazole (9h)



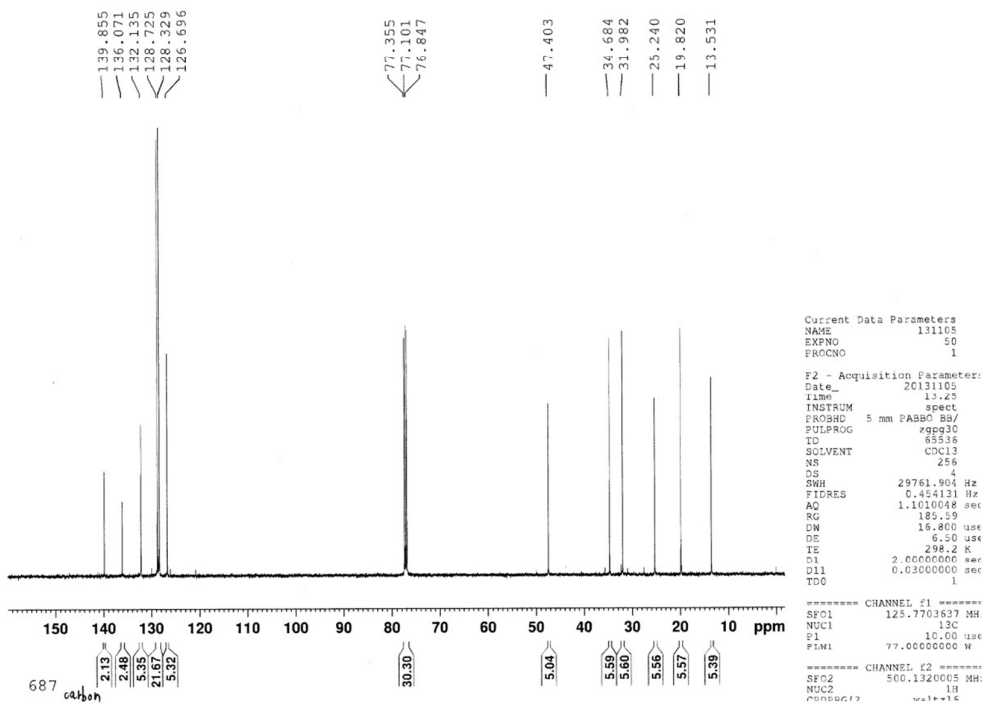
¹³C NMR of 1-Ethyl-5-Phenylethyl-1H-1,2,3-triazole (9h)



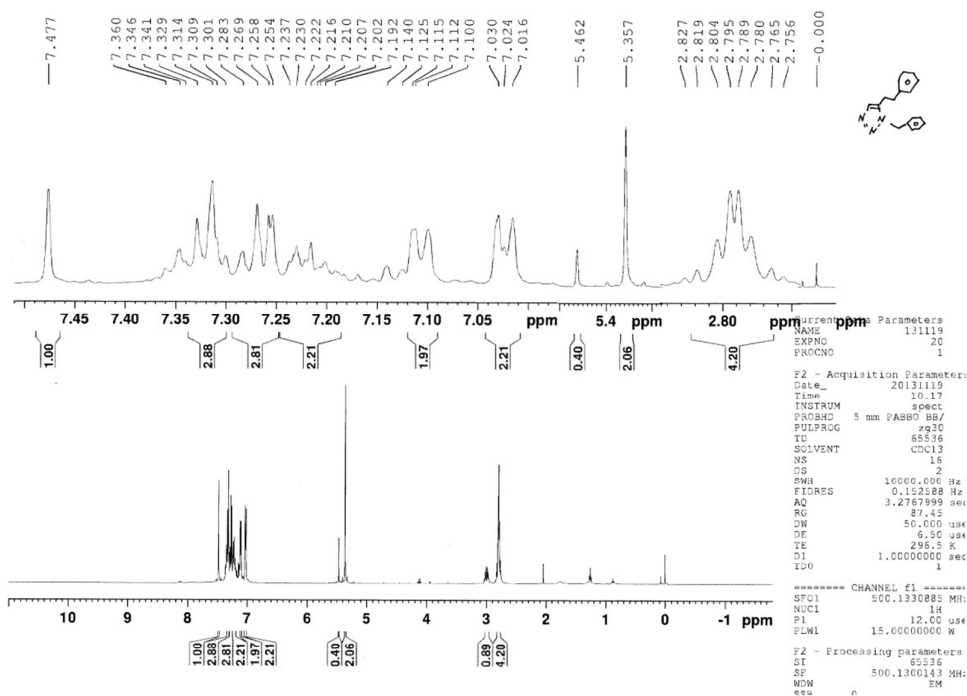
¹H NMR of 1-Butyl-5-phenylethyl-1H-1,2,3-triazole (9i)



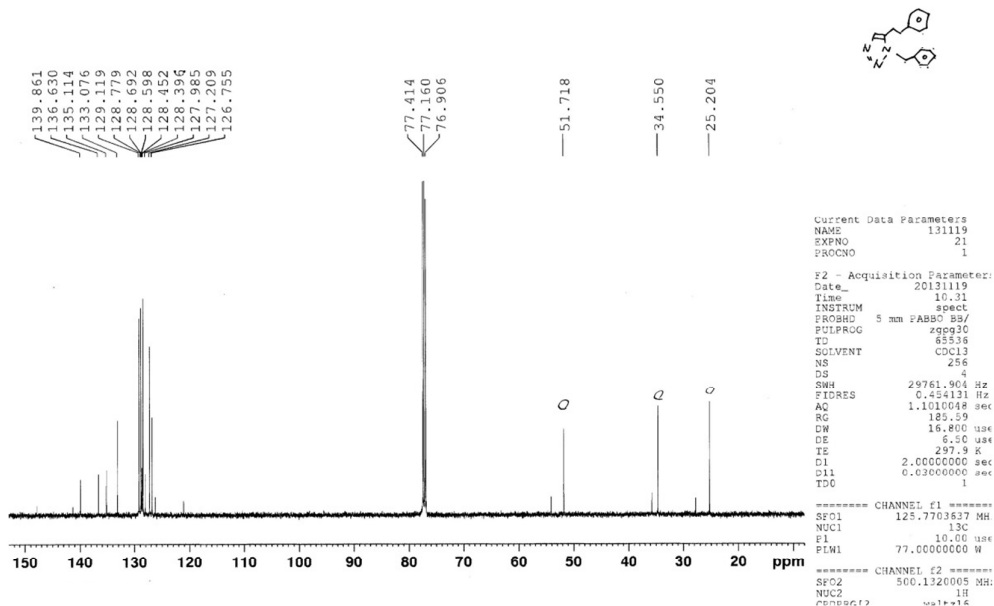
¹³C NMR of 1-Butyl-5-phenylethyl-1H-1,2,3-triazole (9i)



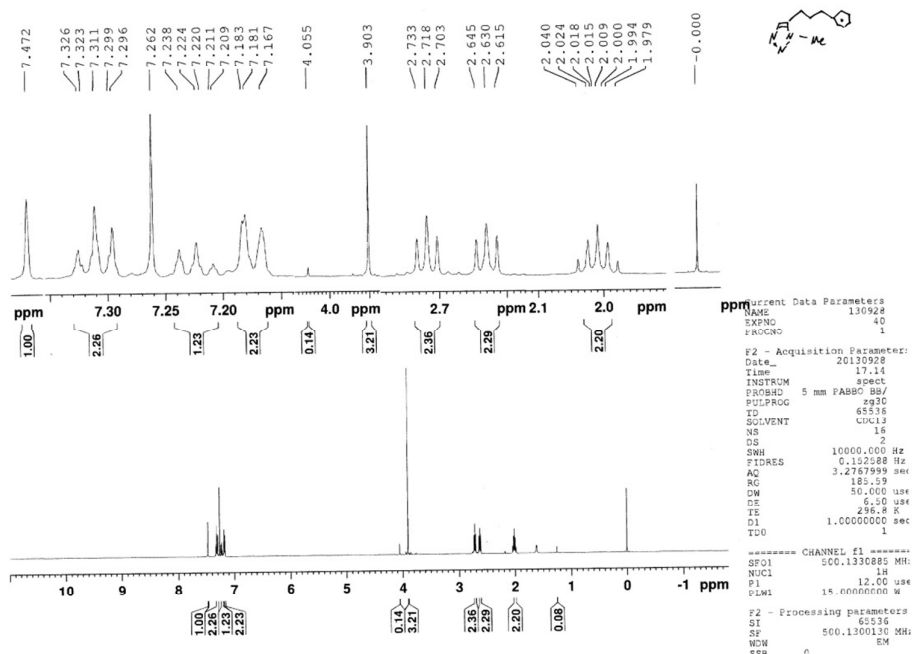
¹H NMR of 1-Benzyl-5-phenylethyl-1H-1,2,3-triazole (9i)



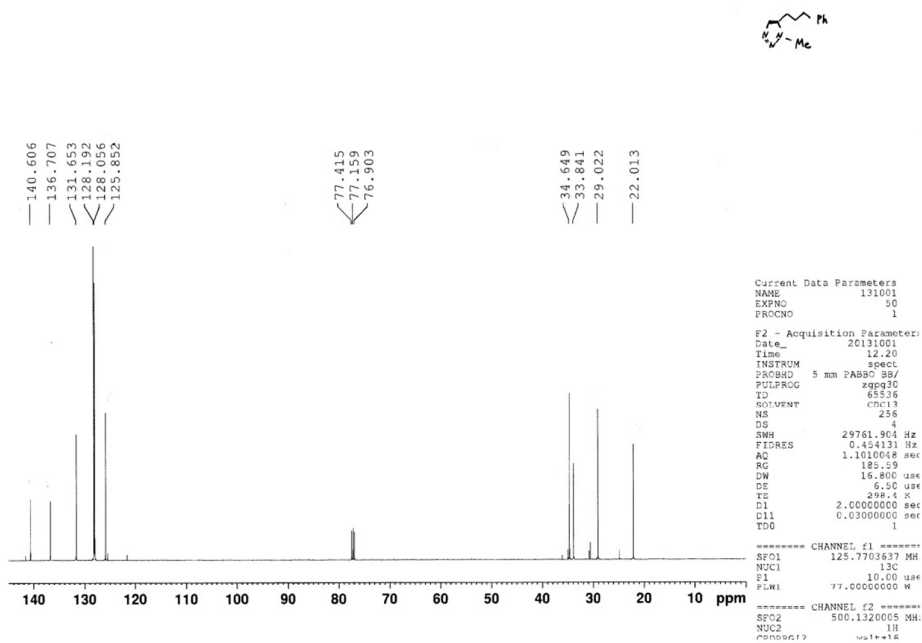
¹³C NMR of 1-Benzyl-5-phenylethyl-1H-1,2,3-triazole (9i)



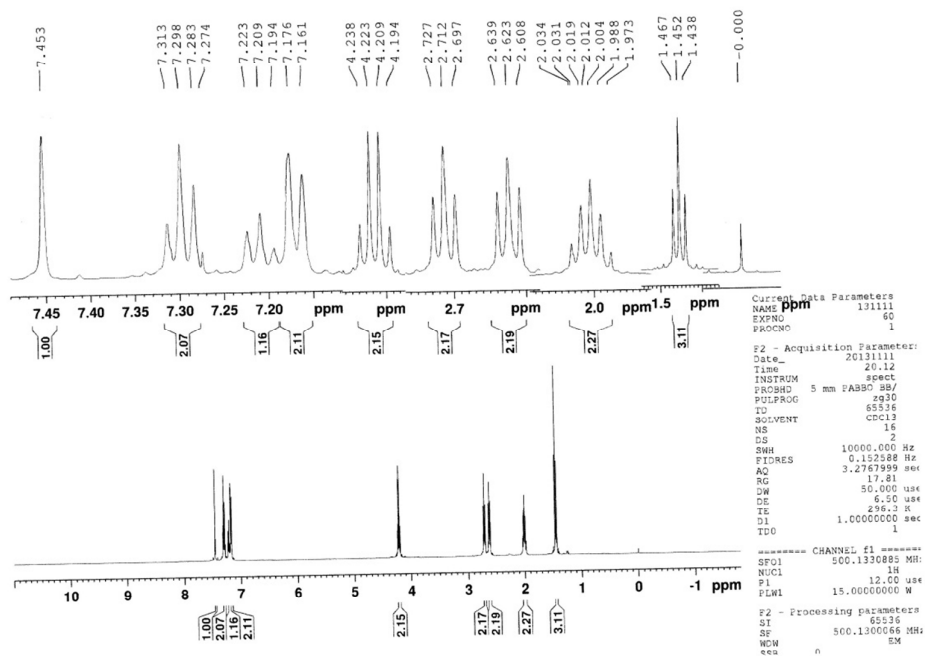
¹H NMR of **1-Methyl-5-(phenylpropyl)-1H-1,2,3-triazole (9k)**



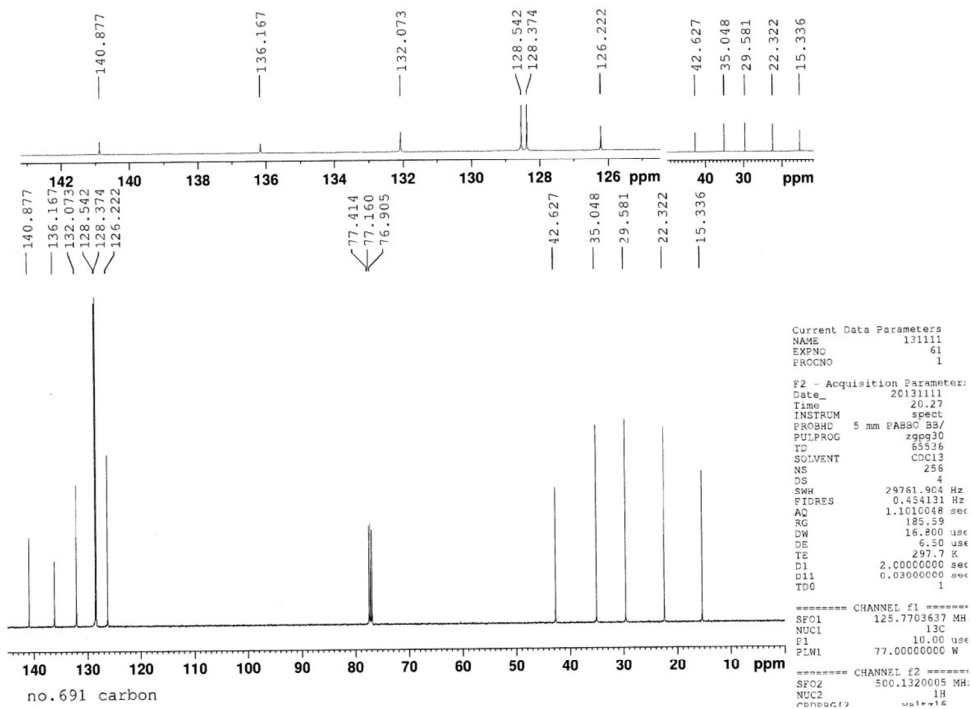
¹³C NMR of **1-Methyl-5-(phenylpropyl)-1H-1,2,3-triazole (9k)**



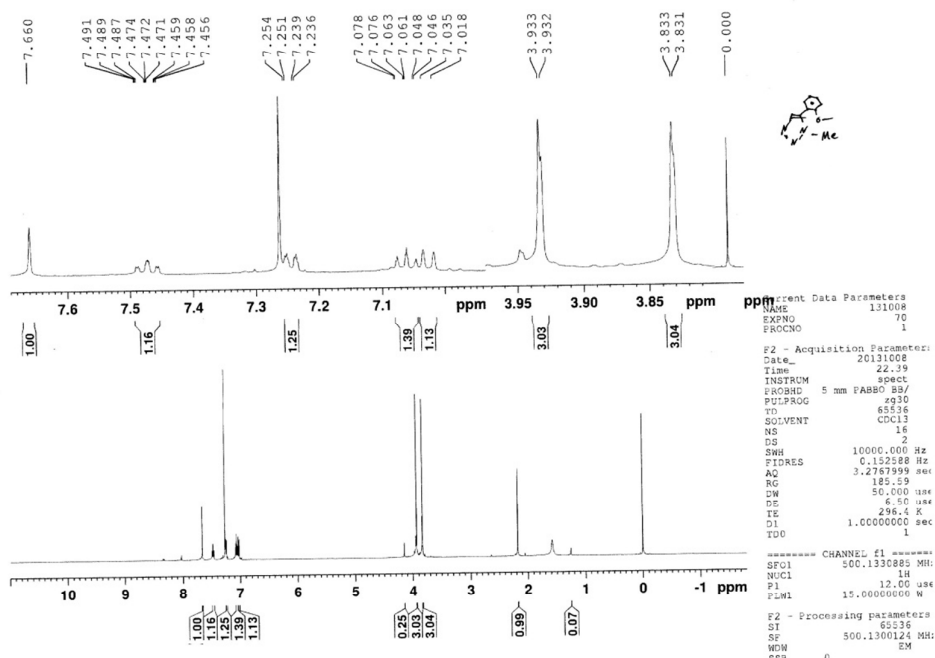
¹H NMR of **5-Phenylpropyl-1-ethyl-1H-1,2,3-triazole (9l)**



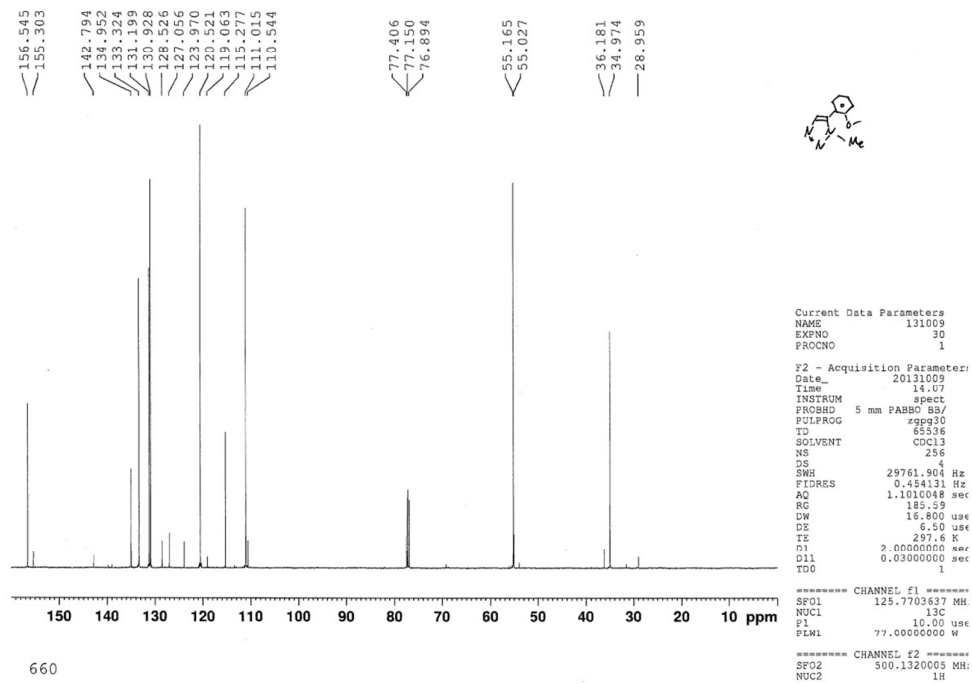
¹³C NMR of **5-Phenylpropyl-1-ethyl-1H-1,2,3-triazole (9l)**



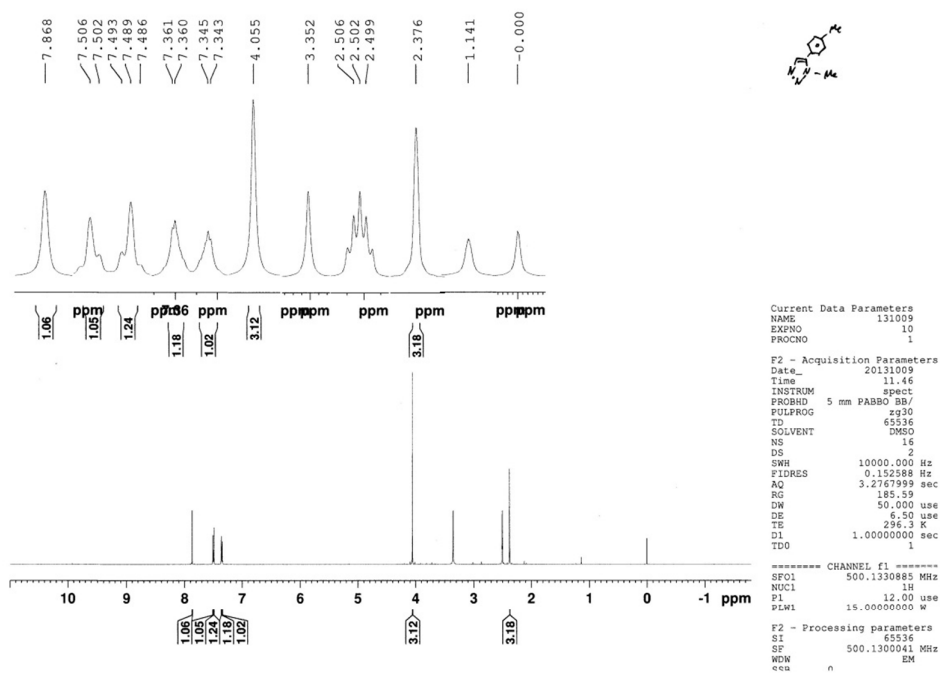
¹H NMR of **5-(2-Methoxy-phenyl)-1-methyl-1H-1,2,3-triazole (9n)**



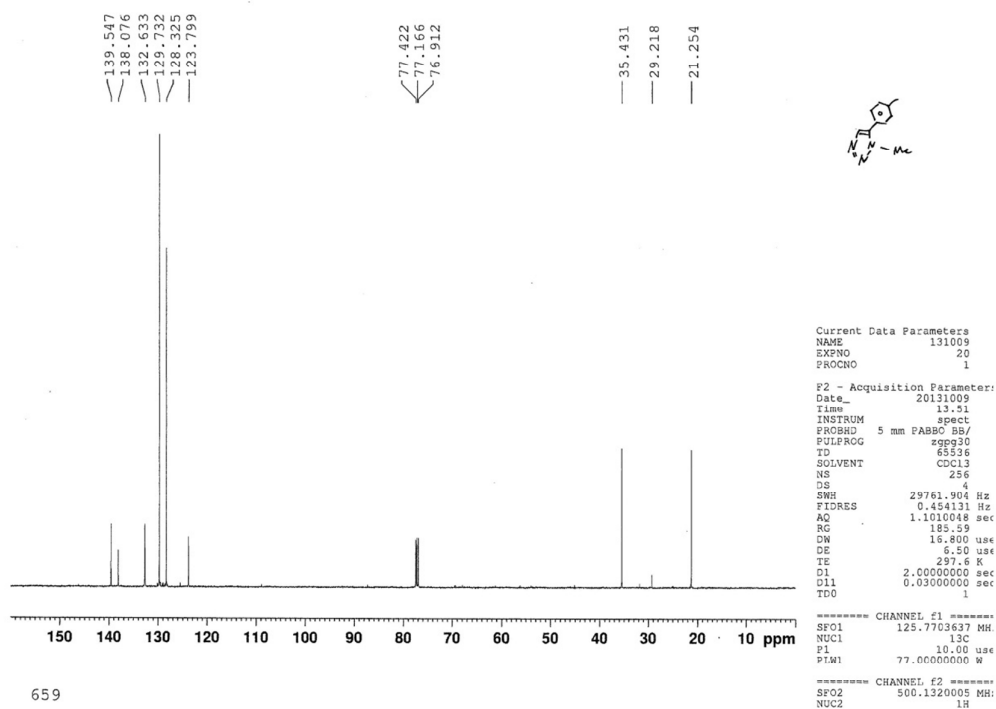
¹³C NMR of **5-(2-Methoxy-phenyl)-1-methyl-1H-1,2,3-triazole (9n)**



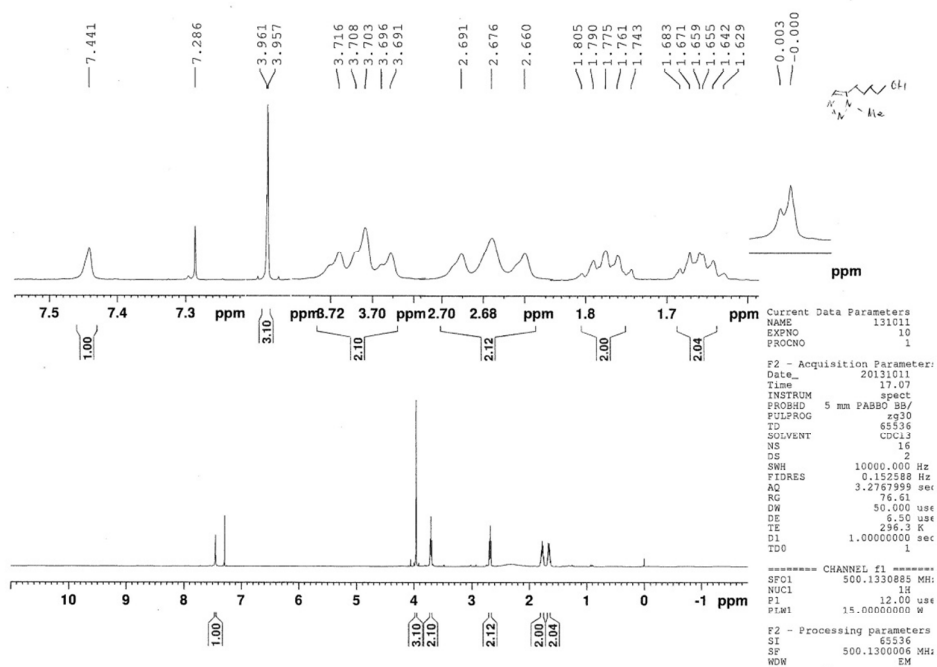
¹H NMR of 5-(4-Methyl-phenyl)-1-methyl-1H-1,2,3-triazole (9o)



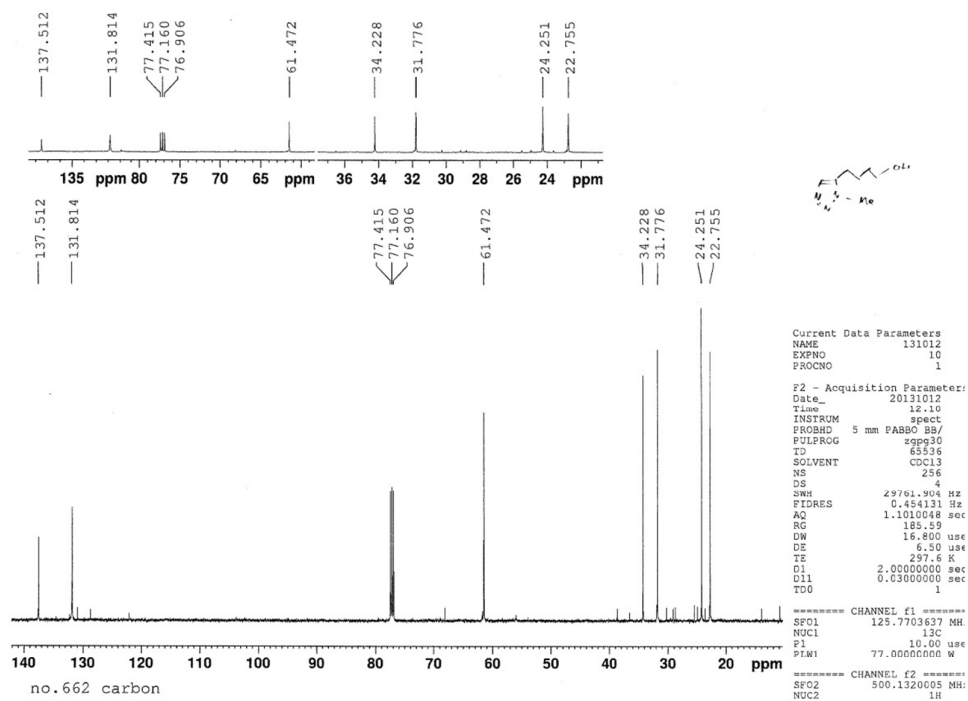
¹³C NMR of 5-(4-Methyl-phenyl)-1-methyl-1H-1,2,3-triazole (9o)



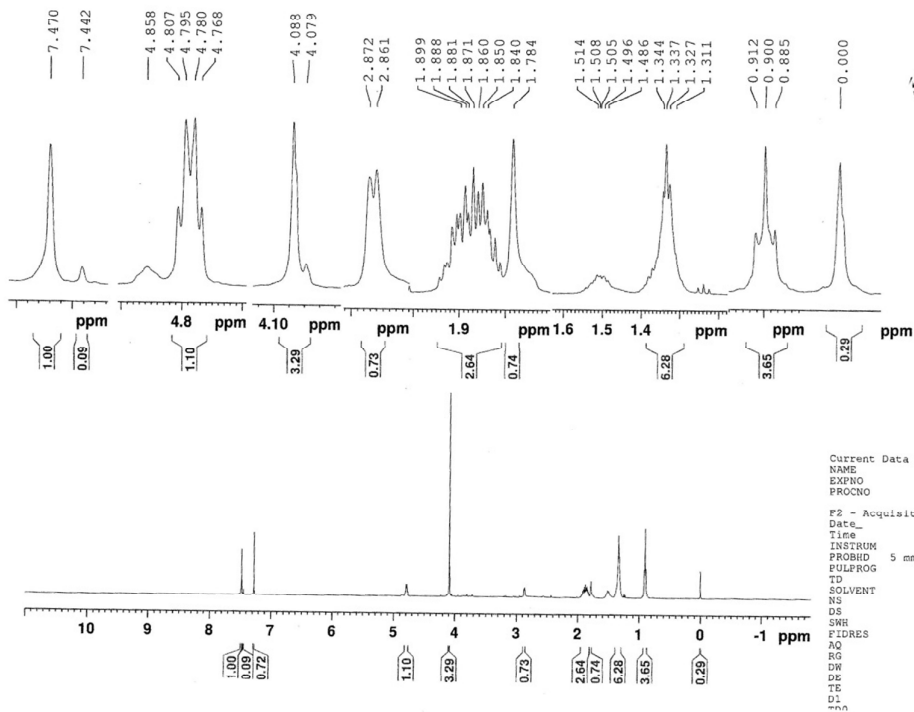
¹H NMR of **5-(Butyl-4-ol)-1-methyl-1H-1,2,3-triazole (9p)**



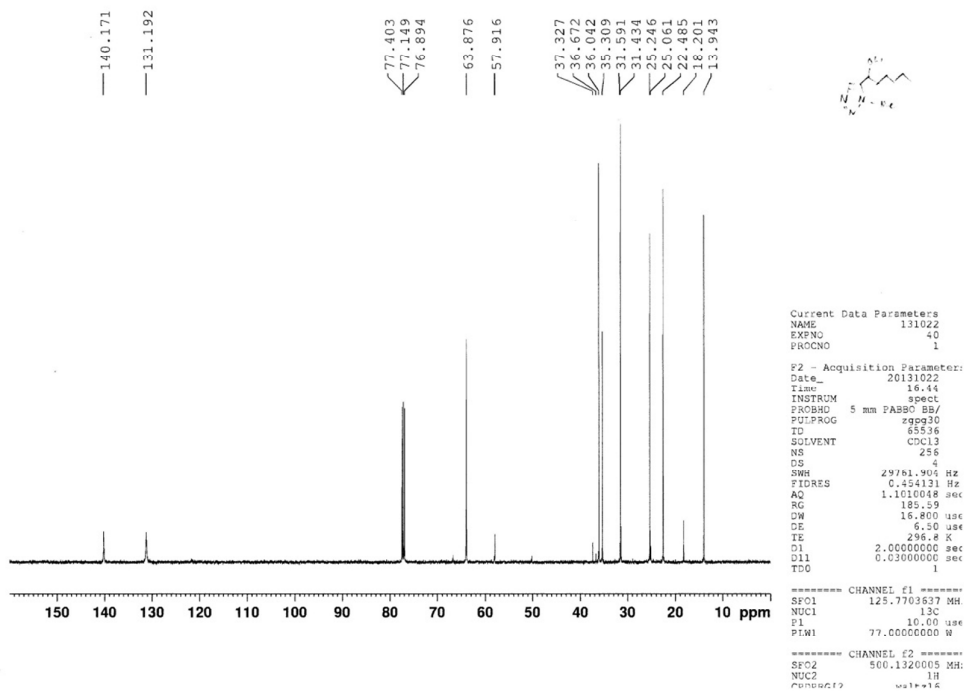
¹³C NMR of **5-(Butyl-4-ol)-1-methyl-1H-1,2,3-triazole (9p)**



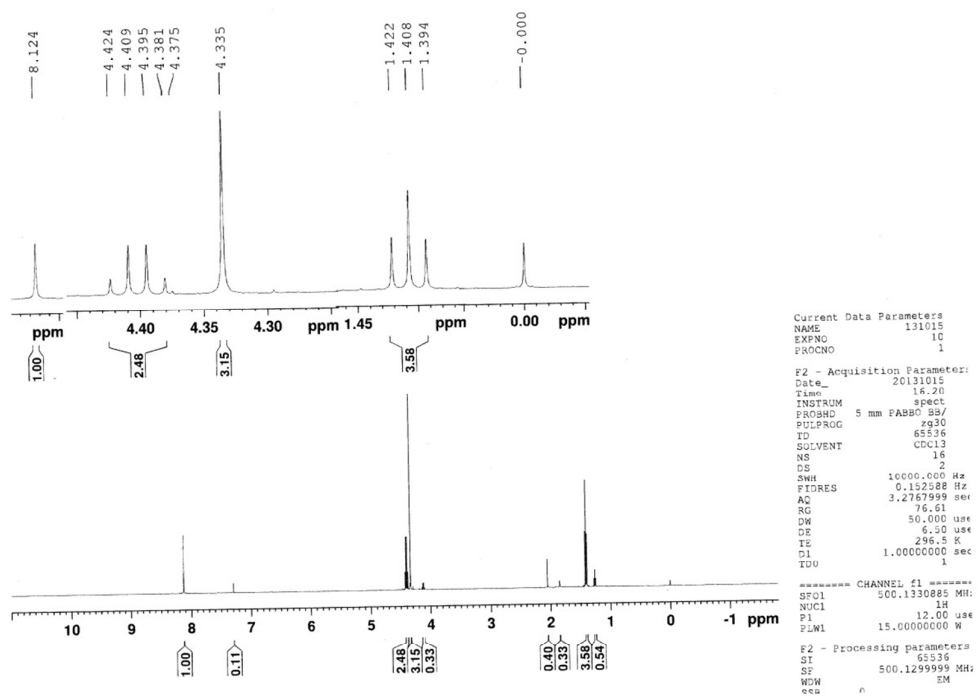
¹H NMR of **5-(Hexyl-1-ol)-1-methyl-1H-1,2,3-triazole (9q)**



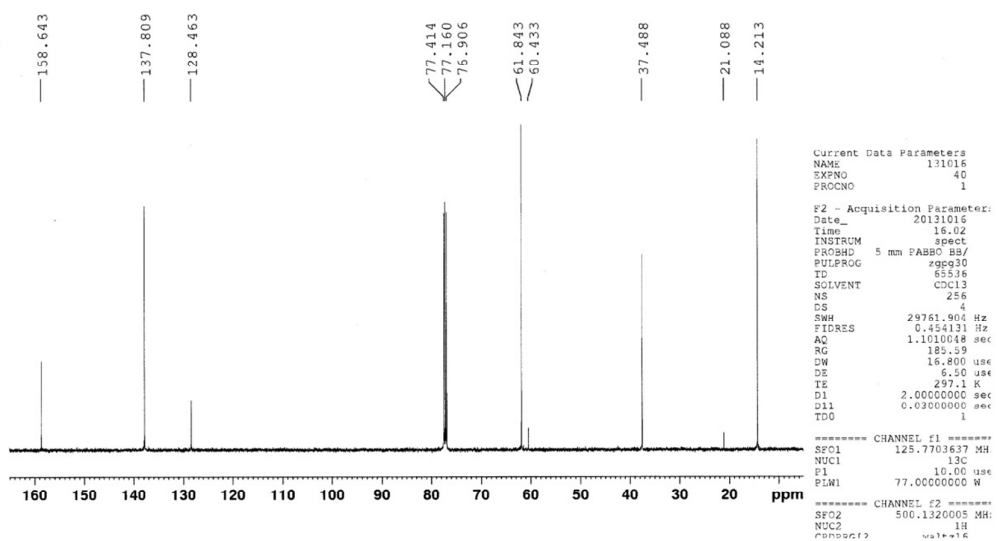
¹³C NMR of **5-(Hexyl-1-ol)-1-methyl-1H-1,2,3-triazole (9q)**



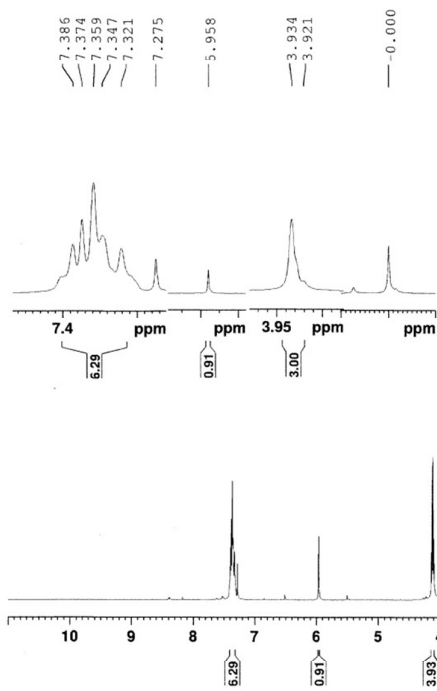
¹H NMR of 5-(Ethylcarboxylate)-1-methyl-1H-1,2,3-triazole (9r)



¹³C NMR of 5-(Ethylcarboxylate)-1-methyl-1H-1,2,3-triazole (9r)



¹H NMR of 5-[1-(Phenyl)-methyl-1-ol]-1-methyl-1H-1,2,3-triazole (9s)



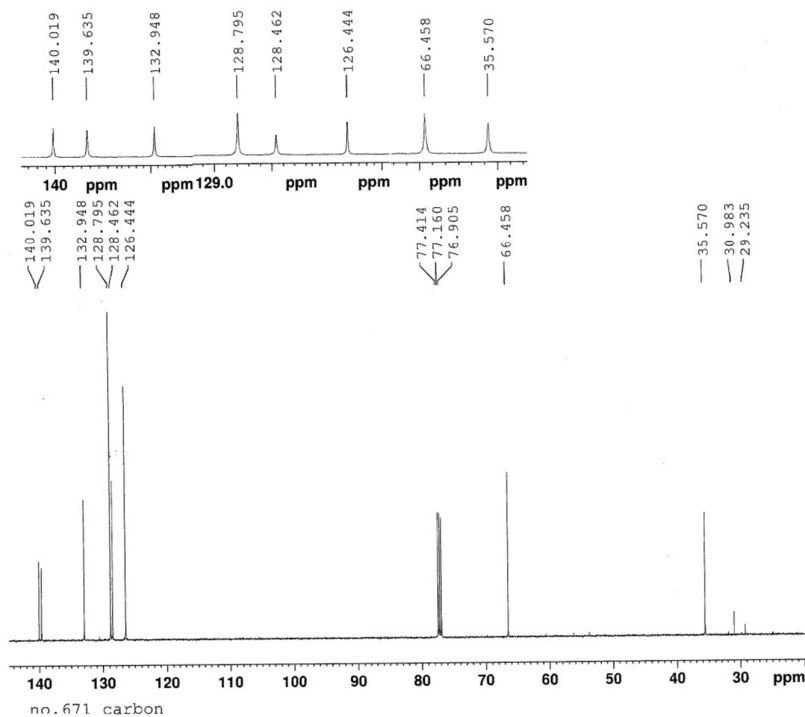
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 NUC1 1H
 P1 12.00 usec
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F2 - Processing parameters:
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¹³C NMR of 5-[1-(Phenyl)-methyl-1-ol]-1-methyl-1H-1,2,3-triazole (9s)



Current Data Parameters
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 PLW1 77.00000000 W

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