Supplementary materials

Imidazolium chloride-Co(III)-*salen-type* complex immobilized on Fe_3O_4@SiO_2 magnetic core-shell nanoparticles as a highly active bi-functional and recyclable nanocatalyst for the copper-, phosphine- and base free Heck-Mozoroki and Sonogashira- Hagihara coupling reactions

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Activity of the catalyst was expressed as turn over frequency (TOF) and calculated from the moles of substrate converted per mole of Co contained in the catalyst using following equation (1):

$$TOF = \frac{\text{moles (Converted substrate)}}{\text{moles of Co (Active site)} \times \text{reaction time (h)}} \qquad (1)$$

Determination of Mn that leached into the solution during the reaction

According to our observations and based on the previously proposed mechanisms, manganese is converted to manganese (II) salt during the reactions (According to the XPS analysis of the mixture). For the coupling reactions, 0.005 mmol Mn has been used, which is activated with 0.65 mmol TFAA (50 μL). This amount was used to prevent contamination of the magnetic heterogeneous catalyst with the metal Mn. Nevertheless, some Mn are found as a metallic form in the mixture (According to XPS analysis of the mixture, Fig. 9), which may be transformed to $MnCl_2$ in the presence of the generated HCl during the reaction (Reaction equation 1, Scheme 3 & 4).

$$Mn_{(s)} + 2HCl_{(aq)} \longrightarrow MnCl_{2\,(aq)} + H_2 \qquad (1)$$

The reaction of Mn with TFAA not only activates the Mn metal for the coupling reactions, but also provides $Mn(O_2CCF_2)$ (*Chemische Berichte*, *124*(3), 515-517). By extraction of the mixture with Et_2O (Experimental part), all manganese is transferred to the aqueous phase. Upon this hypothesis, ICP analysis was carried out for determining of the Mn present in the aqueous phase for the reaction of iodobenzene and *n*-butyl acrylate in the presence of the catalyst **9** (10 mg) and

Mn (0.005 mmol) in EtOH at reflux condition. After completion of the reaction and separation of the catalyst, the ICP results revealed 0.0048 mmol is present in the mixture, that is equal to the total Mn used in the reaction. Moreover, during extraction of the product, the ICP analysis of the organic layer showed no trace of manganese (Experimental section, General procedure for catalytic Heck and Sonogashira reaction catalyzed by Fe_3O_4@SiO_2@Im[Cl]Co(III)-melamine nanocomposite). These results suggest that all manganese used in the reaction has been converted during the reaction to their corresponding Mn salts and transferred to the aqueous phase.
Assuming that $MnCl_2$ is produced, it can be titrated with $AgNO_3$ to produce AgCl sediment (chemical equation 1). We carried out this experiment, which was in line with our expectations. The amount of the precipitated AgCl was approximately 0.0004 mmol. So, according to the chemical reaction (1), there is 0.0002 mmol Mn metal in the mixture. With these results, (0.005-0.0002=0.0048) mmol Mn is leached to the reaction.

$$MnCl_{2\ (aq)} + 2AgNO_{3\ (aq)} \longrightarrow 2AgCl_{\ (s)} + Mn(NO_3)_{2\ (aq)} \qquad (2)$$

Determination of metal loading for compound 8 and its leaching study over the Heck model reaction

The experiments indicated that 0.4 mmol of Co metal per gram of the catalyst was loaded on the compound **8**. Therefore, the catalyst reusability of **8** was checked in the Mizoroki–Heck reaction of iodobenzene (1.0 mmol) and *n*-butyl acrylate (1.3 mmol) in the presence of 0.005 mmol Mn catalyzed by **8** (10 mg). At the end of each reaction, the catalyst **8** was separated, using an external magnetic field, washed with ethanol and reused. The following figure shows the metal leaching of **8**. As could be seen in the figure, the metal leaching in compound **8** is noticeably larger than the catalyst **9**. This reduction in the amount of leaching and the increase in stability may be attributed to the presence of melamine as well as imidazolium moiety (*Applied Catalysis A: General*, *491*, 106-115; *Catalysis Science & Technology*, *5*(4), 2092-2102) in the catalyst. The amount of leaching was 0.35 % in the first run.

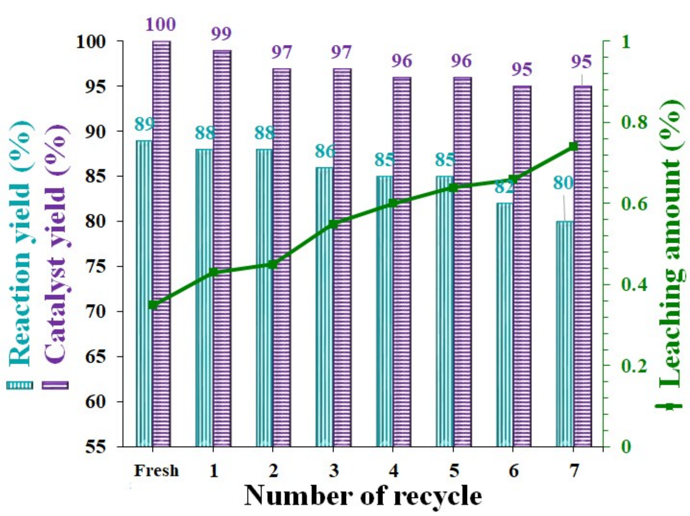


Figure S1 Recyclability and leaching study for compound **8** in the Heck model reaction

*Reaction conditions: iodobenzene (1.0 mmol), n-butyl acrylate (1.3 mmol), Fe_3O_4@SiO_2@Im[Cl]Co(III)-Schiff base complex **8** (10 mg, 0.4 mol% Co), Mn (0.005 mmol), EtOH (3.0 mL), reflux.*

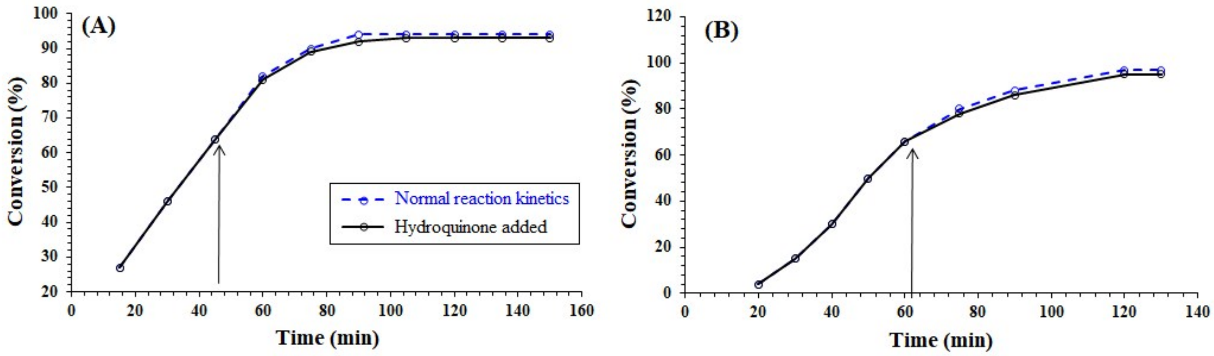
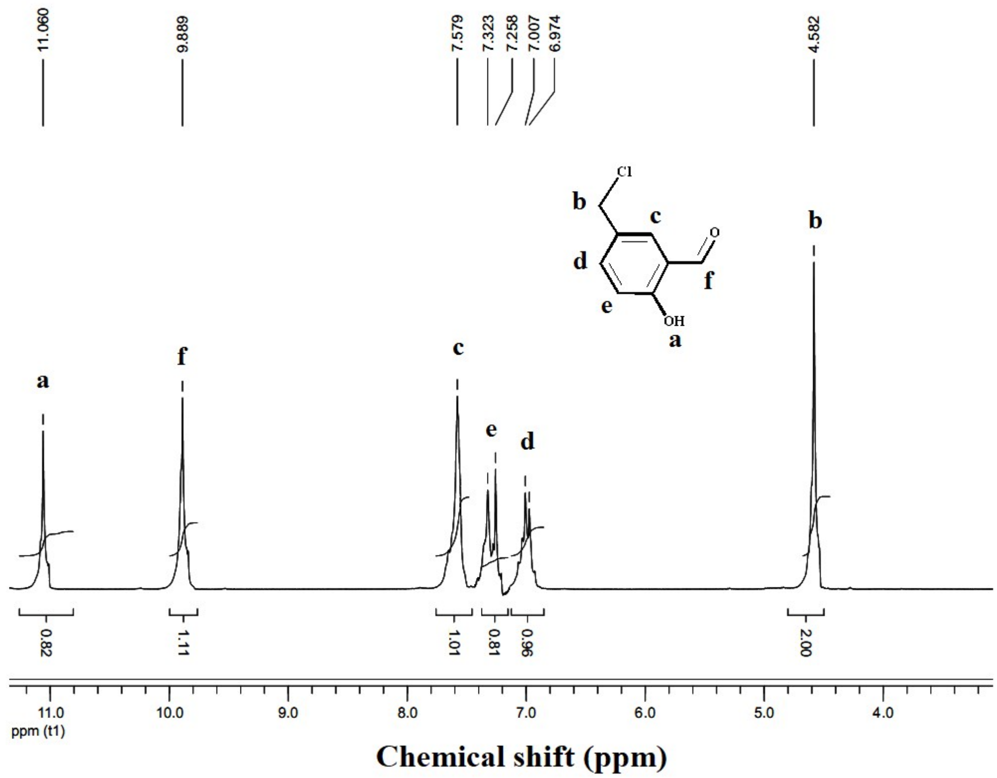
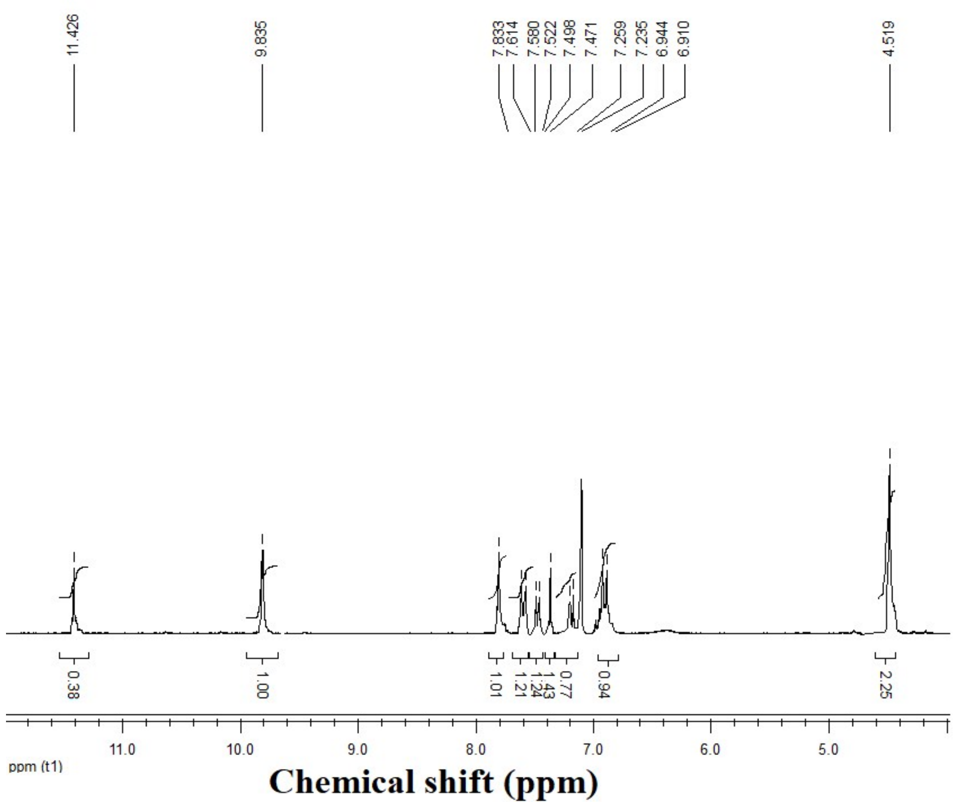


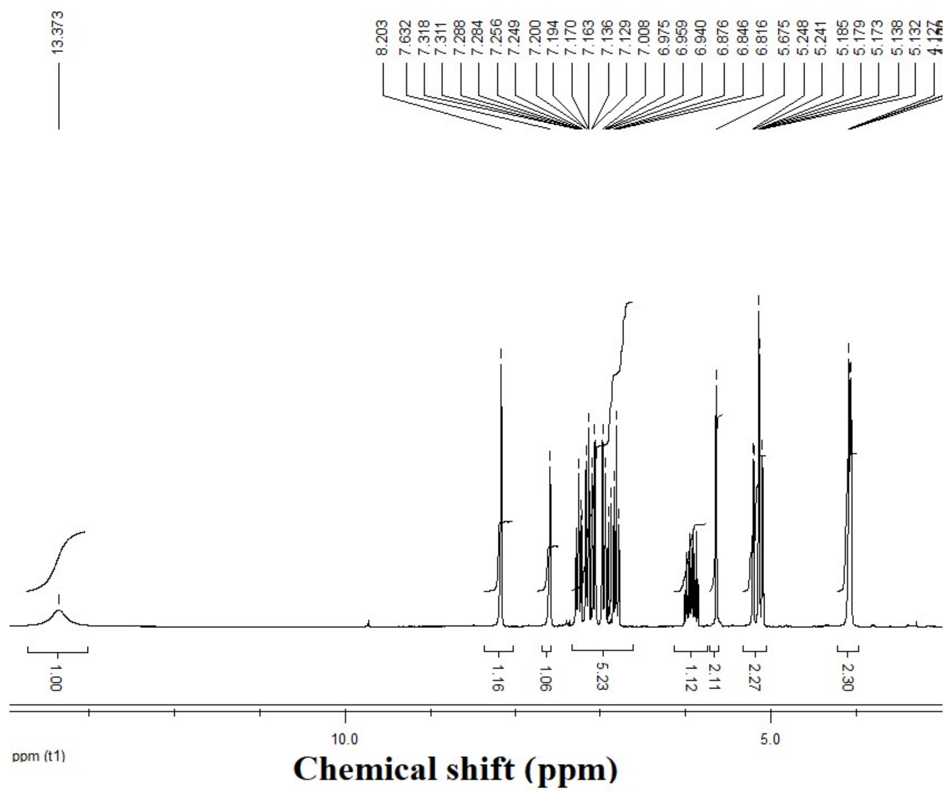
Figure S2 Kinetic plot and effect of hydroquinone as a radical scavenger over the C-C coupling reaction of iodobenzene with (a) *n*-butyl acrylate, and (b) phenyl acetylene catalyzed by Fe_3O_4@SiO_2@Im[Cl]Co(III)-melamine nanocomposite in EtOH/Reflux.



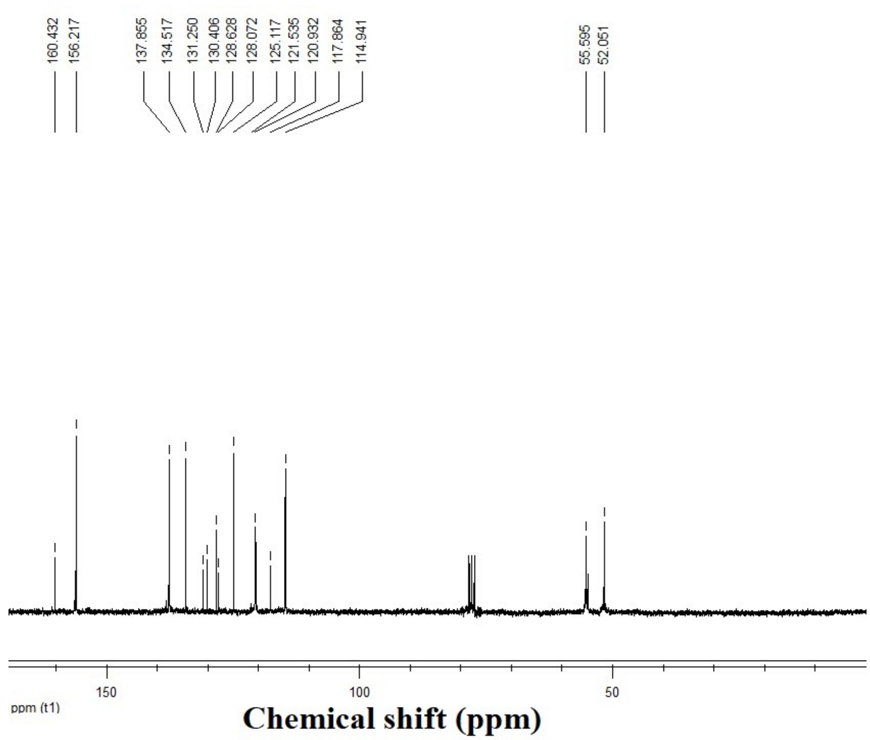
1HNMR spectrum of 2-Hydroxy-5-chloromethyl benzaldehyde (**2**)



1HNMR spectrum of 2-hydroxy-5-imidazole benzaldehyde (**3**)

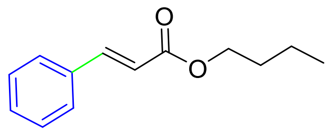


[1]HNMR spectrum of 4-((1H-imidazol-1-yl)methyl)-2-((allylimino)methyl)phenol (**4**)

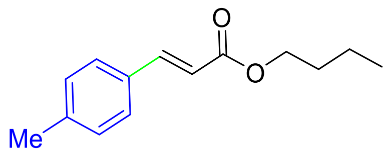


[13]CNMR spectrum of 4-((1H-imidazol-1-yl)methyl)-2-((allylimino)methyl)phenol (**4**)

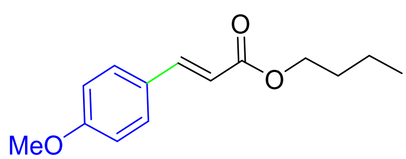
Characterization of some selected compounds:



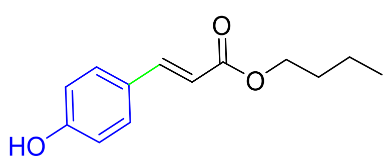
^{1}H-NMR (250 MHz, $CDCl_3$) δ: 0.90 (t, 3H, J= 7.5 Hz), 1.37 (m, 2H), 1.65 (m, 2H), 4.17 (t, 2H, J= 6.7 Hz), 6.53 (d, 1H, J= 16 Hz), 7.60 (d, 2H, J= 8.7 Hz), 7.63 (d, 1H, J= 16.2 Hz), 8.18 (d, 2H, J= 8.7 Hz) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$) δ: 13.7, 19.1, 30.6, 64.8, 122.5, 124.1, 128.5, 140.5, 141.5, 166.0 ppm.



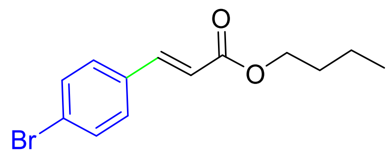
^{1}H-NMR (250 MHz, $CDCl_3$) δ: 0.85 (t, 3H, J= 4.7 Hz), 1.32 (m, 2H), 1.56 (m, 2H), 2.24 (s, 3H), 4.09 (t, 2H, J= 5.0 Hz), 6.30 (dd, 1H, J= 16.0 Hz, J'= 5.9 Hz), 7.06 (m, 2H), 7.29 (m, 2H), 7.52 (dd, 1H, J= 18.2 Hz, J'= 5.5 Hz) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$) δ: 13.7, 19.2, 21.4, 30.8, 64.2, 117.1, 128.3, 129.5, 131.7, 140.5, 144.5, 167.2 ppm.



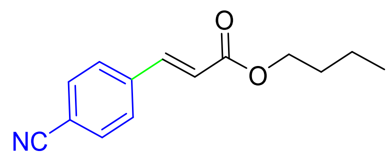
^{1}H NMR (250 MHz, $CDCl_3$) δ: 0.89 (t, 3H, J= 7.5 Hz), 1.35 (m, 2H, J= 7.5 Hz), 1.61 (quint, 2H, J= 5.0 Hz), 3.76 (s, 3H), 4.13 (t, 2H, J= 6.7 Hz), 6.24 (d, 1H, J= 16.0 Hz), 6.83 (d, 2H, J= 5.0 Hz), 7.40 (d, 2H, J= 5.0 Hz), 7.56 (d, 1H, J= 16.0 Hz) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$) δ: 13.8, 19.2, 30.8, 55.36, 64.3, 114.3, 115.8, 129.7, 144.2, 161.3 ppm.



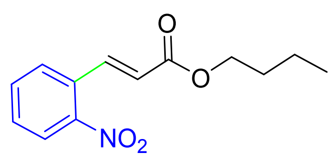
^{1}H-NMR (250 MHz, $CDCl_3$) δ: 0.87 (t, 3H, J= 7. 5 Hz), 1.33 (m, 2H), 1.52 (m, 2H), 4.13 (t, 2H, J= 7.5 Hz), 6.20 (d, 1H, J= 12.5 Hz), 6.80 (d, 3H, J=7.5 Hz), 7.32 (d, 2H, J=10 Hz), 7.55 (d, 1H, J=15 Hz) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$) δ: 13.7, 19.1, 30.7, 64.7, 116.0 (2C), 126.5, 130.0 (2C), 145.1, 158.6, 168.4 ppm.



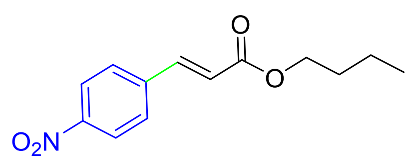
^{1}H-NMR (250 MHz, $CDCl_3$) δ: 0.87 (t, 3H, *J*= 7.5 Hz), 1.34 (m, 2H), 1.60 (m, 2H), 4.12 (t, 2H, *J*= 7.5 Hz), 6.33 (d, 1H, *J*= 17.5 Hz), 7.19-7.30 (m, 2H), 7.40-7.43 (m, 2H), 7.51 (d, 1H, *J*= 17.5 Hz) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$) δ: 13.7, 19.1, 30.7, 64.5, 111.9, 124.4, 129.3 (2C), 132.0 (2C), 133.3, 143.1, 166.7 ppm.



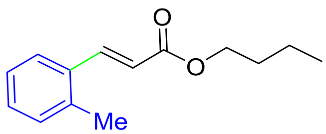
^{1}H-NMR (250 MHz, $CDCl_3$) δ: 0.89 (t, 3H, *J*= 7.2 Hz), 1.35 (m, 2H), 1.64 (m, 2H), 4.16 (t, 2H, *J*= 6.5 Hz), 6.48 (d, 1H, *J*= 16 Hz), 7.59, (m, 5H) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$) δ: 13.6, 19.1, 27.8, 30.6, 64.7, 113.2, 118.3, 121.8, 128.3, 132.5, 138.5, 142.0, 166.1 ppm.



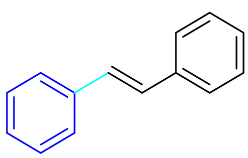
^{1}H-NMR (250 MHz, $CDCl_3$) δ: 0.88 (t, 3H, *J*= 7.5 Hz), 1.35 (m, 2H), 1.62 (m, 2H), 4.14 (t, 2H, *J*= 7.5 Hz), 6.29 (d, 1H, *J*= 17.5 Hz), 7.42-7.57 (m, 2H), 7.94-7.97 (m, 2H), 8.02 (d, 1H, *J*= 17.5 Hz) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$) δ: 13.9, 19.1, 30.6, 64.7, 123.3, 124.6, 129.2, 130.2, 130.3, 133.5, 139.7, 148.2, 165.9 ppm.



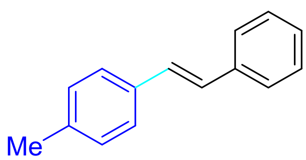
^{1}H-NMR (250 MHz, $CDCl_3$) δ: 0.97 (t, 3H, *J*= 7.2 Hz), 1.45 (m, 2H), 1.67 (m, 2H), 4.23 (t, 2H, *J*= 7.5 Hz), 6.54 (dd, 1H, *J*= 16.0 Hz, *J′*= 6.2 Hz), 7.67 (m, 3H), 8.24 (m, 2H) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$) δ: 13.7, 19.1, 30.6, 64.9, 122.5, 124.1, 128.6, 140.5, 141.5 ppm.



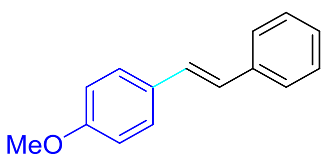
^{1}H-NMR (250 MHz, $CDCl_3$) δ: 0.85 (t, 3H, J= 2.5 Hz), 1.32 (m, 2H), 1.57 (m, 2H), 2.30 (s, 3H), 4.11 (t, 2H), 6.27 (dd, 1H, J= 15.9 Hz, J'= 4.8 Hz), 7.11 (m, 3H), 7.41 (m, 1H), 7.84 (dd, 1H, J= 13.7 Hz, J'= 4.3 Hz) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$) δ: 13.7, 18.9, 19.7, 30.7, 64.3, 119.2, 126.3, 129.9, 130.7, 133.4, 137.5, 142.2, 167.0 ppm.



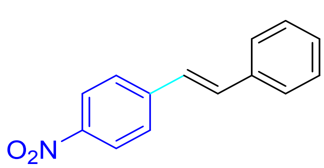
^{1}H-NMR (250 MHz, $CDCl_3$) δ: 7.13 (d, 2H), 7.31 (m, 6H), 7.52 (m, 4H) ppm.



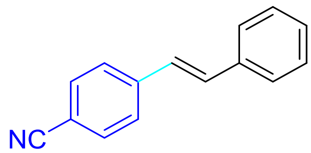
^{1}H-NMR (250 MHz, $CDCl_3$) δ: 2.26 (s, 3H), 6.91-7.64 (m, 9H) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$,) δ: 21.3, 126.4, 126.4, 127.4, 127.7, 128.6, 129.4, 134.5, 137.5 ppm.



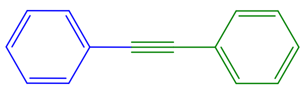
^{1}H NMR (250 MHz, $CDCl_3$) δ: 3.77 (s, 3H), 6.80 (d, 2H, J= 8.5 Hz), 6.89 (d, 1H, J= 16.5 Hz), 6.99 (d, 1H, J= 16.0 Hz), 7.18 (t, 1H, J= 6.5 Hz), 7.28 (t, 2H, J= 7.5 Hz), 7.37 (d, 2H, J= 8.5 Hz), 7.42 (d, 2H, J= 7.5 Hz) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$) δ: 57.5, 116.5, 126.0, 126.5, 127.0, 127.5, 128.0, 129.5, 130.9, 138.8, 160.5 ppm.



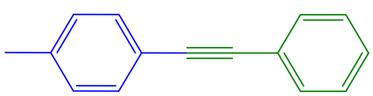
^1H NMR (250 MHz, $CDCl_3$) δ: 7.23 (d, 1H, J= 16.5 Hz), 7.37 (d, 1H, J= 16 Hz), 7.45-7.55 (m, 3H), 7.71 (d, 2H, J= 7.3 Hz), 7.80 (d, 2H, J= 9.3 Hz), 8.40 (d, 2H, J= 9.3 Hz) ppm; ^{13}C NMR (60 MHz, $CDCl_3$) δ: 125.5, 127.0, 128.0, 128.5, 129.0, 130.1, 135.5, 140.5, 149.9, 152.2 ppm.



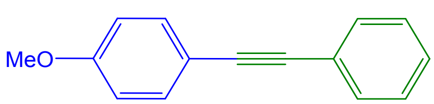
^1H NMR (250 MHz, $CDCl_3$) δ: 7.02 (d, 1H, J= 16.5 Hz), 7.45-7.60 (m, 6H), 7.49 (t, 2H, J= 7.5 Hz), 7.40 (t, 1H, J= 7.45 Hz), 7.20 (d, 1H, J= 16.5 Hz) ppm.



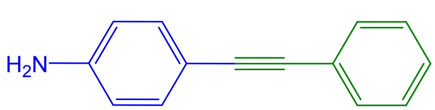
^1H-NMR (250 MHz, $CDCl_3$) δ: 7.23-7.28 (m, 6H), 7.43-7.47 (m, 4H) ppm; ^{13}C-NMR (62.9 MHz, $CDCl_3$) δ: 89.5, 123.3, 128.3, 129.2, 131.6 ppm; MS (m/e) = 178 [M^+]; Elemental Analysis: Calcd. C: 94.33, H: 5.67%, Found. C: 94.11, H: 5.89%.



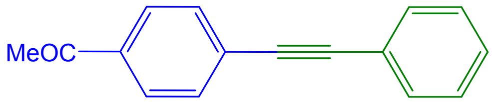
^1H-NMR ($CDCl_3$, 250 MHz) δ: 2.22 (s, 3H, CH_3), 7.14 (d, 2H, J= 8.4 Hz, Ar-H), 7.19-7.42 (m, 7H, Ar-H) ppm; ^{13}C-NMR (62.9 MHz, $CDCl_3$) δ: 21.5, 88.7, 89.6, 120.2, 123.5, 128.1, 128.3, 129.1, 131.5, 131.7, 138.4 ppm; MS (m/e)= 192 [M^+]; Elemental Analysis: Calcd. C: 93.77, H: 6.30%, Found. C: 93.57, H: 6.23%.



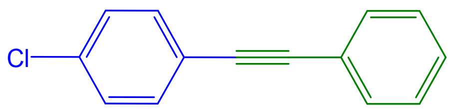
^1H-NMR (250 MHz, $CDCl_3$) δ: 3.76 (s, 3H, CH_3), 6.79 (d, 2H, J= 8.2 Hz, Ar-H), 7.21-7.25 (m, 3H, Ar-H), 7.37-7.44 (m, 4H, Ar-H) ppm; ^{13}C-NMR (62.9 MHz, $CDCl_3$) δ: 55.2, 88.0, 89.4, 114.0, 115.3, 123.6, 127.9, 128.3, 131.4, 133.0, 159.6 ppm; MS (m/e)= 208 [M^+]; Elemental Analysis: Calcd. C: 86.50, H: 5.82%, Found. C: 86.64, H: 5.71%.



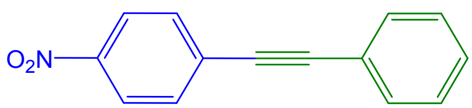
^{1}H-NMR (250 MHz, $CDCl_3$) δ: 3.62 (s, 2H, NH_2), 6.52 (s, 2H, Ar-H), 7.22-7.40 (m, 7H, Ar-H) ppm; ^{13}C-NMR (62.9 MHz, $CDCl_3$) δ: 87.3, 90.2, 112.5, 114.7, 123.9, 127.7, 128.3, 131.3, 132.9, 146.7 ppm.



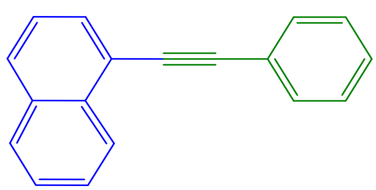
^{1}H-NMR (250 MHz, $CDCl_3$) δ: 2.51 (s, 3H, CH_3), 7.16-7.33 (m, 3H, Ar-H), 7.45-7.54 (m, 4H, Ar-H), 7.86 (d, 2H, J=7.5 Hz, Ar-H) ppm; ^{13}C-NMR (62.9 MHz, $CDCl_3$) δ: 27.0, 88.9, 92.9, 123.0, 128.2, 128.3, 128.8, 129.5, 132.0, 132.2, 136.5, 197.6 ppm.



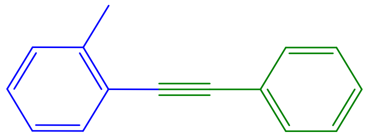
^{1}H NMR (250 MHz, $CDCl_3$) δ: 7.13-7.43 (m, 9H, Ar-H) ppm; ^{13}C NMR (62.9 MHz, $CDCl_3$) δ: 88.2, 90.3, 121.8, 122.9, 128.4, 128.5, 128.7, 131.6, 132.8, 134.2 ppm.



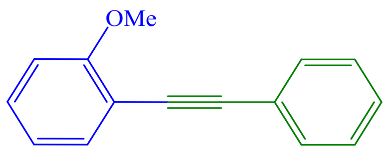
^{1}H-NMR (250 MHz, $CDCl_3$) δ: 7.37-7.41 (m, 3H, Ar-H), 7.54-7.57 (m, 2H, Ar-H), 7.68 (d, 2H, J= 12.5 Hz, Ar-H), 8.22 (d, 2H, J= 7.5 Hz, Ar-H) ppm; ^{13}C-NMR (62.9 MHz, $CDCl_3$) δ: 87.5, 94.7, 122.0, 123.6, 128.5, 129.2, 130.2, 131.8, 132.2, 146.9 ppm; MS (m/e) = 224 [M^+]; Elemental Analysis: Calcd. C: 75.32, H: 4.07, N: 6.27%, Found. C: 75.42, H: 4.21, N: 6.15%.



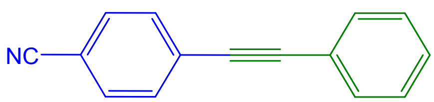
^{1}H-NMR (250 MHz, $CDCl_3$) δ: 7.26-7.74 (m, 11H, Ar-H), 8.36 (d, 1H, J= 8.2 Hz, Ar-H); ^{13}C-NMR (62.9 MHz, $CDCl_3$) δ: 87.5, 94.3, 120.9, 123.4, 125.3, 125.6, 126.2, 126.4, 126.8, 128.3, 128.42, 128.47, 128.8, 130.4, 131.7, 133.2, 133.3, 141.1 ppm; MS (m/e) = 228 [M^+]; Elemental Analysis: Calcd. C: 94.69, H: 5.31%, Found. C: 94.41, H: 5.59%.



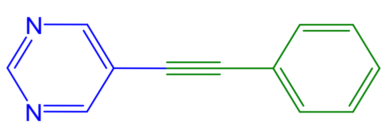
^{1}H NMR (250 MHz, $CDCl_3$) δ: 2.44 (s, 3H, CH_3), 7.14-7.46 (m, 9H, Ar-H) ppm; ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ: 20.7, 86.01, 94.2, 123.0, 125.5, 128.1, 128.30, 128.35, 129.4, 131.5, 131.8, 140.1 ppm; MS (m/e)= 192 [M^+]; Elemental Analysis: Calcd. C: 93.70, H: 6.30%, Found. C: 93.82, H: 6.18%.



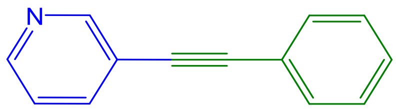
^{1}H NMR (250 MHz, $CDCl_3$) δ: 3.82 (s, 3H, CH_3), 6.87 (d, 2H, J= 7.25 Hz, Ar-H), 7.31-7.33 (m, 3H, Ar-H), 7.45-7.53 (m, 4H, Ar-H) ppm; ^{13}C NMR ($CDCl_3$, 62.9 MHz): δ: 55.8, 85.6, 93.3, 110.6, 112.4, 120.4, 123.5, 128.0, 128.1, 129.7, 131.6, 133.5, 159.8 ppm.



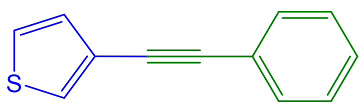
^{1}H-NMR (250 MHz, $CDCl_3$) δ: 7.28-7.31 (m, 3H, Ar-H), 7.44-7.53 (m, 6H, Ar-H) ppm; ^{13}C-NMR (62.9 MHz, $CDCl_3$) δ: 87.7, 93.7, 111.4, 118.5, 122.2, 128.2, 128.5, 129.1, 131.7, 132.03, 132.06 ppm; MS (m/e)= 203 [M^+]; Elemental Analysis: Calcd. C: 88.64, H: 4.47, N: 6.89%, Found. C: 88.77, H: 4.57, N: 6.66%.



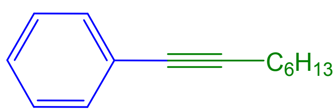
^{1}H-NMR (250 MHz, $CDCl_3$) δ: 7.29-7.48 (m, 5H, Ar-H), 8.77 (s, 2H, Ar-H), 9.06 (s, 1H, Ar-H) ppm; ^{13}C-NMR (62.9 MHz, $CDCl_3$) δ: 82.3, 96.3, 119.9, 121.7, 128.5, 128.7, 129.1, 129.3, 131.7, 139.3, 156.6, 158.5 ppm; MS (m/e)= 180 [M^+]; Elemental Analysis: Calcd. C: 79.98, H: 4.48, N: 15.54%, Found. C: 79.81, H: 4.62, N: 15.57%.



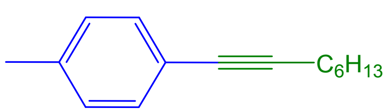
^{1}H-NMR (250 MHz, $CDCl_3$) δ: 7.26-7.29 (m, 4H, Ar-H), 7.44-7.47 (m, 2H, Ar-H), 7.70 (m, 1H, Ar-H), 8.45 (m, 1H, Ar-H), 8.68 (s, 1H, Ar-H) ppm; ^{13}C-NMR (62.9 MHz, $CDCl_3$) δ: 85.9, 92.7, 120.5, 122.4, 123.0, 128.4, 128.8, 131.6, 138.4, 148.4, 152.1 ppm; MS (m/e)= 179 [M^+]; Elemental Analysis: Calcd. C: 87.12, H: 5.07, N: 7.81%, Found. C: 87.27, H: 4.91, N: 7.82%.



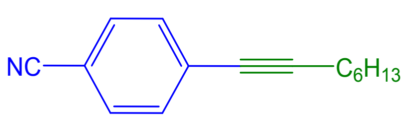
^{1}H-NMR (250 MHz, $CDCl_3$) δ: 7.31-7.42 (m, 5H, Ar-H), 7.59-7.64 (m, 3H, Ar-H) ppm; ^{13}C-NMR (62.9 MHz, $CDCl_3$) δ: 84.7, 89.1, 123.3, 125.5, 127.2, 128.5, 128.7, 129.3, 129.9, 131.7, 132.6 ppm; MS (m/e)= 184 [M^+]; Elemental Analysis: Calcd. C: 78.22, H: 4.39%, Found. C: 78.10, H: 4.46%.



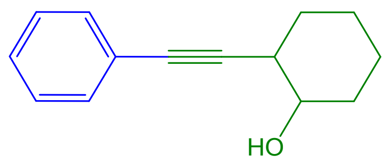
^{1}H-NMR (250 MHz, $CDCl_3$) δ: 0.83 (t, 3H, J= 7.0 Hz, CH_3), 1.20-1.55 (m, 8H, CH_2), 2.31 (t, 2H, J= 7.0 Hz, CH_2), 7.15-7.21 (m, 3H, Ar-H), 7.28-7.33 (m, 2H, Ar-H) ppm; ^{13}C-NMR (62.9 MHz, $CDCl_3$) δ: 14.0, 19.3, 22.5, 28.5, 28.7, 31.3, 80.5, 90.4, 124.0, 127.4, 128.1, 131.5 ppm.



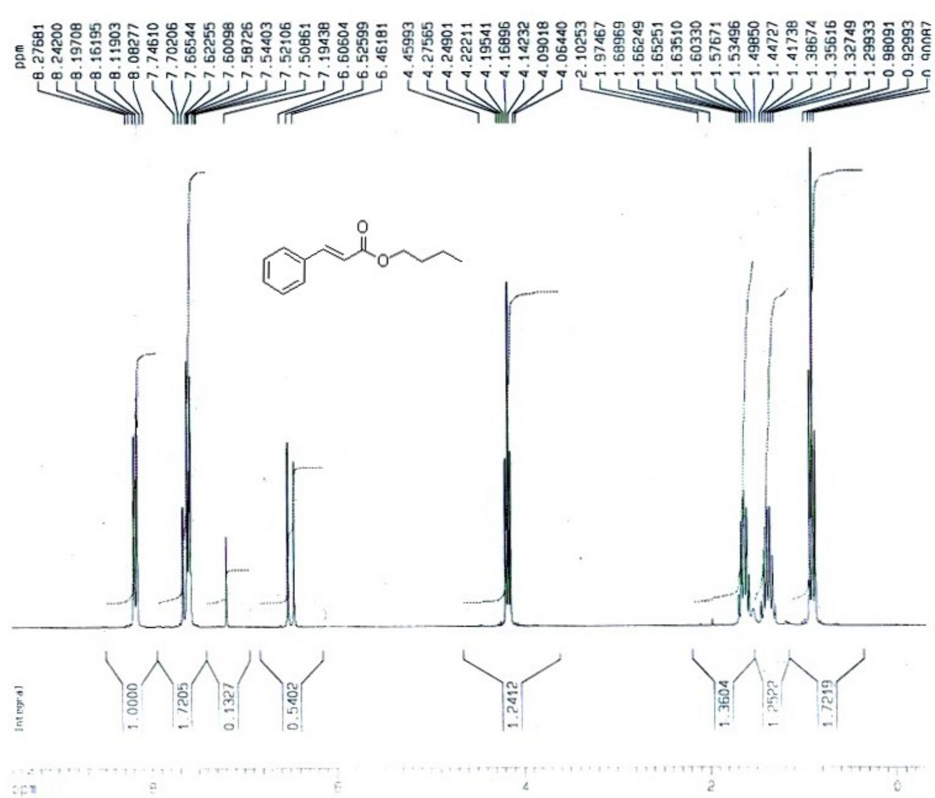
^{1}H-NMR (250 MHz, $CDCl_3$) δ: 0.93 (t, 3H, J= 7.0 Hz, $-CH_2\underline{CH_3}$), 1.31-1.65 (m, 8H, $-CH_2$), 2.35 (s, 3H, CH_3), 2.41 (t, 2H, J= 7.0 Hz, CH_2), 7.10 (d, 2H, J= 8.5 Hz, Ar-H), 7.31 (d, 2H, J= 8.0 Hz, Ar-H) ppm; ^{13}C-NMR (62.9 MHz, $CDCl_3$) δ: 14.0, 19.4, 21.3, 22.5, 28.6, 28.7, 31.3, 80.5, 89.5, 121.0, 128.8, 131.3, 137.3 ppm.



^{1}H-NMR (250 MHz, $CDCl_3$) δ: 0.89 (t, 3H, J= 7.0 Hz, $-CH_2\underline{CH_3}$), 1.24-1.63 (m, 8H, $-CH_2$), 2.41 (t, 2H, J= 7.0 Hz, CH_2), 7.42 (d, 2H, J = 8.75 Hz, Ar-H), 7.56 (d, 2H, J= 8.75 Hz, Ar-H) ppm.



^{1}H-NMR (250 MHz, $CDCl_3$) δ: 1.27-1.29 (s, 1H, OH), 1.55-1.77 (m, 7H, $-CH_2$), 1.98-2.04 (m, 2H, $-CH_2$), 2.27 (s, 1H, CH_2), 7.26-7.31 (m, 3H, Ar-H), 7.40-7.45 (m, 2H, Ar-H) ppm.



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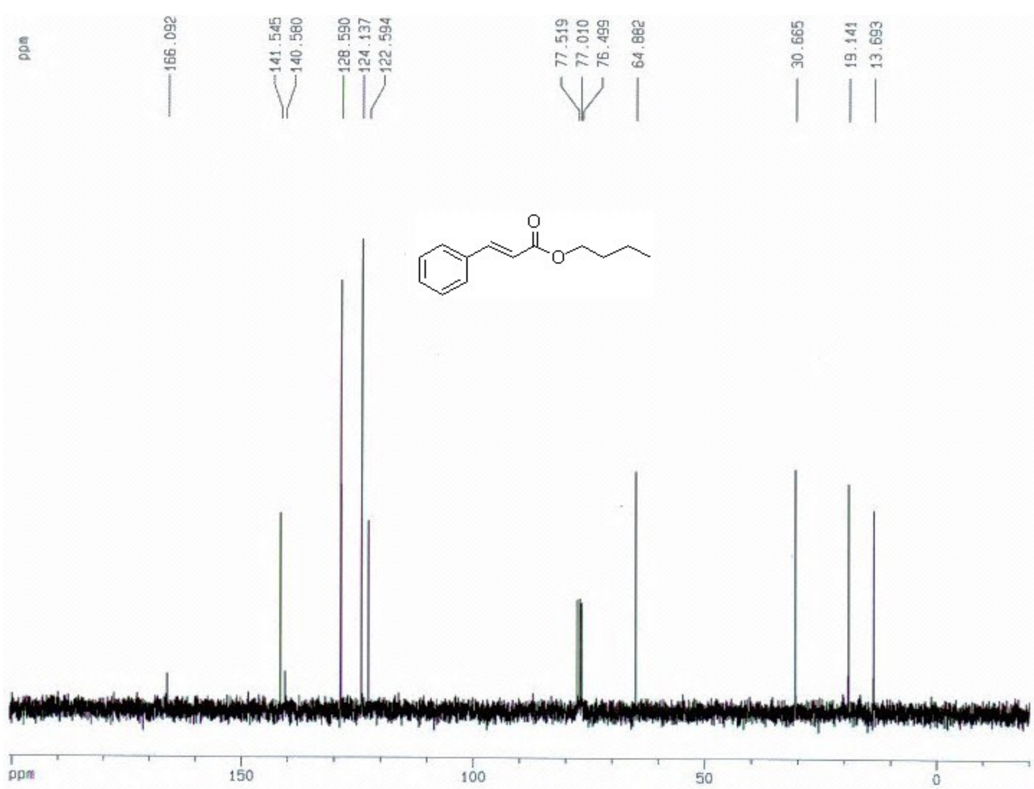

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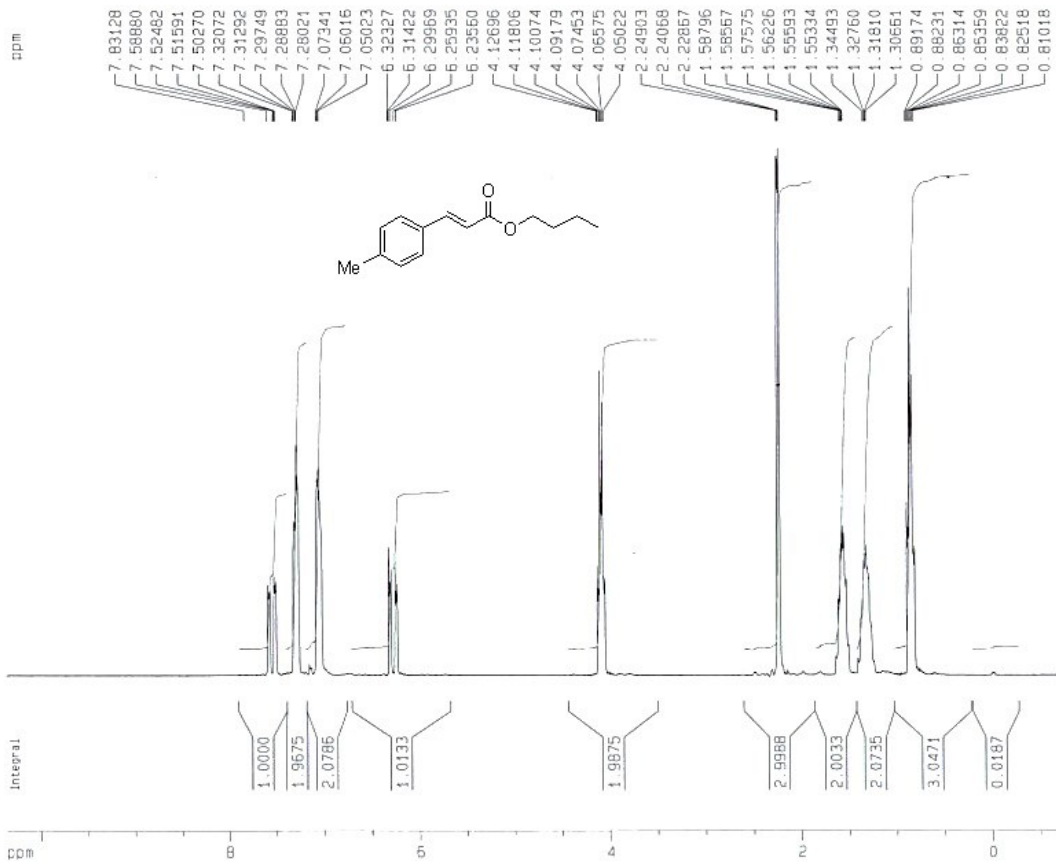

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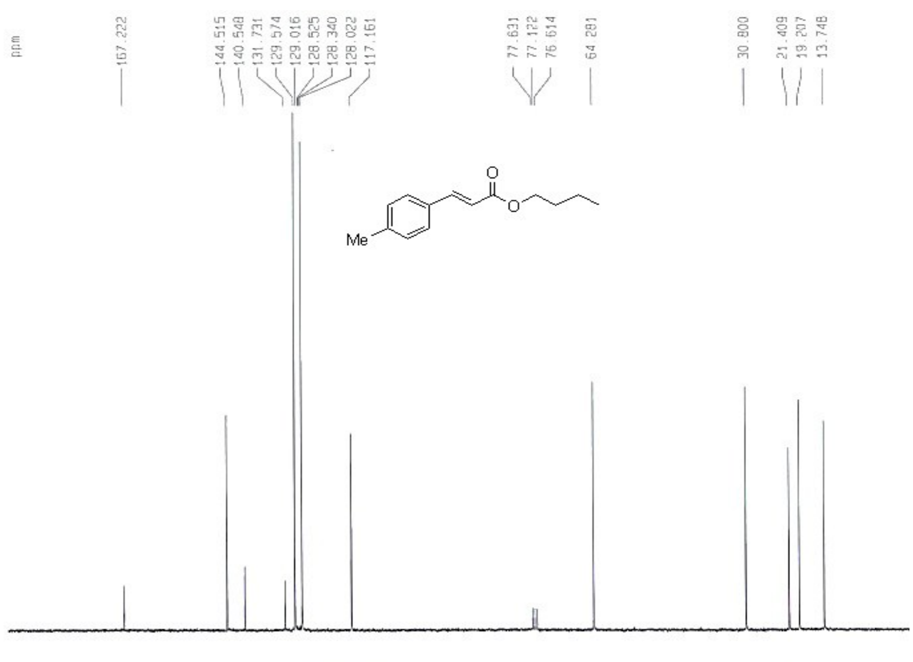

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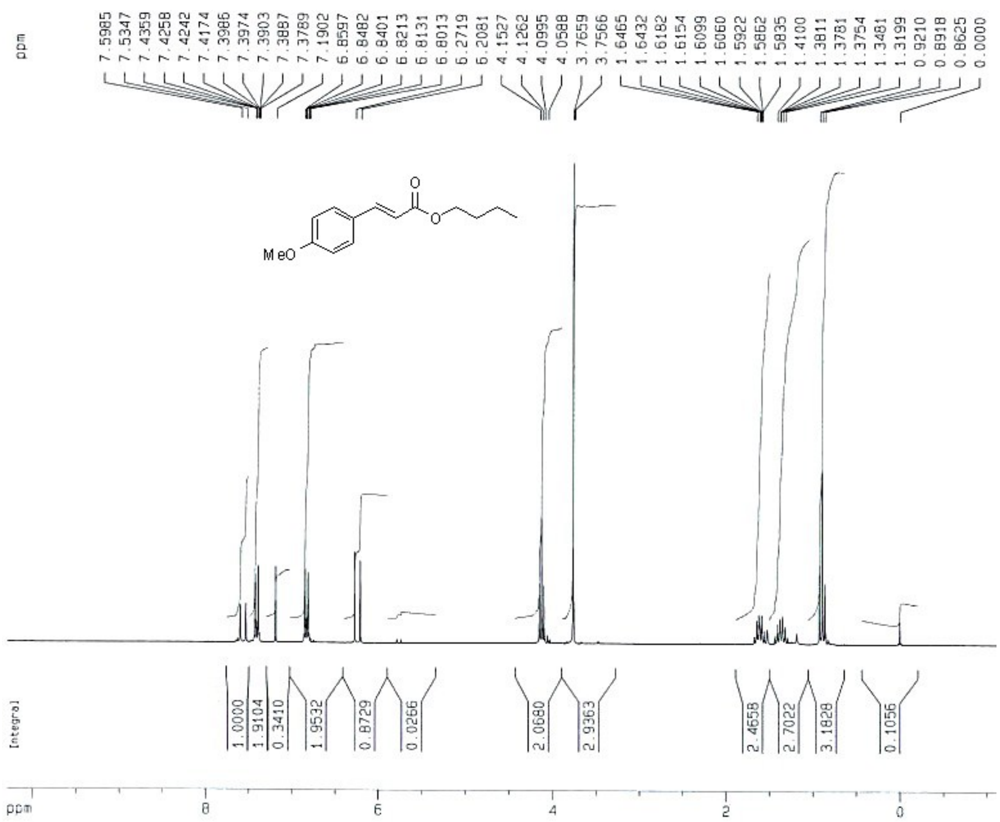
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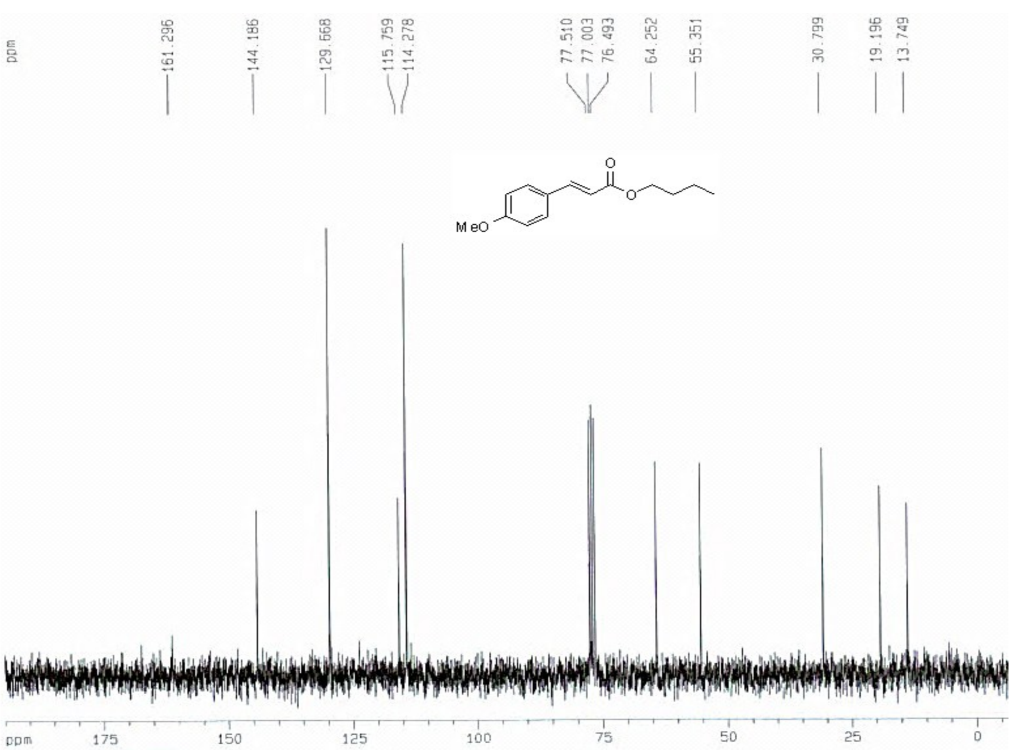
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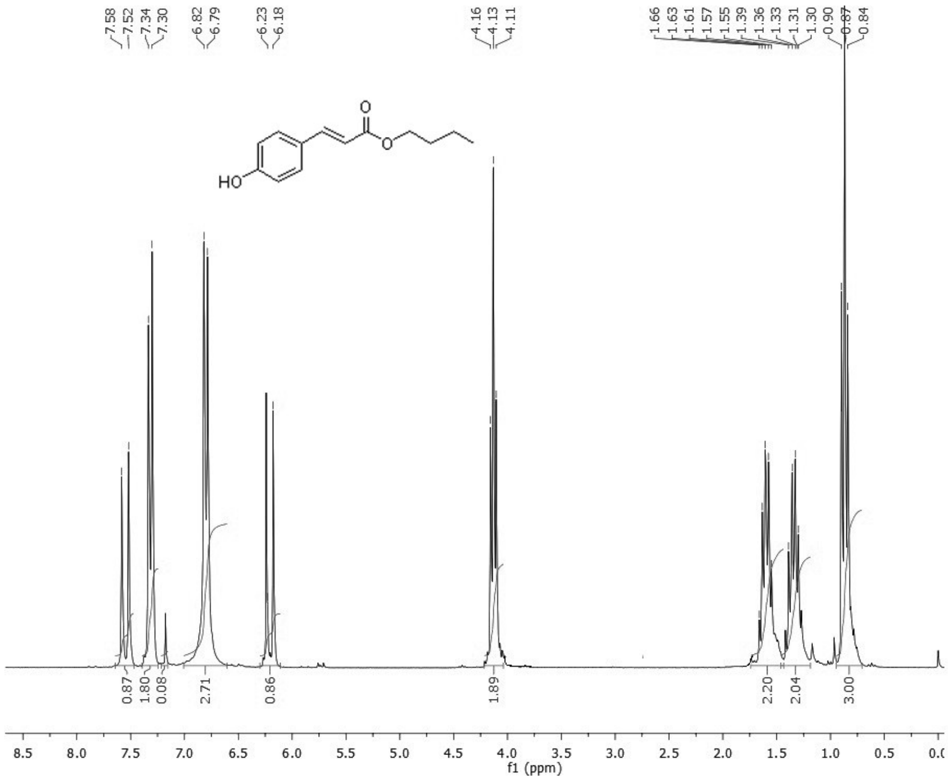
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1.39
1.36
1.33
1.31
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O
HO
O
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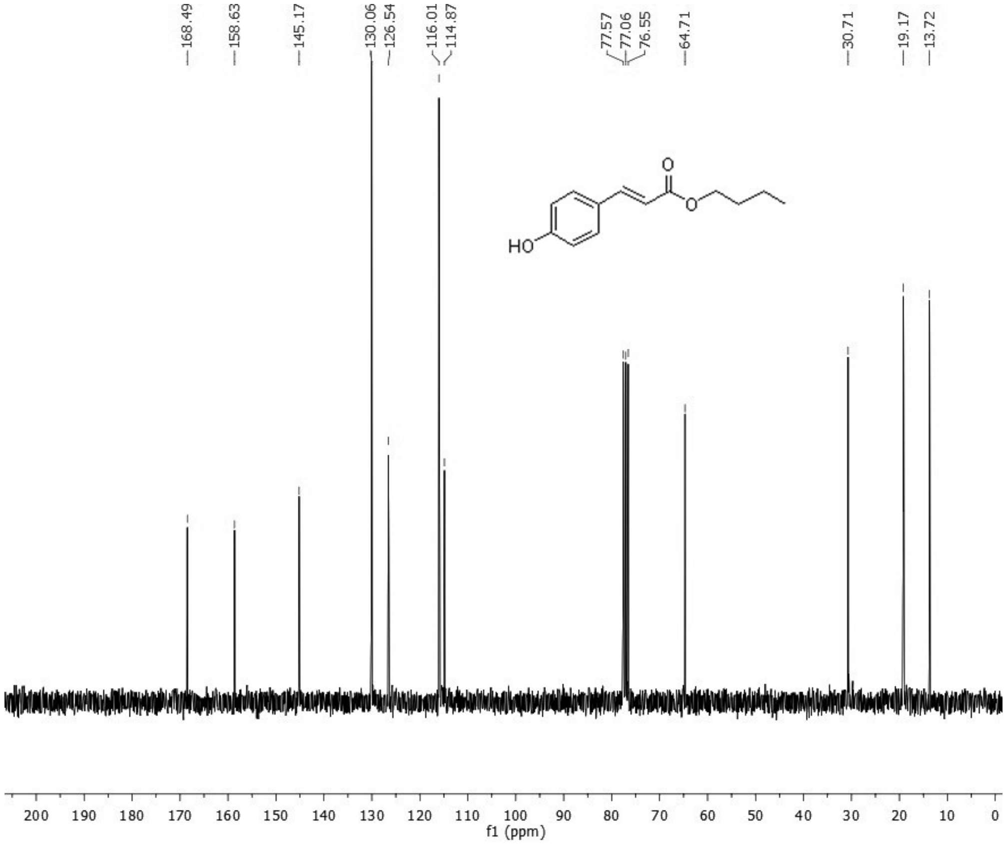
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180
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50
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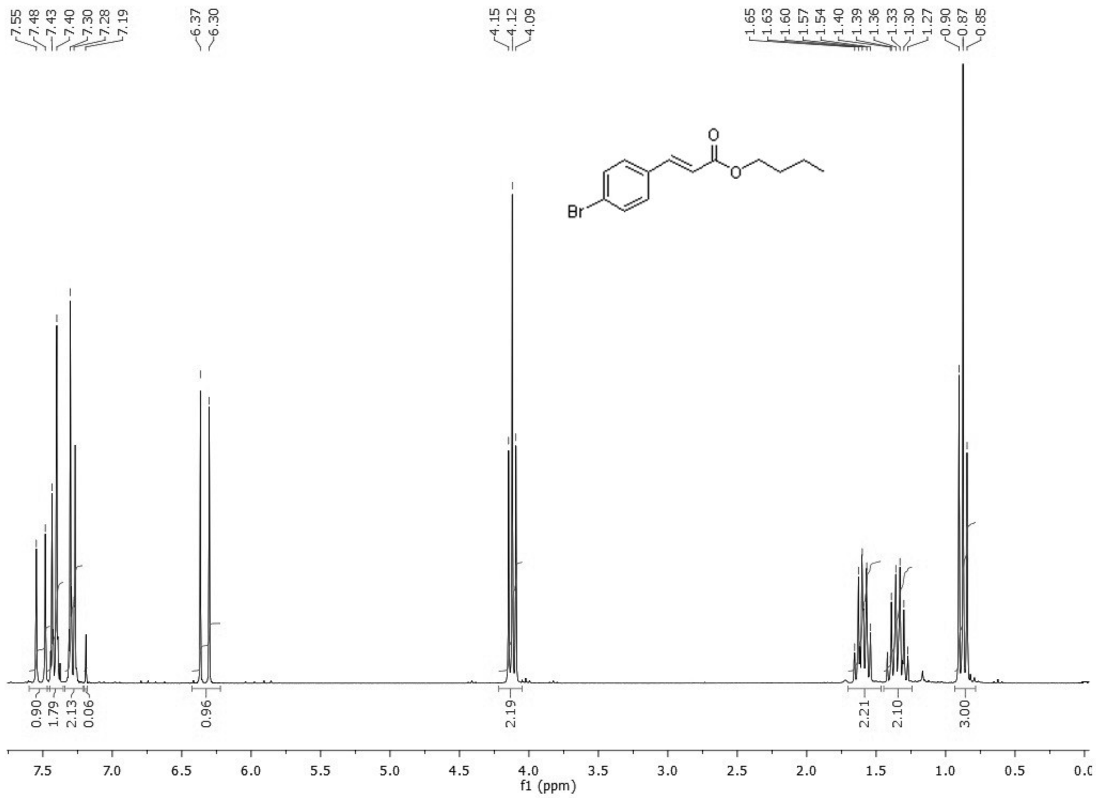
f1 (ppm)



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1.33
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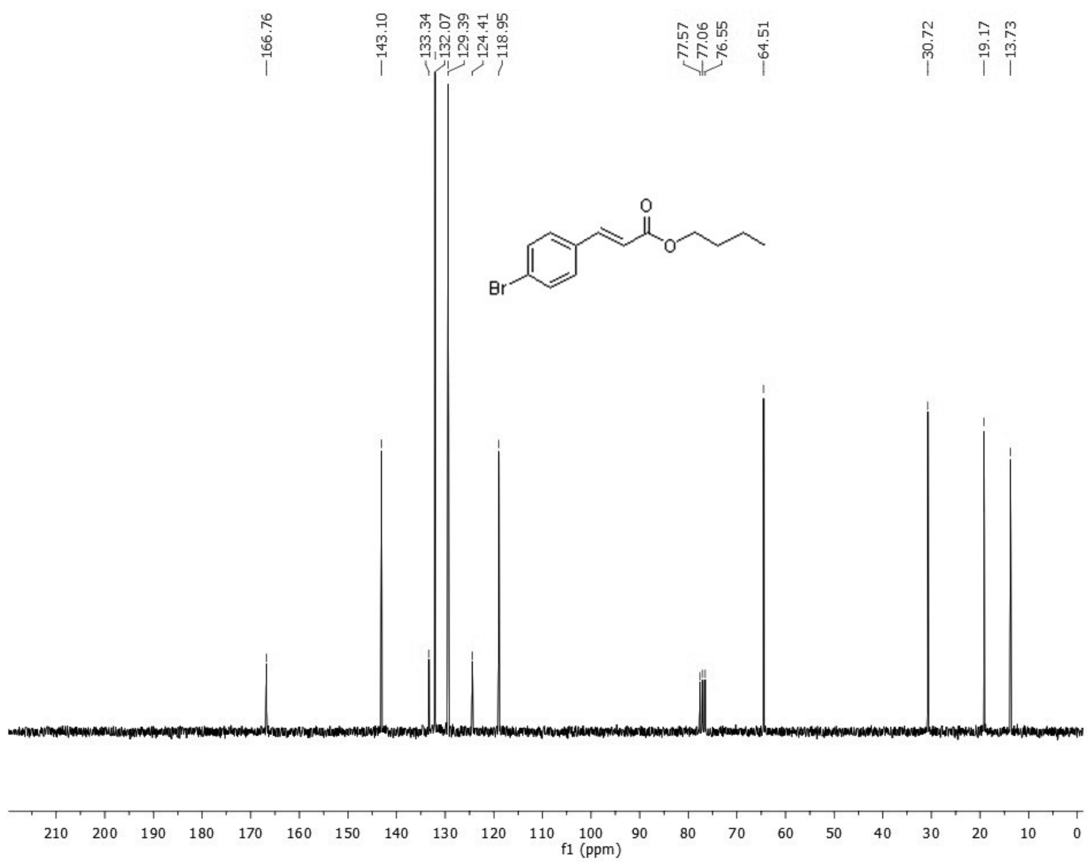

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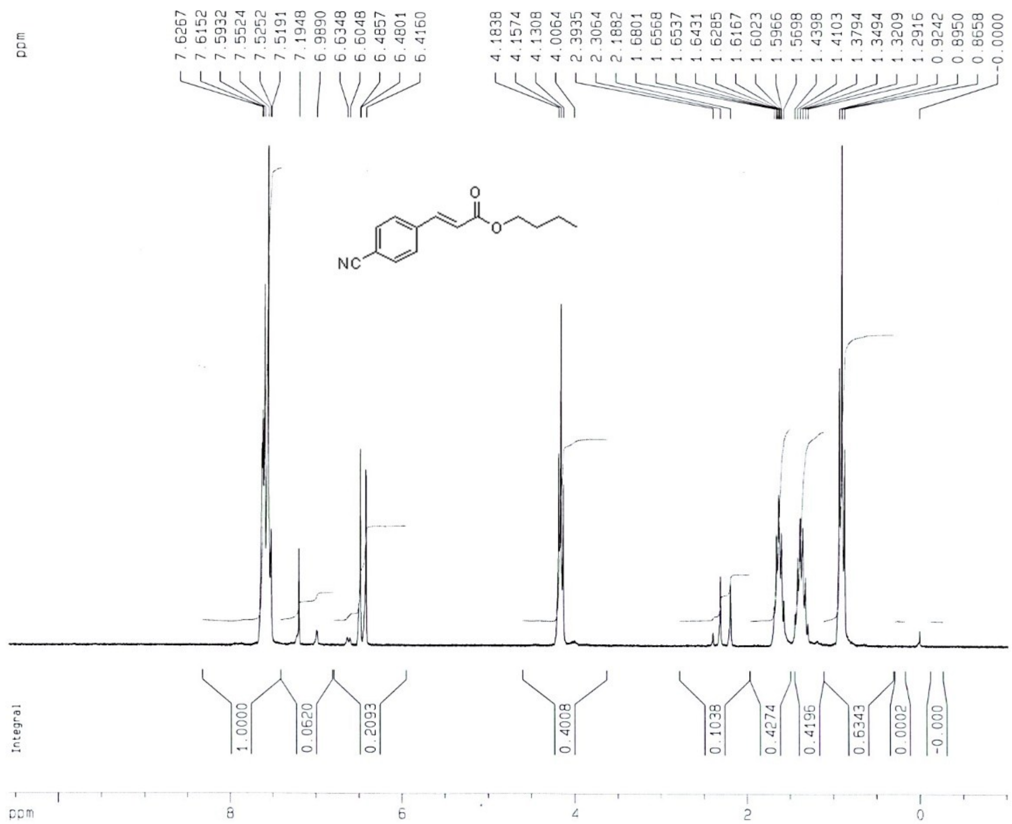
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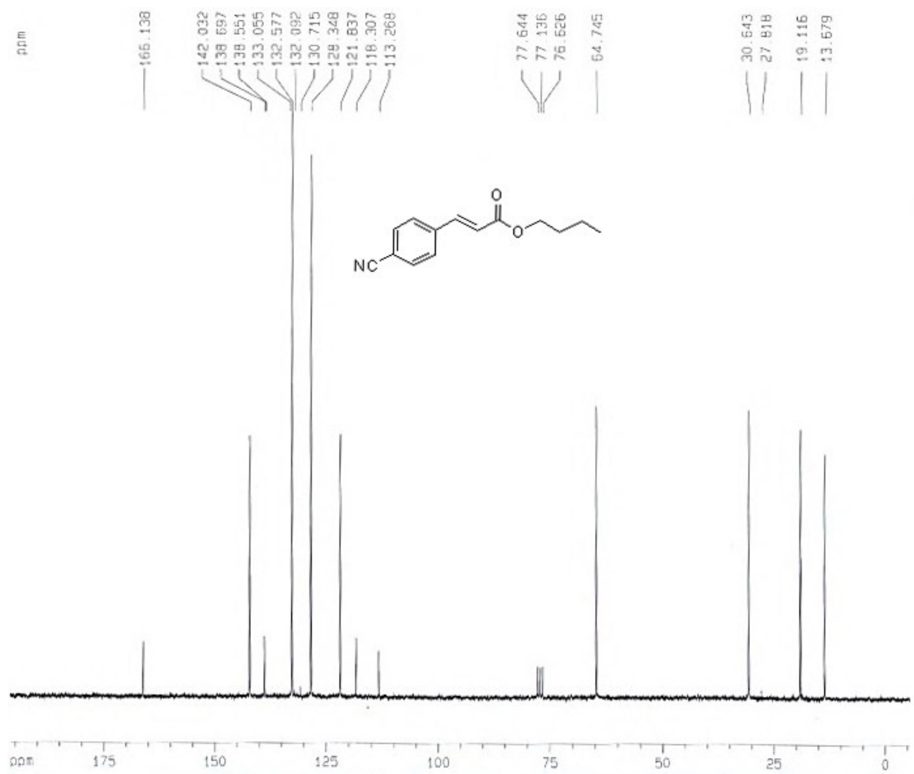
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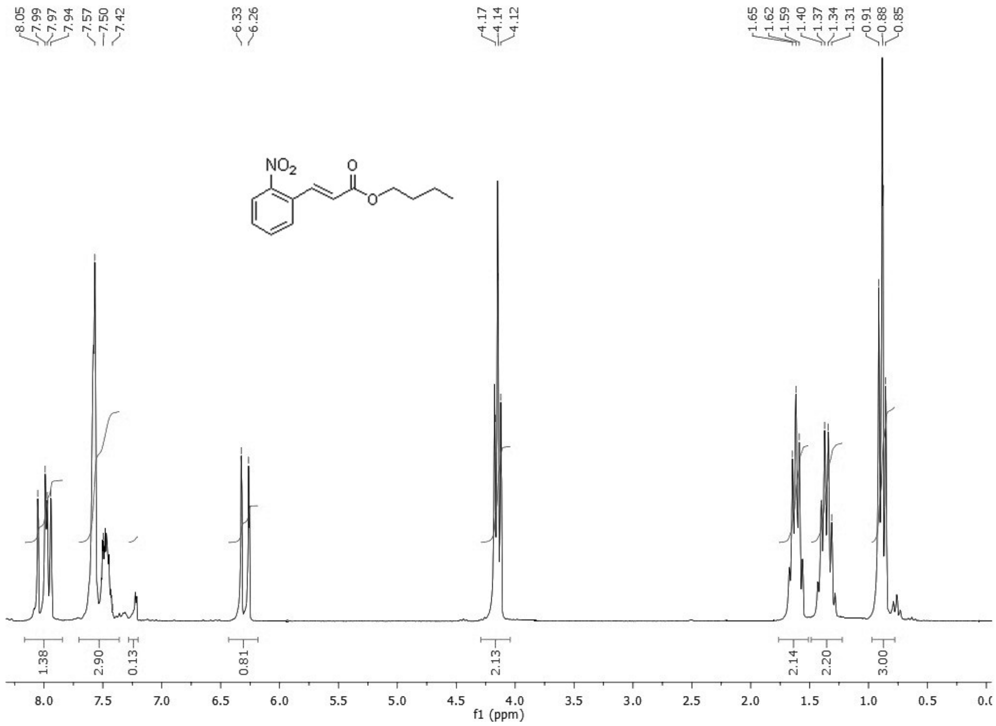
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2.14
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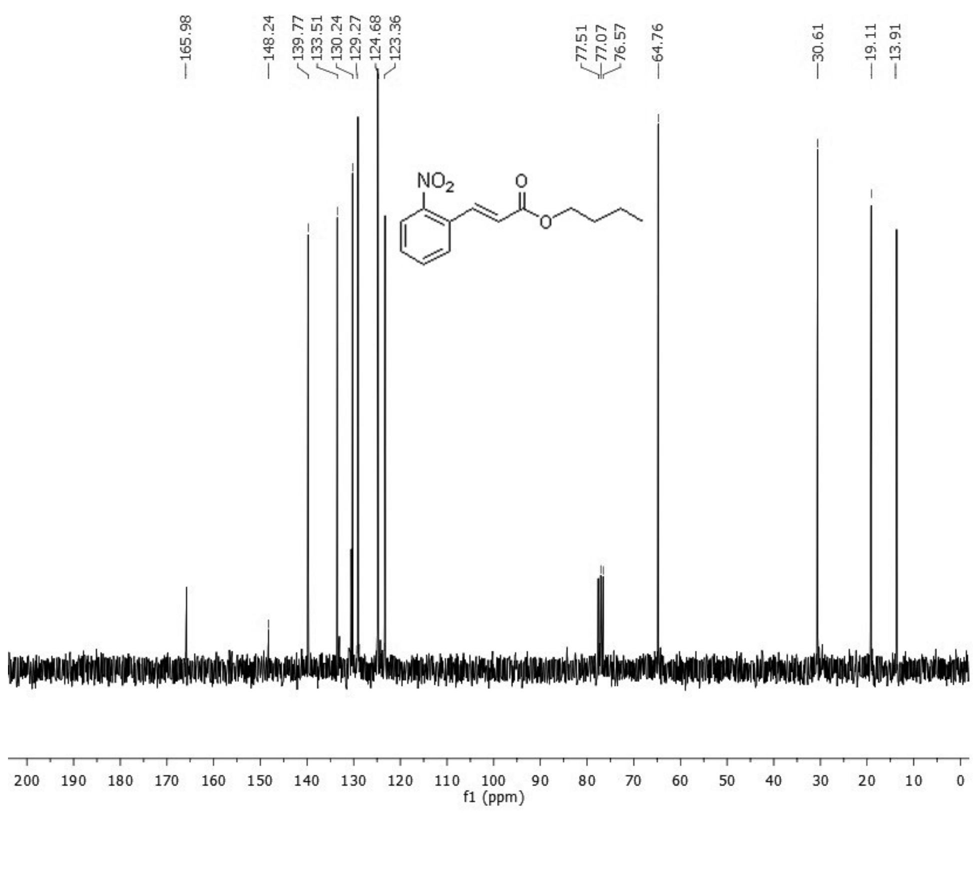
f1 (ppm)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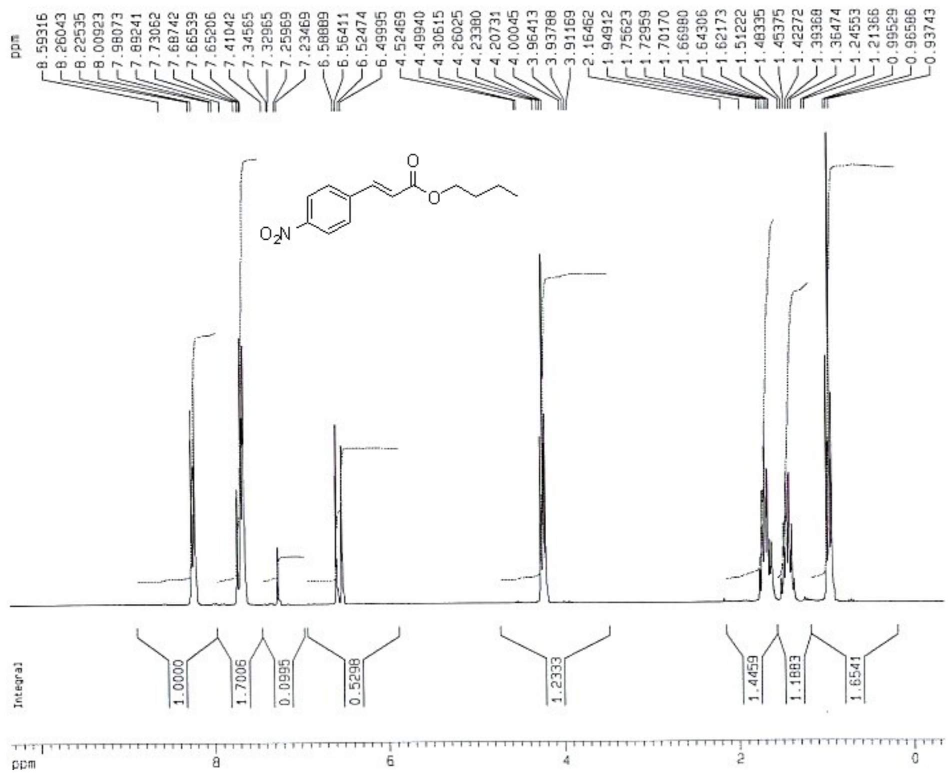
f1 (ppm)



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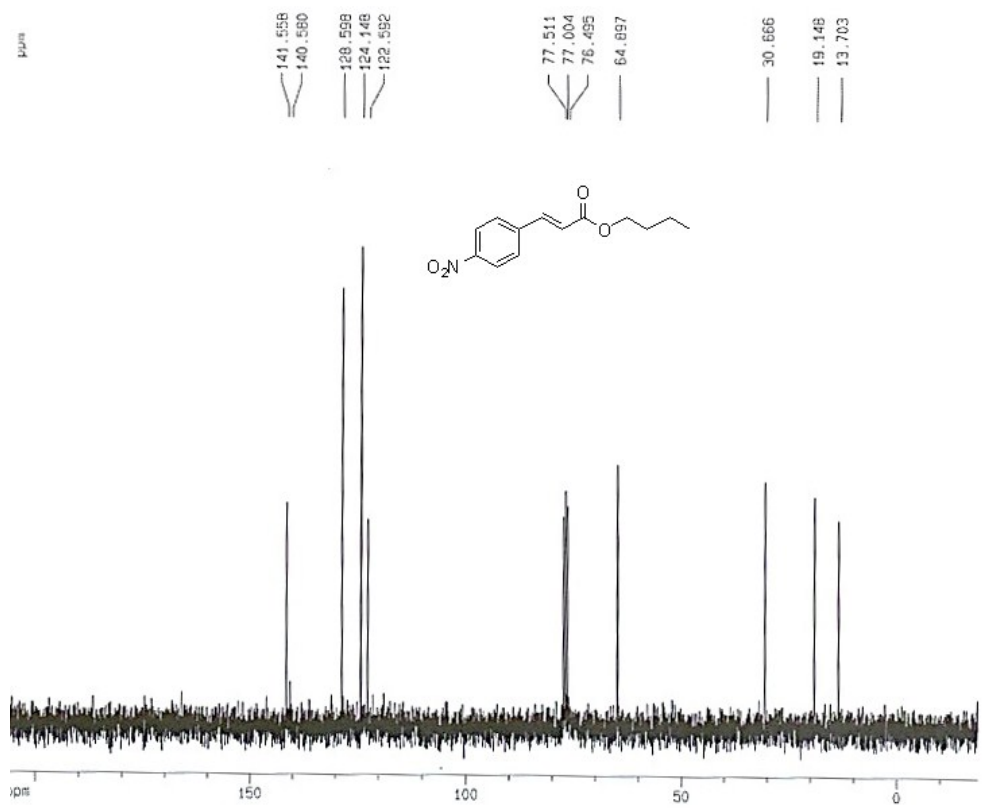

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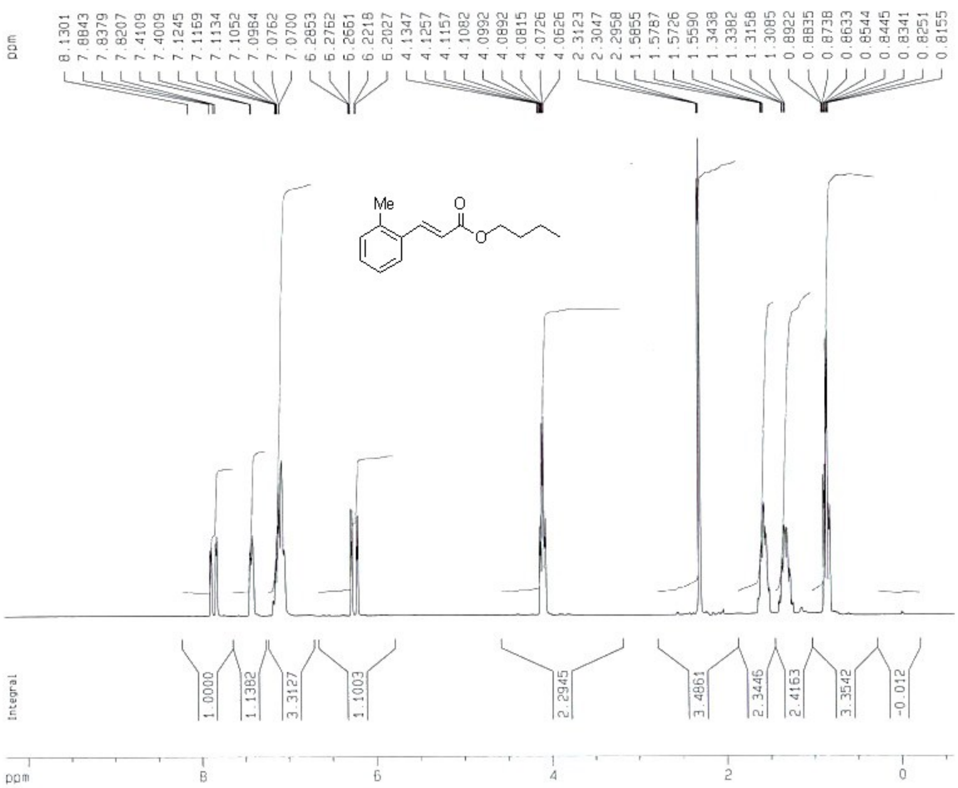
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2
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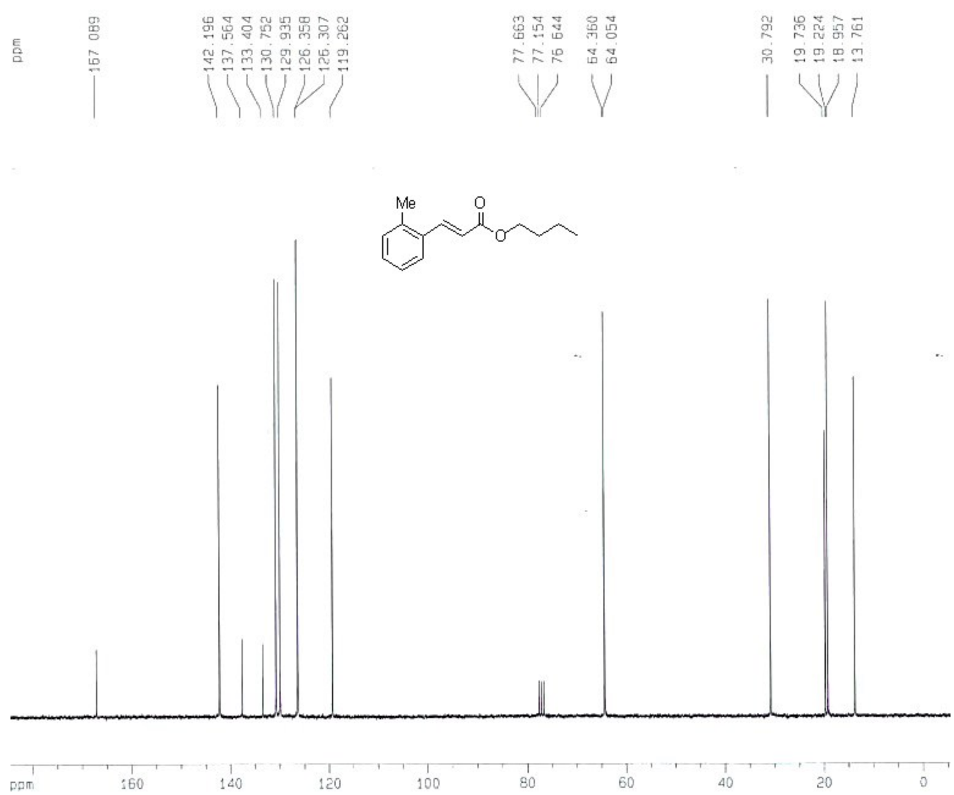
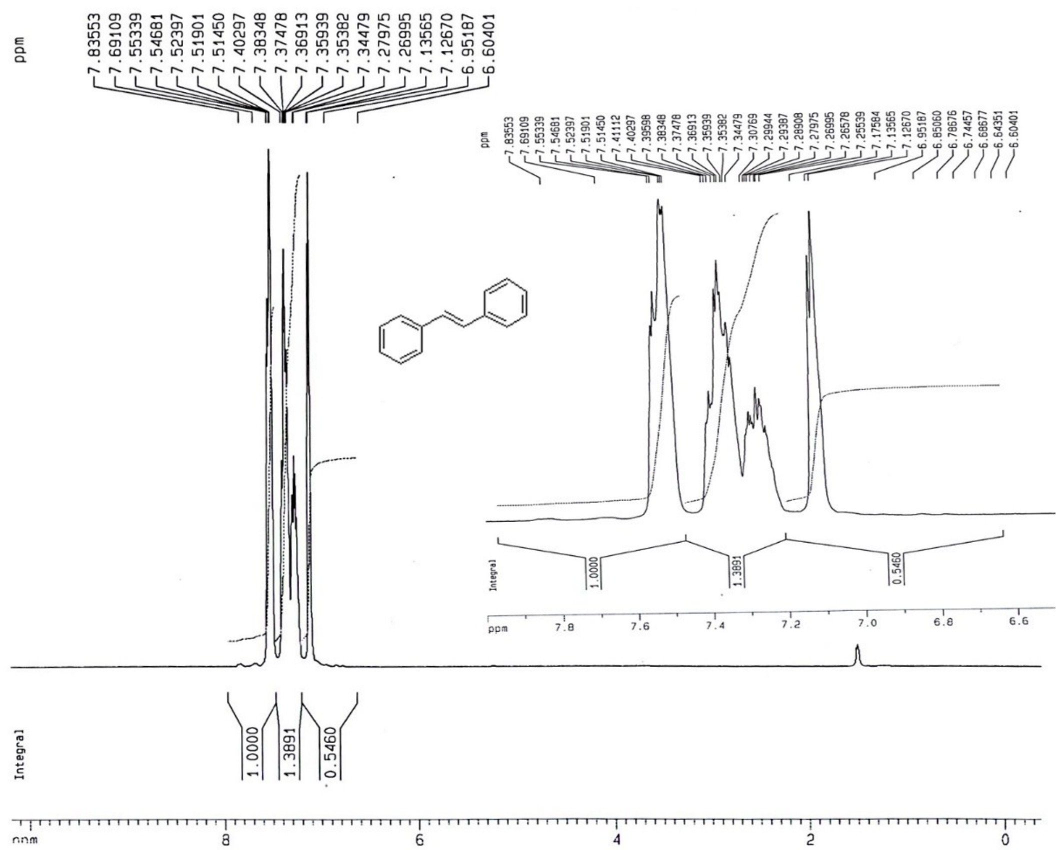
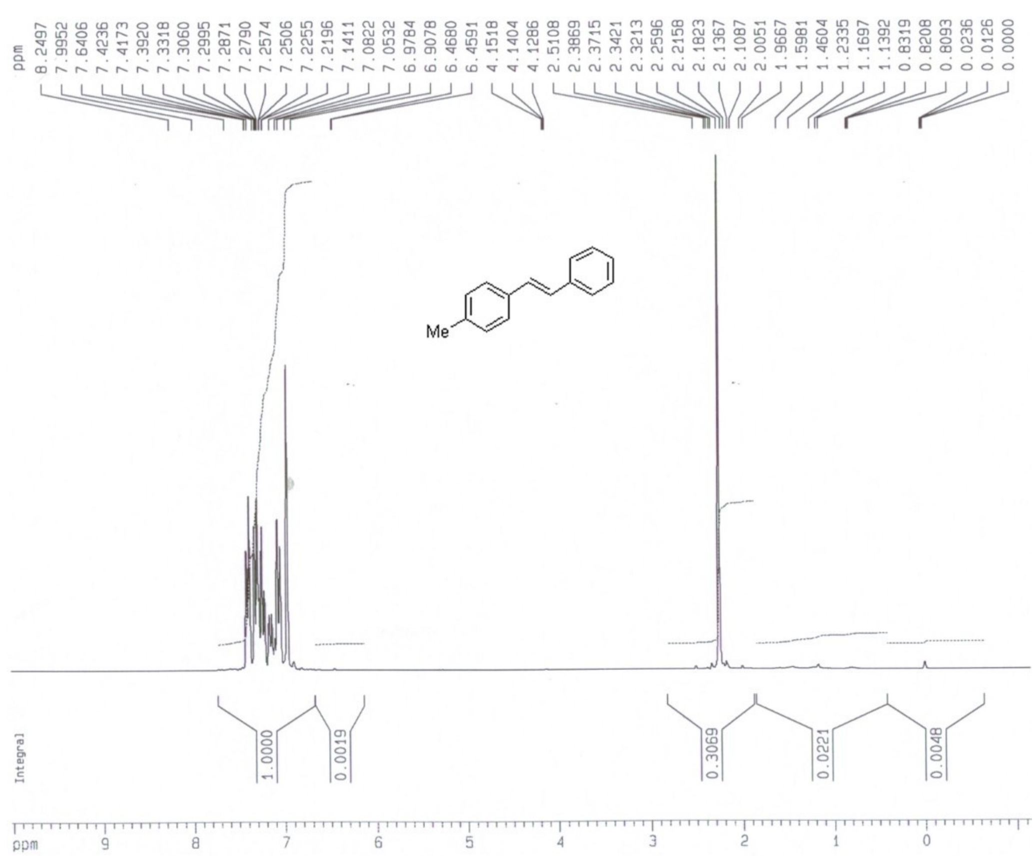
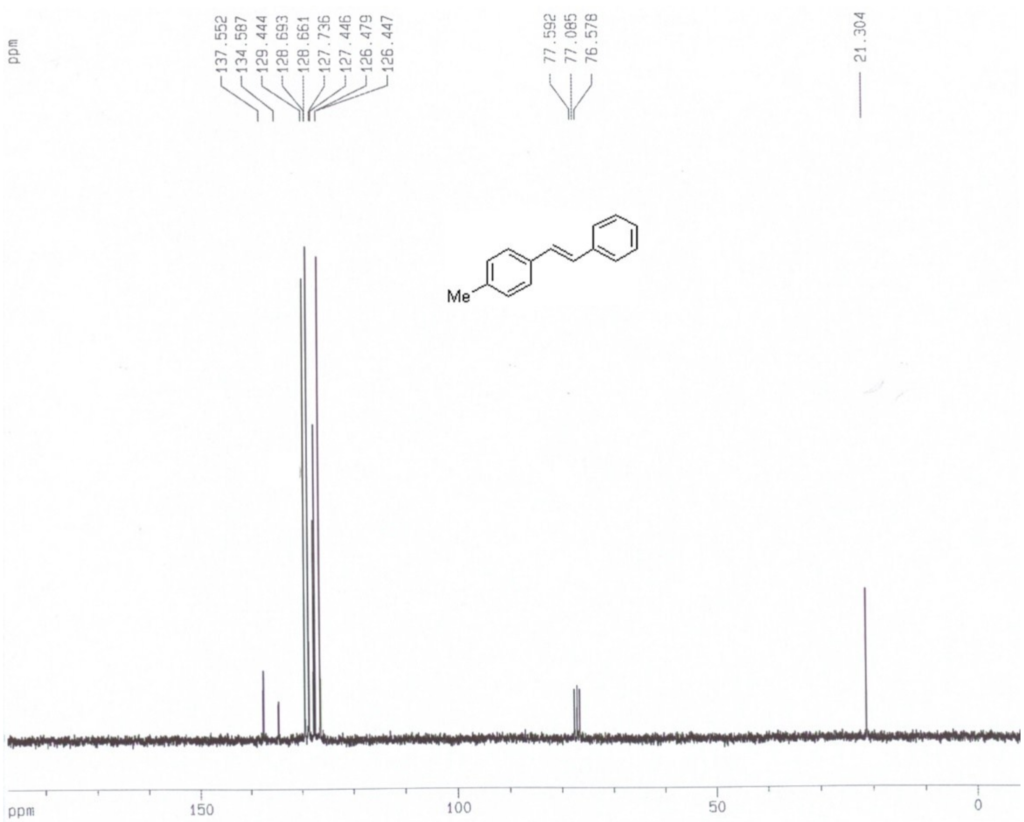
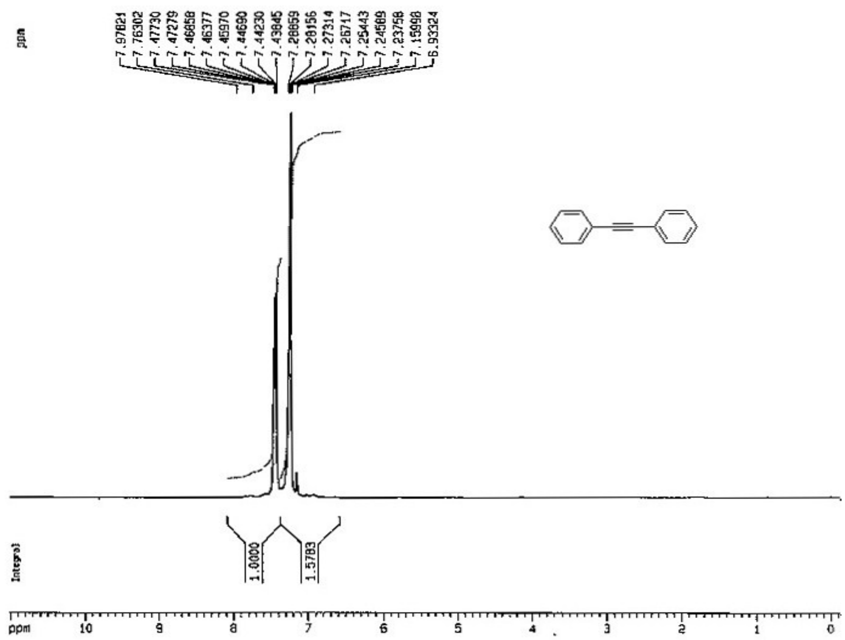

ppm
167.089
142.196
137.564
133.404
130.752
129.935
126.358
126.307
119.262
77.663
77.154
76.644
64.360
64.054
30.792
19.736
19.224
18.957
13.761
Me
O
O
ppm
160
140
120
100
80
60
40
20
0

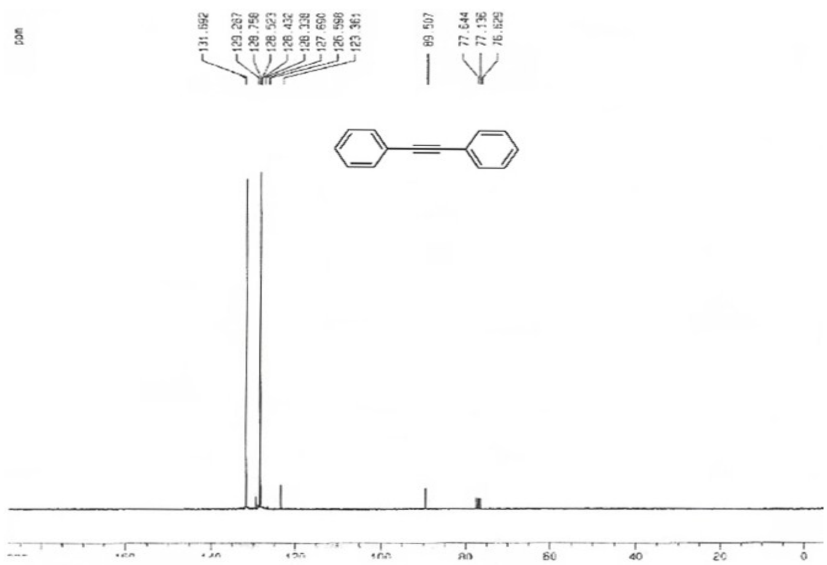


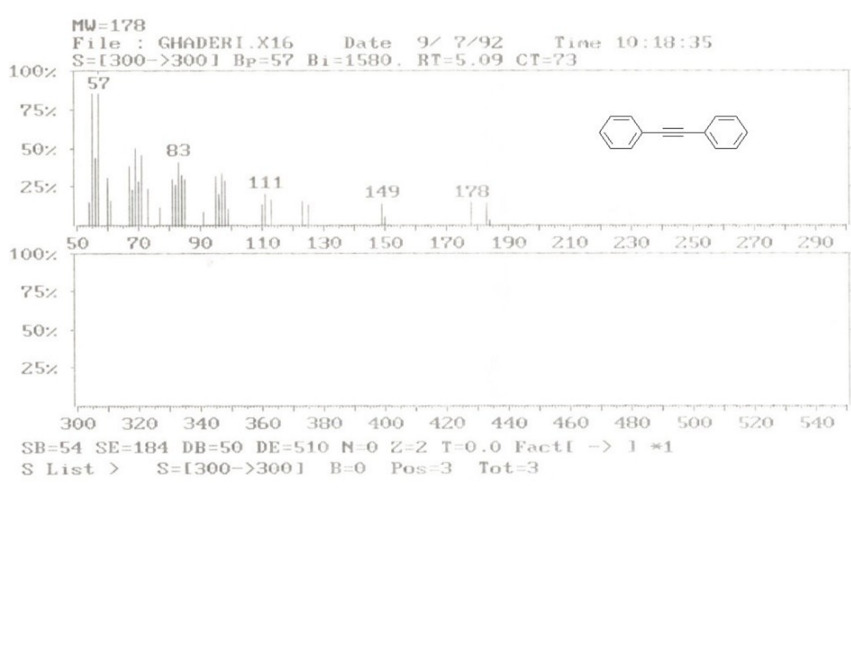


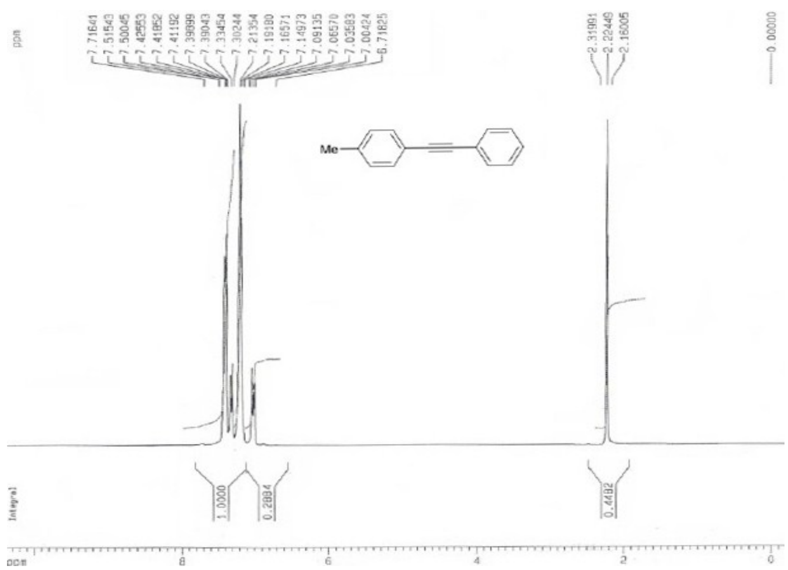


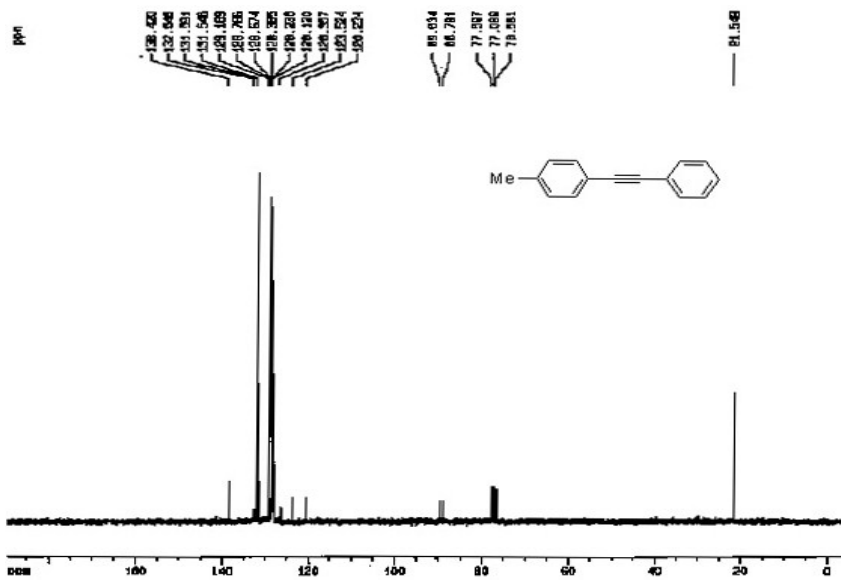
ppm
137.552
134.587
129.444
128.693
128.661
127.736
127.446
126.479
126.447
77.592
77.085
76.578
21.304
Me
ppm
150
100
50
0

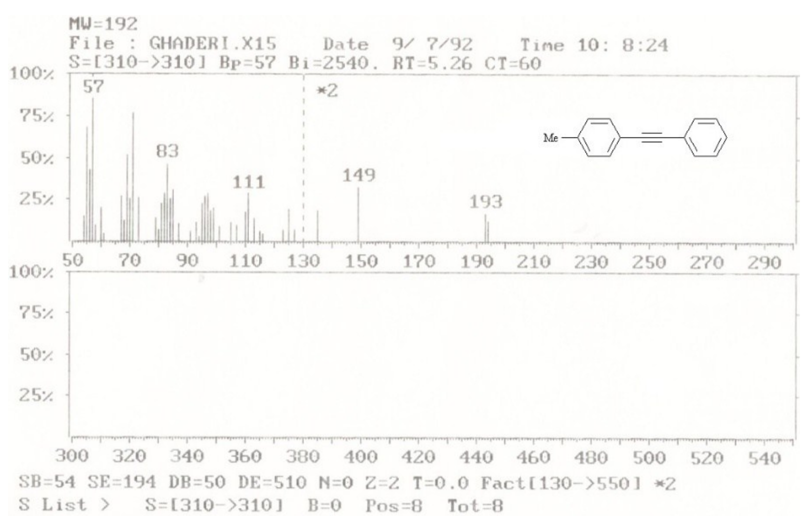

ppm
7.97821
7.76302
7.47730
7.47279
7.46858
7.46377
7.45970
7.44690
7.44230
7.43845
7.28869
7.28156
7.27314
7.26717
7.25443
7.24589
7.23758
7.15998
6.93324
Integral
1.0000
1.5783
ppm 10 9 8 7 6 5 4 3 2 1 0

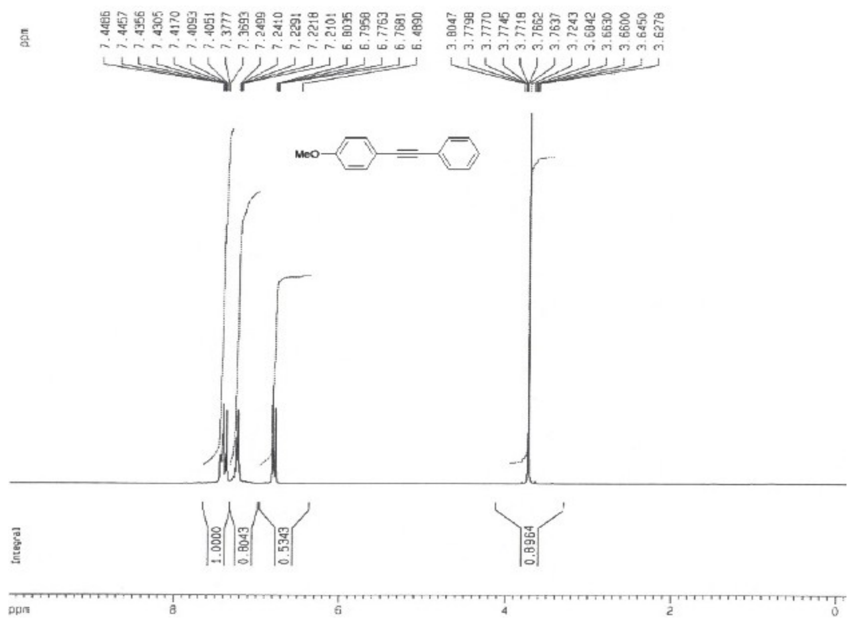

ppm
131.692
129.287
128.758
128.523
128.432
128.338
127.690
126.598
123.361
89.507
77.644
77.136
76.629
80 60 40 20 0

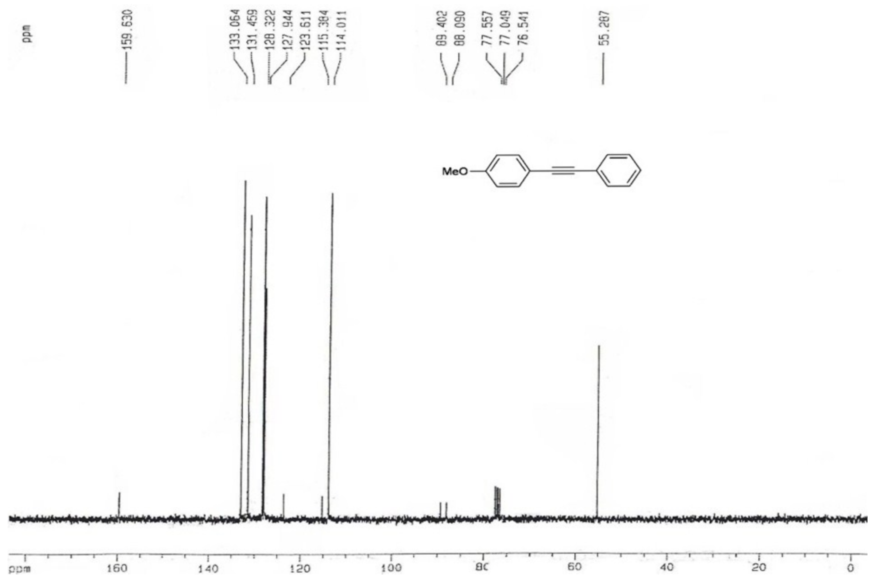

MW=178
File : GHADERI.X16 Date 9/ 7/92 Time 10:18:35
S=[300->300] Bp=57 Bi=1580. RT=5.09 CT=73
100% 75% 50% 25%
57
83
111
149
178
50 70 90 110 130 150 170 190 210 230 250 270 290
100% 75% 50% 25%
300 320 340 360 380 400 420 440 460 480 500 520 540
SB=54 SE=184 DB=50 DE=510 N=0 Z=2 T=0.0 Fact[->] *1
S List > S=[300->300] B=0 Pos=3 Tot=3

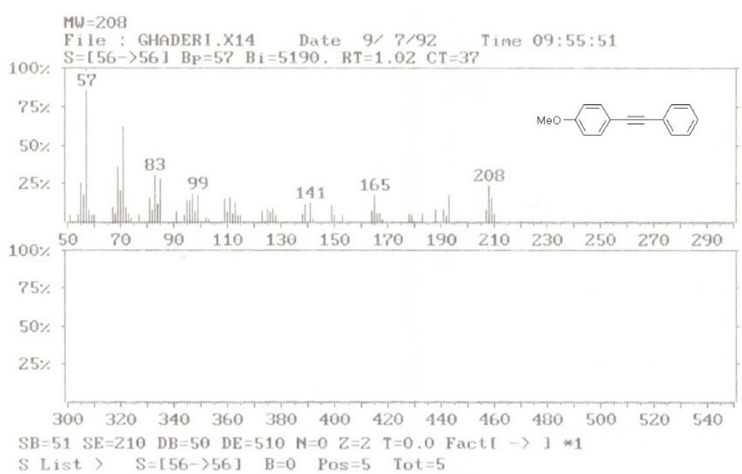

ppm
Me
Integral
1.0000
0.2884
0.4482
0.00000

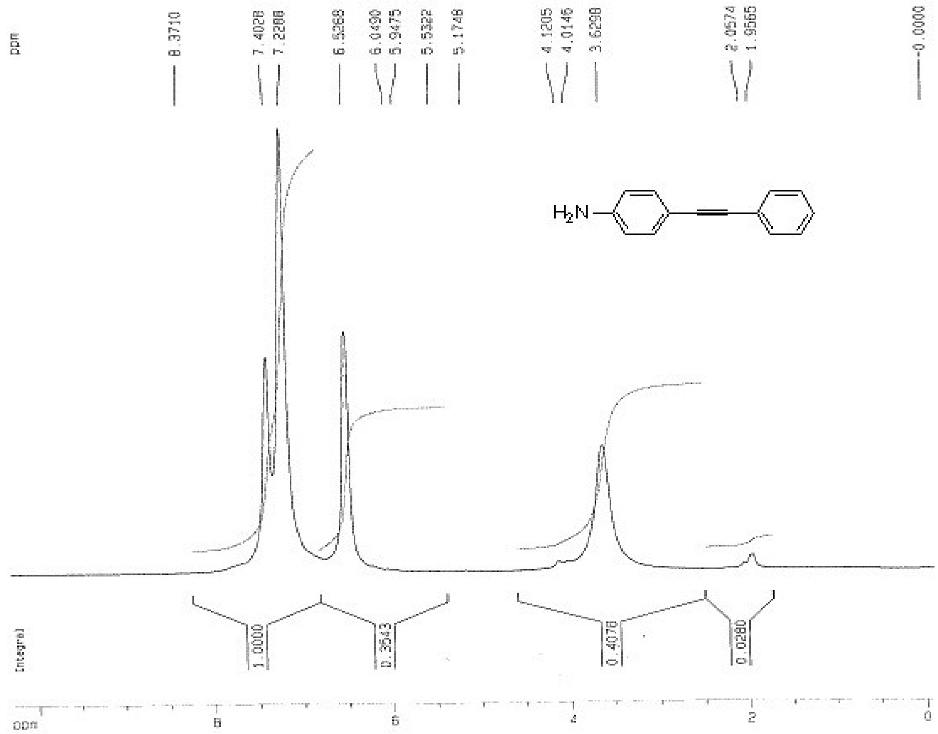

ppm
Me

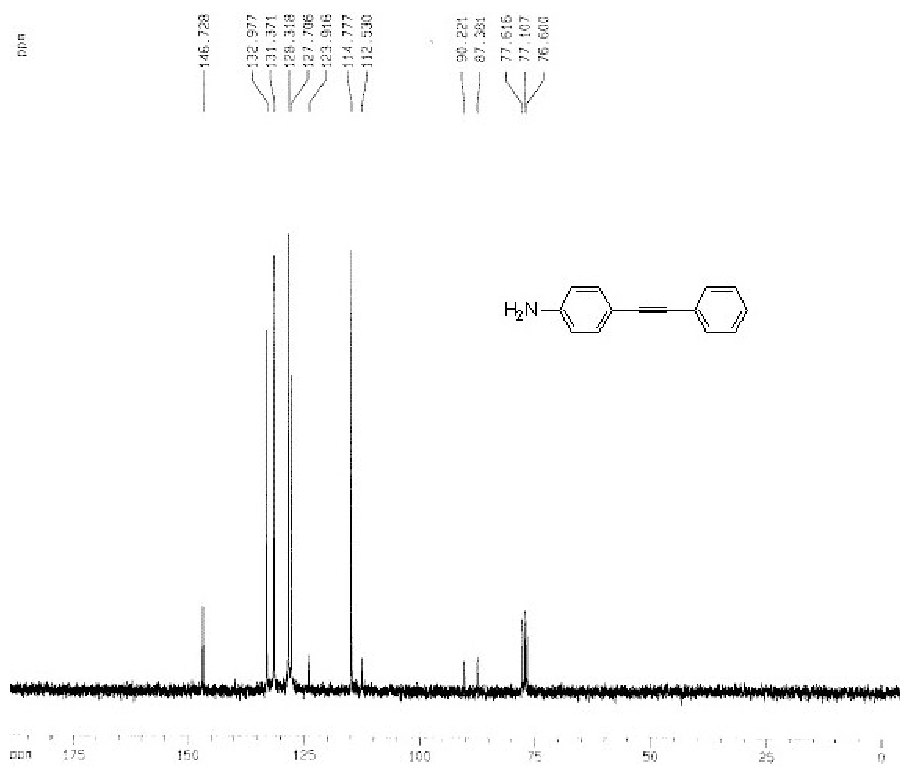

MW=192
File : GHADERI.X15 Date 9/ 7/92 Time 10: 8:24
S=[310->310] Bp=57 Bi=2540. RT=5.26 CT=60
57
*2
83
111
149
193
Me
SB=54 SE=194 DB=50 DE=510 N=0 Z=2 T=0.0 Fact[130->550] *2
S List > S=[310->310] B=0 Pos=8 Tot=8

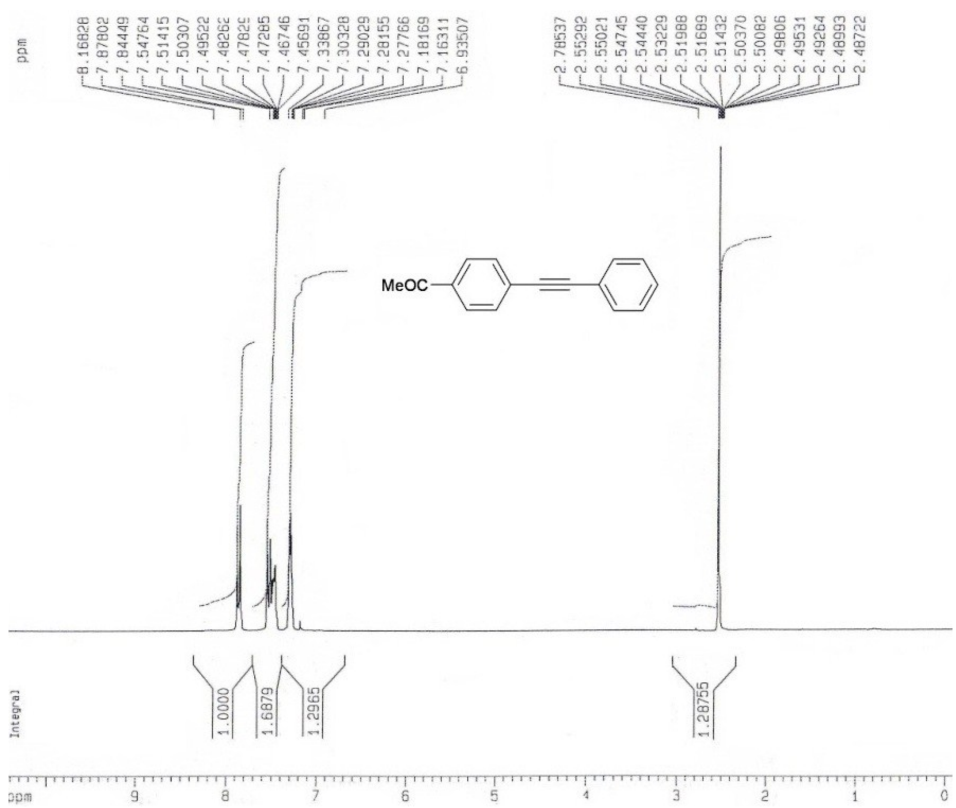

ppm
7.4486
7.4457
7.4356
7.4305
7.4170
7.4093
7.4051
7.3777
7.3693
7.2499
7.2410
7.2291
7.2218
7.2101
6.8035
6.7950
6.7763
6.7681
6.4890
3.8047
3.7798
3.7770
3.7745
3.7718
3.7662
3.7637
3.7243
3.6842
3.6630
3.6600
3.6450
3.6270
MeO
Integral
1.0000
0.8043
0.5343
0.8964
ppm
8
6
4
2
0

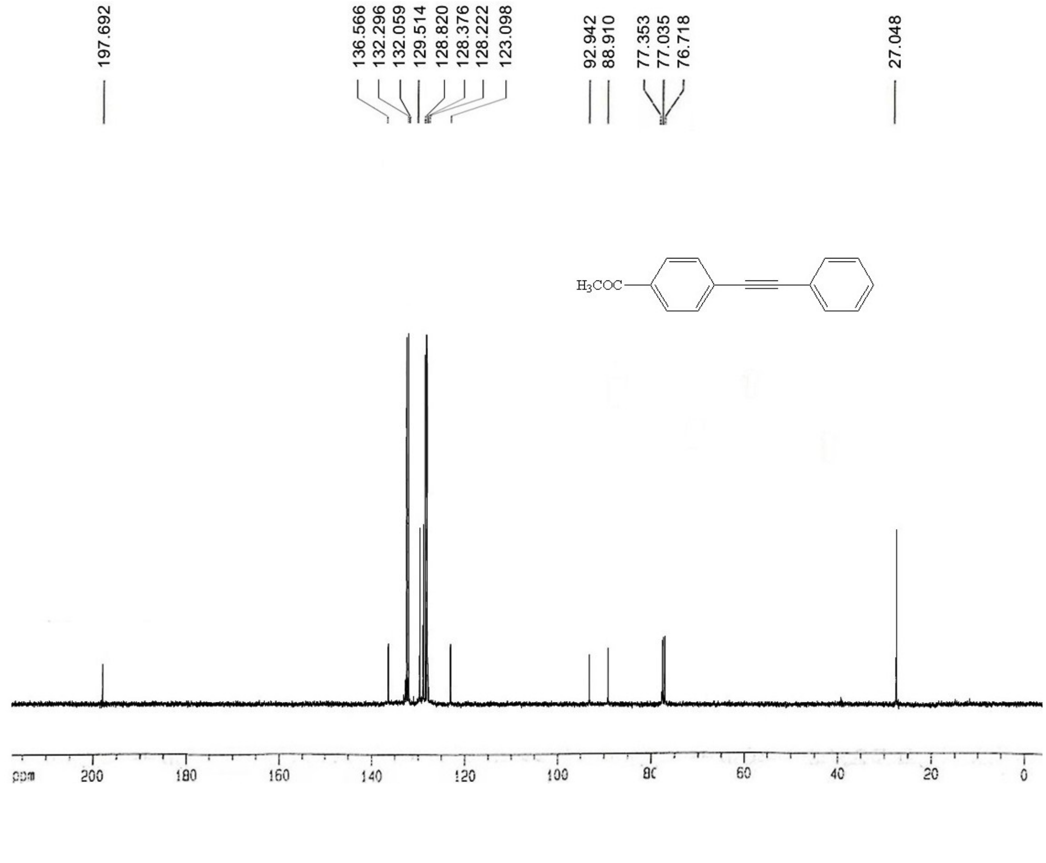

ppm
159.630
133.064
131.459
128.322
127.944
123.611
115.384
114.011
89.402
88.090
77.557
77.049
76.541
55.287
MeO
ppm
160
140
120
100
80
60
40
20
0

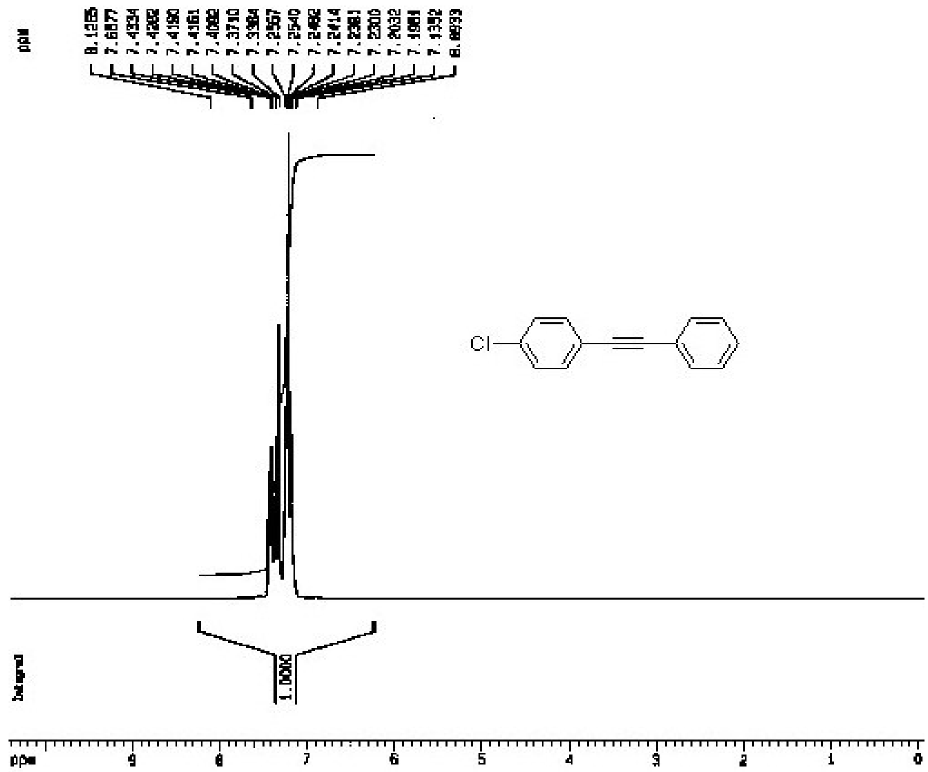

MW=208
File : GHADERI.X14 Date 9/ 7/92 Time 09:55:51
S=[56->56] Bp=57 Bi=5190. RT=1.02 CT=37
100%
75%
50%
25%
57
83
99
141
165
208
MeO
50 70 90 110 130 150 170 190 210 230 250 270 290
100%
75%
50%
25%
300 320 340 360 380 400 420 440 460 480 500 520 540
SB=51 SE=210 DB=50 DE=510 N=0 Z=2 T=0.0 Fact[->] *1
S List > S=[56->56] B=0 Pos=5 Tot=5


ppm
8.3710
7.4028
7.2288
6.5268
6.0490
5.9475
5.5322
5.1748
4.1205
4.0146
3.6298
2.0574
1.9565
-0.0000
H2N
Integral
1.0000
0.3543
0.4076
0.0280
ppm 8 6 4 2 0

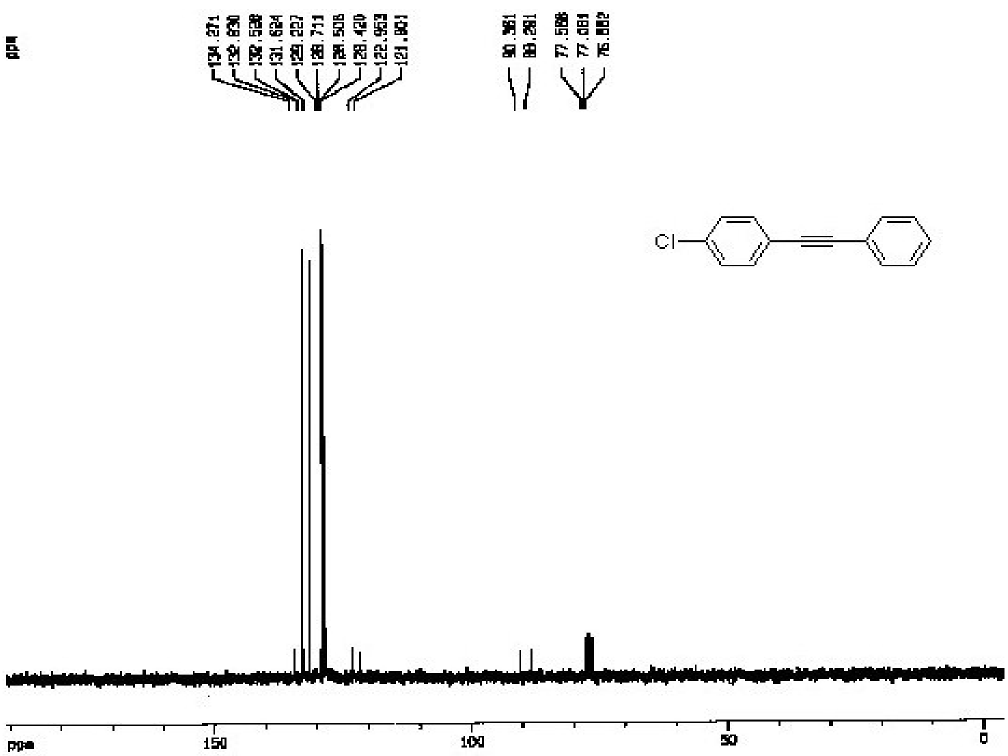

ppm
146.728
132.977
131.371
128.318
127.706
123.916
114.777
112.530
90.221
87.381
77.616
77.107
76.600
H2N
ppm 175 150 125 100 75 50 25 0


MeOC
ppm
8.16828
7.87802
7.84449
7.54764
7.51415
7.50307
7.49522
7.48262
7.47829
7.47285
7.46746
7.45691
7.33867
7.30328
7.29029
7.28155
7.27766
7.18169
7.16311
6.93507
2.78537
2.55292
2.55021
2.54745
2.54440
2.53229
2.51988
2.51689
2.51432
2.50370
2.50082
2.49806
2.49531
2.49264
2.48993
2.48722
Integral
1.0000
1.6879
1.2965
1.28755
ppm
9
8
7
6
5
4
3
2
1
0

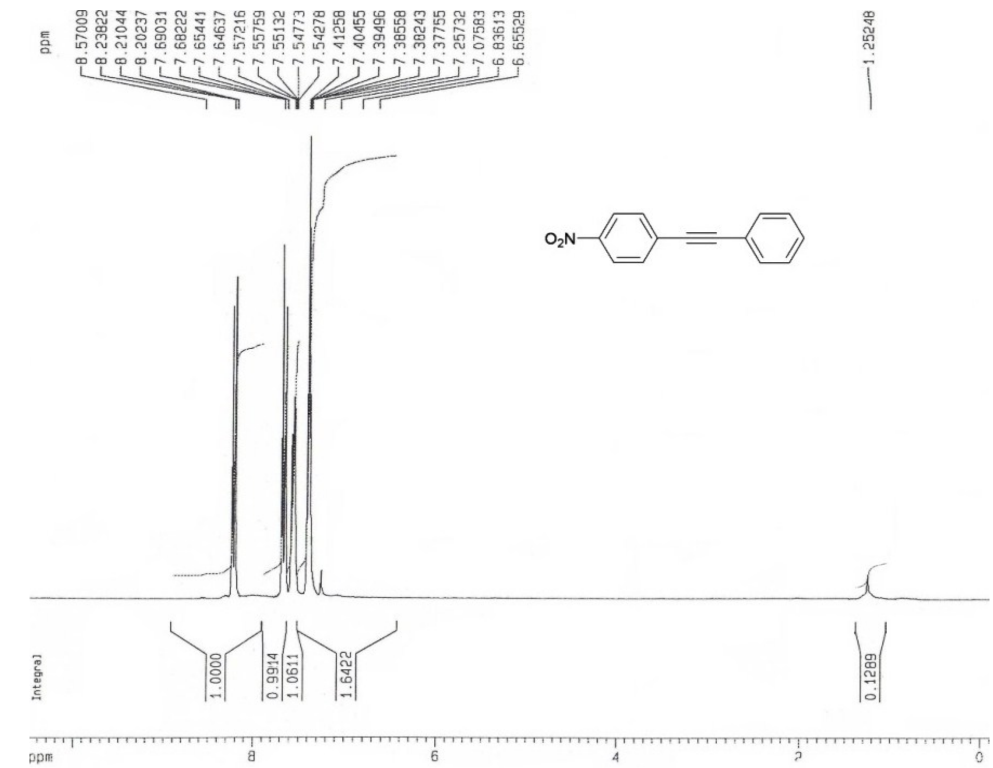

197.692
136.566
132.296
132.059
129.514
128.820
128.376
128.222
123.098
92.942
88.910
77.353
77.035
76.718
27.048
H3COC
ppm
200
180
160
140
120
100
80
60
40
20
0

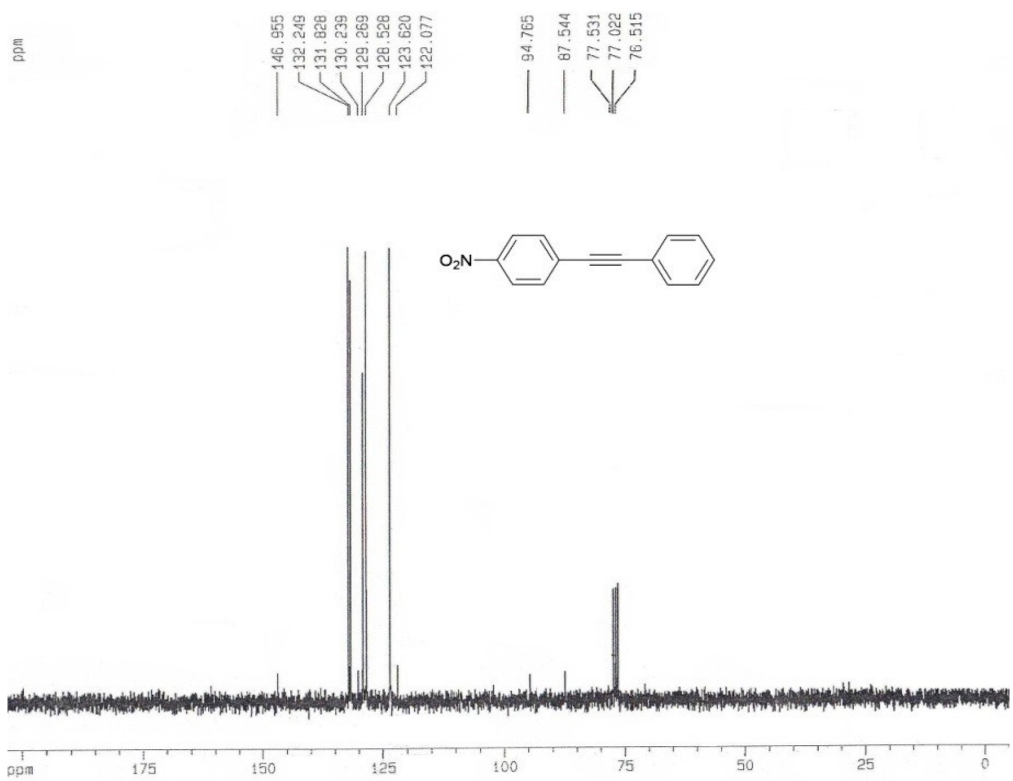


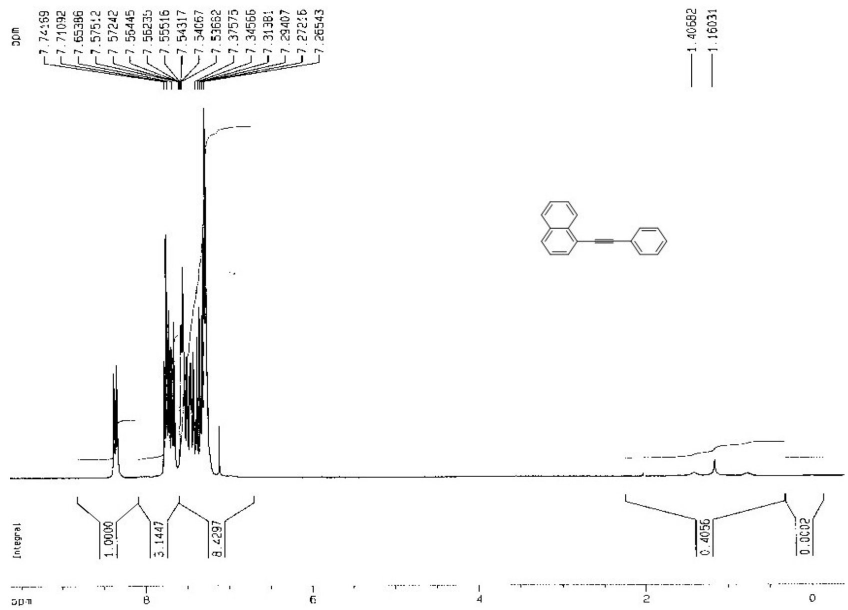
ppm
Cl
1.0000
ppm
9
8
7
6
5
4
3
2
1
0

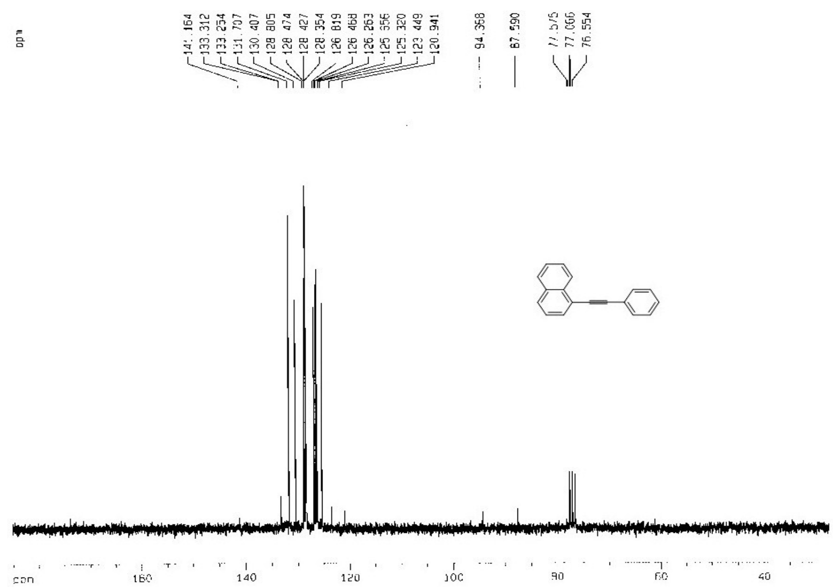


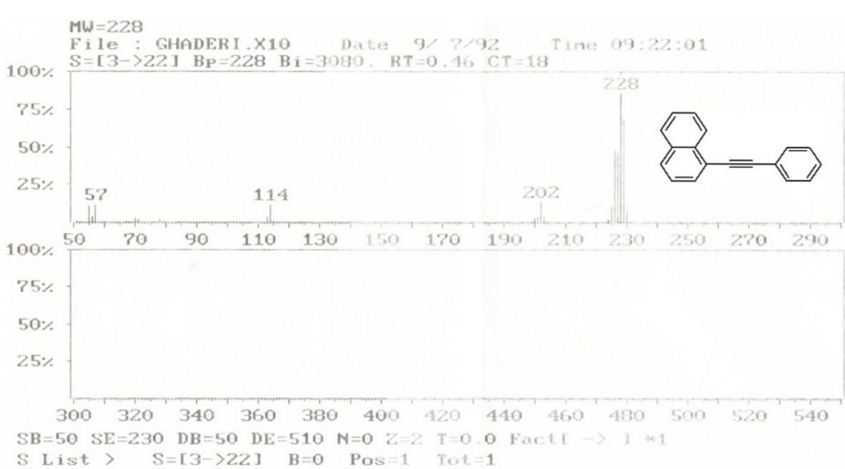
ppm
Cl
ppm
150
100
50
0

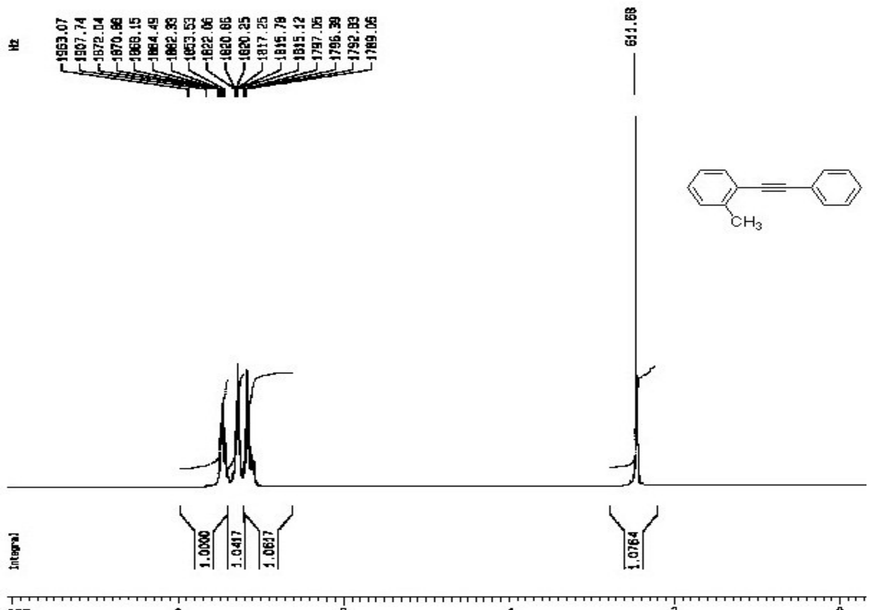

ppm
8.57009
8.23822
8.21044
8.20237
7.69031
7.68222
7.65441
7.64637
7.57216
7.55759
7.55132
7.54773
7.54278
7.41258
7.40455
7.39496
7.38558
7.38243
7.37755
7.25732
7.07583
6.83613
6.65529
1.25248
O_2N
Integral
1.0000
0.9914
1.0611
1.6422
0.1289
ppm
8
6
4
2
0

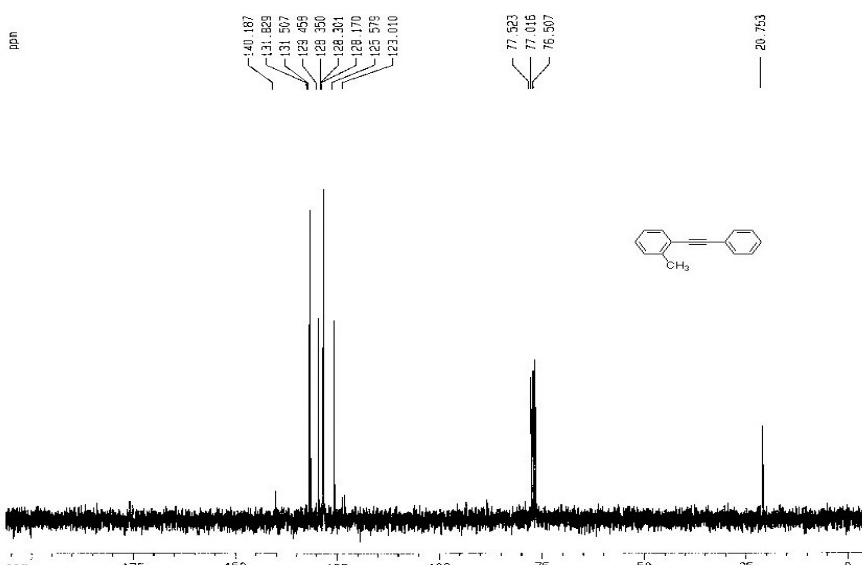

ppm
146.955
132.249
131.828
130.239
129.269
128.528
123.620
122.077
94.765
87.544
77.531
77.022
76.515
O_2N
ppm
175
150
125
100
75
50
25
0

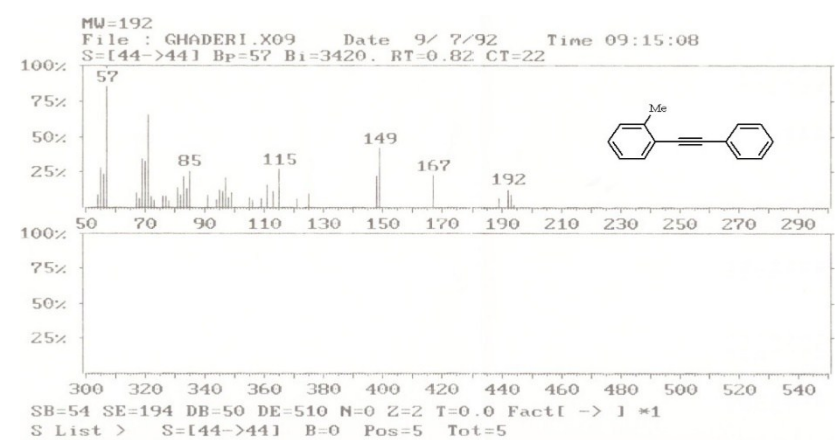

ppm
7.74169
7.71092
7.65386
7.57512
7.57242
7.56445
7.56235
7.55516
7.54317
7.54067
7.53662
7.37575
7.34566
7.31381
7.29407
7.27215
7.26543
1.40682
1.16031
Integral
1.0000
3.1447
8.4297
0.4056
0.0002
ppm
8
6
4
2
0

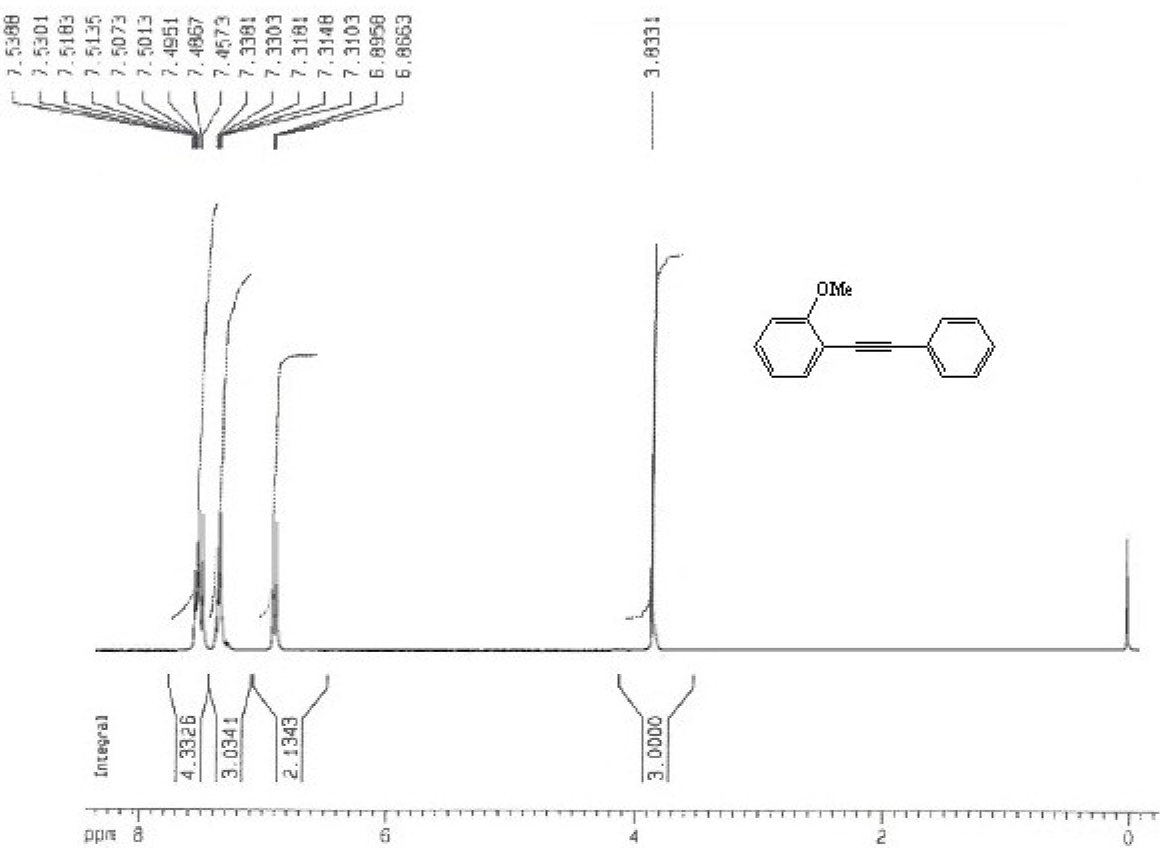

ppm
141.164
133.312
133.254
131.707
130.407
128.805
128.474
128.427
128.354
126.819
126.468
126.263
125.556
125.320
123.449
120.941
94.368
87.590
77.575
77.066
76.554
ppm
160
140
120
100
80
60
40

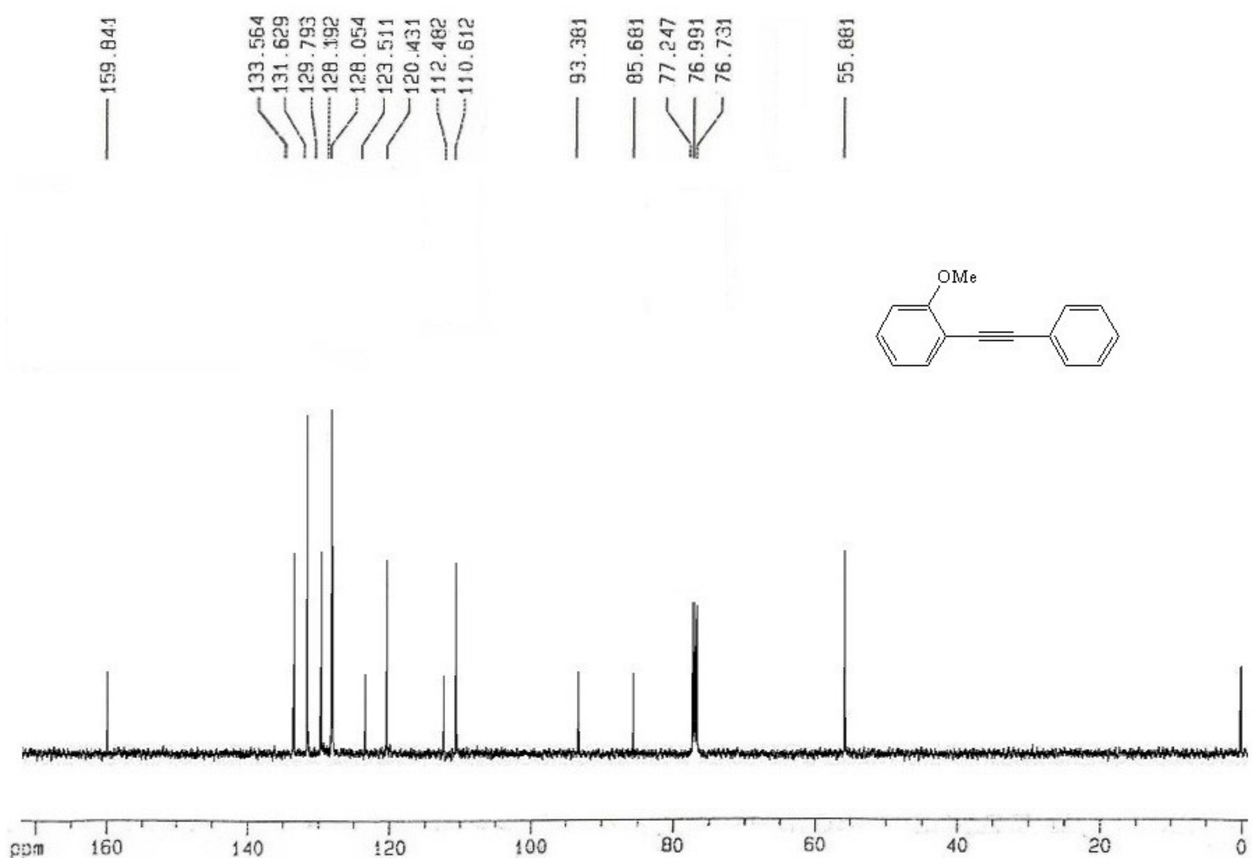

MW=228
File : GHADERI.X10 Date 9/ 7/92 Time 09:22:01
S=[3->22] Bp=228 Bi=3080. RT=0.46 CT=18
100%
75%
50%
25%
228
57
114
202
50 70 90 110 130 150 170 190 210 230 250 270 290
100%
75%
50%
25%
300 320 340 360 380 400 420 440 460 480 500 520 540
SB=50 SE=230 DB=50 DE=510 N=0 Z=2 T=0.0 Fact(->) *1
S List > S=[3->22] B=0 Pos=1 Tot=1

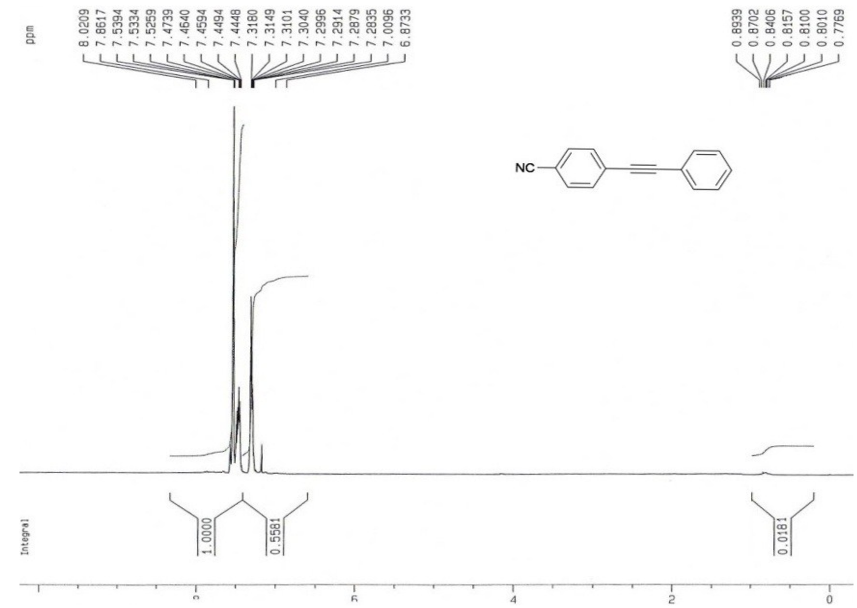

Integral
1.0000
1.0417
1.0637
1.0764
CH_3

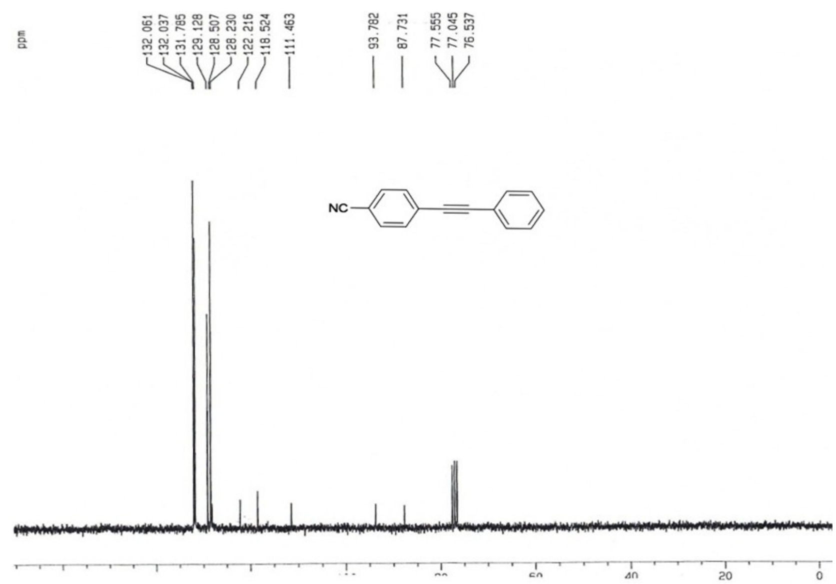

ppm
140.187
131.829
131.507
129.458
128.350
128.301
128.170
125.579
123.010
77.523
77.016
76.507
20.753
CH_3
175
150
125
100
75
50
25
0

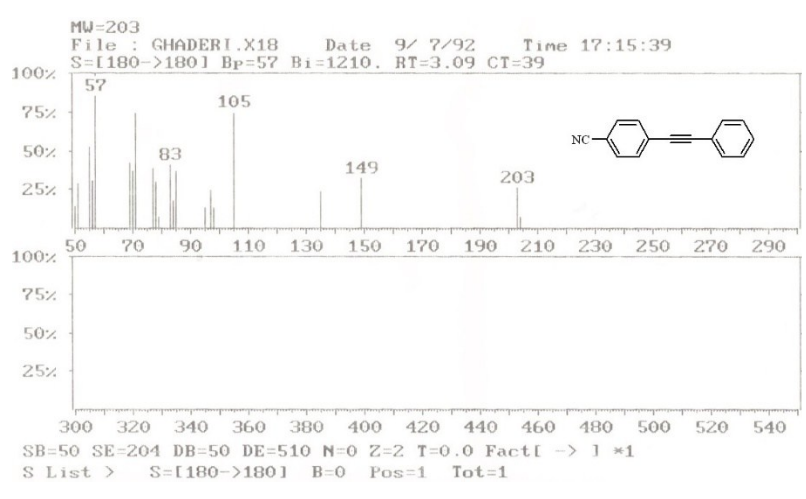

MW=192
File : GHADERI.X09 Date 9/ 7/92 Time 09:15:08
S=[44->44] Bp=57 Bi=3420. RT=0.82 CT=22
57
85
115
149
167
192
Me
SB=54 SE=194 DB=50 DE=510 N=0 Z=2 T=0.0 Fact[->] *1
S List > S=[44->44] B=0 Pos=5 Tot=5

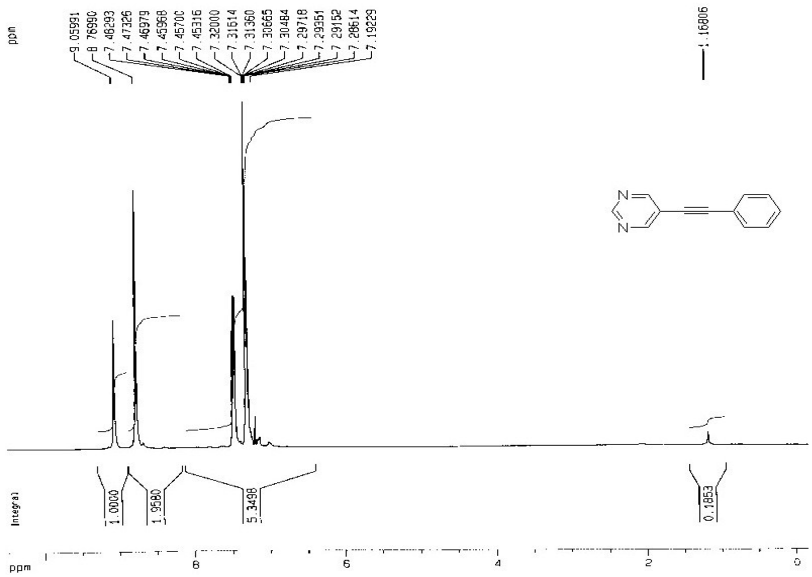

7.5308
7.5301
7.5183
7.5135
7.5073
7.5013
7.4951
7.4867
7.4573
7.3381
7.3303
7.3181
7.3148
7.3103
6.8958
6.8663
3.8331
OMe
Integral
4.3326
3.0341
2.1343
3.0000
ppm
8
6
4
2
0

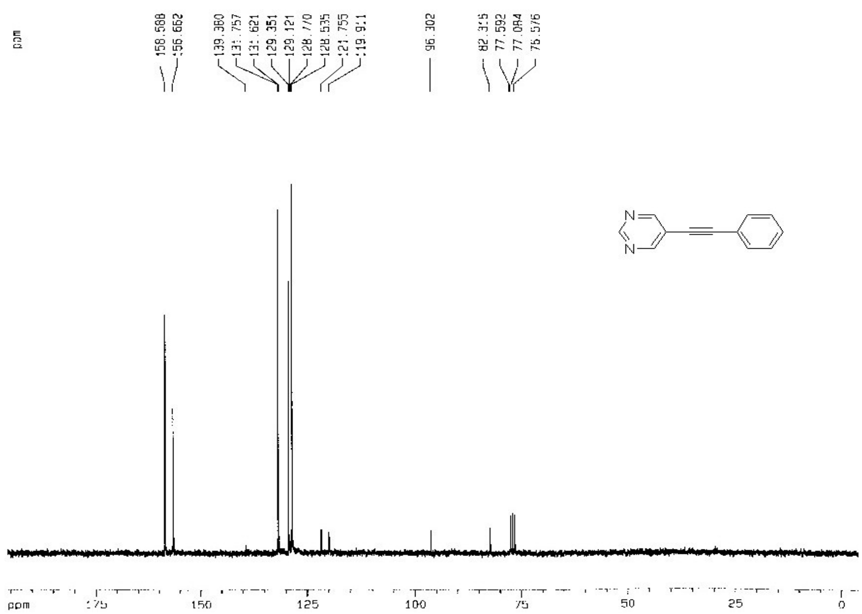

159.844
133.564
131.629
129.793
128.392
128.054
123.511
120.431
112.482
110.612
93.381
85.681
77.247
76.991
76.731
55.881
OMe
ppm
160
140
120
100
80
60
40
20
0

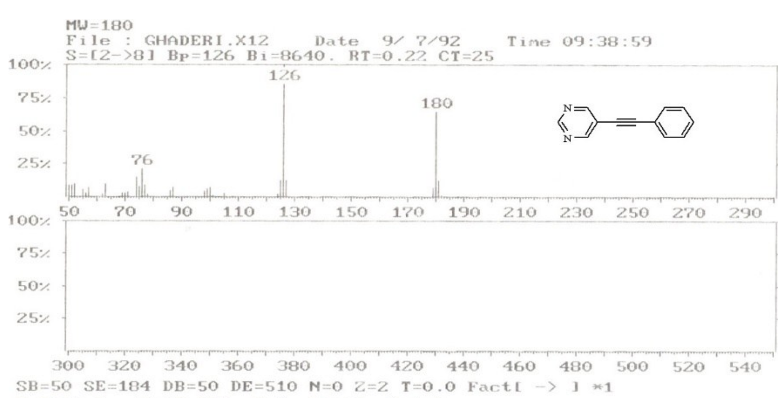

ppm
8.0209
7.8617
7.5394
7.5334
7.5259
7.4739
7.4640
7.4594
7.4494
7.4448
7.3180
7.3149
7.3101
7.3040
7.2996
7.2914
7.2879
7.2835
7.0096
6.8733
0.8939
0.8702
0.8406
0.8157
0.8100
0.8010
0.7769
NC
Integral
1.0000
0.5581
0.0181
4
2
0

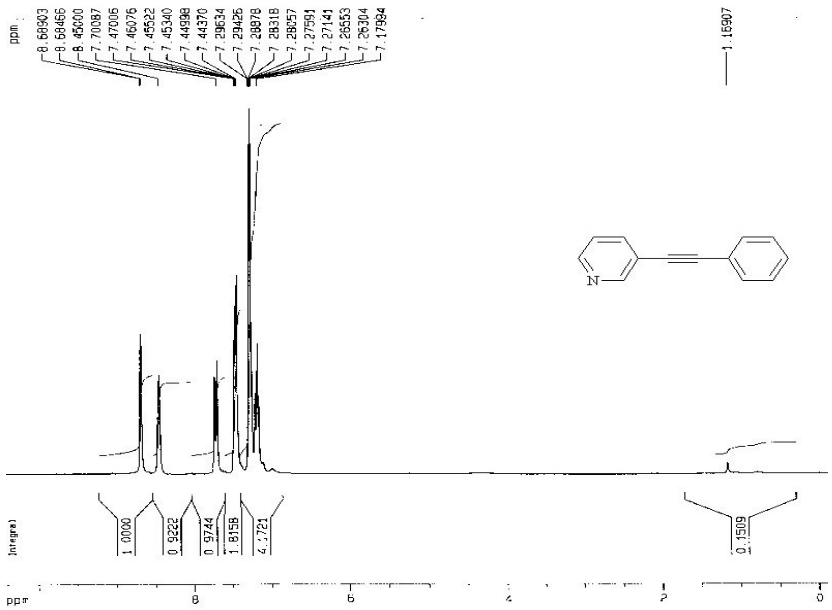

ppm
132.061
132.037
131.785
129.128
128.507
128.230
122.216
118.524
111.463
93.782
87.731
77.555
77.045
76.537
NC
40
20
0

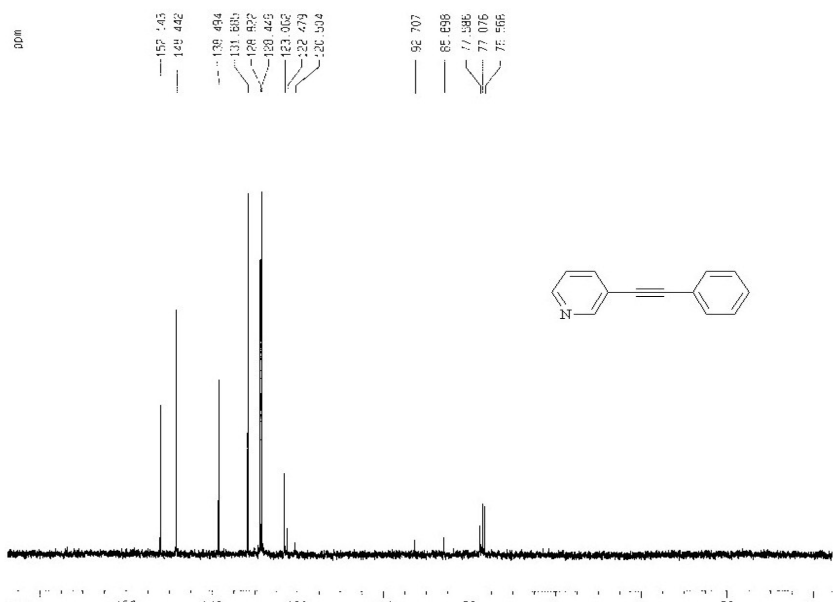

MW=203
File : GHADERI.X18 Date 9/ 7/92 Time 17:15:39
S=[180->180] Bp=57 Bi=1210. RT=3.09 CT=39
100%
75%
50%
25%
57
105
83
149
203
NC
50 70 90 110 130 150 170 190 210 230 250 270 290
300 320 340 360 380 400 420 440 460 480 500 520 540
SB=50 SE=204 DB=50 DE=510 N=0 Z=2 T=0.0 Fact[->] *1
S List > S=[180->180] B=0 Pos=1 Tot=1

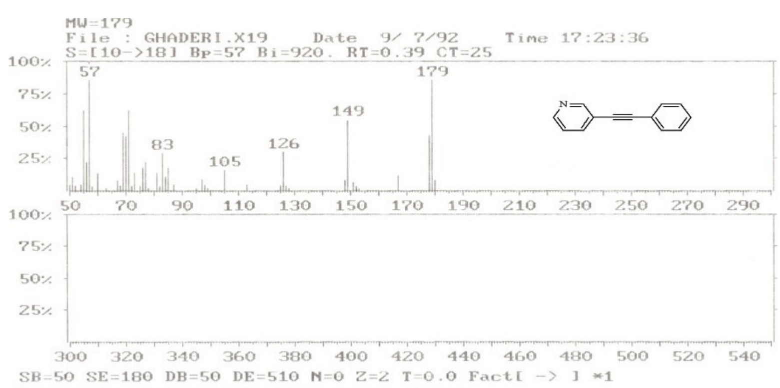

ppm
9.05991
8.76990
7.48293
7.47326
7.46979
7.45968
7.45700
7.45316
7.32000
7.31614
7.31360
7.30665
7.30484
7.29718
7.29351
7.29152
7.28614
7.19229
1.16806
N
N
Integral
1.0000
1.9580
5.3498
0.1853
ppm
8
6
4
2
0

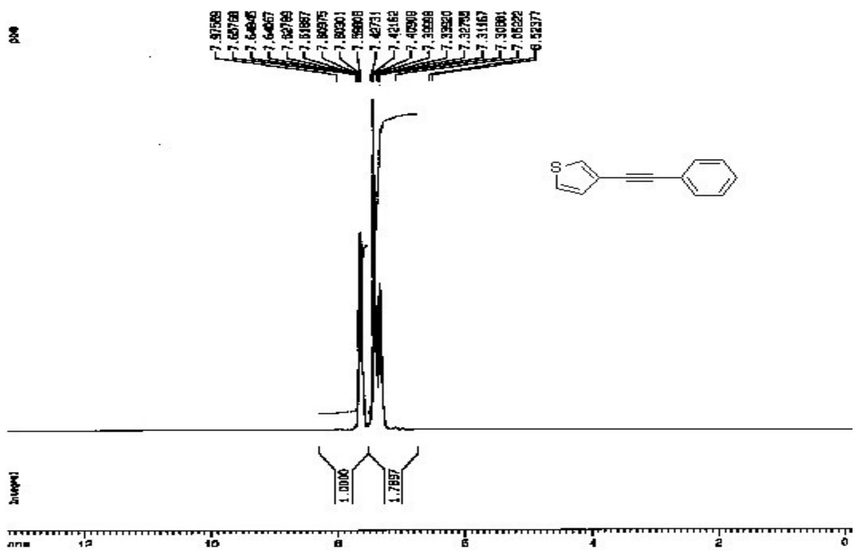

ppm
158.588
156.662
139.360
131.757
131.621
129.351
129.121
128.770
128.535
121.755
119.911
96.302
82.315
77.592
77.084
76.576
N
N
ppm
175
150
125
100
75
50
25
0

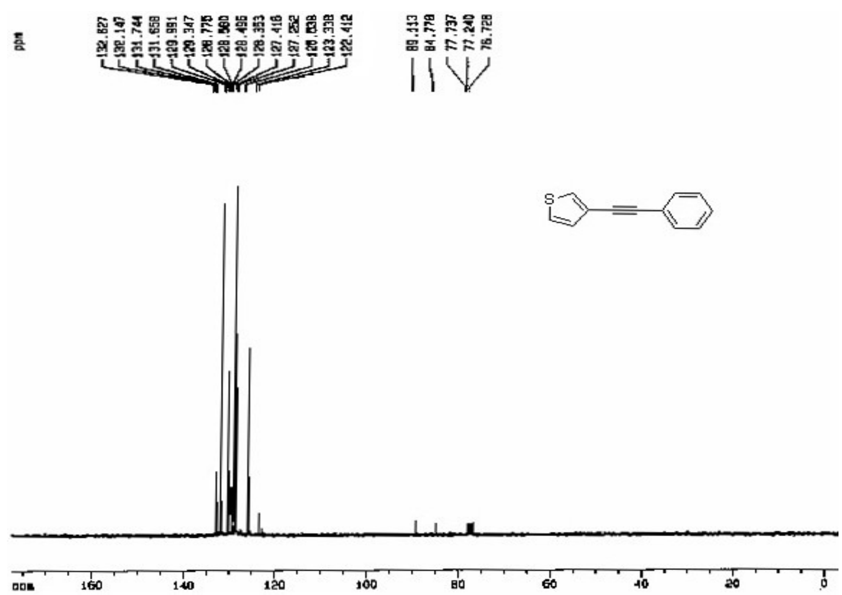

MW=180
File : GHADERI.X12 Date 9/ 7/92 Time 09:38:59
S=[2->8] Bp=126 Bi=8640. RT=0.22 CT=25
100%
75%
50%
25%
126
180
76
N
N
50 70 90 110 130 150 170 190 210 230 250 270 290
100%
75%
50%
25%
300 320 340 360 380 400 420 440 460 480 500 520 540
SB=50 SE=184 DB=50 DE=510 N=0 Z=2 T=0.0 Fact[->] *1
S List > S=[2->8] B=0 Pos=1 Tot=1

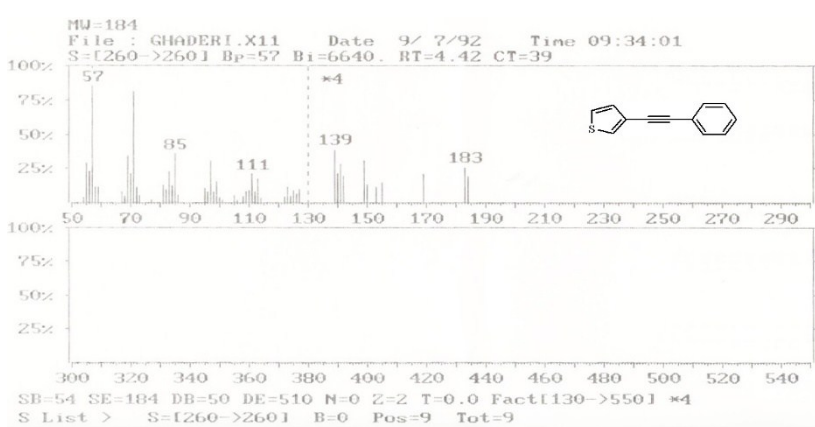

ppm
8.68903
8.68466
8.45600
7.70087
7.47006
7.46076
7.45522
7.45340
7.44998
7.44370
7.29634
7.29426
7.28878
7.28318
7.28057
7.27591
7.27141
7.26553
7.26304
7.17994
1.16907
N
Integral
1.0000
0.9222
0.9744
1.8158
4.1721
0.1509
ppm
8
6
4
2
0

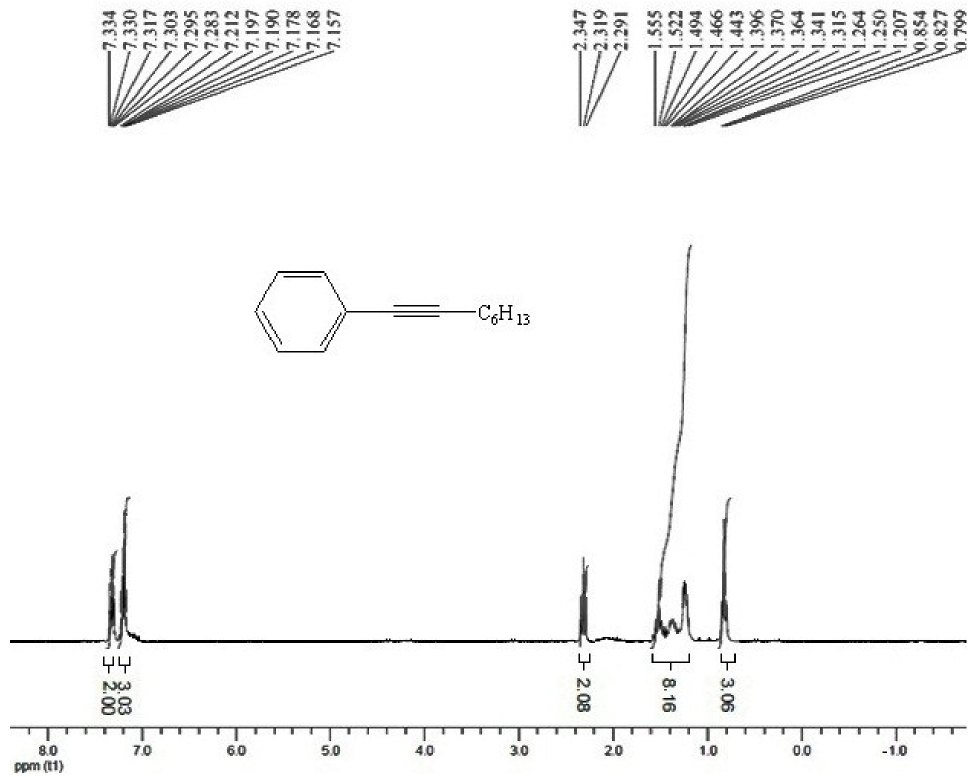

ppm
152.143
149.442
138.494
131.685
128.922
128.449
123.002
122.479
120.504
92.707
85.898
77.386
77.076
76.566
N
ppm
160
140
120
100
80
60
40
20
0

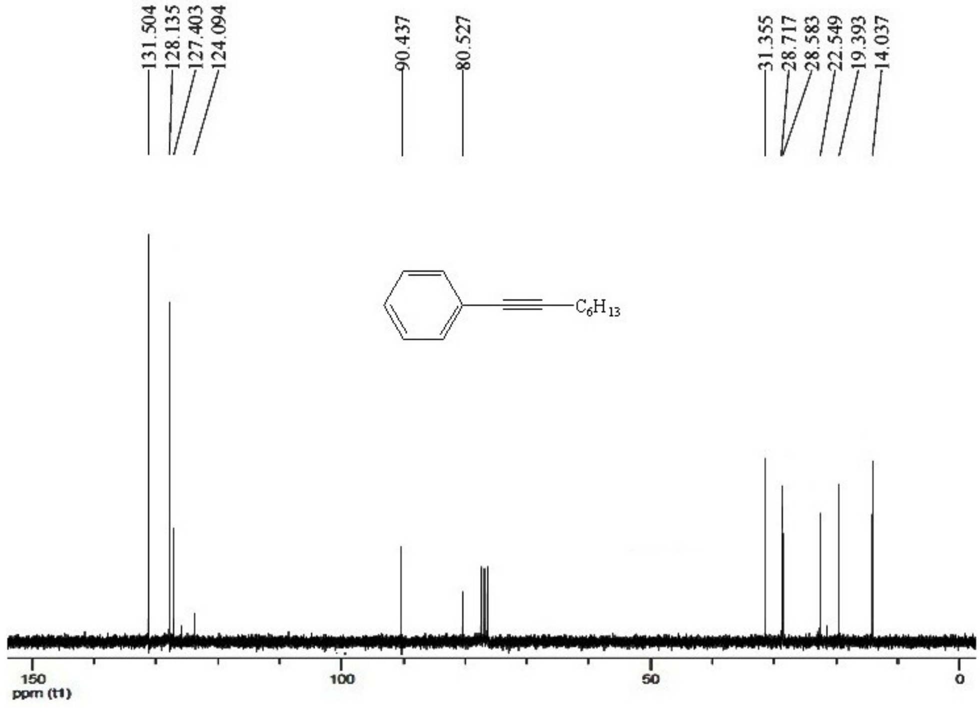

MW=179
File : GHADERI.X19 Date 9/ 7/92 Time 17:23:36
S=[10->18] Bp=57 Bi=920. RT=0.39 CT=25
100%
75%
50%
25%
57
83
105
126
149
179
N
50 70 90 110 130 150 170 190 210 230 250 270 290
100%
75%
50%
25%
300 320 340 360 380 400 420 440 460 480 500 520 540
SB=50 SE=180 DB=50 DE=510 N=0 Z=2 T=0.0 Fact[->] *1
S List > S=[10->18] B=0 Pos=1 Tot=1

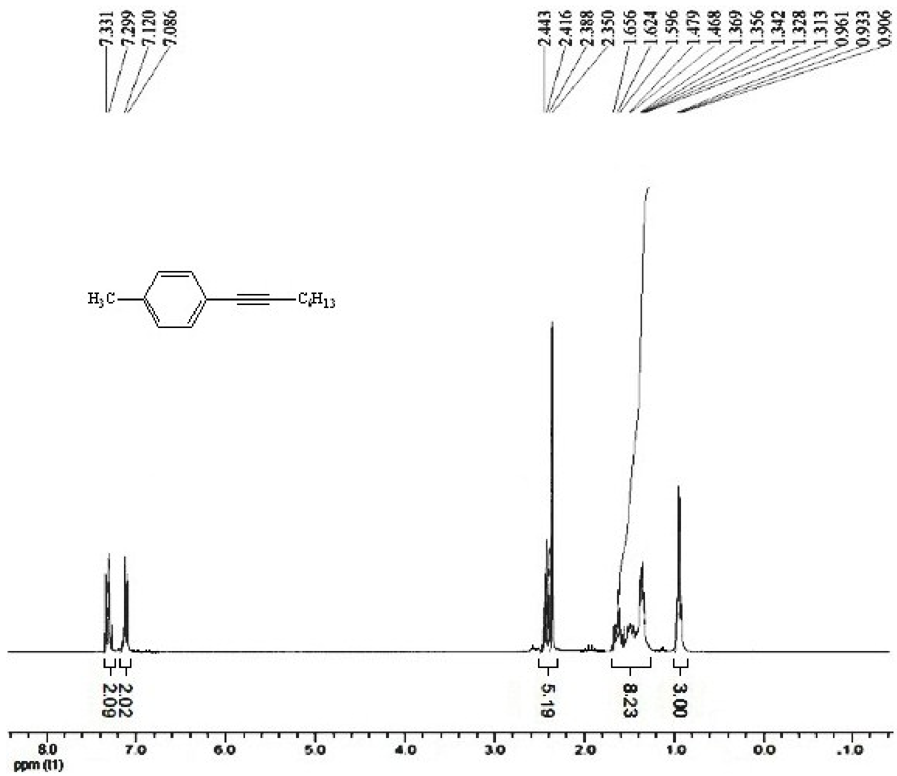


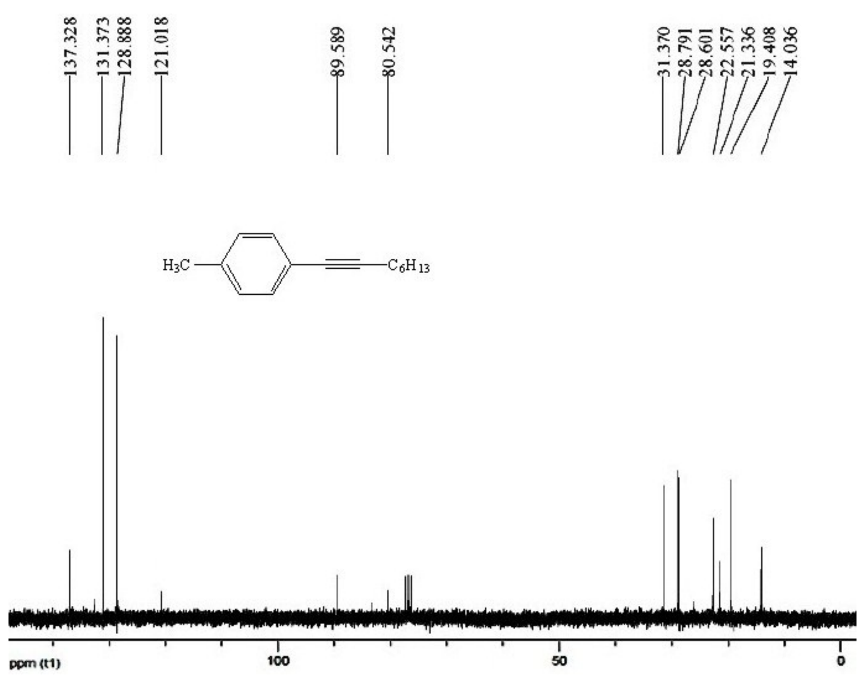


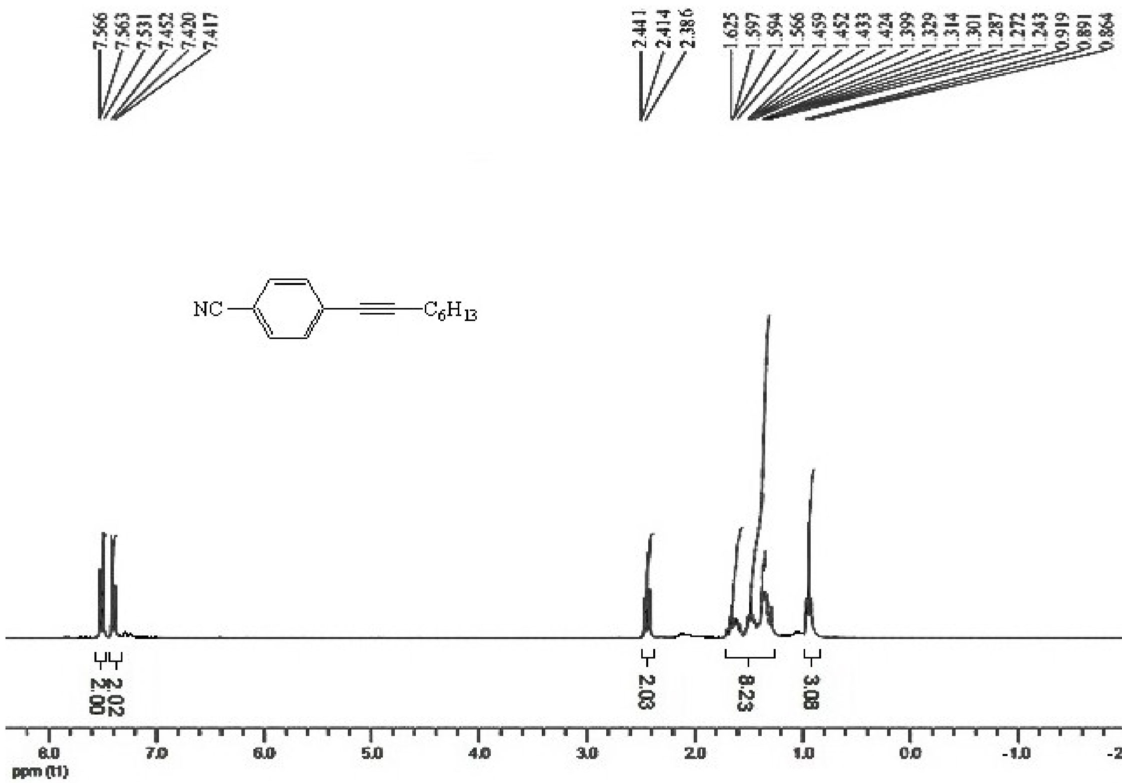

MW=184
File : GHADERI.X11 Date 9/ 7/92 Time 09:34:01
S=[260->260] Bp=57 Bi=6640. RT=4.42 CT=39
SB=54 SE=184 DB=50 DE=510 N=0 Z=2 T=0.0 Fact[130->550] *4
S List > S=[260->260] B=0 Pos=9 Tot=9

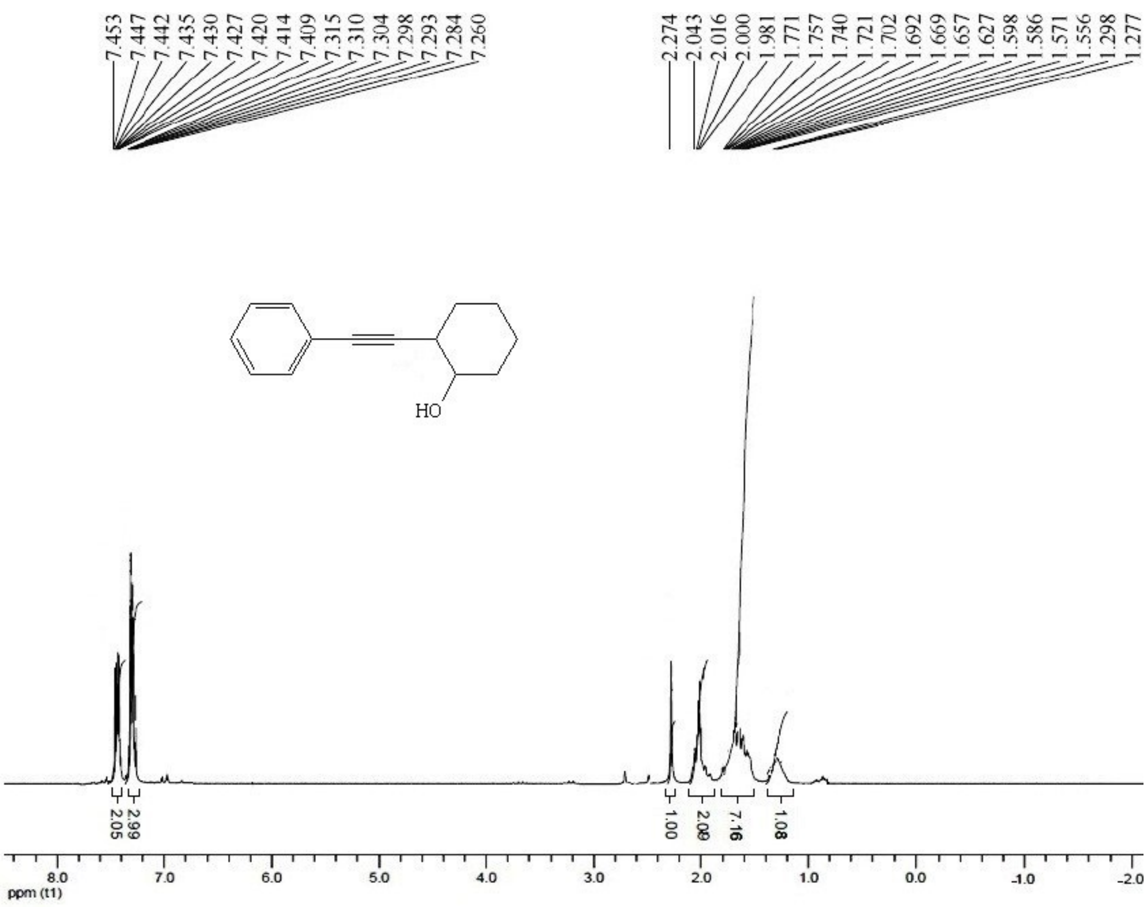

7.334
7.330
7.317
7.303
7.295
7.283
7.212
7.197
7.190
7.178
7.168
7.157
2.347
2.319
2.291
1.555
1.522
1.494
1.466
1.443
1.396
1.370
1.364
1.341
1.315
1.264
1.250
1.207
0.854
0.827
0.799
C_6H_{13}
2.00
3.03
2.08
8.16
3.06
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
ppm (t1)


131.504
128.135
127.403
124.094
90.437
80.527
31.355
28.717
28.583
22.549
19.393
14.037
C_6H_{13}
150
100
50
0
ppm (t1)


7.331
7.299
7.120
7.086
2.443
2.416
2.388
2.350
1.656
1.624
1.596
1.479
1.468
1.369
1.356
1.342
1.328
1.313
0.961
0.933
0.906
H3C
C6H13
2.09
2.02
5.19
8.23
3.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
ppm (t1)


137.328
131.373
128.888
121.018
89.589
80.542
31.370
28.791
28.601
22.557
21.336
19.408
14.036
H3C
C6H13
ppm (t1)
100
50
0


7.566
7.563
7.531
7.452
7.420
7.417
2.441
2.414
2.386
1.625
1.597
1.594
1.566
1.459
1.452
1.433
1.424
1.399
1.329
1.314
1.301
1.287
1.272
1.243
0.919
0.891
0.864
NC
C_6H_{13}
2.00
2.02
2.03
8.23
3.08
8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0 -2.0
ppm (t1)


7.453
7.447
7.442
7.435
7.430
7.427
7.420
7.414
7.409
7.315
7.310
7.304
7.298
7.293
7.284
7.260
2.274
2.043
2.016
2.000
1.981
1.771
1.757
1.740
1.721
1.702
1.692
1.669
1.657
1.627
1.598
1.586
1.571
1.556
1.298
1.277
HO
2.05
2.99
1.00
2.09
7.16
1.08
8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0 -2.0
ppm (t1)