nanomaterials

MDPI

Article

Iron oxide-Cobalt Nanocatalyst for *O-tert*-Boc Protection and *O*-Arylation of Phenols

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Calculations for the size of nanoparticles

In order to build the histogram for the particle size distribution of Fe_3O_4-Co_3O_4, a large number of images (10 to 15) were acquired from each sample (for example, one representative image is shown below Figure S1) and the size of the particles on the border area was measured. It was noted that most of the particles are in the range from 10-30 nm; though the presence of small size nanoparticles cannot be excluded.

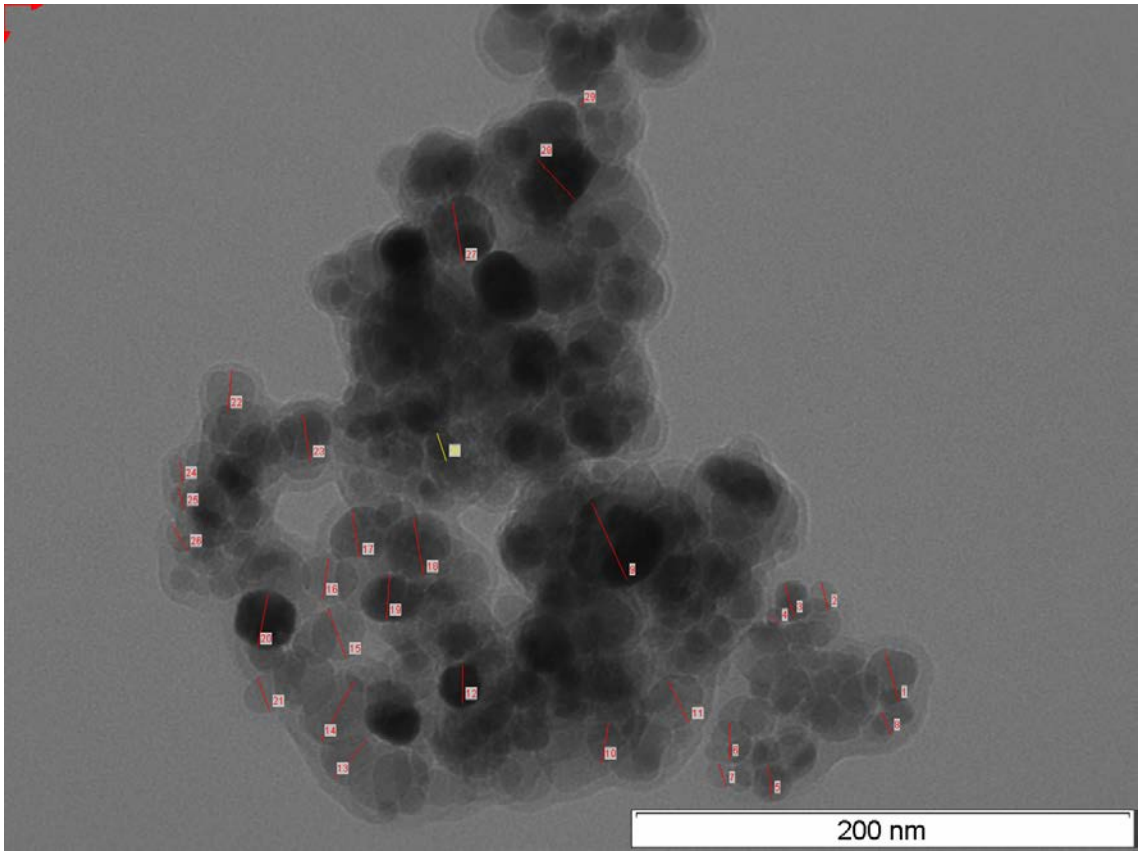


Figure S1. Representative Fe_3O_4-Co_3O_4 TEM image for histogram calculation.

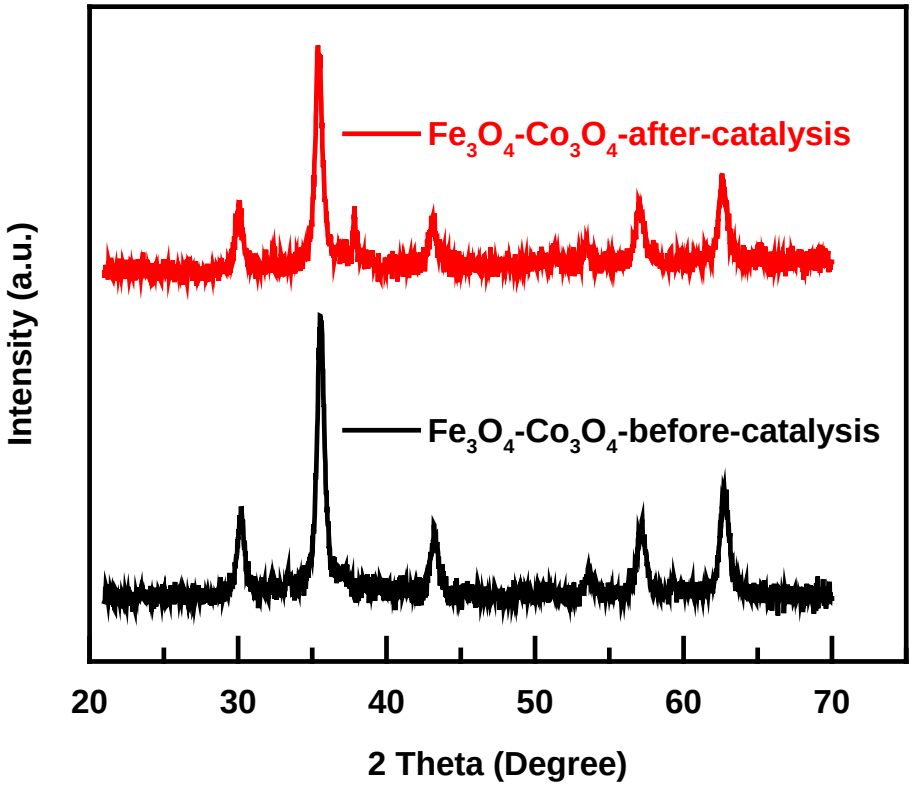
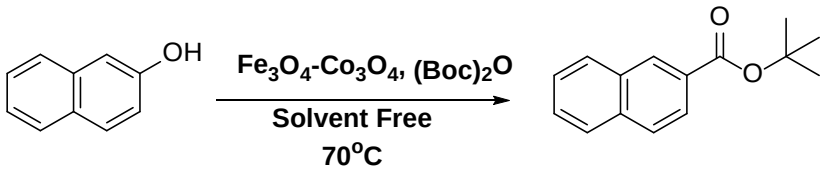


Figure S2. XRD spectra of fresh and reused catalyst.

Standard deviation of catalytic test



1h **2h**

Run	Yield (%)	Standard Deviation (%)
1	85	2.14
2	85	
3	84	
4	82	
5	81	
6	80	

S.D. = √∑Ix-μI²/N-1

X = individual value

μ = mean

N = Number of measurements

μ = 82.83

∑Ix-μI² = 22.8334

N = 6

The comparison of 0.250 g of Fe_3O_4-Co_3O_4 nanocatalyst taken 6 times in parallel for O-*tert*-butoxycarbonylation of 2-naphthol.

Number	Mass of Fe_3O_4-Co_3O_4 Nanocatalysts (mg)[a]	Standard Deviation (mg)
1	250	2.07
2	249	
3	245	
4	245	
5	248	
6	247	

[a]Mass of catalyst after magnetic cleansing, washing and vacuum drying

S.D. = √∑Ix-μI²/N-1

X = individual value

μ = mean

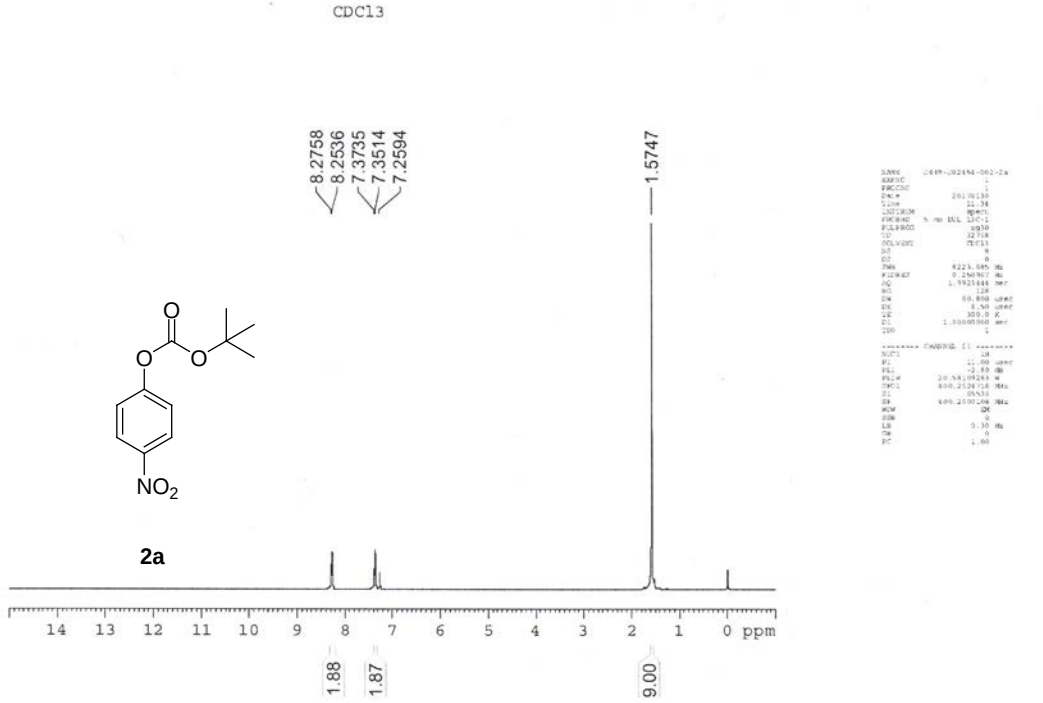
N = Number of measurements

μ = 247.33

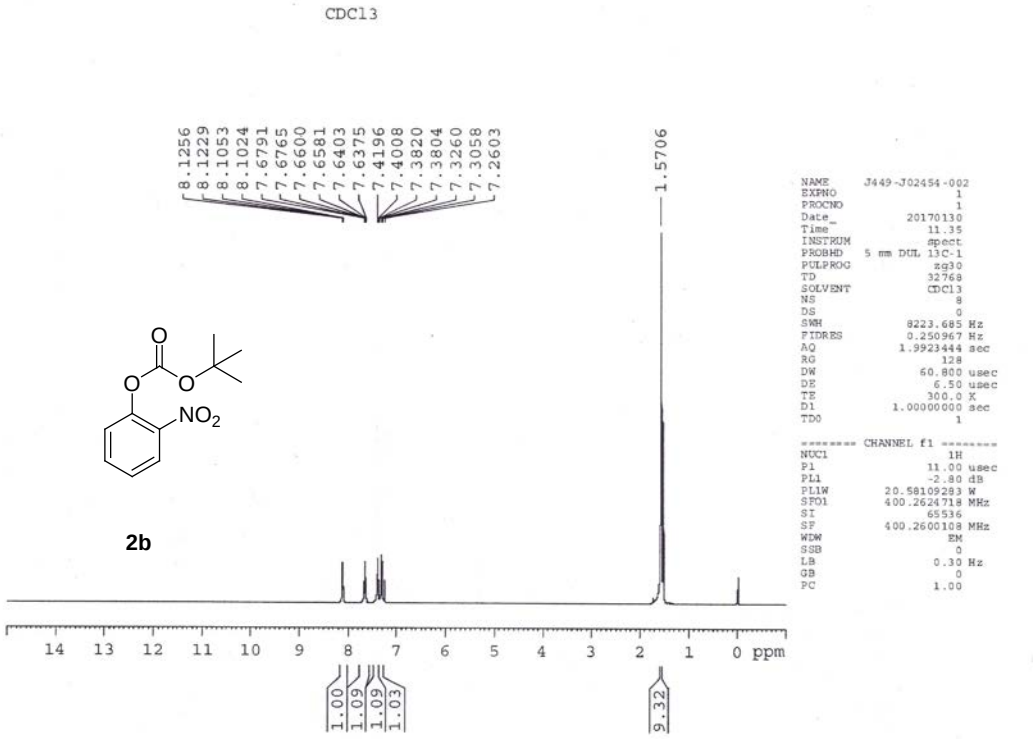
∑Ix-μI² = 21.3334

N = 6

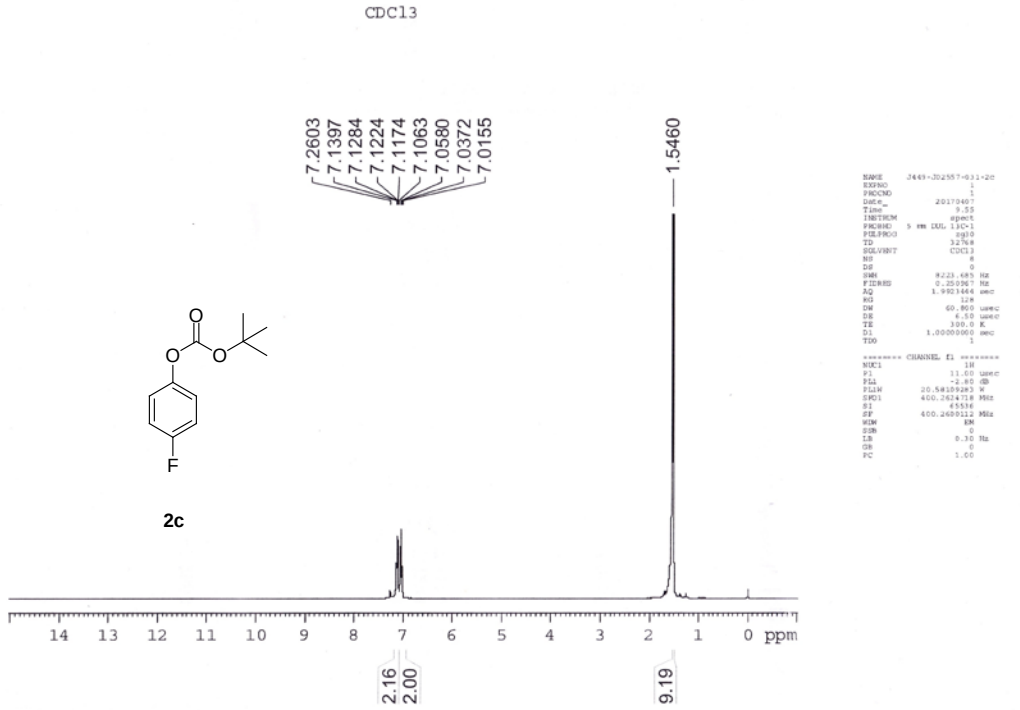
Spectral Data



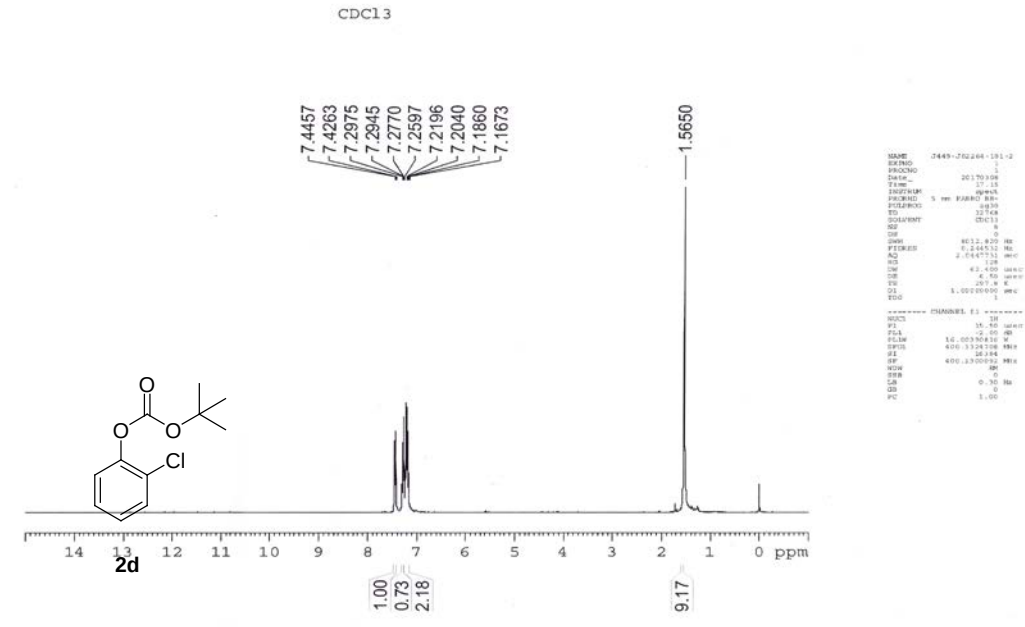
2a. IR (ν, cm^{-1}): 2987.5, 1753.42, 1614.86, 1521.63, 1344.97, ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm): 1.57 (9H, s, CH_3), 7.35-7.37 (2H, d, *J* = 8.8 Hz, HAr), 8.25-8.27 (2H, d, *J* = 8.8 Hz, HAr), LC-MS: 239 [M^+]



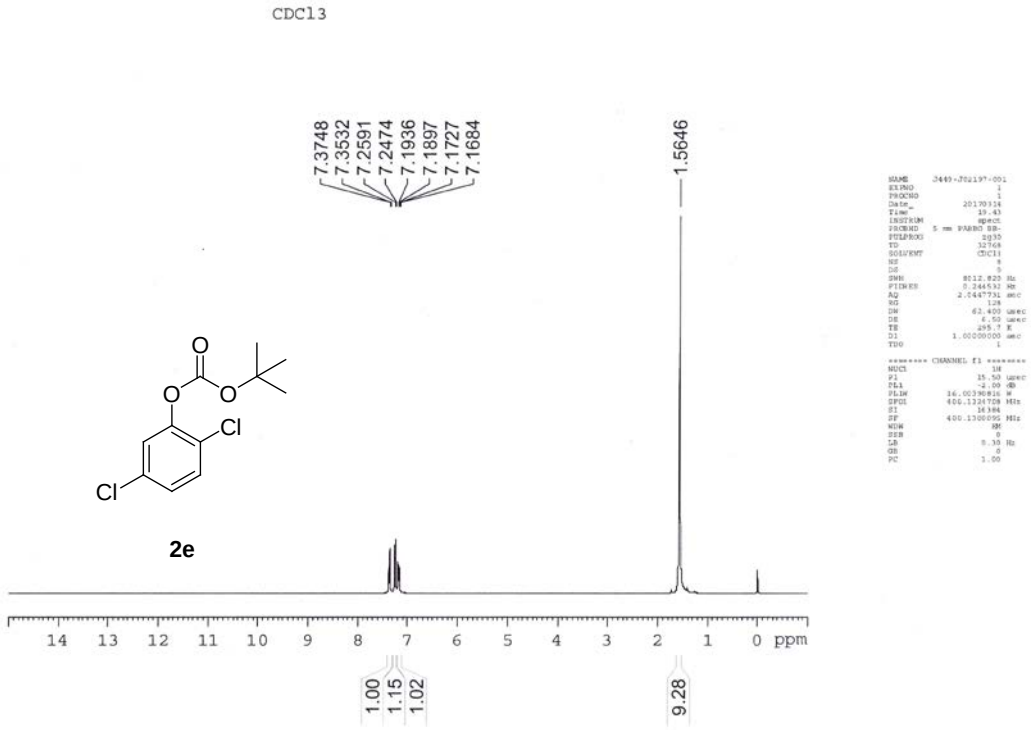
2b. IR (ν, cm^{-1}): 2983.85, 1763.32, 1605.32, 1530.09, 1328.99, ^{1}HNMR (400 MHz, $CDCl_3$, δ, ppm): 1.57 (9H, s, CH_3), 7.30-7.32 (1H, d, *J* = 8.1 Hz, HAr), 7.38-7.41 (1H, t, *J* = 7.5 Hz, HAr), 7.63-7.67 (1H, d, *J* = 1 & 7. 6 Hz, HAr), 8.10-8.12 (1H, dd, *J* = 1 & 8.1 Hz, HAr), LC-MS: 239 [M+1]$^+$



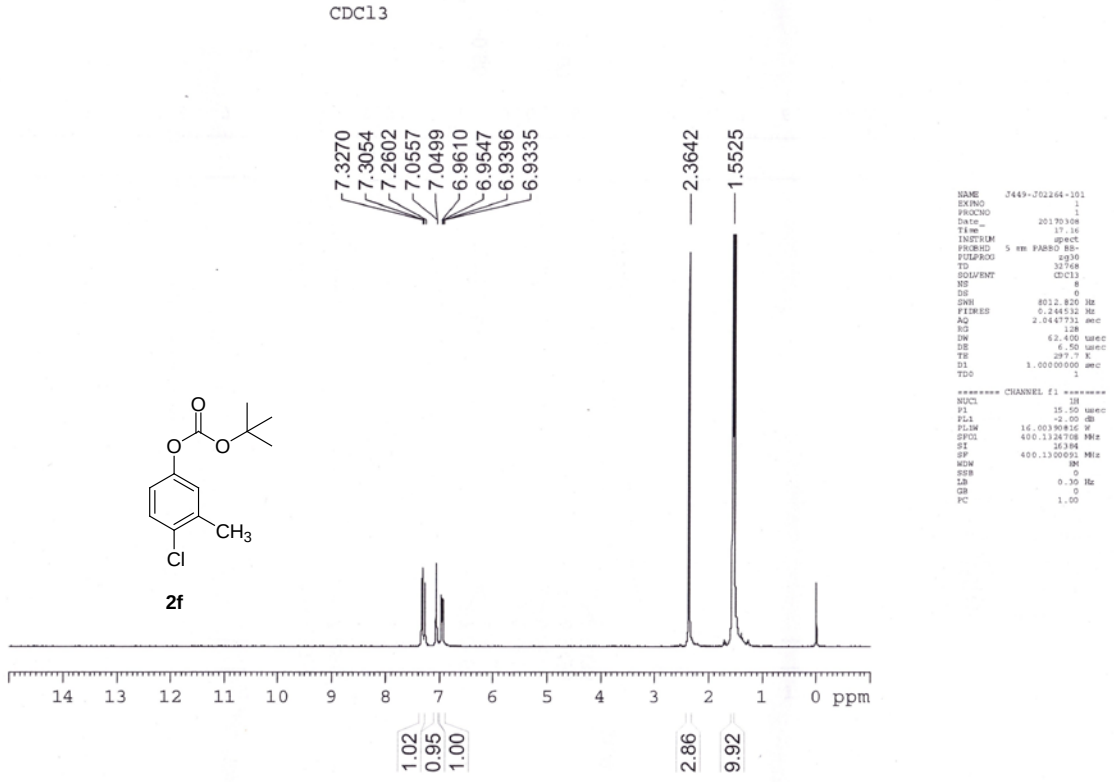
2c. IR (ν, cm^{-1}): 2982.69, 1756.22, 1605, 1505.70, 1371.29, 1141.99, ^{1}HNMR (400 MHz, $CDCl_3$, δ, ppm): 1.54 (9H, s, CH_3), 7.01-7.05 (2H, m, HAr), 7.10-7.13 (2H, m, HAr), LC-MS: 212 $[M+Na]^+$



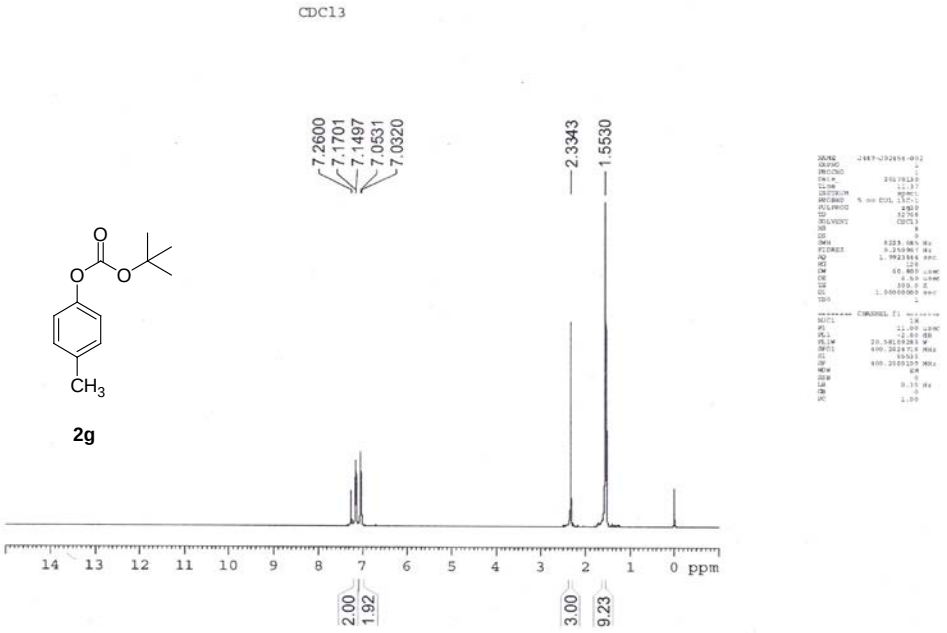
2d. IR (ν, cm^{-1}): 2982.33, 1761.02, 1605, 1251.52, 1062.67, ^{1}HNMR (400 MHz, $CDCl_3$, δ, ppm): 1.56 (9H, s, CH_3), 7.16-7.21 (2H, m, HAr), 7.25-7.29 (1H, m, HAr), 7.42-7.44 (1H, d, *J* = 7.7 Hz, HAr) LC-MS: 228 $[M+18]^+$



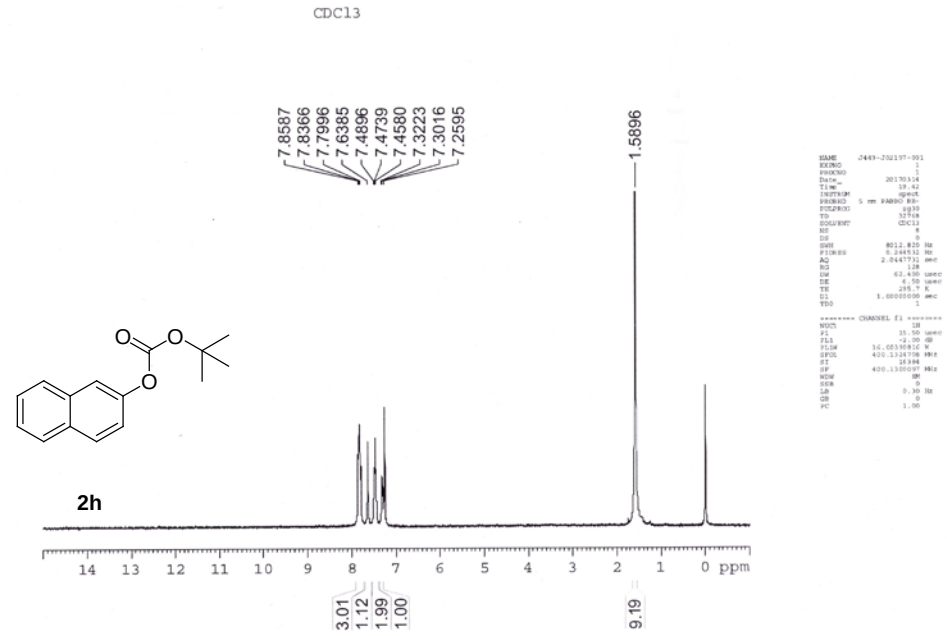
***2e*.** IR (ν, cm^{-1}): 2982.99, 1762.93, 1576.70, 1252.17, 1060.45, ^{1}HNMR (400 MHz, $CDCl_3$, δ, ppm): 1.56 (9H, s, CH_3), 7.16-7.19 (1H, dd, *J* = 1.5 & 8.6 Hz HAr), 7.24 (1H, s, HAr), 7.35-7.37 (1H, d, *J* = 8.6 Hz, HAr), LC-MS: 262 $[M+2]^+$



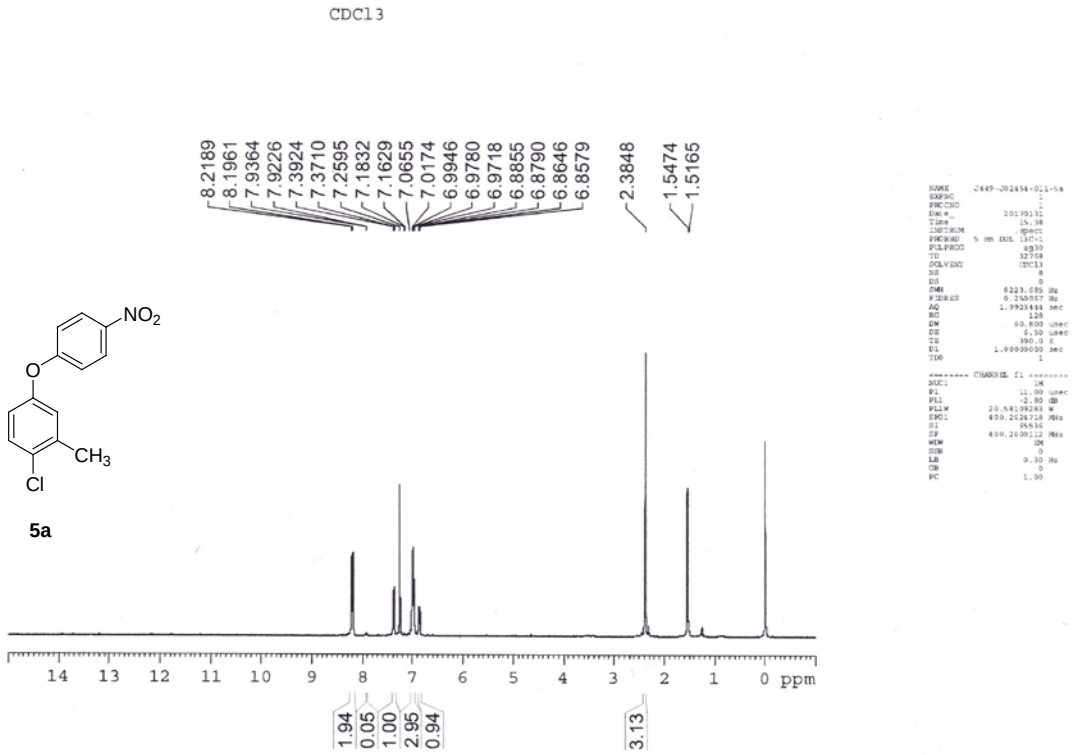
2f. IR (ν, cm^{-1}): 2982.35, 1756.18, 1602, 1250.36, 1060.09, ^{1}HNMR (400 MHz, $CDCl_3$, δ, ppm): 1.55 (9H, s, CH_3), 2.36 (3H, s, CH_3), 6.93-6.96 (1H, dd, *J*= 8.5 & 2.5 Hz HAr), 7.04-7.05 (1H, d, *J*=2.3 Hz, HAr), 7.30-7.32 (1H, d, *J*=8.6 Hz, HAr), LC-MS: 242 $[M+18]^+$



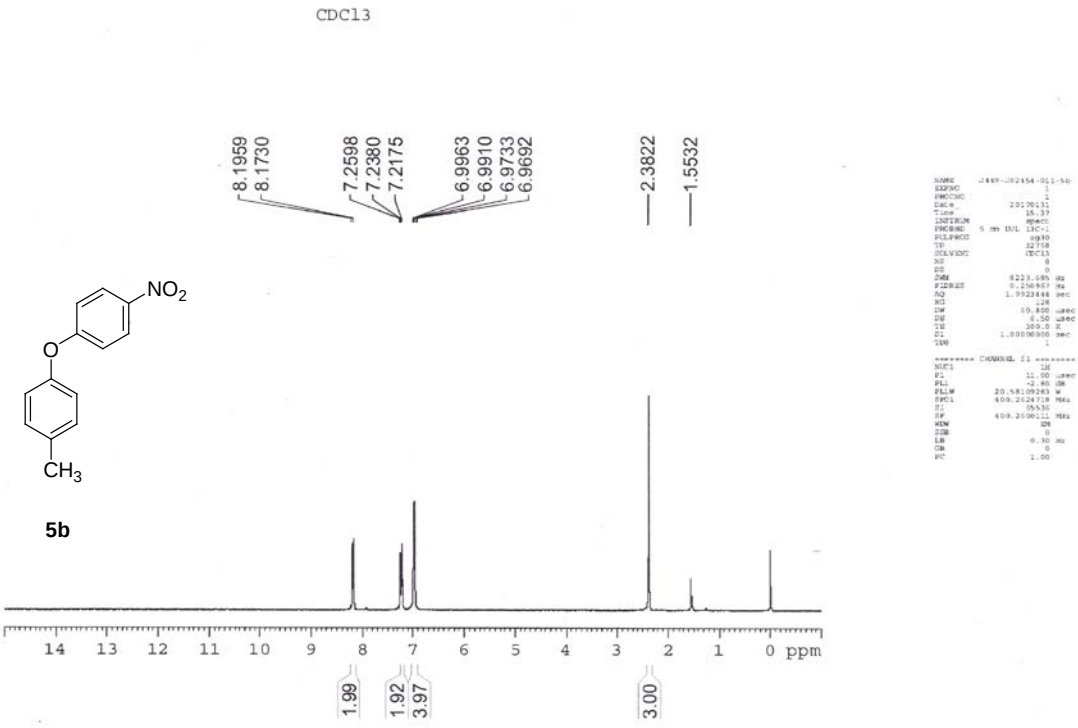
2g. IR (ν, cm^{-1}): 2981.75, 1754.38, 1602, 1254.62, ^{1}HNMR (400 MHz, $CDCl_3$, δ, ppm): 1.55 (9H, s, CH_3), 2.33 (3H, s, CH_3), 7.03-7.05 (2H, d, *J*= 8.4 Hz, HAr), 7.14-7.17 (2H, d, *J*= 8.2 Hz, HAr), LC-MS: 208 $[M+18]^+$



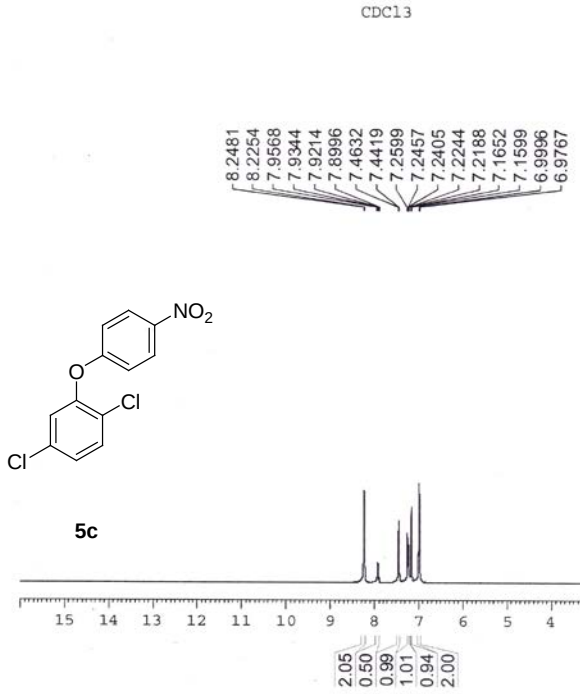
2h. IR (ν, cm^{-1}): 2982.67, 1746.56, 1599.42, 1236.35, ^{1}HNMR (400 MHz, $CDCl_3$, δ, ppm): 1.58 (9H, s, CH_3), 7.30-7.32 (2H, d, *J*= 8.2 Hz, HAr), 7.45-7.48 (2H, m, HAr), 7.63 (1H, S, HAr), 7.79-7.85 (3H, m, HAr), LC-MS: 244 $[M+18]^+$



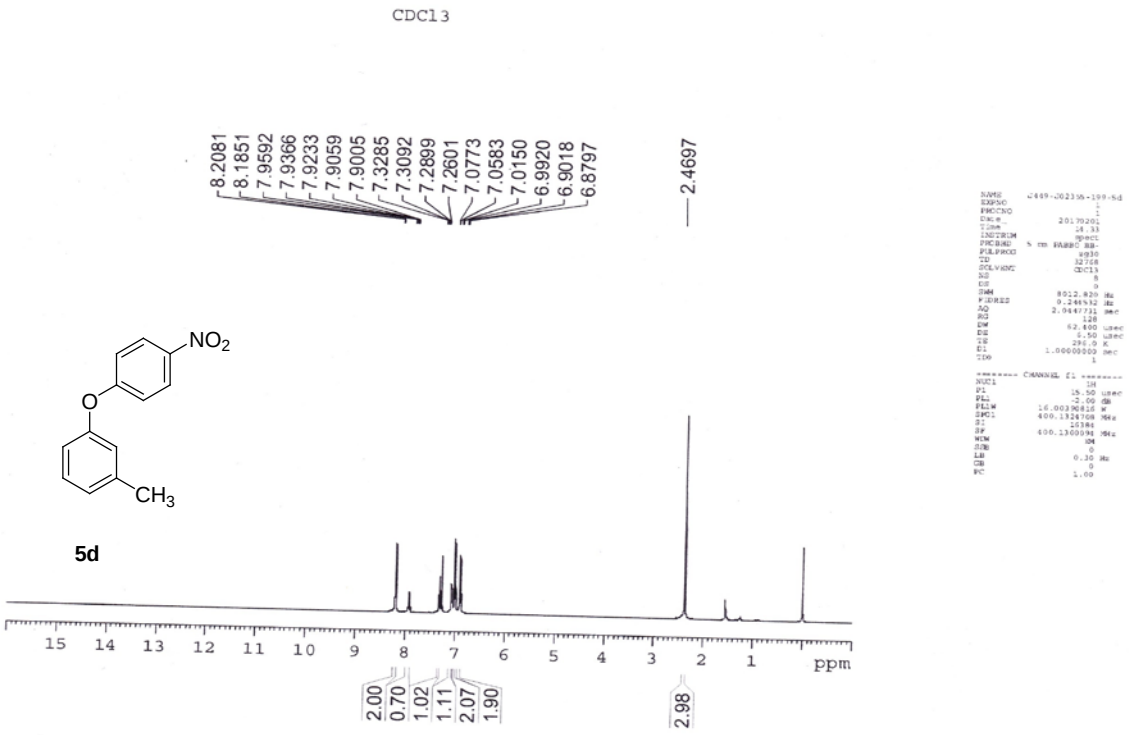
5a. IR (ν, cm^{-1}): 2925.7, 1574.68, 1506.98, 1339.86, 1240.61, ^{1}HNMR (400 MHz, $CDCl_3$, δ, ppm): 2.38 (3H, s, CH_3), 6.85-6.88 (1H, dd, *J* = 2.6 & 8.3 Hz, HAr), 6.97-6.98 (1H, d, *J* =2.4 Hz, HAr), 6.99-7.01 (2H, d, *J* = 9.1 Hz, HAr), 7.37-7.39 (1H, d, *J* = 8.5 Hz, HAr), 8.19-8.21 (2H, d, *J* =9.1 Hz, HAr), LC-MS: 263$[M+1]^+$



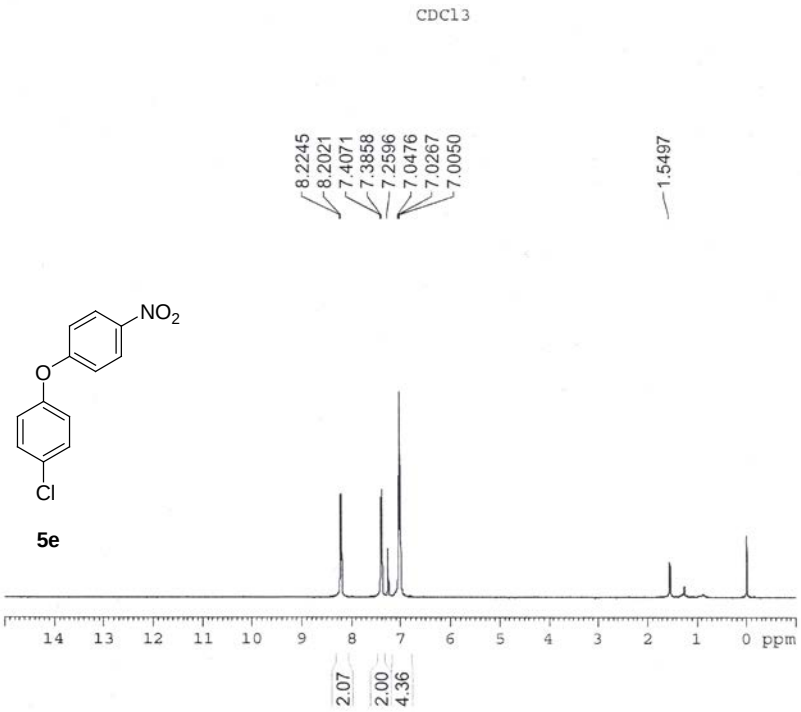
5b. IR (ν, cm^{-1}): 2924, 1587.80, 1505.09, 1338.20, 1243.33, ^{1}HNMR (400 MHz, $CDCl_3$, δ, ppm): 2.38 (3H, s, CH_3), 6.96-6.99 (4H, d, HAr), 7.21-7.23 (2H, d, *J*= 8.2 Hz, HAr), 8.17-8.19 (2H, d, *J* = 9.1 Hz, HAr), LC-MS: 229 $[M-4]^+$



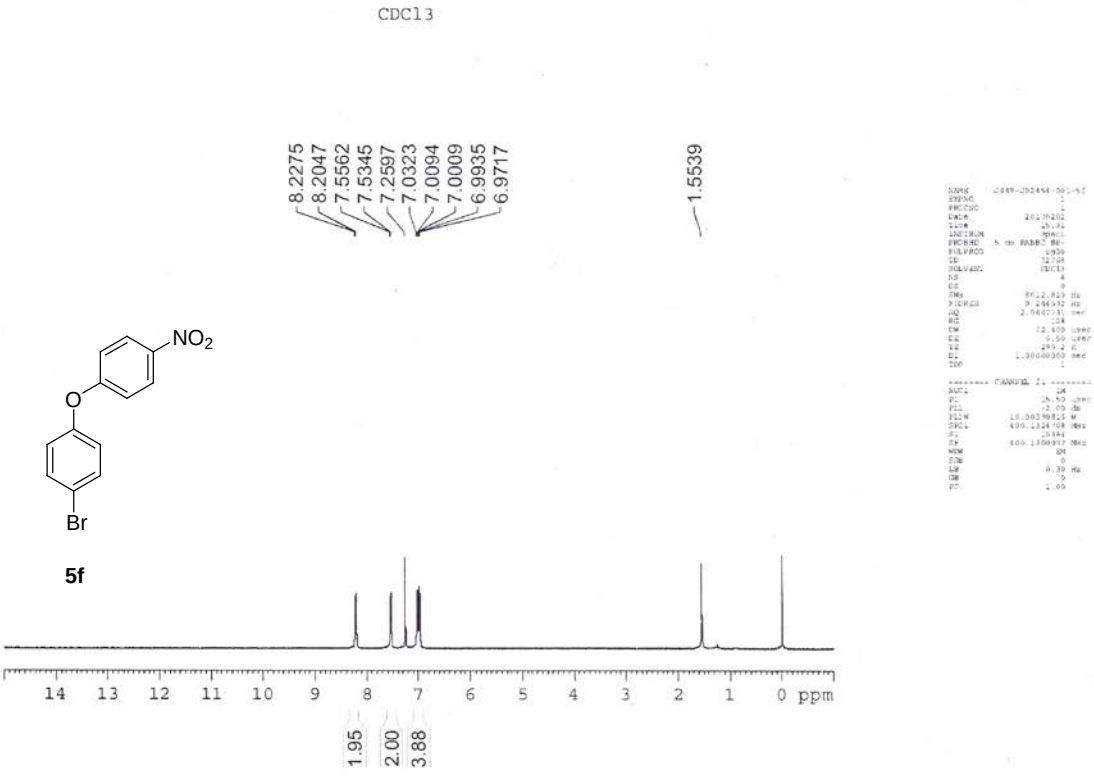
5c. IR (ν, cm^{-1}): 2926.7, 1568.86, 1509.75, 1339.51, 1235.56, 1083.11, ^{1}HNMR (400 MHz, $CDCl_3$, δ, ppm): 6.97-6.99 (2H, d, *J* = 9.1 Hz, HAr), 7.15-7.16 (1H, d, *J* = 2.1 Hz, HAr), 7.21-7.24 (1H, dd, *J* = 2.2 & 8.7 Hz, HAr), 7.44-7.46 (1H, d, *J* = 8.5 Hz, HAr), 8.22-8.24 (2H, d, *J* = 9.1 Hz, HAr), LC-MS: 284 $[M]^+$



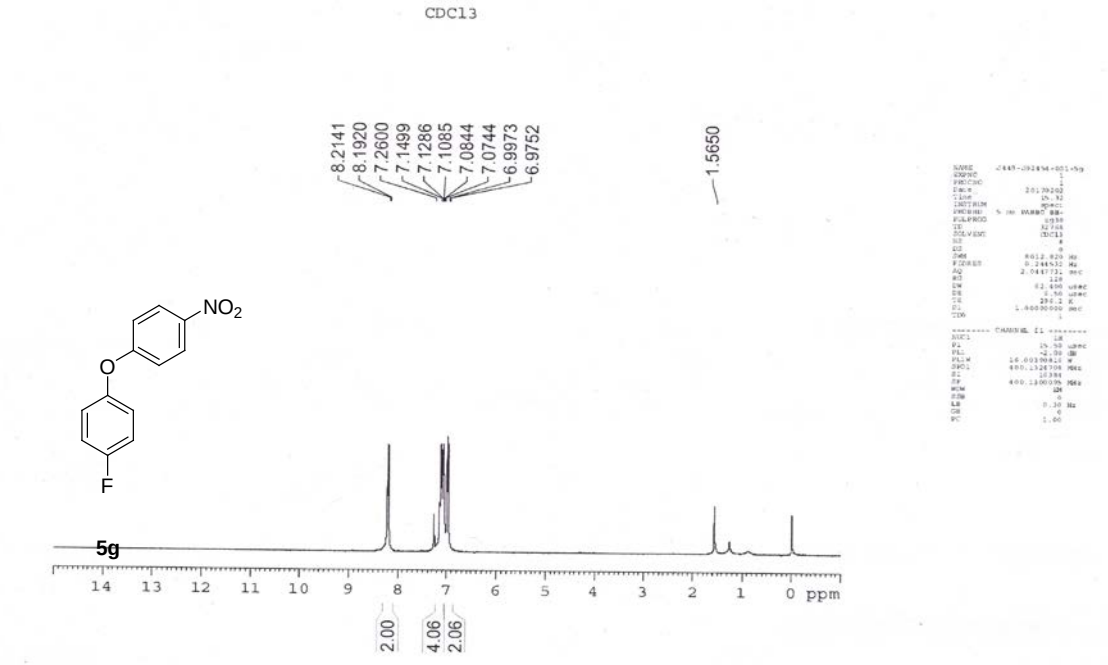
5d. IR (ν, cm^{-1}): 2979.2, 1578.88, 1501.40, 1339.99, 1249.96, ^{1}HNMR (400 MHz, $CDCl_3$, δ, ppm): 2.46 (3H, s, CH_3), 6.87-6.90 (2H, m, HAr), 6.99-7.01 (2H, d, *J*= 9.2 Hz, HAr), 7.05-7.07 (1H, d, *J*= 7.6 Hz, HAr), 7.28-7.32 (1H, t, *J*= 7.7 Hz, HAr), 8.18-8.20 (2H, d, *J*= 9.2 Hz, HAr), LC-MS: 229 $[M+1]^+$



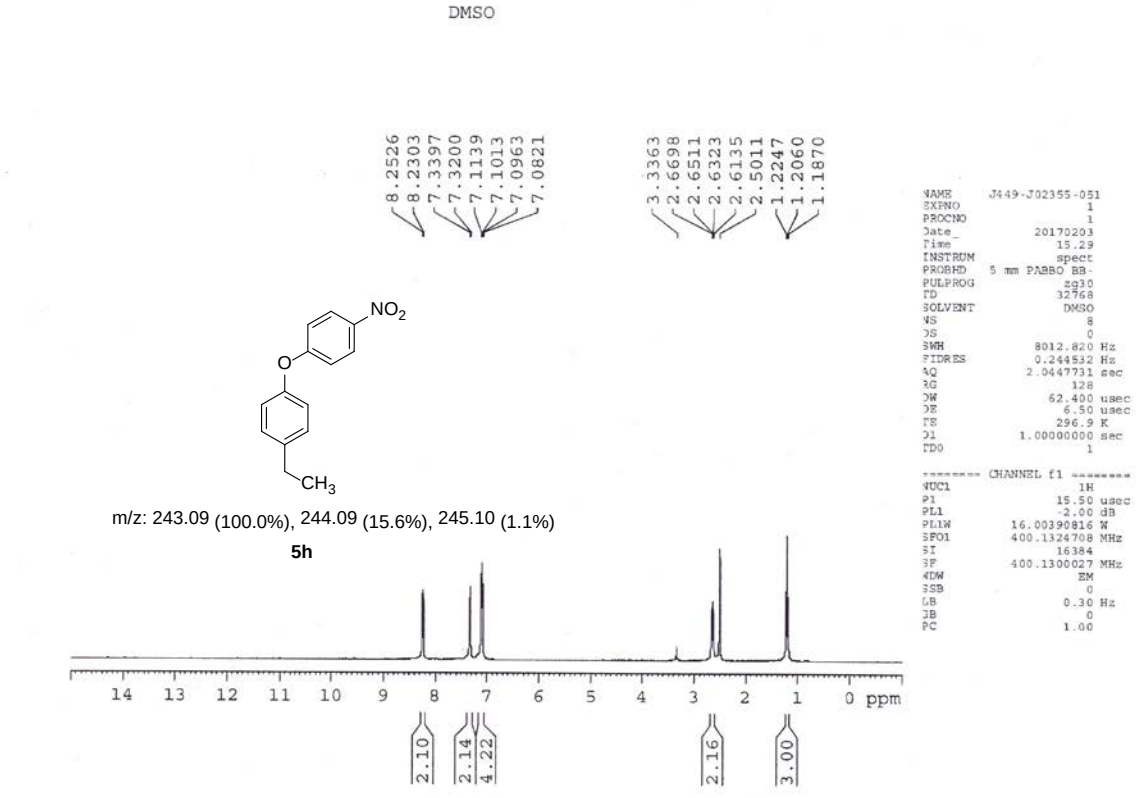
5e. IR (ν, cm^{-1}): 2924, 1610.2, 1598.5, 1510.59, 1340.54, 1244.06, 1088.74, ^{1}HNMR (400 MHz, $CDCl_3$, δ, ppm): 7.00-7.04 (4H, m, HAr), 7.38-7.40 (2H, d, *J* = 8.5 Hz, HAr), 8.20-8.22 (2H, d, *J*= 8.9 Hz, HAr), LC-MS: 249, 250 $[M+1]^+$, $[M+2]^+$



5f. IR (ν, cm^{-1}): 2965.4, 1610, 1509.2, 1339.67, 1241.97, 843.25, ^{1}HNMR (400 MHz, $CDCl_3$, δ, ppm): 6.97-6.99 (2H, d,*J* = 8.7 Hz, HAr), 7.00-7.03 (2H, d, *J* = 9.1 Hz, HAr), 7.53-7.55 (2H, d, *J* = 8.7 Hz, HAr), 8.20-8.22 (2H, d, *J* = 9.1 Hz, HAr), LC-MS: 294 $[M]^+$



5g. IR (ν, cm^{-1}): 2924.62, 1602.71, 1500.65, 1336.94, 1185.39, 1215.03, ^{1}HNMR (400 MHz, $CDCl_3$, δ, ppm): 6.97-6.99 (2H, d, *J* = 8.8 Hz, HAr), 7.07-7.14 (4H, m, HAr), 8.19-8.21 (2H, d, *J* = 8.8 Hz, HAr), LC-MS: 233 $[M-1]^+$



5h. IR (ν, cm^{-1}): 2966.09, 1609.13, 1587.23, 1505. 63, 1340.17, 1242.03, ^{1}HNMR (400 MHz, DMSO-d_6, δ, ppm): 1.18-1.22 (3H, t, *J* = 7.6 Hz, CH_3), 2.61-2.66 (2H, q, *J* = 7.5 Hz, CH_2), 7.08-7.11 (4H, m, HAr), 7.32-7.33 (2H, d, *J* = 7.9 Hz, HAr), 8.23-8.25 (2H, d, *J* = 8.9 Hz, HAr), LC-MS: 243 $[M-1]^+$