SUPPORTING INFORMATION

for

Mechanochemical Indole Synthesis by Rhodium-Catalysed Oxidative Coupling of Acetanilides and Alkynes under Solventless Conditions in a Ball Mill

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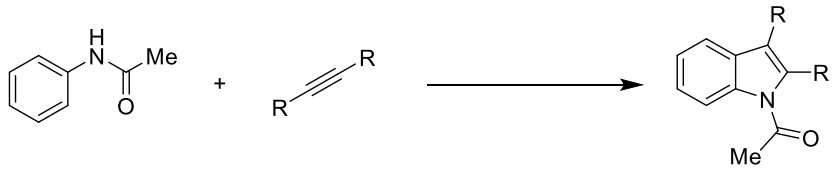
General experimental

Unless otherwise reported, all reactions were performed using grinding vessels in a FRITSCH Planetary micro mill model „Pulverisette 7 premium line“ under an atmosphere of dioxygen. Both vessels (20 mL) and balls (5 mm diameter) were made of ZrO_2. The product mixtures were analysed by thin layer chromatography using TLC silica gel plates (Merck-Schuchardt) with fluorescent indicator (λ = 254 nm). Flash column chromatography was undertaken on silica gel (Acros, 35-70 μm, 60 Å). NMR spectra were recorded on a Varian V-NMRS 600 or a Varian V-NMRS 400 in deuterated chloroform at 25 °C. Chemical shifts (δ) are quoted in ppm, and spin-spin coupling constants (*J*) are reported in Hz, while multiplicities are given by the standard abbreviations: br s (broad singlet), s (singlet), d (doublet), t (triplet), q (quartet) and m (multiplet). Mass spectra were recorded on a Finnigan SSQ 7000 mass spectrometer. IR-Spectra were recorded on a Perkin Elmer 100 FT/IR spectrometer, while the wave numbers of the absorption peaks are given in cm^{-1}. The acetanilides and $[Cp^*Rh(MeCN)_3][SbF_6]_2$ were prepared in accordance with previously published synthetic strategies.[1,2]

Relevant physical properties: N-Phenylacetamide (**1a**): mp = 113 – 116 °C;[3] N-(4-fluorophenyl)acetamide (**1b**): mp = 153 – 155 °C;[4] N-(4-methoxyphenyl)acetamide (**1c**): mp = 130 – 133 °C;[5] N-(*m*-tolyl)acetamide (**1d**): mp = 64 – 66 °C;[6] N-(*o*-tolyl)acetamide (**1e**): mp = 111 – 113 °C;[7] N-(2-methoxyphenyl)acetamide (**1f**): mp = 87 – 90 °C;[4] 1,2-diphenylethyne (**2a**): mp = 59 – 61 °C;[8] prop-1-yn-1-ylbenzene (**2g**): liquid; but-1-yn-1-ylbenzene (**2h**): liquid; pent-1-yn-1-ylbenzene (**2i**): liquid; oct-4-yne (**2j**): liquid.

Optimisation of the reaction conditions[a]



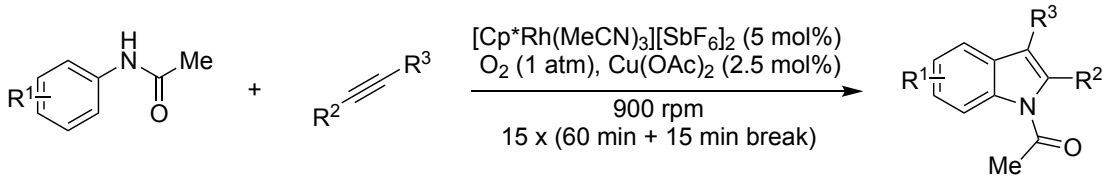
1a **2** **3**

Entry	Cat.	Cat. (mol%)	Redox modulator	Redox modulator (mol%)	**1a** (equiv.)	**2** (equiv.)	R	Time (min.)	Milling balls (mm)	Yield [%][b]
1[c]	$[\{Cp^*Rh(Cl)_2\}_2]$	2.5	$Cu(OAc)_2$	210	1.0	1.1	*n*Pr	15 x 60	16 (5)	6
2[c]	$[\{Cp^*Ir(Cl)_2\}_2]$	2.5	$Cu(OAc)_2$	210	1.0	1.1	*n*Pr	15 x 60	16 (5)	2
3[c]	$[\{Ru(p\text{-cymen})Cl_2\}_2]$	2.5	$Cu(OAc)_2$	210	1.0	1.1	*n*Pr	15 x 60	16 (5)	1
4	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Cu(OAc)_2$	210	1.0	1.1	*n*Pr	15 x 60	16 (5)	27
5	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Cu(OAc)_2$	210	1.0	1.5	Ph	15 x 60	16 (5)	27
6	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Cu(OAc)_2$	210	1.0	2.0	Ph	15 x 60	16 (5)	29
7	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Cu(OAc)_2$	210	1.0	3.0	Ph	15 x 60	16 (5)	37
8	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Cu(OAc)_2$	210	1.0	3.0	Ph	10 x 60	16 (5)	16
9	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Cu(OAc)_2$	210	1.0	3.0	Ph	20 x 60	16 (5)	37
10	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Cu(OAc)_2$	210	1.0	3.0	Ph	15 x 60	30 (5)	42
11	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Cu(OAc)_2$	210	1.0	3.0	Ph	15 x 60	50 (5)	36
12	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Cu(OAc)_2$	210	1.0	3.0	Ph	15 x 60	7 (10)	24
13	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Cu(OAc)_2$	210	1.0	3.0	Ph	15 x 60	1 (15)	-
14	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Cu(OAc)_2$	210	2.0	1.0	Ph	15 x 60	30 (5)	41
15	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Cu(OAc)_2$	210	3.0	1.0	Ph	15 x 60	30 (5)	48
16	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Fe(OAc)_2$	210	3.0	1.0	Ph	15 x 60	30 (5)	5
17	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	NaOAc	210	3.0	1.0	Ph	15 x 60	30 (5)	9
18	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Cu(OAc)_2$	105	3.0	1.0	Ph	15 x 60	30 (5)	29
19	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Cu(OAc)_2$	53	3.0	1.0	Ph	15 x 60	30 (5)	27
20[d]	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Cu(OAc)_2$	2.5	3.0	1.0	Ph	15 x 60	30 (5)	59
21[d]	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Cu(OAc)_2$	2.5	2.0	1.0	Ph	15 x 60	30 (5)	60
22[d]	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Cu(OAc)_2$	2.5	1.0	2.0	Ph	15 x 60	30 (5)	68
23[d]	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Cu(OAc)_2$	2.5	1.0	3.0	Ph	15 x 60	30 (5)	68
24[d,e]	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Cu(OAc)_2$	2.5	1.0	2.0	Ph	15 x 60	30 (5)	76
25[d,e]	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	10.0	$Cu(OAc)_2$	2.5	1.0	2.0	Ph	15 x 60	30 (5)	76
26[d,e]	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	2.5	$Cu(OAc)_2$	2.5	1.0	2.0	Ph	15 x 60	30 (5)	19
27[d,e]	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Cu(OAc)_2$	2.5	1.0	2.0	Ph	18 x 60	30 (5)	76
28[d,e]	$[Cp^*Rh(MeCN)_3][SbF_6]_2$	5.0	$Cu(OAc)_2$	2.5	1.0	2.0	Ph	10 x 60	30 (5)	65

[a] Reaction conditions: 800 rpm, pulverisette 7 premium line, both vessels (20 mL) and balls (5 mm diameter) were made of ZrO_2. [b] Yield determined by ^{1}H-NMR spectroscopy with 1,3,5-trimethoxybenzene as an internal standard. [c] Use of $AgBF_4$ (0.012 g, 0.06 mmol, 10 mol%). [d] Carried out under an atmosphere of dioxygen. [e] Carried out at 900 rpm.

General procedure

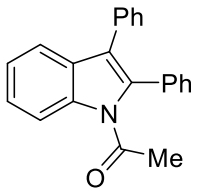
General Procedure: Mechanochemical indole synthesis.



1 **2** **3**

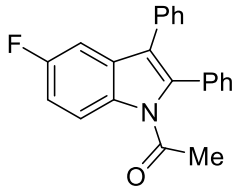
Acetanilide (**1**, 0.6 mmol), alkyne (**2**, 2.0 equiv.), $[Cp^*Rh(MeCN)_3][SbF_6]_2$ (5.0 mol%) and $Cu(OAc)_2$ (2.5 mol%) were transferred to a ball milling vessel (ZrO_2, 20 mL) loaded with 30 grinding balls (ZrO_2, diameter: 5 mm). The ball milling vessel was flushed with dioxygen and placed in the ball mill (milling cycle: 60 min followed by 15 min break, repetitions: 15). The crude was recovered by washing the vessel and balls with EtOAc (5 x 20 mL). Then, the mixture was filtered through a thin layer of SiO_2, concentrated *in vacuo* and purified by flash chromatography (SiO_2, *n*pentane/Et_2O).

1-(2,3-Diphenyl-1*H*-indol-1-yl)ethan-1-one (3a)



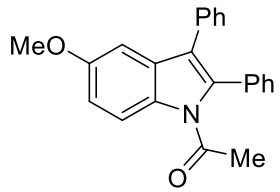
General procedure was followed to afford the title compound as a yellow solid (131.0 mg, 0.42 mmol, 70%). ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.51 (d, J = 8.4 Hz, 1H), 7.59 (d, J = 7.7 Hz, 1H), 7.46–7.24 (m, 12H), 2.03 (s, 3H). $^{13}C\{^1H\}$-NMR (101 MHz, $CDCl_3$): δ = 171.6, 136.9, 135.1, 133.1, 133.0, 130.9, 130.1, 129.3, 128.7, 128.7, 128.3, 127.0, 125.6, 123.9, 123.4, 119.6, 116.3, 28.0. MS (EI^+, 70 eV) *m/z* (%): 312 (22), 311 (76), 270 (25), 269 (100), 268 (24), 267 (26). MS (CI^+, methane) *m/z* (%): 340 (12), 313 (24), 312 (100), 311. IR: ν = 3050, 2930, 2324, 2091, 1691, 1604, 1442, 1366, 1300, 1181, 1021, 927, 748, 690 cm^{-1}. The compound has already been described.[9]

1-(5-Fluoro-2,3-diphenyl-1*H*-indol-1-yl)ethan-1-one (3b)



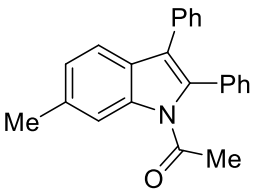
General procedure was followed to afford the title compound colorless solid (89.4 mg, 0.27 mmol, 45%). ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.42 (dd, J = 9.1, 4.7 Hz, 1H), 7.37–7.15 (m, 11H), 7.10 (td, J = 9.1, 2.7 Hz, 1H), 1.97 (s, 3H). $^{13}C\{^{19}F\}$-NMR (101MHz, $CDCl_3$): δ = 171.4, 160.0, 136.6, 133.3, 132.8, 132.7, 130.9, 130.5, 130.0, 129.0, 128.8, 128.5, 127.3, 123.2, 117.7, 113.2, 105.2, 27.9. MS (EI^+, 70 eV) *m/z* (%): 330 (16), 329 (56), 288 (22), 287 (100), 286 (28), 285 (33), 284 (12). MS (CI^+, methane) *m/z* (%): 358 (29), 331 (23), 330 (100), 329 (22). IR: ν = 3061, 2923, 2855, 2103, 1695, 1605, 1445, 1365, 1304, 1227, 1179, 1152, 1103, 1073, 1024, 955, 926, 861, 808, 774, 751, 722, 698 cm^{-1}. The compound has already been described.[10]

1-(5-Methoxy-2,3-diphenyl-1*H*-indol-1-yl)ethan-1-one (3c)



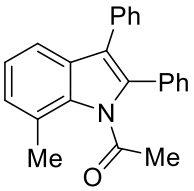
General procedure was followed to afford the title compound as a yellow solid (117.8 mg, 0.35 mmol, 58%). ^{1}H-NMR (600 MHz, $CDCl_3$): δ = 8.08 (d, J = 2.3 Hz, 1H), 7.42 (d, J = 8.6 Hz, 1H), 7.36–7.18 (m, 10H), 6.94 (dd, J = 8.6, 2.4 Hz, 1H), 3.92 (s, 3H), 1.99 (s, 3H). $^{13}C\{^1H\}$-NMR (151 MHz, $CDCl_3$): δ = 172.0, 158.9, 138.0, 133.8, 133.4, 133.3, 130.9, 130.1, 128.7, 128.6, 128.4, 127.0, 123.3, 120.2, 113.2, 100.4, 56.0, 28.2. MS (EI^+, 70 eV) *m/z* (%): 342 (17), 341 (62), 300 (24), 299 (100), 298 (13), 285 (16), 284 (73), 267 (13), 256 (12), 255 (12), 254 (35), 253 (10), 152 (12), 151 (13), 126.0 (12), 57.3 (12). MS (CI^+, methane) *m/z* (%): 370 (12), 343 (24), 342 (100), 341 (26). IR: ν = 3057, 3009. 2922, 2854, 2328, 2103, 1896, 1687, 1603, 1568, 1479, 1437, 1374, 1348, 1311, 1271, 1225, 1188, 1155, 1104, 1073, 1027, 977, 952, 922, 848, 823, 775, 748, 698 cm^{-1}. The compound has already been described.[11]

1-(6-Methyl-2,3-diphenyl-1*H*-indol-1-yl)ethan-1-one (3d)



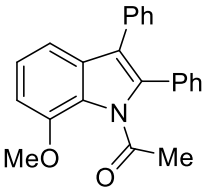
General procedure was followed to afford the title compound as a white solid (136.5 mg, 0.42 mmol, 70%). ^{1}H-NMR (600 MHz, $CDCl_3$): δ = 8.31 (s, 1H), 7.44 (d, *J* = 8.0 Hz, 1H), 7.37–7.33 (m, 5H), 7.30–7.21 (m, 5H), 7.15 (d, *J* = 8.0 Hz, 1H), 2.54 (s, 3H), 2.00 (s, 3H). ^{13}C{^{1}H}-NMR (151 MHz, $CDCl_3$): δ = 171.8, 137.3, 135.8, 134.5, 133.4, 133.3, 130.9, 130.1, 128.7, 128.6, 128.3, 127.1, 127.0, 125.3, 123.5, 119.3, 116.5, 28.1, 22.2. MS (EI$^+$, 70 eV) *m/z* (%): 326 (13), 325 (55), 284 (24), 283 (100), 282 (17), 267 (16). MS (CI$^+$, methane) *m/z* (%): 366 (10), 354 (27), 327 (24), 326 (100), 325 (21). IR: *v* = 3059, 3026, 2924, 2858, 2323, 2109, 1990, 1891, 1691, 1608, 1571, 1481, 1439, 1368, 1342, 1306, 1220, 1192, 1110, 1070, 975, 944, 923, 879, 851, 816, 753, 698 cm^{-1}. The compound has already been described.[11]

1-(7-Methyl-2,3-diphenyl-1*H*-indol-1-yl)ethan-1-one (3e)



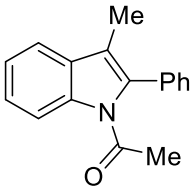
General procedure was followed to afford the title compound as a yellow solid (93.1 mg, 0.29 mmol, 48%). ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.46 (d, *J* = 7.4 Hz, 1H), 7.38–7.33 (m, 5H), 7.30–7.16 (m, 7H), 2.45 (s, 3H), 2.06 (s, 3H). ^{13}C{^{1}H}-NMR (101 MHz, $CDCl_3$): δ = 173.3, 135.7, 135.2, 133.6, 132.5, 131.0, 130.3, 130.2, 128.8, 128.7, 128.3, 127.7, 126.8, 124.7, 123.3, 121.4, 117.7, 28.8, 21.5. MS (EI$^+$, 70 eV) *m/z* (%): 325 (35), 283 (100), 282 (12), 267 (20). MS (CI$^+$, methane) *m/z* (%): 354 (20), 327 (24), 326 (100), 325 (34). IR: *v* = 3058, 2957, 2924, 2858, 2325, 2082, 1897, 1767, 1706, 1606, 1492, 1442, 1412, 1363, 1274, 1187, 1156, 1073, 1019, 955, 920, 881, 848, 816, 783, 747, 697, 666 cm^{-1}. The compound has already been described.[11]

1-(7-Methoxy-2,3-diphenyl-1*H*-indol-1-yl)ethan-1-one (3f)



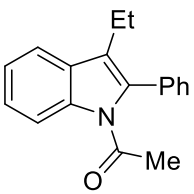
General procedure was followed to afford the title compound as an orange solid (127.1 mg, 0.37 mmol, 62%). ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.40–7.15 (m, 12H), 6.85 (d, *J* = 7.7 Hz, 1H), 3.99 (s, 3H), 2.42 (s, 3H). ^{13}C{^{1}H}-NMR (101 MHz, $CDCl_3$): δ = 173.9, 147.2, 136.3, 133.8, 131.7, 131.3, 131.1, 130.1, 128.4, 128.3, 128.1, 166.5, 125.4, 123.3, 120.0, 112.8, 105.7, 55.9, 29.0. MS (EI$^+$, 70 eV) *m/z* (%): 341 (44), 300 (21), 299 (100), 256 (41), 254 (12). MS (CI$^+$, methane) *m/z* (%): 371 (17), 344 (24), 343 (100), 342 (21). IR: *v* = 3026, 2935, 2844, 2324, 2107, 1728, 1602, 1577, 1492, 1433, 1360, 1292, 1222, 1126, 1069, 1023, 979, 917, 851, 790, 732, 697, 668 cm^{-1}. The compound has already been described.[11]

1-(3-Methyl-2-phenyl-1*H*-indol-1-yl)ethan-1-one (3g)



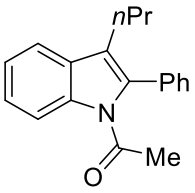
General procedure was followed to afford the title compound as a white solid (115.0 mg, 0.46 mmol, 77%). ^{1}H-NMR (600 MHz, $CDCl_3$): δ = 8.44 (d, *J* = 8.2 Hz, 1H), 7.55–7.52 (m, 1H), 7.51–7.48 (m, 2H), 7.47–7.43 (m, 1H), 7.41–7.37 (m, 3H), 7.33 (td, J = 7.6, 1.0 Hz, 1H), 2.15 (s, 3H), 1.97 (s, 3H). ^{13}C{^{1}H}-NMR (101 MHz, $CDCl_3$): δ = 171.2, 136.9, 135.0, 133.8, 130.4, 128.8, 128.6, 125.5, 123.6, 118.7, 118.3, 116.5, 27.8, 9.4. MS (EI$^+$, 70 eV) *m/z* (%): 250 (14), 249 (64), 208 (17), 207 (100), 206 (59), 204 (17), 130 (18), 128 (11). MS (CI$^+$, methane) *m/z* (%): 500 (14), 499 (31), 457 (14), 278 (15), 251 (17), 250 (100), 249 (29), 236 (10), 208 (17). IR: *v* = 3054, 2922, 2861, 2329, 2104, 1905, 1696, 1600, 1491, 1448, 1368, 1304, 1201, 1159, 1076, 1025, 932, 851, 799, 749, 700, 658 cm^{-1}. The compound has already been described.[9,12]

1-(3-Ethyl-2-phenyl-1*H*-indol-1-yl)ethan-1-one (3h)



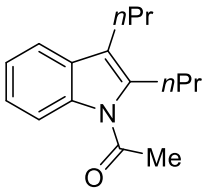
General procedure was followed to afford the title compound as a brown solid (113.2 mg, 0.43 mmol, 72%). ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.52–8.43 (m, 1H), 7.60 (ddd, *J* = 7.5, 1.4, 0.6 Hz, 1H), 7.53–7.46 (m, 3H), 7.43–7.32 (m, 4H), 2.58 (q, *J* = 7.6 Hz, 2H), 1.96 (s, 3H), 1.21 (t, *J* = 7.6 Hz, 3H). ^{13}C{^{1}H}-NMR (101 MHz, $CDCl_3$): δ = 171.2, 137.1, 134.5, 133.7, 130.3, 129.4, 128.8, 128.7, 125.3, 124.4, 123.5, 118.8, 116.7, 27.8, 17.7, 15.1. MS (EI$^+$, 70 eV) *m/z* (%): 264 (34), 263 (100), 221 (21), 206 (34), 204 (15). MS (CI$^+$, methane) *m/z* (%): 527 (14), 292 (19), 265 (19), 264 (100), 263 (30), 222 (11). IR: *v* = 3054, 2963, 2869, 1680, 1601, 1447, 1368, 1308, 1201, 1154, 1016, 919, 822, 756, 702 cm^{-1}. The compound has already been described.[13]

1-(2-Phenyl-3-propyl-1*H*-indol-1-yl)ethan-1-one (3i)



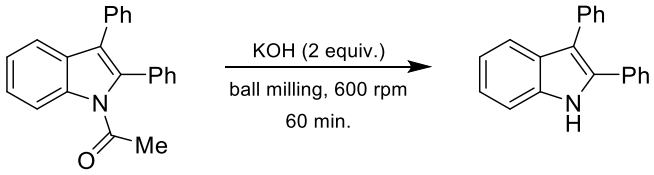
General procedure was followed to afford the title compound as a white solid (116.1 mg, 0.42 mmol, 70%). ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.46 (d, J = 8.3 Hz, 1H), 7.58 (d, *J* = 7.7 Hz, 1H), 7.51–7.44 (m, 3H), 7.42–7.36 (m, 3H), 7.34–7.30 (m, 1H), 2.57-2.49 (m, 2H), 1.95 (s, 3H), 1.66-1.60 (m, 2H), 0.89 (t, *J* = 7.4 Hz, 3H). ^{13}C{^{1}H}-NMR (101 MHz, $CDCl_3$): δ = 171.2, 137.0, 135.0, 133.8, 130.5, 129.8, 128.8, 128.7, 125.3, 123.5, 121.9, 119.0, 116.6, 27.8, 26.5, 23.6, 14.4. MS (EI^+, 70 eV) *m/z* (%): 278 (40), 277 (100), 236 (13), 235 (40), 207 (14), 206 (86), 204 (20). MS (CI^+, methane) *m/z* (%): 318 (15), 306 (27), 279 (26), 278 (100), 277 (24), 236 (10). IR: *v* = 3054, 2956, 2869, 1693, 1598, 1447, 1370, 1303, 1201, 1157, 1084, 1016, 928, 752, 698 cm^{-1}. The compound has already been described.[9]

1-(2,3-Dipropyl-1*H*-indol-1-yl)ethan-1-one (3j)



General procedure was followed to afford the title compound as a white solid (12.8 mg, 0.05 mmol, 9%). ^{1}H-NMR (600 MHz, $CDCl_3$): δ = 7.82-7.70 (m, 1H), 7.53–7.43 (m, 1H), 7.25–7.21 (m, 2H), 3.04–2.92 (m, 2H), 2.77 (s, 3H), 2.72–2.59 (m, 2H), 1.70–1.57 (m, 4H), 1.03–0.95 (m, 6H). ^{13}C{^{1}H}-NMR (151 MHz, $CDCl_3$): δ = 170.0, 138.3, 135.8, 131.1, 123.5, 122.7, 120.2, 118.9, 114.6, 29.1, 27.8, 26.2, 23.7, 23.6, 14.5, 14.3. MS (EI^+, 70 eV) *m/z* (%): 244 (19), 243 (83), 201 (32), 173 (16), 172 (100), 144 (17), 143 (16). MS (CI^+, methane) *m/z* (%): 260 (23), 245 (16), 244 (100), 243 (21). IR: *v* = 2958, 2930, 2868, 1700, 1577, 1457, 1367, 1305, 1202, 1067, 1032, 926, 799, 742, 702 cm^{-1}. The compound has already been described.[9,11]

Mechanochemical cleavage of the acetyl group



3a **4a**

1-(2,3-Diphenyl-1*H*-indol-1-yl)ethan-1-one (**3a**, 186.8 mg, 0.6 mmol) and potassium hydroxide (67.3 mg, 1.2 mmol, 2.0 equiv.) were transferred to a ball milling vessel (ZrO_2, 20 mL) loaded with 30 grinding balls (ZrO_2, diameter: 0.5 cm). The ball milling vessel was placed in the ball mill (60 min at 600 rpm). The crude product was isolated by washing the vessel and balls with EtOAc (5 x 20 mL). The organic phase was washed with a saturated aqueous solution of ammonium chloride (100 mL), dried over $MgSO_4$, concentrated *in vacuo* and purified by flash chromatography (SiO_2, *n*pentane/Et_2O) to afford 2,3-diphenyl-1*H*-indole (**4a**) as a light yellow solid (145.0 mg, 0.54 mmol, 90%).

^{1}H-NMR (600 MHz, $CDCl_3$): δ = 8.21 (s, 1H), 7.75 (d, *J* = 7.9 Hz, 1H), 7.54 – 7.48 (m, 2H), 7.48 – 7.41 (m, 5H), 7.38 – 7.28 (m, 5H), 7.23 – 7.20 (m, 1H). ^{13}C{^{1}H}-NMR (151 MHz, $CDCl_3$): δ = 136.0, 135.2, 134.2, 132.8, 130.3, 128.9, 128.8, 128.7, 128.3, 127.8, 126.4, 122.8, 120.6, 119.8, 115.2, 111.0. MS (EI^+, 70 eV) *m/z* (%): 270 (43), 269 (100), 268 (47), 267 (44), 266 (12), 254 (15), 239 (11), 165 (33), 135 (17), 134 (30), 133 (15), 127 (19), 121 (11). MS (CI^+, methane) *m/z* (%): 298 (16), 271 (26), 270 (100), 269 (31). IR: *v* = 3406, 3055, 2332, 2090, 1600, 1501, 1446, 1371, 1321, 1250, 1179, 1152, 1069, 1026, 965, 909, 826, 743 693 cm^{-1}. The compound has already been described.[9,14]

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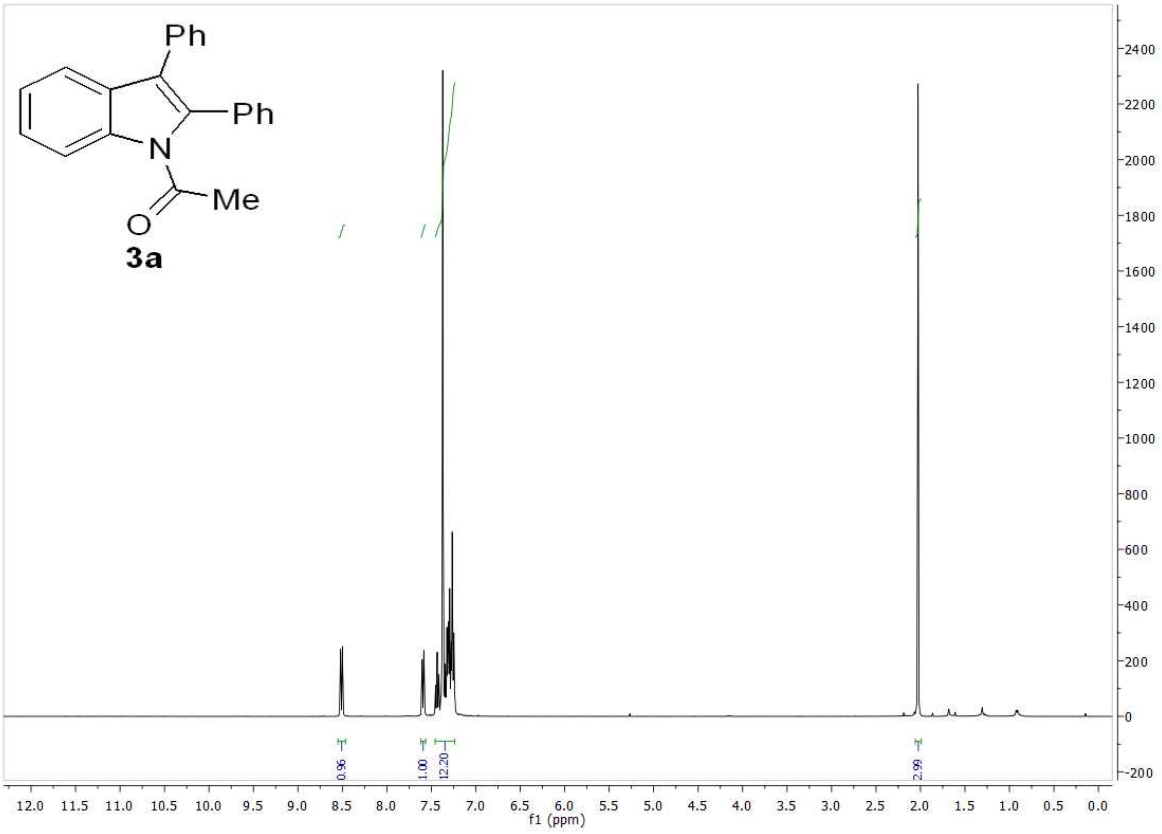
NMR spectra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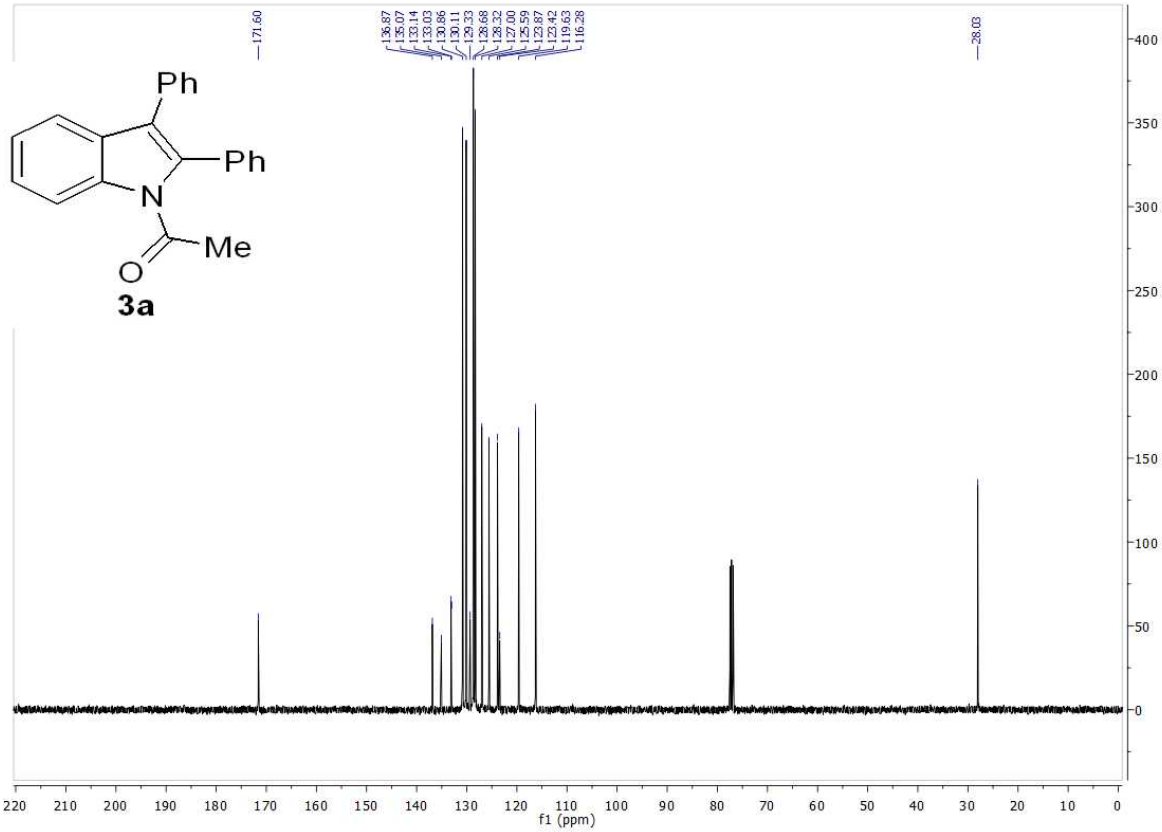
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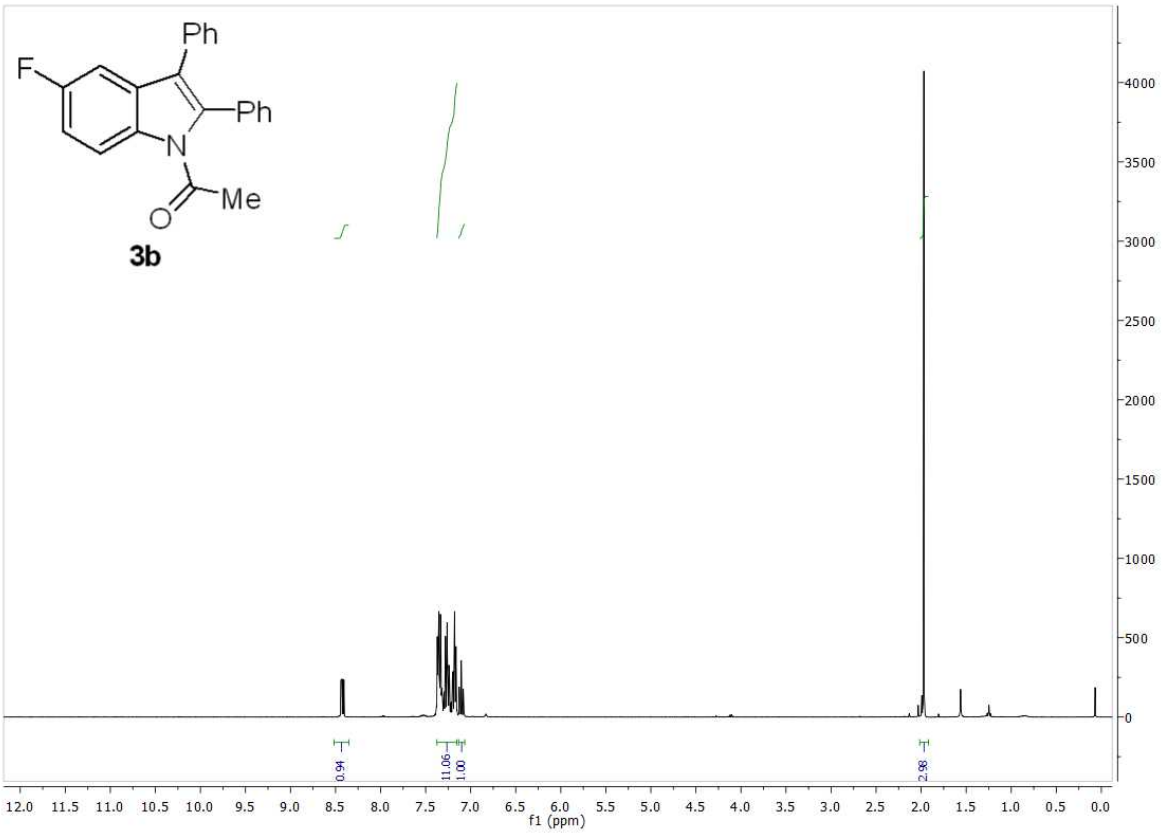
f1 (ppm)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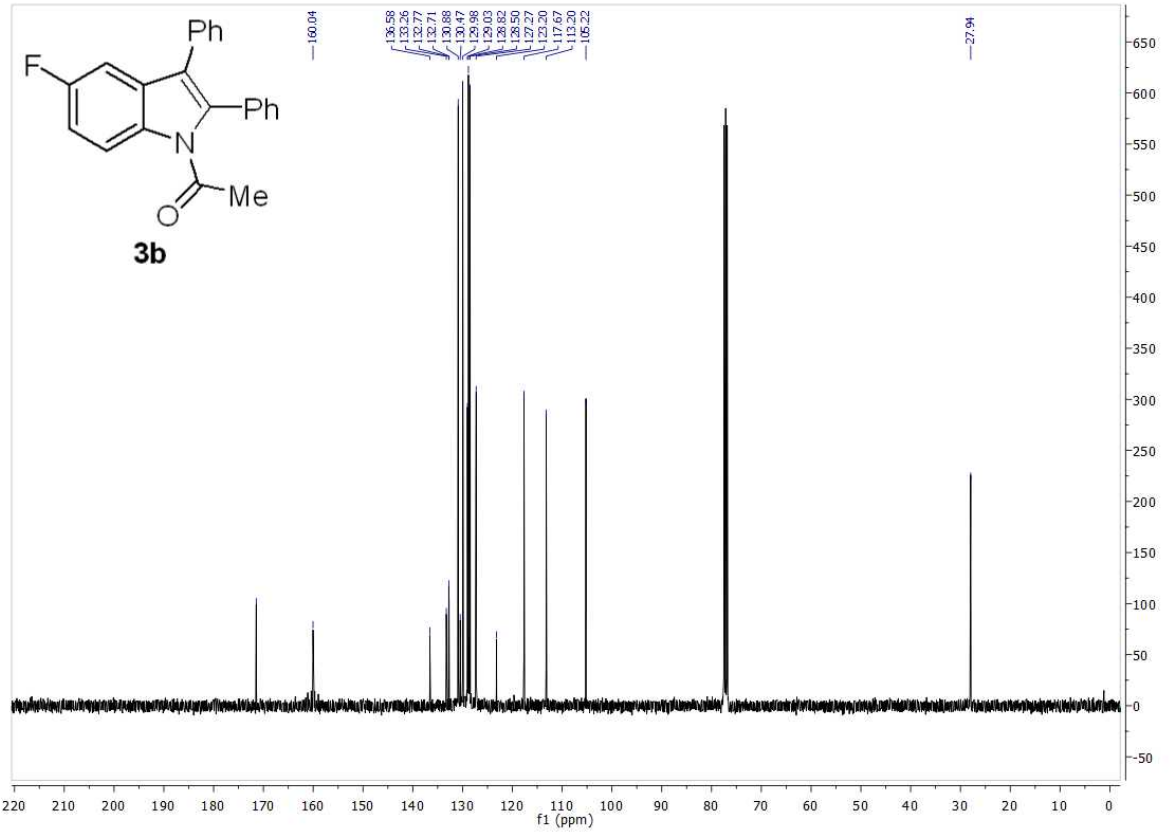
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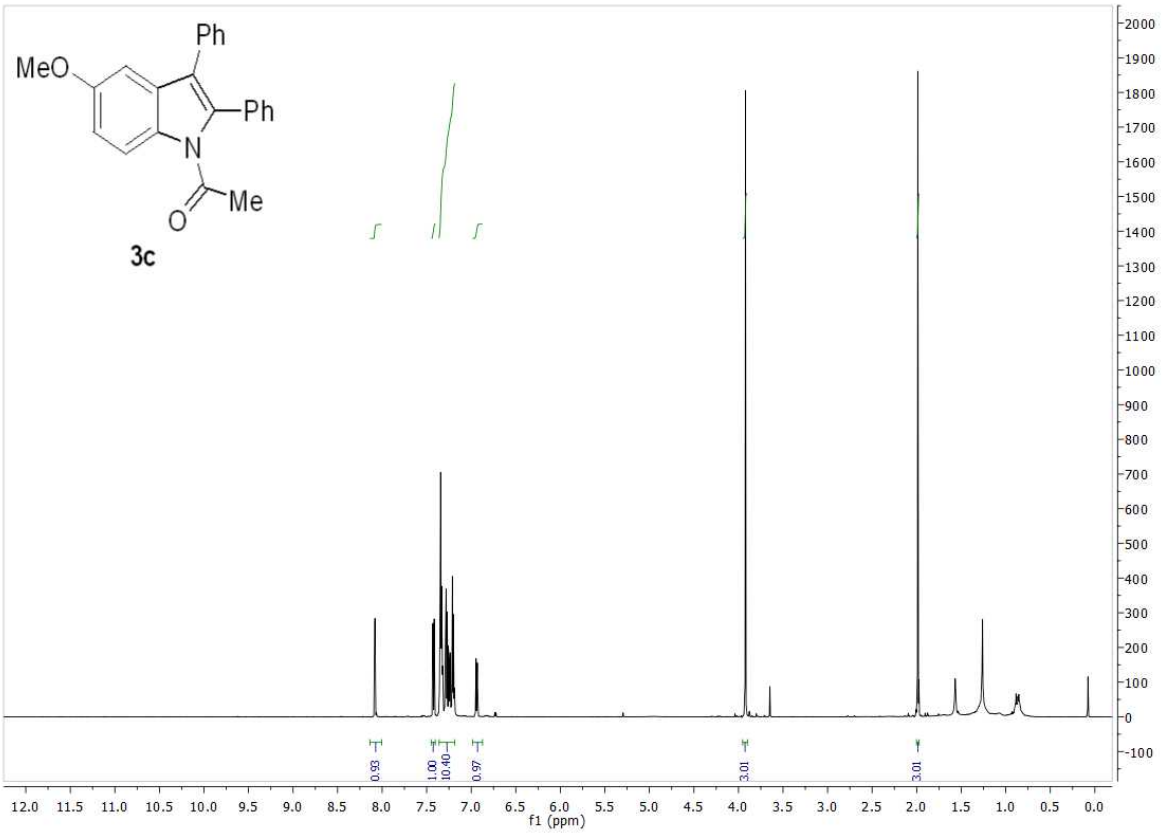
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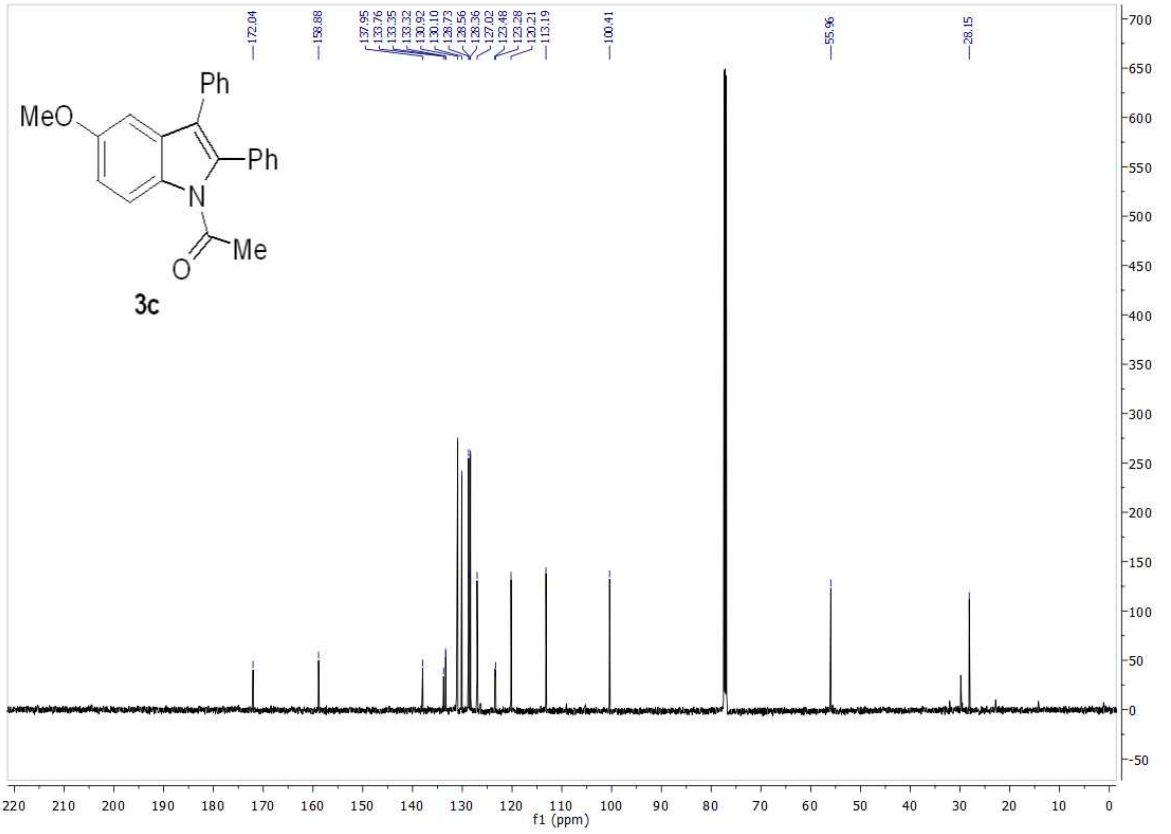
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O
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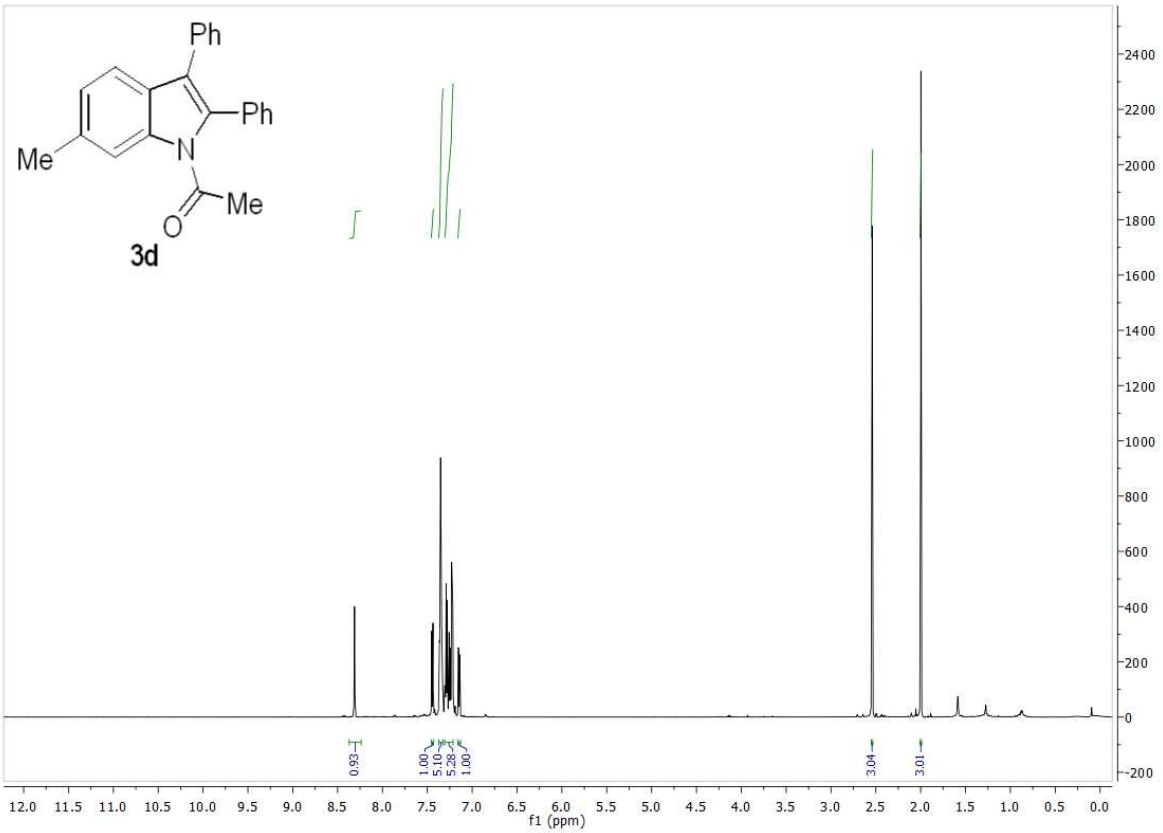
f1 (ppm)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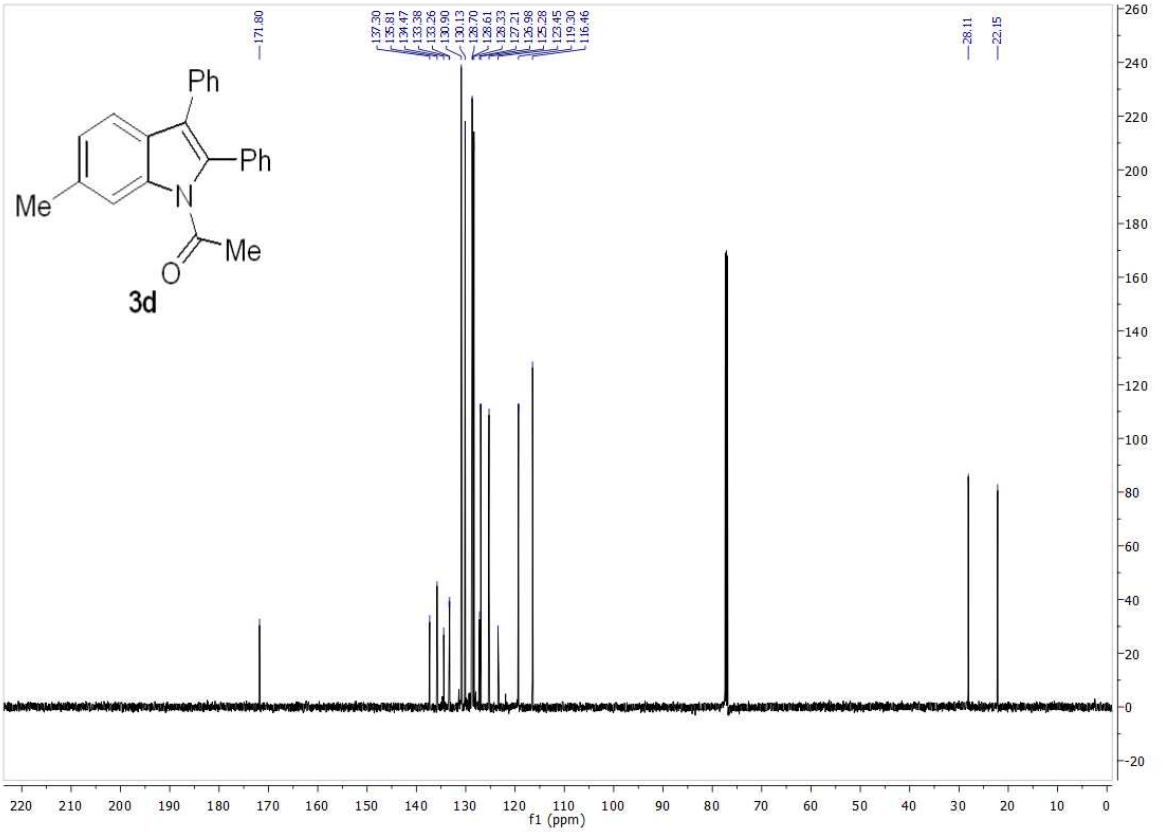
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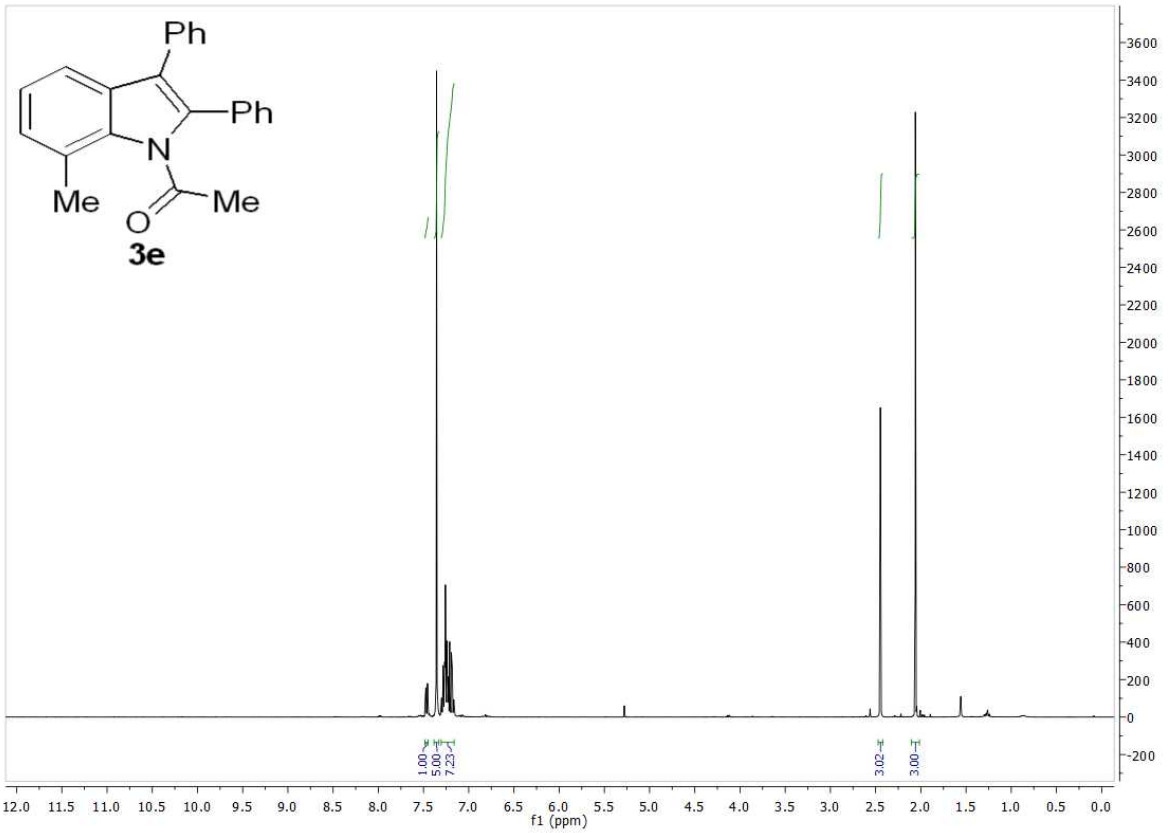
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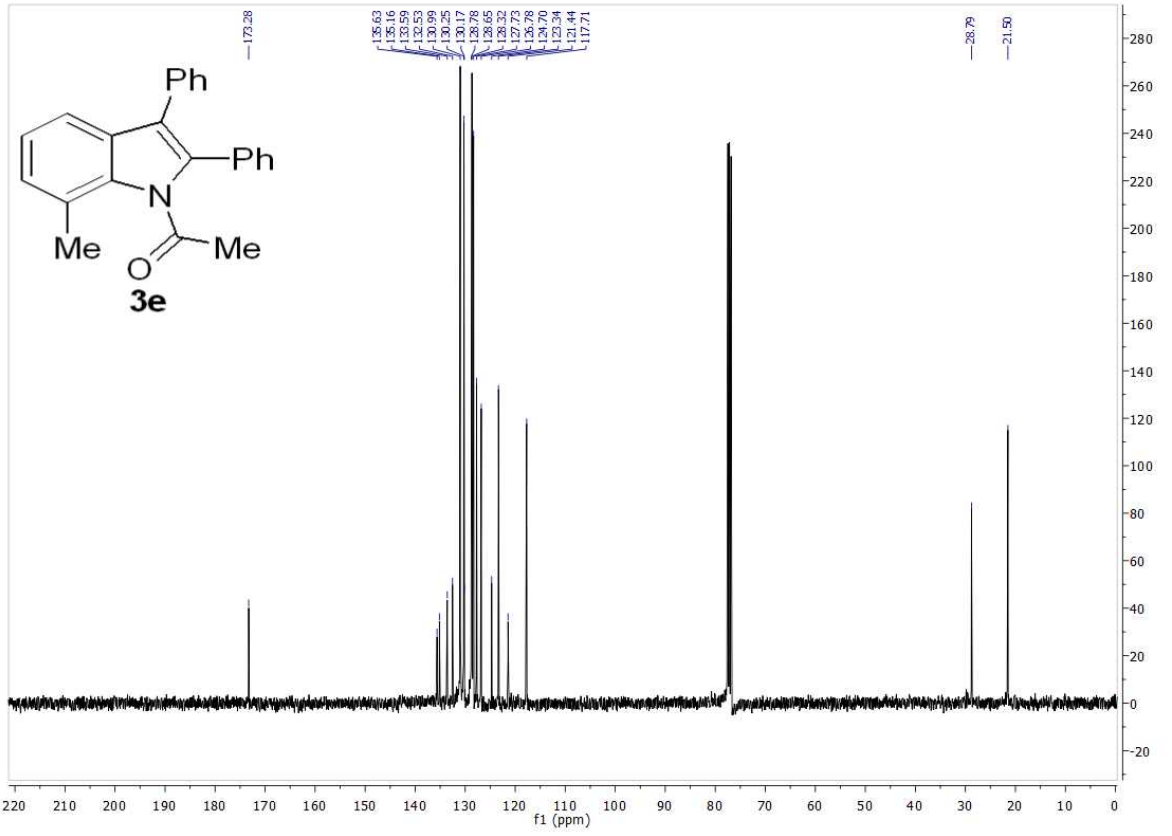
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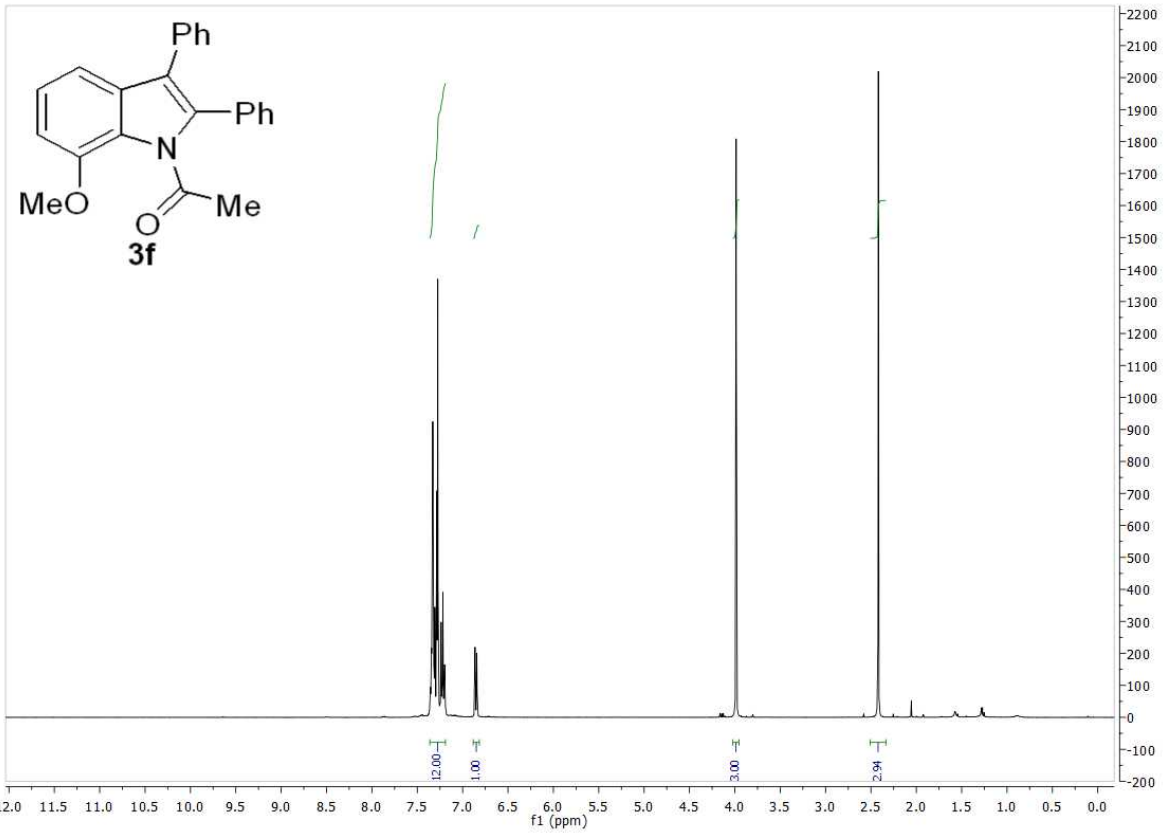
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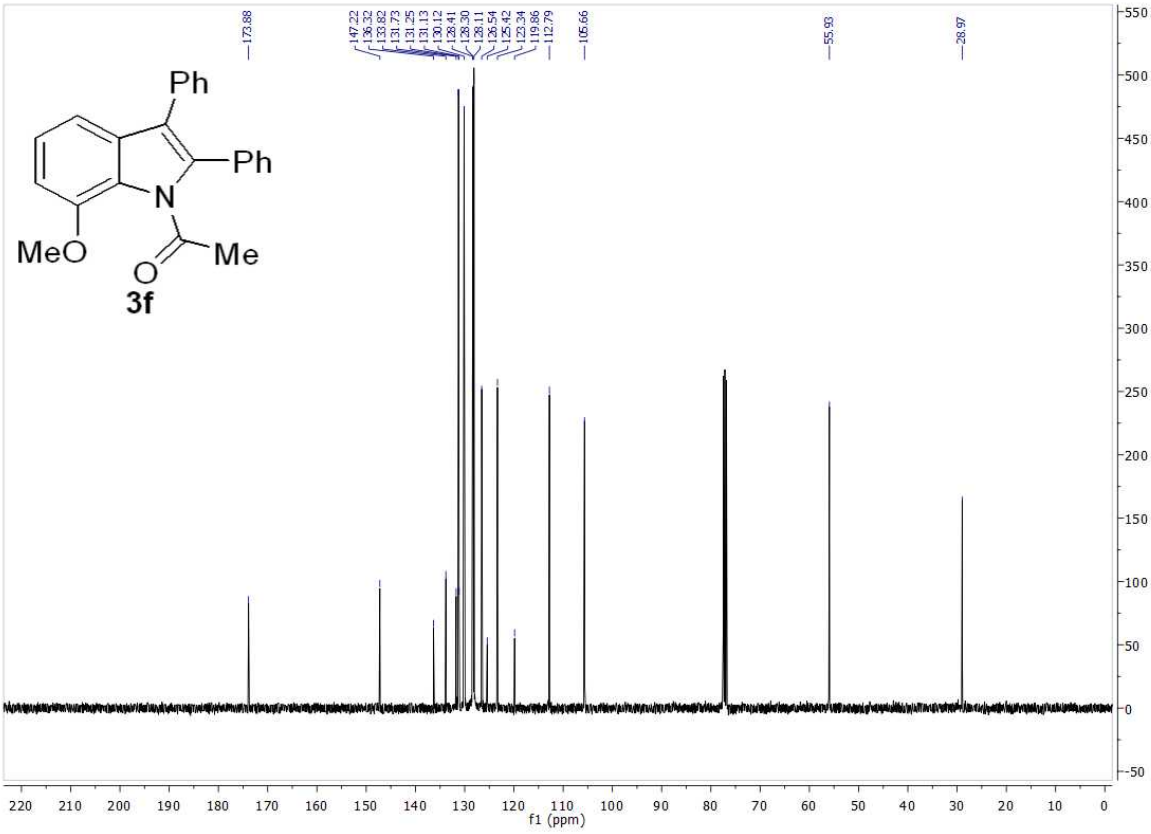
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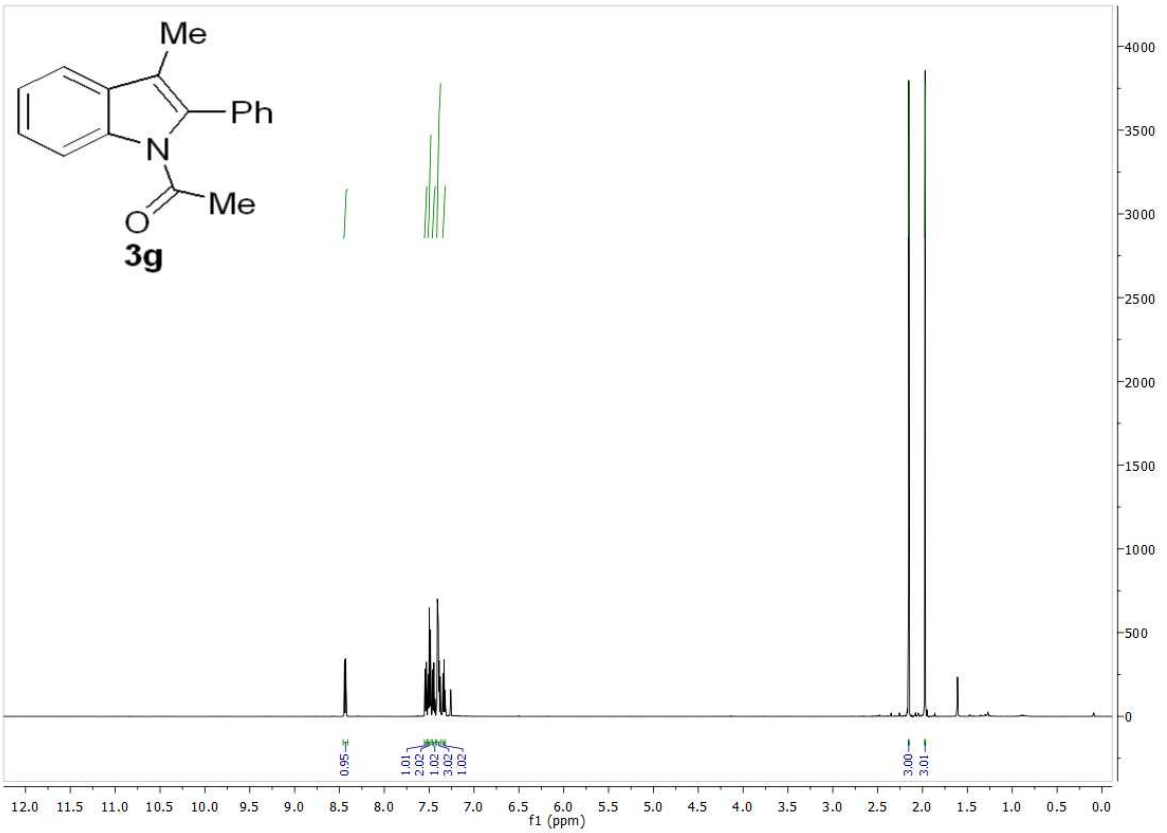
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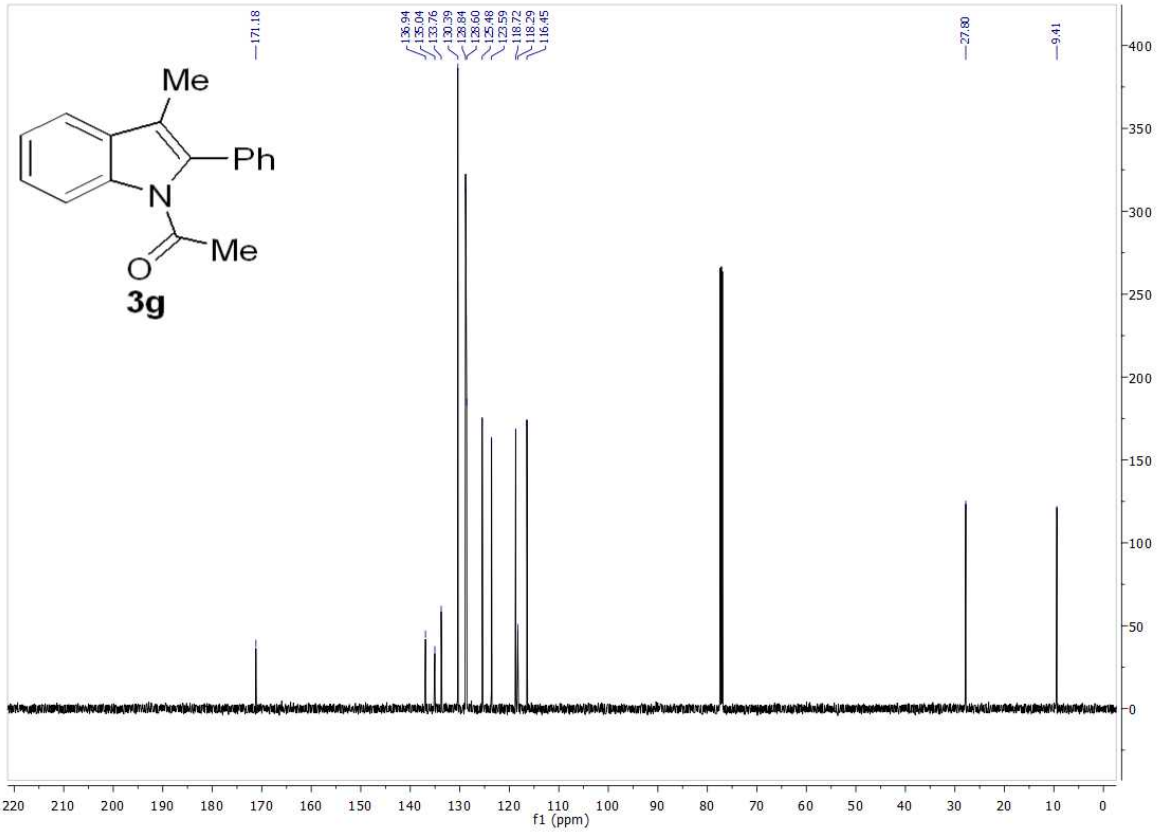
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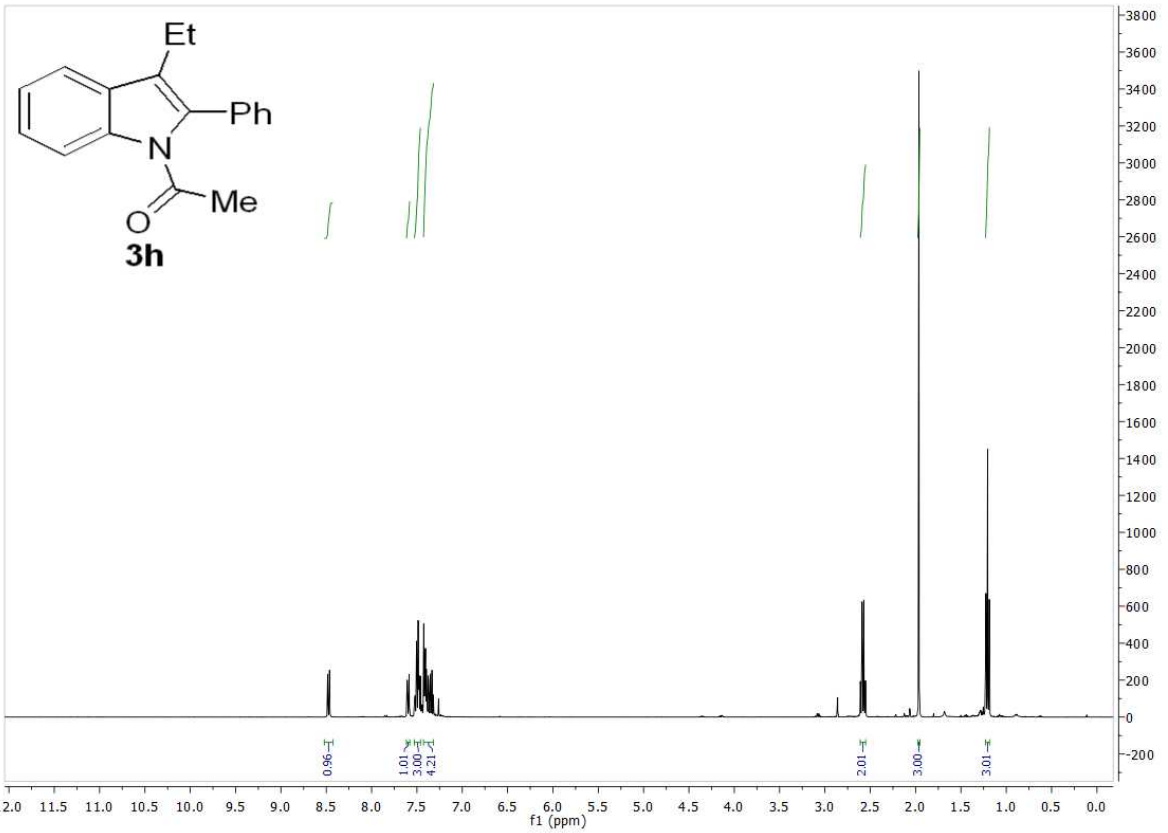
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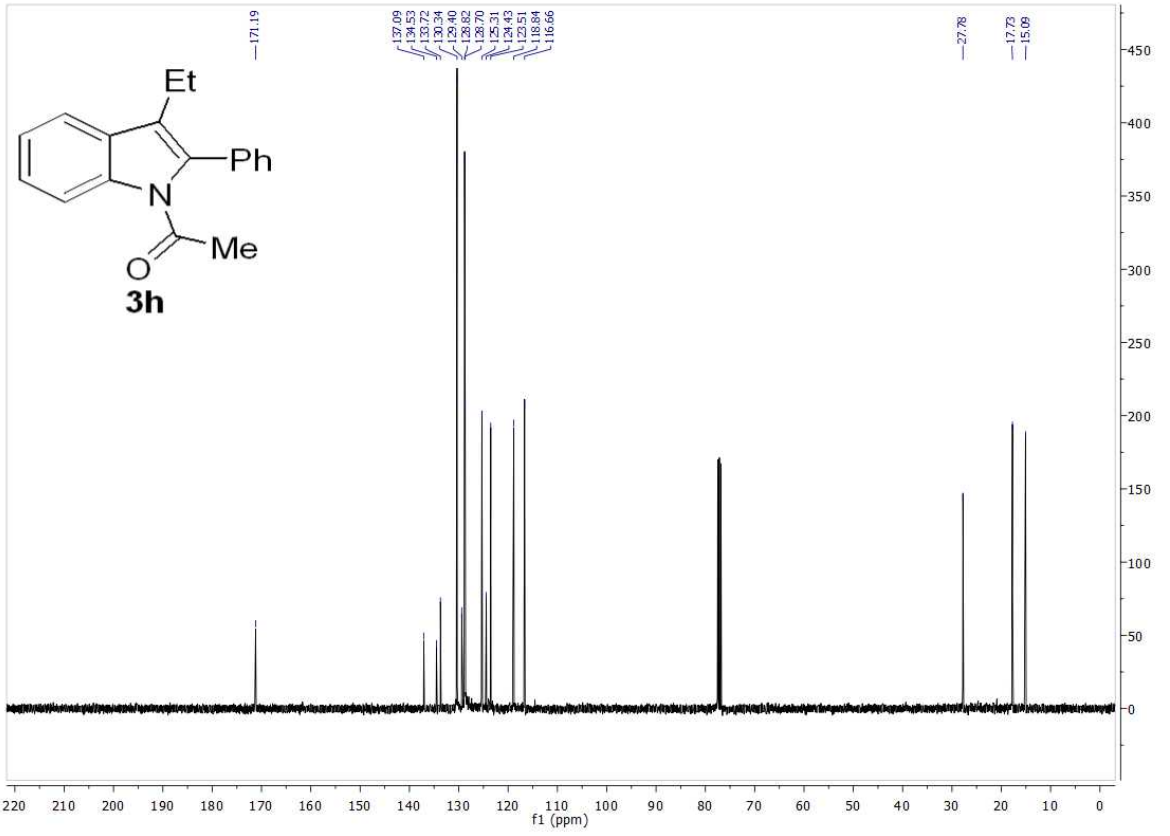
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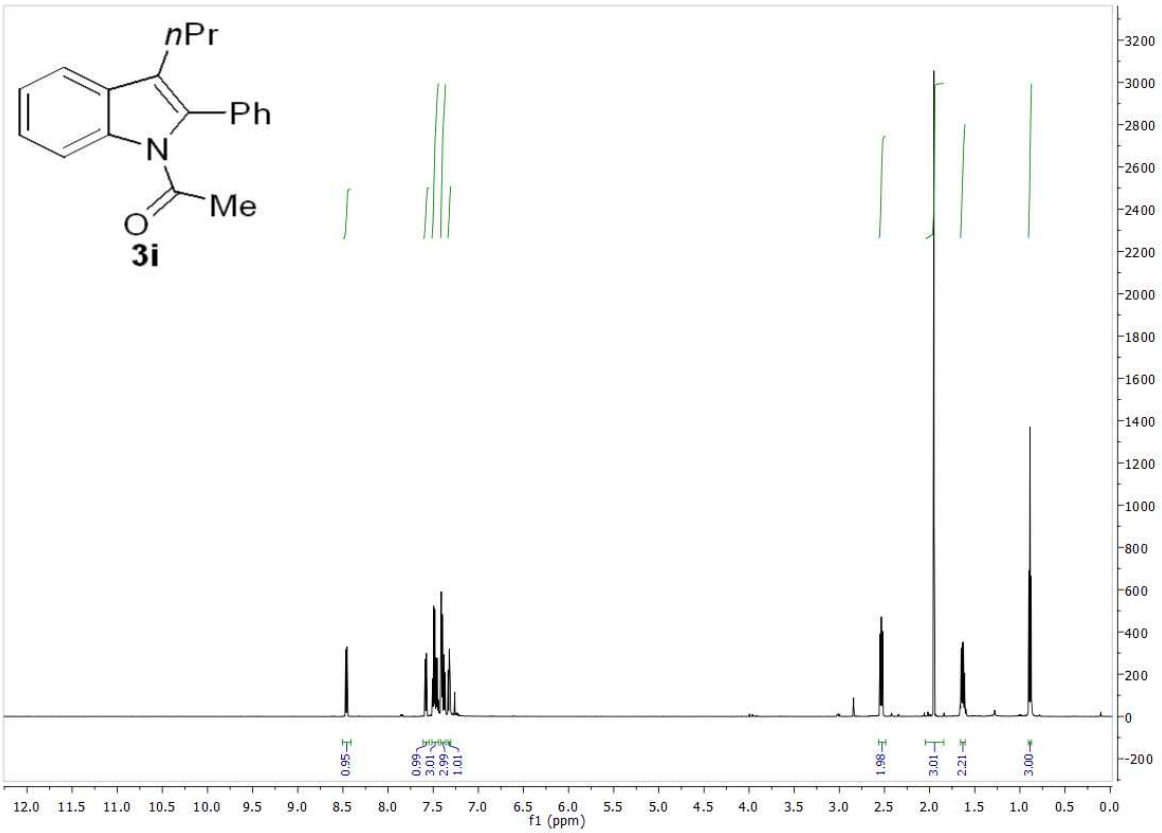
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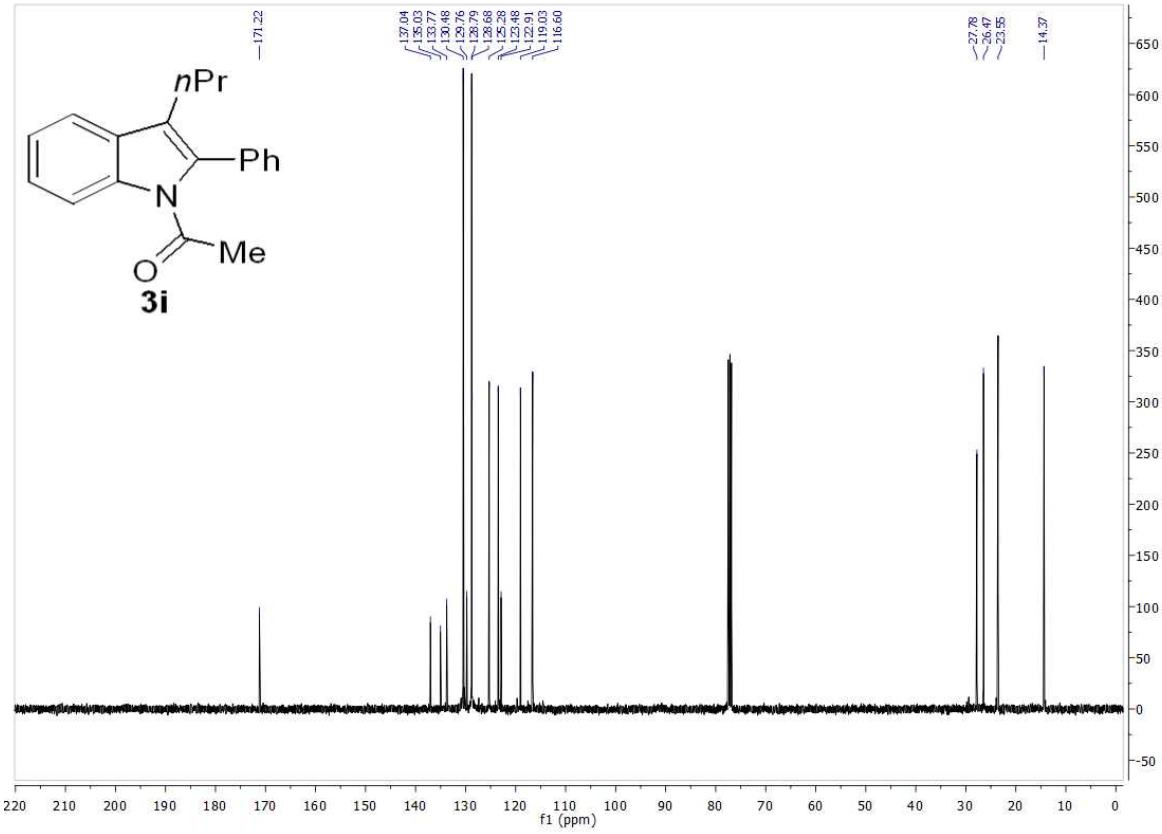
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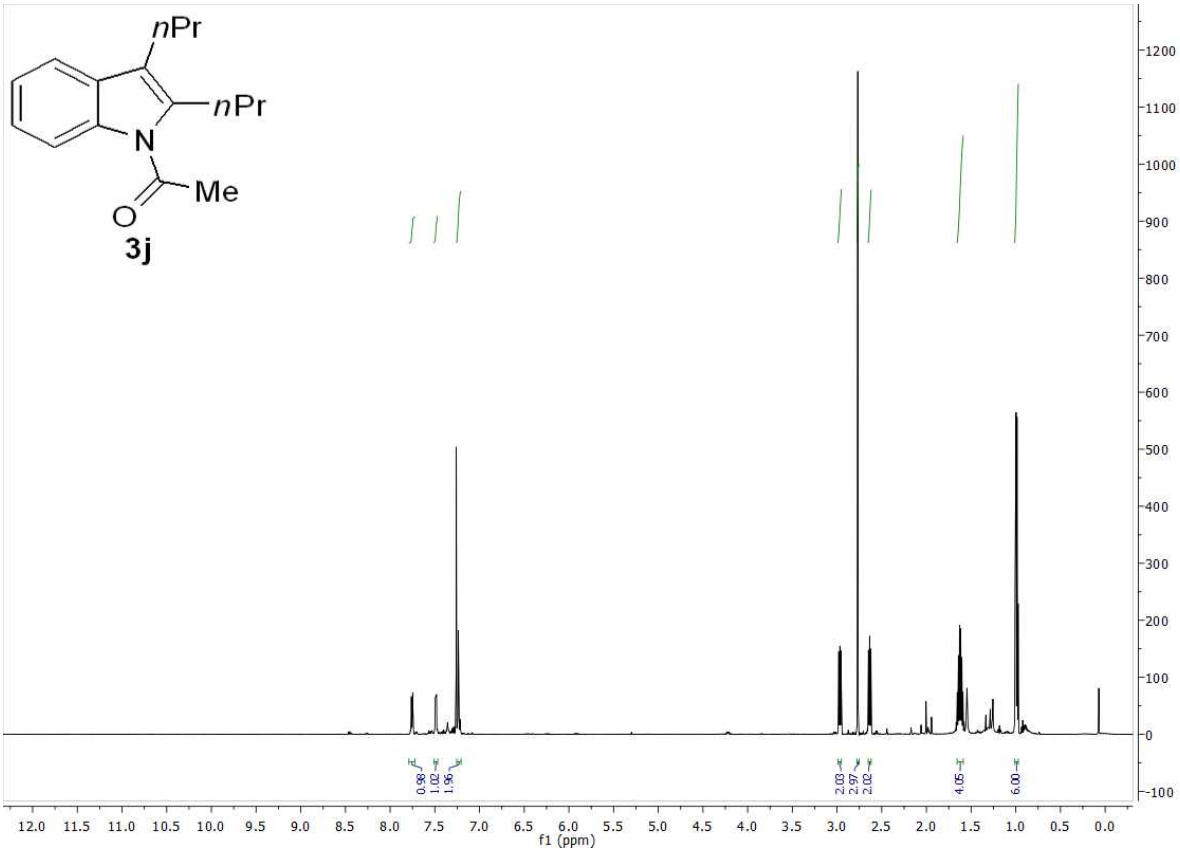
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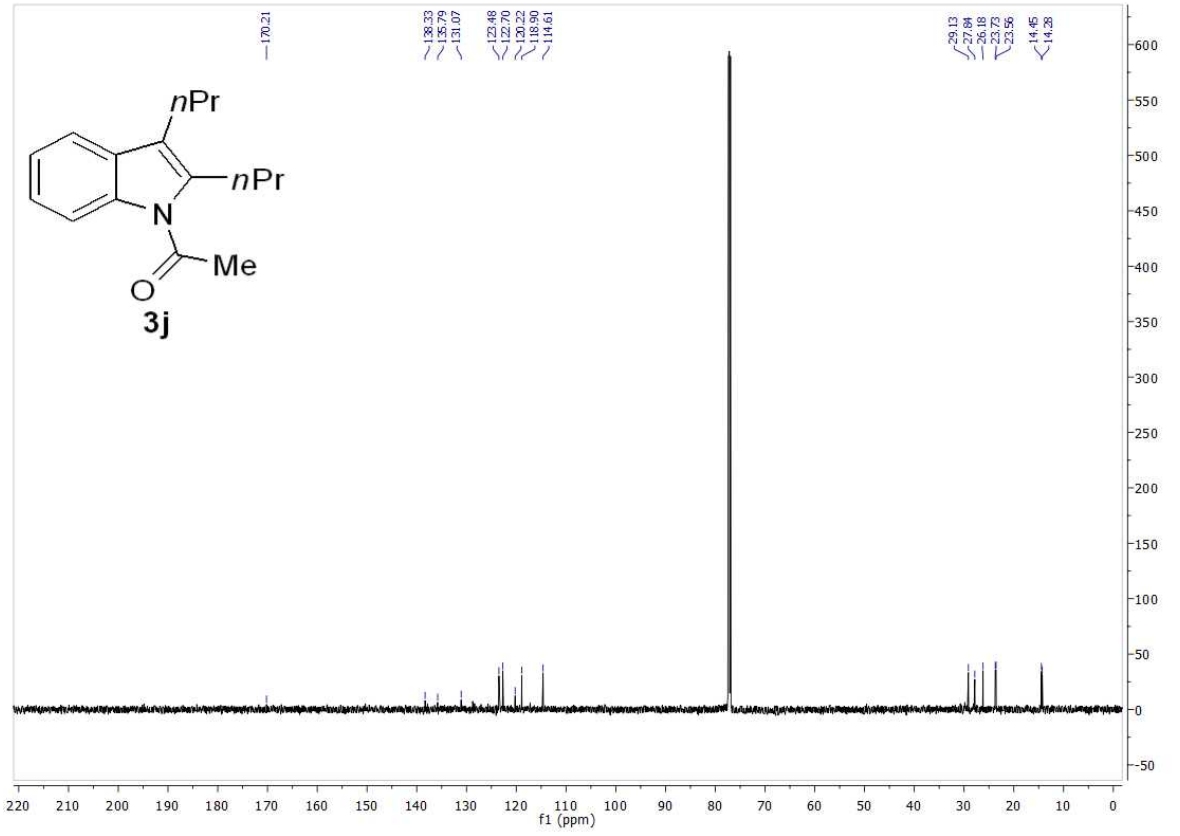
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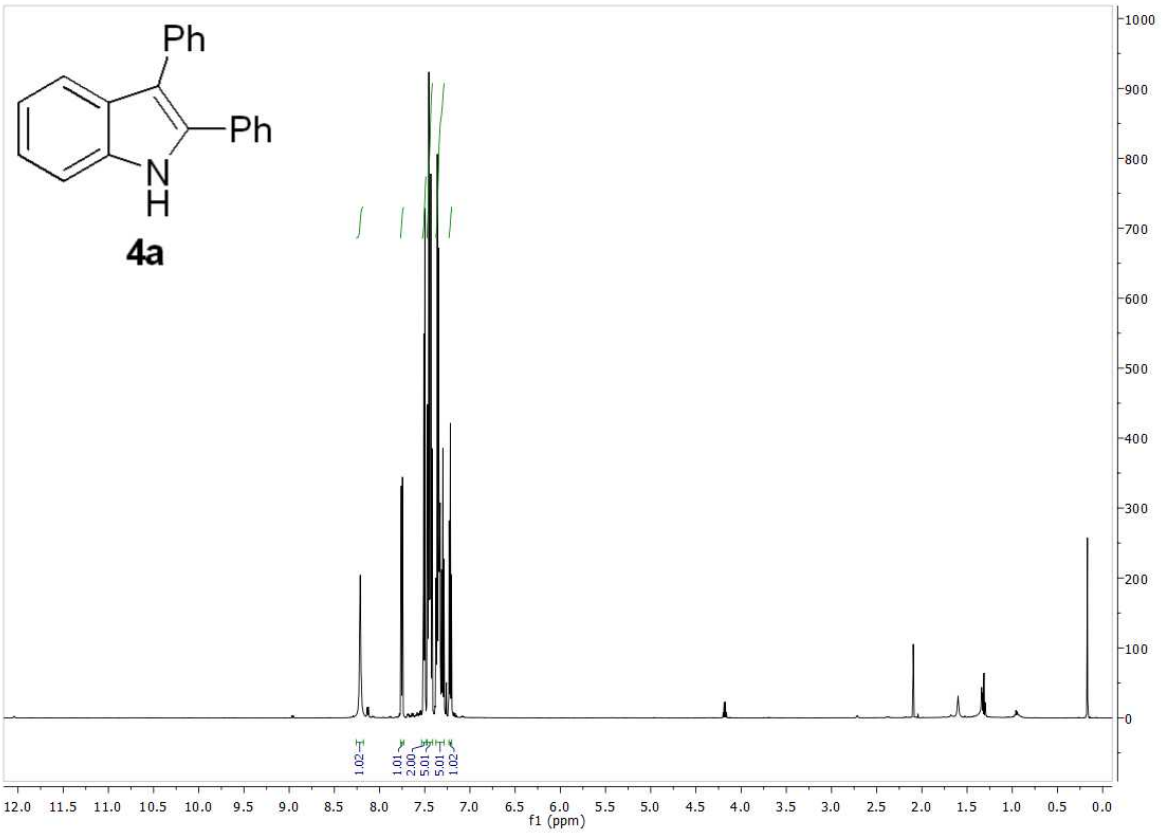
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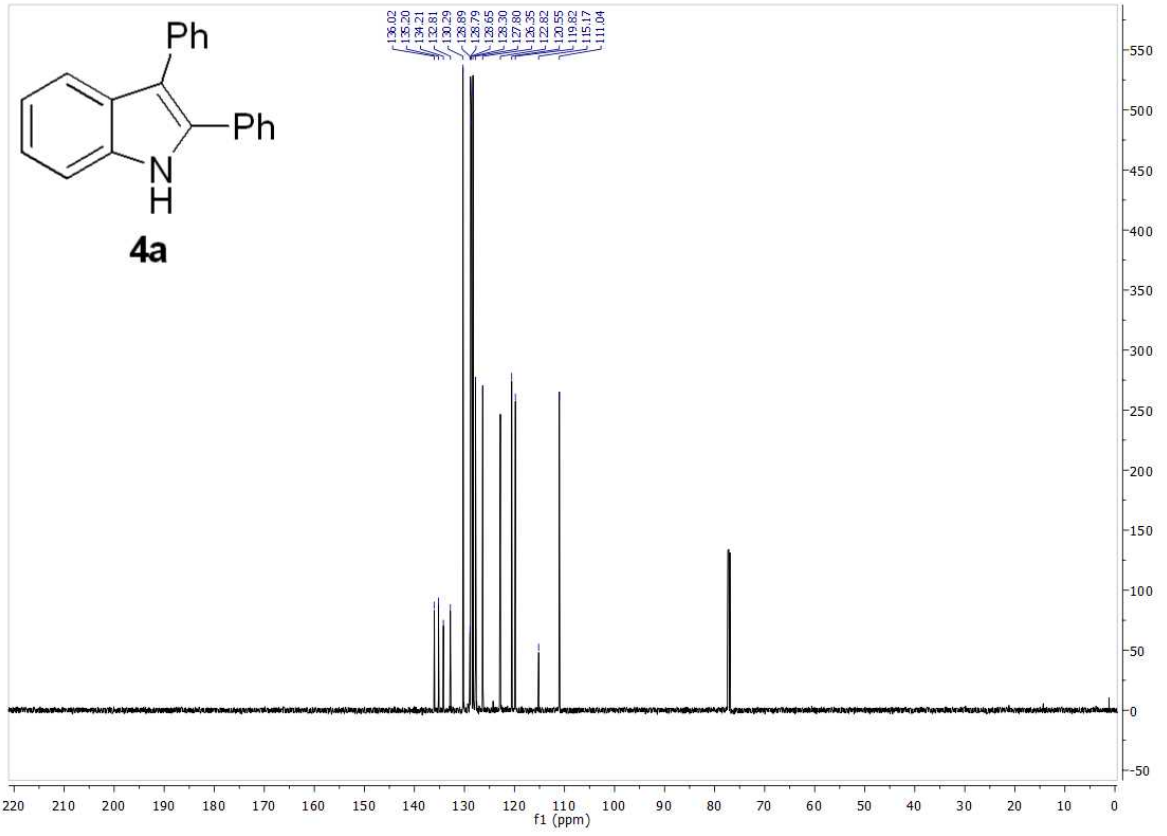
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