Electronic Supporting Information (ESI)

The Thermal C^2-C^6/[2+2] Cyclisation of Enyne-Allenes: Reversible Diradical Formation

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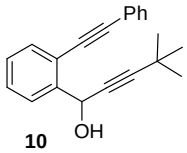
General Methods

All reagents were obtained from commercial sources and used without further purification. All reactions were carried out under nitrogen atmosphere using freshly distilled, anhydrous solvents. Diethyl ether was first dried over calcium hydride, then distilled over sodium. Tetrahydrofuran (THF) and toluene were distilled over potassium. Triethylamine (NEt_3) and dichloromethane (DCM) were distilled over calcium hydride. All reactions were followed by thin layer chromatography (TLC) on Merck silica gel plates (60F-254). Chromatographic purifications were performed on Silica gel 60 (60-230 mesh) from Merck. Preparative TLC's were carried out by using Merck silica gel plates (60F-254). ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AVANCE 400 (400 MHz) spectrometer. Chemical shifts (δ) are reported in ppm. 3-(Diphenylphosphinoyl)-1-[2-(phenylethynyl)-phenyl]-prop-1,2-diene (**6b**),[1] 2-(2-phenylethynyl)benzaldehyde (**9**)[2] and 3-(trimethylsilyl)-1-[2-(2-phenylethynyl)phenyl]prop-2-yn-1-ol (**11**)[3] were synthesised according to reported procedures.

Experimental Procedures

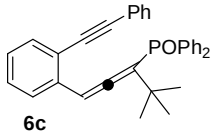


10

4,4-Dimethyl-1-[2-(2-phenylethynyl)phenyl]pent-2-yn-1-ol (10). A solution of ethyl magnesium bromide, prepared from ethyl bromide (909 mg, 8.36 mmol) and magnesium turnings (203 mg, 8.36 mmol) in dry THF (30 mL), was added to a solution of *tert*-butylacetylene (821 mg, 8.36 mmol) in dry THF (10 mL) under nitrogen atmosphere at 0 °C.

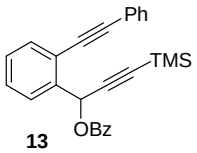
[1] M. Schmittel, S. Kiau, T. Siebert and M. Strittmatter, *Tetrahedron Lett.*, **1996**, *37*, 7691.
[2] R. R. Kevin and R.C. Larock, *J. Org. Chem.*, **2002**, *67*, 86.
[3] T. Kawano, M. Suehiro and I. Ueda, *Chem. Lett.*, **2006**, *35*, 58.

After stirring for 4 h at RT, a solution of 2-(2-phenylethynyl)-benzaldehyde (**9**)[2] (690 mg, 3.34 mmol) in dry THF (10 mL) was added dropwise. After stirring for 2.5 h at RT, it was quenched with saturated NH_4Cl solution. The aqueous layer was extracted with diethyl ether (2×40 mL). The combined organic layers were dried (Na_2SO_4) and concentrated. Purification by column chromatography (silica gel, *n*-hexane/ethyl acetate = 9:1, R_f = 0.28) furnished product **10** in 86% yield (835 mg, 2.89 mmol) as bright yellow oil. IR (KBr, cm^{-1}) 3414, 3060, 2968, 2927, 2901, 2866, 2234, 1600, 1493, 1262, 1066, 987, 757. ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.26 (s, 9H), 2.74 (d, J = 5.6 Hz, 1H), 5.96 (d, J = 5.6 Hz, 1H), 7.31 (td, J = 7.5, 1.3 Hz, 1H), 7.36-7.41 (m, 4H), 7.55-7.59 (m, 3H), 7.74 (dd, J = 7.8, 1.4 Hz, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ = 27.4, 30.8, 63.2, 77.8, 86.7, 94.7, 95.5, 121.4, 122.8, 126.6, 127.9, 128.3, 128.5, 128.7, 131.5, 132.3, 142.9. HRMS-EI (*m/z*) for $C_{21}H_{20}O$ $[M]^+$ calcd 288.151, found 288.151.



6c

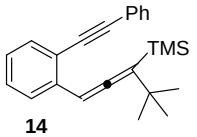
4,4-Dimethyl-3-(diphenylphosphinoyl)-1-[2-(phenyl-ethynyl)phenyl]-pent-1,2-diene (6c). To a solution of **10** (740 mg, 2.56 mmol) and NEt_3 (311 mg, 3.08 mmol) in THF (20 mL) at -78 °C was added chlorodiphenylphosphine (679 mg, 3.08 mmol) in THF (5 mL) over 15 minutes. The reaction mixture was stirred for 1 h at the same temperature and then allowed to slowly warm up to RT. After stirring for 1 h, it was hydrolysed with water and extracted with ethyl acetate. The combined organic layers were dried over magnesium sulfate and concentrated under reduced pressure. After purification by column chromatography (silica gel, *n*-hexane/ethyl acetate = 3:2, R_f = 0.34) **6c** was isolated in 82% yield (994 mg, 2.10 mmol) as white solid. Mp: 38 - 40 °C. IR (KBr, cm^{-1}) 3057, 2962, 2213, 1936, 1599, 1494, 1438, 1190, 1116, 813, 756. ^{1}H-NMR (400 MHz, C_6D_6) δ 1.50 (s, 9H), 6.80-6.84 (m, 1H), 6.88-7.02 (m, 11H), 7.30 (d, J = 7.8 Hz, 1H), 7.36-7.39 (m, 3H), 7.87-7.96 (m, 4H). ^{13}C-NMR (100 MHz, C_6D_6) δ 31.0 (d, $J_{C,P}$ = 2.6 Hz), 38.0 (d, $J_{C,P}$ = 5.1 Hz), 87.9, 94.9, 96.0 (d, $J_{C,P}$ = 14 Hz), 113.0 (d, $J_{C,P}$ = 92 Hz), 121.5 (d, $J_{C,P}$ = 2.6 Hz), 123.5, 126.5 (d, $J_{C,P}$ = 1.7 Hz), 127.4, 127.9, 128.2, 128.4 (d, $J_{C,P}$ = 1.7 Hz), 128.7 (d, $J_{C,P}$ = 1.7 Hz), 131.3 (d, $J_{C,P}$ = 2.6 Hz), 131.5 (d, $J_{C,P}$ = 2.6 Hz), 131.7, 131.8, 131.9, 132.0, 132.8, 135.0 (d, $J_{C,P}$ = 102 Hz), 135.1 (d, $J_{C,P}$ = 104 Hz), 135.3 (d, $J_{C,P}$ = 6.8 Hz), 209.4 (d, $J_{C,P}$ = 6.8 Hz). HRMS-EI (*m/z*) for $C_{33}H_{29}OP$ $[M]^+$ calcd 472.196, found 472.196.



13

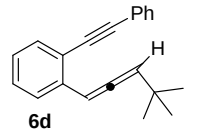
1-[2-(2-Phenylethynyl)phenyl]-3-(trimethylsilyl)prop-2-ynyl benzoate (13). To a solution of 3-(trimethylsilyl)-1-[2-(2-phenylethynyl)phenyl]prop-2-yn-1-ol (**11**)[3] (10.0 g, 32.8 mmol) in 400 mL of THF at -78 °C was added *n*-BuLi (2.5 M in hexane, 14.4 mL, 36.1 mmol). After stirring for 30 min, benzoylchloride (5.07 g, 36.1 mmol) was added. After stirring for 1.5 h, 100 mL of a 10% Na_2CO_3 solution were added. After 1 h, the resulting solution was diluted with 100 mL of water and extracted with diethyl ether (2×100 mL). The combined organic layers were dried and concentrated under reduced pressure. After purification by column chromatography (silica gel, *n*-hexane/ethyl acetate = 9:1, R_f = 0.56) propargyl benzoate **13** was isolated in 95% yield (12.8 g, 31.3 mmol) as a yellow oil. IR (KBr, cm^{-1}) 3063, 3034, 2960, 2900, 2217, 2180, 1725, 1601, 1494, 1451, 1351, 1251, 1177, 1093, 1035, 845, 758. ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.19 (s, 9H), 7.17 (s, 1H), 7.29-7.54 (m, 10H), 7.58 (dd, J = 7.4, 1.4 Hz, 1H), 7.88 (dd, J = 7.8, 1.2 Hz, 1H), 8.04-8.07 (m, 2H). ^{13}C-NMR (100 MHz,

$CDCl_3$) δ -0.24, 64.9, 86.2, 92.8, 95.2, 100.8, 122.8, 123.0, 128.1, 128.2, 128.3, 128.4, 128.5, 128.8, 129.8, 129.9, 131.7, 132.4, 133.0, 138.1, 165.2. HRMS-EI (*m/z*) for $C_{27}H_{24}O_2Si$ $[M]^+$ calcd 408.155, found 408.154.



14

4,4-Dimethyl-1-[2-(phenylethynyl)phenyl]-3-(trimethylsilyl)pent-1,2-diene (14). To a stirred solution of CuCN (218 mg, 2.44 mmol) in diethyl ether (20 mL) at -78 °C was slowly added a solution of *t*-BuLi (1.6 M in pentane, 3.05 mL, 4.88 mmol). After 5 min, the reaction mixture was warmed to 0 °C, and after 15 min it was cooled again to -78 °C. Thereafter a solution of **13** (500 mg, 1.22 mmol) in diethyl ether (10 mL) was then slowly added. The reaction mixture was stirred for 1 h and allowed to warm to 0 °C. After 1 h at 0 °C, water (50 mL) was added and the mixture was extracted with diethyl ether (3 × 30 mL). The combined organic layers were dried and concentrated under reduced pressure. After purification by column chromatography (silica gel, *n*-hexane, R_f = 0.48) **14** was isolated in 48% yield (204 mg, 592 μmol) as a viscous yellow oil. IR (KBr, cm^{-1}) 3059, 3026, 2961, 2900, 2866, 2216, 1922, 1598, 1443, 1362, 1249, 1225, 950, 841, 754. ^{1}H-NMR (400 MHz, C_6D_6) δ 0.21 (s, 9H), 1.19 (s, 9H), 6.86 (td, *J* = 8.1, 1.1 Hz), 6.94-6.98 (m, 3H), 7.05-7.09 (m, 2H), 7.40-7.44 (m, 2H), 7.54 (dd, *J* = 7.8, 1.1 Hz, 1H), 7.67 (d, *J* = 8.1 Hz, 1H). ^{13}C-NMR (100 MHz, C_6D_6) δ 1.42, 31.6, 36.3, 88.6, 89.6, 94.6, 112.2, 120.8, 123.9, 125.2, 126.0, 128.3, 128.5, 128.9, 131.9, 133.0, 138.4, 206.3. Anal. calcd. for $C_{24}H_{28}Si$ (344.56): C, 83.66; H, 8.19. Found C, 83.87; H, 8.38.



6d

4,4-Dimethyl-1-[2-(phenylethynyl)phenyl]-penta-1,2-diene (6d). To a solution of **14** (100 mg, 290 μmol) in 5 mL of methanol was added 10 mL of 1 N KOH and 5 mL of THF. The reaction mixture was stirred for 4 h at room temperature. After extraction with diethyl ether (3 × 15 mL) the organic phase was washed with water. The combined organic layers were dried over $MgSO_4$, and the solvent was removed under reduced pressure. After purification by column chromatography (silica gel, *n*-pentane, R_f = 0.44) **6d** was isolated in 79% yield (62 mg, 228 μmol) as viscous yellow oil. IR (KBr, cm^{-1}) 3058, 3031, 2960, 2901, 2864, 2215, 1947, 1599, 1494, 1445, 1363, 1248, 882, 756. ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.05 (s, 9H), 5.52 (d, *J* = 6.4 Hz, 1H), 6.88 (td, *J* = 7.6, 1.1 Hz), 6.96-6.99 (m, 3H), 7.05 (td, *J* = 7.6, 1.1 Hz, 1H), 7.30 (d, *J* = 6.4 Hz, 1H), 7.40-7.44 (m, 2H), 7.52 (dd, *J* = 7.6, 1.1 Hz, 1H), 7.67 (d, *J* = 7.6 Hz, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 30.3, 32.8, 88.4, 94.8, 95.1, 107.2, 121.6, 123.8, 126.3, 126.8, 128.4, 128.6, 128.8, 131.9, 132.8, 137.3, 203.8. HRMS-EI (*m/z*) for $C_{21}H_{20}$ $[M]^+$ calcd 272.157, found 272.157.

X-ray data collection and structure determinations

X-ray single-crystal diffraction data for **6c** was collected on a STOE IPDS one-circle image plate diffractometer. The structure was solved using the SHELXL-97 crystallographic

software package and refined by the full-matrix least-squares technique.[4] The hydrogen atoms were generated theoretically onto the specific atoms and refined isotropically with fixed thermal factors. The non-H atoms were refined with anisotropic thermal parameters. The crystal parameters, data collection, and refinement results are summarised in Table S1. Selected bond lengths and angles are listed in Table S2. Further details are provided in the ESI.

Table S1. Crystal data and structure refinement details for **6c**.

Empirical formula	$C_{36}H_{32}OP$
Formula weight/g mol^{-1}	511.59
Crystal system, space group	Triclinic, *P*-1
a /Å	9.0854(18)
b /Å	13.430(3)
c /Å	13.560(3)
α (º)	96.34(3)
β (º)	107.73(3)
γ (º)	102.33(3)
V /A^3	1512.0(5)
Z	2
D_{calc} / g·cm^{-3}	1.124
F(000)	542
θ range / (º)	2.44 - 28.23
Reflections collected	6849
Independent reflections	2057
Max. and Min. transmission	0.9874 and 0.9732
Goodness of-fit on F^2	0.723
Final *R* indices [$I > 2\sigma(I)$]	*R*1 = 0.0501, *wR*2 = 0.1118
R indices (all data)	*R*1 = 0.1750, *wR*2 = 0.1455

Table S2. Selected bond distances (Å) and angles (deg) for **6c**

C2-C7	1.439(4)	C7-C8	1.199(4)
C8-C9	1.445(4)	C1-C15	1.463(4)
C15-C16	1.303(4)	C16-C17	1.315(4)
C17-C18	1.551(4)	C17-P1	1.807(3)
C8 C7 C2	179.1(3)	C7 C8 C9	179.2(3)
C18 C17 P1	121.7(2)	C15 C16 C17	176.0(3)
C16 C15 C1	126.0(3)	C16 C17 P1	117.9(2)

[4] (a) G. M. Sheldrick, *SHELXS97, Program for Crystal Structure Determination*; University of Göttingen: Göttingen, Germany, 1997. (b) G. M. Sheldrick, *SHELXL97, Program for Crystal Structural Refinement*; University of Göttingen: Göttingen, Germany, 1997.

Computational Details

DFT calculations[5] were carried out for **6d** by using the Becke's pure gradient-corrected exchange functional[6] in conjunction with the Lee-Yang-Parr non-local correlation functional (BLYP)[7] and with a 6-31G(d) basis set.[8] While unrestricted calculations with a broken spin approach (BS-UBLYP) have been performed for the open-shell singlet state transition state (TS) structures and the intermediates formed from these TSs, restricted method was applied for the closed-shell **6d** and **15d**. It was reported that a broken spin approach applied with DFT method provides good results for the cyclisation of unsaturated systems.[9] The spin projection method of Yamaguchi *et al.*[10] was applied for molecules possessing spin contaminated wave functions. Calculated minima and first order saddle point structures were verified by analyzing the harmonic vibrational frequencies, using analytical second derivatives, which have NIMAG = 0 and 1, respectively.

Table S3. Absolute energies, ZPVEs, and $<S^2>$ values for all computed structures.

	Absolute Energy [Hartree]	ZPVE [kcal mol^{-1}]	$<S^2>$ (S2A)	E+ZPVE [Hartree]
6d	-811.786356	208.262740	0	-811.454468
16	-811.752689	207.397940	0	-811.422179
7d	-811.773786 -811.774078 (T) -811.773485 (S)*	208.094880	1.03 (0.25) (S) 2.03 (2.00) (T)	-811.442217
17	-811.741293	207.783420	0.54 (0)	-811.410169
15d	-811.810029	210.656210	0	-811.474327
18	-811.725095	207.173516	0 (0)	-811.376324

A: After Annihilation. S: Singlet. T: Triplet. * After spin correction.

[5] M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, J. A. Montgomery, Jr., T. Vreven, K. N. Kudin, J. C. Burant, J. M. Millam, S. S. Iyengar, J. Tomasi, V. Barone, B. Mennucci, M. Cossi, G. Scalmani, N. Rega, G. A. Petersson, H. Nakatsuji, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, M. Klene, X. Li, J. E. Knox, H. P. Hratchian, J. B. Cross, V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, P. Y. Ayala, K. Morokuma, G. A. Voth, P. Salvador, J. J. Dannenberg, V. G. Zakrzewski, S. Dapprich, A. D. Daniels, M. C. Strain, O. Farkas, D. K. Malick, A. D. Rabuck, K. Raghavachari, J. B. Foresman, J. V. Ortiz, Q. Cui, A. G. Baboul, S. Clifford, J. Cioslowski, B. B. Stefanov, G. Liu, A. Liashenko, P. Piskorz, I. Komaromi, R. L. Martin, D. J. Fox, T. Keith, M. A. Al-Laham, C. Y. Peng, A. Nanayakkara, M. Challacombe, P. M. W. Gill, B. Johnson, W. Chen, M. W. Wong, C. Gonzalez, and J. A. Pople, Gaussian 03; Gaussian, Inc., Wallingford CT, 2004.
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[7] C. Lee, W. Yang, and R. G. Parr, *Phys. Rev. B* **1988**, *37*, 785.
[8] (a) R. Ditchfield, W. J. Hehre, and J. A. Pople, *J. Chem. Phys.* **1971**, *54*, 724. (b) W. J. Hehre, R. Ditchfield, and J. A. Pople, *J. Chem. Phys.* **1972**, *56*, 2257. (c) P. C. Hariharan, and J. A. Pople, *Theor. Chim. Acta* **1973**, *28*, 213.
[9] a) F. Stahl, D. Moran, P. V. Schleyer, M. Prall, and P. R. Schreiner, *J. Org. Chem.* **2002**, *67*, 1453. b) P. R. Schreiner, A. Navarro-Vazquez, and M. Prall, *Acc. Chem. Res.* **2005**, *38*, 29 and references therein. c) M. Prall, A. Wittkopp, and P. R. Schreiner, *J. Phys. Chem. A* **2001**, *105*, 9265. d) P. G. Wenthold, and M. A. Lipton, *J. Am. Chem. Soc.* **2000**, *122*, 9265.
[10] K. Yamaguchi, F. Jensen, A. Dorigo, and K. N. Houk, *Chem. Phys. Lett.* **1988**, *149*, 537.

6d

C	4.81794	-1.68452	0.04192
C	3.98845	-2.71868	0.51388
C	2.59923	-2.55478	0.48695
C	1.99769	-1.36113	-0.00209
C	2.84283	-0.29251	-0.45904
C	4.24443	-0.49928	-0.43571
C	0.57238	-1.31269	-0.04338
C	-0.65124	-1.43716	-0.05498
C	-2.07358	-1.54250	-0.09795
C	-2.75898	-2.45206	0.75442
C	-4.15563	-2.55827	0.70513
C	-4.89870	-1.76767	-0.19130
C	-4.23257	-0.86595	-1.04253
C	-2.83656	-0.75023	-1.00037
C	2.36727	1.01788	-0.94937
C	1.17199	1.58811	-0.84677
C	0.04303	2.25881	-0.76686
C	-0.39331	3.19766	0.37539
C	0.70964	3.33522	1.45364
C	-0.70484	4.59317	-0.24162
C	-1.68674	2.61541	1.01782
H	5.90570	-1.80001	0.04797
H	4.42075	-3.64961	0.89164
H	1.94186	-3.35642	0.83282
H	4.89269	0.30671	-0.79442
H	-2.18028	-3.06582	1.44936
H	-4.66721	-3.26203	1.36860
H	-5.98852	-1.85448	-0.22735
H	-4.80449	-0.25104	-1.74405
H	-2.31622	-0.05456	-1.66282
H	3.15279	1.61540	-1.43668
H	-0.69622	2.14670	-1.57559
H	0.37588	4.01318	2.25792
H	0.94915	2.35907	1.90642
H	1.63958	3.74529	1.02560
H	-1.07509	5.28590	0.53469
H	-1.47889	4.52128	-1.02574
H	0.19689	5.03753	-0.69585
H	-1.49450	1.62663	1.46502
H	-2.06134	3.28792	1.81002
H	-2.48893	2.49532	0.26929

7d

C	-1.23816	-0.40114	-0.00120
C	-0.06972	0.58564	-0.00309
C	-0.67904	1.94589	-0.00191
C	-2.10789	1.78988	0.00103
C	-2.42865	0.38965	0.00145
C	1.22000	0.29228	-0.00637
C	2.56772	0.00561	-0.00294
C	3.30642	-0.15205	-1.23839
C	4.66887	-0.44178	-1.21842
C	5.36182	-0.58891	0.00492
C	4.66244	-0.43970	1.22435
C	3.29991	-0.14993	1.23671
C	-1.05117	-1.77569	-0.00194
C	-0.09308	3.21450	-0.00298
C	-2.93560	2.93941	0.00298
C	-2.10347	-2.88641	-0.00004
C	-1.36888	-4.25810	-0.00184
C	-2.99702	-2.80774	1.27857
C	-3.00217	-2.80715	-1.27501
C	-2.33701	4.20867	0.00191
C	-0.93026	4.35065	-0.00104
H	-3.44483	-0.00330	0.00356
H	2.77237	-0.03833	-2.18483
H	5.20662	-0.55612	-2.16477
H	6.43116	-0.81635	0.00793
H	5.19520	-0.55242	2.17371
H	2.76086	-0.03458	2.18010
H	-0.00681	-2.11040	-0.00424
H	0.99567	3.32405	-0.00531
H	-4.02525	2.83719	0.00526
H	-2.09234	-5.09127	-0.00058
H	-0.72953	-4.36550	-0.89536
H	-0.72593	-4.36591	0.88905
H	-3.73396	-3.63058	1.28239
H	-2.38403	-2.89466	2.19149
H	-3.54912	-1.85679	1.33721
H	-3.73917	-3.62995	-1.27620
H	-3.55443	-1.85613	-1.33100
H	-2.39288	-2.89371	-2.19043
H	-2.96771	5.10294	0.00337
H	-0.48638	5.35035	-0.00185

18

C	0.55335	1.37115	-0.12505
C	0.88067	-0.31297	-0.86018
C	2.28932	-0.44153	-0.56225
C	2.68567	0.54657	0.41839
C	1.59127	1.44156	0.75534
C	-0.38458	-0.54805	-0.58829
C	-0.78714	1.59130	-0.56182
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C	-1.06829	4.08885	-0.44617
C	4.91339	-0.44070	0.46599
C	4.54350	-1.36025	-0.54351
H	1.66545	2.14480	1.59134
H	-0.96619	1.34285	-1.60669
H	2.93853	-2.09199	-1.81563
H	4.29318	1.22593	1.71352
H	-3.76239	3.38629	-0.33904
H	-3.04020	2.58833	-1.76507
H	-3.56424	1.60947	-0.36472
H	-2.43404	3.46319	1.90714
H	-2.25606	1.68357	1.83705
H	-0.81383	2.71721	2.00313
H	-1.70399	4.91562	-0.08184
H	-0.05946	4.21525	-0.02001
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C	-1.35546	-1.55263	-0.21706
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H	0.15529	-2.96007	0.46776
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H	-4.74060	-2.15362	-0.16398
H	-1.49277	-4.73970	1.07445
H	-3.94219	-4.33891	0.75665

16

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C	-0.12769	1.79054	-0.43985
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17

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15d

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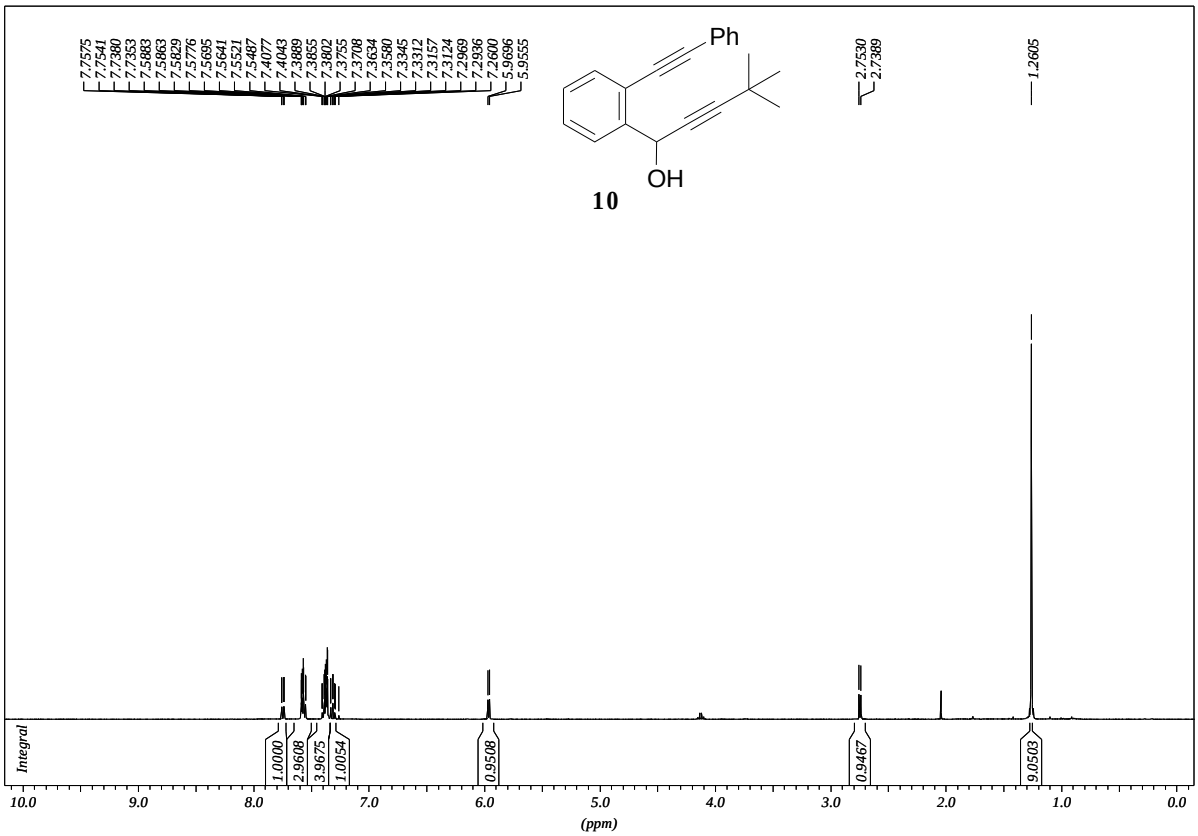
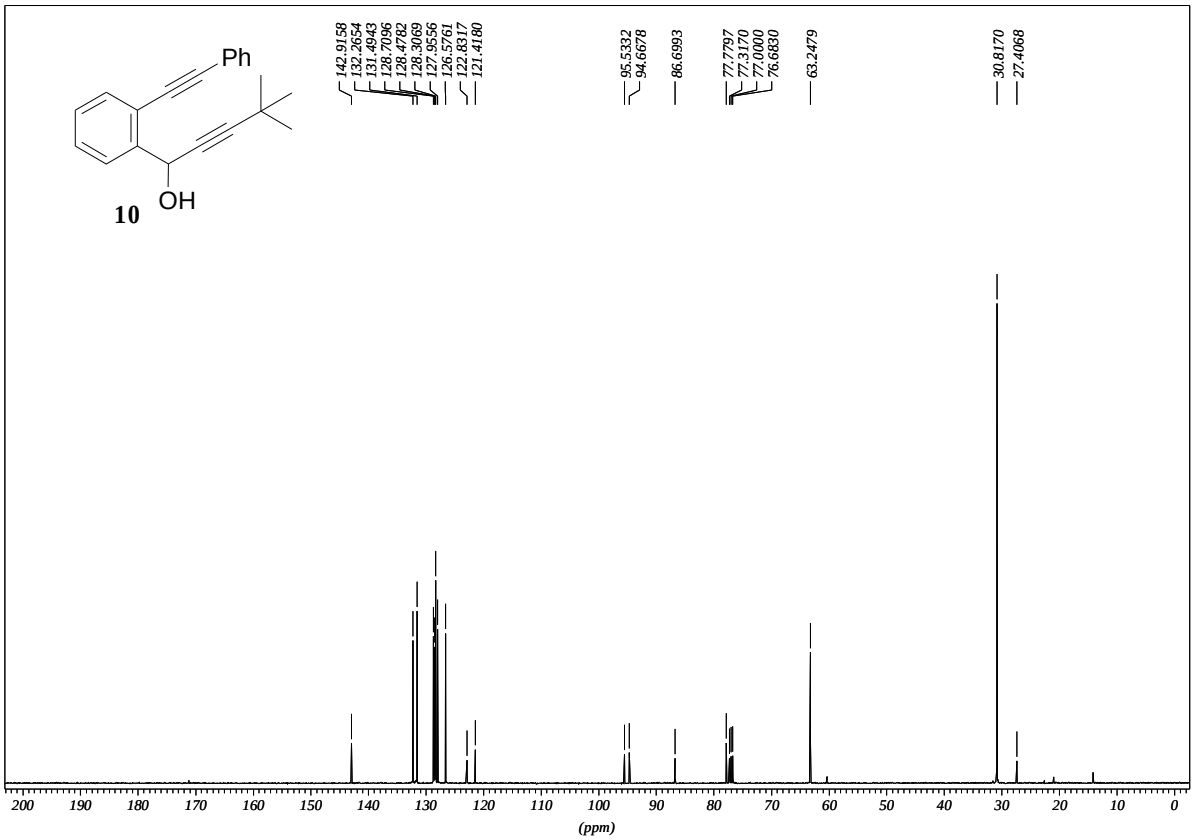
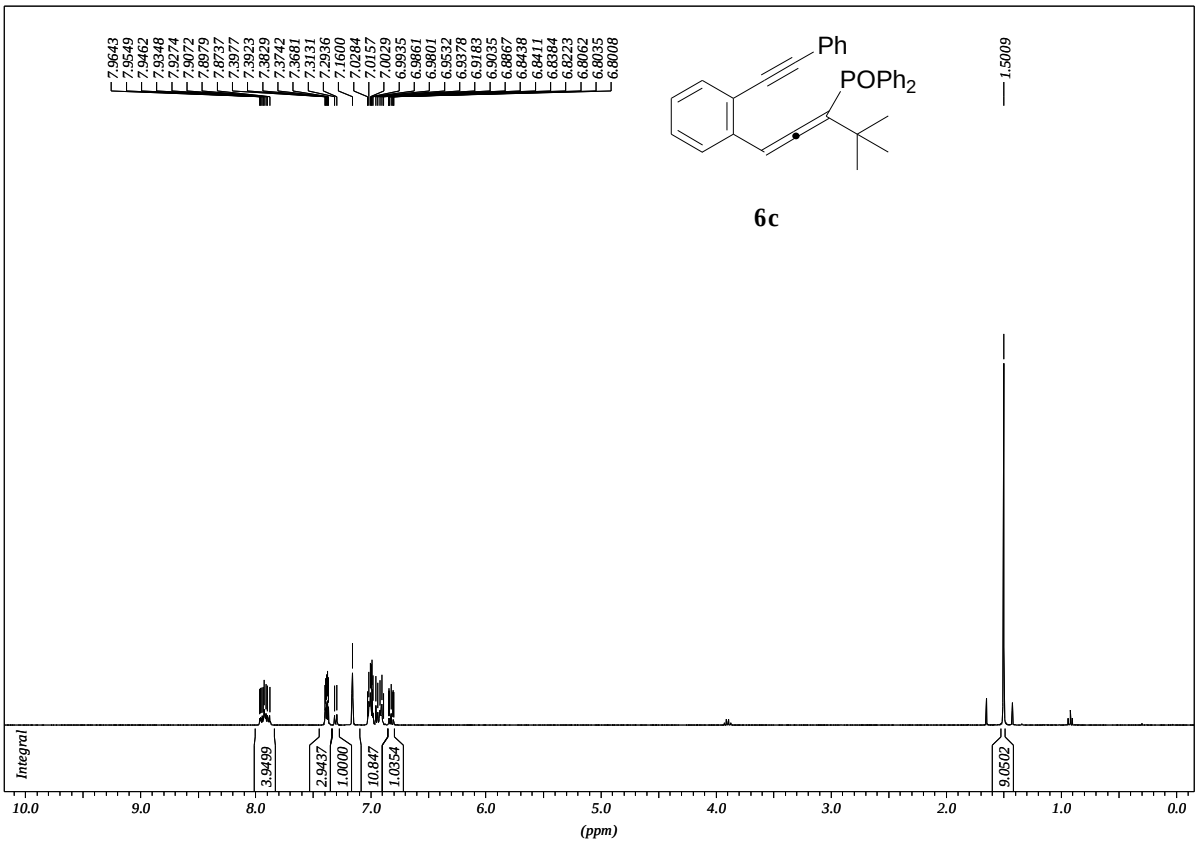
^{1}H and ^{13}C-NMR Spectra



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Ph
POPh2
6c
Integral
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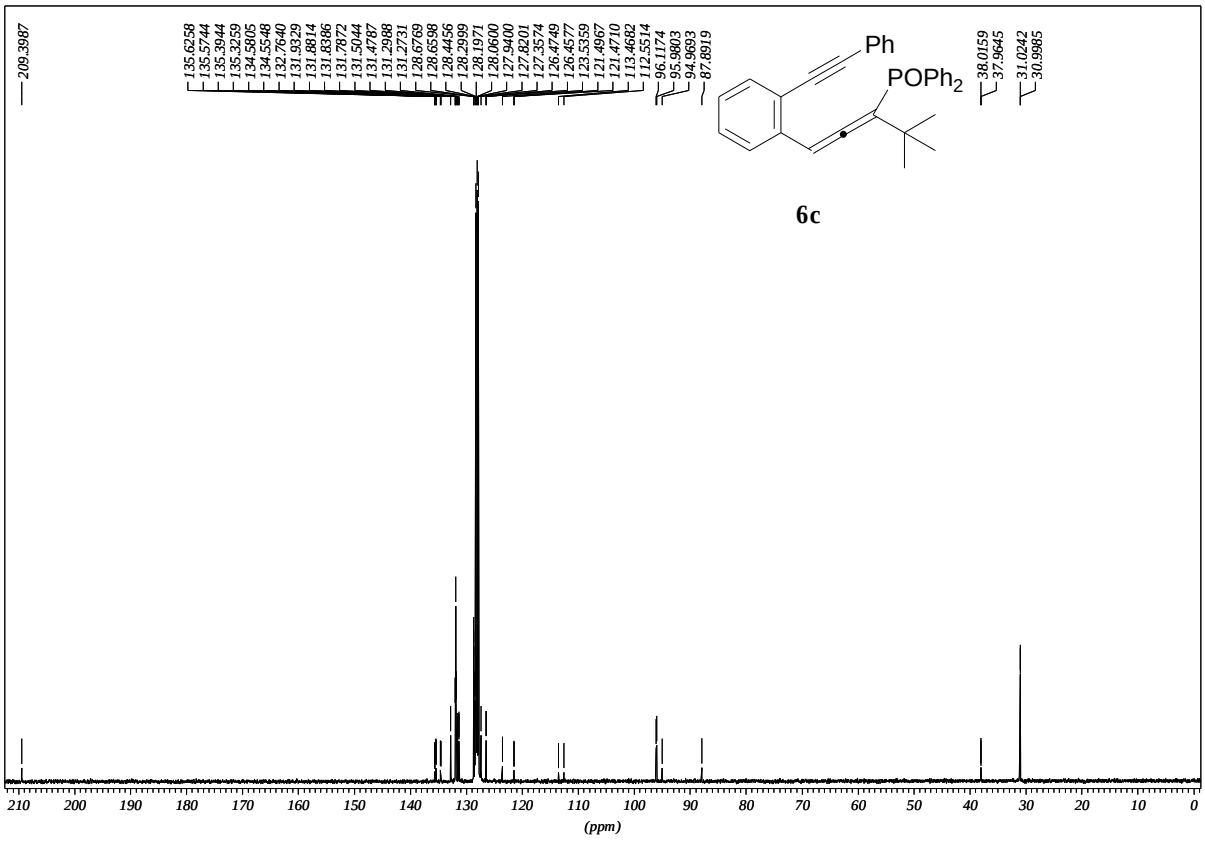
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200
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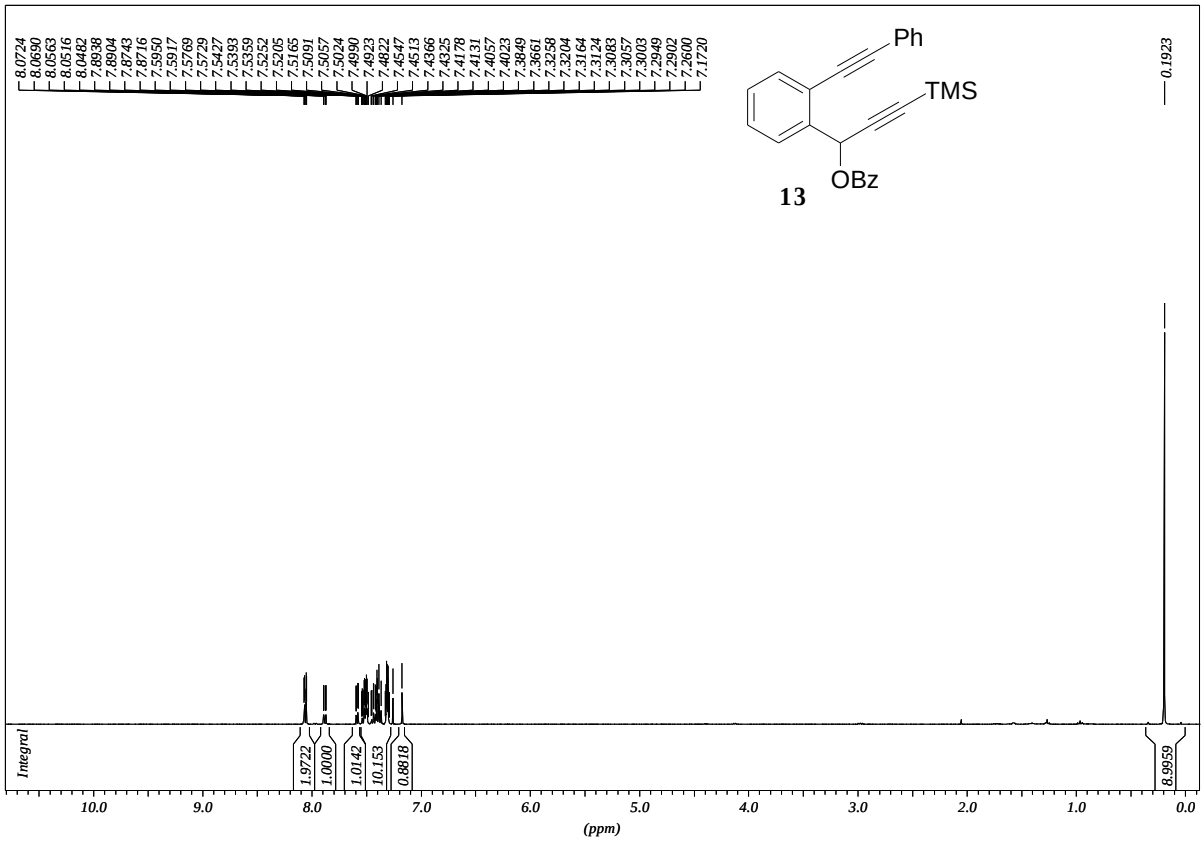
(ppm)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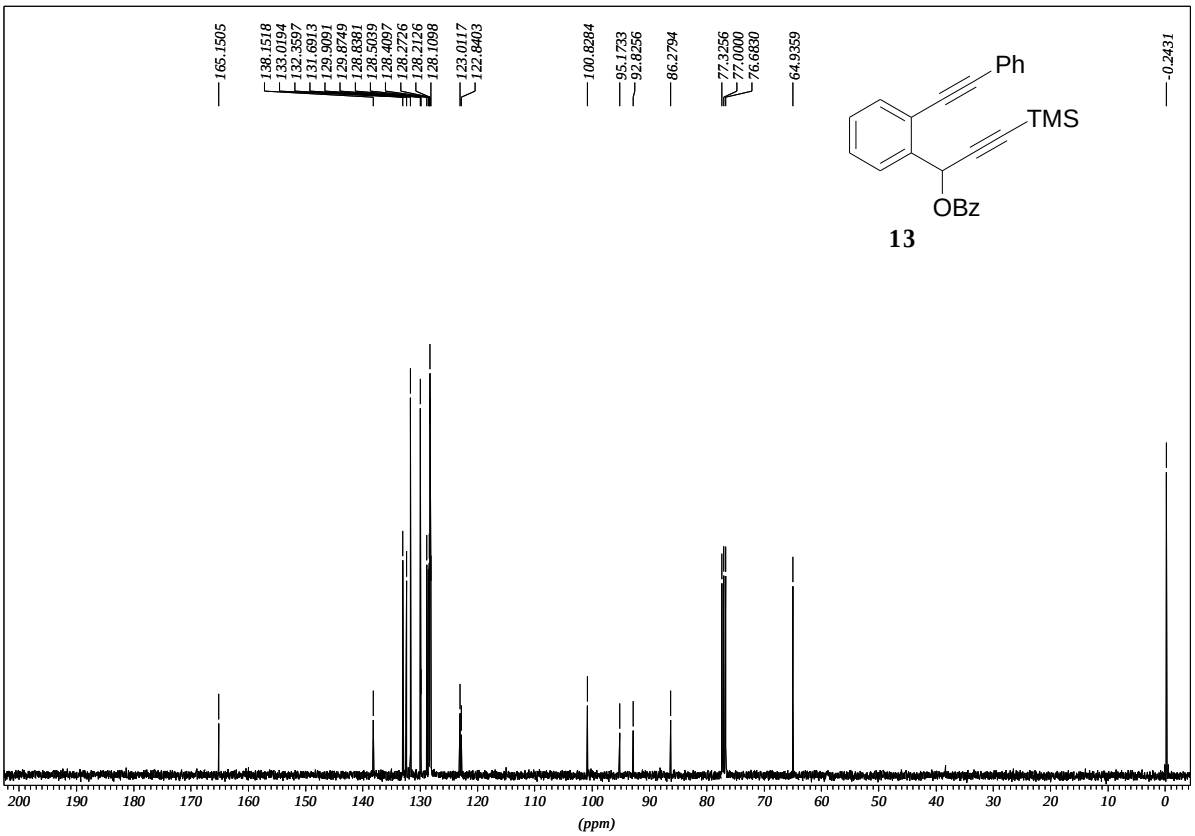
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OBz
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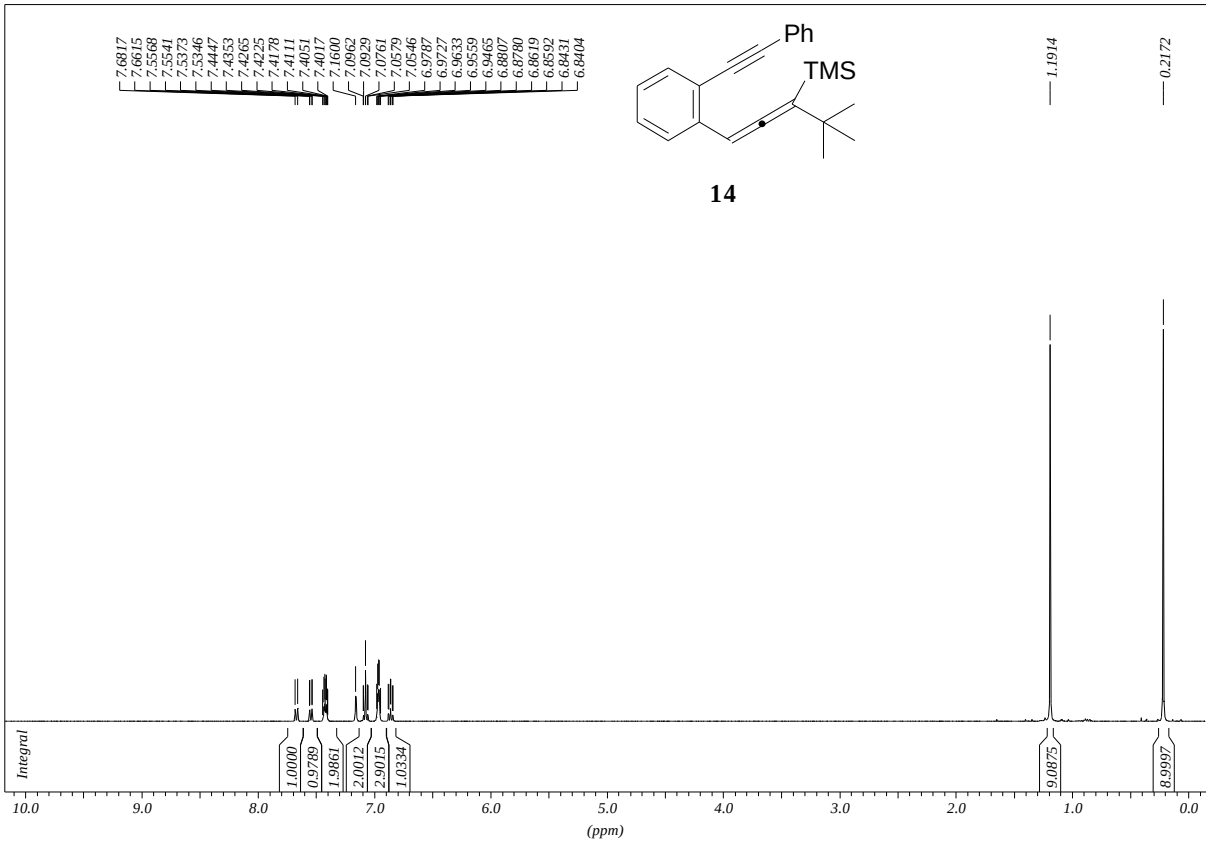
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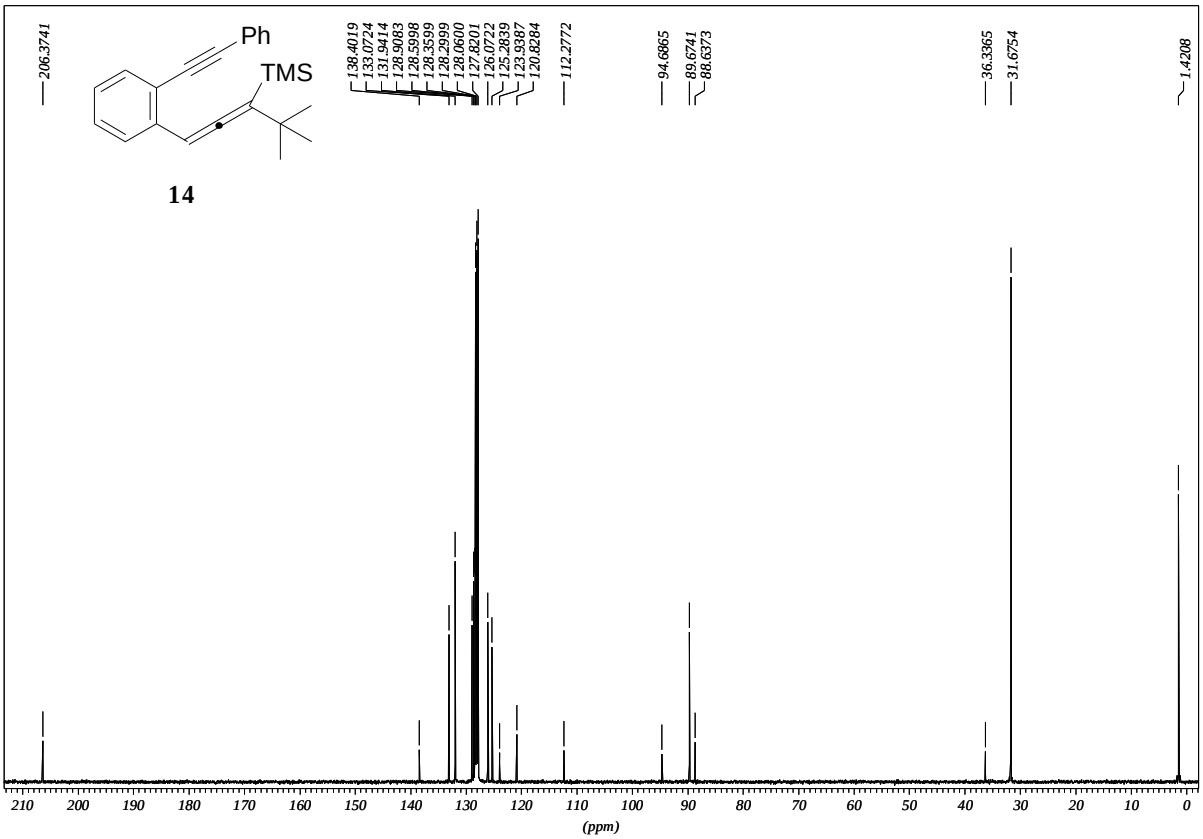
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200
190
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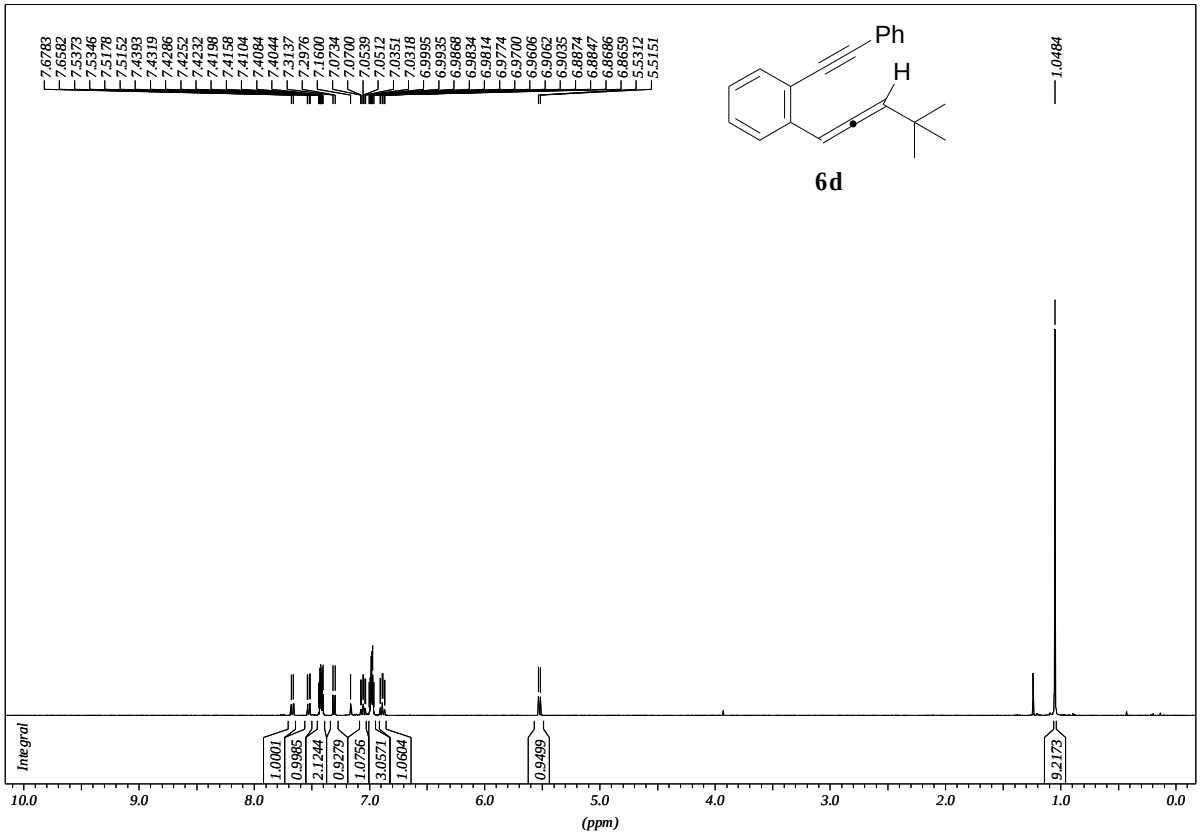
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6.9834
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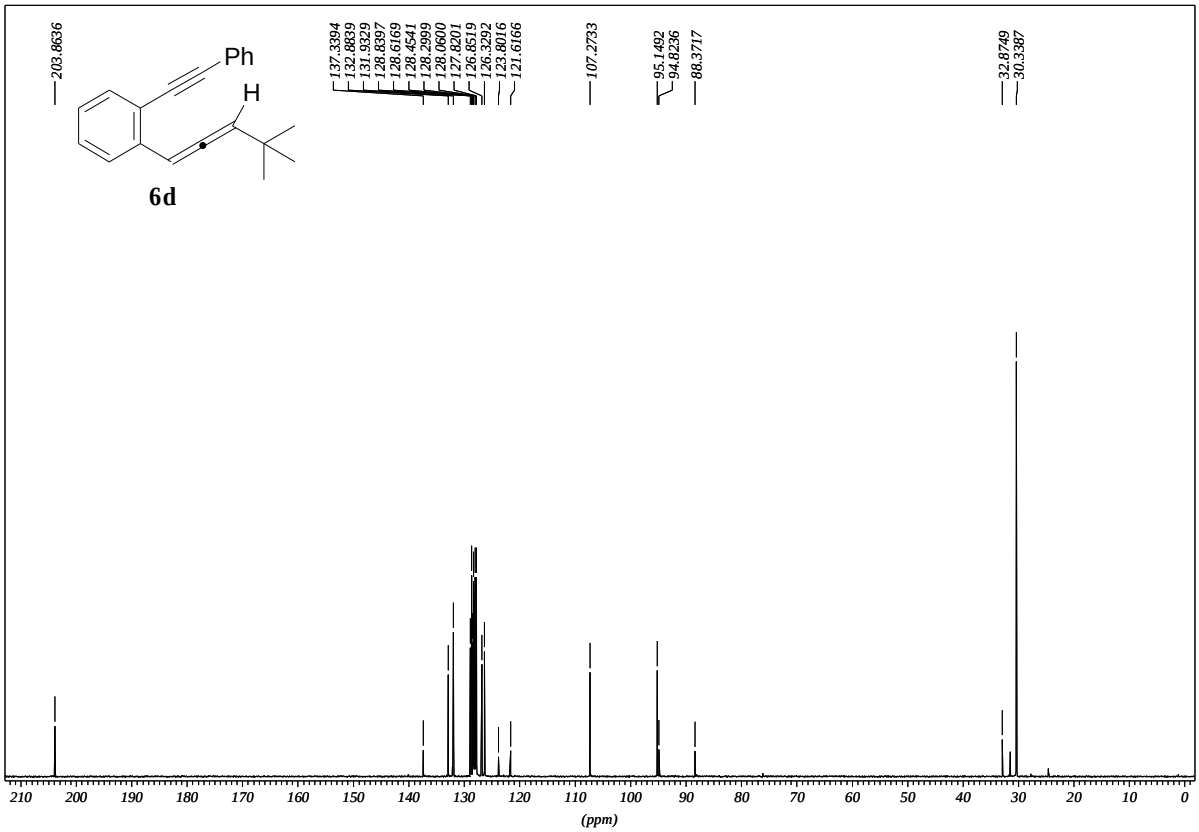
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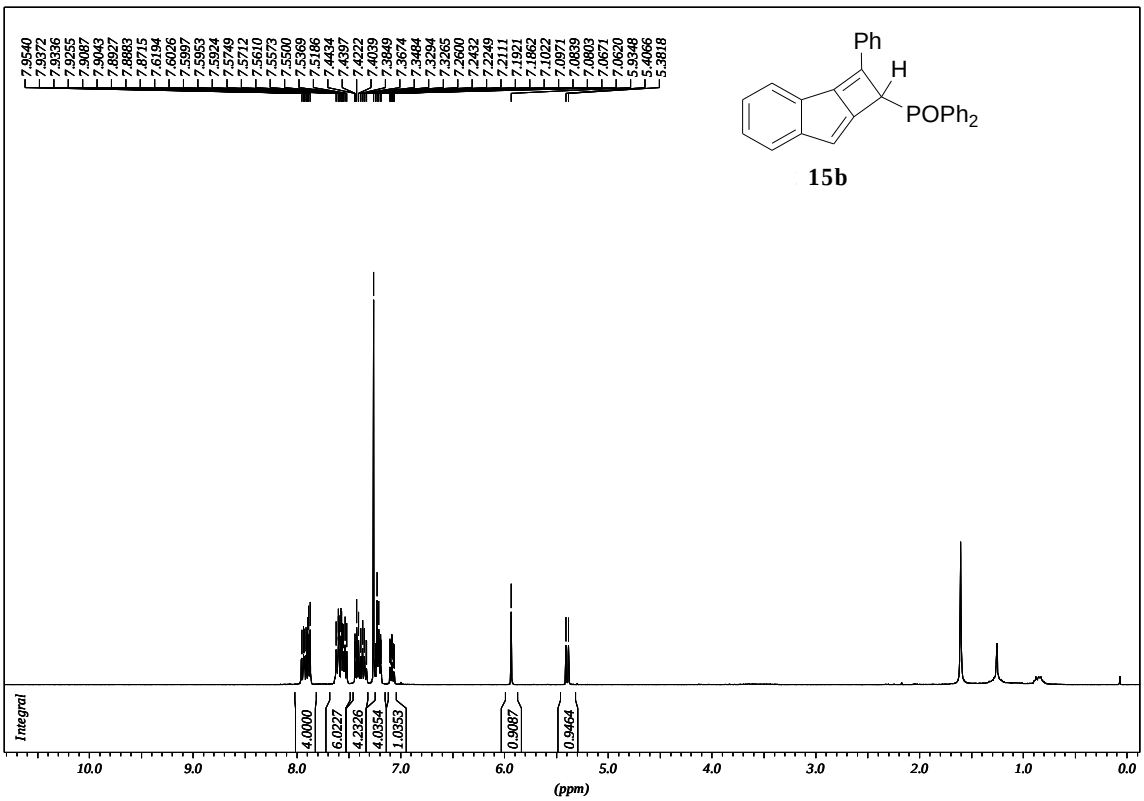
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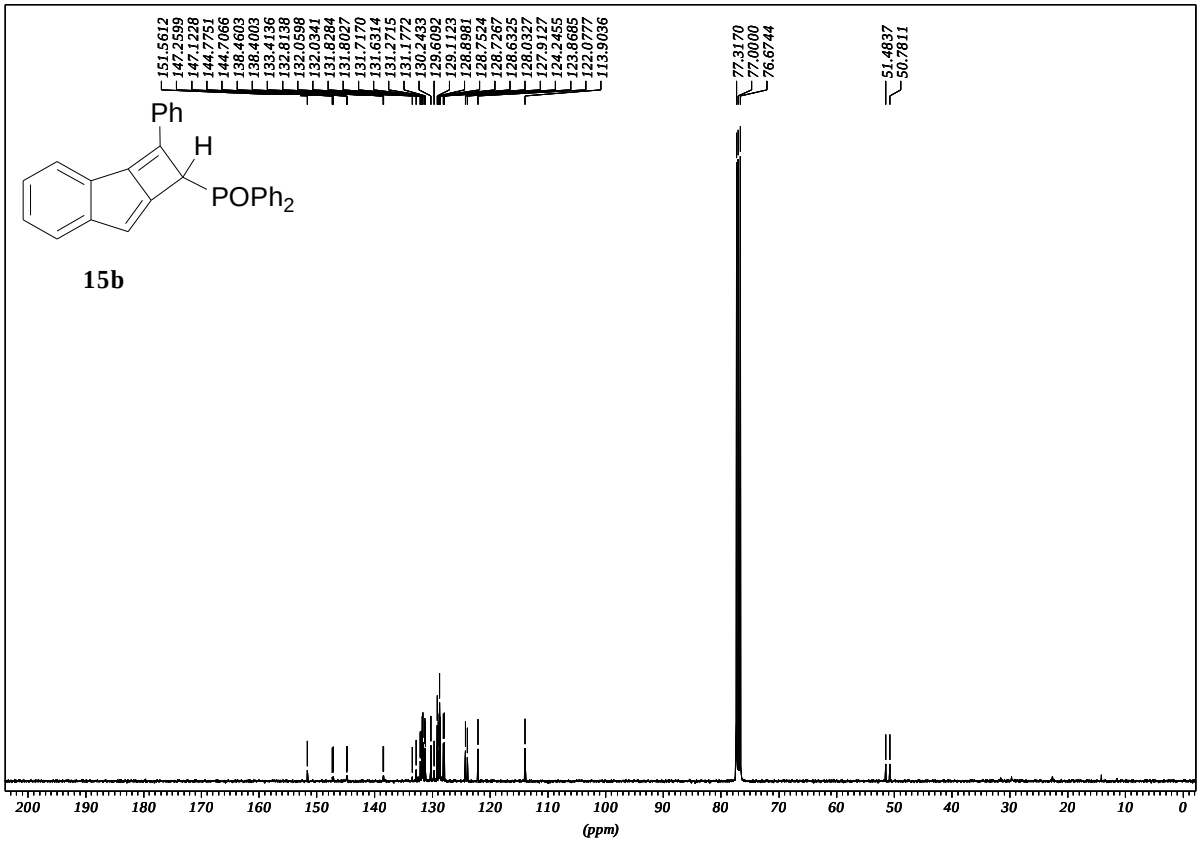
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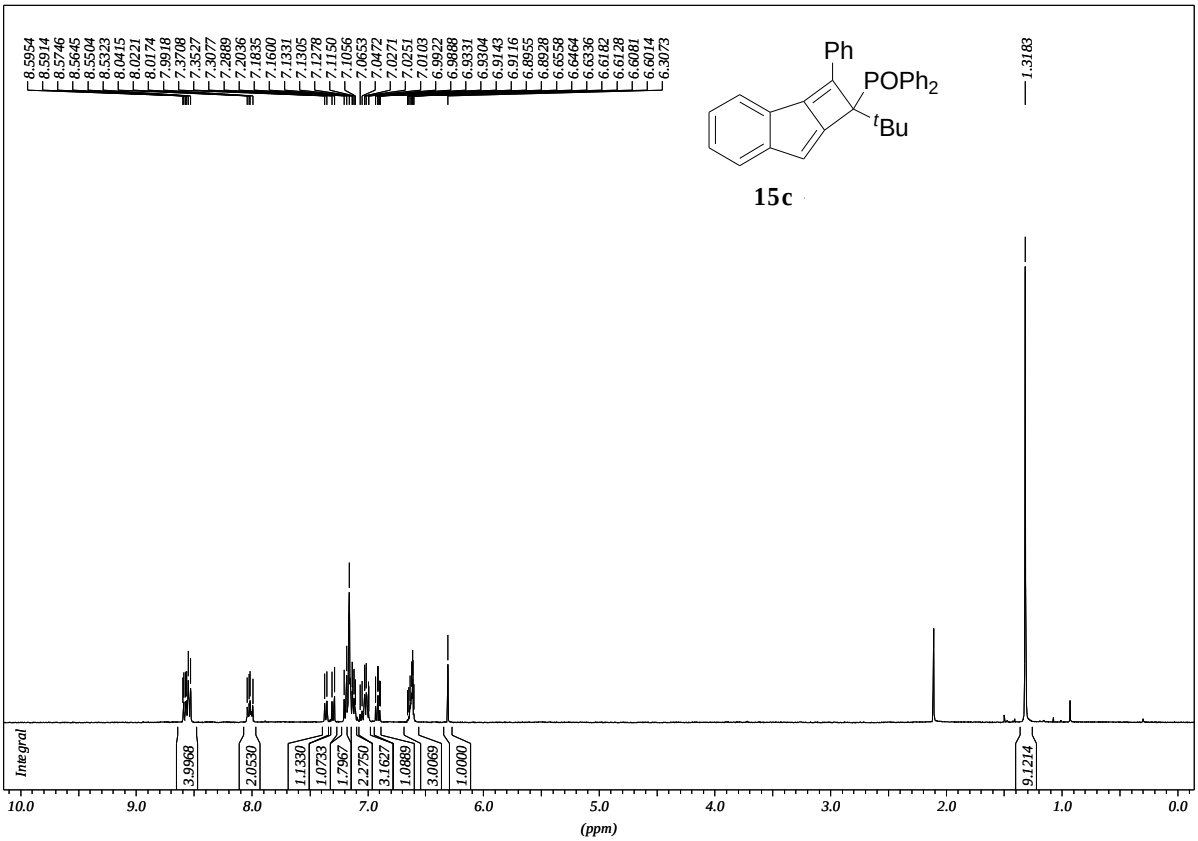
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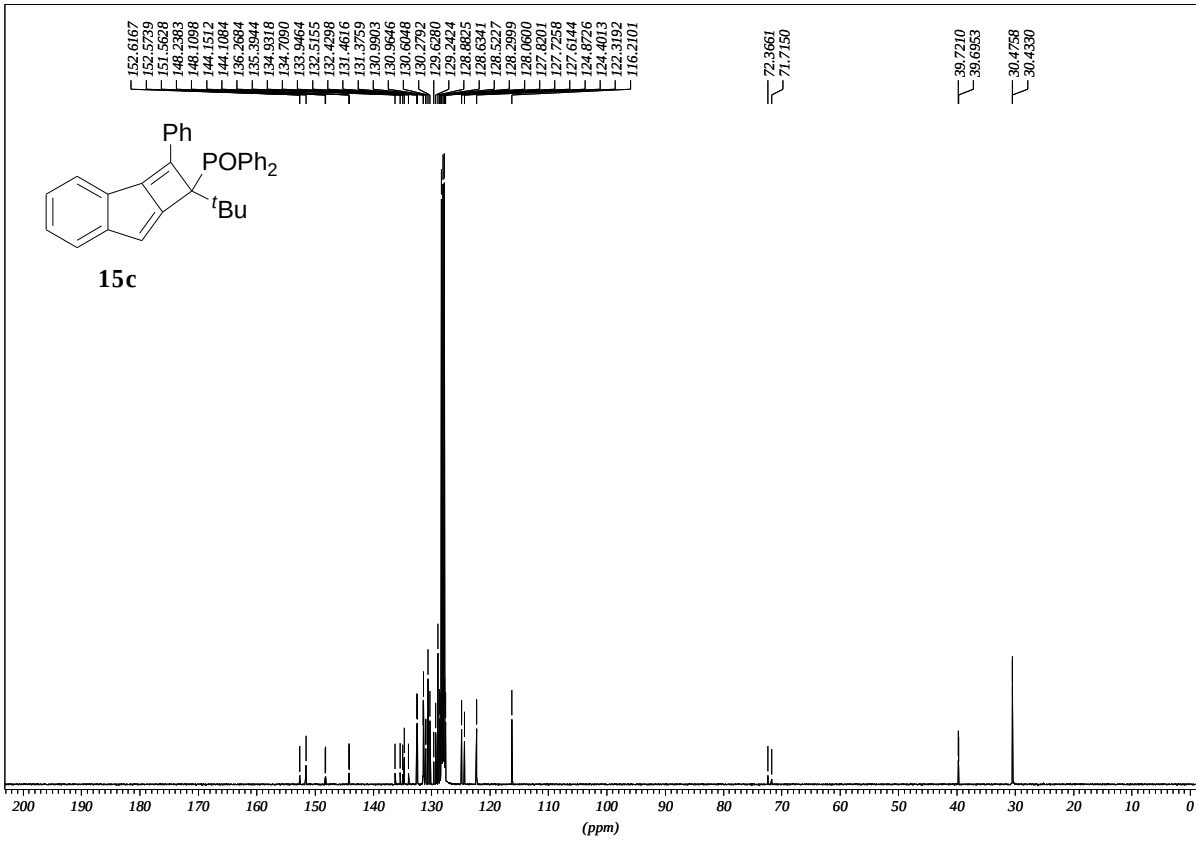
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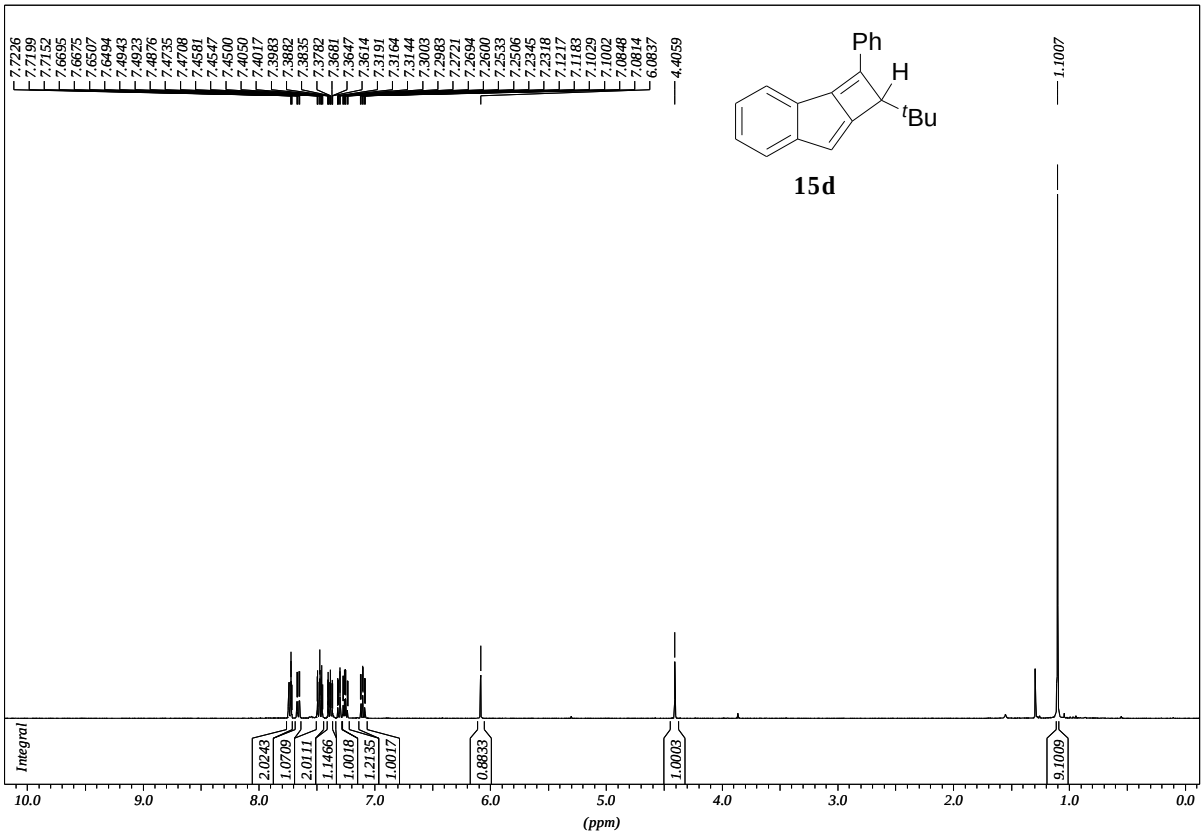
(ppm)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7.7152
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7.6494
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7.4708
7.4581
7.4547
7.4500
7.4050
7.4017
7.3983
7.3882
7.3835
7.3782
7.3681
7.3647
7.3614
7.3191
7.3164
7.3144
7.3003
7.2983
7.2721
7.2694
7.2600
7.2533
7.2506
7.2345
7.2318
7.1217
7.1183
7.1029
7.1002
7.0848
7.0814
6.0837
4.4059
1.1007
Ph
H
tBu
15d
Integral
2.0243
1.0709
2.0111
1.1466
1.0018
1.2135
1.0017
0.8833
1.0003
9.1009
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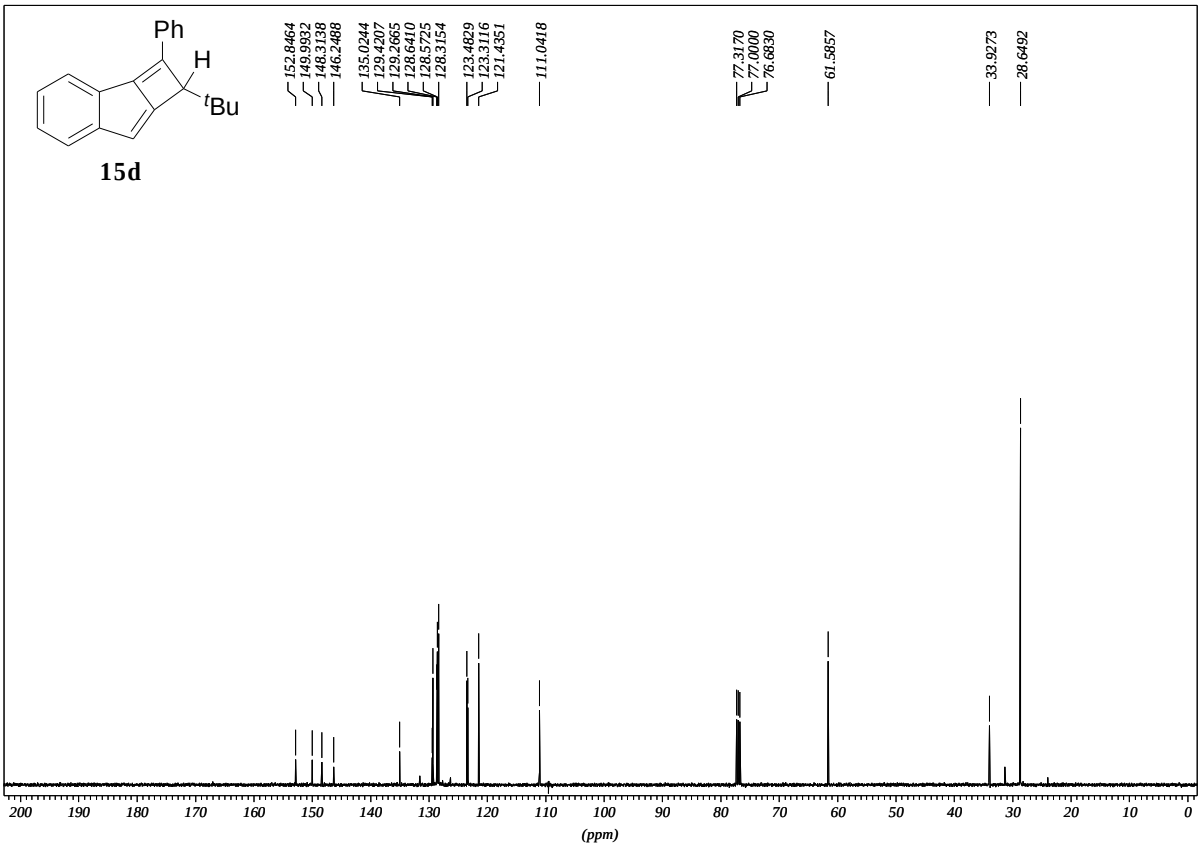
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)


Ph
H
tBu
15d
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123.4829
123.3116
121.4351
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77.3170
77.0000
76.6830
61.5857
33.9273
28.6492
200
190
180
170
160
150
140
130
120
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100
90
80
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60
50
40
30
20
10
0
(ppm)