Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

N-Allenylnitrone Acts as 2-Azadiene in the Cu-Catalyzed Cascade Reaction of *O*-propargylic Oximes with Azodicarboxylates

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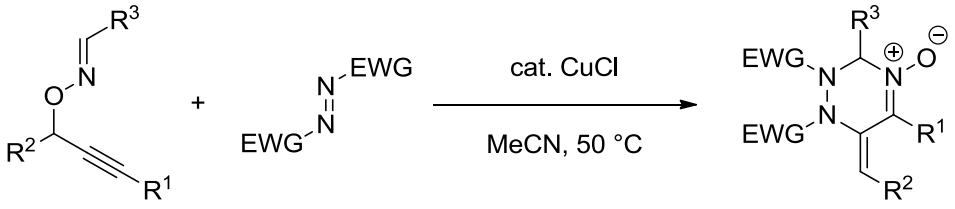


Supporting Information

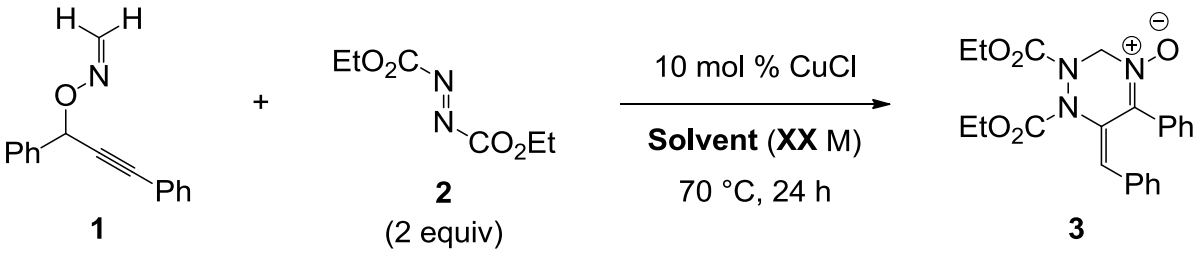
Contents

1. General information

^{1}H and ^{13}C NMR spectra were recorded on a JEOL JNM-ECS400 (400 MHz for ^{1}H and 100 MHz for ^{13}C) spectrometer. Chemical shifts are reported in ppm relative to $CHCl_3$ (for ^{1}H, δ 7.24), and $CDCl_3$ (for ^{13}C, δ 77.00). ^{1}H NMR data are reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, sext = sextet, dd = double doublet, dq = double quartet, tq = triple quartet, br = broad, m = multiplet) and coupling constants (Hz). Infrared spectra were recorded on a JASCO FT/IR-4100 spectrometer. Optically rotations were measured on a Jasco P-1020 digital polarimeter with a sodium lamp and reported as follows; [α] T ºC D (c = g/100 mL, solvent). High-resolution mass spectra analysis was performed on a Bruker Daltonics solariX FT-ICR-MS spectrometer at the Instrumental Analysis Center for Chemistry, Graduate School of Science, Tohoku University. X-ray crystallographic data was obtained by Rigaku XtaLAB mini diffractometer at the Instrumental Analysis Center for Chemistry, Graduate School of Science, Tohoku University. Flash column chromatography was performed with Kanto Chemical silica gel 60N (spherical, neutral, 40-50 μm). Analytical thin layer chromatography (TLC) was performed on Merck precoated TLC plates (silica gel 60 F_{254}). All reactions were carried out under argon atmosphere. Anhydrous MeCN, 1,4-dioxane, toluene, 1,2-dichloroethane, EtOAc, DMF were purchased from WAKO and used as received. CuCl was purchased from WAKO. Substrates **1** were synthesized in accordance with the literature method[1].

[1] I. Nakamura, Y. Kudo, T. Araki, D. Zhang, E. Kwon, M. Terada, *Synthesis*, **2012**, *44*, 1542.
G. Gao, D. Moore, R. Xie, L. Pu, *Org. Lett.*, **2002**, *4*, 4143

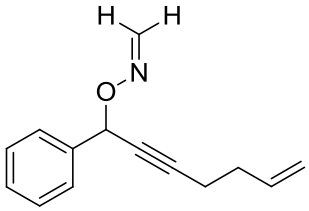
2. Solvent effect



Entry	Solvent	**XX**	**3** (%)[a]	**1** (%)[b]
1	MeCN	0.5	63	<1
2	1,4-dioxane	0.5	32	<1
3	Toluene	0.5	35	<1
4	1,2-DCE	0.5	35	<1
5	EtOAc	0.5	30	5
6	DMF	0.5	26	<1
7	MeCN	1.0	56	<1
8	MeCN	0.25	66	<1

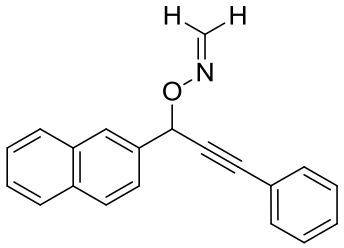
[a]Isolated yield. [b1]H NMR yield using CH_2Br_2 as an internal standard.

3. Analytical data of 1



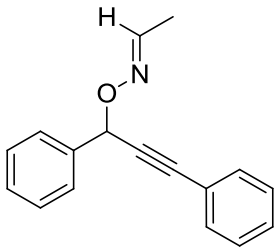
1e

Formaldehyde *O*-1-phenylhept-6-en-2-yn-1-yl oxime (1e). ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.29-2.34 (m, 2H), 2.39-2.43 (m, 2H), 5.02-5.11 (m, 2H), 5.82-5.90 (m, 2H), 6.53 (d, *J* = 8.2 Hz, 1H), 7.09 (d, *J* = 7.8 Hz, 1H), 7.33-7.41 (m, 3H), 7.54-7.56 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.7, 32.6, 75.5, 77.9, 88.0, 115.7, 127.8, 128.3, 128.5, 136.7, 137.9, 138.2. IR (neat) 3066, 3033, 2979, 2909, 2843, 2284, 2228, 1641, 1494, 1454, 1333, 1300, 1273, 1136, 984, 919, 883 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 236.1046, found. 236.1045.



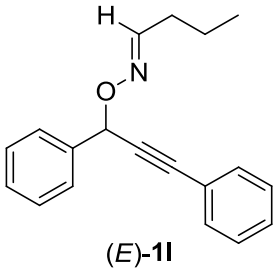
1i

Formaldehyde *O*-1-(2-naphthyl)-3-phenylprop-2-ynyl oxime (1i). mp 61-62 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.25 (s, 1H), 6.57 (d, *J* = 8.0 Hz, 1H), 7.15 (d, *J* = 8.0 Hz, 1H), 7.32-7.33 (m, 3H), 7.49-7.54 (m, 4H), 7.72 (d, *J* = 8.5 Hz, 1H), 7.84-7.90 (m, 3H), 8.08 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 75.9, 86.4, 88.1, 122.4, 125.4, 126.3, 126.5, 127.2, 127.7, 128.2, 128.3, 128.5, 128.6, 131.9, 133.1, 133.4, 135.0, 138.7. IR (neat) 3055, 2950, 2900, 2226, 1598, 1489, 1442, 1365, 1307, 1123, 977, 948, 917, 874, 817 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 308.1046, found. 308.1045.



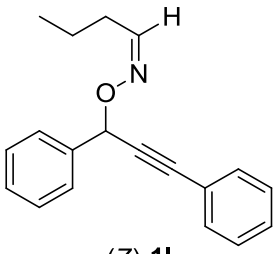
(*E*)-**1k**

(*E*)-Acetaldehyde *O*-1,3-diphenylprop-2-ynyl oxime ((*E*)-1k). mp 61-62 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.83 (d, *J* = 6.0 Hz, 3H), 6.01 (s, 1H), 7.28-7.43 (m, 6H), 7.48-7.55 (m, 3H), 7.61-7.63 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 15.3, 75.2, 86.9, 87.4, 122.6, 127.9, 128.2, 128.5, 128.5, 128.6, 131.9, 137.8, 148.4. IR (neat) 3062, 3032, 2999, 2917, 2227, 1597, 1489, 1442, 1300, 1276, 1016, 997, 977, 889 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 272.1046, found. 272.1045.



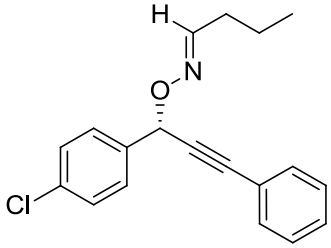
(*E*)-**1l**

(*E*)-Butyraldehyde *O*-1,3-diphenylprop-2-ynyl oxime ((*E*)-1l). ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.94 (t, *J* = 7.2 Hz, 3H), 1.52 (sext, *J* = 7.2 Hz, 2H), 2.20 (q, *J* = 7.2 Hz, 2H), 6.02 (s, 1H), 7.25-7.41 (m, 6H), 7.47-7.51 (m, 3H), 7.62-7.64 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.6, 20.0, 31.4, 75.2, 87.0, 87.4, 122.6, 128.0, 128.2, 128.5, 128.5, 128.6, 131.9, 137.8, 152.5. IR (neat) 3063, 3032, 2961, 2933, 2872, 2228, 1598, 1489, 1454, 1299, 1277, 1027, 963, 922, 880 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 300.1359, found. 300.1359.



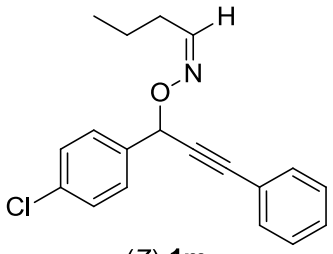
(*Z*)-**1l**

(*Z*)-Butyraldehyde *O*-1,3-diphenylprop-2-ynyl oxime ((*Z*)-1l). ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.95 (t, *J* = 7.3 Hz, 3H), 1.51 (sext, *J* = 7.3 Hz, 2H), 2.31-2.45 (m, 2H), 6.05 (s, 1H), 6.77 (t, *J* = 5.5 Hz, 1H), 7.29-7.43 (m, 6H), 7.48-7.51 (m, 2H), 7.60-7.62 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.8, 19.5, 27.9, 75.3, 87.1, 87.3, 122.6, 127.7, 128.1, 128.4, 128.5, 128.5, 131.9, 138.1, 153.3. IR (neat) 3062, 3032, 2961, 2931, 2872, 2229, 1598, 1489, 1454, 1443, 1299, 1279, 1029, 964, 912, 876 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 300.1359, found. 300.1359.



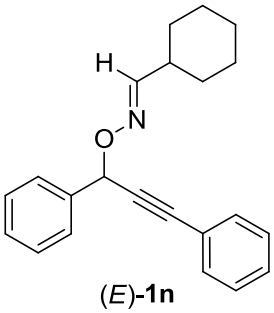
(*S*,*E*)-**1m**

(*S*,*E*)-Butyraldehyde *O*-1-(4chlorophenyl)-3-phenylprop-2-ynyl oxime ((*S*,*E*)-1m). $[\alpha]^{20}_{D}$ = -6.80 (*c* 1.00, $CHCl_3$), HPLC analysis Chiralcel IA-3 (Hexane/IPA = 99/1, 1.0 mL/min, 254 nm, 30 ºC), 6.3, 7.2 min (major), (99% ee). ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.94 (t, *J* = 7.3 Hz, 3H), 1.52 (sext, *J* = 7.3 Hz, 2H), 2.20 (q, *J* = 7.3 Hz, 2H), 5.98 (s, 1H), 7.29-7.39 (m, 5H), 7.46-7.50 (m, 3H), 7.54-7.56 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.5, 19.9, 31.4, 74.4, 86.4, 87.7, 122.3, 128.2, 128.6, 128.6, 129.3, 131.9, 134.4, 136.4, 152.7. IR (neat) 3054, 2961, 2931, 2873, 2228, 1597, 1489, 1442, 1408, 1298, 1090, 1032, 1016, 920, 883, 826 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 334.0969, found. 334.0969.



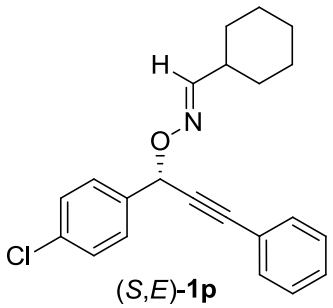
(*Z*)-**1m**

(*Z*)-Butyraldehyde *O*-1-(4chlorophenyl)-3-phenylprop-2-ynyl oxime ((*Z*)-1m). ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.95 (t, *J* = 7.3 Hz, 3H), 1.51 (sext, *J* = 7.3 Hz, 2H), 2.31-2.41 (m, 2H), 6.01 (s, 1H), 6.76 (t, *J* = 5.5 Hz, 1H), 7.28-7.39 (m, 5H), 7.48-7.55 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.8, 19.5, 27.9, 74.6, 86.5, 87.5, 122.3, 128.2, 128.6, 129.2, 131.9, 134.3, 136.7, 153.6. IR (neat) 3057, 2960, 2931, 2873, 2230, 1597, 1489, 1442, 1408, 1298, 1288, 1089, 1032, 1015, 966, 912, 876, 824 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 334.0969, found. 334.0969.



(*E*)-**1n**

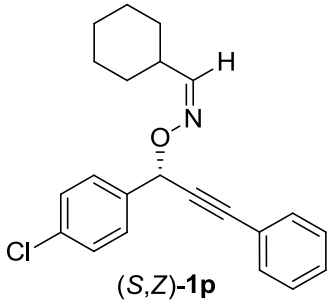
(*E*)-Cyclohexanecaboxaldehyde *O*-1,3-diphenylprop-2-ynyl oxime ((*E*)-1n). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.17-1.31 (m, 5H), 1.64-1.81 (m, 5H), 2.24-2.32 (m, 1H), 6.00 (s, 1H), 7.29-7.42 (m, 7H), 7.48-7.51 (m, 2H) 7.61-7.63 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.3, 25.8, 30.3, 38.5, 75.2, 87.0, 87.4, 122.6, 128.0, 128.2, 128.4, 128.5, 128.6, 131.8, 137.8, 156.4. IR (neat) 3063, 3032, 2925, 2851, 2228, 1598, 1489, 1449, 1301, 1028, 1016, 998, 923, 883 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 340.1672, found. 340.1671.



(*S*,*E*)-**1p**

(*S*,*E*)-Cyclohexanecaboxaldehyde *O*-1-(4chlorophenyl)-3-phenylprop-2-ynyl oxime ((*S*,*E*)-1p). mp 51-52 °C. $[\alpha]^{21}_{D}$ = -9.72 (*c* 1.01, $CHCl_3$), HPLC analysis Chiralcel IA-3 (Hexane/IPA = 99/1, 1.0 mL/min, 254 nm, 30 ºC), 6.1, 6.8 min (major), (99% ee). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.17-1.30 (m, 5H), 1.58-1.75 (m, 5H), 2.25-2.27 (m, 1H), 5.96 (s, 1H), 7.25-7.38 (m, 6H), 7.47-7.50 (m, 2H), 7.55 (d, *J* = 8.2 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.3, 25.7, 30.3, 38.5, 74.4, 86.4, 87.6, 122.3, 128.2, 128.6, 129.3, 131.8, 134.4, 136.5, 156.6. IR (neat) 3055, 2926, 2851, 2228, 1597, 1489, 1449, 1408, 1301, 1089, 1015, 911,

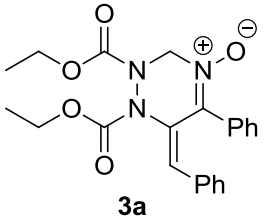
823 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 374.1282, found. 374.1282.



(*S*,*Z*)-**1p**

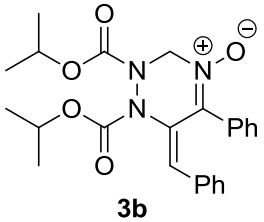
(*S*,*Z*)-Cyclohexanecaboxaldehyde *O*-1-(4chlorophenyl)-3-phenylprop-2-ynyl oxime ((*S*,*Z*)-1p). mp 91-93 °C. $[\alpha]^{22}_D$ = +24.53 (*c* 0.42, $CHCl_3$), HPLC analysis Chiralcel IA-3 (Hexane/IPA = 99/1, 1.0 mL/min, 254 nm, 30 ºC), 7.2, 9.2 min (major), (99% ee). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.14-1.34 (m, 5H), 1.64-1.83 (m, 5H), 2.91-2.95 (m, 1H), 5.99 (s, 1H), 6.58 (d, *J* = 7.3 Hz, 1H), 7.31-7.33 (m, 3H), 7.37 (d, *J* = 8.2 Hz, 1H), 7.48-7.51 (m, 2H), 7.54 (d, *J* = 8.2 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.1, 25.1, 25.8, 29.4, 29.5, 34.5, 74.5, 86.5, 87.6, 122.3, 128.2, 129.1, 131.9, 134.3, 136.8, 157.5. IR (neat) 3056, 2925, 2851, 2230, 1597, 1489, 1449, 1408, 1301, 1090, 1016, 957, 908, 883, 834 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 374.1282, found. 374.1282.

4. Analytical data of 3



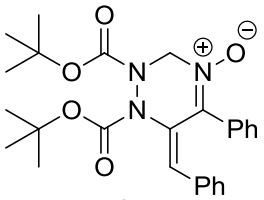
3a

(*E*)-6-benzylidene-1,2-bis(ethoxycarbonyl)-5-phenyl-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide (3a). mp 123-124 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.31 (t, *J* = 6.9 Hz, 3H), 1.39 (t, *J* = 7.1 Hz, 3H), 4.27-4.39 (m, 4H), 5.13 (d, *J* = 14.2 Hz, 1H), 5.90 (d, *J* = 14.2 Hz, 1H), 6.79-6.81 (m, 2H), 6.87-6.91 (m, 2H), 6.93-6.97 (m, 1H), 7.01-7.05 (m, 3H), 7.08 (s, 1H), 7.38-7.39 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.3, 14.3, 63.6, 63.6, 68.0, 124.3, 127.2, 127.3, 127.6, 127.7, 128.9, 129.3, 130.3, 132.3, 132.7, 140.5, 153.9. IR (neat) 3058, 3026, 2982, 2936, 1731, 1512, 1490, 1444, 1401, 1375, 1299, 1213, 1179, 1095, 1075, 1021 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 432.1530, found. 432.1529.



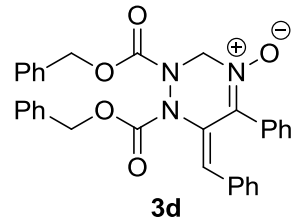
3b

(*E*)-6-benzylidene-1,2-bis(isopropoxycarbonyl)-5-phenyl-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide (3b). mp 97-98 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.25 (br, 3H), 1.33 (d, *J* = 6.2 Hz, 3H), 1.35 (d, *J* = 6.4 Hz, 3H), 1.39 (d, *J* = 6.2 Hz, 3H), 5.05-5.12 (m, 3H), 5.90 (br, 1H), 6.79-6.80 (m, 2H), 6.87-6.91 (m, 2H), 6.93-6.95 (m, 1H), 7.02-7.04 (m, 4H), 7.37-7.39 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.8, 21.9, 22.0, 68.1, 71.7, 71.9, 124.5, 127.3, 127.4, 127.6, 127.8, 128.9, 129.3, 130.4, 132.2, 132.9, 140.6, 153.4. IR (neat) 3056, 3026, 2981, 2937, 1722, 1512, 1434, 1374, 1292, 1215, 1180, 1105, 1075, 953 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 460.1843, found. 460.1843.



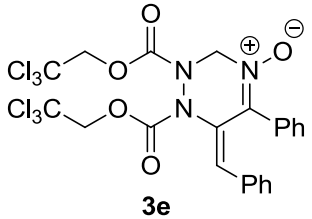
3c

(*E*)-6-benzylidene-1,2-bis(*tert*-butoxycarbonyl)-5-phenyl-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide (3c). mp 124-125 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.50 (s, 9H), 1.58 (s, 9H), 5.07 (br, 1H), 5.86 (br, 1H), 6.78-6.80 (m, 2H), 6.87-6.94 (m, 3H), 7.00-7.03 (m, 4H), 7.37-7.39 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 27.7, 27.9, 67.5, 68.7, 83.0, 124.6, 127.0, 127.0, 127.2, 127.7, 128.5, 129.0, 130.1, 131.3, 132.8, 140.2, 152.1, 152.8. IR (neat) 3057, 2977, 2933, 1720, 1511, 1432, 1368, 1299, 1248, 1146, 1095, 1075, 944, 898, 851 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 488.2156, found. 488.2156.



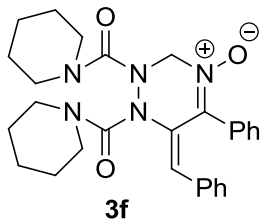
3d

(*E*)-6-benzylidene-1,2-bis((benzyloxy)carbonyl)-5-phenyl-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide (3d). mp 84-86 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.11-5.35 (m, 5H), 5.91 (br, 1H), 6.71 (d, *J* = 7.8 Hz, 2H), 6.87 (t, *J* = 7.6 Hz, 2 H), 6.93-7.06 (m, 5H), 7.30-7.38 (m, 12H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 68.1, 69.0, 124.2, 127.1, 127.3, 127.6, 127.8, 127.9, 128.4, 128.5, 128.5, 128.6, 128.9, 129.3, 129.8, 130.3, 132.4, 132.5, 134.8, 134.9, 140.4, 153.7. IR (neat) 3061, 3033, 2957, 1729, 1494, 1445, 1391, 1288, 1213, 1176, 1075, 978 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 556.1843, found. 556.1843.



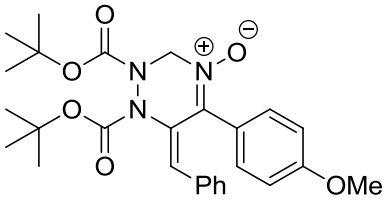
3e

(*E*)-6-benzylidene-1,2-bis(2,2,2-trichloroethoxycarbonyl)-5-phenyl-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide (3e). mp 94-96 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.72 (br, 1H), 4.84 (d, *J* = 11.9 Hz, 1H), 5.07 (br, 1H), 5.23 (d, *J* = 13.7 Hz, 1H), 6.00 (d, *J* = 14.7 Hz, 1H), 6.77 (d, *J* = 7.8 Hz, 2H), 6.89 (t, *J* = 7.3 Hz, 2H), 6.95-7.09 (m, 5H), 7.20 (br, 1H), 7.38-7.39 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 67.6, 75.9, 76.1, 94.2, 94.4, 123.3, 127.2, 127.4, 127.5, 128.0, 128.9, 129.7, 130.4, 132.3, 133.8, 140.4, 152.0, 152.6. IR (neat) 3058, 3027, 2958, 1744, 1512, 1491, 1434, 1387, 1279, 1205, 1109, 1058, 923, 891, 815 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 635.9192, found. 635.9191.



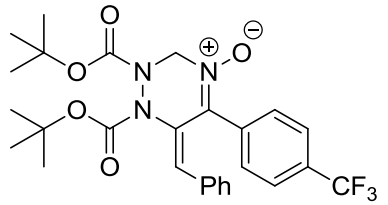
3f

(*E*)-6-benzylidene-5-phenyl-1,2-di(piperidine-1-carbonyl)-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide (3f). mp 211-212 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.64-1.66 (m, 12H), 3.42 (br, 4H), 3.52 (br, 4H), 5.26 (br, 2H), 6.65 (s, 1H), 6.86 (br, 5H), 7.00-7.02 (m, 3H), 7.48-7.50 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.3, 24.3, 25.6, 25.9, 46.7, 47.1, 73.5, 125.6, 126.8, 127.2, 127.2, 129.0, 129.1, 129.2, 129.8, 130.7, 134.2, 139.8, 158.8, 158.8. IR (neat) 3058, 3023, 2988, 2937, 2856, 1674, 1443, 1419, 1246, 1208, 1140, 1073, 1012, 956, 912, 854 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 510.2476, found. 510.2475.



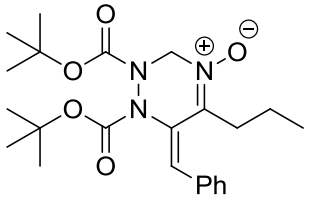
3g

(*E*)-6-benzylidene-1,2-bis(*tert*-butoxycarbonyl)-5-(4-methoxyphenyl)-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide (3g). mp 117-118 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.49 (s, 9H), 1.57 (s, 9H), 3.67 (s, 3H), 5.05 (br, 1H), 5.85 (br, 1H), 6.53 (d, *J* = 8.9 Hz, 2H), 6.80 (d, *J* = 7.3 Hz, 2H), 6.90-6.99 (m, 4H), 7.36 (d, *J* = 8.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 28.0, 28.2, 55.2, 67.5, 68.9, 83.3, 112.9, 120.2, 125.0, 127.3, 127.4, 128.9, 131.8, 132.1, 133.2, 140.3, 152.4, 152.9, 160.1. IR (neat) 2977, 2936, 1719, 1606, 1503, 1367, 1294, 1248, 1147, 1091, 1031, 923, 850, 830 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 518.2262, found. 518.2261.



3h

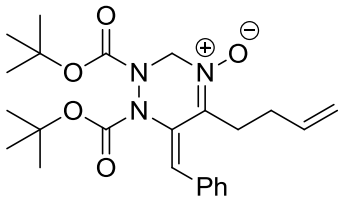
(*E*)-6-benzylidene-1,2-bis(*tert*-butoxycarbonyl)-5-(4-(trifluoromethyl)phenyl)-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide (3h). mp 160-161 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.51 (s, 9H), 1.58 (s, 9H), 5.07 (d, *J* = 14.2 Hz, 1H), 5.86 (br, 1H), 6.74-6.76 (m, 2H), 6.87-6.90 (m, 2H), 6.94-6.96 (m, 1H), 7.09 (br, 1H), 7.24 (d, *J* = 8.2 Hz, 2H), 7.44 (d, *J* = 7.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 28.0, 28.2, 68.3, 69.4, 83.6, 123.5 (q, *J* = 272 Hz), 124.3 (q, *J* = 3.8 Hz), 124.7, 127.8, 128.1, 128.7, 130.8 (q, *J* = 32 Hz), 130.9, 131.3, 131.8, 133.0, 139.2, 152.2, 153.0. IR (neat) 2980, 2933, 1724, 1478, 1411, 1392, 1369, 1321, 1245, 1149, 1127, 1066 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 556.2030, found. 556.2030.



3i

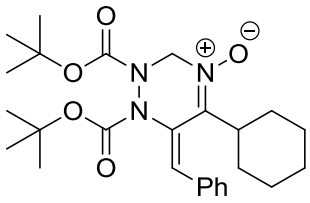
(*E*)-6-benzylidene-1,2-bis(*tert*-butoxycarbonyl)-5-propyl-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide (3i). mp 119-120 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.55 (t, *J* = 7.3 Hz, 3H), 1.35 (m, 2H), 1.50 (s, 9H), 1.52 (s, 9H), 2.16 (br, 1H), 2.41 (br, 1H), 4.93 (br, 1H), 5.60 (br, 1H), 7.03 (br, 1H), 7.21-7.23 (m, 2H), 7.35-7.37 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.4, 18.0, 27.9, 28.0, 28.4, 67.3, 68.6, 83.1, 125.9, 128.2, 128.2, 128.3, 129.3, 134.6, 143.8, 152.1, 152.8. IR (neat) 2975, 2933, 1723, 1521, 1444, 1368, 1296, 1247, 1145, 1115, 1057, 938, 854 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 454.2312, found.

454.2312.



3j

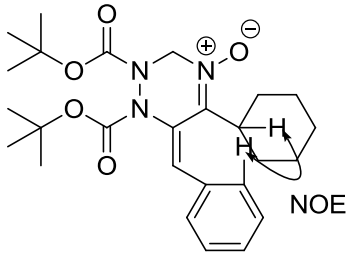
(*E*)-6-benzylidene-5-(but-3-en-1-yl)-1,2-bis(*tert*-butoxycarbonyl)-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide (3j). mp 110-111 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.50 (s, 9H), 1.53 (s, 9H), 2.03-2.11 (m, 2H), 2.30 (br, 1H), 2.54 (br, 1H), 4.74-4.79 (m, 2H), 4.92 (br, 1H), 5.36-5.46 (m, 1H), 5.61 (br, 1H), 7.03 (br, 1H), 7.22-7.24 (m, 2H), 7.35-7.37 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.4, 28.1, 28.2, 28.6, 67.6, 68.7, 83.3, 115.4, 125.9, 128.4, 128.5, 128.6, 134.7, 136.5, 143.2, 152.3, 153.1. IR (neat) 2977, 2933, 1723, 1520, 1444, 1368, 1290, 1247, 1228, 1145, 1060, 913, 854 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 466.2312, found. 466.2312.

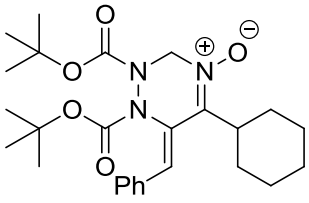


(*E*)-**3k**

(*E*)-6-benzylidene-1,2-bis(*tert*-butoxycarbonyl)-5-cyclohexyl-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide ((*E*)-3k). mp 121-123 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.44-0.58 (br, 2H), 1.11 (tq, J = 3.4, 13.1 Hz, 1H), 1.30-1.39 (m, 3H), 1.49 (s, 9H), 1.52 (s, 9H), 1.49-1.54 (m, 2H), 2.05 (br, 1H), 2.21 (dq, J = 2.7, 12.4 Hz, 1H), 2.36 (dq, J = 2.3, 12.4 Hz, 1H), 4.88 (br, 1H), 5.43-5.57 (br, 1H), 7.00 (br, 1H), 7.21-7.23 (m, 2H), 7.36-7.38 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.6, 24.9, 25.6, 25.9, 27.8, 27.9, 39.9, 67.8, 69.6, 82.9, 126.1, 127.9, 128.1, 128.2, 129.4, 135.0, 145.5, 152.1, 152.9. IR (neat) 2977, 2928, 2852, 1723, 1524, 1452, 1368, 1288, 1239, 1147, 1118, 1057, 938, 848 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 494.2625, found. 494.2625.

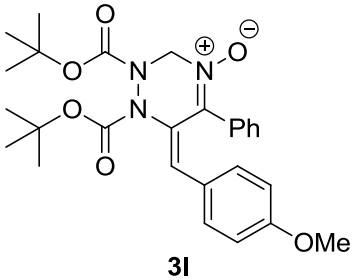
The stereochemistry was determined by NOE experiment as shown below.





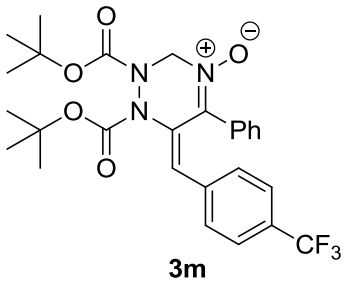
(*Z*)-**3k**

(*Z*)-6-benzylidene-1,2-bis(*tert*-butoxycarbonyl)-5-cyclohexyl-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide ((*Z*)-3k). mp 126-127 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.15 (br, 9H), 1.35-1.37 (m, 2H), 1.46-1.49 (m, 2H), 1.52 (s, 9H), 1.74- 1.77 (m, 2H), 1.86-1.89 (m, 2H), 2.17-2.31 (m, 2H), 3.36 (br, 1H), 4.90 (br, 1H), 5.57 (br, 1H), 6.79 (s, 1H), 7.29-7.35 (m, 3H), 7.88 (br, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.8, 26.5, 26.9, 27.6, 28.3, 37.4, 67.9, 69.4, 83.7, 124.6, 126.4, 128.4, 128.7, 129.9, 135.2, 145.9, 151.7, 152.3. IR (neat) 2978, 2928, 2855, 1731, 1523, 1448, 1368, 1302, 1248, 1224, 1142, 1058, 848 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 494.2625, found. 494.2625.



3l

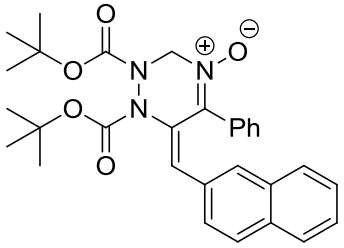
(*E*)-1,2-bis(*tert*-butoxycarbonyl)-6-(4-methoxybenzylidene)-5-phenyl-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide (3l). mp 87-89 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.49 (s, 9H), 1.57 (s, 9H), 3.66 (s, 3H), 5.06 (br, 1H), 5.86 (br, 1H), 6.42 (d, *J* = 8.7 Hz, 2H), 6.73 (d, *J* = 8.2 Hz, 2H), 6.95 (s, 1H), 7.06-7.08 (m, 4H), 7.43-7.44 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 27.8, 28.0, 54.9, 67.4, 68.5, 83.0, 112.7, 123.2, 125.2, 127.2, 127.7, 129.1, 130.2, 130.3, 131.4, 140.6, 152.2, 153.0, 158.84. IR (neat) 2977, 2934, 1720, 1605, 1508, 1368, 1294, 1250, 1149, 1031, 903, 851 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 518.2262, found. 518.2261.



3m

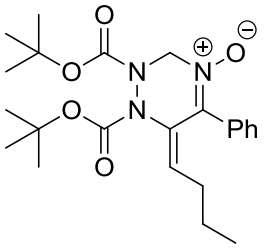
(*E*)-1,2-bis(*tert*-butoxycarbonyl)-5-phenyl-6-(4-(trifluoromethyl)benzylidene)-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide (3m). mp 96-98 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.52 (s, 9H), 1.58 (s, 9H), 5.08 (d, *J* = 14.0 Hz, 1H), 5.82 (br, 1H), 6.87 (d, *J* = 8.2 Hz, 2H), 6.98-7.04 (m, 4H), 7.11 (d, *J* = 8.0 Hz, 2H), 7.28 (br, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 28.1, 28.2, 68.9, 70.7, 83.7, 123.8 (q, *J* = 272 Hz), 124.2 (q, *J* = 3.8

Hz), 127.0, 127.7, 128.2, 128.5, 129.0, 129.1 (q, *J* = 33 Hz), 129.5, 130.5, 137.3, 139.6, 152.3, 152.3. IR (neat) 2979, 2933, 1724, 1615, 1491, 1369, 1321, 1248, 1149, 1124, 1066, 1017, 905, 851 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 556.2030, found. 556.2030.



3n

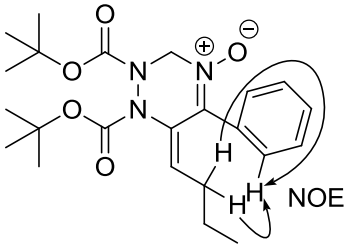
(*E*)-1,2-bis(*tert*-butoxycarbonyl)-6-(2-naphthalenylmethylidene)-5-phenyl-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide (3n). mp 160-161 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 1.51 (s, 9H), 1.59 (s, 9H), 5.09 (d, *J* = 14.7 Hz, 1H), 5.85 (d, *J* = 14.7 Hz, 1H), 6.73-6.76 (m, 1H), 6.83-6.92 (m, 3H), 7.16 (br, 1H), 7.26 (br, 1H), 7.32-7.42 (m, 5H), 7.49-7.51 (m, 1H), 7.58-7.60 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 28.2, 28.3, 68.6, 83.4, 83.6, 125.6, 126.0, 126.1, 126.4, 126.9, 127.4, 127.4, 127.9, 128.6, 129.1, 129.1, 130.3, 131.2, 132.3, 132.4, 140.5, 152.6, 152.7. IR (neat) 2978, 2933, 1722, 1509, 1491, 1433, 1369, 1300, 1251, 1150, 1094, 1075, 910, 850 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 538.2312, found. 538.2312.

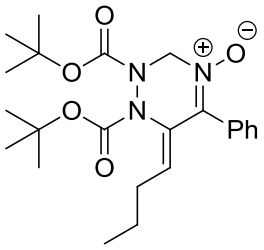


(*E*)-**3o**

(*E*)-1,2-bis(*tert*-butoxycarbonyl)-5-phenyl-6-propyl-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide ((*E*)-3o). mp 90-91 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 0.66 (t, *J* = 7.2 Hz, 3H), 1.20-1.23 (m, 2H), 1.29-1.44 (m, 2H), 1.51 (s, 9H), 1.53 (s, 9H), 4.98 (br, 1H), 5.74 (br, 1H), 5.89 (br, 1H), 7.45-7.50 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.3, 22.1, 27.9, 28.1, 29.9, 67.0, 68.3, 82.9, 125.1, 128.5, 129.3, 129.7, 130.7, 134.9, 140.6, 152.5, 152.9. IR (neat) 2975, 2932, 2872, 1719, 1519, 1494, 1455, 1435, 1367, 1303, 1251, 1149, 1074, 981, 904, 892, 874, 850 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 454.2312, found. 454.2312.

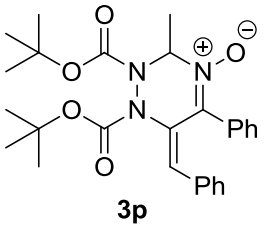
The stereochemistry was determined by NOE experiment as shown below.





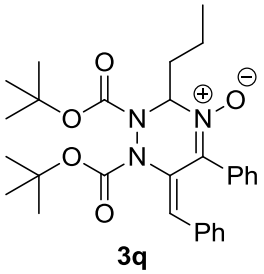
(*Z*)-**3o**

(*Z*)-1,2-bis(*tert*-butoxycarbonyl)-5-phenyl-6-propyl-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide ((*Z*)-3o). mp 102-104 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.87 (t, *J* = 7.4 Hz, 3H), 1.36-1.43 (m, 2H), 1.52 (s, 9H), 1.53 (s, 9H), 2.17 (br, 1H), 2.33 (br, 1H), 4.98 (d, *J* = 13.5 Hz, 1H), 5.48 (dd, *J* = 9.3, 5.6 Hz, 1H), 5.73 (br, 1H), 7.47 (br, 5H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.8, 21.7, 28.0, 28.1, 30.8, 66.8, 83.2, 83.4, 128.1, 128.4, 129.2, 129.7, 130.0, 133.4, 141.5, 151.9, 152.1. IR (neat) 2975, 2933, 2872, 1724, 1531, 1441, 1391, 1368, 1333, 1151, 1069, 876, 849 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 454.2312, found. 454.2312.



3p

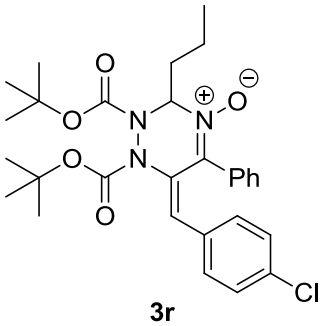
(*E*)-6-benzylidene-1,2-bis(*tert*-butoxycarbonyl)-3-methyl-5-phenyl-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide (3p). mp 130-131 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.50 (s, 9H), 1.58 (s, 9H), 1.80 (d, *J* = 6.4 Hz, 3H), 5.98 (br, 1H), 6.76-6.78 (m, 2H), 6.84-6.90 (m, 3H), 6.96-6.99 (m, 3H), 7.10 (br, 1H), 7.31-7.33 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 17.7, 27.9, 28.0, 77.2, 82.5, 83.1, 125.1, 126.8, 127.1, 127.1, 127.8, 128.7, 128.8, 128.9, 130.5, 133.7, 138.4, 152.3, 152.7. IR (neat) 2978, 2936, 1717, 1479, 1444, 1367, 1316, 1276, 1254, 1150, 1113, 1055, 952, 849 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 502.2312, found. 502.2312.



3q

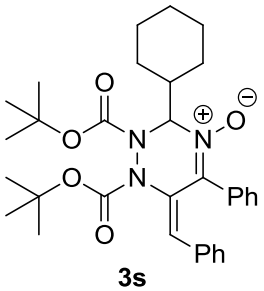
(*E*)-6-benzylidene-1,2-bis(*tert*-butoxycarbonyl)-5-phenyl-3-propyl-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide (3q). mp 120-121 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.04 (t, *J* = 7.3 Hz, 3H), 1.50 (s, 9H), 1.58 (s, 9H), 1.61-1.68 (m, 1H), 1.69-1.78 (m, 1H), 1.85-1.94 (m, 1H), 2.33-2.41 (m, 1H), 5.80 (d, *J* = 10.1 Hz, 1H), 6.76-6.77 (m, 2H), 6.82-6.90 (m, 3H), 6.94-6.97 (m, 3H), 7.14 (br, 1H), 7.30-7.32 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.2, 18.5, 27.9, 30.0, 32.3, 80.1, 82.4, 83.0, 124.8, 126.7, 127.0, 127.0, 127.4, 128.6, 128.7, 128.9, 130.5, 133.7, 138.5, 152.0, 153.1. IR (neat) 3056, 2974, 2933, 2875, 1718, 1476, 1443,

1367, 1324, 1150, 1126, 1071, 950, 848 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 530.2625, found. 530.2625.



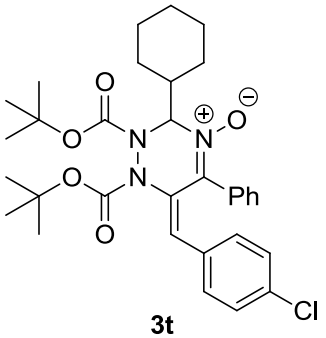
3r

(*E*)-1,2-bis(*tert*-butoxycarbonyl)-6-(4-chlorobenzylidene)-5-phenyl-3-propyl-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide (3r). mp 149-150 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 1.04 (t, *J* = 7.3 Hz, 3H), 1.50 (s, 9H), 1.57 (s, 9H), 1.57 (m, 1H), 1.73 (br, 1H), 1.90 (br, 1H), 2.36 (br, 1H), 5.80 (br, 1H), 6.68 (d, *J* = 8.7 Hz, 2H), 6.81 (d, *J* = 8.2 Hz, 2H), 7.00-7.10 (m, 4H), 7.27-7.29 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.5, 18.8, 28.1, 28.1, 33.3, 79.8, 81.4, 82.9, 125.4, 125.9, 127.3, 127.4, 128.8, 129.1, 130.1, 130.7, 132.3, 132.8, 138.4, 152.2, 153.6. IR (neat) 3055, 2976, 2932, 2875, 1718, 1591, 1479, 1368, 1253, 1152, 1126, 1087, 1014, 952, 908, 849 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 564.2236, found. 564.2235.



3s

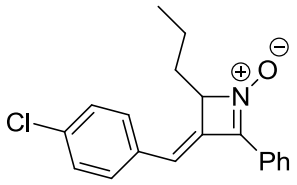
(*E*)-6-benzylidene-1,2-bis(*tert*-butoxycarbonyl)-3-cyclohexyl-5-phenyl-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide (3s). mp 151-152 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 1.24-1.36 (m, 4H), 1.49 (s, 9H), 1.59 (s, 9H), 1.45-1.63 (m, 2H), 1.71-1.93 (m, 4H), 2.23 (br, 1H), 5.68 (br, 1H), 6.77-6.79 (m, 2H), 6.84-6.86 (m, 3H), 6.95-6.97 (m, 3H), 7.28-7.33 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.0, 26.0, 26.2, 28.1, 28.3, 28.4, 39.6, 82.8, 83.4, 84.2, 125.1, 125.6, 126.7, 127.2, 127.2, 128.8, 129.1, 129.6, 130.8, 134.5, 138.6, 152.1, 153.7. IR (neat) 3055, 2979, 2932, 2856, 1720, 1481, 1455, 1392, 1367, 1248, 1149, 1114, 1020, 853 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 570.2938, found. 570.2940.



3t

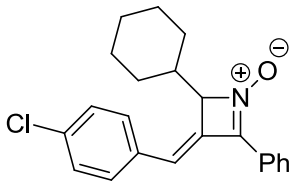
(*E*)-1,2-bis(*tert*-butoxycarbonyl)-6-(4-chlorobenzylidene)-3-cyclohexyl-5-phenyl-1,2,3,6-tetrahydro-1,2,4-triazine 4-oxide (3t). mp 154-155 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.25-1.33 (m, 4H), 1.50 (s, 9H), 1.59 (s, 9H), 1.46-1.61 (m, 2H), 1.72-1.90 (m, 2H), 2.23 (br, 1H), 5.68 (br, 1H), 6.70 (d, *J* = 8.2 Hz, 2H), 6.81 (d, *J* = 8.2 Hz, 2H), 6.99-7.06 (m, 3H), 7.29 (d, *J* = 6.9 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.9, 26.1, 27.5, 28.1, 28.2, 28.4, 29.7, 39.5, 83.0, 83.5, 84.7, 123.0, 126.3, 127.4, 127.4, 128.9, 129.6, 130.2, 130.8, 132.5, 133.1, 138.1, 152.1, 153.5. IR (neat) 3055, 2977, 2928, 2855, 1719, 1481, 1368, 1305, 1255, 1150, 1088, 1014, 956, 850 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 604.2549, found. 604.2549.

5. Analytical data of 4



4m

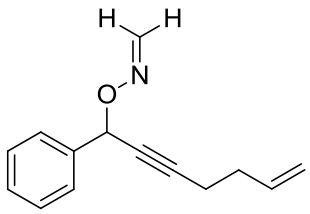
(4*E*)-4-(4-chlorobenzylidene)-1-phenyl-3-propylazetidin-2-one (4m). mp 108-109 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 7.6 Hz, 3H), 1.43 (sext, *J* = 7.6 Hz, 2H), 1.77-1.86 (m, 1H), 2.04-2.11 (m, 1H), 5.36 (dd, *J* = 5.0, 3.7 Hz, 1H), 6.75 (s, 1H), 7.19-7.21 (m, 2H), 7.33-7.35 (m, 2H), 7.43-7.50 (m, 3H), 8.09-8.11 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.8, 17.0, 29.6, 82.6, 115.3, 126.2, 126.9, 128.8, 128.8, 129.0, 130.6, 131.2, 133.4, 134.1, 148.3. IR (neat) 3058, 2959, 2932, 2873, 1685, 1544, 1490, 1449, 1415, 1406, 1090, 1011, 859 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 334.0969, found. 334.0969.



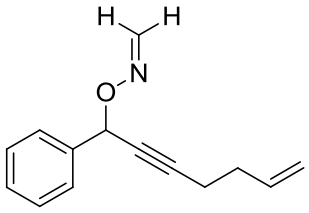
4p

(4*E*)-4-(4-chlorobenzylidene)-3-cyclohexyl-1-phenylazetidin-2-one (4p). mp 155-156 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.07-1.25 (m, 4H), 1.59-1.92 (m, 7H), 5.27 (s, 1H), 6.77 (s, 1H), 7.18-7.20 (m, 2H), 7.34-7.36 (m, 2H), 7.42-7.51 (m, 3H), 8.10-8.12 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.8, 25.8, 26.2, 26.5, 29.7, 37.0, 87.2, 115.2, 126.1, 126.8, 128.7, 128.9, 129.2, 130.0, 130.5, 133.2, 134.2, 148.6. IR (neat) 3057, 2927, 2853, 1685, 1544, 1489, 1415, 1405, 1241, 1091, 1011, 856 cm^{-1}. HRMS (ESI) calcd. for $(M+Na)^+$ 374.1282, found. 374.1282.

5. ^{1}H and ^{13}C NMR charts of 1, 3 and 4



1e


H
H
N
O

1e

PPM
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115.663
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77.000
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75.465
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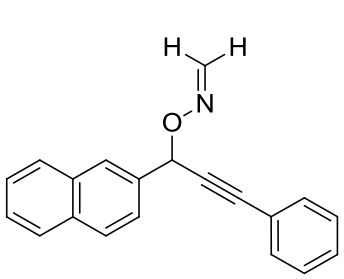
18.687



1i

1.00
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1.02
4.08
3.06
1.02
1.00
1.00
PPM
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6
4
2
0
8.079
7.902
7.882
7.865
7.841
7.732
7.710
7.535
7.515
7.505
7.497
7.489
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7.322
7.254
7.165
7.145
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6.248
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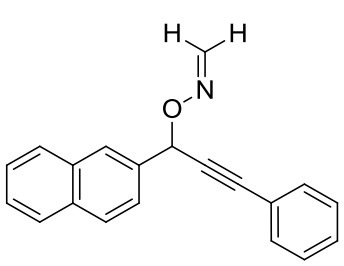
H
H
N
O

1i

PPM
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128.449
128.297
128.221
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126.247
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122.347
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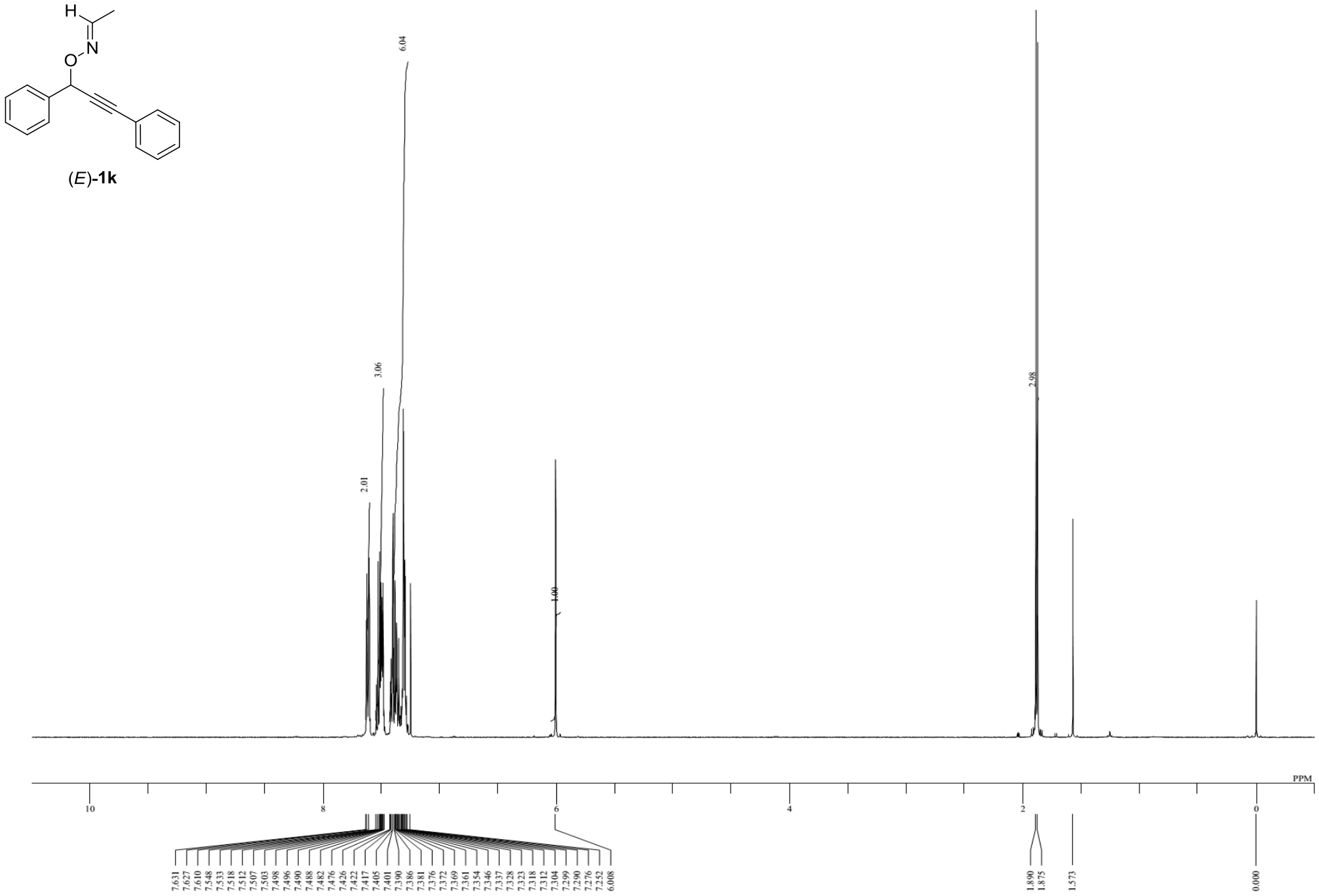
77.315
77.000
76.676
75.904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1.00
2.98
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7.610
7.548
7.533
7.518
7.512
7.507
7.503
7.498
7.496
7.490
7.488
7.482
7.476
7.426
7.422
7.417
7.405
7.401
7.390
7.386
7.381
7.376
7.372
7.369
7.361
7.354
7.346
7.337
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7.323
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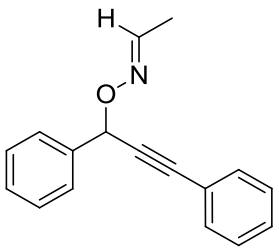
H
N
O

(*E*)-**1k**

PPM
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127.925
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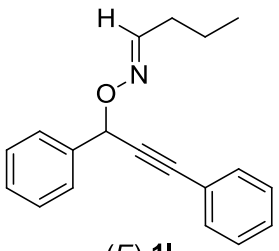
75.227
15.310


H
N
O

(*E*)-**1l**

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6.08
1.00
3.05
2.07
3.05
PPM
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8
6
4
2
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7.633
7.615
7.508
7.505
7.503
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7.489
7.484
7.474
7.427
7.423
7.418
7.406
7.401
7.387
7.376
7.372
7.369
7.361
7.355
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7.337
7.323
7.316
7.312
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7.299
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7.249
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2.196
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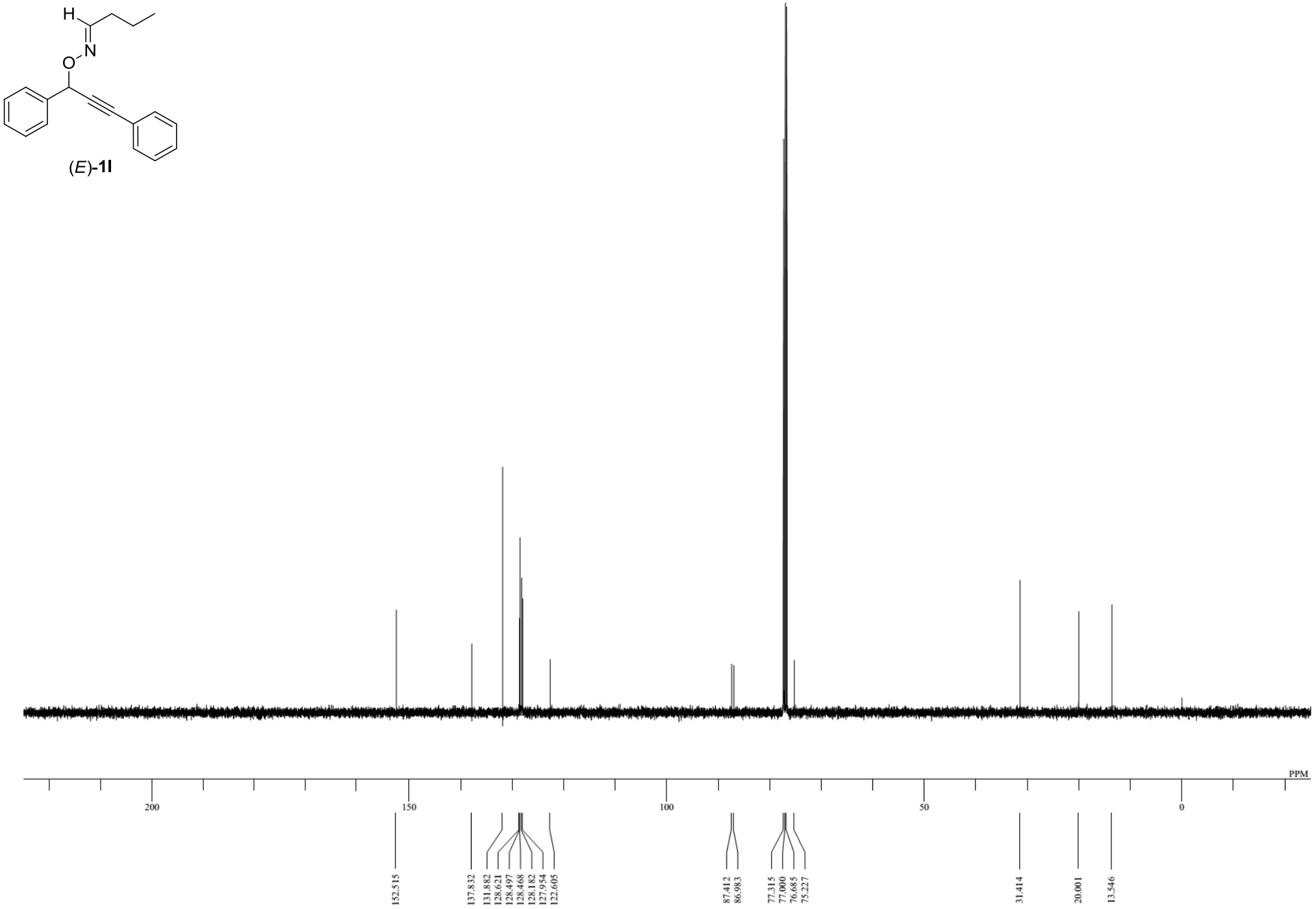
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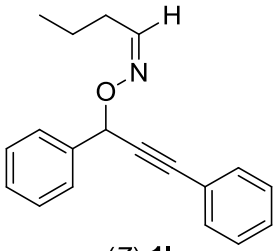
31.414
20.001
13.546


H
N
O

(*Z*)-**1l**

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6.01
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1.00
2.07
2.02
3.09
PPM
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8
6
4
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7.619
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7.504
7.496
7.485
7.480
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7.422
7.418
7.405
7.401
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7.370
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7.359
7.352
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7.334
7.322
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7.311
7.303
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6.767
6.753
6.046
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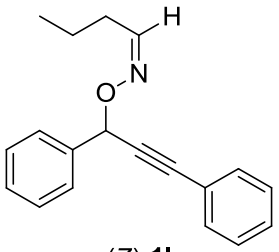
H
N
O

(*Z*)-**1l**

PPM
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87.116
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77.000
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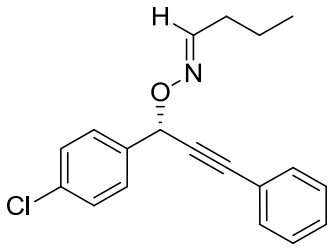
75.331
27.868
19.534
13.813


H
N
O
Cl

(*S*,*E*)-**1m**

2.03
2.06
5.00
1.00
2.03
2.00
3.00
PPM
10
8
6
4
2
0
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7.557
7.540
7.498
7.495
7.486
7.479
7.474
7.463
7.386
7.380
7.376
7.359
7.353
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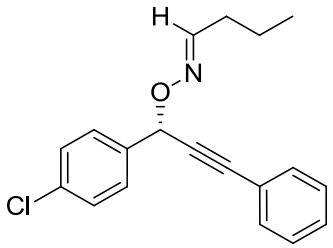
H
N
O
Cl

(*S*,*E*)-**1m**

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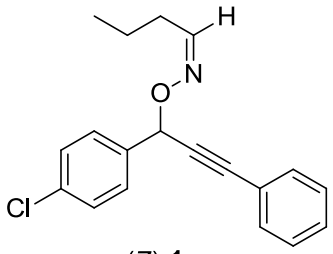
13.537


H
N
O
Cl

(*Z*)-**1m**

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5.00
0.99
1.00
2.05
2.00
3.08
PPM
10
8
6
4
2
0
7.552
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7.526
7.503
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7.490
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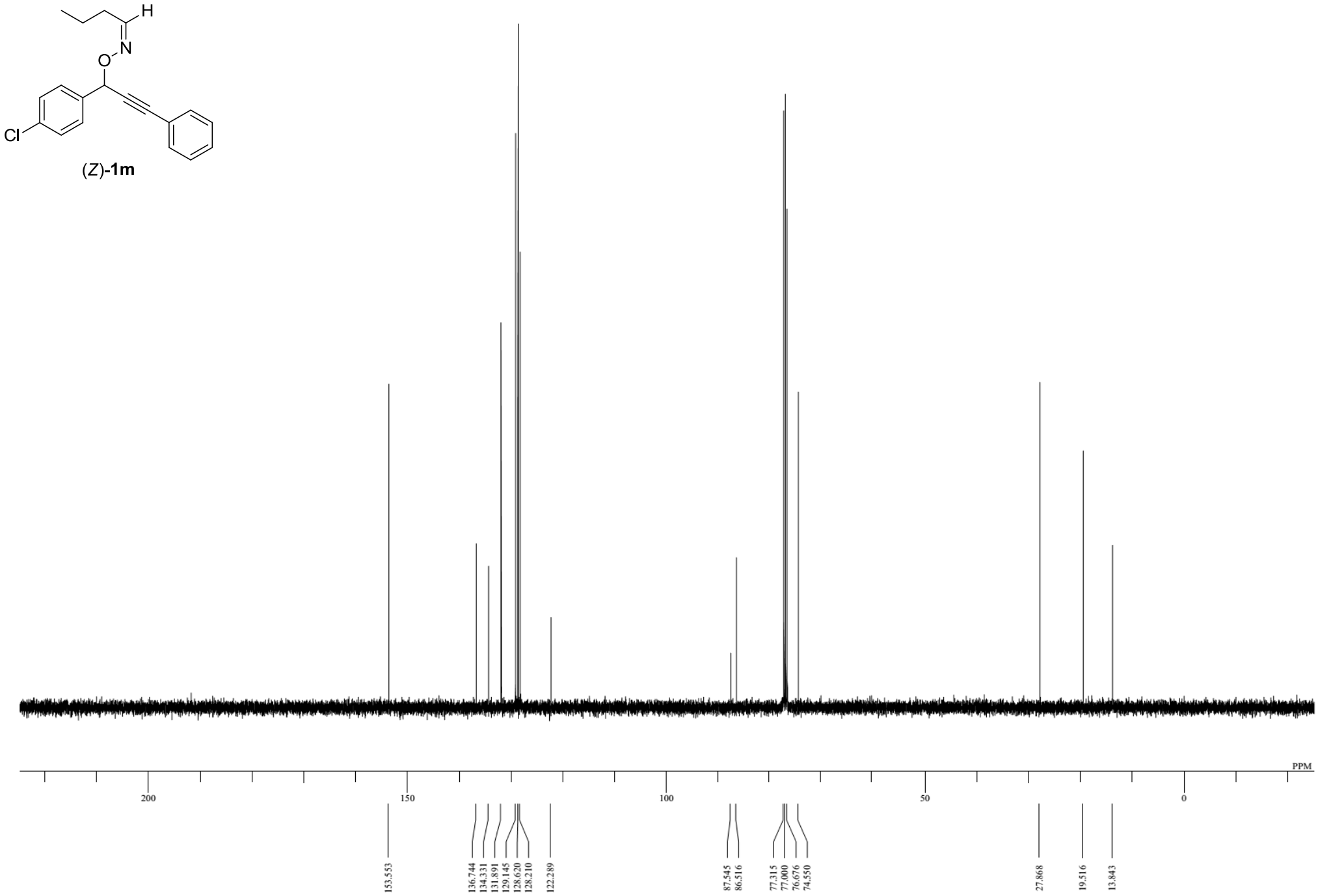
H
N
O
Cl
(Z)-1m
PPM
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150
100
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129.145
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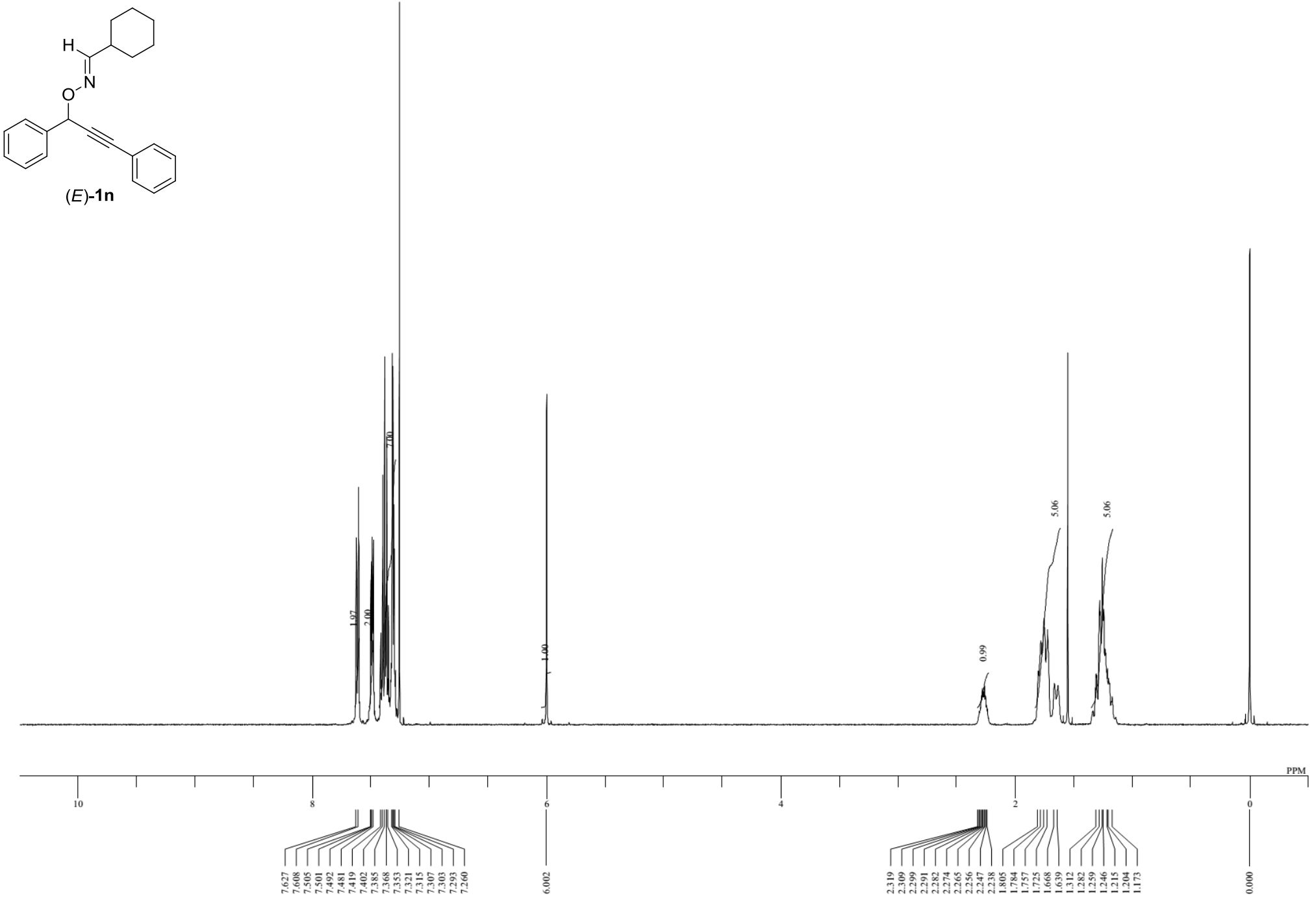
74.550
27.868
19.516
13.843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5.06
PPM
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8
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4
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7.321
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7.260
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2.256
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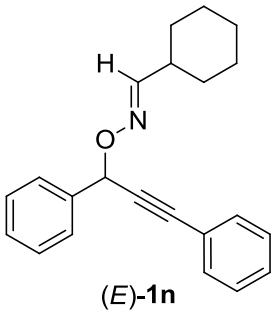
H
N
O

(*E*)-**1n**

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100
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0
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137.841
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128.573
128.459
128.421
128.154
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87.002
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77.000
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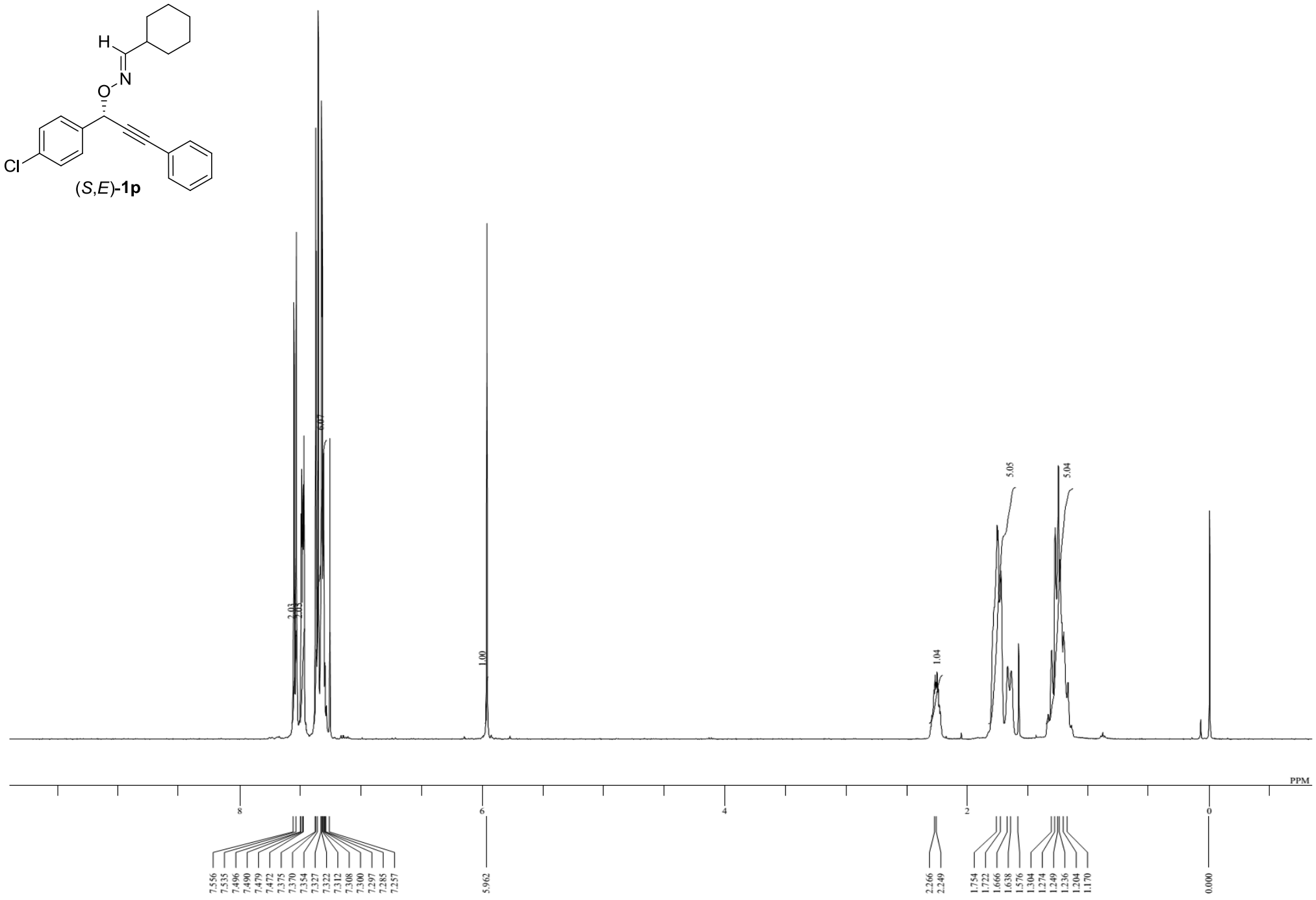
25.293


H
N
O
Cl
(S,E)-1p
2.03
2.03
6.07
1.00
1.04
5.05
5.04
PPM
8
6
4
2
0
7.556
7.535
7.496
7.490
7.479
7.472
7.375
7.370
7.354
7.327
7.322
7.312
7.308
7.300
7.297
7.285
7.257
5.962
2.266
2.249
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1.666
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1.576
1.304
1.274
1.249
1.236
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1.170
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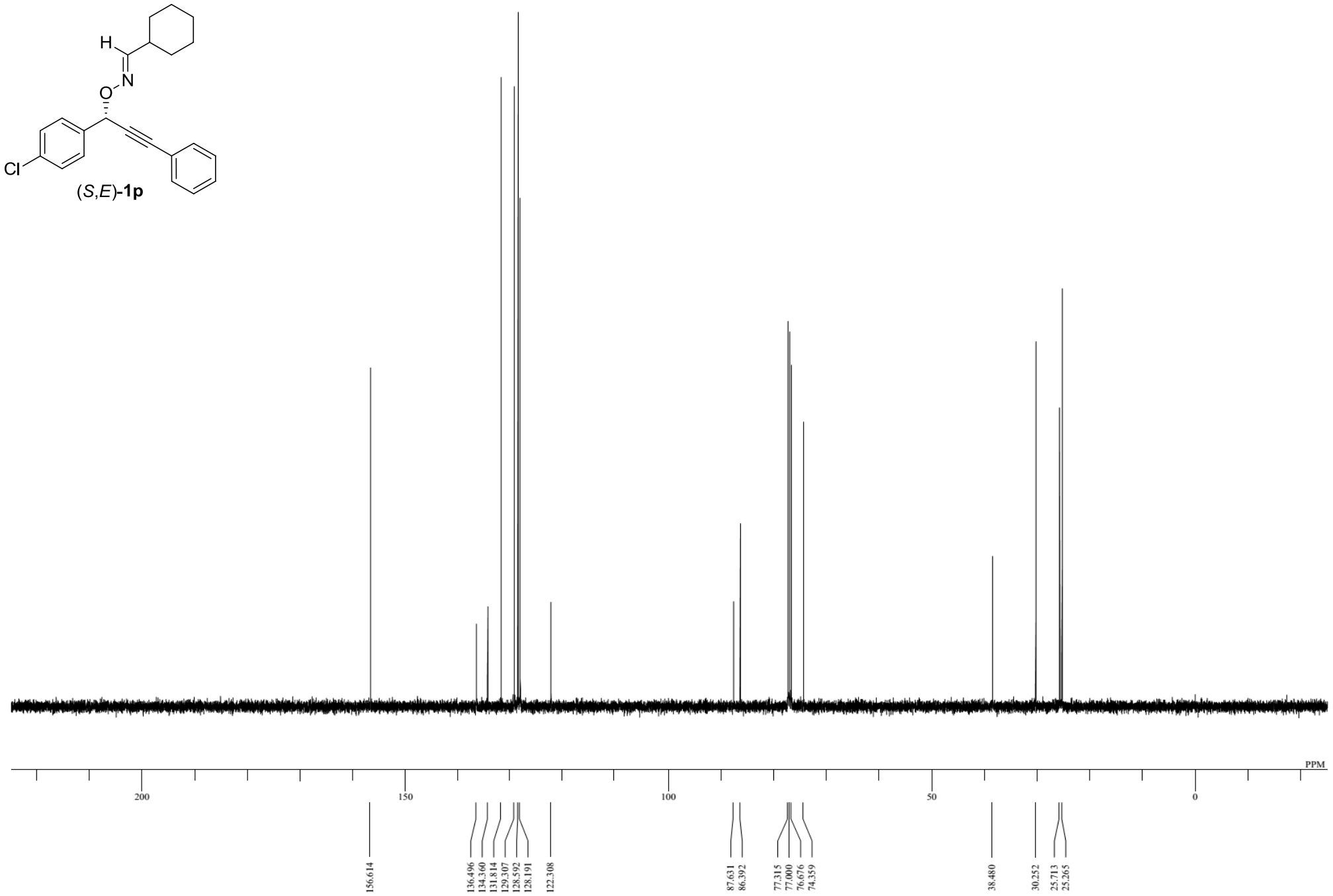
H
N
O
Cl
(S,E)-1p
PPM
200
150
100
50
0
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136.496
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129.307
128.592
128.191
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86.392
77.315
77.000
76.676
74.359
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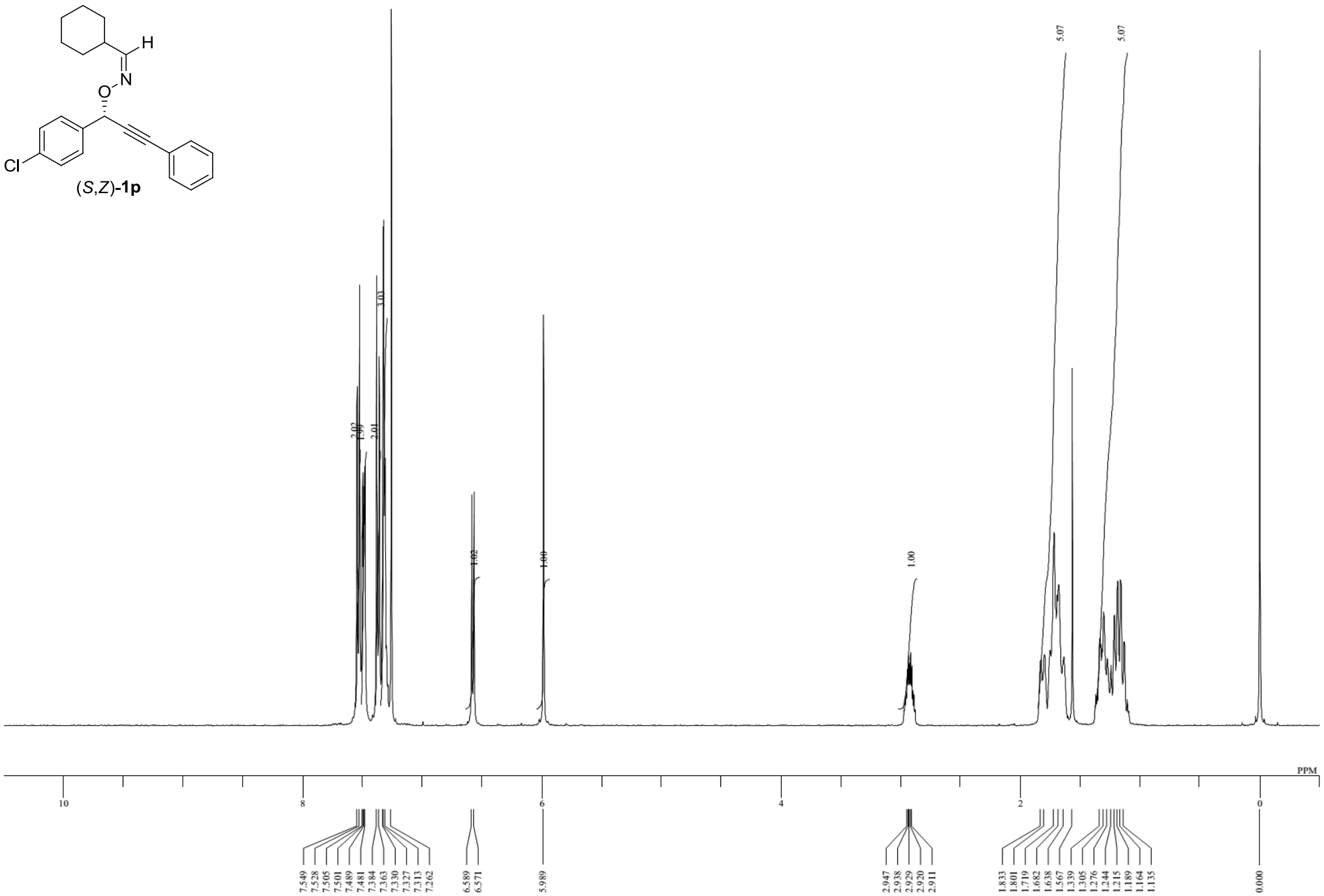
30.252
25.713
25.265


(S,Z)-1p
2.02
1.99
2.01
3.03
1.02
1.00
1.00
5.07
5.07
PPM
10
8
6
4
2
0
7.549
7.528
7.505
7.501
7.489
7.481
7.384
7.363
7.330
7.327
7.313
7.262
6.589
6.571
5.989
2.947
2.938
2.929
2.920
2.911
1.833
1.801
1.719
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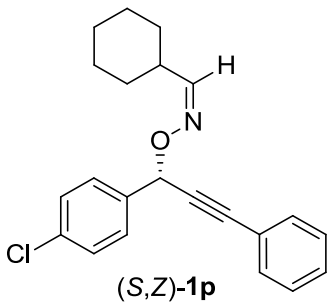
1.164
1.135
0.000


H
N
O
Cl

(*S*,*Z*)-**1p**

PPM
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150
100
50
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136.801
134.284
131.891
129.106
128.620
128.220
122.318
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77.000
76.676
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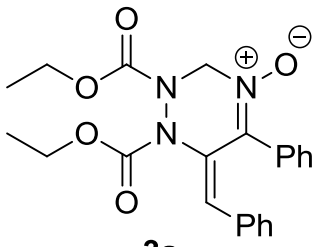
29.508
29.432
25.799
25.122
25.103


O
O
N
N
⊕
N
O
⊖
O
O
Ph
Ph

3a

2.05
1.00
3.03
1.04
2.05
2.01
1.00
1.00
4.05
3.02
2.98
PPM
10
8
6
4
2
0
7.394
7.378
7.264
7.080
7.058
7.043
7.025
7.012
6.973
6.955
6.937
6.911
6.891
6.874
6.812
6.794
5.921
5.886
5.151
5.116
4.390
4.372
4.354
4.334
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4.280
4.272
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1.308
1.291
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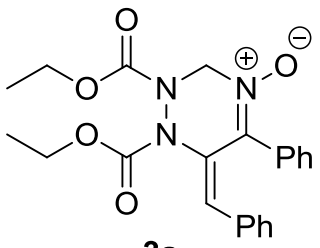
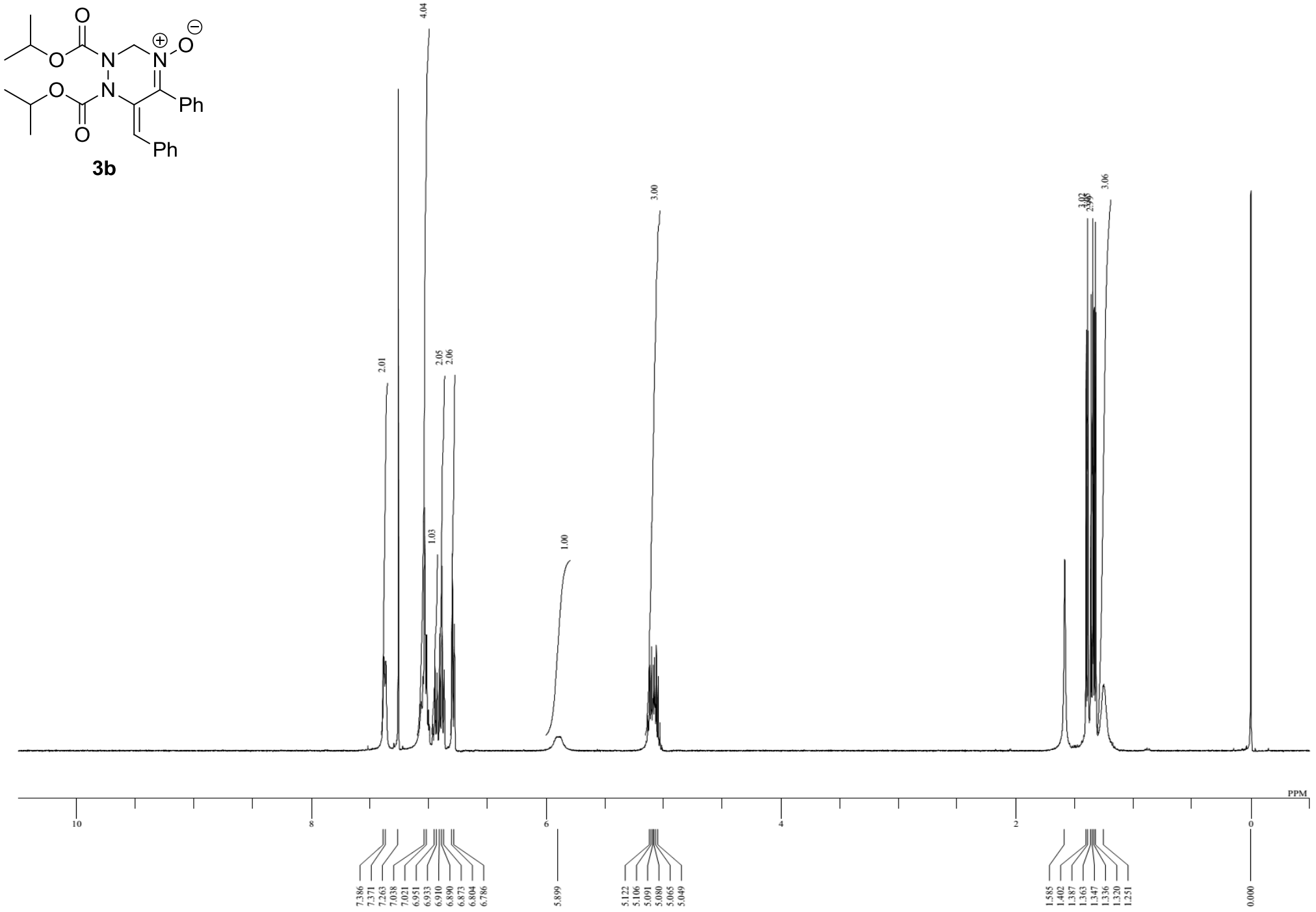
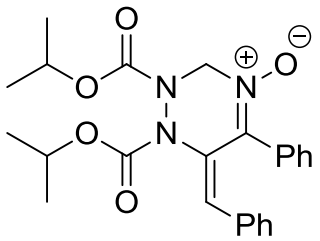

O
O
N
N
⊕
N
O
⊖
O
O
Ph
Ph

3a

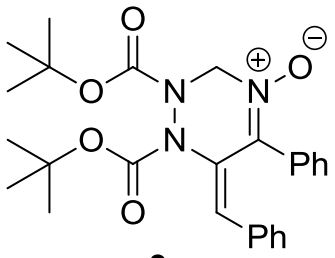
PPM
200
150
100
50
0
153.898
140.454
132.711
132.254
130.337
129.307
128.850
127.715
127.563
127.334
127.191
124.330
77.324
77.000
76.685
68.037
63.642
63.604
14.328
14.252


3b
4.04
2.01
2.05
2.06
1.03
1.00
3.00
3.06
PPM
10
8
6
4
2
0
7.386
7.371
7.263
7.038
7.021
6.951
6.933
6.910
6.890
6.873
6.804
6.786
5.899
5.122
5.106
5.091
5.080
5.065
5.049
1.585
1.402
1.387
1.363
1.347
1.336
1.320
1.251
0.000



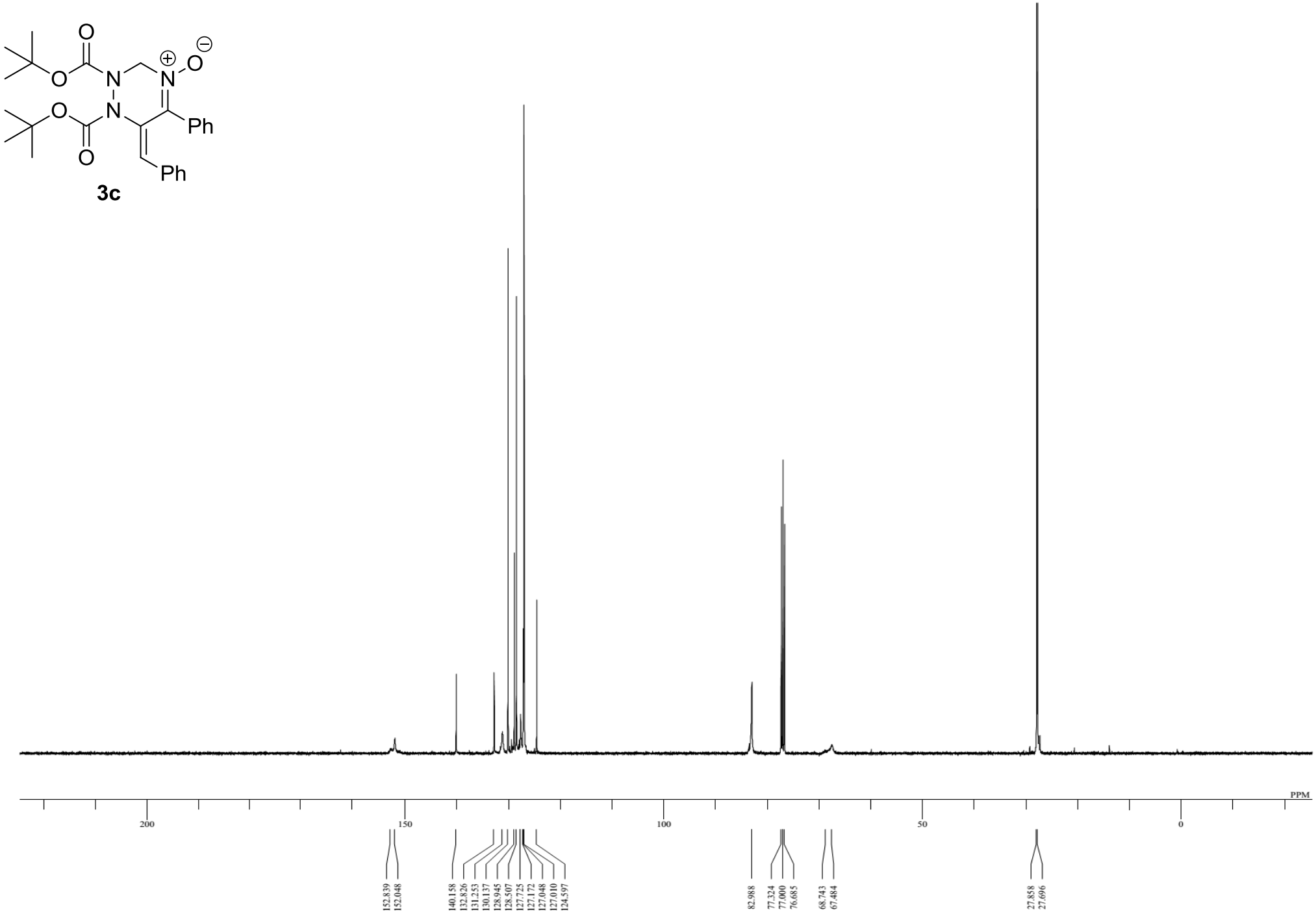
3b

PPM
200
150
100
50
0
153.421
140.587
132.883
132.158
130.394
129.336
128.850
127.830
127.563
127.401
127.277
124.502
77.324
77.000
76.685
71.851
71.699
68.075
21.966
21.870
21.803


O
O
N
N
⊕
N
⊖
O
O
O
Ph
Ph

3c

9.05
9.06
4.03
3.01
2.05
2.02
1.03
1.00
PPM
10
8
6
4
2
0
7.385
7.371
7.263
7.027
7.012
6.998
6.941
6.923
6.904
6.885
6.867
6.800
6.783
5.857
5.065
1.576
1.502
0.000


3c
PPM
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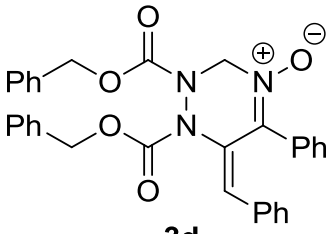
100
50
0
152.839
152.048
140.158
132.826
131.253
130.137
128.945
128.507
127.725
127.172
127.048
127.010
124.597
82.988
77.324
77.000
76.685
68.743
67.484
27.858
27.696


O
Ph
O
N
N
O
Ph
O
N
Ph
O
Ph

3d

12.06
5.00
3.03
1.99
1.00
5.10
PPM
10
8
6
4
2
0
7.383
7.304
7.260
7.057
7.039
7.022
7.010
6.991
6.971
6.951
6.933
6.892
6.873
6.855
6.723
6.703
5.914
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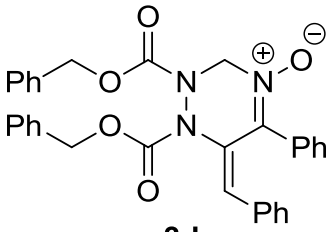
5.340
5.319
5.309
5.293
5.262
5.223
5.194
5.138
5.105
0.000


O
Ph
O
N
N
O
Ph
O
N
Ph
O
Ph

3d

PPM
200
150
100
50
0
153.650
140.377
134.866
134.752
132.530
132.416
130.318
129.756
129.298
128.878
128.554
128.497
128.449
128.411
127.906
127.801
127.591
127.305
127.143
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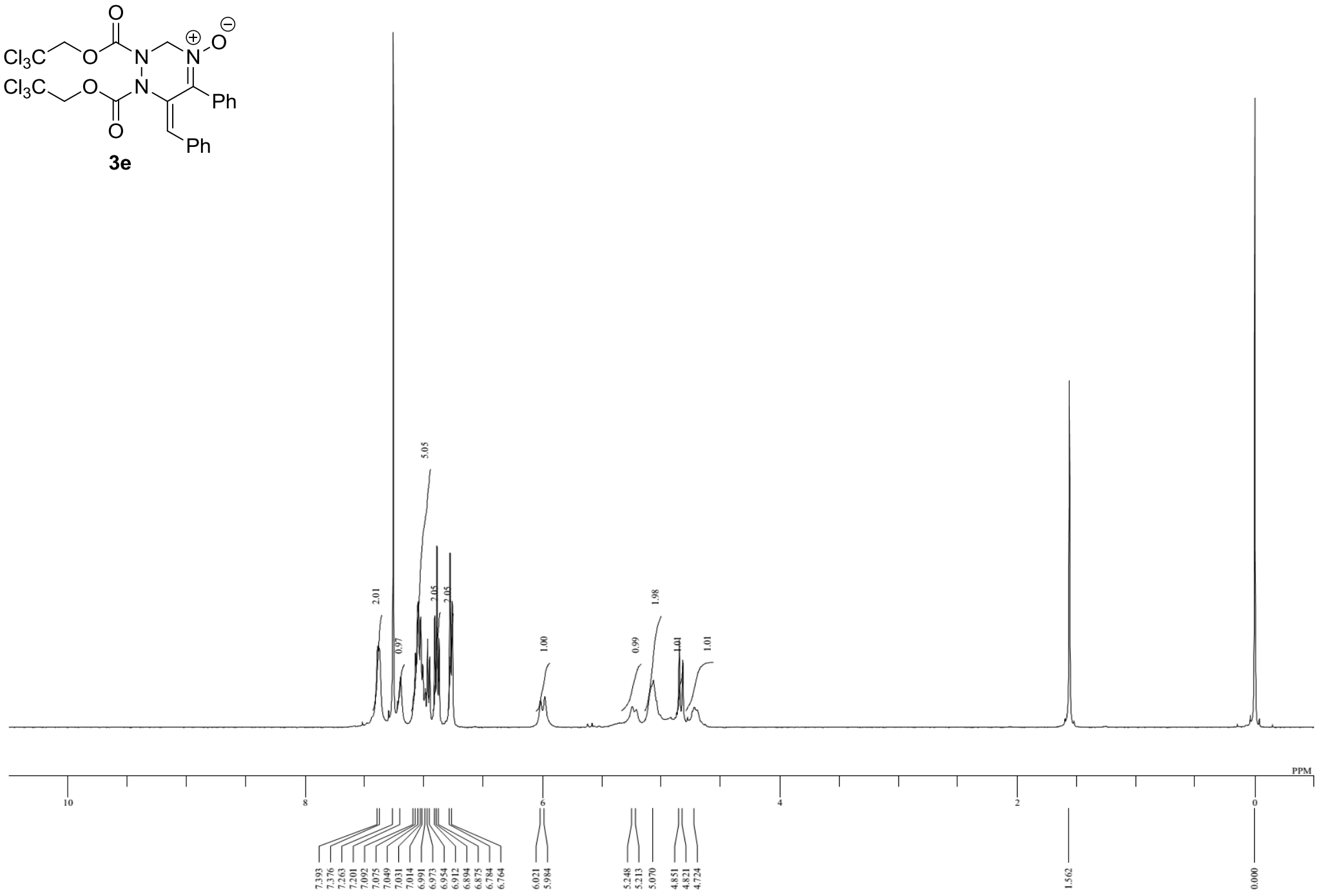
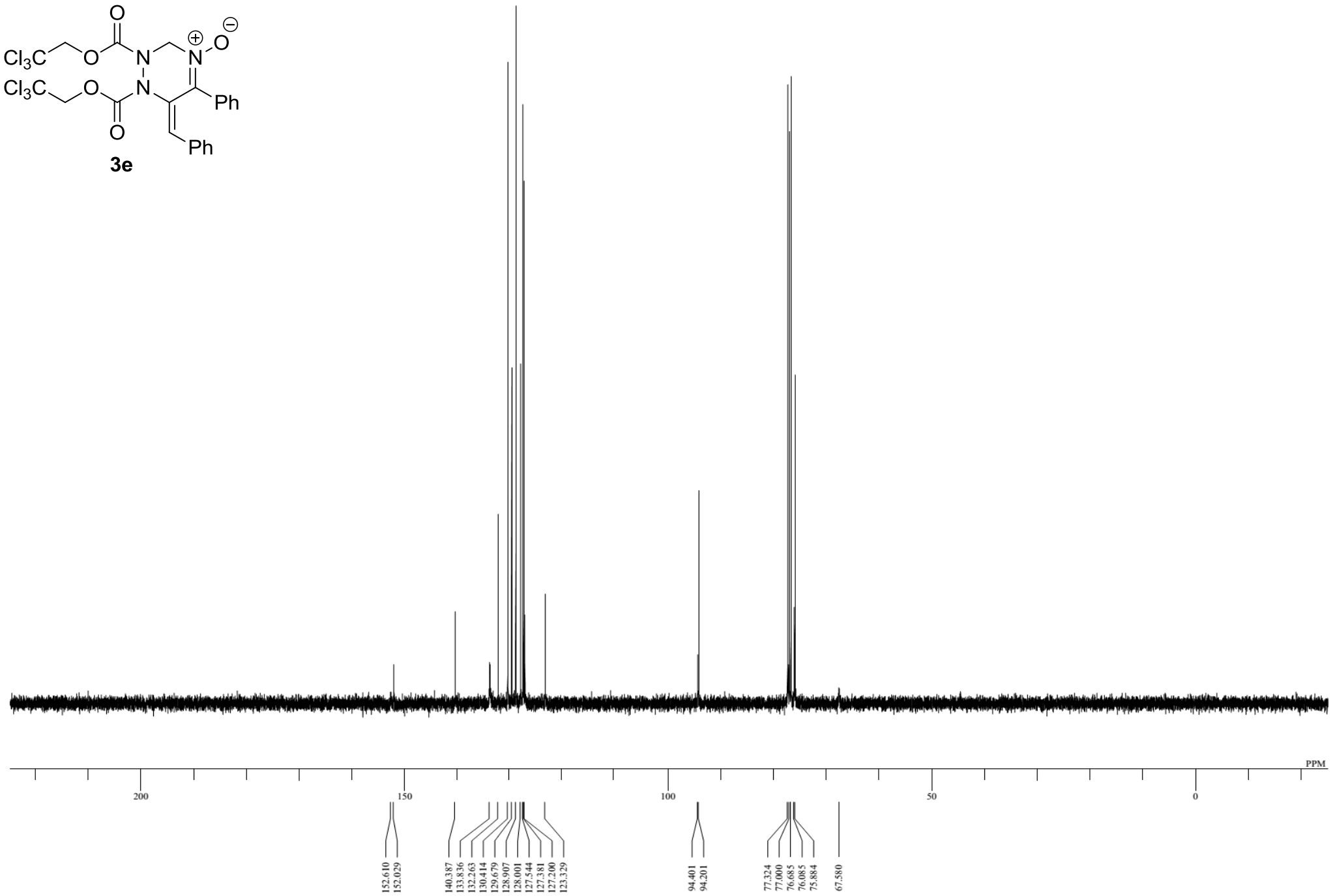
77.000
76.685
69.010
68.047


3e
2.01
0.97
5.05
2.05
2.05
1.00
0.99
1.98
1.01
1.01
PPM
10
8
6
4
2
0
7.393
7.376
7.263
7.201
7.092
7.075
7.049
7.031
7.014
6.991
6.973
6.954
6.912
6.894
6.875
6.784
6.764
6.021
5.984
5.248
5.213
5.070
4.851
4.821
4.724
1.562
0.000


Cl3C
O
N
N
O
Ph
Ph
3e
PPM
200
150
100
50
0
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152.029
140.387
133.836
132.263
130.414
129.679
128.907
128.001
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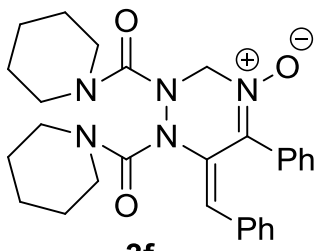
127.381
127.200
123.329
94.401
94.201
77.324
77.000
76.685
76.085
75.884
67.580


O
N
N
N
⊕
O
⊖
N
N
Ph
O
Ph

3f

12.01
5.03
4.00
4.03
3.05
2.03
2.01
1.00
PPM
10
8
6
4
2
0
7.503
7.495
7.489
7.486
7.479
7.272
7.268
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7.004
7.000
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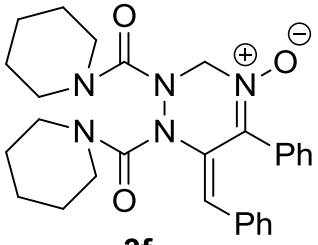
1.644
0.000


O
N
N
N
O
N
N
Ph
O
Ph

3f

PPM
200
150
100
50
0
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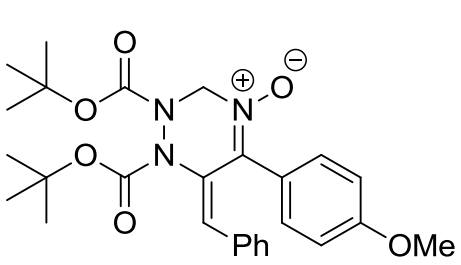
134.237
130.661
129.832
129.222
129.117
128.955
127.219
127.162
126.800
125.637
77.324
77.000
76.685
73.510
47.061
46.737
25.913
25.608
24.330
24.292


O
O
N
N
⊕
N
O
⊖
O
O
O
Ph
OMe

3g

9.05
9.00
4.01
2.01
2.03
2.00
1.00
1.03
3.00
PPM
10
8
6
4
2
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7.372
7.352
7.263
6.992
6.973
6.956
6.938
6.919
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6.810
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6.544
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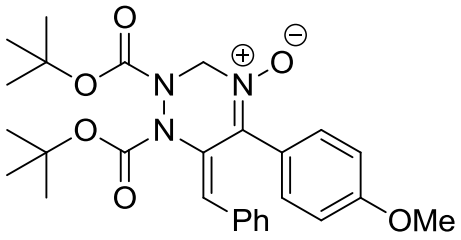
O
O
N
N
⊕
N
O
⊖
O
O
Ph
OMe

3g

PPM
200
150
100
50
0
160.105
152.906
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140.339
133.198
132.073
131.787
128.945
127.420
127.315
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120.154
112.898
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77.315
77.000
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68.924
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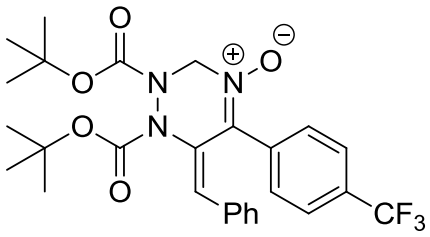
28.182
28.020


O
O
N
N
O
O
O
O
Ph
CF3

3h

9.07
9.06
2.02
2.08
2.00
2.01
1.00
1.00
0.99
1.00
PPM
10
8
6
4
2
0
7.448
7.428
7.263
7.252
7.232
7.086
6.956
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6.756
6.738
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5.088
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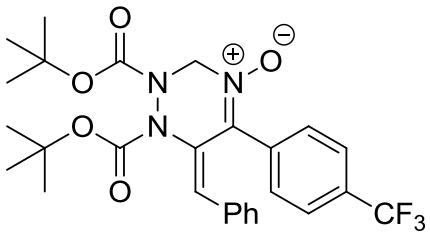
0.000


O
O
N
N
O
O
O
O
N
Ph
CF3

3h

PPM
200
150
100
50
0
152.992
152.239
139.205
132.988
131.806
131.310
131.253
130.928
130.890
130.604
130.280
128.669
128.058
127.782
127.515
124.845
124.731
124.330
124.292
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122.137
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83.636
77.324
77.000
76.685
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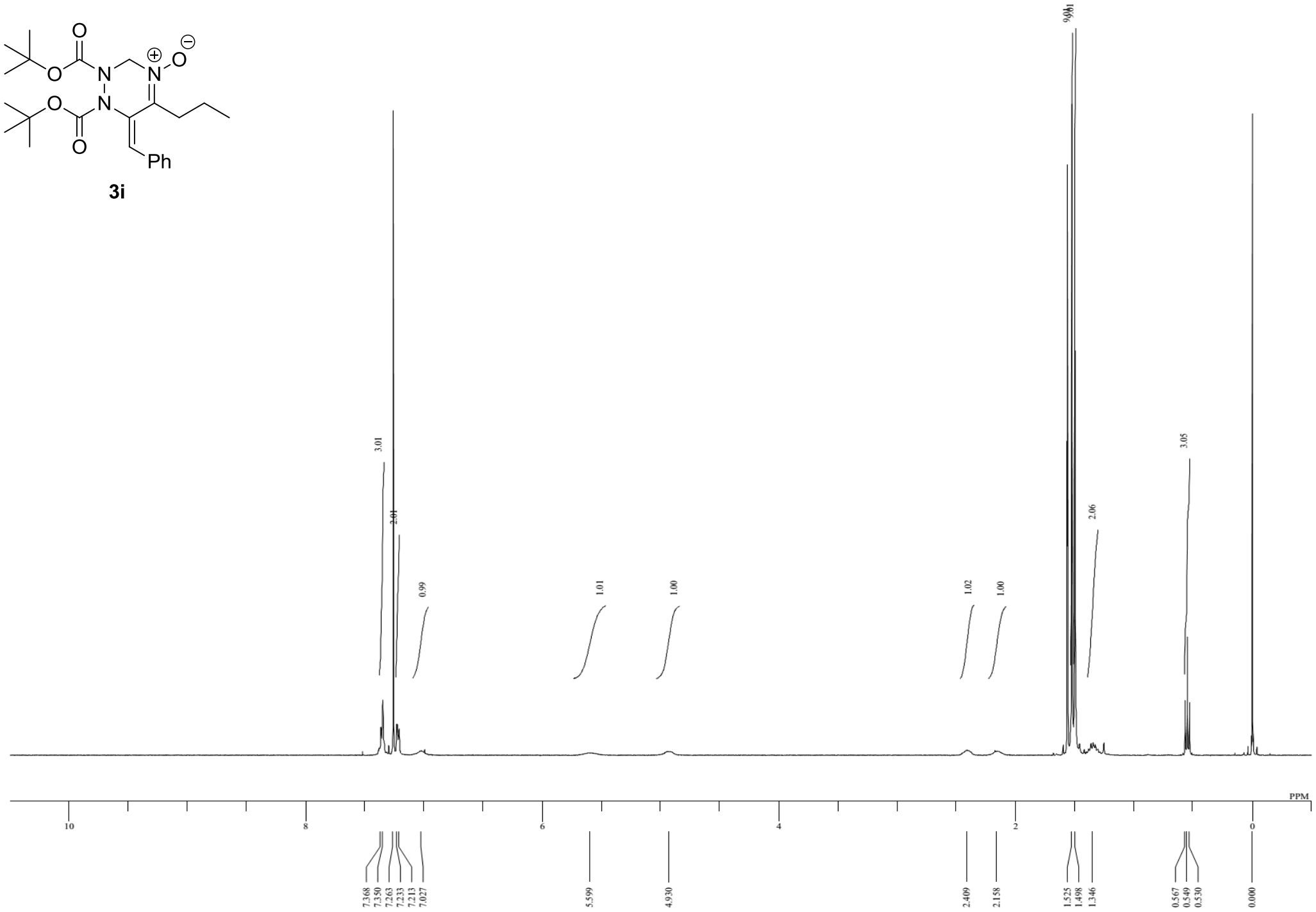
68.247
28.182
28.030


3i
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2.01
0.99
1.01
1.00
1.02
1.00
9.00
2.06
3.05
PPM
10
8
6
4
2
0
7.368
7.350
7.263
7.233
7.213
7.027
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4.930
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2.158
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1.346
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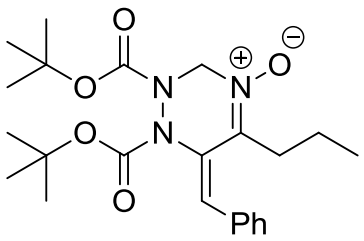
0.000



3i

PPM
200
150
100
50
0
152.811
152.115
143.753
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129.317
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128.230
128.154
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77.000
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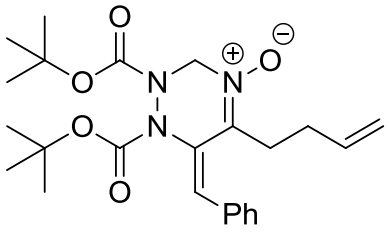
67.284
28.421
27.992
27.915
18.009
13.413


O
O
N
N
⊕
N
O
⊖
O
O
O
Ph

3j

3.04
2.04
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1.02
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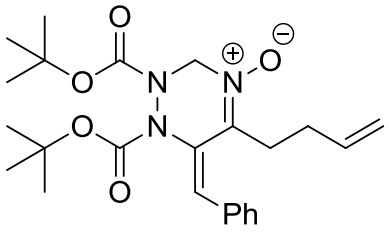
1.00
2.03
0.99
0.99
2.04
PPM
10
8
6
4
2
0



3j

PPM
200
150
100
50
0
153.080
152.260
143.183
136.537
134.678
128.557
128.480
128.414
125.849
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77.365
77.041
76.726
68.707
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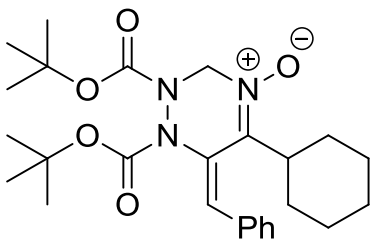
26.411



(*E*)-**3k**

3.07
1.99
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1.00
1.02
1.01
1.06
1.01
20.01
3.06
1.06
2.04
PPM
10
8
6
4
2
0
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7.266
7.228
7.221
7.210
7.001
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5.432
4.880
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2.406
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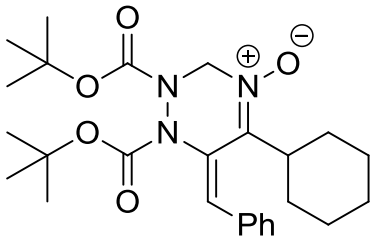
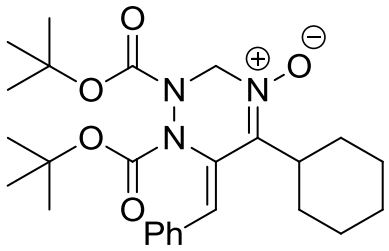
2.376
2.349
2.345
2.319
2.313
2.260
2.252
2.229
2.222
2.198
2.191
2.167
2.159
2.053
1.535
1.516
1.489
1.392
1.363
1.330
1.297
1.167
1.158
1.150
1.134
1.126
1.117
1.102
1.093
1.084
1.069
1.060
1.050
0.578
0.441
0.000


O
O
N
N
O
O
O
Ph

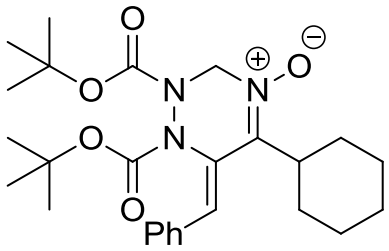
(*E*)-**3k**

PPM
200
150
100
50
0
152.906
152.134
145.488
134.962
129.355
128.173
128.125
127.887
126.075
82.902
77.315
77.000
76.676
69.620
67.751
39.891
27.877
27.791
25.942
25.608
24.940
24.559



(*Z*)-**3k**

9.07
9.08
1.97
3.05
1.05
1.00
1.01
1.01
2.01
2.01
2.01
2.02
2.06
PPM
10
8
6
4
2
0
7.882
7.347
7.306
7.289
7.267
6.791
5.566
4.904
3.360
2.309
2.175
1.890
1.860
1.769
1.740
1.536
1.516
1.490
1.461
1.369
1.347
1.150
0.000



(*Z*)-**3k**

PPM
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150
100
50
0
152.304
151.646
145.878
135.218
129.926
128.725
128.410
126.427
124.606
83.693
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77.000
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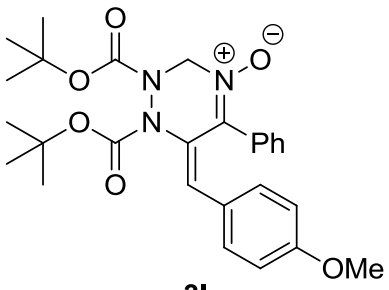
69.353
67.923
37.441
28.259
27.639
26.877
26.533
25.780
-0.020


O
O
N
N
⊕
N
O
⊖
O
O
Ph
OMe

3l

2.05
3.07
1.00
2.02
2.01
1.00
1.00
3.07
9.04
9.07
PPM
10
8
6
4
2
0
7.443
7.428
7.263
7.076
7.060
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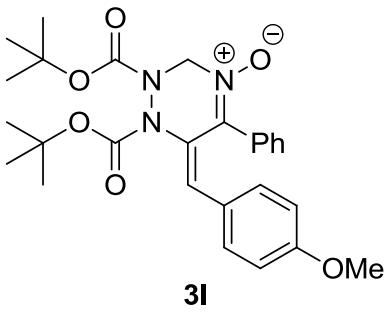
6.717
6.429
6.407
5.861
5.055
3.663
1.565
1.492
0.000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N
⊕
N
O
⊖
O
O
Ph
OMe

3l

PPM
200
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100
50
0
158.837
153.020
152.210
140.568
131.405
130.280
130.242
129.079
127.744
127.229
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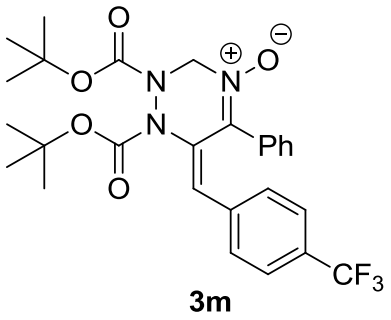
76.685
68.533
67.427
54.851
27.953
27.791


O
O
N
N
⊕
N
O
⊖
O
O
Ph
CF3

3m

8.95
9.02
4.01
2.01
2.02
1.04
1.01
1.00
PPM
10
8
6
4
2
0
7.280
7.263
7.123
7.103
7.044
7.027
7.018
6.998
6.983
6.880
6.860
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5.096
5.061
1.579
1.517
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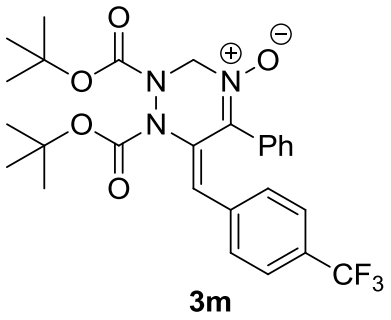
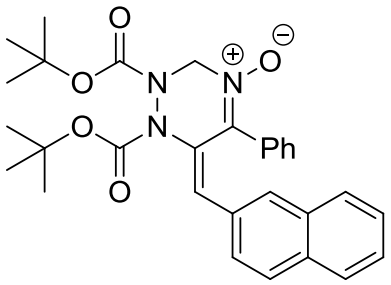

O
O
N
N
⊕
N
O
⊖
O
O
Ph
O
CF3

3m

PPM
200
150
100
50
0
152.342
152.295
139.585
137.249
130.479
129.564
129.459
129.240
128.954
128.916
128.592
128.468
128.229
127.848
127.648
126.952
125.140
124.215
124.177
124.139
124.101
122.432
119.724
83.731
77.324
77.000
76.685
70.660
68.896
28.240
28.116



3n

1.06
1.05
5.03
1.06
1.00
3.08
1.03
0.99
1.00
8.99
9.07
PPM
10
8
6
4
2
0
7.599
7.593
7.577
7.514
7.497
7.492
7.418
7.400
7.372
7.360
7.353
7.345
7.336
7.330
7.324
7.264
7.250
7.162
6.919
6.915
6.898
6.894
6.868
6.850
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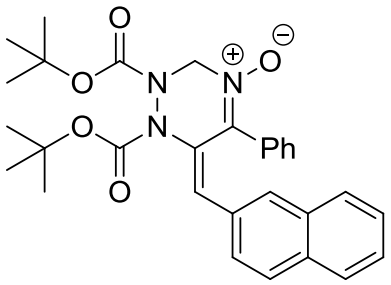
5.835
5.109
5.072
1.591
1.512
0.000



3n

PPM
200
150
100
50
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152.563
140.511
132.349
132.320
131.214
130.900
130.299
129.098
129.050
128.554
127.915
127.410
127.353
126.943
126.361
126.094
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125.551
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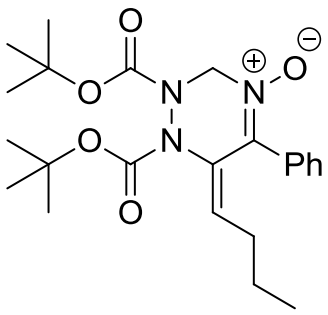
77.315
77.000
76.685
68.629
28.306
28.144


O
O
N
N
⊕
N
O
⊖
O
O
Ph

(*E*)-**3o**

9.08
9.02
5.05
3.03
2.05
2.02
1.00
0.98
1.00
PPM
10
8
6
4
2
0
7.500
7.467
7.448
7.263
5.892
5.738
4.977
1.583
1.526
1.512
1.437
1.414
1.399
1.365
1.352
1.331
1.312
1.294
1.234
1.217
1.199
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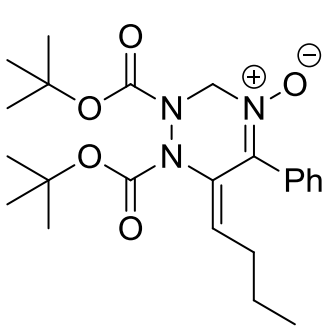
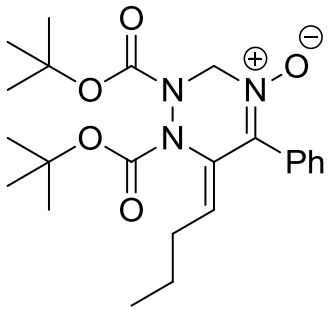
0.655
0.637
0.000



(*E*)-**3o**

PPM
200
150
100
50
0
152.944
152.525
140.635
134.895
130.719
129.660
129.260
128.507
125.074
82.892
77.315
77.000
76.676
68.342
66.989
29.870
28.058
27.944
22.099
13.299



(*Z*)-**3o**

5.01
8.902
0.99
1.04
1.00
1.01
1.03
1.97
3.03
PPM
10
8
6
4
2
0
7.468
7.265
5.733
5.495
5.481
5.472
5.458
5.001
4.967
2.334
2.173
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1.433
1.414
1.396
1.372
1.355
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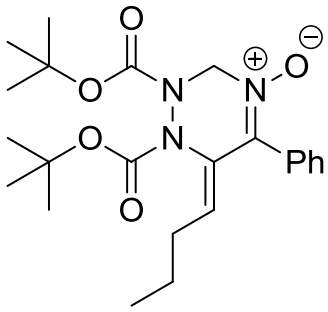
0.877
0.858
0.000


O
O
N
N
O
O
N
Ph
O

(*Z*)-**3o**

PPM
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150
100
50
0
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151.867
141.483
133.360
129.984
129.708
129.184
128.421
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77.315
77.000
76.676
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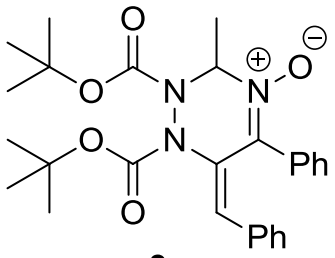
28.068
28.001
21.680
13.823



3p

2.08
0.97
3.00
3.06
2.07
1.00
3.04
9.05
9.05
PPM
10
8
6
4
2
0
7.326
7.312
7.265
7.263
7.099
6.990
6.988
6.981
6.975
6.959
6.902
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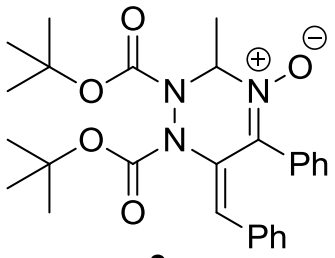

3p

PPM
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100
50
0
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130.528
128.917
128.754
128.716
127.791
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127.067
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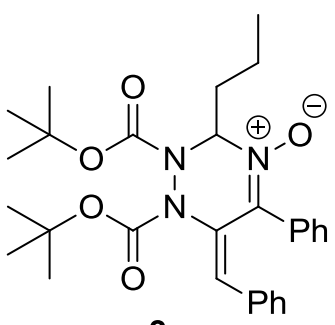
O
O
N
N
O
O
O
O
N
Ph
Ph

3q

2.05
0.98
3.07
3.04
2.06
1.00
1.03
1.06
1.08
1.08
9.07
9.04
3.02
PPM
10
8
6
4
2
0
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7.314
7.307
7.300
7.257
7.137
6.975
6.972
6.965
6.960
6.957
6.945
6.935
6.904
6.886
6.868
6.864
6.844
6.828
6.823
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2.411
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2.387
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2.362
2.353
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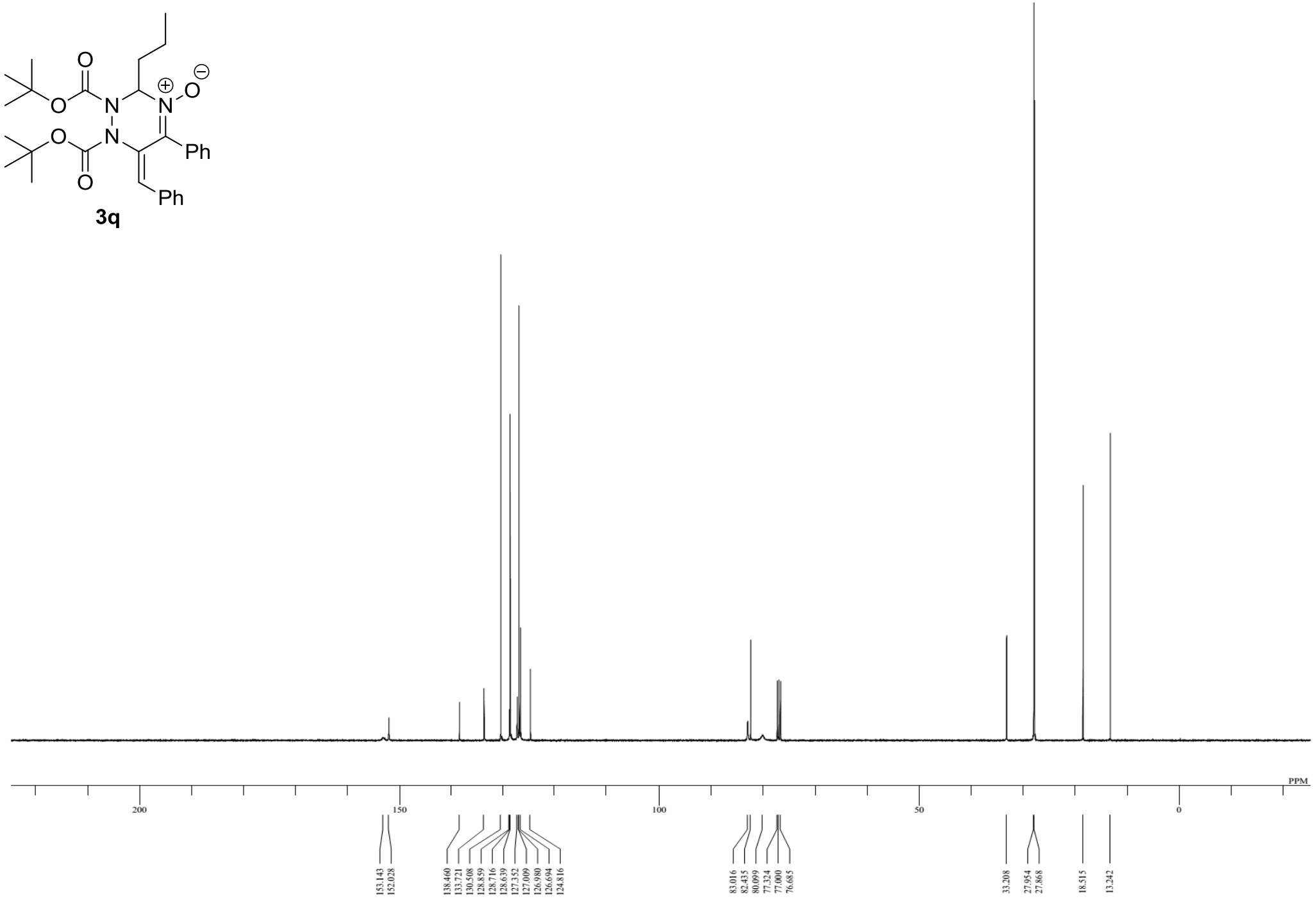
1.021
0.000


O
O
N
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⊕
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⊖
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O
N
Ph
O
Ph
3q
PPM
200
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100
50
0
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130.508
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128.716
128.639
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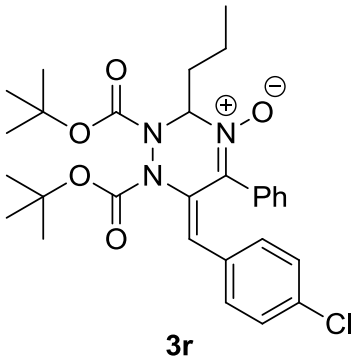
13.242


O
O
N
N
⊕
N
O
⊖
O
O
Ph
Cl

3r

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4.08
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2.06
1.00
1.06
1.01
1.08
10.02
9.08
3.03
PPM
10
8
6
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2
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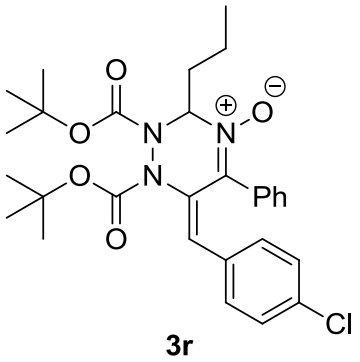
O
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⊕
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⊖
O
O
Ph
Cl

3r

PPM
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153.640
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132.769
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130.690
130.071
129.060
128.784
127.439
127.334
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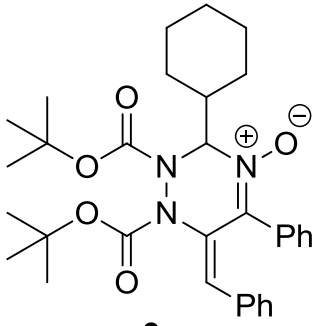
O
O
N
N
O
O
O
Ph
Ph

3s

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3.02
2.10
1.00
1.06
4.01
10.07
10.02
4.06
PPM
10
8
6
4
2
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7.330
7.325
7.315
7.307
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7.254
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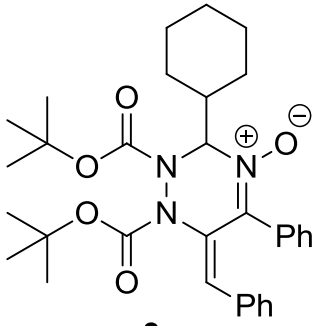
O
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⊕
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⊖
O
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Ph
Ph

3s

PPM
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127.238
127.191
126.666
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125.045
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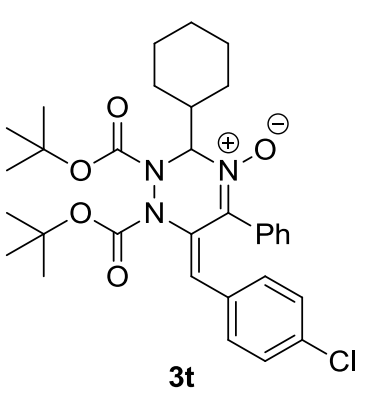
25.961


O
O
N
N
O
O
O
O
Ph
Cl

3t

2.04
2.99
1.99
2.06
1.00
1.07
4.05
20.01
4.00
PPM
10
8
6
4
2
0
7.296
7.279
7.266
7.060
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7.028
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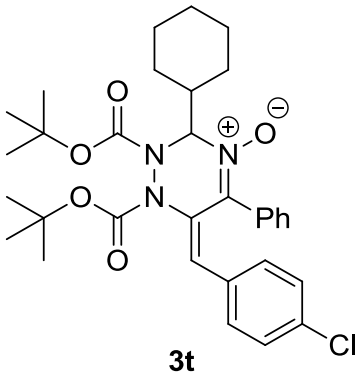
O
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N
Ph
Cl

3t

PPM
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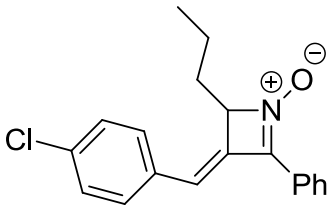
Cl
N
O
Ph

4m

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1.01
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2.01
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PPM
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8
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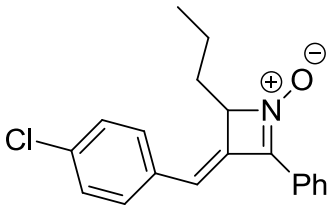
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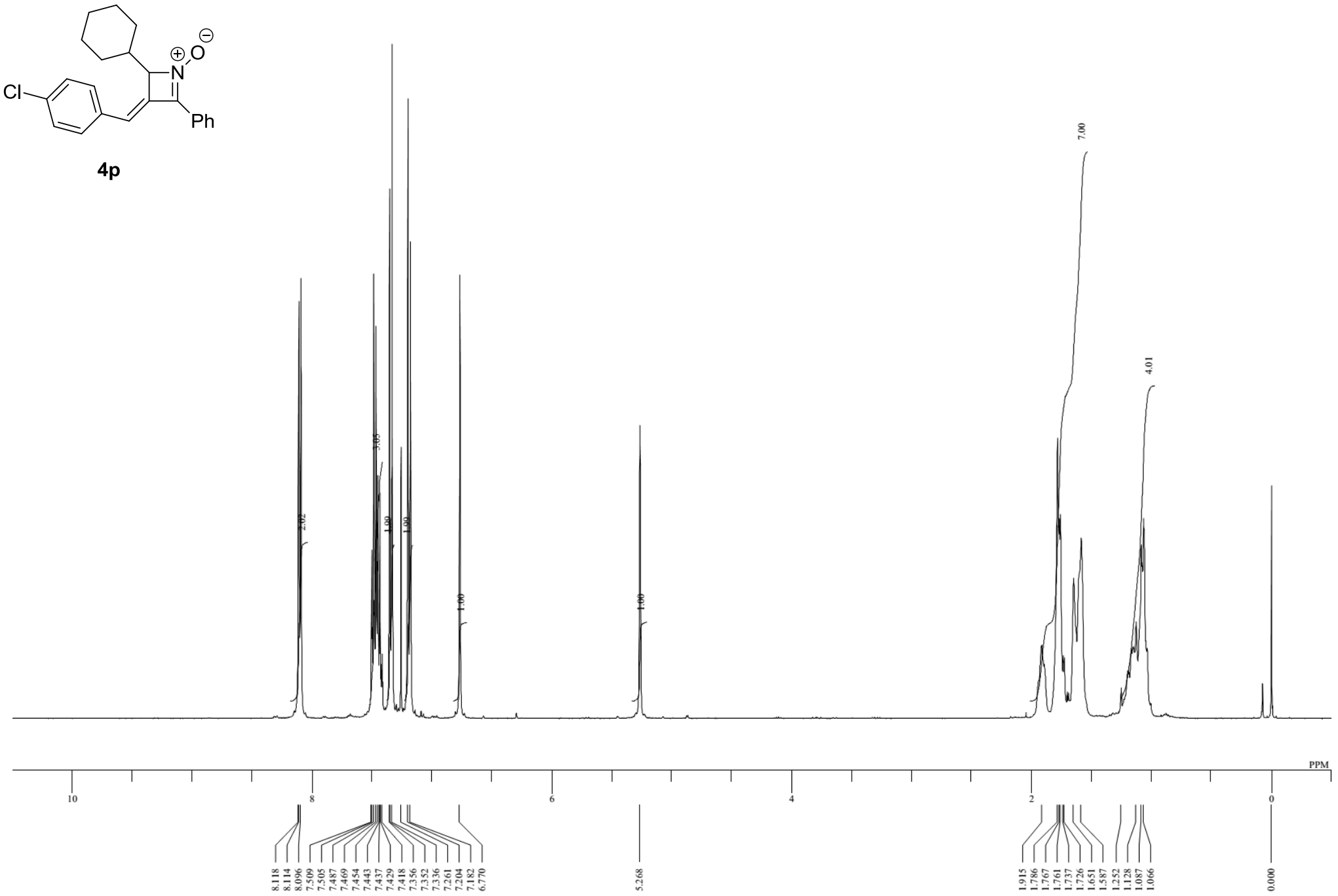
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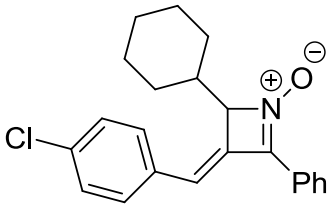
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77.315
77.000
76.676
36.945
29.708
26.533
26.200
25.828
25.799

7. X-ray crystallographic analysis of 3c

Experimental

Data Collection

A colorless prism crystal of $C_{26}H_{31}N_3O_5$ having approximate dimensions of 0.200 x 0.100 x 0.100 mm was mounted on a glass fiber. All measurements were made on a Rigaku XtaLAB mini diffractometer using graphite monochromated Mo-Kα radiation.

The crystal-to-detector distance was 50.00 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive triclinic cell with dimensions:

a	=	6.770(2) Å	α	=	74.170(5)^{o}
b	=	11.166(3) Å	β	=	82.524(6)^{o}
c	=	17.891(4) Å	γ	=	85.510(6)^{o}
V	=	1288.8(6) Å^3			

For Z = 2 and F.W. = 465.55, the calculated density is 1.200 g/cm^3. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

P-1 (#2)

The data were collected at a temperature of -123 $\pm$ 1^{o}C to a maximum 2θ value of 55.0^{o}. A total of 570 oscillation images were collected. A sweep of data was done using ω oscillations from -70.0 to 120.0^{o} in 1.0^{o} steps. The exposure rate was 120.0 [sec./^{o}]. The detector swing angle was 30.00^{o}. A second sweep was performed using ω oscillations from -70.0 to 120.0^{o} in 1.0^{o} steps. The exposure rate was 120.0 [sec./^{o}]. The detector swing angle was 30.00^{o}. Another sweep was performed using ω oscillations from -70.0 to 120.0^{o} in 1.0^{o} steps. The exposure rate was 120.0 [sec./^{o}]. The detector swing angle was 30.00^{o}. Another sweep was performed using ω oscillations from -70.0 to 120.0^{o} in 1.0^{o} steps. The exposure rate was 120.0 [sec./^{o}]. The detector swing angle was 30.00^{o}. Another sweep was performed using ω oscillations from -70.0 to 120.0^{o} in 1.0^{o} steps. The exposure rate was 120.0 [sec./^{o}]. The detector swing angle was 30.00^{o}. Another sweep was performed using ω oscillations from -70.0 to 120.0^{o} in 1.0^{o} steps. The exposure rate was 120.0 [sec./^{o}]. The detector swing angle was 30.00^{o}. The crystal-to-detector distance was 50.00 mm. Readout was performed in the 0.146 mm pixel mode.

Data Reduction

Of the 14009 reflections that were collected, 5896 were unique (R_{int} = 0.0620). Data were collected and processed using CrystalClear (Rigaku).

The linear absorption coefficient, μ, for Mo-Kα radiation is 0.836 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.679 to 0.992. The data were

corrected for Lorentz and polarization effects. A correction for secondary extinction[2] was applied (coefficient = 0.010980).

Structure Solution and Refinement

The structure was solved by direct methods[3] and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[4] on F^2 was based on 5896 observed reflections and 308 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0608$$

$$wR2 = [\Sigma \, (w \, (Fo^2 - Fc^2)^2 \,) / \, \Sigma \, w(Fo^2)^2]^{1/2} = 0.1512$$

The standard deviation of an observation of unit weight[5] was 1.02. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.26 and -0.26 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[6]. Anomalous dispersion effects were included in Fcalc[7]; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley[8]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[9]. All calculations were performed using the CrystalStructure[10] crystallographic software package except for refinement, which was performed using SHELXL-97[11].

References

(1) CrystalClear: Rigaku Corporation, 1999. CrystalClear Software User's Guide, Molecular Structure Corporation, (c) 2000.J.W.Pflugrath (1999) Acta Cryst. D55, 1718-1725.

(2) Larson, A.C. (1970), Crystallographic Computing, 291-294. F.R. Ahmed, ed. Munksgaard, Copenhagen (equation 22, with V replaced by the cell volume).

(3) SHELX97: Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

(4) Least Squares function minimized: (SHELXL97)

$\Sigma w(F_o^2-F_c^2)^2$ where w = Least Squares weights.

(5) Standard deviation of an observation of unit weight:

$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations
N_V = number of variables

(6) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(7) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(8) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(9) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(10) CrystalStructure 4.0: Crystal Structure Analysis Package, Rigaku Corporation (2000-2010). Tokyo 196-8666, Japan.

(11) SHELX97: Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{26}H_{31}N_3O_5$
Formula Weight	465.55
Crystal Color, Habit	colorless, prism
Crystal Dimensions	0.200 X 0.100 X 0.100 mm
Crystal System	triclinic
Lattice Type	Primitive
Lattice Parameters	a = 6.770(2) Å b = 11.166(3) Å c = 17.891(4) Å α = 74.170(5) ^{o} β = 82.524(6) ^{o} γ = 85.510(6) ^{o} V = 1288.8(6) Å^3
Space Group	P-1 (#2)
Z value	2
D_{calc}	1.200 g/cm^3
F_{000}	496.00
μ(MoKα)	0.836 cm^{-1}

B. Intensity Measurements

Diffractometer	XtaLAB mini
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Voltage, Current	50kV, 12mA
Temperature	-123.0^{o}C
Detector Aperture	75 mm (diameter)
Data Images	570 exposures
ω oscillation Range	-70.0 - 120.0^{o}
Exposure Rate	120.0 sec./^{o}
Detector Swing Angle	30.00^{o}
ω oscillation Range	-70.0 - 120.0^{o}
Exposure Rate	120.0 sec./^{o}
Detector Swing Angle	30.00^{o}
ω oscillation Range	-70.0 - 120.0^{o}
Exposure Rate	120.0 sec./^{o}
Detector Swing Angle	30.00^{o}
ω oscillation Range	-70.0 - 120.0^{o}
Exposure Rate	120.0 sec./^{o}
Detector Swing Angle	30.00^{o}
ω oscillation Range	-70.0 - 120.0^{o}

Exposure Rate	120.0 sec./^{o}
Detector Swing Angle	30.00^{o}
ω oscillation Range	-70.0 - 120.0^{o}
Exposure Rate	120.0 sec./^{o}
Detector Swing Angle	30.00^{o}
Detector Position	50.00 mm
Pixel Size	0.146 mm
$2\theta_{max}$	55.0^{o}
No. of Reflections Measured	Total: 14009 Unique: 5896 (R_{int} = 0.0620)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.679 - 0.992) Secondary Extinction (coefficient: 1.09800e-002)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELX97)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\Sigma\ w\ (Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1/ [\ \sigma^2(Fo^2) + (0.0620 \cdot P)^2 + 0.0737 \cdot P\ ]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$
$2\theta_{max}$ cutoff	55.0^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	5896
No. Variables	308
Reflection/Parameter Ratio	19.14
Residuals: R1 (I>2.00σ(I))	0.0608
Residuals: R (All reflections)	0.1148
Residuals: wR2 (All reflections)	0.1512
Goodness of Fit Indicator	1.022
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.26 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.26 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
O1	0.6127(2)	0.0756(2)	0.10149(8)	2.98(3)
O2	0.3464(2)	0.1858(2)	0.04699(8)	2.75(3)
O3	-0.0403(2)	-0.2410(2)	0.22622(9)	3.34(4)
O4	0.1611(2)	0.2367(2)	0.20742(9)	2.78(3)
O5	-0.1599(2)	0.1789(2)	0.22199(9)	3.36(4)
N6	0.0953(3)	0.0766(2)	0.1635(1)	2.49(4)
N7	0.0628(3)	-0.1462(2)	0.2213(1)	2.46(4)
N8	0.2991(3)	0.0465(2)	0.1628(1)	2.45(4)
C9	0.4370(3)	0.1028(2)	0.1023(2)	2.29(4)
C10	0.2249(3)	-0.1499(2)	0.2558(1)	2.17(4)
C11	0.4661(3)	-0.0045(2)	0.2788(2)	2.39(4)
C12	0.2974(3)	-0.2701(2)	0.3066(2)	2.36(4)
C13	0.4992(3)	-0.3040(2)	0.2977(2)	2.69(4)
C14	0.3415(3)	-0.0368(2)	0.2357(1)	2.19(4)
C15	0.5011(3)	-0.0657(2)	0.3601(2)	2.35(4)
C16	0.0166(3)	0.1678(2)	0.2014(2)	2.61(4)
C17	-0.0150(3)	-0.0299(2)	0.1669(2)	2.66(4)
C18	0.4675(4)	0.2661(2)	-0.0206(2)	2.86(4)
C19	0.5738(4)	-0.4128(2)	0.3465(2)	3.44(5)
C20	0.3437(4)	-0.1053(2)	0.4175(2)	3.09(5)
C21	0.1689(4)	-0.3470(2)	0.3639(2)	3.15(5)
C22	0.5663(4)	-0.1727(3)	0.5155(2)	4.01(6)
C23	0.1167(4)	0.3440(2)	0.2421(2)	3.02(5)
C24	0.6925(4)	-0.0782(3)	0.3830(2)	3.20(5)
C25	0.3766(4)	-0.1590(3)	0.4942(2)	3.96(6)
C26	0.2460(4)	-0.4546(3)	0.4127(2)	3.93(6)
C27	0.4482(5)	-0.4873(3)	0.4039(2)	4.11(6)
C28	0.7241(4)	-0.1326(3)	0.4596(2)	3.99(6)
C29	0.6082(4)	0.3374(3)	0.0082(2)	4.04(6)
C30	0.0241(4)	0.2989(3)	0.3259(2)	4.68(6)
C31	0.5770(4)	0.1875(3)	-0.0709(2)	4.23(6)
C32	0.3238(4)	0.3904(3)	0.2377(2)	4.16(6)
C33	0.3084(4)	0.3514(3)	-0.0630(2)	4.28(6)
C34	-0.0131(4)	0.4411(3)	0.1920(2)	4.39(6)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}
H11	0.5410	0.0669	0.2536	2.86
H13	0.5866	-0.2527	0.2581	3.23
H17A	-0.0073	-0.0422	0.1139	3.19
H17B	-0.1569	-0.0141	0.1847	3.19
H19	0.7119	-0.4356	0.3402	4.13
H20	0.2114	-0.0951	0.4038	3.71
H21	0.0301	-0.3259	0.3695	3.78
H22	0.5886	-0.2095	0.5684	4.82
H24	0.8023	-0.0487	0.3453	3.83
H25	0.2672	-0.1868	0.5326	4.75
H26	0.1597	-0.5064	0.4524	4.71
H27	0.4996	-0.5611	0.4376	4.94
H28	0.8559	-0.1426	0.4739	4.79
H29A	0.7087	0.2793	0.0351	4.84
H29B	0.5330	0.3804	0.0446	4.84
H29C	0.6743	0.3986	-0.0363	4.84
H30A	-0.1090	0.2693	0.3268	5.62
H30B	0.0133	0.3675	0.3508	5.62
H30C	0.1080	0.2305	0.3544	5.62
H31A	0.6785	0.1331	-0.0422	5.07
H31B	0.6414	0.2419	-0.1191	5.07
H31C	0.4822	0.1364	-0.0839	5.07
H32A	0.3804	0.4183	0.1829	5.00
H32B	0.4095	0.3228	0.2659	5.00
H32C	0.3154	0.4602	0.2616	5.00
H33A	0.2405	0.4018	-0.0295	5.13
H33B	0.2114	0.3012	-0.0755	5.13
H33C	0.3706	0.4064	-0.1115	5.13
H34A	0.0538	0.4669	0.1386	5.26
H34B	-0.0363	0.5136	0.2135	5.26
H34C	-0.1410	0.4061	0.1915	5.26

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O1	0.0255(8)	0.0415(9)	0.040(1)	0.0020(7)	-0.0036(7)	-0.0018(8)
O2	0.0312(8)	0.0351(9)	0.0317(9)	0.0009(7)	-0.0052(7)	0.0021(7)
O3	0.0338(9)	0.0368(9)	0.059(1)	-0.0079(8)	-0.0101(8)	-0.0140(8)
O4	0.0271(8)	0.0336(9)	0.047(1)	-0.0044(7)	-0.0056(7)	-0.0119(7)
O5	0.0236(8)	0.045(1)	0.061(2)	-0.0026(7)	0.0001(7)	-0.0184(9)
N6	0.0211(9)	0.032(1)	0.041(1)	-0.0007(8)	-0.0058(8)	-0.0077(9)
N7	0.0235(9)	0.031(1)	0.040(1)	-0.0040(8)	-0.0046(8)	-0.0095(9)
N8	0.0208(9)	0.034(1)	0.033(1)	-0.0009(8)	-0.0056(7)	0.0009(8)
C9	0.028(2)	0.027(1)	0.031(2)	-0.000(1)	-0.0066(9)	-0.006(1)
C10	0.022(1)	0.029(1)	0.030(2)	-0.0018(9)	0.0006(8)	-0.0076(9)
C11	0.025(1)	0.029(1)	0.035(2)	-0.0035(9)	-0.0020(9)	-0.006(1)
C12	0.031(1)	0.028(1)	0.031(2)	-0.005(1)	-0.0052(9)	-0.007(1)
C13	0.034(2)	0.031(2)	0.039(2)	-0.002(1)	-0.008(1)	-0.009(1)
C14	0.023(1)	0.027(1)	0.029(2)	-0.0020(9)	-0.0029(9)	-0.0017(9)
C15	0.032(2)	0.028(1)	0.030(2)	-0.004(1)	-0.0041(9)	-0.0076(9)
C16	0.028(2)	0.034(2)	0.036(2)	-0.001(1)	-0.0067(9)	-0.005(1)
C17	0.025(1)	0.035(2)	0.041(2)	0.002(1)	-0.0101(9)	-0.008(1)
C18	0.047(2)	0.030(2)	0.027(2)	-0.000(1)	-0.001(1)	-0.001(1)
C19	0.045(2)	0.035(2)	0.053(2)	0.005(2)	-0.020(2)	-0.011(2)
C20	0.040(2)	0.043(2)	0.034(2)	-0.008(1)	-0.002(1)	-0.010(1)
C21	0.042(2)	0.039(2)	0.037(2)	-0.009(1)	0.001(1)	-0.007(1)
C22	0.077(2)	0.043(2)	0.031(2)	-0.008(2)	-0.016(2)	-0.002(2)
C23	0.042(2)	0.039(2)	0.037(2)	-0.008(1)	-0.004(1)	-0.013(1)
C24	0.035(2)	0.050(2)	0.036(2)	-0.005(1)	-0.005(1)	-0.009(2)
C25	0.063(2)	0.052(2)	0.033(2)	-0.018(2)	0.006(2)	-0.008(2)
C26	0.073(2)	0.037(2)	0.035(2)	-0.016(2)	-0.002(2)	0.001(1)
C27	0.075(2)	0.032(2)	0.051(2)	0.001(2)	-0.028(2)	-0.005(2)
C28	0.051(2)	0.060(2)	0.042(2)	0.001(2)	-0.020(2)	-0.009(2)
C29	0.061(2)	0.037(2)	0.051(2)	-0.010(2)	-0.005(2)	-0.003(2)
C30	0.063(2)	0.072(2)	0.045(2)	-0.022(2)	0.004(2)	-0.017(2)
C31	0.072(2)	0.049(2)	0.036(2)	-0.001(2)	0.005(2)	-0.010(2)
C32	0.054(2)	0.056(2)	0.056(2)	-0.022(2)	-0.001(2)	-0.024(2)
C33	0.063(2)	0.044(2)	0.045(2)	0.003(2)	-0.012(2)	0.007(2)
C34	0.063(2)	0.035(2)	0.068(2)	0.005(2)	-0.012(2)	-0.012(2)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
O1	C9	1.204(3)	O2	C9	1.335(3)
O2	C18	1.485(3)	O3	N7	1.290(3)
O4	C16	1.324(3)	O4	C23	1.487(3)
O5	C16	1.211(3)	N6	N8	1.394(3)
N6	C16	1.406(3)	N6	C17	1.436(3)
N7	C10	1.320(3)	N7	C17	1.499(3)
N8	C9	1.377(3)	N8	C14	1.430(3)
C10	C12	1.488(3)	C10	C14	1.477(3)
C11	C14	1.343(4)	C11	C15	1.470(3)
C12	C13	1.390(3)	C12	C21	1.396(3)
C13	C19	1.390(3)	C15	C20	1.389(3)
C15	C24	1.395(3)	C18	C29	1.509(4)
C18	C31	1.511(4)	C18	C33	1.517(4)
C19	C27	1.374(4)	C20	C25	1.379(4)
C21	C26	1.388(3)	C22	C25	1.372(4)
C22	C28	1.377(4)	C23	C30	1.512(4)
C23	C32	1.518(4)	C23	C34	1.509(4)
C24	C28	1.376(4)	C26	C27	1.389(4)

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C11	H11	0.950	C13	H13	0.950
C17	H17A	0.990	C17	H17B	0.990
C19	H19	0.950	C20	H20	0.950
C21	H21	0.950	C22	H22	0.950
C24	H24	0.950	C25	H25	0.950
C26	H26	0.950	C27	H27	0.950
C28	H28	0.950	C29	H29A	0.980
C29	H29B	0.980	C29	H29C	0.980
C30	H30A	0.980	C30	H30B	0.980
C30	H30C	0.980	C31	H31A	0.980
C31	H31B	0.980	C31	H31C	0.980
C32	H32A	0.980	C32	H32B	0.980
C32	H32C	0.980	C33	H33A	0.980
C33	H33B	0.980	C33	H33C	0.980
C34	H34A	0.980	C34	H34B	0.980
C34	H34C	0.980			

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
C9	O2	C18	119.79(15)	C16	O4	C23	120.87(16)
N8	N6	C16	117.46(19)	N8	N6	C17	111.36(16)
C16	N6	C17	121.46(16)	O3	N7	C10	125.09(17)
O3	N7	C17	112.04(17)	C10	N7	C17	122.77(18)
N6	N8	C9	122.60(16)	N6	N8	C14	110.66(14)
C9	N8	C14	126.34(16)	O1	C9	O2	127.11(17)
O1	C9	N8	122.44(17)	O2	C9	N8	110.43(16)
N7	C10	C12	119.61(19)	N7	C10	C14	118.38(17)
C12	C10	C14	121.66(18)	C14	C11	C15	128.06(18)
C10	C12	C13	118.65(17)	C10	C12	C21	121.64(18)
C13	C12	C21	119.68(18)	C12	C13	C19	120.25(19)
N8	C14	C10	111.80(18)	N8	C14	C11	120.10(18)
C10	C14	C11	127.99(17)	C11	C15	C20	121.17(19)
C11	C15	C24	120.92(17)	C20	C15	C24	117.8(2)
O4	C16	O5	127.9(3)	O4	C16	N6	109.89(17)
O5	C16	N6	122.2(3)	N6	C17	N7	112.14(18)
O2	C18	C29	109.73(18)	O2	C18	C31	109.79(18)
O2	C18	C33	101.87(17)	C29	C18	C31	112.0(2)
C29	C18	C33	111.9(2)	C31	C18	C33	111.0(2)
C13	C19	C27	120.1(3)	C15	C20	C25	121.0(3)
C12	C21	C26	119.3(2)	C25	C22	C28	119.5(3)
O4	C23	C30	109.84(19)	O4	C23	C32	101.49(18)
O4	C23	C34	109.7(2)	C30	C23	C32	111.3(3)
C30	C23	C34	112.9(2)	C32	C23	C34	110.97(19)
C15	C24	C28	120.8(2)	C20	C25	C22	120.4(3)
C21	C26	C27	120.7(2)	C19	C27	C26	119.9(3)
C22	C28	C24	120.5(3)				

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C14	C11	H11	116.0	C15	C11	H11	116.0
C12	C13	H13	119.9	C19	C13	H13	119.9
N6	C17	H17A	109.2	N6	C17	H17B	109.2
N7	C17	H17A	109.2	N7	C17	H17B	109.2
H17A	C17	H17B	107.9	C13	C19	H19	119.9
C27	C19	H19	119.9	C15	C20	H20	119.5
C25	C20	H20	119.5	C12	C21	H21	120.3
C26	C21	H21	120.3	C25	C22	H22	120.3
C28	C22	H22	120.3	C15	C24	H24	119.6
C28	C24	H24	119.6	C20	C25	H25	119.8
C22	C25	H25	119.8	C21	C26	H26	119.7
C27	C26	H26	119.7	C19	C27	H27	120.1
C26	C27	H27	120.1	C22	C28	H28	119.8
C24	C28	H28	119.8	C18	C29	H29A	109.5
C18	C29	H29B	109.5	C18	C29	H29C	109.5
H29A	C29	H29B	109.5	H29A	C29	H29C	109.5
H29B	C29	H29C	109.5	C23	C30	H30A	109.5
C23	C30	H30B	109.5	C23	C30	H30C	109.5
H30A	C30	H30B	109.5	H30A	C30	H30C	109.5
H30B	C30	H30C	109.5	C18	C31	H31A	109.5
C18	C31	H31B	109.5	C18	C31	H31C	109.5
H31A	C31	H31B	109.5	H31A	C31	H31C	109.5
H31B	C31	H31C	109.5	C23	C32	H32A	109.5
C23	C32	H32B	109.5	C23	C32	H32C	109.5
H32A	C32	H32B	109.5	H32A	C32	H32C	109.5
H32B	C32	H32C	109.5	C18	C33	H33A	109.5
C18	C33	H33B	109.5	C18	C33	H33C	109.5
H33A	C33	H33B	109.5	H33A	C33	H33C	109.5
H33B	C33	H33C	109.5	C23	C34	H34A	109.5
C23	C34	H34B	109.5	C23	C34	H34C	109.5
H34A	C34	H34B	109.5	H34A	C34	H34C	109.5
H34B	C34	H34C	109.5				

Table 8. Torsion Angles(O)
(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C9	O2	C18	C29	-56.3(3)	C9	O2	C18	C31	67.3(3)
C9	O2	C18	C33	-174.98(17)	C18	O2	C9	O1	-7.1(4)
C18	O2	C9	N8	174.46(17)	C16	O4	C23	C30	61.1(3)
C16	O4	C23	C32	179.00(15)	C16	O4	C23	C34	-63.6(2)
C23	O4	C16	O5	-0.8(3)	C23	O4	C16	N6	177.16(14)
N8	N6	C16	O4	20.5(2)	N8	N6	C16	O5	-161.43(16)
C16	N6	N8	C9	-93.6(3)	C16	N6	N8	C14	79.67(19)
N8	N6	C17	N7	40.3(3)	C17	N6	N8	C9	119.85(19)
C17	N6	N8	C14	-66.9(2)	C16	N6	C17	N7	-104.71(19)
C17	N6	C16	O4	163.51(16)	C17	N6	C16	O5	-18.4(3)
O3	N7	C10	C12	-1.6(3)	O3	N7	C10	C14	171.72(16)
O3	N7	C17	N6	177.95(15)	C10	N7	C17	N6	-5.6(3)
C17	N7	C10	C12	-177.59(16)	C17	N7	C10	C14	-4.3(3)
N6	N8	C9	O1	-179.1(2)	N6	N8	C9	O2	-0.6(3)
N6	N8	C14	C10	55.3(3)	N6	N8	C14	C11	-121.19(18)
C9	N8	C14	C10	-131.8(2)	C9	N8	C14	C11	51.7(3)
C14	N8	C9	O1	8.7(4)	C14	N8	C9	O2	-172.72(19)
N7	C10	C12	C13	133.9(2)	N7	C10	C12	C21	-48.0(3)
N7	C10	C14	N8	-19.8(3)	N7	C10	C14	C11	156.33(18)
C12	C10	C14	N8	153.36(17)	C12	C10	C14	C11	-30.5(3)
C14	C10	C12	C13	-39.2(3)	C14	C10	C12	C21	138.9(2)
C14	C11	C15	C20	-43.8(4)	C14	C11	C15	C24	140.7(2)
C15	C11	C14	N8	168.31(18)	C15	C11	C14	C10	-7.6(4)
C10	C12	C13	C19	177.0(2)	C10	C12	C21	C26	-176.3(2)
C13	C12	C21	C26	1.8(4)	C21	C12	C13	C19	-1.2(4)
C12	C13	C19	C27	-0.1(4)	C11	C15	C20	C25	-177.85(19)
C11	C15	C24	C28	178.3(2)	C20	C15	C24	C28	2.6(4)
C24	C15	C20	C25	-2.2(4)	C13	C19	C27	C26	0.7(5)
C15	C20	C25	C22	1.1(4)	C12	C21	C26	C27	-1.2(4)
C25	C22	C28	C24	0.6(4)	C28	C22	C25	C20	-0.2(4)
C15	C24	C28	C22	-1.9(4)	C21	C26	C27	C19	-0.0(5)

Table 9. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
O1	N6	3.534(3)	O1	C11	3.101(3)
O1	C14	2.878(3)	O1	C18	2.822(3)
O1	C29	2.944(3)	O1	C31	3.029(3)
O2	O4	3.159(3)	O2	N6	2.595(2)
O2	C16	3.289(3)	O3	N6	3.571(3)
O3	C12	2.808(3)	O3	C21	2.921(3)
O4	N8	2.537(3)	O4	C9	3.073(3)
O4	C11	3.329(3)	O4	C14	3.132(3)
O4	C17	3.573(3)	O5	N8	3.499(3)
O5	C17	2.831(3)	O5	C23	2.848(3)
O5	C30	3.004(4)	O5	C34	3.052(3)
N6	C10	2.766(3)	N6	C11	3.370(3)
N7	N8	2.676(3)	N7	C13	3.553(3)
N7	C16	3.419(3)	N7	C21	3.026(3)
C9	C10	3.598(3)	C9	C11	3.083(3)
C9	C16	3.291(3)	C9	C17	3.428(3)
C9	C29	2.932(3)	C9	C31	3.028(4)
C10	C15	3.162(4)	C10	C20	3.274(4)
C11	C12	3.151(4)	C11	C13	3.262(4)
C12	C15	3.171(4)	C12	C20	3.110(4)
C12	C27	2.780(3)	C13	C14	3.046(3)
C13	C15	3.155(4)	C13	C20	3.503(4)
C13	C26	2.764(3)	C14	C16	3.039(3)
C14	C17	2.835(3)	C14	C20	3.133(3)
C15	C22	2.789(4)	C16	C30	2.988(4)
C16	C34	3.004(4)	C19	C21	2.782(4)
C20	C21	3.437(4)	C20	C28	2.747(4)
C24	C25	2.750(4)			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O1	H11	2.677	O1	H29A	2.359
O1	H29B	3.298	O1	H31A	2.462
O1	H31C	3.418	O2	H17A	3.492
O2	H29A	2.701	O2	H29B	2.586
O2	H29C	3.291	O2	H31A	2.705
O2	H31B	3.293	O2	H31C	2.588
O2	H33A	2.528	O2	H33B	2.469
O2	H33C	3.204	O3	H17A	2.558
O3	H17B	2.531	O3	H21	2.572
O4	H11	3.134	O4	H30A	2.710
O4	H30B	3.295	O4	H30C	2.589
O4	H32A	2.515	O4	H32B	2.471
O4	H32C	3.202	O4	H34A	2.620
O4	H34B	3.295	O4	H34C	2.663
O5	H17A	3.548	O5	H17B	2.422
O5	H30A	2.432	O5	H30C	3.355
O5	H34A	3.495	O5	H34C	2.455
N6	H11	3.582	N7	H21	2.849
N8	H11	2.514	N8	H17A	2.706
N8	H17B	3.164	C9	H11	2.800
C9	H17A	3.483	C9	H29A	2.723
C9	H29B	3.081	C9	H31A	2.825
C9	H31C	3.225	C10	H11	3.343
C10	H13	2.622	C10	H17A	3.073
C10	H17B	3.129	C10	H20	2.864
C10	H21	2.695	C11	H13	2.935
C11	H20	2.653	C11	H24	2.655
C11	H32B	3.591	C12	H19	3.267
C12	H20	2.932	C12	H26	3.261
C13	H21	3.269	C13	H27	3.254
C14	H13	2.783	C14	H17A	3.431
C14	H17B	3.582	C14	H20	2.928
C15	H13	3.104	C15	H25	3.263
C15	H28	3.264	C16	H17A	3.178
C16	H17B	2.527	C16	H30A	2.791
C16	H30C	3.150	C16	H34A	3.240
C16	H34C	2.757	C19	H26	3.246

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C20	H11	3.224	C20	H22	3.246
C20	H24	3.244	C21	H13	3.267
C21	H20	3.127	C21	H27	3.268
C22	H20	3.240	C22	H24	3.243
C24	H11	2.712	C24	H13	3.510
C24	H20	3.244	C24	H22	3.247
C25	H28	3.232	C26	H19	3.250
C27	H13	3.251	C27	H21	3.270
C28	H25	3.233	C29	H31A	2.661
C29	H31B	2.744	C29	H31C	3.341
C29	H33A	2.658	C29	H33B	3.343
C29	H33C	2.752	C30	H32A	3.343
C30	H32B	2.694	C30	H32C	2.700
C30	H34A	3.353	C30	H34B	2.718
C30	H34C	2.717	C31	H29A	2.661
C31	H29B	3.342	C31	H29C	2.743
C31	H33A	3.338	C31	H33B	2.693
C31	H33C	2.686	C32	H29B	3.587
C32	H30A	3.345	C32	H30B	2.697
C32	H30C	2.695	C32	H34A	2.644
C32	H34B	2.729	C32	H34C	3.336
C33	H29A	3.347	C33	H29B	2.703
C33	H29C	2.701	C33	H31A	3.339
C33	H31B	2.690	C33	H31C	2.687
C34	H30A	2.680	C34	H30B	2.758
C34	H30C	3.351	C34	H32A	2.644
C34	H32B	3.334	C34	H32C	2.734
H11	H13	3.537	H11	H20	3.449
H11	H24	2.595	H11	H32B	2.983
H13	H19	2.337	H13	H24	3.579
H19	H27	2.324	H20	H21	3.175
H20	H25	2.319	H20	H30C	3.535
H21	H26	2.338	H22	H25	2.323
H22	H28	2.327	H24	H28	2.317
H26	H27	2.335	H29A	H31A	2.444
H29A	H31B	2.996	H29A	H31C	3.538
H29A	H33A	3.559	H29B	H31A	3.538

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H29B	H32A	2.684	H29B	H33A	2.485
H29B	H33B	3.564	H29B	H33C	3.066
H29C	H31A	2.993	H29C	H31B	2.621
H29C	H33A	2.922	H29C	H33C	2.583
H30A	H32B	3.584	H30A	H32C	3.590
H30A	H34A	3.576	H30A	H34B	2.953
H30A	H34C	2.512	H30B	H32A	3.587
H30B	H32B	2.982	H30B	H32C	2.526
H30B	H34B	2.597	H30B	H34C	3.071
H30C	H32A	3.582	H30C	H32B	2.516
H30C	H32C	2.990	H30C	H34C	3.580
H31A	H33B	3.582	H31A	H33C	3.578
H31B	H33A	3.578	H31B	H33B	2.985
H31B	H33C	2.509	H31C	H33A	3.579
H31C	H33B	2.514	H31C	H33C	2.971
H32A	H34A	2.417	H32A	H34B	2.972
H32A	H34C	3.524	H32B	H34A	3.521
H32C	H34A	2.982	H32C	H34B	2.607
H33A	H34A	3.351			

Table 11. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
O1	$O5^{1}$	3.293(3)	O1	$N6^{1}$	3.586(3)
O1	$C17^{1}$	2.943(3)	O1	$C31^{2}$	3.497(4)
O3	$C13^{3}$	3.266(3)	O3	$C19^{3}$	3.494(3)
O5	$O1^{3}$	3.293(3)	O5	$C11^{3}$	3.262(3)
O5	$C24^{3}$	3.568(3)	N6	$O1^{3}$	3.586(3)
N7	$C31^{2}$	3.492(3)	N8	$C31^{2}$	3.447(4)
C9	$C31^{2}$	3.448(4)	C10	$C31^{2}$	3.527(4)
C11	$O5^{1}$	3.262(3)	C13	$O3^{1}$	3.266(3)
C17	$O1^{3}$	2.943(3)	C19	$O3^{1}$	3.494(3)
C24	$O5^{1}$	3.568(3)	C25	$C28^{4}$	3.568(4)
C27	$C27^{5}$	3.528(4)	C28	$C25^{4}$	3.568(4)
C31	$O1^{2}$	3.497(4)	C31	$N7^{2}$	3.492(3)
C31	$N8^{2}$	3.447(4)	C31	$C9^{2}$	3.448(4)
C31	$C10^{2}$	3.527(4)	C33	$C34^{6}$	3.500(4)
C34	$C33^{6}$	3.500(4)			

Symmetry Operators:

(1)	X+1,Y,Z	(2)	-X+1,-Y,-Z
(3)	X-1,Y,Z	(4)	-X+1,-Y,-Z+1
(5)	-X+1,-Y-1,-Z+1	(6)	-X,-Y+1,-Z

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O1	H17A[1]	2.803	O1	H17B[1]	2.280
O1	H31C[2]	2.610	O3	H13[3]	2.522
O3	H19[3]	2.993	O3	H24[3]	3.440
O3	H31B[2]	3.103	O3	H33B[4]	3.315
O3	H34B[5]	2.809	O5	H11[3]	2.387
O5	H24[3]	2.879	O5	H29A[3]	3.435
O5	H32B[3]	3.325	N6	H24[3]	3.571
N7	H13[3]	3.445	N7	H24[3]	3.036
N7	H31A[2]	3.418	N7	H31B[2]	2.876
N8	H31A[2]	3.307	N8	H31B[2]	3.497
N8	H31C[2]	2.986	C9	H17B[1]	3.298
C9	H31A[2]	3.286	C9	H31C[2]	2.783
C10	H24[3]	3.364	C10	H31B[2]	2.917
C10	H31C[2]	3.409	C11	H17B[1]	2.881
C12	H31B[2]	3.257	C12	H32C[5]	3.312
C13	H31B[2]	3.329	C13	H32C[5]	3.248
C14	H17B[1]	3.410	C14	H31B[2]	3.481
C14	H31C[2]	3.262	C15	H17B[1]	3.588
C16	H11[3]	3.424	C16	H24[3]	3.281
C17	H11[3]	3.435	C17	H24[3]	3.227
C17	H31A[2]	3.342	C17	H31B[2]	3.512
C19	H21[1]	3.416	C19	H32C[5]	3.104
C19	H34B[6]	3.521	C20	H28[3]	3.350
C21	H19[3]	3.433	C21	H30B[5]	3.511
C21	H32C[5]	3.211	C22	H27[7]	2.913
C22	H30C[8]	3.315	C24	H17B[1]	3.454
C24	H20[1]	3.565	C24	H21[1]	3.488
C25	H27[7]	3.119	C25	H28[3]	3.576
C25	H30A[9]	3.426	C26	H26[10]	3.384
C26	H27[7]	3.416	C26	H30B[5]	3.130
C26	H32C[5]	3.072	C27	H22[7]	3.309
C27	H27[7]	3.094	C27	H32C[5]	3.022
C28	H20[1]	3.344	C28	H21[1]	3.446
C28	H30C[8]	3.517	C29	H29B[11]	3.143
C29	H29C[11]	3.504	C29	H33A[11]	3.293
C30	H19[12]	3.552	C30	H22[8]	3.361
C30	H25[9]	3.037	C31	H13[2]	3.534

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C31	H17A[2]	3.327	C32	H33C[11]	3.354
C33	H29B[11]	3.371	C33	H32A[11]	3.509
C33	H34A[13]	3.247	C33	H34B[13]	3.400
C33	H34C[13]	3.271	C34	H19[12]	3.562
C34	H29C[11]	3.533	C34	H33A[13]	3.444
C34	H33B[13]	3.358	C34	H33C[13]	3.124
H11	O5[1]	2.387	H11	C16[1]	3.424
H11	C17[1]	3.435	H11	H17B[1]	2.487
H13	O3[1]	2.522	H13	N7[1]	3.445
H13	C31[2]	3.534	H13	H17B[1]	3.172
H13	H31B[2]	3.064	H13	H31C[2]	3.113
H13	H33B[2]	3.539	H13	H33C[2]	3.477
H17A	O1[3]	2.803	H17A	C31[2]	3.327
H17A	H31A[2]	2.691	H17A	H31B[2]	3.193
H17A	H33B[4]	3.563	H17B	O1[3]	2.280
H17B	C9[3]	3.298	H17B	C11[3]	2.881
H17B	C14[3]	3.410	H17B	C15[3]	3.588
H17B	C24[3]	3.454	H17B	H11[3]	2.487
H17B	H13[3]	3.172	H17B	H24[3]	2.774
H17B	H31C[4]	3.580	H19	O3[1]	2.993
H19	C21[1]	3.433	H19	C30[6]	3.552
H19	C34[6]	3.562	H19	H21[1]	2.734
H19	H30A[6]	3.480	H19	H30B[6]	2.864
H19	H32C[5]	3.592	H19	H34B[6]	2.812
H19	H34C[6]	3.577	H20	C24[3]	3.565
H20	C28[3]	3.344	H20	H24[3]	3.044
H20	H28[3]	2.588	H21	C19[3]	3.416
H21	C24[3]	3.488	H21	C28[3]	3.446
H21	H19[3]	2.734	H21	H24[3]	3.292
H21	H26[10]	3.372	H21	H28[3]	3.195
H21	H30B[5]	3.541	H22	C27[7]	3.309
H22	C30[8]	3.361	H22	H26[7]	3.569
H22	H27[7]	2.708	H22	H30A[9]	3.546
H22	H30B[8]	3.368	H22	H30C[8]	2.583
H22	H32B[8]	2.890	H24	O3[1]	3.440
H24	O5[1]	2.879	H24	N6[1]	3.571
H24	N7[1]	3.036	H24	C10[1]	3.364

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H24	C16[1]	3.281	H24	C17[1]	3.227
H24	H17B[1]	2.774	H24	H20[1]	3.044
H24	H21[1]	3.292	H24	H30A[1]	3.567
H25	C30[9]	3.037	H25	H27[7]	3.067
H25	H28[3]	3.059	H25	H30A[9]	2.552
H25	H30B[9]	3.051	H25	H30C[9]	3.017
H26	C26[10]	3.384	H26	H21[10]	3.372
H26	H22[7]	3.569	H26	H26[10]	2.592
H26	H27[7]	3.467	H26	H30B[5]	2.891
H26	H32C[5]	3.550	H27	C22[7]	2.913
H27	C25[7]	3.119	H27	C26[7]	3.416
H27	C27[7]	3.094	H27	H22[7]	2.708
H27	H25[7]	3.067	H27	H26[7]	3.467
H27	H27[7]	2.917	H27	H32C[5]	3.478
H28	C20[1]	3.350	H28	C25[1]	3.576
H28	H20[1]	2.588	H28	H21[1]	3.195
H28	H25[1]	3.059	H28	H30C[8]	2.995
H29A	O5[1]	3.435	H29A	H33A[11]	3.577
H29B	C29[11]	3.143	H29B	C33[11]	3.371
H29B	H29B[11]	2.749	H29B	H29C[11]	2.719
H29B	H33A[11]	2.908	H29B	H33C[11]	3.087
H29C	C29[11]	3.504	H29C	C34[11]	3.533
H29C	H29B[11]	2.719	H29C	H29C[11]	3.514
H29C	H32A[11]	2.899	H29C	H33A[11]	2.913
H29C	H34A[11]	2.655	H30A	C25[9]	3.426
H30A	H19[12]	3.480	H30A	H22[9]	3.546
H30A	H24[3]	3.567	H30A	H25[9]	2.552
H30A	H32B[3]	3.527	H30B	C21[14]	3.511
H30B	C26[14]	3.130	H30B	H19[12]	2.864
H30B	H21[14]	3.541	H30B	H22[8]	3.368
H30B	H25[9]	3.051	H30B	H26[14]	2.891
H30C	C22[8]	3.315	H30C	C28[8]	3.517
H30C	H22[8]	2.583	H30C	H25[9]	3.017
H30C	H28[8]	2.995	H31A	N7[2]	3.418
H31A	N8[2]	3.307	H31A	C9[2]	3.286
H31A	C17[2]	3.342	H31A	H17A[2]	2.691
H31A	H31C[2]	3.391	H31B	O3[2]	3.103

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H31B	N7[2]	2.876	H31B	N8[2]	3.497
H31B	C10[2]	2.917	H31B	C12[2]	3.257
H31B	C13[2]	3.329	H31B	C14[2]	3.481
H31B	C17[2]	3.512	H31B	H13[2]	3.064
H31B	H17A[2]	3.193	H31C	O1[2]	2.610
H31C	N8[2]	2.986	H31C	C9[2]	2.783
H31C	C10[2]	3.409	H31C	C14[2]	3.262
H31C	H13[2]	3.113	H31C	H17B[4]	3.580
H31C	H31A[2]	3.391	H32A	C33[11]	3.509
H32A	H29C[11]	2.899	H32A	H33C[11]	2.626
H32A	H34C[1]	3.255	H32B	O5[1]	3.325
H32B	H22[8]	2.890	H32B	H30A[1]	3.527
H32B	H34C[1]	3.258	H32C	C12[14]	3.312
H32C	C13[14]	3.248	H32C	C19[14]	3.104
H32C	C21[14]	3.211	H32C	C26[14]	3.072
H32C	C27[14]	3.022	H32C	H19[14]	3.592
H32C	H26[14]	3.550	H32C	H27[14]	3.478
H32C	H33C[11]	3.283	H33A	C29[11]	3.293
H33A	C34[13]	3.444	H33A	H29A[11]	3.577
H33A	H29B[11]	2.908	H33A	H29C[11]	2.913
H33A	H34A[13]	2.986	H33A	H34B[13]	3.596
H33A	H34C[13]	3.216	H33B	O3[4]	3.315
H33B	C34[13]	3.358	H33B	H13[2]	3.539
H33B	H17A[4]	3.563	H33B	H34A[13]	3.067
H33B	H34B[13]	3.061	H33B	H34C[13]	3.395
H33C	C32[11]	3.354	H33C	C34[13]	3.124
H33C	H13[2]	3.477	H33C	H29B[11]	3.087
H33C	H32A[11]	2.626	H33C	H32C[11]	3.283
H33C	H34A[13]	3.138	H33C	H34B[13]	3.021
H33C	H34C[13]	2.699	H34A	C33[13]	3.247
H34A	H29C[11]	2.655	H34A	H33A[13]	2.986
H34A	H33B[13]	3.067	H34A	H33C[13]	3.138
H34B	O3[14]	2.809	H34B	C19[12]	3.521
H34B	C33[13]	3.400	H34B	H19[12]	2.812
H34B	H33A[13]	3.596	H34B	H33B[13]	3.061
H34B	H33C[13]	3.021	H34C	C33[13]	3.271
H34C	H19[12]	3.577	H34C	H32A[3]	3.255

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H34C	H32B[3]	3.258	H34C	H33A[13]	3.216
H34C	H33B[13]	3.395	H34C	H33C[13]	2.699

Symmetry Operators:

(1) X+1,Y,Z
(2) -X+1,-Y,-Z
(3) X-1,Y,Z
(4) -X,-Y,-Z
(5) X,Y-1,Z
(6) X+1,Y-1,Z
(7) -X+1,-Y-1,-Z+1
(8) -X+1,-Y,-Z+1
(9) -X,-Y,-Z+1
(10) -X,-Y-1,-Z+1
(11) -X+1,-Y+1,-Z
(12) X-1,Y+1,Z
(13) -X,-Y+1,-Z
(14) X,Y+1,Z