Electronic Supplementary Information

Metal-free decarbonylative alkylation-aminoxidation of styrene derivatives with aliphatic aldehydes and N-hydroxyphthalimide

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I. General information

Unless otherwise noted, all commercially available compounds were used as provided without further purification. Dry solvents (toluene, benzene, chlorobenzene o-dichlorobenzene, 1,2-dichloroethane, dioxane, acetonitrile, dimethylformamide) were used as commercially available;

Thin-layer chromatography (TLC) was performed using E. Merck silica gel 60 F254 precoated plates (0.25 mm) or Sorbent Silica Gel 60 F254 plates. The developed chromatography was analyzed by UV lamp (254 nm). High-resolution mass spectra (HRMS) were obtained from a JEOL JMS-700 instrument (ESI). Melting points are uncorrected. Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker Avance 400 spectrometer at ambient temperature. Chemical shifts for ^{1}H NMR spectra are reported in parts per million (ppm) from tetramethylsilane with the solvent resonance as the internal standard (chloroform: δ 7.26 ppm). Chemical shifts for ^{13}C NMR spectra are reported in parts per million (ppm) from tetramethylsilane with the solvent as the internal standard ($CDCl_3$: δ 77.16 ppm). Data are reported as following: chemical shift, multiplicity (s = singlet, d = doublet, dd = doublet of doublets, t = triplet, q = quartet, m = multiplet, br = broad signal), coupling constant (Hz), and integration.

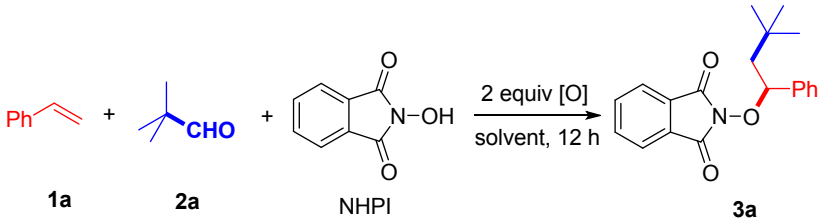
II. General experimental procedures

A general experimental procedure is described as following:

To a solution of NHPI (0.2 mmol, 1 equiv.), styrene (0.6 mmol, 3equiv.) and aldehyde (0.6mmol, 3 equiv.) in mixed solvent of chlorobenzene (0.5 mL) and acetonitrile (0.5 mL) at ambient temperature. DTBP (0.4 mmol, 2.0 equiv.) was added with vigorous stirring. The reaction mixture was stirring at 130 °C (oil bath temperature) for 12 h under air. Afterwards the resulting mixture was cooled to room temperature, transferred to silica gel column directly and purified by column chromatography on silica gel with a mixture of EtOAc/petroleum ether as eluent to afford the pure product.

III. Condition optimization

Table S1: Condition optimization [a]



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

entry	ratio **1a**:**2a**:NHPI	[O] (2 equiv)	Solvent	Temp. (°C)	yield [%][b]
1	1:1:1	DTBP	PhCl	130	15
2	3:3:1	DTBP	PhCl	130	36
3	**3:3:1**	**DTBP**	**PhCl/CH_3CN**	**130**	**80**
4	3:3:1	DTBP	$^{o}C_6H_4Cl_2/CH_3CN$	130	54
5	3:3:1	DTBP	DCE /CH_3CN	130	53

6	3:3:1	DTBP	toluene /CH_3CN	130	51
7	3:3:1	DTBP	benzene /CH_3CN	130	63
8	3:3:1	DTBP	CH_3CN	130	47
9	3:3:1	TBHP	PhCl/CH_3CN	130	55
10	3:3:1	DCP	PhCl/CH_3CN	130	50
11	3:3:1	DTBP	PhCl/CH_3CN	110	36
12	3:3:1	DTBP	PhCl/CH_3CN	150	74

[a] Conditions: **1a** (3 equiv, 0.6 mmol), **2a** (3 equiv, 0.6 mmol), NHPI (0.2 mmol), DTBP (2.0 equiv, 0.4 mmol), solvent (1.0 mL or mixed solvents in 1:1 volume ratio), reacted for 12 h under air unless otherwise noted. [b] Isolated yields.

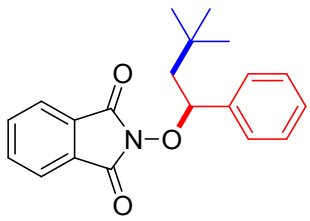
Table S2: Optimization of the ratio of mixed solvent [a]

Entry	**ratio PhCl : CH_3CN**	**Yield (%)[b]**
1	**1:1**	**80**
2	1:4	61
3	2:3	72
4	3:2	74
5	4:1	70

[a] Conditions: **1a** (3 equiv, 0.6 mmol), **2a** (3 equiv, 0.6 mmol), NHPI (0.2 mmol), DTBP (2.0 equiv, 0.4 mmol), solvent (1.0 mL or mixed solvents), reacted for 12 h under air unless otherwise noted. [b] Isolated yields.

IV. Spectra data of products 3a-3p, 4a

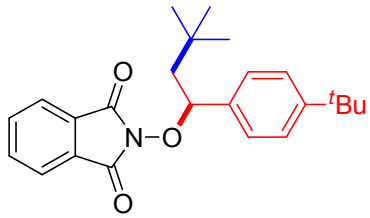
(3a) 2-(3,3-dimethyl-1-phenylbutoxy)isoindoline-1,3-dione



The title compound was prepared according to the general procedure described above by the reaction between 2-hydroxyisoindoline-1,3-dione (**NHPI**) with styrene (**1a**) and pivalaldehyde(**2a**), and purified by flash column chromatography as white solid (51.9 mg, 80%).

M.p. 132-134 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72–7.63 (m, 4H), 7.45-7.43 (m, 2H), 7.29-7.27 (m, 3H), 5.54-5.51 (m, 1H), 2.23-2.18 (m, 1H), 1.88-1.75 (m, 1H), 1.06 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.76, 139.78, 134.27, 128.95, 128.88, 128.34, 128.24, 123.31, 86.96, 48.90, 30.62, 30.14. IR(cm^{-1}): 2933, 1726, 1516, 1466, 1193, 1111. HRMS: calcd. for $[M+Na]^+$ $C_{20}H_{21}NNaO_3$: 346.1419, found: 346.1389.

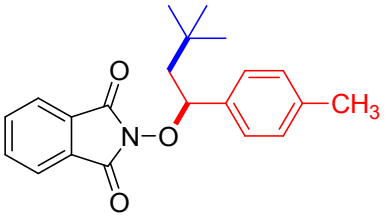
(3b) 2-(1-(4-(tert-butyl)phenyl)-3,3-dimethylbutoxy)isoindoline-1,3-dione



The title compound was prepared according to the general procedure described above by the reaction between 2-hydroxyisoindoline-1,3-dione (**NHPI**) with 1-(tert-butyl)-4-vinylbenzene (**1b**) and pivalaldehyde(**2a**), and purified by flash column chromatography as white solid (66 mg, 87%).

M.p. 136-138 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 – 7.64 (m, 4H), 7.37 – 7.34 (m, 2H), 7.30 – 7.26 (m, 2H), 5.56-5.53 (m, 1H), 2.20-2.14 (m, 1H), 1.78-1.73 (m, 1H), 1.25 (s, 9H), 1.06 (s, 9H).^{13}C NMR (101 MHz, $CDCl_3$) δ 163.89, 151.83, 136.77, 134.24, 129.06, 127.79, 125.24, 123.27, 86.74, 49.09, 34.68, 31.38, 30.64, 30.17. IR(cm^{-1}): 2950, 1779, 1723, 1503, 1463, 1354, 978, 696. HRMS: calcd. for $[M+Na]^+$ $C_{24}H_{29}NNaO_3$: 402.2045, found: 402.2046.

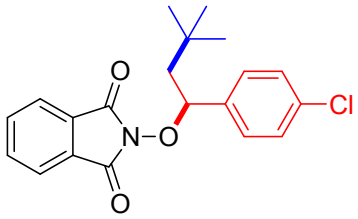
(3c) 2-(3,3-dimethyl-1-(*p*-tolyl)butoxy)isoindoline-1,3-dione



The title compound was prepared according to the general procedure described above by the reaction between 2-hydroxyisoindoline-1,3-dione (**NHPI**) with 1-methyl-4-vinylbenzene (**1c**) and pivalaldehyde(**2a**), and purified by flash column chromatography as white solid (51 mg, 76%).

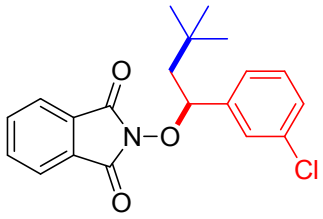
M.p. 101-102 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 – 7.63 (m, 4H), 7.32 (d, J = 8.0 Hz, 2H), 7.08 (d, J = 8.0 Hz, 2H),5.51-5.48(m, 1H), 2.28 (s, 3H), 2.22-2.16 (m, 1H), 1.79-1.74 (m, 1H), 1.05 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.82, 138.74, 136.80, 134.24, 129.04, 128.98, 128.22, 123.31, 86.83, 48.90, 30.59, 30.16, 21.34. IR(cm^{-1}): 2941, 1796, 1723, 1522, 1454, 962. HRMS: calcd.for$[M+Na]^+C_{21}H_{23}NNaO_3$: 360.1576, found: 360.1575.

(3d) 2-(1-(4-chlorophenyl)-3,3-dimethylbutoxy)isoindoline-1,3-dione



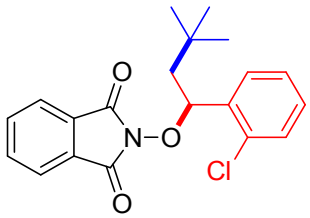
The title compound was prepared according to the general procedure described above by the reaction between 2-hydroxyisoindoline-1,3-dione (**NHPI**) with 1-chloro-4-vinylbenzene (**1d**) and pivalaldehyde(**2a**), and purified by flash column chromatography as white solid (40.2 mg, 56%).
M.p. 138-140 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 – 7.66(m, 4H), 7.40 – 7.37 (m, 2H), 7.27 (s, 1H), 7.25 (s, 1H), 5.52-549 (m, 1H), 2.19-2.13 (m, 1H), 1.73-1.69(m, 1H), 1.05 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.74, 138.49, 134.73, 134.41, 129.57, 128.85, 128.61, 123.43, 86.22, 49.12, 30.62, 30.13. IR(cm^{-1}): 2958, 1779, 1723, 1597, 1488, 1345, 696. HRMS: calcd. for $[M+Na]^+$ $C_{20}H_{20}ClNNaO_3$: 380.1029, found: 380.1027.

(3e) 2-(1-(3-chlorophenyl)-3,3-dimethylbutoxy)isoindoline-1,3-dione



The title compound was prepared according to the general procedure described above by the reaction between 2-hydroxyisoindoline-1,3-dione (**NHPI**) with 1-chloro-3-vinylbenzene (**1e**) and pivalaldehyde(**2a**), and purified by flash column chromatography as white solid (50.5 mg, 71%).
M.p. 147-148 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 – 7.66 (m, 4H), 7.43-7.29 (m, 1H), 7.39 – 7.36 (m, 1H), 7.24-7.23 (m, 2H), 5.49-5.46 (m, 1H), 2.18-2.12 (m, 1H), 1.72-1.67 (m, 1H), 1.07 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.75, 142.17, 134.43, 134.14, 129.73, 129.01, 128.87, 128.24, 126.21, 123.46, 86.33, 49.28, 30.67, 30.12. IR(cm^{-1}): 2958, 1788, 1732, 1572, 1463, 1370, 687. HRMS: calcd. for $[M+Na]^+$ $C_{20}H_{20}ClNNaO_3$: 380.1029, found: 380.1024.

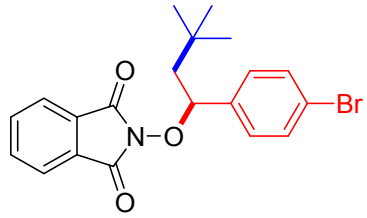
(3f) 2-(1-(2-chlorophenyl)-3,3-dimethylbutoxy)isoindoline-1,3-dione



The title compound was prepared according to the general procedure described above by the reaction between 2-hydroxyisoindoline-1,3-dione (**NHPI**) with 1-chloro-2-vinylbenzene (**1f**) and pivalaldehyde(**2a**), and purified by flash column chromatography as white solid (30 mg, 42%).
M.p. 123-124 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, J = 7.6 Hz, 1H), 7.73 – 7.66 (m, 4H), 7.37 – 7.33 (m, 1H), 7.21 – 7.20 (m, 2H), 6.20-6.17 (m, 1H), 2.14-2.08 (m, 1H), 1.72-1.68 (m, 1H), 1.12 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.78, 137.88, 134.39, 133.47, 129.73, 129.11,

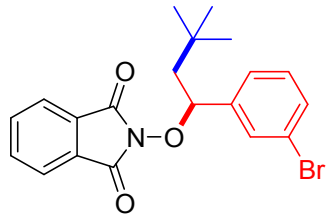
129.01, 127.20, 123.45, 82.61, 49.11, 30.90, 30.14. IR(cm^{-1}): 2950, 1788, 1723, 1480, 1429, 1370, 696. HRMS: calcd. for $[M+Na]^+$ $C_{20}H_{20}ClNNaO_3$: 380.1029, found: 380.1021.

(3g) 2-(1-(4-bromophenyl)-3,3-dimethylbutoxy)isoindoline-1,3-dione



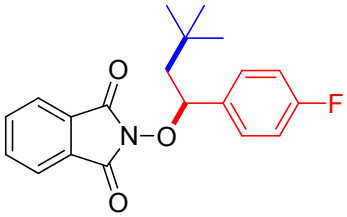
The title compound was prepared according to the general procedure described above by the reaction between 2-hydroxyisoindoline-1,3-dione (**NHPI**) with 1-bromo-4-vinylbenzene (**1g**) and pivalaldehyde(**2a**), and purified by flash column chromatography as white solid (50.5 mg, 63%). M.p. 121-123 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 – 7.66 (m, 4H), 7.44 – 7.40 (m, 2H), 7.34 – 7.32(m, 2H), 5.51-5.48 (m, 1H), 2.18-2.12 (m, 1H), 1.72-1.68 (m, 1H), 1.05 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.77, 139.04, 134.44, 131.58, 129.86, 128.86, 123.46, 122.99, 86.29, 49.17, 30.65, 30.14. IR(cm^{-1}):2958, 1779, 1740, 1597, 1488, 1370, 687. HRMS: calcd. for $[M+Na]^+$ $C_{20}H_{20}BrNNaO_3$: 424.0524, found: 424.0516.

(3h) 2-(1-(3-bromophenyl)-3,3-dimethylbutoxy)isoindoline-1,3-dione



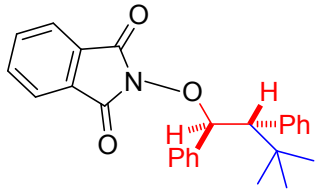
The title compound was prepared according to the general procedure described above by the reaction between 2-hydroxyisoindoline-1,3-dione (**NHPI**) with 1-bromo-3-vinylbenzene (**1h**) and pivalaldehyde(**2a**), and purified by flash column chromatography as white solid (52.3 mg, 65%). M.p. 144-145 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 – 7.66 (m, 4H), 7.58 (t, J = 2.0 Hz, 1H), 7.44 – 7.38 (m, 2H), 7.18 (t, J = 7.8 Hz, 1H), 5.48-5.45 (m, 1H), 2.17-2.11 (m, 1H), 1.72-1.67 (m, 1H), 1.07 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.78, 142.50, 134.45, 131.96, 131.18, 130.05, 128.95, 126.69, 123.49, 122.33, 86.35, 49.38, 30.71, 30.14. IR(cm^{-1}): 2933, 1726, 1559, 1466, 1363, 689.HRMS: calcd. for $[M+Na]^+$ $C_{20}H_{20}BrNNaO_3$: 424.0524, found: 424.0519.

(3i) 2-(1-(4-fluorophenyl)-3,3-dimethylbutoxy)isoindoline-1,3-dione



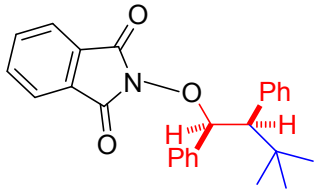
The title compound was prepared according to the general procedure described above by the reaction between 2-hydroxyisoindoline-1,3-dione (**NHPI**) with 1-fluoro-4-vinylbenzene (**1i**) and pivalaldehyde(**2a**), and purified by flash column chromatography as white solid (48.3 mg, 71%).
M.p. 145-146 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 – 7.67 (m, 4H), 7.46-7.42 (m, 2H), 6.99 (t, *J* = 8.8Hz, 2H), 5.53-5.50 (m, 1H), 2.23-2.18 (m, 1H), 1.79-1.74 (m, 1H), 1.07 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.31, 163.78, 161.85, 135.70, 135.67, 134.40, 130.15, 130.07, 128.85, 123.41, 115.45, 115.24, 86.24, 48.96, 30.60, 30.15. IR(cm^{-1}): 2942, 1726, 1516, 1457, 1355, 1136. HRMS: calcd. for $[M+Na]^+$ $C_{20}H_{20}FNNaO_3$: 364.1325, found: 364.1319.

(3j) 2-(trans-3,3-dimethyl-1,2-diphenylbutoxy)isoindoline-1,3-dione



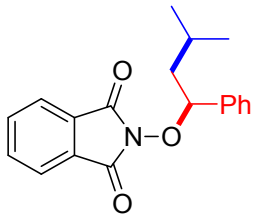
The title compound was prepared according to the general procedure described above by the reaction between 2-hydroxyisoindoline-1,3-dione (**NHPI**) with (E)-1,2-diphenylethene(**1j**) and pivalaldehyde(**2a**), and purified by flash column chromatography as white solid(42mg, 53%, trans:cis=1.1:1).
M.p. 153-154 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 – 7.61 (m, 4H), 7.19 (s, 2H), 7.01 – 6.97(m, 8H), 5.97 (d, *J* = 10.0 Hz, 1H), 3.29 (d, *J* = 10.4 Hz, 1H), 1.21 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.64, 140.93, 137.85, 134.25, 129.50, 128.92, 128.36, 127.52, 126.03, 123.29, 90.51, 60.42, 34.76, 30.05. IR(cm^{-1}): 2921, 1826, 1580, 1516, 1479, 1193, 1046. HRMS: calcd. for $[M+Na]^+$ $C_{26}H_{25}NNaO_3$: 422.1732, found: 422.1727.

(3j') 2-(*cis*-3,3-dimethyl-1,2-diphenylbutoxy)isoindoline-1,3-dione



The title compound was prepared according to the general procedure described above by the reaction between 2-hydroxyisoindoline-1,3-dione (**NHPI**) with *cis*-1,2-diphenylethene (**1j'**) and pivalaldehyde(**2a**), and purified by flash column chromatography as white solid(48 mg, 60%, trans:cis=3:1).
M.p. 153-154 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 – 7.61 (m, 4H), 7.21 (m, 5H), 7.16 – 7.10 (m, 5H), 6.18 (d, *J* = 4.8Hz, 1H), 2.83 (d, *J* = 4.8 Hz, 1H), 1.05 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.82, 138.71, 138.29, 134.24, 129.05, 128.69, 128.53, 127.71, 127.47, 126.64, 123.24, 88.41, 62.93, 34.88, 29.66. IR(cm^{-1}): 2921, 1826, 1580, 1516, 1479, 1193, 1046. HRMS: calcd. for $[M+Na]^+$ $C_{26}H_{25}NNaO_3$: 422.1732, found: 422.1727.

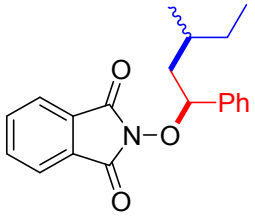
(3k) 2-(3-methyl-1-phenylbutoxy)isoindoline-1,3-dione



The title compound was prepared according to the general procedure described above by the reaction between 2-hydroxyisoindoline-1,3-dione (**NHPI**) with styrene (**1a**) and isobutyraldehyde(**2b**), and purified by flash column chromatography as a colorless oil (45.2 mg, 74%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 – 7.65 (m, 4H), 7.47-7.44 (m, 2H), 7.34-7.29 (m, 3H), 5.44-5.41 (m, 1H), 2.15 – 2.08 (m, 1H), 1.81-1.72 (m, 2H), 1.04 (d, J = 6.4 Hz, 3H), 0.98 (d, J = 6.4 Hz, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 163.84, 138.62, 134.32, 129.08, 128.96, 128.40, 128.26, 123.39, 87.78, 44.11, 25.08, 22.83, 22.69. IR(cm^{-1}): 2941, 1796, 1732, 1589, 1496, 1370, 1180.HRMS: calcd. for $[M+Na]^+$ $C_{19}H_{19}NNaO_3$: 332.1263, found: 332.1263.

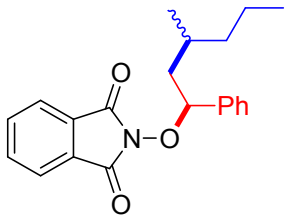
(3l) 2-((3-methyl-1-phenylpentyl)oxy)isoindoline-1,3-dione [1]



The title compound was prepared according to the general procedure described above by the reaction between 2-hydroxyisoindoline-1,3-dione (**NHPI**) with styrene (**1a**) and 2-methylbutanal(**2c**), and purified by flash column chromatography as a colorless oil (47.0 mg, 73%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 – 7.64 (m, 4H), 7.47-7.45(m, 2H), 7.31 – 7.29 (m, 3H), 5.47-5.44 (m, 1H), 2.27 – 2.20 (m, 1H), 1.78 – 1.70 (m, 1H), 1.59 – 1.58 (m, 1H), 1.45 – 1.40 (m, 1H), 1.29-1.21 (m, 1H), 1.05 (d, J = 6.4 Hz, 3H), 0.90 (t, J = 7.6 Hz, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 163.85, 139.00, 134.33, 129.03, 128.94, 128.38, 128.16, 123.38, 87.43, 42.51, 31.18, 29.78, 19.10, 11.27. IR(cm^{-1}): 2966, 1796, 1732, 1496, 1454, 1362, 1180. HRMS: calcd. for $[M+Na]^+$ $C_{20}H_{21}NNaO_3$: 346.1419, found: 346.1385.

(4m) 2-((3-methyl-1-phenylhexyl)oxy)isoindoline-1,3-dione[1]

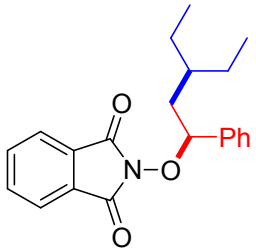


The title compound was prepared according to the general procedure described above by the

[1] Diastereoselectivities were not determined.

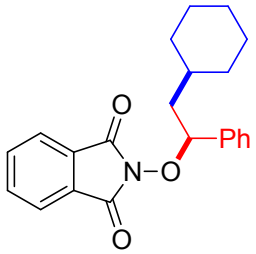
reaction between 2-hydroxyisoindoline-1,3-dione (**NHPI**) with styrene (**1a**) and 2-methylpentanal (**2d**), and purified by flash column chromatography as a colorless oil (45.5 mg, 68%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72-7.65 (m, 4H), 7.46 – 7.44 (m, 2H), 7.33-7.30 (m, 3H), 5.43 (t, *J* = 7.4 Hz, 1H), 2.03 – 1.92 (m, 2H), 1.58 – 1.42 (m, 2H), 1.33 – 1.29 (m, 2H), 1.19 – 1.14 (m, 1H), 0.95 (d, *J* = 6.6 Hz, 3H), 0.87 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.85, 138.34, 134.32, 129.13, 128.96, 128.41, 128.38, 128.28, 128.03, 126.33, 123.40, 123.38, 88.01, 87.37, 41.81, 39.40, 29.52, 20.41, 19.95, 19.86, 14.38. IR(cm^{-1}): 2950, 1736, 1504, 1454, 1336. HRMS: calcd. for $[M+Na]^+$ $C_{21}H_{23}NNaO_3$: 360.1576, found: 360.1578.

(3n) 2-((3-ethyl-1-phenylpentyl)oxy)isoindoline-1,3-dione



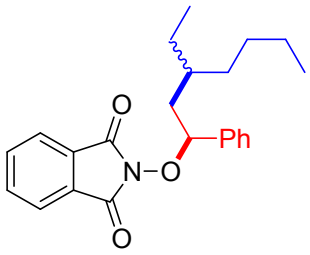
The title compound was prepared according to the general procedure described above by the reaction between 2-hydroxyisoindoline-1,3-dione (**NHPI**) with styrene (**1a**) and 2-ethylbutanal (**2e**), and purified by flash column chromatography as a colorless oil (45.2 mg, 71%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 – 7.64 (m, 4H), 7.47-7.45 (m, 2H), 7.31 – 7.30 (m, 3H), 5.42(t, *J* = 7.0 Hz, 1H), 2.17 – 2.10 (m, 1H), 1.85 – 1.80 (m, 1H), 1.48 – 1.37 (m, 5H), 0.90 – 0.85 (m, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.84, 138.79, 134.32, 129.05, 128.96, 128.38, 128.26, 123.38, 87.85, 38.61, 36.90, 25.53, 25.28, 10.68, 10.60. IR(cm^{-1}): 2958, 1796, 1732, 1505, 1471, 1370, 1172. HRMS: calcd. for $[M+Na]^+$ $C_{21}H_{23}NNaO_3$: 360.1576, found: 360.1571.

(3o) 2-(2-cyclohexyl-1-phenylethoxy)isoindoline-1,3-dione



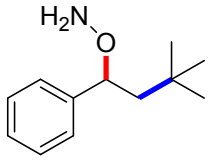
The title compound was prepared according to the general procedure described above by the reaction between 2-hydroxyisoindoline-1,3-dione (**NHPI**) with styrene (**1a**) and 2-ethylhexanal (**2f**), and purified by flash column chromatography as white solid (43.2 mg, 62%).
M.p. 133-135 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 – 7.65 (m, 4H), 7.46 – 7.44 (m, 2H), 7.32 – 7.29 (m, 3H), 5.48-5.45 (m, 1H), 2.15-2.07 (m, 1H), 1.95 (d, *J* = 12.8 Hz, 1H), 1.81 – 1.71 (m, 5H), 1.54 – 1.49 (m, 1H), 1.27 – 1.17(m, 3H), 1.05 – 0.96 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.85, 138.74, 134.32, 129.05, 128.95, 128.39, 128.23, 123.39, 87.20, 42.82, 34.34, 33.61, 33.24, 26.64, 26.28. IR(cm^{-1}): 2916, 2849, 1796, 1715, 1505, 1463, 1446, 1362, 1180. HRMS: calcd. for $[M+Na]^+$ $C_{22}H_{23}NNaO_3$: 372.1576, found: 372.1598.

(3p) 2-((3-ethyl-1-phenylheptyl)oxy)isoindoline-1,3-dione[1]



The title compound was prepared according to the general procedure described above by the reaction between 2-hydroxyisoindoline-1,3-dione (**NHPI**) with styrene (**1a**) and 2-ethylhexanal (**2g**), and purified by flash column chromatography as a colorless oil (45.2 mg, 61%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 – 7.65 (m, 4H), 7.47-7.44 (m, 2H), 7.30 (d, *J* = 6.0 Hz, 3H), 5.42 (t, *J* = 7.0 Hz, 1H), 2.13– 2.12 (m, 1H), 1.84-1.78 (m, 1H), 1.50-1.44 (m, 2H), 1.40 – 1.37 (m, 2H), 1.27 (s, 5H), 0.89 – 0.85 (m, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.85, 138.82, 138.73, 134.31, 129.05, 128.97, 128.38, 128.30, 128.26, 123.38, 87.89, 87.84, 39.08, 38.94, 35.50, 32.89, 32.67, 28.72, 28.60, 26.02, 25.75, 23.17, 23.13, 14.26, 14.25, 10.61, 10.54. IR(cm^{-1}): 2950, 2933, 1788, 1732, 1597, 1496, 1345, 1188. HRMS: calcd. for $[M+Na]^+$ $C_{23}H_{27}NNaO_3$: 388.1889, found: 388.1864.

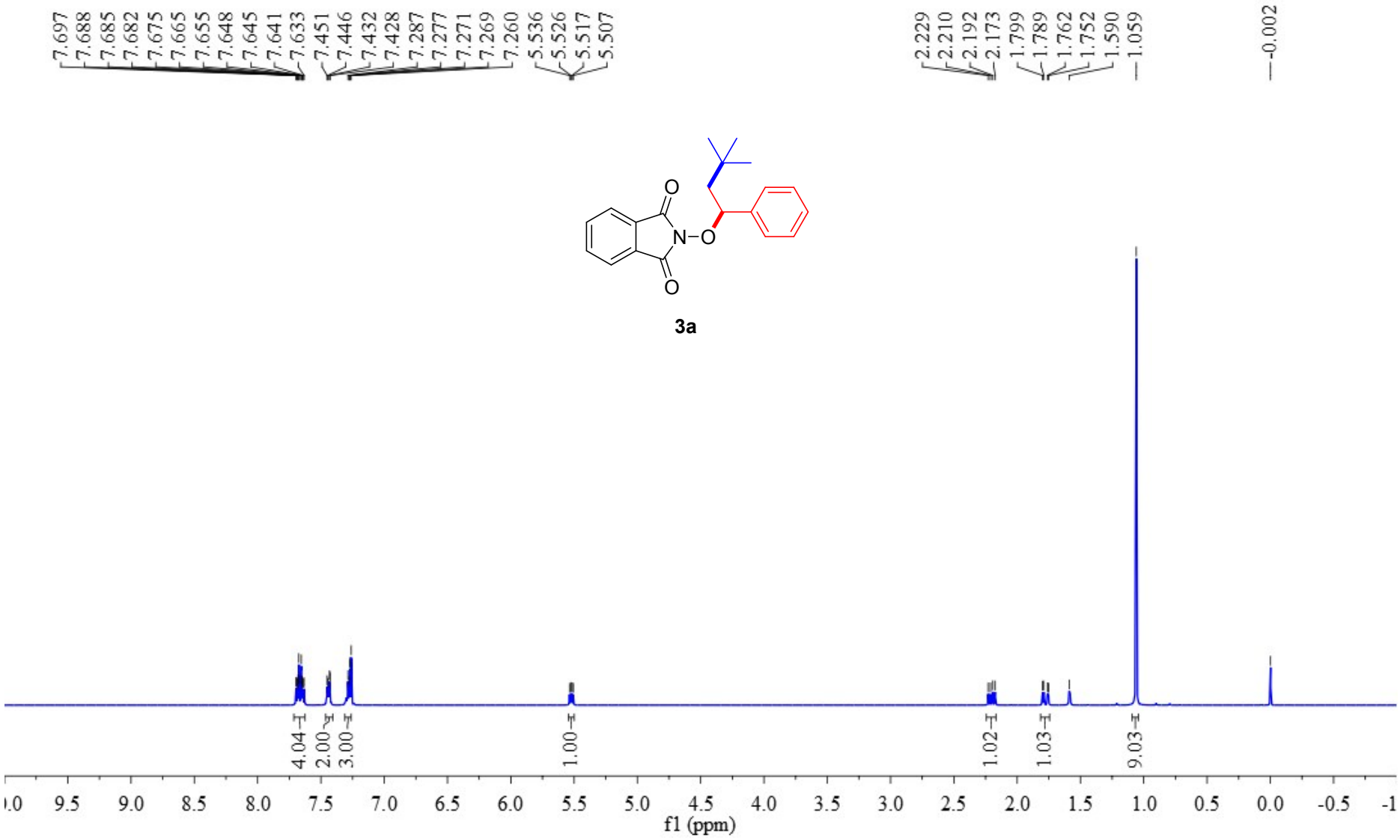
(4a) O-(3,3-dimethyl-1-phenylbutyl)hydroxylamine



To a solution of **3a** (64.6mg, 0.2 mmol) in 10% MeOH in $CHCl_3$ (2.0 mL) was added hydrazine monohydrate (0.03 mL, 0.6 mm), and the reaction mixture was stirred at room temperature. Upon completion (TLC monitoring, 16 h), a white precipitate appeared in a colorless reaction solution. The reaction mixture was passed through a short plug of silica gel washing with 30% EtOAc in hexane (30 mL). The filtrate was concentrated, and the residue was taken up in hexane (10 mL). The solution was acidified to pH=3 with H_2SO_4 in ether/hexane (1:3). A white precipitate formed and was separated by filtration washing with ether/hexane (1:3, 10 mL). The collected white precipitate was partitioned between ether (5 mL) and aqueous saturated K_2CO_3 solution (5 mL), and the aqueous phase was extracted with additional portions of ether (5 mL x 3). The combined organic layers were dried and concentrated to give O-(3,3-dimethyl-1-phenylbutyl)hydroxylamine as a colorless oil (29.7 mg, 77%);

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.28 (m, 5H), 5.13 (s, 2H), 4.62-4.59 (m, 1H), 1.79-1.74 (m, 1H), 1.48-1.44 (m, 1H), 0.97 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.75, 128.70, 127.66, 126.73, 85.56, 50.28, 30.59, 30.26. IR(cm^{-1}): 2949, 1581, 1464, 1377. HRMS: calcd. for $[M+H]^+$ $C_{12}H_{20}NO$: 194.1545, found: 194.1539.

VI. Copies of 1H and 13C NMR spectra of products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7.446
7.432
7.428
7.287
7.277
7.271
7.269
7.260
5.536
5.526
5.517
5.507
2.229
2.210
2.192
2.173
1.799
1.789
1.762
1.752
1.590
1.059
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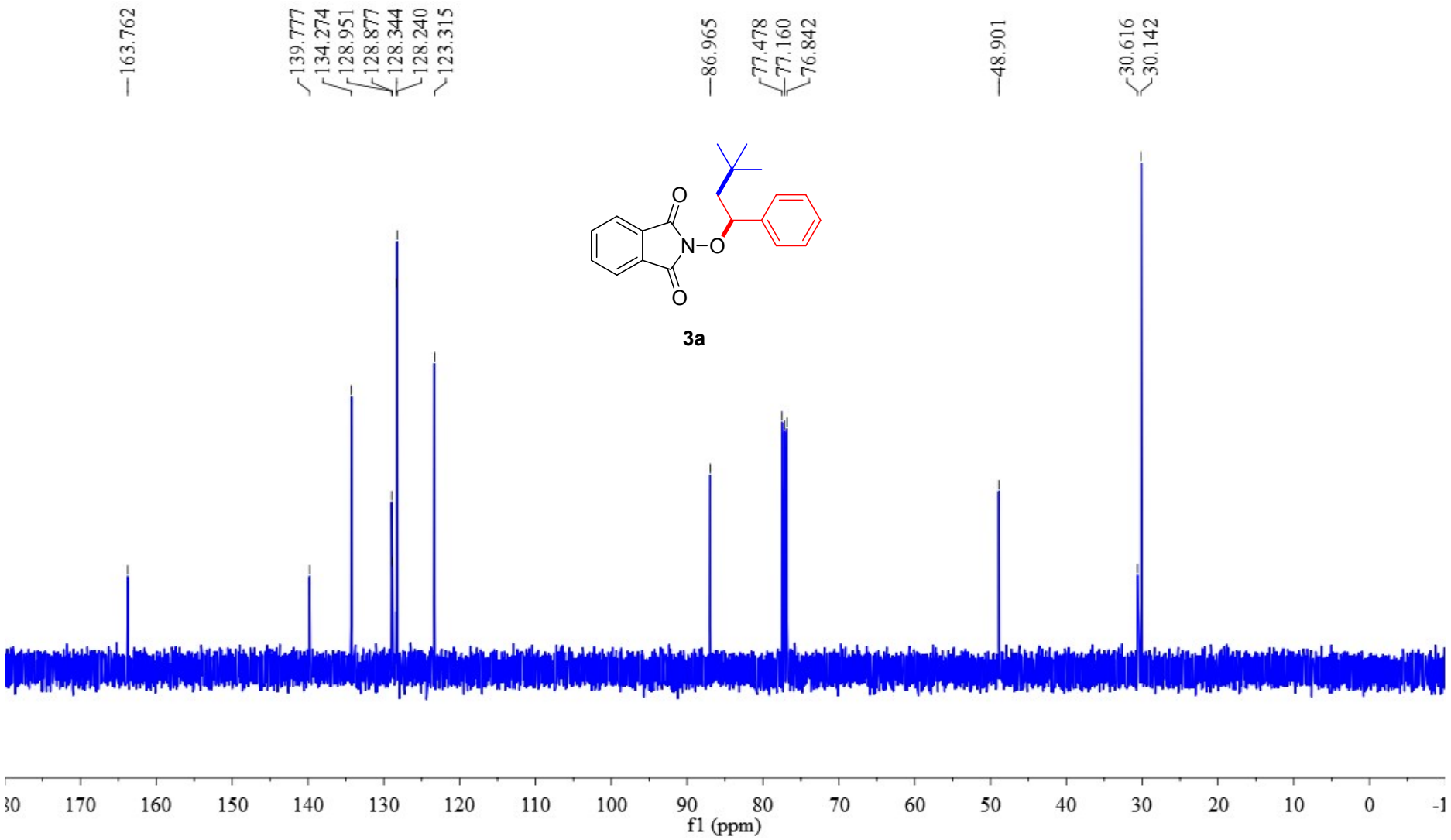
O
3a
4.04
2.00
3.00
1.00
1.02
1.03
9.03
f1 (ppm)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128.240
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77.160
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48.901
30.616
30.142
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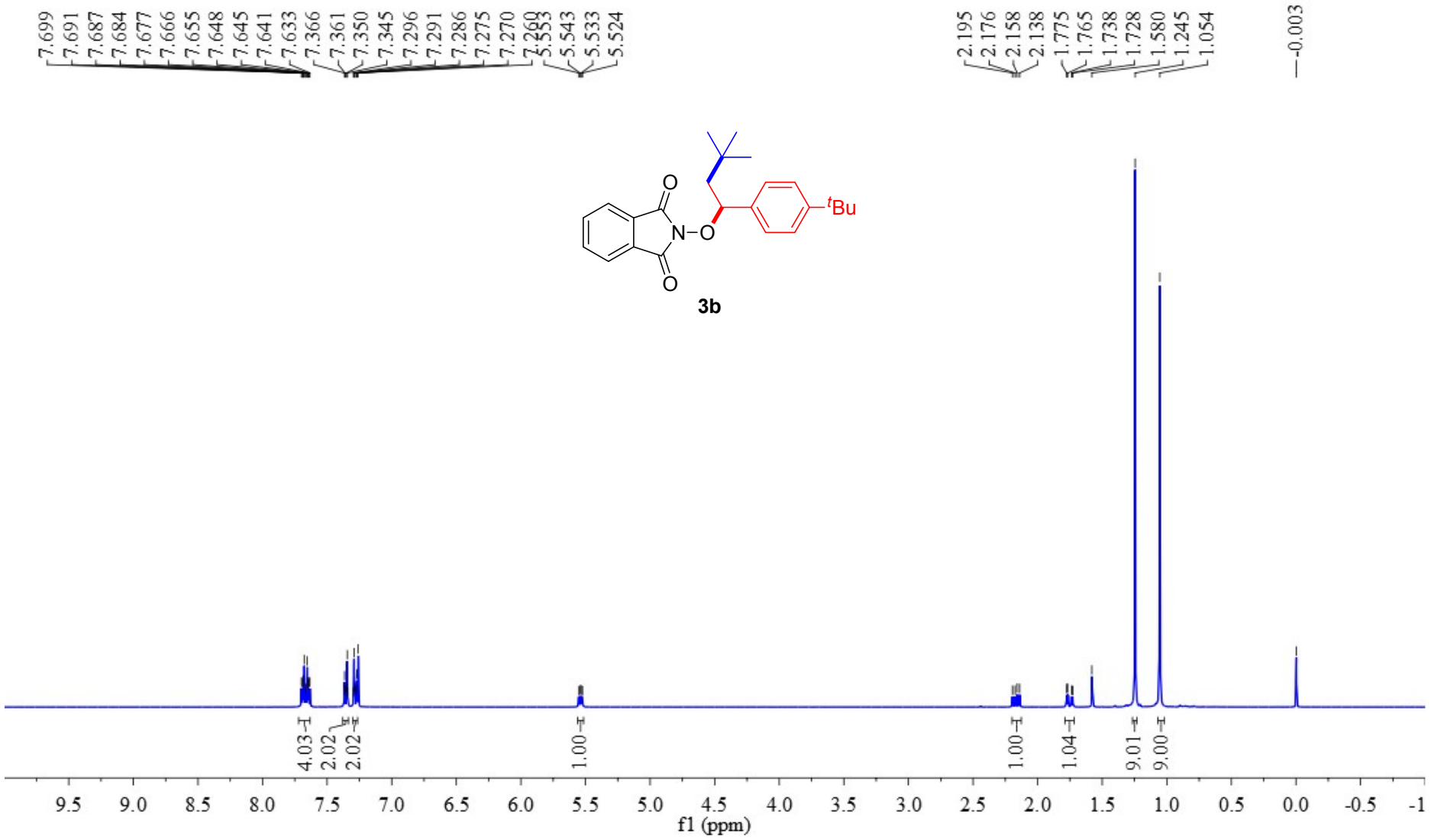
N–O
O
3a
f1 (ppm)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7.648
7.645
7.641
7.633
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7.350
7.345
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7.286
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5.524
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2.158
2.138
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1.765
1.738
1.728
1.580
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N–O
O
tBu
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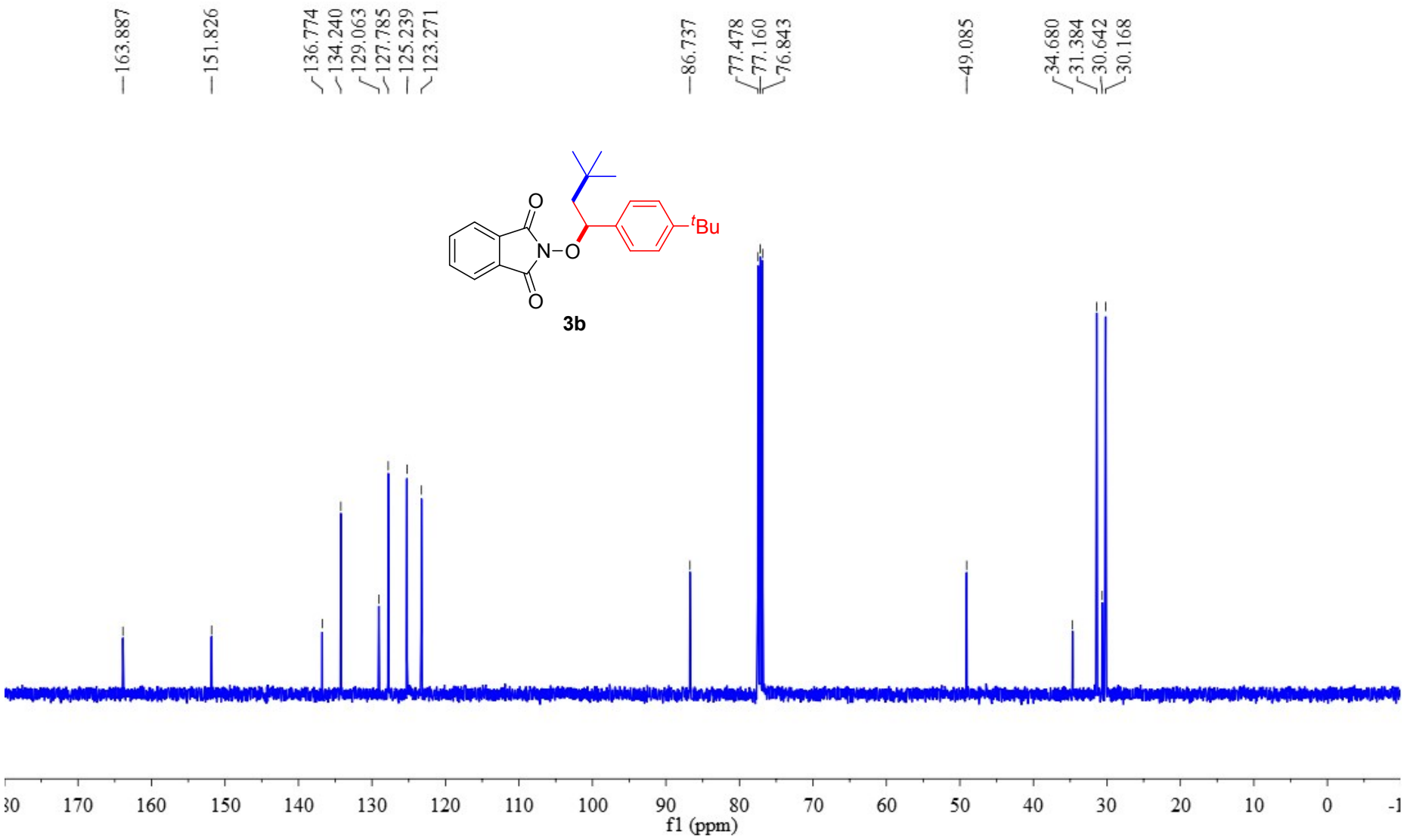
2.02
1.00
1.00
1.04
9.01
9.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)

3b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49.085
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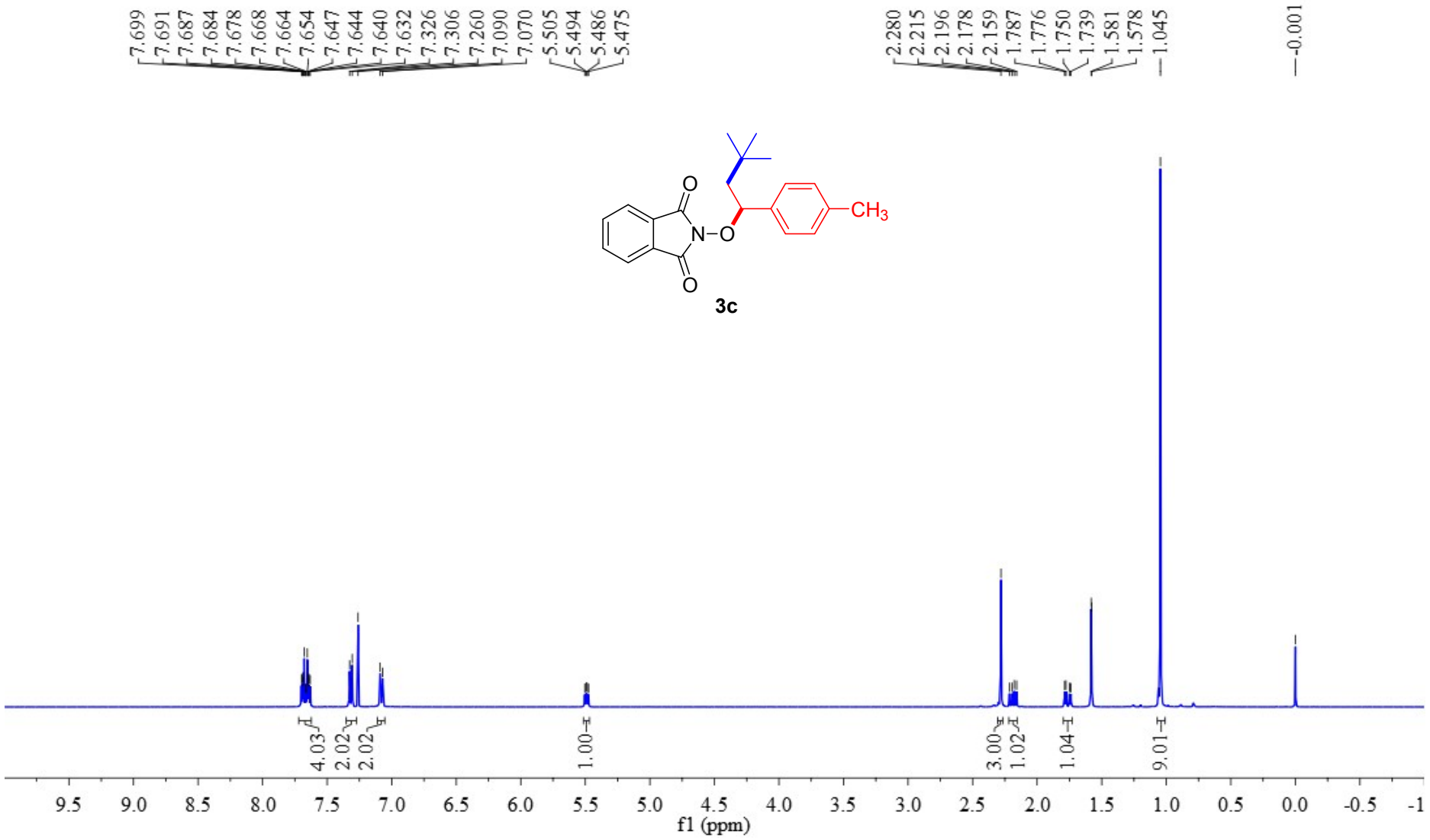
31.384
30.642
30.168
O
N–O
O
tBu
3b
f1 (ppm)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7.306
7.260
7.090
7.070
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5.494
5.486
5.475
2.280
2.215
2.196
2.178
2.159
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3c
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2.02
2.02
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3.00
1.02
1.04
9.01
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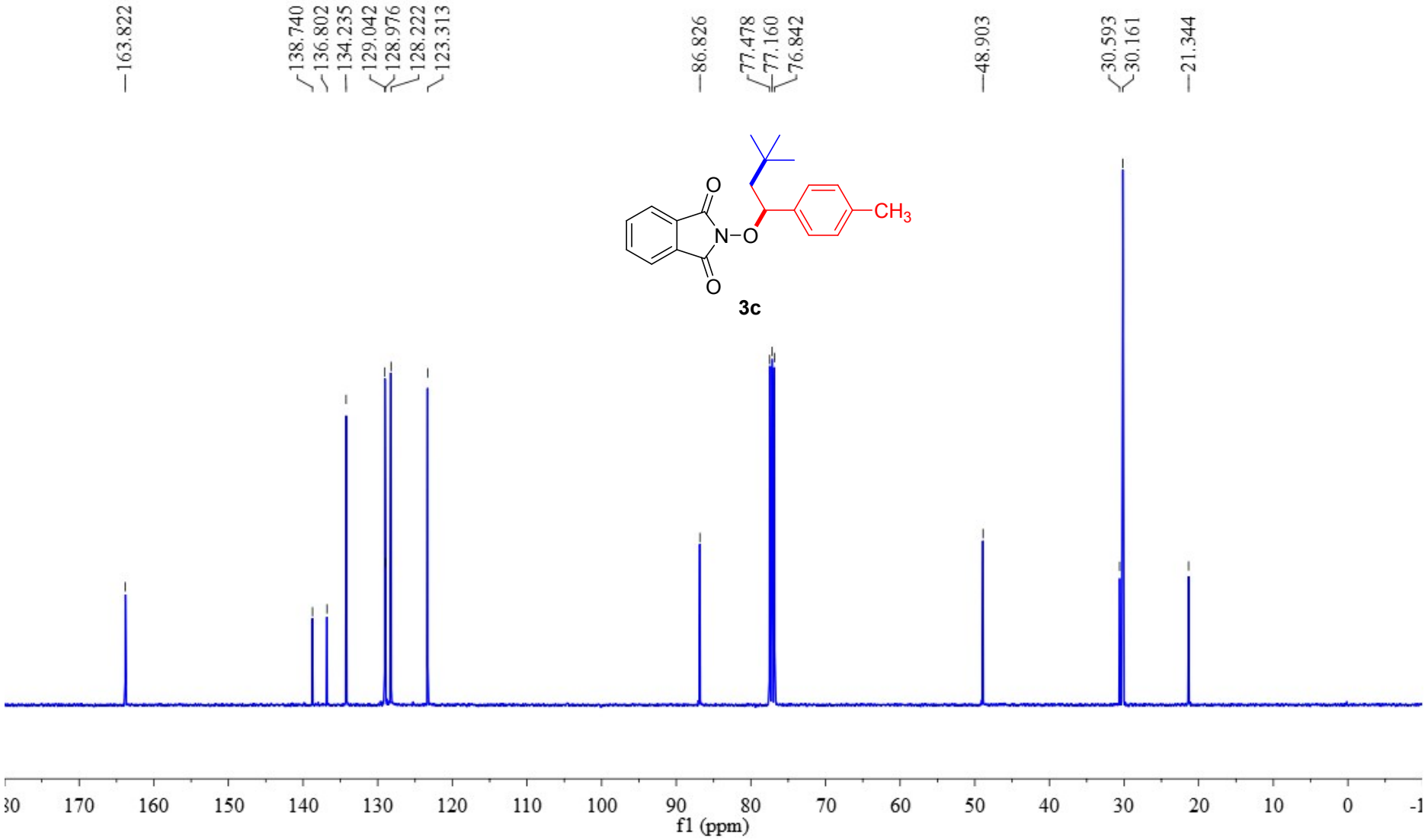
f1 (ppm)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30.161
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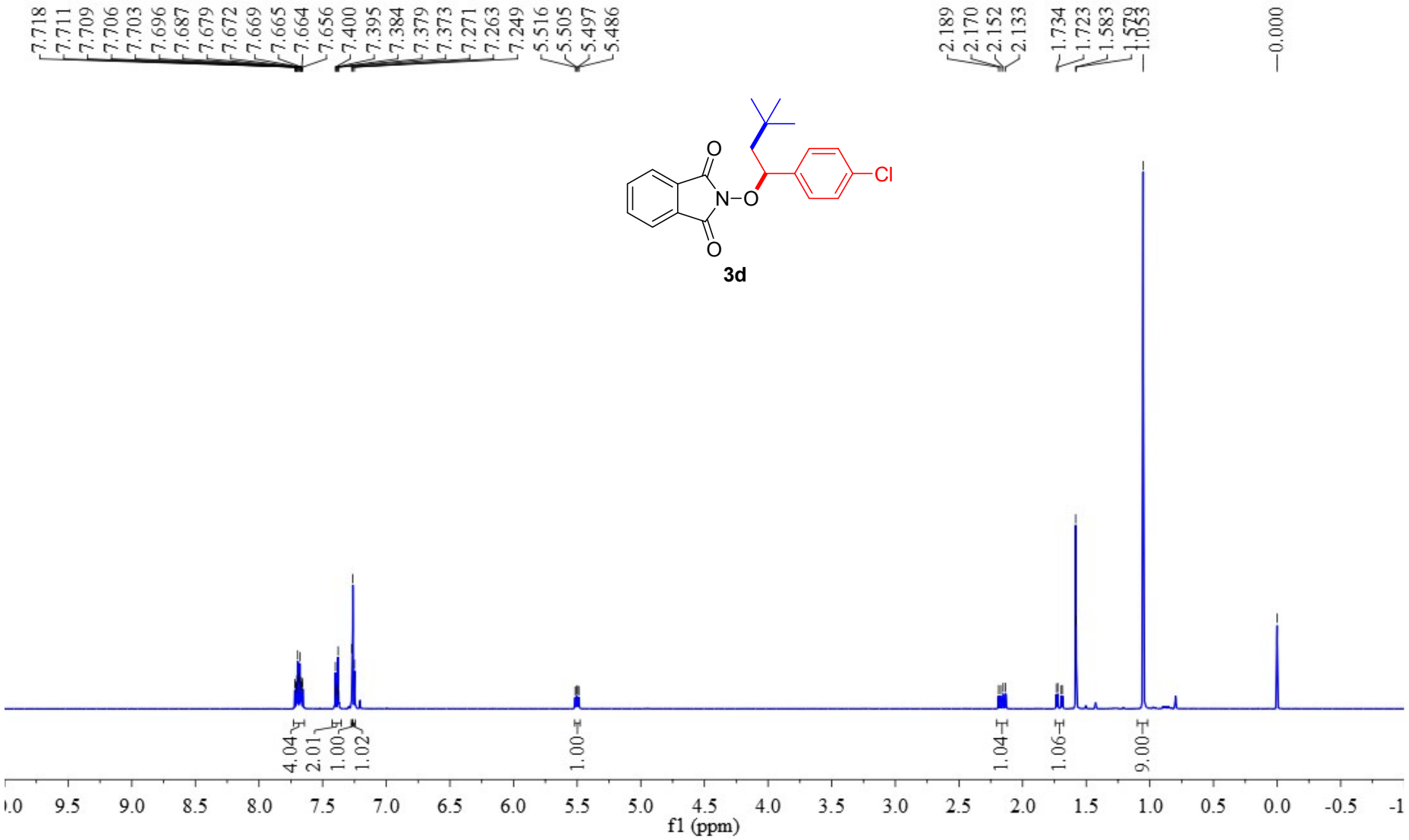
O
N–O
O
CH3
3c
f1 (ppm)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7.656
7.400
7.395
7.384
7.379
7.373
7.271
7.263
7.249
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5.505
5.497
5.486
2.189
2.170
2.152
2.133
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1.723
1.583
1.573
1.053
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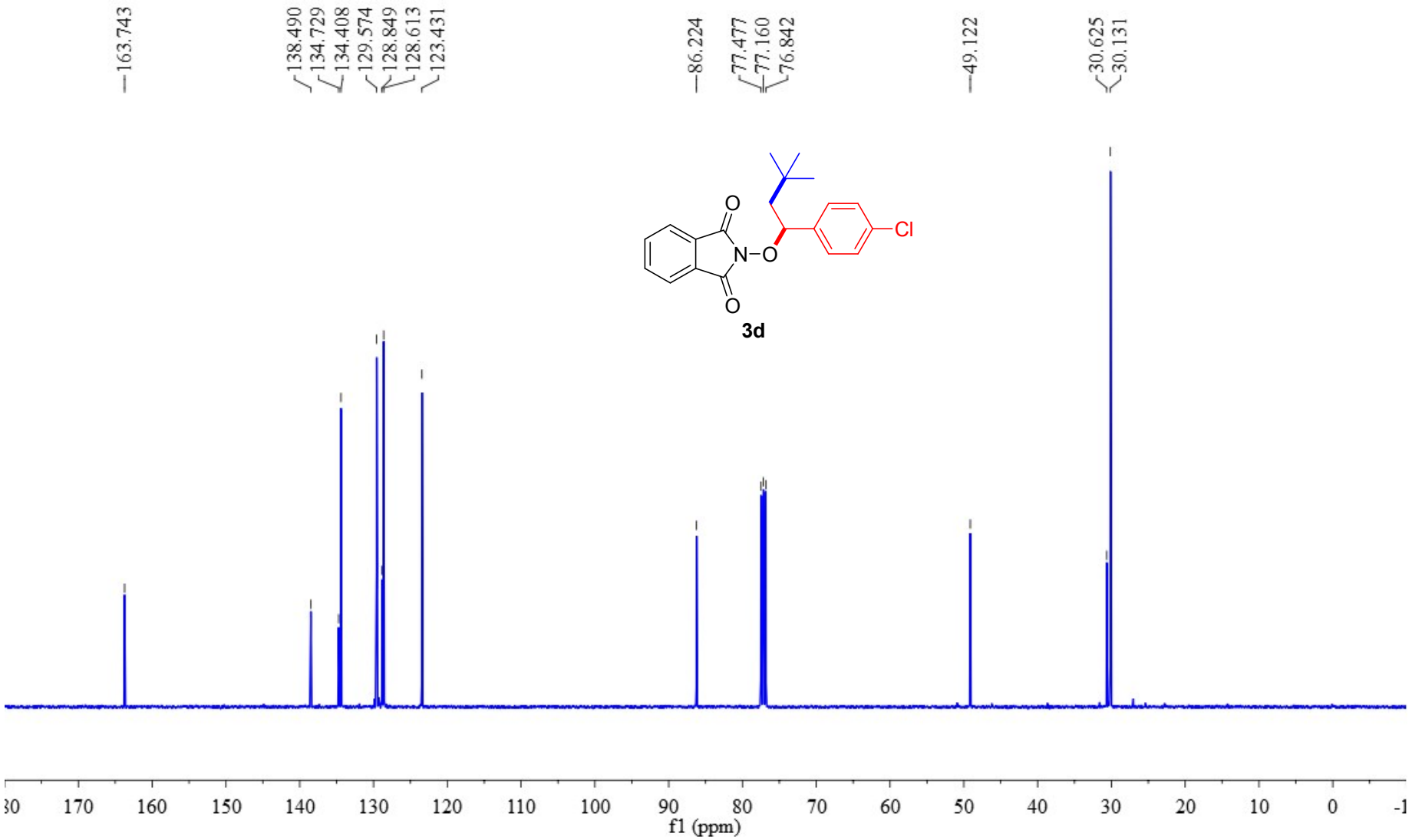
f1 (ppm)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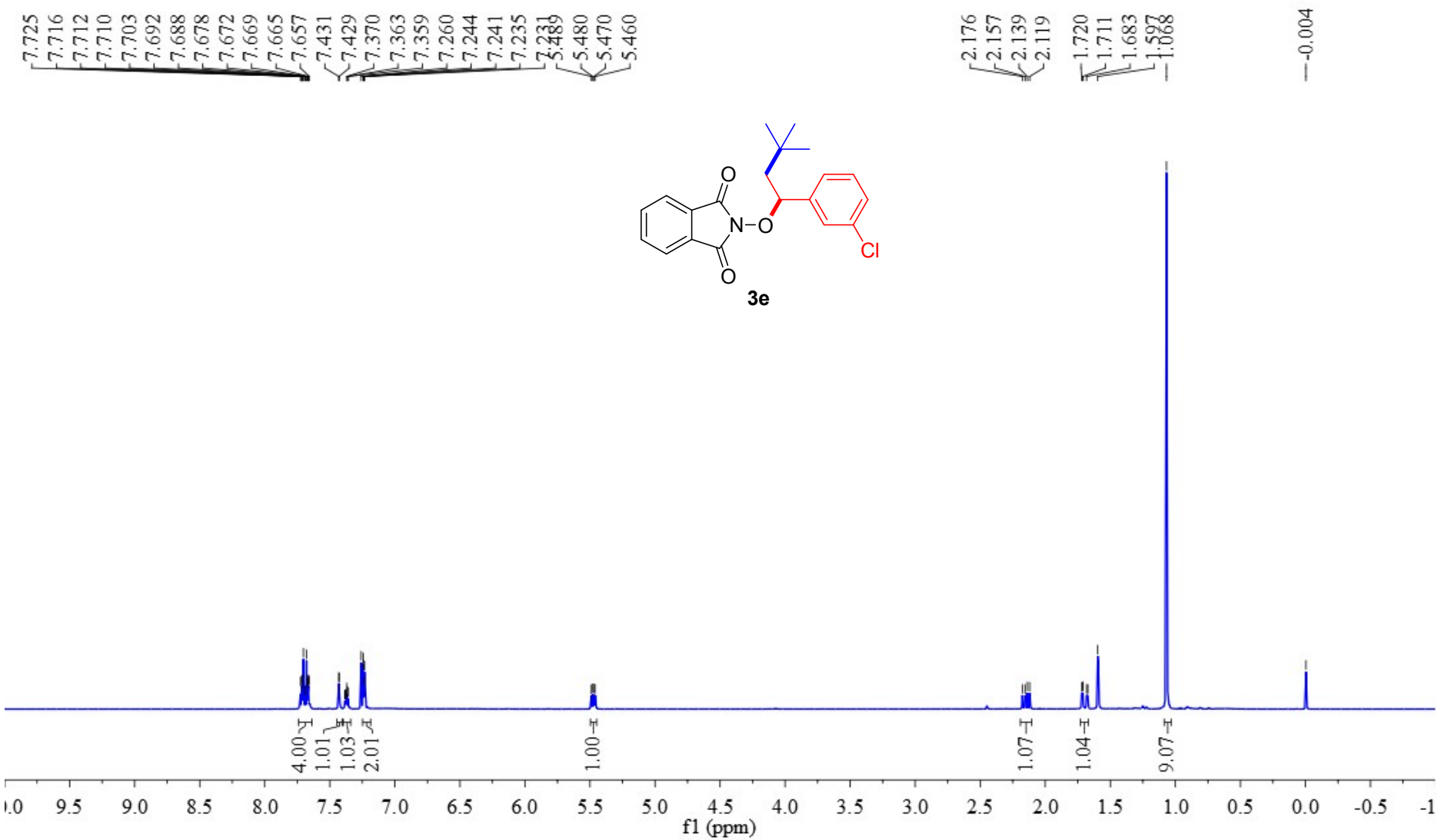
f1 (ppm)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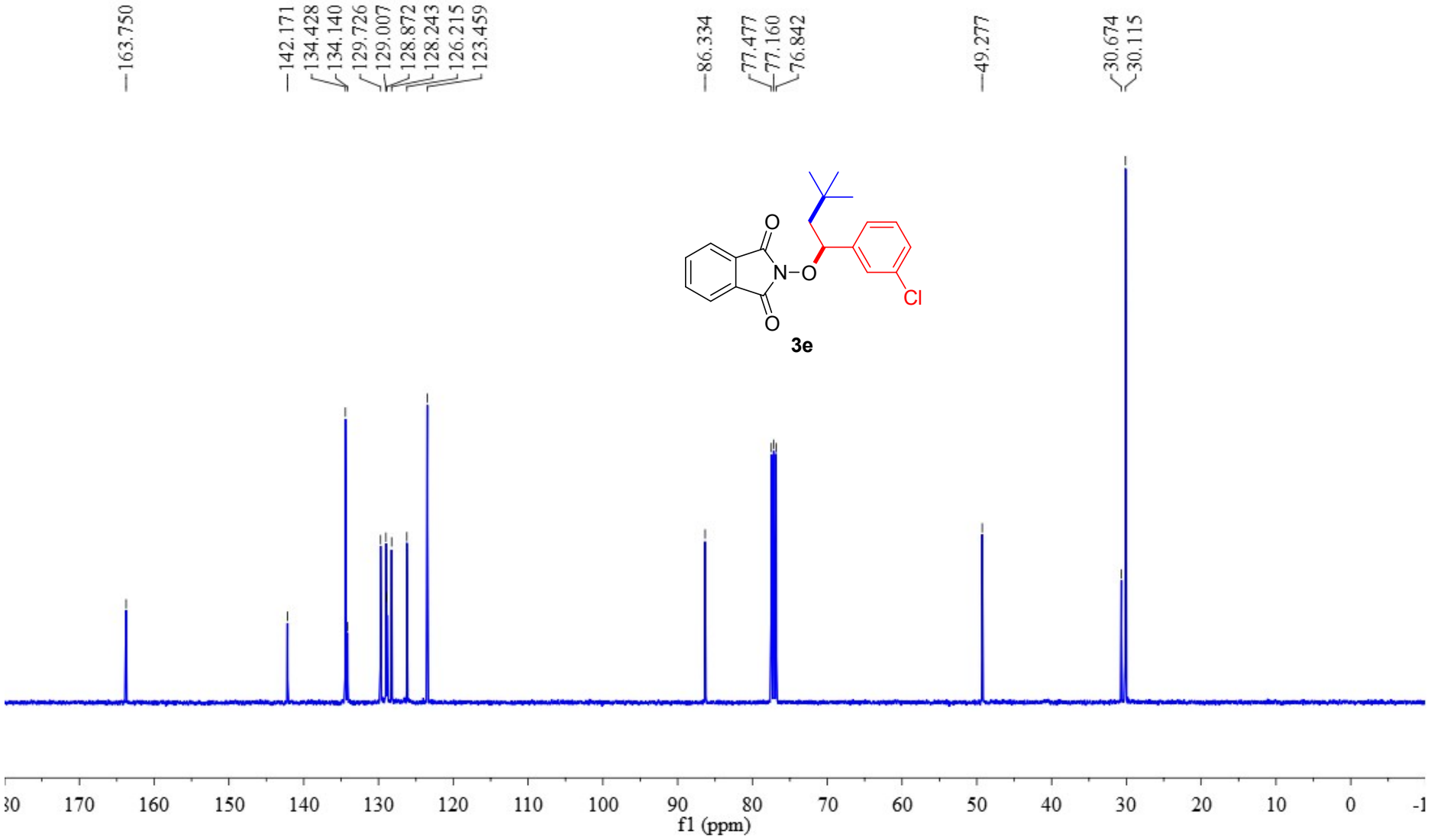
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Cl
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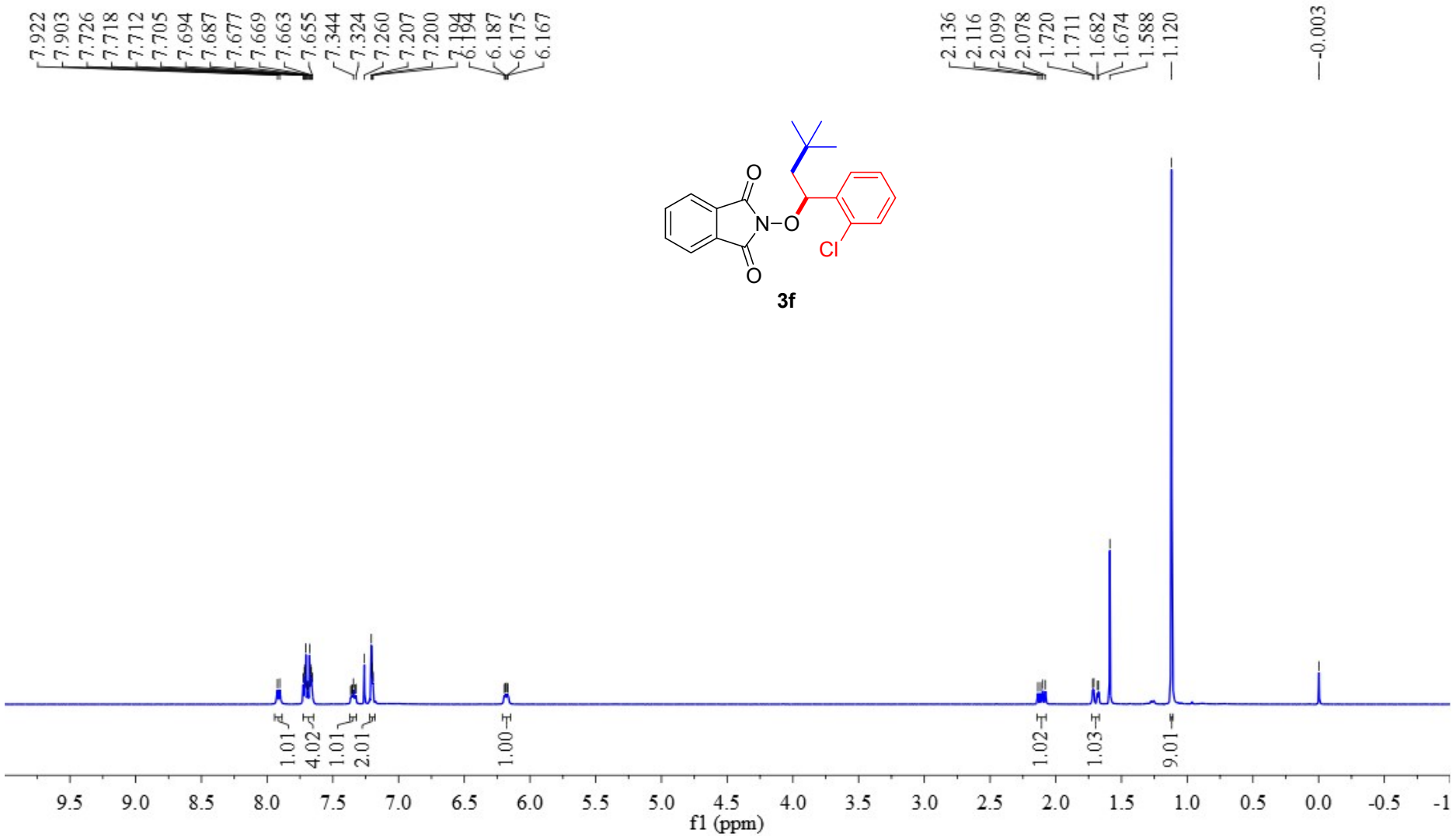
f1 (ppm)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6.167
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2.01
1.00
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1.03
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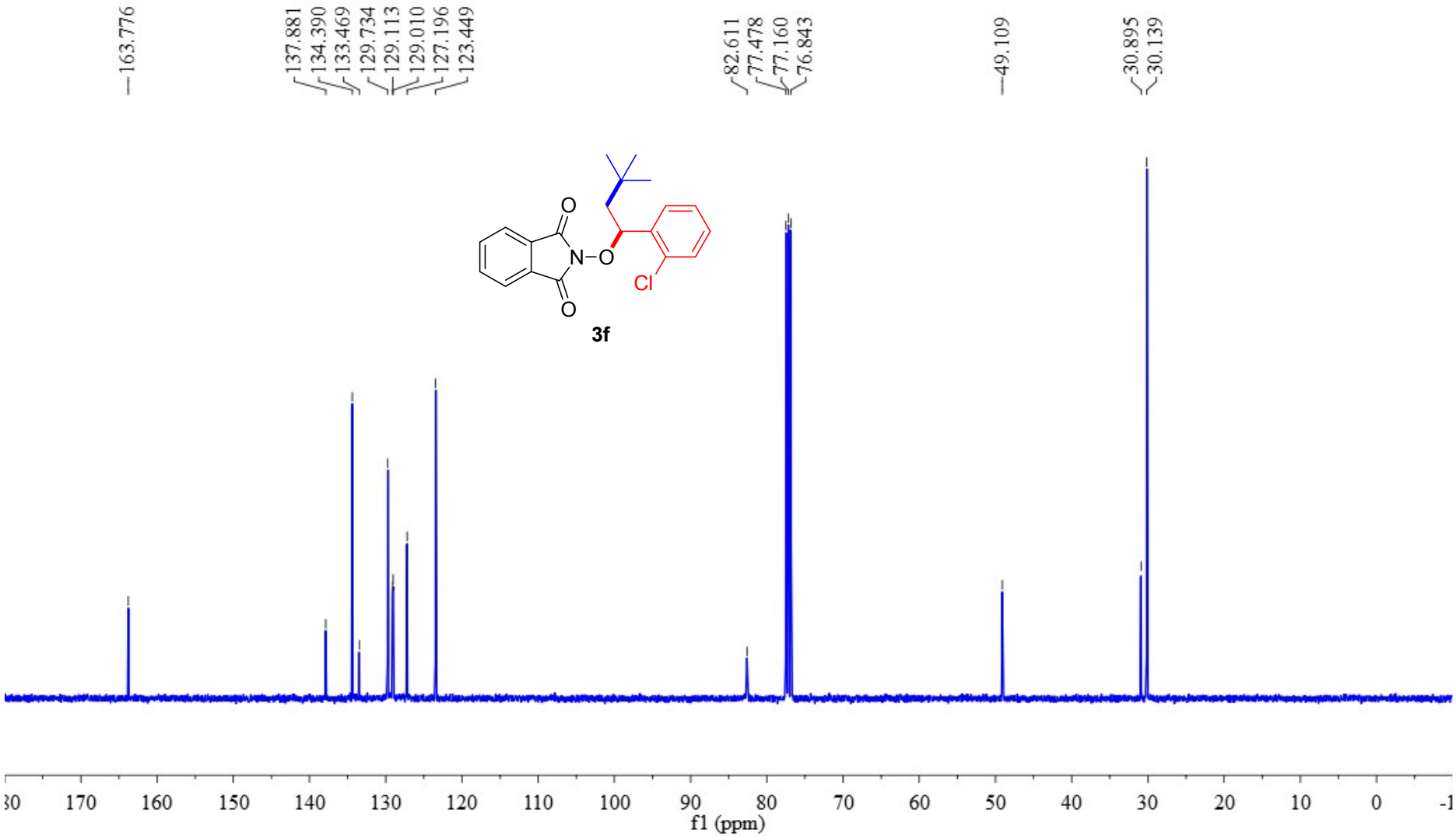
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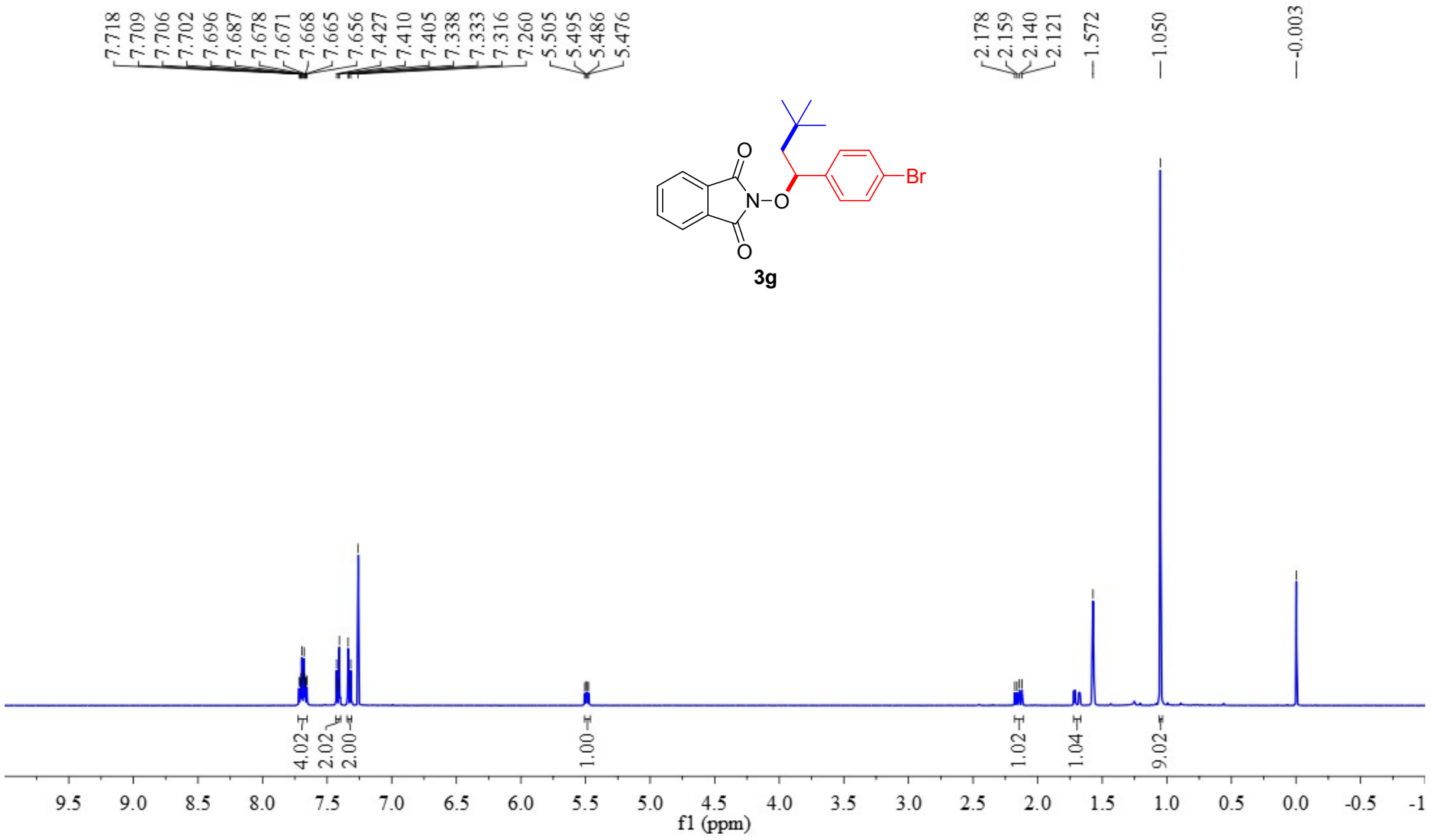
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7.260
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1.00
1.02
1.04
9.02
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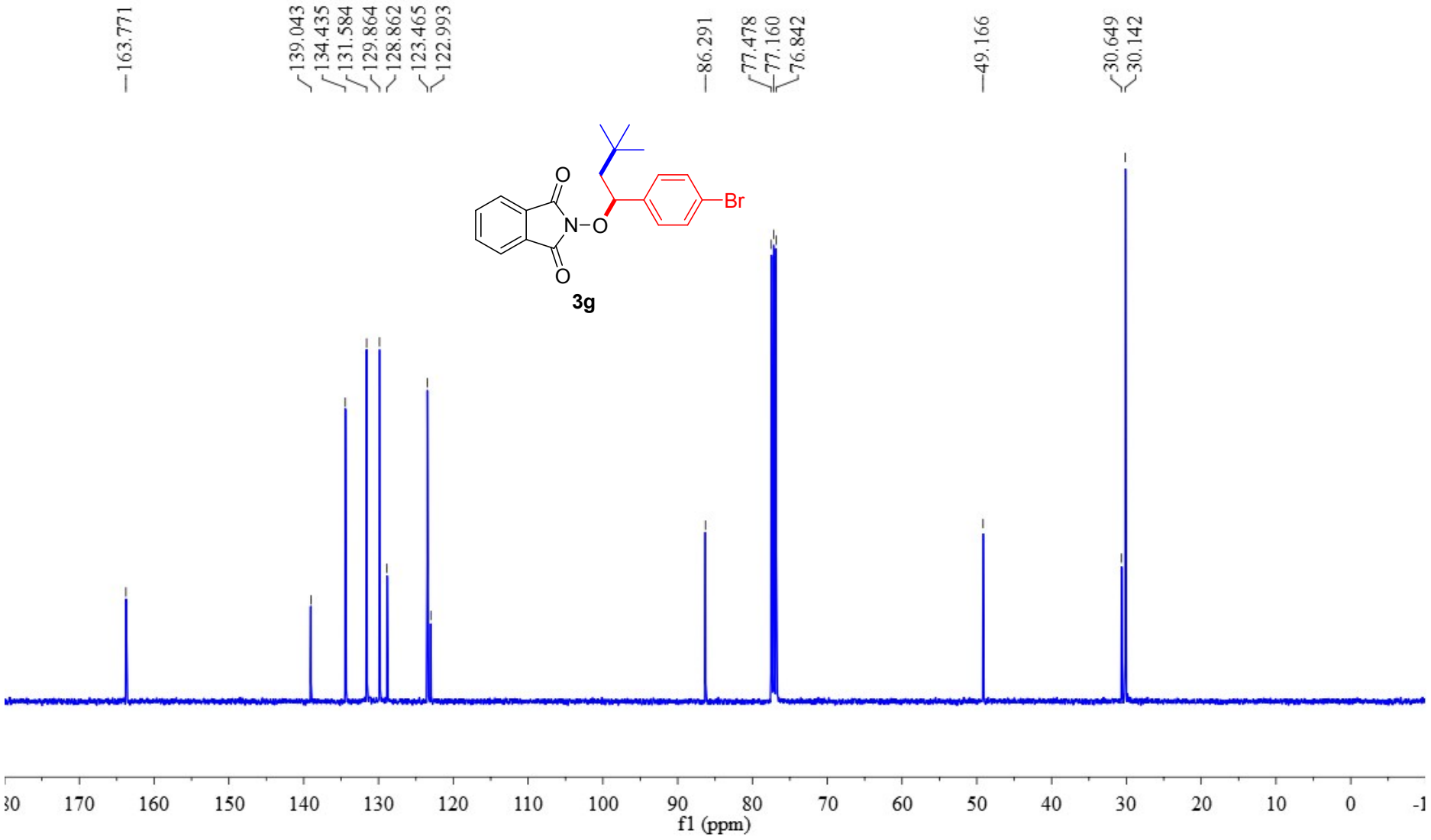
f1 (ppm)

3g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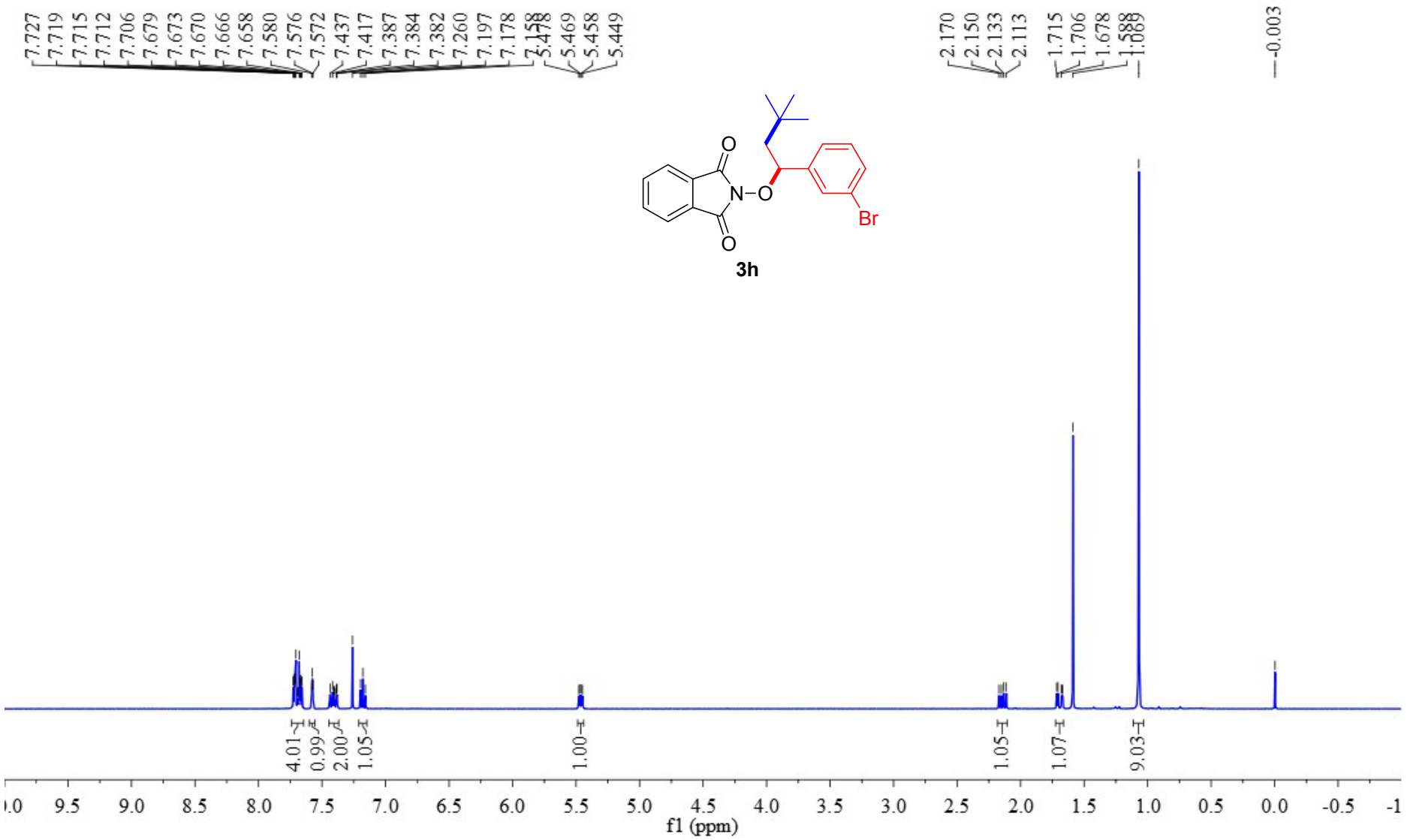
3g
f1 (ppm)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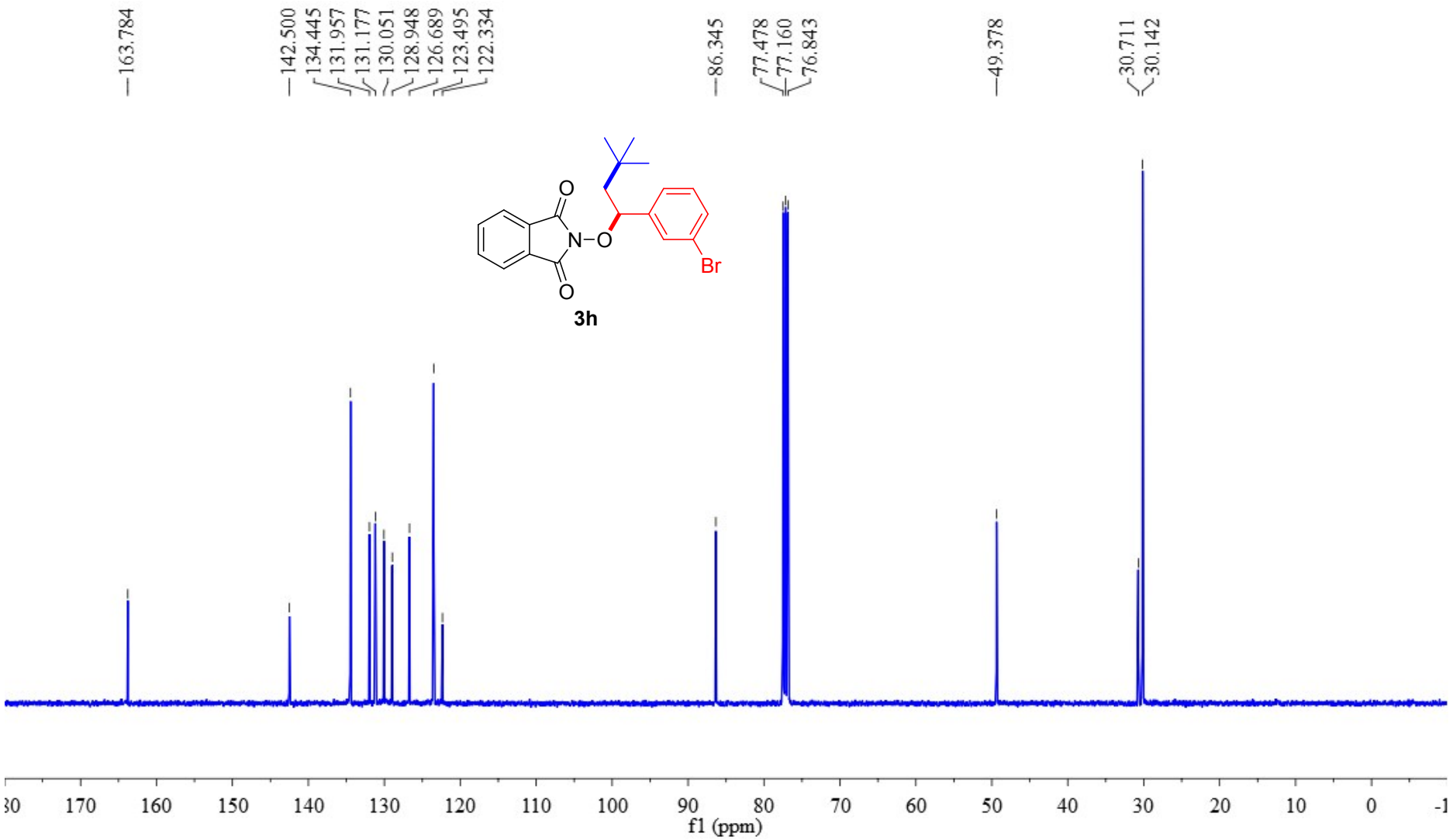
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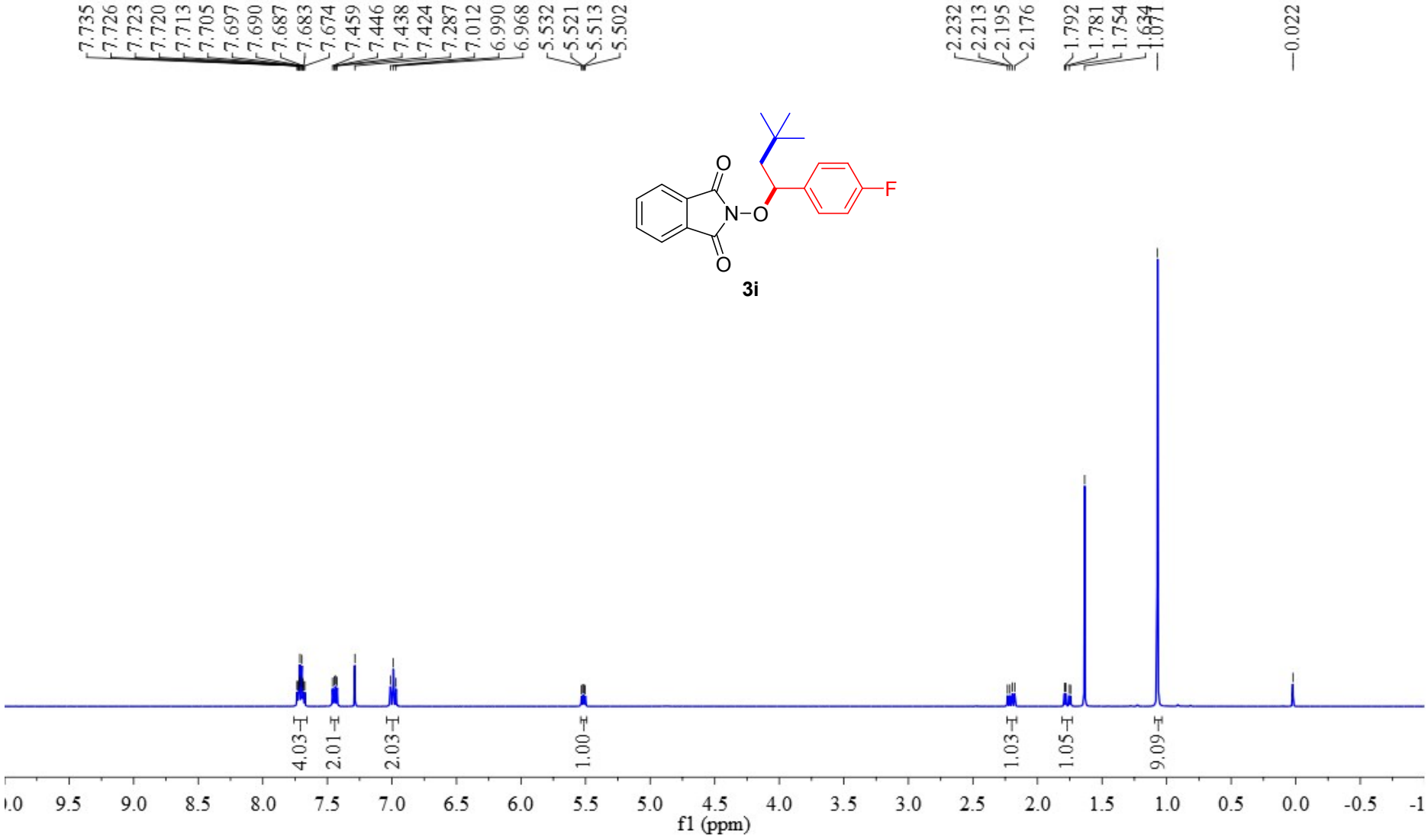
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2.213
2.195
2.176
1.792
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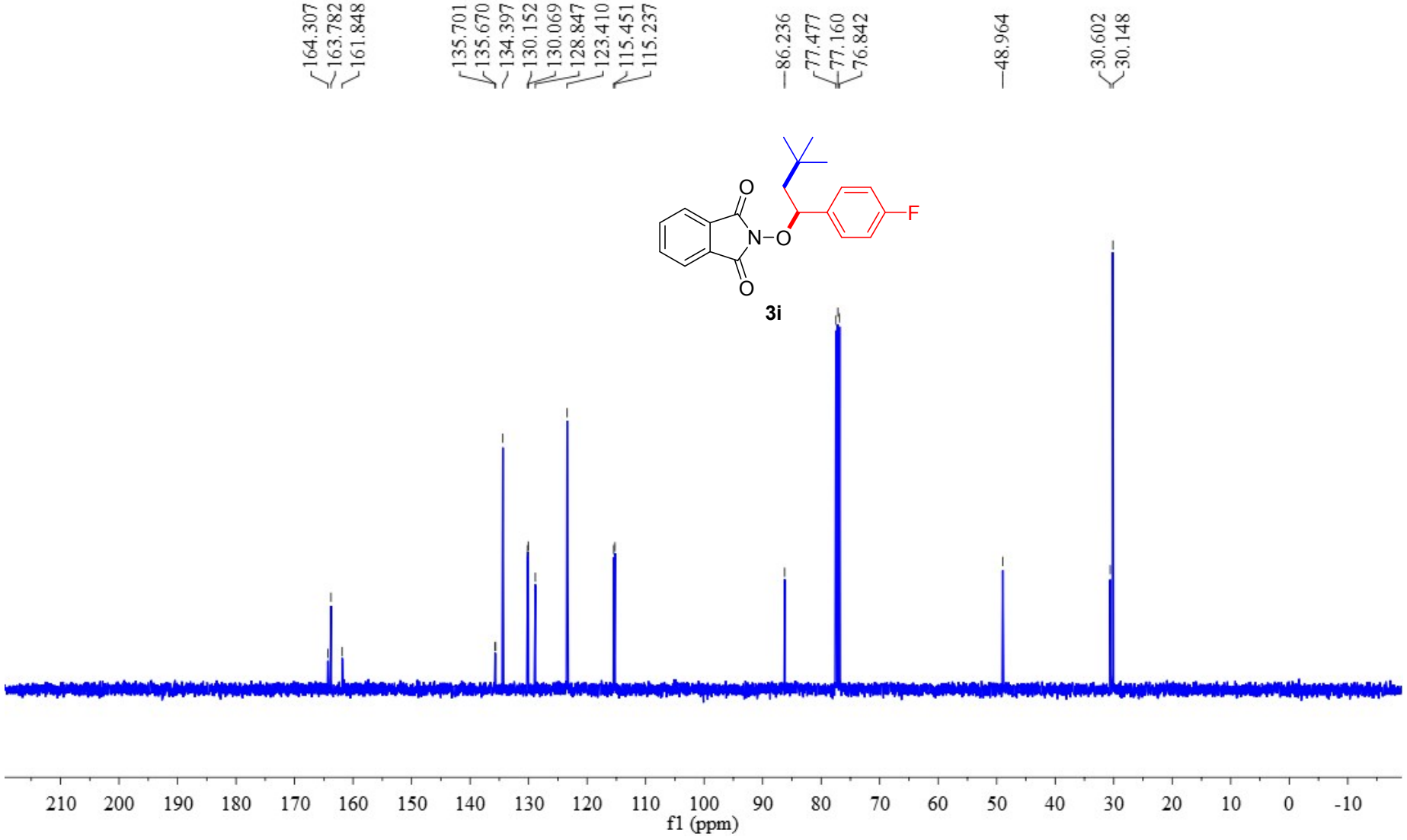
1.754
1.634
1.071
0.022
O
N–O
O
F
3i
4.03
2.01
2.03
1.00
1.03
1.05
9.09
f1 (ppm)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161.848
135.701
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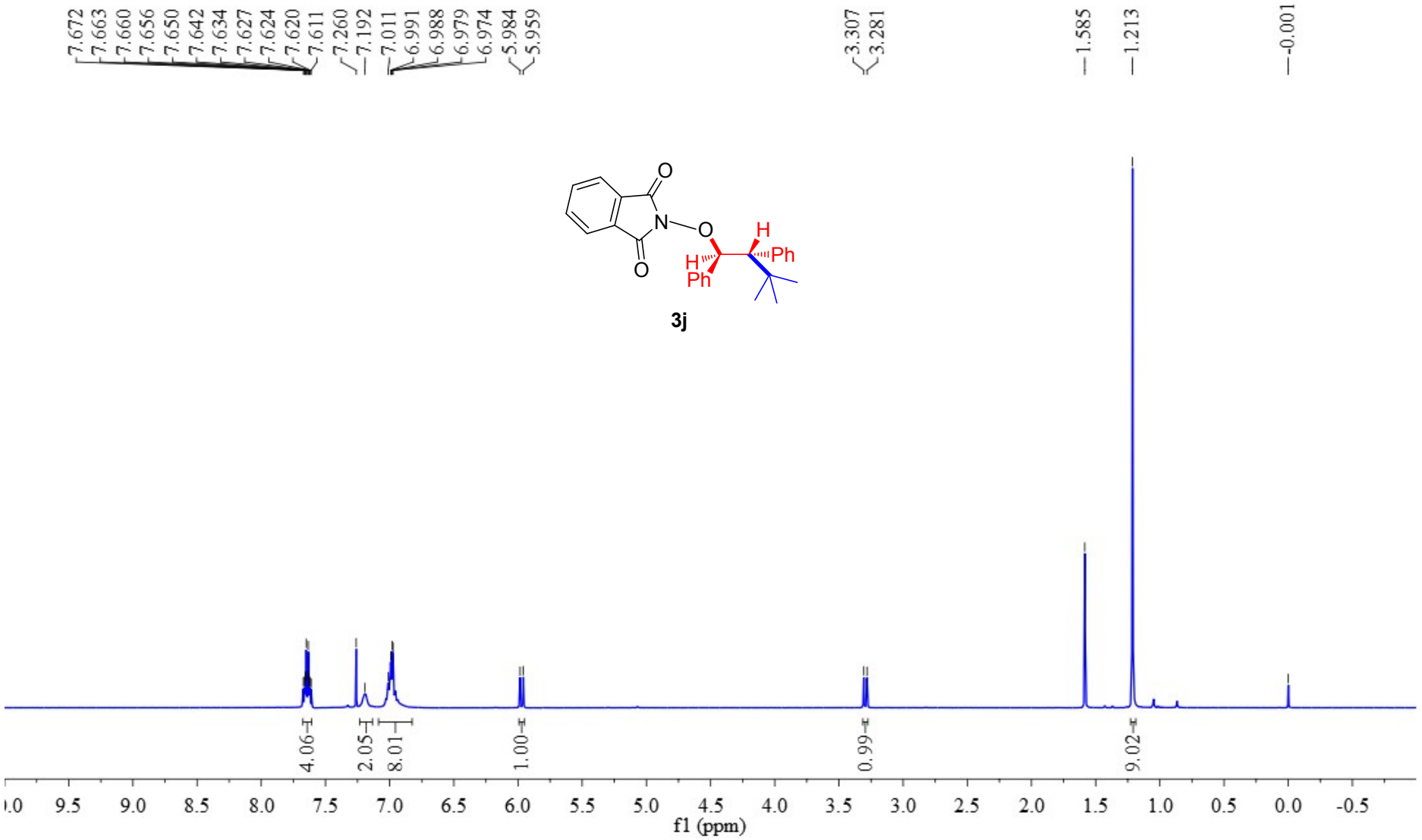
134.397
130.152
130.069
128.847
123.410
115.451
115.237
86.236
77.477
77.160
76.842
48.964
30.602
30.148
O
N–O
O
F
3i
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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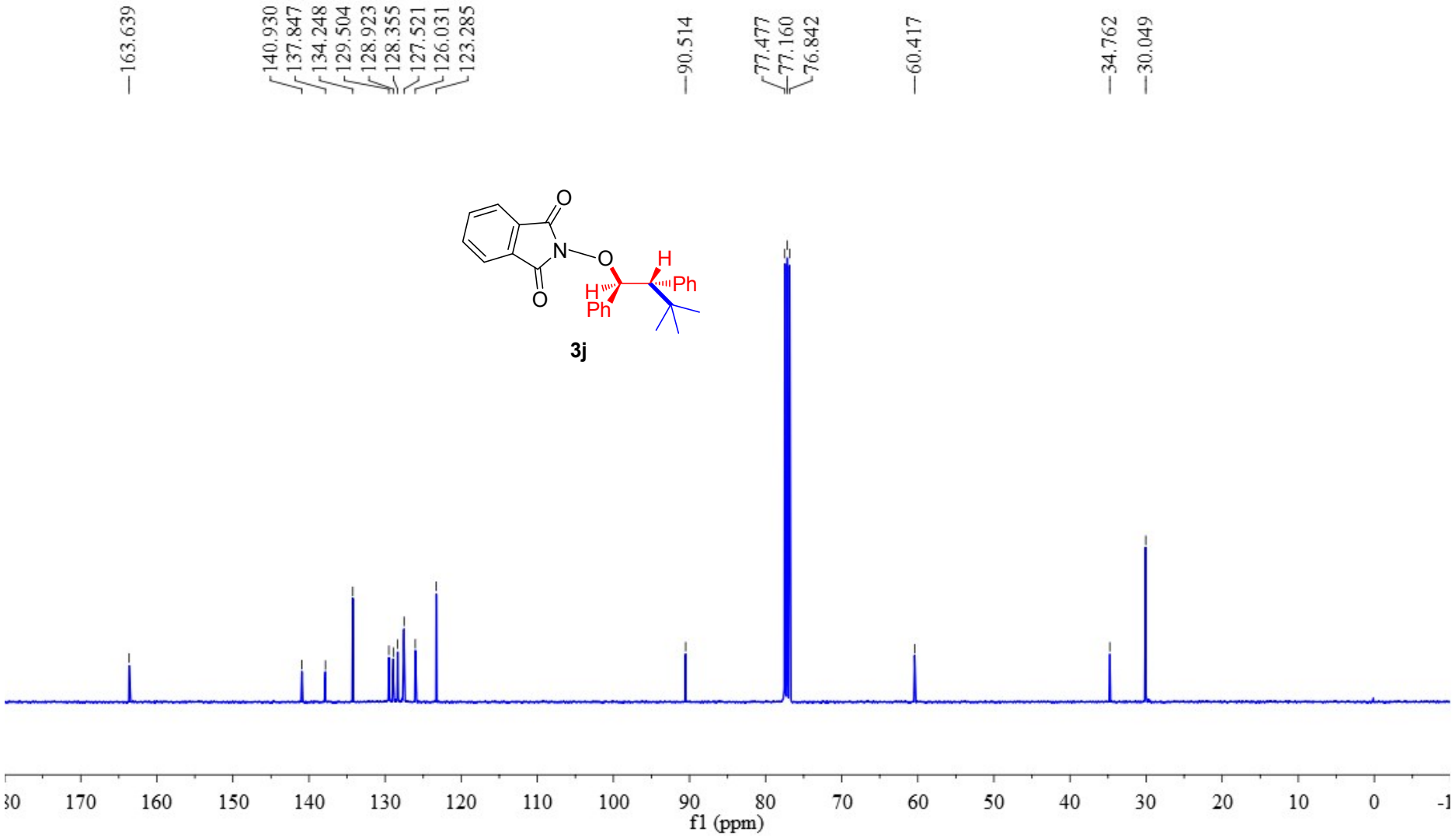
7.663
7.660
7.656
7.650
7.642
7.634
7.627
7.624
7.620
7.611
7.260
7.192
7.011
6.991
6.988
6.979
6.974
5.984
5.959
3.307
3.281
1.585
1.213
0.001
O
N
O
O
H
Ph
H
Ph
3j
4.06
2.05
8.01
1.00
0.99
9.02
f1 (ppm)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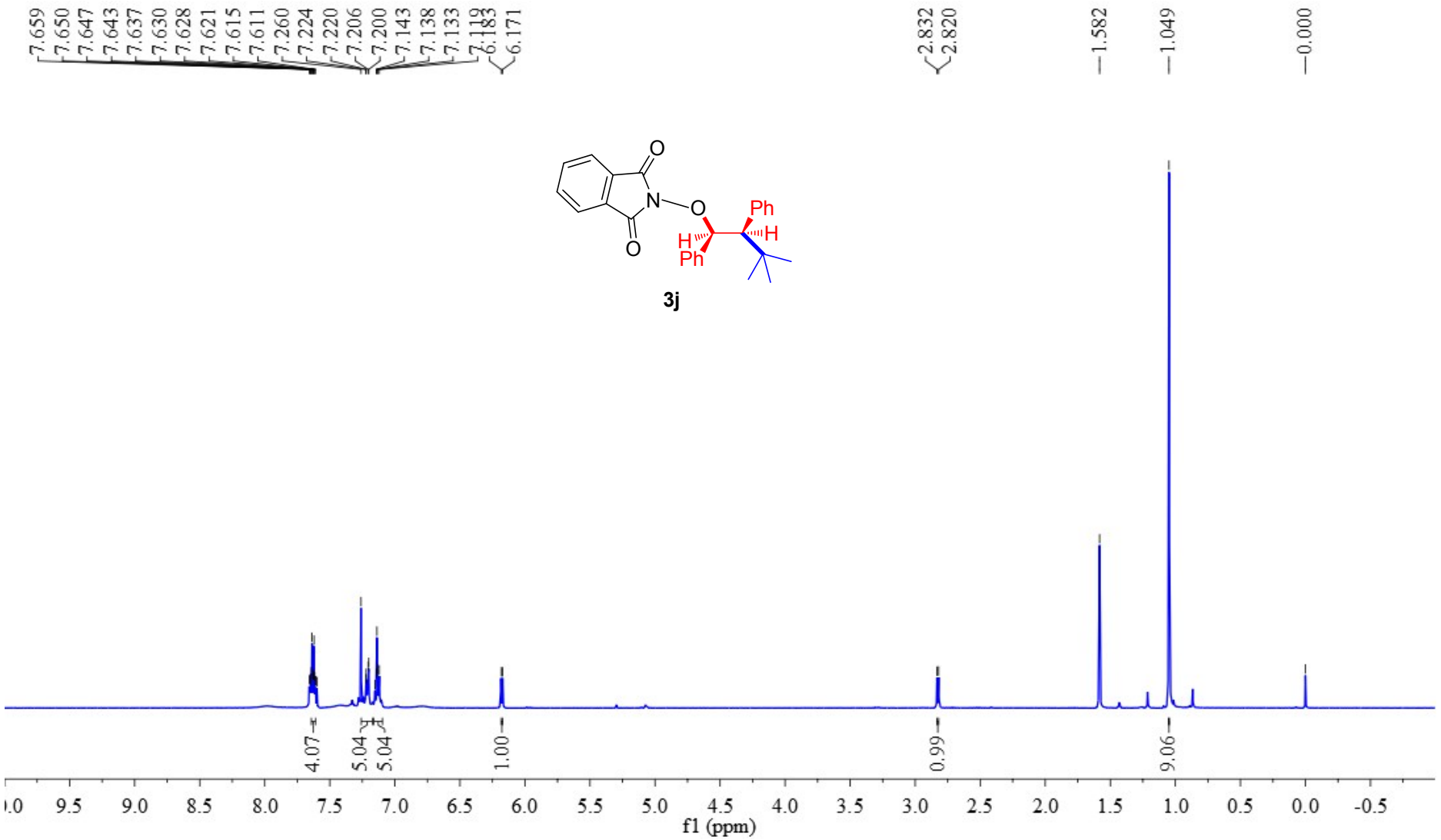
140.930
137.847
134.248
129.504
128.923
128.355
127.521
126.031
123.285
90.514
77.477
77.160
76.842
60.417
34.762
30.049
O
N
O
O
H
Ph
H
Ph
3j
f1 (ppm)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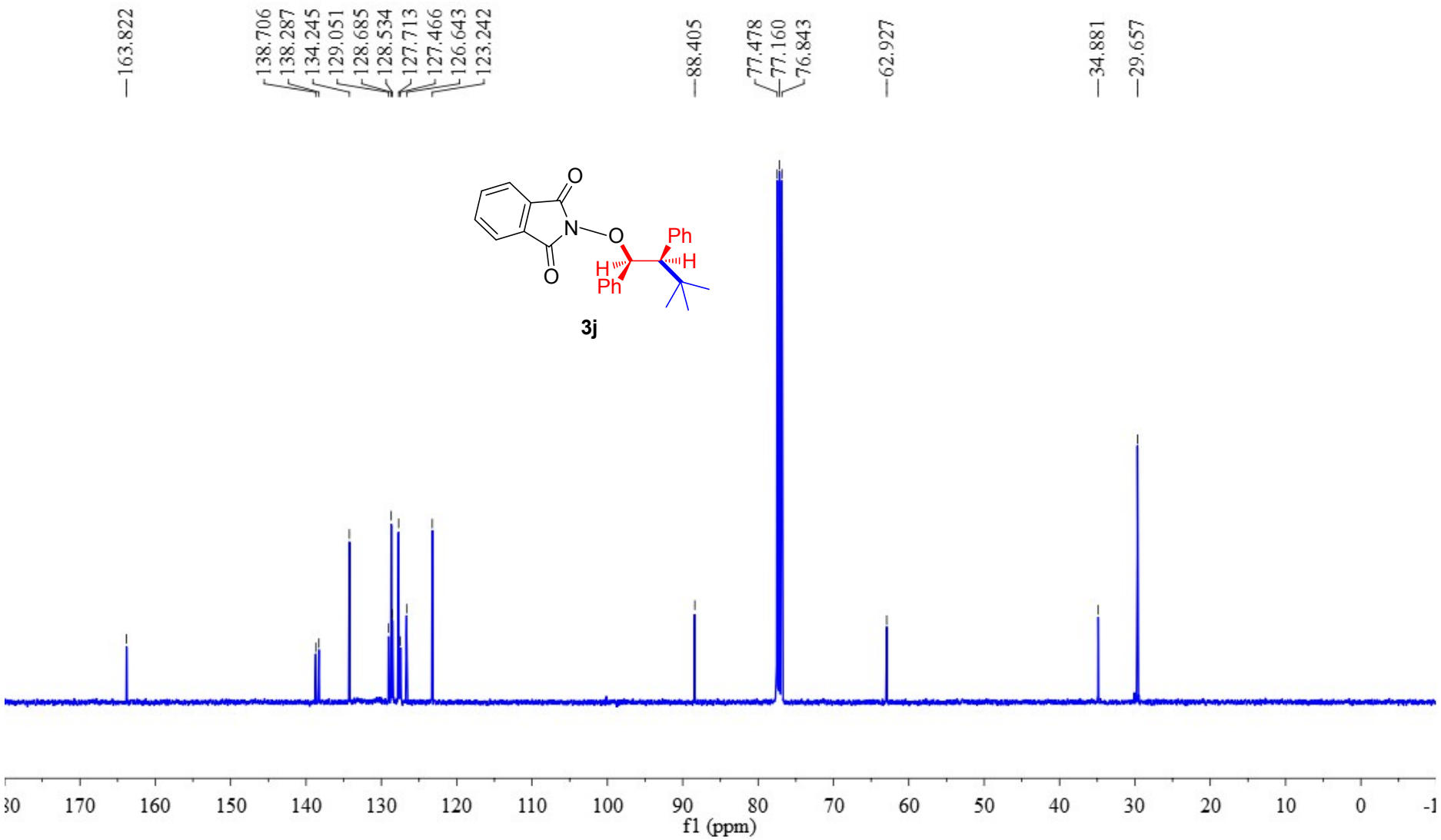
7.650
7.647
7.643
7.637
7.630
7.628
7.621
7.615
7.611
7.260
7.224
7.220
7.206
7.200
7.143
7.138
7.133
7.119
6.183
6.171
2.832
2.820
1.582
1.049
0.000
O
N
O
O
Ph
H
H
Ph
3j
4.07
5.04
5.04
1.00
0.99
9.06
f1 (ppm)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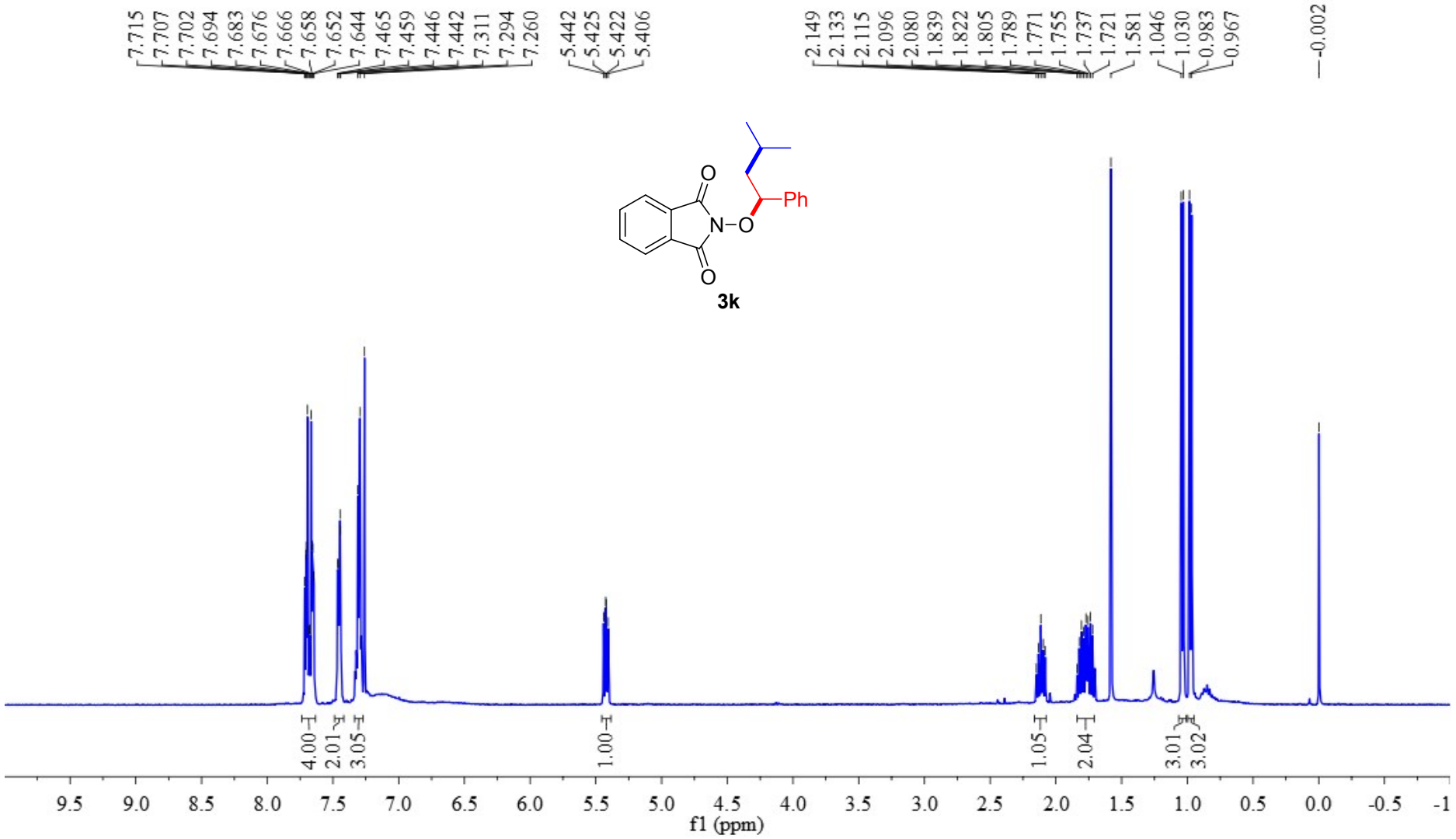
138.706
138.287
134.245
129.051
128.685
128.534
127.713
127.466
126.643
123.242
88.405
77.478
77.160
76.843
62.927
34.881
29.657
O
N
O
O
Ph
H
H
Ph
3j
f1 (ppm)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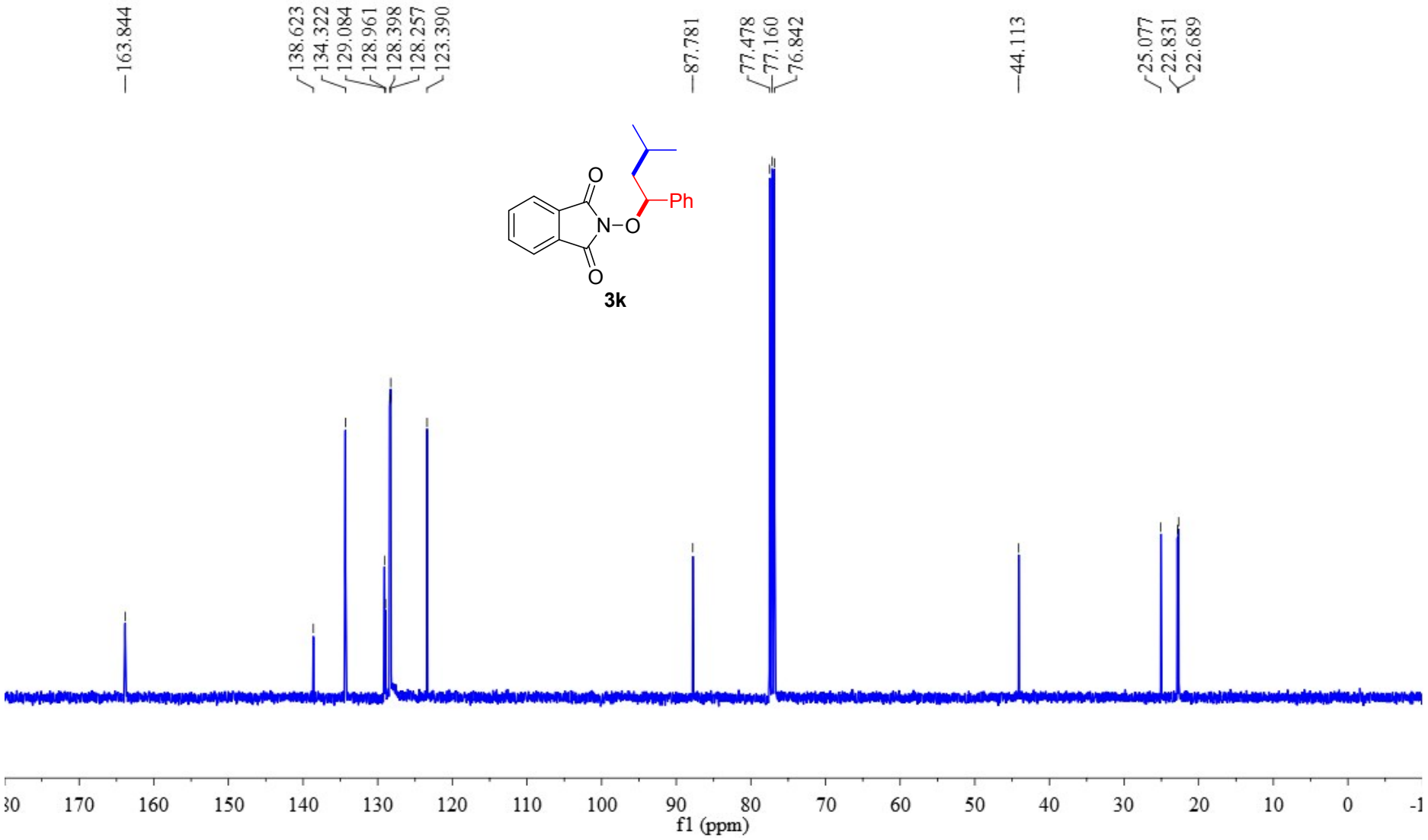
7.707
7.702
7.694
7.683
7.676
7.666
7.658
7.652
7.644
7.465
7.459
7.446
7.442
7.311
7.294
7.260
5.442
5.425
5.422
5.406
2.149
2.133
2.115
2.096
2.080
1.839
1.822
1.805
1.789
1.771
1.755
1.737
1.721
1.581
1.046
1.030
0.983
0.967
-0.002
O
N–O
O
Ph
3k
4.00
2.01
3.05
1.00
1.05
2.04
3.01
3.02
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)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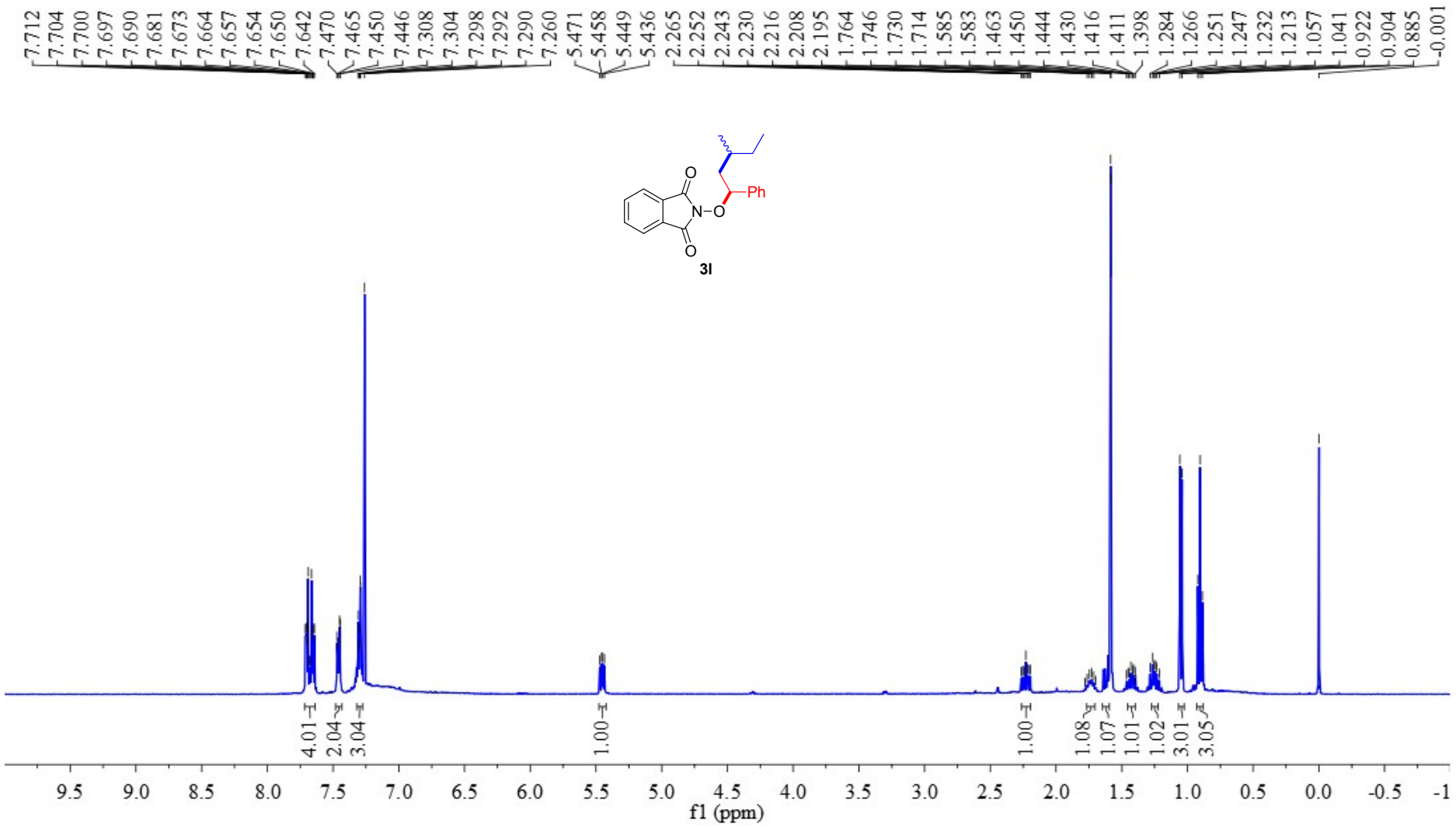
134.322
129.084
128.961
128.398
128.257
123.390
87.781
77.478
77.160
76.842
44.113
25.077
22.831
22.689
O
N–O
O
Ph
3k
f1 (ppm)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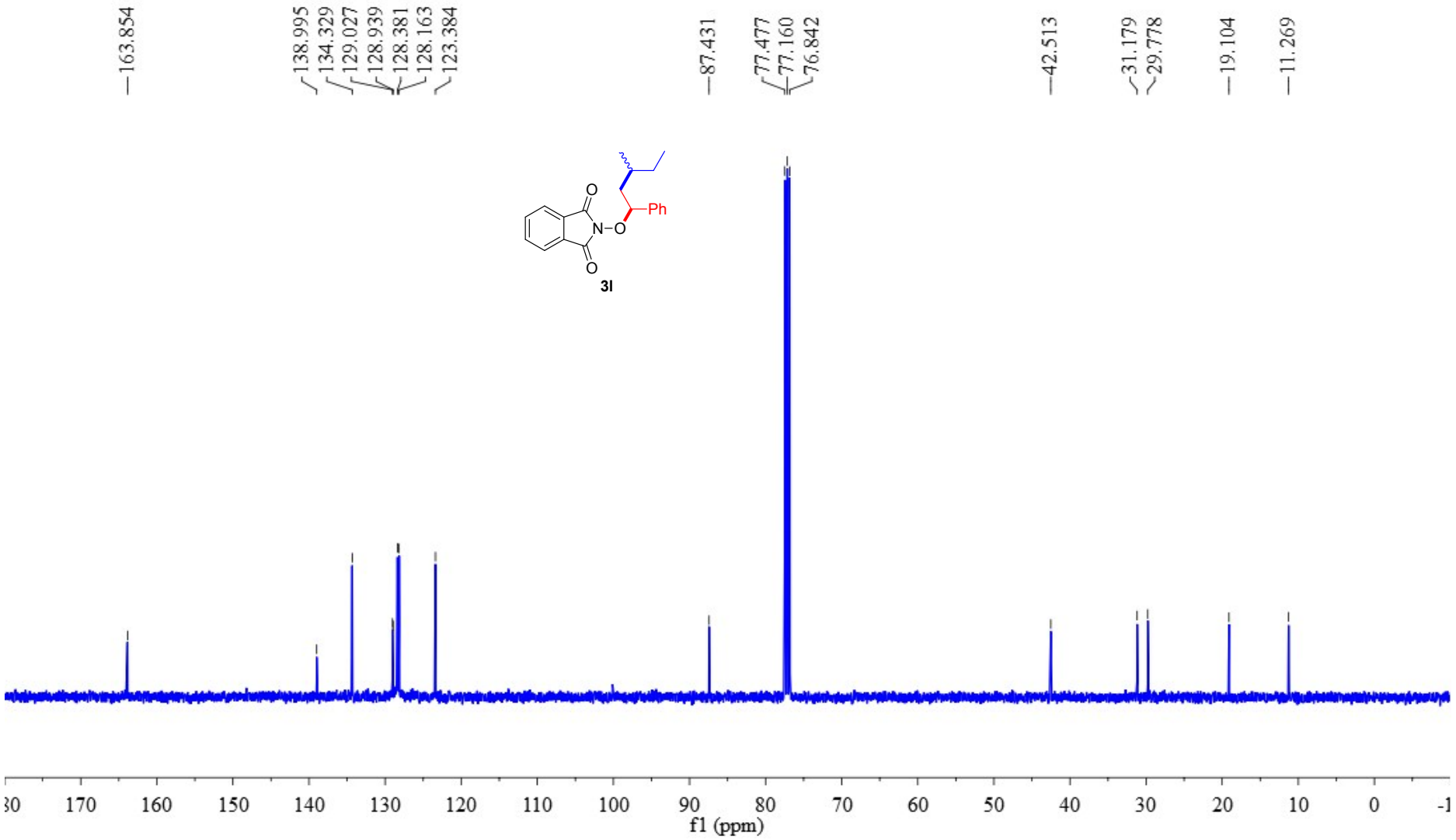
7.704
7.700
7.697
7.690
7.681
7.673
7.664
7.657
7.654
7.650
7.642
7.470
7.465
7.450
7.446
7.308
7.304
7.298
7.292
7.290
7.260
5.471
5.458
5.449
5.436
2.265
2.252
2.243
2.230
2.216
2.208
2.195
1.764
1.746
1.730
1.714
1.585
1.583
1.463
1.450
1.444
1.430
1.416
1.411
1.398
1.284
1.266
1.251
1.247
1.232
1.213
1.057
1.041
0.922
0.904
0.885
-0.001
O
N–O
O
Ph
3l
4.01
2.04
3.04
1.00
1.00
1.08
1.07
1.01
1.02
3.01
3.05
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1
f1 (ppm)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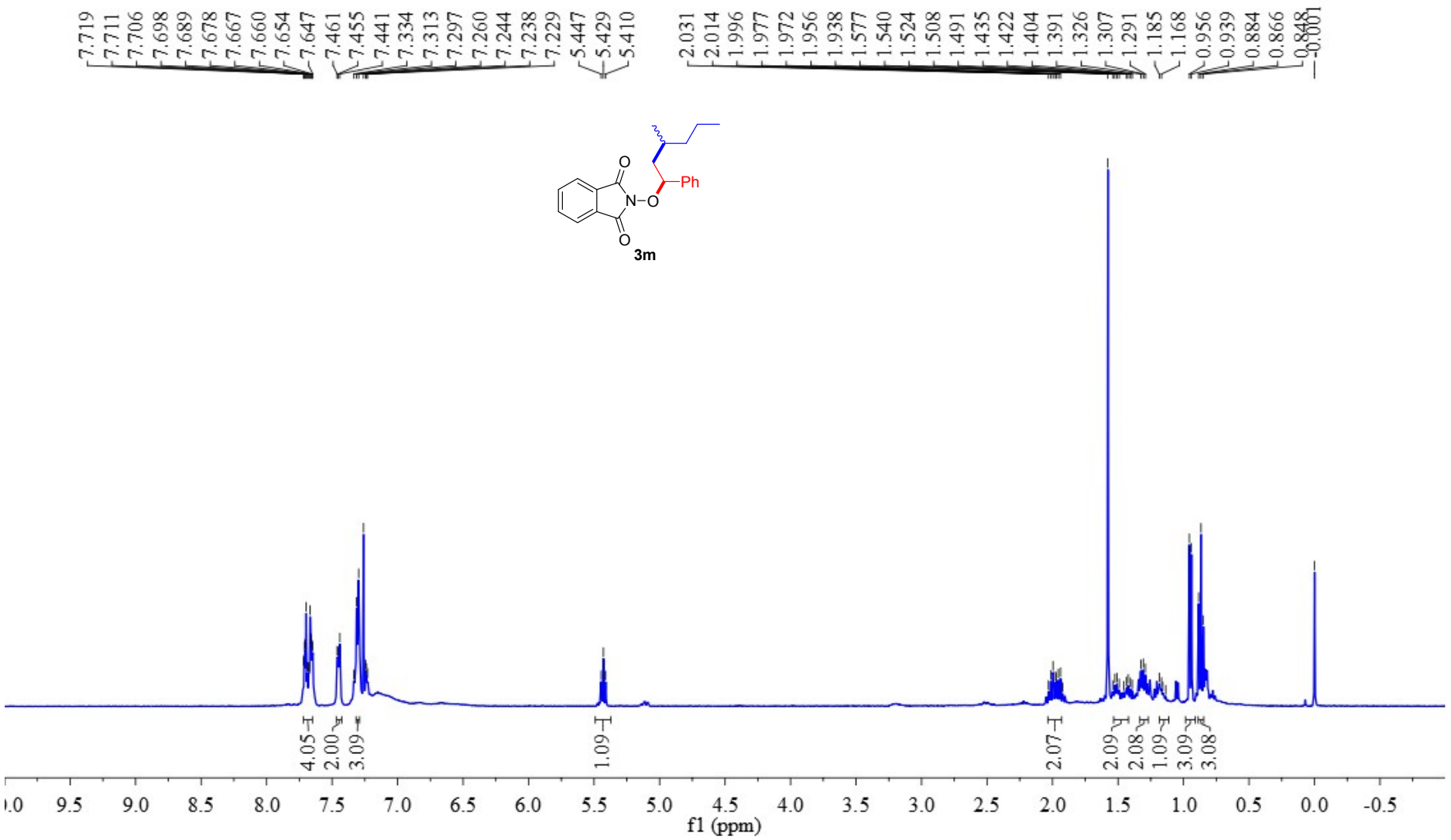
134.329
129.027
128.939
128.381
128.163
123.384
87.431
77.477
77.160
76.842
42.513
31.179
29.778
19.104
11.269
O
N–O
O
Ph
3l
f1 (ppm)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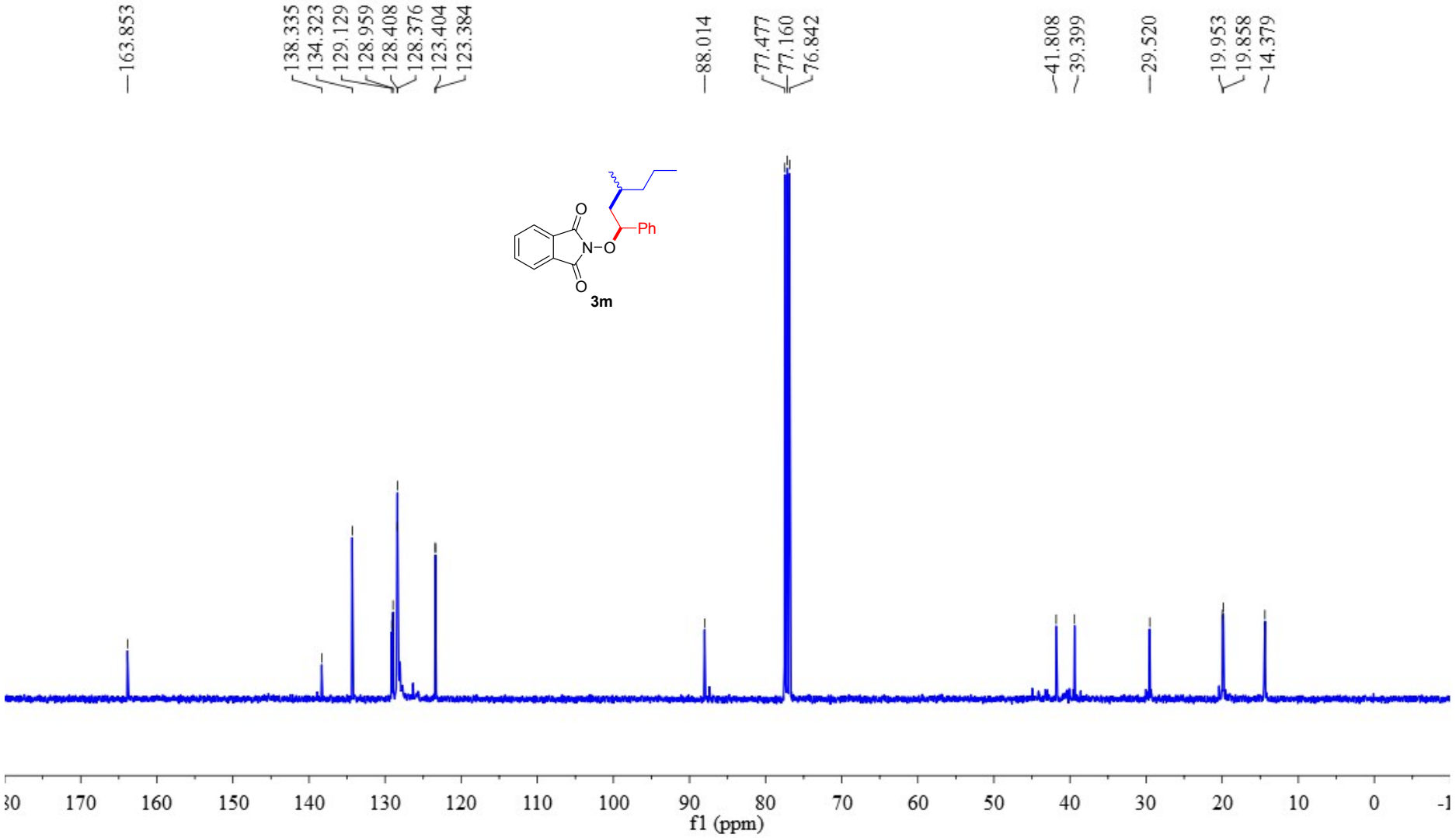
7.706
7.698
7.689
7.678
7.667
7.660
7.654
7.647
7.461
7.455
7.441
7.334
7.313
7.297
7.260
7.244
7.238
7.229
5.447
5.429
5.410
2.031
2.014
1.996
1.977
1.972
1.956
1.938
1.577
1.540
1.524
1.508
1.491
1.435
1.422
1.404
1.391
1.326
1.307
1.291
1.185
1.168
0.956
0.939
0.884
0.866
0.848
0.001
O
N–O
O
Ph
3m
4.05
2.00
3.09
1.09
2.07
2.09
2.08
1.09
3.09
3.08
f1 (ppm)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138.335
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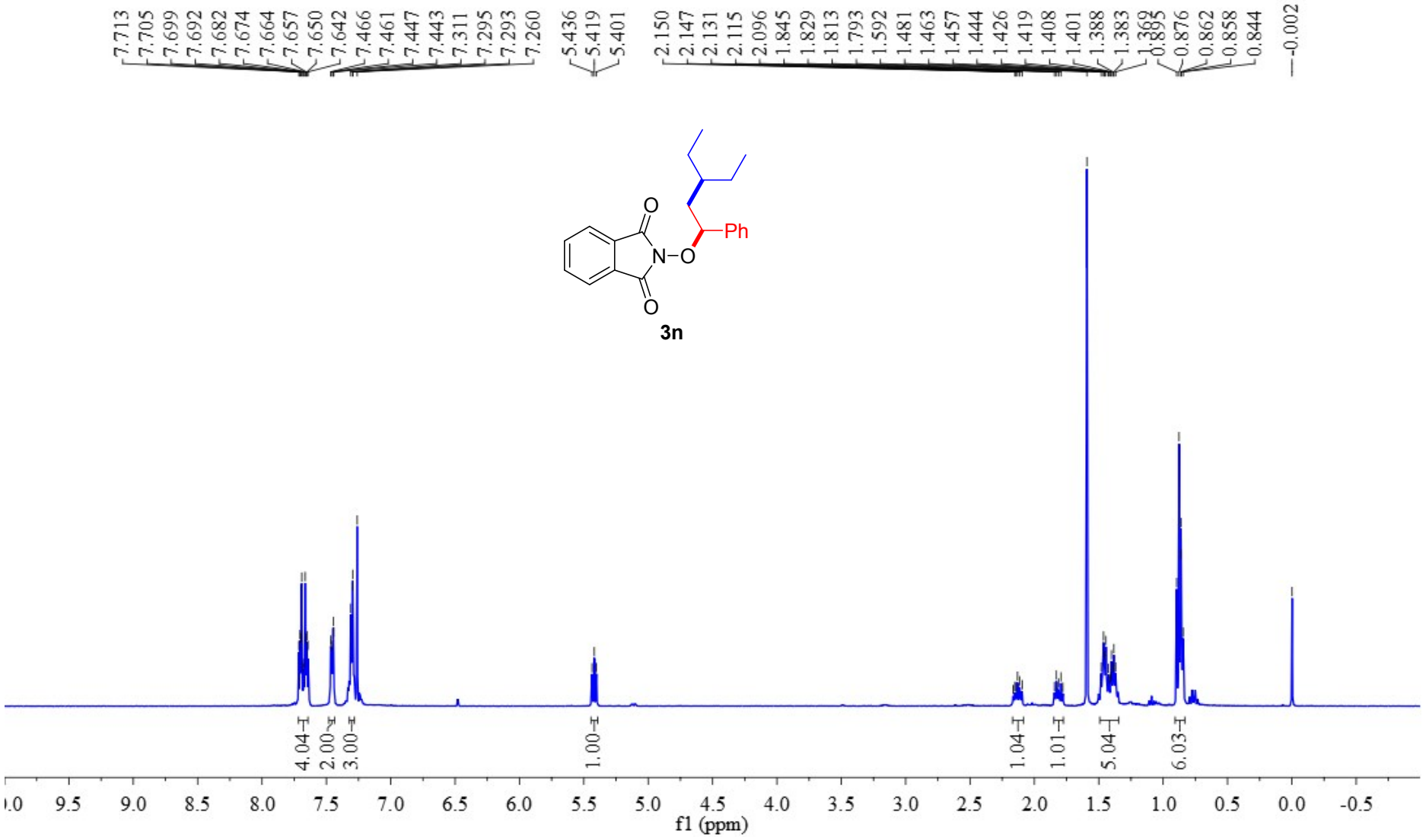
128.408
128.376
123.404
123.384
88.014
77.477
77.160
76.842
41.808
39.399
29.520
19.953
19.858
14.379
O
N–O
Ph
O
3m
f1 (ppm)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7.699
7.692
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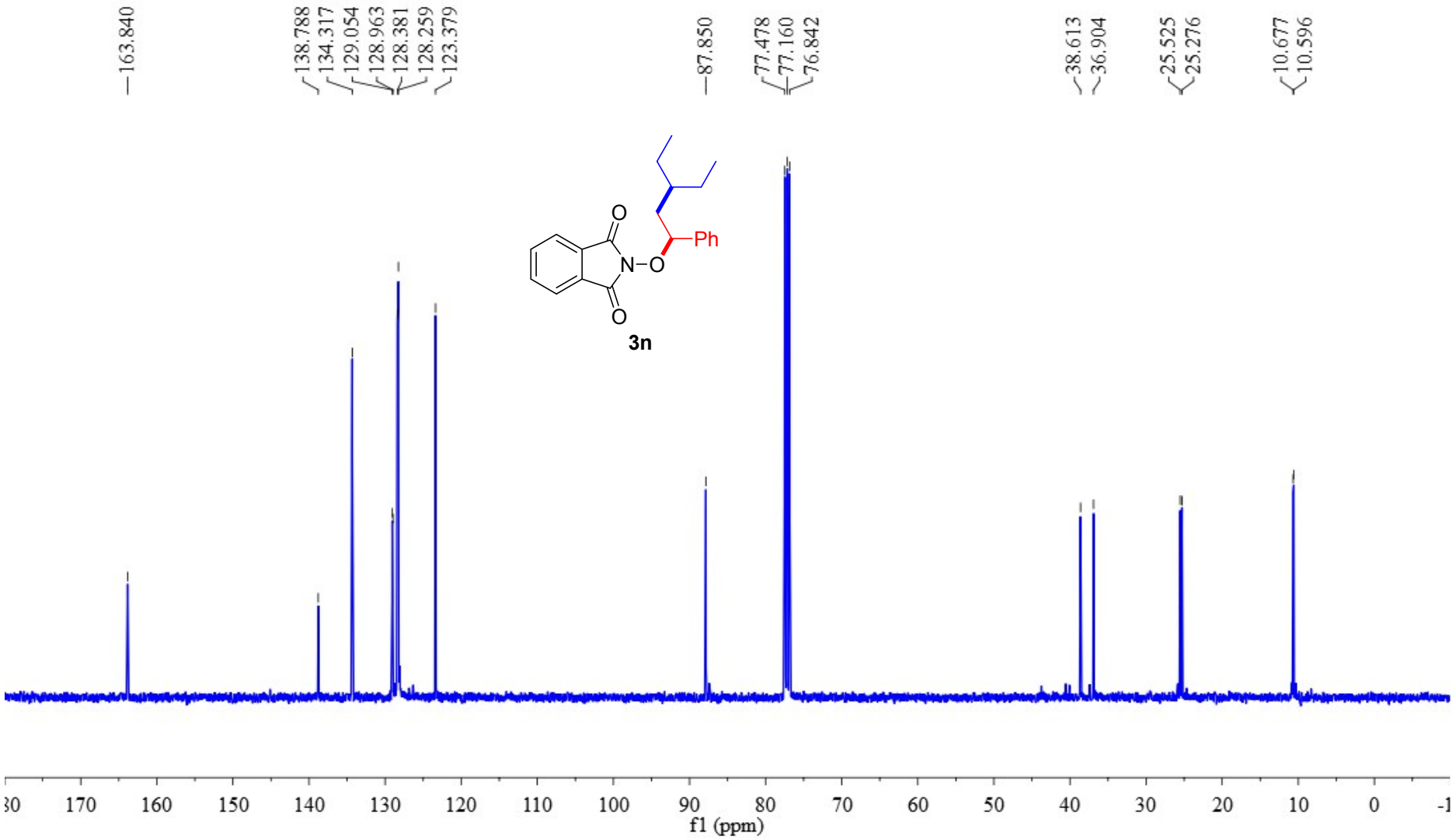
7.657
7.650
7.642
7.466
7.461
7.447
7.443
7.311
7.295
7.293
7.260
5.436
5.419
5.401
2.150
2.147
2.131
2.115
2.096
1.845
1.829
1.813
1.793
1.592
1.481
1.463
1.457
1.444
1.426
1.419
1.408
1.401
1.388
1.383
0.895
0.876
0.862
0.858
0.844
-0.002
O
N–O
O
Ph
3n
4.04
2.00
3.00
1.00
1.04
1.01
5.04
6.03
f1 (ppm)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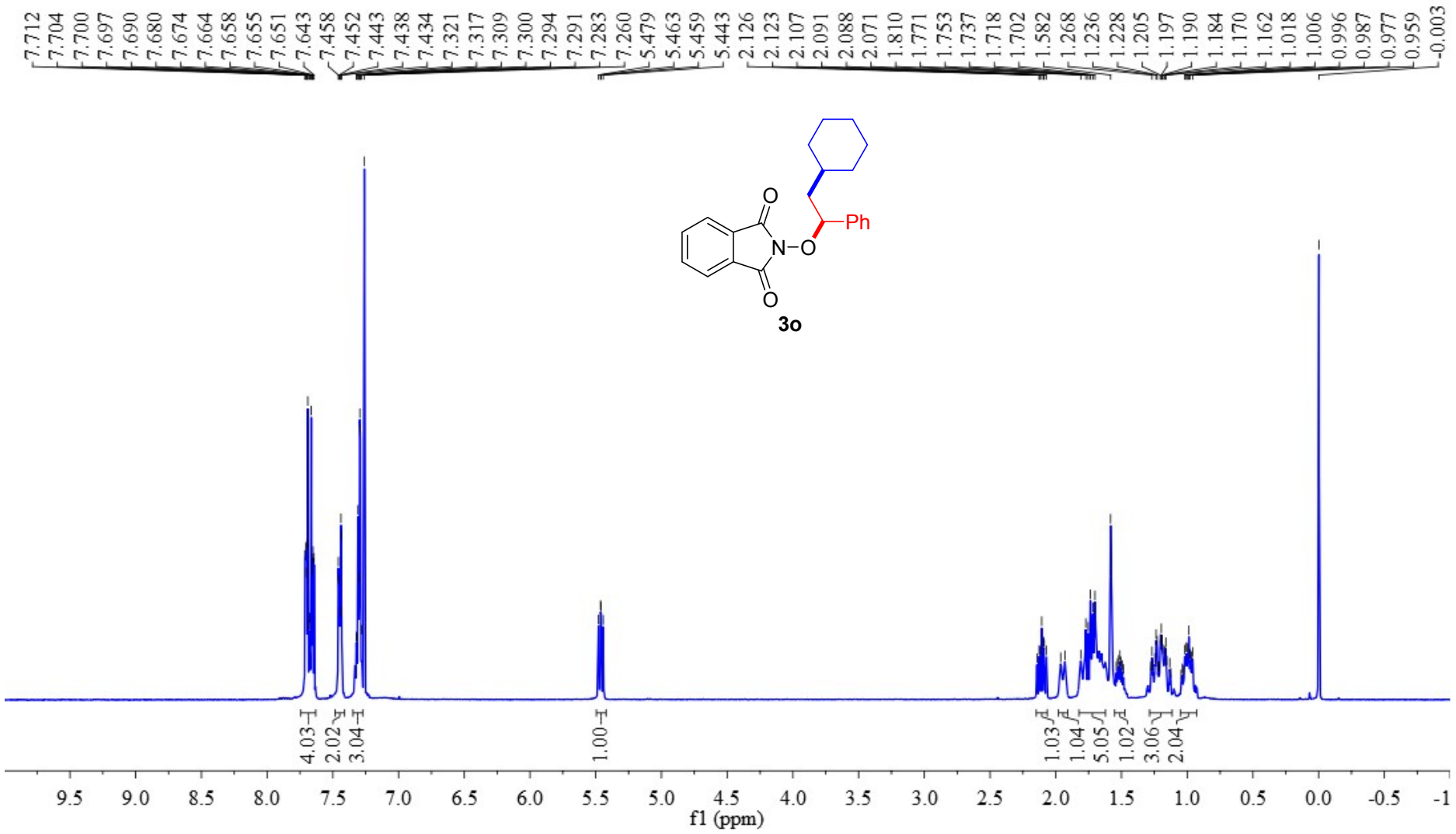
128.381
128.259
123.379
87.850
77.478
77.160
76.842
38.613
36.904
25.525
25.276
10.677
10.596
O
N–O
Ph
O
3n
f1 (ppm)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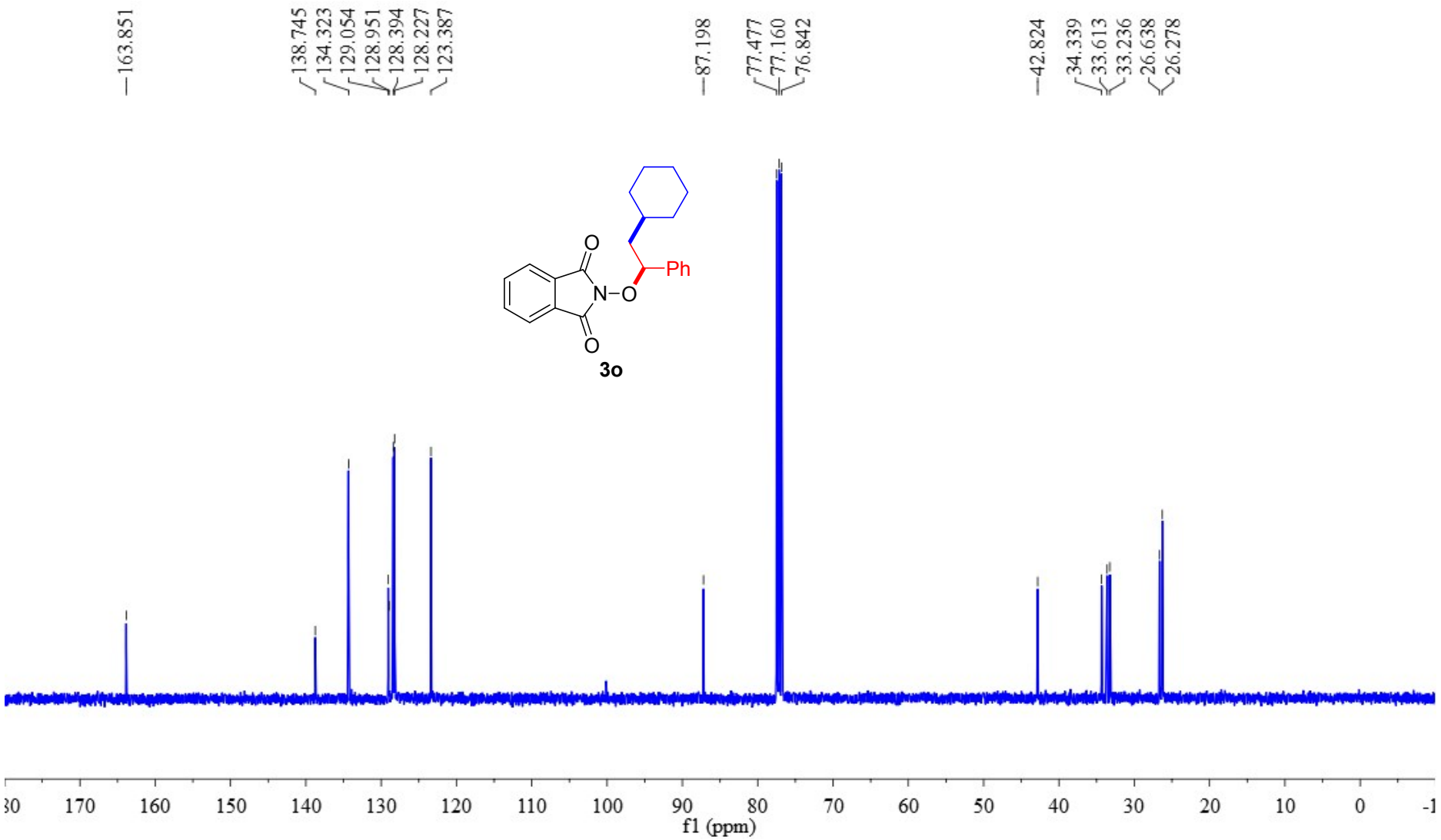
7.704
7.700
7.697
7.690
7.680
7.674
7.664
7.658
7.655
7.651
7.643
7.458
7.452
7.443
7.438
7.434
7.321
7.317
7.309
7.300
7.294
7.291
7.283
7.260
5.479
5.463
5.459
5.443
2.126
2.123
2.107
2.091
2.088
2.071
1.810
1.771
1.753
1.737
1.718
1.702
1.582
1.268
1.236
1.228
1.205
1.197
1.190
1.184
1.170
1.162
1.018
1.006
0.996
0.987
0.977
0.959
-0.003
O
N–O
O
Ph
3o
4.03
2.02
3.04
1.00
1.03
1.04
5.05
1.02
3.06
2.04
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1
f1 (ppm)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128.951
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128.227
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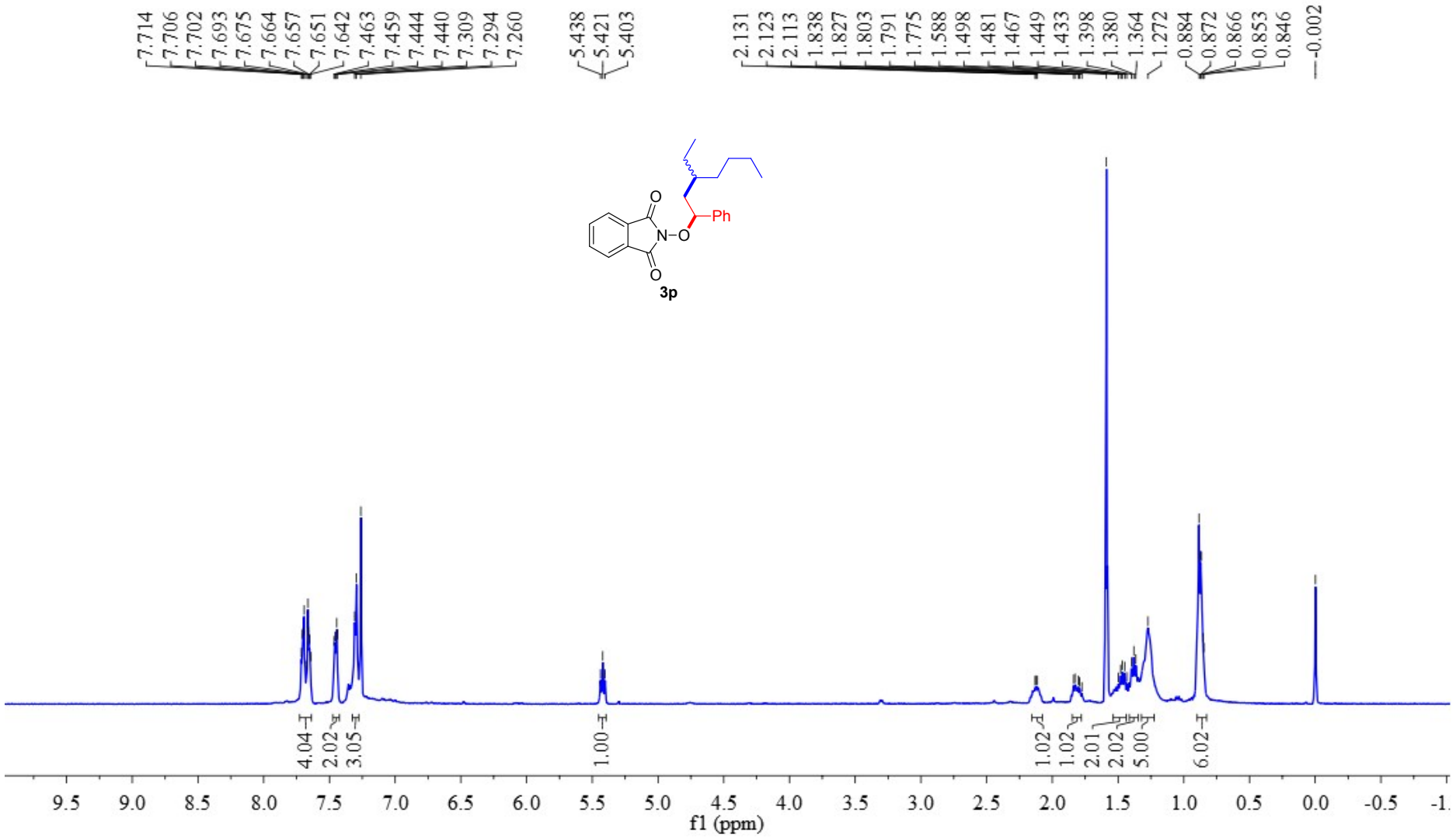
77.477
77.160
76.842
42.824
34.339
33.613
33.236
26.638
26.278
O
N–O
Ph
O
3o
f1 (ppm)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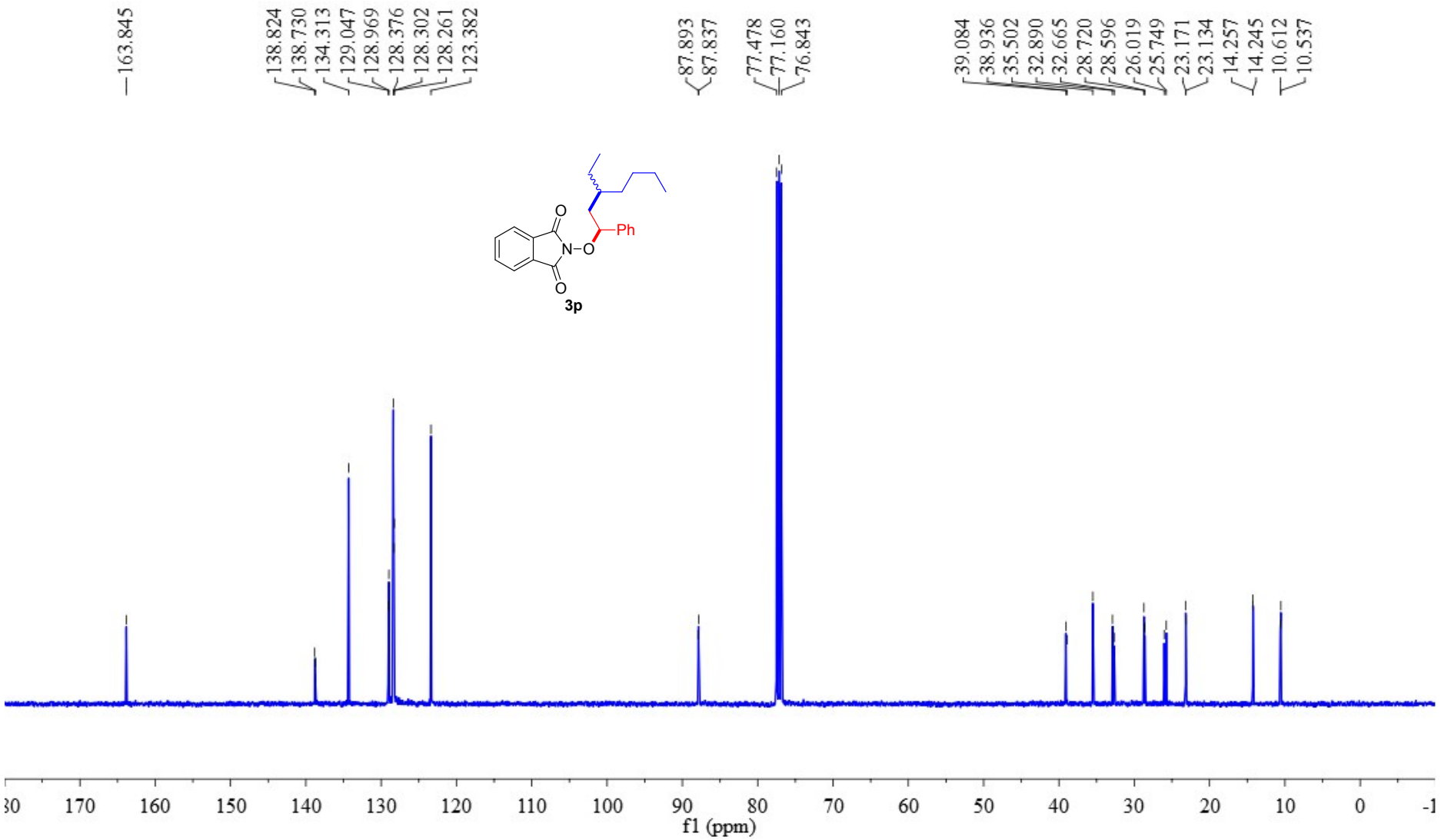
7.463
7.459
7.444
7.440
7.309
7.294
7.260
5.438
5.421
5.403
2.131
2.123
2.113
1.838
1.827
1.803
1.791
1.775
1.588
1.498
1.481
1.467
1.449
1.433
1.398
1.380
1.364
1.272
0.884
0.872
0.866
0.853
0.846
-0.002
O
N–O
O
Ph
3p
4.04
2.02
3.05
1.00
1.02
1.02
2.01
2.02
5.00
6.02
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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128.302
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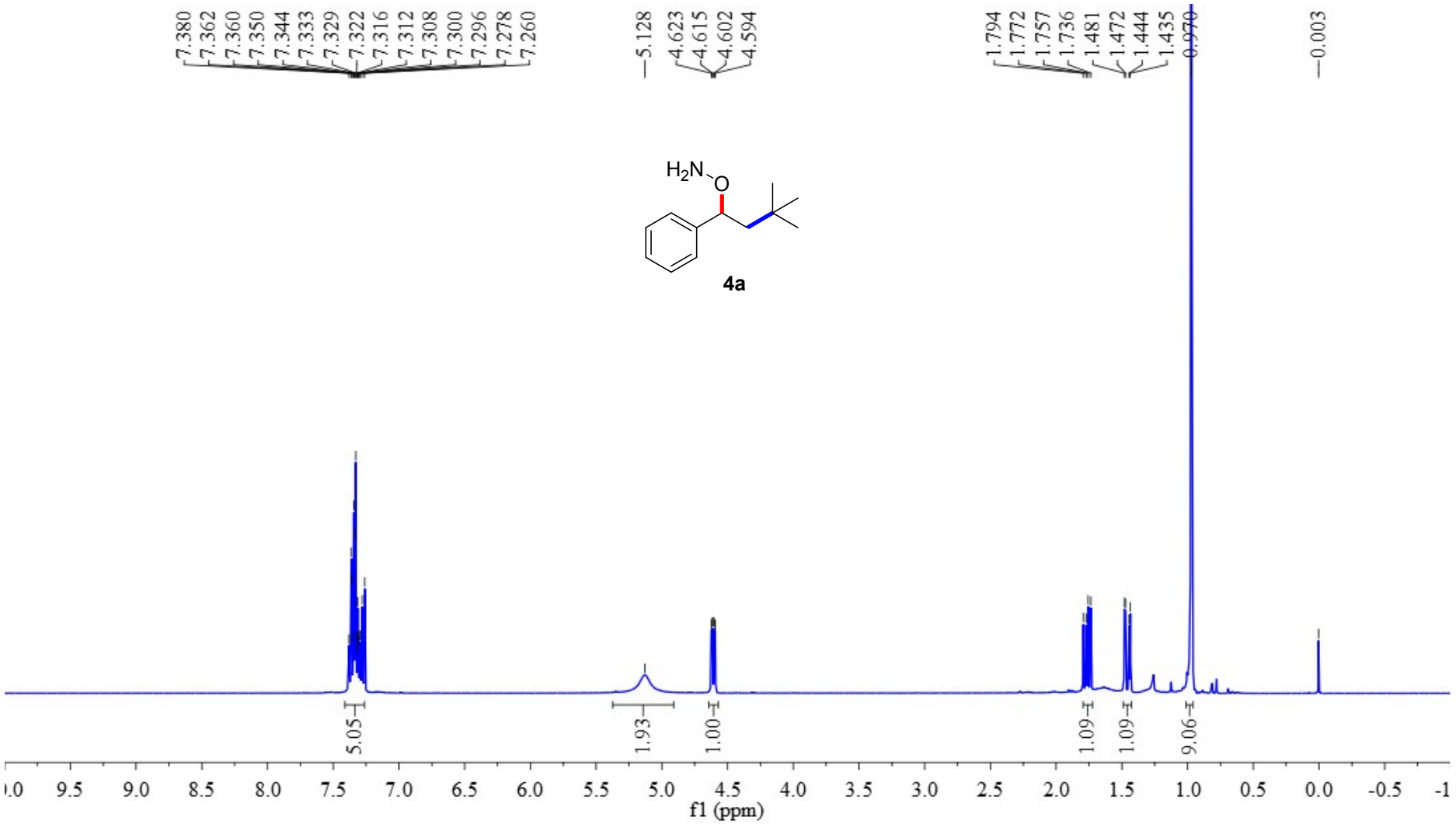
87.837
77.478
77.160
76.843
39.084
38.936
35.502
32.890
32.665
28.720
28.596
26.019
25.749
23.171
23.134
14.257
14.245
10.612
10.537
O
N–O
Ph
O
3p
f1 (ppm)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7.333
7.329
7.322
7.316
7.312
7.308
7.300
7.296
7.278
7.260
5.128
4.623
4.615
4.602
4.594
1.794
1.772
1.757
1.736
1.481
1.472
1.444
1.435
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0.003
H2N
O
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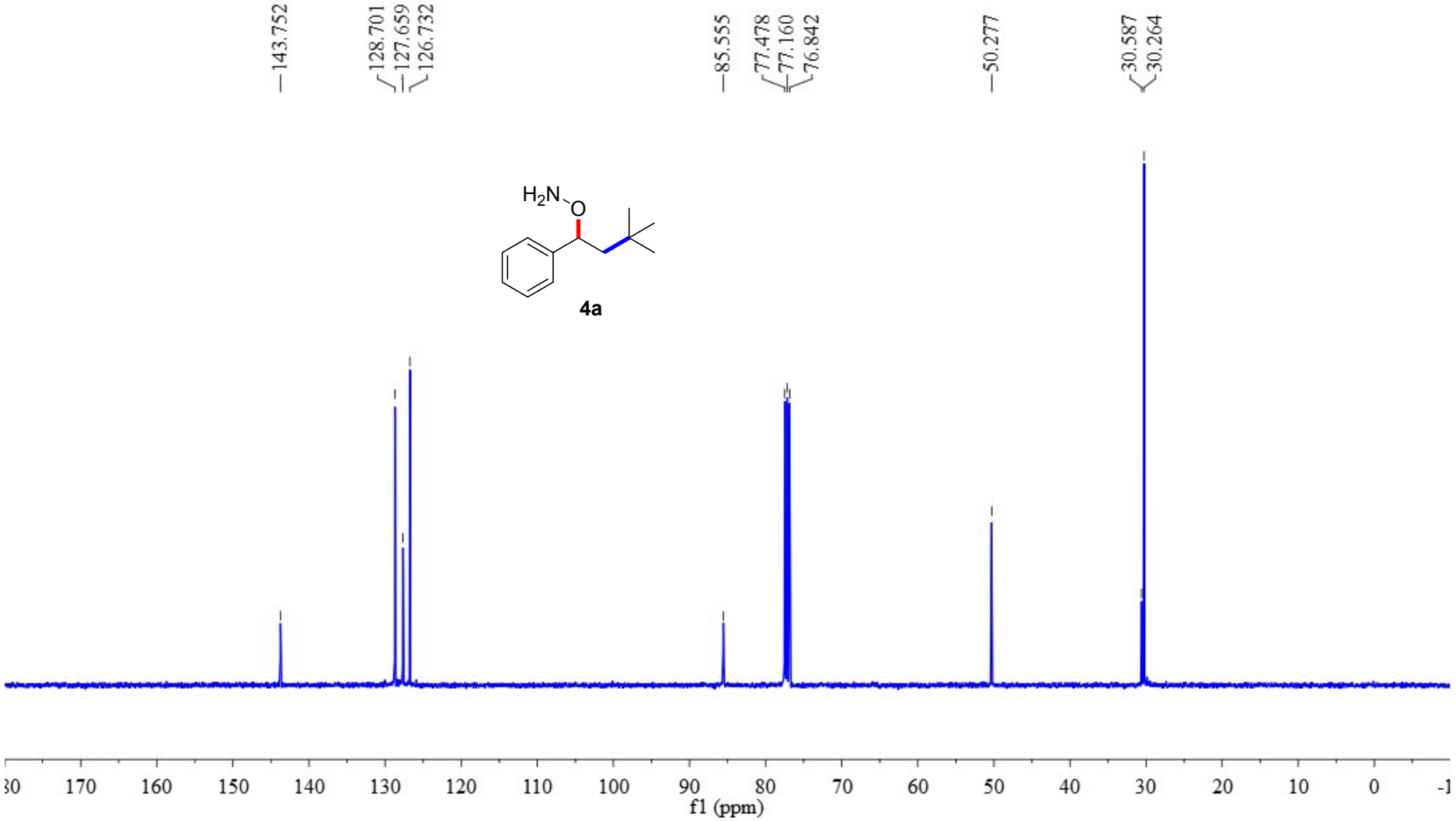
1.93
1.00
1.09
1.09
9.06
f1 (ppm)


143.752
128.701
127.659
126.732
85.555
77.478
77.160
76.842
50.277
30.587
30.264
H2N
O
4a
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)