Supporting Information

Electrifying Phthalimide-*N*-Oxyl (PINO) Radical Chemistry: Anodically Induced Dioxygenation of Vinyl Arenes with *N*-hydroxyphthalimide

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*Corresponding author

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1. Picture of an Electrochemical Cell

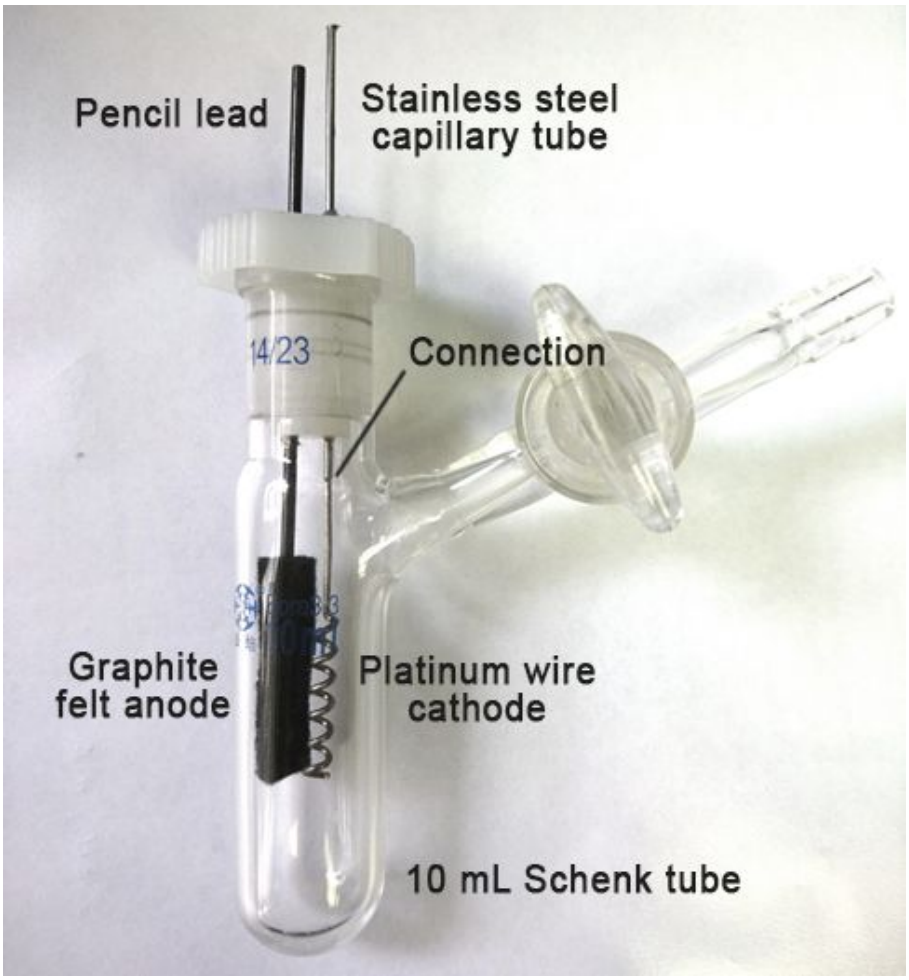
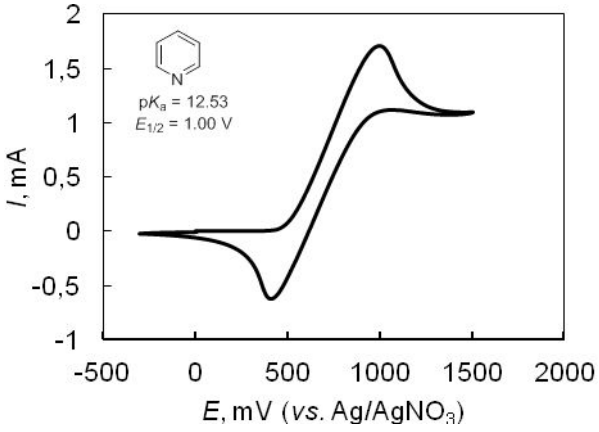
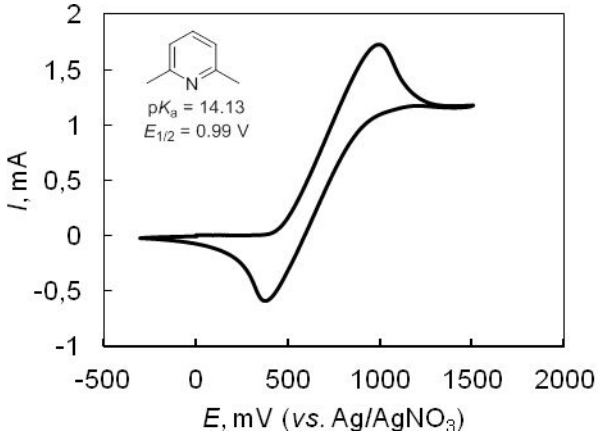


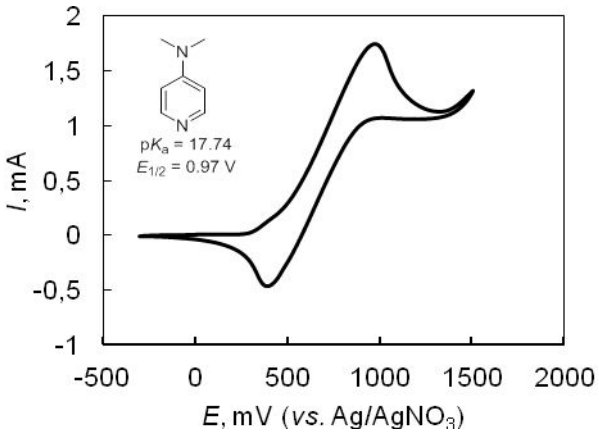
Figure S1. Electrochemical cell with electrodes employed in the present work

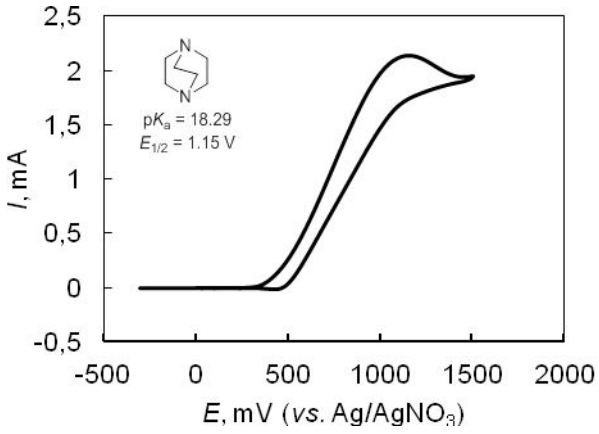
2. Cyclic Voltammetry Studies

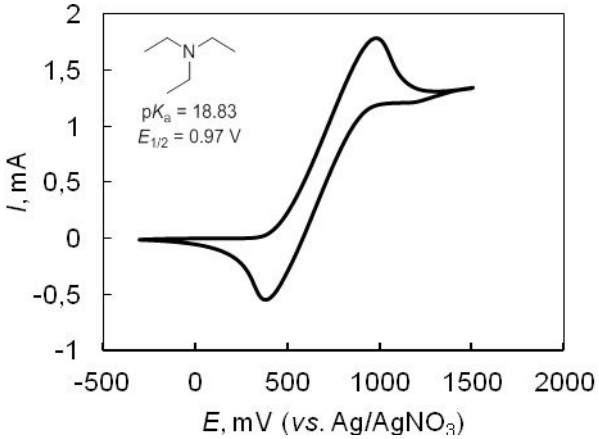
Table S1. CV curves of 0.1 M solution of NHPI **2a** in 0.1 M [pyH]ClO_4 / MeCN in the presence of various bases (0.1 M) (working glassy-carbon electrode (d = 3 mm), scan rate 0.1 V/s)

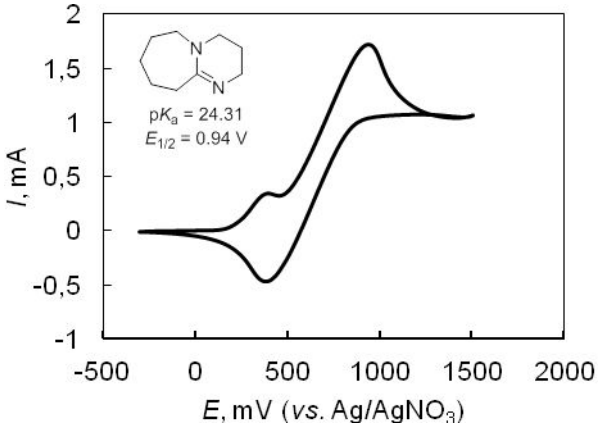












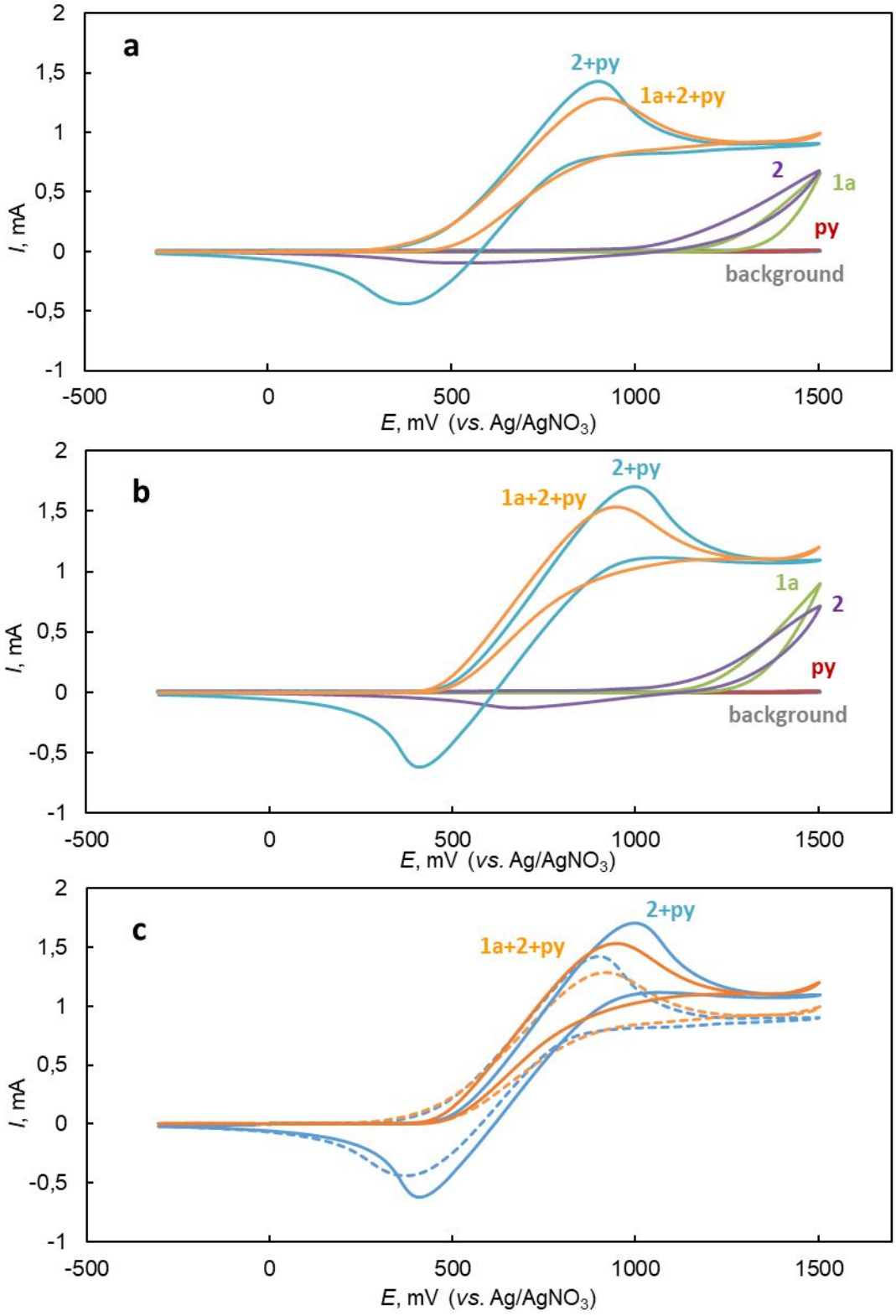
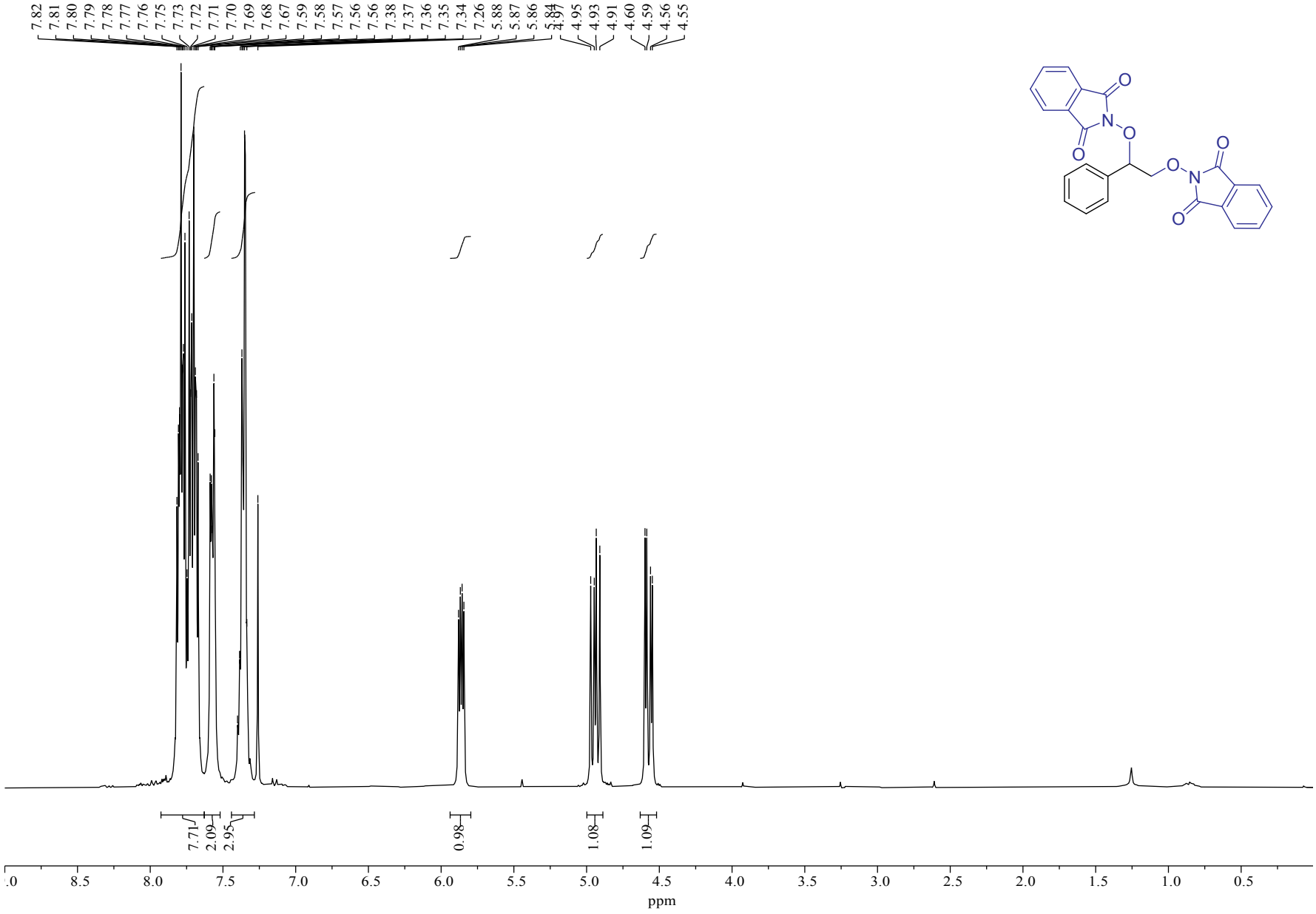


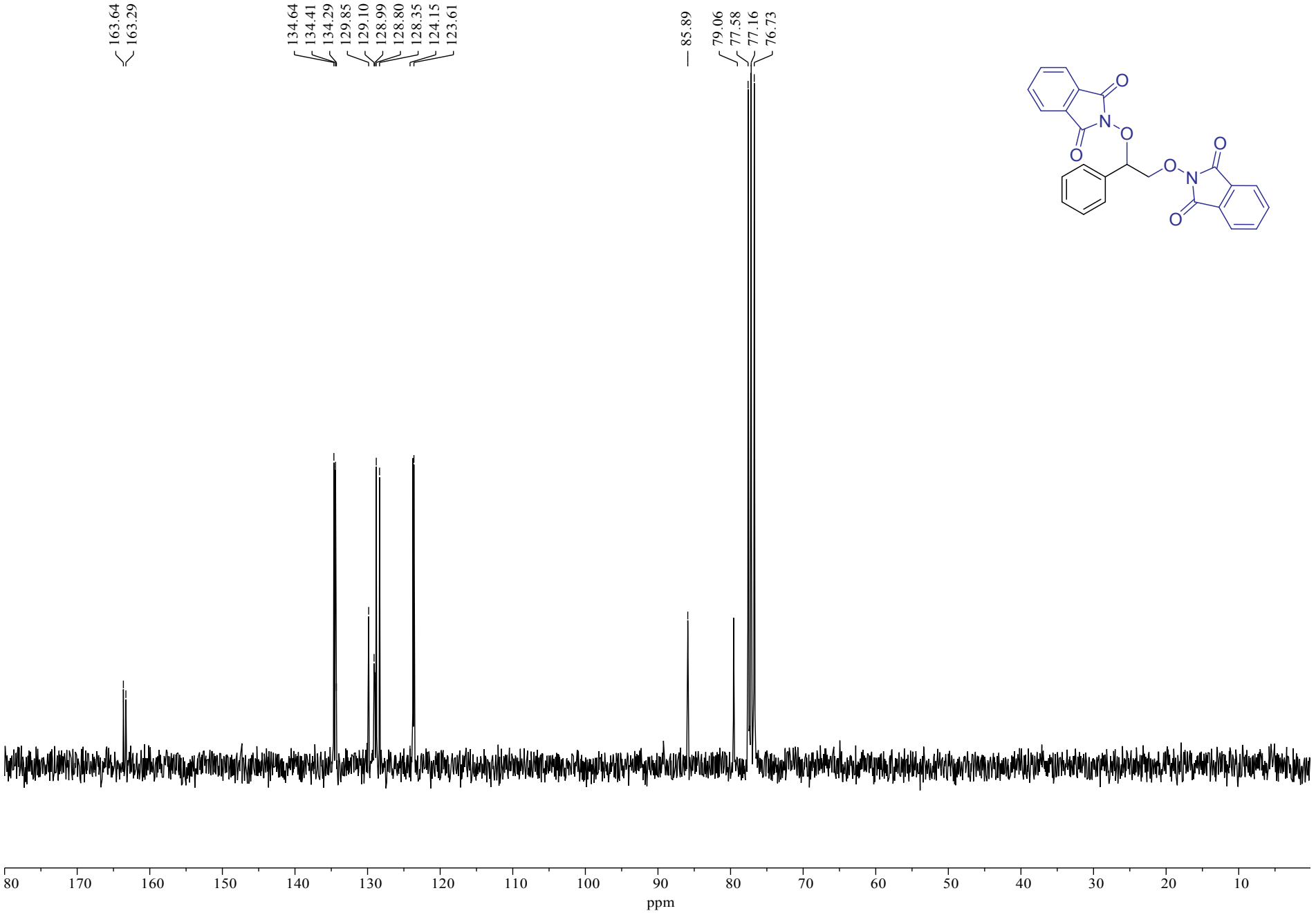
Figure S2. CV curves: 0.1 M solution of NHPI **2** and pyridine (orange), styrene **1a**, NHPI **2**, and pyridine (blue), styrene **1a** (green), pyridine (red) and NHPI **2** (violet) in 0.1 M *n*-Bu_4NBF_4 / MeCN **(a)** and 0.1 M [pyH]ClO_4 / MeCN **(b)**; 0.1 M solution of NHPI **2** and pyridine (orange), styrene **1a**, NHPI **2**, and pyridine (blue) in 0.1 M *n*-Bu_4NBF_4 / MeCN (dashed line) and 0.1 M [pyH]ClO_4 / MeCN (solid line) **(c)** (working glassy-carbon electrode (d = 3 mm), scan rate 0.1 V/s)

3. Copies of NMR Spectra

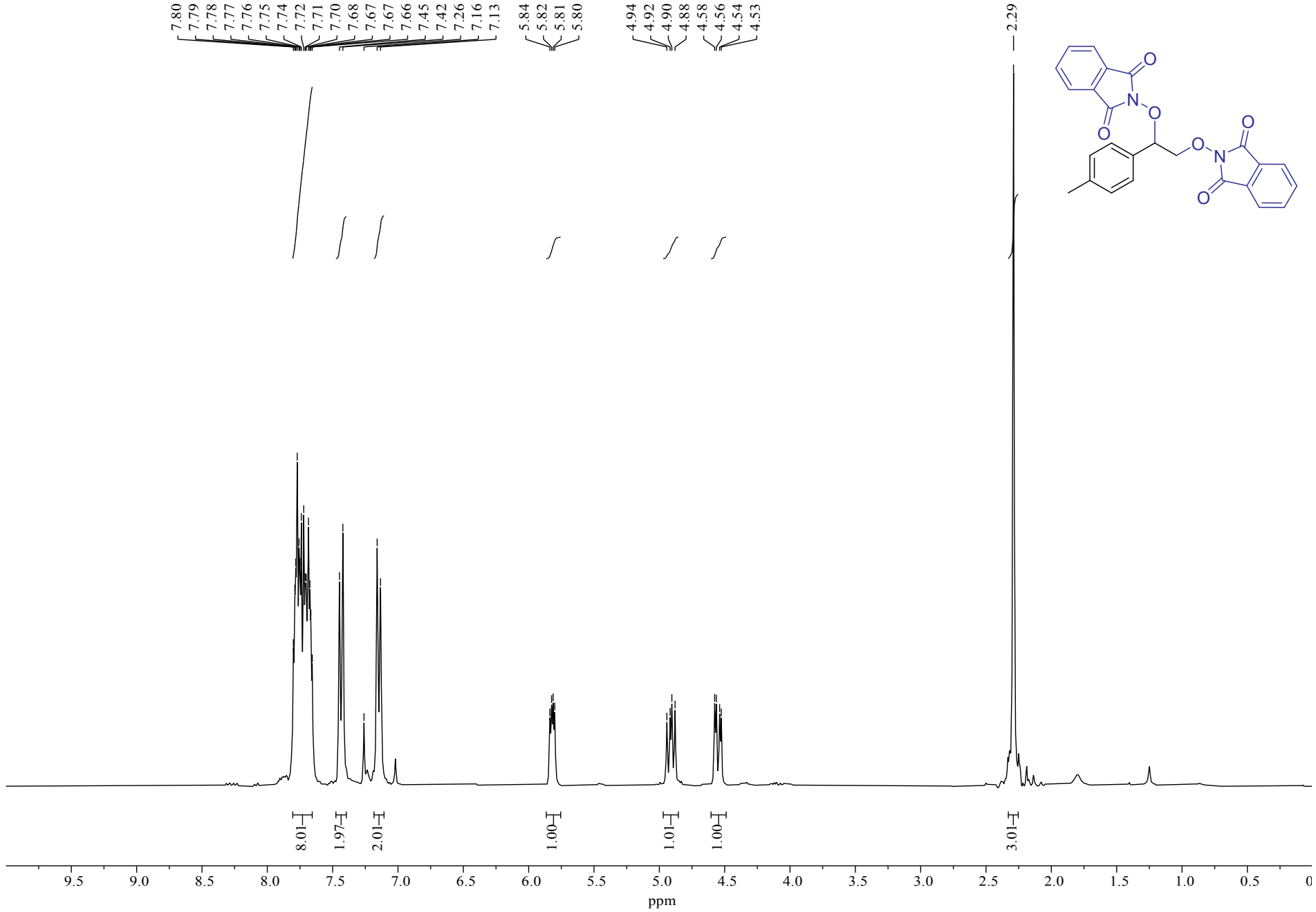
^{1}H NMR spectra of 3a [300.13 MHz, $CDCl_3$]



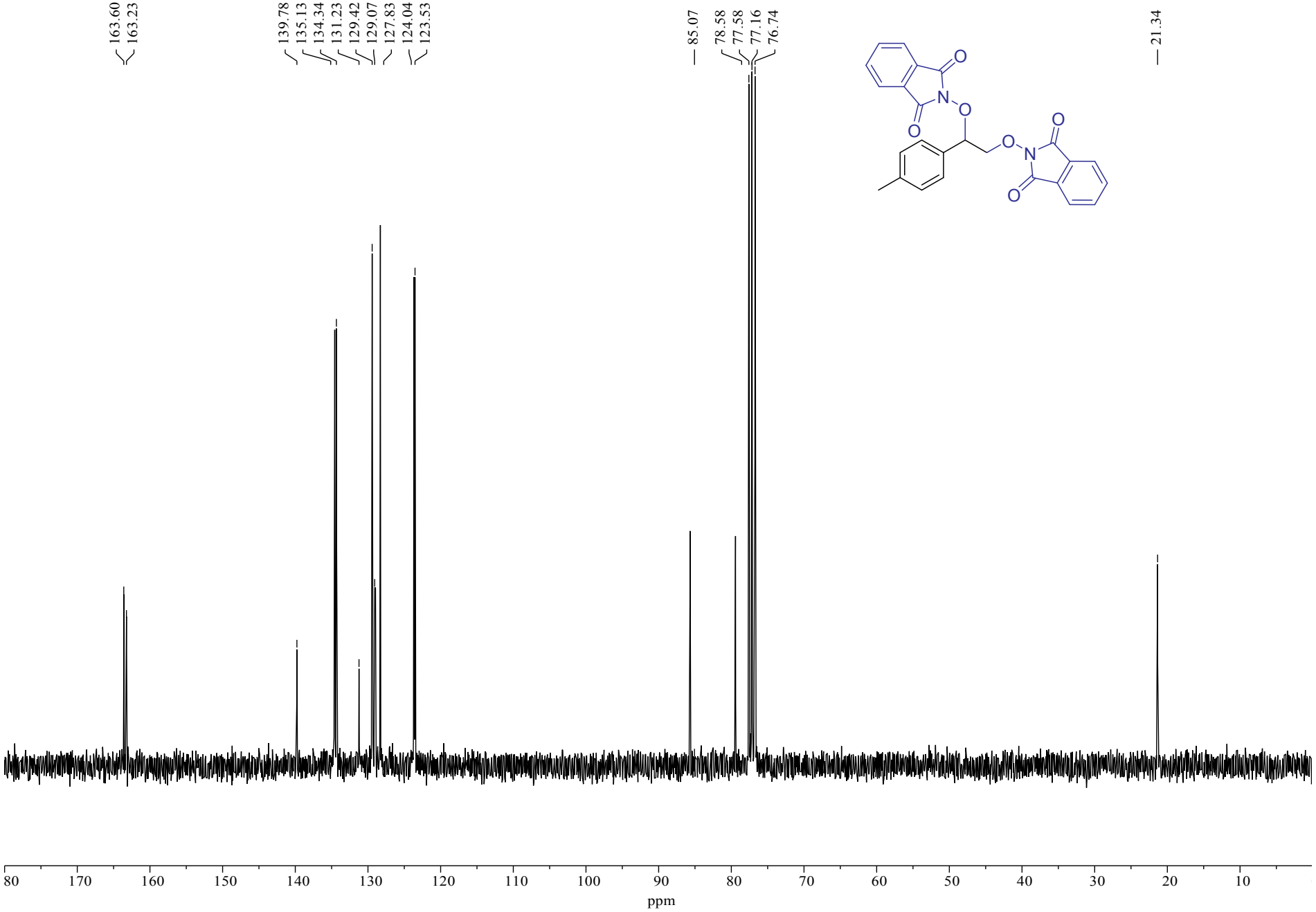
$^{13}C\{^{1}H\}$ NMR spectra of 3a [75.47 MHz, $CDCl_3$]



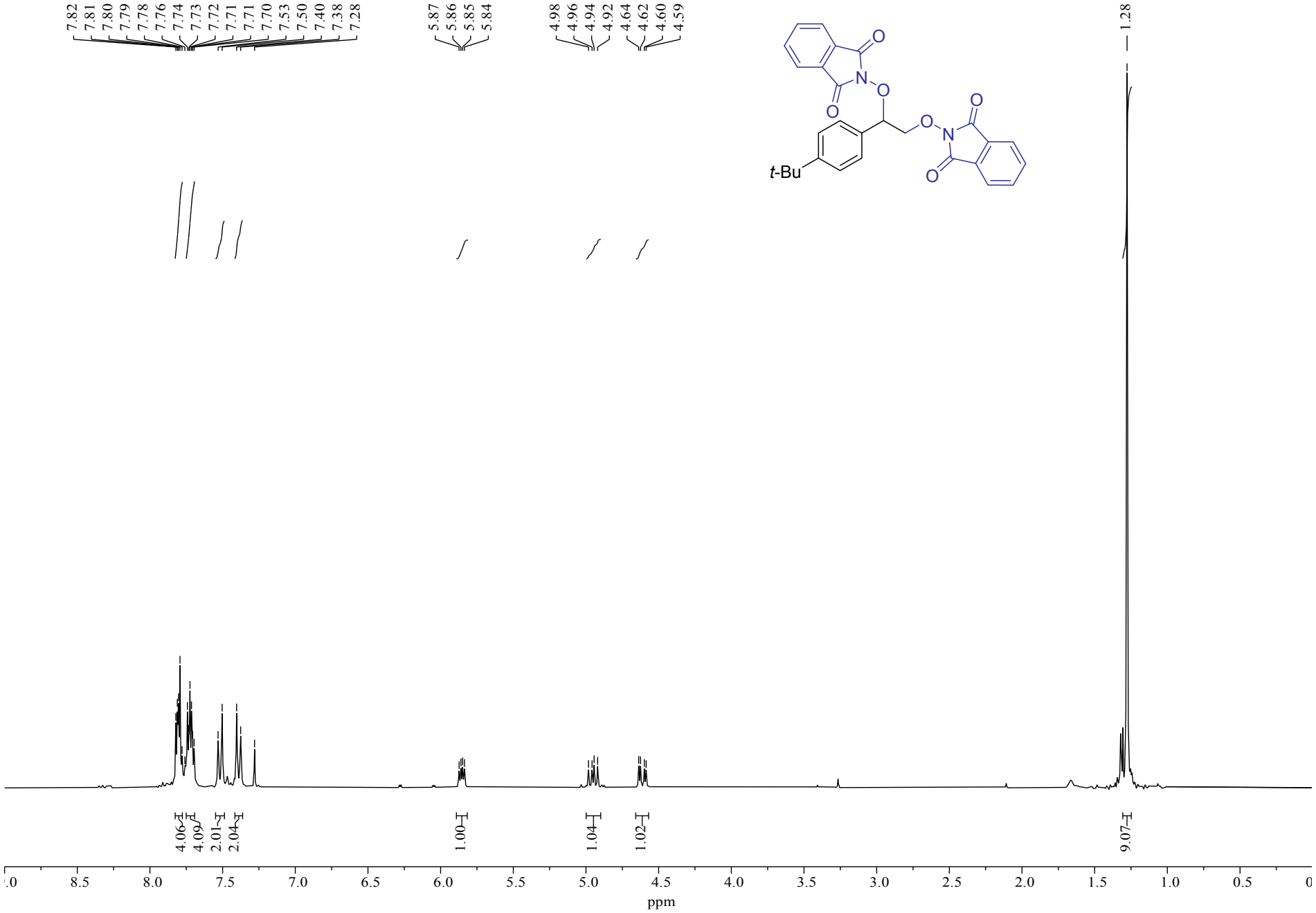
^{1}H NMR spectra of 3b [300.13 MHz, $CDCl_3$]



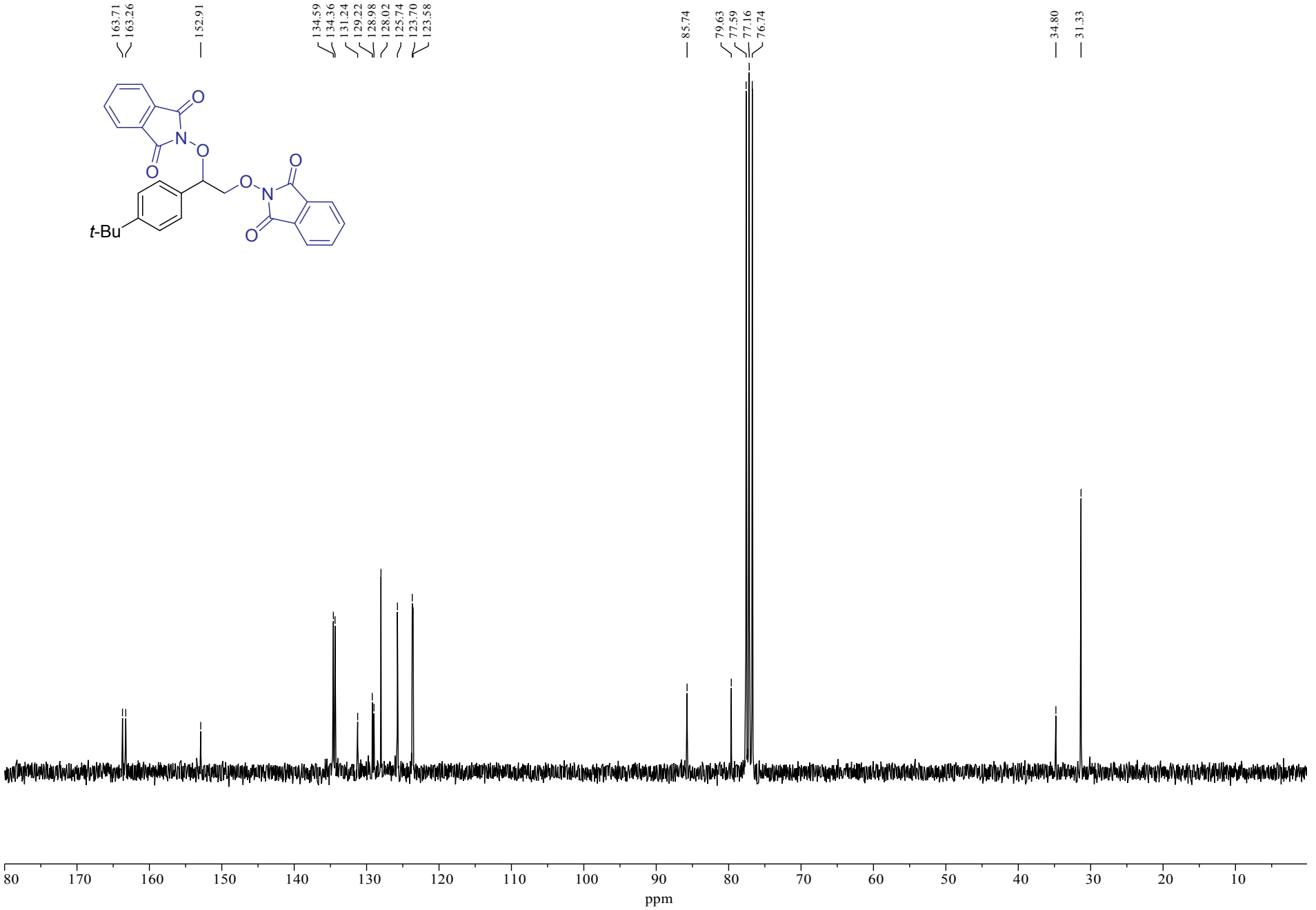
$^{13}C\{^{1}H\}$ NMR spectra of 3b [75.47 MHz, $CDCl_3$]



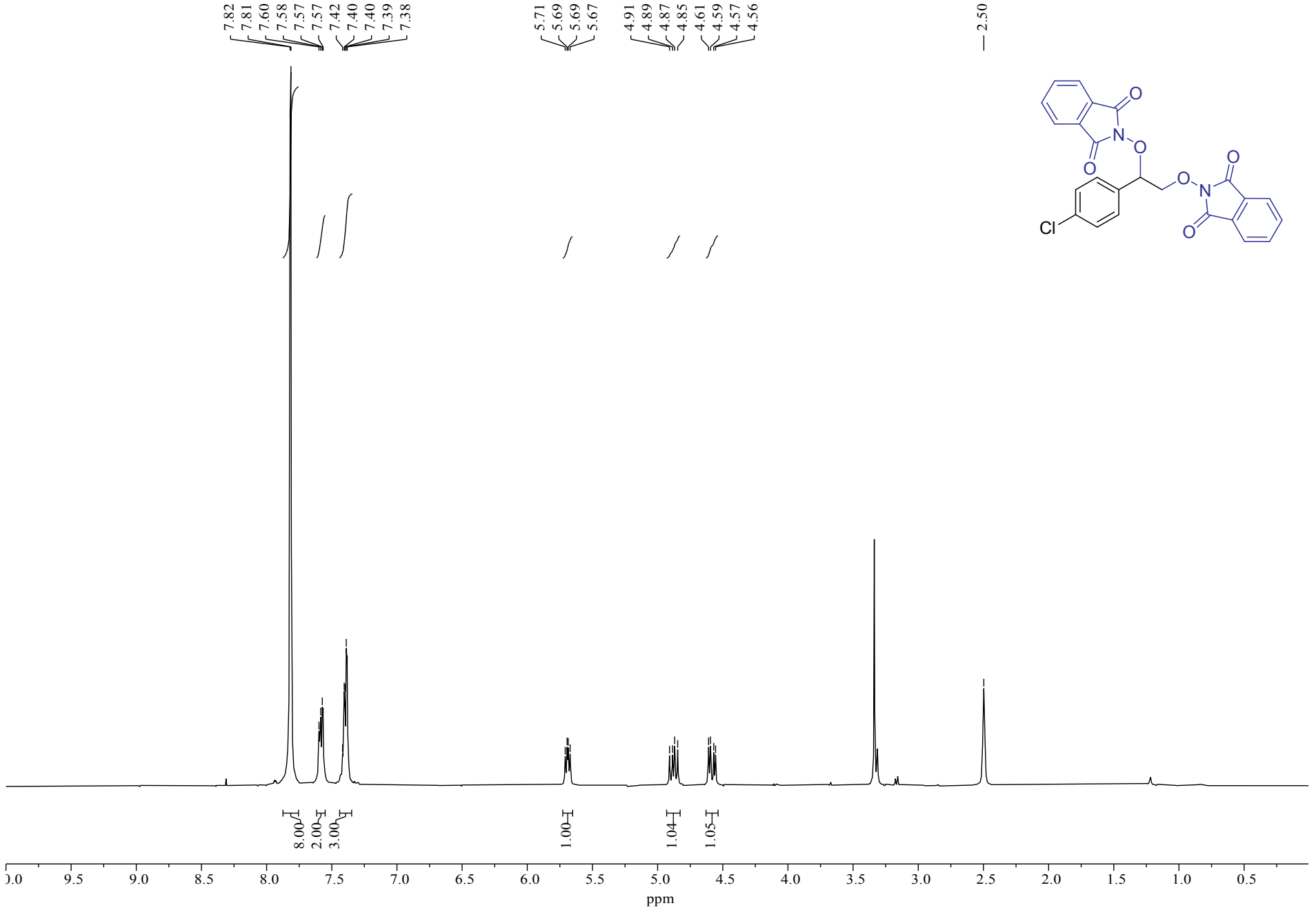
^{1}H NMR spectra of 3c [300.13 MHz, $CDCl_3$]



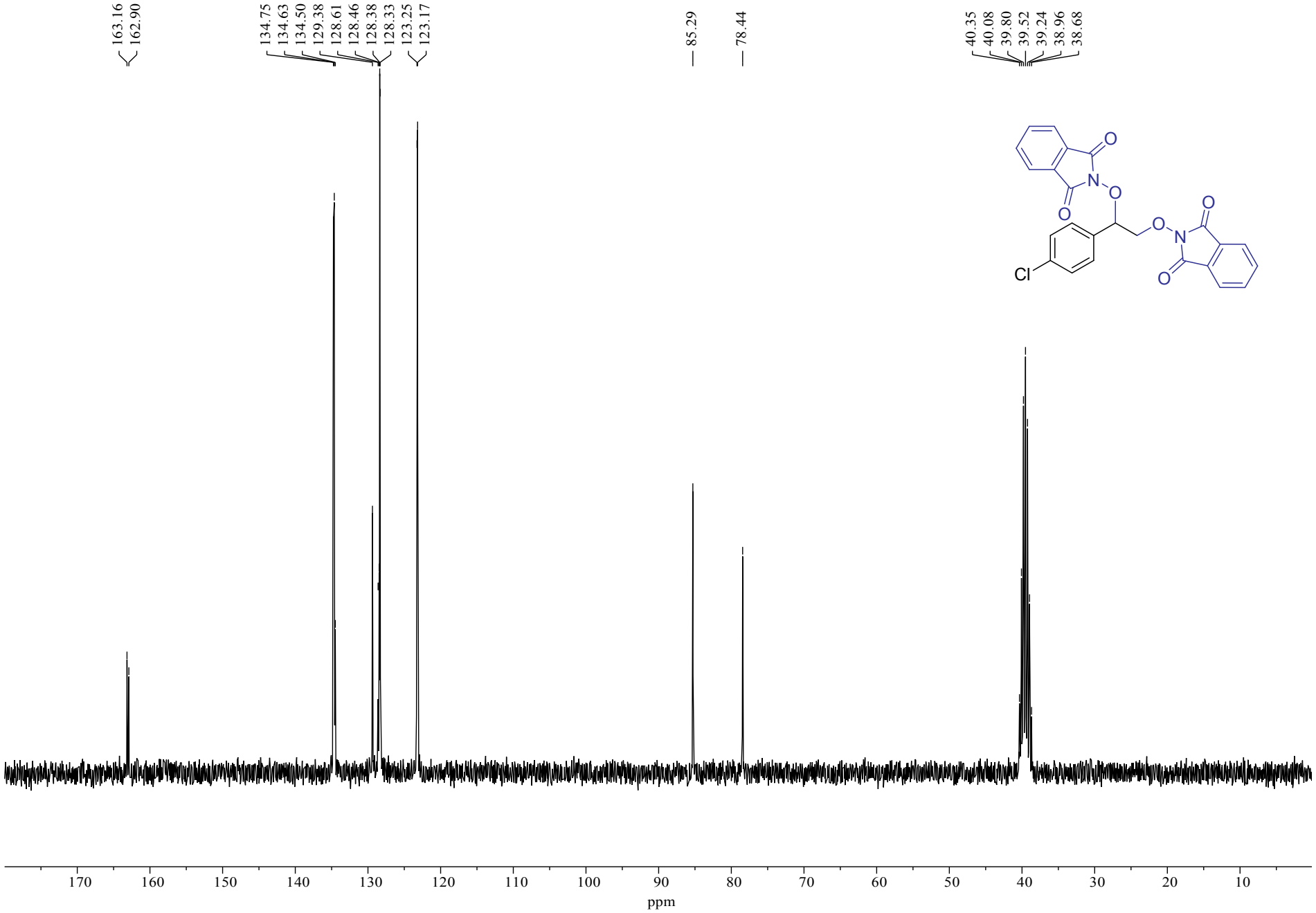
$^{13}C\{^{1}H\}$ NMR spectra of 3c [75.47 MHz, $CDCl_3$]



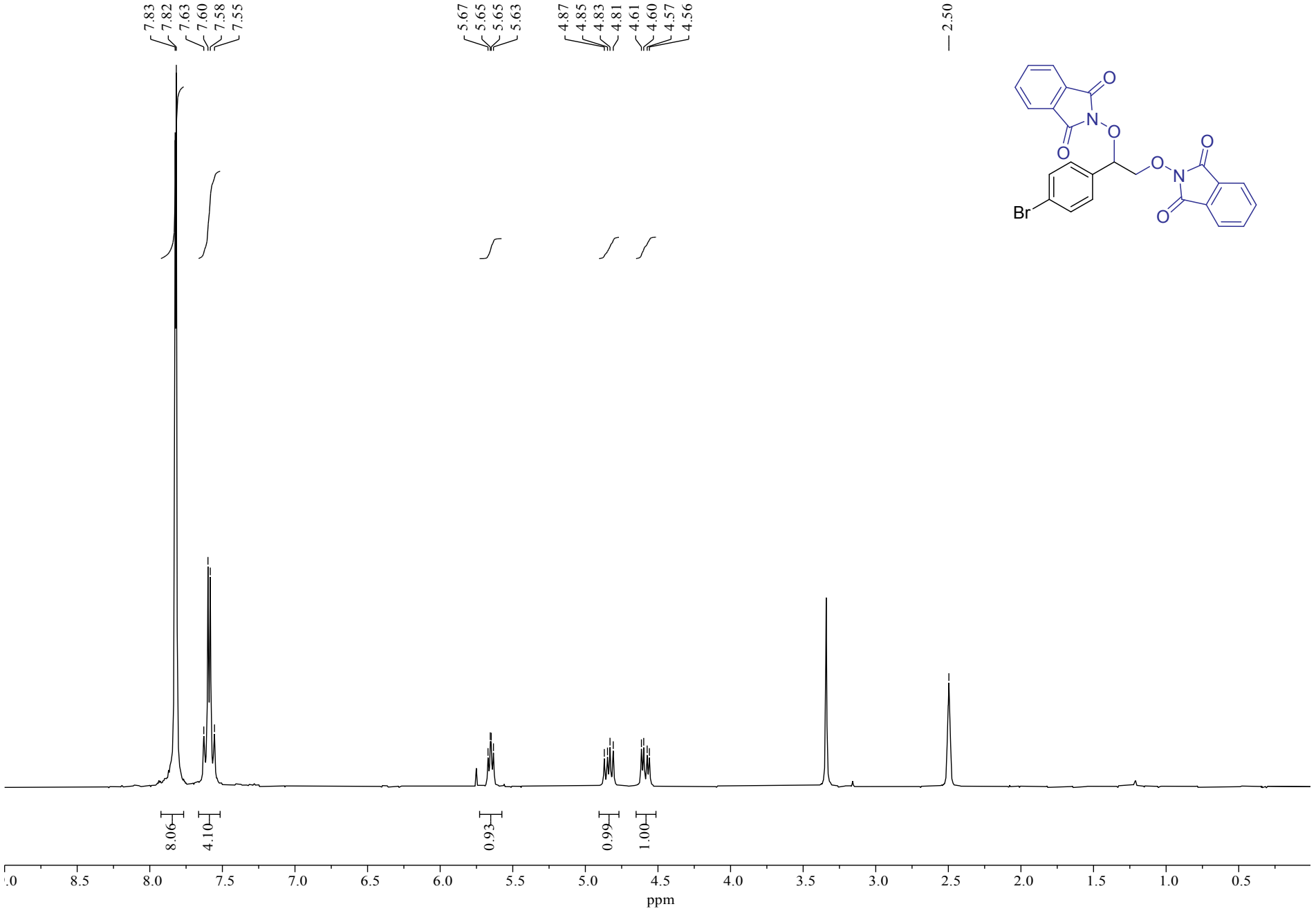
^{1}H NMR spectra of 3d [300.13 MHz, DMSO-d6]



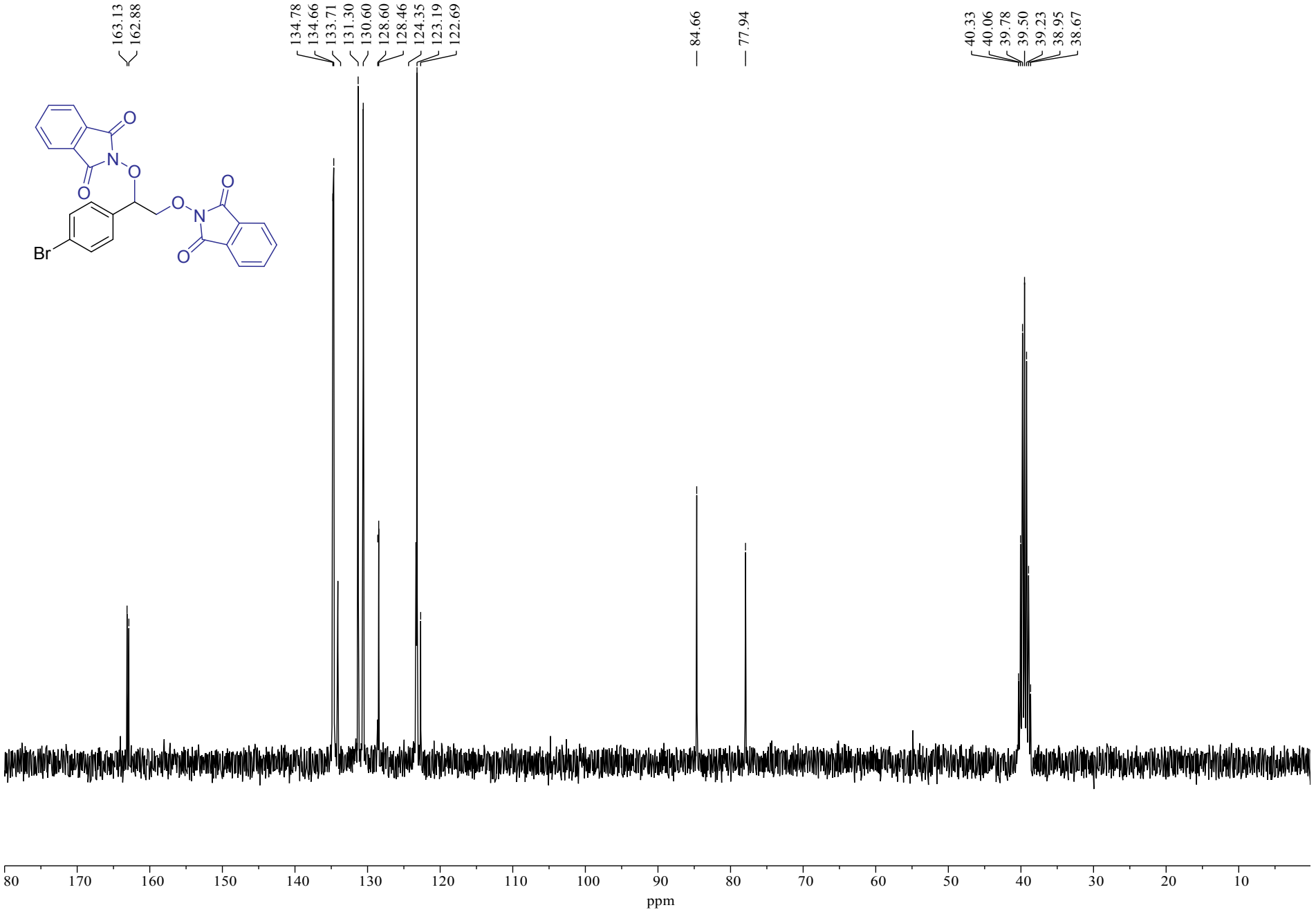
$^{13}C\{^{1}H\}$ NMR spectra of 3d [75.47 MHz, DMSO-d6]



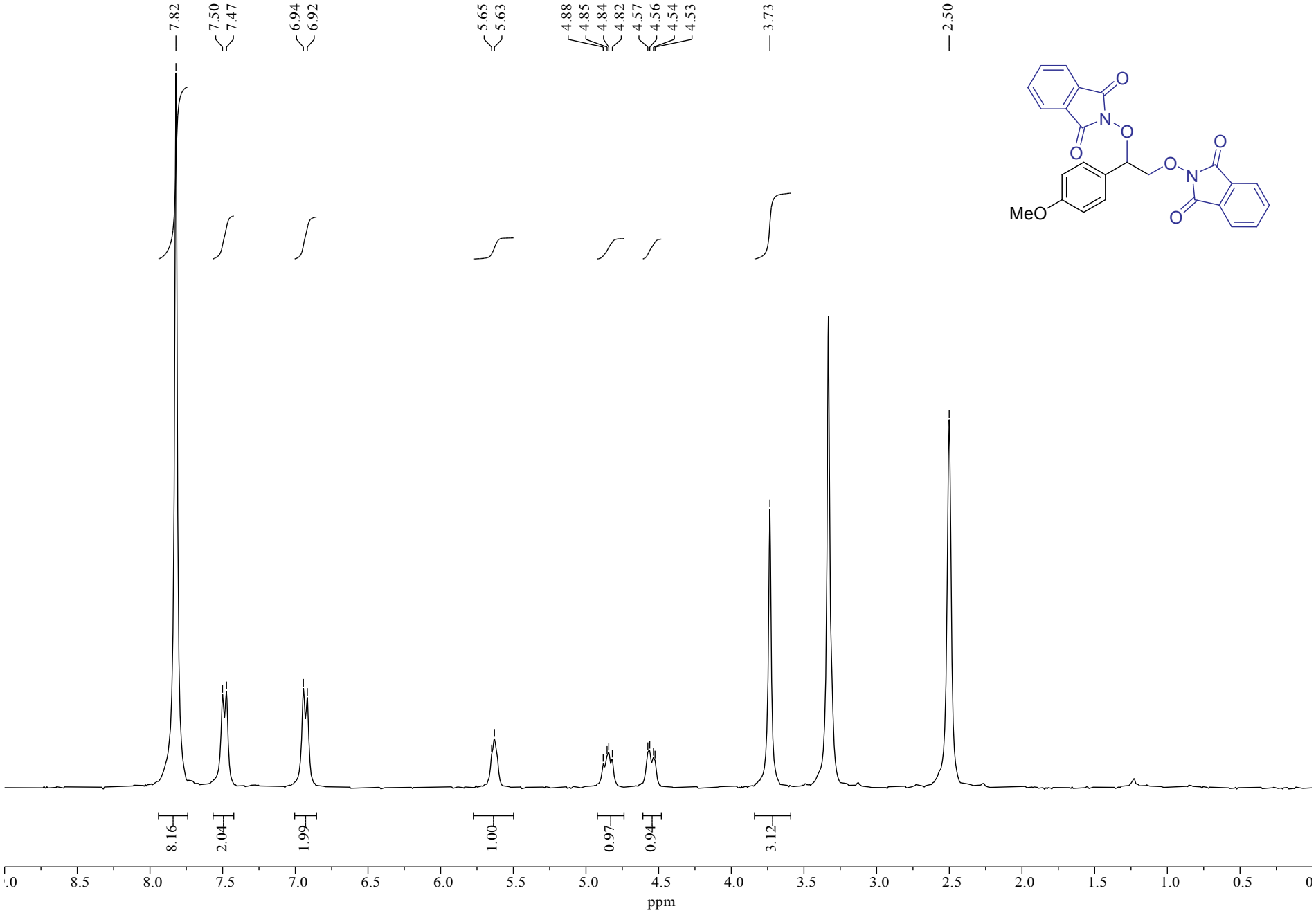
^{1}H NMR spectra of 3e [300.13 MHz, DMSO-d6]



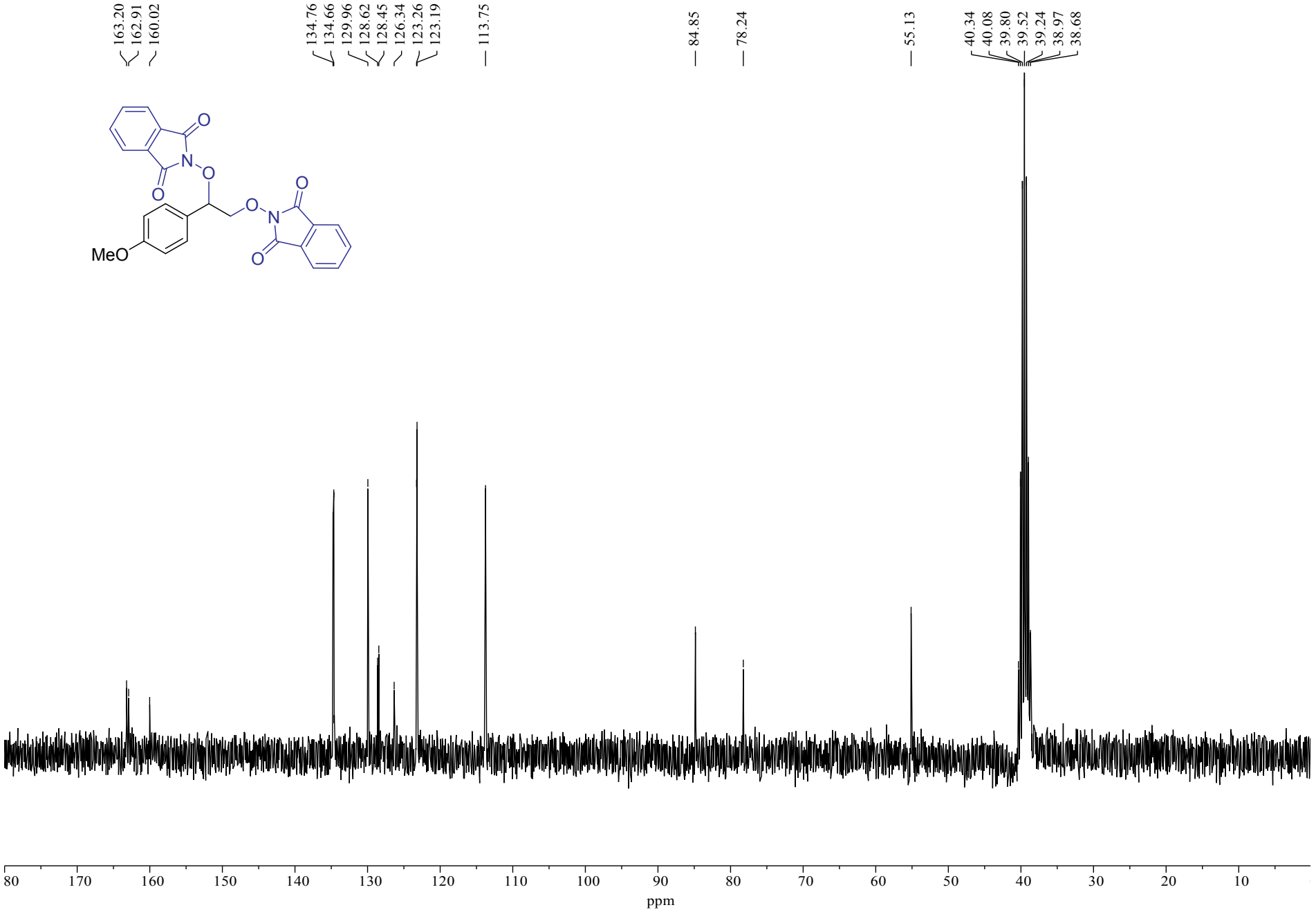
$^{13}C\{^{1}H\}$ NMR spectra of 3e [75.47 MHz, DMSO-d6]



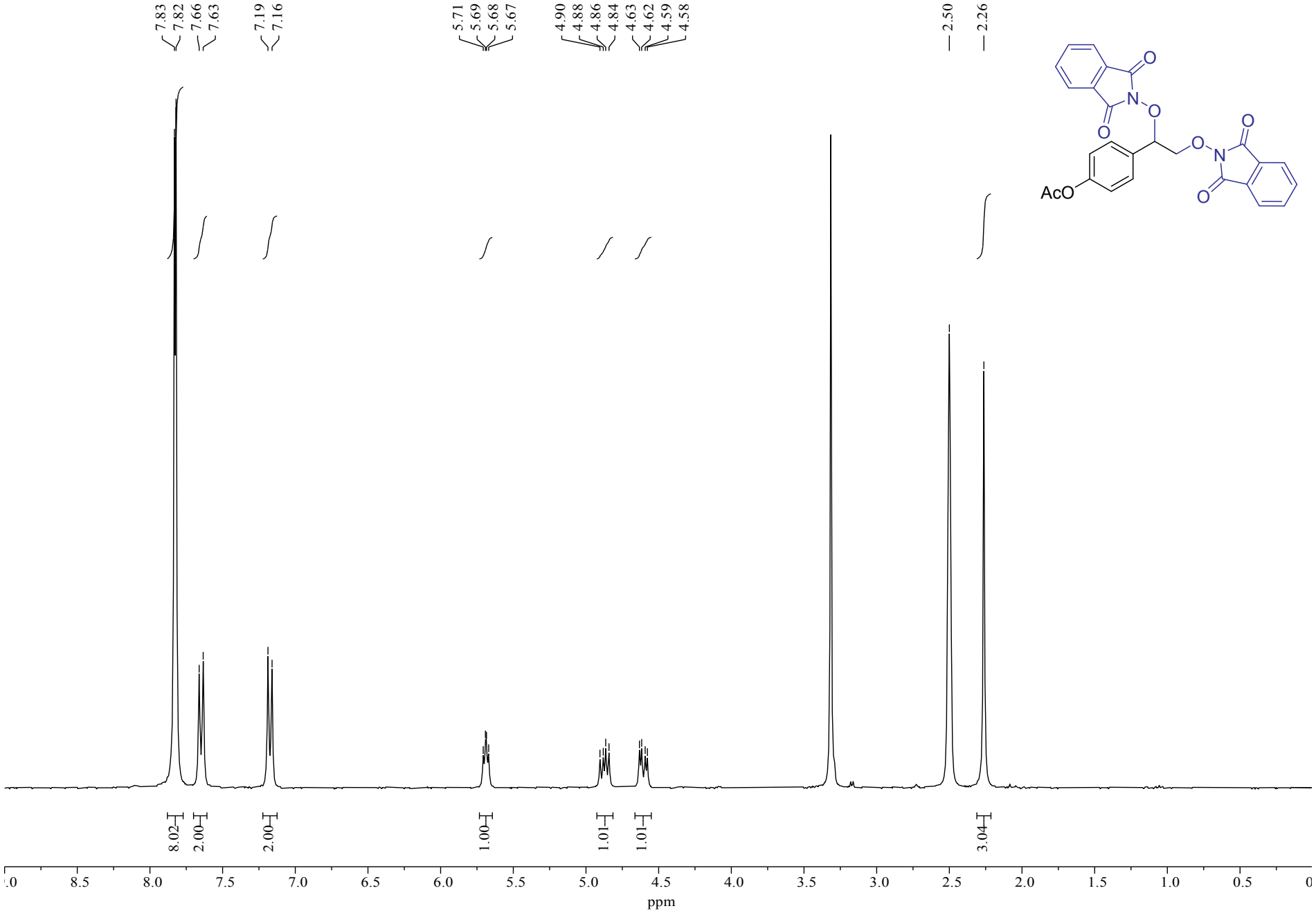
[1]H NMR spectra of 3f [300.13 MHz, DMSO-d6]



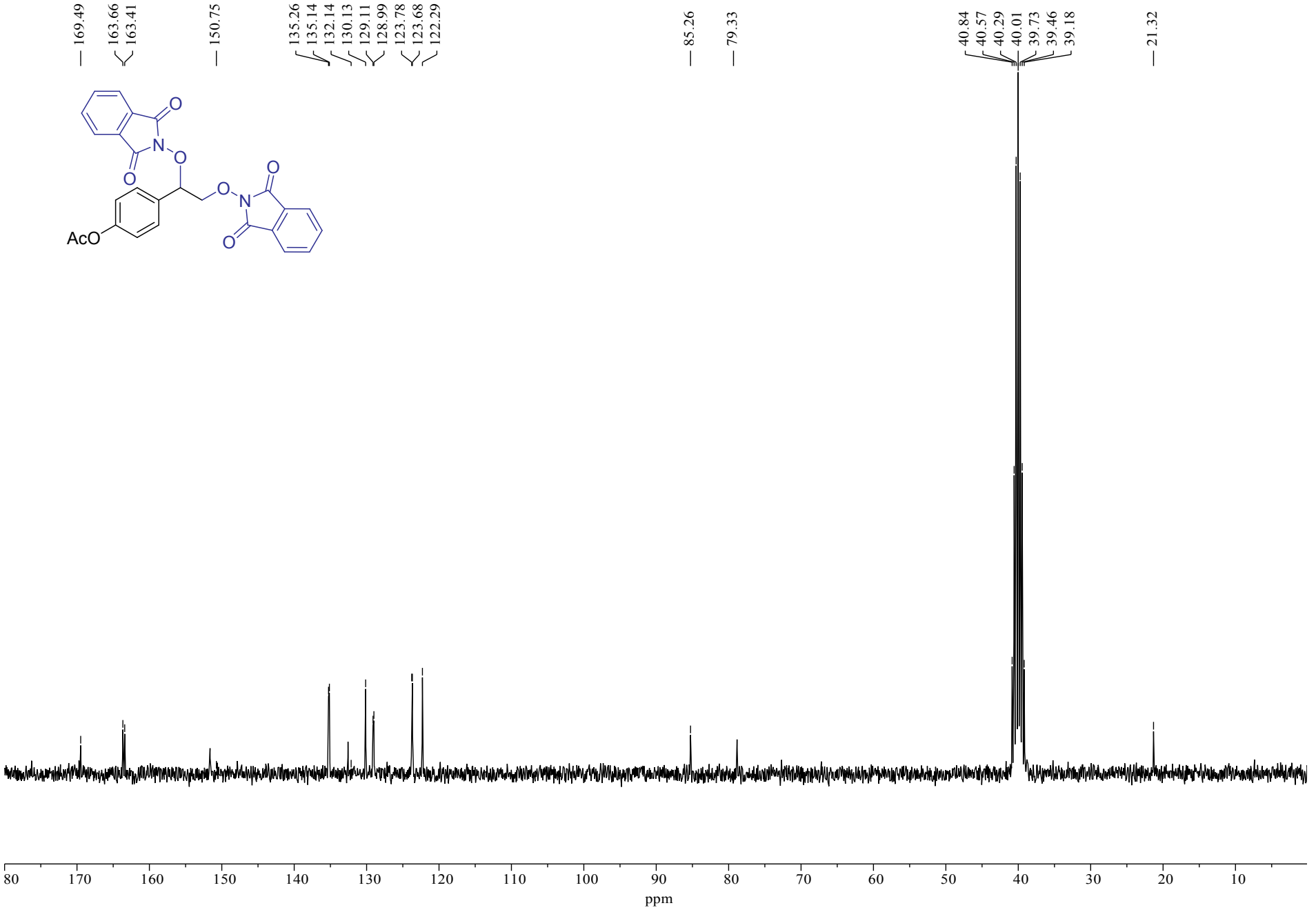
$^{13}C\{^{1}H\}$ NMR spectra of 3f [75.47 MHz, DMSO-d6]



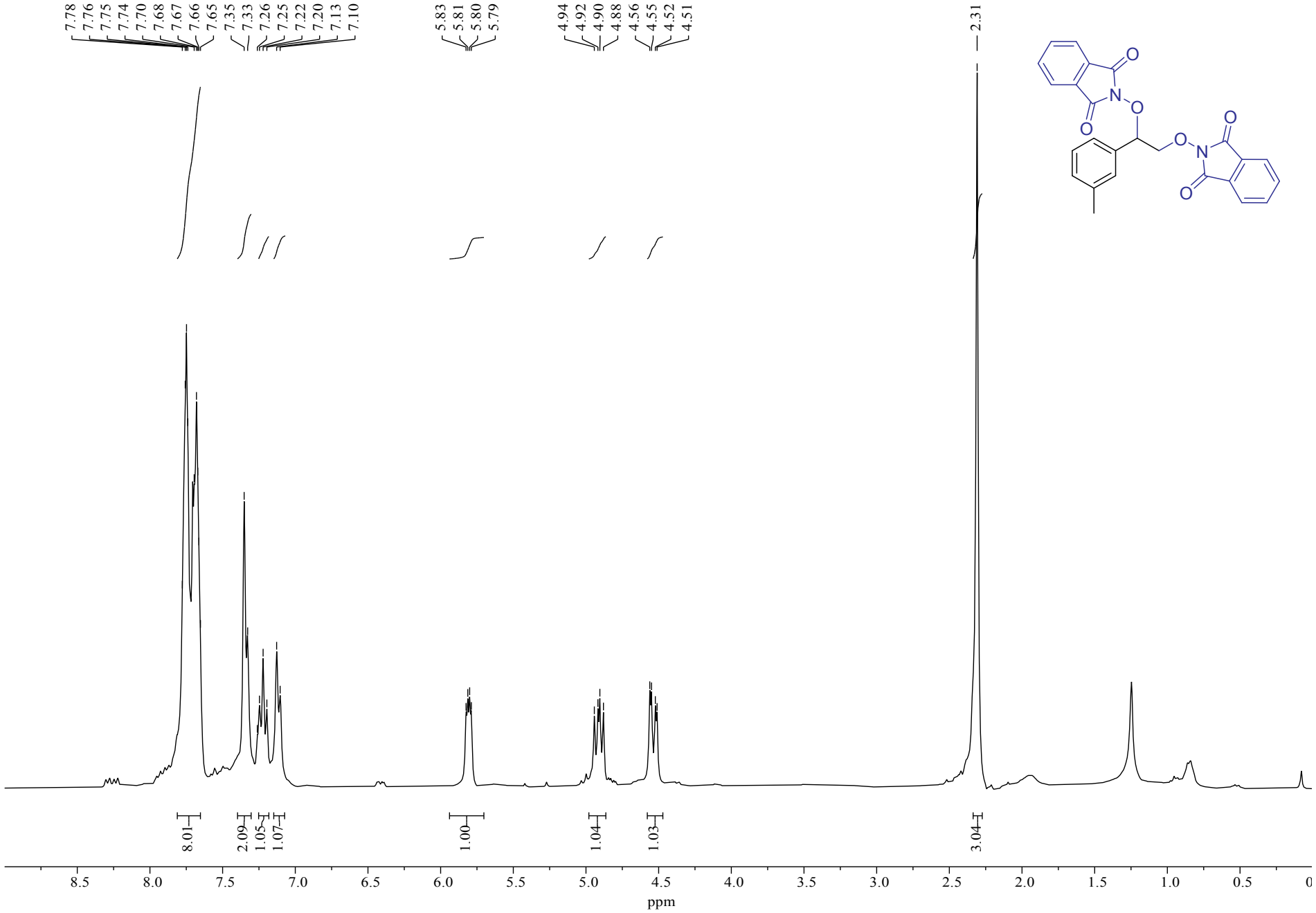
^{1}H NMR spectra of 3g [300.13 MHz, DMSO-d6]



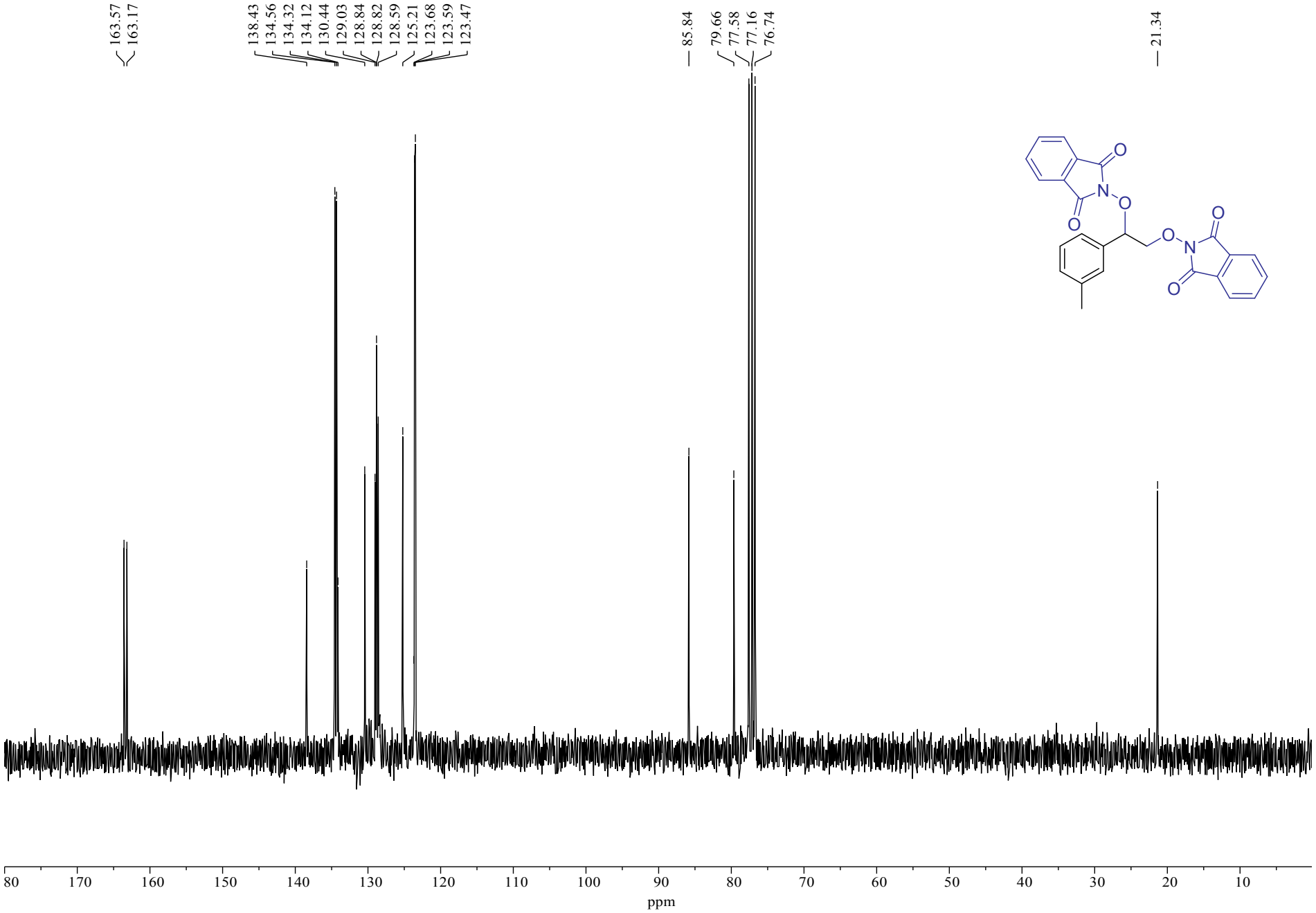
$^{13}C\{^{1}H\}$ NMR spectra of 3g [75.47 MHz, DMSO-d6]



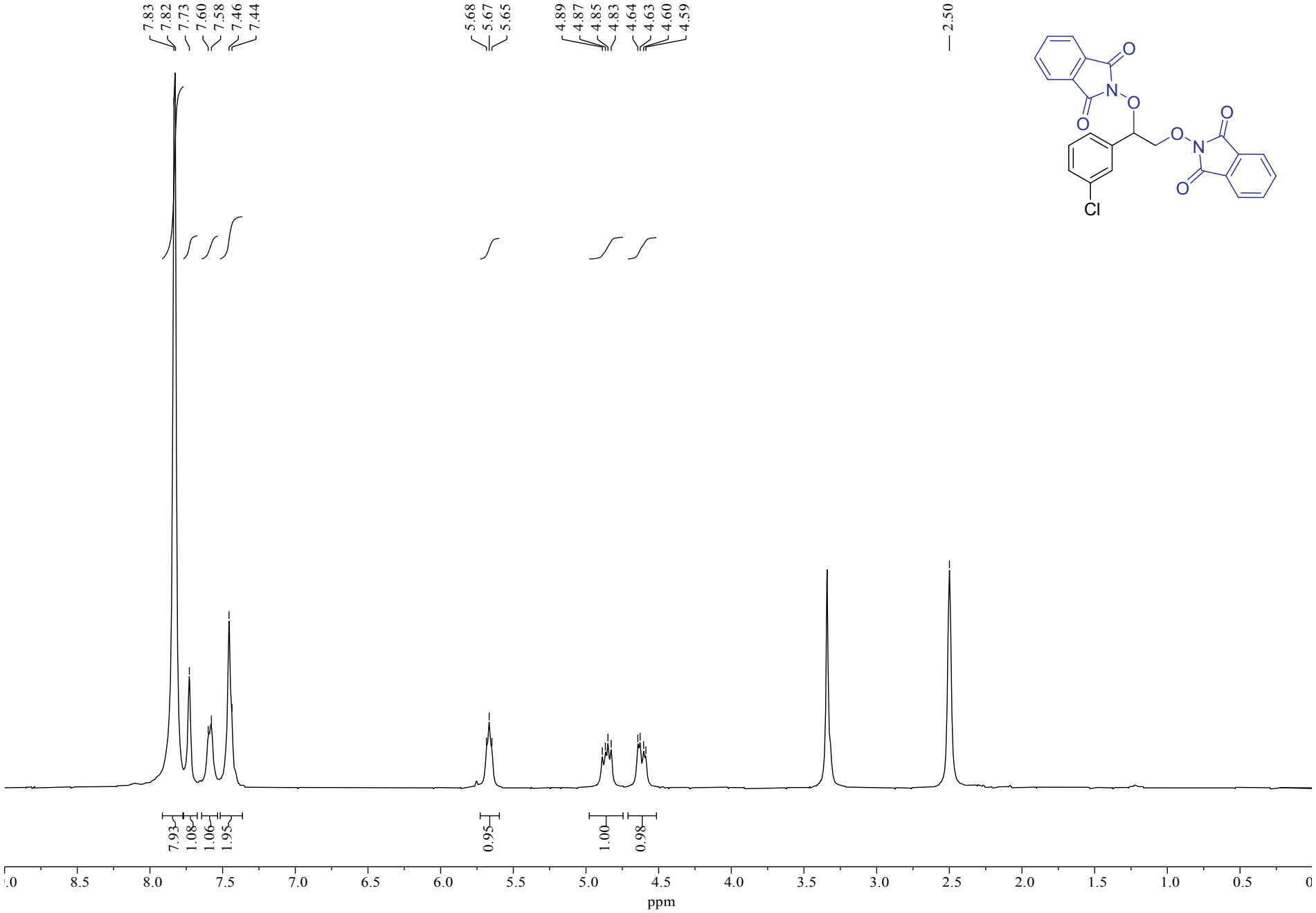
^{1}H NMR spectra of 3h [300.13 MHz, $CDCl_3$]



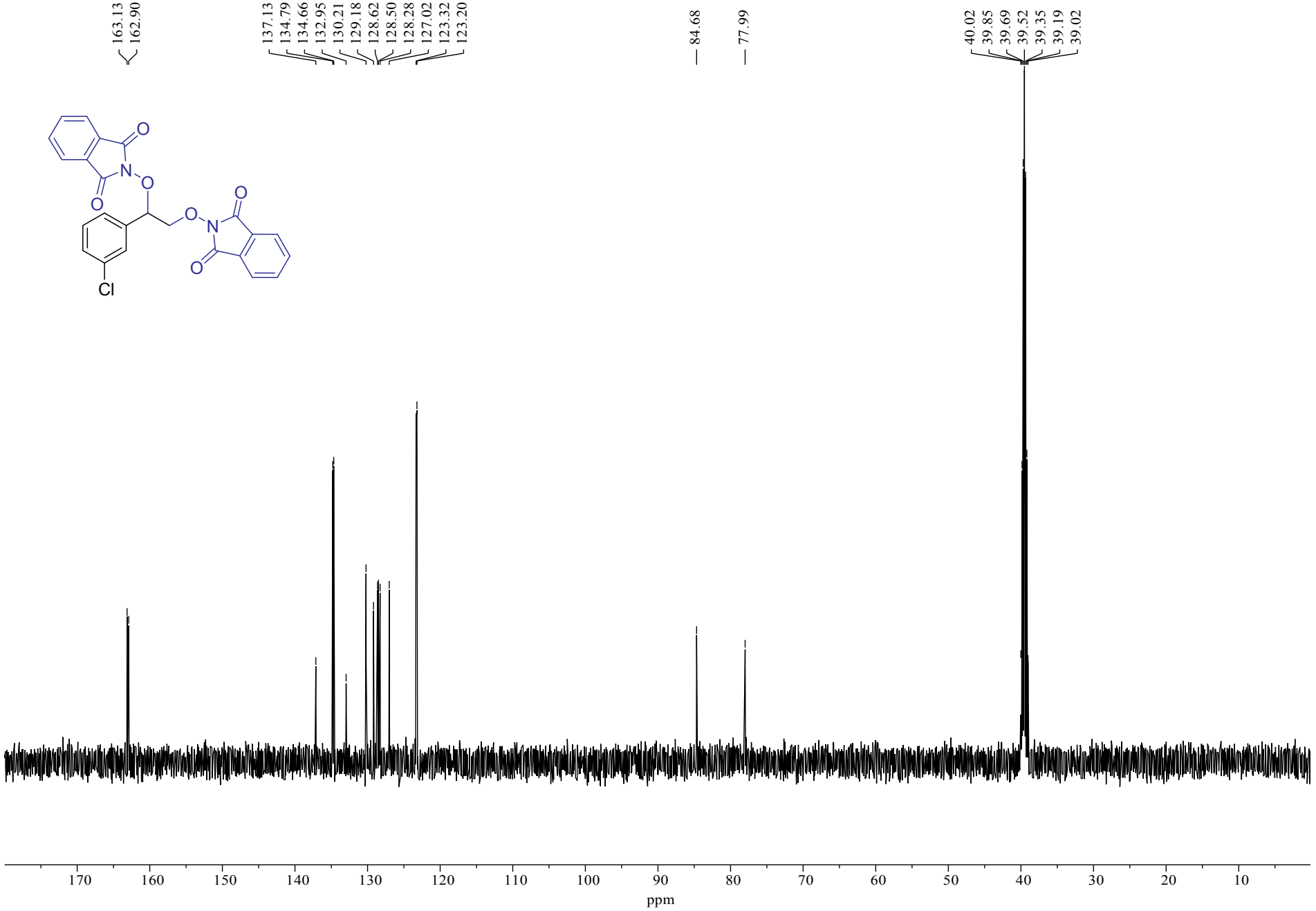
$^{13}C\{^{1}H\}$ NMR spectra of 3h [75.47 MHz, $CDCl_3$]



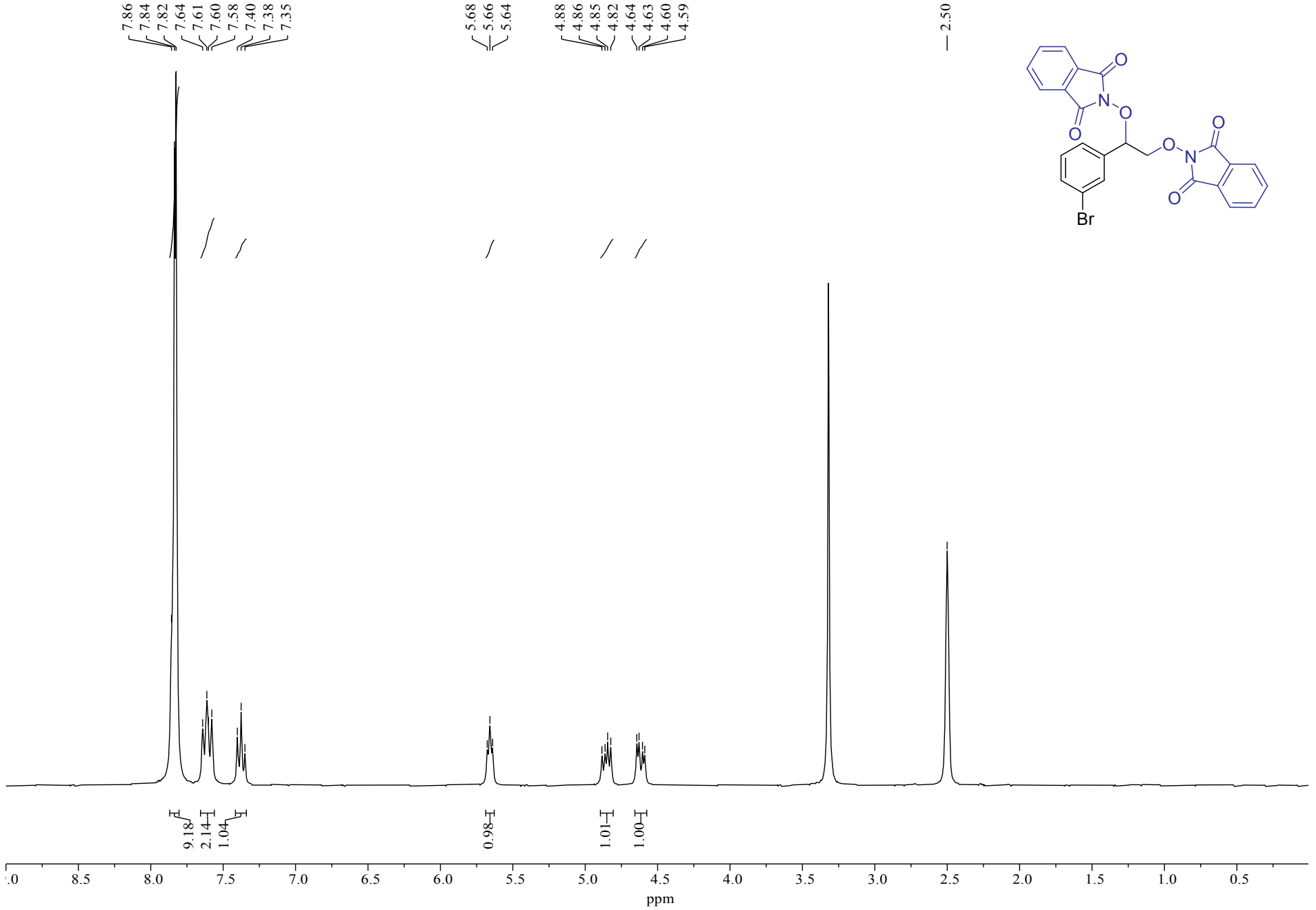
^{1}H NMR spectra of 3i [300.13 MHz, DMSO-d6]



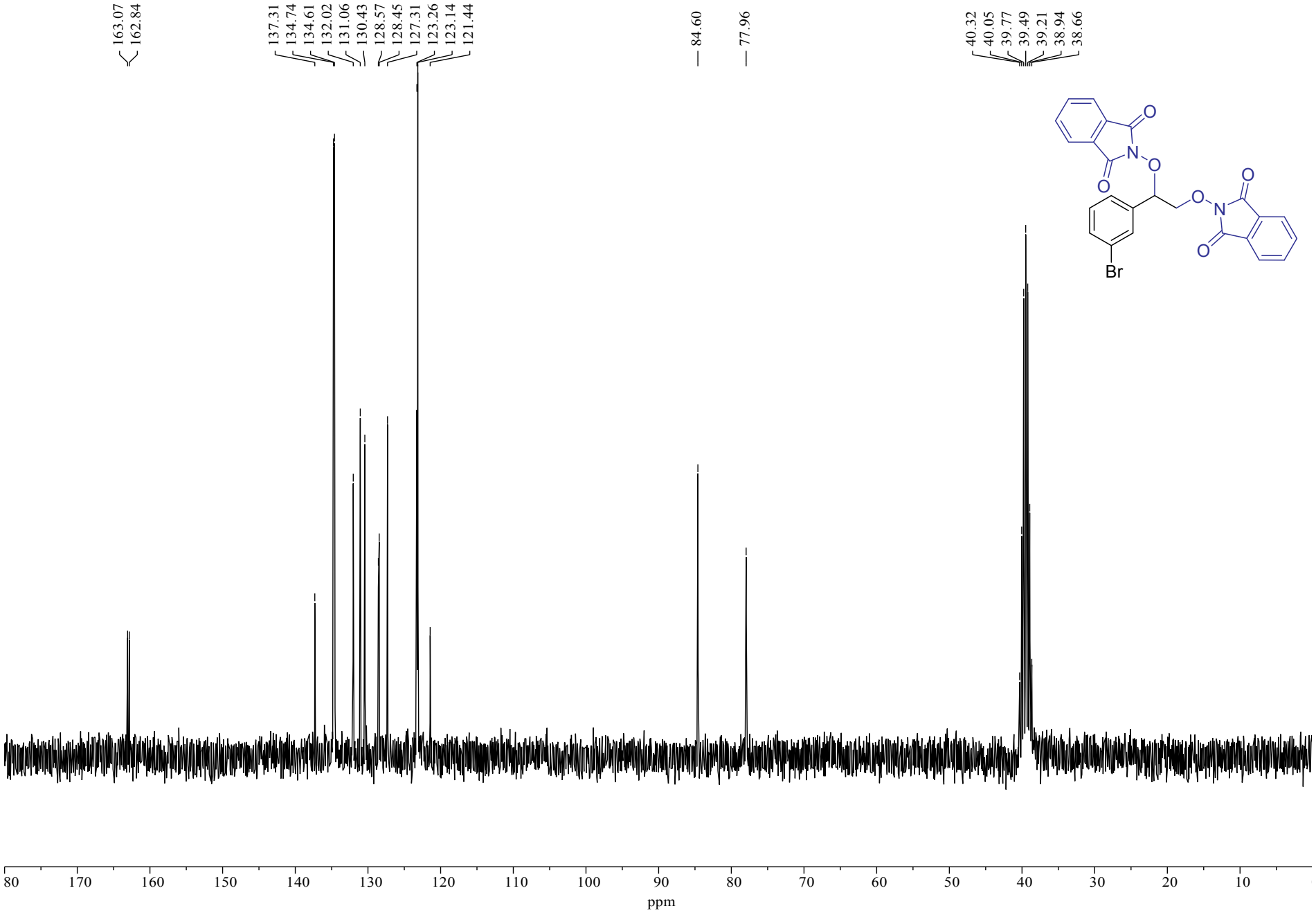
$^{13}C\{^{1}H\}$ NMR spectra of 3i [75.47 MHz, DMSO-d6]



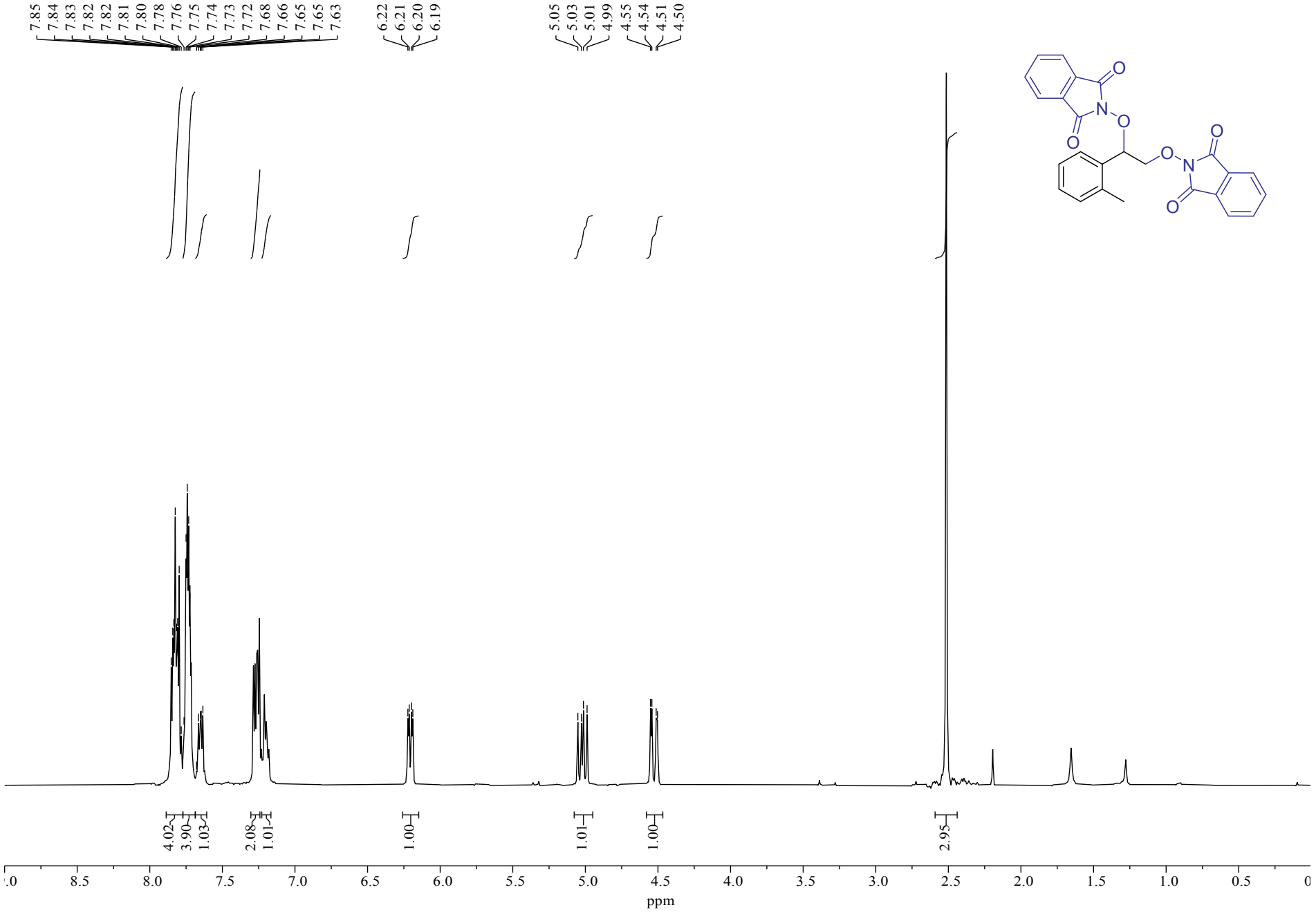
^{1}H NMR spectra of 3j [300.13 MHz, DMSO-d6]



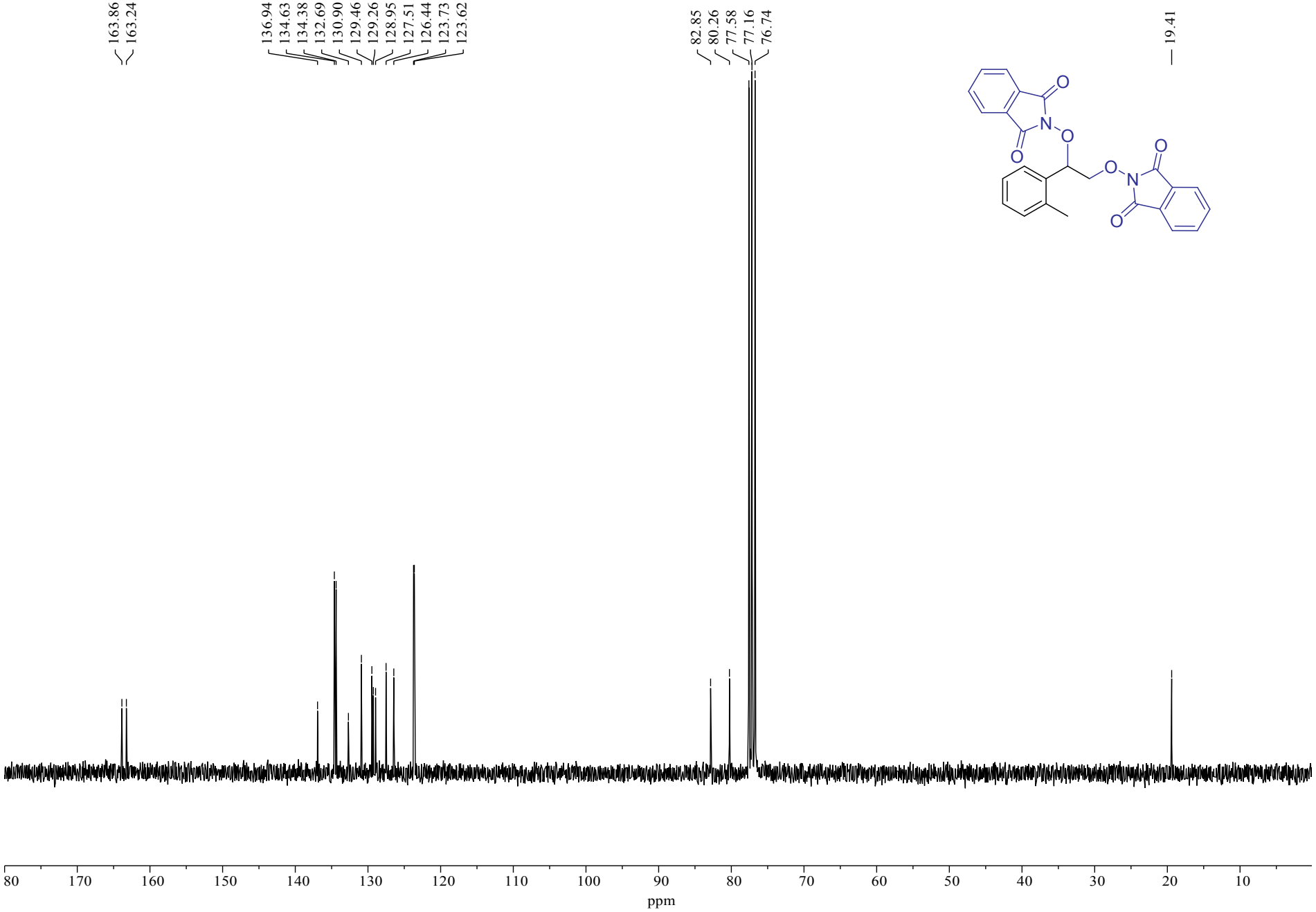
$^{13}C\{^{1}H\}$ NMR spectra of 3j [75.47 MHz, DMSO-d6]



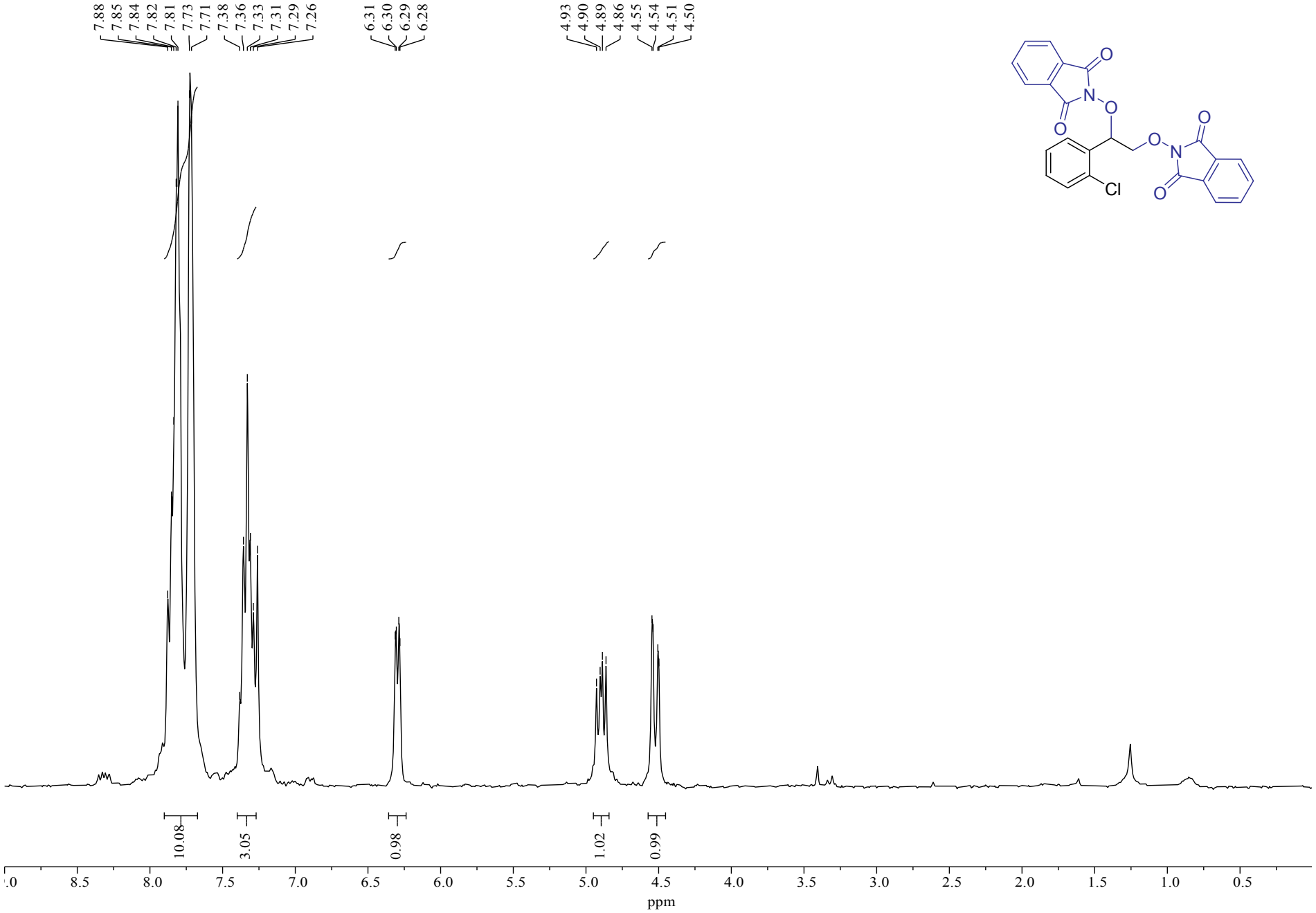
^{1}H NMR spectra of 3k [300.13 MHz, $CDCl_3$]



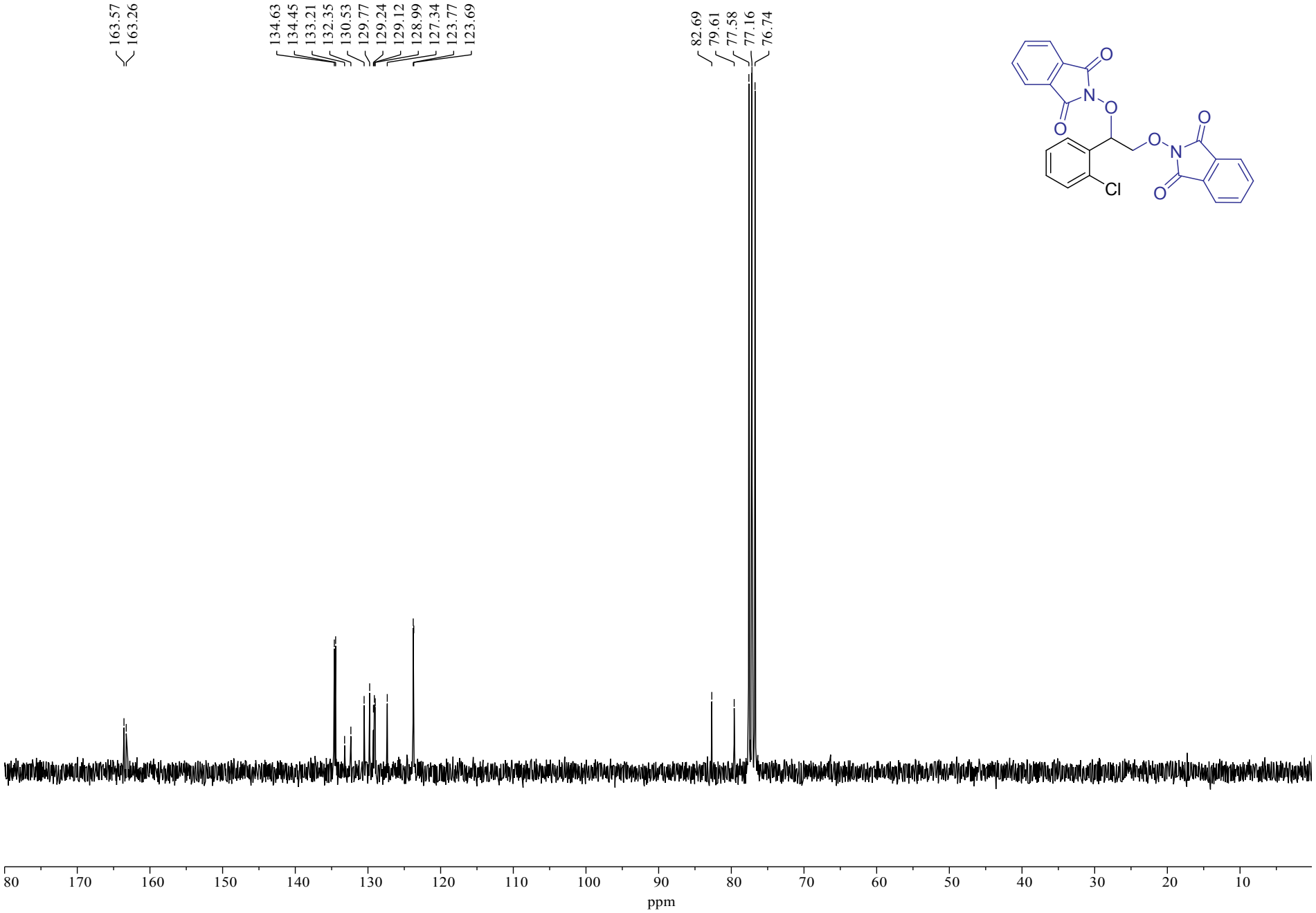
$^{13}C\{^{1}H\}$ NMR spectra of 3k [75.47 MHz, $CDCl_3$]



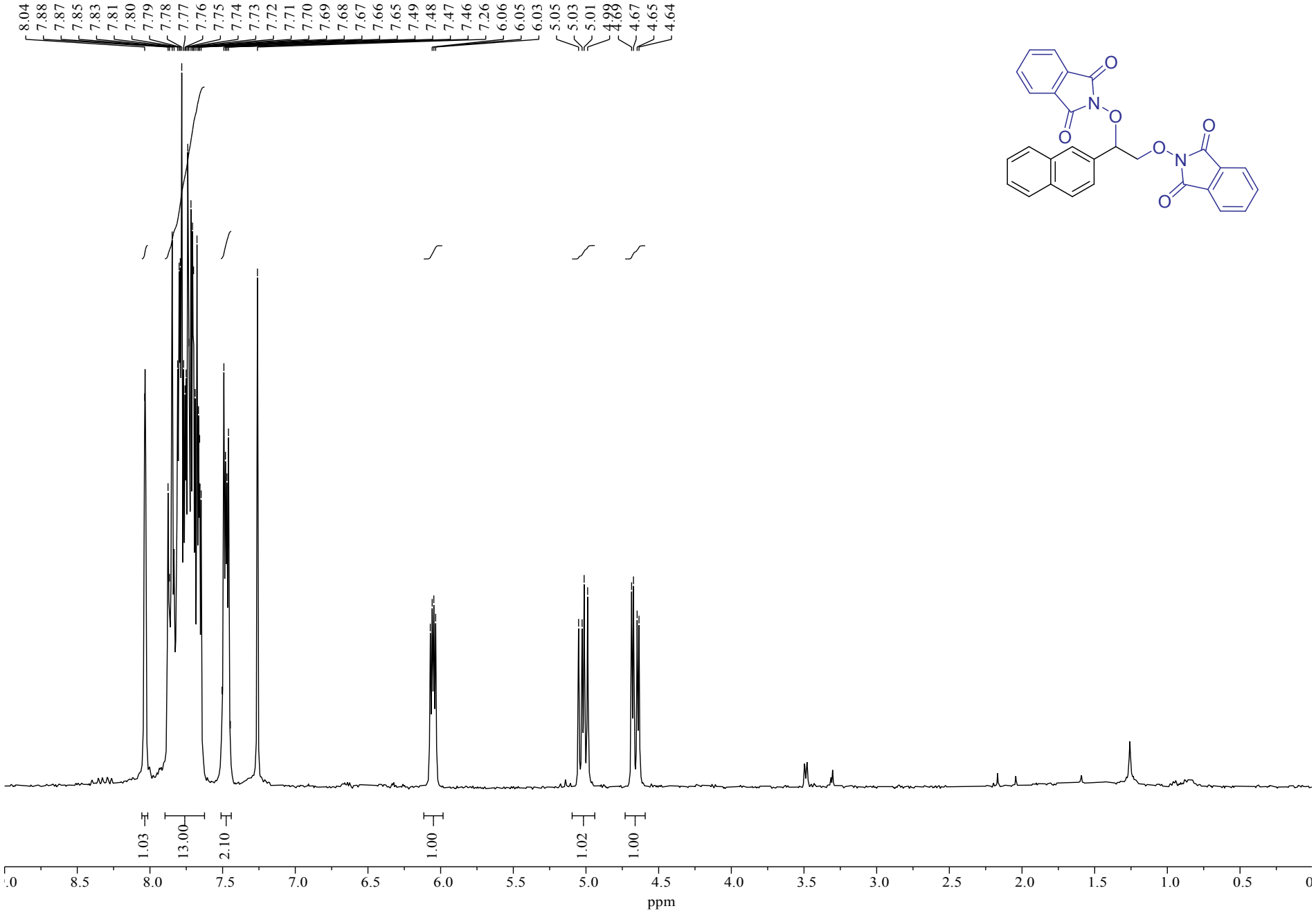
^{1}H NMR spectra of 3l [300.13 MHz, $CDCl_3$]



$^{13}C\{^{1}H\}$ NMR spectra of 3l [75.47 MHz, $CDCl_3$]



^{1}H NMR spectra of 3m [300.13 MHz, $CDCl_3$]



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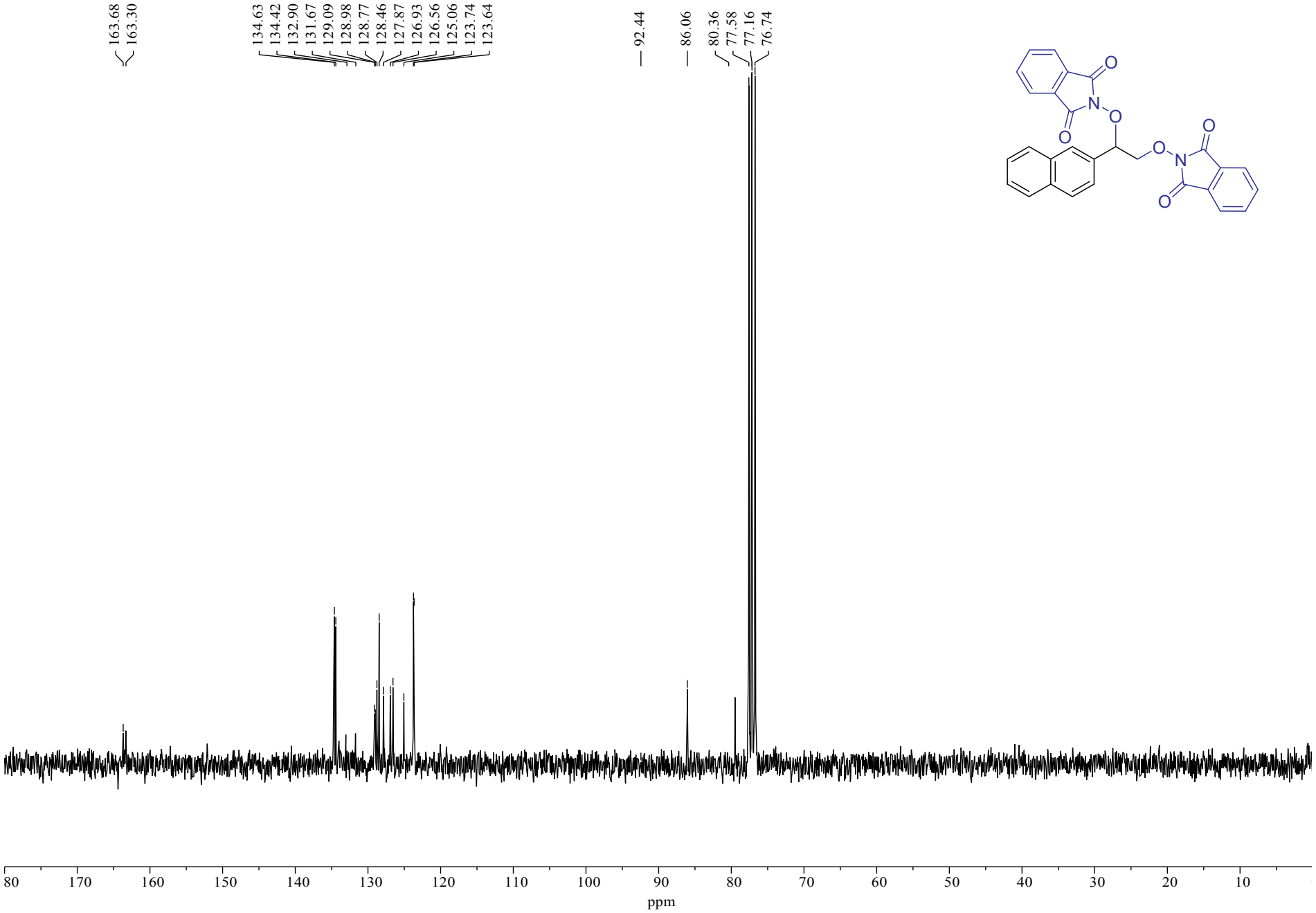
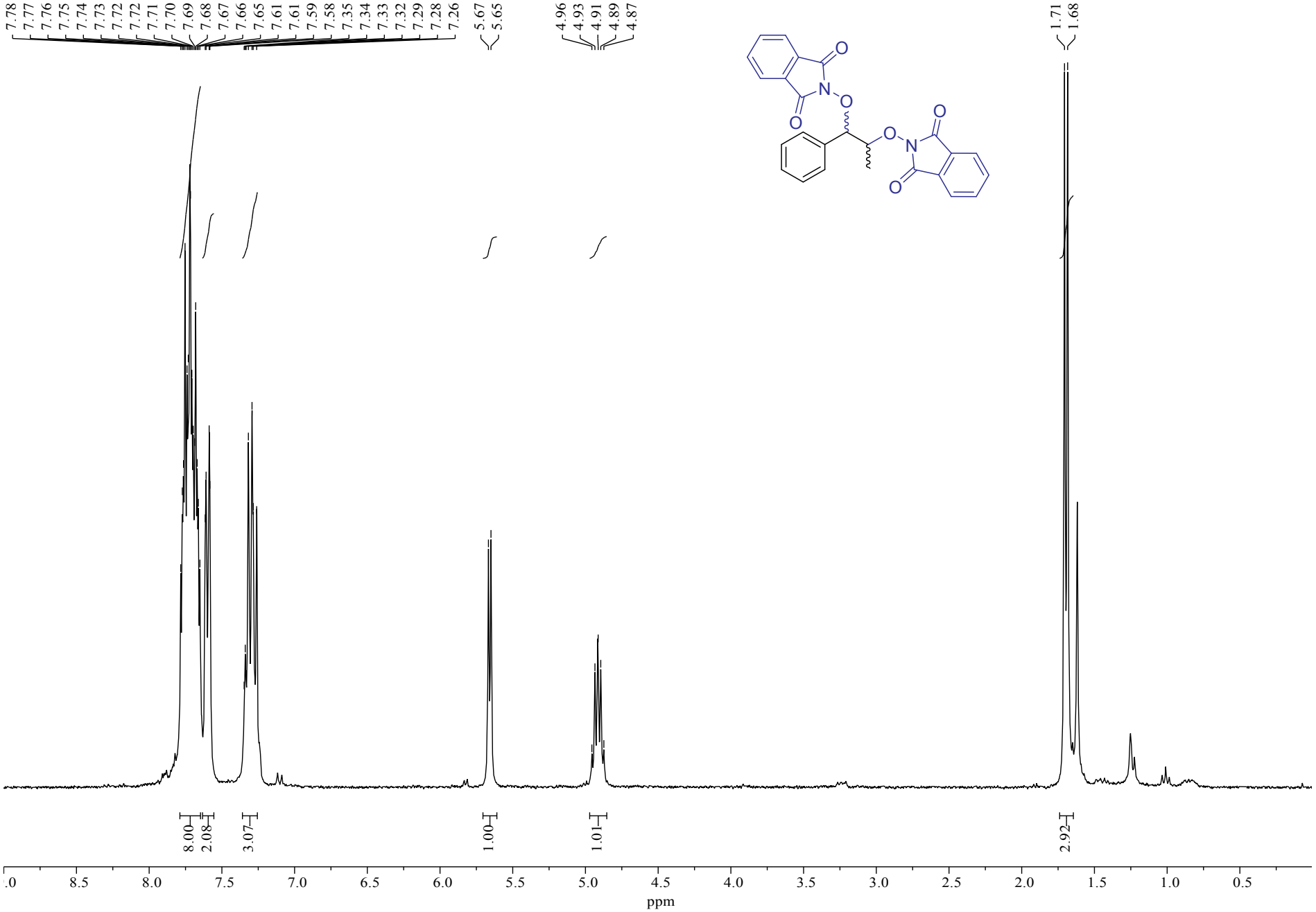
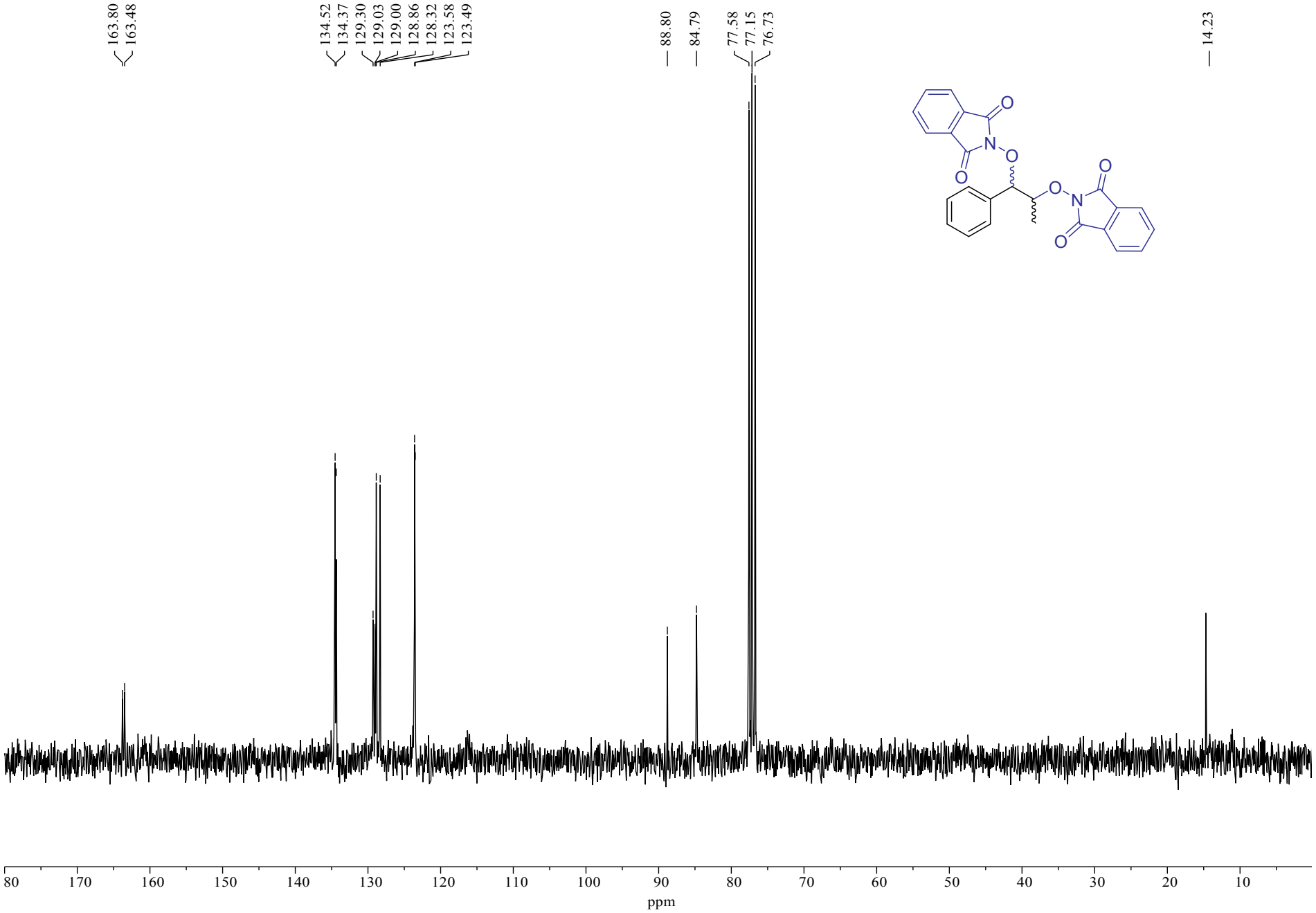


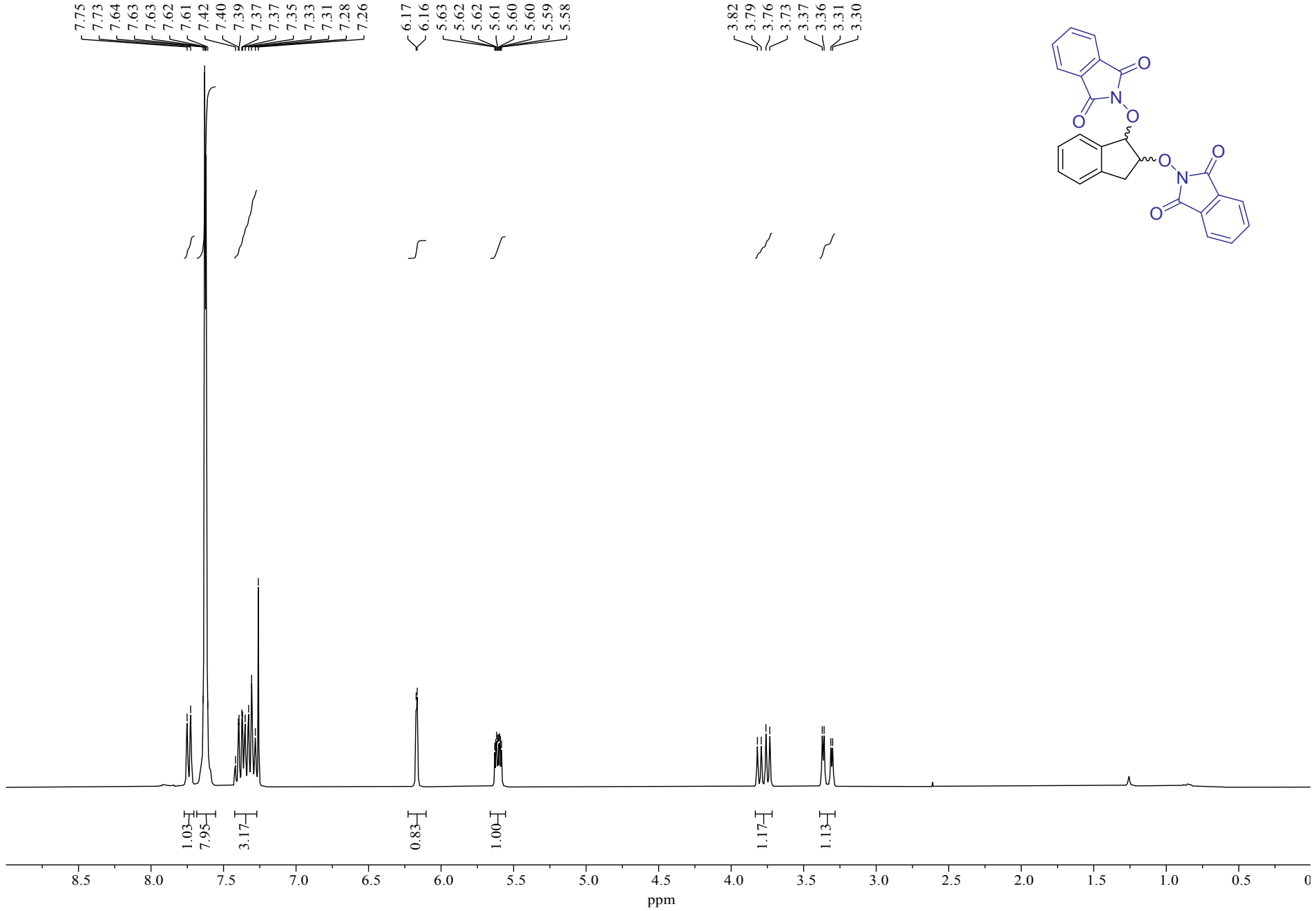
^{1}H NMR spectra of 3n [300.13 MHz, $CDCl_3$]



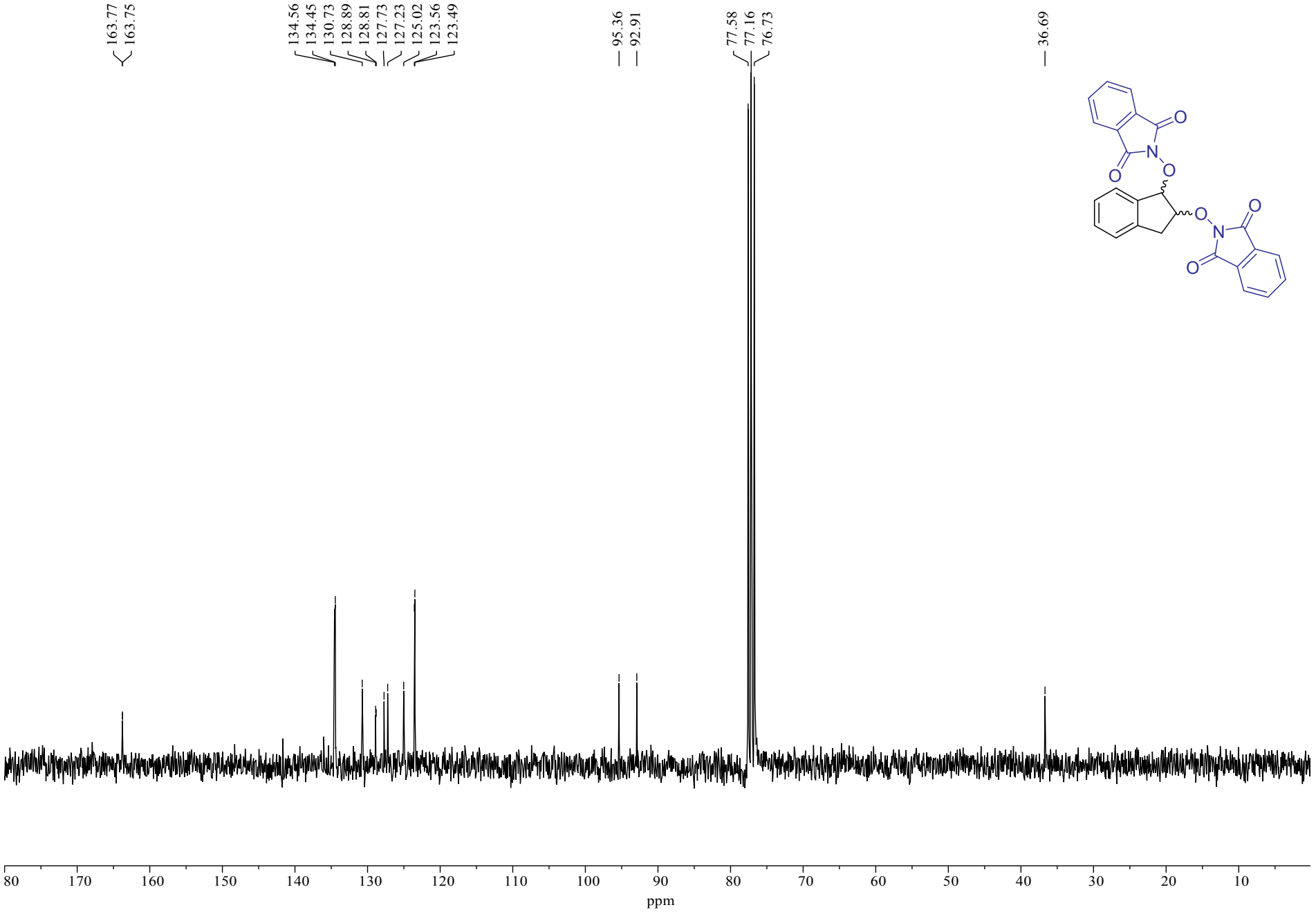
$^{13}C\{^{1}H\}$ NMR spectra of 3n [75.47 MHz, $CDCl_3$]



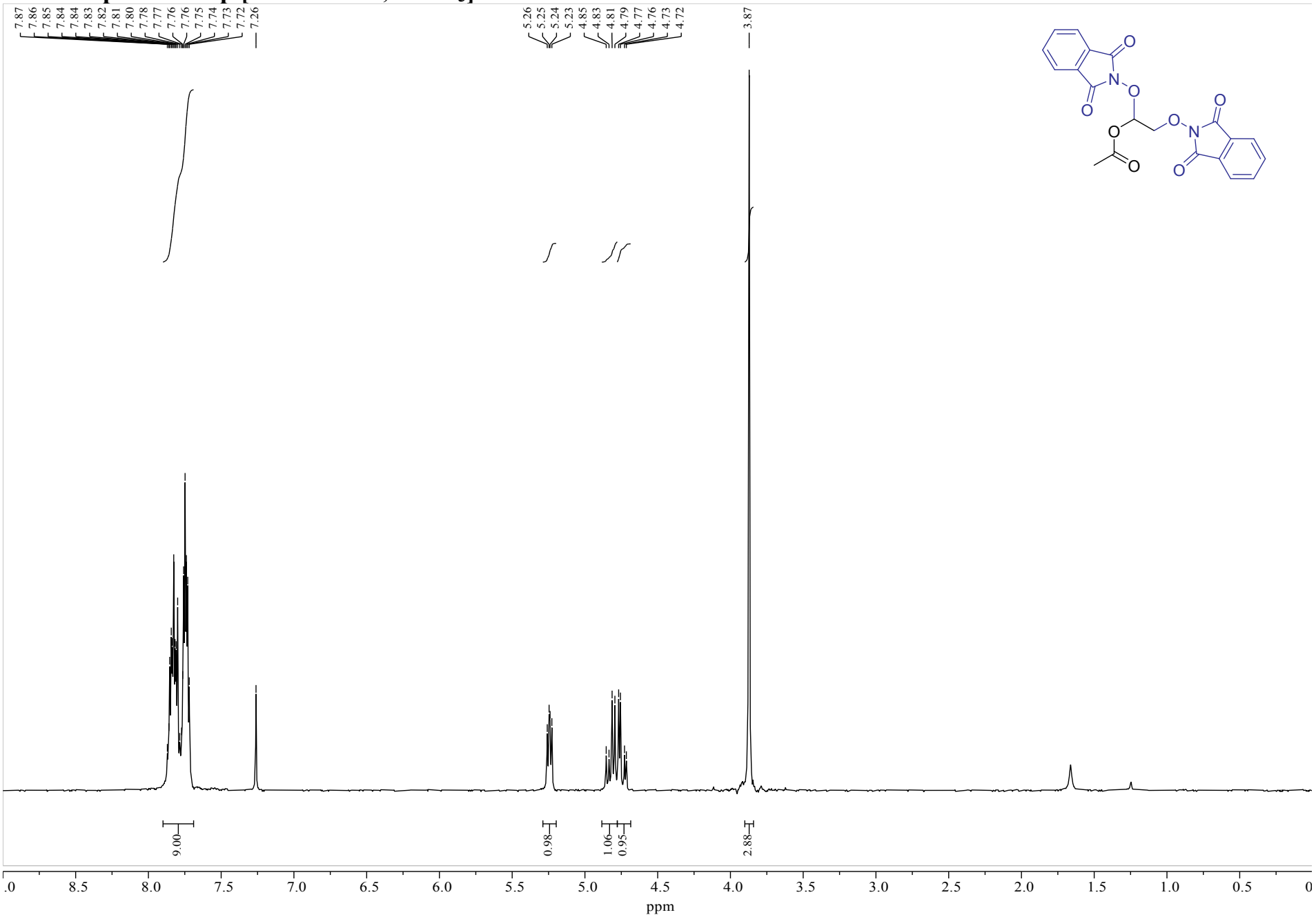
^{1}H NMR spectra of 3o [300.13 MHz, $CDCl_3$]



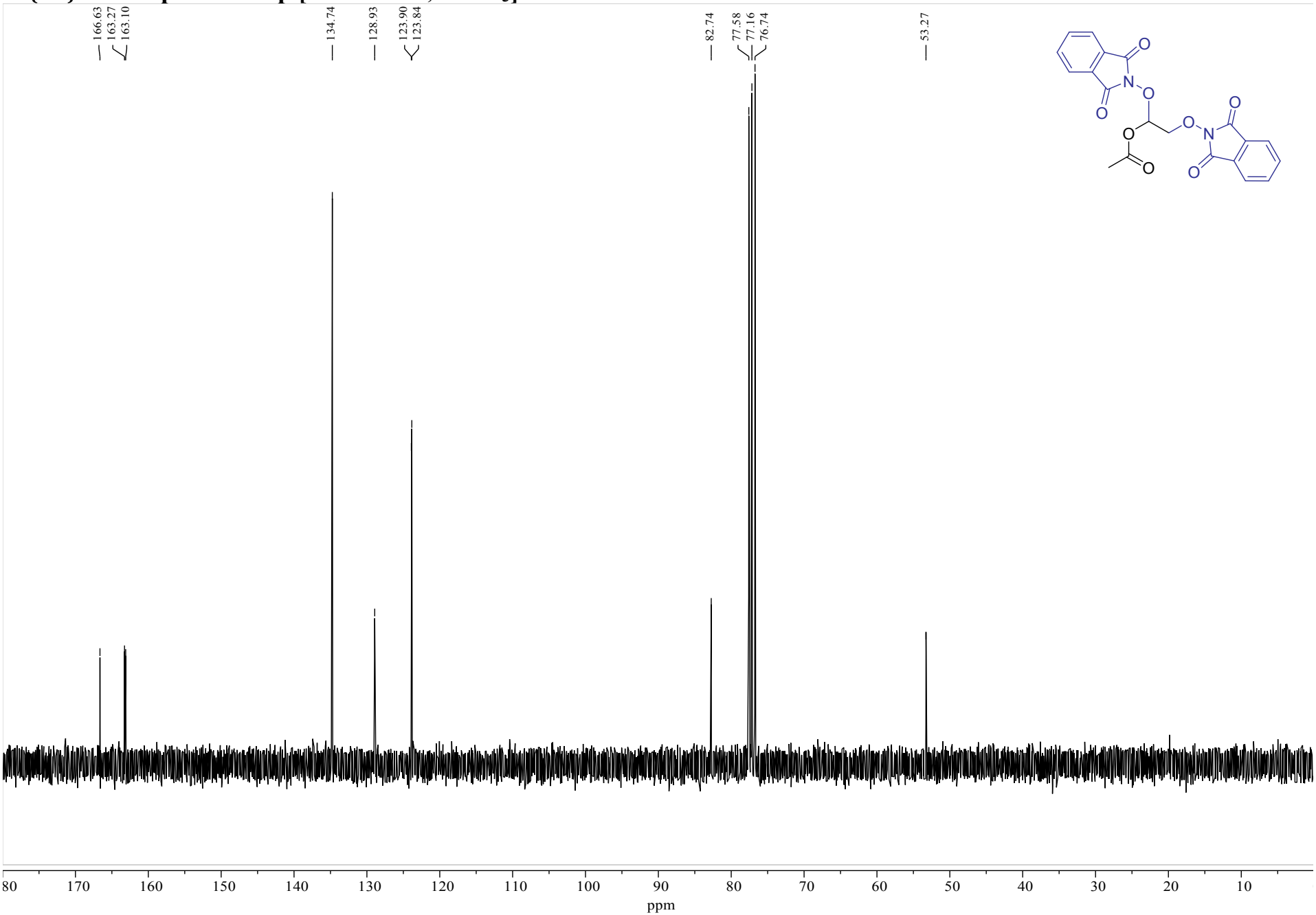
$^{13}C\{^{1}H\}$ NMR spectra of 3o [75.47 MHz, $CDCl_3$]



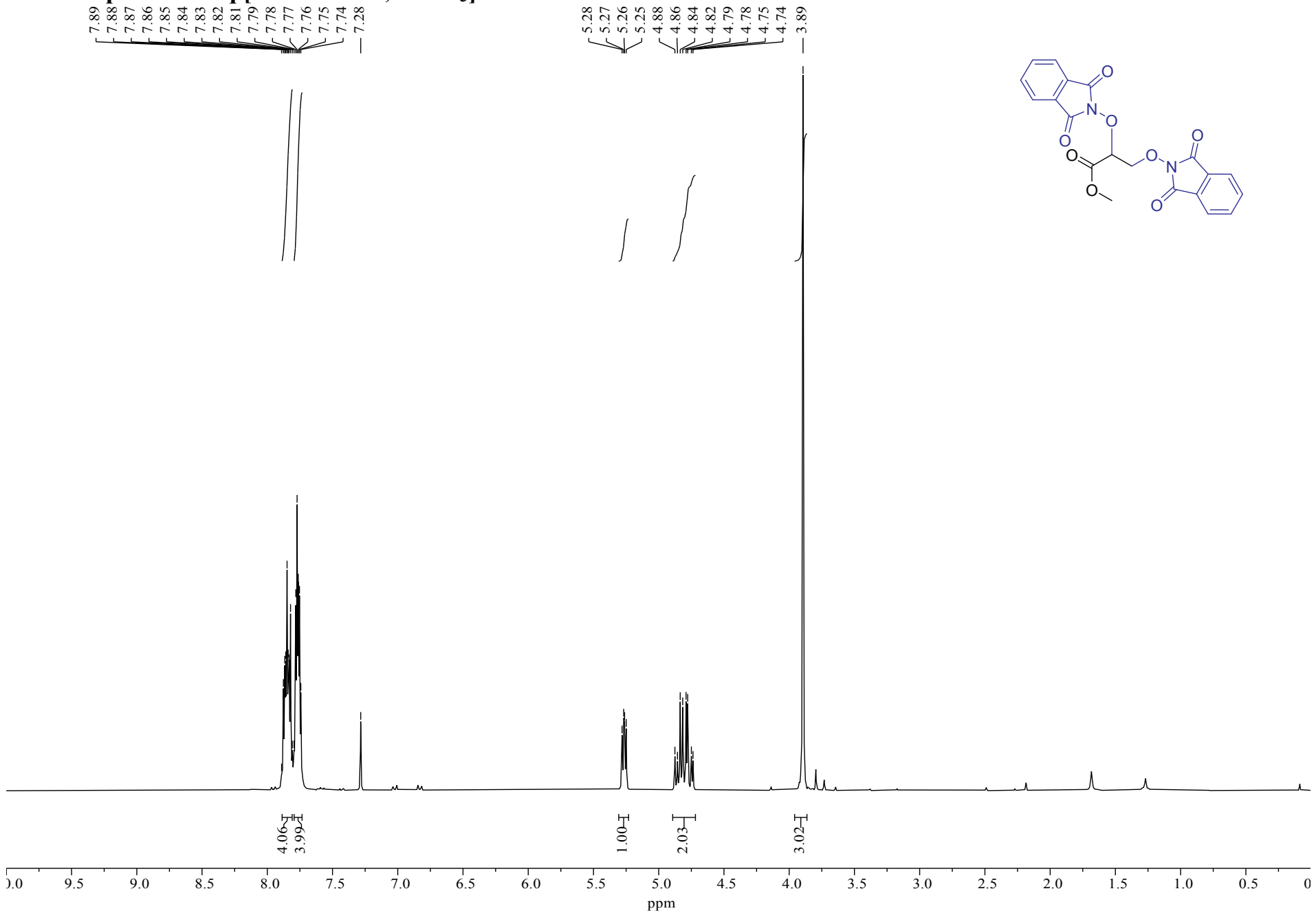
^{1}H NMR spectra of 3p [300.13 MHz, $CDCl_3$]



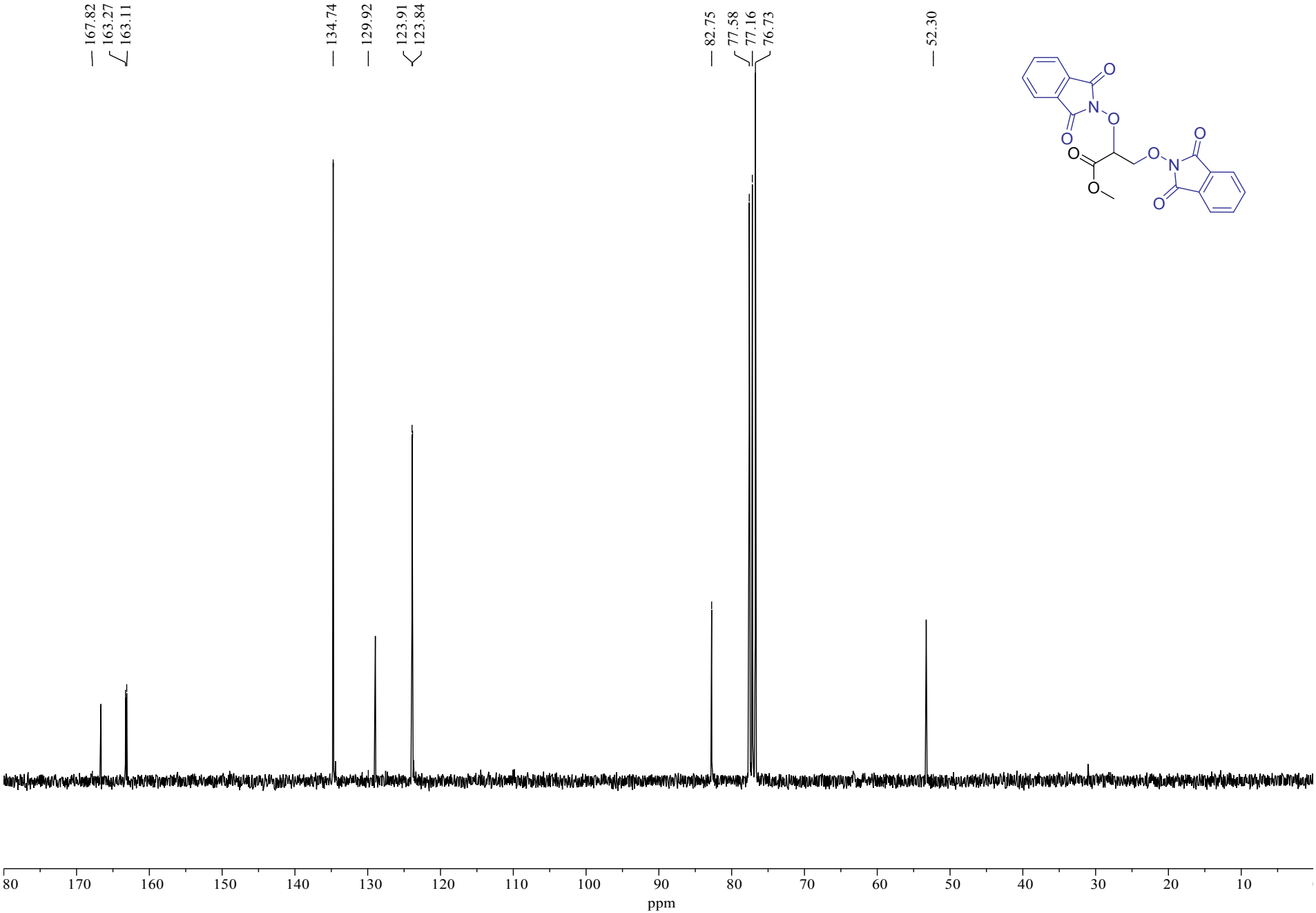
$^{13}C\{^{1}H\}$ NMR spectra of 3p [75.47 MHz, $CDCl_3$]



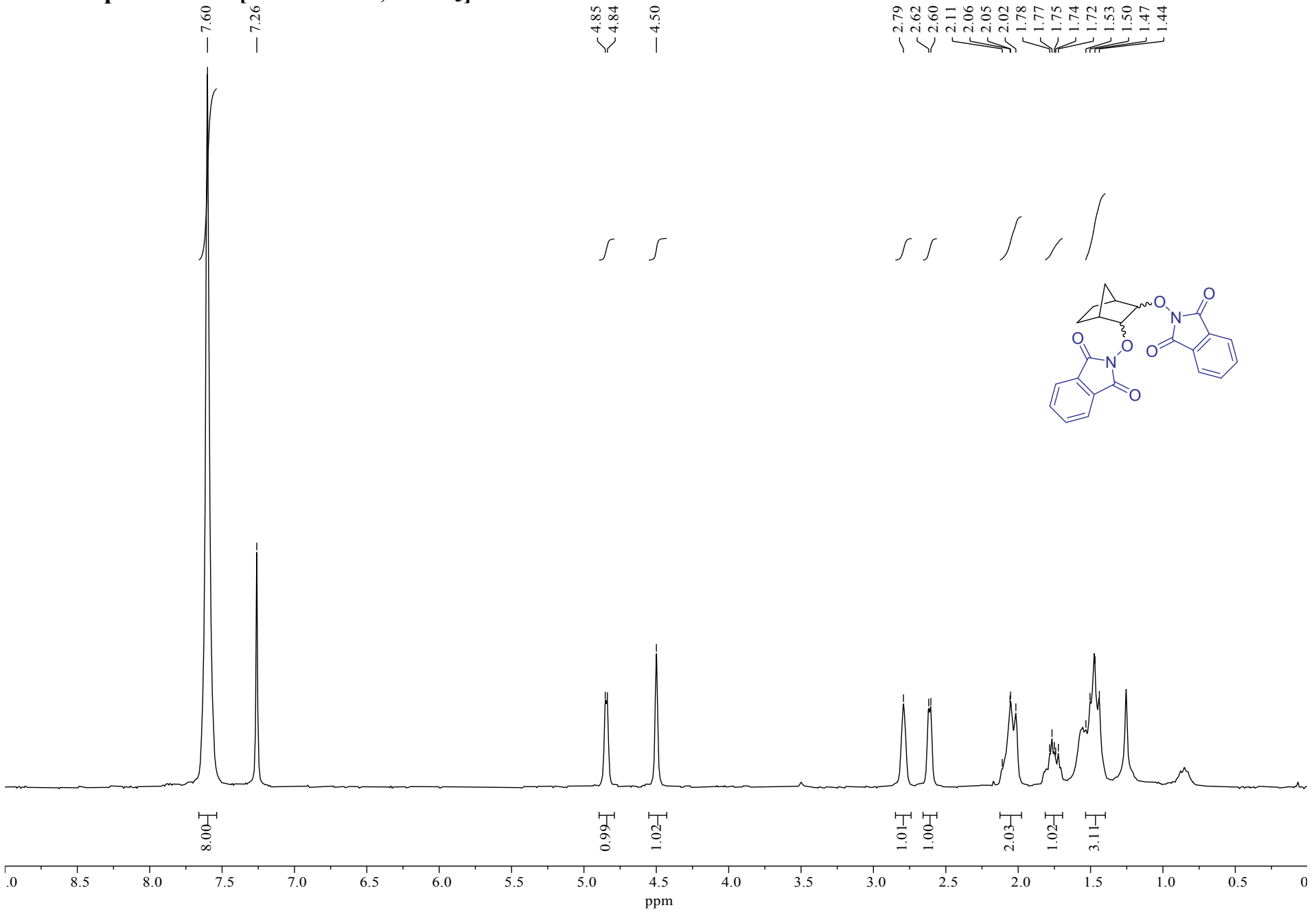
^{1}H NMR spectra of 3q [300.13 MHz, $CDCl_3$]



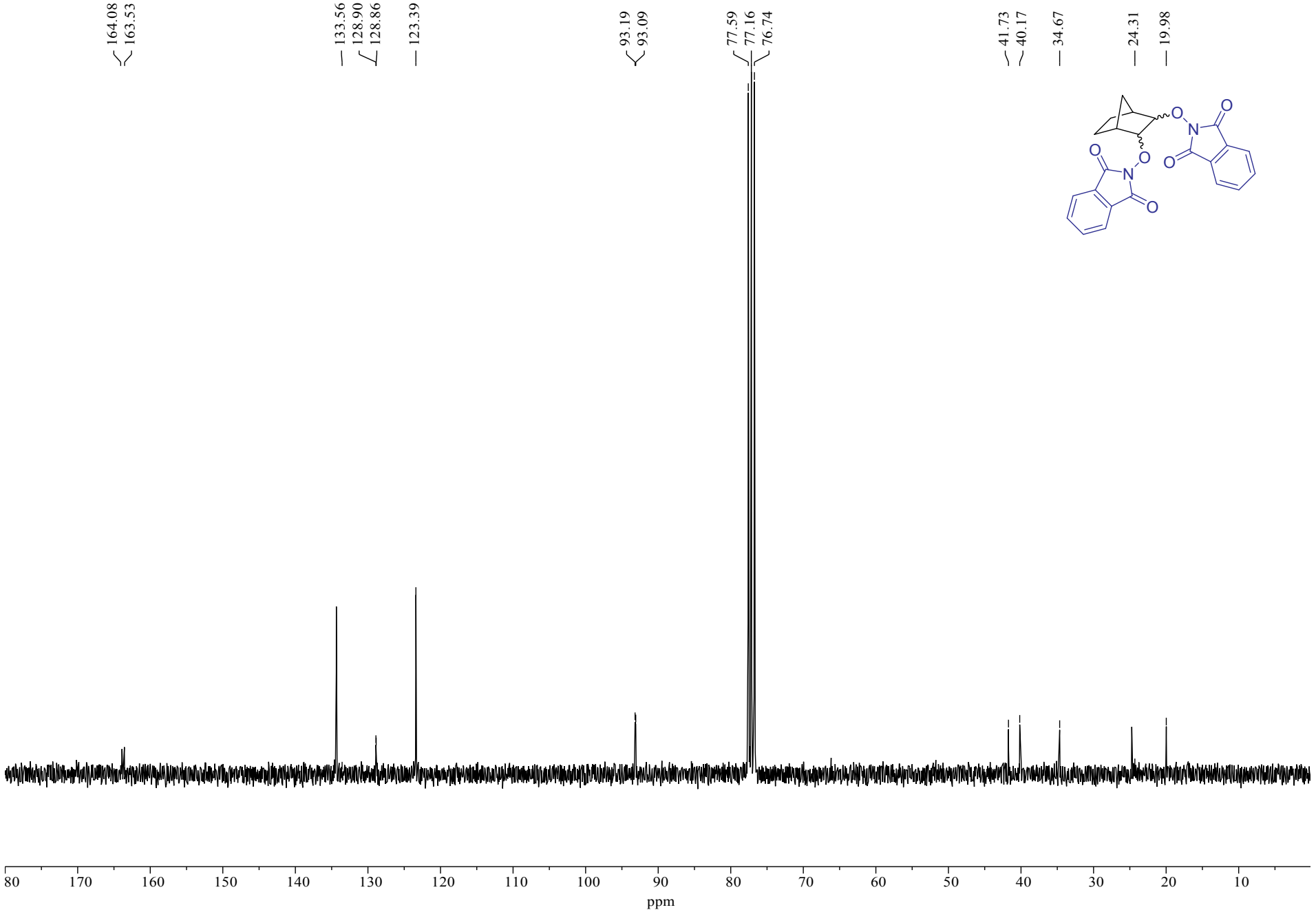
$^{13}C\{^{1}H\}$ NMR spectra of 3q [75.47 MHz, $CDCl_3$]



^{1}H NMR spectra of 3r [300.13 MHz, $CDCl_3$]



$^{13}C\{^{1}H\}$ NMR spectra of 3r [75.47 MHz, $CDCl_3$]



7. Copies of HRMS Spectra

HRMS (ESI) spectra of 3a

Display Report

Analysis Info

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Comment	C24H16N2O6 mH 429.1081 calibrant added, CH3CN		

Acquisition Parameter

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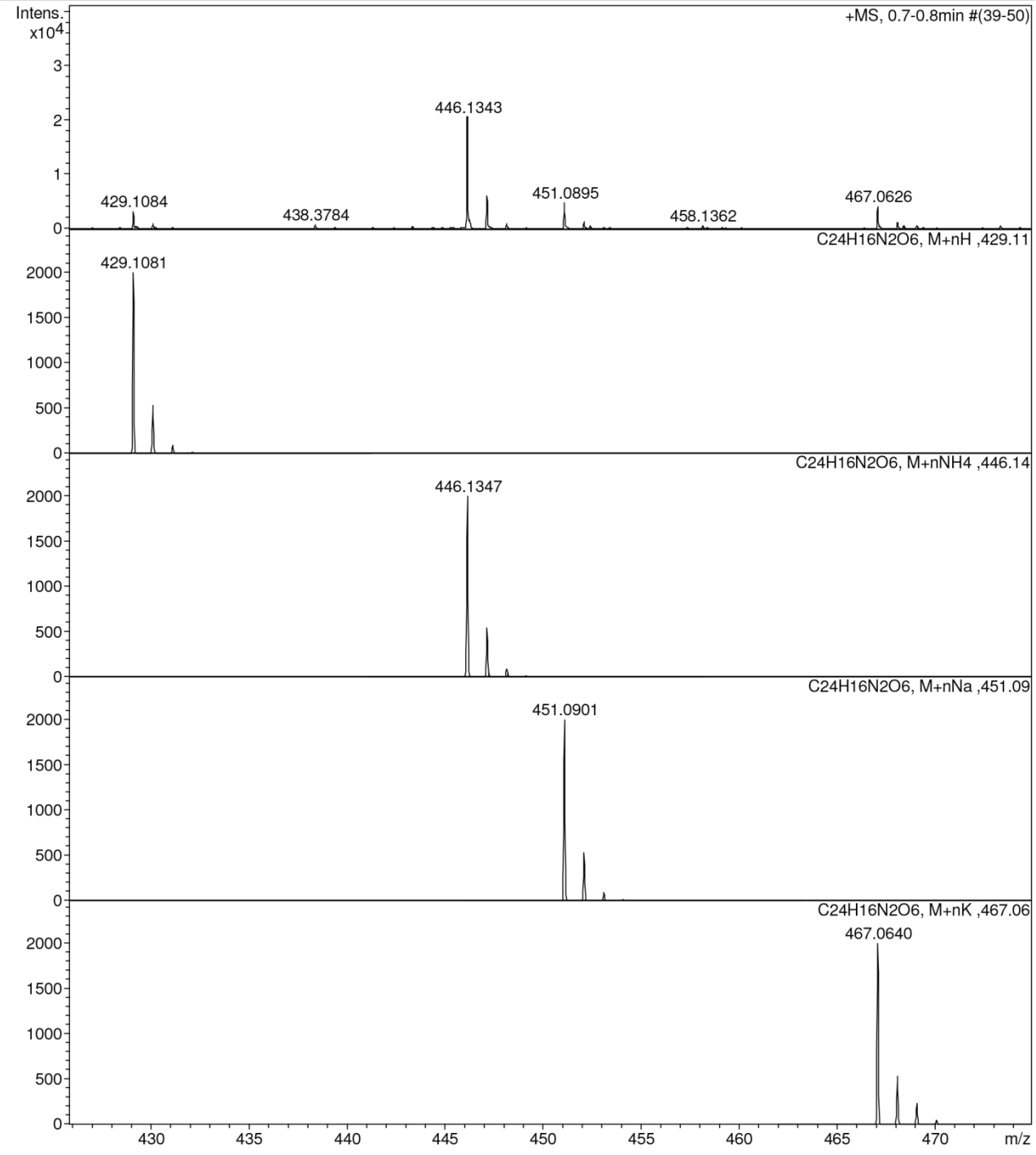

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HRMS (ESI) spectra of 3b

Display Report

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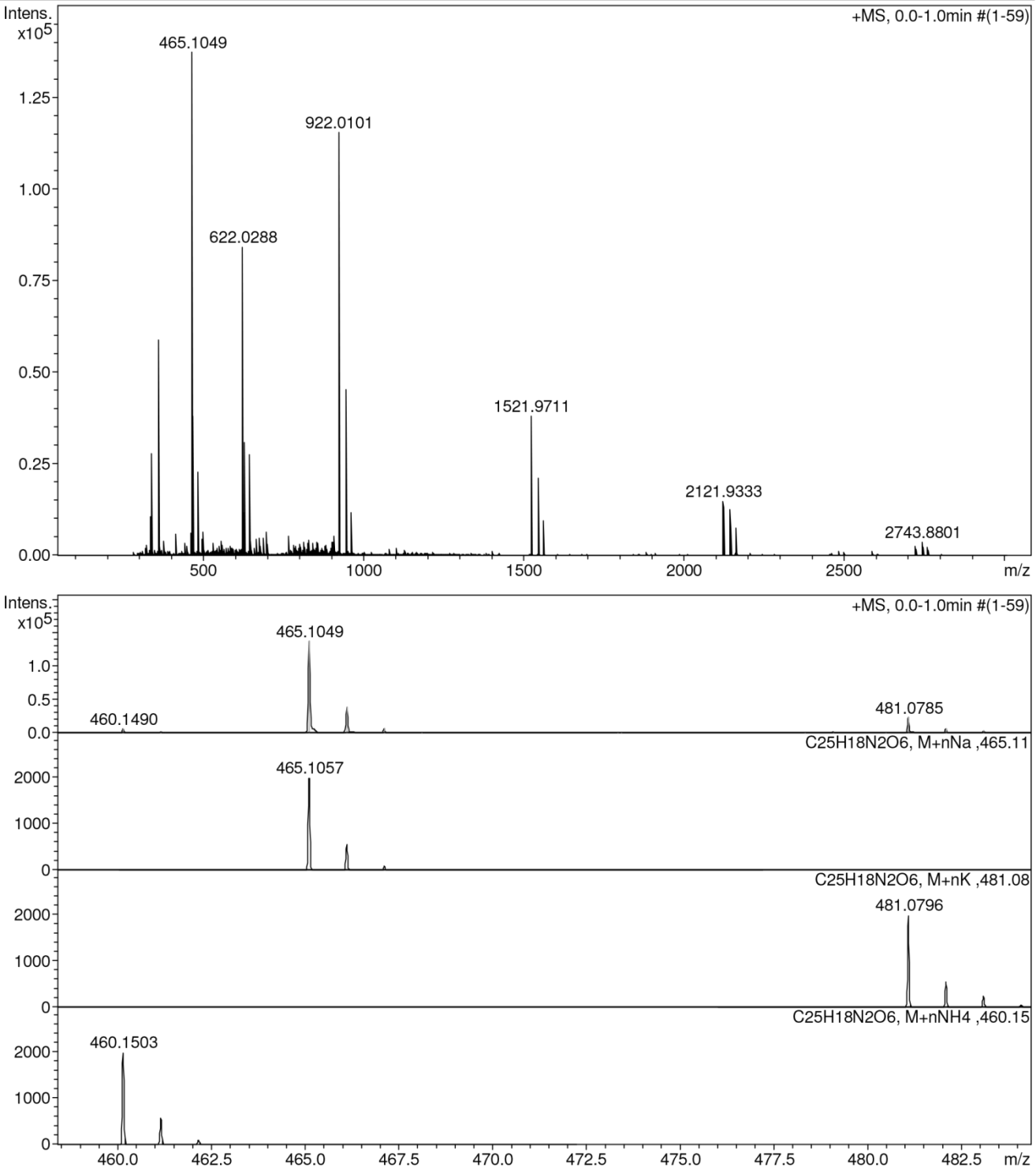

HRMS (ESI) spectra of 3c

Display Report

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Acquisition Parameter

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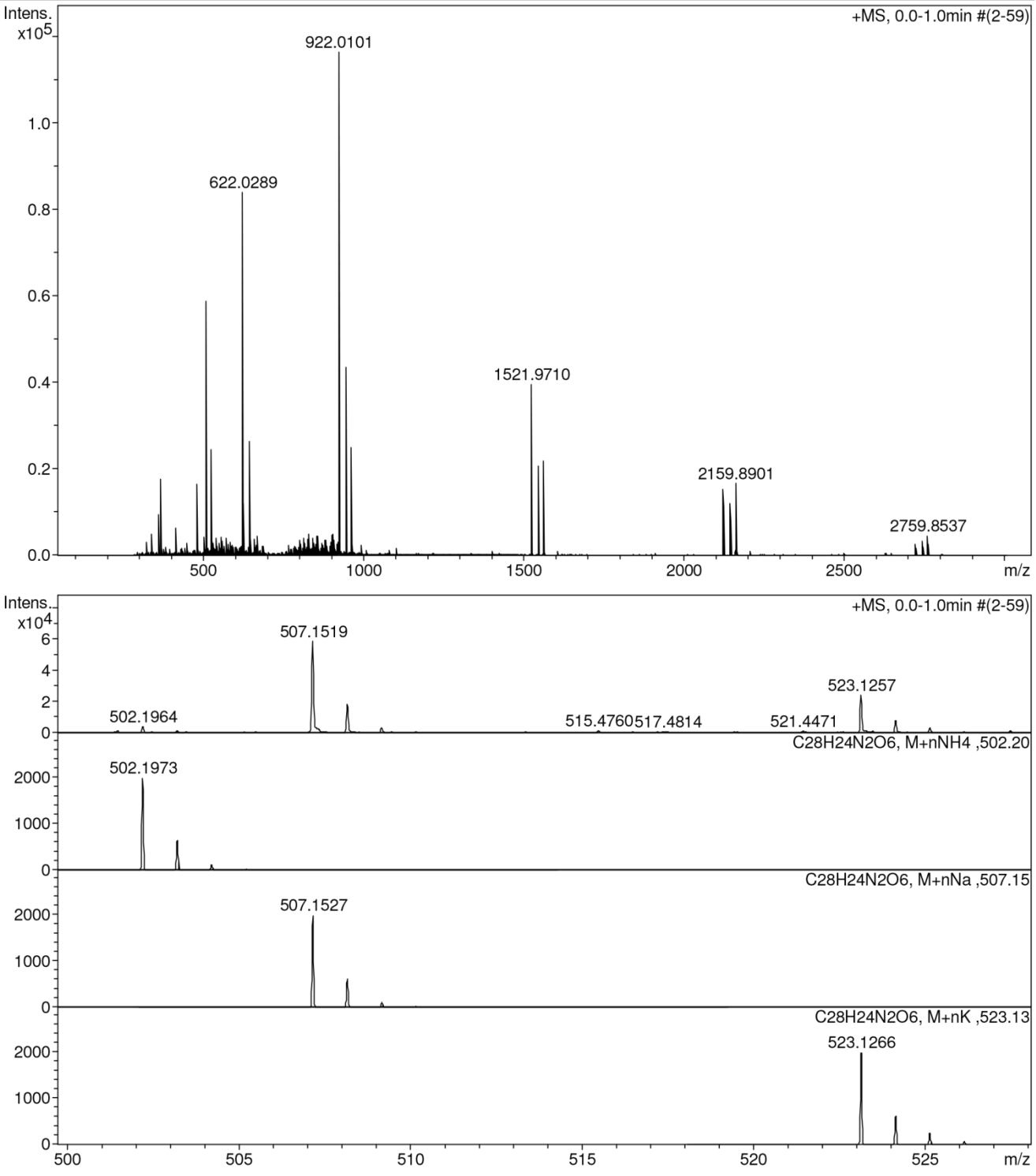
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS (ESI) spectra of 3d

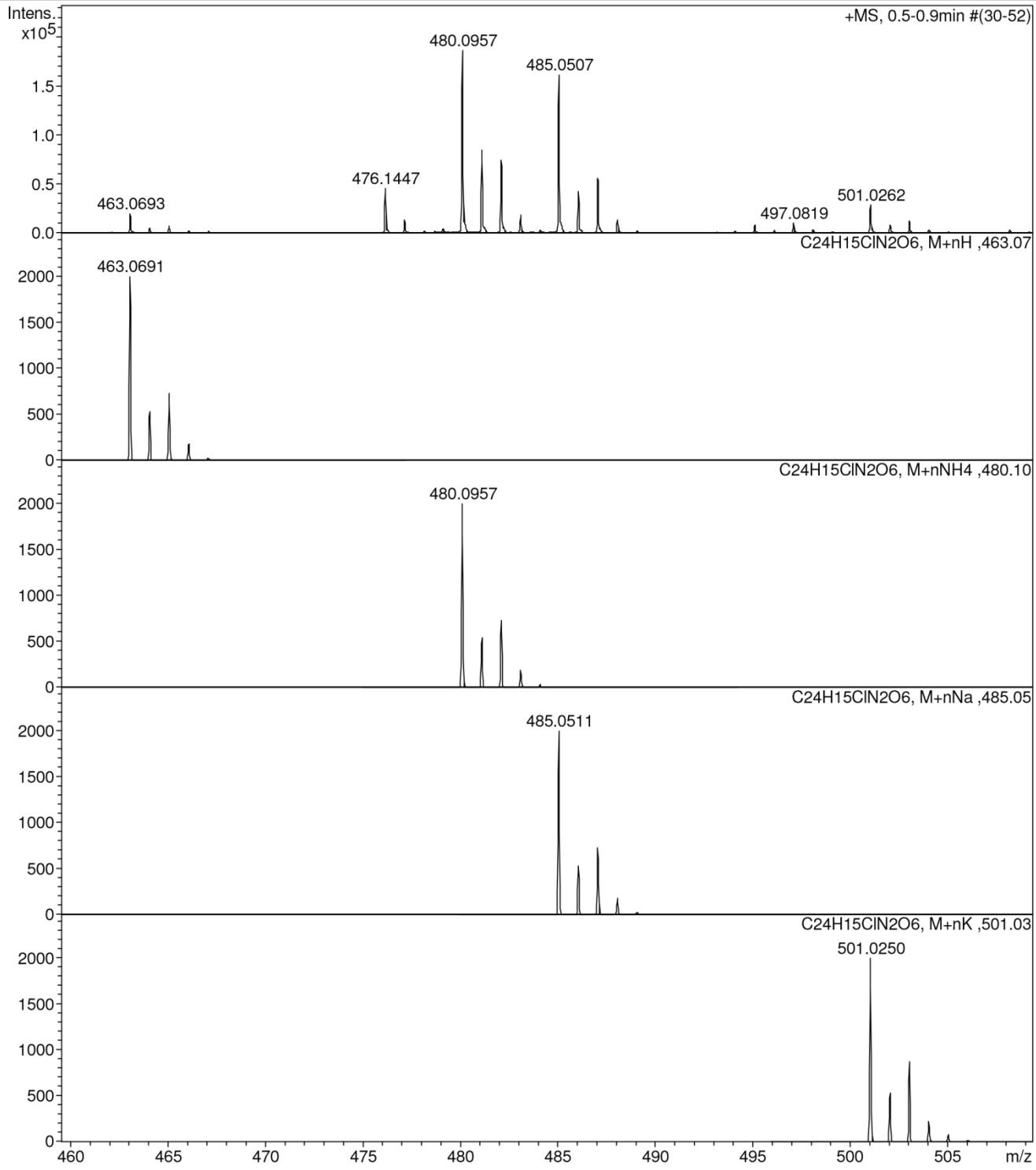
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2021\Dzunov\0701006.d	Acquisition Date	01.07.2021 9:57:45
Method	tune_50-1600.m	Operator	BDAL@DE
Sample Name	/TERN MD83	Instrument / Ser#	micrOTOF 10248
Comment	C24H15ClN2O6 mH 463.0691 clb added CH3CN		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	1600 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS (ESI) spectra of 3e

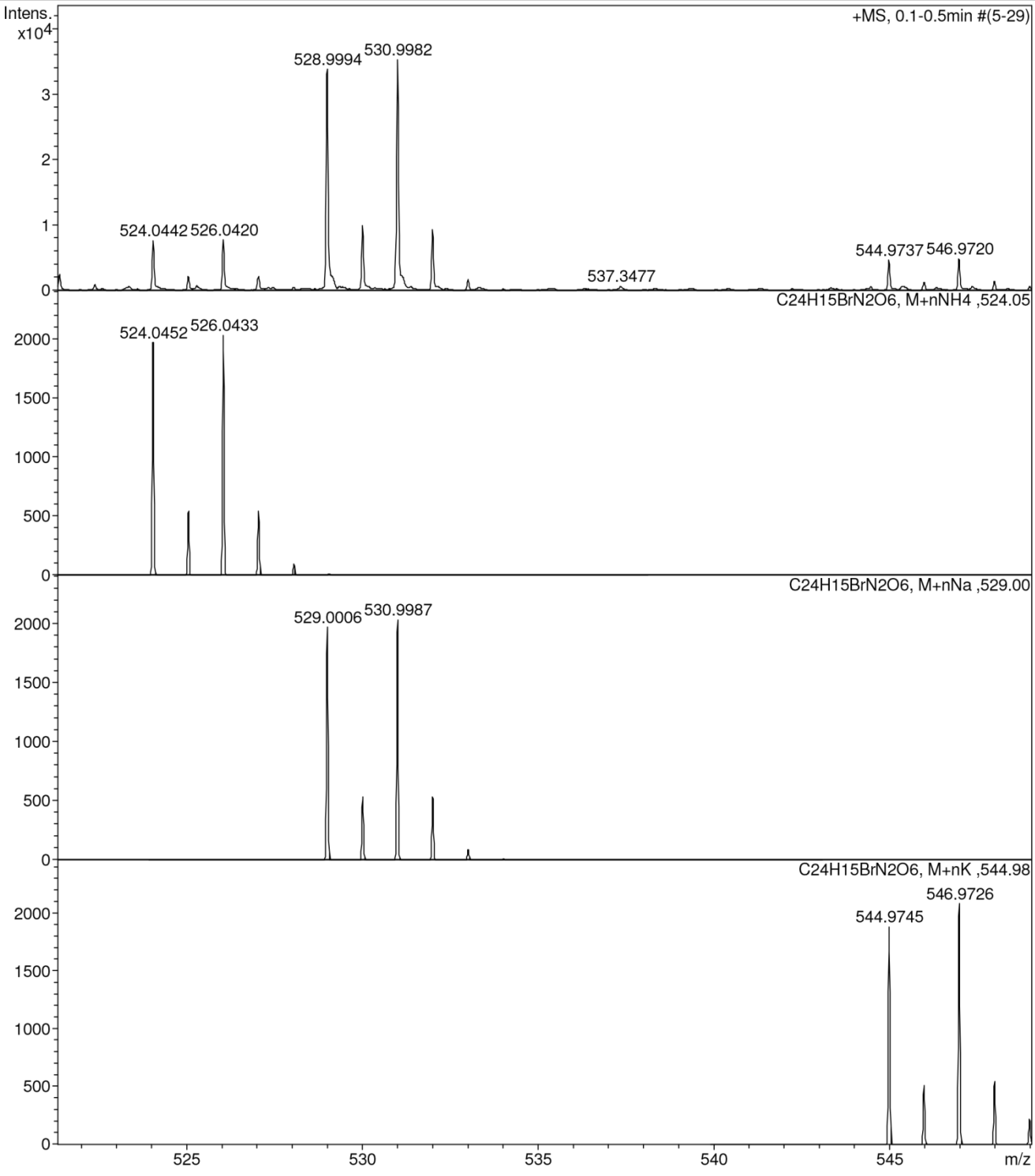
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2021\Segida\0608015.d	Acquisition Date	08.06.2021 19:06:09
Method	tune_50-1600.m	Operator	BDAL@DE
Sample Name	/TERN OSP102	Instrument / Ser#	micrOTOF 10248
Comment	C24H15BrN2O6 mH 507.0186 calibrant added CH3CN		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	1600 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS (ESI) spectra of 3f

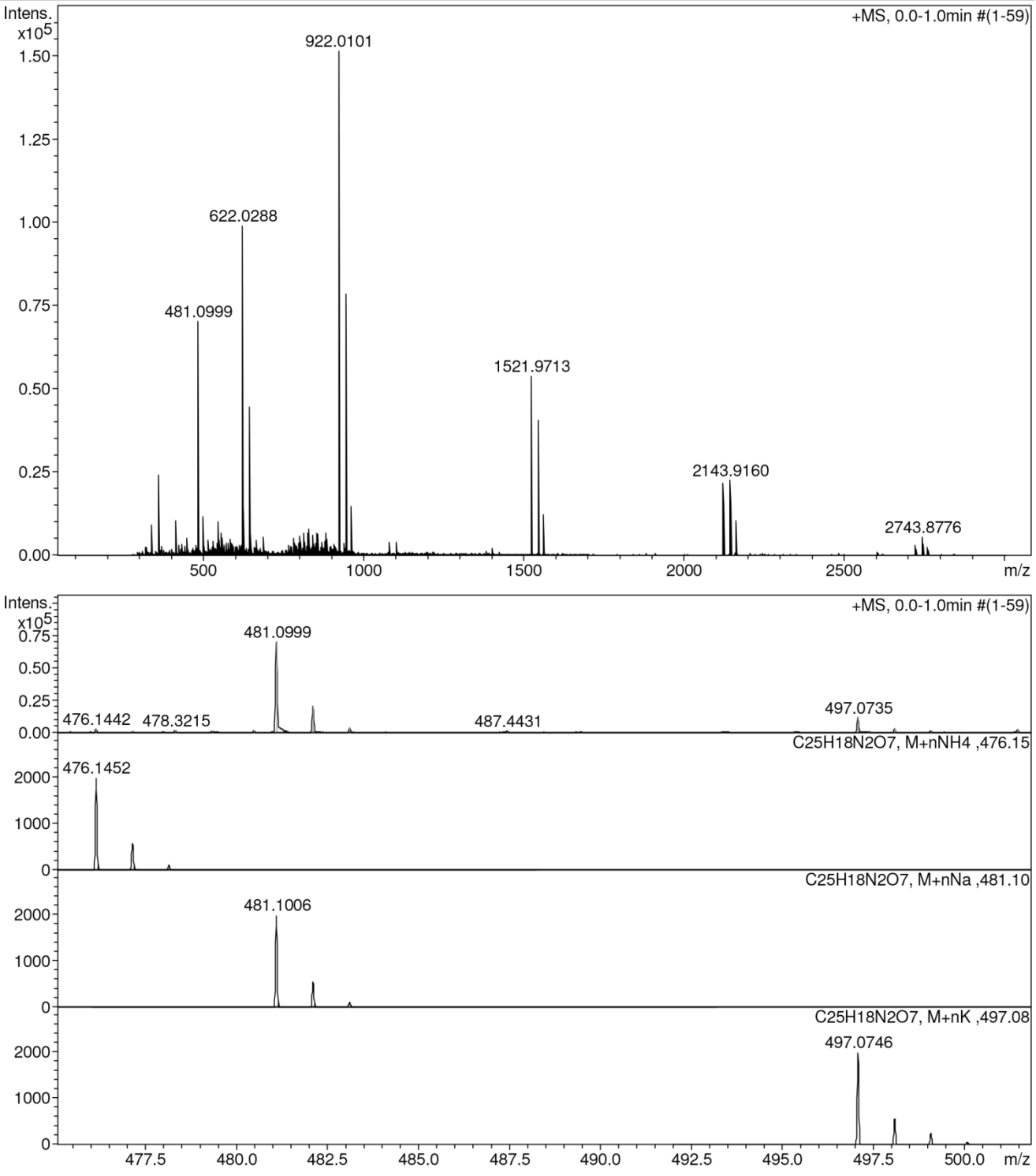
Display Report

Analysis Info

Analysis Name	D:\Data\Chizhov\Terentiev\Dzyukov\mp78_&clb.d	Acquisition Date	07.06.2021 15:34:34
Method	tune_wide.m	Operator	BDAL@DE
Sample Name	/TERN MP78	Instrument / Ser#	micrOTOF 10248
Comment	CH3OH 100 %, dil. 2000, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS (ESI) spectra of 3g

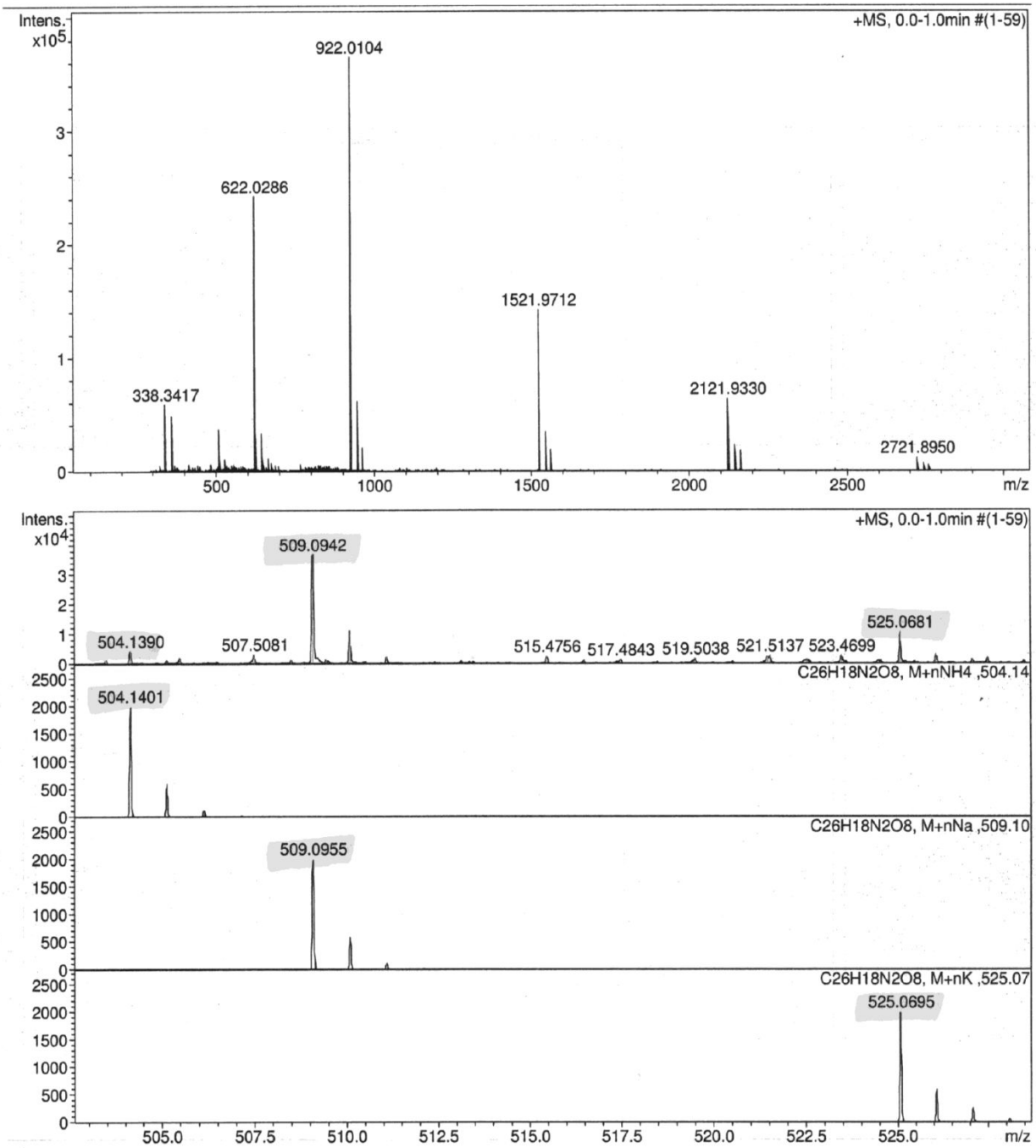
Display Report

Analysis Info

Analysis Name	D:\Data\Chizhov\Terentiev\Segida\md80_&clb.d	Acquisition Date	17.06.2021 13:04:58
Method	tune_wide.m	Operator	BDAL@DE
Sample Name	/TERN MD80	Instrument / Ser#	micrOTOF 10248
Comment	CH3OH 100 %, dil. 2000, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Bruker Compass DataAnalysis 4.0 printed: 17.06.2021 13:18:21 Page 1 of 1

HRMS (ESI) spectra of 3h

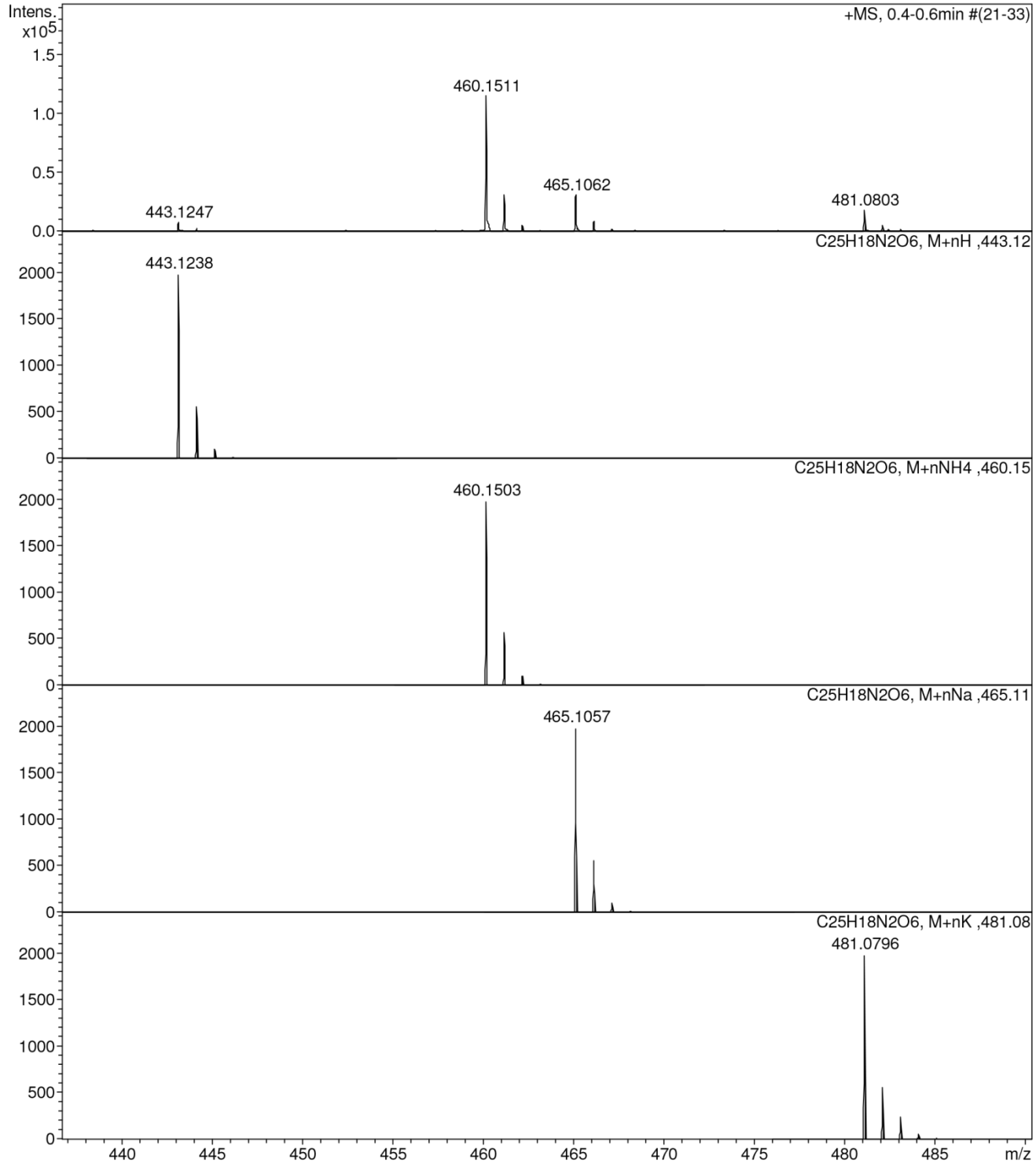
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2021\Segida\0608014.d	Acquisition Date	08.06.2021 18:56:40
Method	tune_50-1600.m	Operator	BDAL@DE
Sample Name	/TERN OSP105	Instrument / Ser#	micrOTOF 10248
Comment	C25H18N2O6 mH 443.1237 calibrant added CH3CN		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	1600 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS (ESI) spectra of 3i

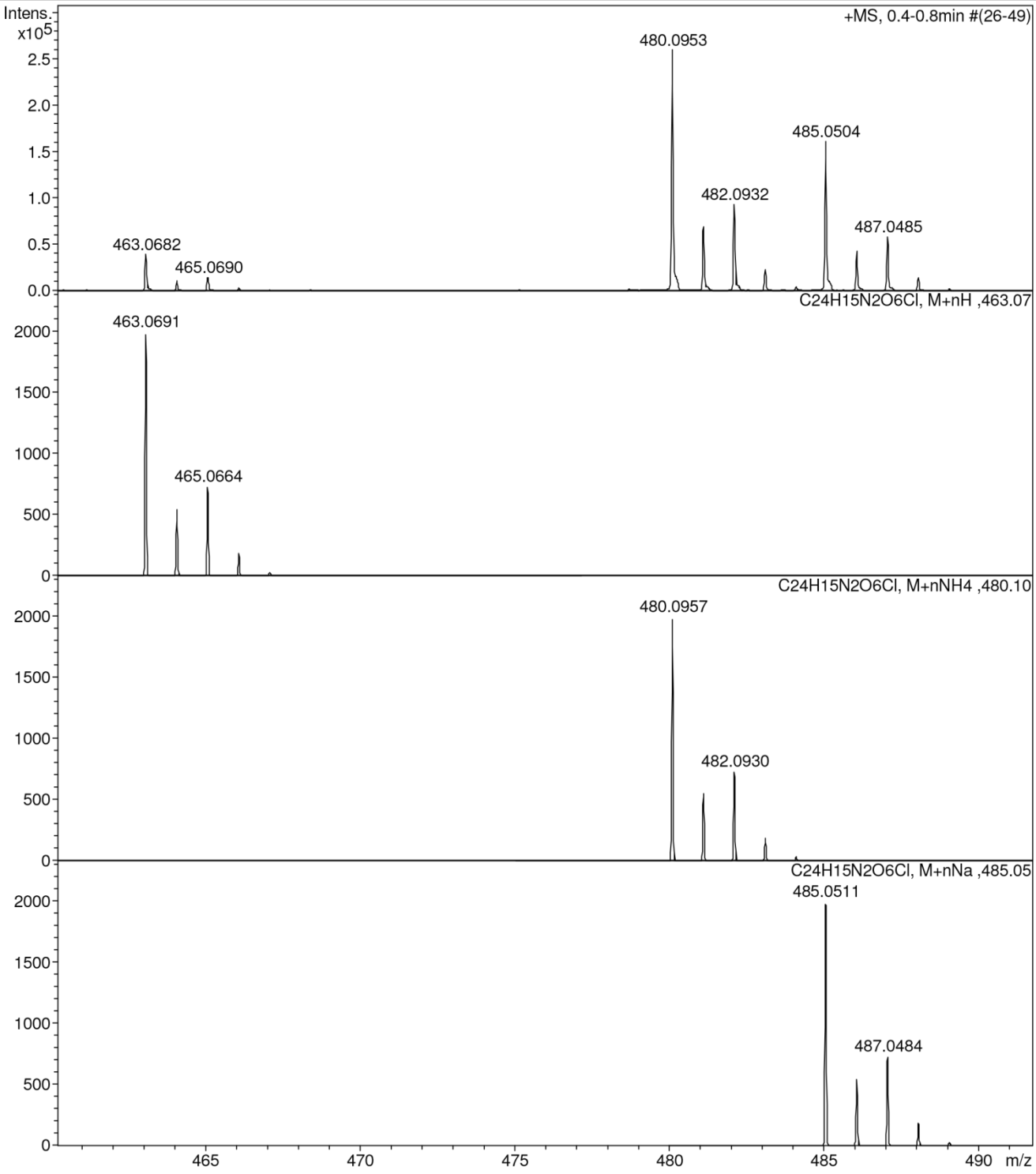
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2021\Segida\0610002.d	Acquisition Date	10.06.2021 9:41:20
Method	tune_50-1600.m	Operator	BDAL@DE
Sample Name	/TERN OS106	Instrument / Ser#	micrOTOF 10248
Comment	C24H15ClN2O6 mH 463.0691 calibrant added CH3OH		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	1600 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS (ESI) spectra of 3j

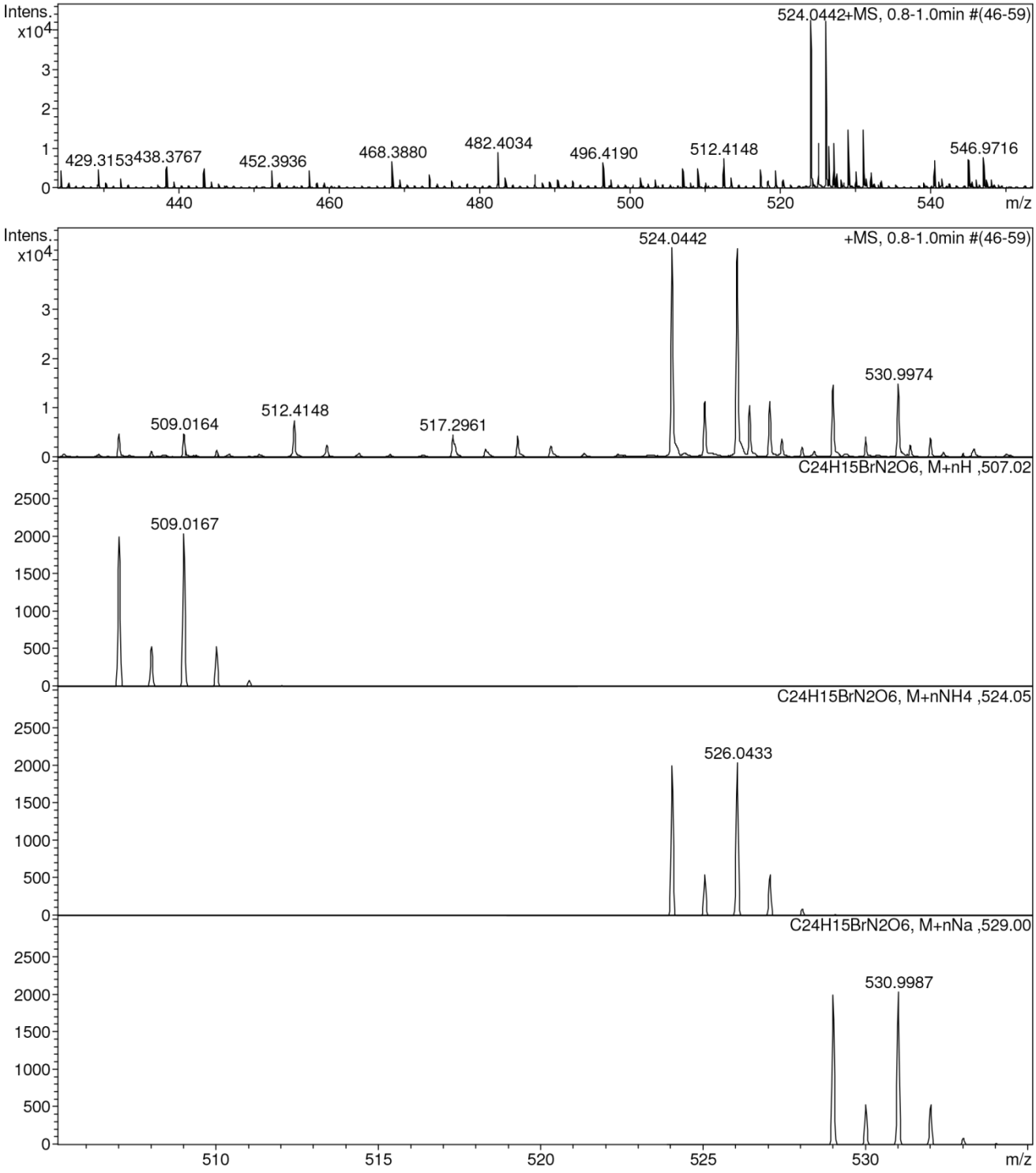
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2021\Fedorova\0701048.d	Acquisition Date	01.07.2021 18:14:58
Method	tune_50-1600.m	Operator	BDAL@DE
Sample Name	/TERN UF-128_2	Instrument / Ser#	micrOTOF 10248
Comment	C24H16N2O6 mH 429.1081 clb added CH3CN		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	1600 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS (ESI) spectra of 3k

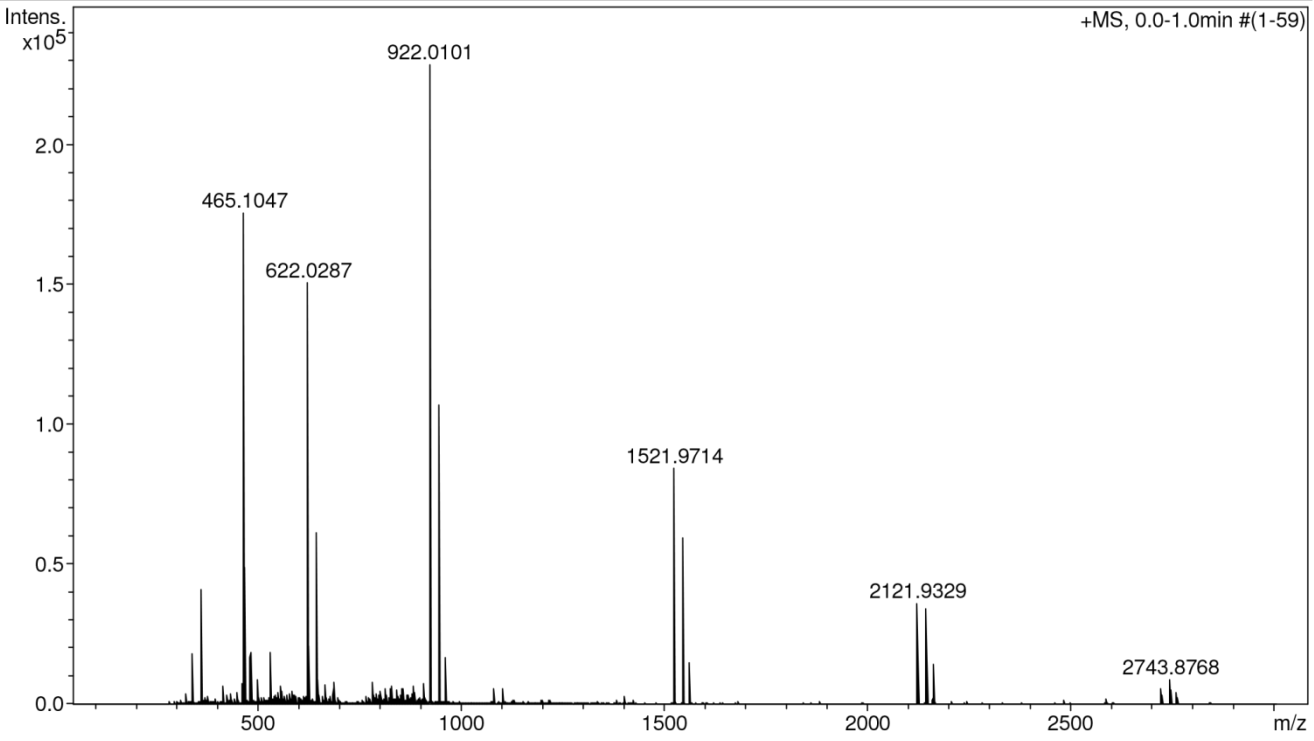
Display Report

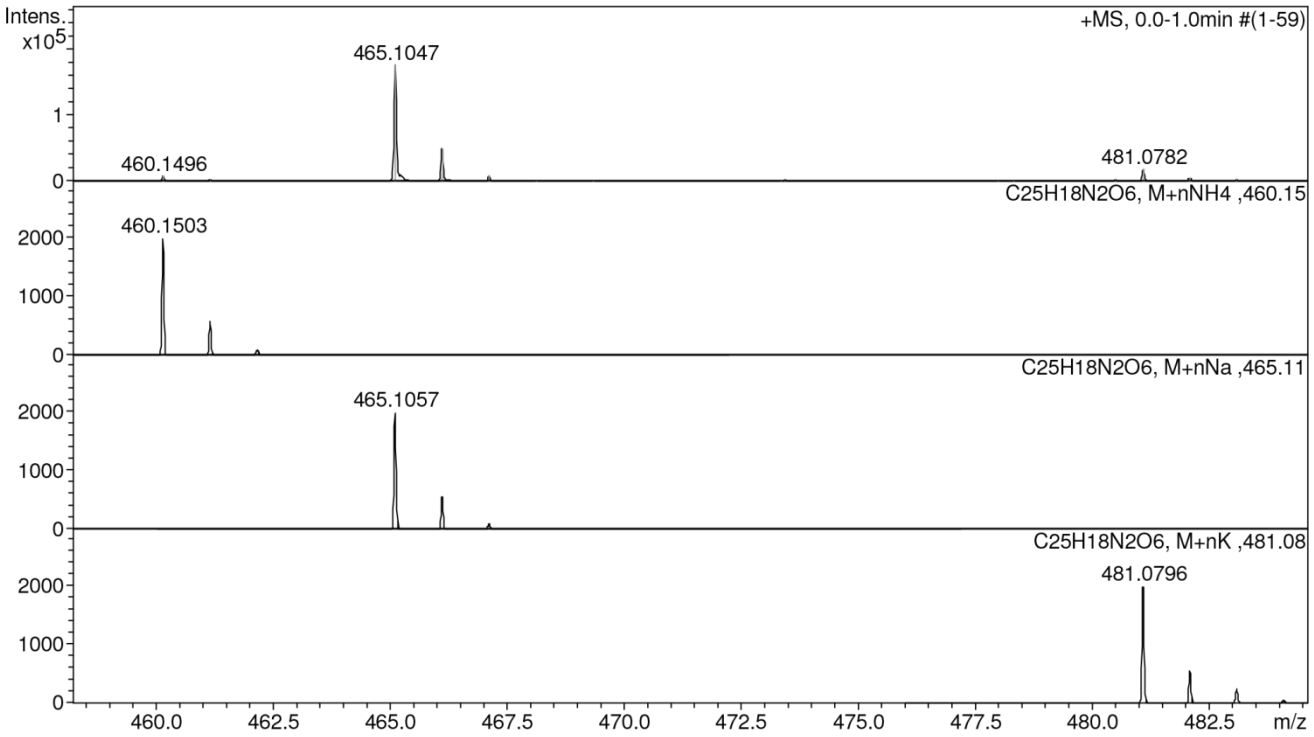
Analysis Info

Analysis Name	D:\Data\Chizhov\Terentiev\Segida\osp100_&clb.d	Acquisition Date	07.06.2021 15:29:47
Method	tune_wide.m	Operator	BDAL@DE
Sample Name	/TERN OSP100	Instrument / Ser#	micrOTOF 10248
Comment	CH3OH 100 %, dil. 2000, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste





HRMS (ESI) spectra of 3l

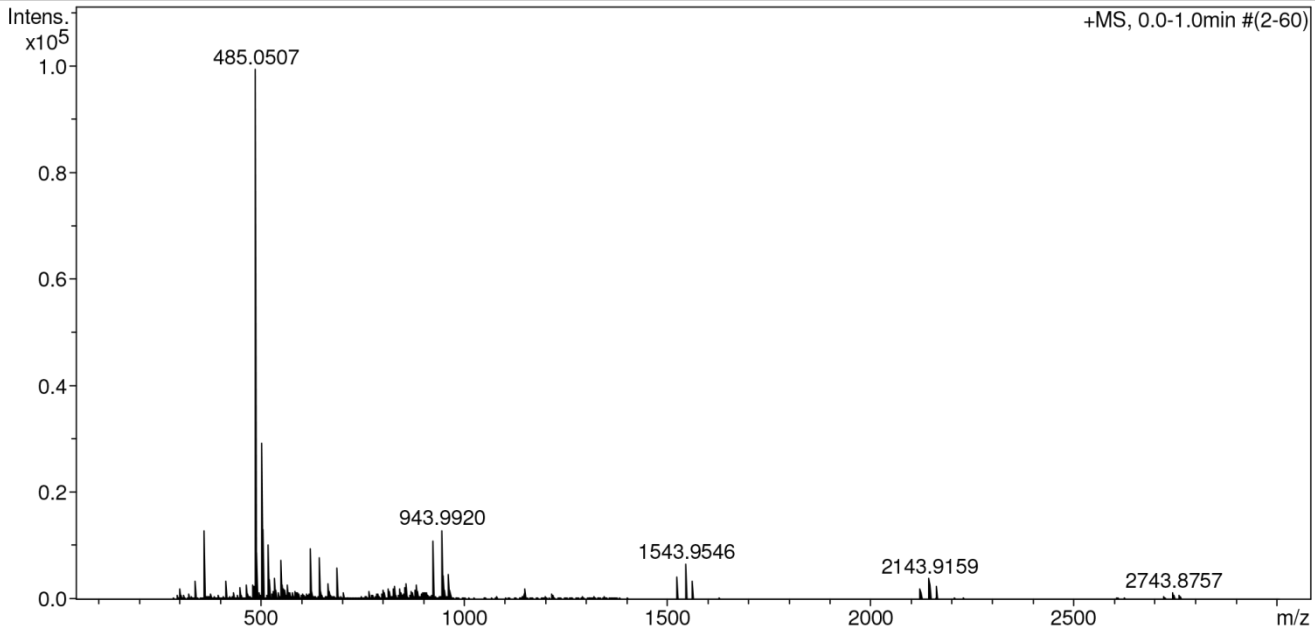
Display Report

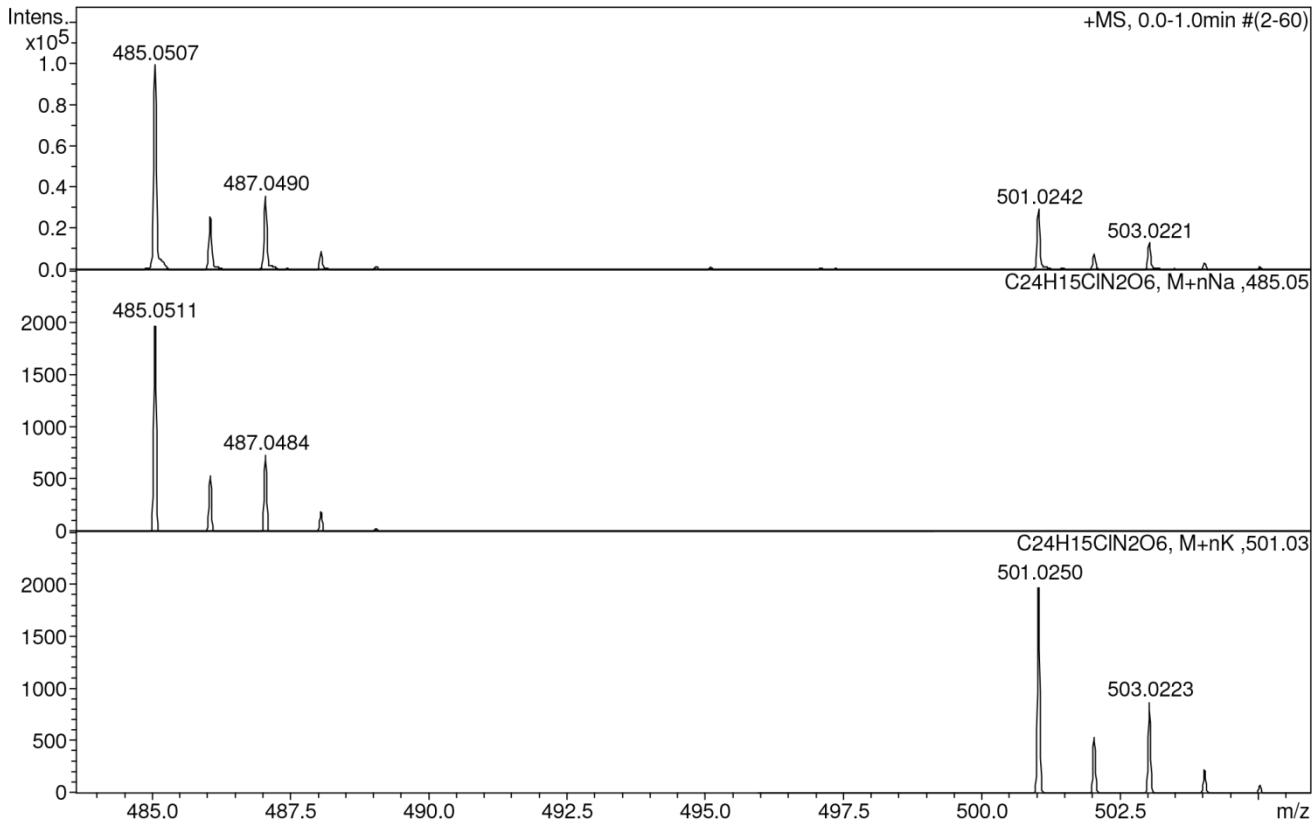
Analysis Info

Analysis Name	D:\Data\Chizhov\Terentiev\Segida\osp110_&clb.d	Acquisition Date	22.06.2021 16:19:53
Method	tune_wide.m	Operator	BDAL@DE
Sample Name	/TERN OSP110	Instrument / Ser#	micrOTOF 10248
Comment	CH3OH 100 %, dil, 1000, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste





HRMS (ESI) spectra of 3m

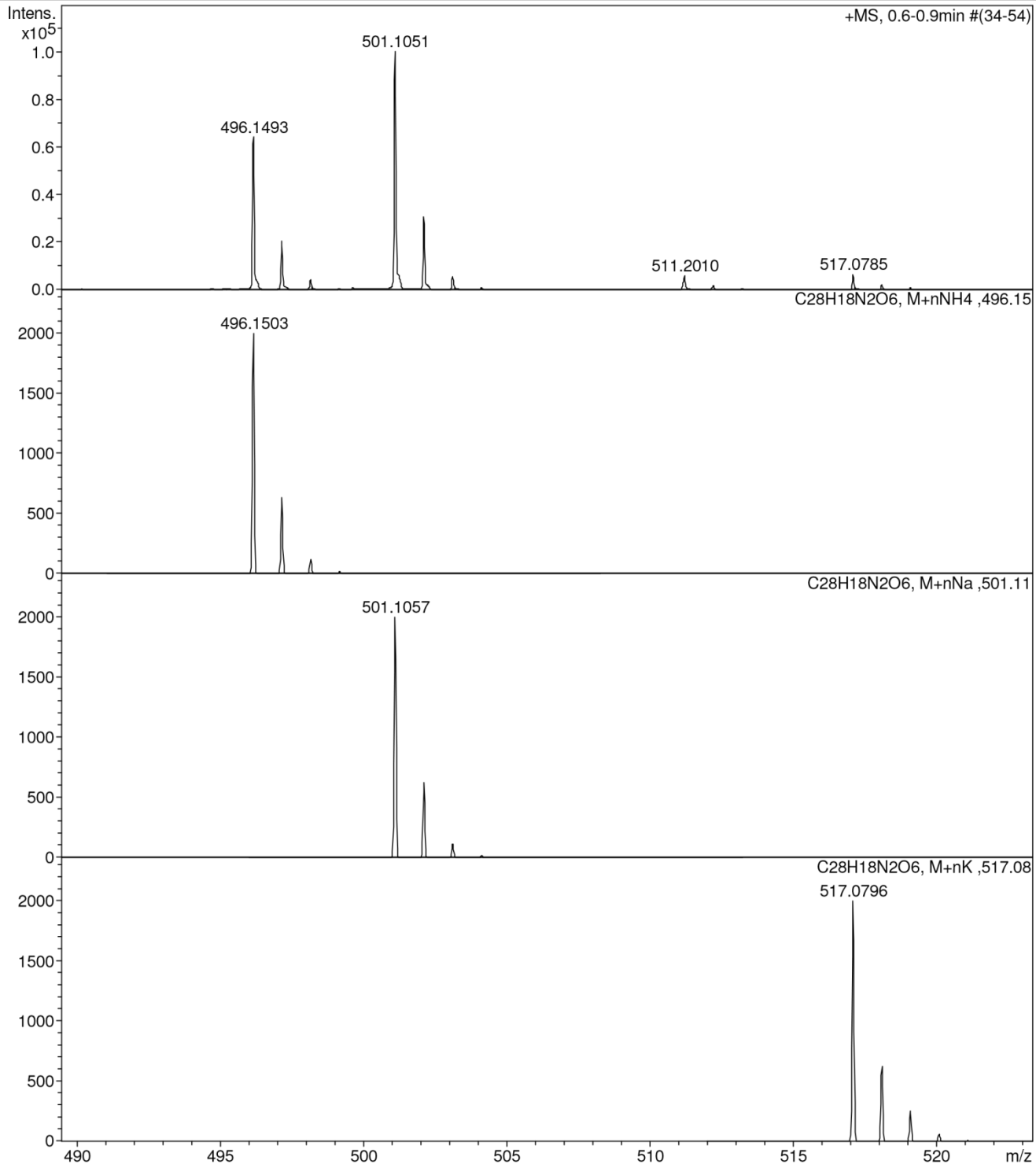
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2021\Dzunov\0701005.d	Acquisition Date	01.07.2021 9:52:39
Method	tune_50-1600.m	Operator	BDAL@DE
Sample Name	/TERN MD82	Instrument / Ser#	micrOTOF 10248
Comment	C28H18N2O6 mH 479.1237 clb added CH3CN		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	1600 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS (ESI) spectra of 3n

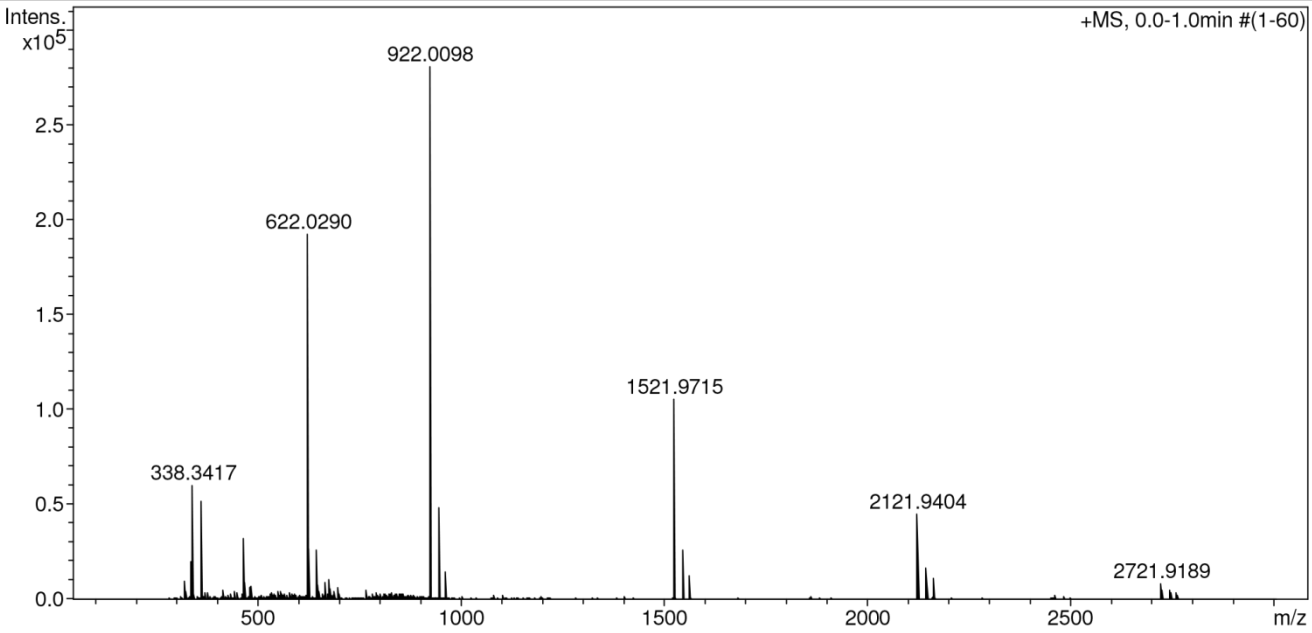
Display Report

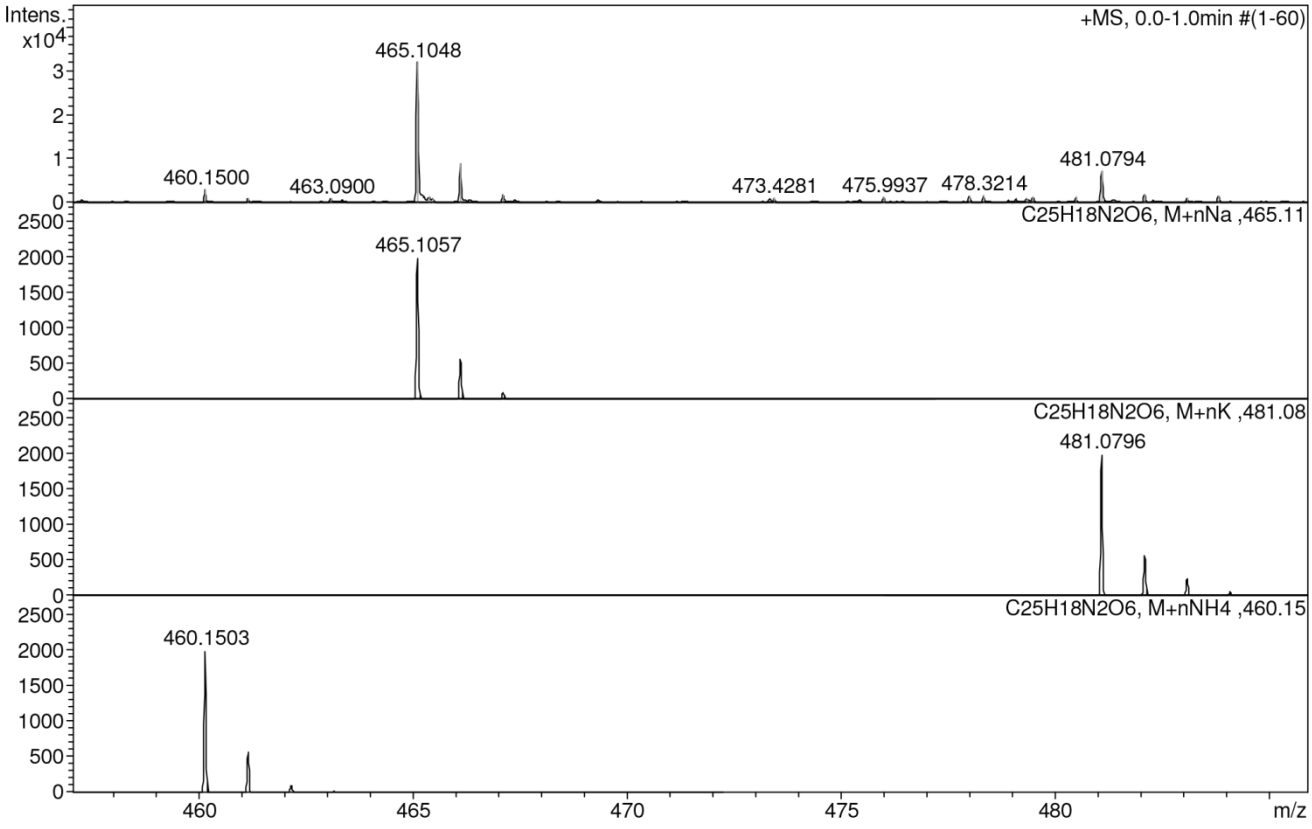
Analysis Info

Analysis Name	D:\Data\Chizhov\Terentiev\Segida\osp108_&clb.d	Acquisition Date	17.06.2021 13:13:51
Method	tune_wide.m	Operator	BDAL@DE
Sample Name	/TERN OSP108	Instrument / Ser#	micrOTOF 10248
Comment	CH3OH 100 %, dil. 2000, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste





HRMS (ESI) spectra of 3o

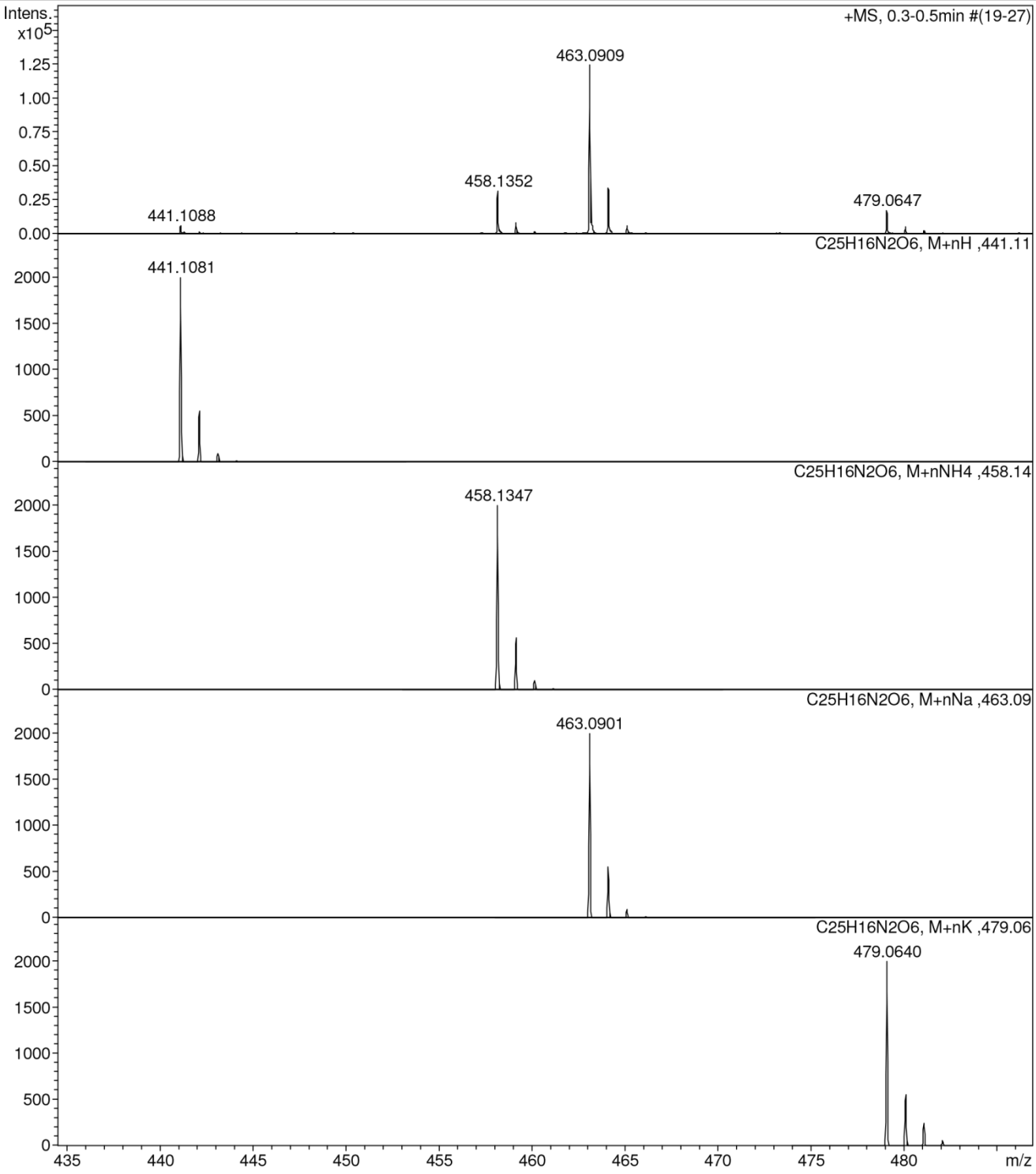
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2021\Segida\0709003.d	Acquisition Date	09.07.2021 9:01:06
Method	tune_50-1600.m	Operator	BDAL@DE
Sample Name	/TERN OSP113	Instrument / Ser#	micrOTOF 10248
Comment	C25H16N2O6 mH 441.1081 calibrant added, CH3CN		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	1600 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS (ESI) spectra of 3p

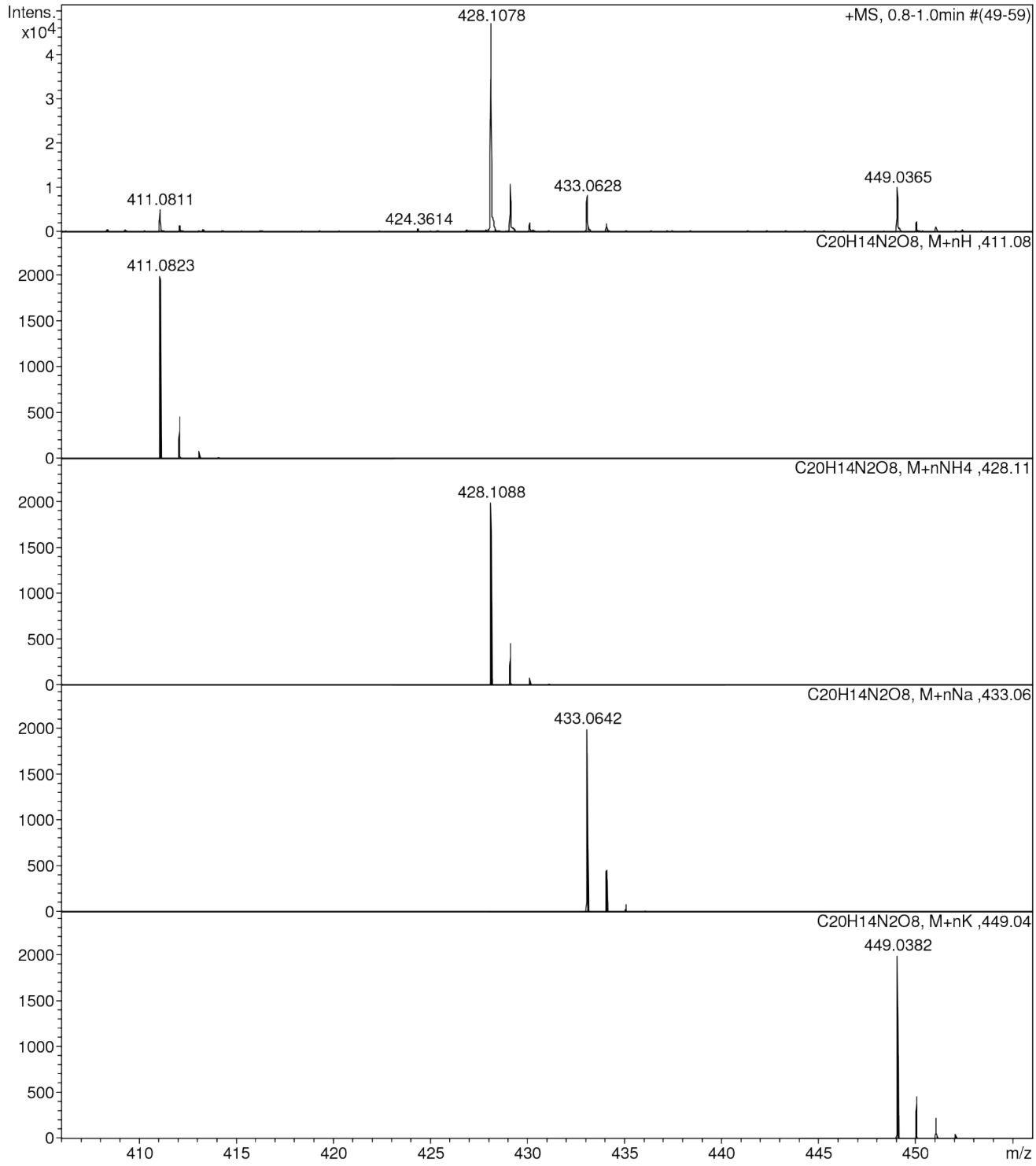
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2021\Dvoretsky\1019001.d	Acquisition Date	19.10.2021 8:38:34
Method	tune_50-1600.m	Operator	BDAL@DE
Sample Name	/TERN AD159_1	Instrument / Ser#	micrOTOF 10248
Comment	C20H14N2O8 mH 411.0822 calibrant added, CH3CN		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	1600 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS (ESI) spectra of 3q

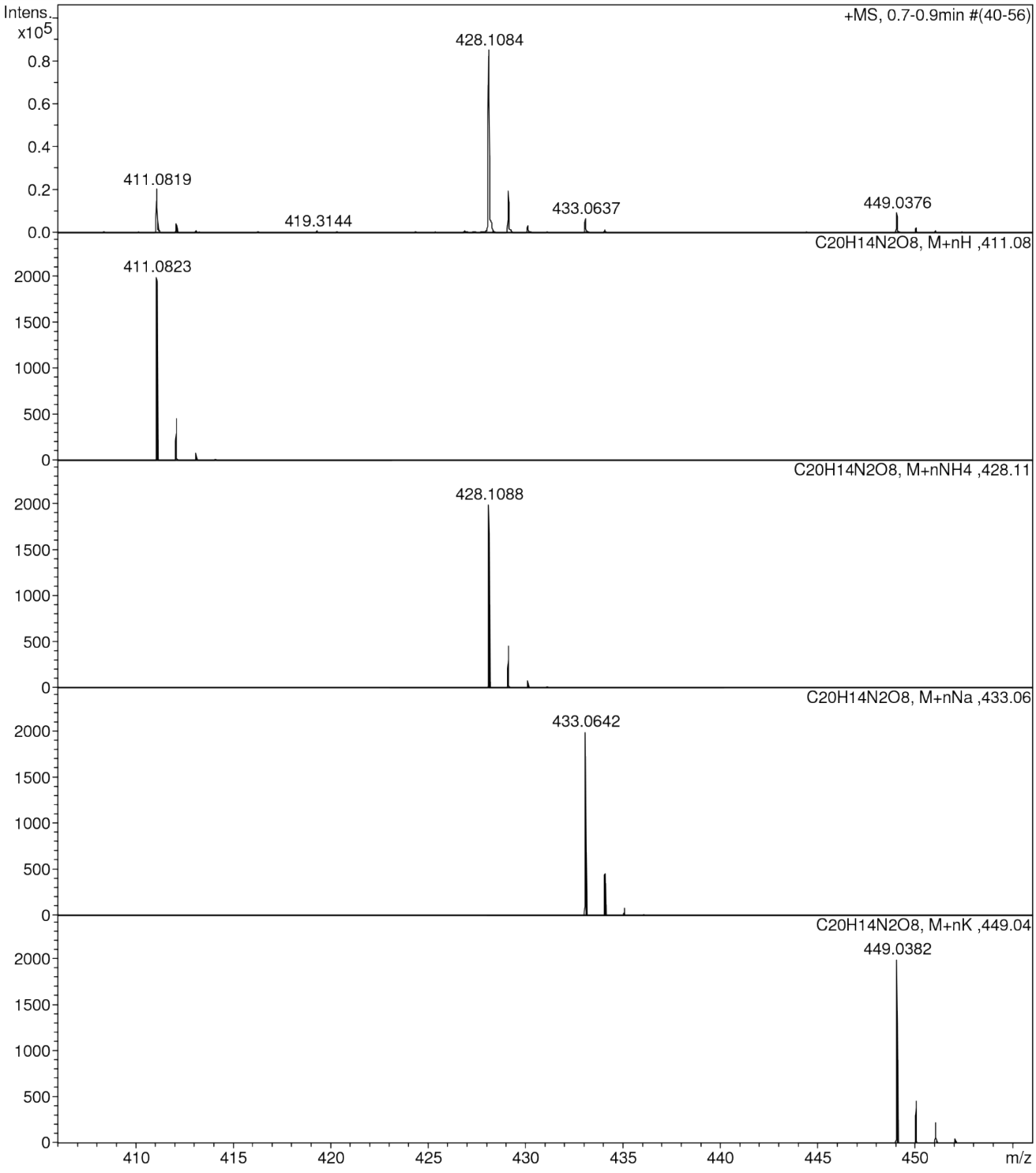
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2021\Dvoretsky\1019002.d	Acquisition Date	19.10.2021 9:14:13
Method	tune_50-1600.m	Operator	BDAL@DE
Sample Name	/TERN AD159_2	Instrument / Ser#	micrOTOF 10248
Comment	C20H14N2O8 mH 411.0822 calibrant added, CH3CN		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active			Set Dry Heater	200 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	1600 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS (ESI) spectra of 3r

Display Report

Analysis Info

Analysis Name	D:\Data\Chizhov\Terentiev\Segida\osp131_&clb.d	Acquisition Date	25.10.2021 17:43:25
Method	tune_wide.m	Operator	BDAL@DE
Sample Name	/TERN OSP131	Instrument / Ser#	micrOTOF 10248
Comment	CH3OH 100 %, dil. 200, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste