

Direct Synthesis of Amides from Carboxylic Acids and Amines using $\text{B}(\text{OCH}_2\text{CF}_3)_3$

Rachel M. Lanigan, Pavel Starkov, and Tom D. Sheppard*

*Department of Chemistry, University College London,
Christopher Ingold Laboratories, 20 Gordon St, London, WC1H 0AJ, UK*

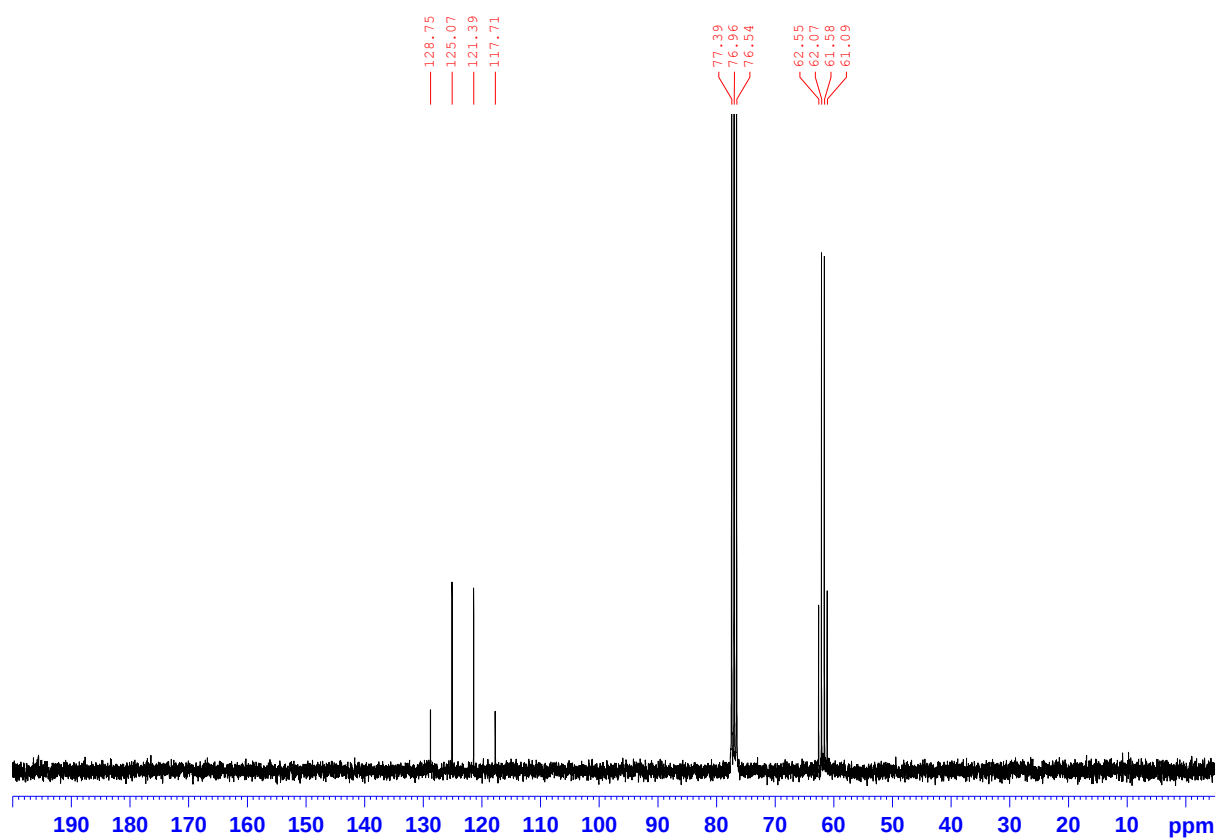
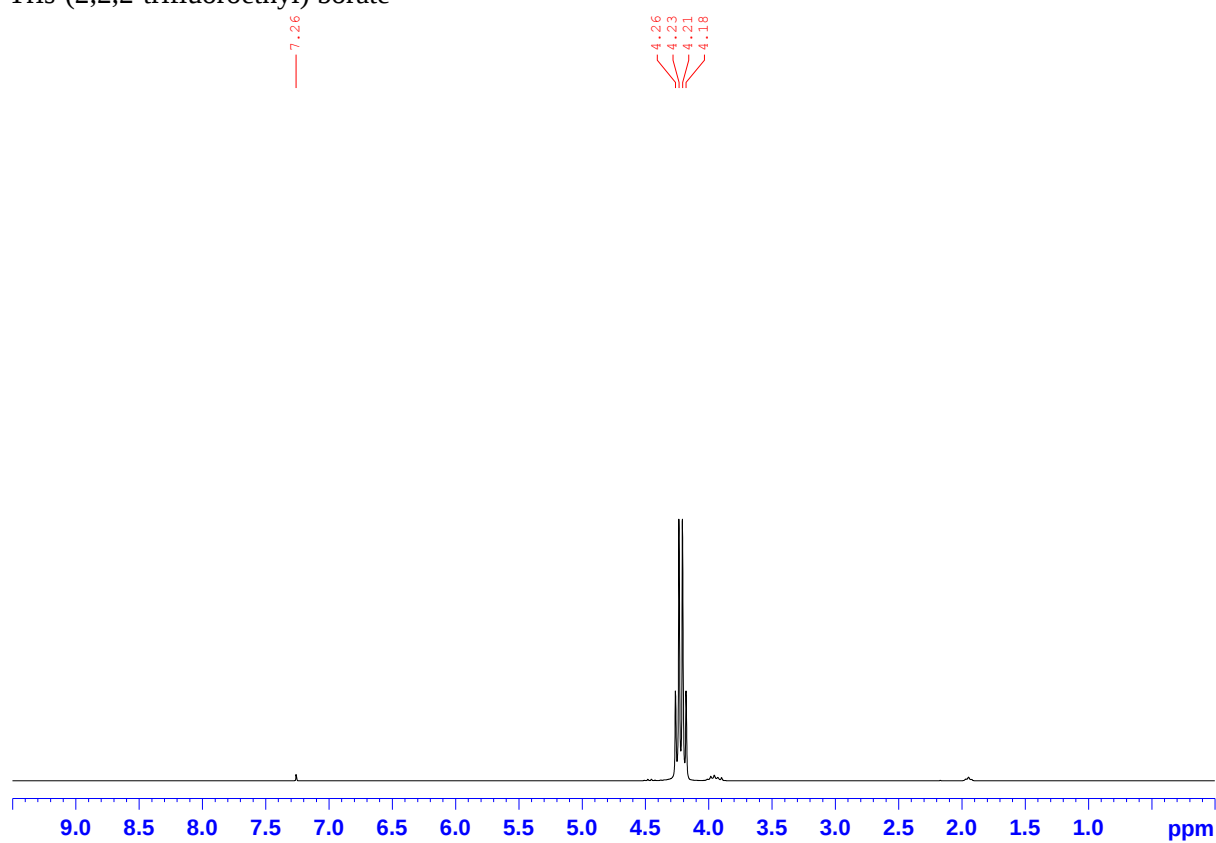
tom.sheppard@ucl.ac.uk

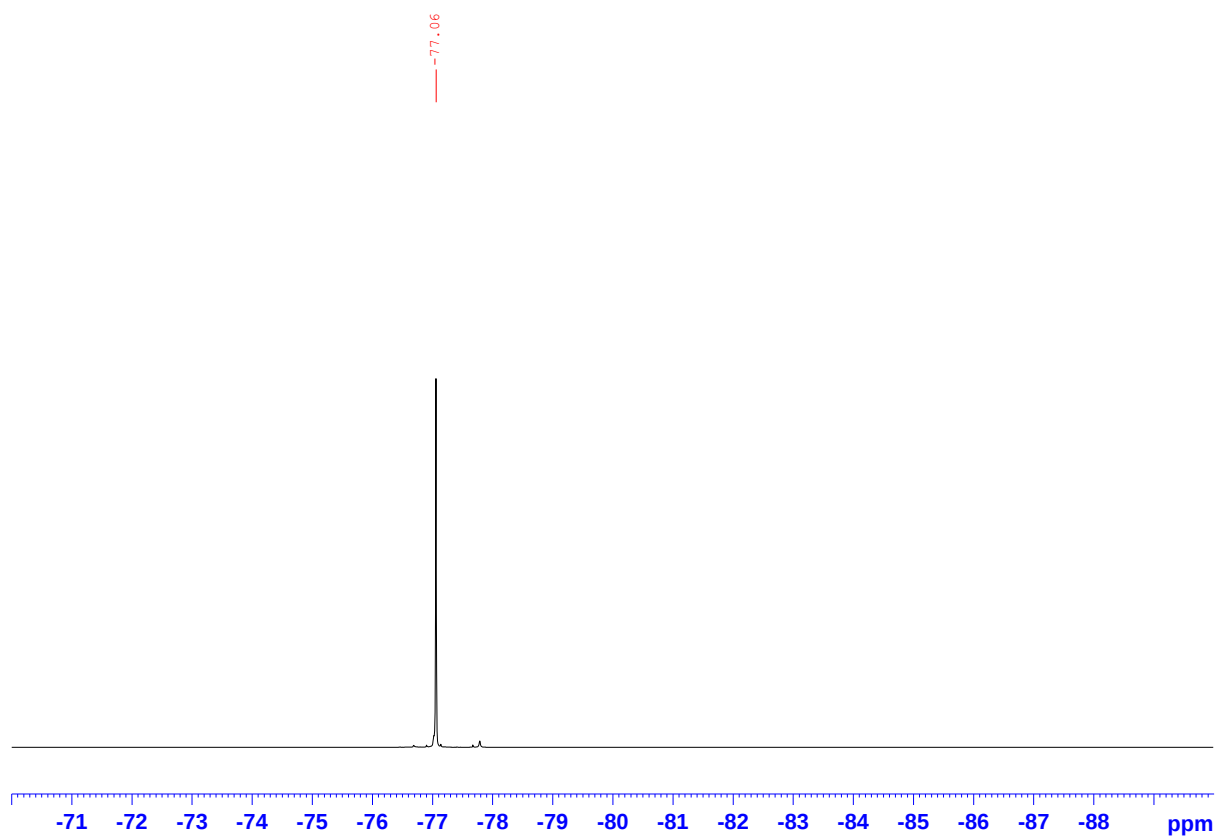
Table of Contents

1. ^1H and ^{13}C NMR Spectra	1
2. Determination of enantiopurity of chiral amides	67

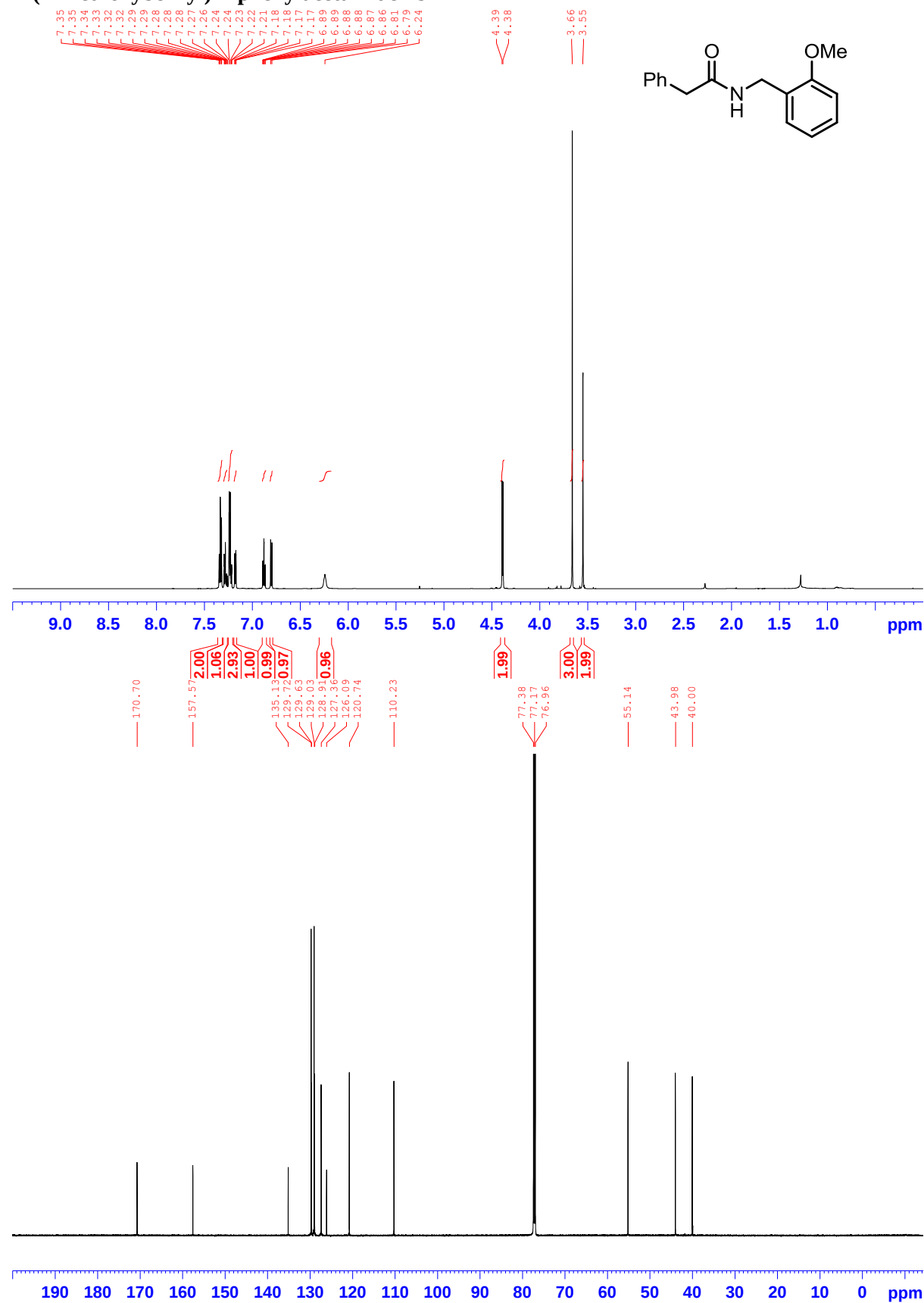
1 NMR Spectra

Tris-(2,2,2-trifluoroethyl) borate

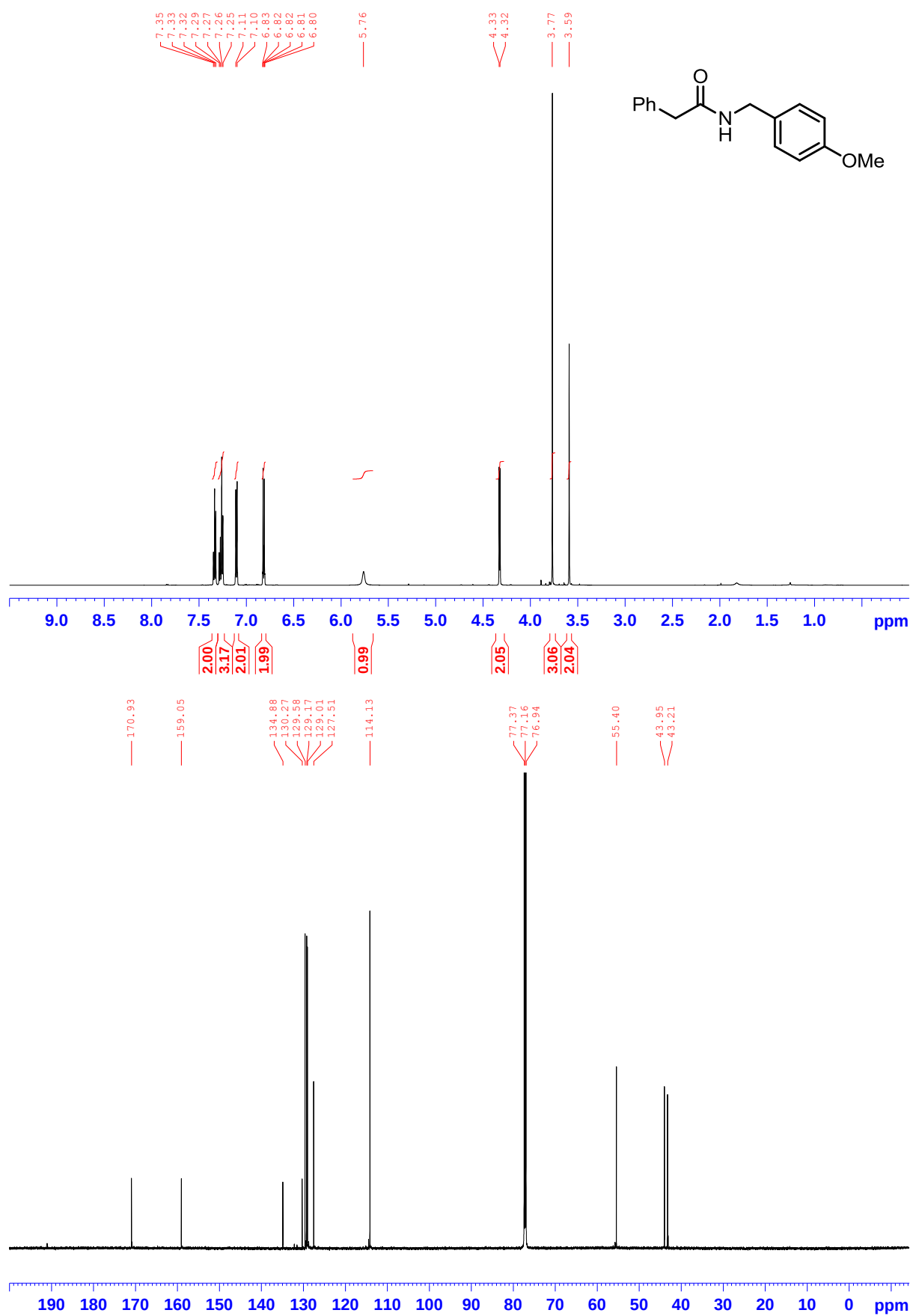




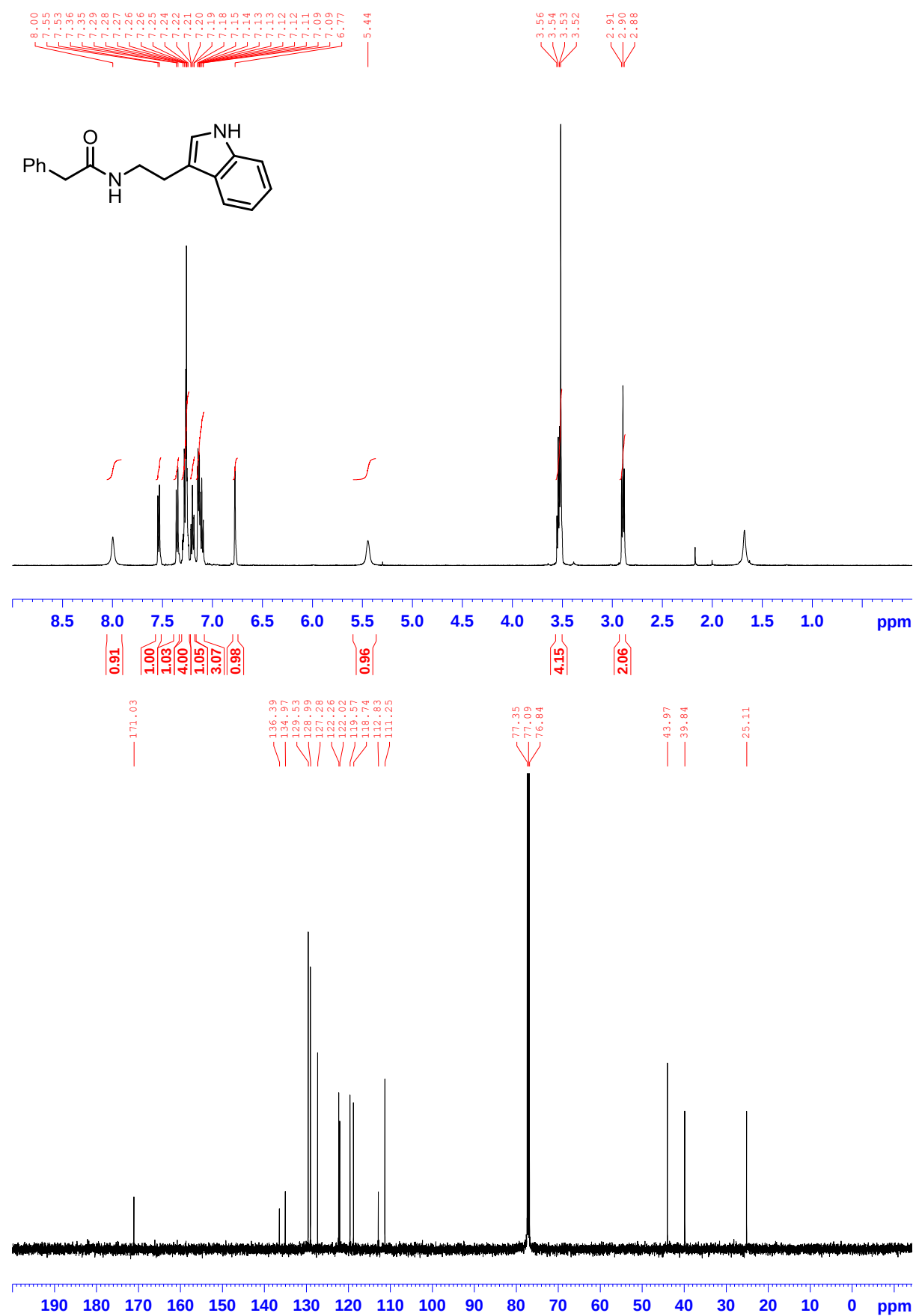
***N*-(2-Methoxybenzyl)-2-phenylacetamide 2b**



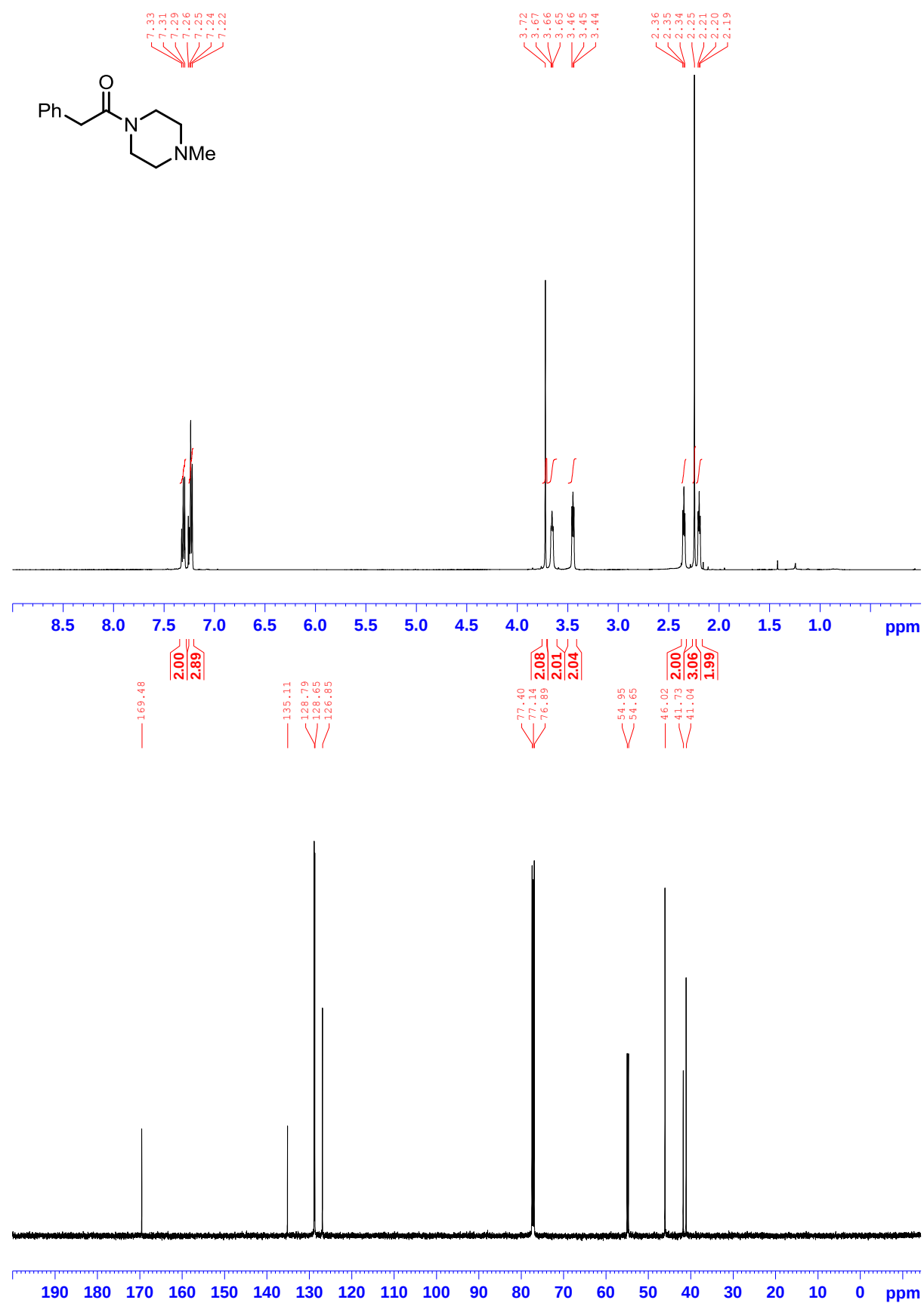
***N*-(4-Methoxybenzyl)-2-phenylacetamide 2c**



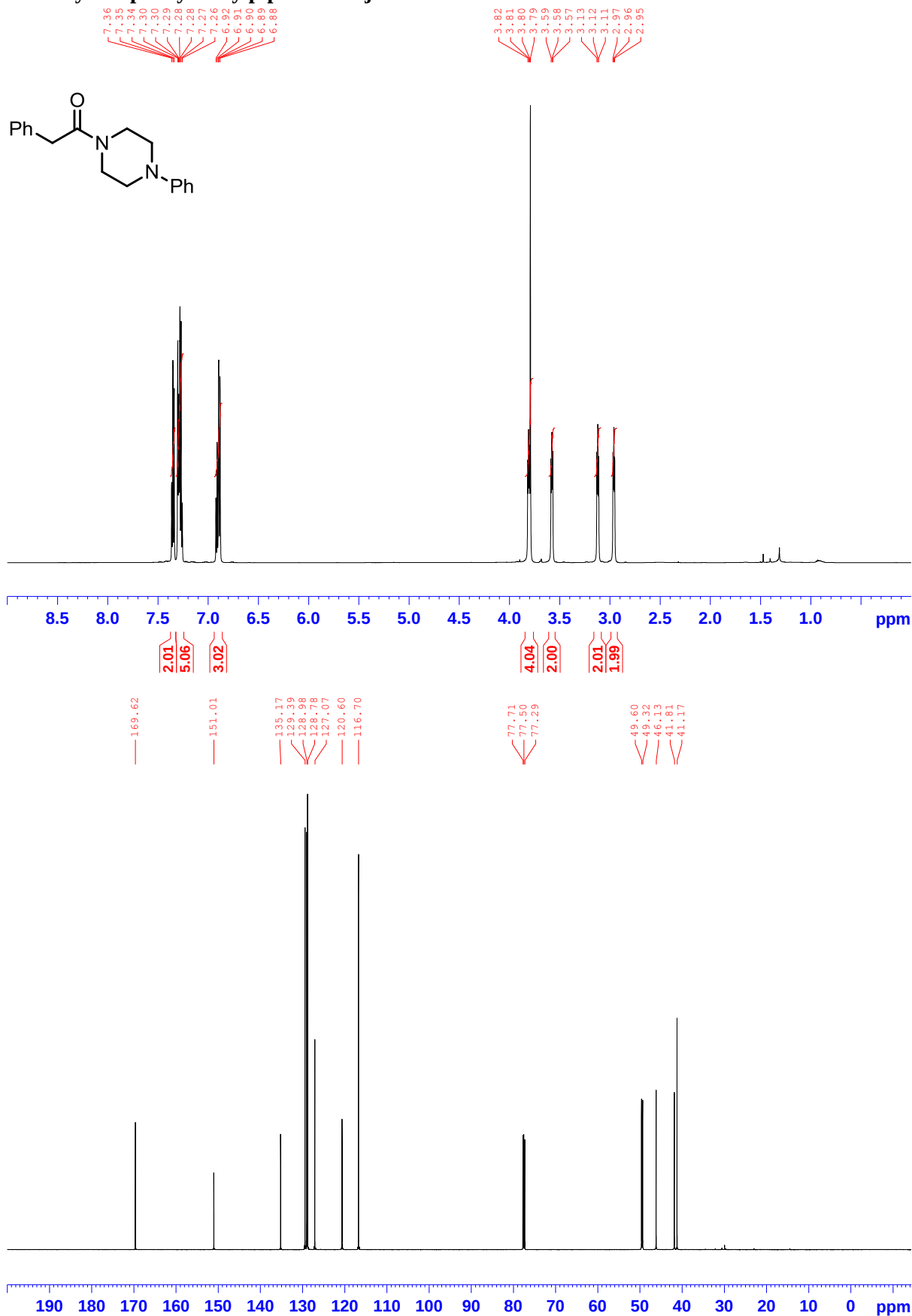
***N*-(2-(1H-Indol-3-yl)ethyl)-2-phenylacetamide 2f**



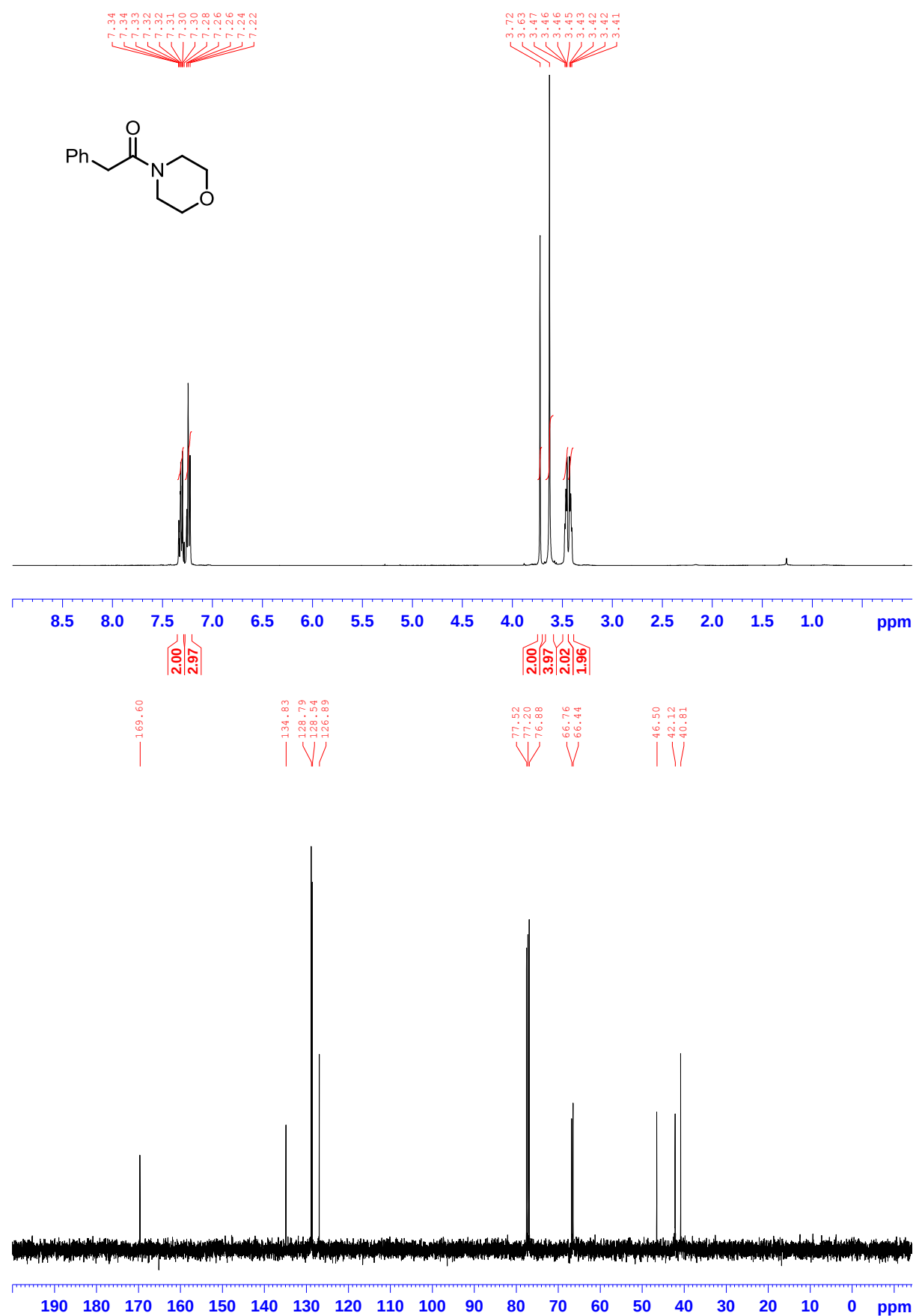
***N*-Methyl-*N'*-phenylacetylpiiperazine 2i**



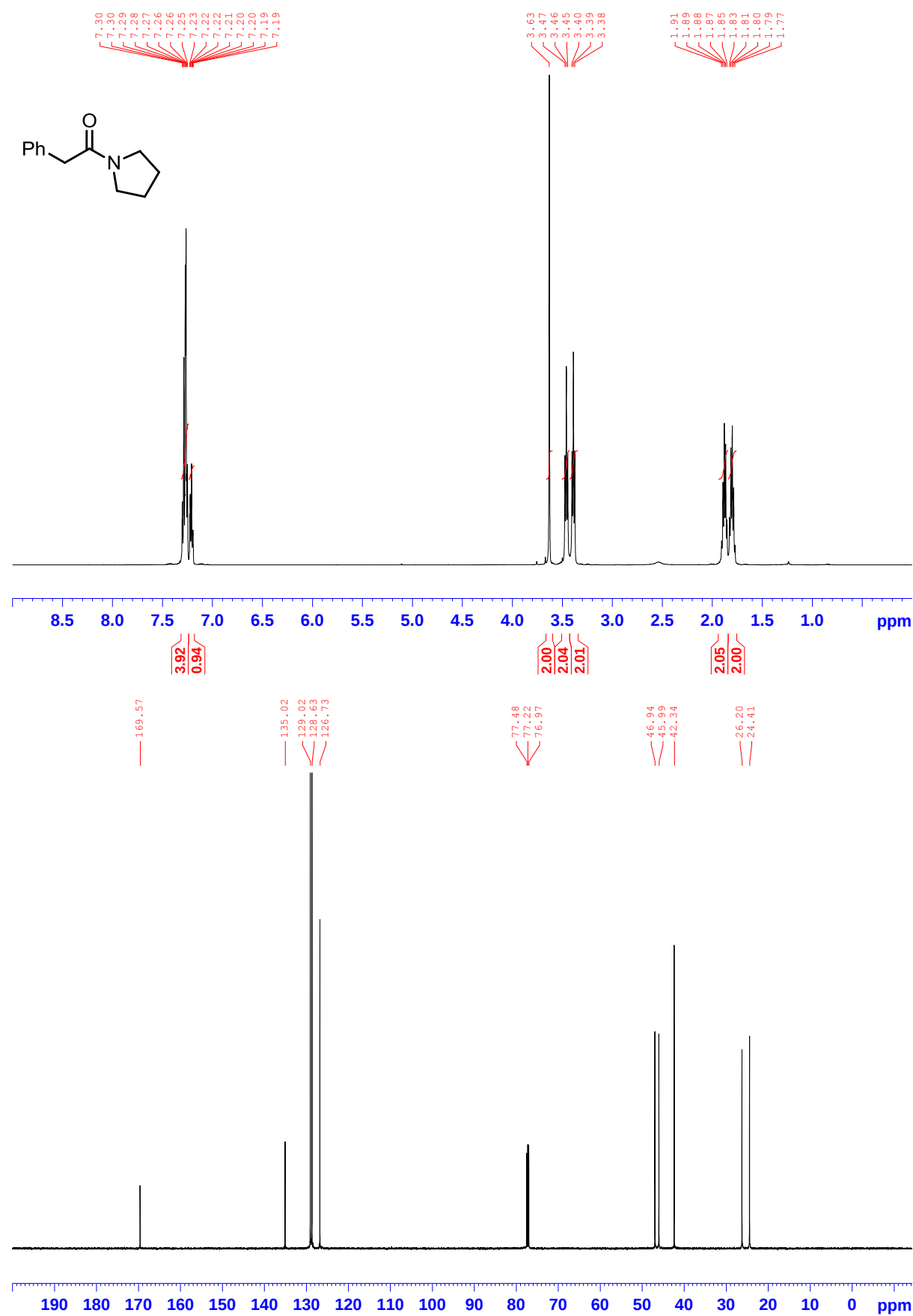
***N*-Phenyl-*N'*-phenylacetylpiiperazine 2j**



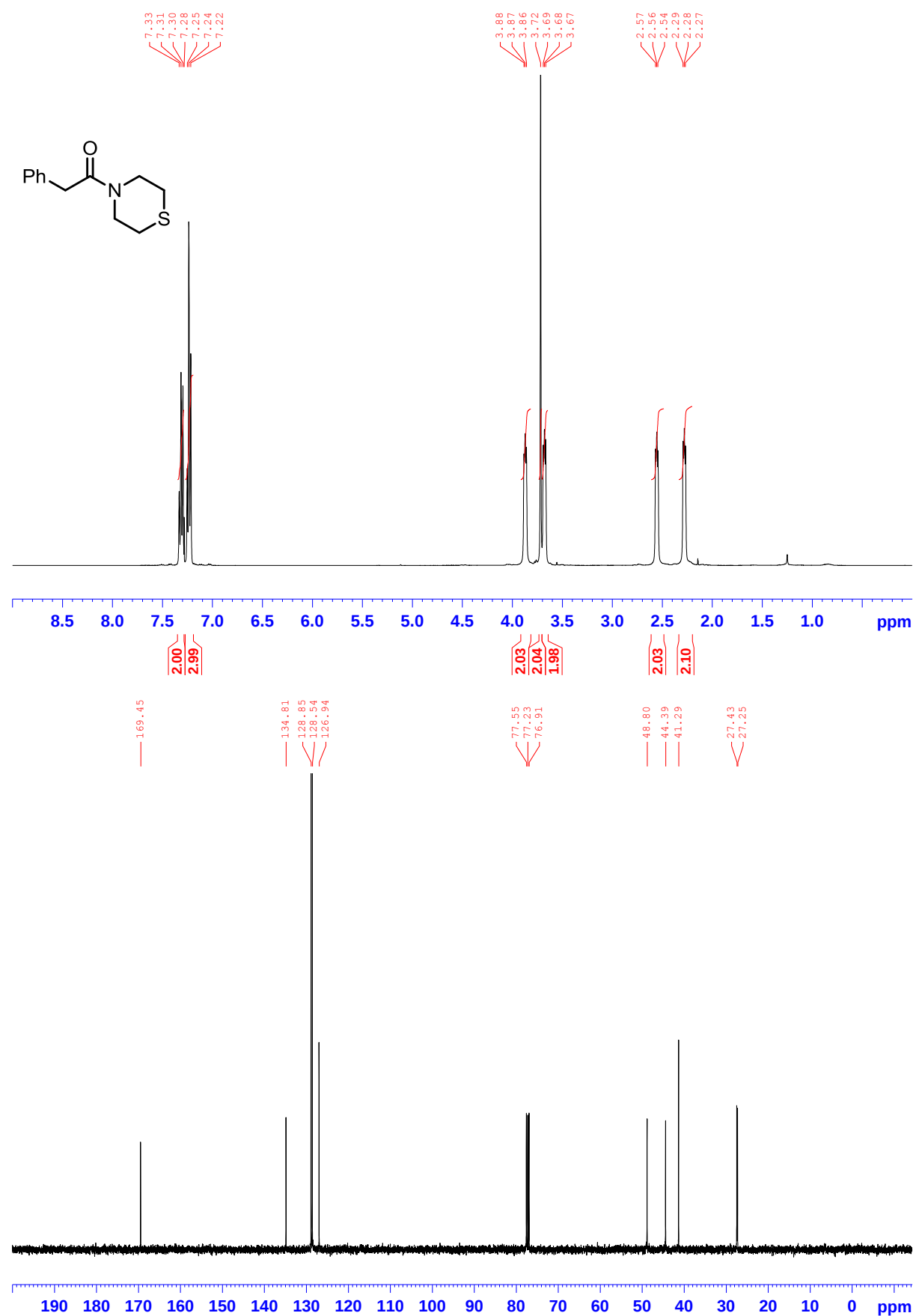
N-Phenylacetylmorpholine 2k



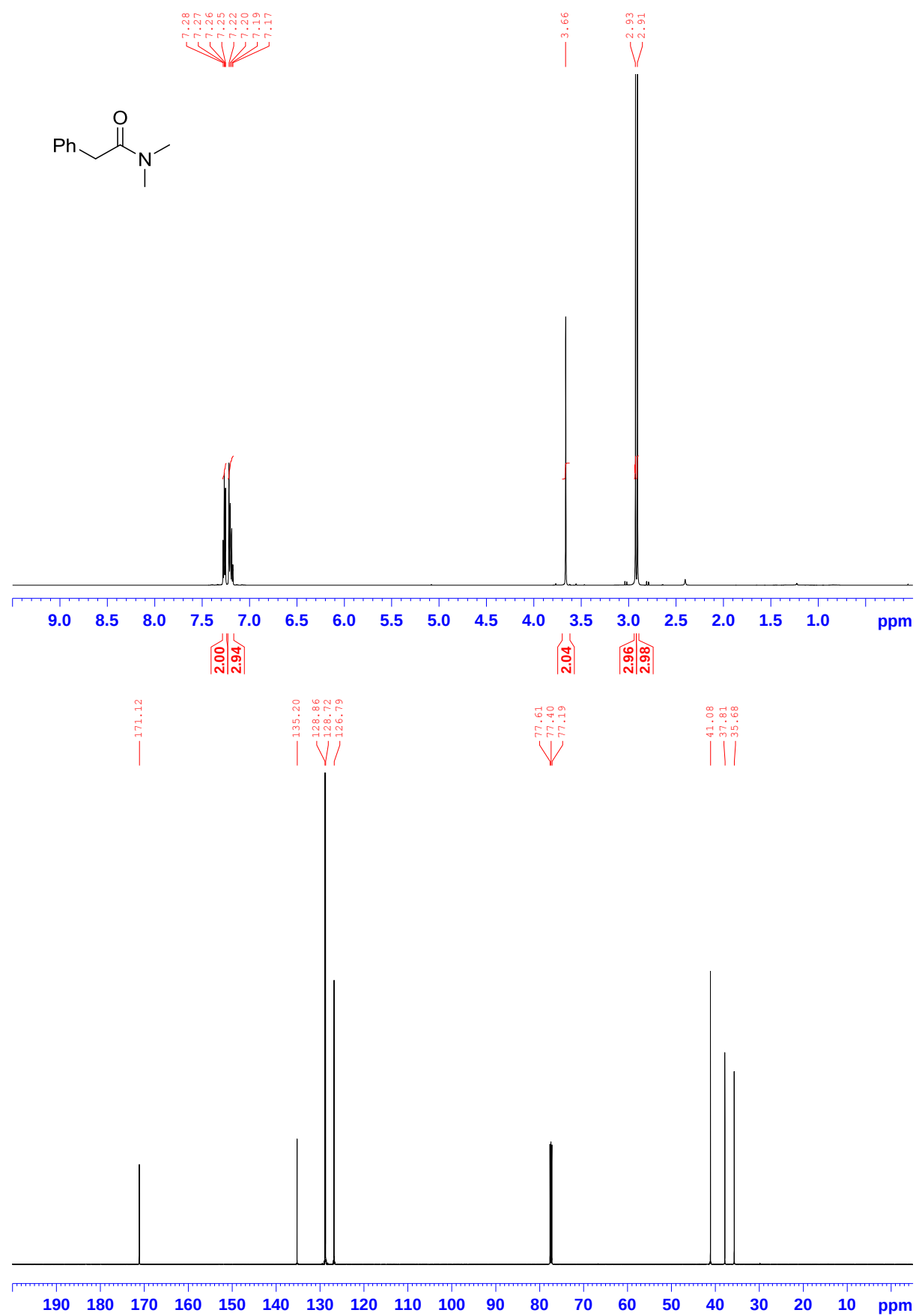
2-Phenyl-1-(pyrrolidin-1-yl)ethanone 21



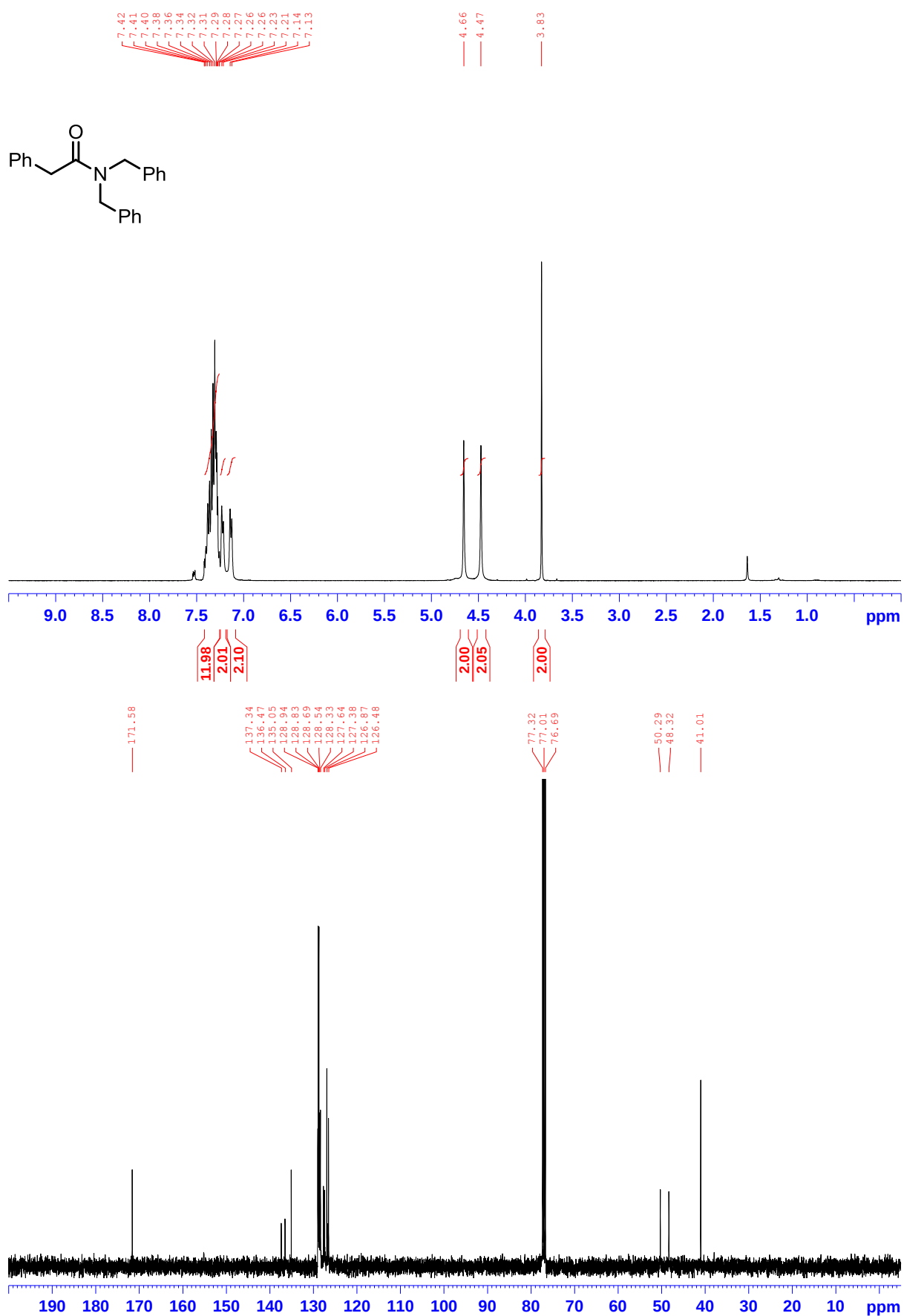
N-Phenylacetylthiomorpholine 2m



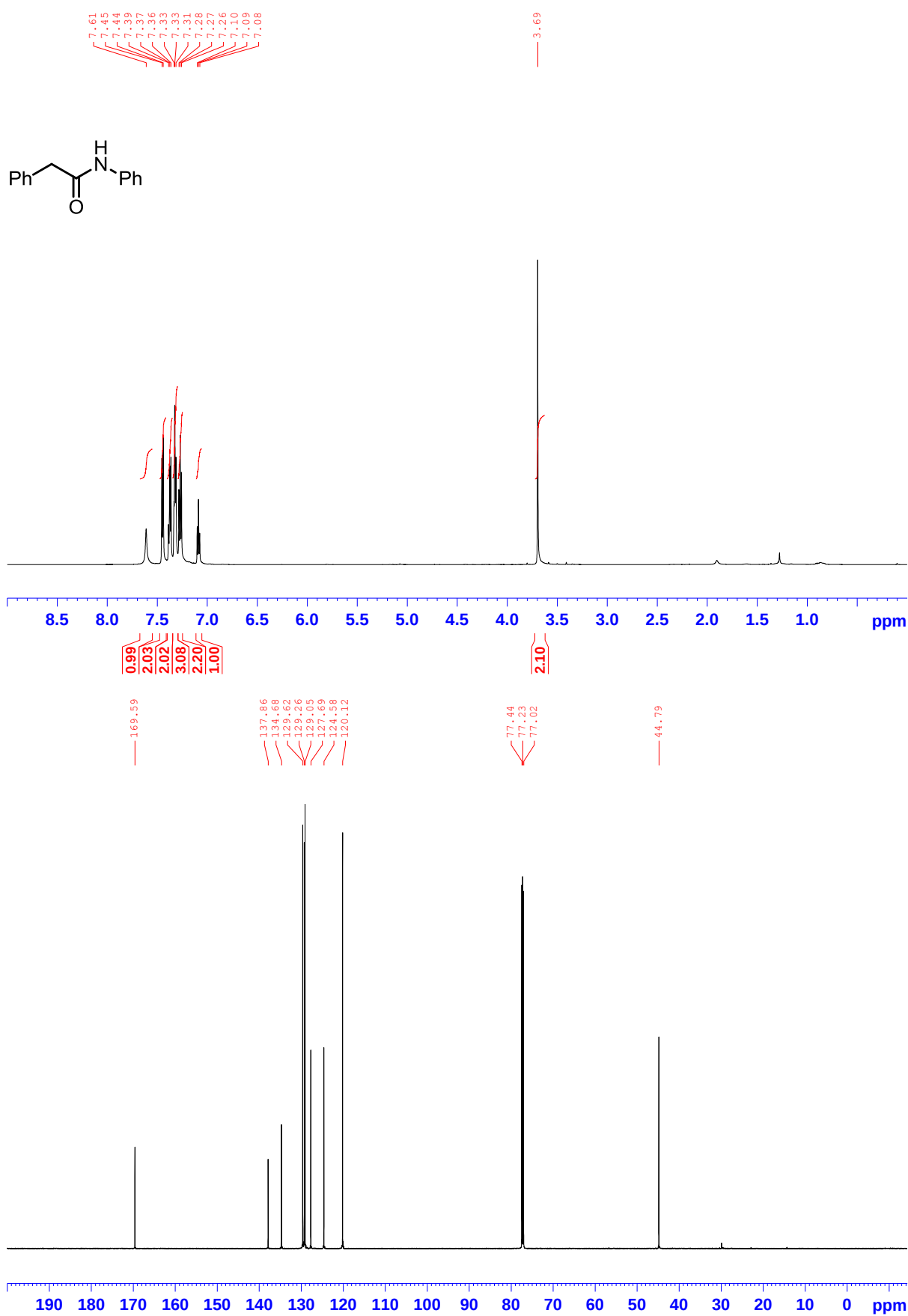
***N,N*-Dimethyl-2-phenylacetamide 2n**



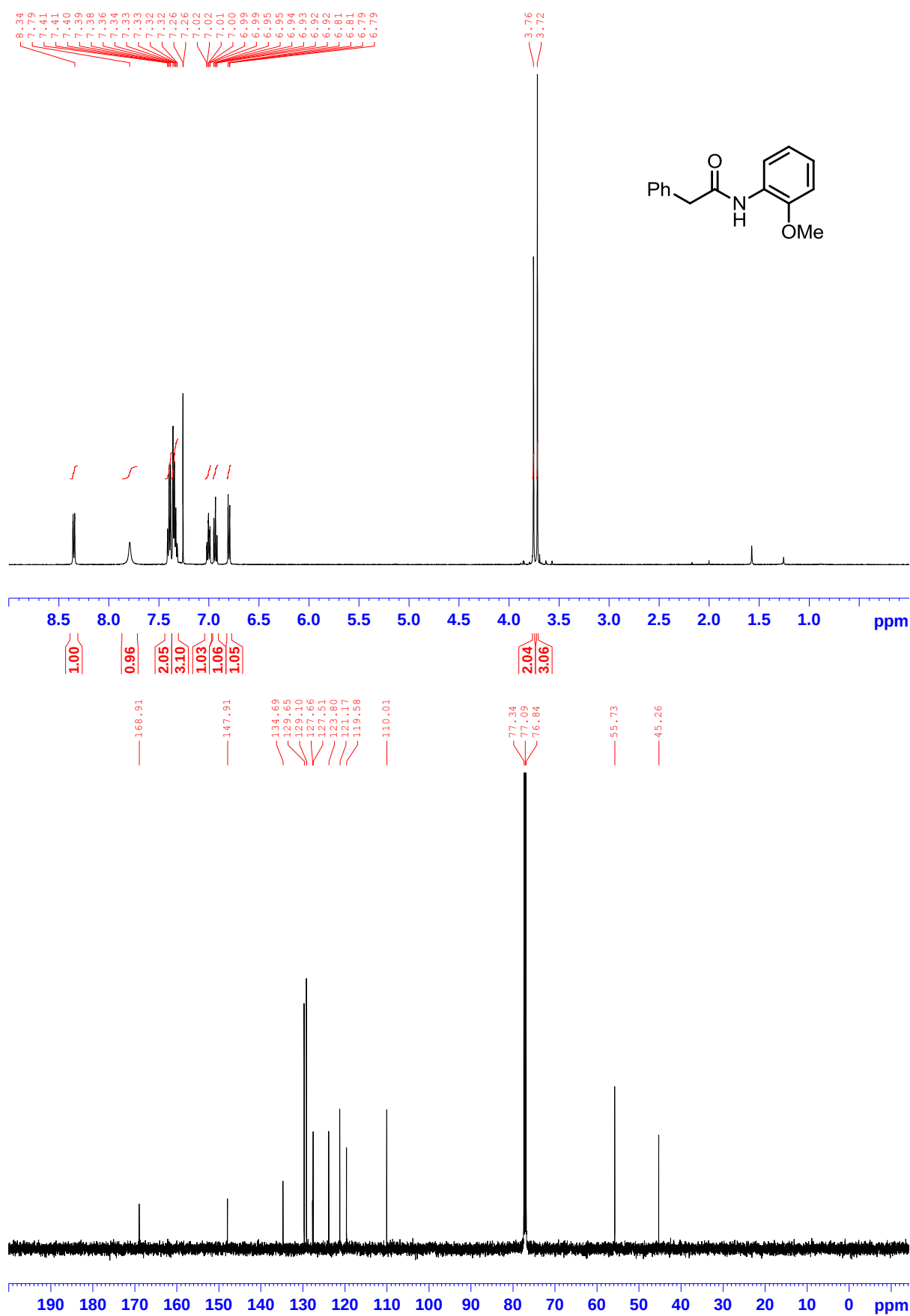
***N,N*-Dibenzyl-2-phenylacetamide 2o**



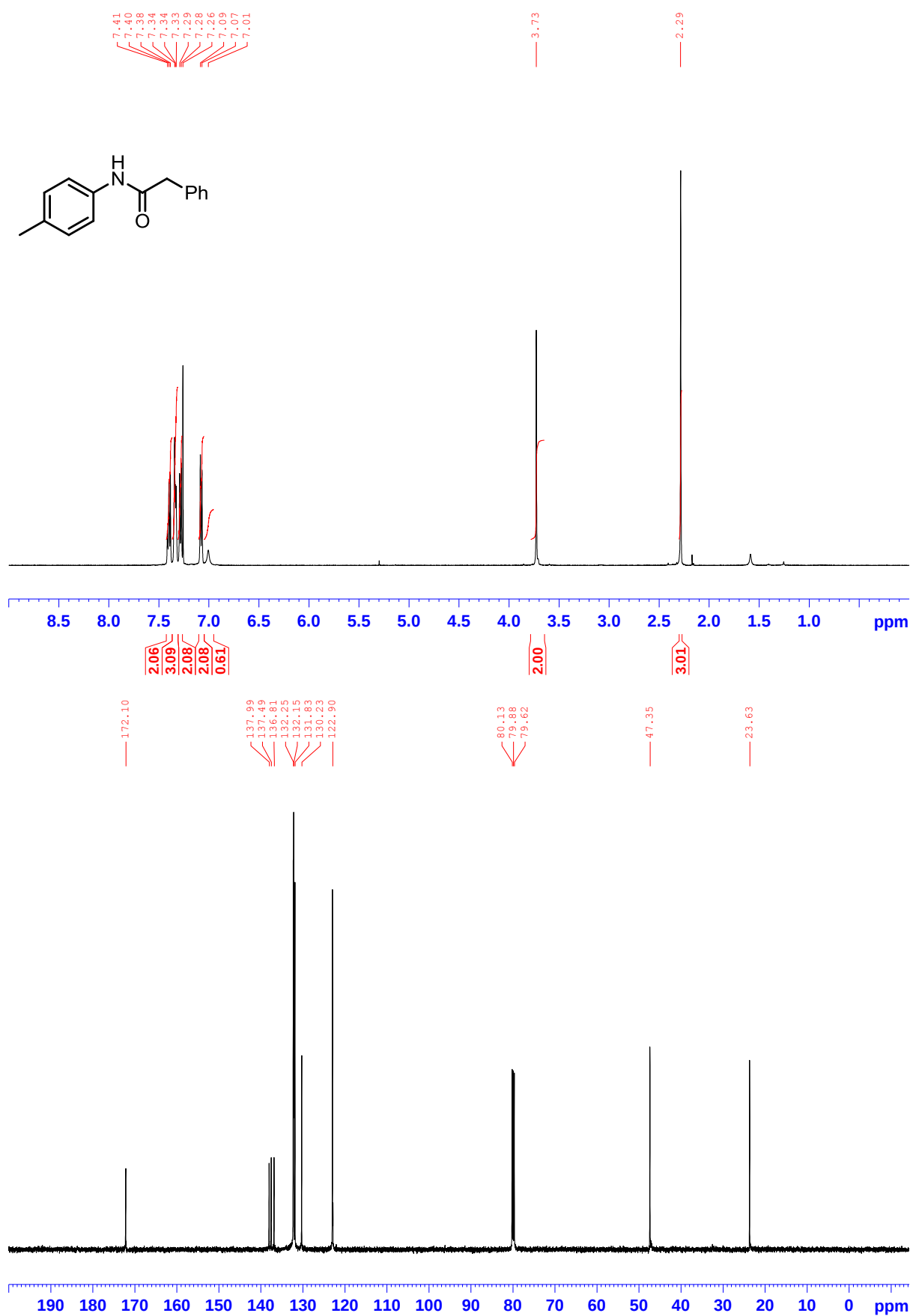
N,2-Diphenylacetamide 2p



***N*-(2-Methoxyphenyl)-2-phenylacetamide 2q**



2-Phenyl-N-p-tolylacetamide 2r

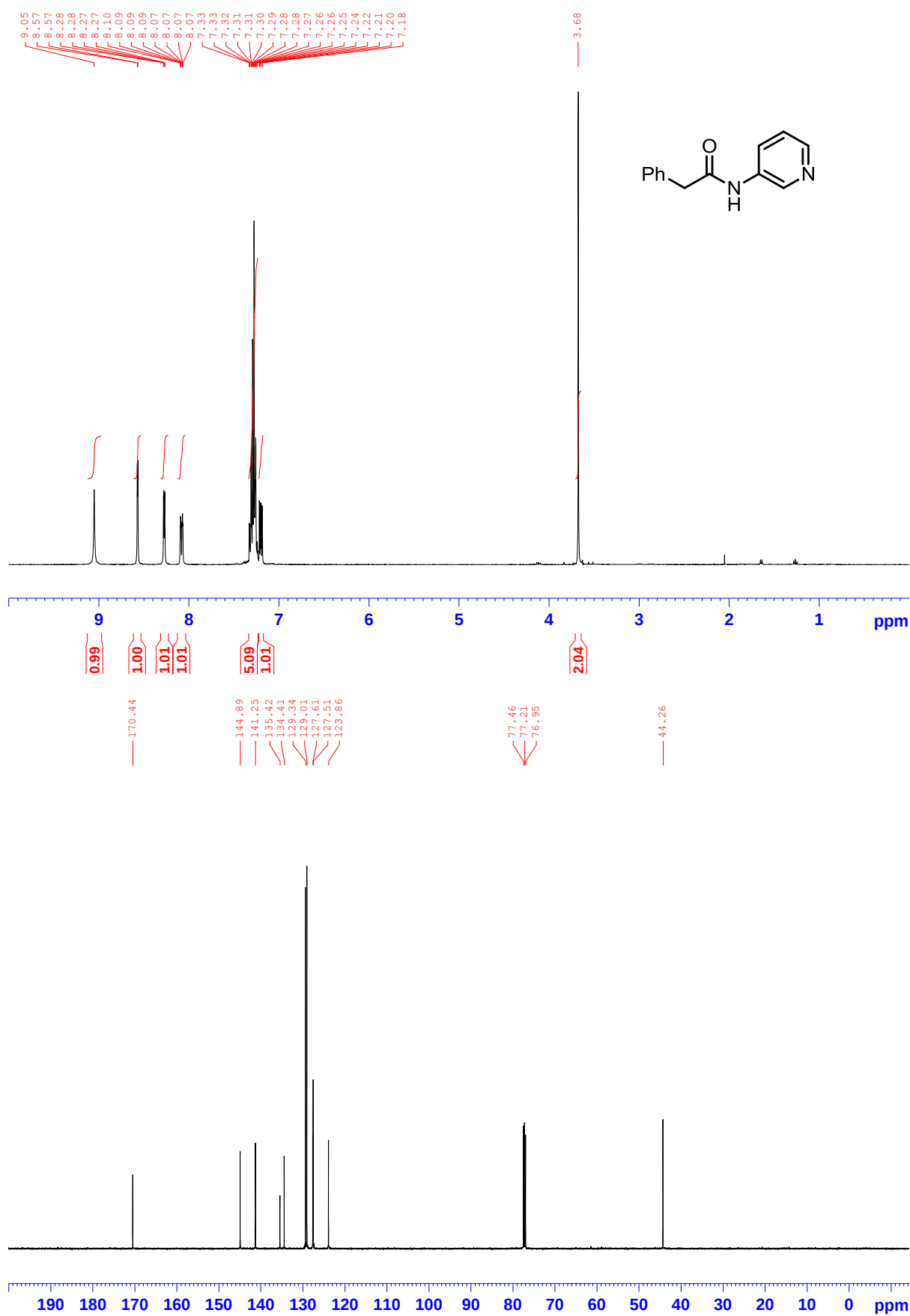


COc1ccc(NC(=O)Cc2ccccc2)cc1

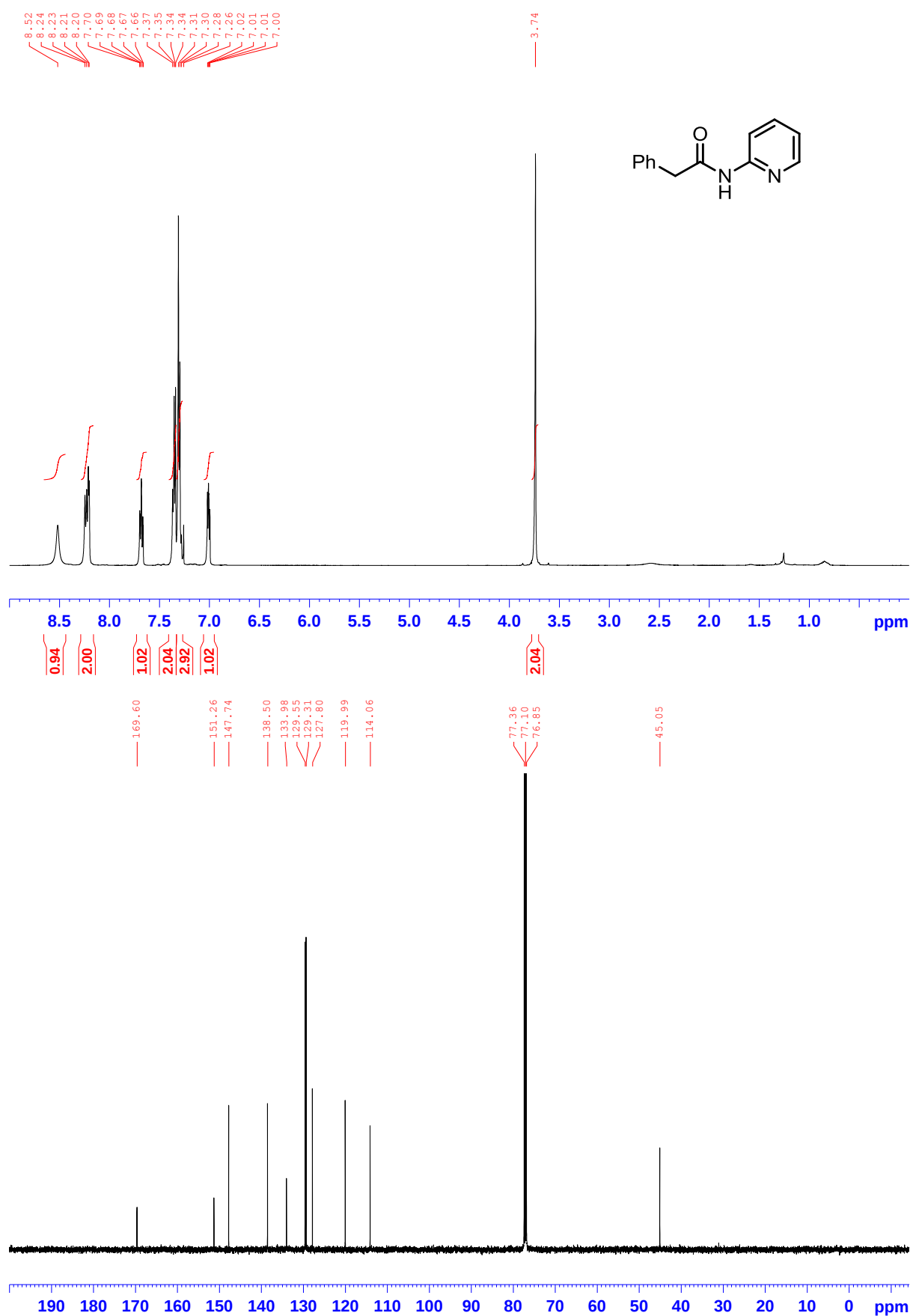
¹H NMR spectrum (400 MHz, CDCl₃) of N-(4-methoxyphenyl)-2-phenylacetamide. The spectrum shows peaks in the aromatic region (6.80-7.41 ppm) and aliphatic region (3.73-3.77 ppm). Integration values are provided for the peaks.

¹³C NMR spectrum (100 MHz, CDCl₃) of N-(4-methoxyphenyl)-2-phenylacetamide. The spectrum shows peaks in the aromatic region (114.15-169.01 ppm) and aliphatic region (44.76-55.54 ppm).

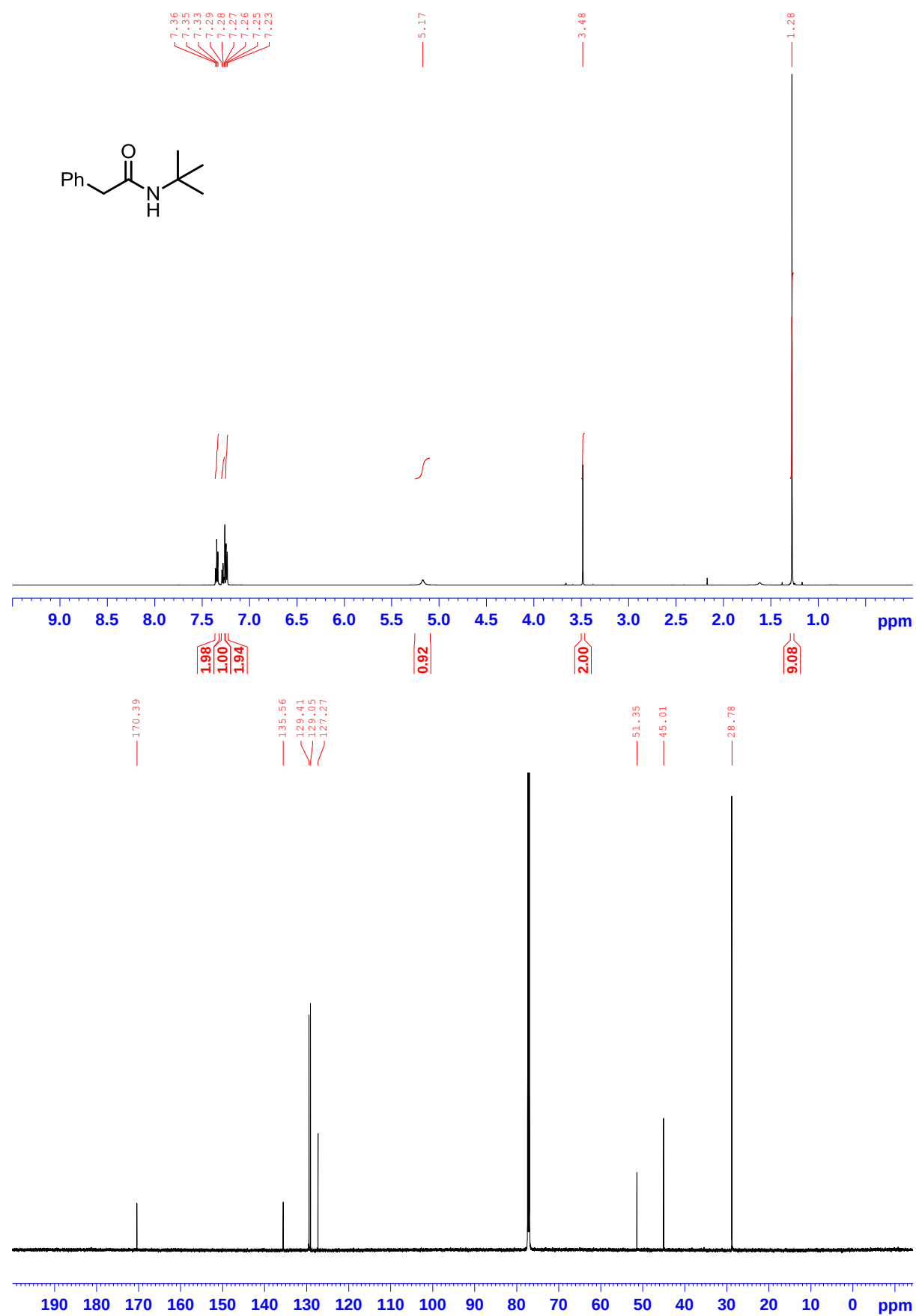
N-(Pyridin-3-yl)-2-phenylacetamide 2t



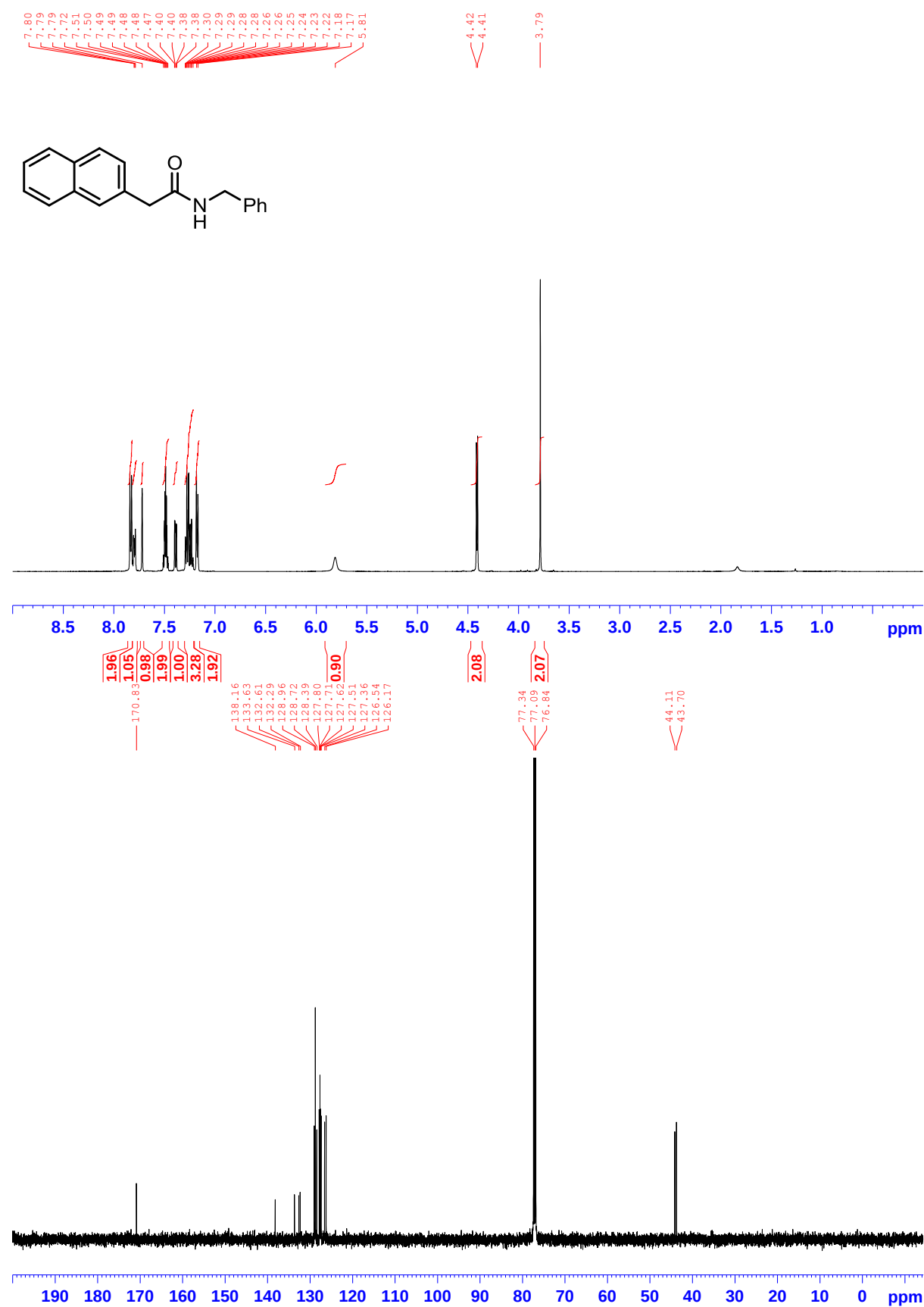
***N*-(Pyridin-2-yl)-2-phenylacetamide 2u**



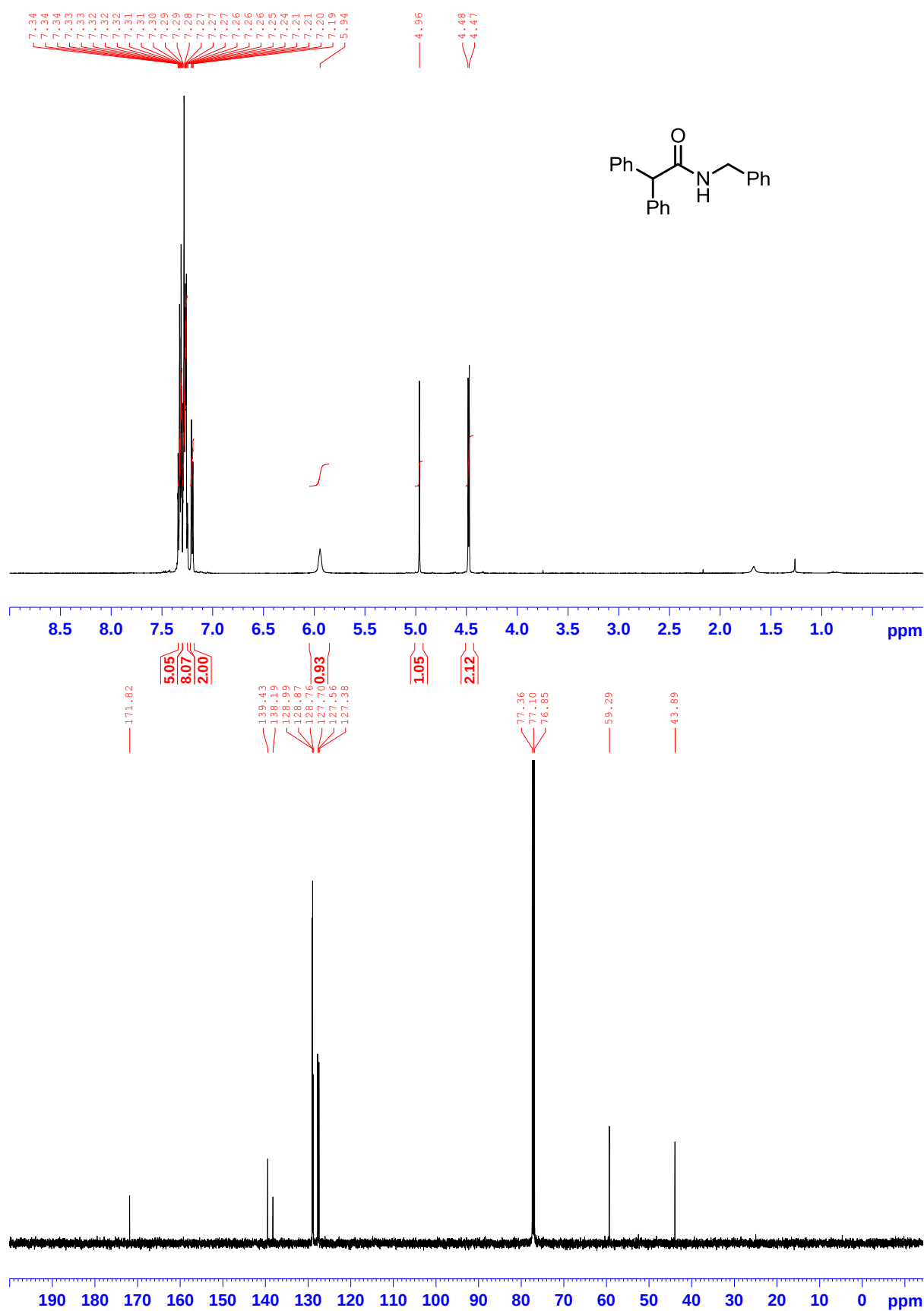
***N*-tert-Butyl-2-phenylacetamide 2v**



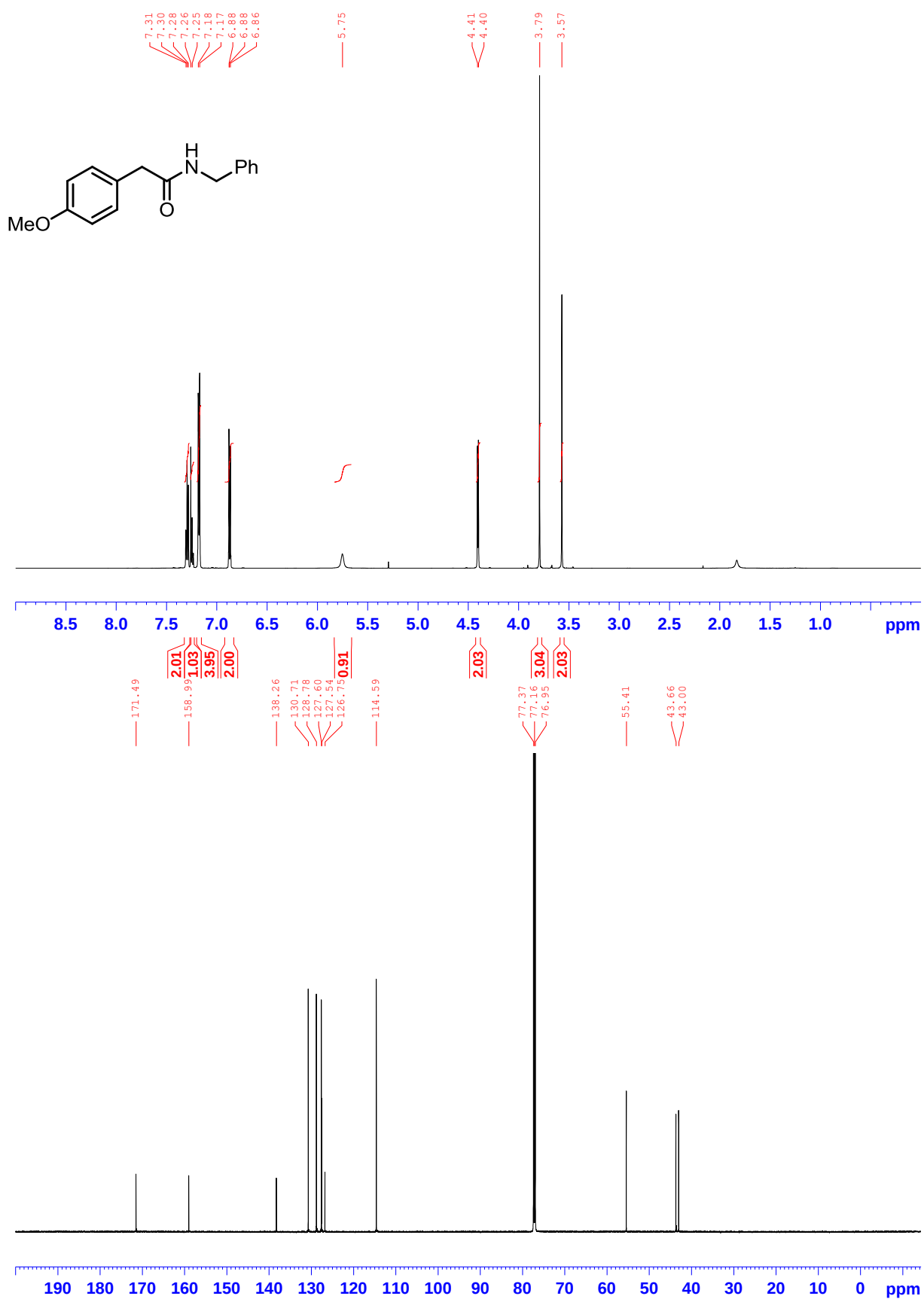
***N*-Benzyl-2-(naphthalen-2-yl)acetamide 3a**



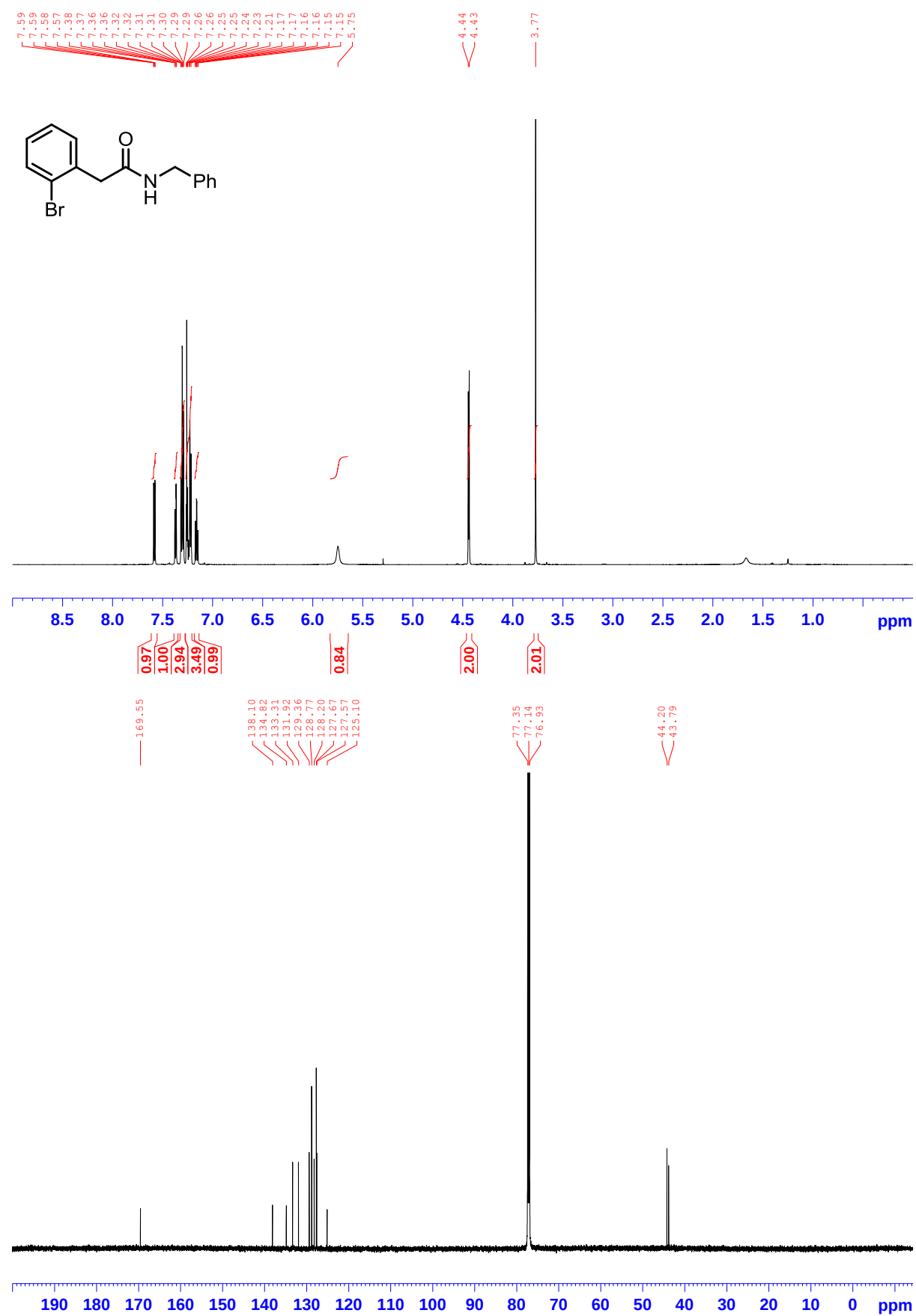
***N*-Benzyl-2,2-diphenylacetamide 3b**



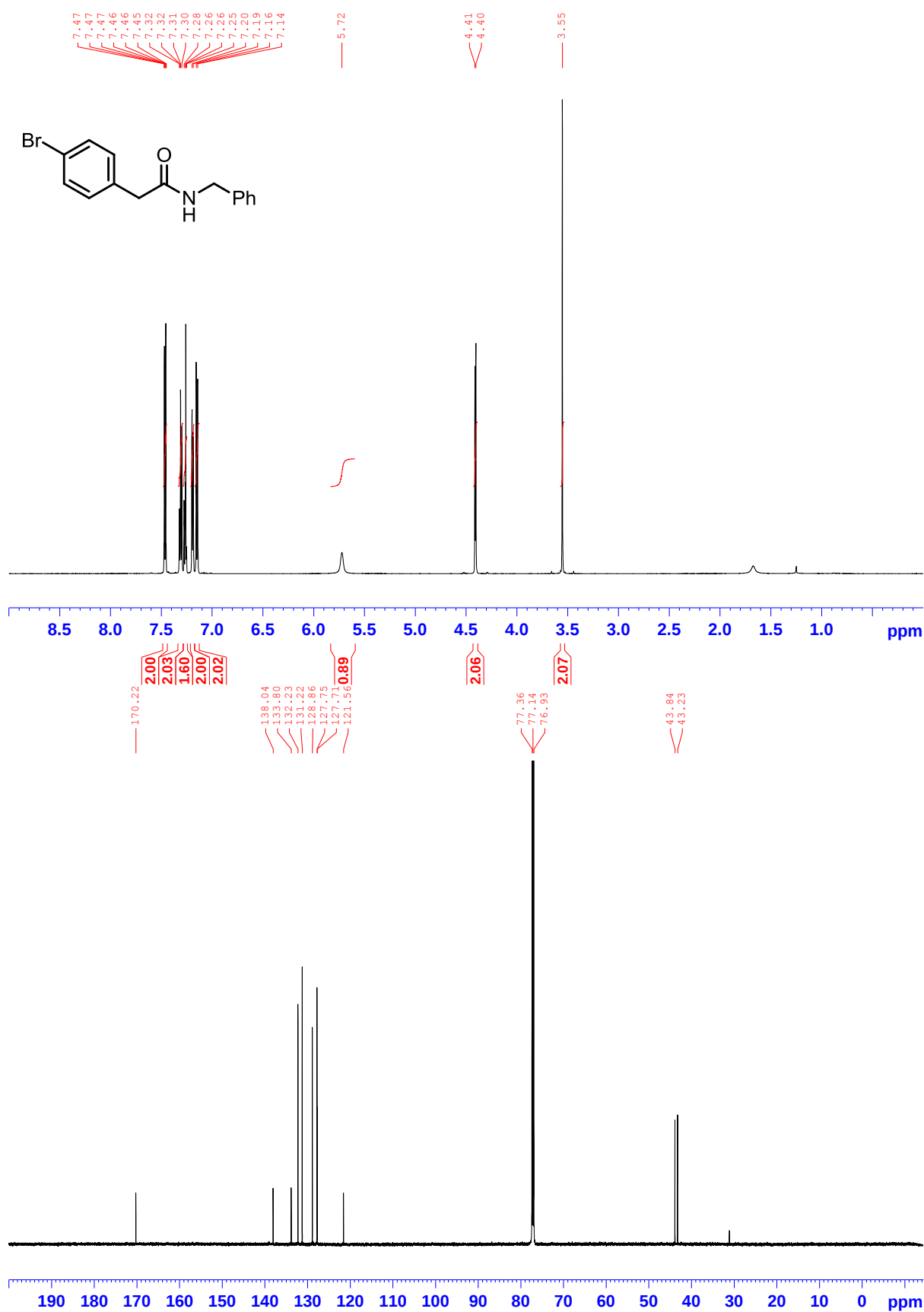
***N*-Benzyl-2-(4-methoxyphenyl)acetamide 3c**



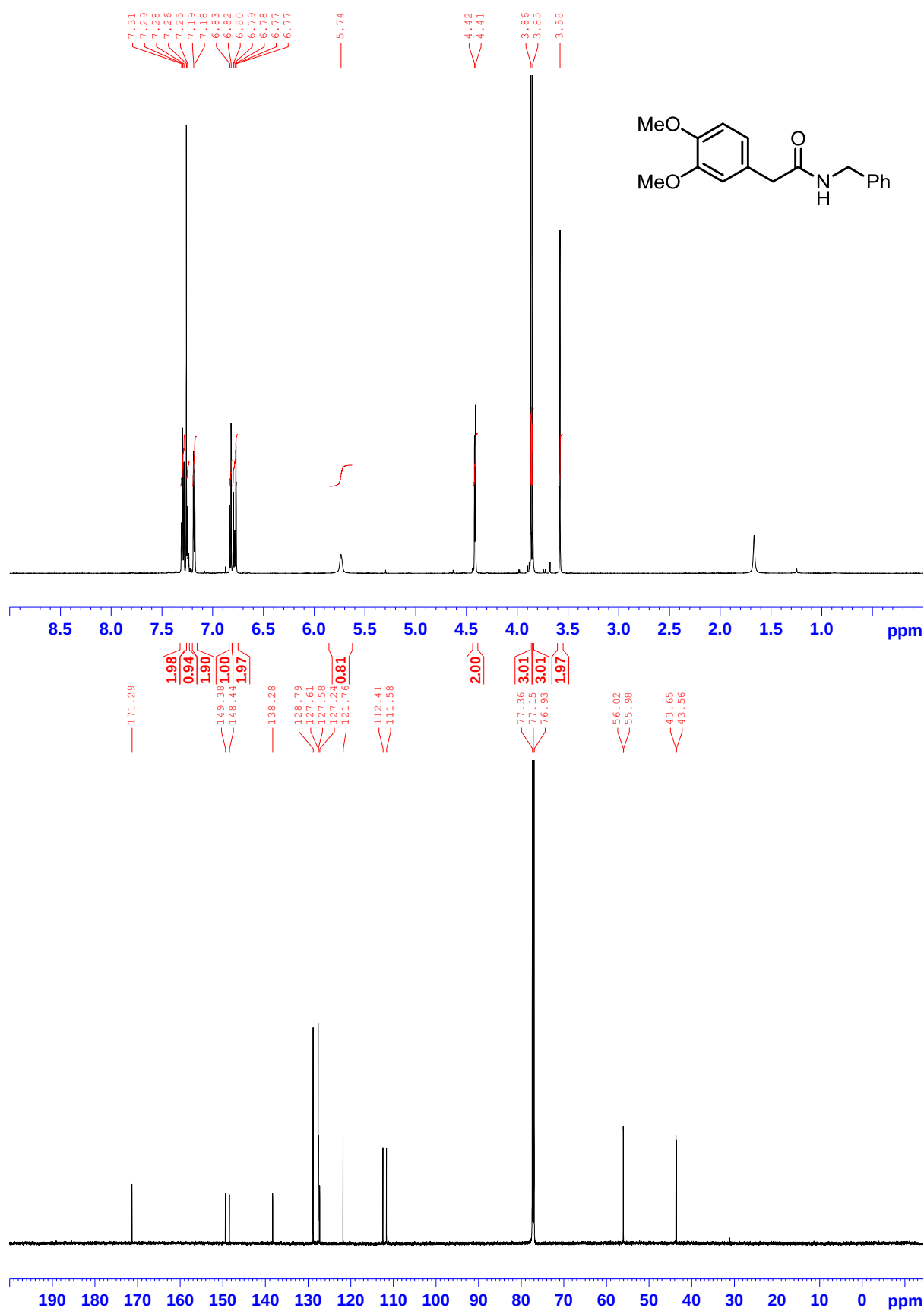
N-Benzyl-2-(2-bromophenyl)acetamide 3d



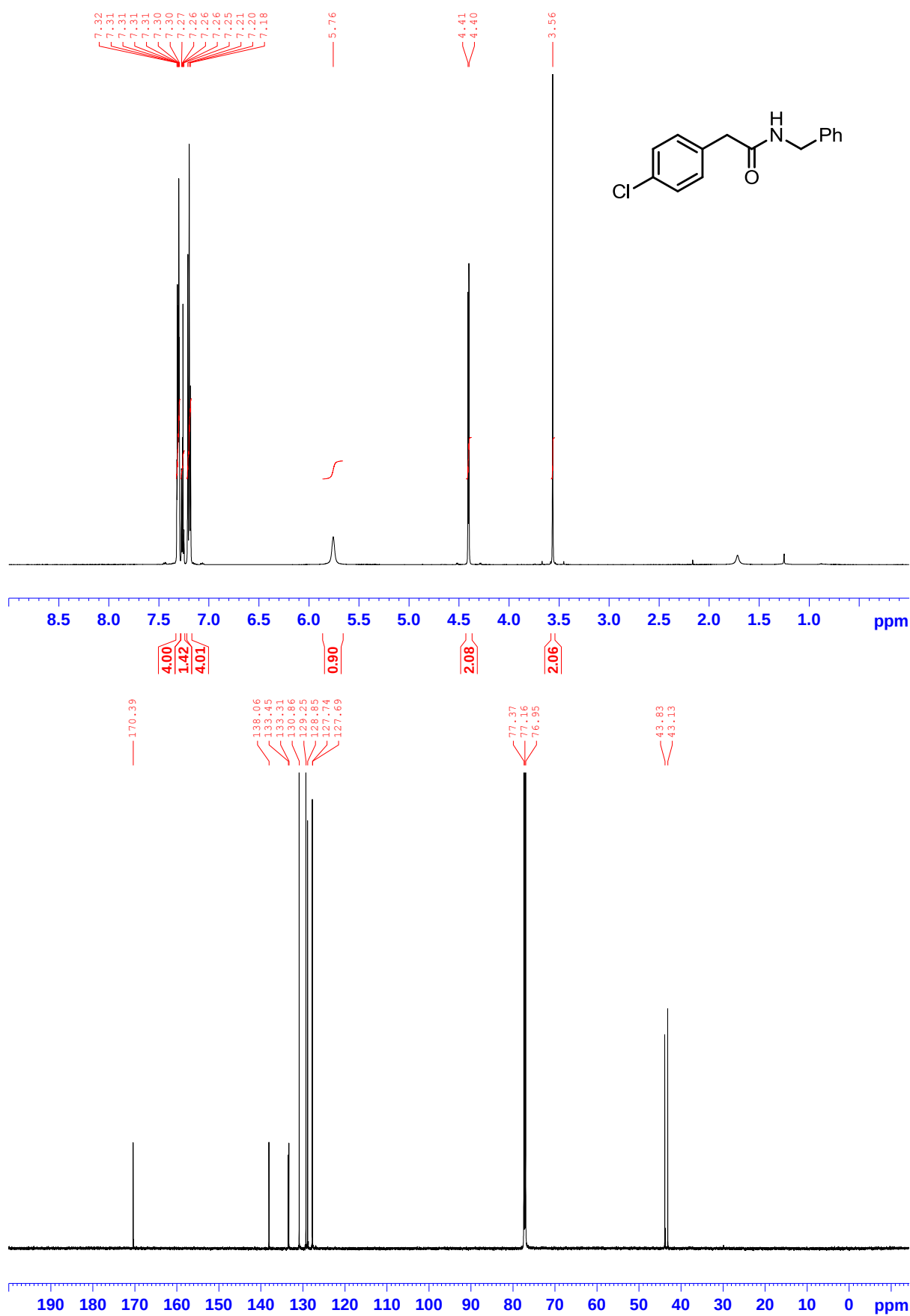
***N*-Benzyl-2-(4-bromophenyl)acetamide 3e**



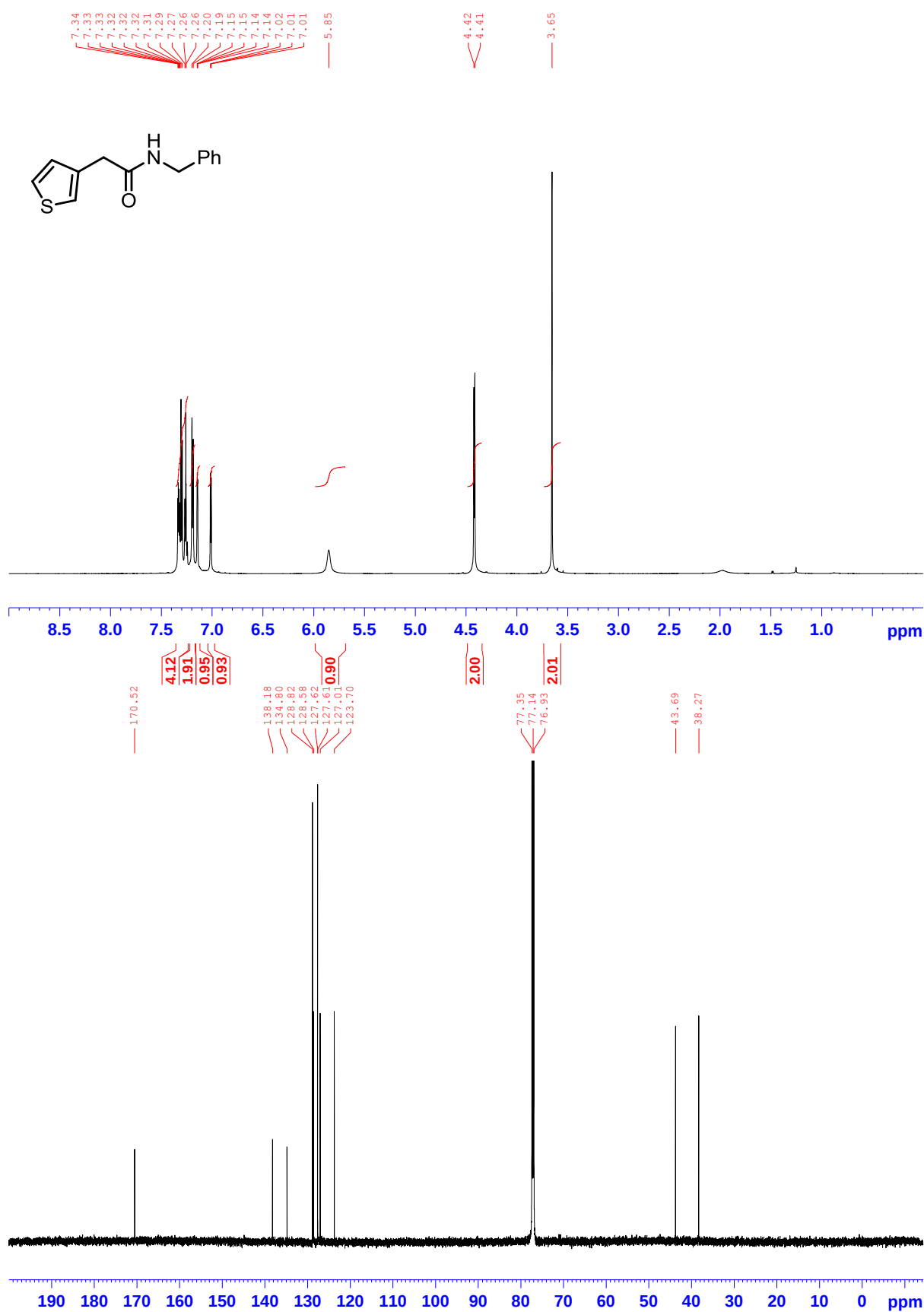
***N*-Benzyl-2-(3,4-dimethoxyphenyl)acetamide 3f**



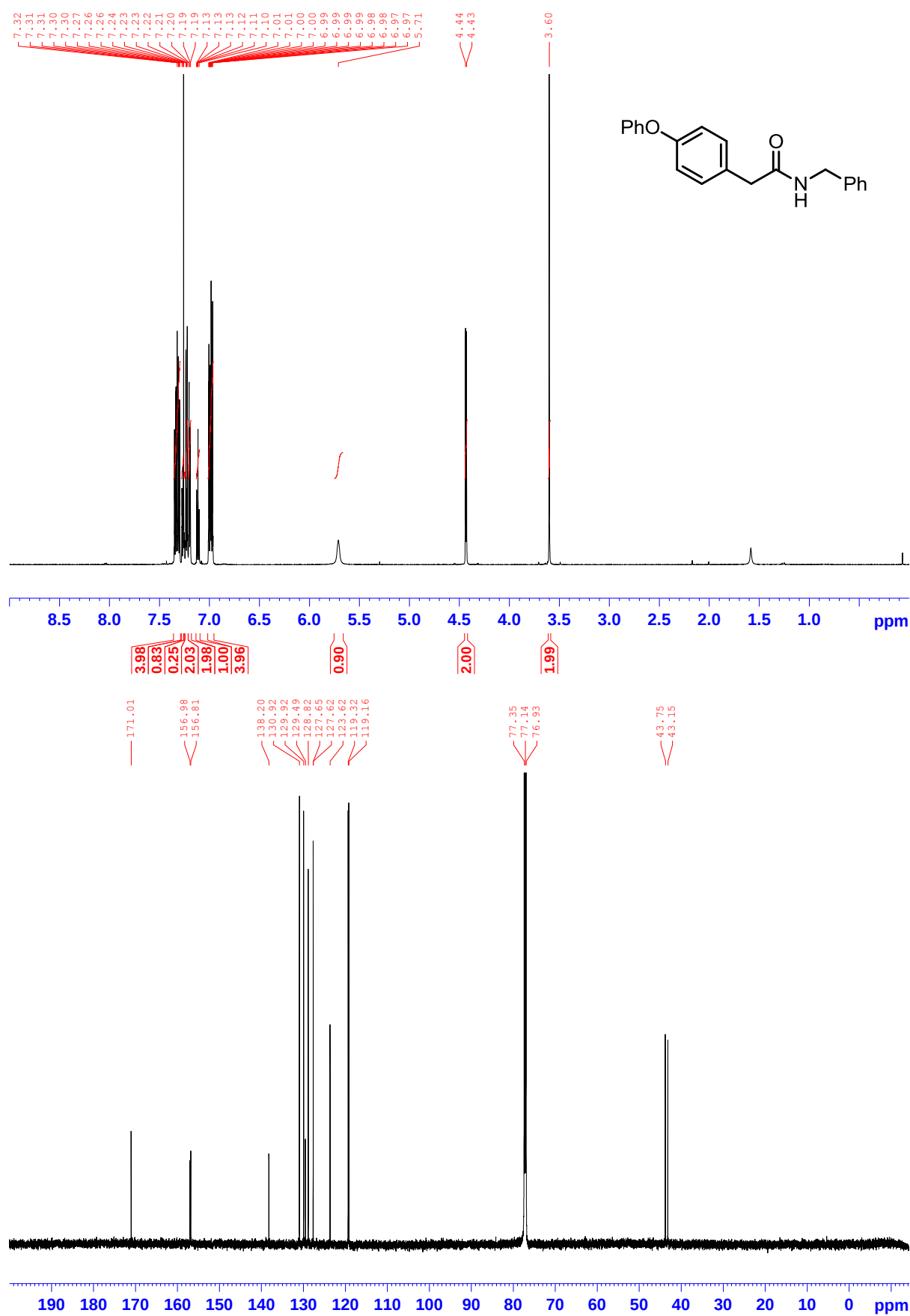
***N*-Benzyl-2-(4-chlorophenyl)acetamide 3g**



***N*-Benzyl-2-(thiophen-3-yl)acetamide 3h**



***N*-Benzyl-2-(4-phenoxyphenyl)acetamide 3i**



CCN(C(=O)NCC1=CC=CC=C1)C(C)(C)C

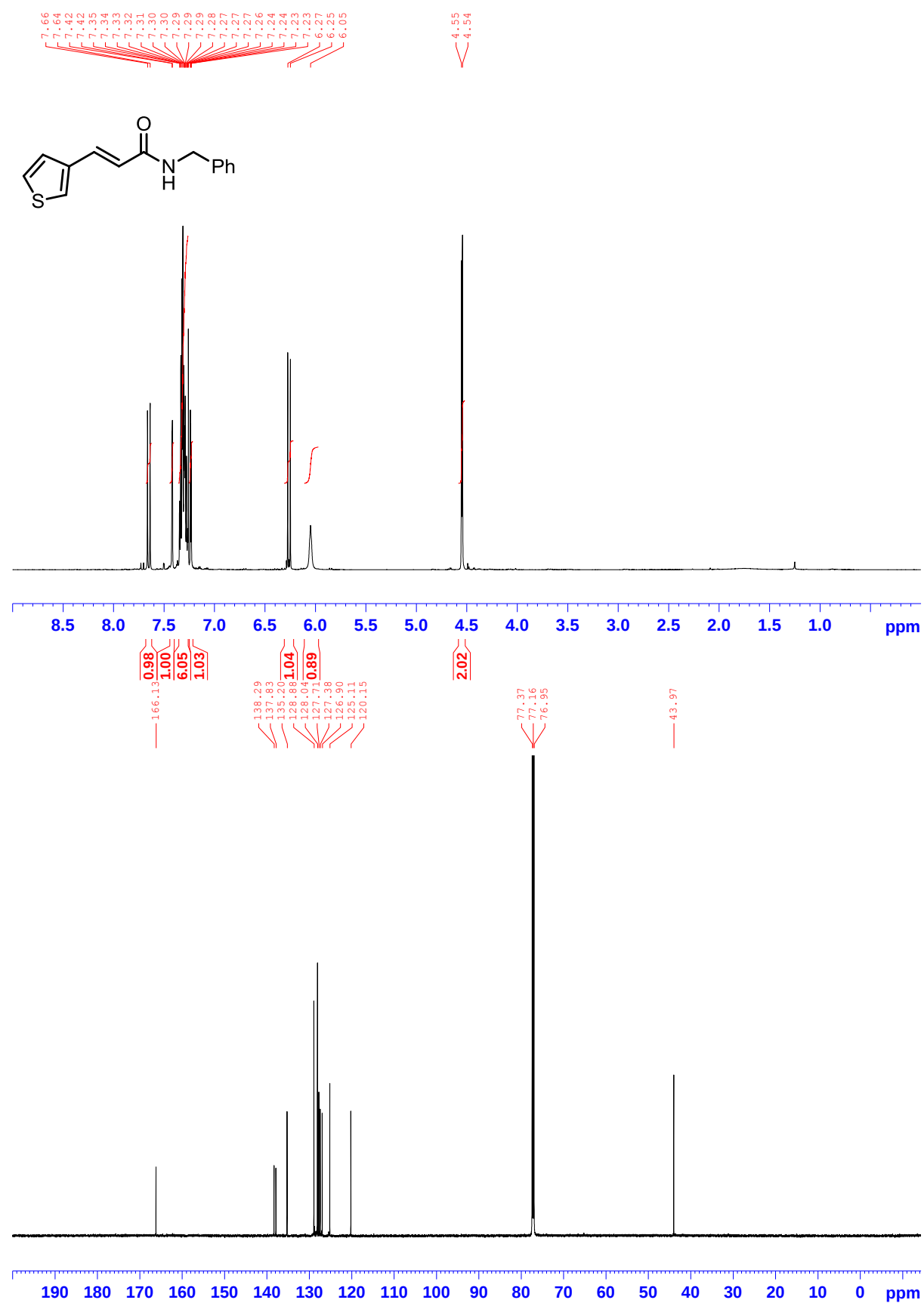
¹H NMR (400 MHz, CDCl₃)

Chemical Shift (ppm)	Integration
7.29, 7.27, 7.26, 7.25, 7.23, 7.22, 7.21	2.23, 3.03
6.52	0.94
4.41, 4.40	2.08
3.83	2.06
2.89	3.08
1.40	9.00

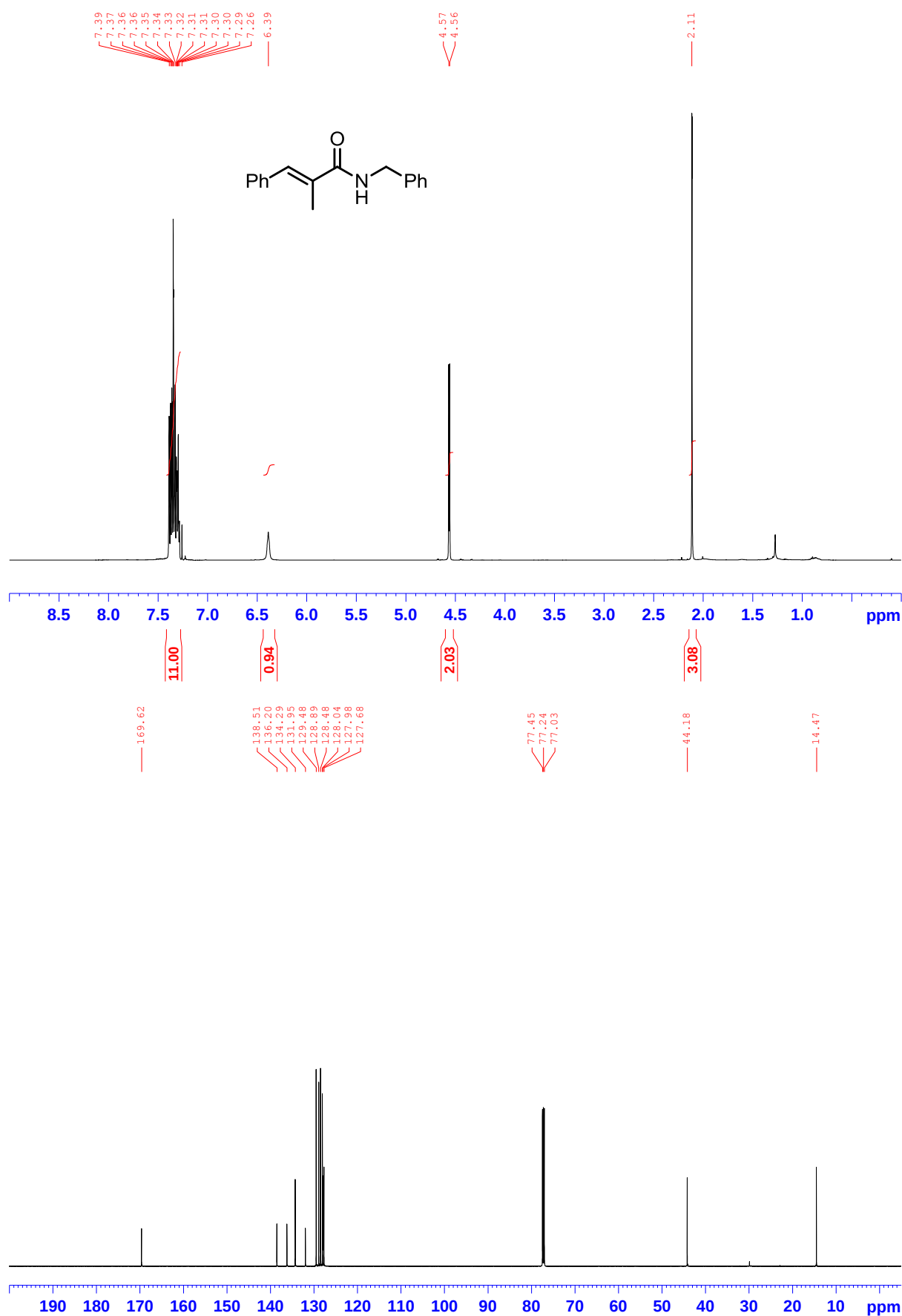
¹³C NMR (100 MHz, CDCl₃)

Chemical Shift (ppm)
169.09
155.92
138.22
128.60, 127.49, 127.39
80.61, 77.27, 76.95, 76.63
53.40
43.32
35.73
28.21

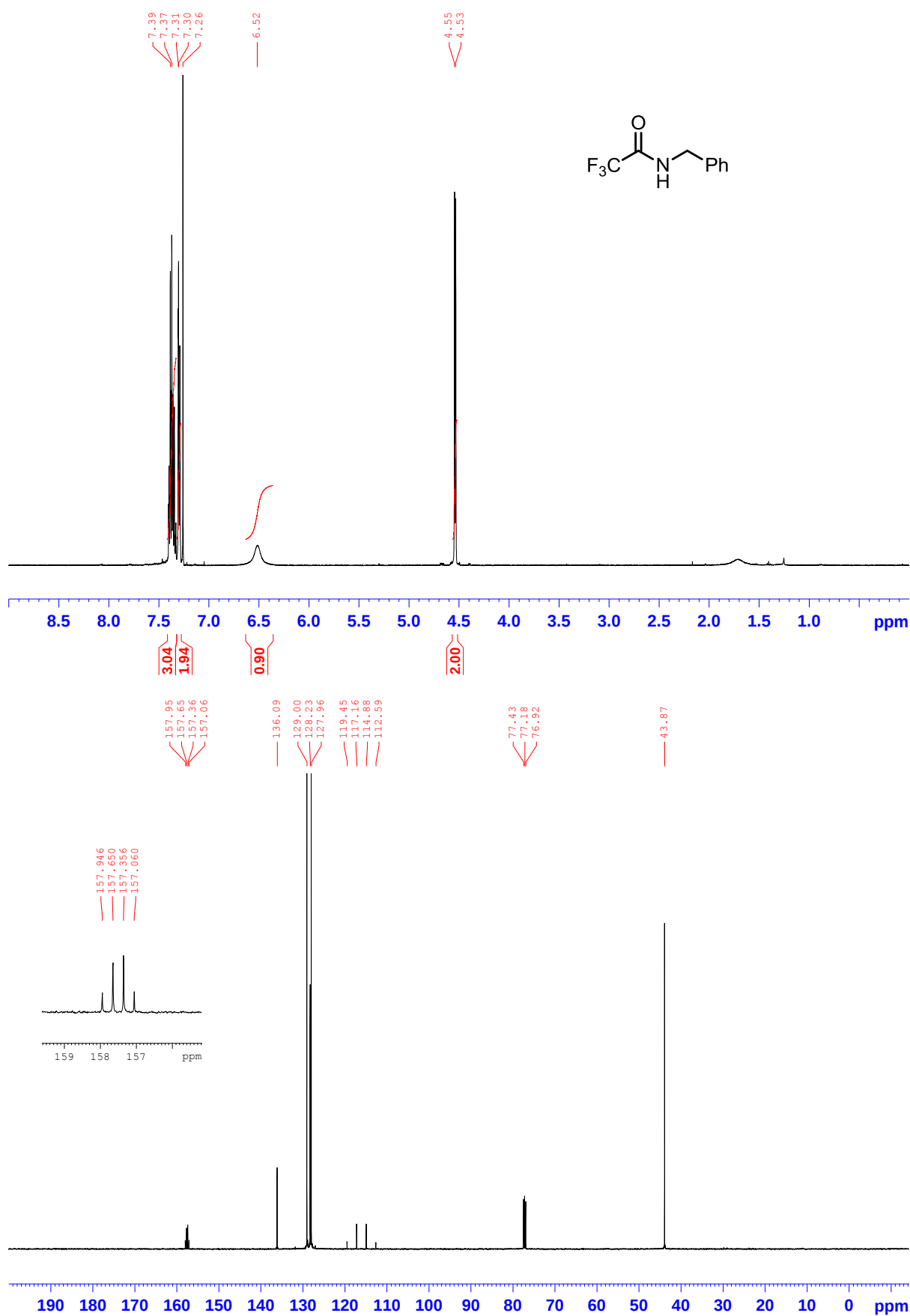
(E)-N-Benzyl-3-(thiophen-3-yl)acrylamide 3m



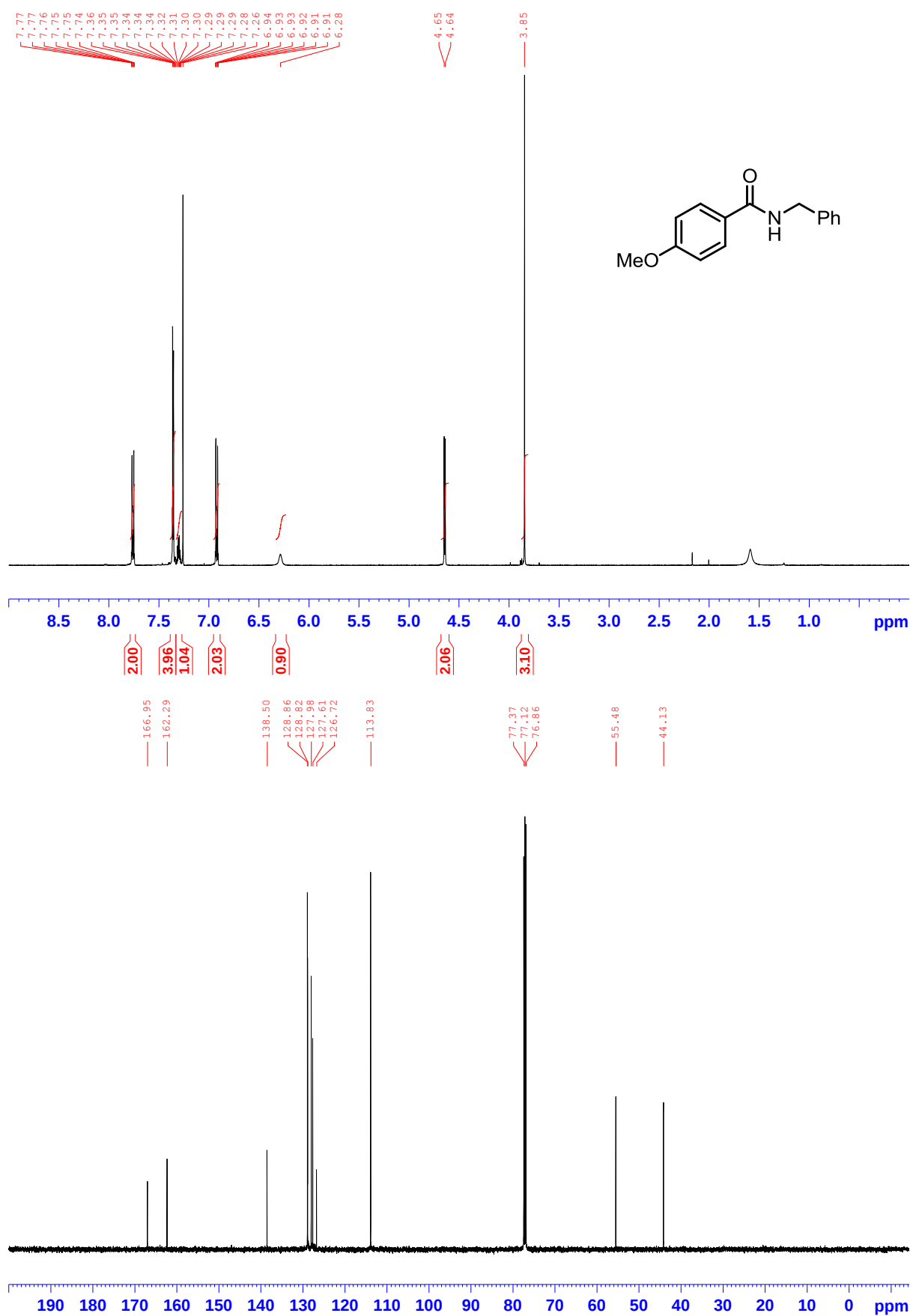
(E)-N-Benzyl-2-methyl-3-phenylacrylamide 3n



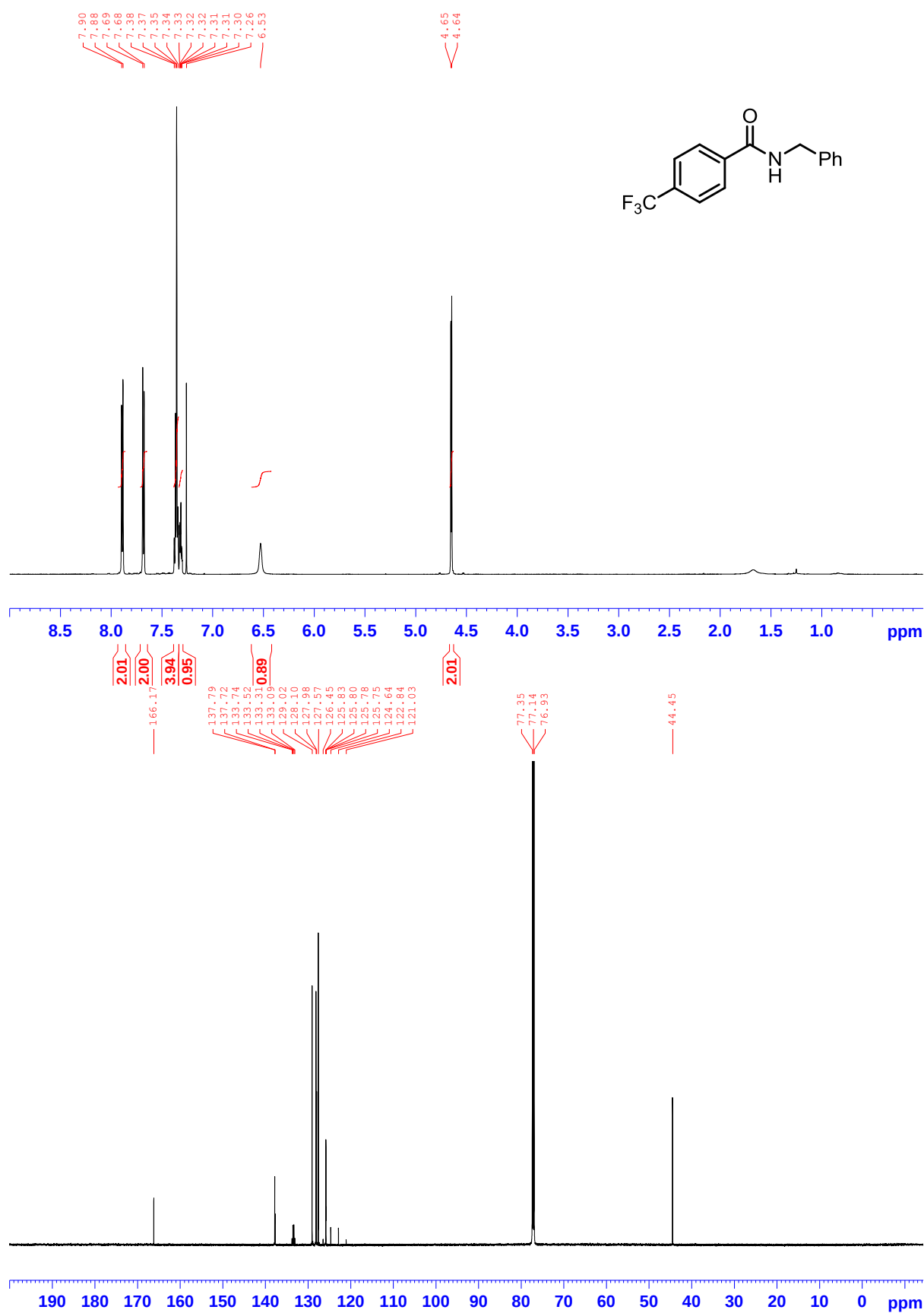
N-Benzyl-2,2,2-trifluoroacetamide 3p



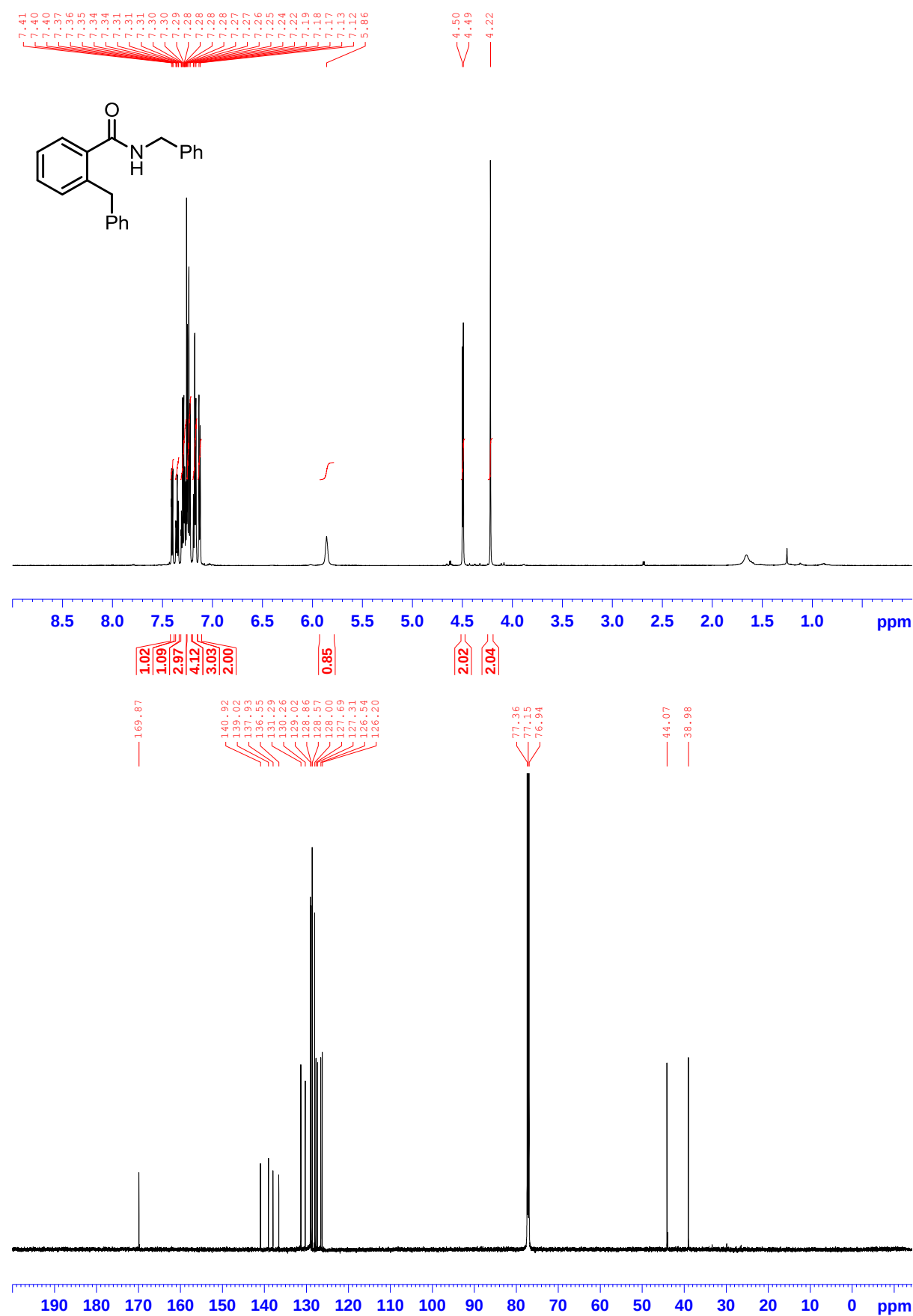
***N*-Benzyl-4-methoxybenzamide 3s**



***N*-Benzyl-4-(trifluoromethyl)benzamide 3t**



N,2-Dibenzylbenzamide 3u



Chemical Structure: NCCc1ccccc1C(=O)c2ccc(I)cc2

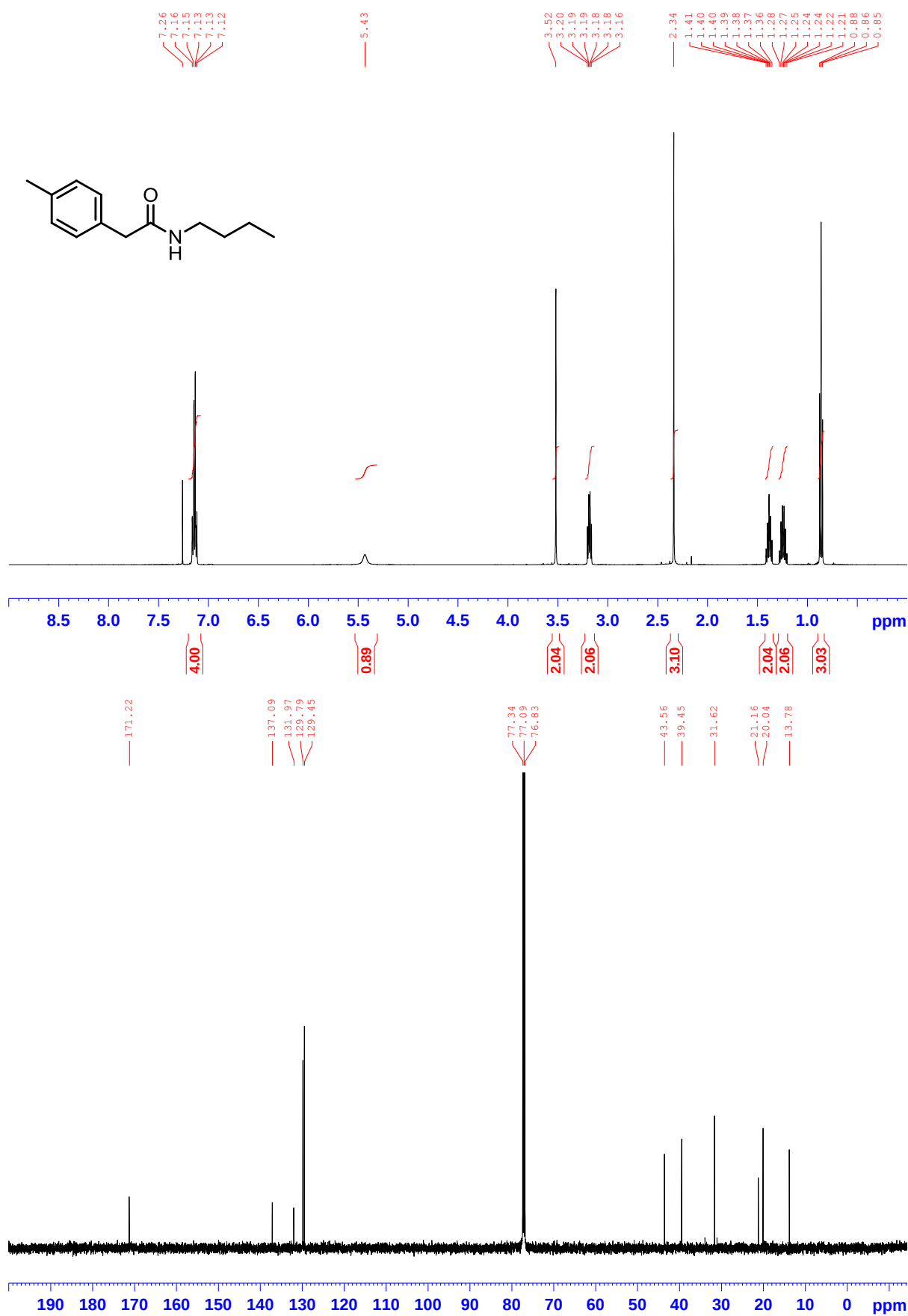
¹H NMR Spectrum (CDCl₃):

- Chemical shift range: 4.46 – 9.11 ppm
- Integration values: 0.97, 1.97, 1.98, 3.94, 0.97, 2.00

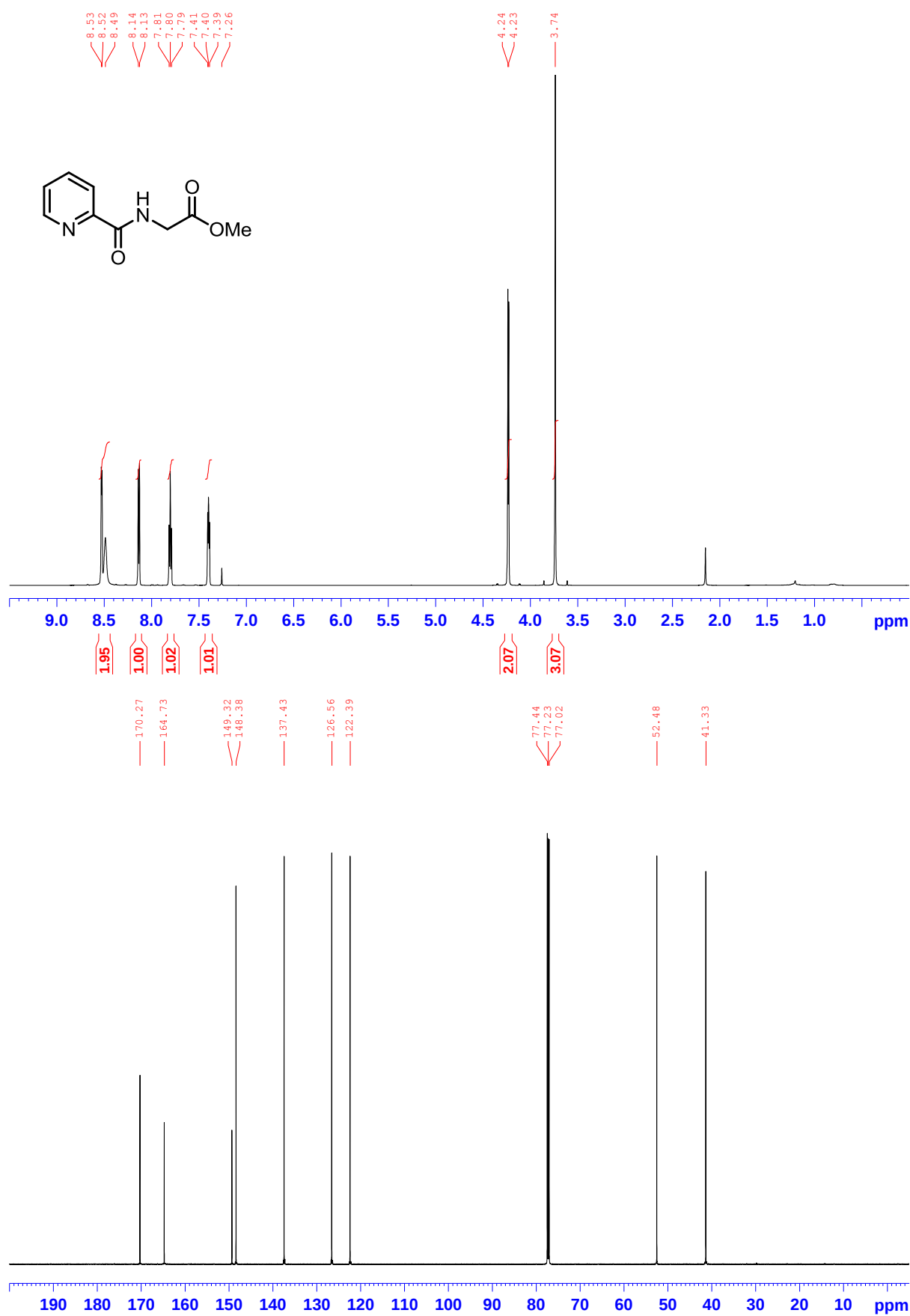
¹³C NMR Spectrum (CDCl₃):

- Chemical shift range: 39.15 – 165.56 ppm

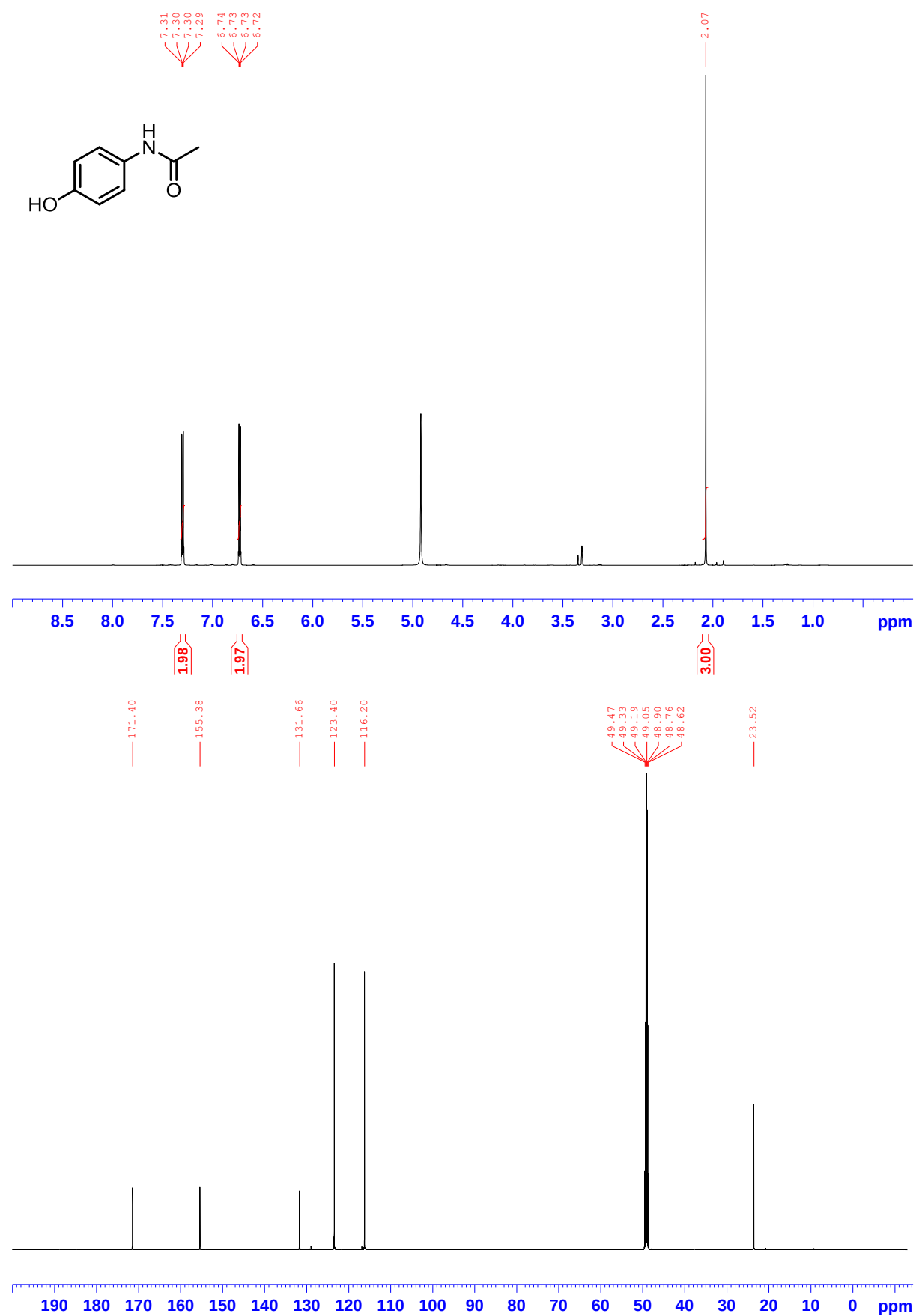
***N*-Butyl-2-*p*-tolylacetamide 4c**



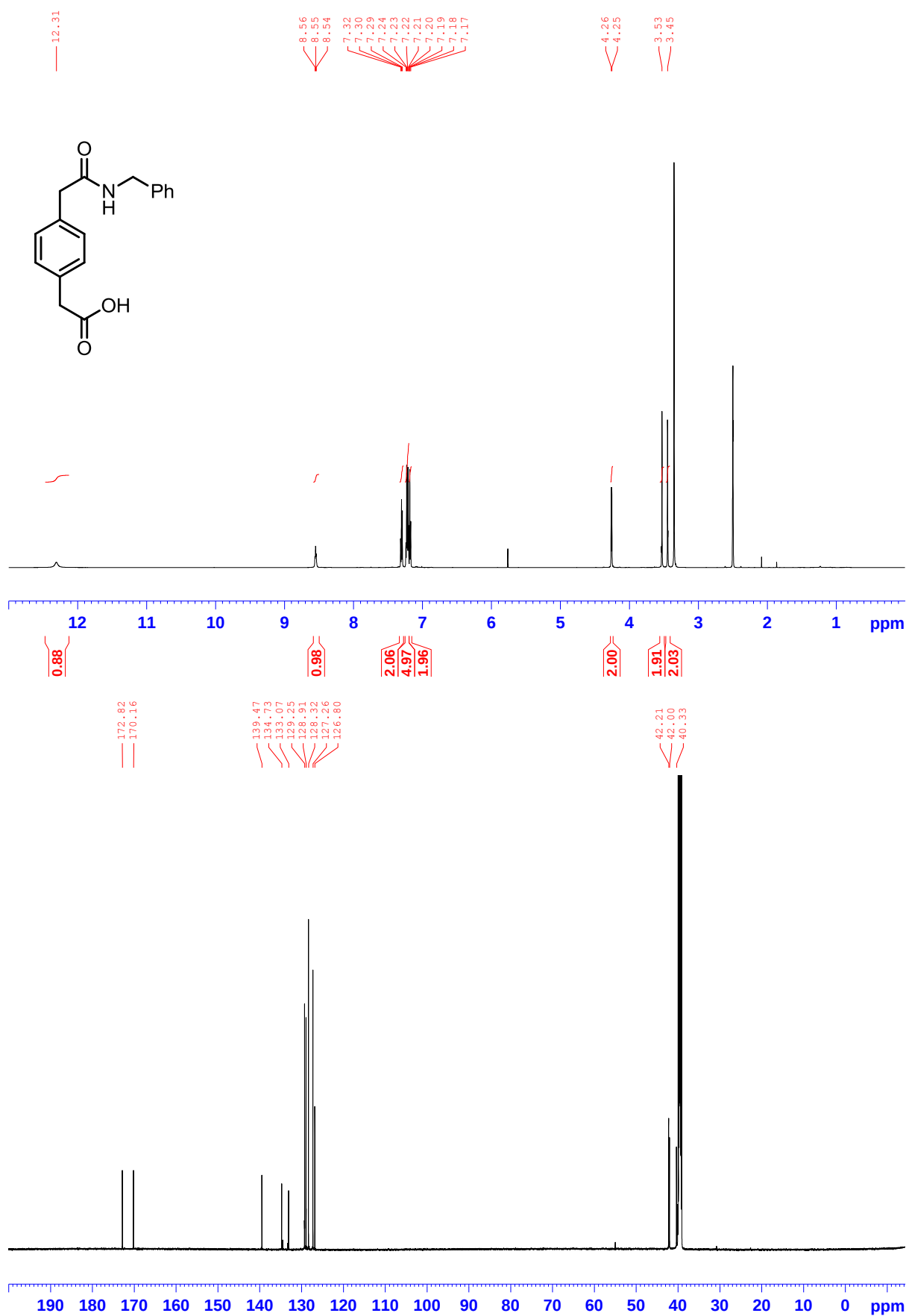
Methyl 2-(picolinamido)acetate 4e



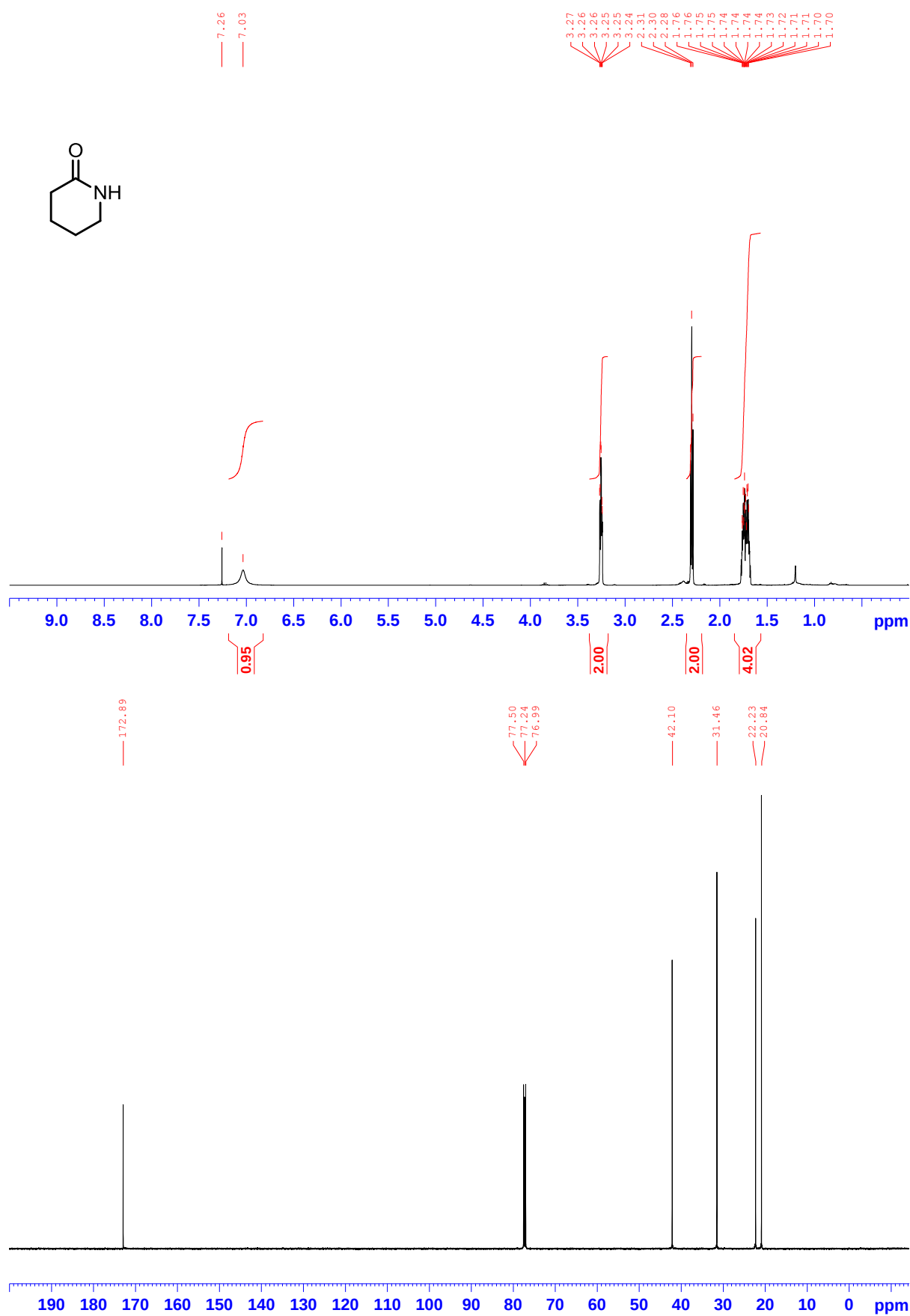
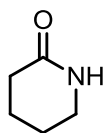
***N*-(4-Hydroxyphenyl)acetamide 4g**



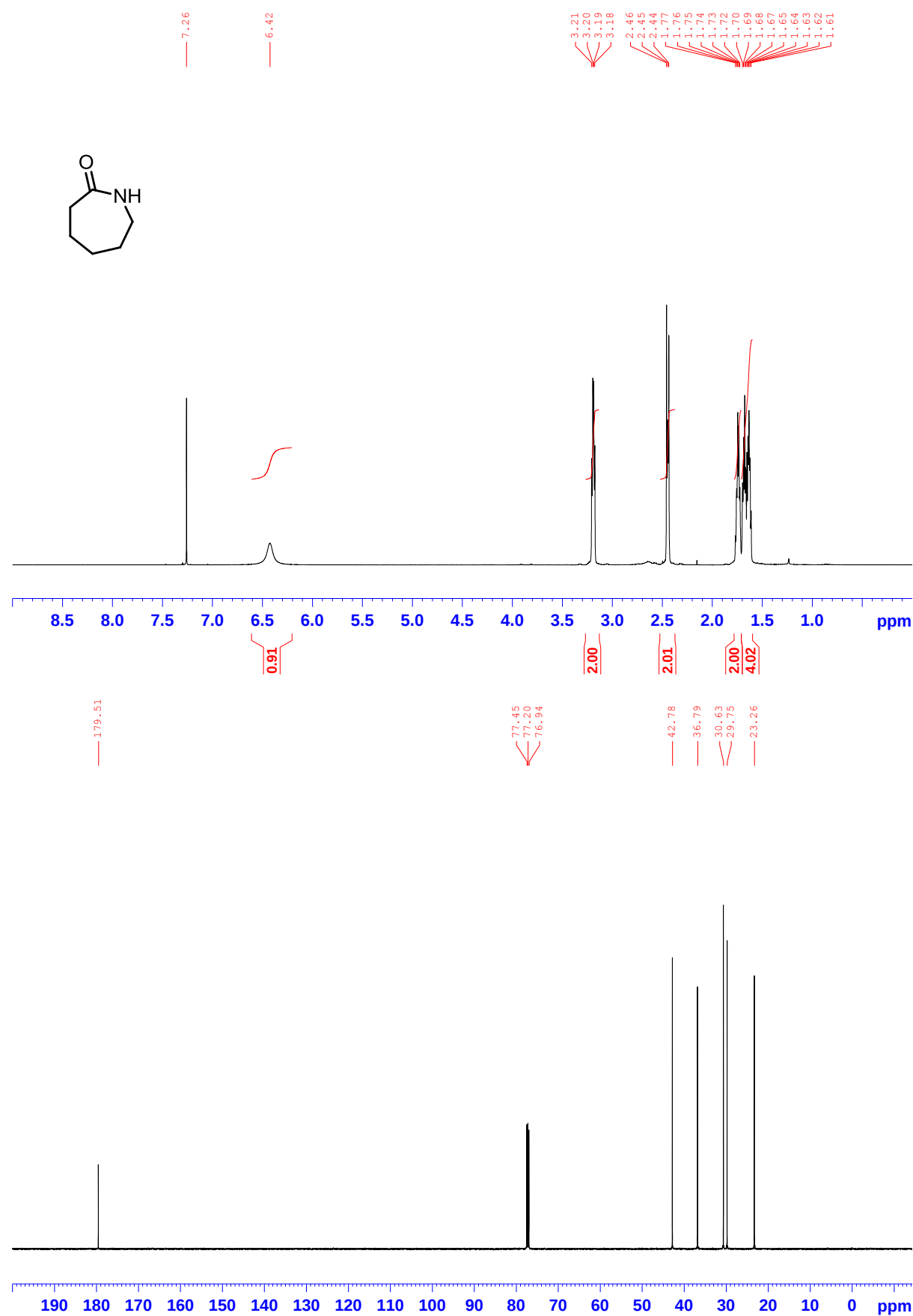
2-(4-(2-(Benzylamino)-2-oxoethyl)phenyl)acetic acid 4h



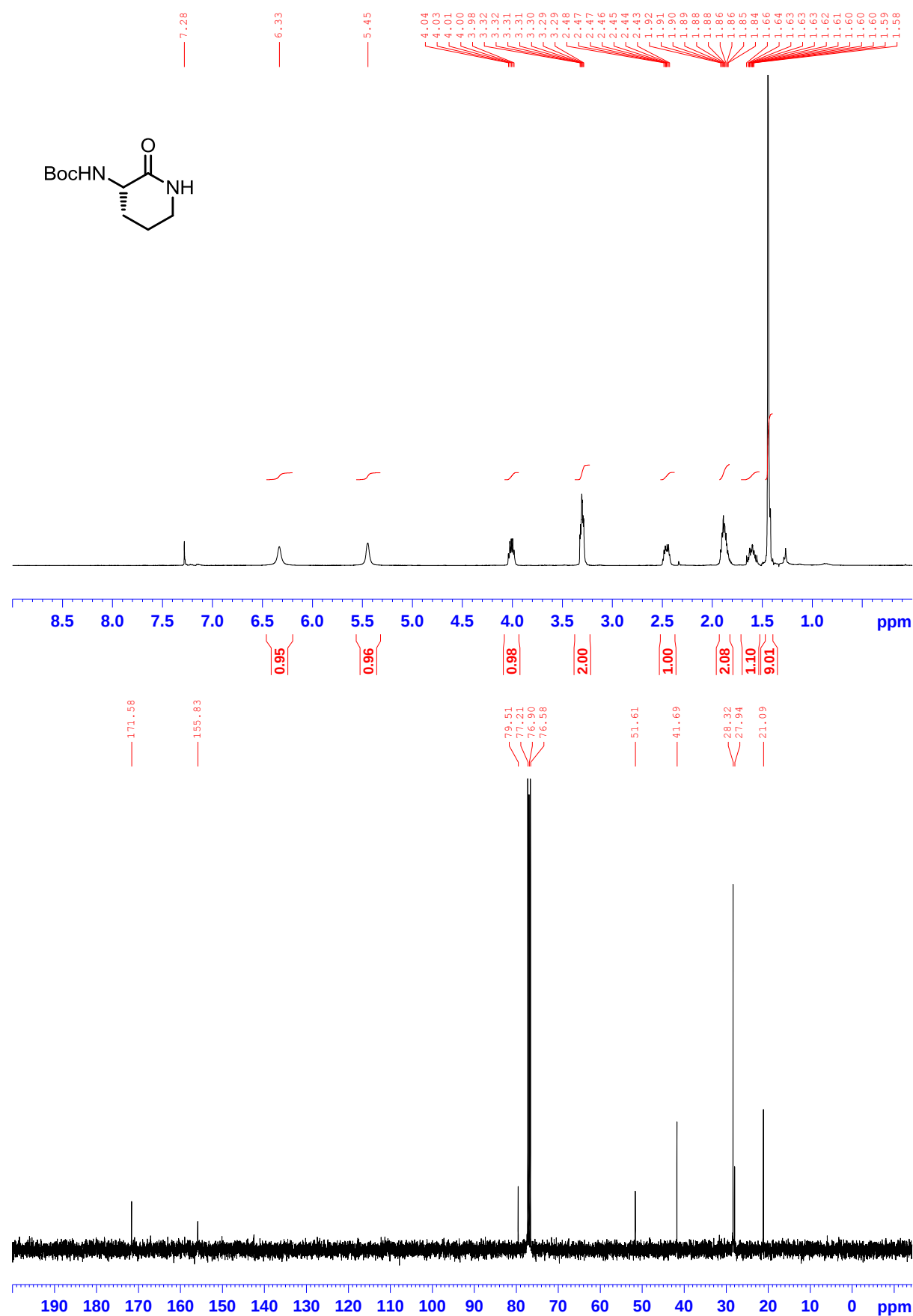
Piperidin-2-one 5a



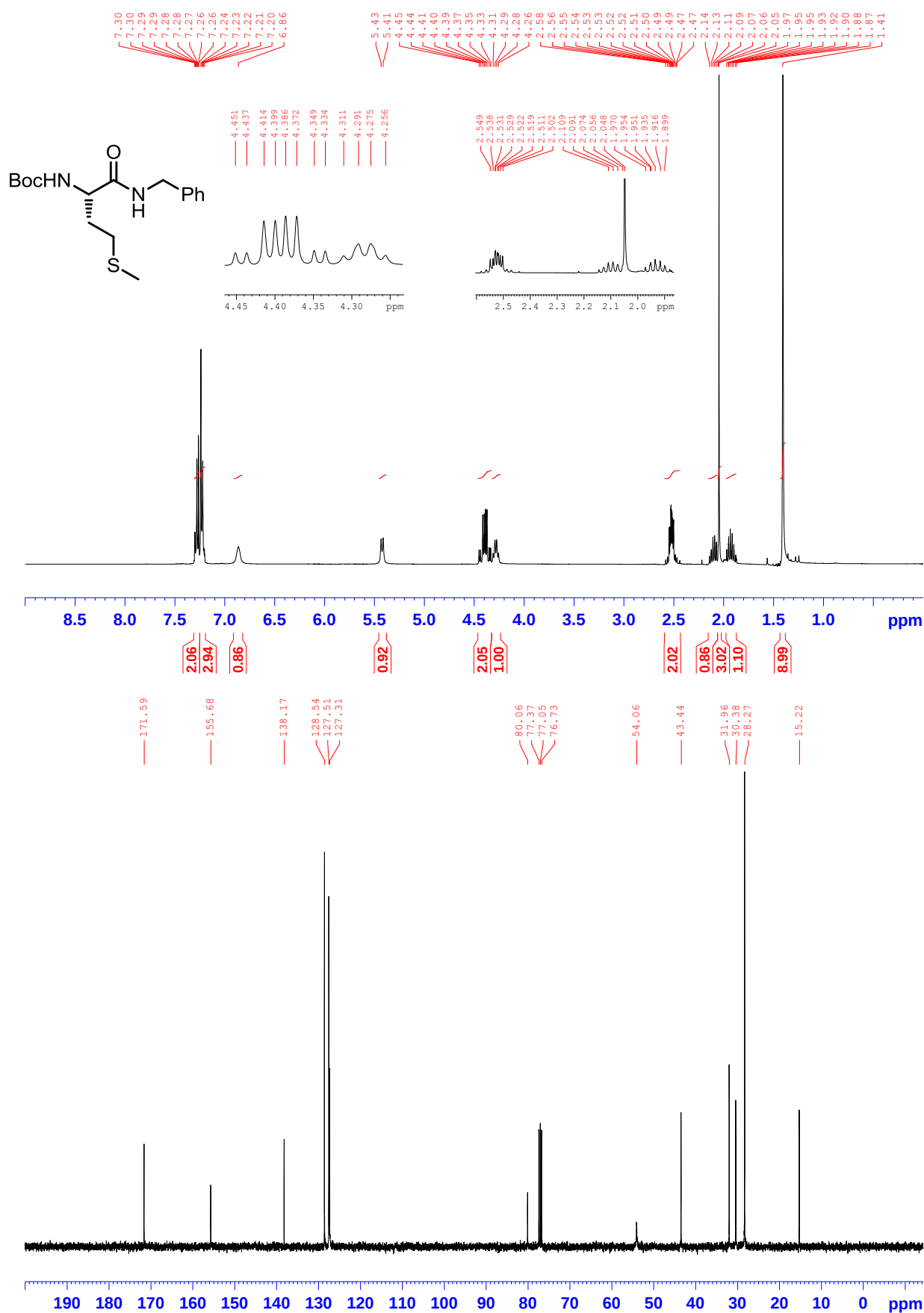
Azepan-2-one 5b



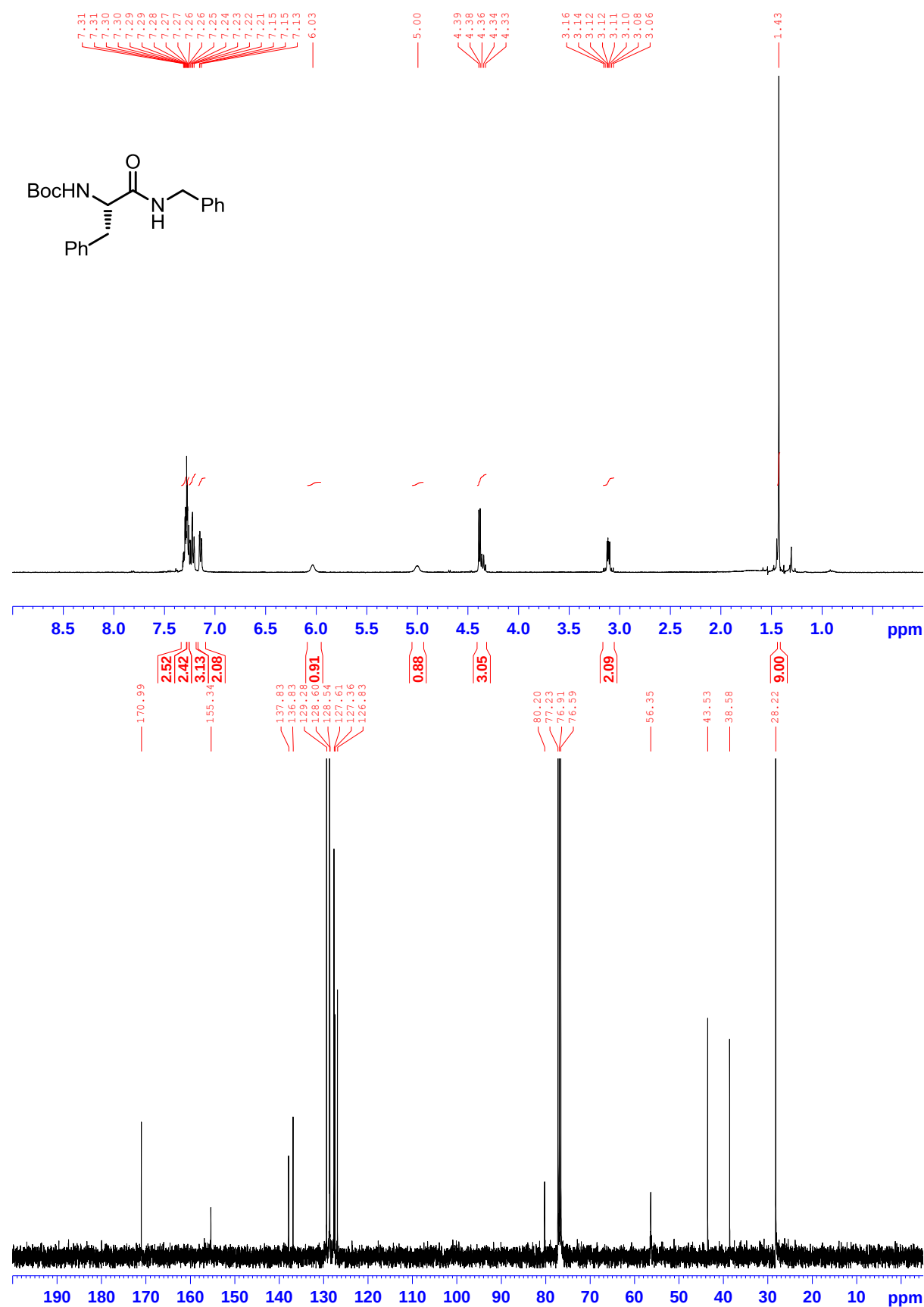
(S)-tert-Butyl 2-oxopiperidin-3-ylcarbamate 5c



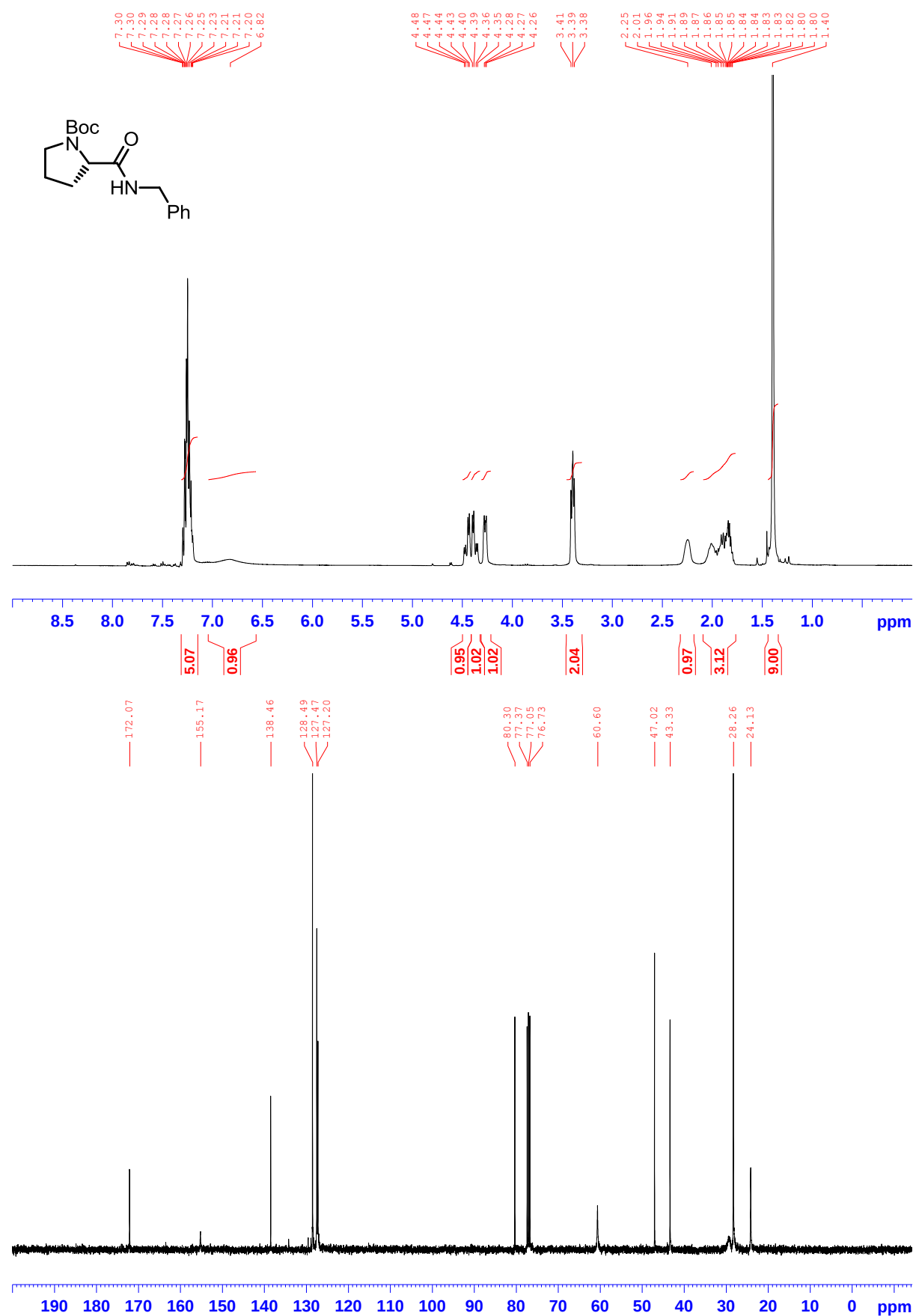
Boc-L-Methionine-benzamide 6a



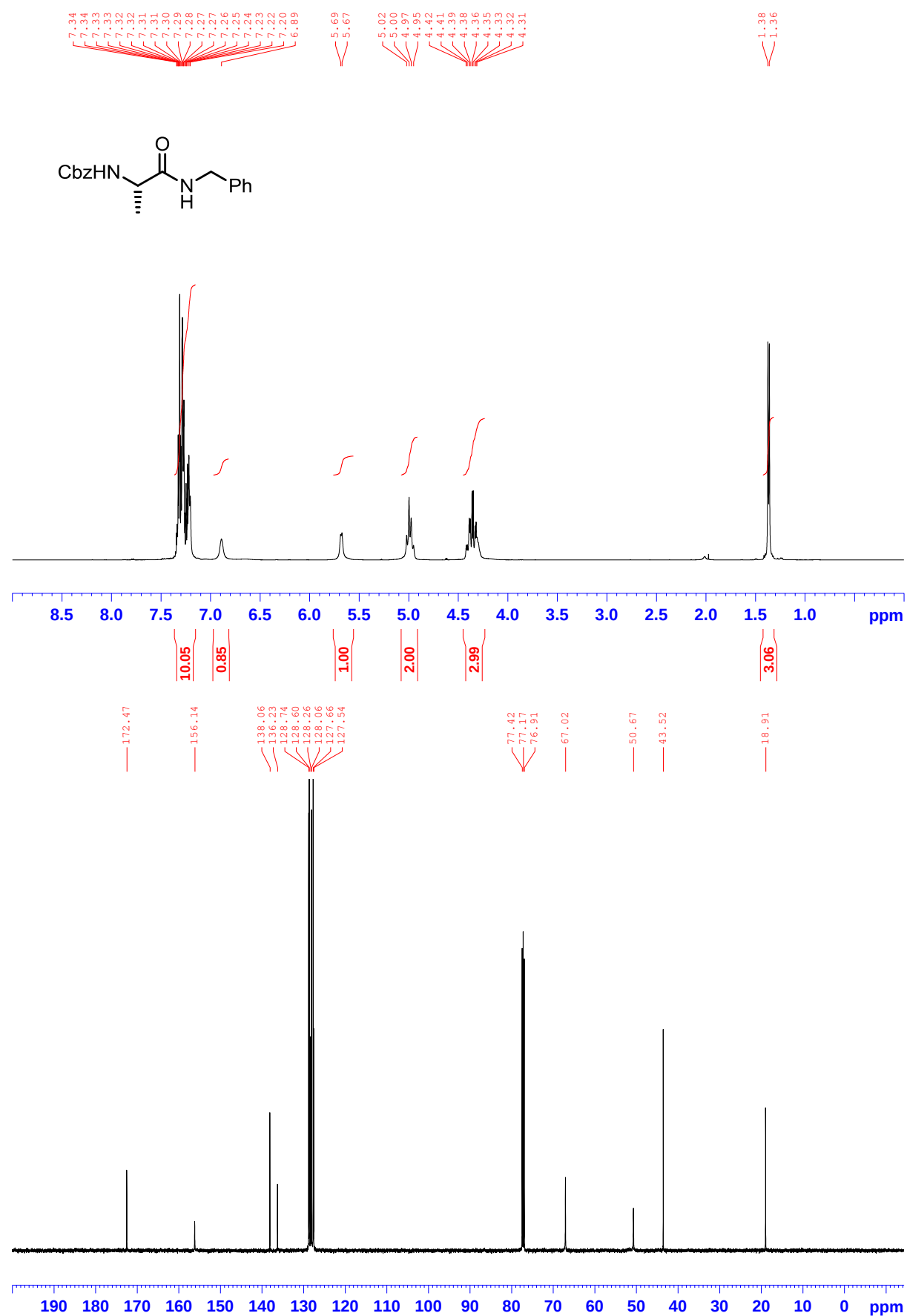
Boc-L-Phenylalanine-benzamide 6c



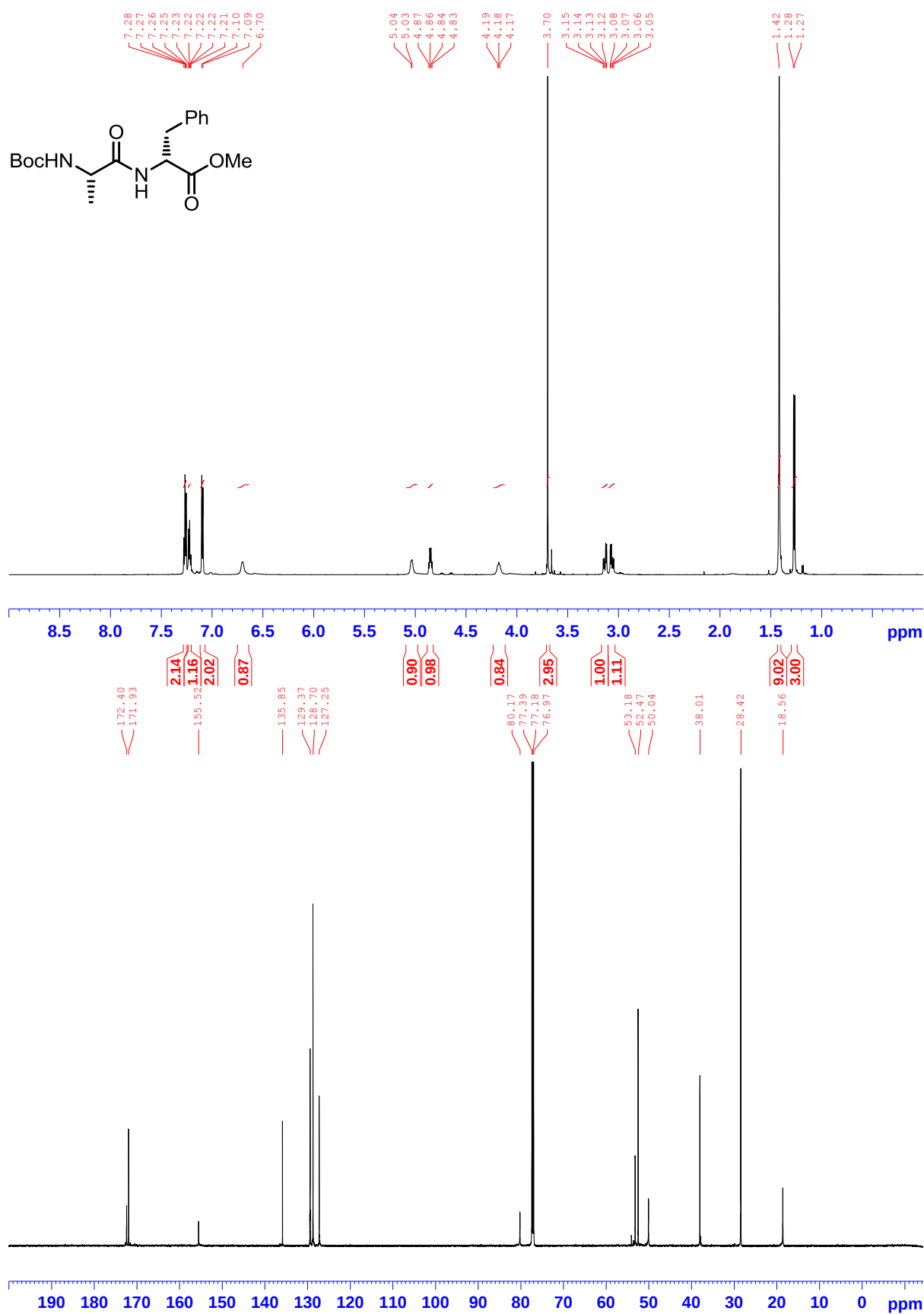
Boc-L-Proline-benzamide 6d



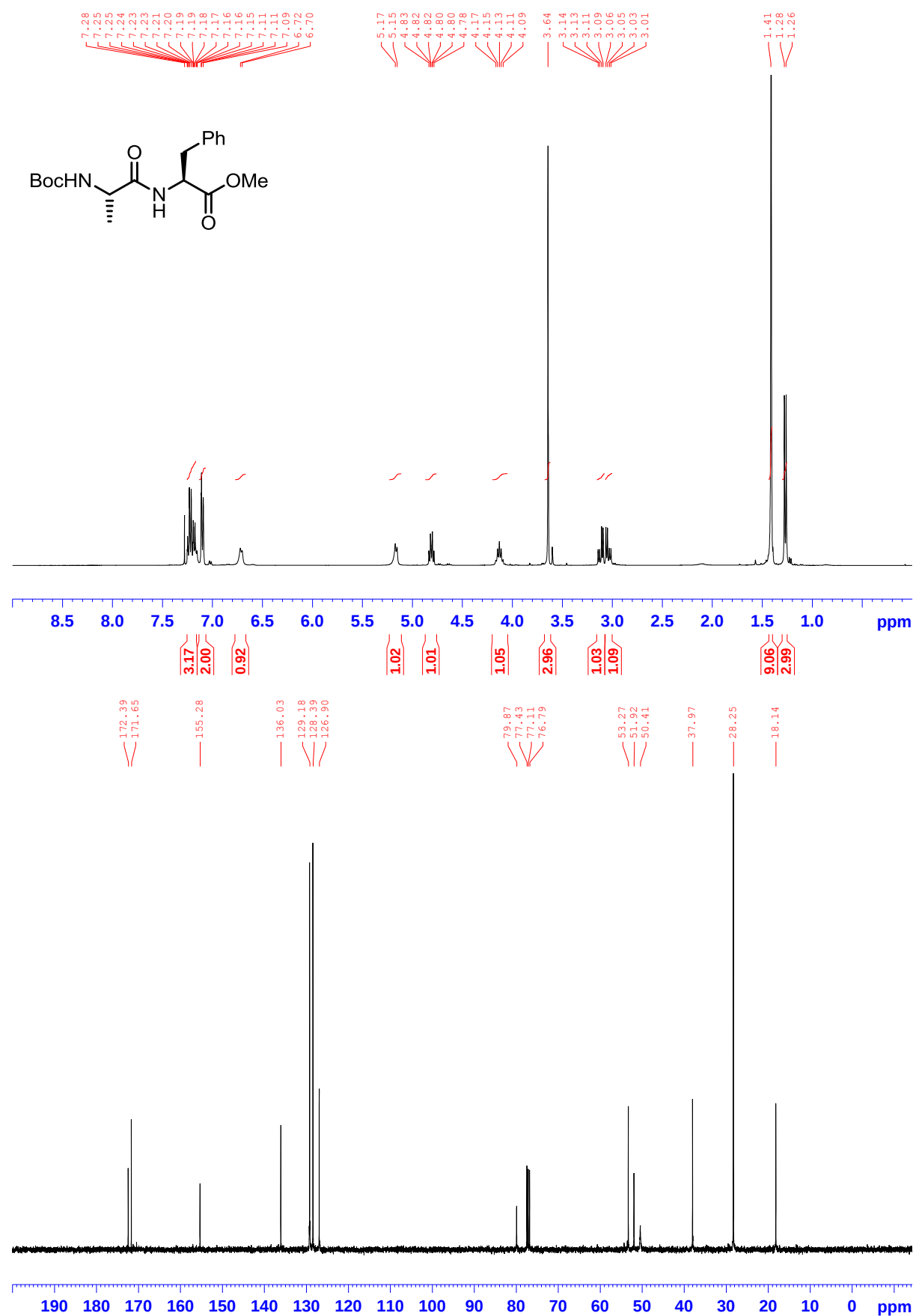
Cbz-L-Alanine-benzamide 6e



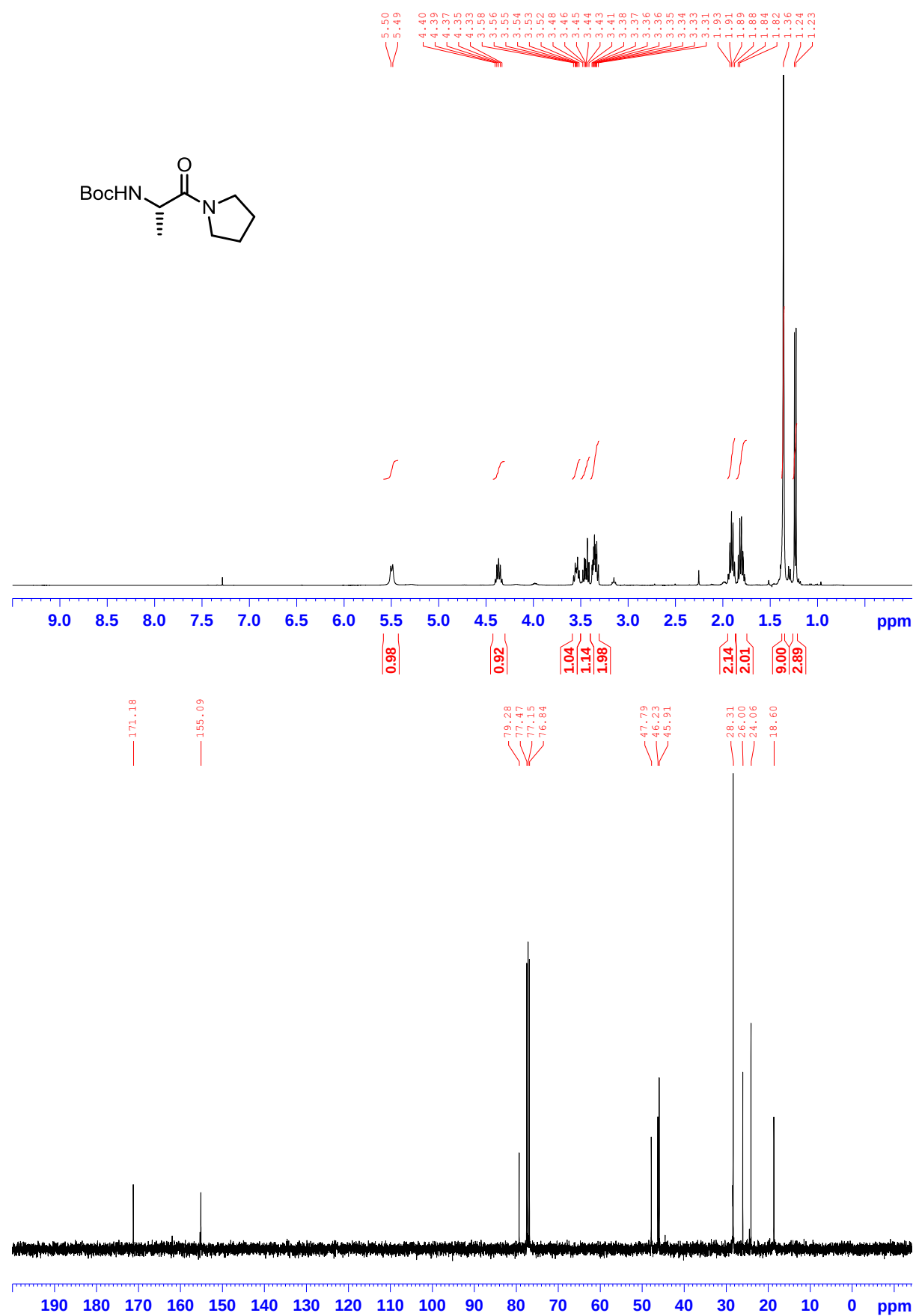
Boc-L-Ala-D-Phe-OMe 6f



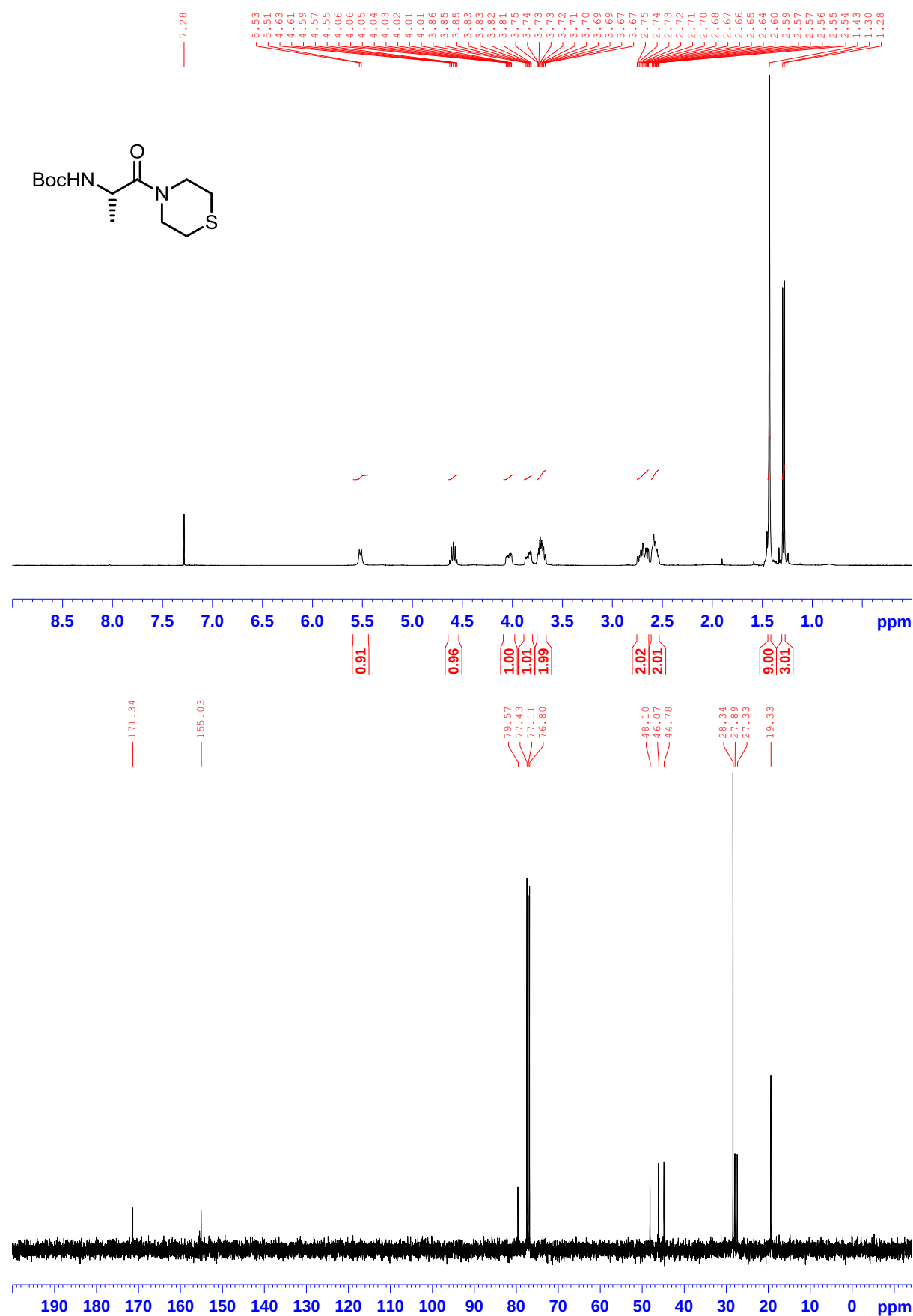
Boc-L-Ala-L-Phe-OMe 6g



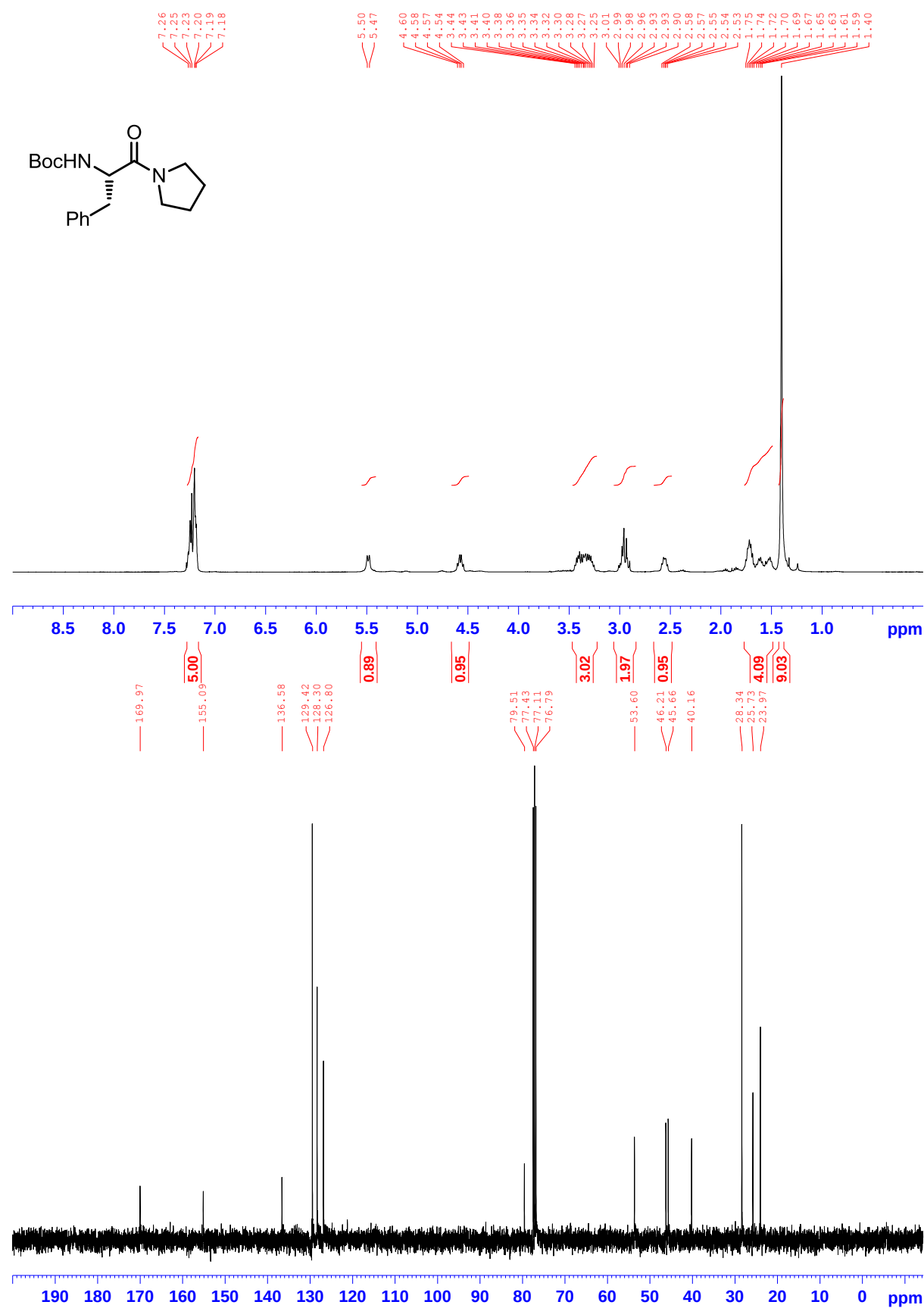
Boc-L-Alanine pyrrolidine amide 6h



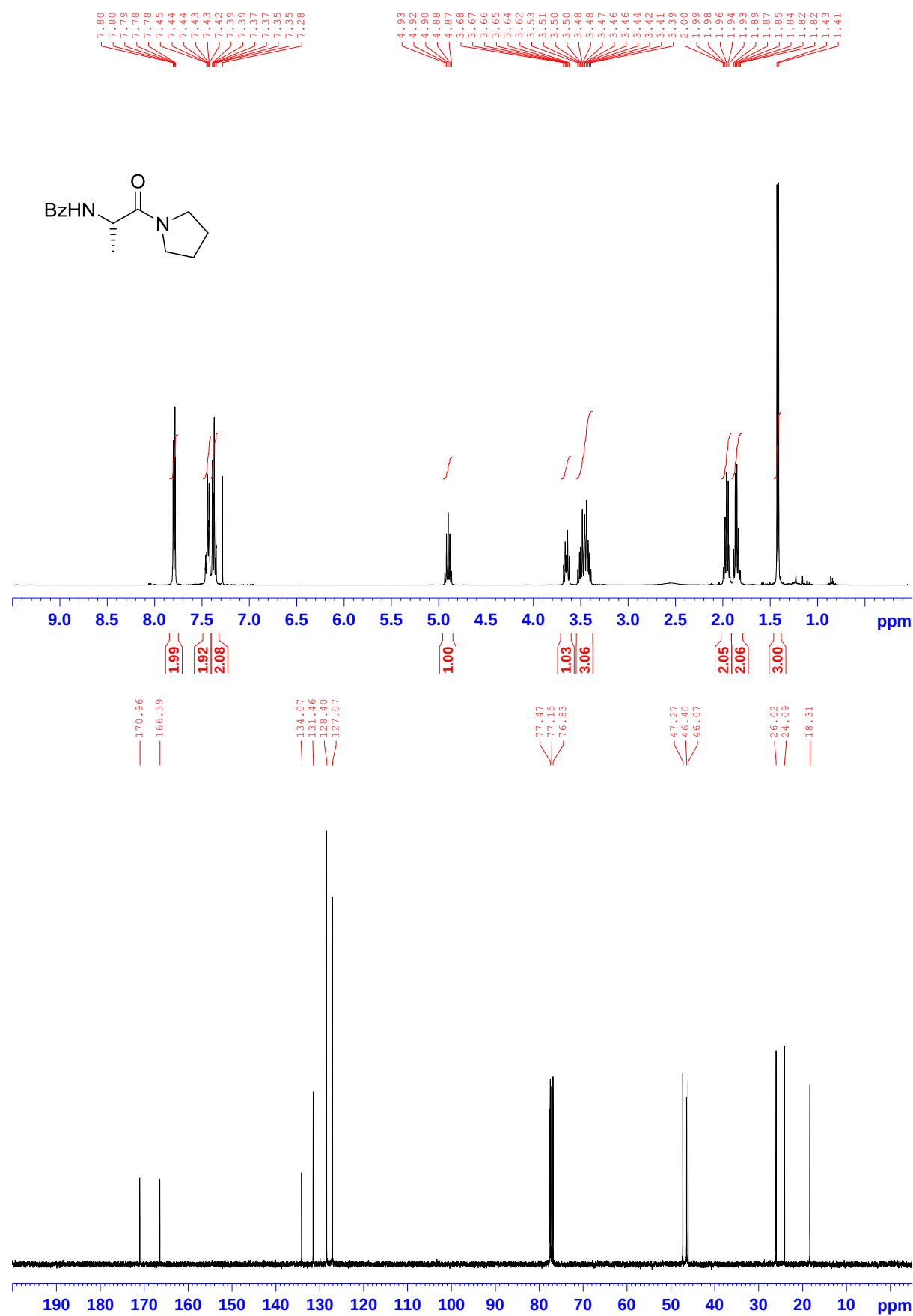
Boc-L-Alanine thiomorpholine amide 6i



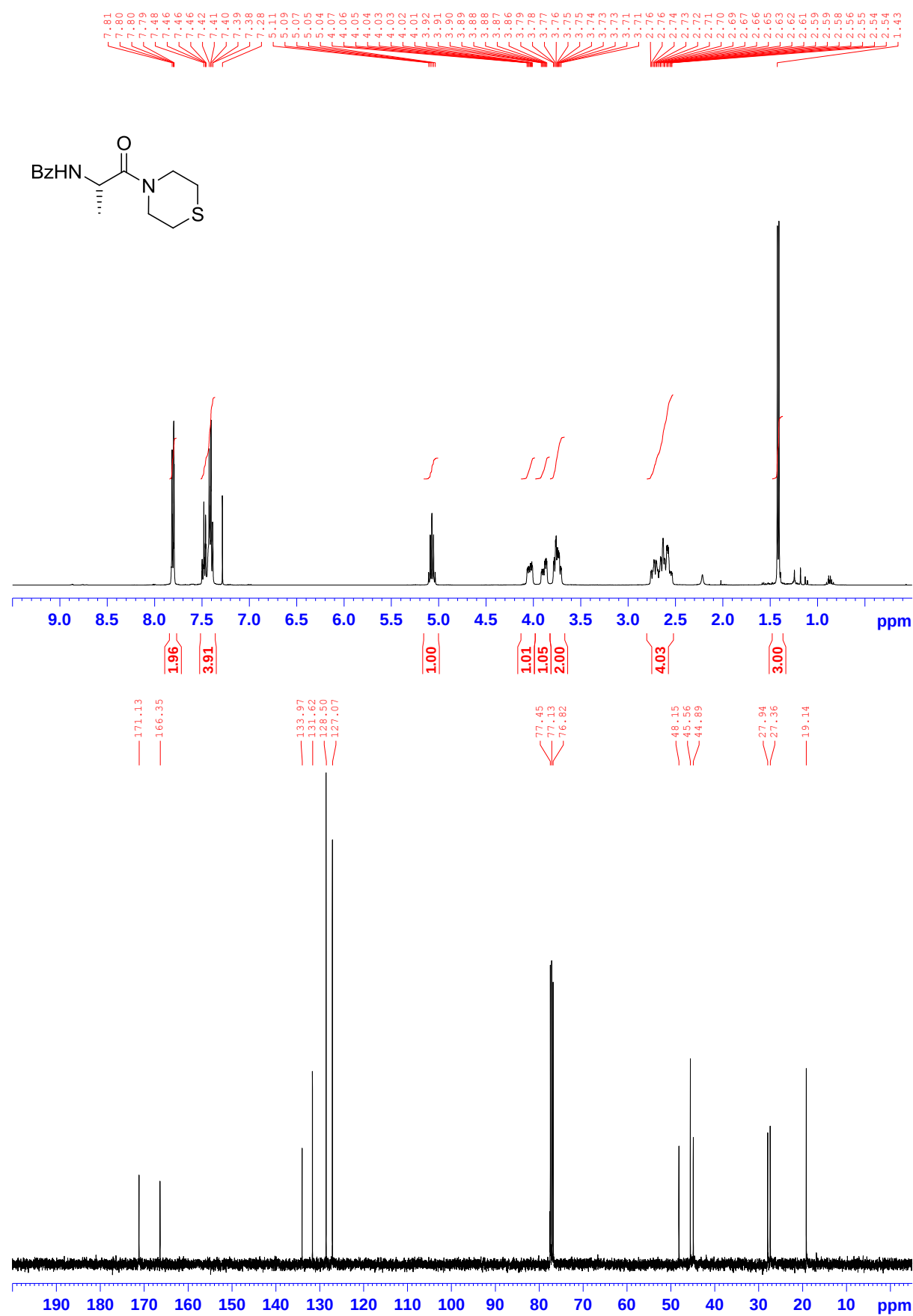
Boc-L-Phenylalanine pyrrolidine amide 6j



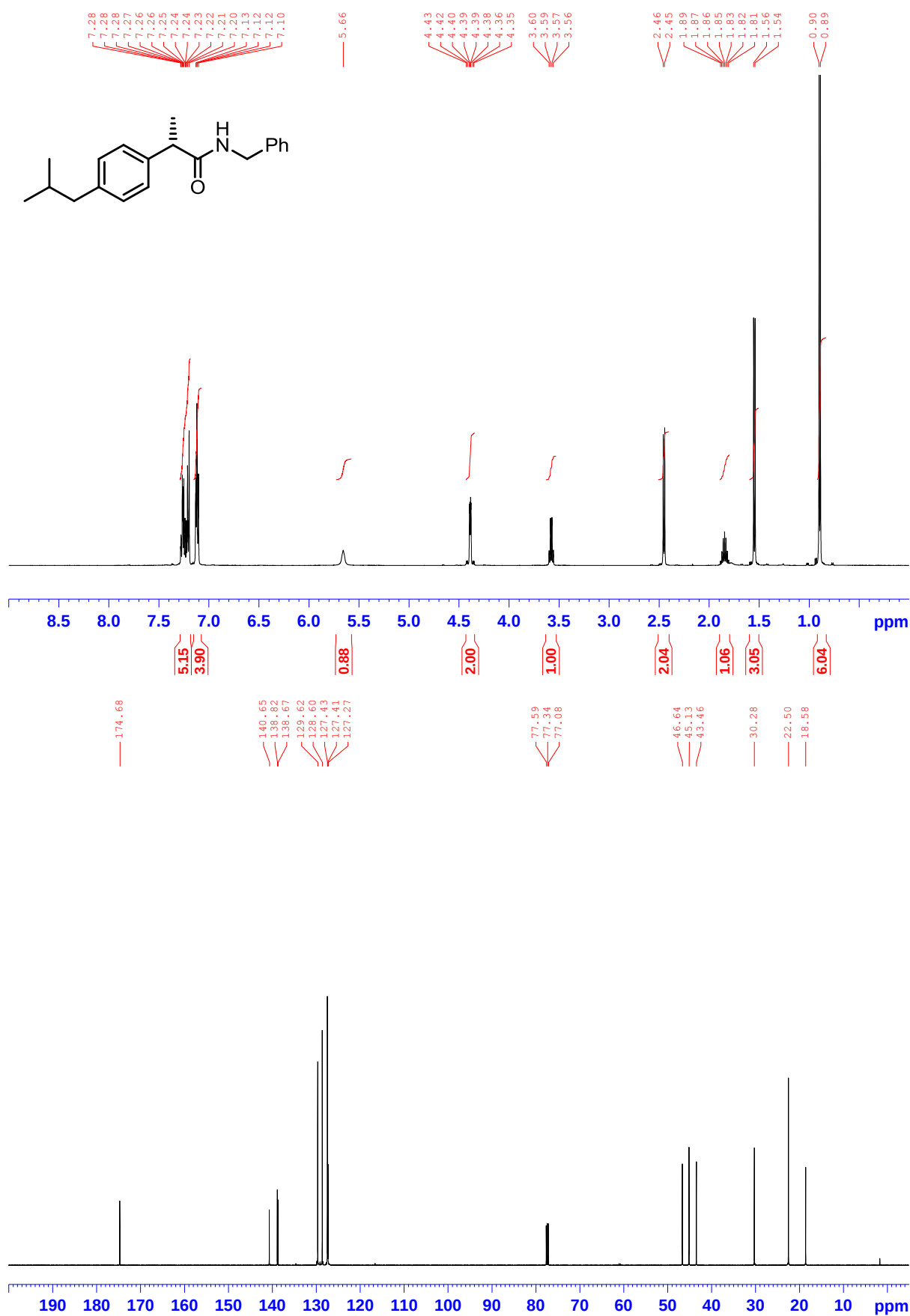
Benz-L-Alanine-pyrrolidine amide 6k



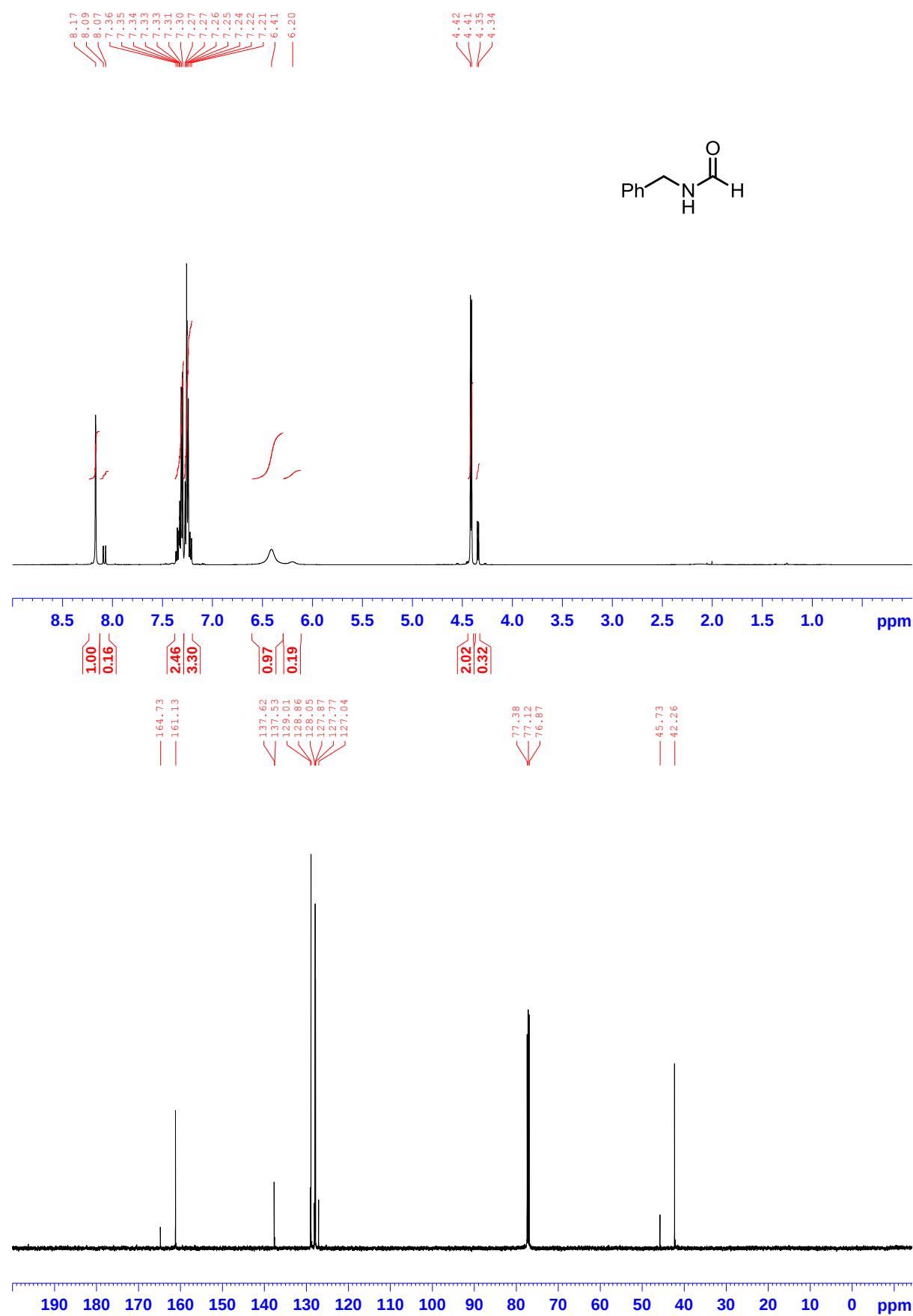
Benz-L-Alanine-thiomorpholine amide 6l



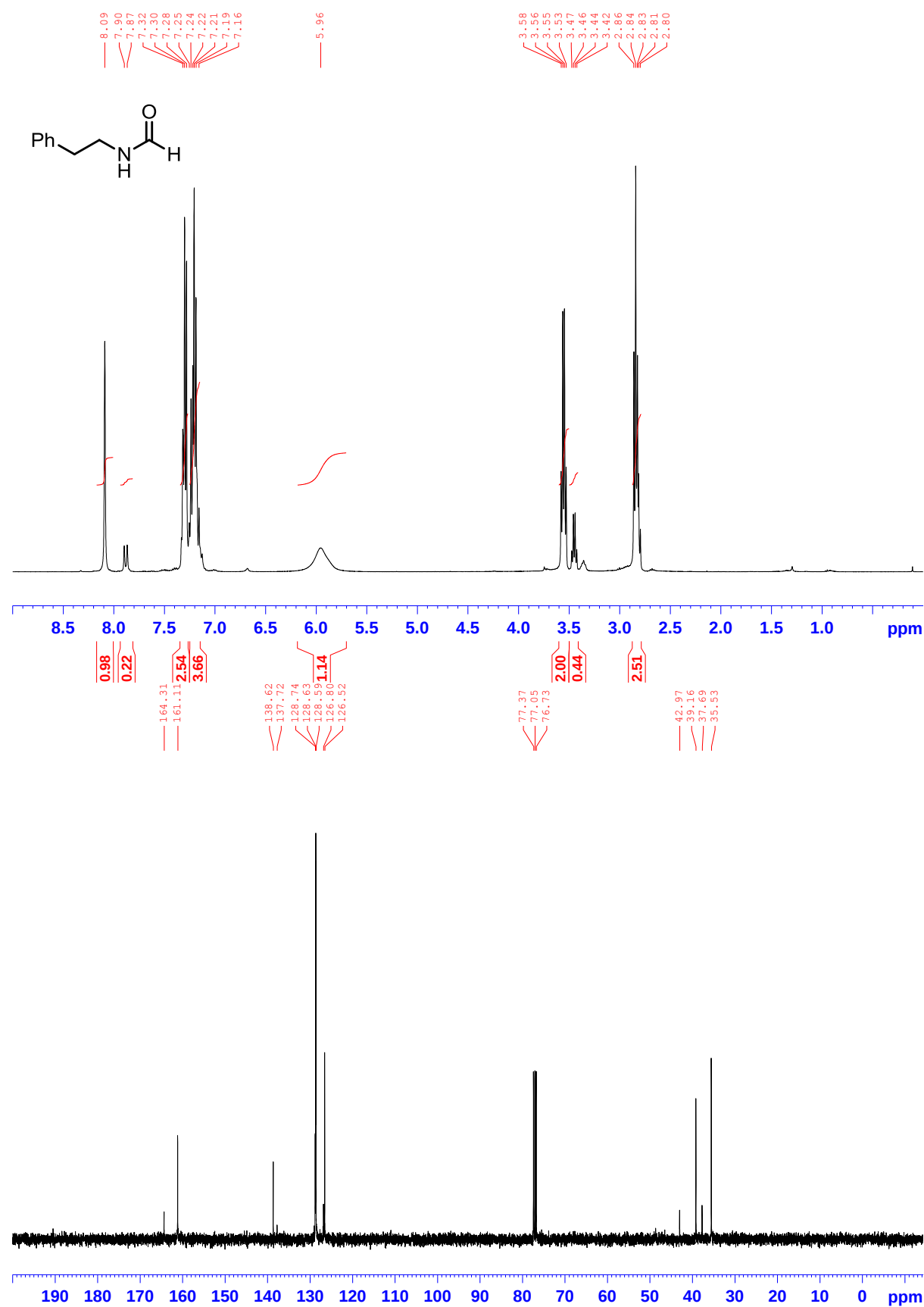
(S)-N-Benzyl-2-(4-isobutylphenyl)propanamide 6m



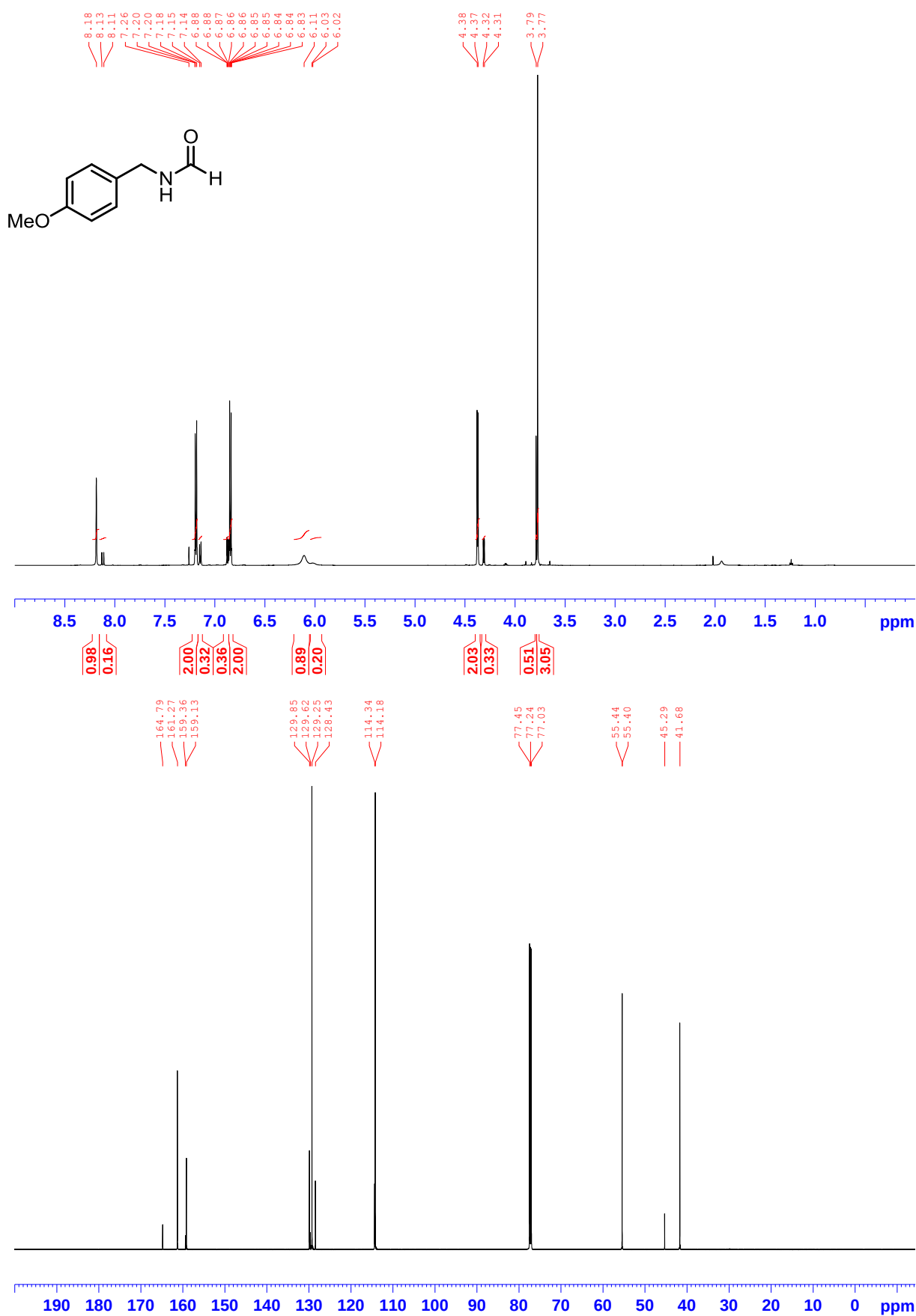
N-Benzylformamide 7a



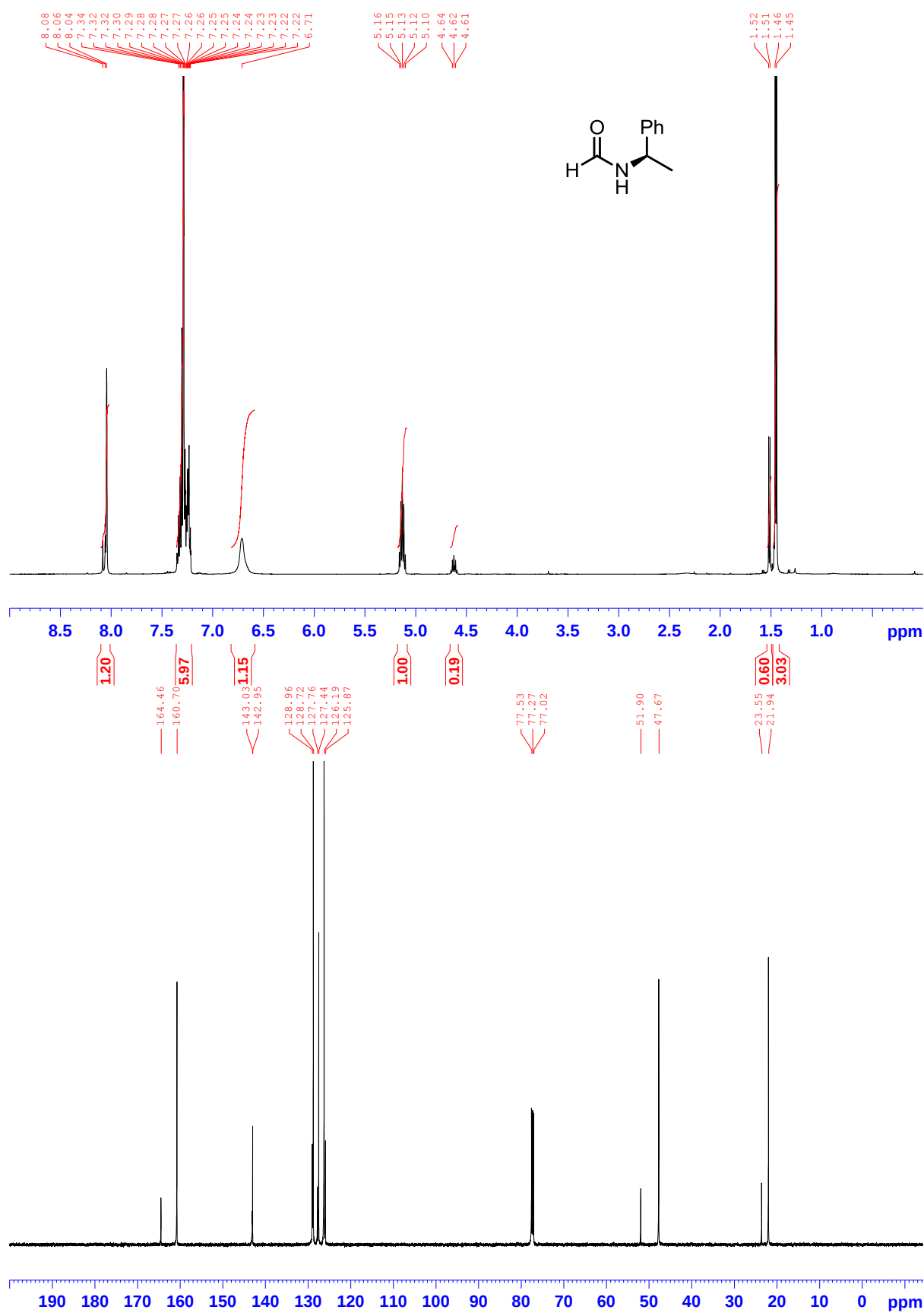
N-Phenethylformamide 7b



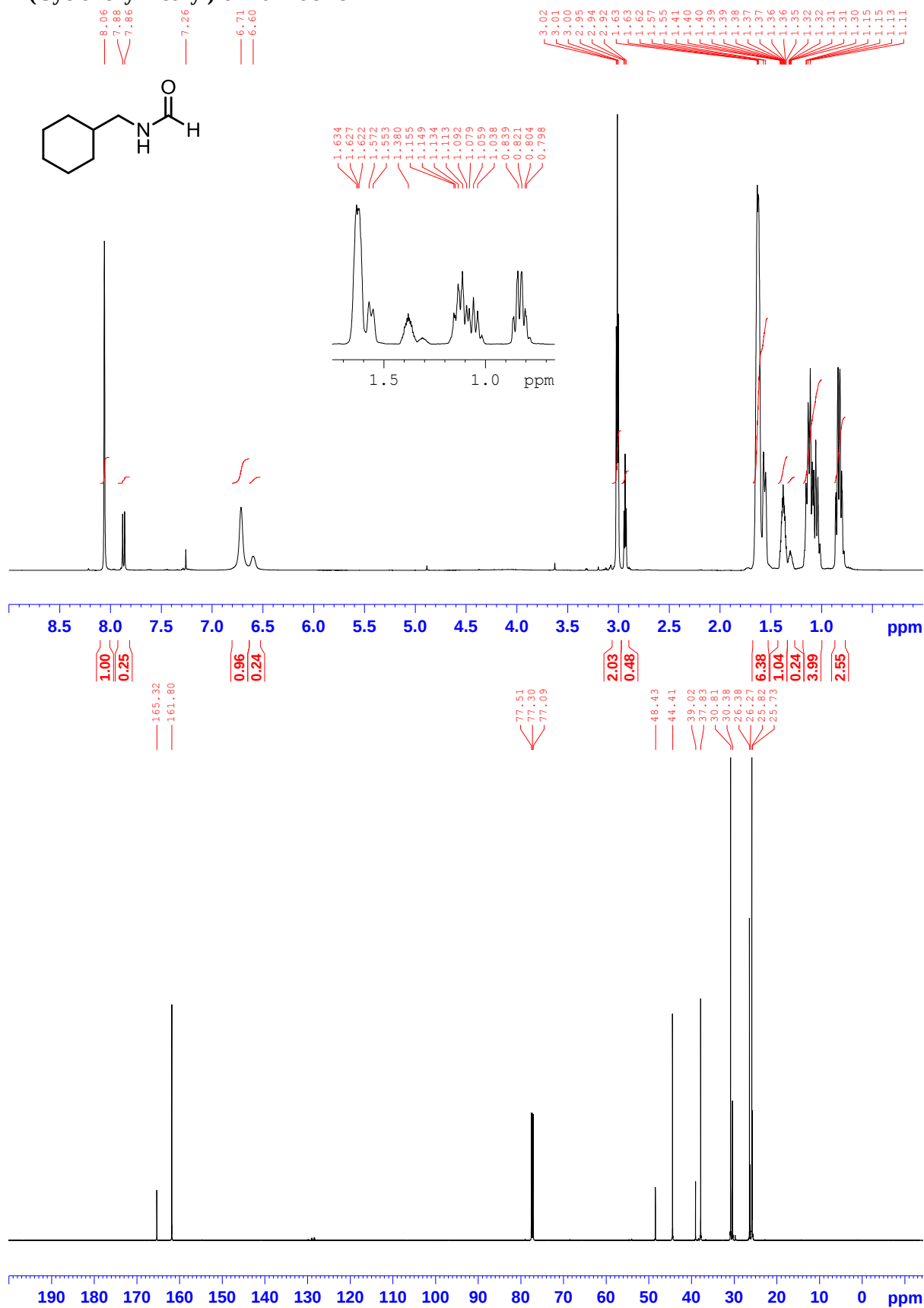
***N*-(4-Methoxybenzyl)formamide 7c**



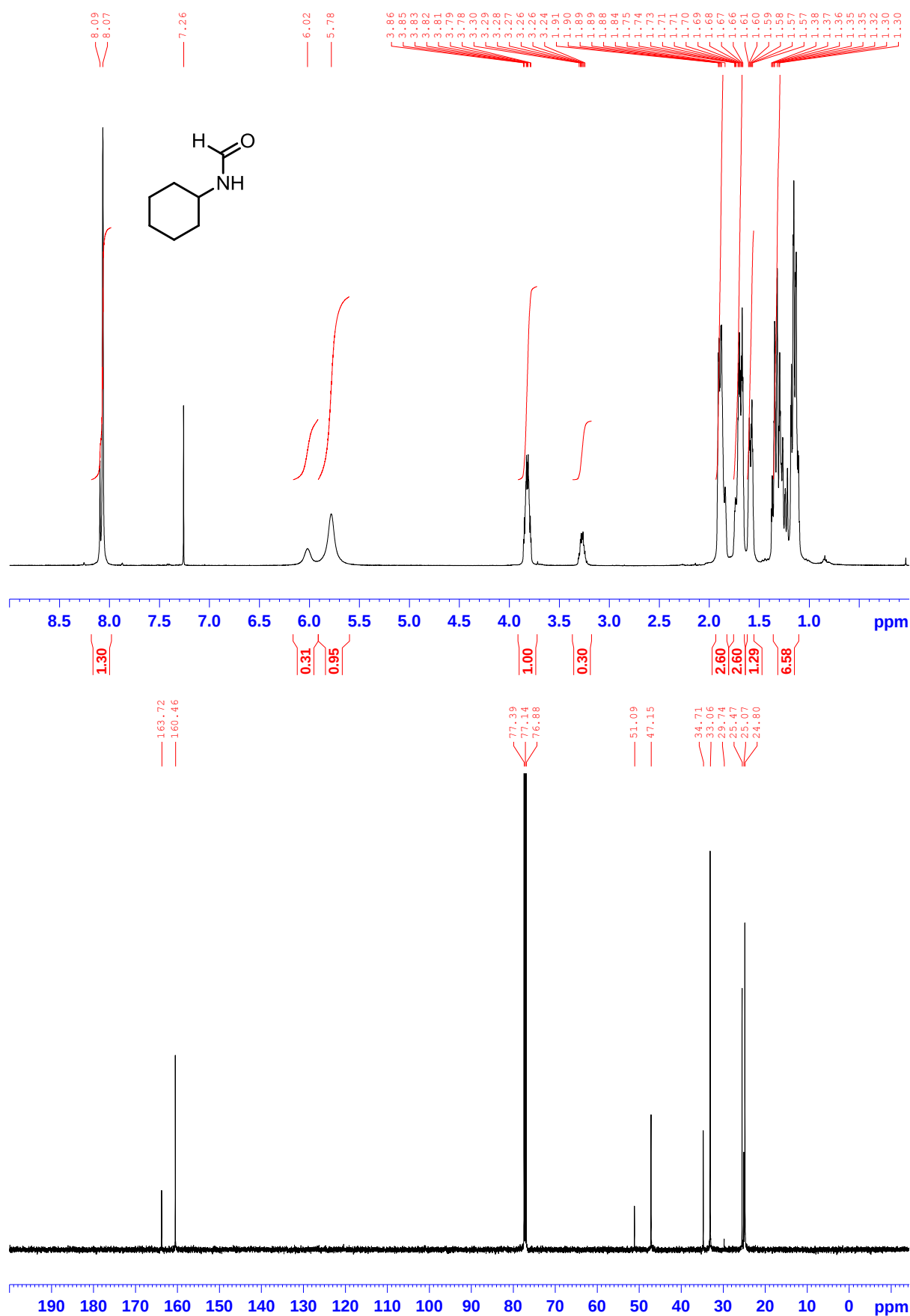
(R)-N-(1-Phenylethyl)formamide 7d



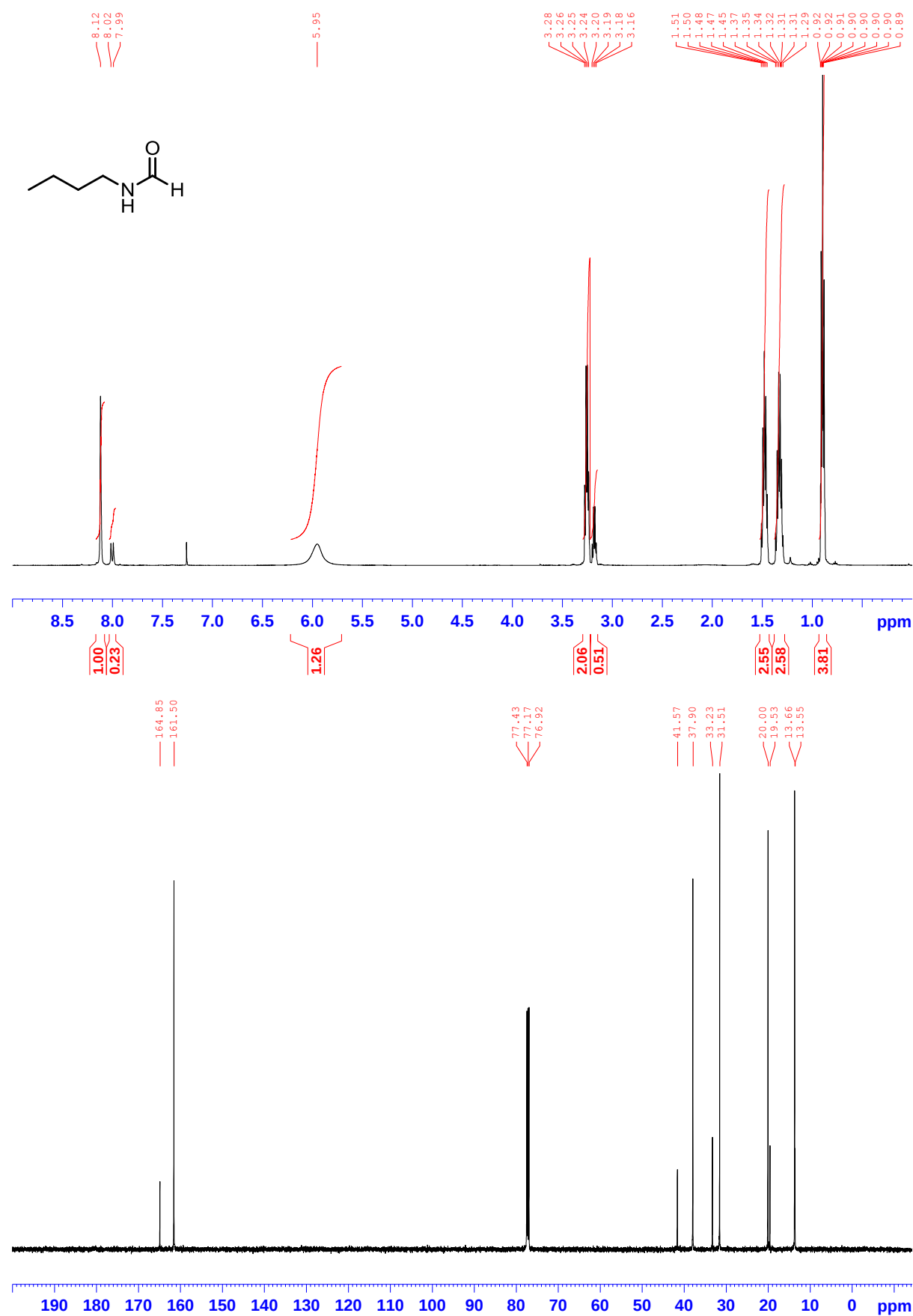
***N*-(Cyclohexylmethyl)formamide 7e**



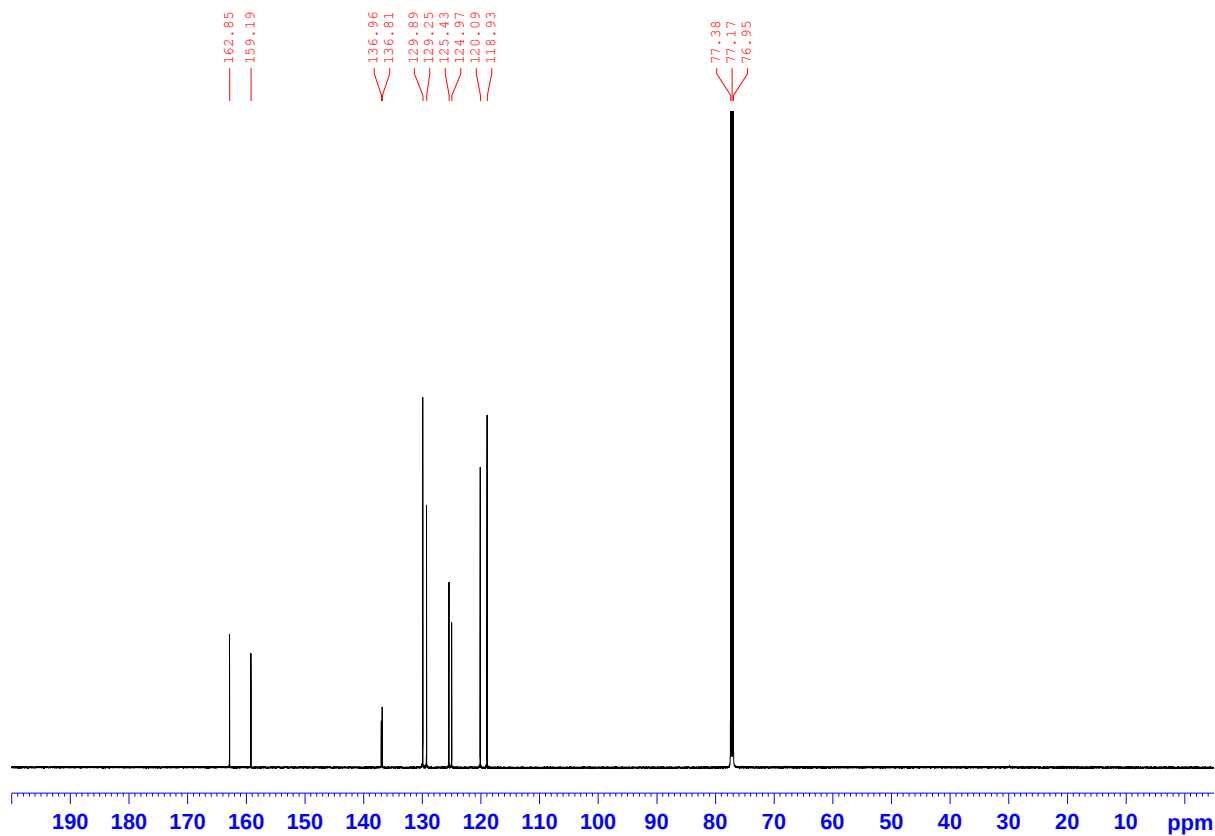
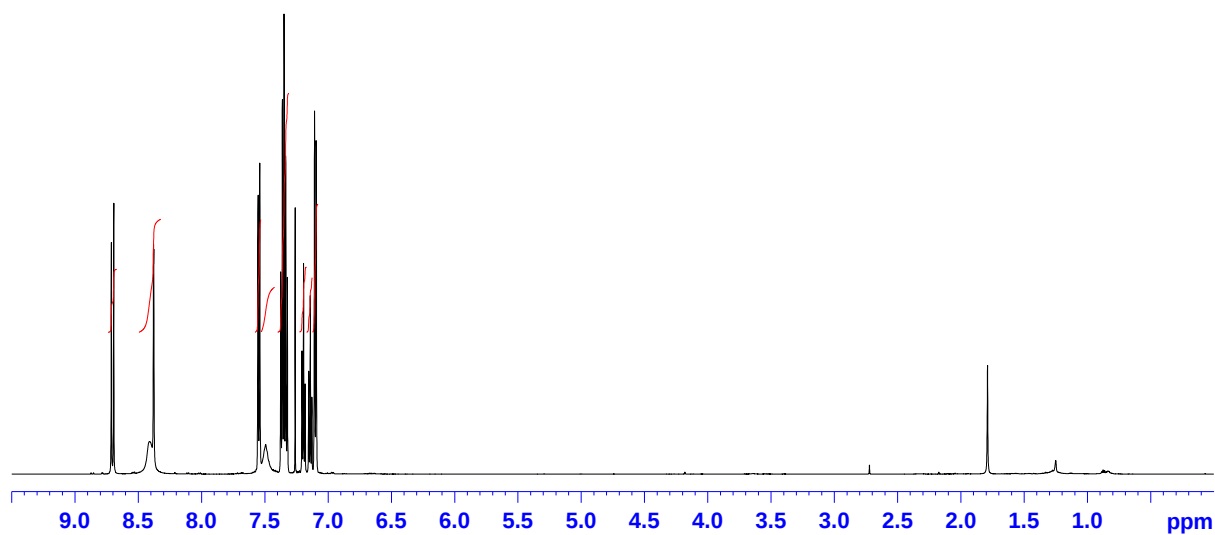
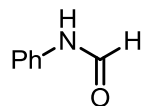
N-Cyclohexylformamide 7f



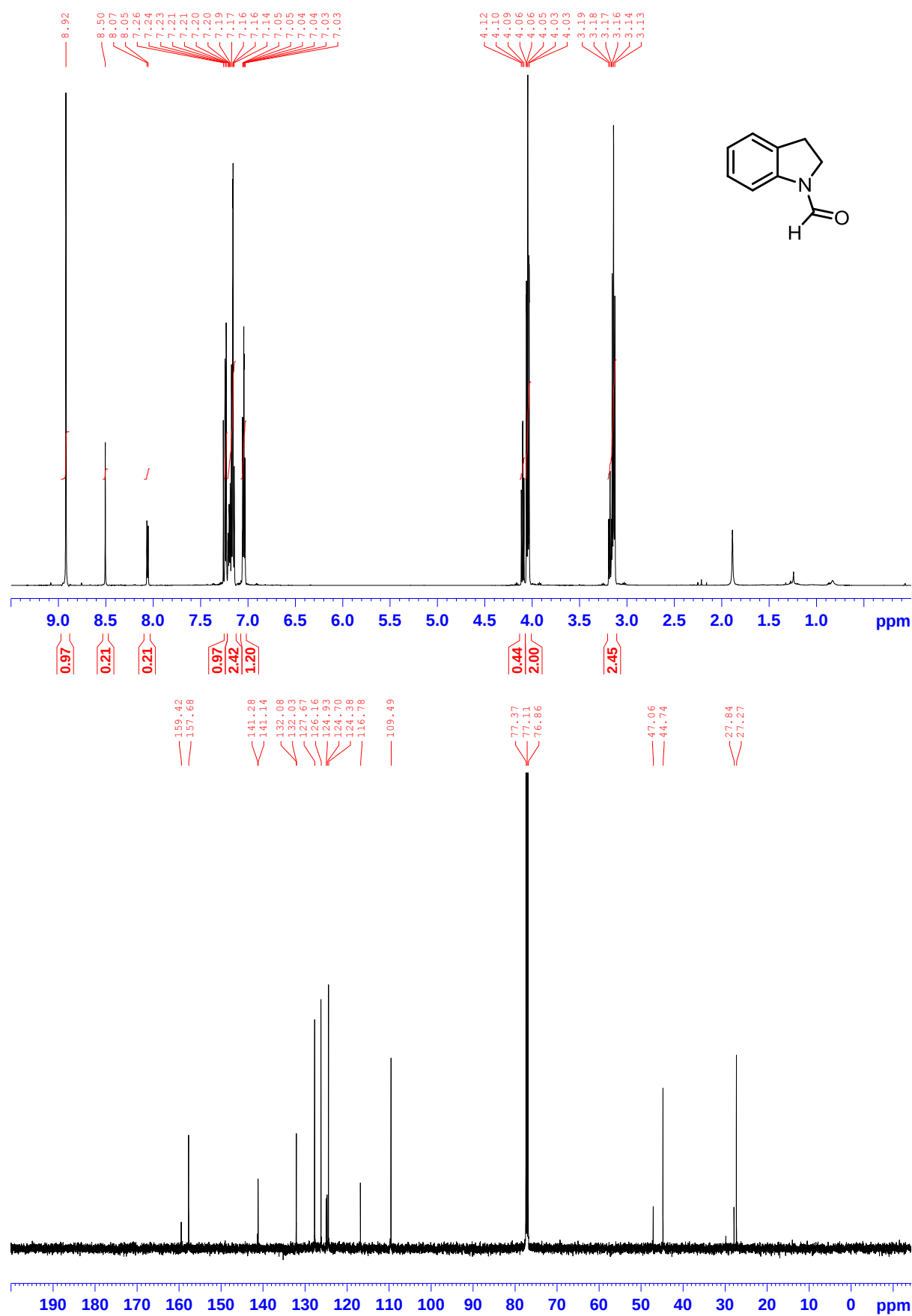
N-Butylformamide 7g



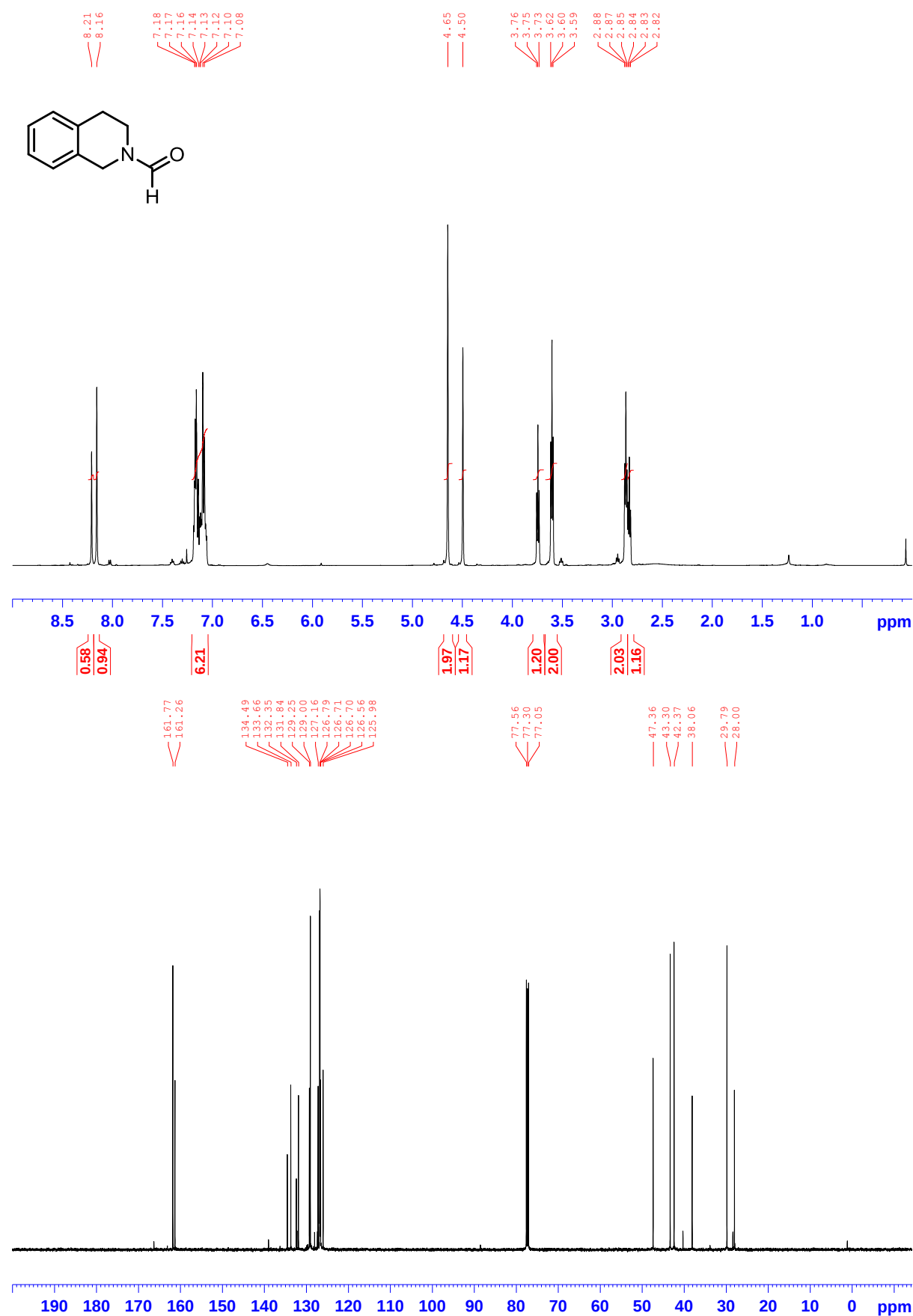
N-Phenylformamide 7h



1-Indolinecarbaldehyde 7i



3,4-Dihydro-2(1H)-isoquinolinecarbaldehyde 7j



2 Determination of enantiomeric excess and derivatisation

Compounds **6h** and **6i** were converted to the corresponding *N*-benzoyl derivatives **6k** and **6l** as described below in order to measure the enantiomeric ratio.

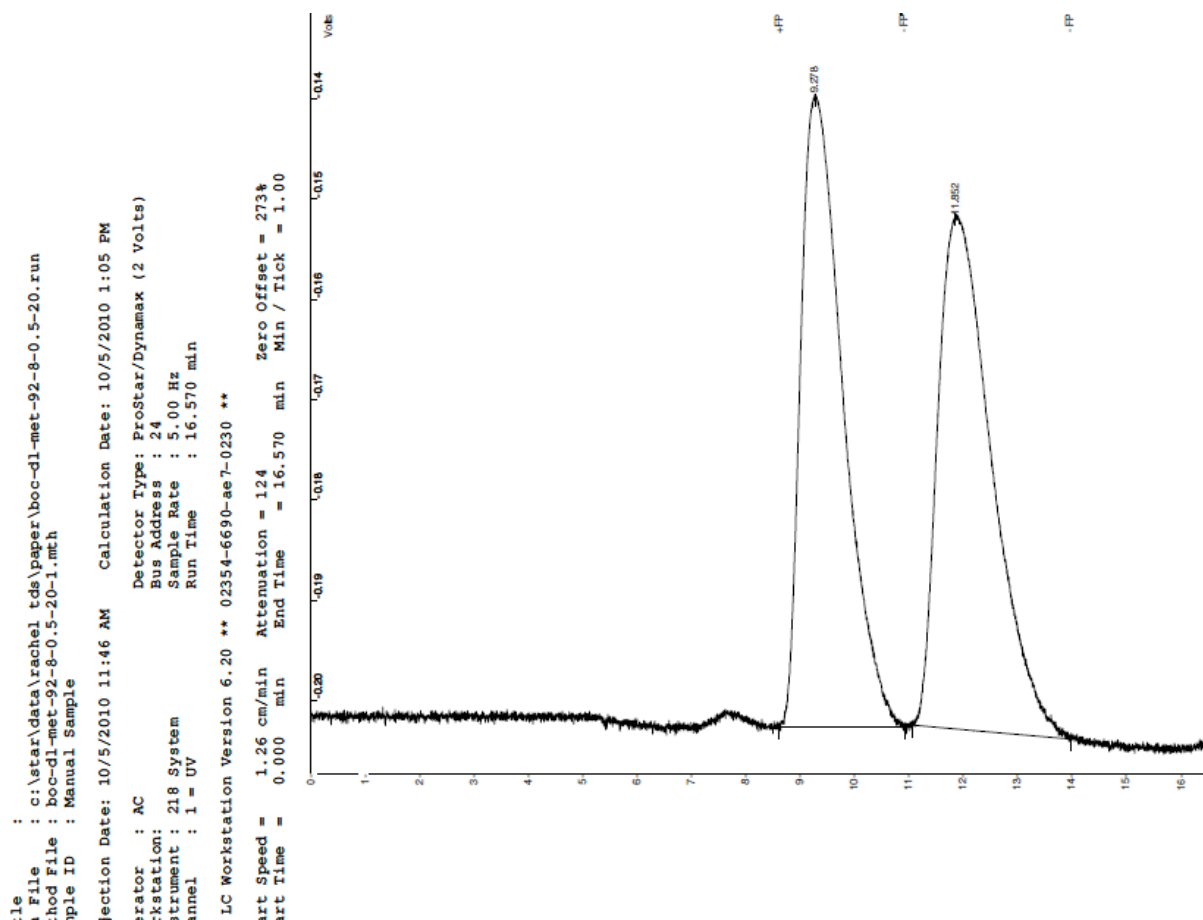
Representative Procedure for Boc deprotection

Boc-L-Alanine pyrrolidine amide was stirred in a 1:1 solution of TFA and CH₂Cl₂ (5 ml) for 1 h. The reaction mixture was concentrated *in vacuo* to yield the crude salt. The product was carried forward to the next step without further purification.

Representative Procedure for benzoyl protection

Benzoyl chloride (2 eq) was added dropwise to L-alanine pyrrolidine amide TFA salt (1 eq) and triethylamine (3 eq) in CH₂Cl₂ at 0 °C. The reaction mixture was allowed to warm to room temperature and stirred for 2 h. The reaction mixture was then diluted with CH₂Cl₂, washed with water (10 ml), dried over MgSO₄ and concentrated *in vacuo*. The crude product was purified by column chromatography (PE-EtOAc 2:1) to yield the *N*-benzoyl amino acid amide.

Boc-DL-Methionine-benzamide



Print Date: Tue Oct 05 13:05:35 2010

Page 1 of 1

Title :
 Run File : c:\star\data\rachel tds\paper\boc-dl-met-92-8-0.5-20.run
 Method File : boc-dl-met-92-8-0.5-20-1.mth
 Sample ID : Manual Sample

Injection Date: 10/5/2010 11:46 AM Calculation Date: 10/5/2010 1:05 PM

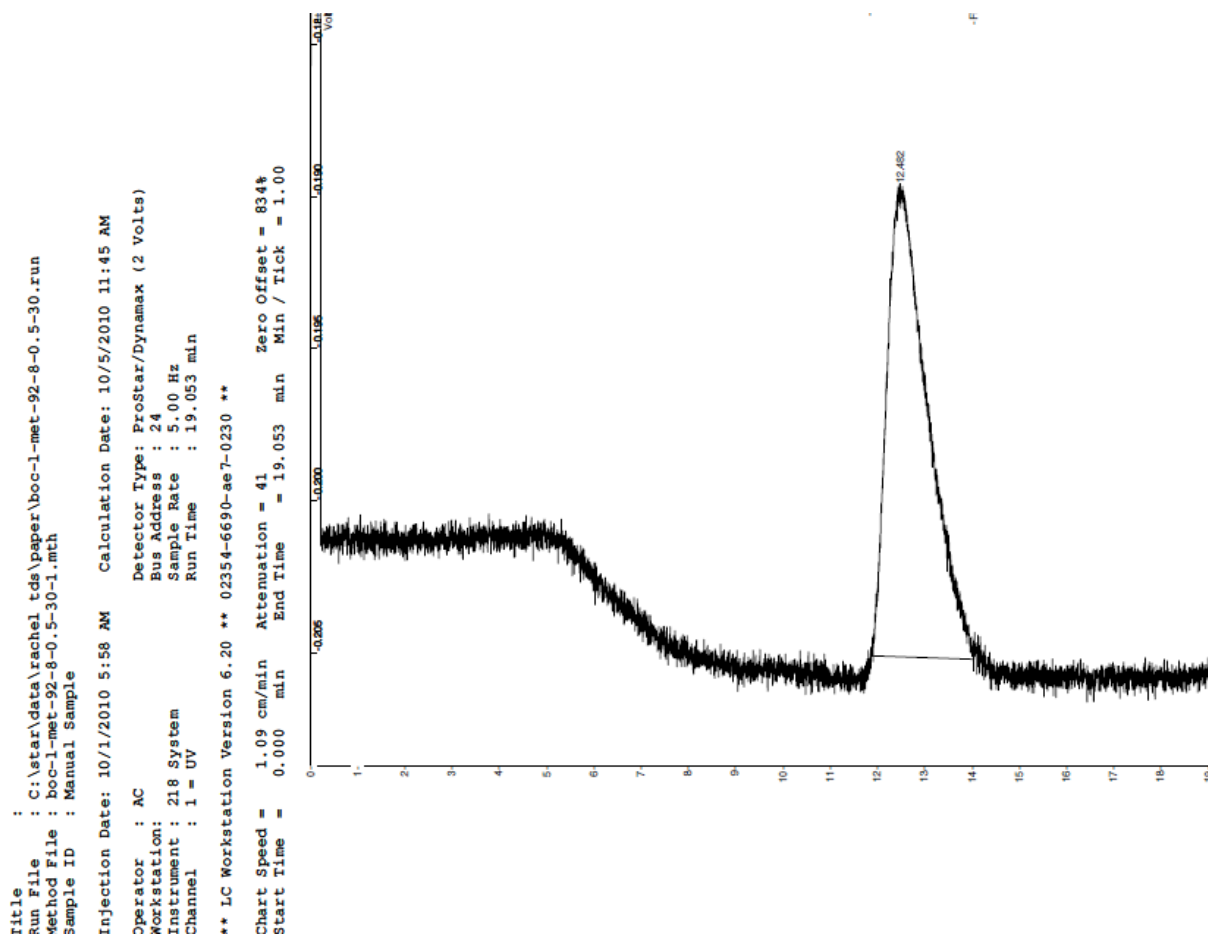
Operator : AC
 Workstation:
 Instrument : 218 System
 Channel : 1 - UV
 Detector Type: ProStar/Dynamax (2 Volts)
 Bus Address : 24
 Sample Rate : 5.00 Hz
 Run Time : 16.570 min

** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		49.0595	9.278	0.000	3391570	BB	49.7	
2		50.9406	11.852	0.000	3521614	BB	62.9	
Totals:		100.0001		0.000	6913184			

Boc-L-Methionine-benzamide 5a



Print Date: Tue Oct 05 13:06:20 2010

Page 1 of 1

Title :
 Run File : C:\star\data\rachel tds\paper\boc-l-met-92-8-0.5-30.run
 Method File : boc-l-met-92-8-0.5-30-1.mth
 Sample ID : Manual Sample

Injection Date: 10/1/2010 5:58 AM Calculation Date: 10/5/2010 11:45 AM

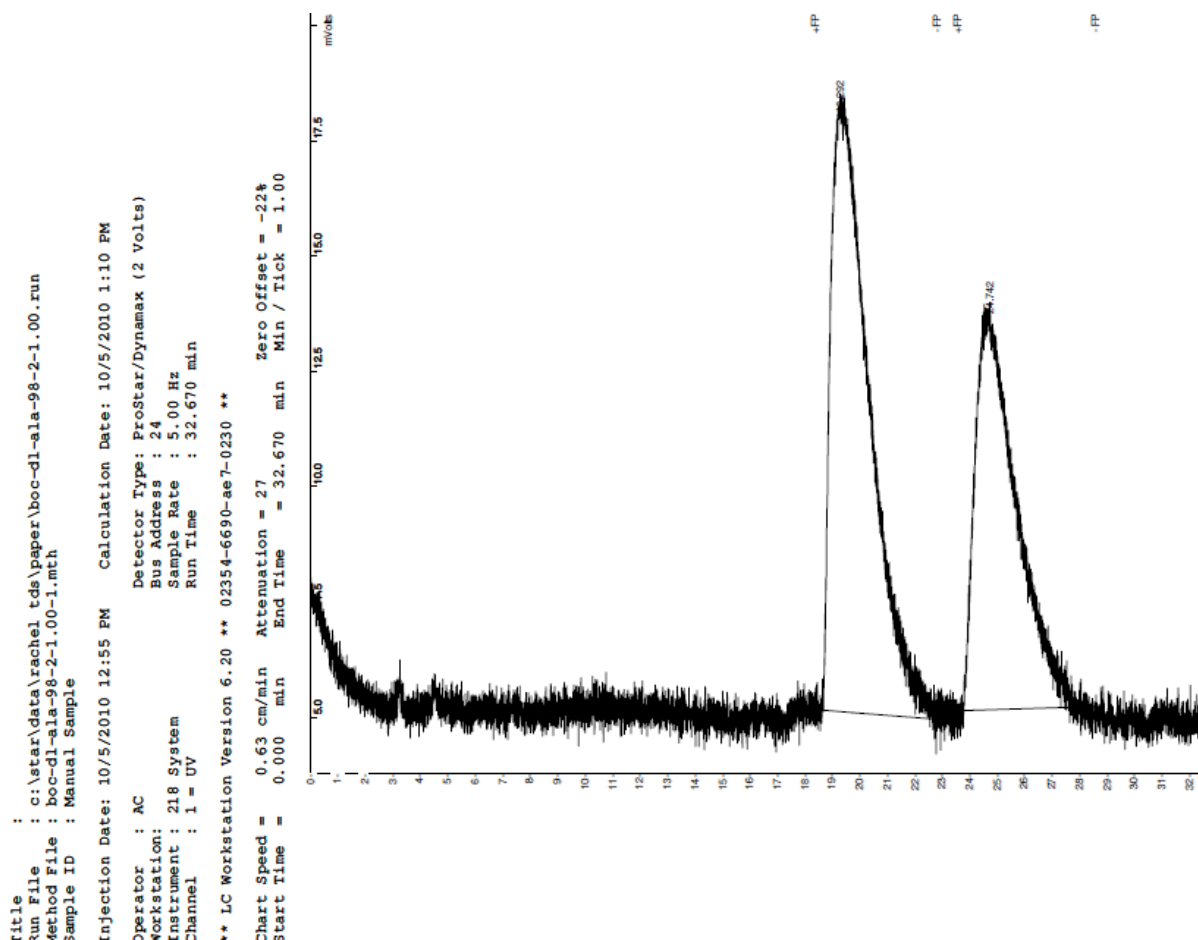
Operator : AC
 Workstation: Detector Type: ProStar/Dynamax (2 Volts)
 Instrument : 218 System Bus Address : 24
 Channel : 1 = UV Sample Rate : 5.00 Hz
 Run Time : 19.053 min

** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		100.0000	12.482	0.000	919716	BB	55.2	
Totals:		100.0000		0.000	919716			

Boc-DL-Alanine benzamide



Print Date: Tue Oct 05 13:00:47 2010

Page 1 of 1

Title :
 Run File : c:\star\data\rachel tds\paper\boc-dl-ala-98-2-1.00.run
 Method File : boc-dl-ala-98-2-1.00-1.mth
 Sample ID : Manual Sample

Injection Date: 10/5/2010 12:55 PM Calculation Date: 10/5/2010 1:10 PM

Operator : AC
 Workstation: 218 System
 Instrument : 1 - UV
 Detector Type: ProStar/Dynamax (2 Volts)
 Bus Address : 24
 Sample Rate : 5.00 Hz
 Run Time : 32.670 min

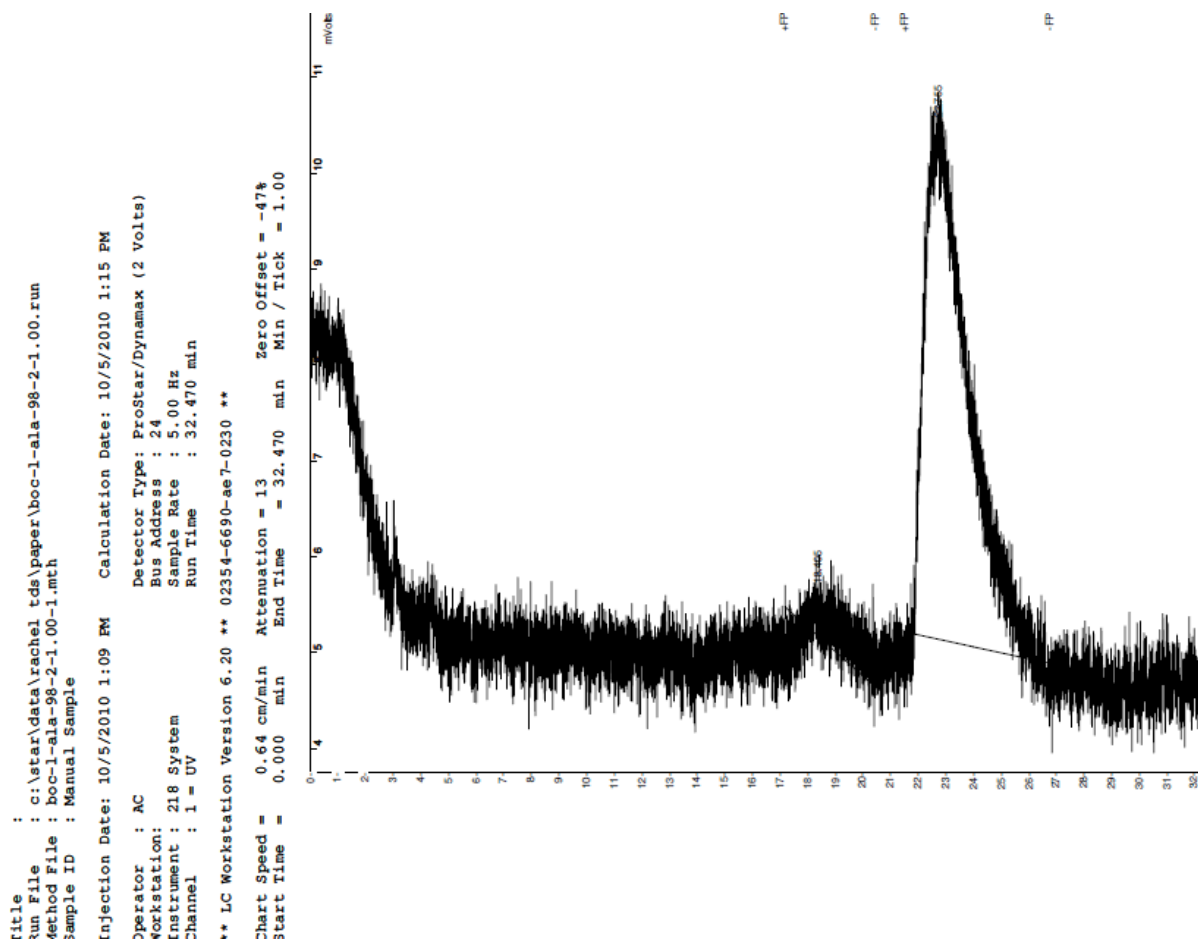
** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		59.1229	19.292	0.000	1295464	BB	85.5	
2		40.8771	24.742	0.000	895673	BB	89.4	
Totals:		100.0000		0.000	2191137			

Total Unidentified Counts : 2191137 counts

Boc-L-Alanine-benzamide 6b



Print Date: Tue Oct 05 13:01:59 2010

Page 1 of 1

Title :
 Run File : c:\star\data\rachel tds\paper\boc-l-ala-98-2-1.00.run
 Method File : boc-l-ala-98-2-1.00-1.mth
 Sample ID : Manual Sample

Injection Date: 10/5/2010 1:09 PM Calculation Date: 10/5/2010 1:15 PM

Operator : AC
 Workstation: ProStar/Dynamax (2 Volts)
 Instrument : 218 System
 Channel : 1 - UV
 Bus Address : 24
 Sample Rate : 5.00 Hz
 Run Time : 32.470 min

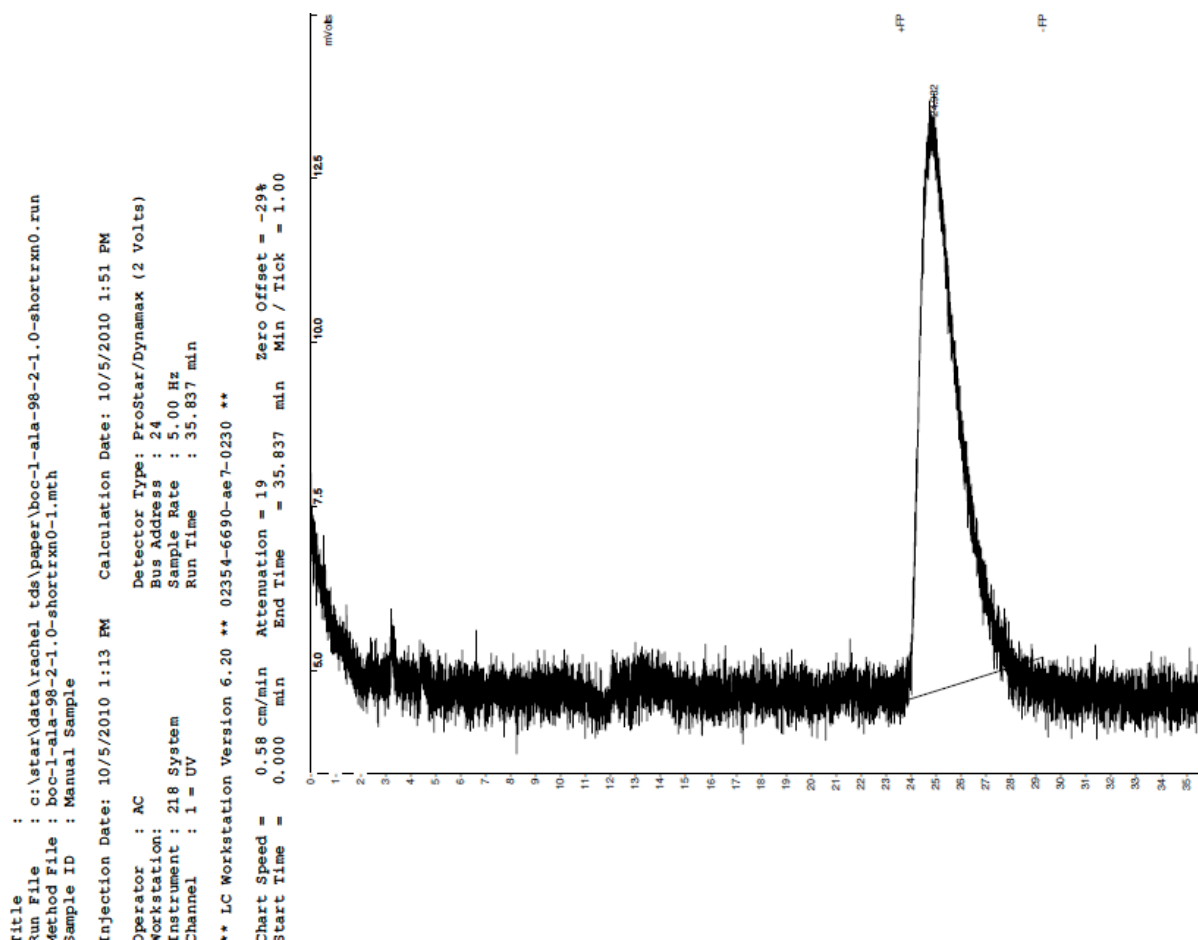
** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		3.5331	18.405	0.000	21603	BB	0.2	
2		96.4669	22.755	0.000	589851	BB	80.1	
Totals:		100.0000		0.000	611454			

Total Unidentified Counts : 611454 counts

Boc-L-Alanine benzamide 6b (8 h reaction time)



Print Date: Tue Oct 05 13:01:20 2010

Page 1 of 1

Title :
 Run File : c:\star\data\rachel tds\paper\boc-1-ala-98-2-1.0-shortrxn0.run
 Method File : boc-1-ala-98-2-1.0-shortrxn0-1.mth
 Sample ID : Manual Sample

Injection Date: 10/5/2010 1:13 PM Calculation Date: 10/5/2010 1:51 PM

Operator : AC
 Workstation: Detector Type: ProStar/Dynamax (2 Volts)
 Instrument : 218 System Bus Address : 24
 Channel : 1 - UV Sample Rate : 5.00 Hz
 Run Time : 35.837 min

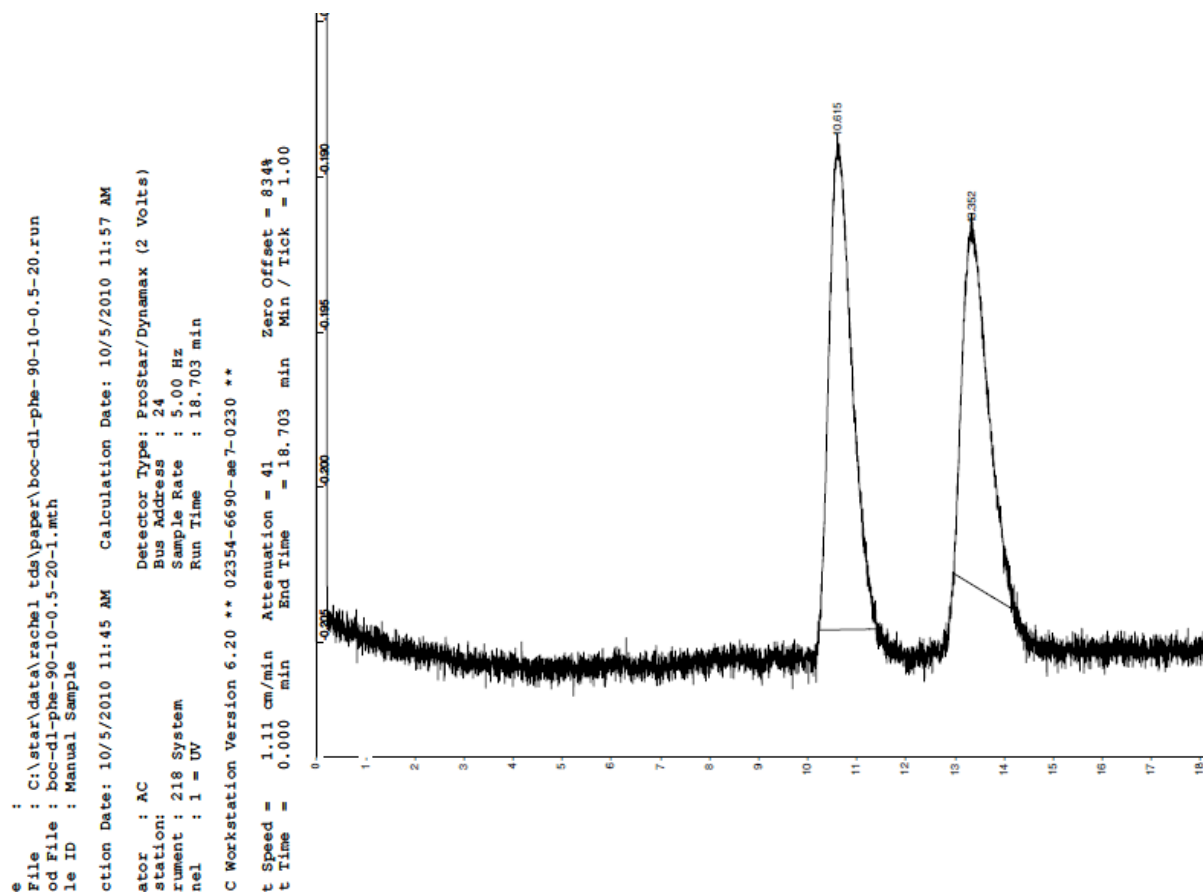
** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		100.0000	24.932	0.000	878660	BB	90.3	
Totals:		100.0000		0.000	878660			

Total Unidentified Counts : 878660 counts

Boc-DL-Phenylalanine-benzamide



Print Date: Tue Oct 05 13:08:07 2010

Page 1 of 1

Title :
 Run File : C:\star\data\rachel tds\paper\boc-dl-phe-90-10-0.5-20.run
 Method File : boc-dl-phe-90-10-0.5-20-1.mth
 Sample ID : Manual Sample

Injection Date: 10/5/2010 11:45 AM Calculation Date: 10/5/2010 11:57 AM

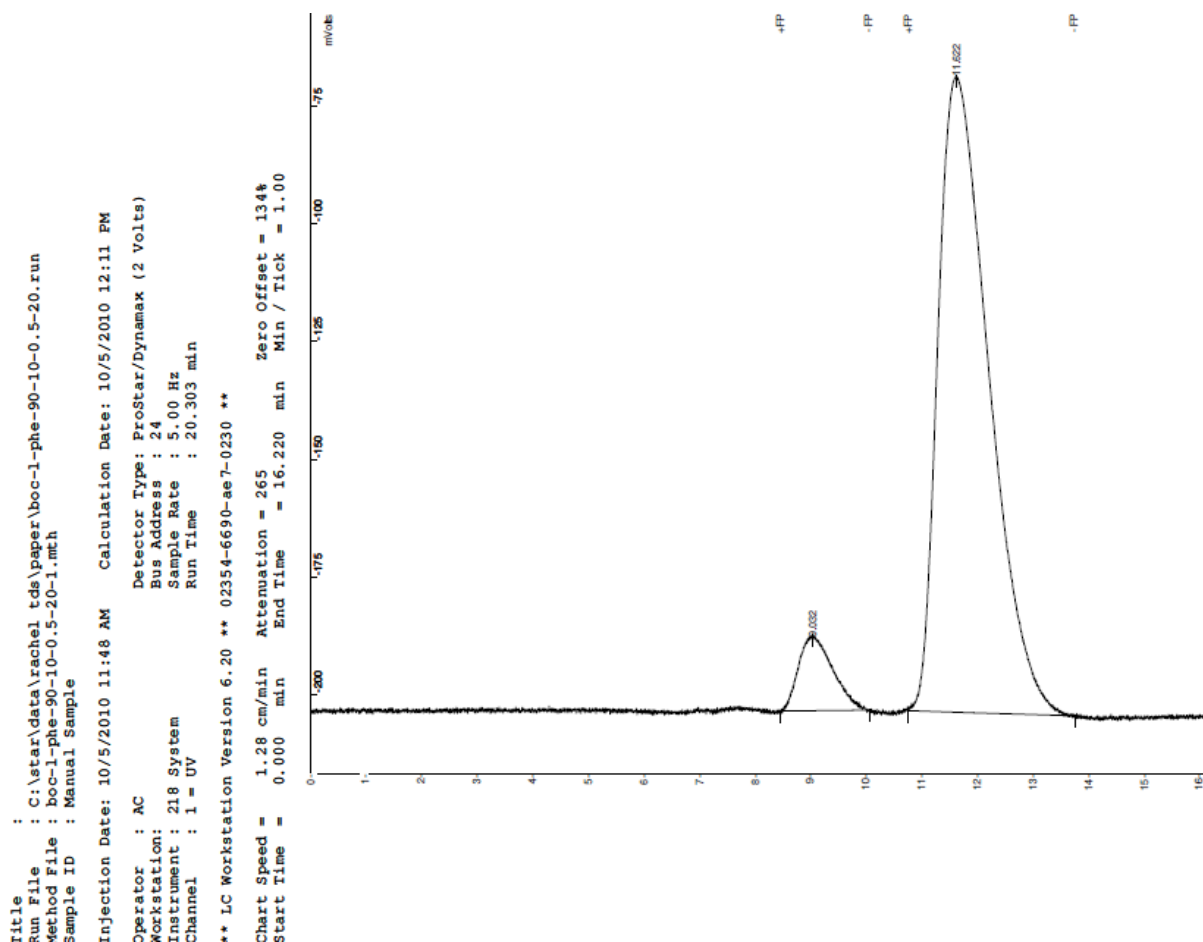
Operator : AC
 Workstation: Detector Type: ProStar/Dynamax (2 Volts)
 Instrument : 218 System Bus Address : 24
 Channel : 1 = UV Sample Rate : 5.00 Hz
 Run Time : 18.703 min

** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		54.8020	10.615	0.000	505270	BB	29.9	
2		45.1980	13.352	0.000	416721	BB	31.0	
Totals:		100.0000		0.000	921991			

Boc-L-Phenylalanine-benzamide 6c



Print Date: Tue Oct 05 12:55:10 2010

Page 1 of 1

Title :
 Run File : C:\star\data\rachel tds\paper\boc-1-phe-90-10-0.5-20.run
 Method File : boc-1-phe-90-10-0.5-20-1.mth
 Sample ID : Manual Sample

Injection Date: 10/5/2010 11:48 AM Calculation Date: 10/5/2010 12:11 PM

Operator : AC Detector Type: ProStar/Dynamax (2 Volts)
 Workstation: Bus Address : 24
 Instrument : 218 System Sample Rate : 5.00 Hz
 Channel : 1 - UV Run Time : 20.303 min

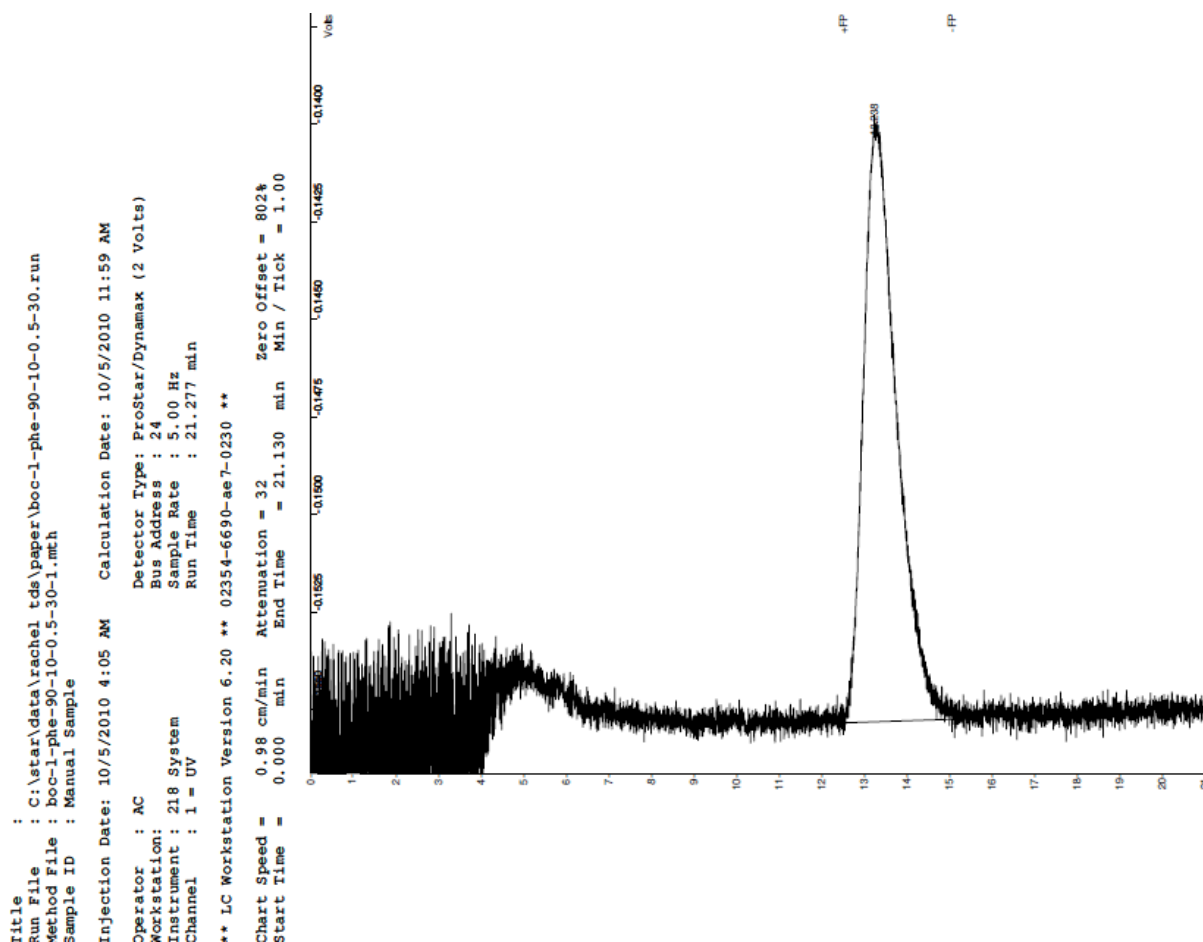
** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		7.3005	9.032	0.000	674385	BB	40.5	
2		92.6995	11.622	0.000	8563088	BB	59.5	
Totals:		100.0000		0.000	9237473			

Total Unidentified Counts : 9237473 counts

Boc-L-Phenylalanine-benzamide 6c (8 h reaction time)



Print Date: Tue Oct 05 13:08:48 2010

Page 1 of 1

Title :
 Run File : C:\star\data\rachel tds\paper\boc-l-phe-90-10-0.5-30.run
 Method File : boc-l-phe-90-10-0.5-30-1.mth
 Sample ID : Manual Sample

Injection Date: 10/5/2010 4:05 AM Calculation Date: 10/5/2010 11:59 AM

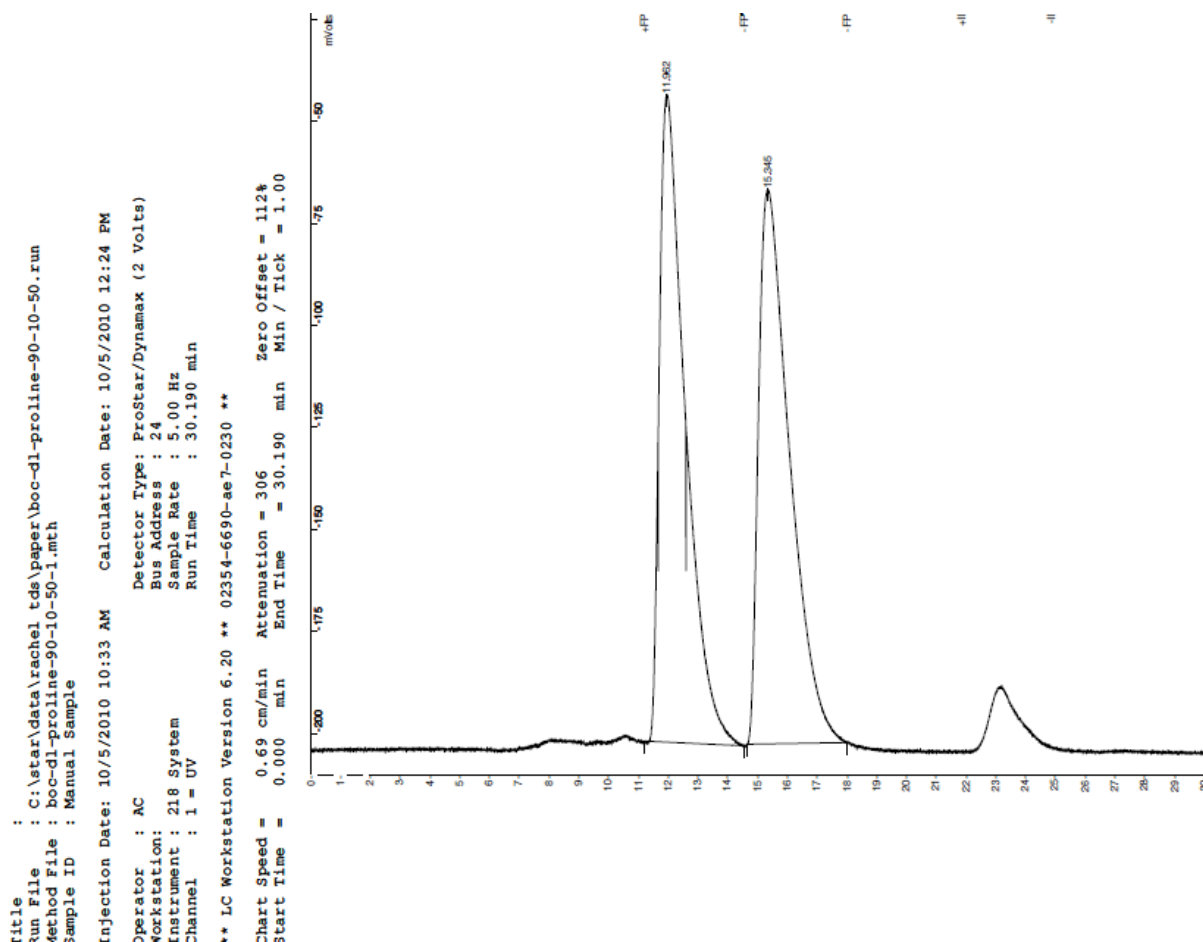
Operator : AC Detector Type: ProStar/Dynamax (2 volts)
 Workstation: Bus Address : 24
 Instrument : 218 System Sample Rate : 5.00 Hz
 Channel : 1 - UV Run Time : 21.277 min

** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		100.0000	13.238	0.000	835578	BB	48.4	
Totals:		100.0000		0.000	835578			

Boc-DL-Proline-benzamide



Print Date: Tue Oct 05 13:14:30 2010

Page 1 of 1

Title :
 Run File : C:\star\data\rachel tds\paper\boc-dl-proline-90-10-50.run
 Method File : boc-dl-proline-90-10-50-1.mth
 Sample ID : Manual Sample

Injection Date: 10/5/2010 10:33 AM Calculation Date: 10/5/2010 12:24 PM

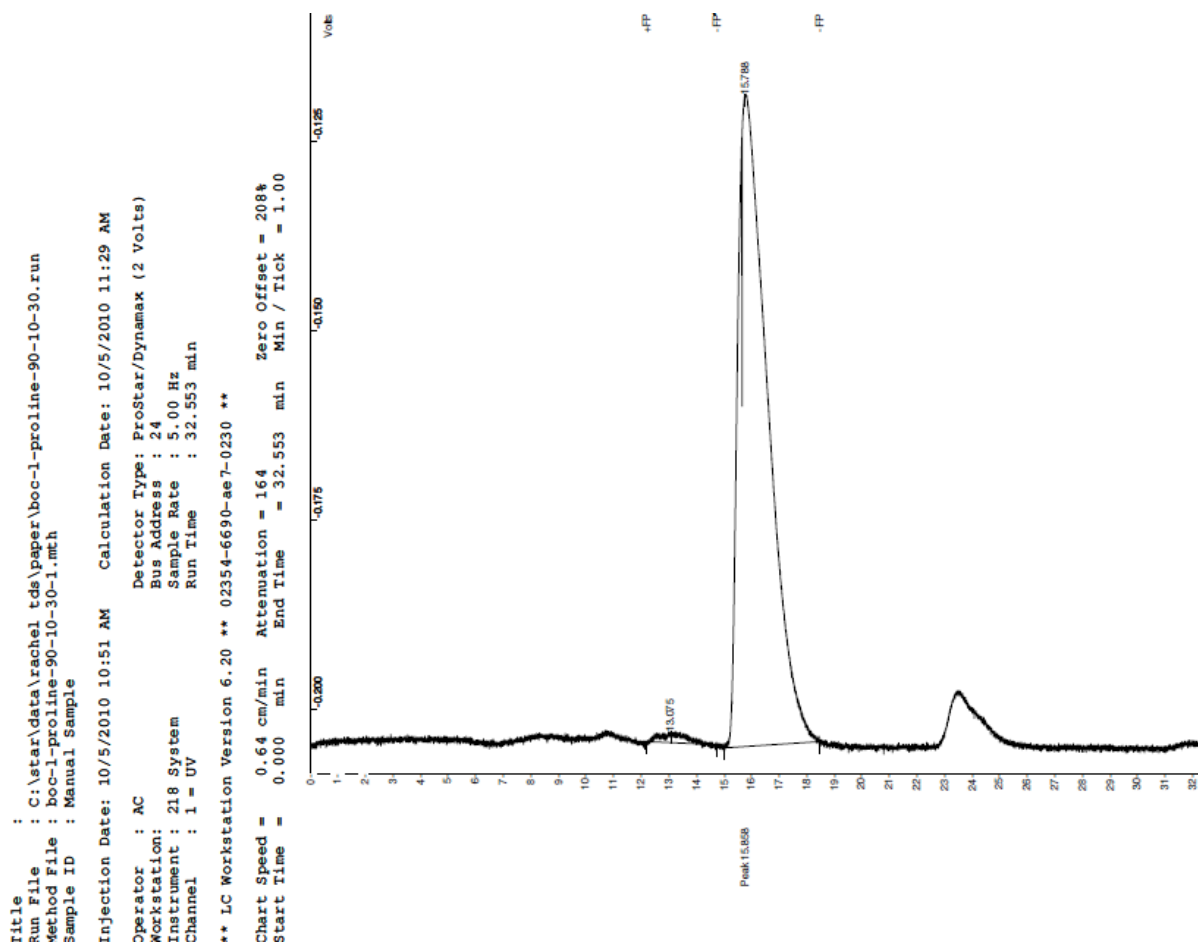
Operator : AC
 Workstation: ProStar/Dynamax (2 Volts)
 Instrument : 218 System
 Channel : 1 - UV
 Bus Address : 24
 Sample Rate : 5.00 Hz
 Run Time : 30.190 min

** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		49.3556	11.962	0.000	9605417	BB	54.5	
2		50.6444	15.345	0.000	9856222	BB	67.0	
Totals:		100.0000		0.000	19461639			

Boc-L-Proline-benzamide 6d



Print Date: Tue Oct 05 13:13:45 2010

Page 1 of 1

Title :
 Run File : C:\star\data\rachel tds\paper\boc-l-proline-90-10-30.run
 Method File : boc-l-proline-90-10-30-1.mth
 Sample ID : Manual Sample

Injection Date: 10/5/2010 10:51 AM Calculation Date: 10/5/2010 11:29 AM

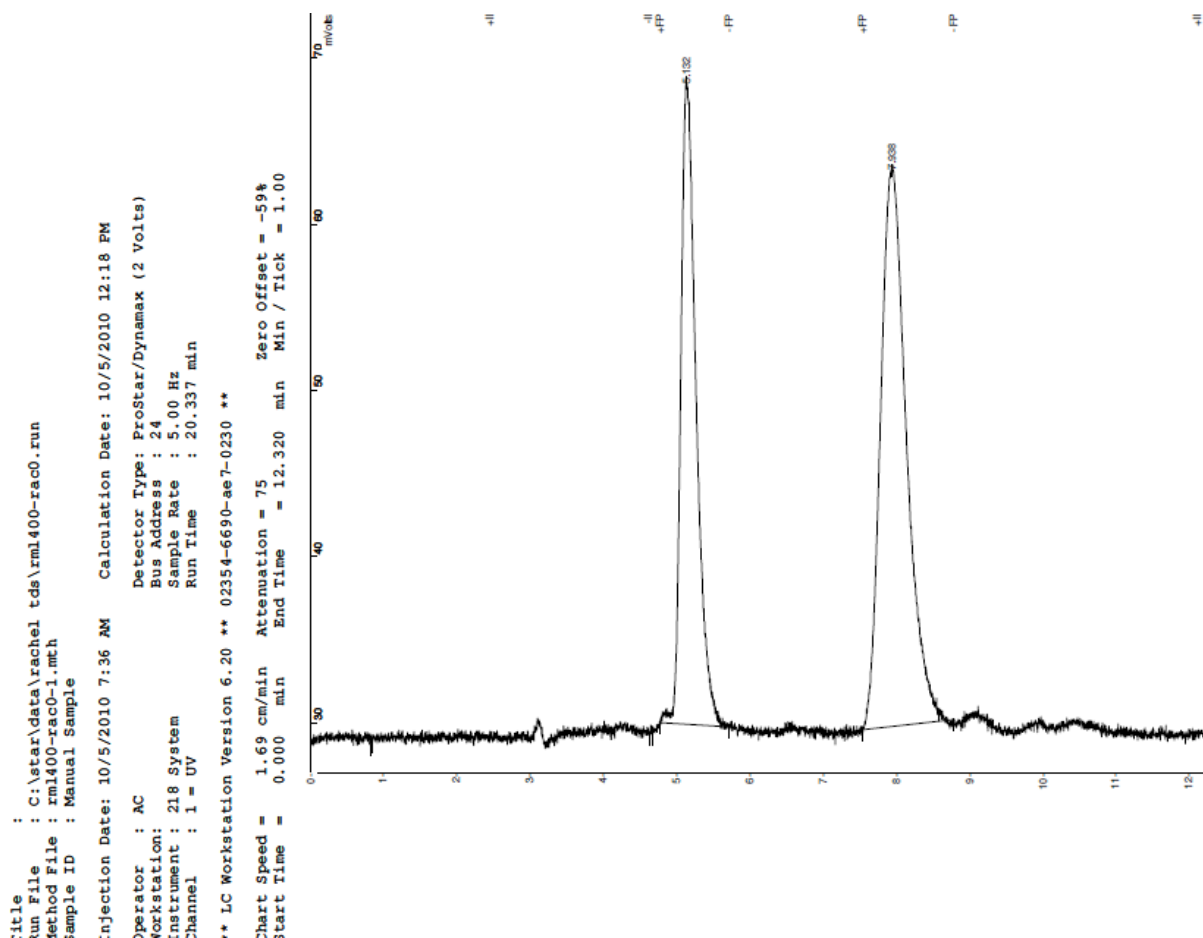
Operator : AC Detector Type: ProStar/Dynamax (2 Volts)
 Workstation: Bus Address : 24
 Instrument : 218 System Sample Rate : 5.00 Hz
 Channel : 1 - UV Run Time : 32.553 min

** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		1.2271	13.075	0.000	80230	BB	2.9	
2	Peak 15.858	98.7729	15.788	-0.031	6458176	BB	71.4	
Totals:		100.0000		-0.031	6538406			

Cbz-DL-Alanine-benzamide



Print Date: Tue Oct 05 13:16:14 2010

Page 1 of 1

Title :
 Run File : C:\star\data\rachel tds\rml400-rac0.run
 Method File : rml400-rac0-1.mth
 Sample ID : Manual Sample

Injection Date: 10/5/2010 7:36 AM Calculation Date: 10/5/2010 12:18 PM

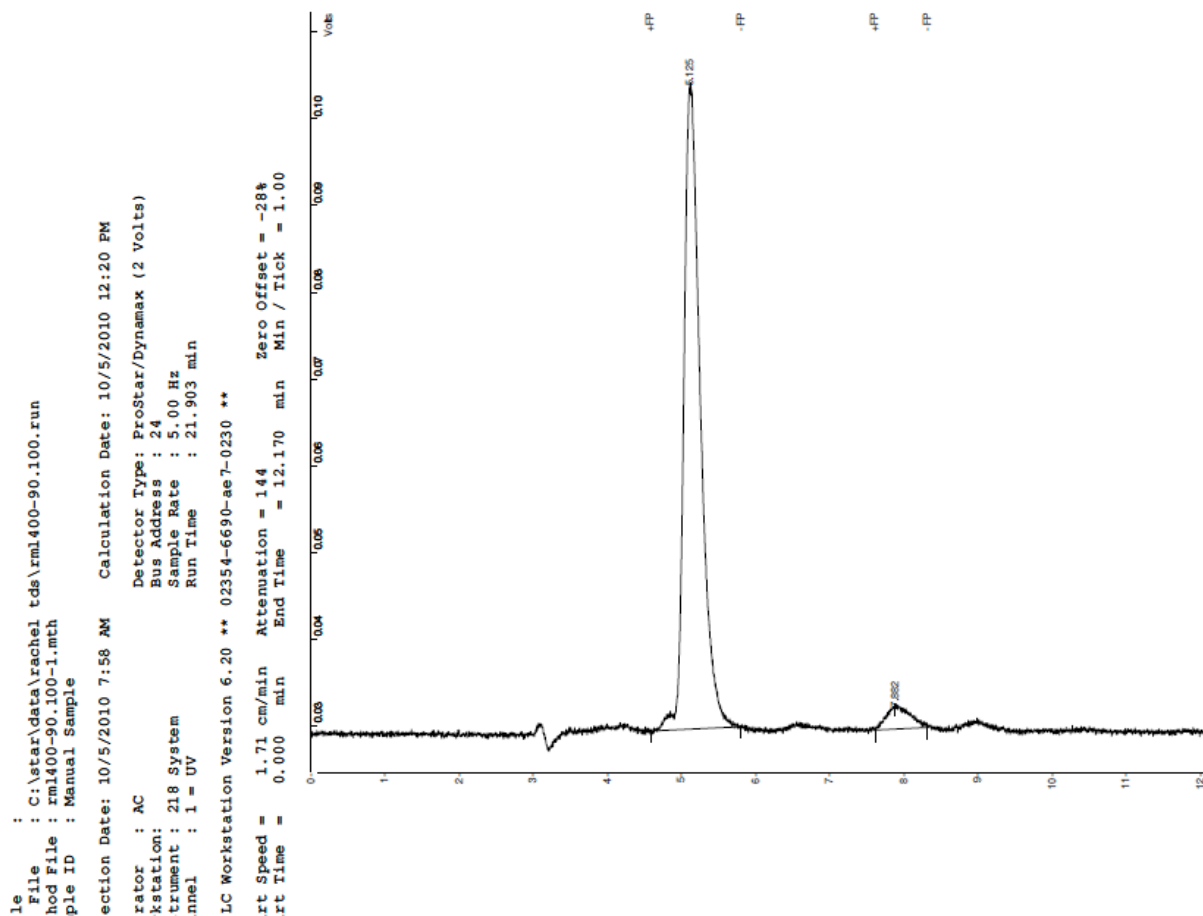
Operator : AC
 Workstation: ProStar/Dynamax (2 Volts)
 Instrument : 218 System
 Channel : 1 - UV
 Bus Address : 24
 Sample Rate : 5.00 Hz
 Run Time : 20.337 min

** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		40.5417	5.132	0.000	562471	BB	13.0	
2		59.4583	7.938	0.000	824918	BB	22.3	
Totals:		100.0000		0.000	1387389			

Cbz-L-Alanine-benzamide 6e



Print Date: Tue Oct 05 13:16:37 2010

Page 1 of 1

Title :
 Run File : C:\star\data\rachel tds\rml400-90.100.run
 Method File : rml400-90.100-1.mth
 Sample ID : Manual Sample

Injection Date: 10/5/2010 7:58 AM Calculation Date: 10/5/2010 12:20 PM

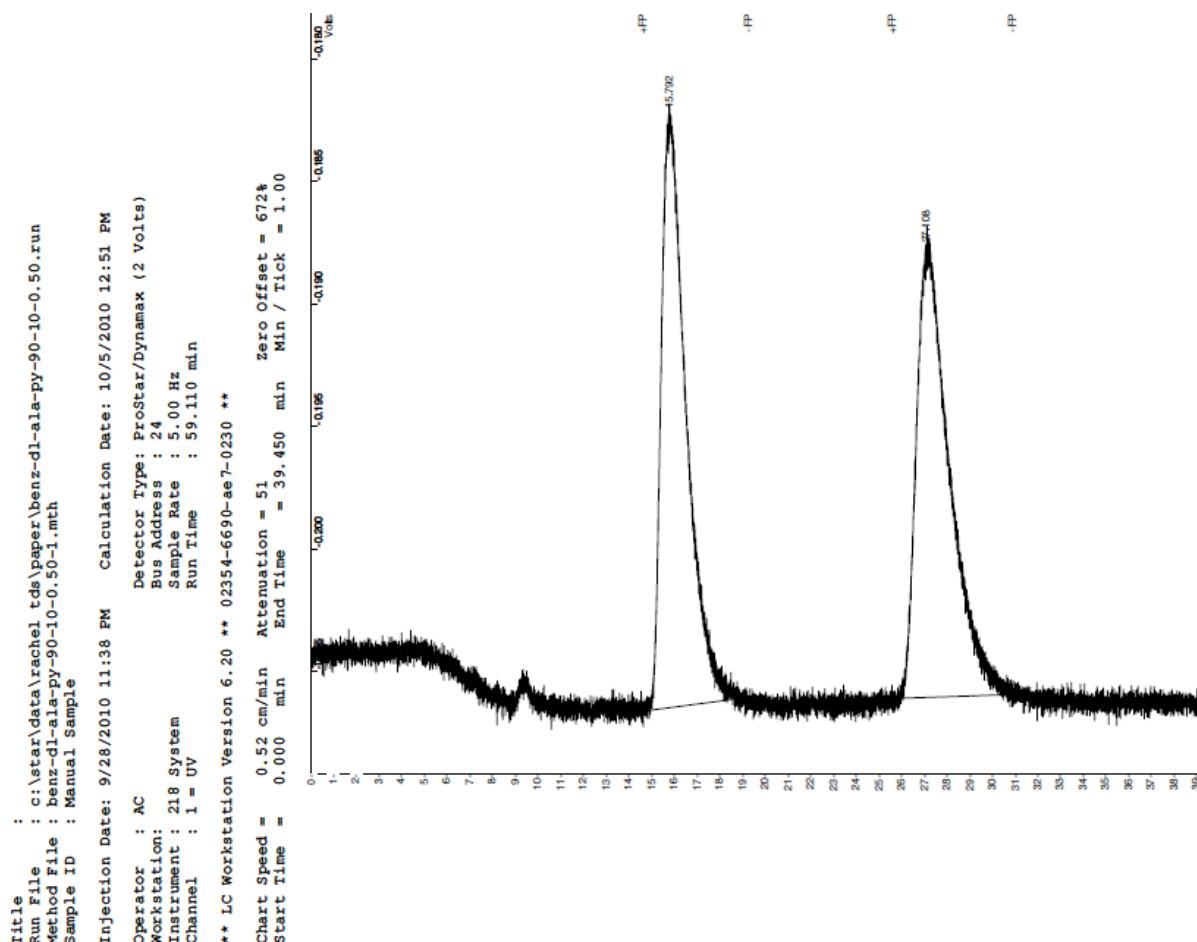
Operator : AC Detector Type: ProStar/Dynamax (2 volts)
 Workstation: Bus Address : 24
 Instrument : 218 System Sample Rate : 5.00 Hz
 Channel : 1 - UV Run Time : 21.903 min

** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		95.0551	5.125	0.000	1148915	BB	13.7	
2		4.9449	7.882	0.000	59768	BB	20.2	
Totals:		100.0000		0.000	1208683			

Benz-DL-Alanine pyrrolidine amide



Print Date: Tue Oct 05 12:57:18 2010

Page 1 of 1

Title :
 Run File : c:\star\data\rachel tds\paper\benz-dl-ala-py-90-10-0.50.run
 Method File : benz-dl-ala-py-90-10-0.50-1.mth
 Sample ID : Manual Sample

Injection Date: 9/28/2010 11:38 PM Calculation Date: 10/5/2010 12:51 PM

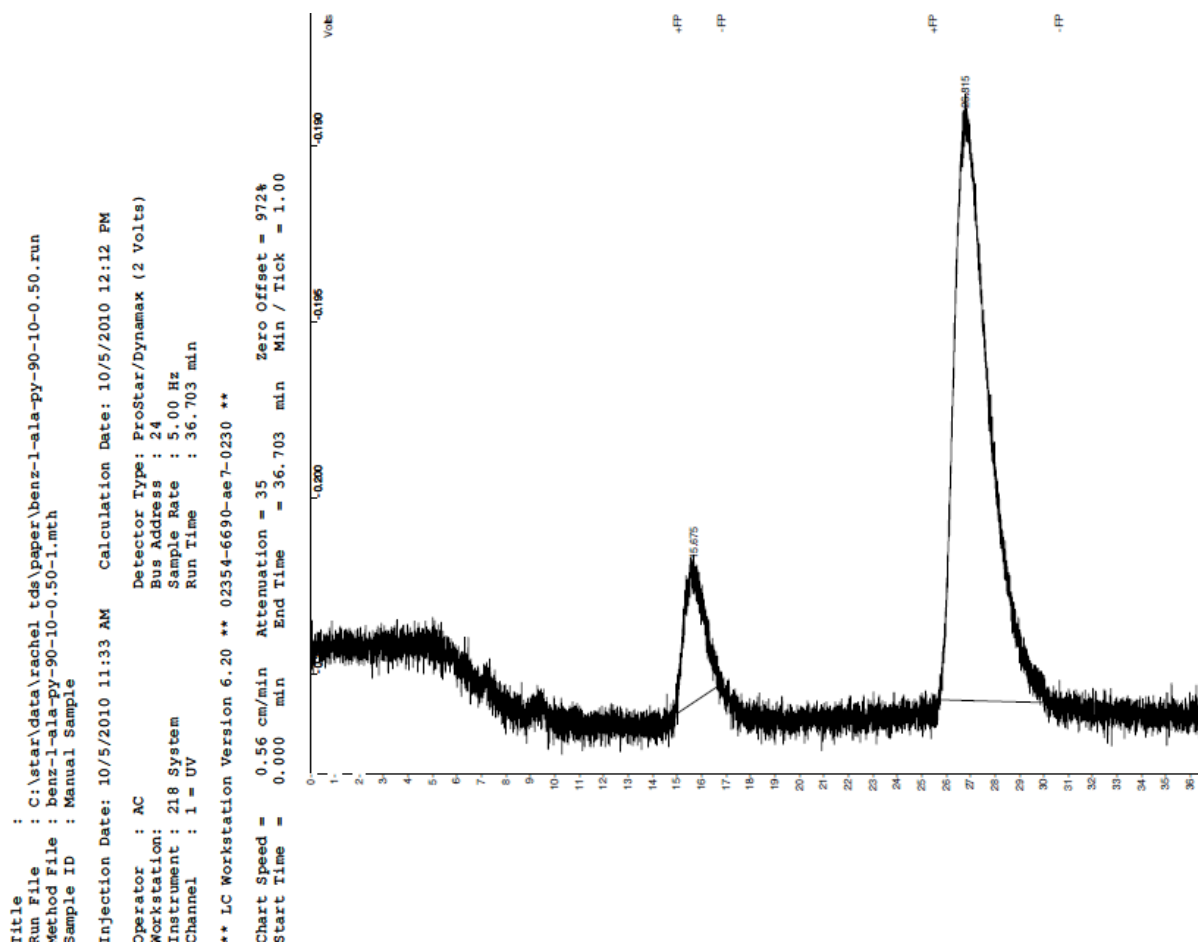
Operator : AC
 Workstation: ProStar/Dynamax (2 Volts)
 Instrument : 218 System
 Channel : 1 = UV
 Bus Address : 24
 Sample Rate : 5.00 Hz
 Run Time : 59.110 min

** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		49.7030	15.792	0.000	1846888	BB	66.3	
2		50.2970	27.108	0.000	1868960	BB	83.7	
Totals:		100.0000		0.000	3715848			

Benz-L-Alanine pyrrolidine amide 6k (obtained from 6h)



Print Date: Tue Oct 05 13:01:28 2010

Page 1 of 1

Title :
 Run File : C:\star\data\rachel tds\paper\benz-l-ala-py-90-10-0.50.run
 Method File : benz-l-ala-py-90-10-0.50-1.mth
 Sample ID : Manual Sample

Injection Date: 10/5/2010 11:33 AM Calculation Date: 10/5/2010 12:12 PM

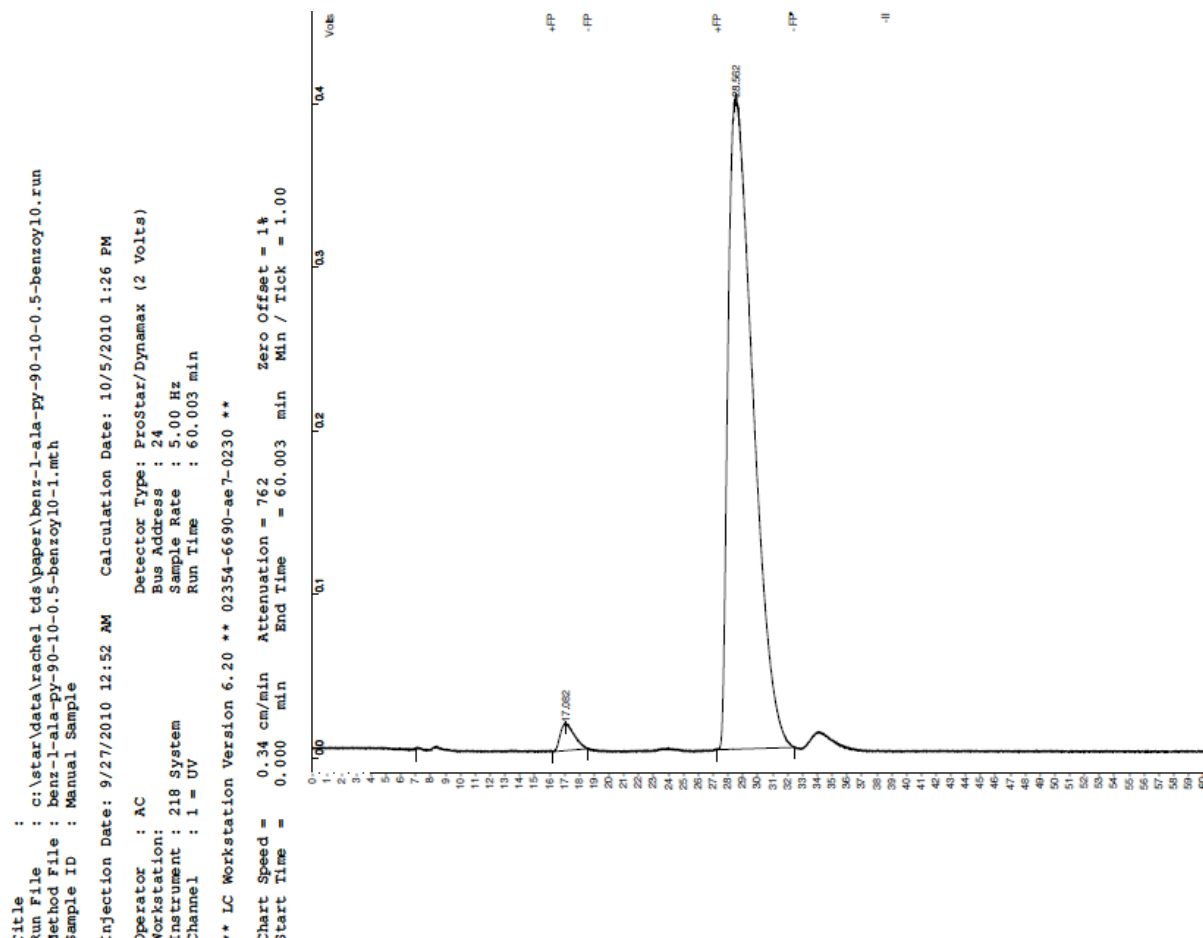
Operator : AC Detector Type: ProStar/Dynamax (2 Volts)
 Workstation: Bus Address : 24
 Instrument : 218 System Sample Rate : 5.00 Hz
 Channel : 1 - UV Run Time : 36.703 min

** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		11.8045	15.675	0.000	221509	BB	50.8	
2		88.1955	26.815	0.000	1654964	BB	85.5	
Totals:		100.0000		0.000	1876473			

Benz-L-Alanine pyrrolidine amide 6k (Directly from *N*-benzoyl alanine)



Print Date: Tue Oct 05 13:17:12 2010

Page 1 of 1

Title :
 Run File : c:\star\data\rachel tds\paper\benz-l-ala-py-90-10-0.5-benzoyl0.run
 Method File : benz-l-ala-py-90-10-0.5-benzoyl0-1.mth
 Sample ID : Manual Sample

Injection Date: 9/27/2010 12:52 AM Calculation Date: 10/5/2010 1:26 PM

Operator : AC
 Workstation: ProStar/Dynamax (2 Volts)
 Instrument : 218 System
 Channel : 1 - UV
 Bus Address : 24
 Sample Rate : 5.00 Hz
 Run Time : 60.003 min

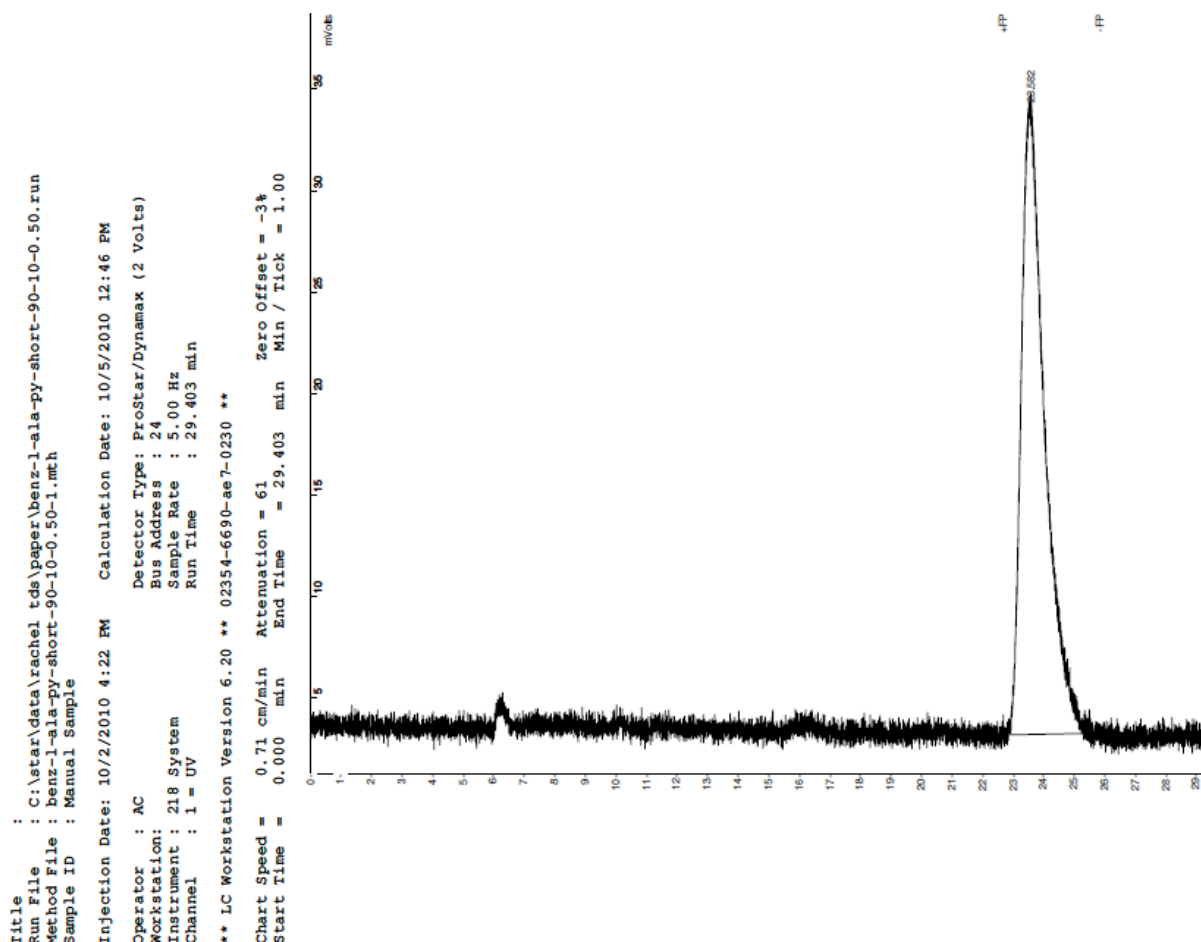
** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		2.2581	17.082	0.000	1070689	BB	61.8	
2		97.7419	28.562	0.000	46345164	BB	110.7	
Totals:		100.0000		0.000	47415853			

Total Unidentified Counts : 47415852 counts

Benz-L-Alanine pyrrolidine amide 6k (obtained from 6h; 8 h reaction time)



Print Date: Tue Oct 05 13:01:52 2010

Page 1 of 1

Title :
 Run File : C:\star\data\rachel tds\paper\benz-l-ala-py-short-90-10-0.50.run
 Method File : benz-l-ala-py-short-90-10-0.50-1.mth
 Sample ID : Manual Sample

Injection Date: 10/2/2010 4:22 PM Calculation Date: 10/5/2010 12:46 PM

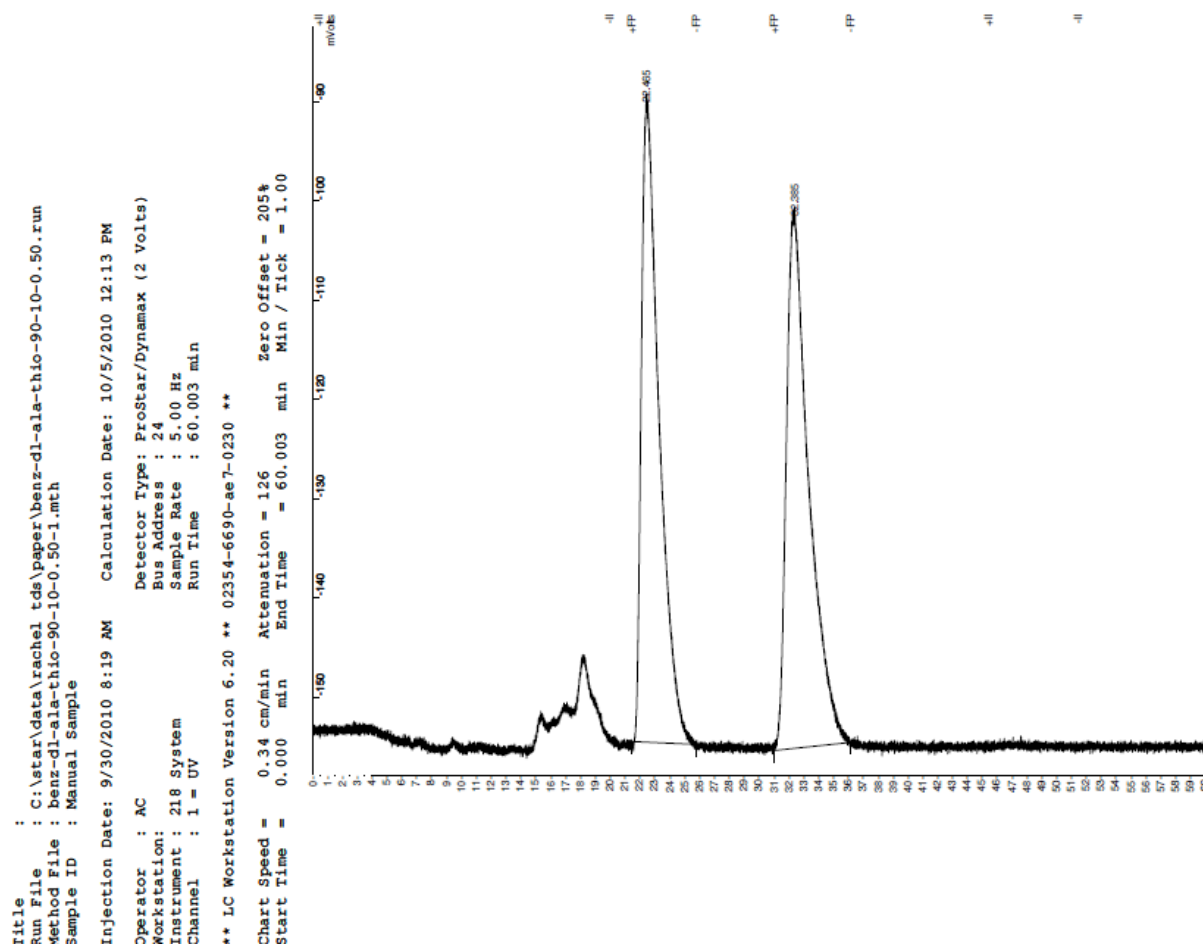
Operator : AC
 Workstation: ProStar/Dynamax (2 Volts)
 Instrument : 218 System
 Channel : 1 - UV
 Bus Address : 24
 Sample Rate : 5.00 Hz
 Run Time : 29.403 min

** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		100.0000	23.582	0.000	1660522	BB	45.4	
Totals:		100.0000		0.000	1660522			

Benz-DL-Alanine thiomorpholine amide



Print Date: Tue Oct 05 13:02:36 2010

Page 1 of 1

Title :
 Run File : C:\star\data\rachel tds\paper\benz-dl-ala-thio-90-10-0.50.run
 Method File : benz-dl-ala-thio-90-10-0.50-1.mth
 Sample ID : Manual Sample

Injection Date: 9/30/2010 8:19 AM Calculation Date: 10/5/2010 12:13 PM

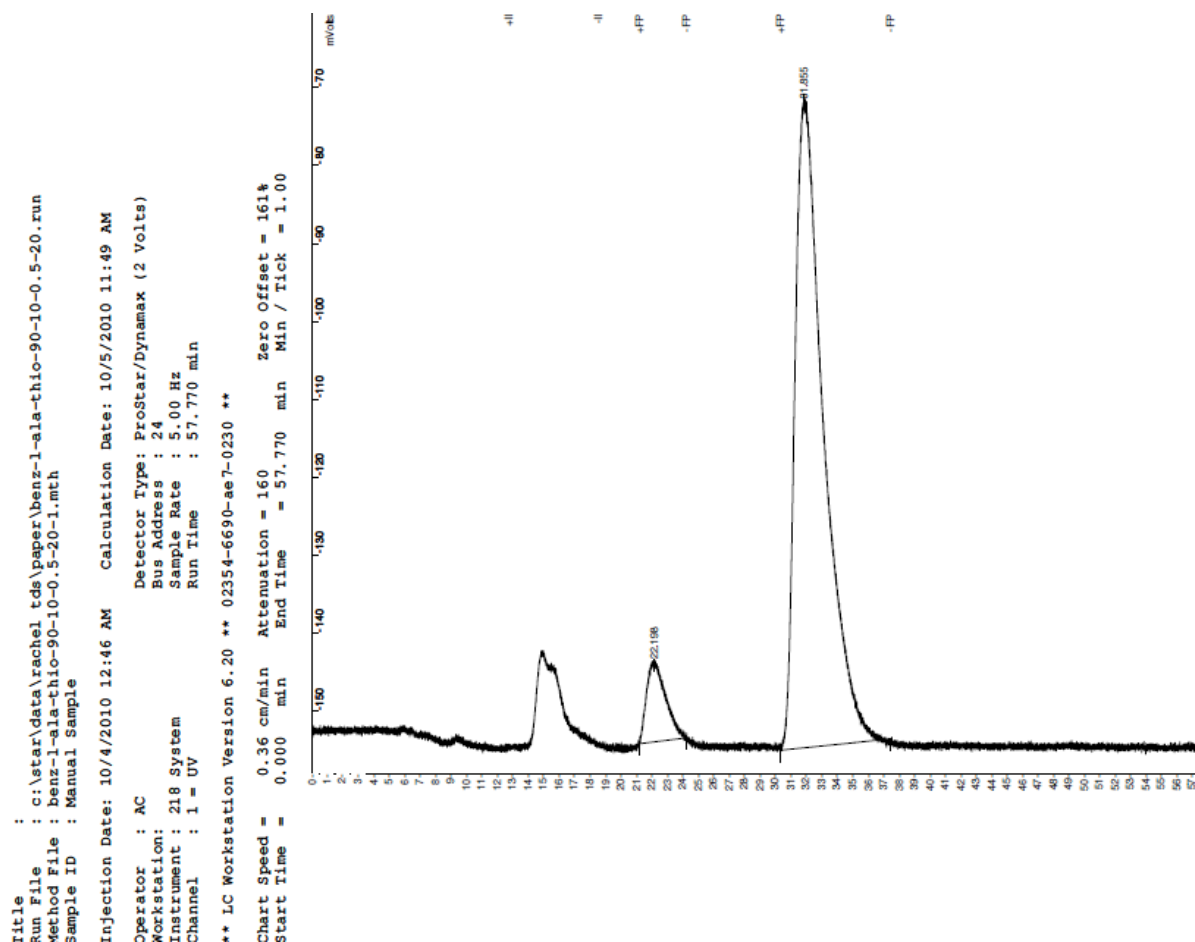
Operator : AC
 Workstation: ProStar/Dynamax (2 Volts)
 Instrument : 218 System
 Channel : 1 - UV
 Bus Address : 24
 Sample Rate : 5.00 Hz
 Run Time : 60.003 min

** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		48.5606	22.465	0.000	5356351	BB	73.2	
2		51.4394	32.385	0.000	5673891	BB	91.1	
Totals:		100.0000		0.000	11030242			

Benz-L-Alanine thiomorpholine amide 6l (obtained from 6i)



Print Date: Tue Oct 05 12:54:28 2010

Page 1 of 1

Title :
 Run File : c:\star\data\rachel tds\paper\benz-l-ala-thio-90-10-0.5-20.run
 Method File : benz-l-ala-thio-90-10-0.5-20-1.mth
 Sample ID : Manual Sample

Injection Date: 10/4/2010 12:46 AM Calculation Date: 10/5/2010 11:49 AM

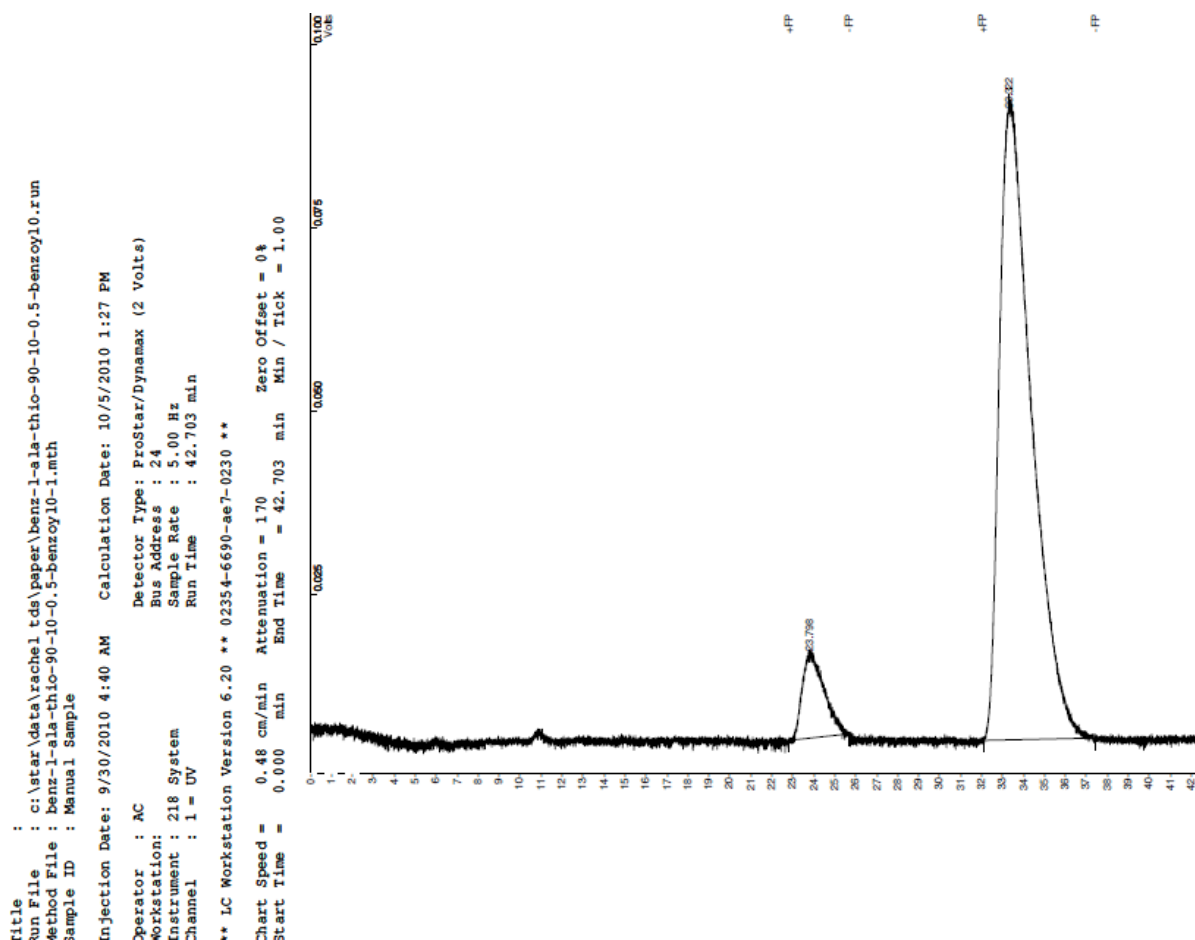
Operator : AC
 Workstation: ProStar/Dynamax (2 Volts)
 Instrument : 218 System
 Channel : 1 - UV
 Bus Address : 24
 Sample Rate : 5.00 Hz
 Run Time : 57.770 min

** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		7.6265	22.198	0.000	879805	BB	80.3	
2		92.3735	31.855	0.000	10656364	BB	112.2	
Totals:		100.0000		0.000	11536169			

Benz-L-Alanine thiomorpholine amide 6l (directly from *N*-benzoyl alanine)



Print Date: Tue Oct 05 13:16:25 2010

Page 1 of 1

Title :
 Run File : c:\star\data\rachel tds\paper\benz-l-ala-thio-90-10-0.5-benzoyl0.run
 Method File : benz-l-ala-thio-90-10-0.5-benzoyl0-1.mth
 Sample ID : Manual Sample

Injection Date: 9/30/2010 4:40 AM Calculation Date: 10/5/2010 1:27 PM

Operator : AC
 Workstation: ProStar/Dynamax (2 Volts)
 Instrument : 218 System Bus Address : 24
 Channel : 1 = UV Sample Rate : 5.00 Hz
 Run Time : 42.703 min

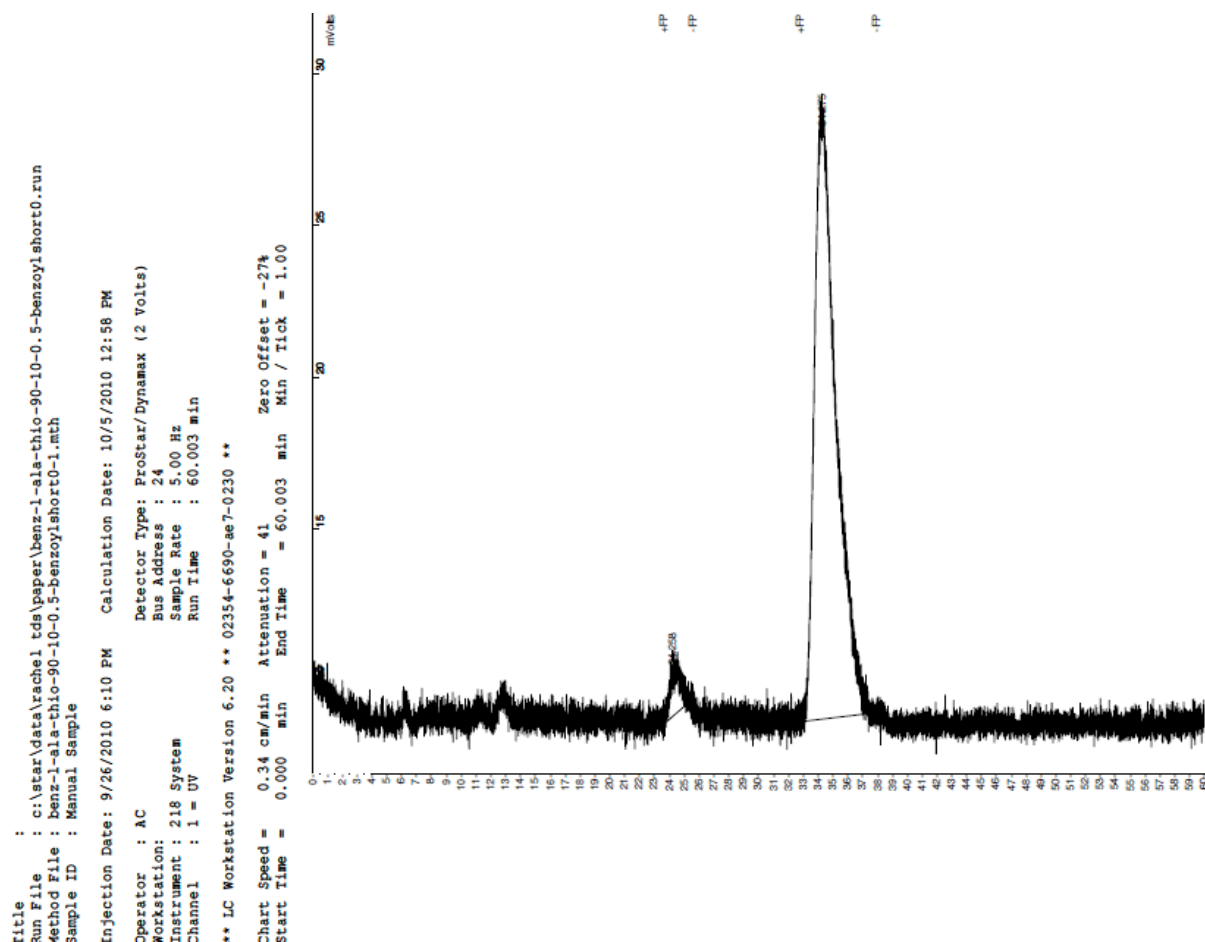
** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		8.2508	23.798	0.000	835734	BB	68.9	
2		91.7492	33.322	0.000	9293362	BB	96.1	
Totals:		100.0000		0.000	10129096			

Total Unidentified Counts : 10129096 counts

Benz-L-Alanine thiomorpholine amide 6l (directly from *N*-benzoyl alanine; 8 h reaction time)



Print Date: Tue Oct 05 12:59:51 2010

Page 1 of 1

Title :
 Run File : c:\star\data\rachel tds\paper\benz-l-ala-thio-90-10-0.5-benzoylshort0.run
 Method File : benz-l-ala-thio-90-10-0.5-benzoylshort0-1.mth
 Sample ID : Manual Sample

Injection Date: 9/26/2010 6:10 PM Calculation Date: 10/5/2010 12:58 PM

Operator : AC
 Workstation: ProStar/Dynamax (2 Volts)
 Instrument : 218 System
 Channel : 1 - UV
 Bus Address : 24
 Sample Rate : 5.00 Hz
 Run Time : 60.003 min

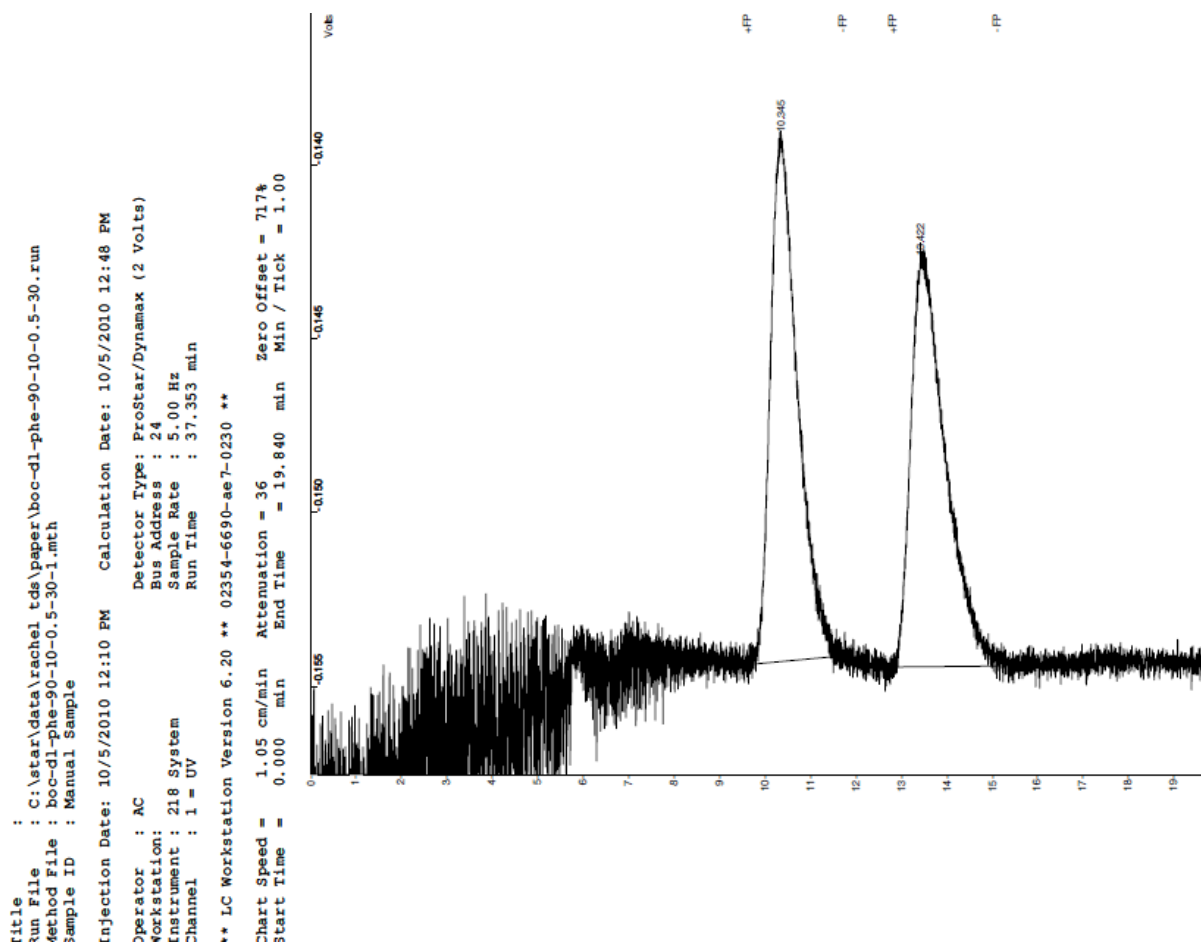
** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		3.7822	24.258	0.000	80261	BB	4.4	
2		96.2178	34.275	0.000	2041845	BB	85.3	
Totals:		100.0000		0.000	2122106			

Total Unidentified Counts : 2122106 counts

Boc-DL-Phenylalanine pyrrolidine amide



Print Date: Tue Oct 05 12:55:52 2010

Page 1 of 1

Title :
 Run File : C:\star\data\rachel tds\paper\boc-dl-phe-90-10-0.5-30.run
 Method File : boc-dl-phe-90-10-0.5-30-1.mth
 Sample ID : Manual Sample

Injection Date: 10/5/2010 12:10 PM Calculation Date: 10/5/2010 12:48 PM

Operator : AC Detector Type: ProStar/Dynamax (2 Volts)
 Workstation: Bus Address : 24
 Instrument : 218 System Sample Rate : 5.00 Hz
 Channel : 1 - UV Run Time : 37.353 min

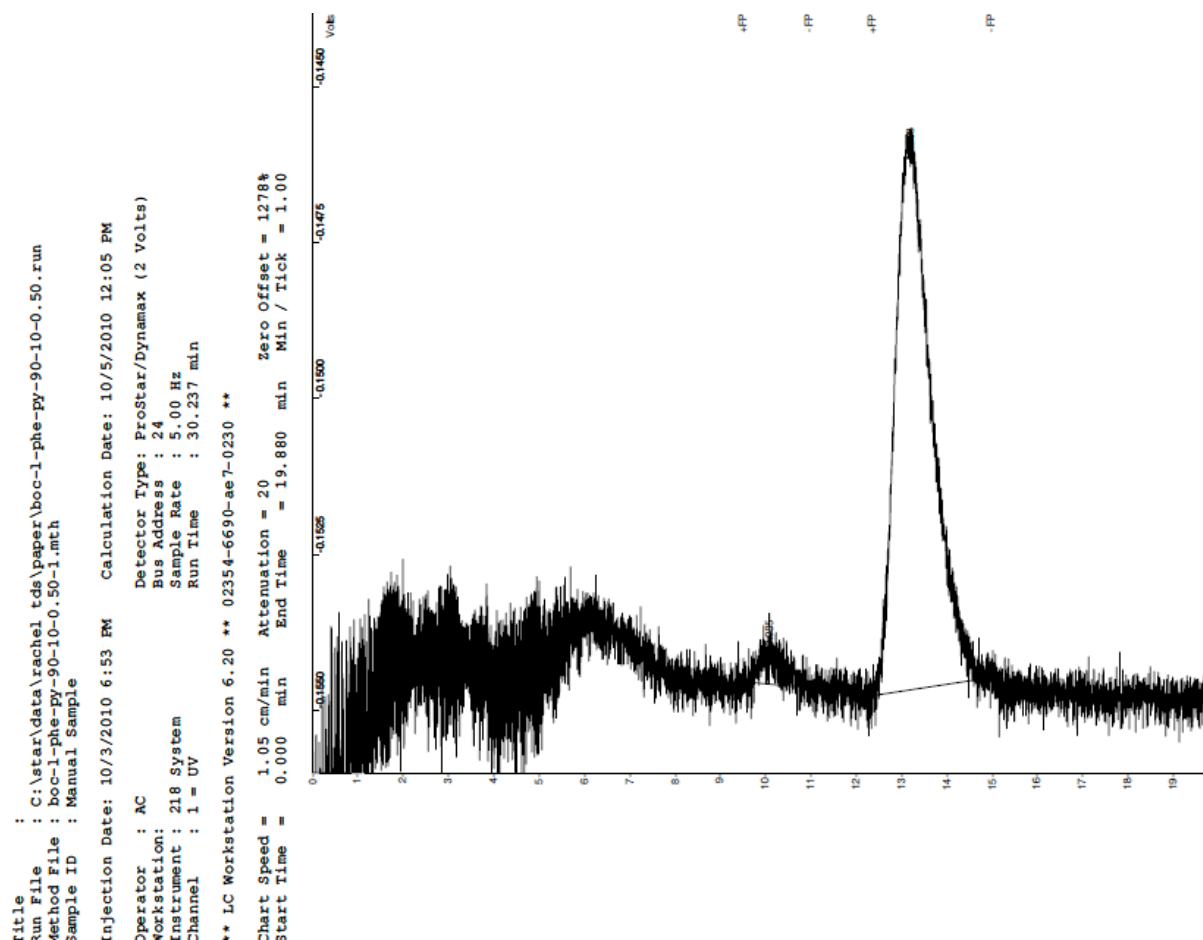
** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		50.1391	10.345	0.000	630998	BB	36.6	
2		49.8609	13.422	0.000	627496	BB	45.9	
Totals:		100.0000		0.000	1258494			

Total Unidentified Counts : 1258494 counts

Boc-L-Phenylalanine pyrrolidine amide 6j



Print Date: Tue Oct 05 12:56:49 2010

Page 1 of 1

Title :
 Run File : C:\star\data\rachel tds\paper\boc-l-phe-py-90-10-0.50.run
 Method File : boc-l-phe-py-90-10-0.50-1.mth
 Sample ID : Manual Sample

Injection Date: 10/3/2010 6:53 PM Calculation Date: 10/5/2010 12:05 PM

Operator : AC Detector Type: ProStar/Dynamax (2 Volts)
 Workstation: Bus Address : 24
 Instrument : 218 System Sample Rate : 5.00 Hz
 Channel : 1 - UV Run Time : 30.237 min

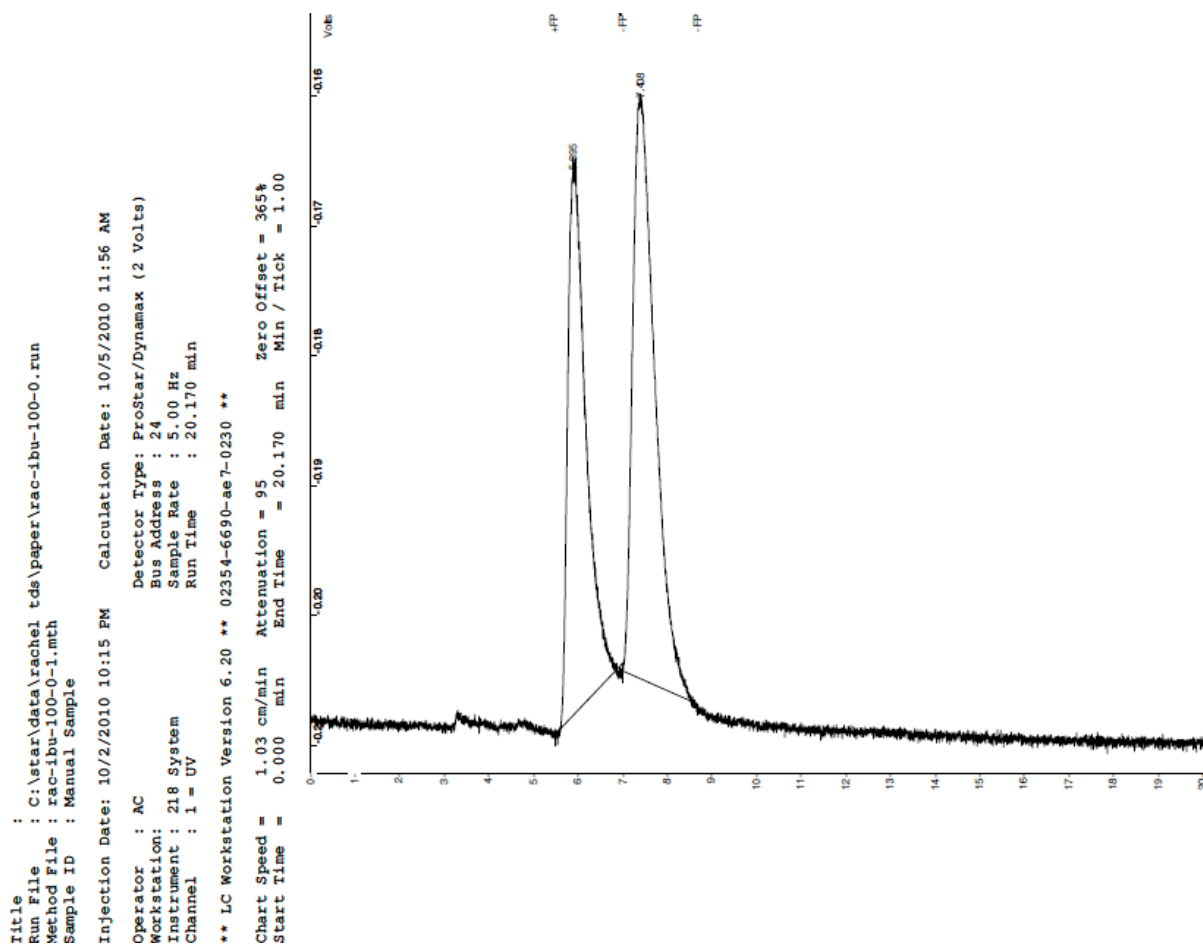
** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		3.7329	10.085	0.000	19136	BB	1.7	
2		96.2671	13.208	0.000	493486	BB	48.0	
Totals:		100.0000		0.000	512622			

Total Unidentified Counts : 512621 counts

(±)-N-Benzyl-2-(4-isobutylphenyl)propanamide



Print Date: Tue Oct 05 13:18:30 2010

Page 1 of 1

Title :
 Run File : C:\star\data\rachel tds\paper\rac-ibu-100-0.run
 Method File : rac-ibu-100-0-1.mth
 Sample ID : Manual Sample

Injection Date: 10/2/2010 10:15 PM Calculation Date: 10/5/2010 11:56 AM

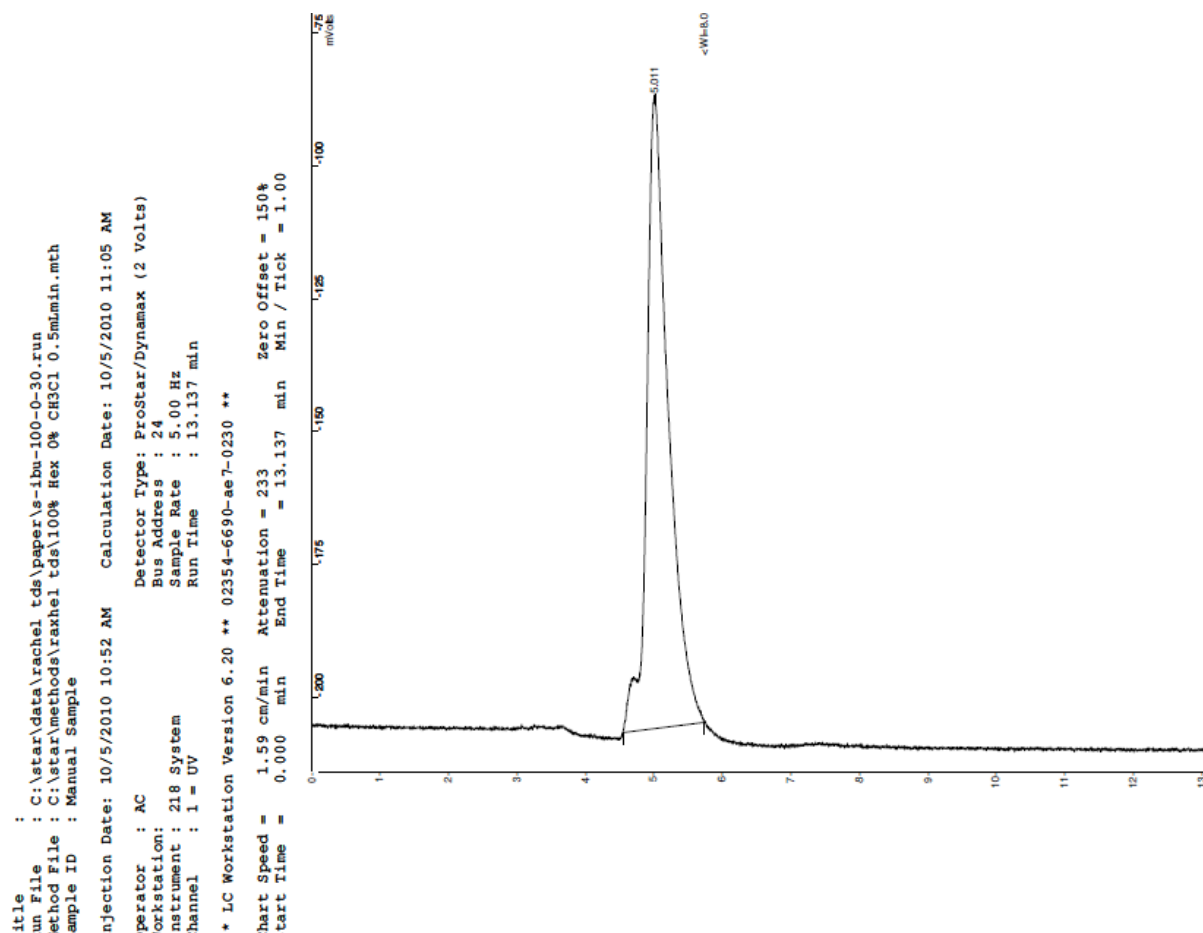
Operator : AC
 Workstation : 218 System
 Instrument : 1 - UV
 Detector Type: ProStar/Dynamax (2 Volts)
 Bus Address : 24
 Sample Rate : 5.00 Hz
 Run Time : 20.170 min

** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		43.5612	5.895	0.000	1194116	BB	26.2	
2		56.4388	7.408	0.000	1547124	BB	31.5	
Totals:		100.0000		0.000	2741240			

(S)-N-Benzyl-2-(4-isobutylphenyl)propanamide 6m



Print Date: Tue Oct 05 13:19:27 2010

Page 1 of 1

Title :
 Run File : C:\star\data\rachel tds\paper\s-ibu-100-0-30.run
 Method File : C:\star\methods\raxhel tds\100% Hex 0% CH3Cl 0.5mLmin.mth
 Sample ID : Manual Sample

Injection Date: 10/5/2010 10:52 AM Calculation Date: 10/5/2010 11:05 AM

Operator : AC Detector Type: ProStar/Dynamax (2 Volts)
 Workstation: Bus Address : 24
 Instrument : 218 System Sample Rate : 5.00 Hz
 Channel : 1 - UV Run Time : 13.137 min

** LC Workstation Version 6.20 ** 02354-6690-ae7-0230 **

Run Mode : Analysis
 Peak Measurement: Peak Area
 Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		100.0000	5.011	0.000	2624127	BB	19.0	
Totals:		100.0000		0.000	2624127			