



Supporting Information

for

Expanded use of benzamidines in the Groebke–Blackburn–Bienaymé synthesis of imidazoles and their biological evaluation

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Beilstein J. Org. Chem. **2026**, 22, 1344–1350. [doi:10.3762/bjoc.22.108](https://doi.org/10.3762/bjoc.22.108)

Docking results, VCP ATPase activity from a malachite green enzymatic assay, synthetic procedures for BITIs, and nuclear magnetic resonance spectroscopy data for 3b–h, 3j–l, and 3r

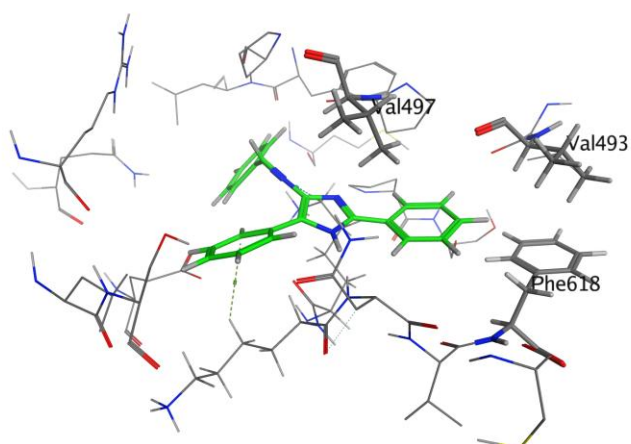


Figure S1: Molecular docking was performed using the cryo-EM structure of VCP (PDB ID: 5FTJ) in MOE. The top-ranked docking pose suggests that compound **1** (HBITI) occupies the reported allosteric binding and engages residues Val493, Val 497, Phe618 which are key residues for allosteric binding of VCP inhibitors. Protein residues are shown as sticks and the ligand is shown in green.

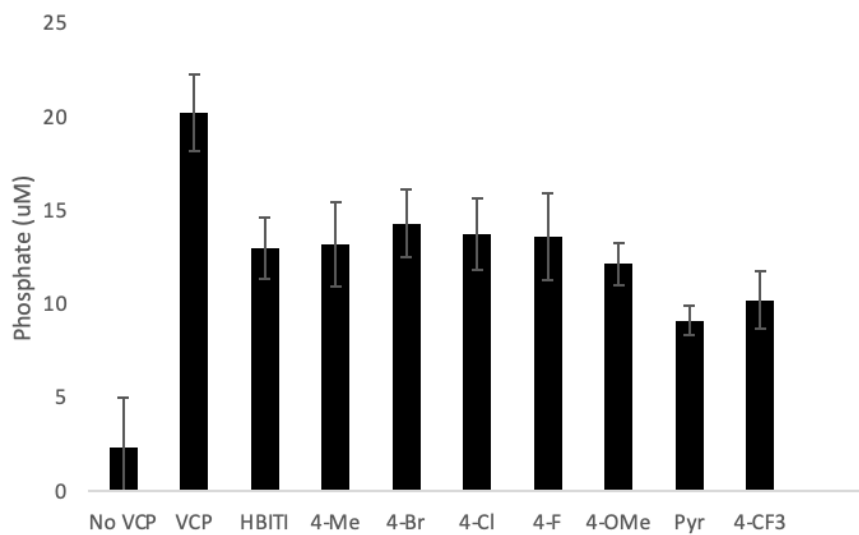


Figure S2: Effect of compounds (100 micromolar) on VCP ATPase activity using a malachite green enzymatic assay.

General

All reagents and solvents were purchased from commercial suppliers and used as received unless noted otherwise. Flash column chromatography separations were done on a Biotage Select purification system, monitoring at 254 and 280 nm. NMR spectra were recorded on a Bruker Advance spectrometer at 25 °C, operating at 500 MHz for ^1H and 126 MHz for ^{13}C NMR. ^1H NMR and ^{13}C NMR data are reported as follows: chemical shift in ppm (δ), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet). Coupling constants are measured in hertz (Hz). The chemical shifts are expressed in ppm downfield from TMS as an internal standard. High-resolution mass spectra (HRMS) were recorded using electrospray ionization (ESI) in positive mode on a JEOL AccuTOF-CS spectrometer, reported as m/z values for $[\text{M} + \text{H}]^+$. Reactions were monitored by thin-layer chromatography (TLC) on silica gel aluminum slides. All microwave experiments were conducted in an Anton-Paar Monowave 450. The reactions were carried out in a 30 mL glass vial with a septum. The reaction was irradiated at the prescribed temperature for the stipulated time and then cooled to 55 °C.

General procedure for synthesis of 2,4-benzyl-5-imino tricyclic imidazoles (BITIs)

In the reaction vessel, benzamidine hydrochloric acid (0.05 g, 1 equiv, 0.5 mmol) and sodium sulfate (0.3 g, 4 equiv, 2 mmol) were suspended in anhydrous DMF (2.5 mL). Benzaldehyde (0.4 g, 0.4 mL, 8 equiv, 4 mmol), and trimethylsilanecarbonitrile (0.1 g, 0.1 mL, 2 equiv, 1 mmol) were added and the solution was microwave irradiated at 220 °C for 20 min. The reaction mixture was extracted with water and ethyl acetate to remove the DMF. The solvent was removed and the crude product was purified by column chromatography (Hex/EtOAc).

(E)-N-Benzylidene-4-phenyl-2-(p-tolyl)-1H-imidazol-5-amine (3b) A light yellow powder was obtained (52 mg, 37%). R_f 0.88 (silica gel, 5:1 hexane/EtOAc) ^1H NMR (500 MHz, CDCl_3) δ 9.37 (s, 1H), 9.32 (s, 1H), 7.96-7.99 (m, 4H), 7.80 (d, J = 8.2 Hz, 2H), 7.42-7.48 (m, 5H), 7.31 (t, J = 3.2 Hz, 1H), 7.27 (d, J = 7.9 Hz, 2H), 2.40 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 156.5, 144.6, 19.3, 137.1, 130.5, 129.7, 128.7, 128.6, 127.2, 127.0, 126.4, 125.2, 21.4 $[\text{M} + \text{H}]^+$ (ESI): calc'd for $\text{C}_{23}\text{H}_{19}\text{N}_3$, 338.426; found 338.164

(E)-N-Benzylidene-2-(4-bromophenyl)-4-phenyl-1H-imidazol-5-amine (3c) A dark, brown powder was obtained (81 mg, 50%). R_f 0.8 (silica gel, 5:1 hexane/EtOAc) ^1H NMR (500 MHz, CDCl_3) δ 9.39 (s, 1H), 9.31 (s, 1H), 7.96-7.98 (m, 4H), 7.79 (d, J = 8.6 Hz, 2H), 7.60 (d, J = 8.6 Hz, 2H), 7.44-7.50 (m, 5H), 7.33 (t, J = 7.4 Hz, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 157.0, 143.2, 137.0, 132.2, 130.7, 128.9, 128.7, 128.6, 127.4, 126.7, 126.5, 123.3 $[\text{M} + \text{H}]^+$ (ESI): calc'd for, $\text{C}_{22}\text{H}_{16}\text{N}_3\text{Br}$, 402.295, 404.051; found 402.059, 404.060

(E)-N-Benzylidene-2-(4-chlorophenyl)-4-phenyl-1H-imidazol-5-amine (3d) A light yellow powder was obtained (50 mg, 35%). R_f 0.55 (silica gel, 5:1 hexane/EtOAc) ^1H NMR (500 MHz, CDCl_3) δ 9.41 (s, 1H), 9.31 (s, 1H), 7.96-7.98 (m, 4H), 7.85 (d, J = 8.6 Hz, 2H), 7.47 (m, 8H), 7.33 (t, J = 7.4 Hz, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 156.9, 145.5, 143.2, 137.0, 135.0, 130.6, 129.2, 128.8, 128.7, 128.6, 128.2, 126.5 $[\text{M} + \text{H}]^+$ (ESI): calc'd for, $\text{C}_{22}\text{H}_{18}\text{N}_3\text{Cl}$, 358.100; 360.001 found 358.111; 360.111

(E)-N-Benzylidene-2-(4-fluorophenyl)-4-phenyl-1H-imidazol-5-amine (3e) A tan, brown powder was obtained (57 mg, 43%). R_f 0.63 (silica gel, 5:1 hexane/EtOAc) ^1H NMR (500 MHz, CDCl_3) δ 9.43 (s, 1H), 9.31 (s, 1H), 7.95-7.98 (m, 4H), 7.86-7.89 (m, 2H), 7.43-7.49 (m, 5H), 7.32 (t, J = 7.4 Hz, 1H), 7.15 (t, J = 8.6 Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 116.0, 116.2, 124.9, 126.1, 126.2, 126.4, 127.2, 127.3, 128.6, 128.7, 128.8, 129.6, 130.6, 137.0, 143.6, 145.4, 156.8, 162.3, 164.3 $[\text{M}+\text{H}]^+$ (ESI): calc'd for, $\text{C}_{22}\text{H}_{16}\text{N}_3\text{F}$, 342.389; found 342.141

(E)-N-Benzylidene-2-(4-methoxyphenyl)-4-phenyl-1H-imidazol-5-amine (3f) A tan, brown powder was obtained (80 mg, 70%). R_f 0.61 (silica gel, 5:1 hexanes/EtOAc) ^1H NMR (500 MHz, CDCl_3) δ 9.87 (s, 1H), 9.30 (s, 1H), 8.01 (d, J = 5.5 Hz, 2H), 7.95 (d, J = 6.7 Hz, 2H), 7.86 (d, J = 8.8 Hz, 2H), 7.41-7.46 (m, 5H), 7.27-7.30 (m, 2H), 6.95 (d, J = 8.8 Hz, 2H), 3.83 (s, 3H) ^{13}C NMR (126 MHz, CDCl_3) δ 162.7, 160.4, 156.3, 144.8, 137.3, 130.4, 128.7, 128.6, 126.5, 122.7, 114.3, 55.4 $[\text{M}+\text{H}]^+$ (ESI): calc'd for $\text{C}_{23}\text{H}_{19}\text{N}_3\text{O}$, 354.425; found 354.161 [1]

(E)-N-Benzylidene-4-phenyl-2-(pyridin-4-yl)-1H-imidazol-5-amine (3g) A tan, brown powder was obtained (70 mg, 27%). R_f 0.57 (silica gel, 6:1 hexane/EtOAc) ^1H NMR (500 MHz, CDCl_3) δ 9.74 (s, 1H), 9.35 (s, 1H), 8.72-8.73 (m, 2H), 8.00-8.01 (m, 4H), 7.81-7.82 (m, 2H), 7.47-7.53 (m, 6H), 7.37 (t, J = 7.4 Hz, 1H) ^{13}C NMR (126 MHz, CDCl_3) δ 119.1, 119.2, 126.8, 127.3, 127.7, 128.7, 128.8, 130.9, 136.9, 137.0, 141.4, 150.3 $[\text{M}+\text{H}]^+$ (ESI): calc'd for $\text{C}_{21}\text{H}_{16}\text{N}_4$, 324.38; found 325.146

(E)-N-Benzylidene-4-phenyl-2-(4-(trifluoromethyl)phenyl)-1H-imidazol-5-amine (3h) A light yellow powder was obtained (20 mg, 12%). R_f 0.67 (silica gel, 5:1 hexane/EtOAc) ^1H NMR (500 MHz, CDCl_3) δ 9.55 (s, 1H), 9.27 (s, 1H), 7.98 (d, J = 8.1 Hz, 2H), 7.91-7.94 (m, 4H), 7.66 (d, J = 8.2 Hz, 2H), 7.39-7.44 (m, 5H), 7.28 (t, J = 7.4 Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 125.4, 126.0, 126.5, 127.6, 128.6, 128.8, 128.9, 130.7, 142.6, 145.8, 147.3 $[\text{M}+\text{H}]^+$ (ESI): calc'd for, $\text{C}_{23}\text{H}_{18}\text{N}_3\text{F}_3$, 392.130; found 392.138

4-(E)-N-(4-(tert-Butyl)benzylidene)-4-(4-(tert-butyl)phenyl)-2-(4-methoxyphenyl)-1H-imidazol-5-amine (3j) A dark brown powder was obtained (132 mg, 70%). R_f 0.56 (silica gel, 5:1 hexanes/EtOAc) ^1H NMR (500 MHz, CDCl_3) δ 9.29 (s, 1H), 9.19 (s, 1H), 7.92 (dd, J = 4.9, 8.3 Hz, 4H), 7.85 (d, J = 8.8 Hz, 2H), 7.49 (dd, J = 5.6, 8.3 Hz, 4H), 7.00 (d, J = 8.8 Hz, 2H), 3.87 (s, 3H), 1.37 (s, 9H), 1.36 (s, 9H) ^{13}C NMR (126 MHz, CDCl_3) δ 31.2, 31.3, 34.6, 34.9, 55.4, 114.4, 122.7, 124.2, 125.5, 125.7, 126.0, 126.7, 128.4, 134.4, 144.1, 145.2, 150.1, 153.7, 155.9, 160.4 $[\text{M}+\text{H}]^+$ (ESI): calc'd for $\text{C}_{31}\text{H}_{35}\text{N}_3\text{O}$, 466.641; found 466.287

(E)-1-(4-(tert-Butyl)phenyl)-N-(4-(4-(tert-butyl)phenyl)-2-(4-chlorophenyl)-1H-imidazol-5-yl)methanimine (3k) A yellow, crystalline solid was obtained (86 mg, 36%). R_f 0.35 (silica gel, 9:1 hexanes/EtOAc). ^1H NMR (500 MHz, CDCl_3) δ 9.20 (br, 1H), 7.92 (d, J = 8.1 Hz, 2H), 7.84 (d, J = 8.1 Hz, 2H), 7.77 (d, J = 8.2 Hz, 2H), 7.43-7.46 (m, 4H), 7.30 (d, J = 7.8 Hz, 2H), 1.34 (s, 18H). ^{13}C NMR (126 MHz, CDCl_3) δ 31.22, 31.27, 34.61, 34.93, 125.5, 125.6, 126.5, 126.7, 128.4, 128.9, 134.5, 134.6, 143.2, 150.1, 153.9 $[\text{M}+\text{H}]^+$ (ESI): calc'd for $\text{C}_{30}\text{H}_{32}\text{ClN}_3$, 470.228, 472.232; found 470.235, 472.238

(E)-1-(4-(tert-Butyl)phenyl)-N-(4-(4-(tert-butyl)phenyl)-2-(4-(trifluoromethyl)phenyl)-1H-imidazol-5-yl)methanimine (3l) A yellow-green, crystalline solid was obtained (41 mg, 19%). R_f 0.42 (silica gel, 9:1 hexanes/EtOAc). ^1H NMR (500 MHz, CDCl_3) δ 9.24 (s, 1H), 8.05 (d, J = 8.0 Hz, 2H), 7.94 (d, J = 8.2 Hz, 2H), 7.89 (d, J = 8.1 Hz, 2H), 7.62 (d, J = 8.1 Hz, 2H), 7.45-7.49 (m, 4H), 1.36 (s, 9H), 1.35 (s, 9H) ^{13}C NMR (126 MHz, CDCl_3) δ 31.30, 34.67, 3.98, 125.5, 125.6, 125.8, 126.5, 128.5, 133.3, 134.5, 142.6, 150.4, 154.0, 156.4 $[\text{M}+\text{H}]^+$ (ESI): calc'd for $\text{C}_{31}\text{H}_{32}\text{F}_3\text{N}_3$, 504.255; found 504.263

(E)-1-(3,4-Dimethylphenyl)-N-(4-(3,4-dimethylphenyl)-2-phenyl-1H-imidazol-5-yl)methanimine (3r) A dark brown powder was obtained (44 mg, 59%). *R*_f 0.56 (silica gel, 5:1 hexanes/EtOAc) ¹H NMR (500 MHz, CDCl₃) δ 10.35 (s, 1H), 9.07 (s, 1H), 7.68 (d, *J* = 7.4 Hz, 3H), 7.58-7.59 (m, 1H), 7.44-7.48 (m, 2H), 7.10-7.17 (m, 4H), 7.01-7.03 (m, 2H), 2.14 (s, 12H). ¹³C NMR (126 MHz, CDCl₃) δ 19.62, 19.85, 19.99, 20.02, 124.1, 125.5, 126.3, 127.5, 128.0, 129.6, 129.9, 135.1, 135.6, 136.7, 139.5, 144.3, 145.2, 156.2, 162.8 [M+H]⁺(ESI): calc'd for C₂₆H₂₅N₃, 380.507 ; found 380.211

References

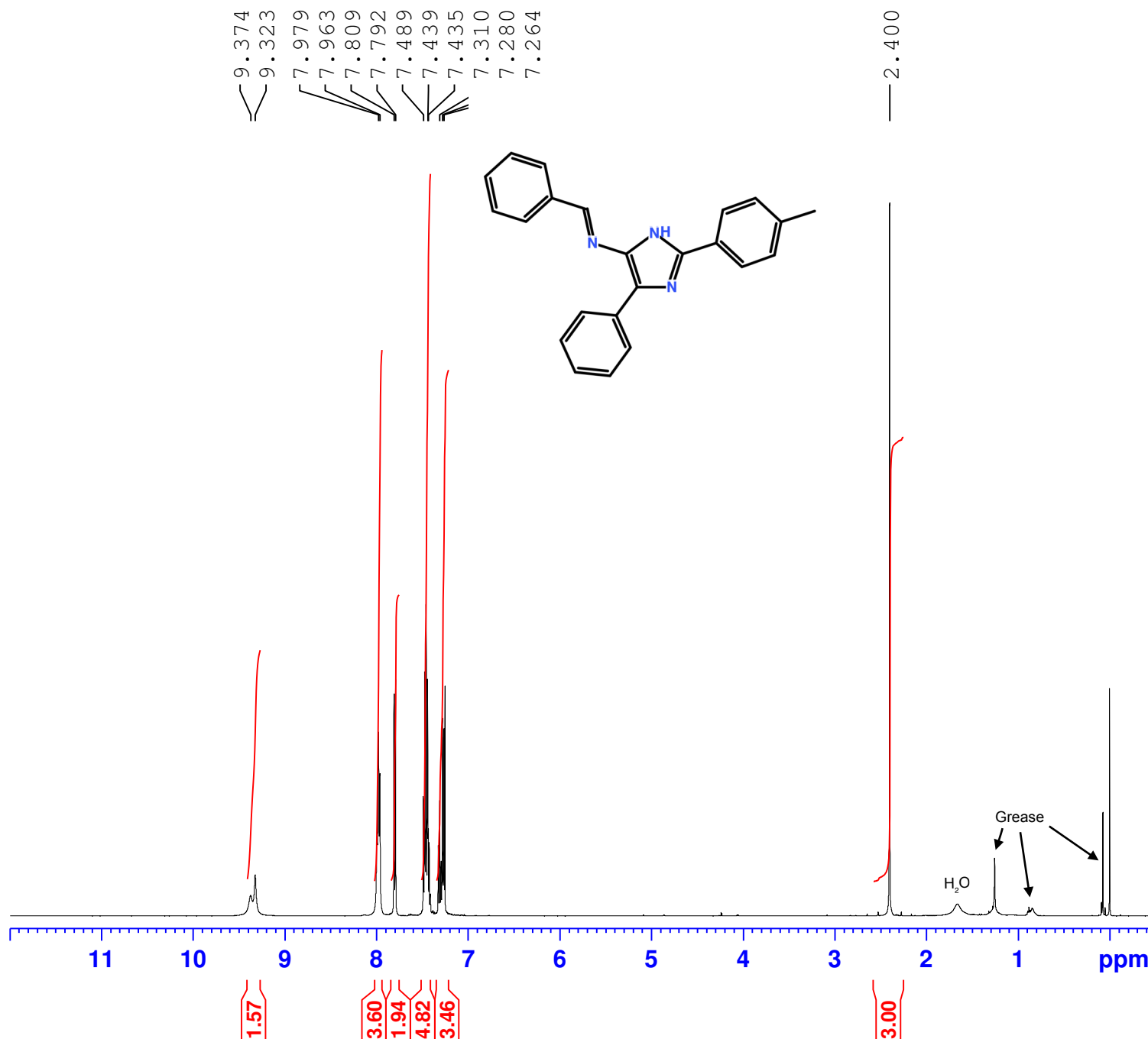
1. Bell, C. E.; Shaw, A. Y.; De Moliner, F.; Hulme, C. *Tetrahedron* **2014**, 70, 54–59.



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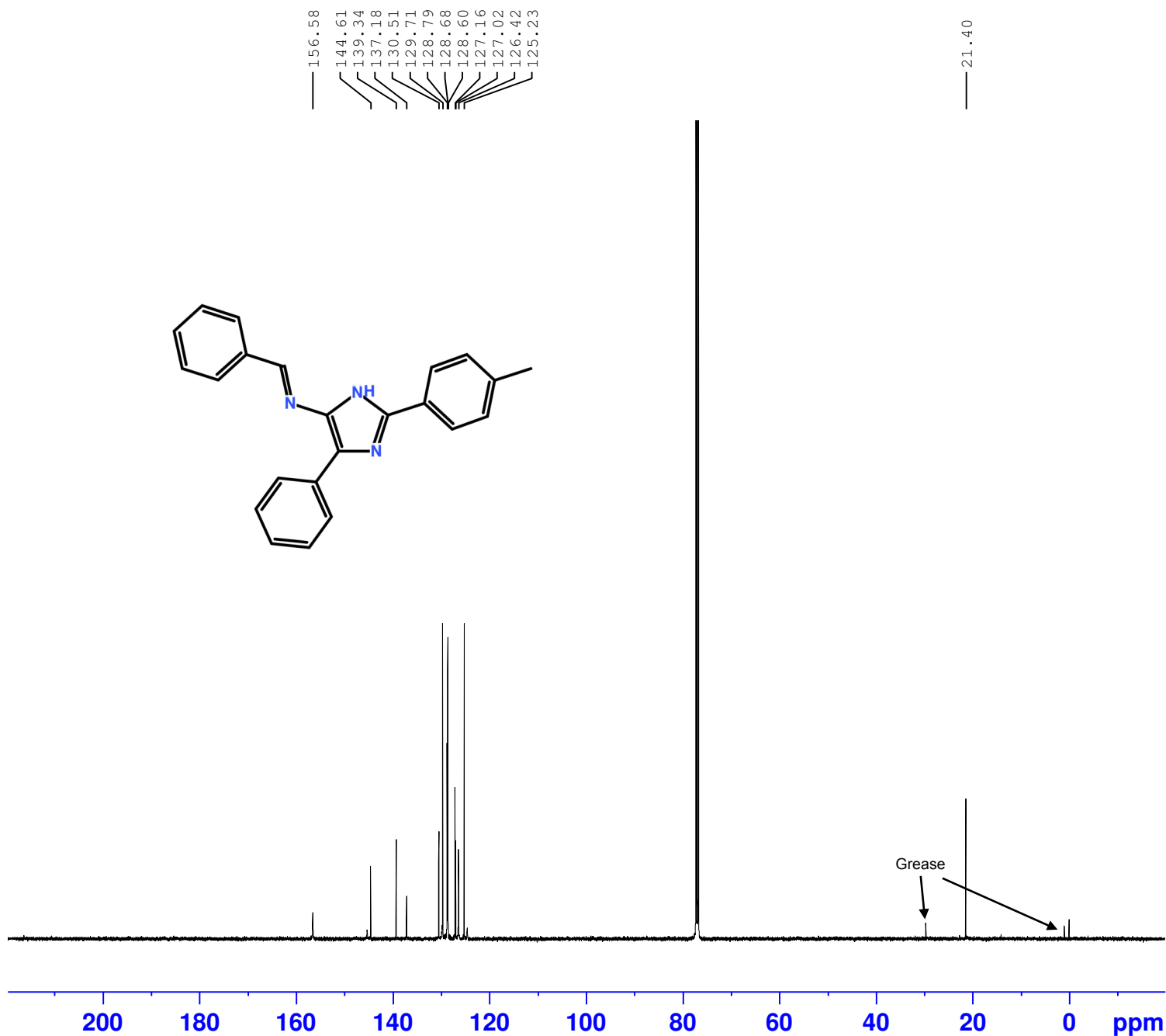




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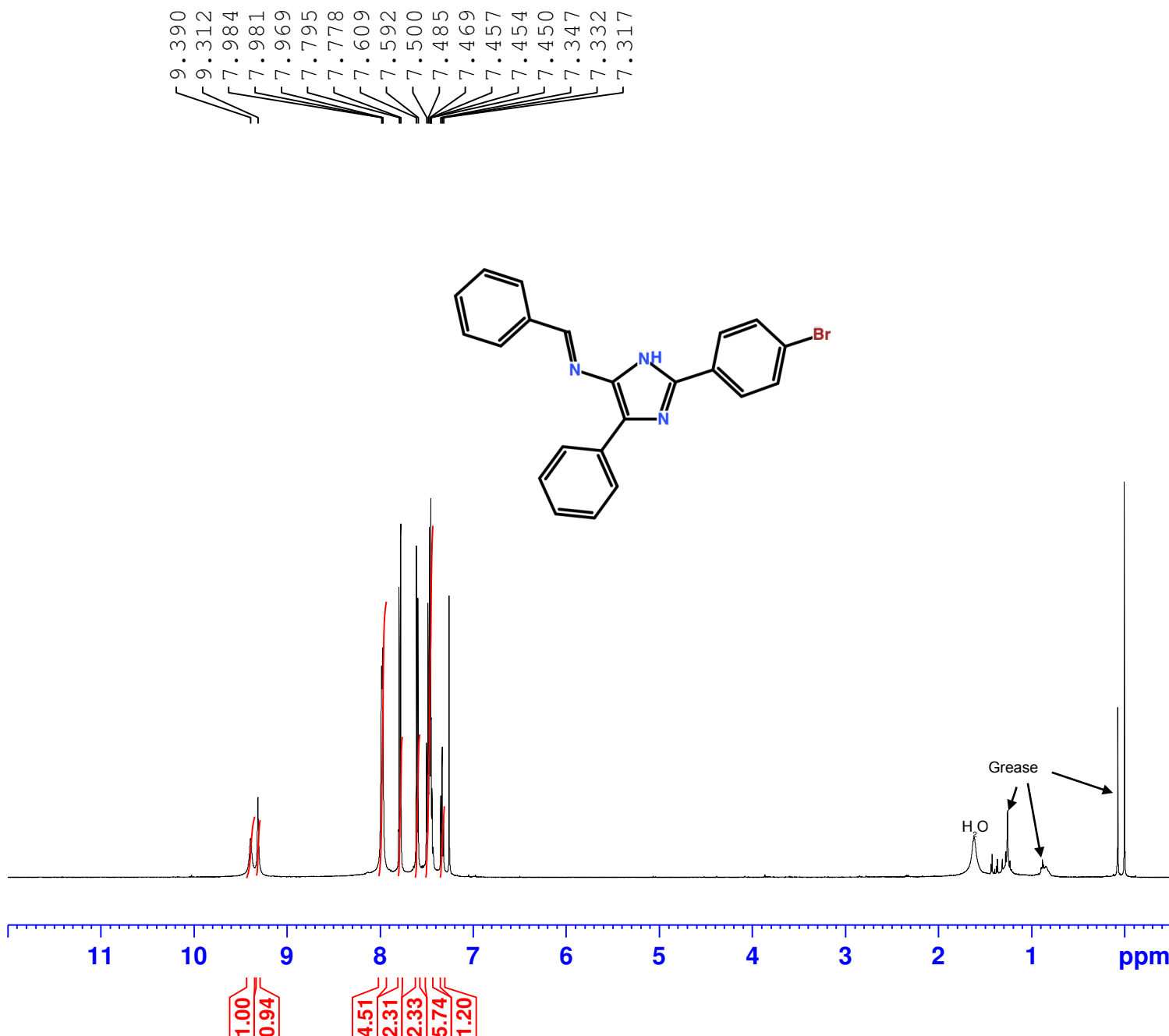


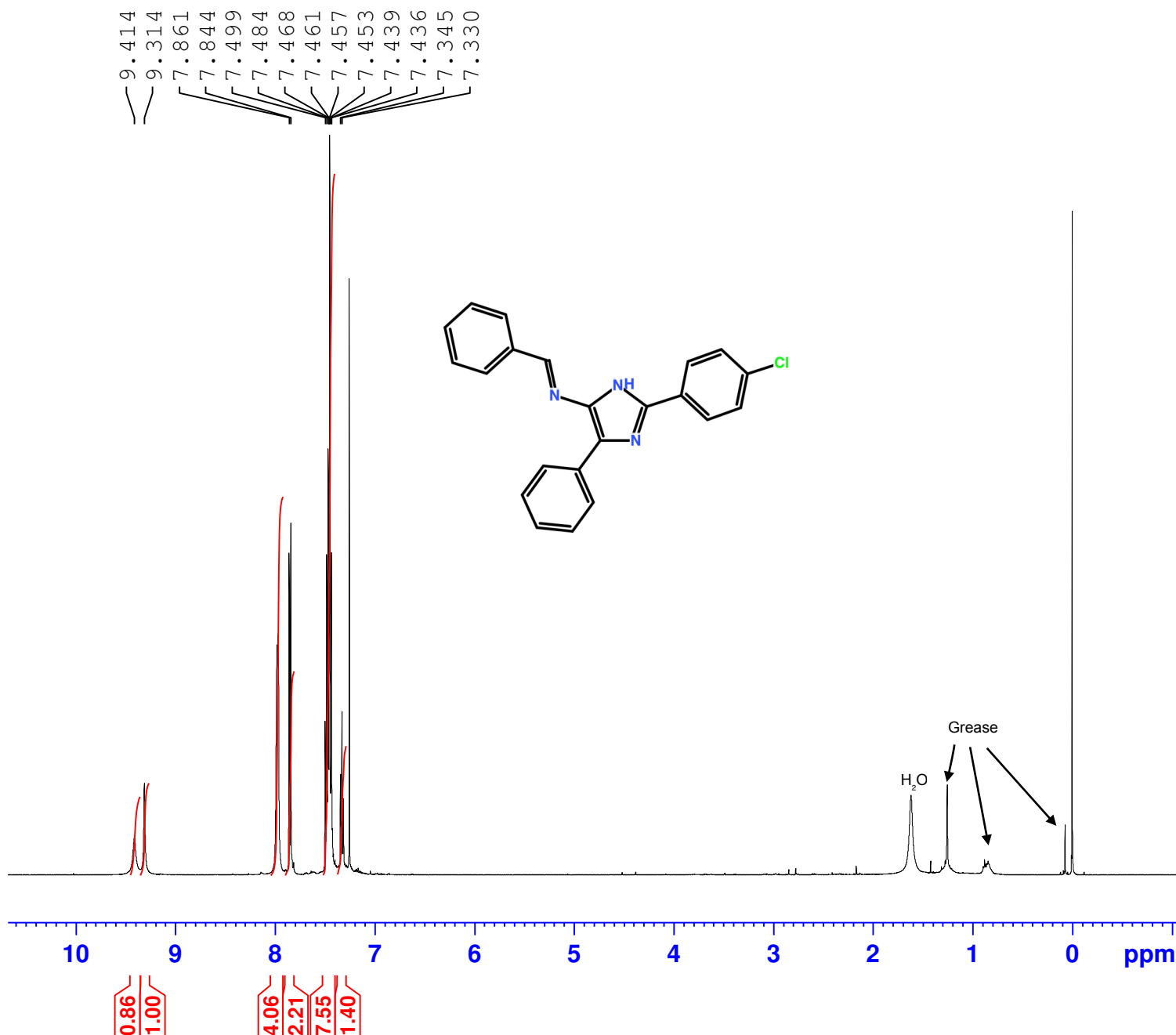


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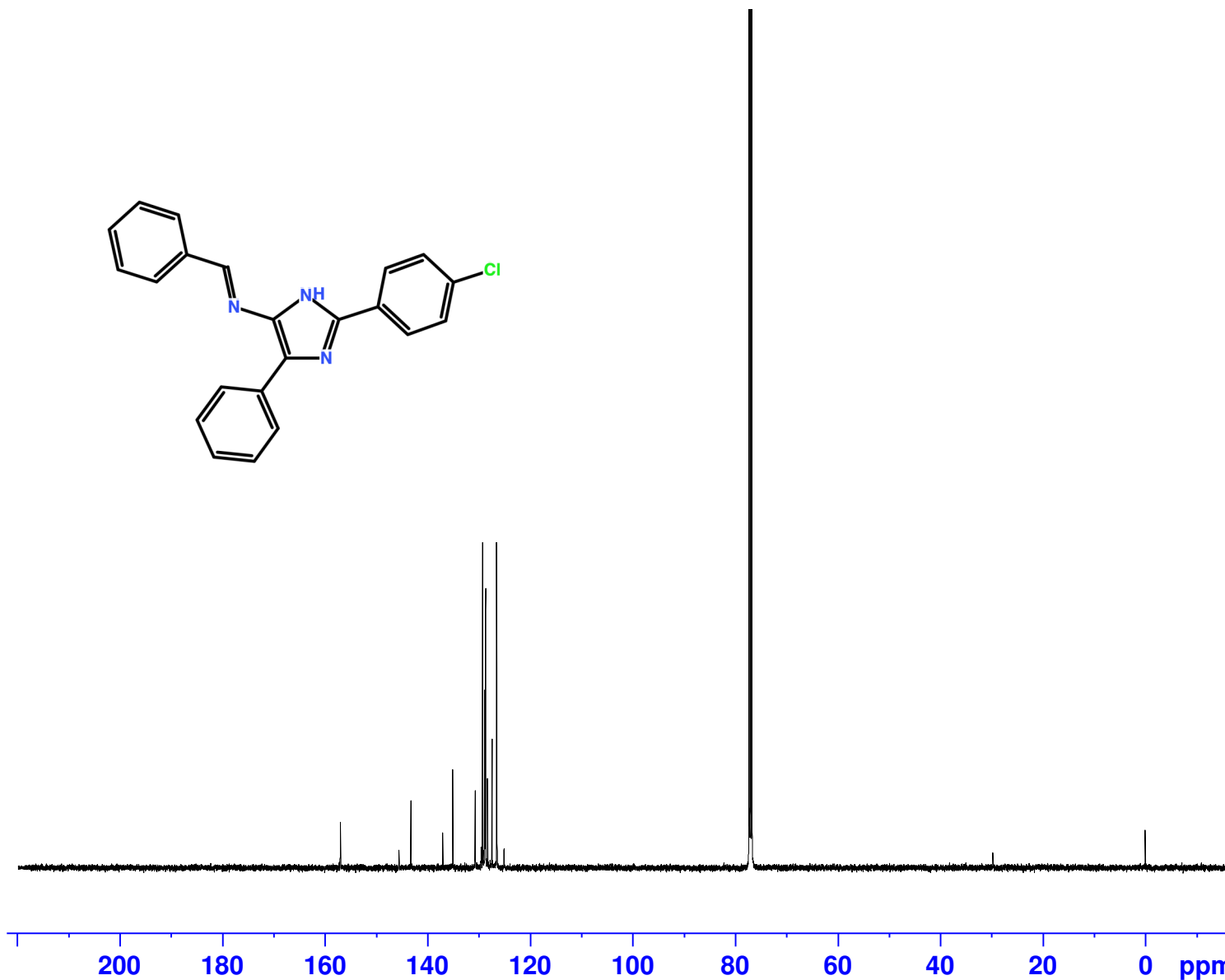
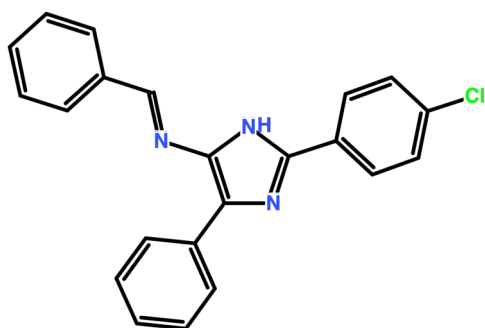


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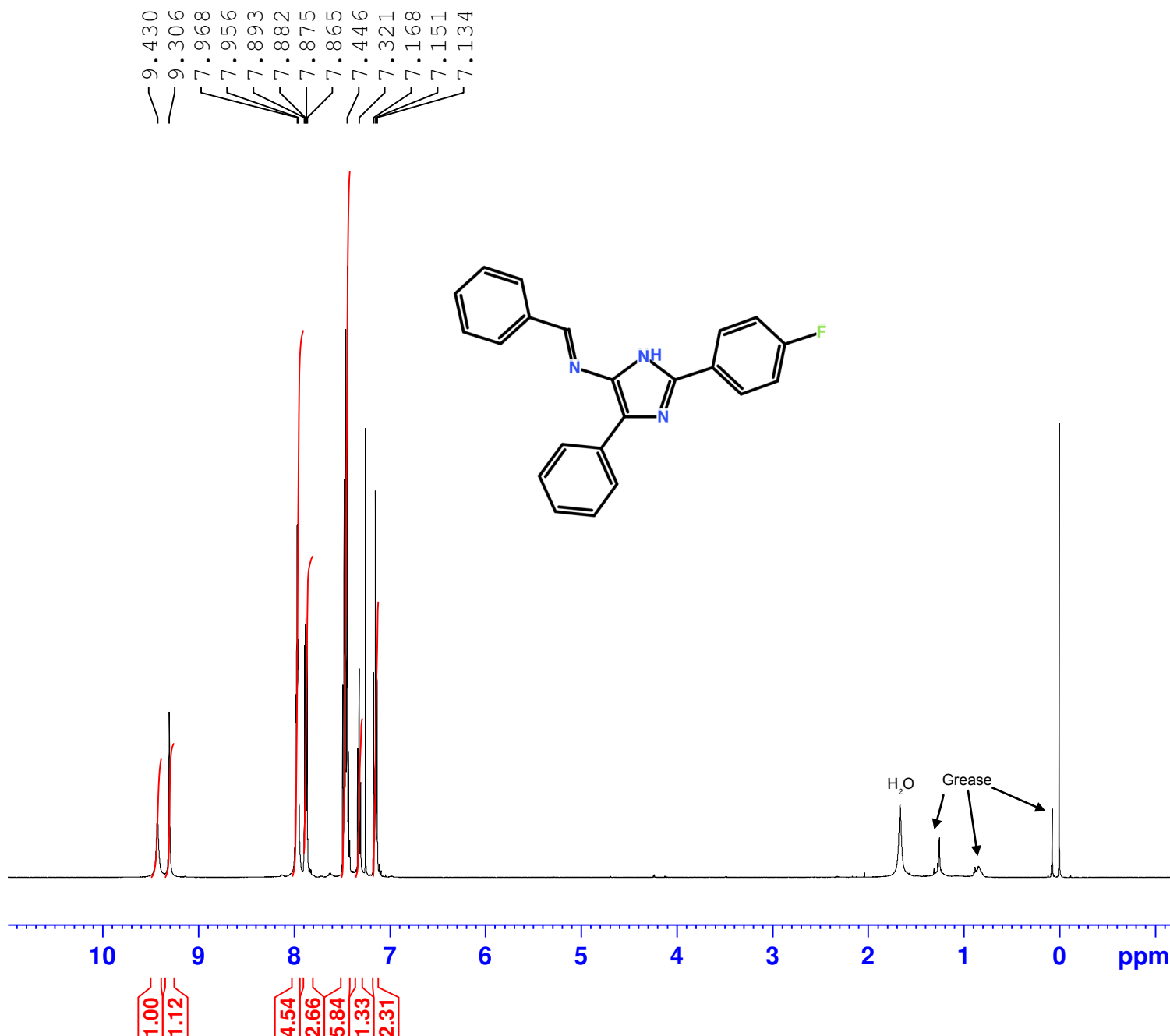




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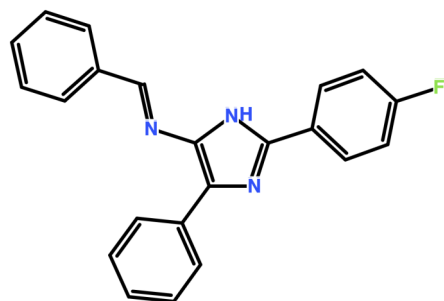




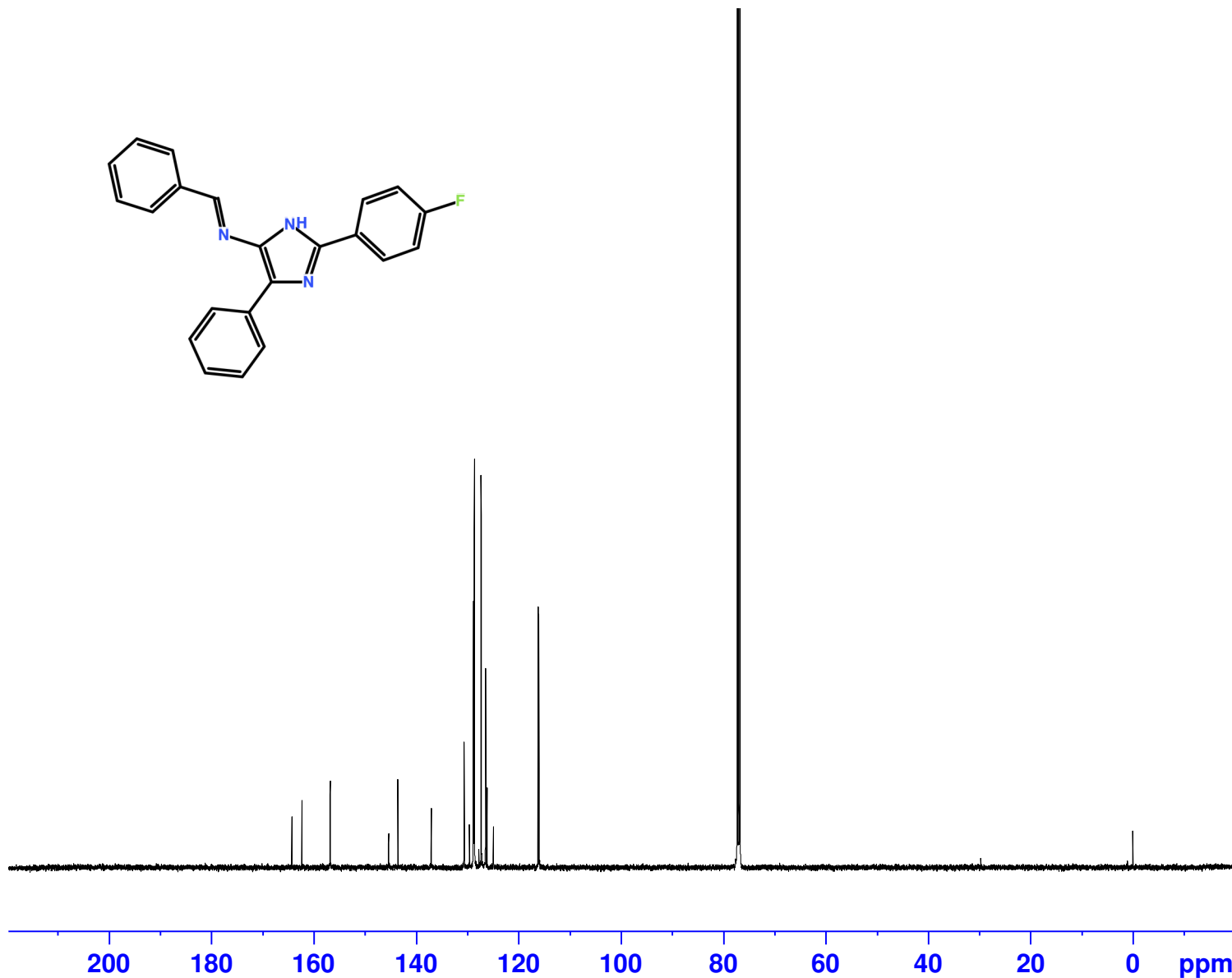
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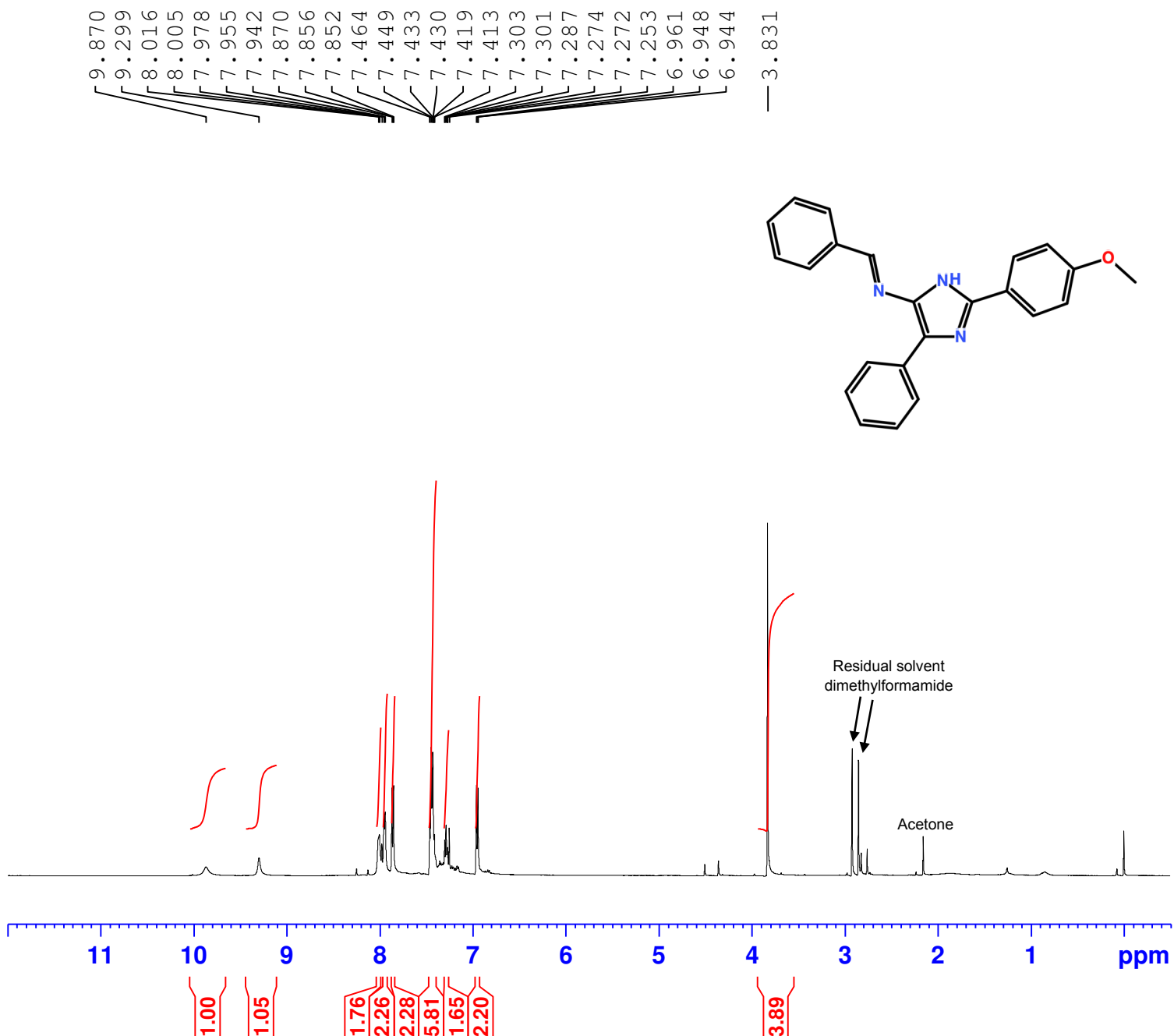
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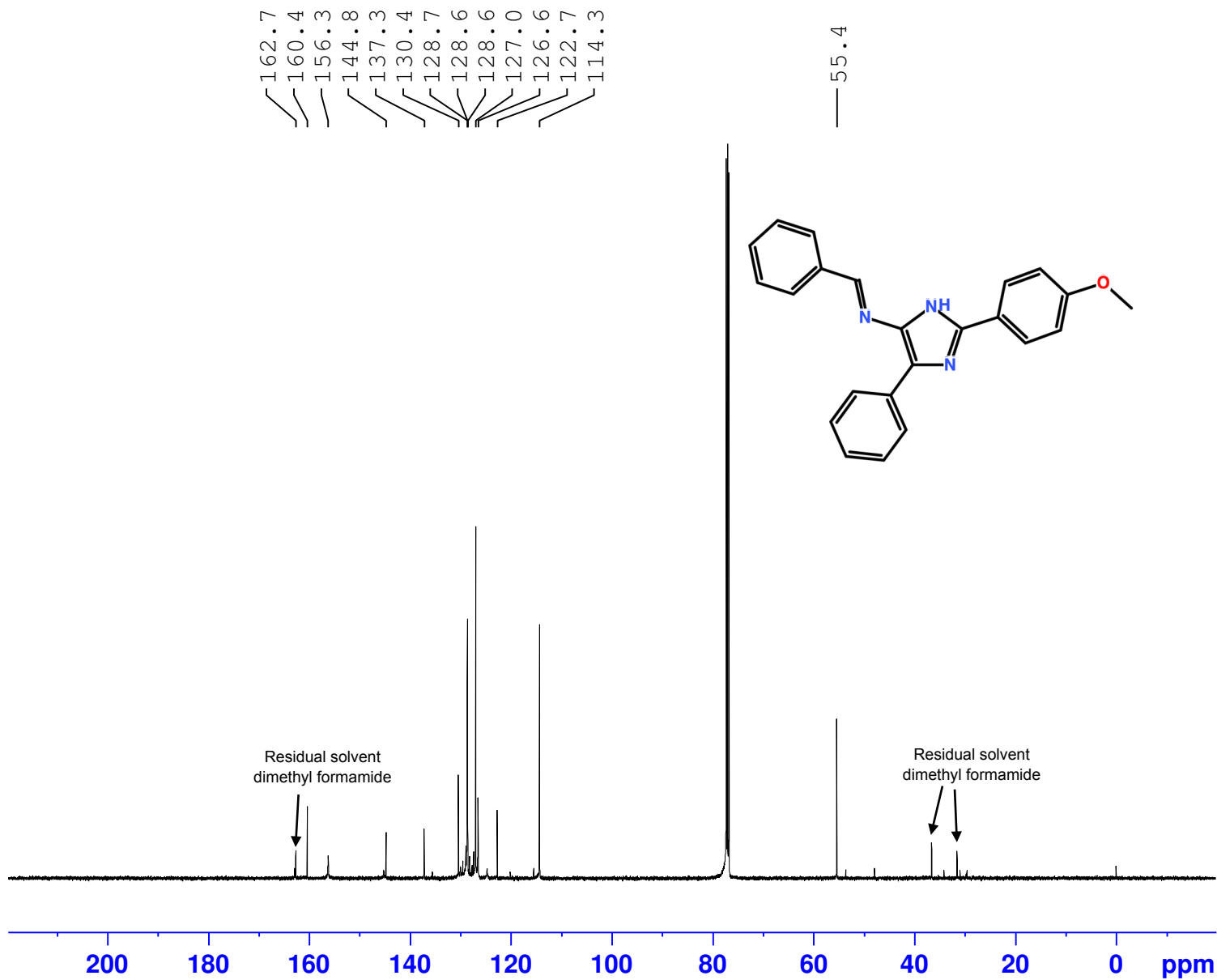




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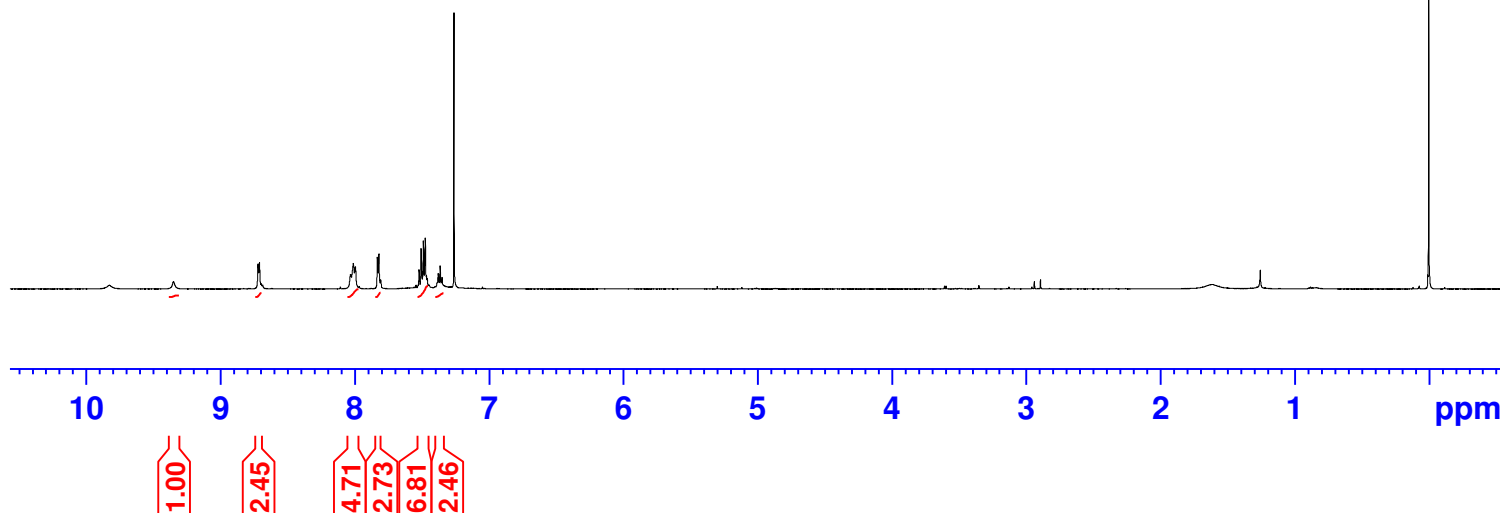
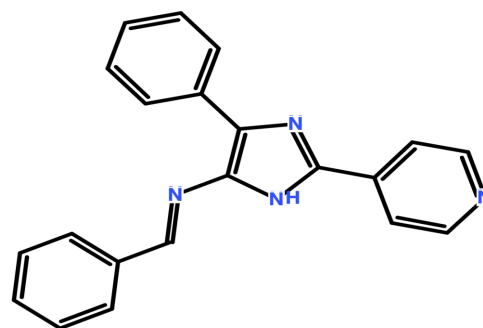
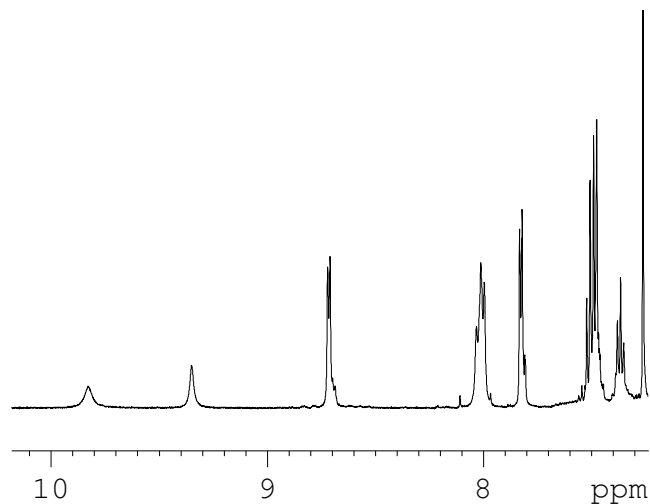
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7.466
7.462
7.458
7.443
7.401
7.386
7.378
7.363
7.348
7.330
7.280
7.277

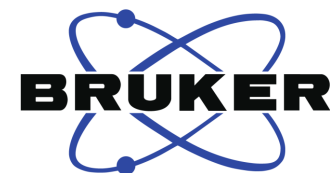
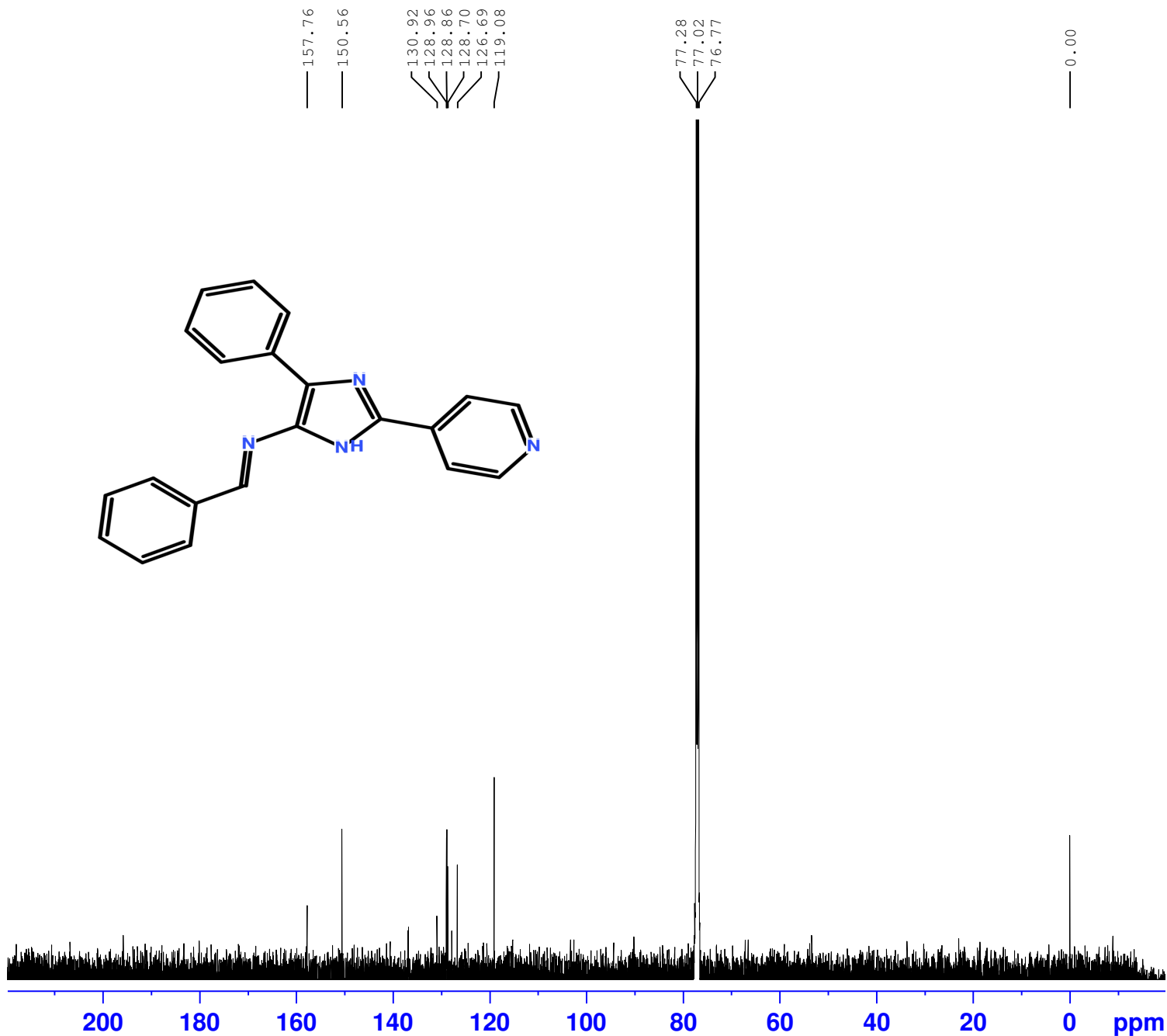


Current Data Parameters
NAME HT-GBB-PIMI-6-2-26-GroupB
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20260605
Time 10.16
INSTRUM Avance
PROBHD 2173362_0026 (PI HR-TBO500S1-BBF/H/F/D-5.0-2 FB)
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2768000 sec
RG 101
DW 50.000 usec
DE 10.79 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
FO 3.33 usec
P1 10.00 usec
PLW1 22.34400000 W

F2 - Processing parameters
SI 65536
SF 500.1300130 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





Current Data Parameters

NAME	KJW-GBB-4-17-25
EXPNO	3
PROCNO	1

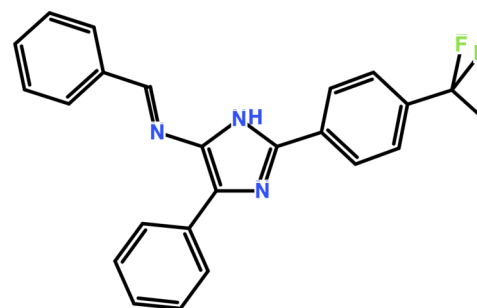
F2 - Acquisition Parameters

Date_	20250421
Time	20.21
INSTRUM	Avance
PROBHD	2173362_0026 (PI BB-TBO500S1-BBF/H/F/D-5.0-2 FB)
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	3072
DS	4
SWH	30120.482 Hz
FIDRES	0.919204 Hz
AQ	1.0878976 sec
RG	101
DW	16.600 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	125.7703643 MHz
NUC1	13C
P0	3.33 usec
P1	10.00 usec
PLW1	81.01100000 W
SFO2	500.1320005 MHz
NUC2	1H
CPDPRG2	waltz65
PCPD2	80.00 usec
PLW2	22.34400000 W
PLW12	0.34912000 W
PLW13	0.17561000 W

F2 - Processing parameters

SI	32768
SF	125.7577885 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

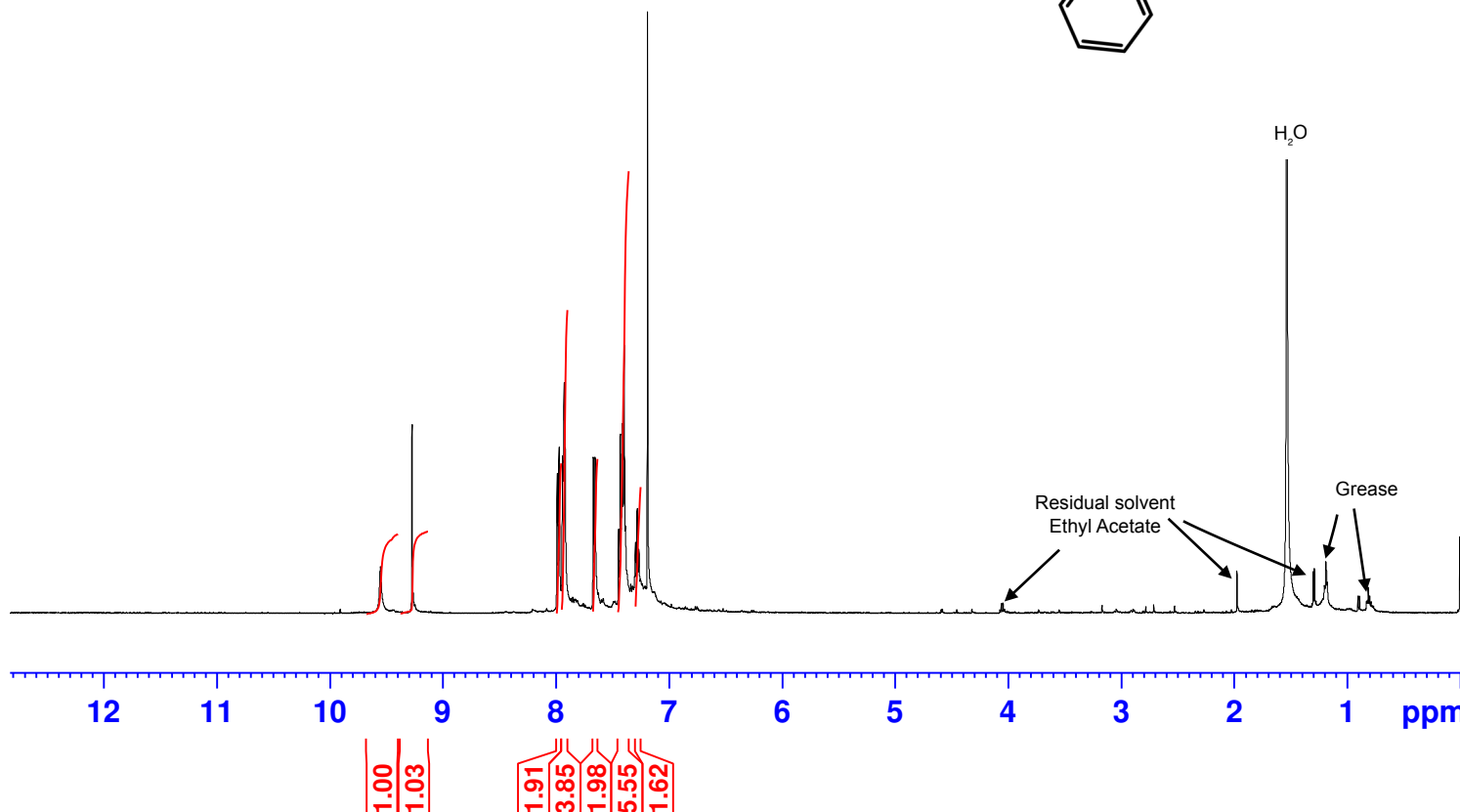
9.553
9.273
7.987
7.971
7.941
7.935
7.926
7.920
7.668
7.652
7.445
7.430
7.414
7.410
7.395
7.295
7.280
7.266

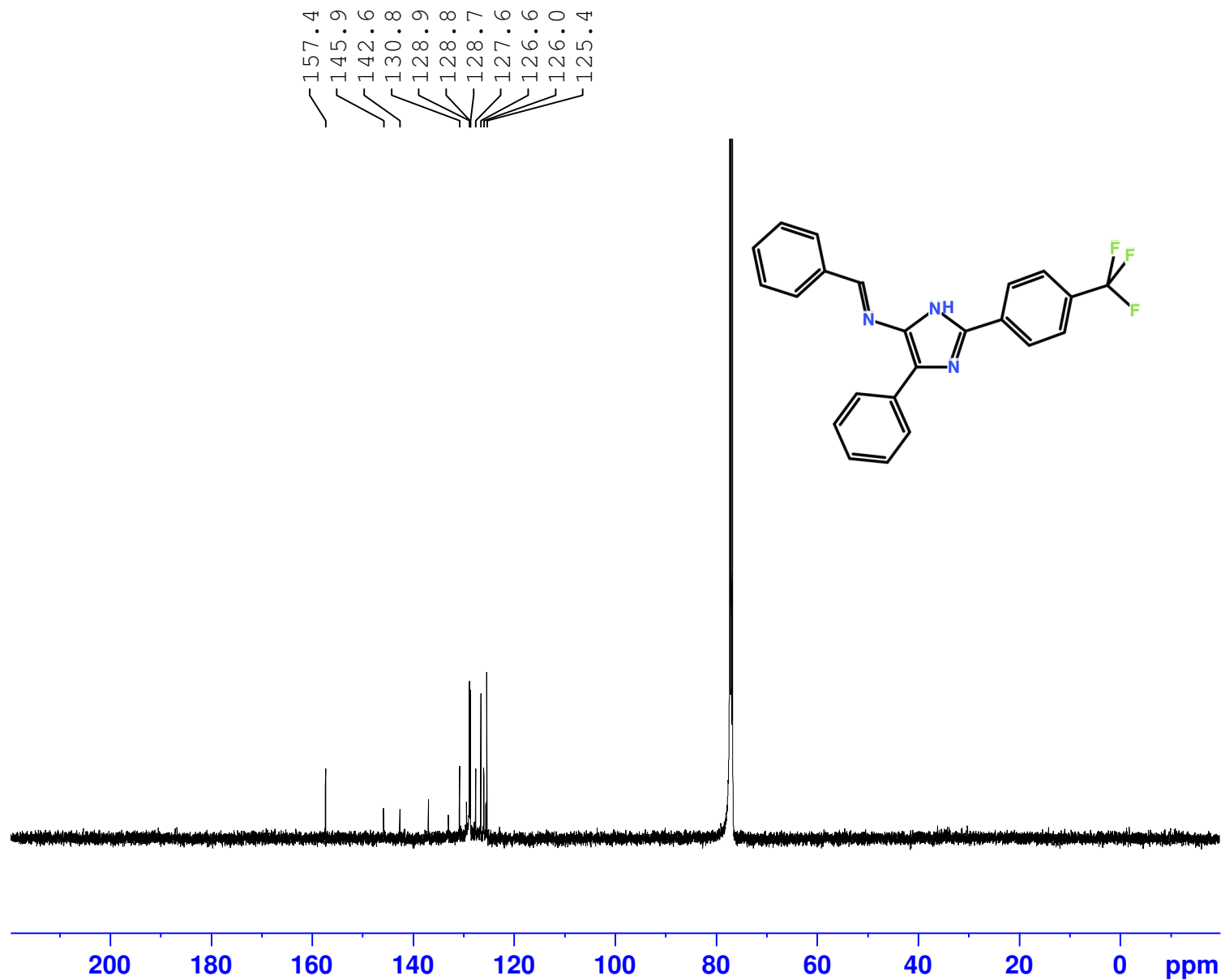
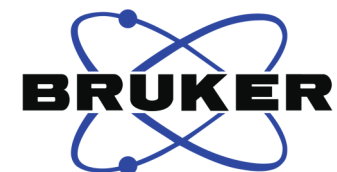


Current Data Parameters
NAME GBB-6-10-25-C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20250618
Time 15.38
INSTRUM Avance
PROBHD Z173362_0026 (PI HR-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000
FIDRES 0.305176
AQ 3.2768000
RG 101
DW 50.000
DE 10.79
TE 298.0
D1 1.00000000
TD0 1
SF01 500.1330883
NUC1 1H
P0 3.33
P1 10.00
PLW1 22.34400000

F2 - Processing parameters
SI 65536
SF 500.1300474
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00

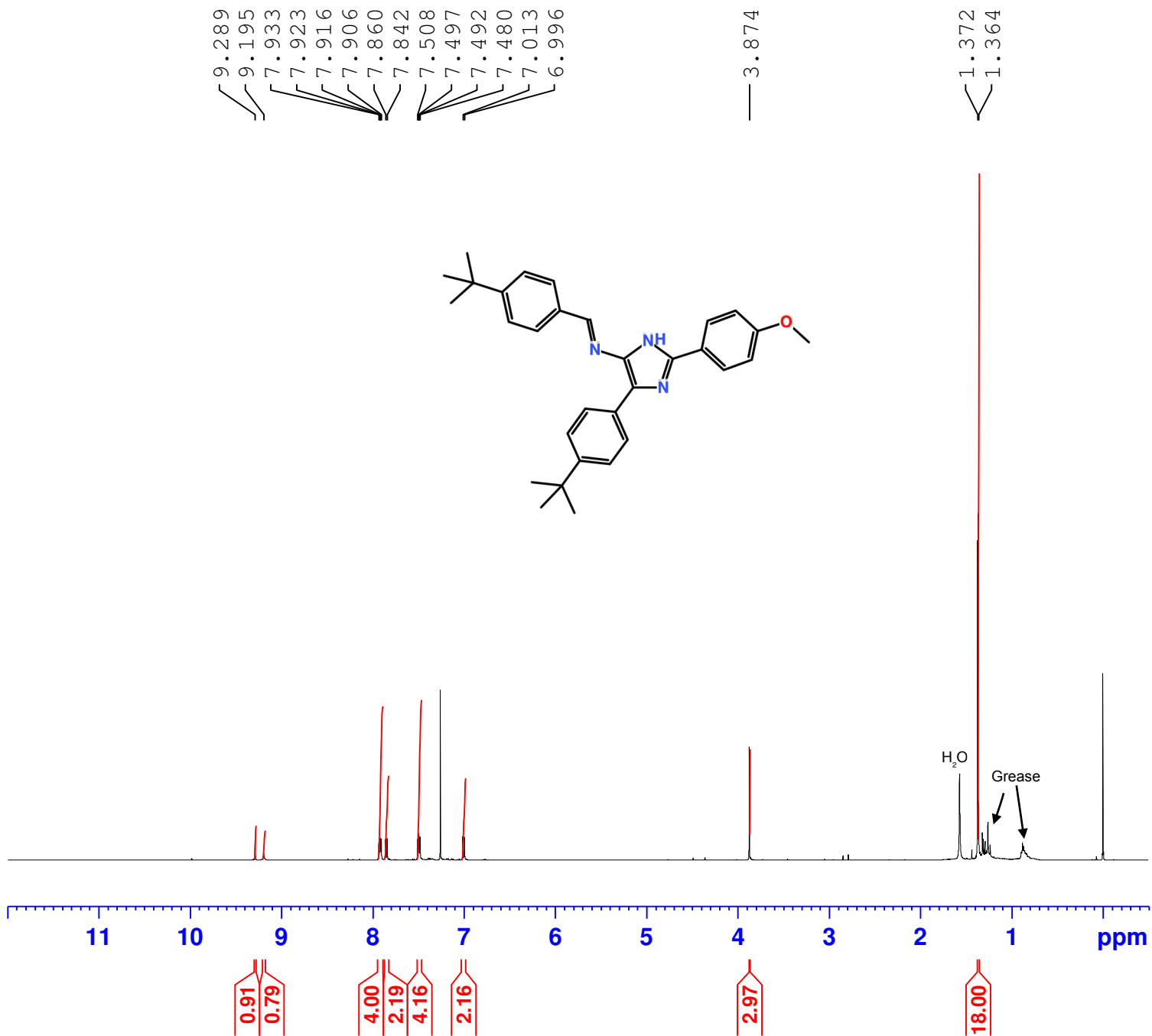




Current Data Parameters
 NAME GBB-6-10-25-C
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20250618
 Time 23.34
 INSTRUM Avance
 PROBHD Z173362_0026 (PI HR-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 5120
 DS 4
 SWH 30120.482
 FIDRES 0.919204
 AQ 1.0878976
 RG 101
 DW 16.600
 DE 6.50
 TE 298.0
 D1 2.00000000
 D11 0.03000000
 TD0 1
 SFO1 125.7703643
 NUC1 13C
 P0 3.33
 P1 10.00
 PLW1 81.01100000
 SFO2 500.1320005
 NUC2 1H
 CPDPRG2 waltz65
 PCPD2 80.00
 PLW2 22.34400000
 PLW12 0.34912000
 PLW13 0.17561000

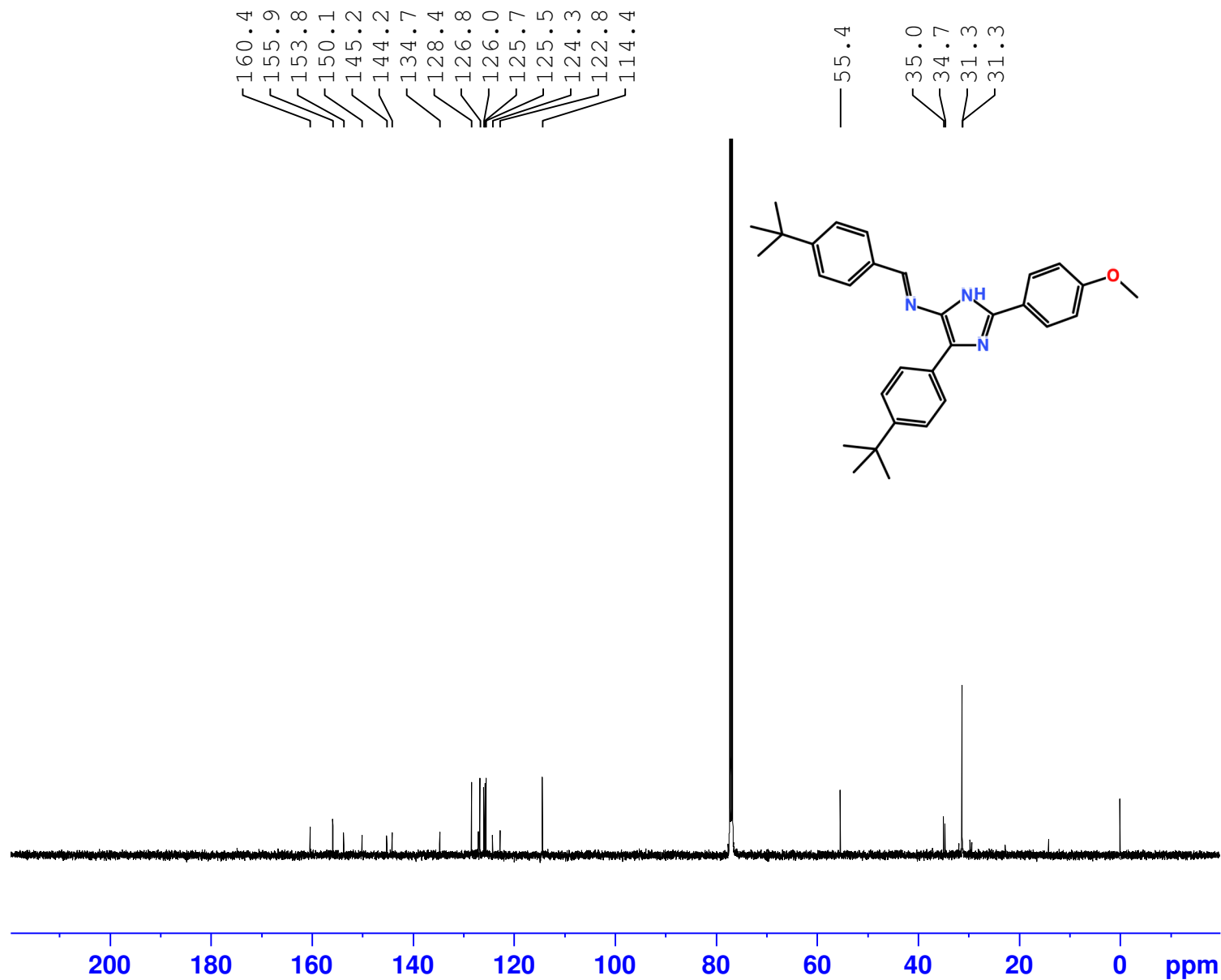
F2 - Processing parameters
 SI 32768
 SF 125.7577885
 WDW EM
 SSB 0
 LB 1.00
 GB 0
 PC 1.40



Current Data Parameters
 NAME t-bad & methoxy bam
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20250521
 Time 10.39
 INSTRUM Avance
 PROBHD Z173362_0026 (PI HR-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10000.000
 FIDRES 0.305176
 AQ 3.2768000
 RG 101
 DW 50.000
 DE 10.79
 TE 298.0
 D1 1.00000000
 TD0 1
 SF01 500.1330883
 NUC1 1H
 P0 3.33
 P1 10.00
 PLW1 22.34400000

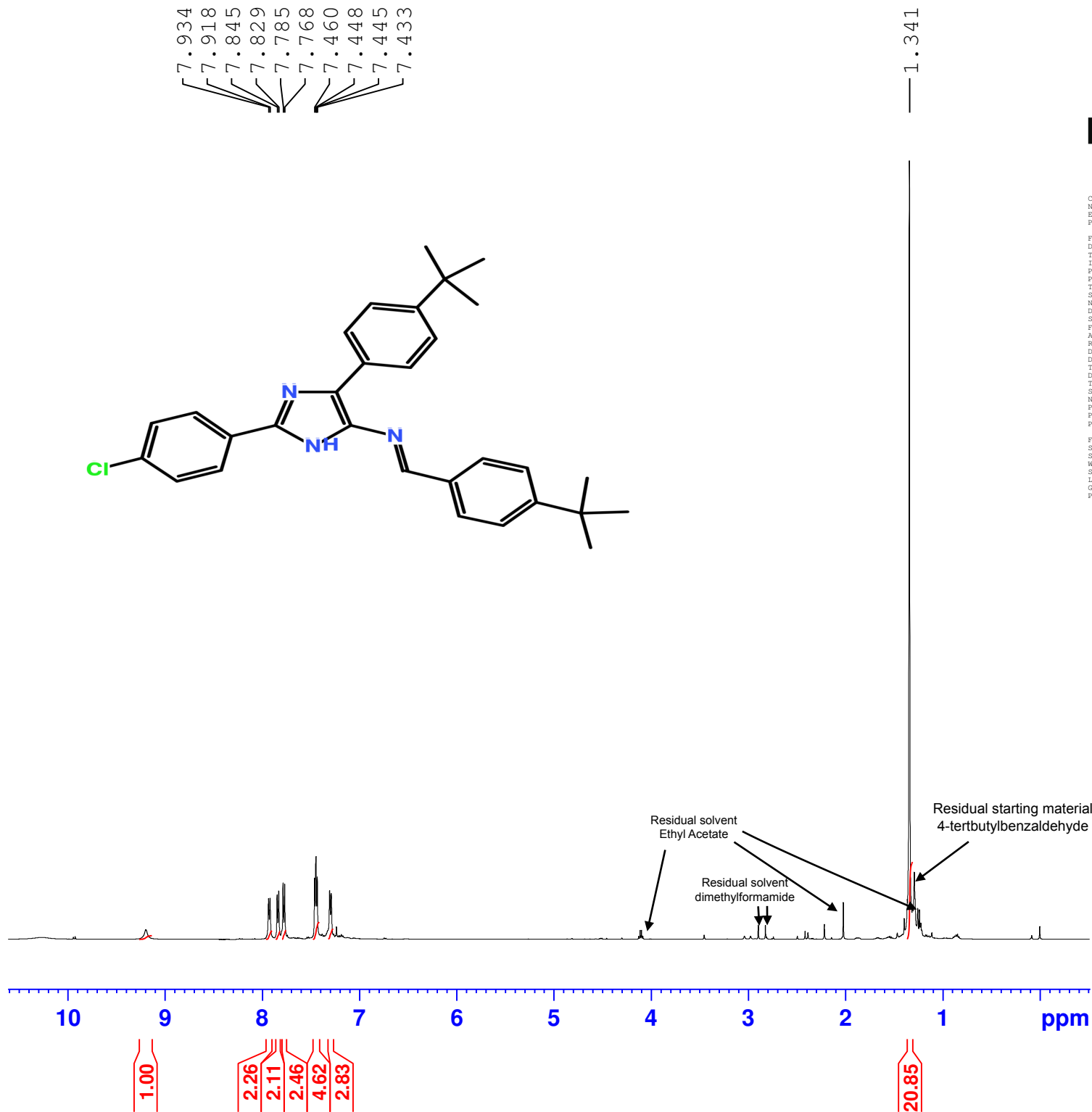
F2 - Processing parameters
 SI 65536
 SF 500.1300123
 WDW EM
 SSB 0
 LB 0.30
 GB 0
 PC 1.00



Current Data Parameters
 NAME t-bad & methoxy bam
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20250521
 Time 15.28
 INSTRUM Avance
 PROBHD Z173362_0026 (PI HR-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 5120
 DS 4
 SWH 30120.482
 FIDRES 0.919204
 AQ 1.0878976
 RG 101
 DW 16.600
 DE 6.50
 TE 298.0
 D1 2.00000000
 D11 0.03000000
 TD0 1
 SFO1 125.7703643
 NUC1 13C
 P0 3.33
 P1 10.00
 PLW1 81.01100000
 SFO2 500.1320005
 NUC2 1H
 CPDPRG2 waltz65
 PCPD2 80.00
 PLW2 22.34400000
 PLW12 0.34912000
 PLW13 0.17561000

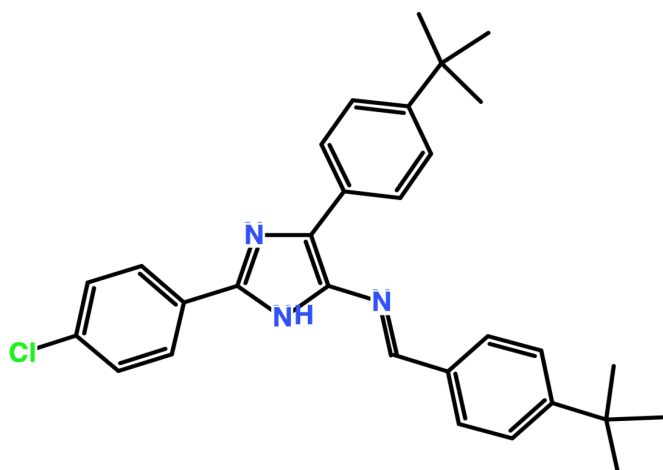
F2 - Processing parameters
 SI 32768
 SF 125.7577895
 WDW EM
 SSB 0
 LB 1.00
 GB 0
 PC 1.40



Current Data Parameters
NAME GBB-Cl-BAMI + tBu
EXPNO 1
PROCNO 1

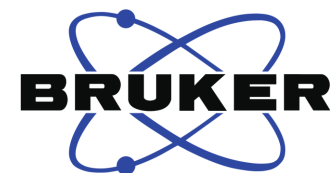
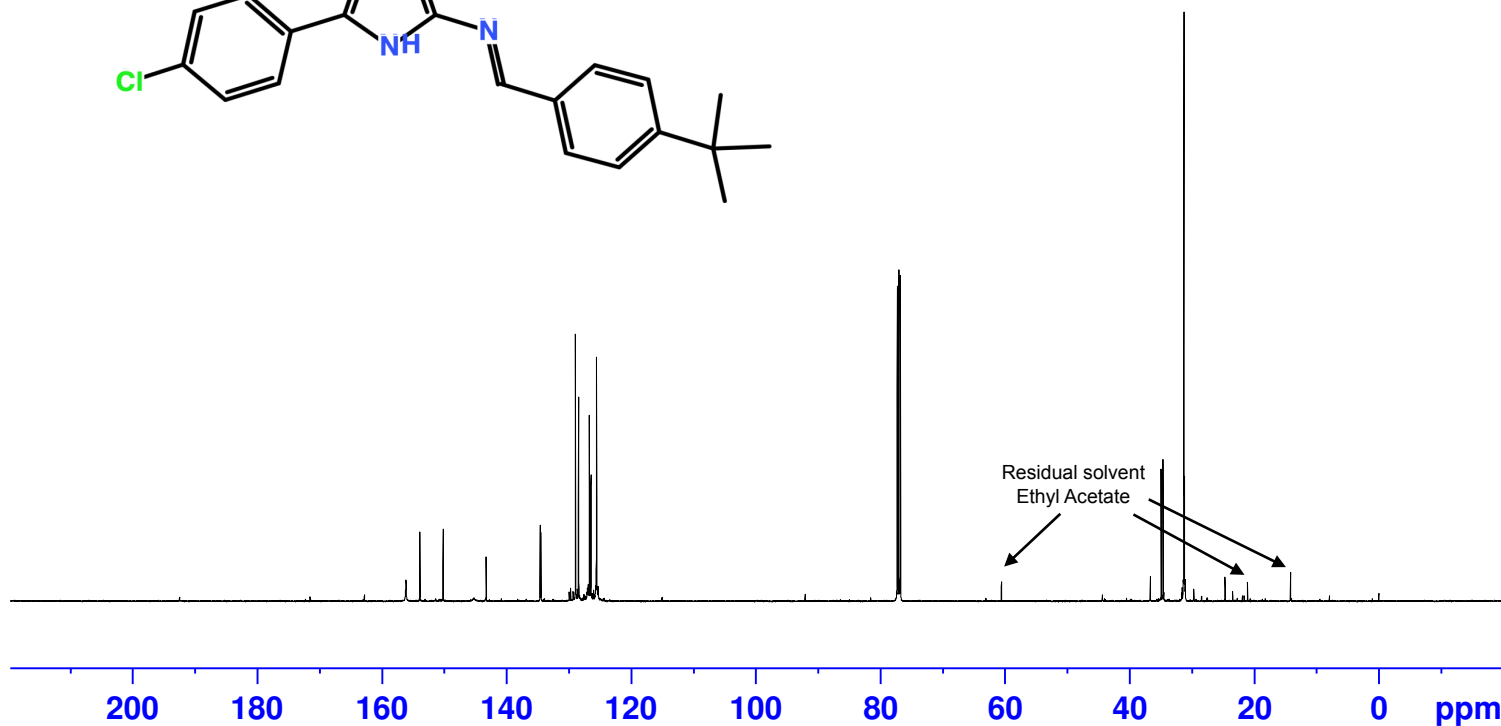
F2 - Acquisition Parameters
Date_ 20260305
Time 20.00
INSTRUM Avance
PROBHD Z173362_0026 (PI HR-TBO500S1-BBF/H/F/D-5.0-2 FB)
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2768000 sec
RG 45.2
DW 50.000 usec
DE 10.79 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.34400000 W

F2 - Processing parameters
SI 65536
SF 500.1300240 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



153.92
150.19
143.27
134.61
134.49
128.94
128.47
128.42
126.71
126.46
125.57
125.54

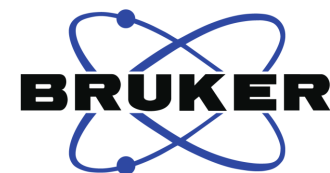
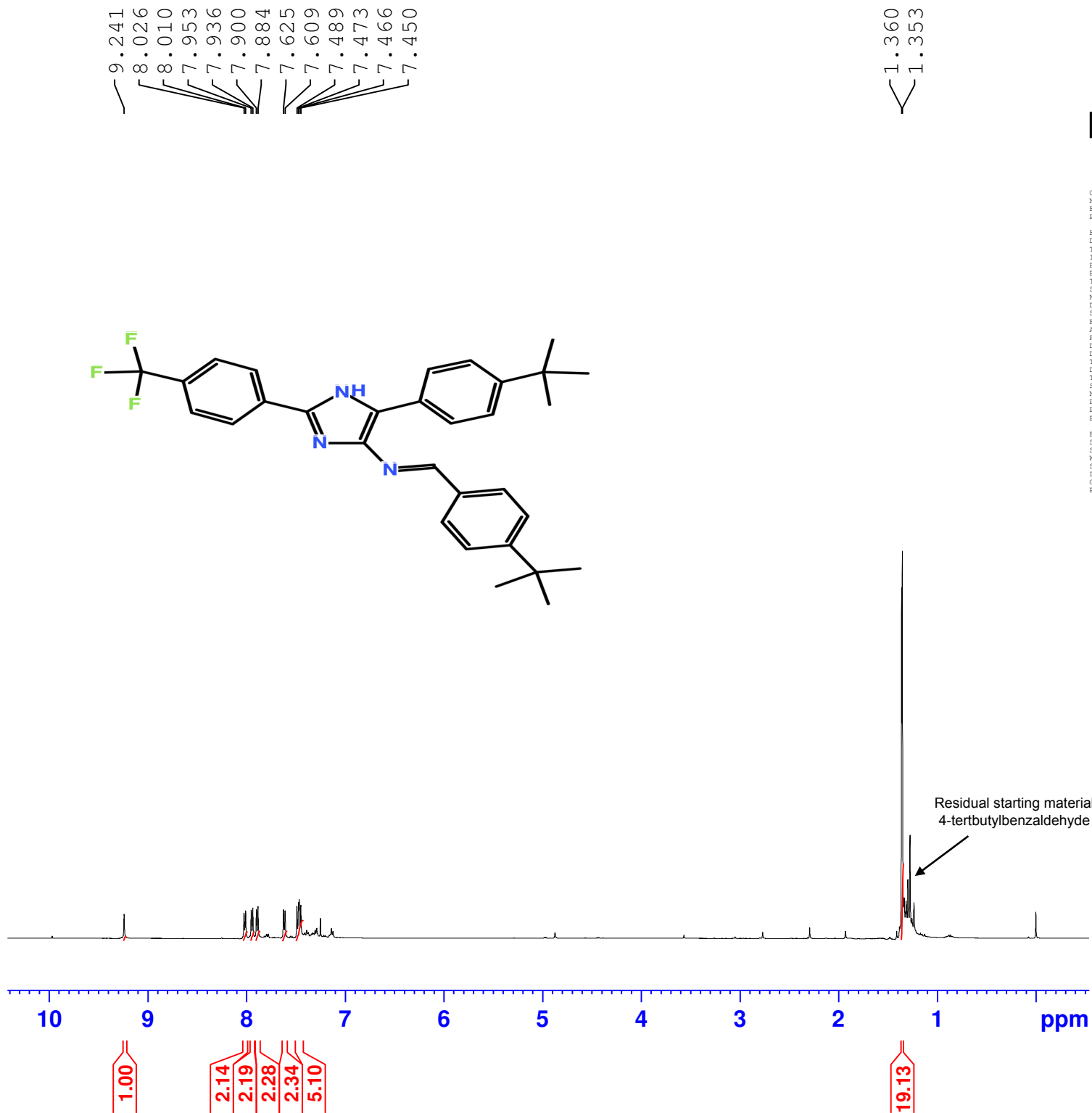
34.93
34.61
31.27
31.22



Current Data Parameters
NAME GBB-Cl-BAMI + tBu
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20260306
Time 0.32
INSTRUM Avance
PROBHD 2173362_0026 (PI HR-TBO500S1-BBF/H/F/D-5.0-2 FB)
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 5120
DS 4
SWH 30120.482 Hz
FIDRES 0.919204 Hz
AQ 1.0878976 sec
RG 101
DW 16.600 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 81.01100000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG2 waltz65
PCPD2 80.00 usec
PLW2 22.34400000 W
PLW12 0.34912000 W
PLW13 0.17561000 W

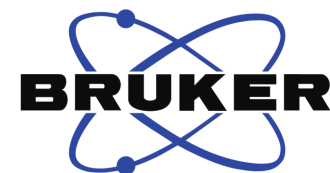
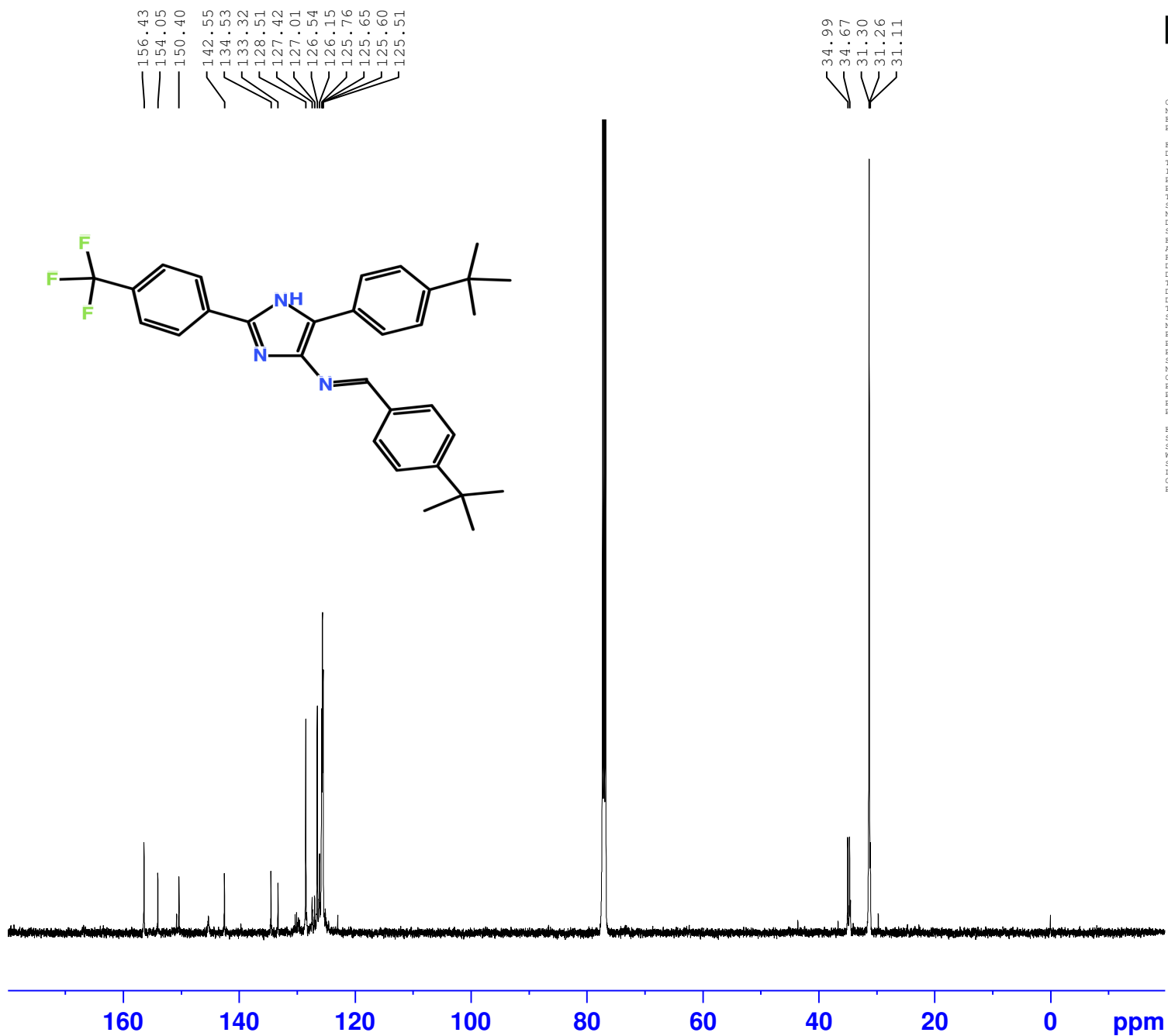
F2 - Processing parameters
SI 32768
SF 125.7577952 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Current Data Parameters
 NAME GBB-CF3-BAMI + tBu
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20260305
 Time_ 19.51
 INSTRUM Avance
 PROBHD Z173362_0026 (PI HR-TBO500S1-BBF/H/F/D-5.0-2 FB)
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 16
 DS 2
 SWH 10000.000 Hz
 FIDRES 0.305176 Hz
 AQ 3.2768000 sec
 RG 101
 DW 50.000 usec
 DE 10.79 usec
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1
 SFO1 500.1330883 MHz
 NUC1 1H
 FO 3.33 usec
 P1 10.00 usec
 PLW1 22.34400000 W

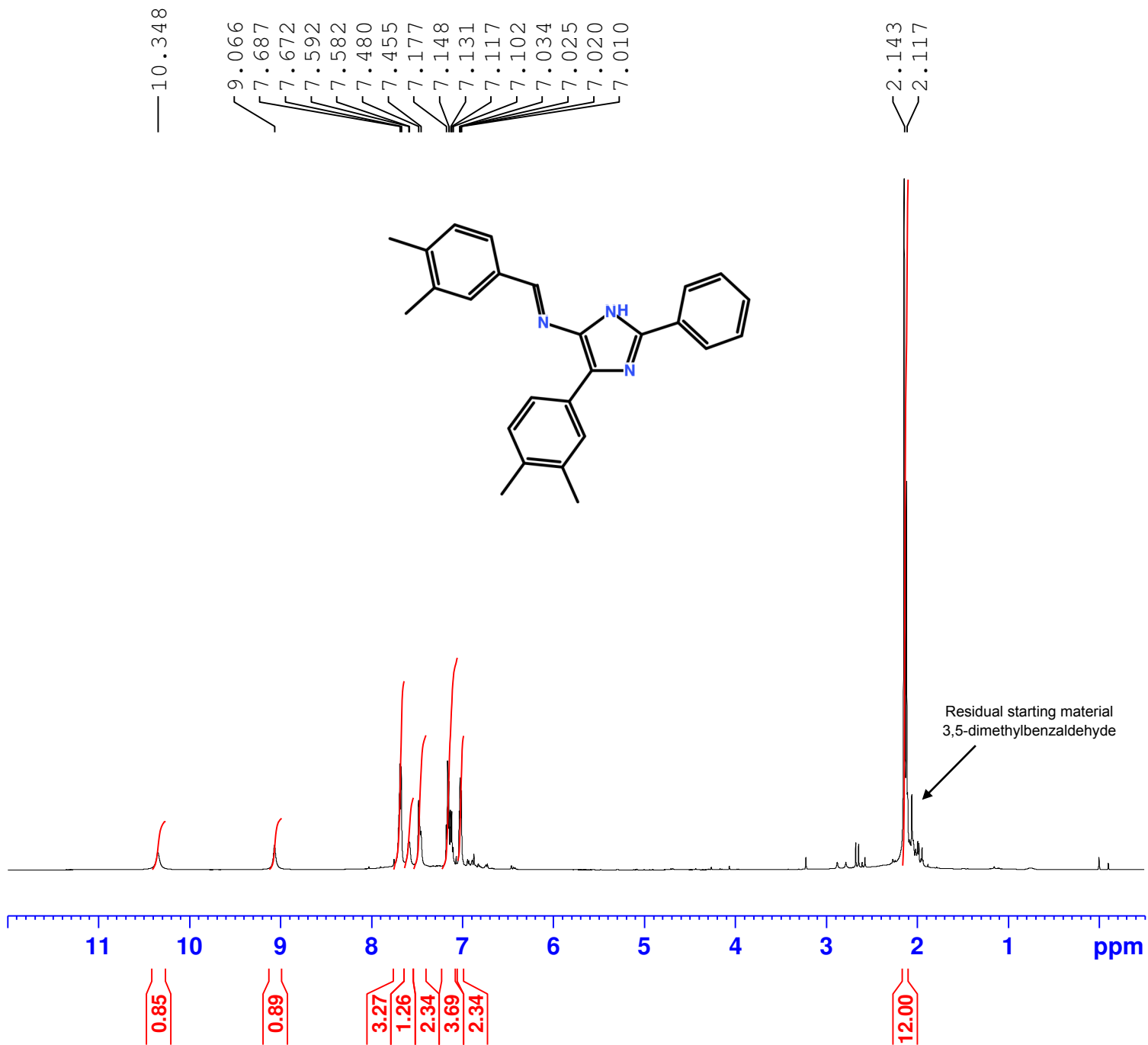
F2 - Processing parameters
 SI 65536
 SF 500.1300162 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
NAME GBB-CF3-BAMI + tBu
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20260306
Time 5.05
INSTRUM Avance
PROBHD Z173362_0026 (PI-BB-TBO500S1-BBF/H/F/D-5.0-2 FB)
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 5120
DS 4
SWH 30120.482 Hz
FIDRES 0.919204 Hz
AQ 1.0878976 sec
RG 101
DW 16.600 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 81.01100000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG2 waltz165
PCPD2 80.00 usec
PLW2 22.34400000 W
PLW12 0.34912000 W
PLW13 0.17561000 W

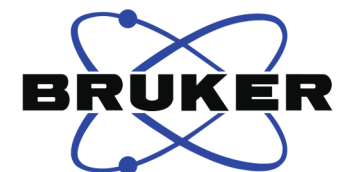
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Current Data Parameters
 NAME GBB-3,4-dimethylbenz
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20250529
 Time 12.31
 INSTRUM Avance
 PROBHD Z173362_0026 (PI HR-
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 10000.000
 FIDRES 0.305176
 AQ 3.2768000
 RG 32
 DW 50.000
 DE 10.79
 TE 298.0
 D1 1.00000000
 TD0 1
 SF01 500.1330883
 NUC1 1H
 P0 3.33
 P1 10.00
 PLW1 22.34400000

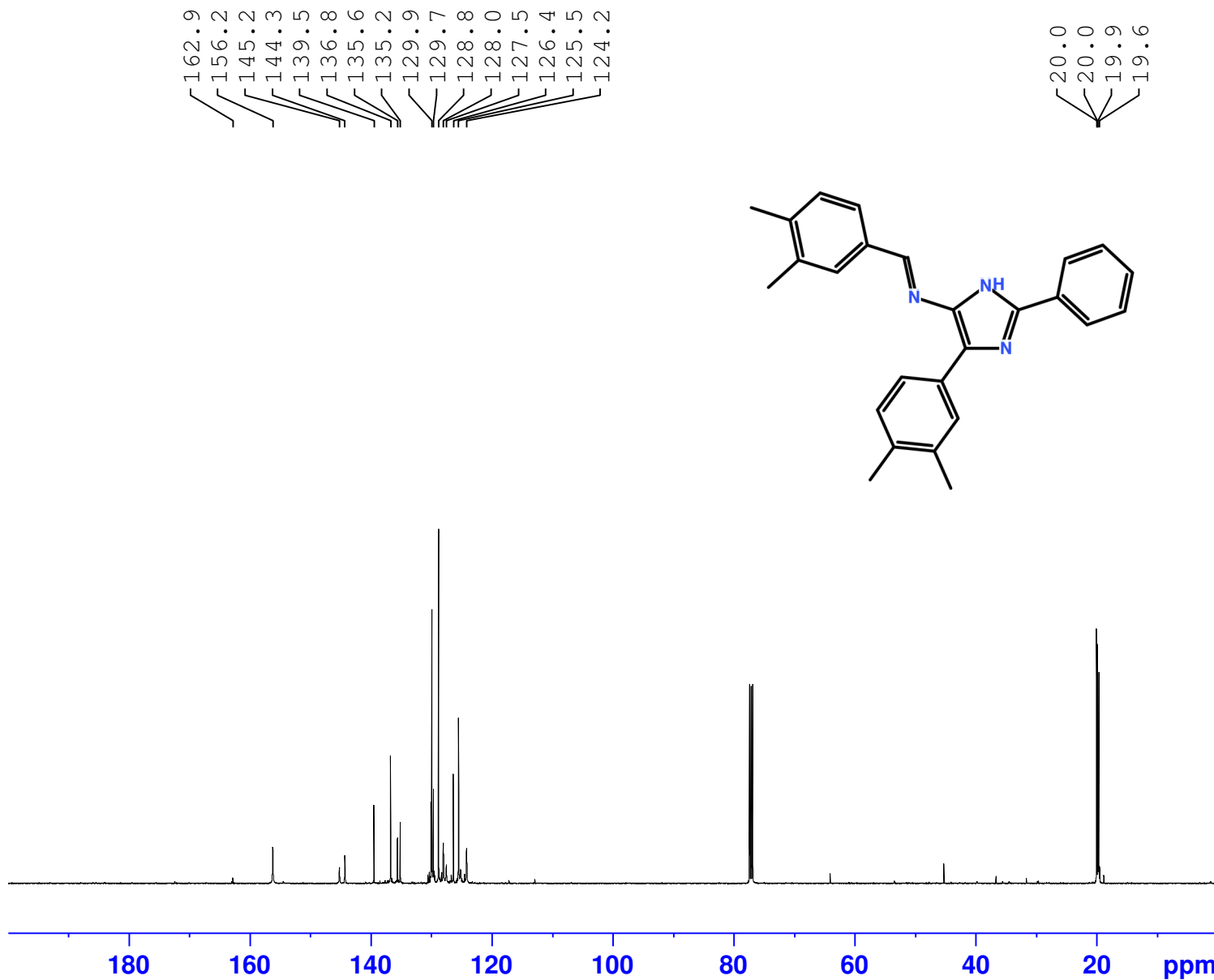
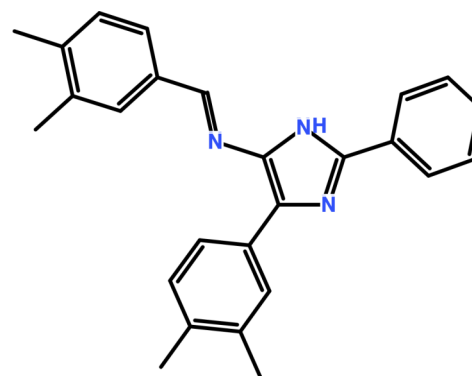
F2 - Processing parameters
 SI 65536
 SF 500.1301090
 WDW EM
 SSB 0
 LB 0.30
 GB 0
 PC 1.00



Current Data Parameters
 NAME GBB-3,4-dimethylbenz
 EXPNO 5
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20250529
 Time 18.19
 INSTRUM Avance
 PROBHD Z173362_0026 (PI HR-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 5120
 DS 4
 SWH 30120.482
 FIDRES 0.919204
 AQ 1.0878976
 RG 101
 DW 16.600
 DE 6.50
 TE 298.0
 D1 2.00000000
 D11 0.03000000
 TD0 1
 SFO1 125.7703643
 NUC1 13C
 P0 3.33
 P1 10.00
 PLW1 81.01100000
 SFO2 500.1320005
 NUC2 1H
 CPDPRG2 waltz65
 PCPD2 80.00
 PLW2 22.34400000
 PLW12 0.34912000
 PLW13 0.17561000

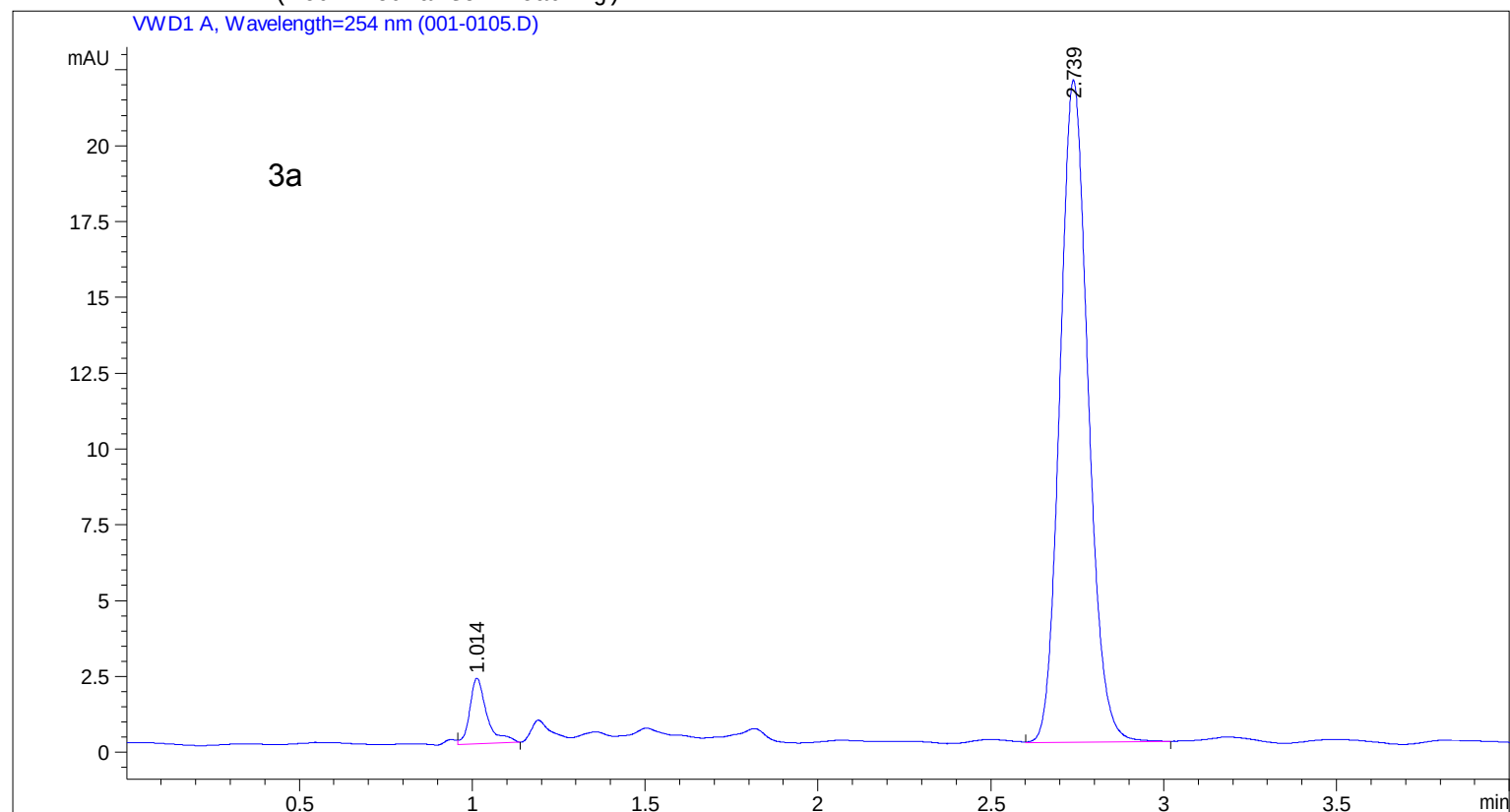
F2 - Processing parameters
 SI 32768
 SF 125.7577885
 WDW EM
 SSB 0
 LB 1.00
 GB 0
 PC 1.40



Sample Name:

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :    1
Acq. Instrument : LC                        Location  : Vial 1
Injection Date  : 7/22/2026 11:54:13 AM      Inj       :    5
                                           Inj Volume: Manually

Sequence File   : C:\CHEM32\1\SEQUENCE\KEM.S
Method          : C:\CHEM32\1\METHODS\KAM.M
Last changed    : 7/22/2026 11:48:47 AM by SYSTEM
                  (modified after loading)
=====
```



```
=====
                        Area Percent Report
=====
```

```
Sorted By           :      Signal
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.014	VB	0.0512	7.45310	2.16099	5.6793
2	2.739	VB	0.0876	123.78002	21.83794	94.3207

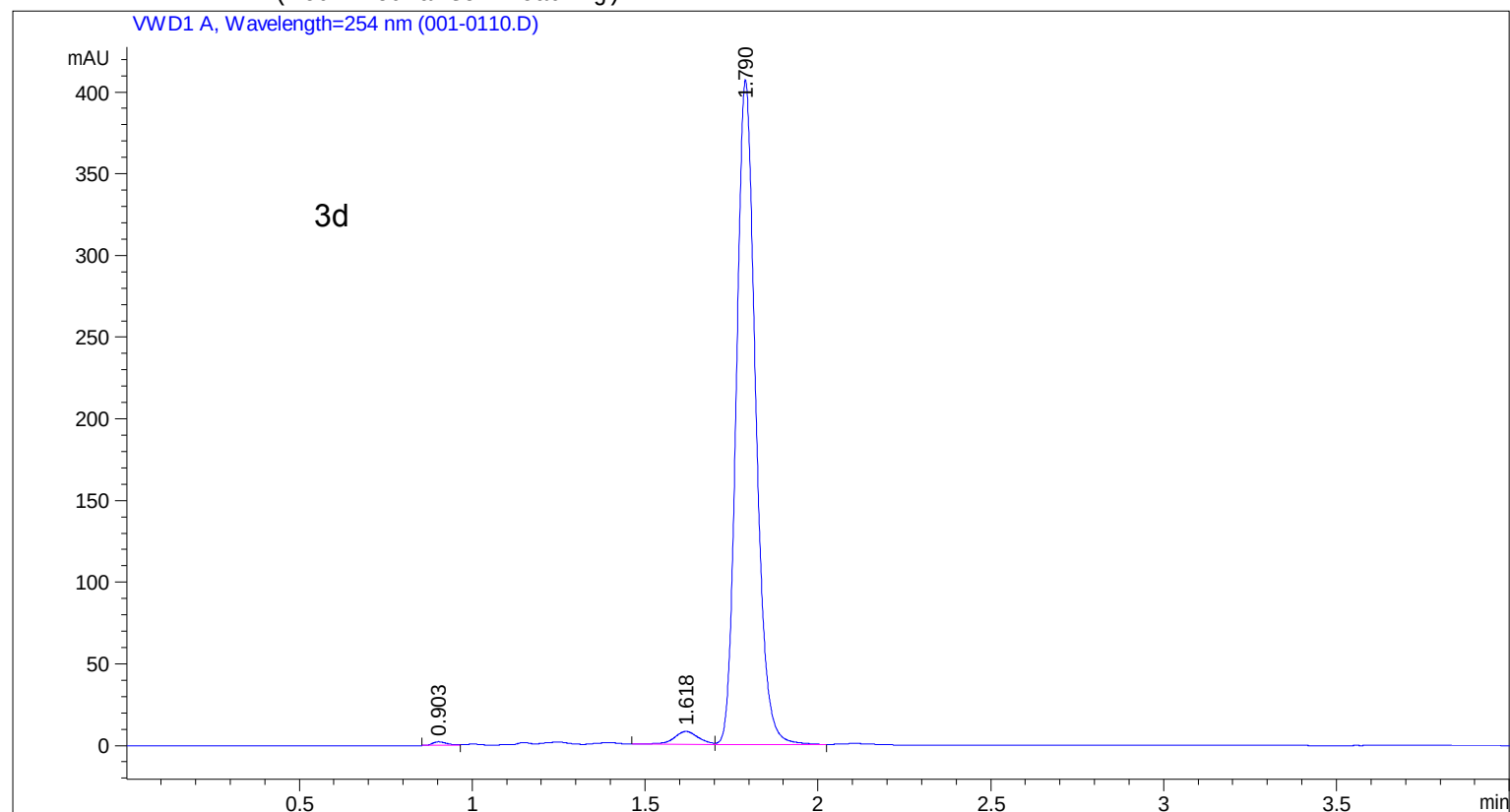
```
Totals :                      131.23313   23.99892
```

```
=====
*** End of Report ***
```

Sample Name:

```
=====
Acq. Operator   : SYSTEM                      Seq. Line :    1
Acq. Instrument : LC                        Location : Vial 1
Injection Date  : 7/22/2026 1:14:44 PM      Inj       :   10
                                           Inj Volume: Manually

Sequence File   : C:\CHEM32\1\SEQUENCE\KEM.S
Method          : C:\CHEM32\1\METHODS\KAM.M
Last changed    : 7/22/2026 1:00:56 PM by SYSTEM
                  (modified after loading)
=====
```



```
=====
                        Area Percent Report
=====
```

```
Sorted By           :      Signal
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	0.903	BV	0.0442	6.68037	2.27947	0.4122
2	1.618	BV	0.0732	38.35580	7.89205	2.3666
3	1.790	VV	0.0602	1575.68213	406.72134	97.2212

```
Totals :                      1620.71829  416.89286
```

```
=====
*** End of Report ***
```

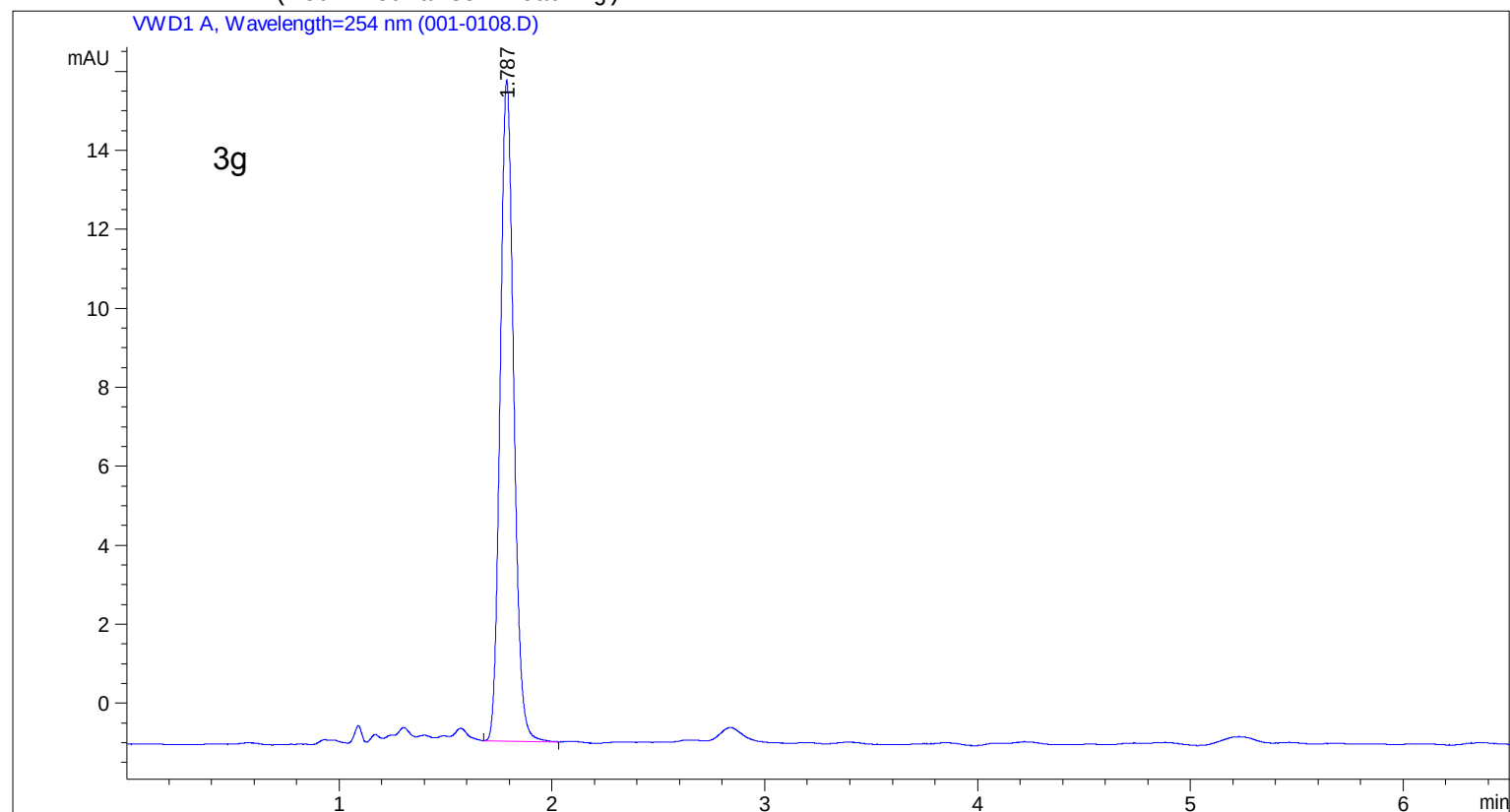
Sample Name:

```

=====
Acq. Operator   : SYSTEM                      Seq. Line :    1
Acq. Instrument : LC                        Location  : Vial 1
Injection Date  : 6/16/2026 2:16:36 PM      Inj       :    8
                                           Inj Volume: Manually

Sequence File   : C:\CHEM32\1\SEQUENCE\KEM.S
Method          : C:\CHEM32\1\METHODS\KAM.M
Last changed    : 6/16/2026 2:17:56 PM by SYSTEM
                  (modified after loading)
=====

```



```

=====
                        Area Percent Report
=====

```

```

Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.787	BB	0.0688	74.36150	16.74156	100.0000

```
Totals :                      74.36150  16.74156
```

```

=====
*** End of Report ***

```