Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

Microwave-assisted multicomponent synthesis of antiproliferative 2,4-dimethoxy-tetrahydropyrimido[4,5-b]quinolin-6(7*H*)-ones

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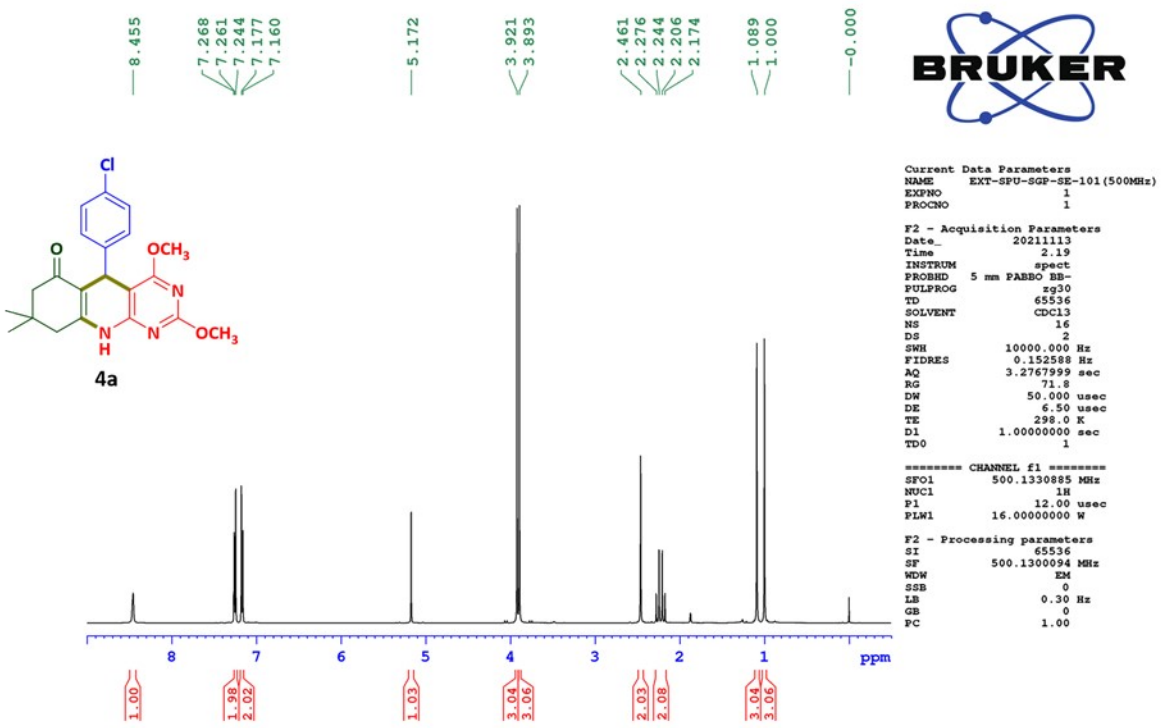

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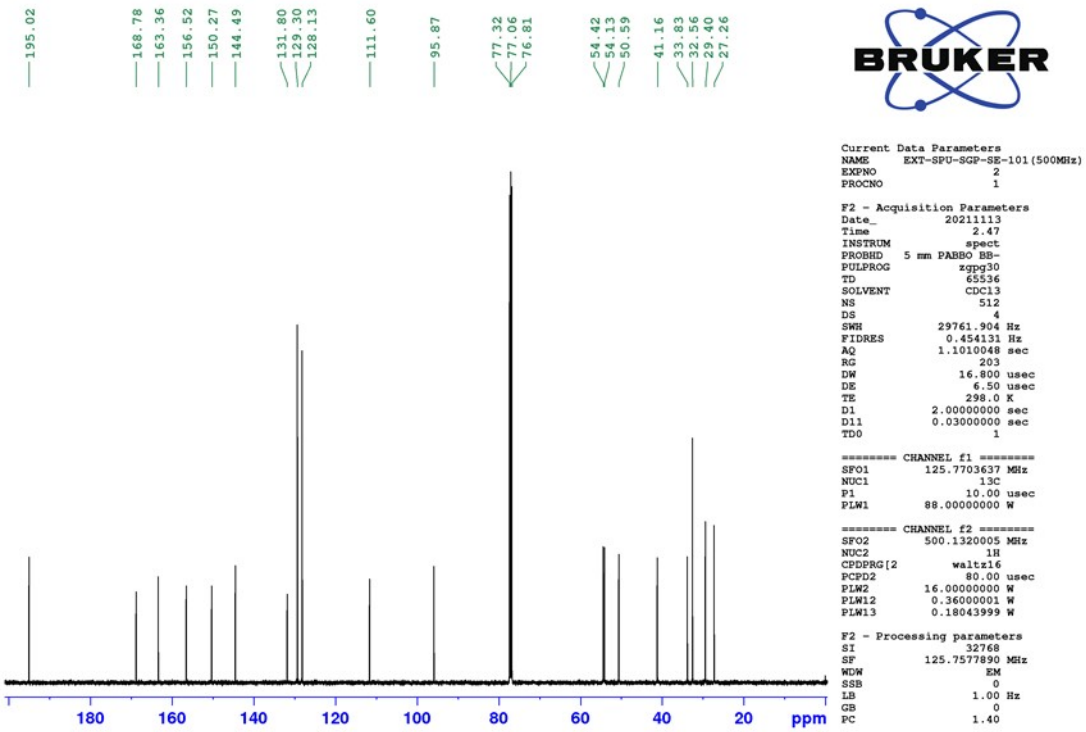

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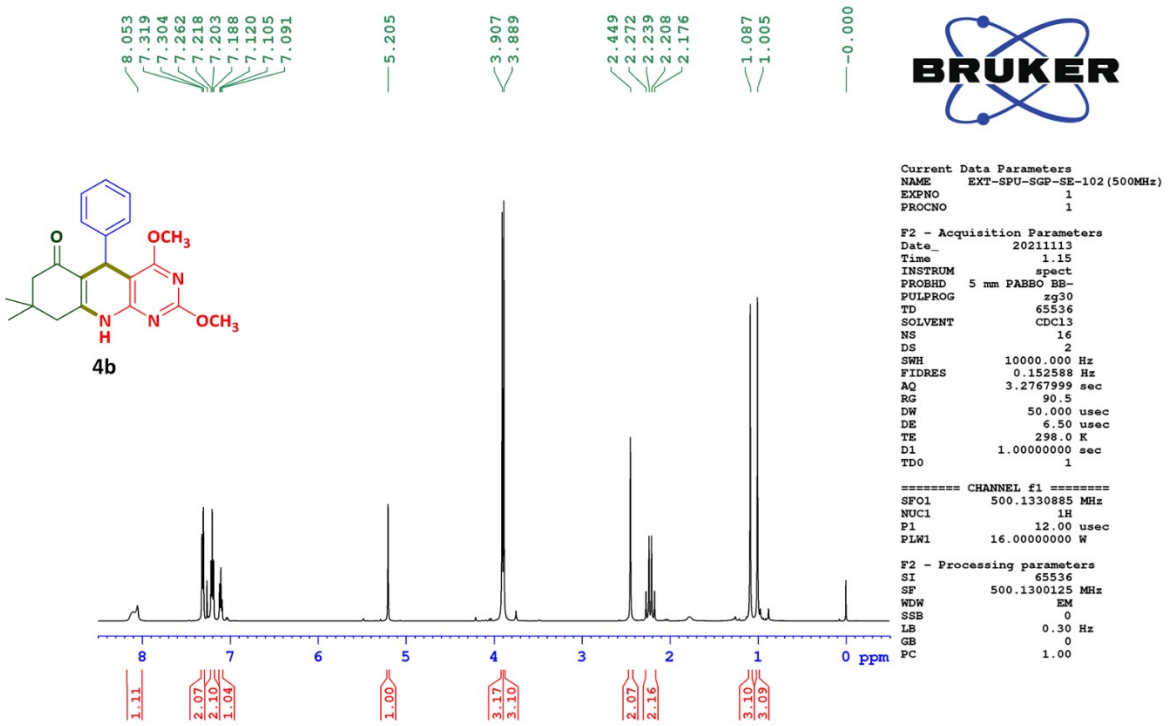

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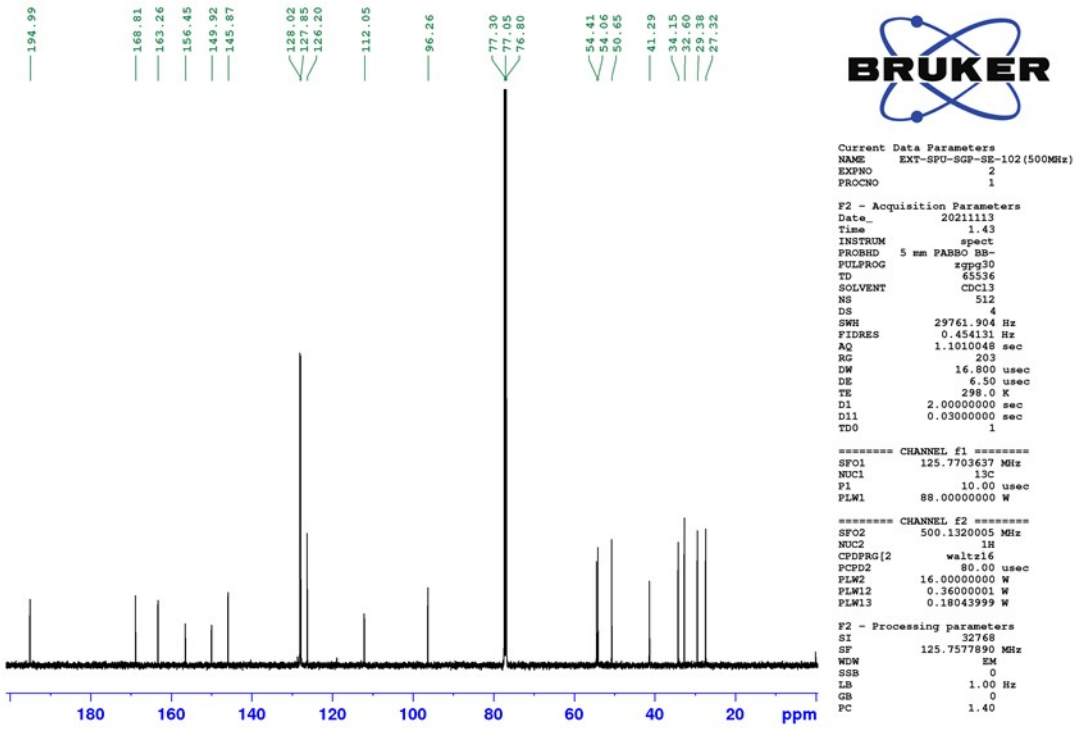

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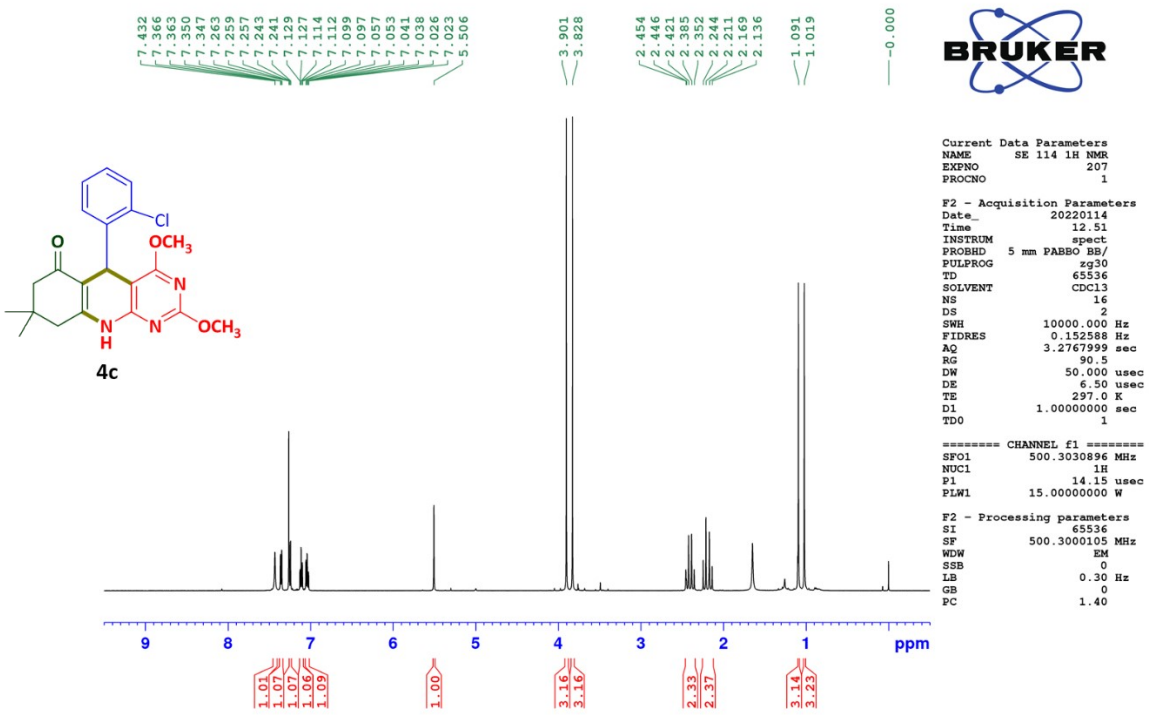

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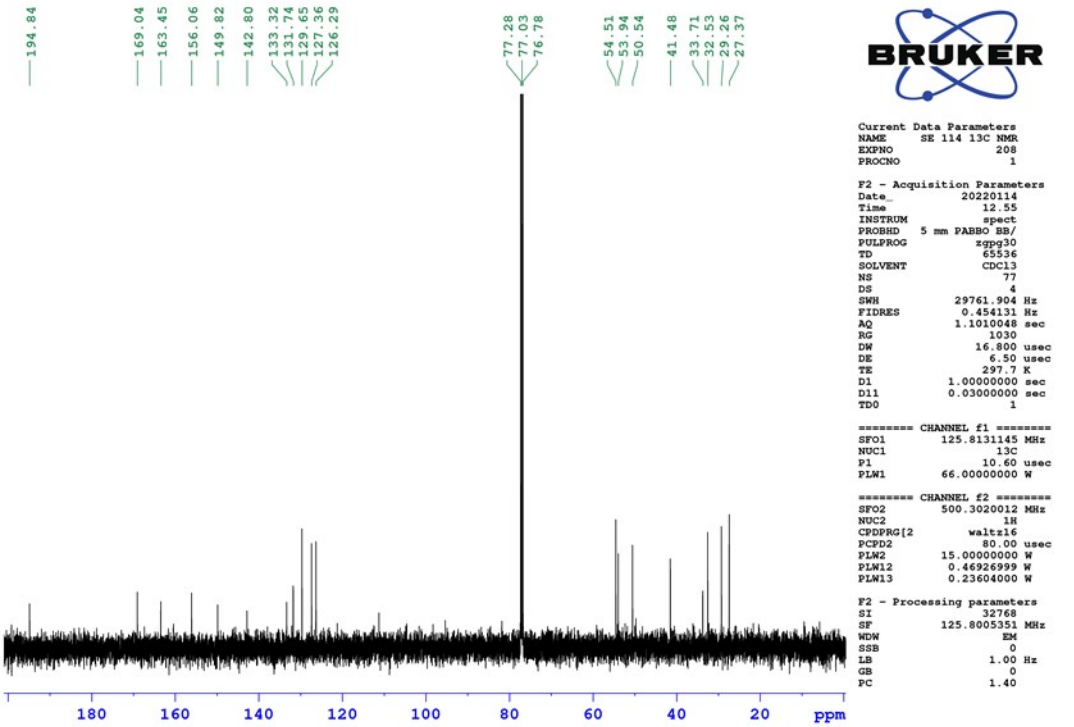

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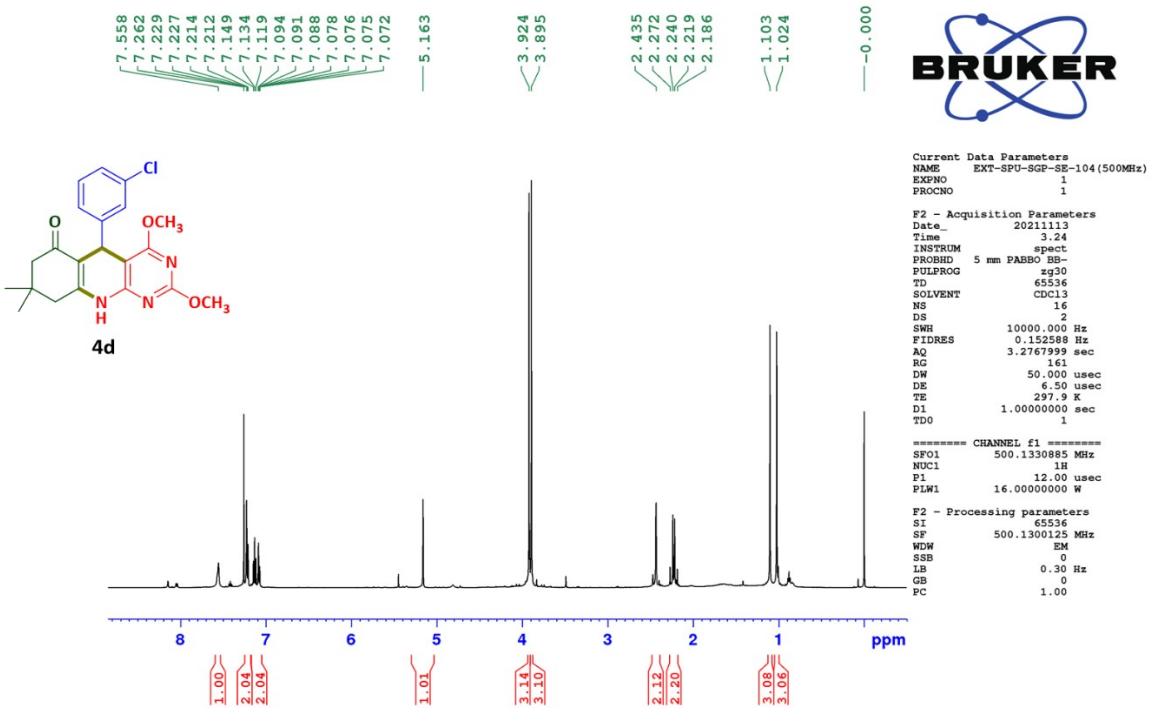

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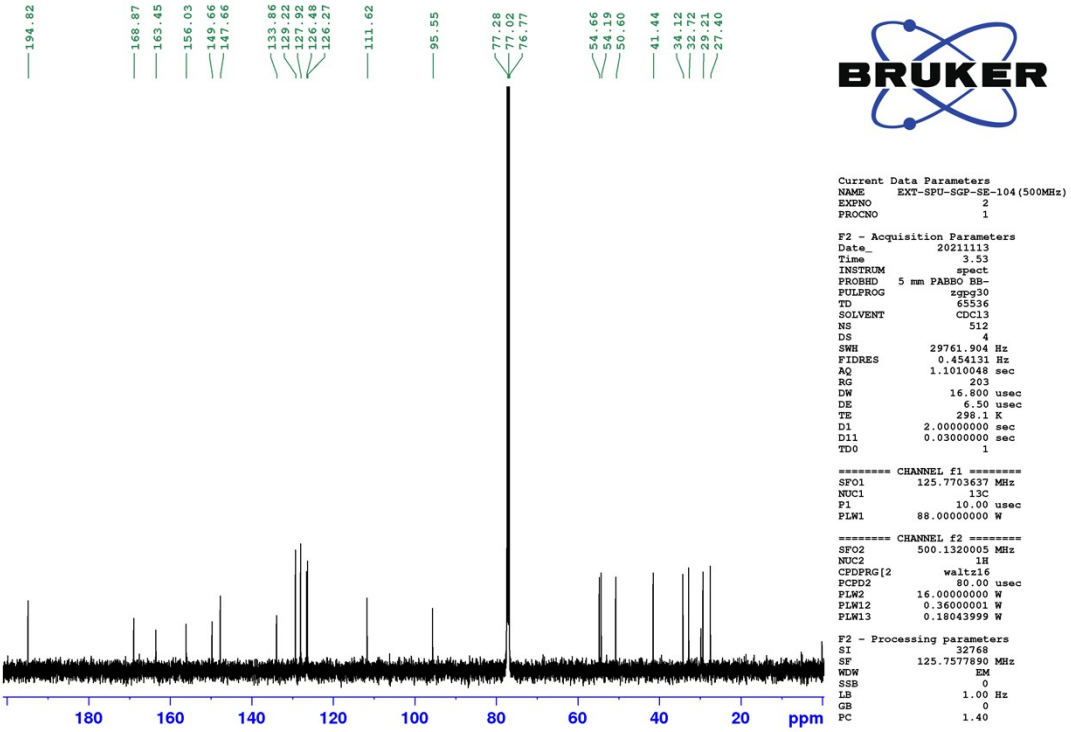

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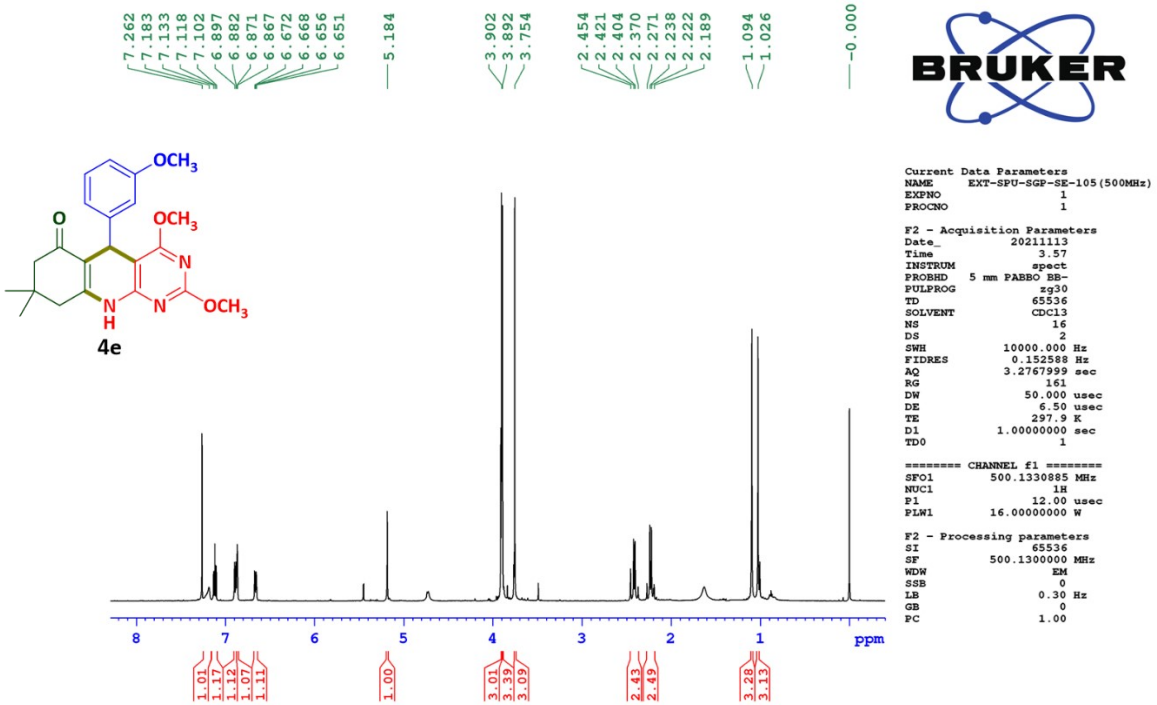

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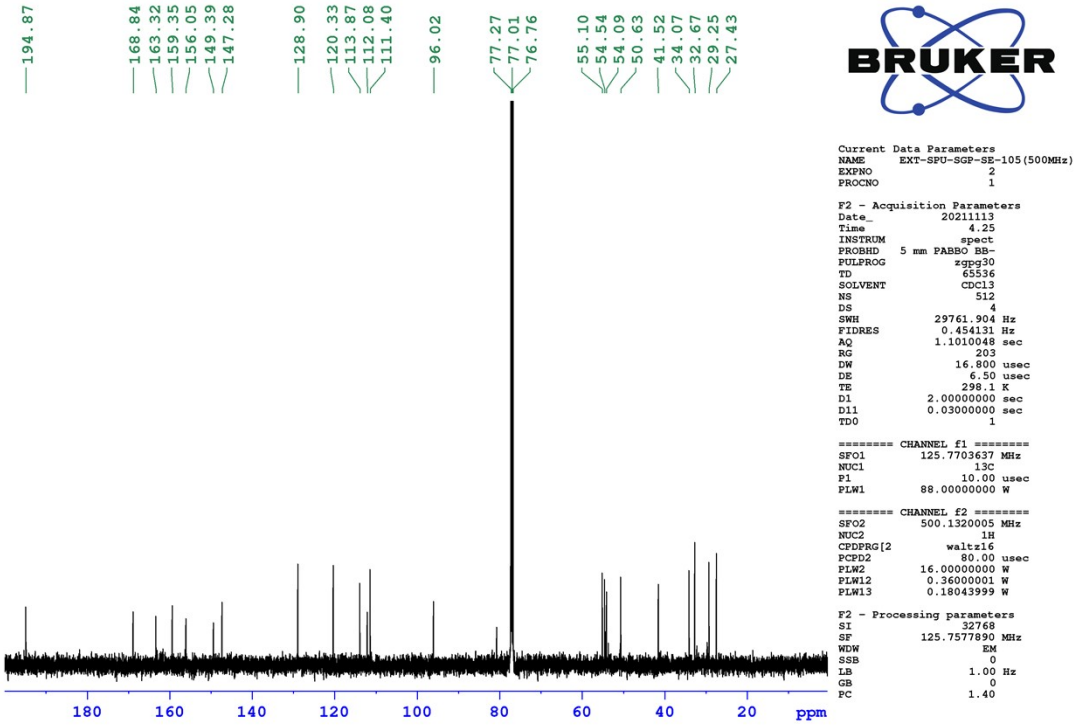

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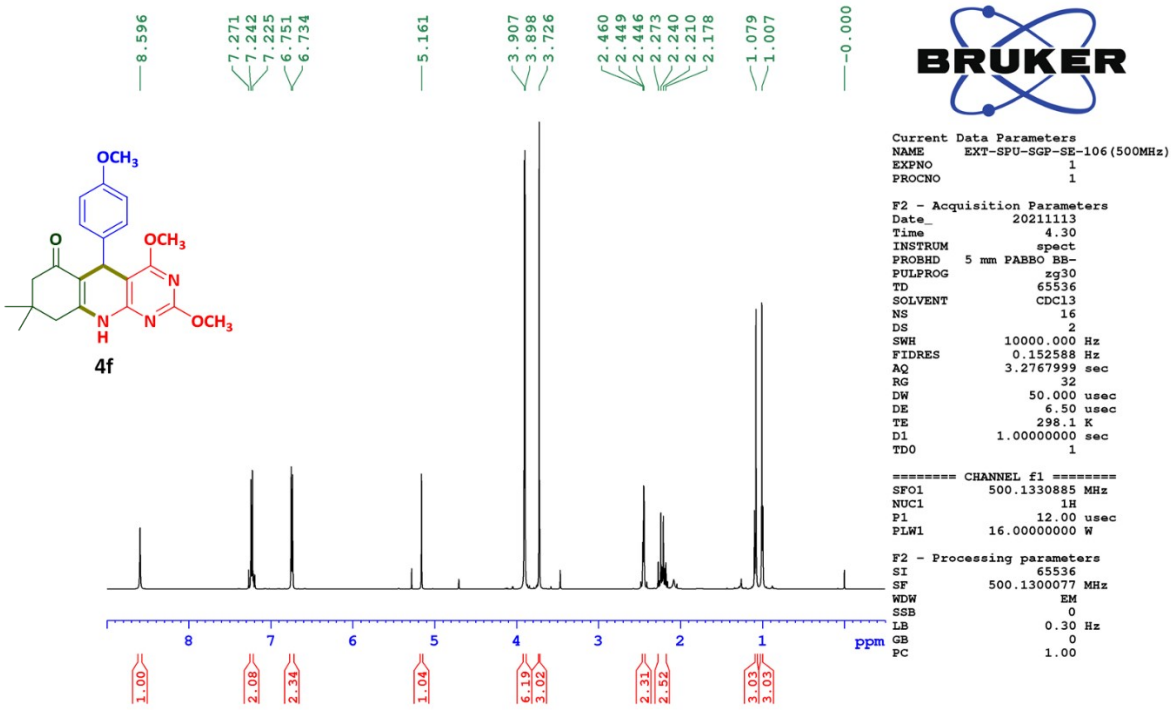

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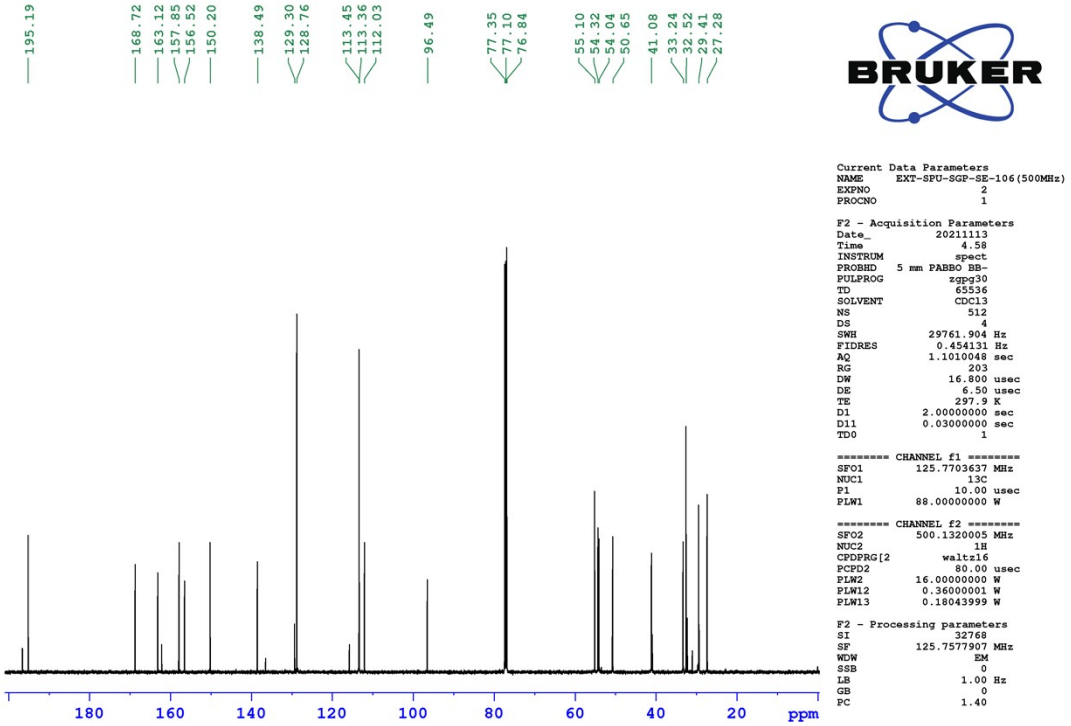

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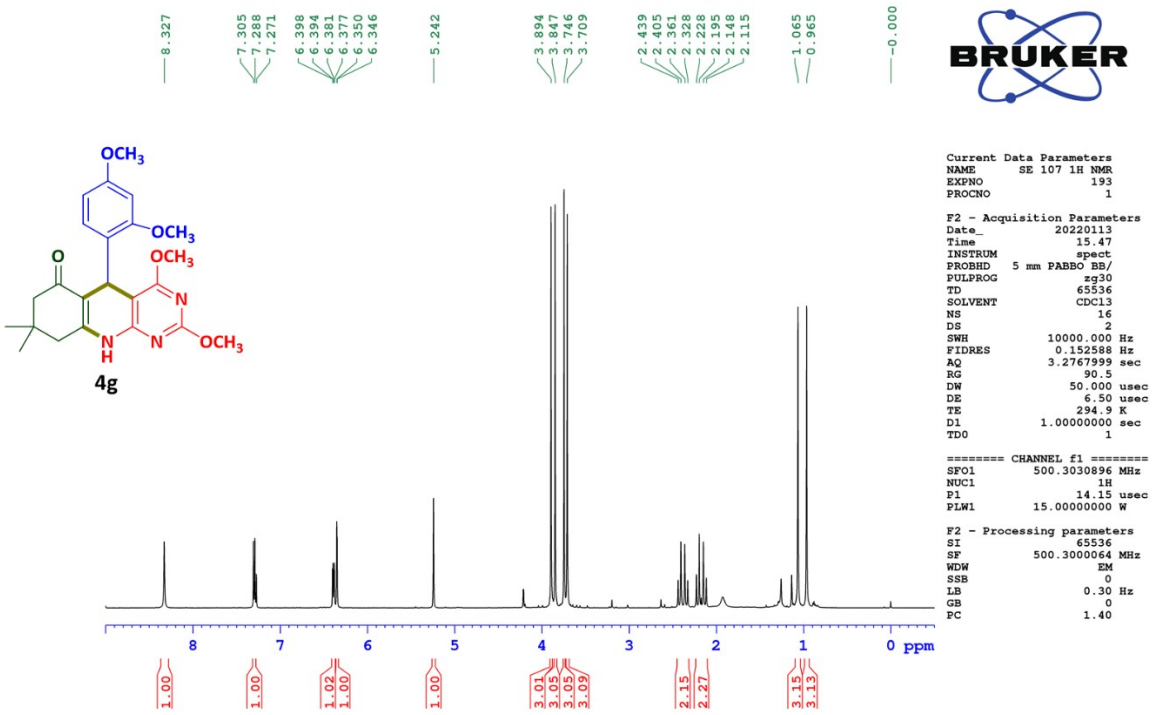

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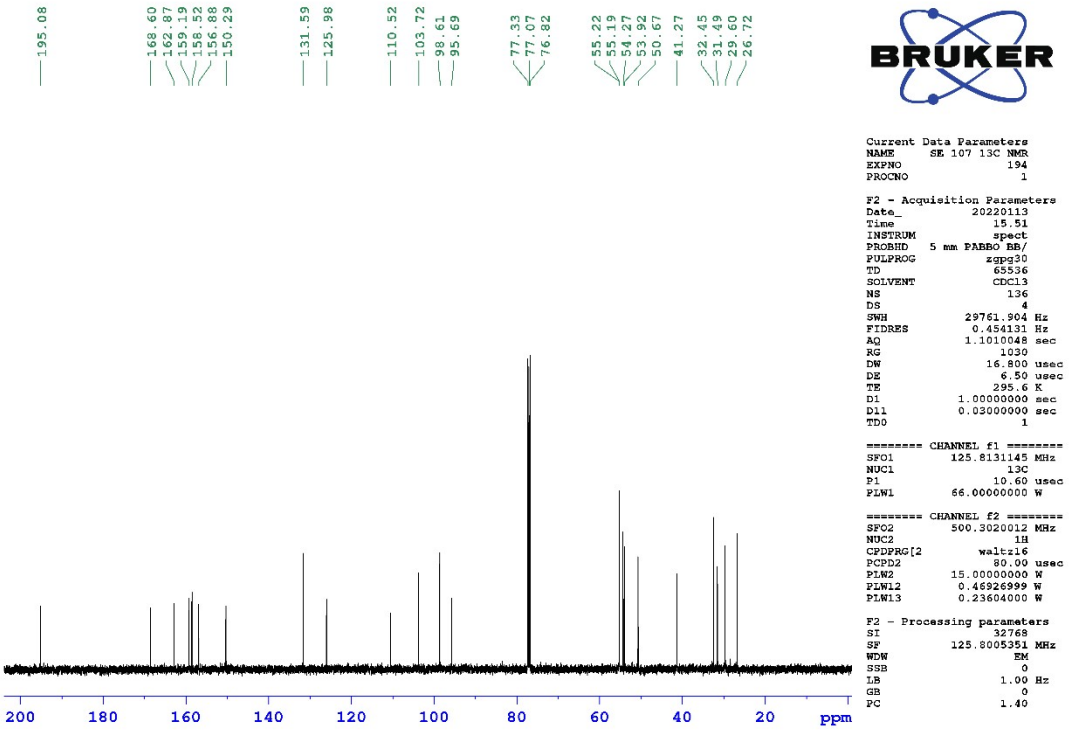

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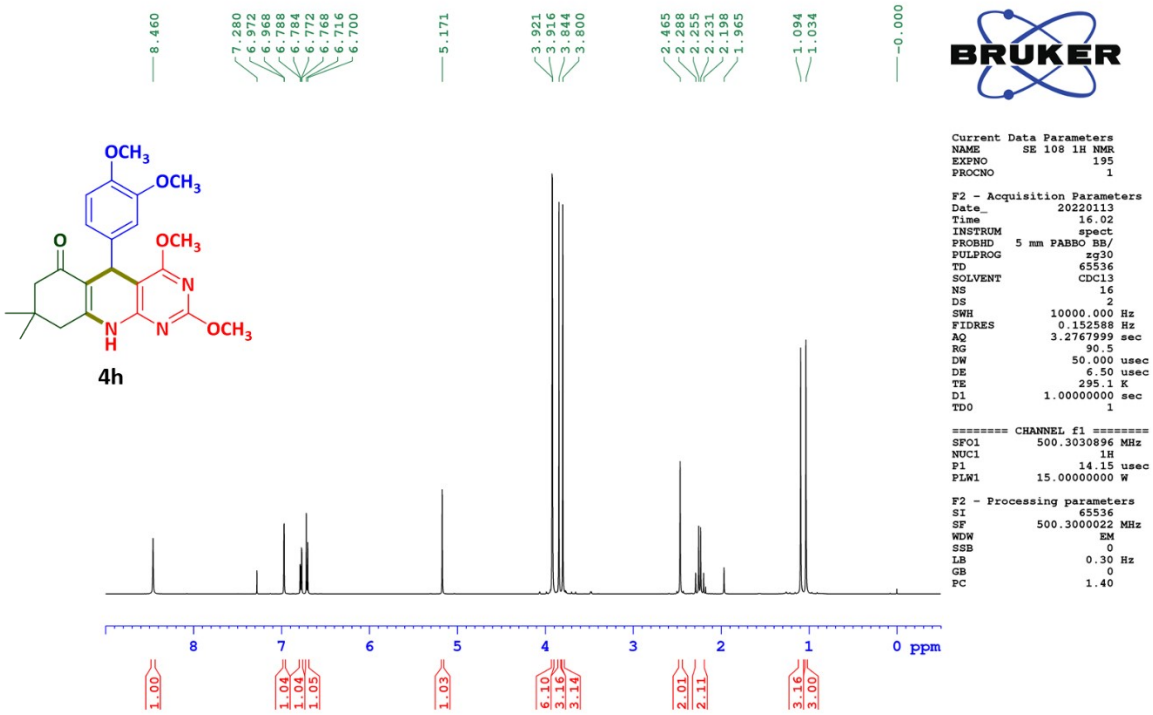

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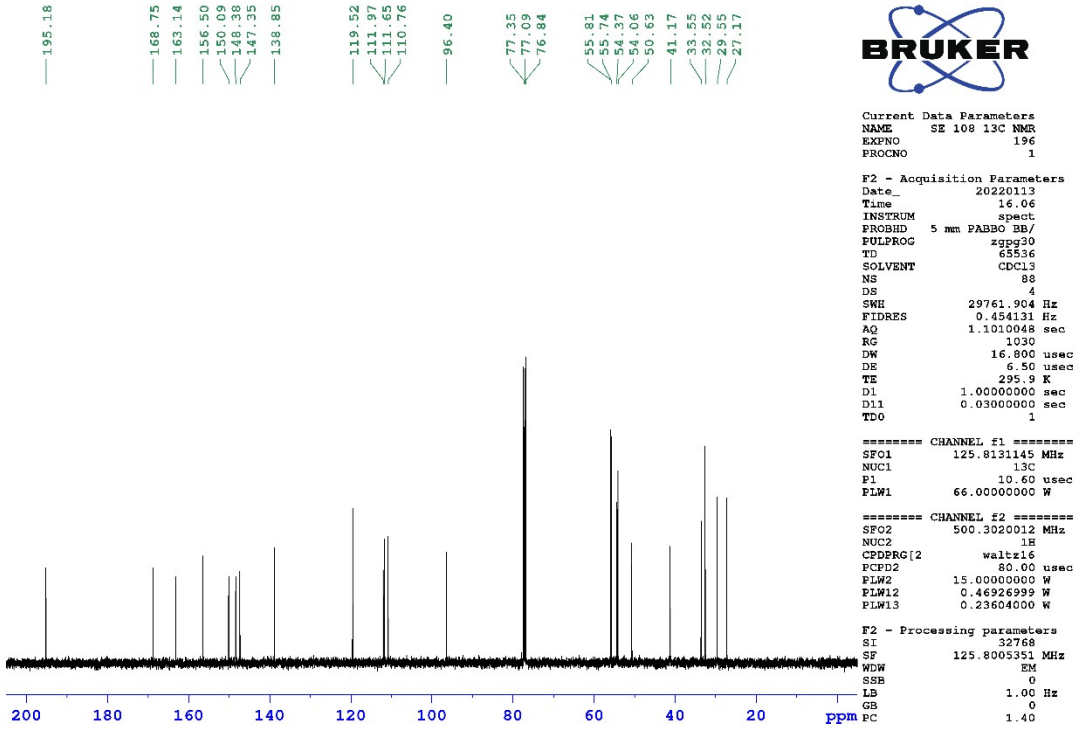

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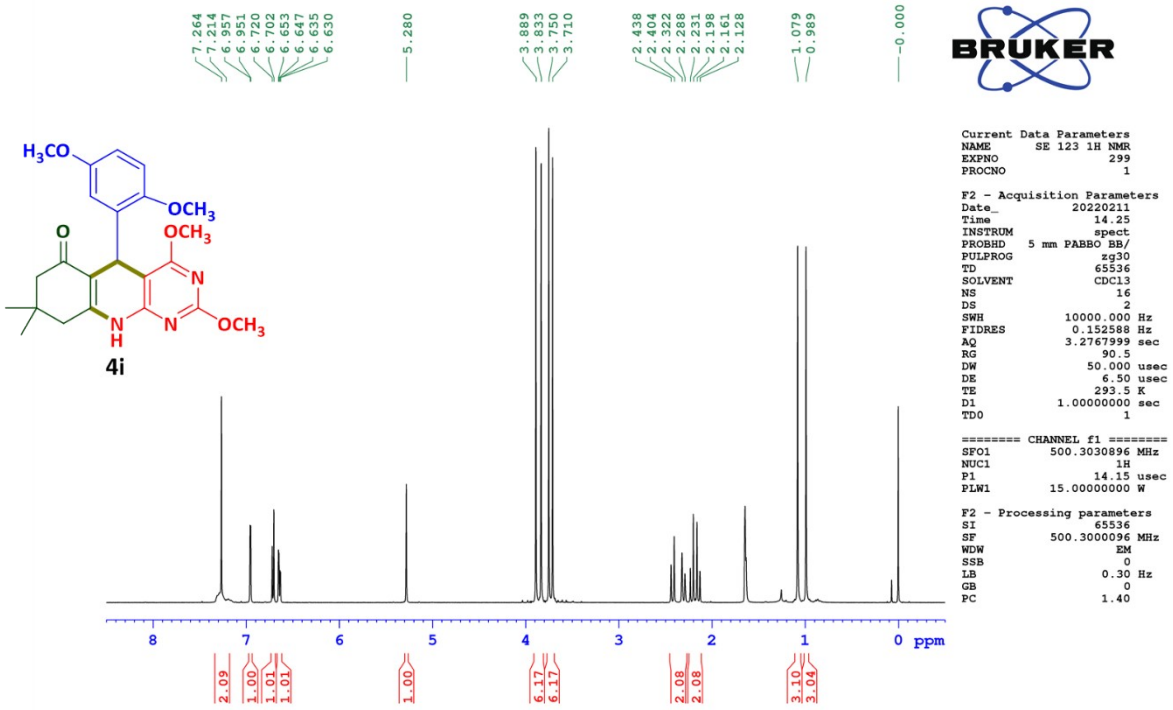

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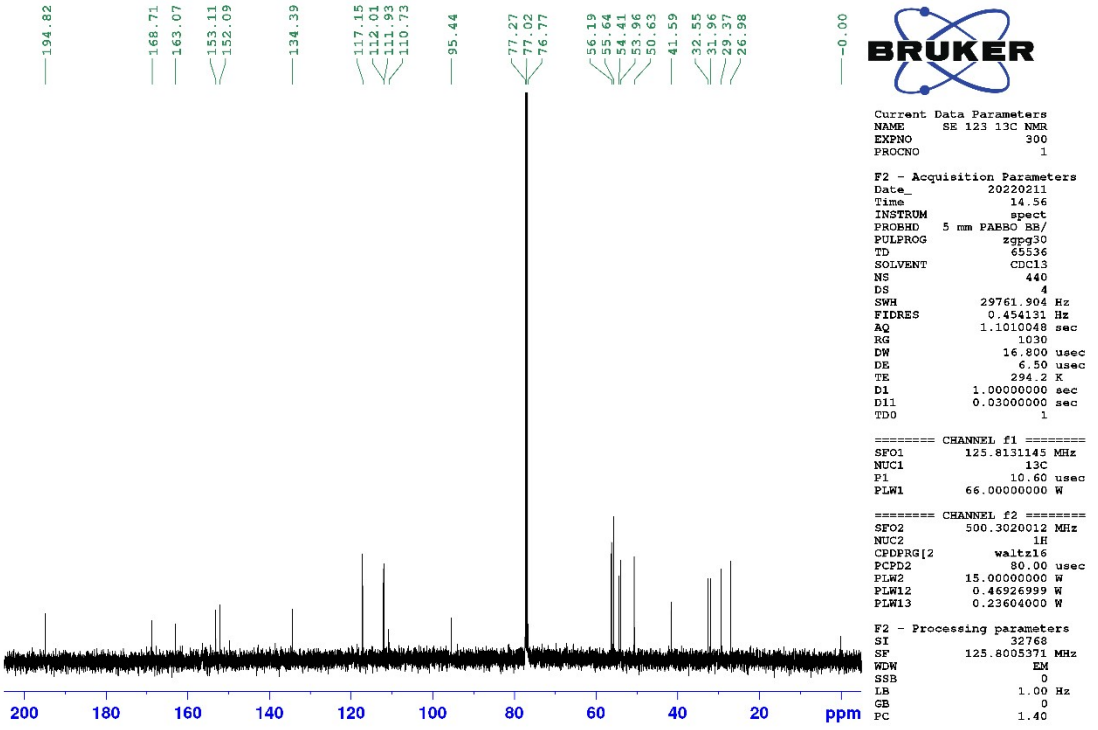

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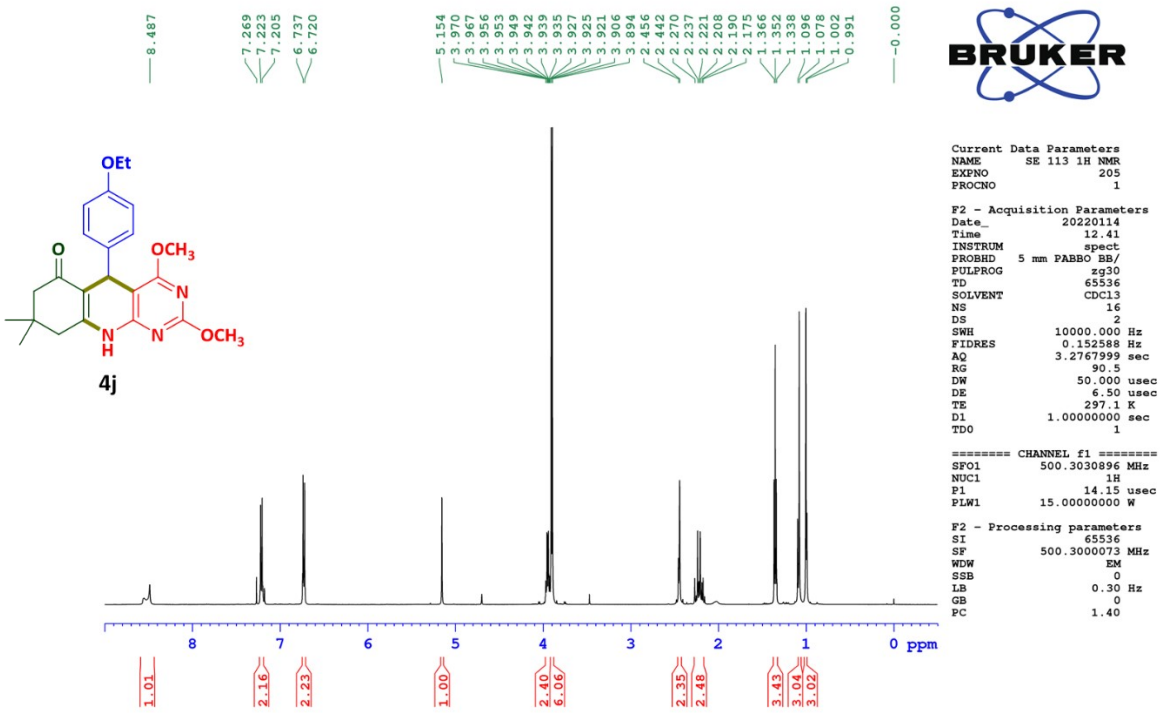

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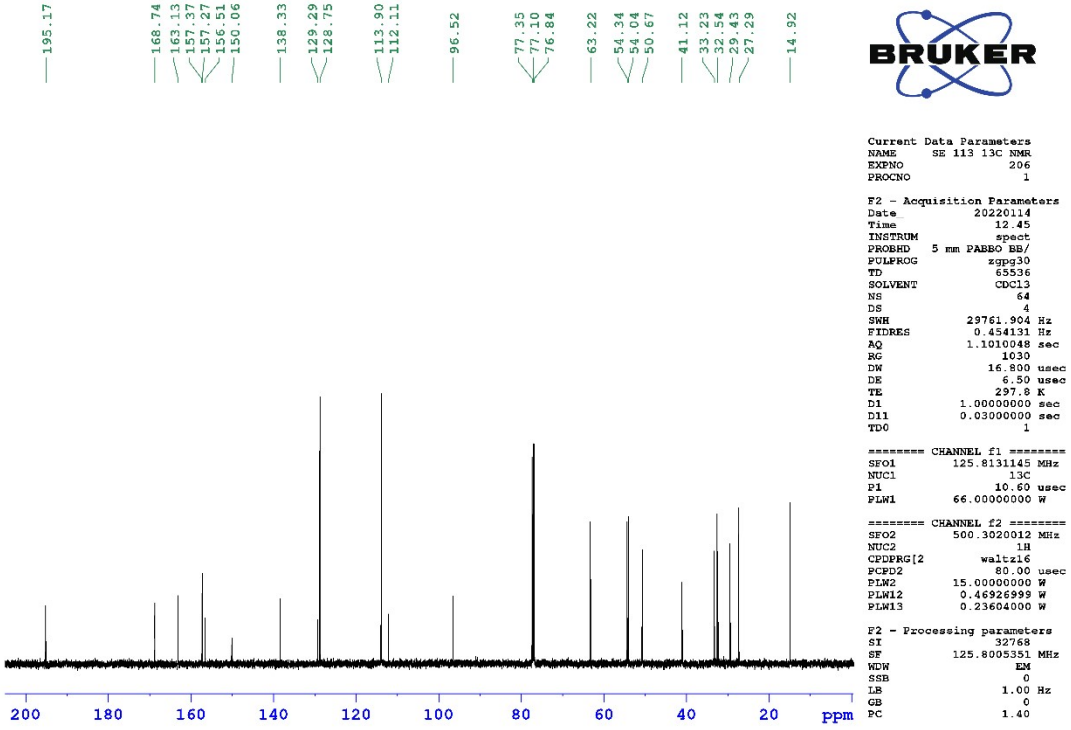

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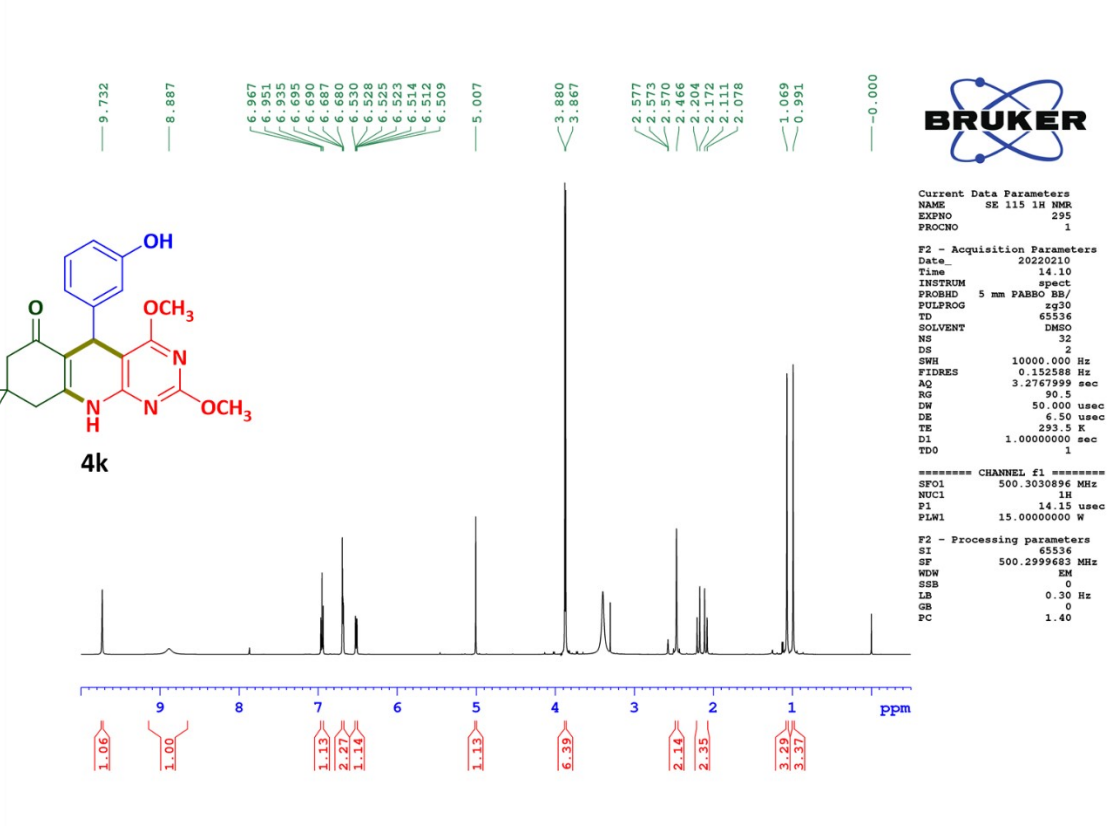

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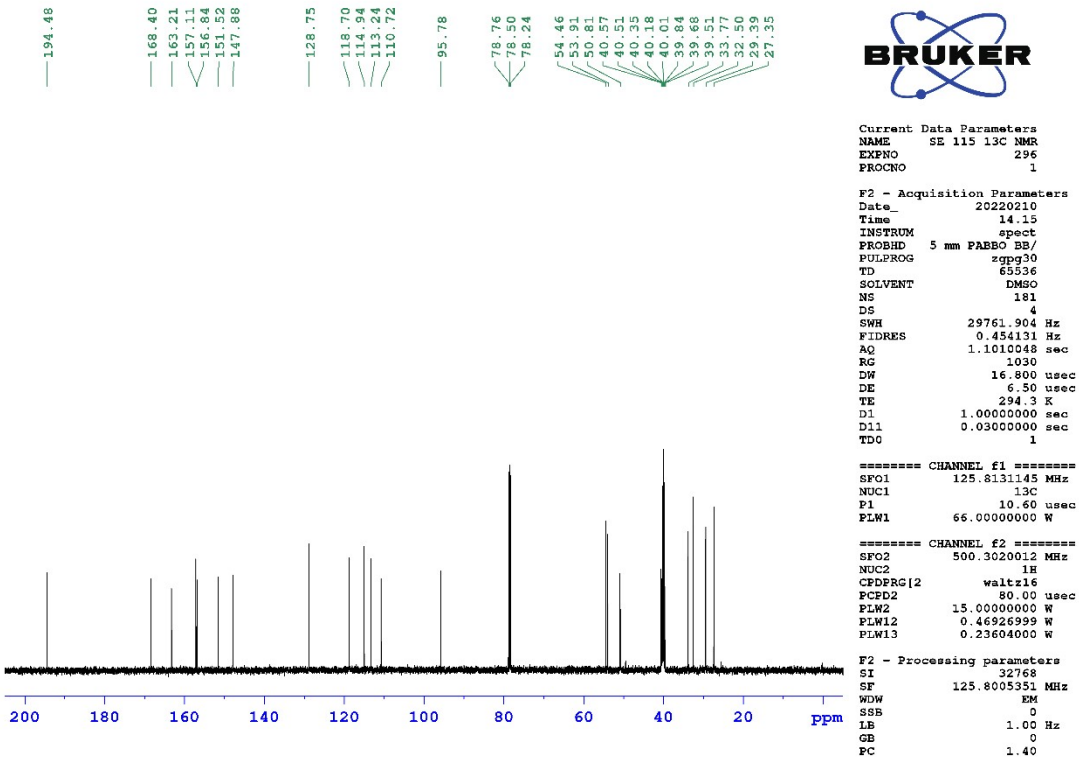

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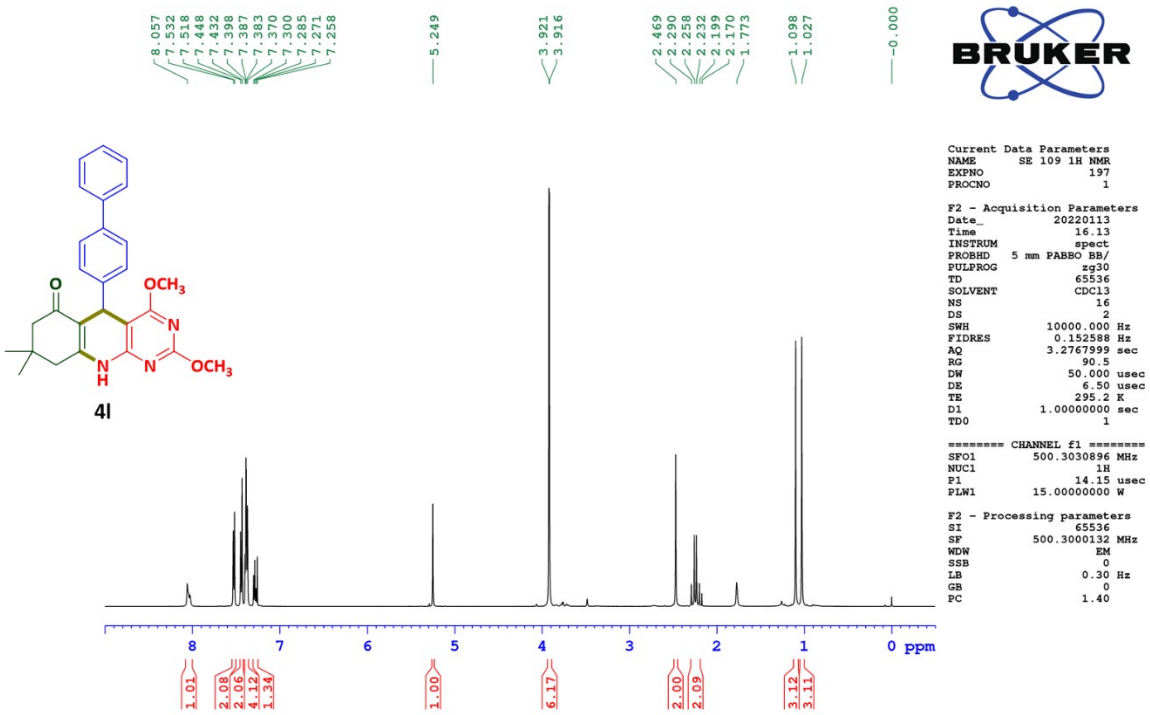

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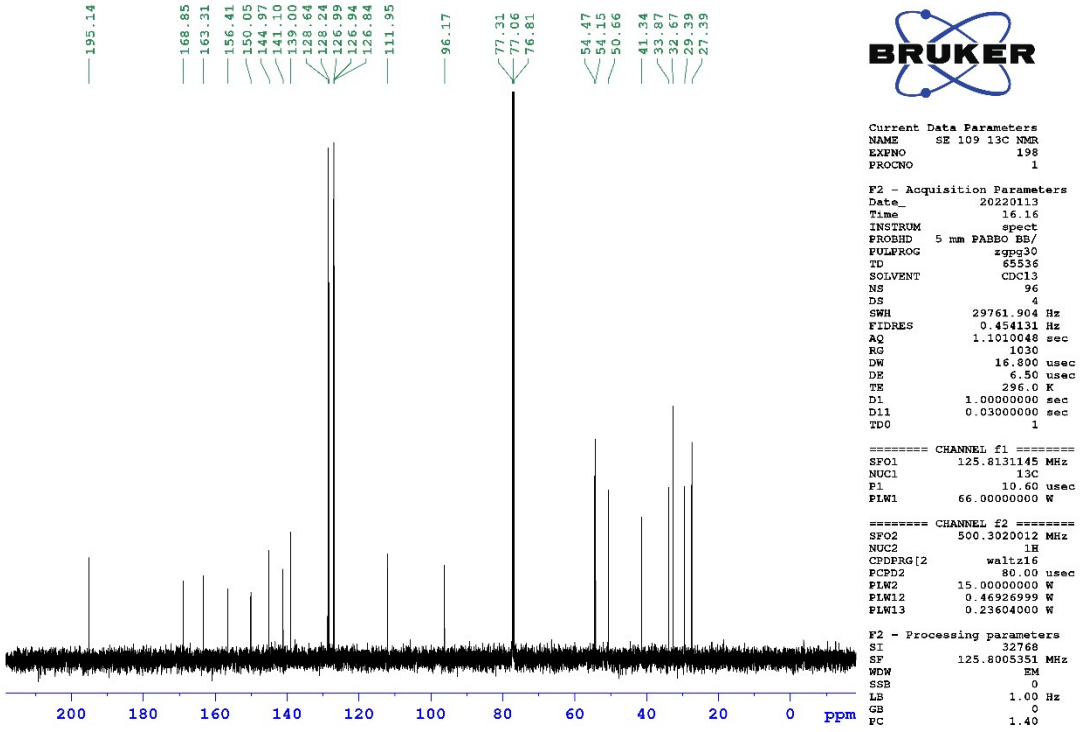

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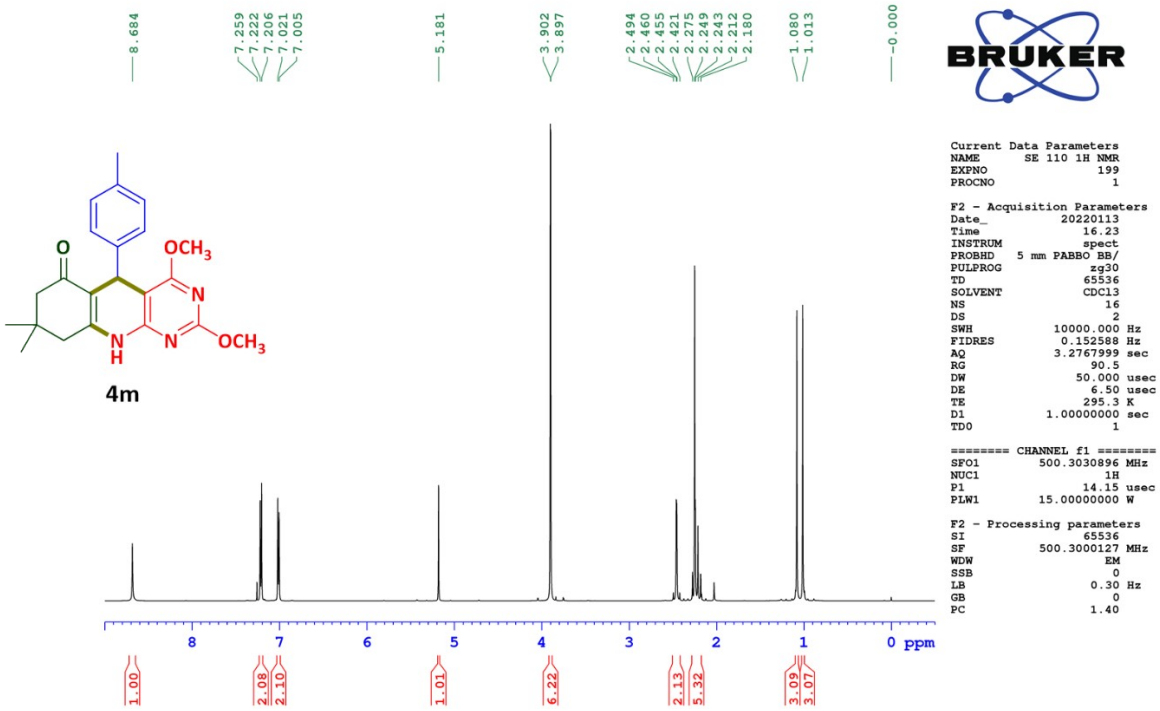

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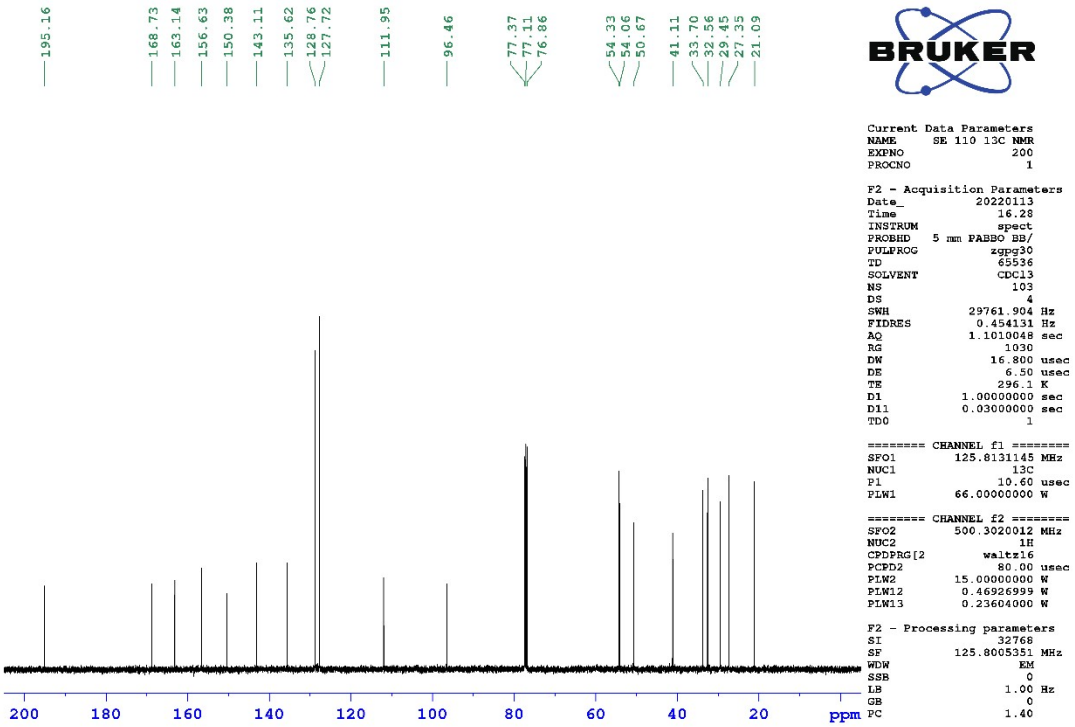

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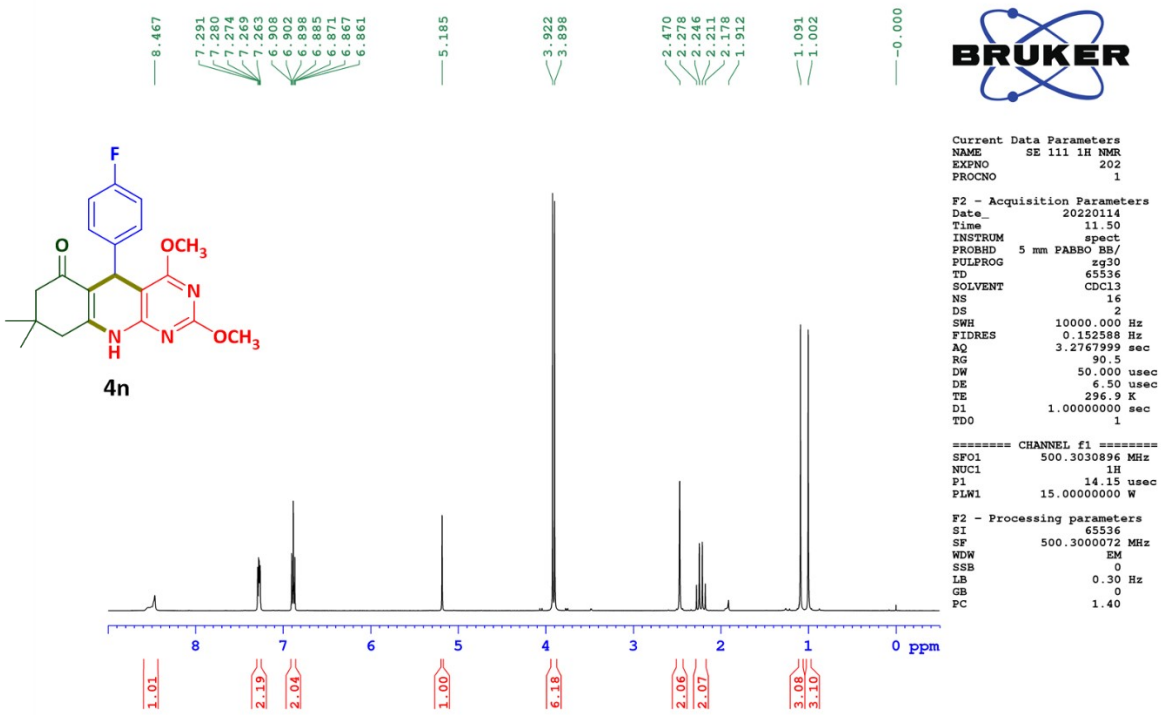

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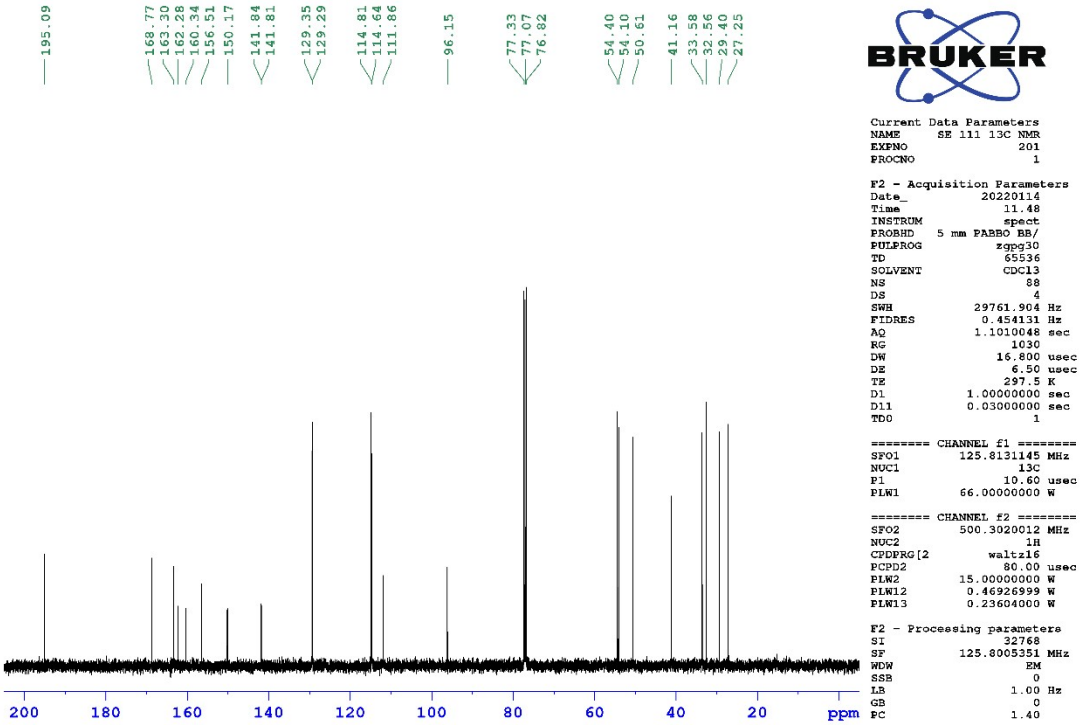

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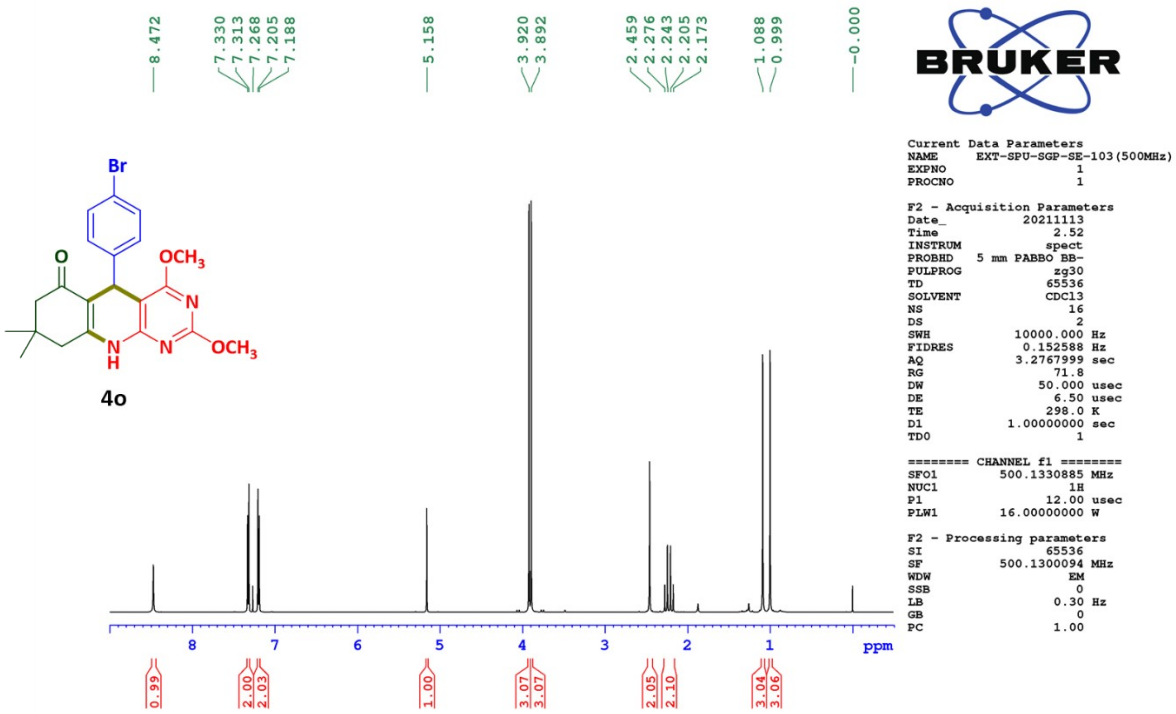

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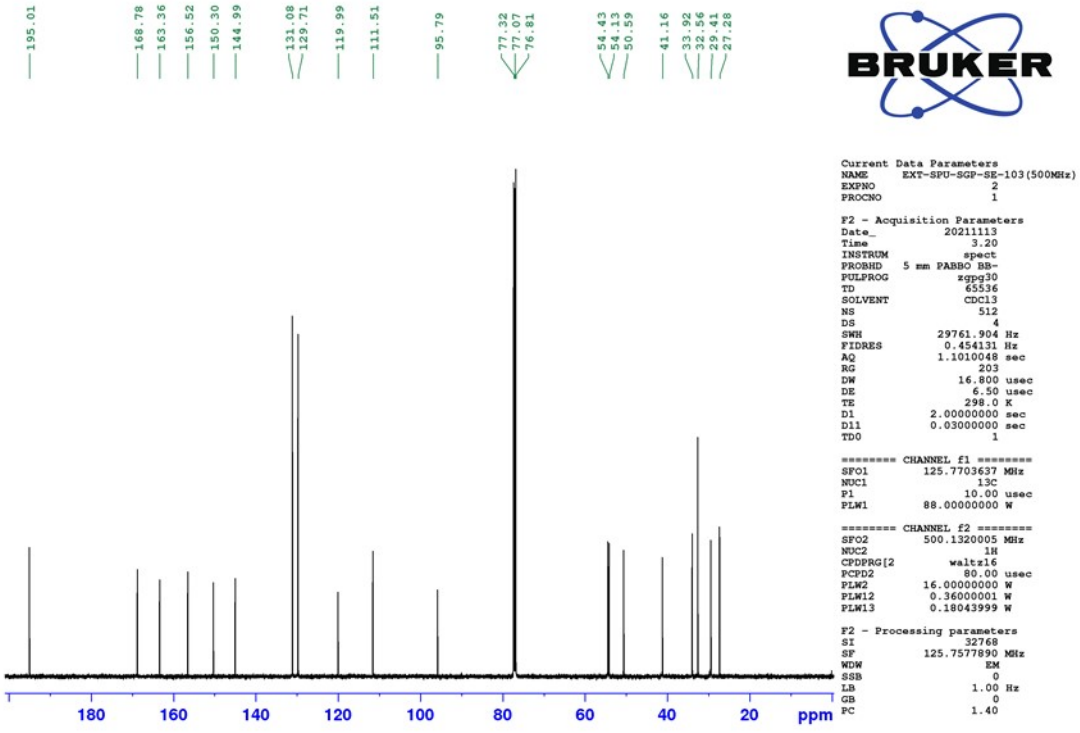

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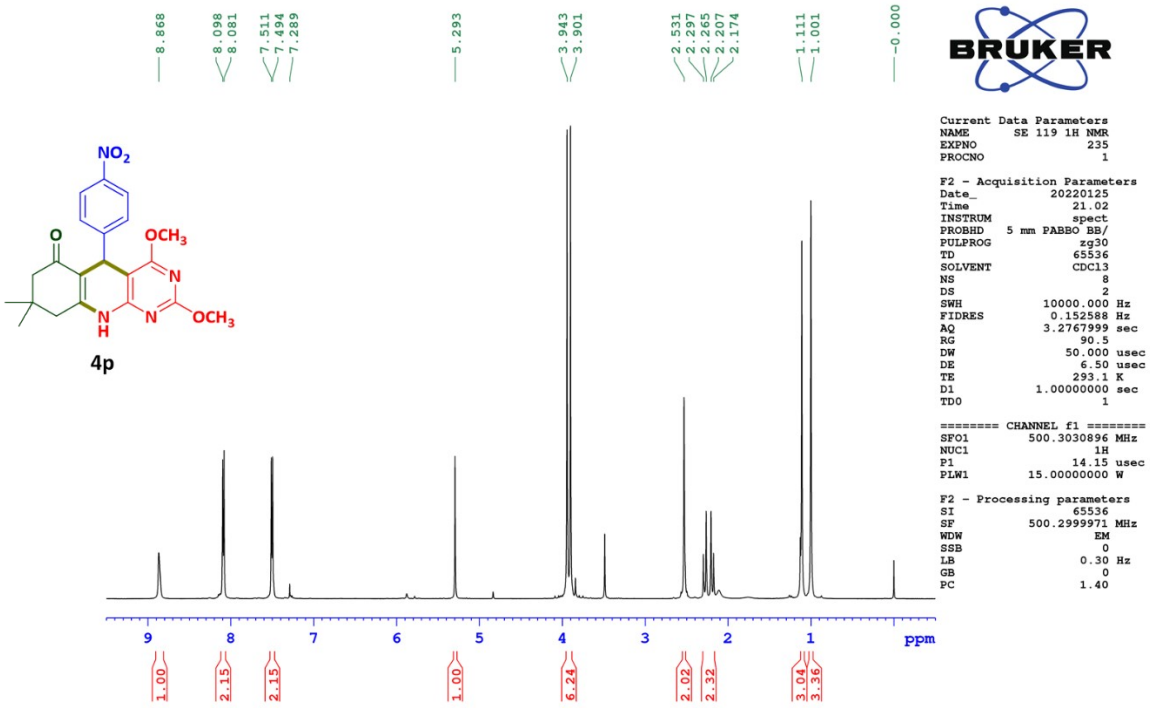

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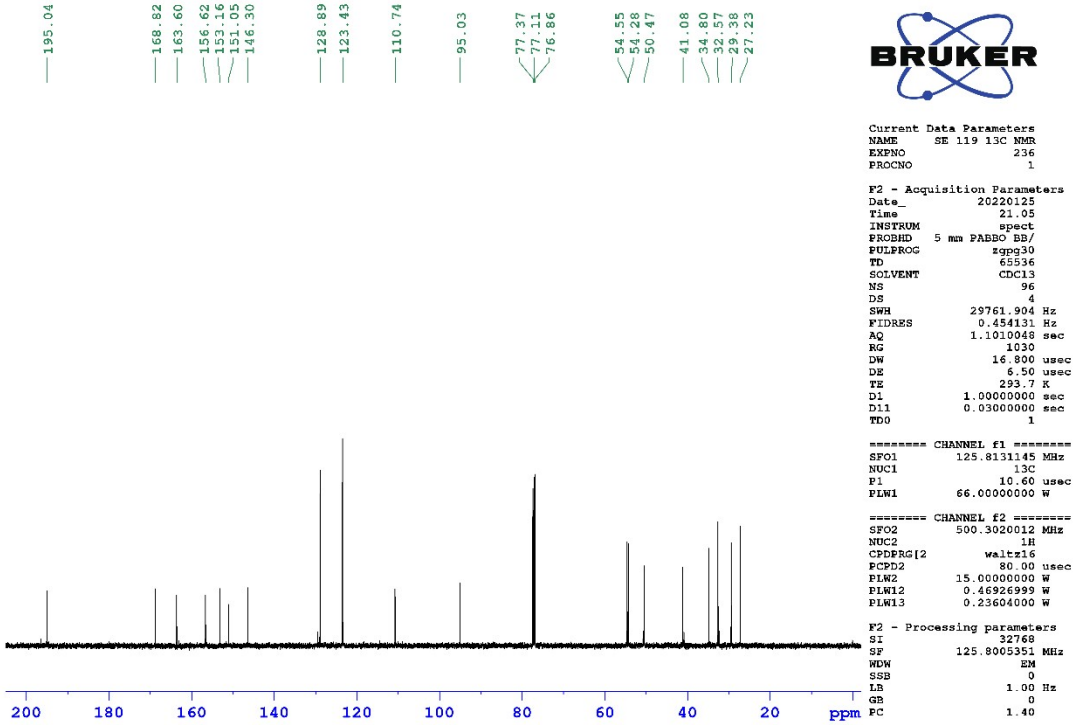

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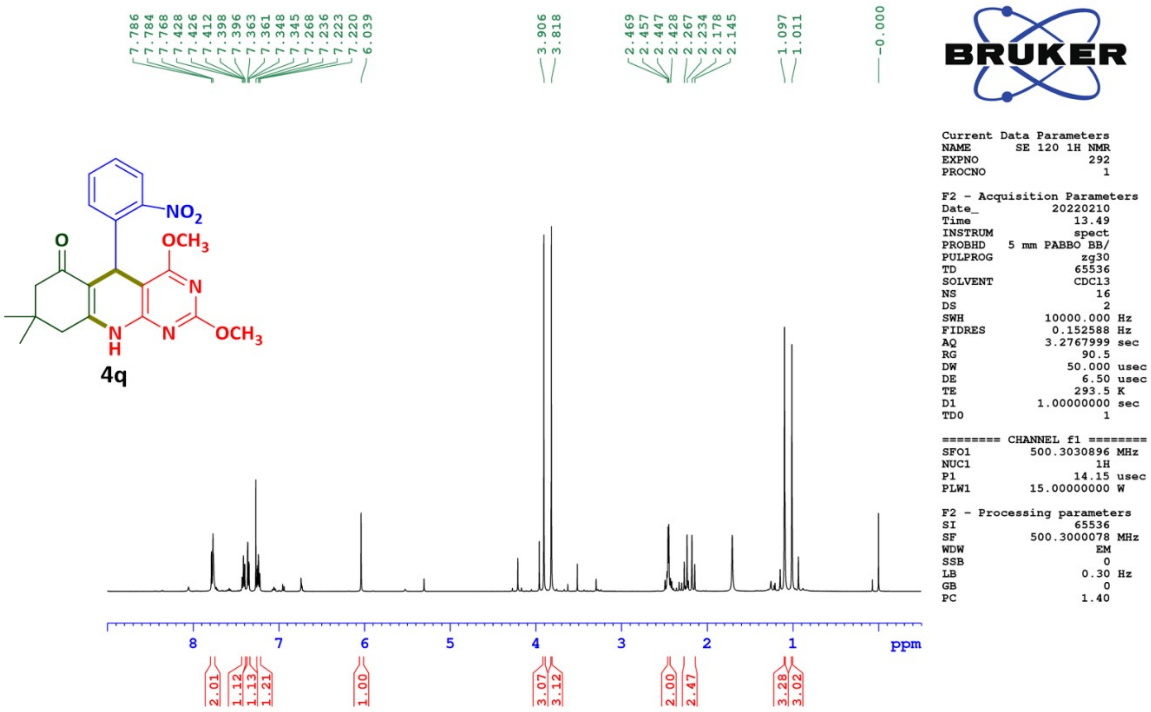

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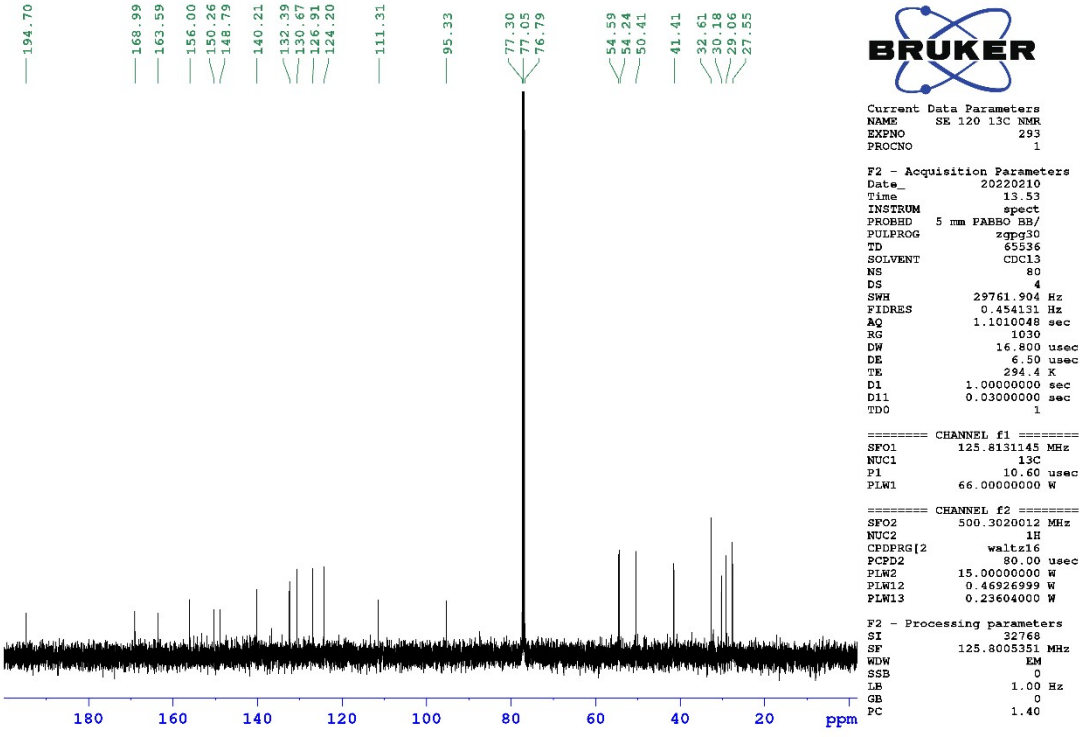

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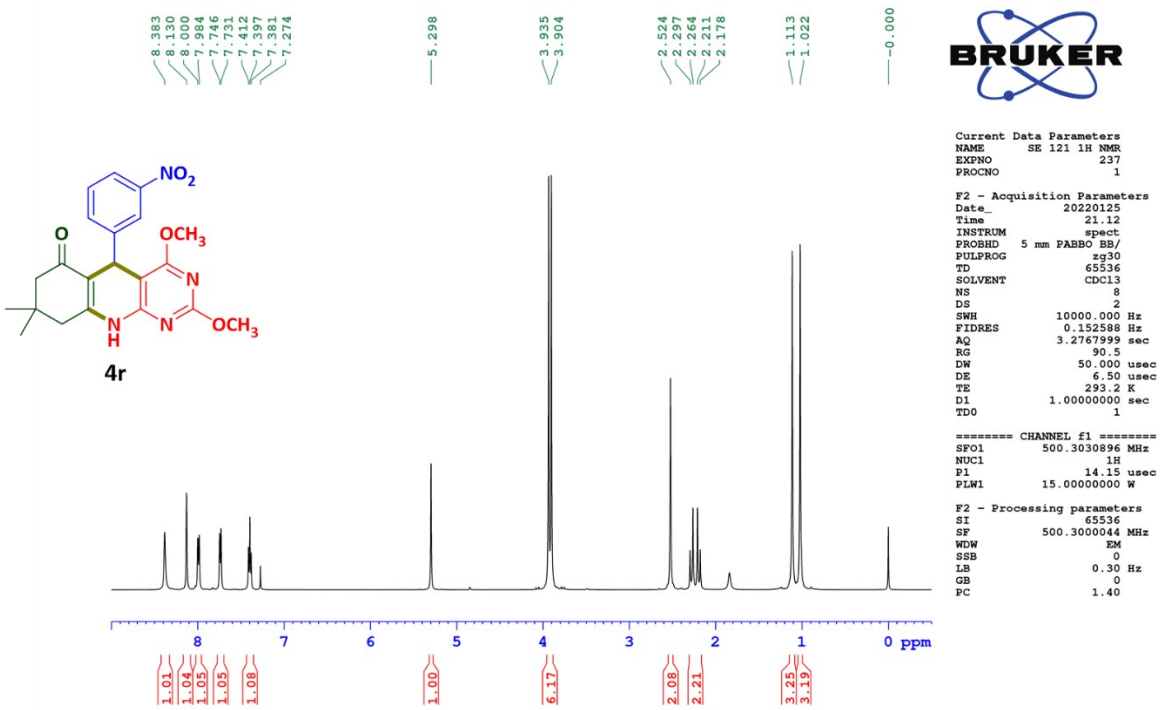

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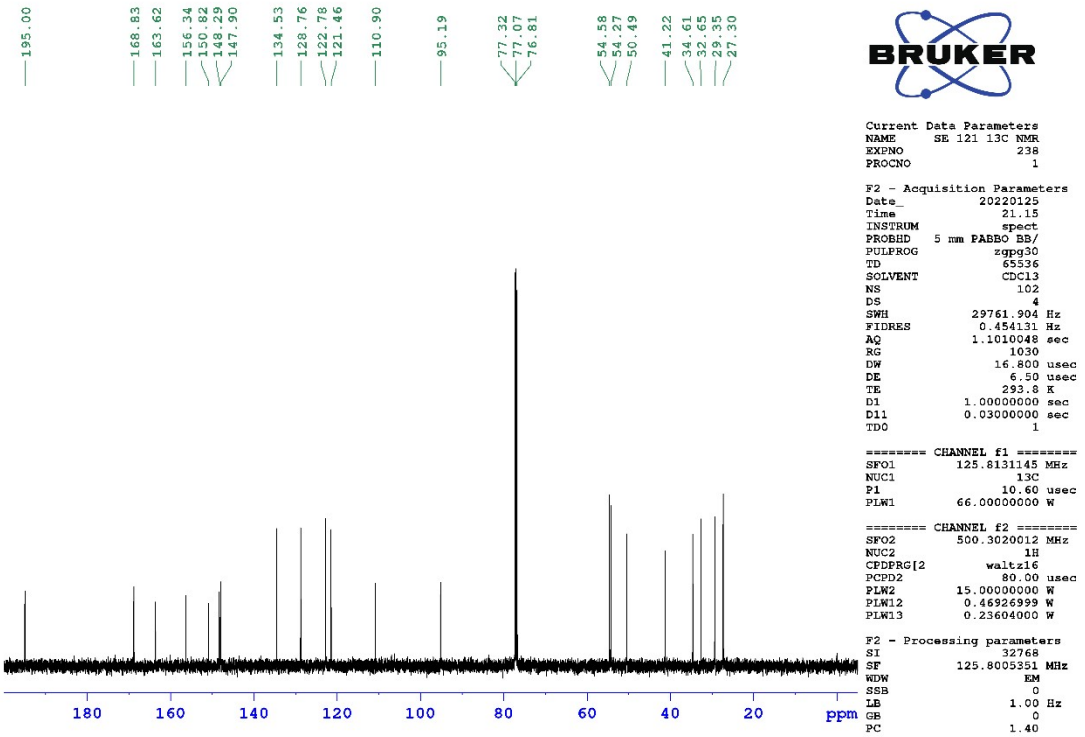

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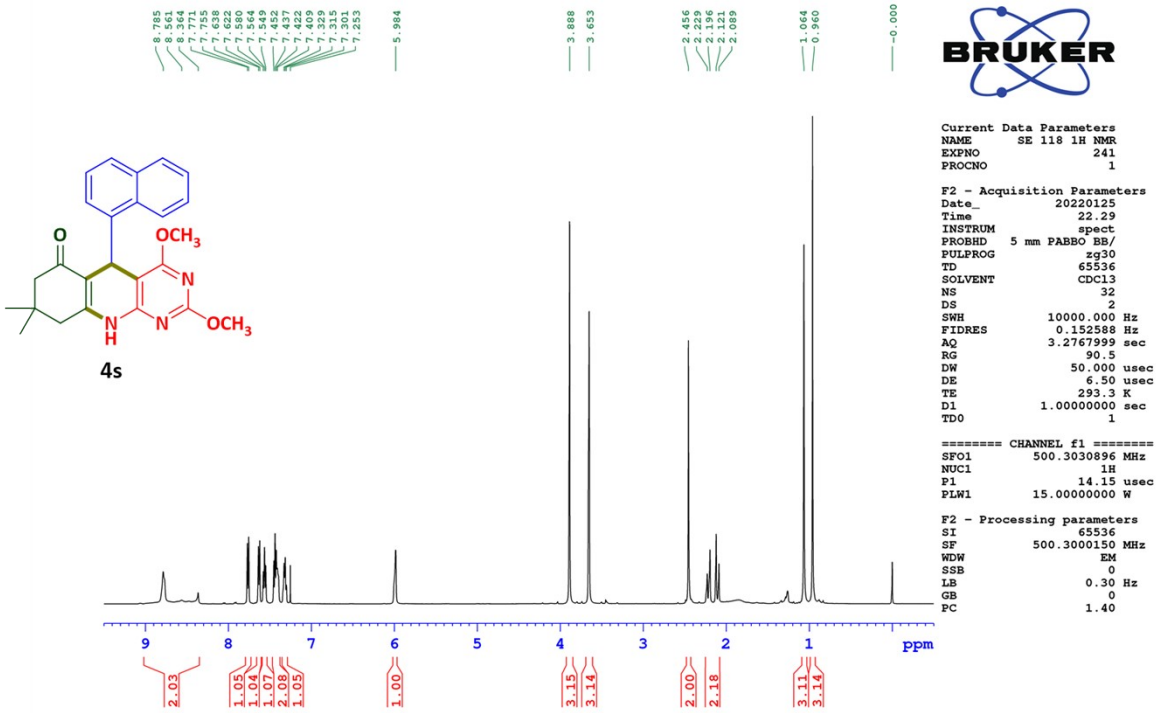

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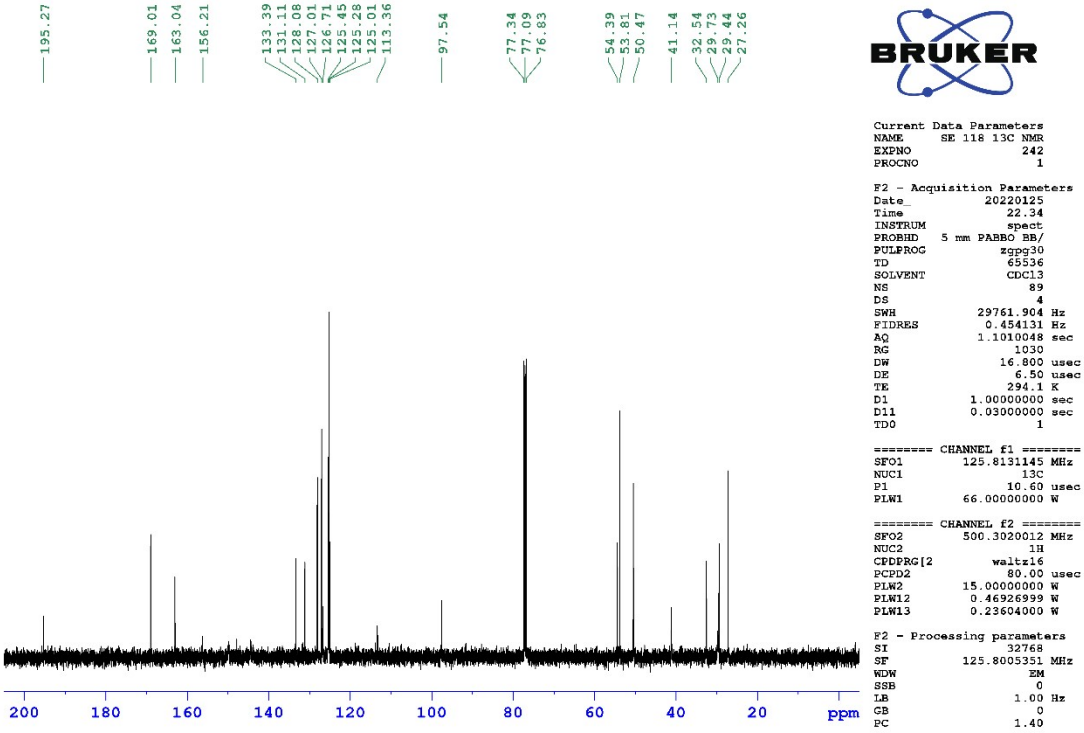

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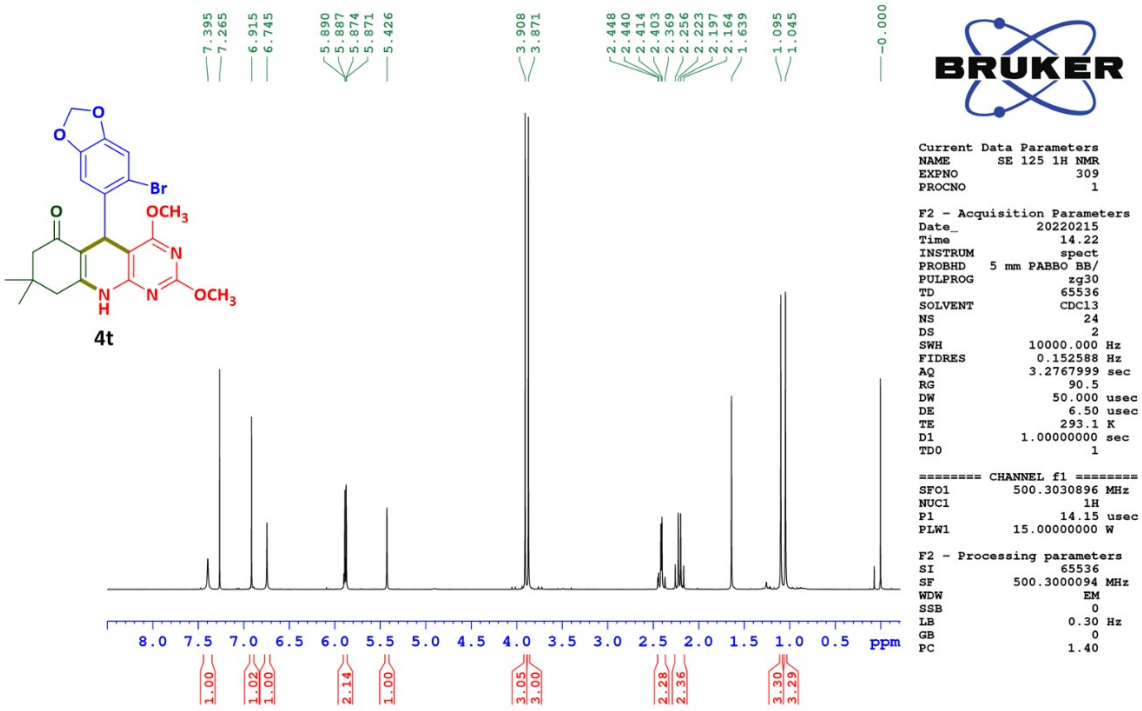

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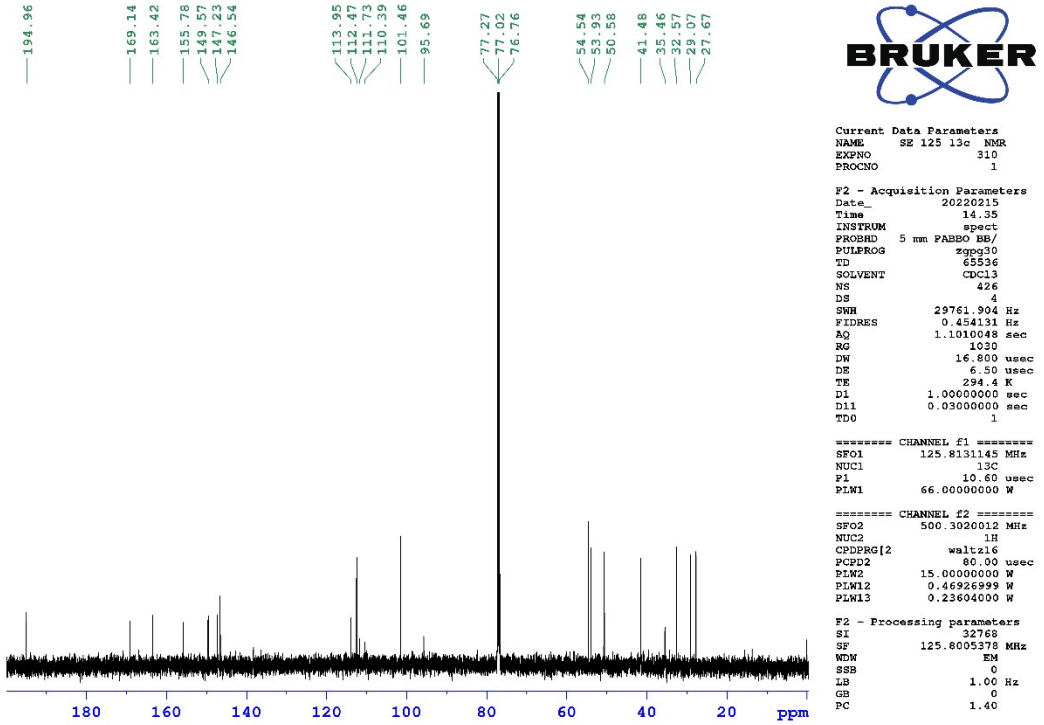

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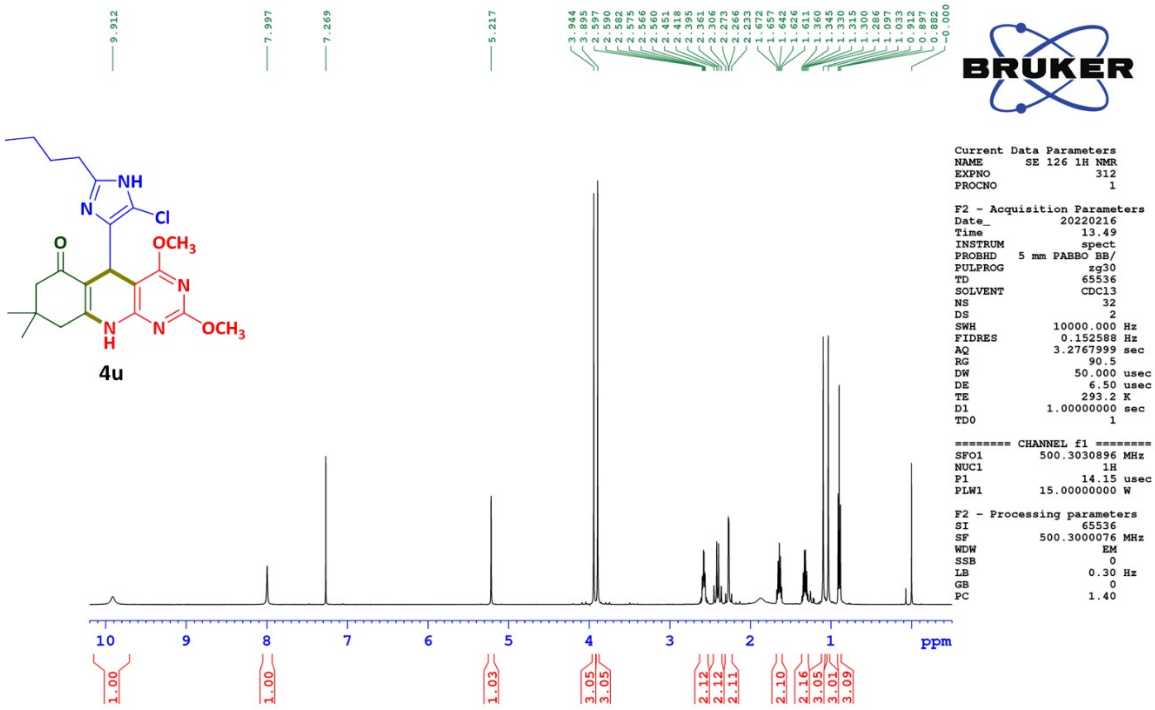


Figure **S42:** ^{13}C-NMR (125 MHz, $CDCl_3$) of Compound **4u.**

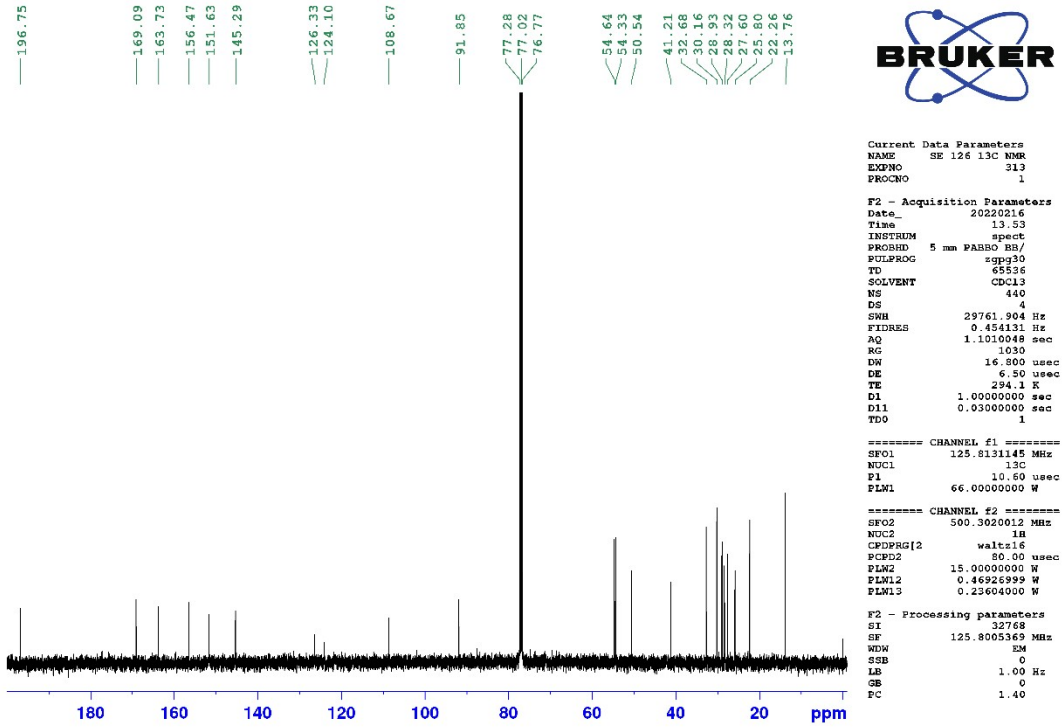


Figure **S43:** ^{1}H-NMR (500 MHz, $CDCl_3$) of Compound **4v.**

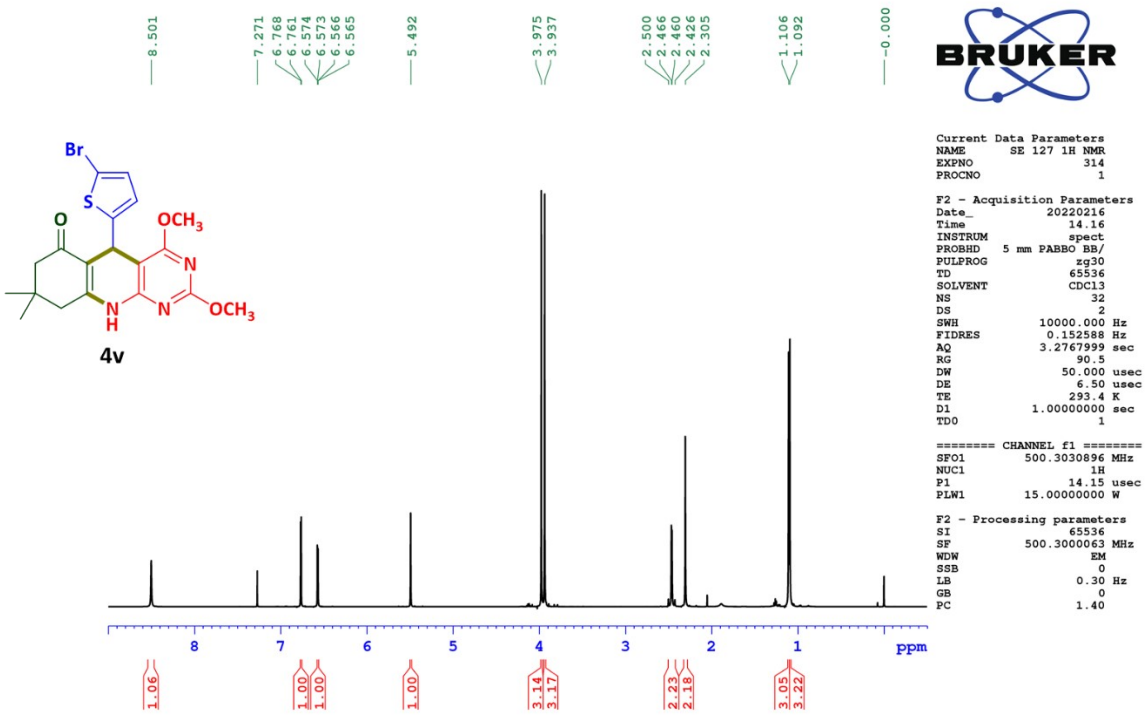


Figure **S44:** ^{13}C-NMR (125 MHz, $CDCl_3$) of Compound **4v.**

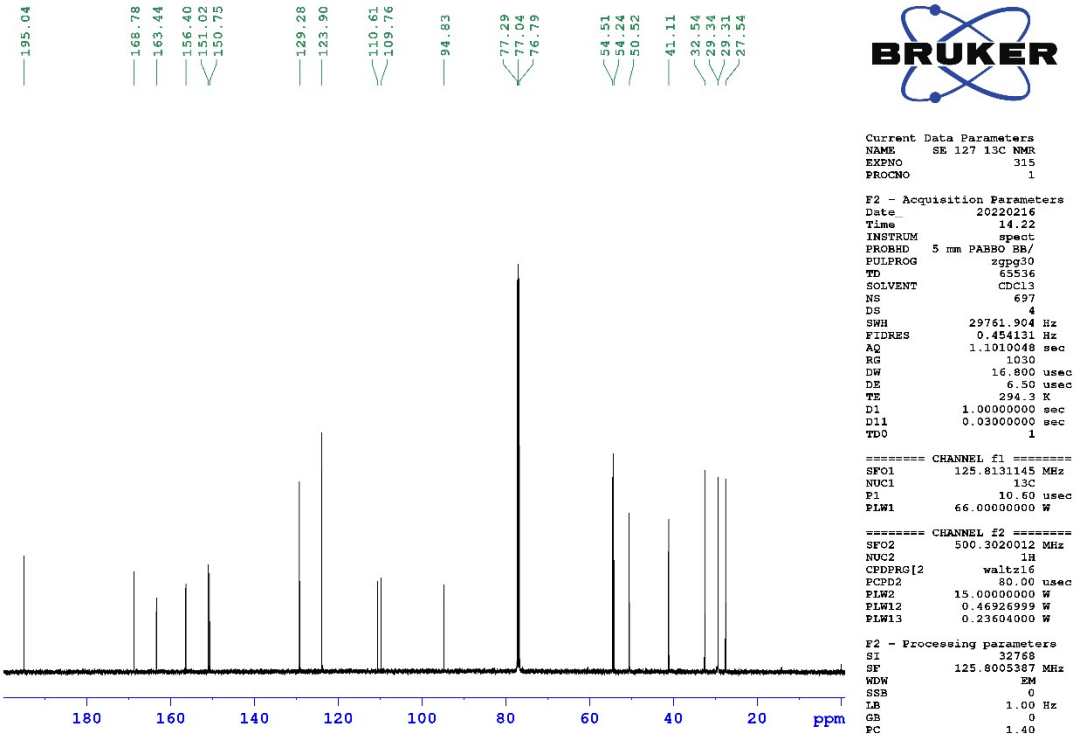


Figure **S45:** HRMS of Compound **4a.**

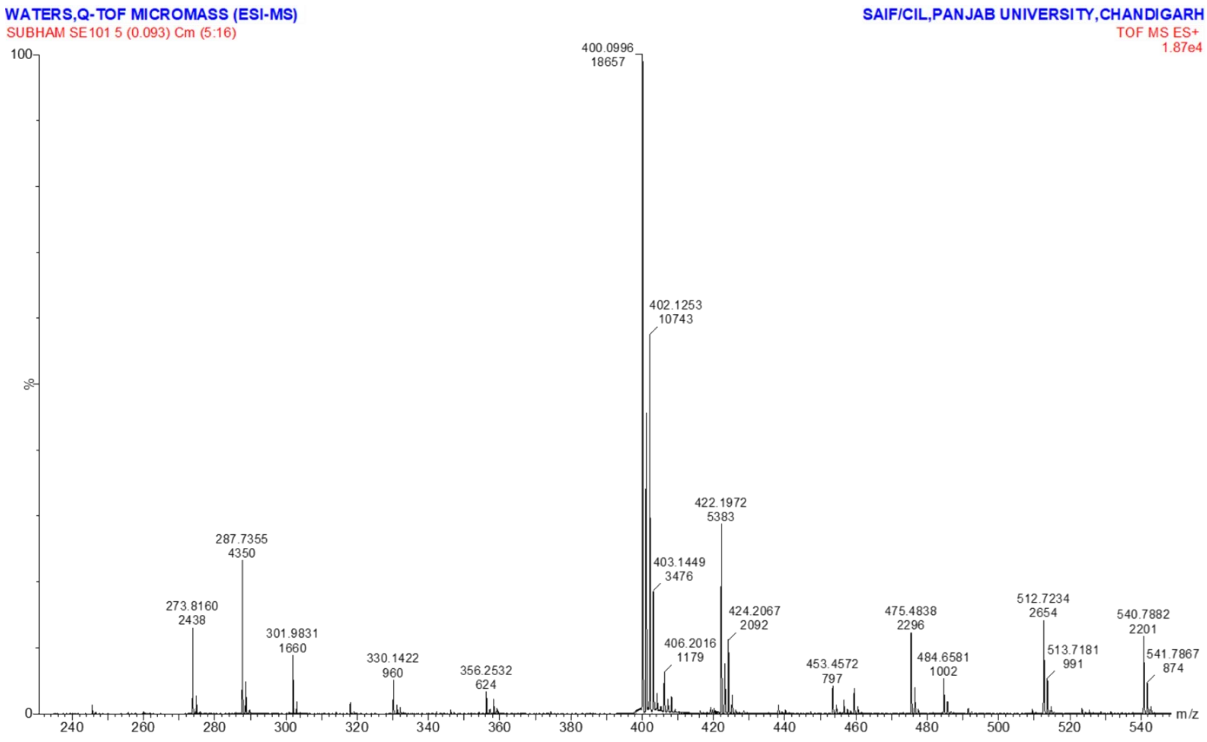


Figure **S46:** LCMS of Compound **4b.**

Retention Time (MS)	MS Area	Mol. Weight or Ion
5.244	550930	365.20 I
		364.20 I

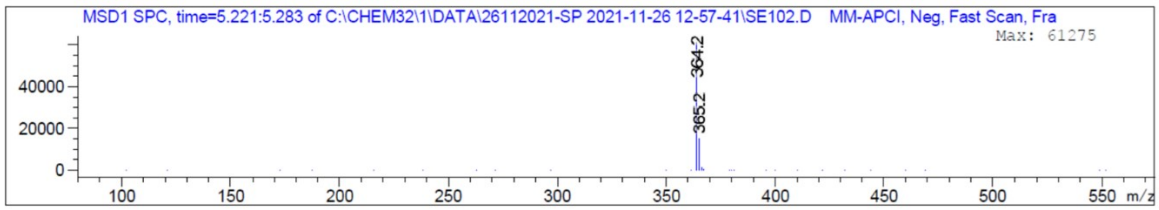


Figure **S47:** HRMS of Compound **4c.**

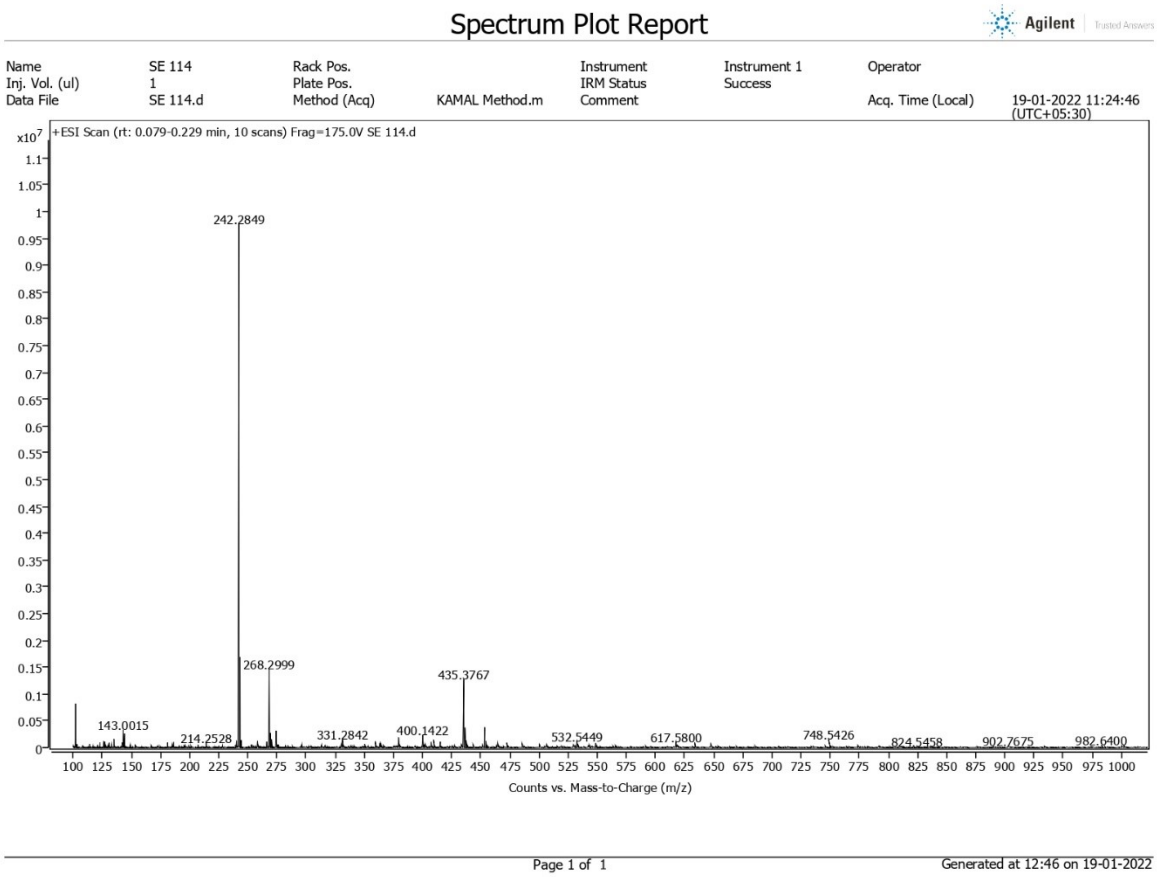


Figure **S48:** LCMS of Compound **4d.**

Retention Time (MS)	MS Area	Mol. Weight or Ion
5.591	1183032	400.20 I
		399.20 I
		398.20 I

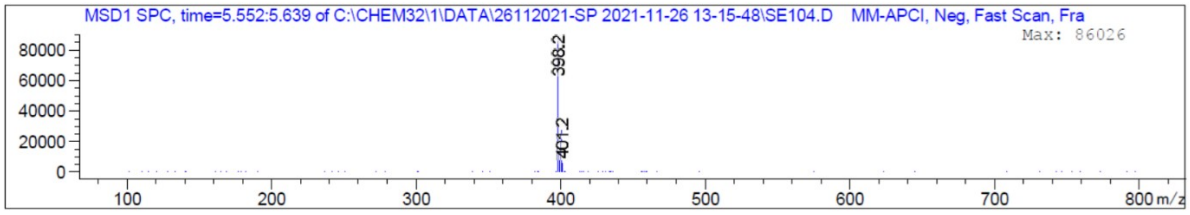


Figure **S49:** LCMS of Compound **4e.**

Retention Time (MS)	MS Area	Mol. Weight or Ion
5.187	942895	395.20 I
		394.20 I

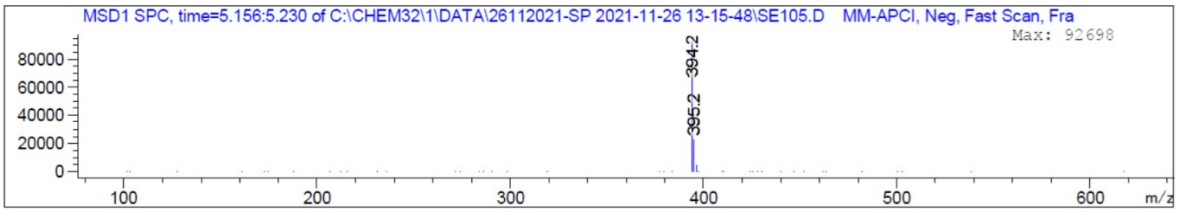


Figure **S50:** LCMS of Compound **4f.**

Retention Time (MS)	MS Area	Mol. Weight or Ion
5.152	469870	395.20 I
		394.20 I

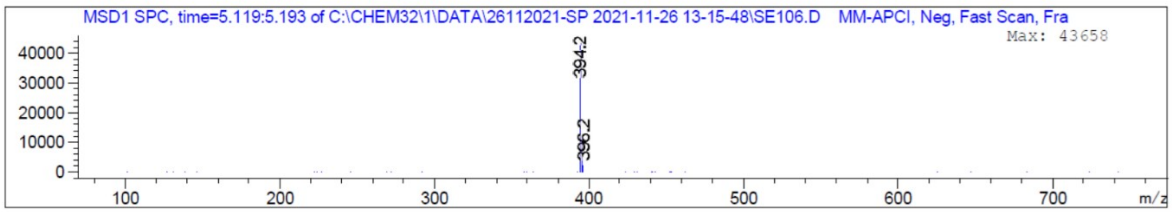


Figure **S51:** HRMS of Compound **4g.**

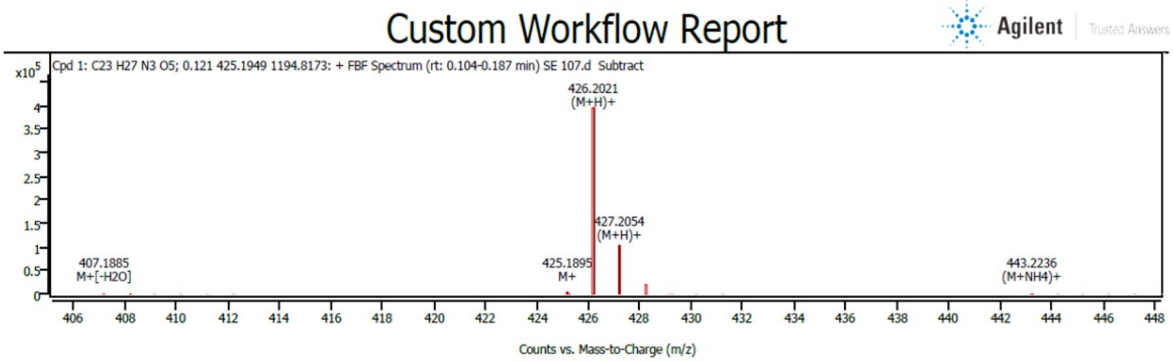


Figure **S52:** HRMS of Compound **4h.**

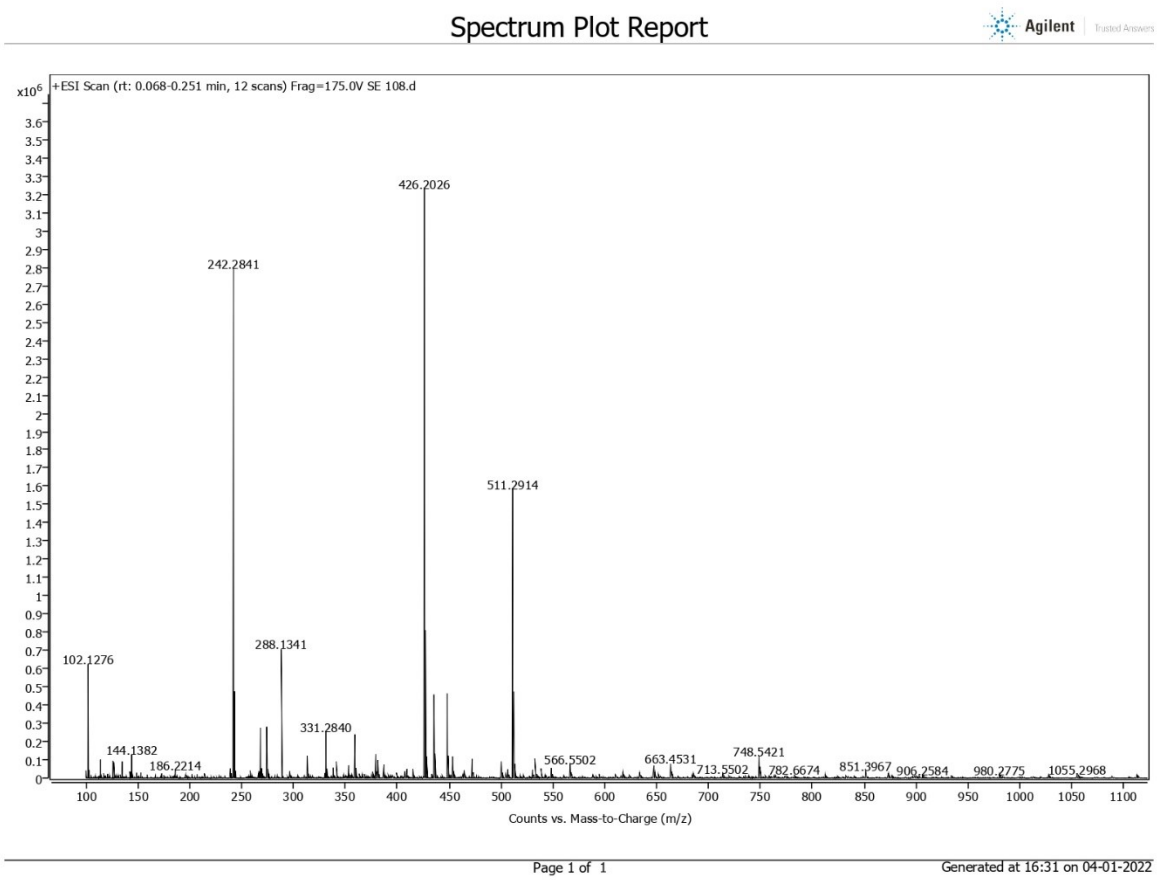


Figure **S53:** HRMS of Compound **4i.**

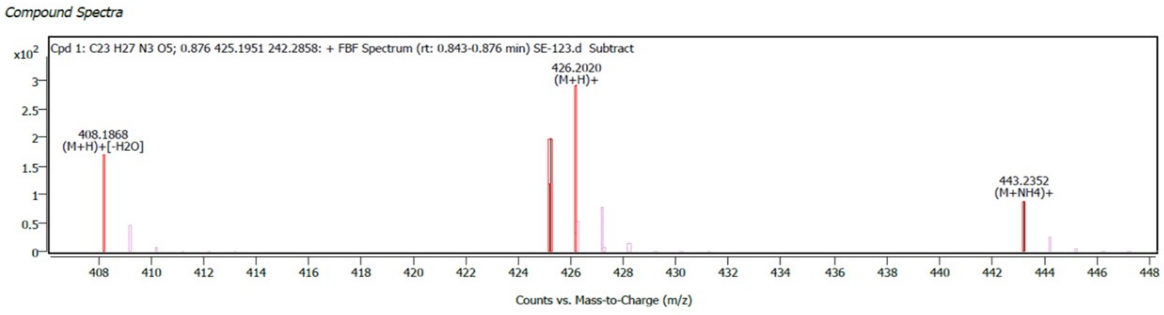


Figure **S54:** HRMS of Compound **4j.**

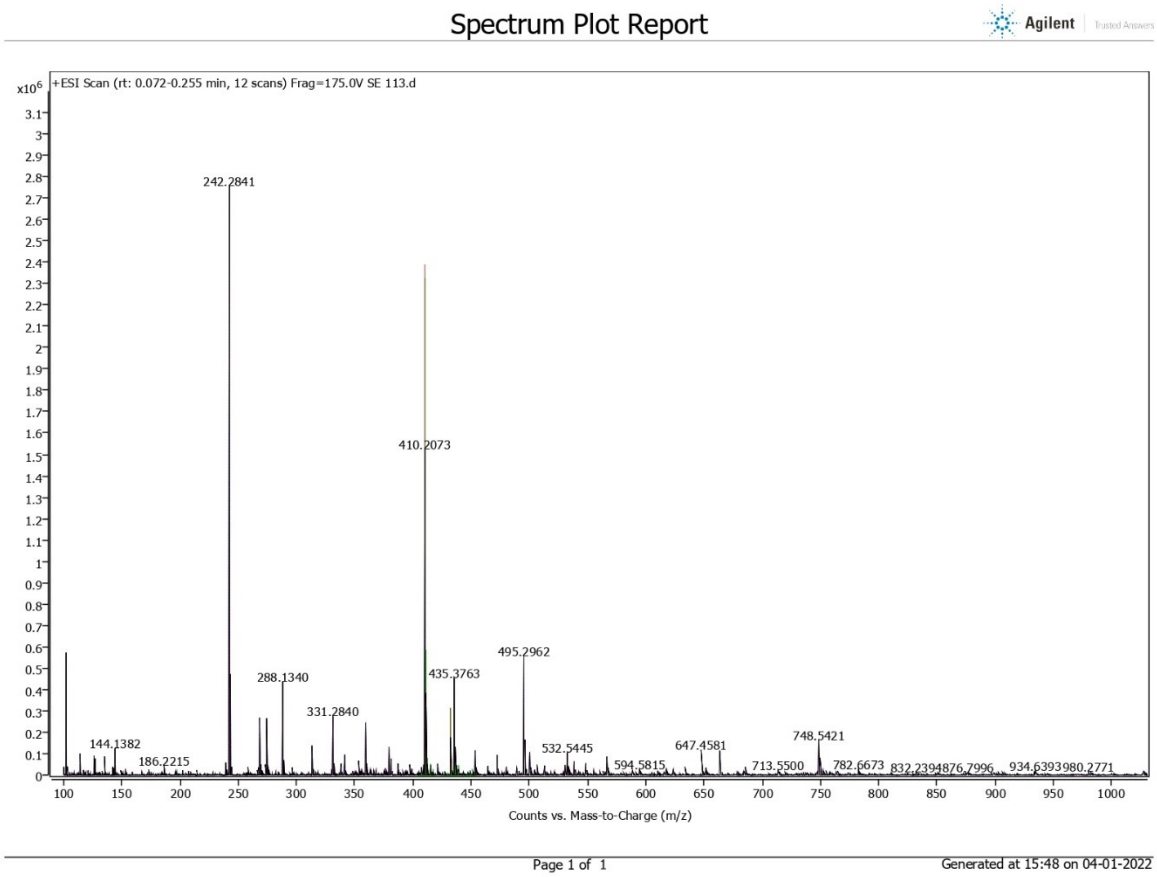


Figure **S55:** HRMS of Compound **4k.**

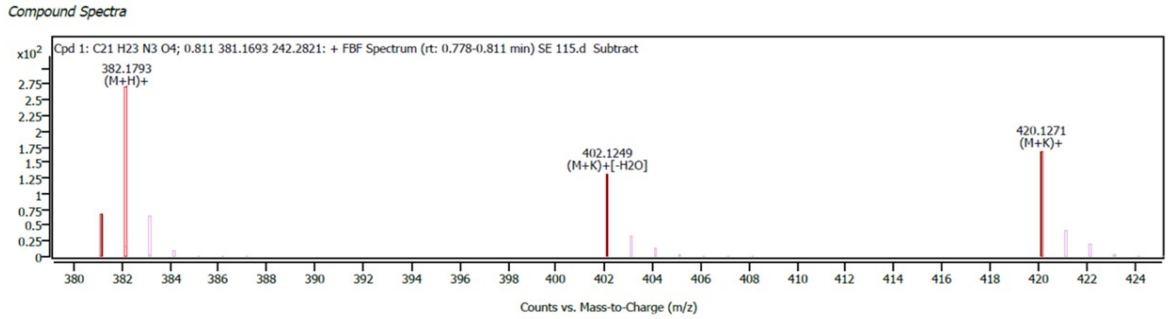


Figure **S56:** HRMS of Compound **4l.**

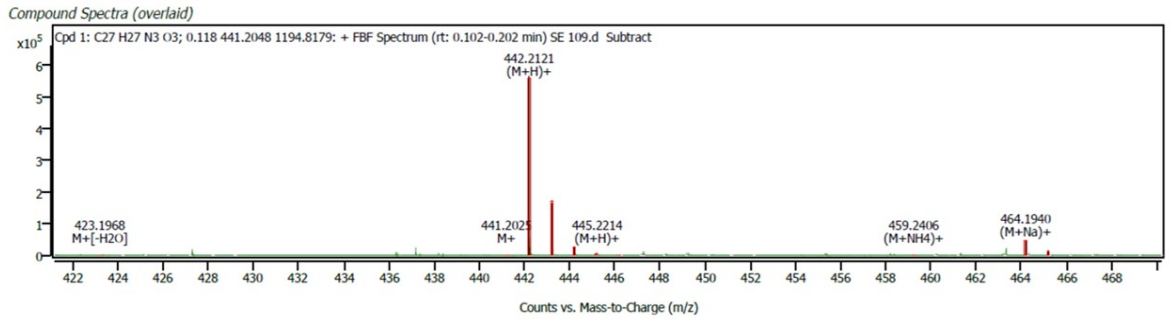


Figure **S57:** HRMS of Compound **4m.**

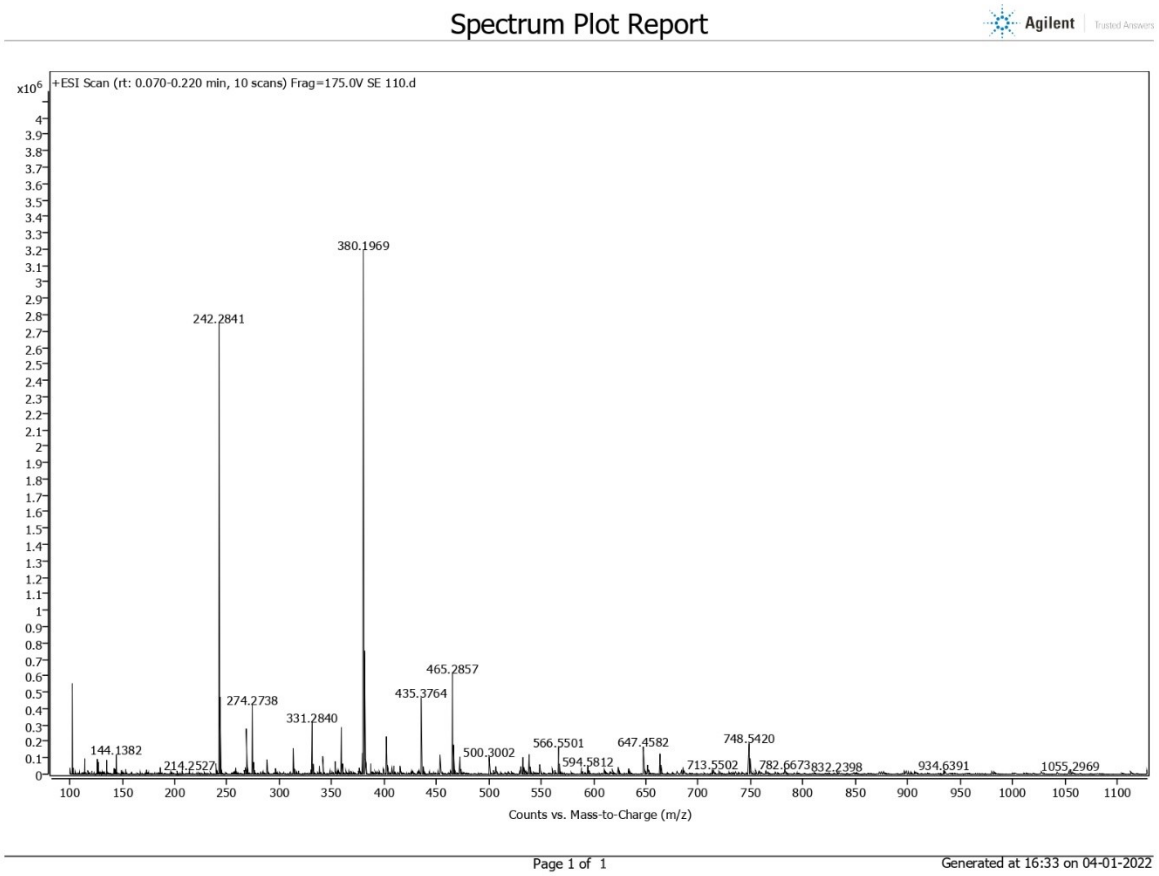


Figure **S58:** HRMS of Compound **4n.**

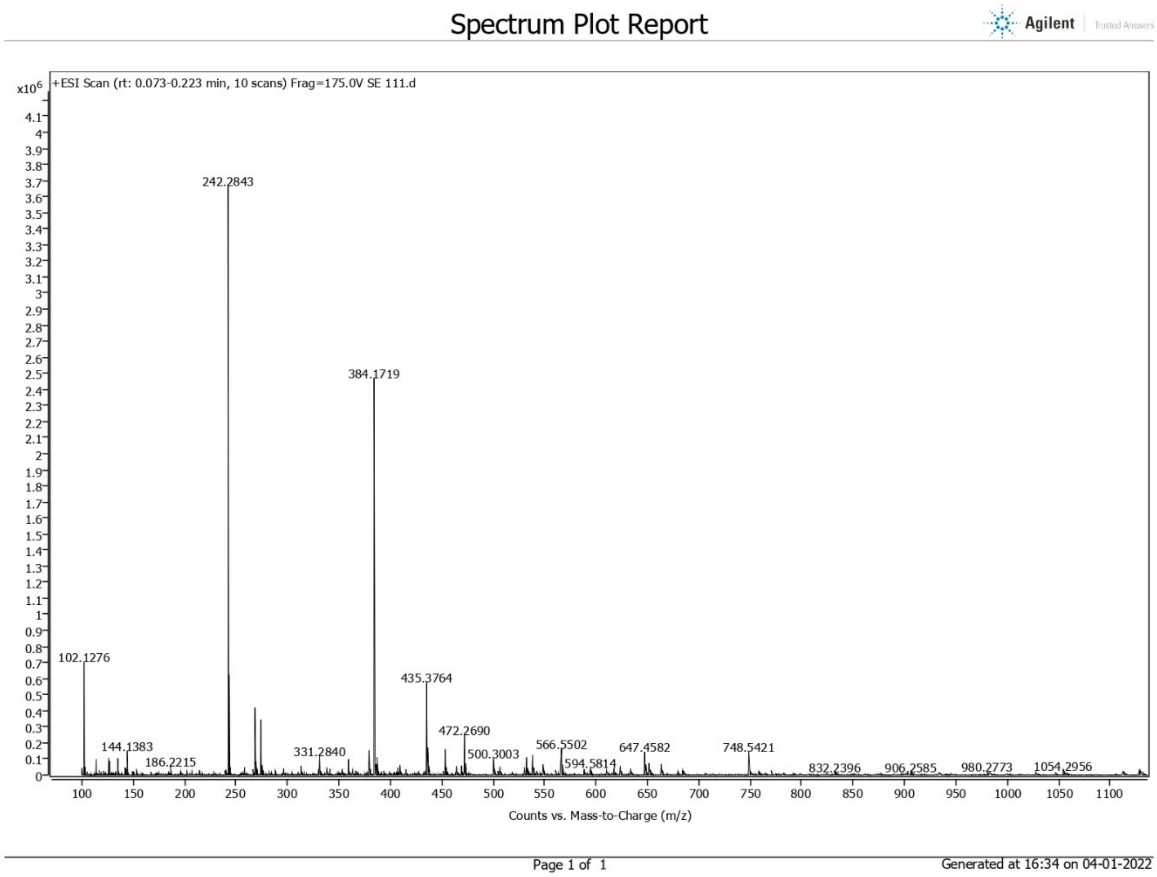


Figure **S59:** LCMS of Compound **4o.**

Retention Time (MS)	MS Area	Mol. Weight or Ion
5.694	3213958	445.20 I
		445.00 I
		444.20 I
		444.00 I
		443.20 I
		442.20 I
		442.00 I

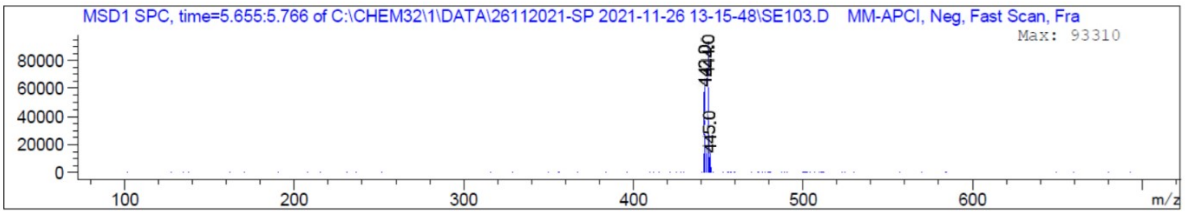


Figure **S60:** HRMS of Compound **4p.**

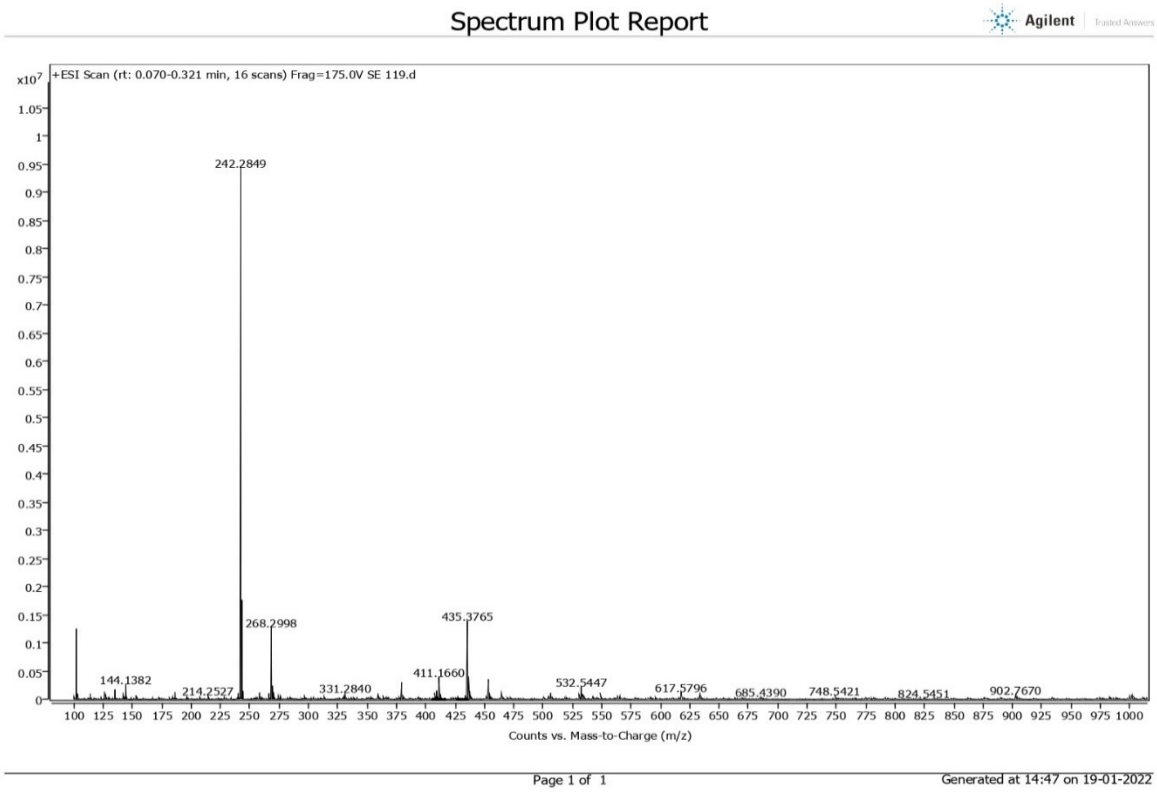


Figure **S61:** HRMS of Compound **4q.**

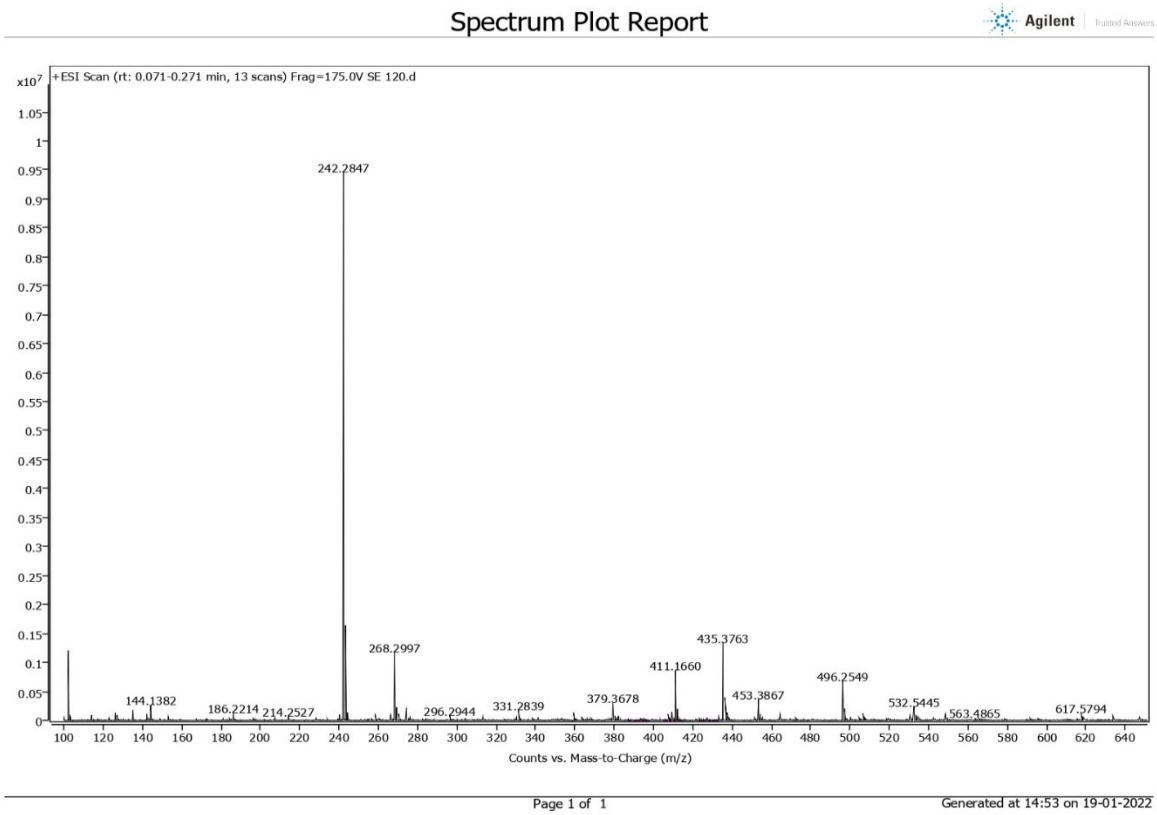


Figure **S62:** HRMS of Compound **4r.**

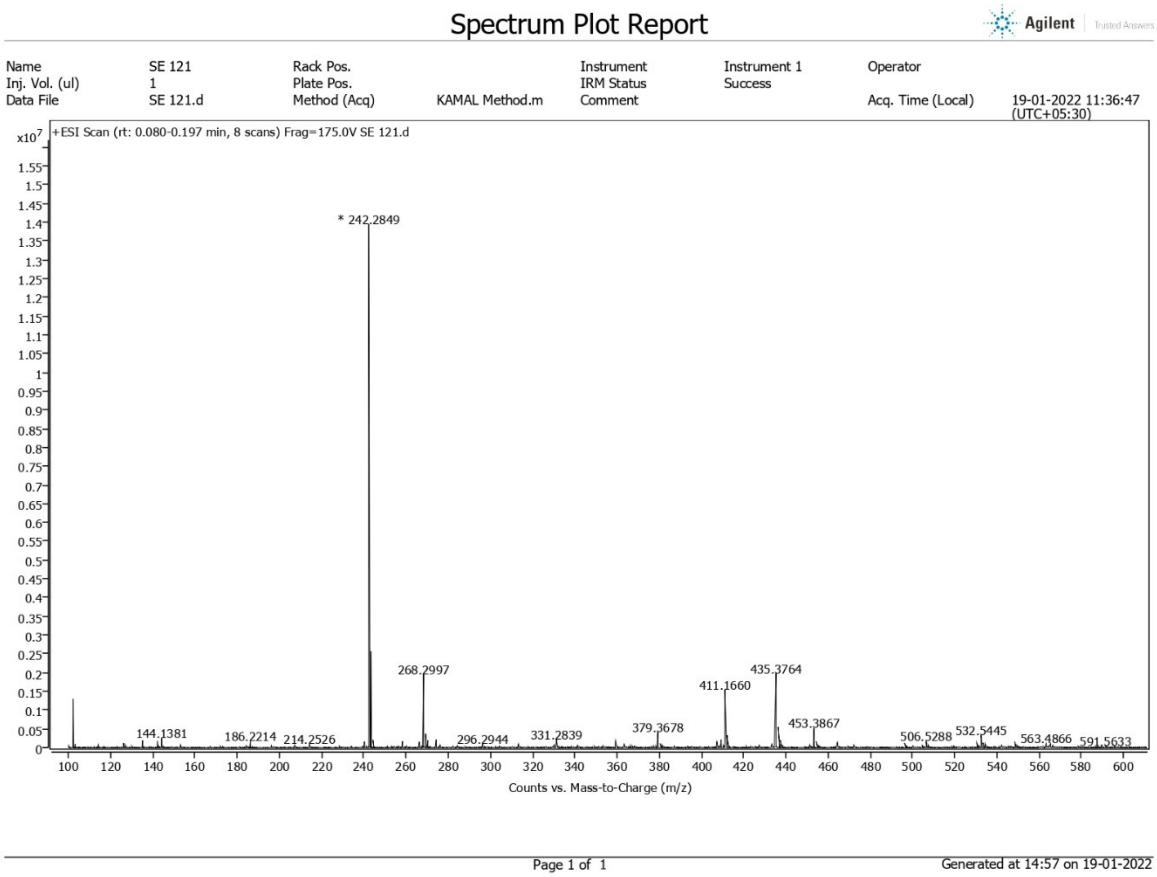


Figure **S63:** HRMS of Compound **4s.**

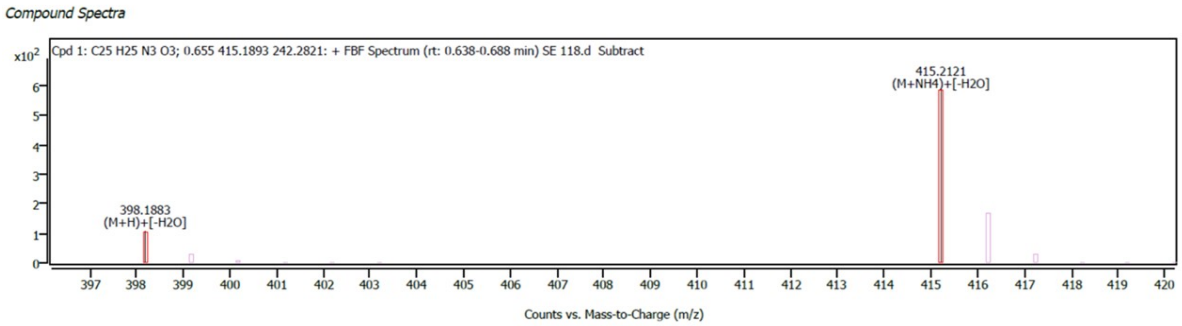


Figure **S64:** HRMS of Compound **4t.**

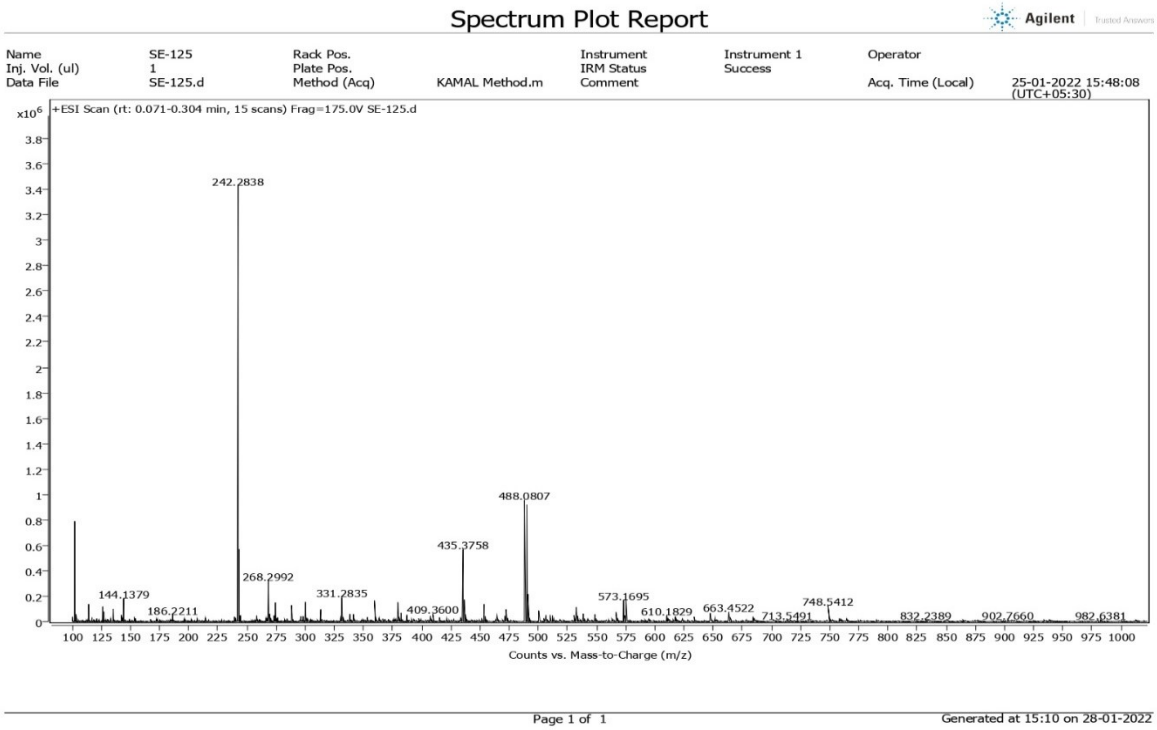


Figure **S65:** HRMS of Compound **4u.**

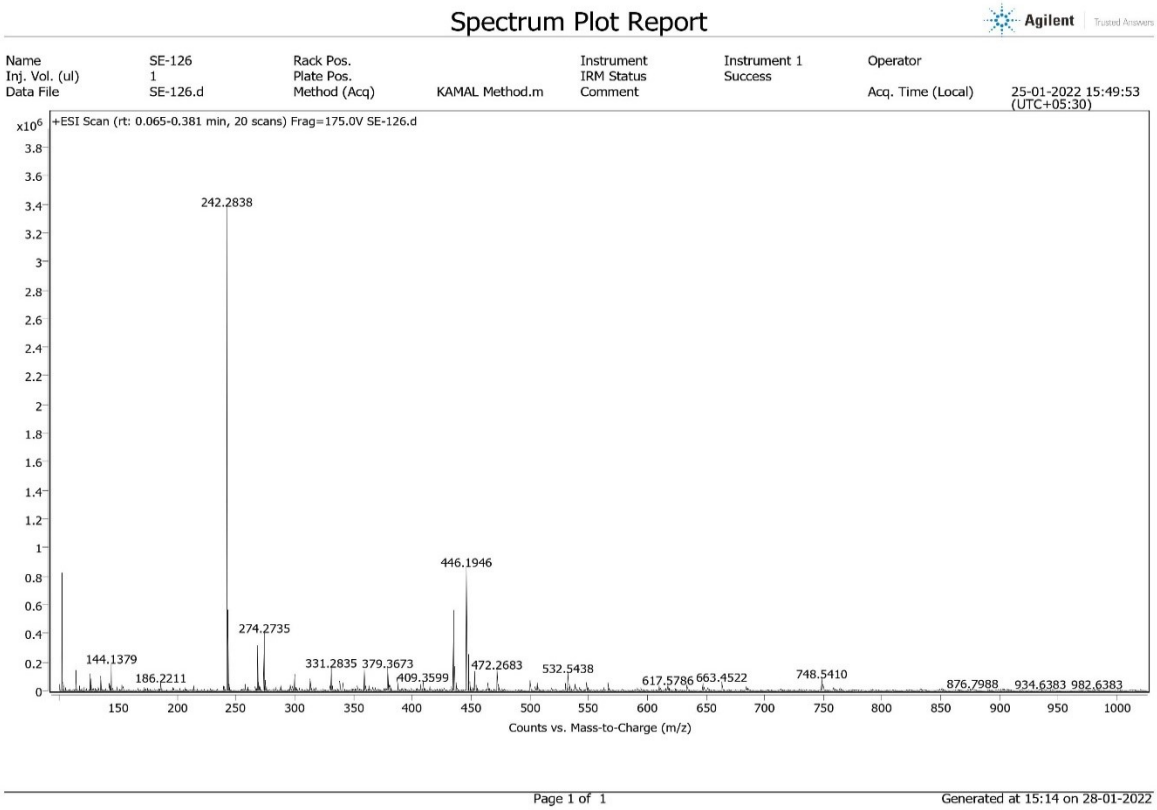
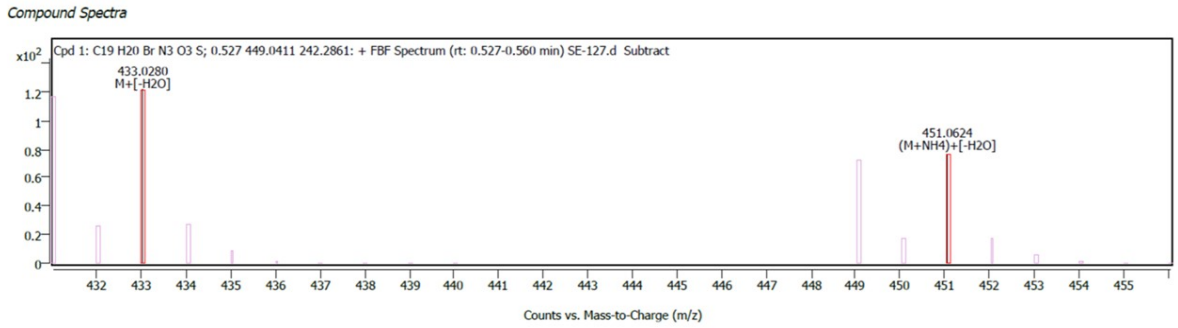


Figure **S66:** HRMS of Compound **4v.**



Crystal X-ray report of compound **4f [CCDC: 2157101]**.

checkCIF (basic structural check) running

Checking for embedded fcf data in CIF ...
Found embedded fcf data in CIF. Extracting fcf data from uploaded CIF, please wait **. . .**

checkCIF/PLATON (basic structural check)

Structure factors have been supplied for datablock(s) 106

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary
Please wait while processing .... Interpreting this report

Structure factor report

Datablock: 106

Bond precision: C-C = 0.0033 A Wavelength=0.71073

Cell: a=17.3963(6) b=22.485(1) c=14.0877(9)
alpha=90 beta=127.7461(13) gamma=90

Temperature: 296 K

	Calculated	Reported
Volume	4357.3(4)	4357.3(4)
Space group	C 2/c	C 2/c
Hall group	-C 2yc	-C 2yc
Moiety formula	C22 H25 N3 O4	?
Sum formula	C22 H25 N3 O4	C22 H25 N3 O4
Mr	395.42	395.45
Dx,g cm-3	1.206	1.206
Z	8	8
Mu (mm-1)	0.084	0.084
F000	1679.9	1680.0
F000'	1680.63	
h,k,lmax	20,26,16	20,26,16
Nref	3835	3835
Tmin,Tmax	0.979,0.984	0.979,0.984
Tmin'	0.979	

Correction method= # Reported T Limits: Tmin=0.979 Tmax=0.984 AbsCorr = MULTI-SCAN

Data completeness= 1.000 Theta(max)= 25.000

R(reflections)= 0.0468(2826) wR2(reflections)= 0.1368(3835)

S = 1.045 Npar= 293

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level A

PLAT601_ALERT_2_A Unit Cell Contains Solvent Accessible VOIDS of . 221 Ang**3

Author Response: Application of procedure SQUEEZE (program PLATON) did not bring about a significant improvement on refinement and therefore was not retained for the final refinement. The compound is therefore considered as a DESOLVATE with no solvent content.

Alert level B

PLAT919_ALERT_3_B Reflection # Likely Affected by the Beamstop ... 1 Check

Author Response: The crystal which was studied was small in size and some reflections having small 2theta values, may have been affected by beam stop.

Alert level C

PLAT220_ALERT_2_C NonSolvent Resd 1 C Ueq(max)/Ueq(min) Range 3.8 Ratio
PLAT222_ALERT_3_C NonSolvent Resd 1 H Uiso(max)/Uiso(min) Range 4.4 Ratio
PLAT241_ALERT_2_C High 'MainMol' Ueq as Compared to Neighbors of C15 Check
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of O2 Check
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of C17 Check
PLAT334_ALERT_2_C Small Aver. Benzene C-C Dist C14 -C19 1.37 Ang.
PLAT934_ALERT_3_C Number of (Iobs-Icalc)/Sigma(W) > 10 Outliers .. 1 Check

Alert level G

PLAT066_ALERT_1_G Predicted and Reported Tmin&Tmax Range Identical ? Check
PLAT068_ALERT_1_G Reported F000 Differs from Calcd (or Missing)... Please Check
PLAT128_ALERT_4_G Alternate Setting for Input Space Group C2/c I2/a Note
PLAT301_ALERT_3_G Main Residue Disorder .....(Resd 1) 7% Note
PLAT398_ALERT_2_G Deviating C-O-C Angle From 120 for O3 . 109.4 Degree
PLAT398_ALERT_2_G Deviating C-O-C Angle From 120 for O3A . 108.8 Degree
PLAT413_ALERT_2_G Short Inter XH3 .. XHn H21C ..H22C . 1.99 Ang.
1/2-x,3/2-y,1-z = 7_566 Check
PLAT793_ALERT_4_G Model has Chirality at C7 (Centro SPGR) R Verify
PLAT860_ALERT_3_G Number of Least-Squares Restraints ..... 1 Note
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary . Please Do !
PLAT909_ALERT_3_G Percentage of I>2sig(I) Data at Theta(Max) Still 43% Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ..... 4.5 Low
PLAT965_ALERT_2_G The SHELXL WEIGHT Optimisation has not Converged Please Check
PLAT967_ALERT_5_G Note: Two-Theta Cutoff Value in Embedded .res .. 50.0 Degree
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 4 Info

1 **ALERT level A** = Most likely a serious problem - resolve or explain
1 **ALERT level B** = A potentially serious problem, consider carefully
7 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
15 **ALERT level G** = General information/check it is not something unexpected

3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
11 ALERT type 2 Indicator that the structure model may be wrong or deficient
7 ALERT type 3 Indicator that the structure quality may be low
2 ALERT type 4 Improvement, methodology, query or suggestion
1 ALERT type 5 Informative message, check

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

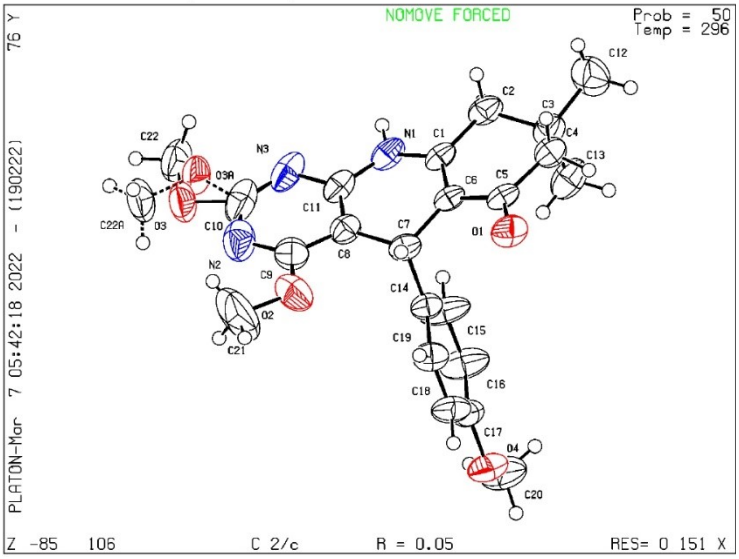
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 19/02/2022; check.def file version of 19/02/2022

Datablock 106 - ellipsoid plot



Crystal X-ray report of compound **4o [CCDC: 2149724]**.

checkCIF (basic structural check) running

Checking for embedded fcf data in CIF ...
Found embedded fcf data in CIF. Extracting fcf data from uploaded CIF, please wait ..

checkCIF/PLATON (basic structural check)

Structure factors have been supplied for datablock(s) 103

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary
Please wait while processing .... Interpreting this report

Structure factor report

Datablock: 103

Bond precision: C-C = 0.0032 A Wavelength=0.71073

Cell: a=6.0680(2) b=13.0347(5) c=13.5866(5)
alpha=103.302(3) beta=95.6327(18) gamma=103.2902(19)

Temperature: 296 K

	Calculated	Reported
Volume	1004.76(7)	1004.76(7)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C21 H22 Br N3 O3	?
Sum formula	C21 H22 Br N3 O3	C21 H22 Br N3 O3
Mr	444.32	444.32
Dx,g cm-3	1.469	1.469
Z	2	2
Mu (mm-1)	2.072	2.072
F000	456.0	456.0
F000'	455.60	
h,k,lmax	7,15,16	7,15,16
Nref	3554	3531
Tmin,Tmax	0.602,0.813	0.625,0.820
Tmin'	0.590	

Correction method= # Reported T Limits: Tmin=0.625 Tmax=0.820 AbsCorr = MULTI-SCAN

Data completeness= 0.994 Theta(max)= 24.993

R(reflections)= 0.3315(2771) wR2(reflections)= 0.0762(3531)

S = 1.029 Npar= 261

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.594 24 Report

Alert level G

PLAT793_ALERT_4_G Model has Chirality at C8 (Centro SPGR) S Verify
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary . Please Do !
PLAT909_ALERT_3_G Percentage of I>2sig(I) Data at Theta(Max) Still 50% Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ..... 3.9 Low
PLAT965_ALERT_2_G The SHELXL WEIGHT Optimisation has not Converged Please Check
PLAT967_ALERT_5_G Note: Two-Theta Cutoff Value in Embedded .res .. 50.0 Degree
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 9 Info

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1 ALERT type 5 Informative message, check

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Publication of your CIF in other journals

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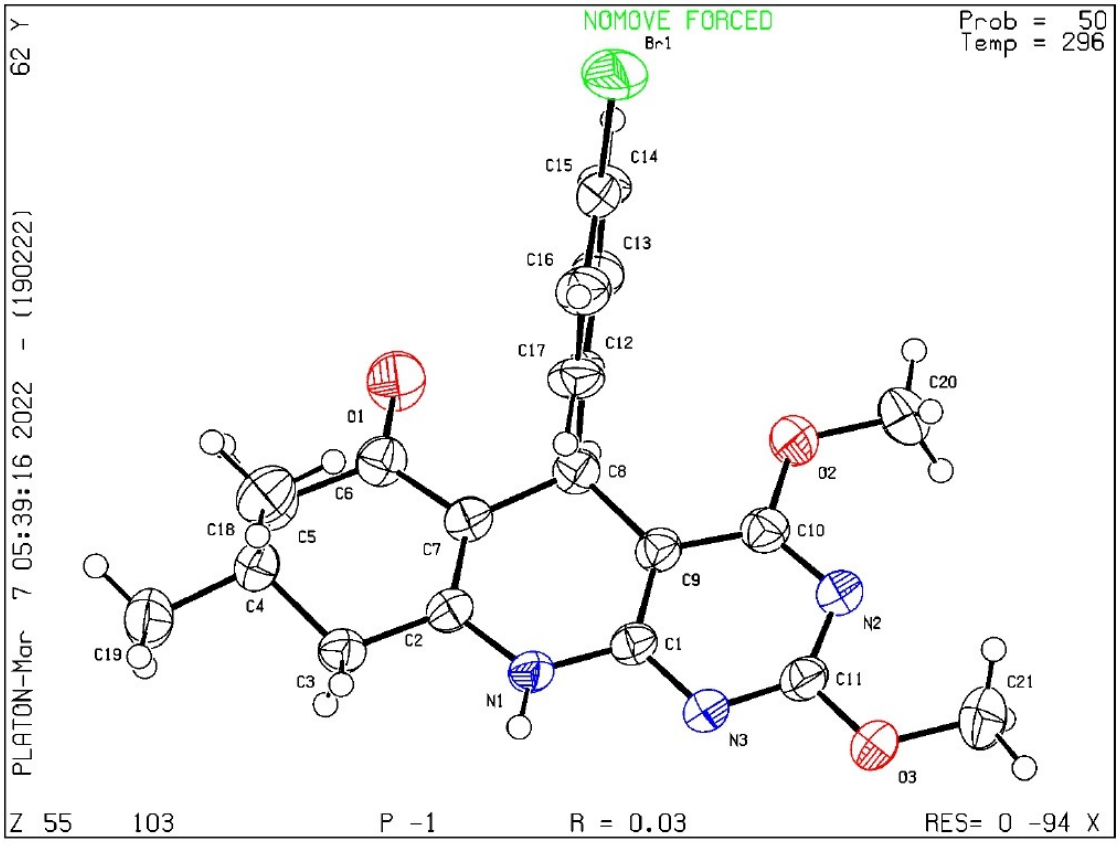
PLATON version of 19/02/2022; check.def file version of 19/02/2022

Datablock 103 - ellipsoid plot



4. Green chemistry metrics analysis

The following formulae were used for calculating Atom Economy (AE), Atom Efficiency (AEf), Carbon Efficiency (CE), Reaction Mass Efficiency (RME), Optimum Efficiency (OE), Mass Productivity (MP), Mass Intensity (MI) and Process Mass Intensity (PMI), E factor, Solvent and Water Intensity (SI and WI).

$$AE = \frac{Molecular\ weight\ of\ product}{Total\ molecular\ weight\ of\ reactants} \times 100$$

$$AEf = AE \times \%yield$$

$$CE = \frac{Amount\ of\ carbon\ in\ the\ product}{Total\ carbon\ present\ in\ the\ reactant} \times 100$$

$$RME = \frac{Mass\ of\ isolated\ product}{Total\ mass\ of\ reactant} \times 100$$

$$OE = \frac{RME}{AE} \times 100$$

$$PMI = \frac{Total\ mass\ of\ input\ material\ in\ the\ whole\ process}{Mass\ of\ product}$$

$$MP = \frac{1}{PMI} \times 100$$

$$E\ Factor = PMI - 1$$

$$SI = \frac{Total\ mass\ of\ solvents\ excluding\ water\ in\ the\ whole\ process}{Mass\ of\ product}$$

$$WI = \frac{Total\ mass\ of\ water\ in\ the\ whole\ process}{Mass\ of\ product}$$

4.1. Green chemistry metrics analysis for conventional method

Materials used for metrics calculations:4-Chlorobenzaldehyde **1a** (140 mg, 1 mmol), Dimedone **2** (140 mg, 1 mmol), 6-amino-2,4-dimethoxypyrimidine **3** (155 mg, 1 mmol), Product **4a** (290 mg, 0.73 mmol), Acetic acid (3.147 g, 3 mL), water (20 g, 20 mL), diethyl ether (3.57 g, 5 mL), petroleum ether (3.35 g, 5 mL).

$$AE = \frac{399.88}{140.57 + 140.18 + 155.15} \times 100 = \frac{399.88}{435.9} \times 100 = 91.74$$

$$AEf = 91.74 \times 73\% = 66.97$$

$$CE = \frac{21 \times 0.0007252}{7 \times 0.001 + 8 \times 0.001 + 6 \times 0.001} \times 100 = 72.52$$

$$RME = \frac{290}{435} \times 100 = 66.67$$

$$OE = \frac{66.67}{91.74} \times 100 = 72.67$$

$$PMI = \frac{30.50}{0.290} = 105.17$$

$$MP = \frac{1}{105.17} \times 100 = 0.95$$

$$E\ Factor = 105.17 - 1 = 104.17$$

$$SI = \frac{3.147 + 3.57 + 3.35}{0.290} = 34.71$$

$$WI = \frac{20}{0.290} = 68.97$$

4.2. Green chemistry metrics analysis for microwave method

Materials used for metrics calculations: 4-Chlorobenzaldehyde **1a** (140 mg, 1 mmol), Dimedone **2** (140 mg, 1 mmol), 6-amino-2,4-dimethoxypyrimidine **3** (155 mg, 1 mmol), Product **4a** (333 mg, 0.83 mmol), Acetic acid (3.147 g, 3 mL), water (20 g, 20 mL), diethyl ether (3.57 g, 5 mL), petroleum ether (3.35 g, 5 mL).

$$AE = \frac{399.88}{140.57 + 140.18 + 155.15} \times 100 = \frac{399.88}{435.9} \times 100 = 91.74$$

$$AEf = 91.74 \times 84\% = 77.06$$

$$CE = \frac{21 \times 0.0008328}{7 \times 0.001 + 8 \times 0.001 + 6 \times 0.001} \times 100 = 83.28$$

$$RME = \frac{333}{435} \times 100 = 76.55$$

$$OE = \frac{76.55}{91.74} \times 100 = 83.44$$

$$PMI = \frac{30.50}{0.333} = 91.59$$

$$MP = \frac{1}{91.59} \times 100 = 1.09$$

$$E\ Factor = 91.59 - 1 = 90.59$$

$$SI = \frac{3.147 + 3.57 + 3.35}{0.333} = 30.23$$

$$WI = \frac{20}{0.333} = 60.06$$