

Registered Address: Chemicea Pharmaceuticals Pvt Ltd, Platinum Springs,  
Unit No- A- 205&206 Second floor, Plot No: G-6, G-20 to G 24, Taloja- MIDC, Opp. Dena Bank  
Navi Mumbai 410208, MH, INDIA. Email- [info@chemicea.com](mailto:info@chemicea.com) Mob- +91 9284897978

## CERTIFICATE OF ANALYSIS

Analysis Date: Jun 2024

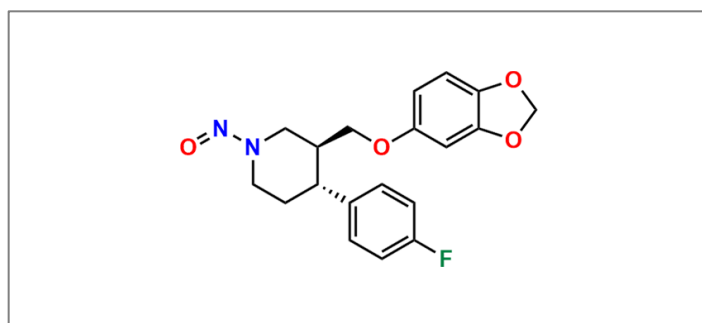
Re-test Date: May 2027

**Product Name: N-Nitroso Paroxetine**

### Identification:

Chemical Name: (3S,4R)-3-((benzo[d][1,3]dioxol-5-yloxy)methyl)-4-(4-fluorophenyl)-1-nitrosopiperidine

Synonyms : NA  
CAS Number : 2361294-43-9  
CAT Number : CP-P3015  
Molecular Formula : C<sub>19</sub>H<sub>19</sub>FN<sub>2</sub>O<sub>4</sub>  
Molecular Weight : 358.37  
Batch Number : CP-PAR-NIT-0624



Storage Condition : Tightly closed, Inert atmosphere at 2 to 8° C  
Shipping condition : All Products are stable to be shipped at room temperature, if specific condition required it is mention in the COA

### Analytical Information:

Sr. No.	Test	Result
1)	Description	White to Off White Solid
2)	Solubility	Soluble in Methanol/DMSO
3)	MASS By LCMS	Confirm to structure
4)	Purity By HPLC	99.96%
5)	<sup>1</sup> H NMR	Confirm to structure
6)	<sup>13</sup> C NMR	Confirm to structure
7)	IR	Confirm to structure
8)	Weight loss by TGA	0.147%
9)	Potency	99.81%

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Summary of Analysis	Specifications	Result
1H NMR spectrum	Confirms to Structure	Confirms
13C NMR spectrum	Confirms to Structure	Confirms
Mass spectrum	Confirms to Structure	Confirms
IR spectrum	Confirms to Structure	Confirms
Purity by HPLC	Not less than 95%	99.96%
Weight loss by TGA	Information Only	0.147%
Potency = $[100 - 0.147\% \text{ (Wt. loss by TGA)}] \times [99.96\% \text{ (HPLC Purity)}] / 100$	Information Only	99.81%

**Summary:**

The compound was analysed for its structure and purity. 1H-NMR and 13C NMR spectra were consistent with the structure and the compound was found pure by NMR. Mass spectrum confirmed to the structure. The IR signals corresponding to important functional groups with their wavelength range were observed and further validated our conclusions. HPLC purity obtained was 99.96%. TGA showed 0.147% loss due to Volatiles was detected at the end of the analysis. The Potency of the compound was 99.81%.

**Prepared and Reviewed By**

Ms. Priyanka Salunkhe  
(Quality Control)

**Approved By**

Ms. Rohini Gagare  
(Quality Assurance)

**Note:** Product supplied by Chemicea are for R&D purpose only and not for human consumptions.

## HPLC Method of Analysis

The purity of the reference substance was determined by the following analysis methods:

**Mobile Phase A:** 10mm Ammonium acetate in 1 Lit H<sub>2</sub>O

**Mobile Phase B:** 950 ml Acetonitrile added 50ml H<sub>2</sub>O

**Flow:** 1ml/min

**Run Time:** 25 Min

**Detector:** PDA 235 nm

**Temperature:** 30° C

**Injection Volume:** 10µl

**Preparation of Sample:** 1000ppm

**Column:** Water symmetry C18

**Diluent:** Mobile Phase A 30% + Mobile Phase B 70%

<b>Gradient</b>				
<b>Sr.No</b>	<b>Time</b>	<b>Flow</b>	<b>%A</b>	<b>%B</b>
1	0.0	1.00	90.0	10.0
2	2.0	1.00	90.0	10.0
3	10	1.00	2.0	98.0
4	20	1.00	2.0	98.0
5	21	1.00	90.0	10.0
6	25	1.00	90.0	10.0

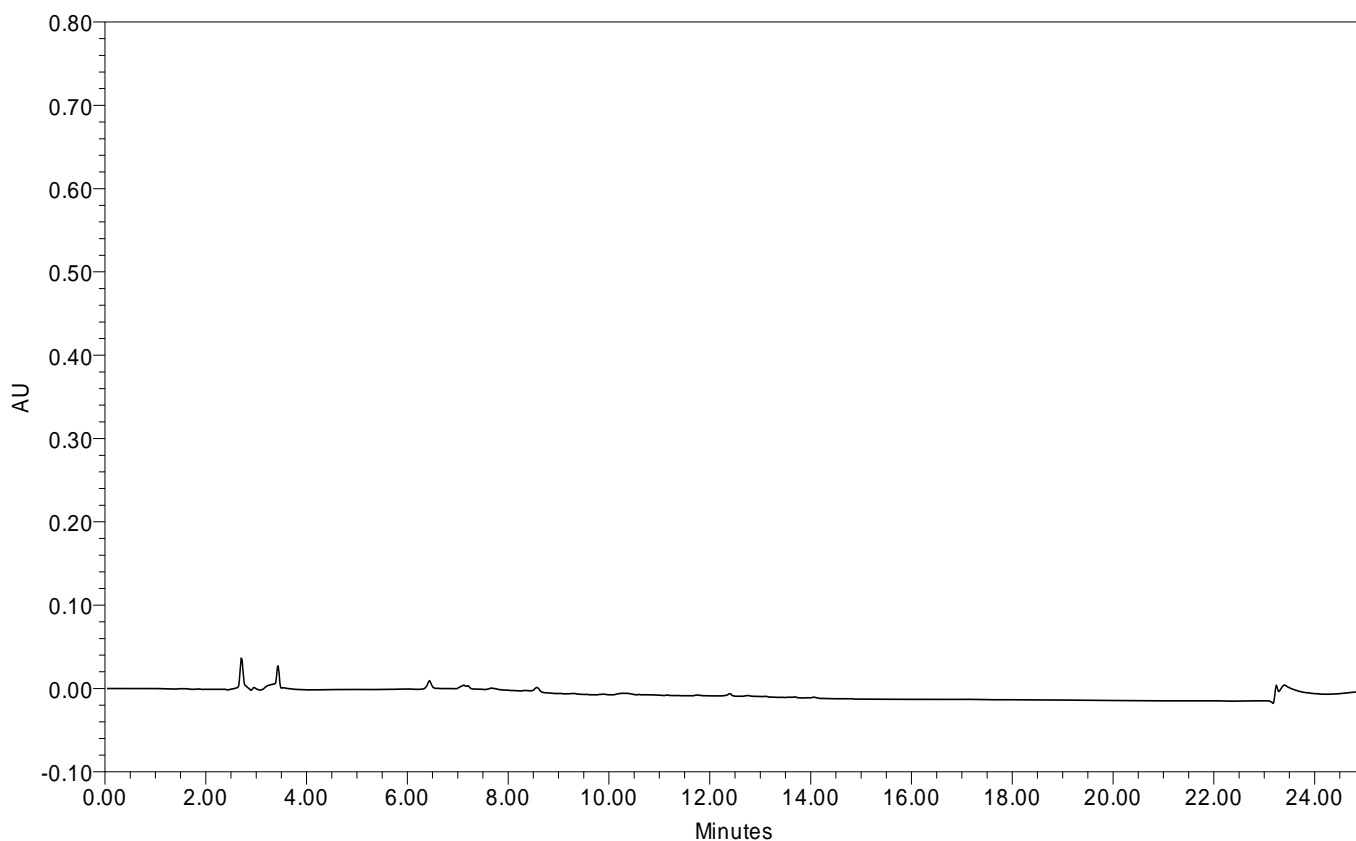
## SAMPLE INFORMATION

Sample Name:	Blank	Acquired By:	System
Sample Type:	Unknown	Sample Set Name:	24 Jun 2024
Vial:	45	Acq. Method Set:	INHOUSE_25MINFAST
Injection #:	1	Processing Method:	Default
Injection Volume:	10.00 ul	Channel Name:	235.0nm
Run Time:	25.0 Minutes	Proc. Chnl. Descr.:	PDA 235.0 nm

Date Acquired: 6/24/2024 12:52:03 PM IST

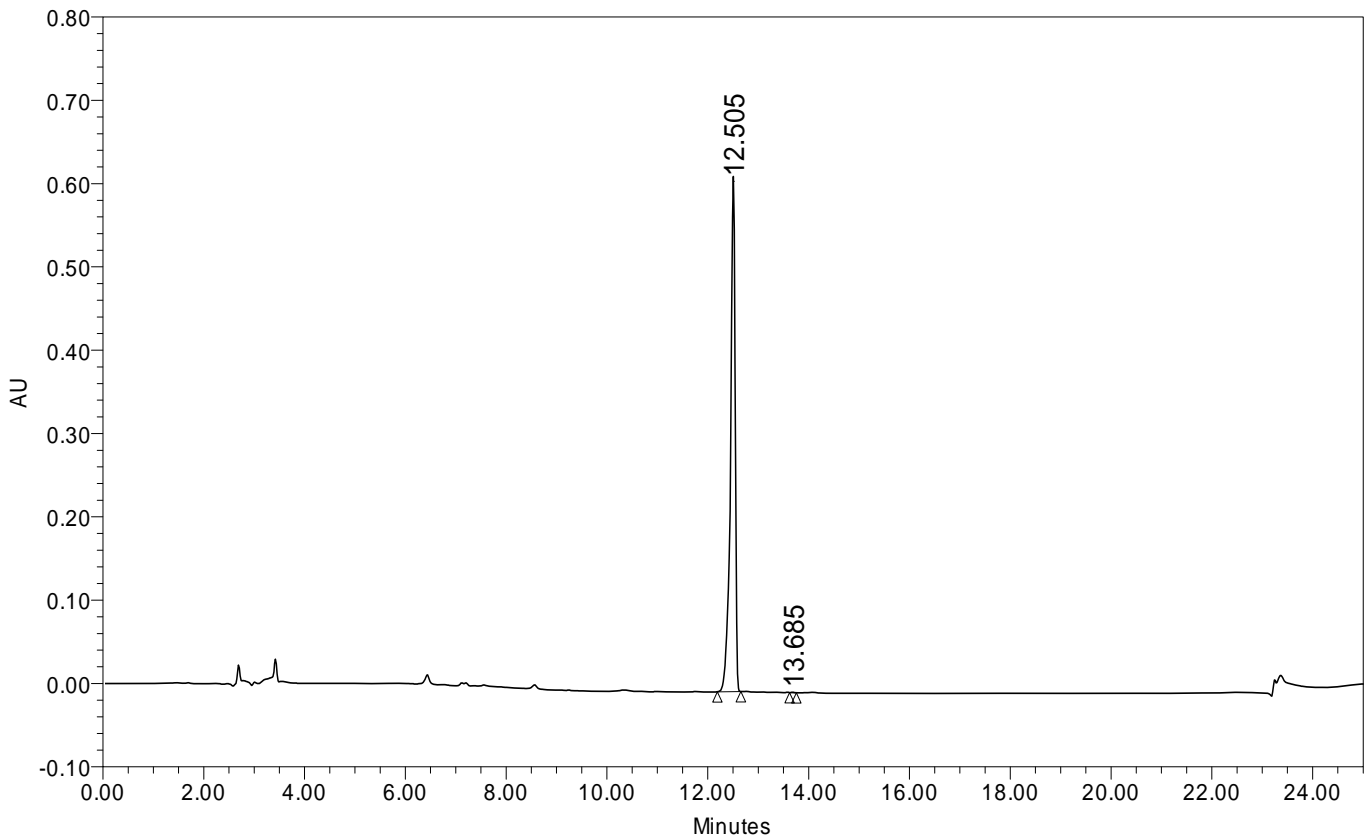
Date Processed: 6/24/2024 1:46:55 PM IST

Batch No:



## SAMPLE INFORMATION

Sample Name: CP-PAR-NIT-0624	Acquired By: System
Sample Type: Unknown	Sample Set Name: 24 Jun 2024
Vial: 46	Acq. Method Set: INHOUSE_25MINFAST
Injection #: 1	Processing Method: Default
Injection Volume: 10.00 ul	Channel Name: 235.0nm @ 1
Run Time: 25.0 Minutes	Proc. Chnl. Descr.: PDA 235.0 nm
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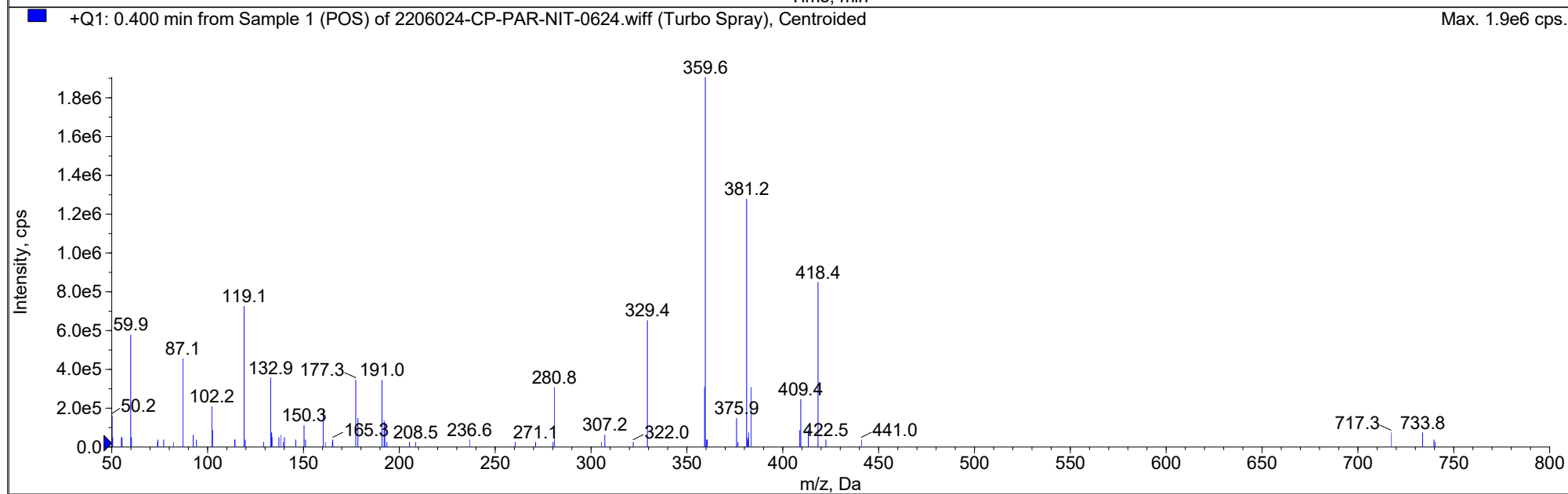
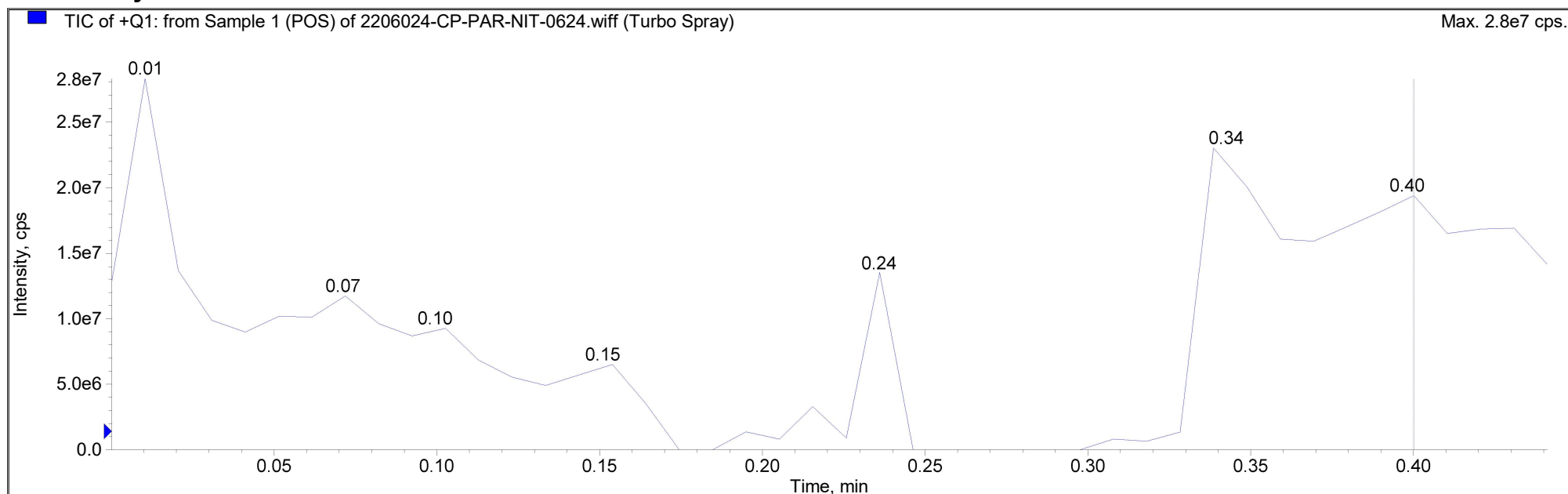


	RT	Area	% Area	Height
1	12.505	4014829	99.96	614006
2	13.685	1506	0.04	369

**Result:** The HPLC purity of the compound is 99.96%.



Mass Analysis:



Polarity/Scan Type: Positive Q1 MS

Page 1 of 1

**Mass Result:** Observed mass is consistent with the structure.

CHEMICEA PHARMACEUTICALS PVT LTD  
 Spectrometer: Bruker 400MHz  
 Solvent: DMSO  
 Number of Scans: 9  
 N-NITROSO PAROXETINE IMPURITY  
 BATCH NO: CP-PAR-NIT-0624  
 1H-NMR  
 22-JUNE-2024

### 1H NMR Analysis

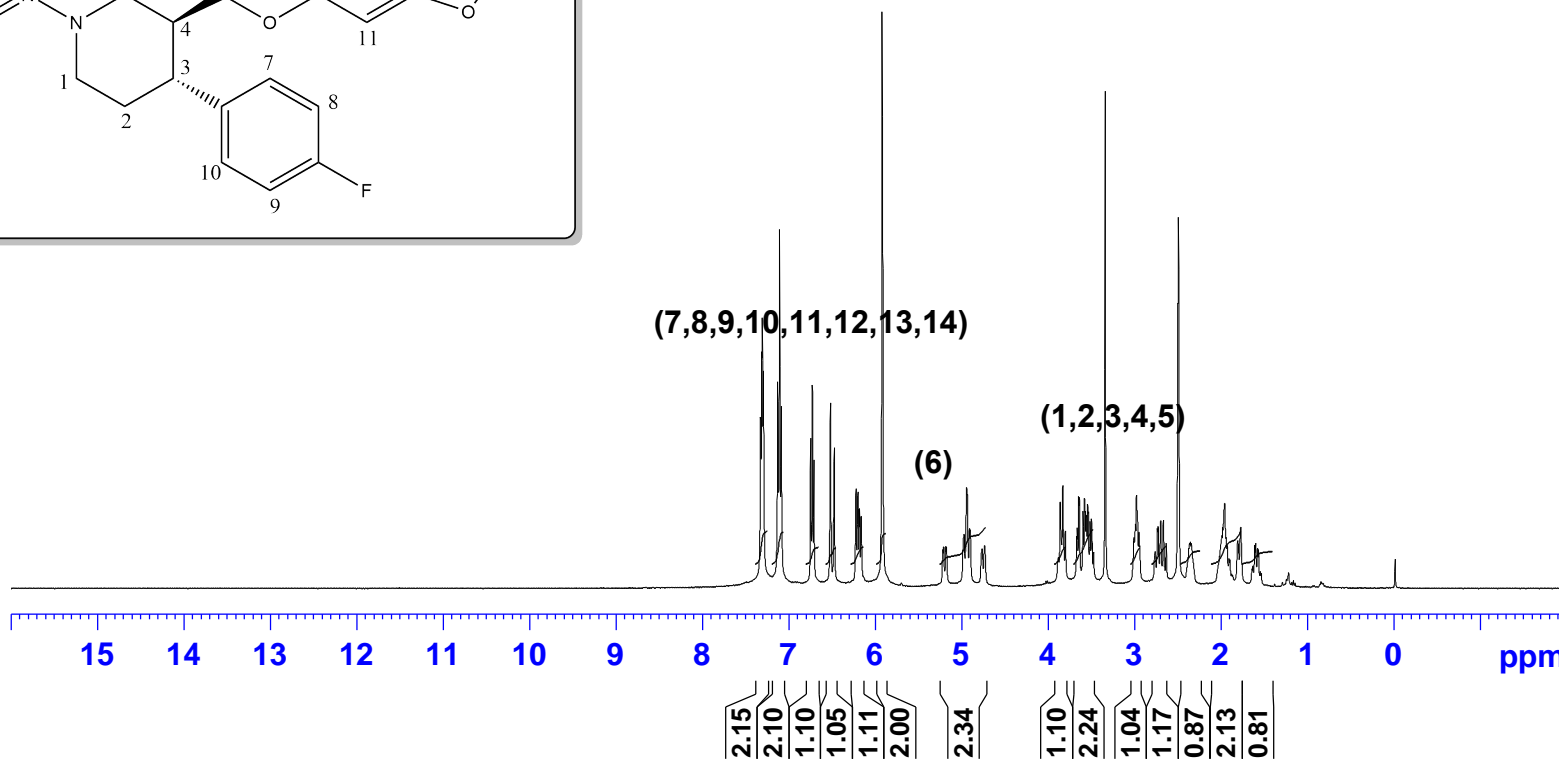
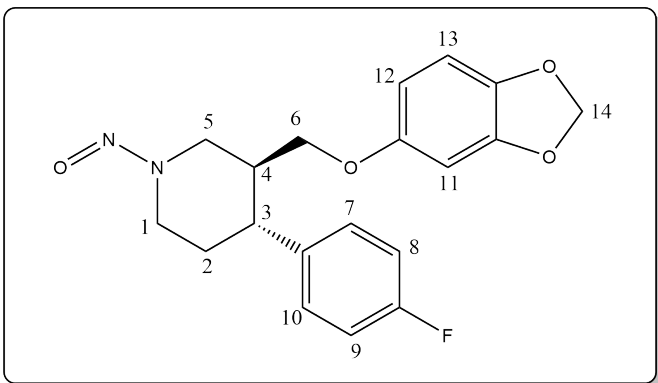


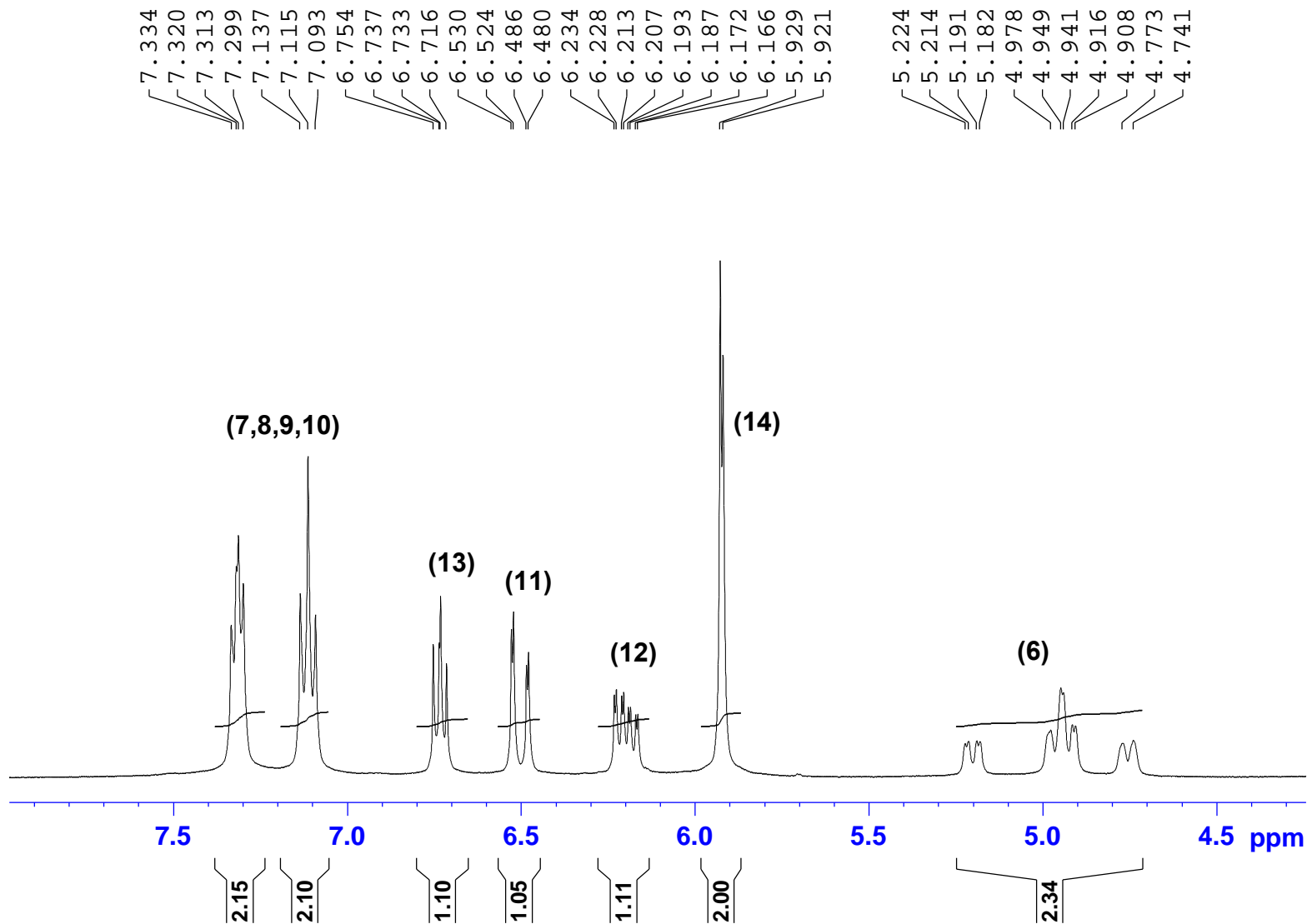
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 6.716  
 6.530  
 6.524  
 6.486  
 6.480  
 6.234  
 6.228  
 6.213  
 6.207  
 6.193  
 6.187  
 6.172  
 6.166  
 5.929  
 5.921  
 4.949  
 4.941  
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 3.652  
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 FIDRES 0.244532 Hz  
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 TE 0 K  
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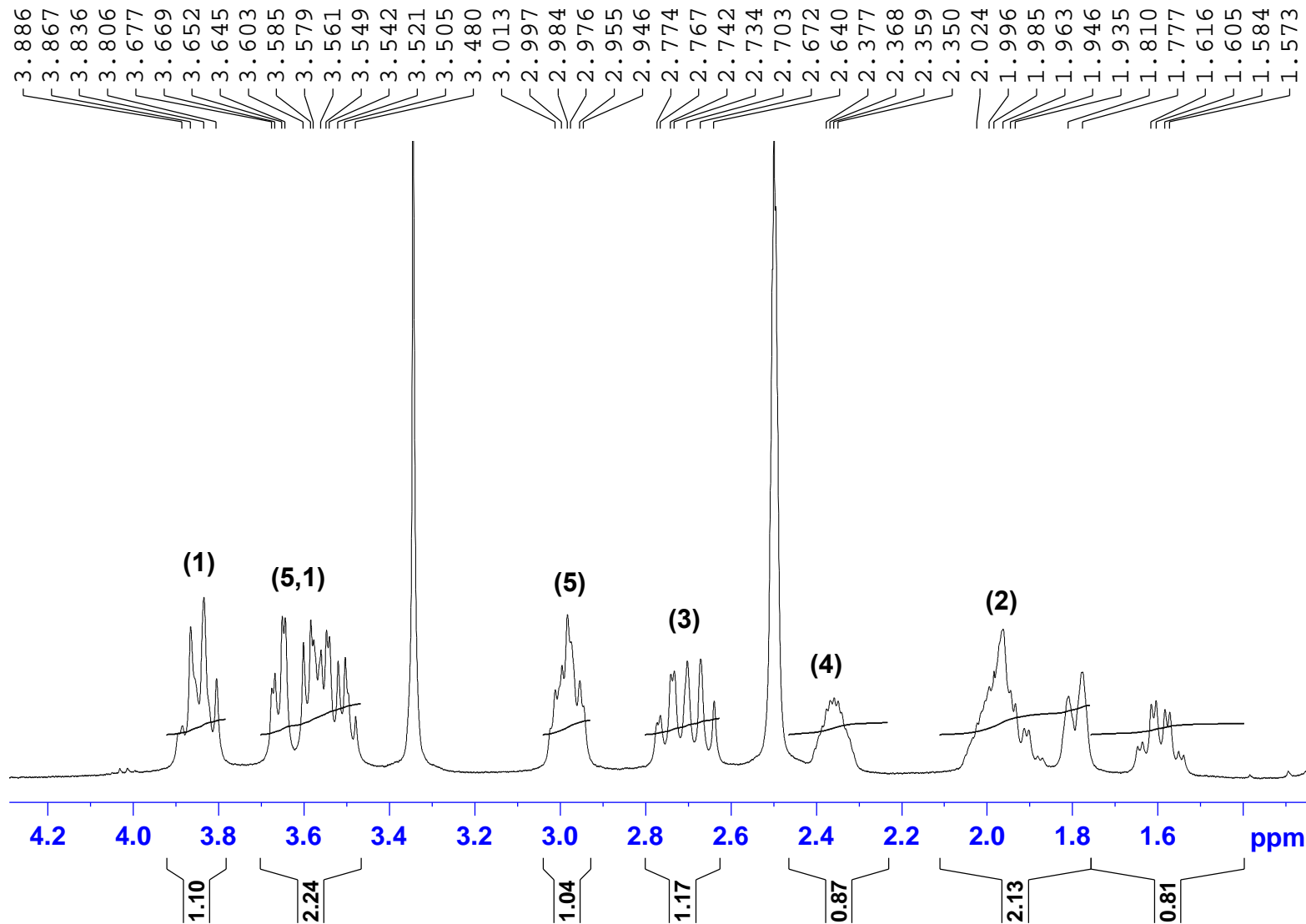




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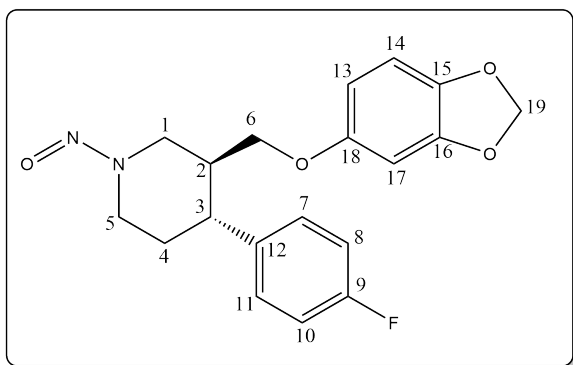
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5	29569.3	2855.430	7.1370	5.35
6	29641.6	2846.581	7.1149	9.31
7	29713.4	2837.801	7.0929	4.73
8	30821.3	2702.353	6.7544	3.89
9	30877.5	2695.472	6.7372	3.94
10	30889.9	2693.965	6.7334	5.29
11	30945.8	2687.124	6.7163	3.33
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13	31576.7	2609.992	6.5235	4.82
14	31698.7	2595.068	6.4862	3.29
15	31718.4	2592.668	6.4802	3.65
16	32524.4	2494.116	6.2339	2.42
17	32544.4	2491.677	6.2278	2.58
18	32593.8	2485.637	6.2127	2.40
19	32613.9	2483.177	6.2066	2.51
20	32657.7	2477.821	6.1932	2.06
21	32677.7	2475.370	6.1871	2.07
22	32727.1	2469.336	6.1720	1.86
23	32747.2	2466.879	6.1658	1.87
24	33522.1	2372.137	5.9290	15.00
25	33548.7	2368.878	5.9209	12.24
26	35828.5	2090.133	5.2242	1.01
27	35860.4	2086.238	5.2144	1.08
28	35936.7	2076.903	5.1911	1.09
29	35965.9	2073.338	5.1822	1.07
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31	36728.0	1980.162	4.9493	2.62
32	36753.7	1977.017	4.9415	2.48
33	36836.1	1966.947	4.9163	1.55
34	36864.6	1963.452	4.9076	1.52
35	37304.8	1909.632	4.7730	1.02
36	37410.0	1896.779	4.7409	1.11
37	40207.5	1554.732	3.8860	0.81
38	40270.4	1547.040	3.8668	2.24
39	40371.6	1534.666	3.8358	2.66
40	40468.3	1522.850	3.8063	1.49

41	40892.7	1470.960	3.6766	1.35
42	40916.1	1468.099	3.6694	1.57
43	40972.7	1461.173	3.6521	2.39
44	40995.1	1458.442	3.6453	2.37
45	41134.6	1441.384	3.6027	2.01
46	41191.4	1434.434	3.5853	2.33
47	41213.5	1431.731	3.5785	2.05
48	41269.4	1424.903	3.5615	1.90
49	41311.2	1419.796	3.5487	2.19
50	41334.4	1416.956	3.5416	2.10
51	41401.0	1408.814	3.5213	1.74
52	41455.3	1402.174	3.5047	1.80
53	41535.5	1392.361	3.4801	0.94
54	43065.4	1205.317	3.0126	1.33
55	43117.3	1198.967	2.9968	1.68
56	43159.5	1193.810	2.9839	2.41
57	43186.6	1190.499	2.9756	2.02
58	43254.7	1182.162	2.9548	1.46
59	43283.3	1178.674	2.9460	1.10
60	43847.4	1109.704	2.7736	0.85
61	43870.6	1106.860	2.7665	0.96
62	43952.3	1096.874	2.7416	1.55
63	43977.8	1093.758	2.7338	1.60
64	44078.2	1081.479	2.7031	1.75
65	44180.0	1069.038	2.6720	1.78
66	44283.4	1056.392	2.6404	1.17
67	45146.9	950.813	2.3765	1.05
68	45173.7	947.540	2.3683	1.17
69	45203.8	943.856	2.3591	1.21
70	45233.7	940.202	2.3500	1.16
71	46300.1	809.817	2.0241	0.85
72	46391.8	798.607	1.9961	1.37
73	46428.9	794.076	1.9848	1.60
74	46499.1	785.485	1.9633	2.20
75	46555.8	778.556	1.9460	1.32
76	46592.8	774.025	1.9346	1.13
77	46662.0	765.564	1.9135	0.77
78	46695.3	761.504	1.9033	0.75
79	47001.3	724.090	1.8098	1.24
80	47109.2	710.888	1.7768	1.58
81	47531.6	659.243	1.6477	0.51
82	47567.9	654.806	1.6367	0.57
83	47635.1	646.595	1.6161	1.12
84	47672.9	641.971	1.6046	1.17
85	47739.8	633.789	1.5841	1.04
86	47777.2	629.215	1.5727	1.00
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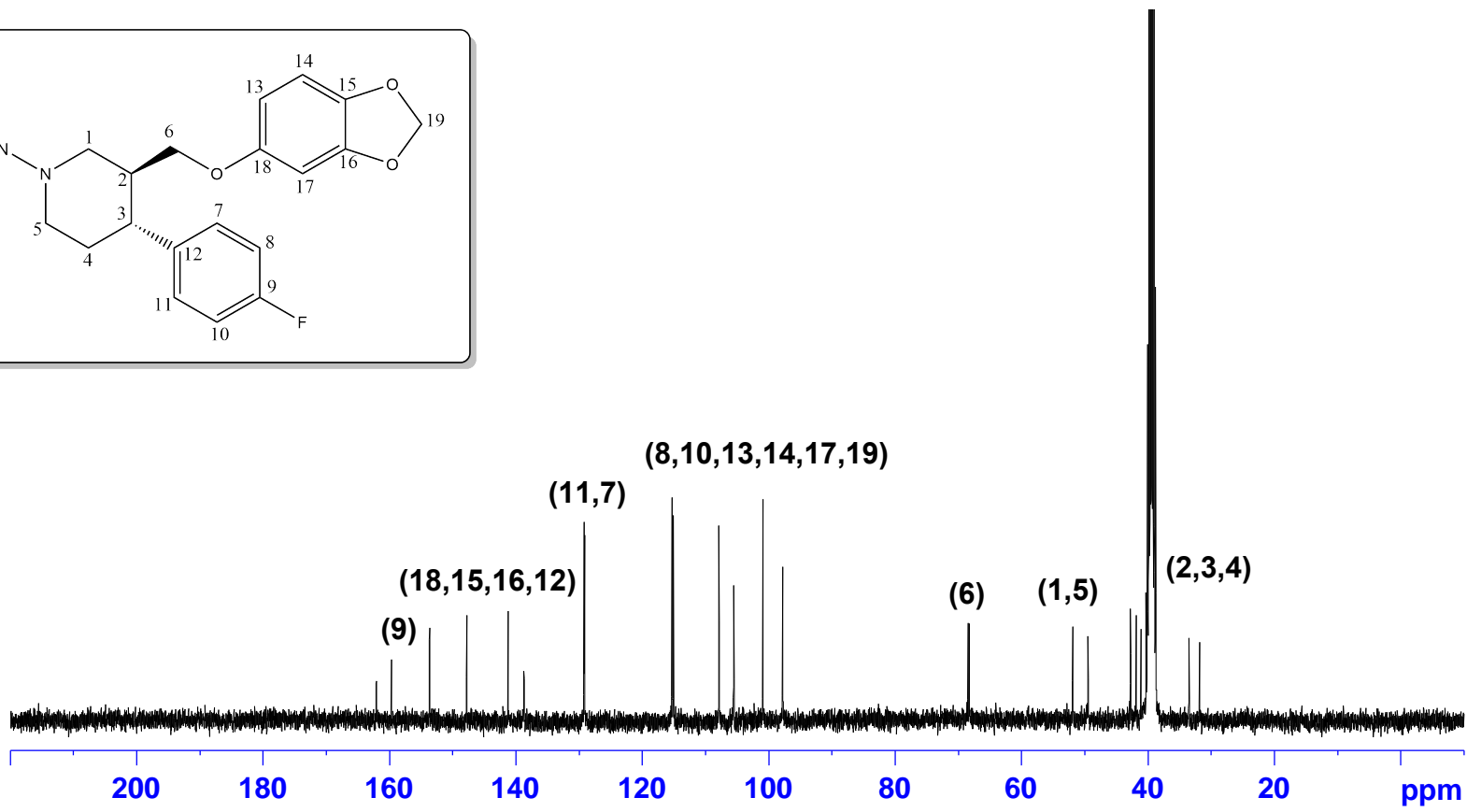
**Result:** The <sup>1</sup>H-NMR is consistent with the structure. The NMR was performed according with the solubility of compound e.g. DMSO-D<sub>6</sub>-D<sub>2</sub>O, CDCl<sub>3</sub> as the solvent. The peaks at 2.5 ppm and 3.78 ppm are due to residual DMSO and moisture in the NMR solvent as well as and 7.26 ppm and 1.56 respectively in CDCl<sub>3</sub>. The exchangeable protons like NH and OH protons cannot be observed due to exchange with D<sub>2</sub>O.

CHEMICEA PHARMACEUTICALS PVT LTD  
 Spectrometer: Bruker 100MHz  
 Solvent: DMSO  
 Number of Scans: 1323  
 N-NITROSO PAROXETINE IMPURITY  
 BATCH NO: CP-PAR-NIT-0624  
 13C-NMR  
 22-JUNE-2024

### 13C NMR ANALYSIS



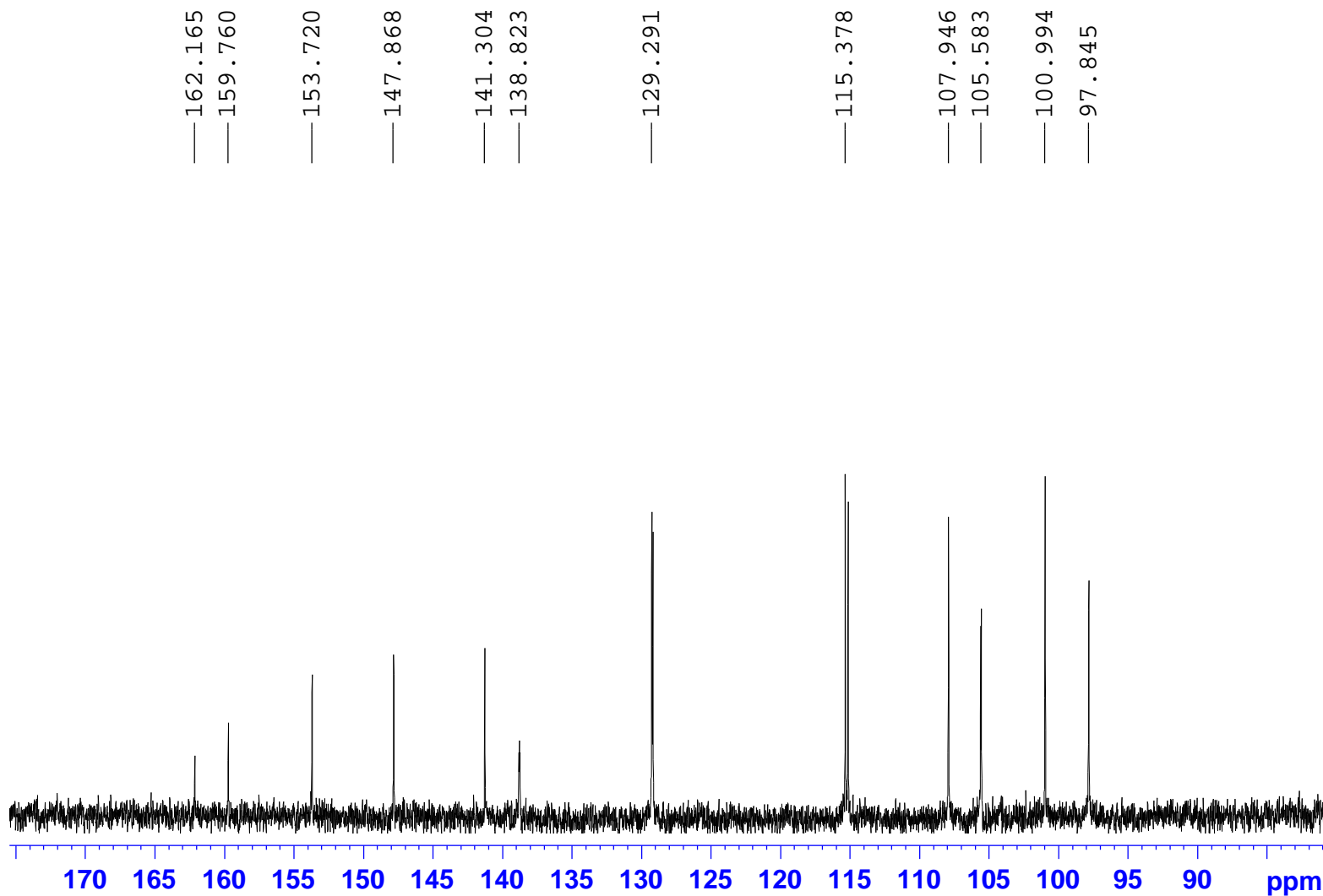
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 DE 6.50 usec  
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 D11 0.03000000 sec  
 TD0 1  
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 P0 3.67 usec  
 P1 11.00 usec  
 PLW1 41.08399963 W  
 SFO2 400.0896004 MHz  
 NUC2 1H  
 CPDPRG[2] waltz65  
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 PLW12 0.45554000 W  
 PLW13 0.22913000 W

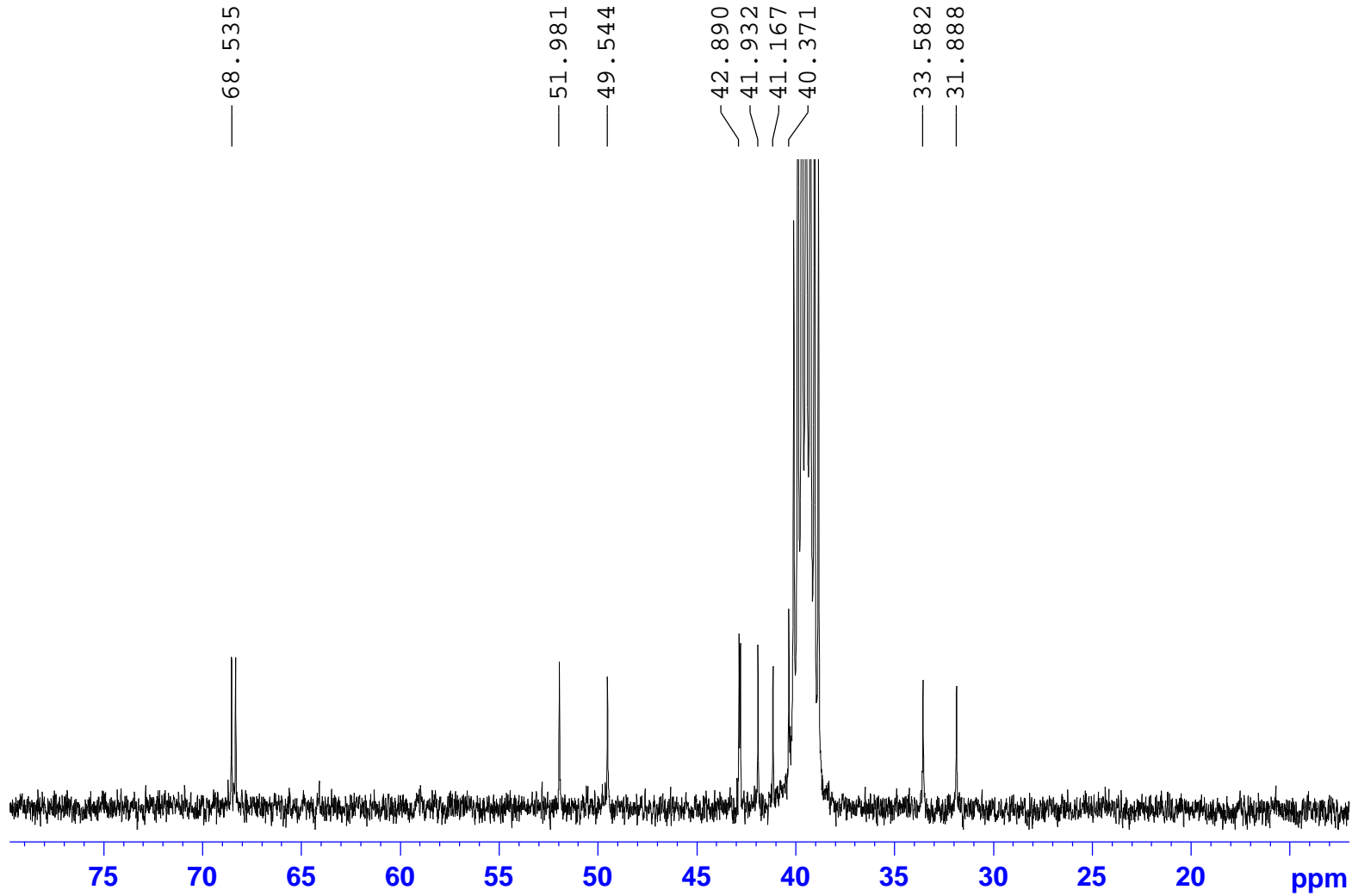
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 P0 3.67 usec  
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 SFO2 400.0896004 MHz  
 NUC2 1H  
 CPDPRG[2] waltz65  
 PCPD2 90.00 usec  
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Current Data Parameters
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PROCNO    1

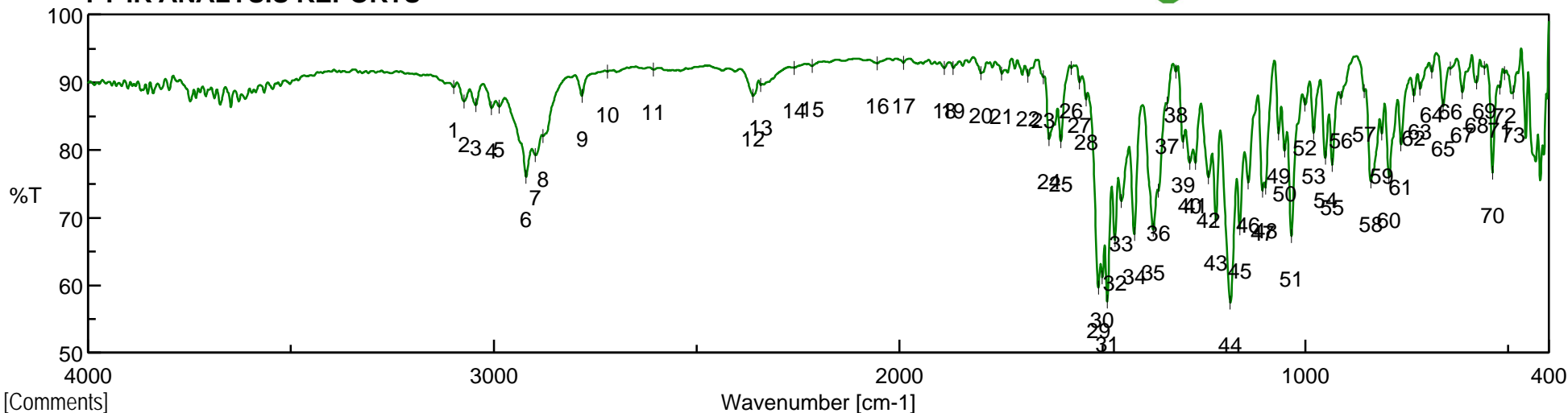
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3	10380.5	15464.580	153.7200	0.56
4	11028.7	14875.882	147.8683	0.64
5	11755.8	14215.463	141.3036	0.67
6	12030.6	13965.865	138.8226	0.30
7	13086.4	13006.937	129.2907	1.20
8	14627.4	11607.282	115.3779	1.35
9	15450.7	10859.575	107.9456	1.18
10	15712.4	10621.848	105.5826	0.82
11	16220.7	10160.208	100.9938	1.34
12	16569.5	9843.408	97.8448	0.92
13	19816.0	6894.750	68.5347	0.61
14	21649.5	5229.393	51.9809	0.57
15	21919.4	4984.242	49.5440	0.51
16	22656.5	4314.798	42.8897	0.67
17	22762.6	4218.441	41.9319	0.63
18	22847.3	4141.468	41.1667	0.55
19	22935.4	4061.450	40.3714	0.77
20	23687.5	3378.402	33.5818	0.50
21	23875.1	3207.993	31.8879	0.49

**Result:**The <sup>13</sup>C-NMR is consistent with the structure.

### FT-IR ANALYSIS REPORTS



[Comments]  
 Sample name CP-PAR-NIT-0624  
 Comment KBr Pellet AR#042617  
 User Laxminarayana  
 Division QC

#### Results of Peak Find

No.	Position	Intensity	No.	Position	Intensity	No.	Position	Intensity	No.	Position	Intensity
1	3099.05	89.2244	2	3073.01	87.1557	3	3044.09	86.5706	4	3005.52	86.1641
5	2986.23	86.3401	6	2920.66	75.9582	7	2897.52	79.1562	8	2878.24	81.988
9	2782.78	87.9348	10	2720.1	91.5542	11	2606.32	91.8198	12	2361.41	87.9281
13	2342.12	89.5428	14	2259.2	92.1056	15	2215.81	92.3832	16	2054.78	92.7578
17	1990.18	92.8644	18	1889.9	92.0692	19	1867.72	92.0491	20	1799.26	91.2986
21	1748.16	91.2135	22	1683.55	90.9352	23	1645.95	90.709	24	1631.48	81.5746
25	1602.56	81.2825	26	1576.52	92.0707	27	1556.27	89.9588	28	1540.85	87.456
29	1509.99	59.6363	30	1500.35	61.1209	31	1487.81	57.5221	32	1469.49	66.6478
33	1454.06	72.3983	34	1421.28	67.4929	35	1375	68.1495	36	1362.46	73.9435
37	1341.25	86.7868	38	1319.07	91.571	39	1301.72	81.1814	40	1285.32	78.075
41	1270.86	78.0786	42	1239.04	75.9517	43	1220.72	69.6239	44	1185.04	57.419
45	1161.9	68.3419	46	1140.69	75.1822	47	1105.98	73.8974	48	1098.26	74.3851
49	1065.48	82.4121	50	1051.01	79.8441	51	1034.62	67.2261	52	1000.87	86.6539
53	979.661	82.5061	54	950.734	78.803	55	933.378	77.7561	56	912.165	87.6538
57	855.275	88.6256	58	838.883	75.2859	59	811.885	82.407	60	793.564	75.9002
61	764.637	80.7759	62	732.817	88.0078	63	717.39	88.9875	64	688.463	91.5164
65	660.5	86.4456	66	642.179	91.9834	67	613.252	88.5137	68	578.54	89.9551
69	558.291	92.0911	70	539.007	76.5948	71	520.686	89.1884	72	509.115	91.3028
73	487.902	88.4753									

[Measurement Information]  
 Model Name FT/IR-4100typeA  
 Serial Number B088661016  
 Measurement Date 24/Jun/2024 10:44  
 Light Source Standard  
 Detector TGS  
 Accumulation 16  
 Resolution 4 cm-1  
 Zero Filling On  
 Apodization Cosine  
 Gain Auto (16)  
 Aperture Auto (7.1 mm)  
 Scanning Speed Auto (2 mm/sec)  
 Filter Auto (30000 Hz)

Analysed by:

Reviewed by:

**Results:** The signals of the IR spectrum are consistent with the structure. 16

## IR Interpretation

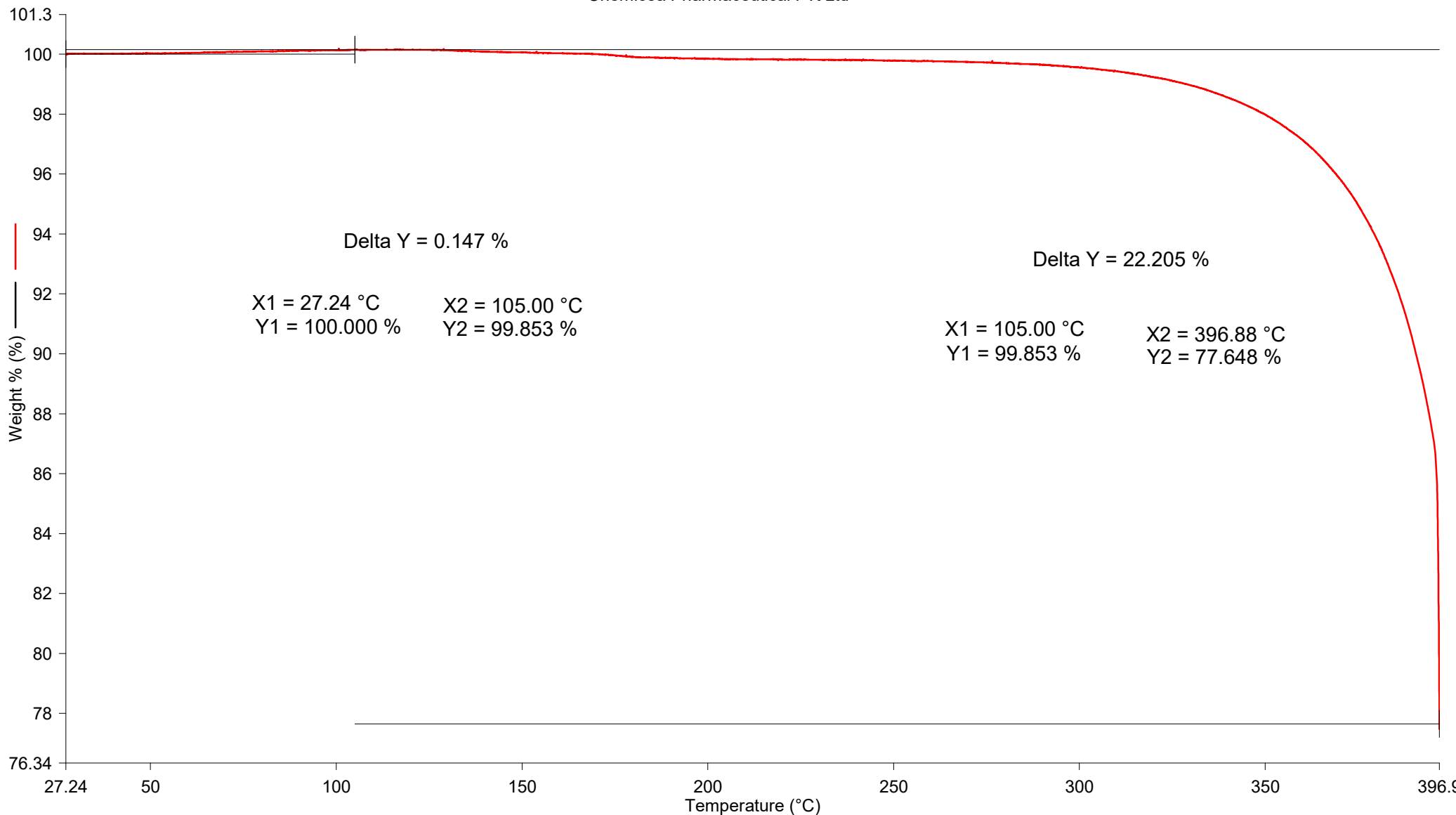
<b>Wave Number (cm-1)</b>	<b>Type of Bond</b>
1500.35	C=C Stretching
1509.99 and 1362.46	N=O Stretching
1034.62	C-F Stretching
1161.9	C-O-C Stretching

Filename: C:\Progra...\CP-PAR-NIT-0624-24-06-024.tg1d  
Data Collected: 6/24/2024 5:50:59 PM  
Baseline Filename:  
Operator ID: Shubham  
Sample ID: CP-PAR-NIT-0624  
Sample Weight: 3.492 mg  
Comment:

CP-PAR-NIT-0624: CP-PAR-NIT-0624-24-06-024  
Weight % (%) : Steps: 1-2



Chemicea Pharmaceutical Pvt Ltd



6/24/2024 5:56:34 PM

1) Heat from 30.00°C to 400.00°C at 10.00°C/min 18 2) Hold for 1.0 min at 400.00°C

**Results:** A 0.147% loss due to volatiles was detected at the end of the analysis.