Supporting Information

Design, Synthesis and Diversification of Natural Product-Inspired Hydantoin-Fused Tetrahydroazepino Indoles

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Table of contents

I The Role of N-methyl-D-aspartic acid (NMDA) receptor in schizophrenia

The aberrant regulatory mechanism of glutamate transmission on N-methyl-D-aspartic acid (NMDA) receptor has been reported as one of the neuropathology in schizophrenia.[1] The receptor is a heterotetramer composed of two structure subunits of NMDA receptor 1 (NR1) and NR2.[2] The extracellular domain of these two subunits were responsible for modulatory and ligand binding functions, where the NR1 binds the co-agonist glycine, and the NR2 binds the neurotransmitter glutamate. The membrane channel domain is responsible for the entrance of calcium ion. The receptor requires the binding of glutamate from NR2 subunit to activate the receptor, and requires the co-agonist of glycine binding for the efficient opening of the ion channel.[3] Modulation the glycine binding site of NMDA receptor may improve cognitive function and negative symptoms in schizophrenia.[4] D-amino acid oxidase (DAO) was found to be involved in the activation process of the NMDA receptor.[5] The substrates of DAO, especially the D-amino acid of D-serine, may bind the glycine site of the NMDA receptor as a co-agonist.[6,7] This in turn may regulate the NMDA receptor in opening its calcium channel. Moreover,

D-serine has also been found to inhibit the α-amino-3-hydroxy-5-methyl-4-isoxazole propionic acid (AMPA) receptor-mediated current in rat hippocampus neurons.[8] Thus, DAO may play role in the pathogenesis of schizophrenia.

D-serine is a full agonist at the allosteric glycine binding site of the NMDA receptor, and was reported to improve negative, cognitive symptoms, and symptoms poorly addressed by the standard D2 antagonist in schizophrenia.[9] Inhibition of DAO became essential to increase D-serine levels in the brain. The DAOI does not seem to be used alone in the treatment of schizophrenia, a meta-analysis evaluating the potential of modulators of the NMDA receptor as adjunctive therapy for schizophrenia in 29 published clinical trials was reported that D-serine, glycine structure analogue of mucolytic agent N-acetyl-cysteine (NAC) and glycine transporter inhibitor sarcosine as adjuncts to non-clozapine antipsychotics may benefit the treatment of negative and total symptoms of chronic schizophrenia.[10] In our ethnic group, the glycine transporter inhibitor sarcosine has been reported with better adjunctive than D-serine for long-term stable schizophrenia and acute schizophrenia[11], and high dose may be essential for acute schizophrenia.[12] D-alanine may be a

better adjunctive.[13] These results suggest indirect NMDA glycine site treatment may be better than direct full agonist, especially in our population.

Sodium benzoate is a bacteriostatic and fungistatic food preservative. It is a safe and easy to obtain DAO inhibitor. It was put in a clinical trial with doses of above 1g and showed beneficial results in the treatment of schizophrenia.[14] Although in the short term up to six weeks of clinical trial, it did not showed toxic effects. However, it has been reported that sodium benzoate under the combination with ascorbic acid (vitamin C) may form benzene, a known carcinogen (http://www.fda.gov/Food/FoodSafety/FoodContaminantsAdulteration/ChemicalContaminants/Benzene/ucm055815.htm). The Food Standards Agency (FSA) at United Kingdom even suggested that sodium benzoate combining with certain artificial colors may be linked to hyperactivity behavior (http://www.myomancy.com/2007/09/food-colorings-and-hyperactivity/). This indicates that DAOI better than sodium benzoate is essential to meet the treatment need.

References:

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(10) Singh, S. P.; Singh,V. *CNS Drugs*, **2011**, *25*, 859-85.

(11) Lane, H. Y. *Arch. Gen. Psychiatry*, **2005**, *62*, 1196-204.

(12) Lane, H. Y. *Biol. Psychiatry*, **2008**, *63*, 9-12.

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II. DAO enzymatic assay. Porcine kidney DAO (pkDAO) was used in the DAO enzymatic assay**.** A purified DAO enzyme was obtained from porcine kidney (Sigma-Aldrich, USA). The DAO enzymatic activity assay was modified as specified by Oguri *et al*.[1] DAO activity was measured by using substrate *D*-alanine reaction-produced hydrogen peroxide (H_2O_2) to further react with 3-(4-hydroxyphenyl) propionic acid (HPPA). HPPA was oxidized by H_2O_2 and peroxidase to become the fluorogenic dimer measured to represent DAO activity. For porcine kidney DAO, the DAO substrate was prepared in 50 mM *D*-alanine (dissolved in 0.2 M Tris–HCl buffer, pH 8.3). A 100 ul of *D*-alanine solution was mixed with 4 ul (in 100%) dimethyl sulfoxide, DMSO) of different concentrations of candidate compounds ranging from 12.21 uM, 24.41 uM, 48.83 μM, 97.66 μM, 195.31 μM, 390.63 μM, 0.78 mM, 1.56 mM, 3.13 mM, 6.25 mM, 12.50 mM, and 25.00 mM. Ten microliters of *D*-alanine and candidate compound mixture was incubated with 220 ul of Reaction Master Mix in black 96 well plate at 37℃ for 5 min. The

Reaction Master Mix contained 110 ul of 5 U/mL porcine kidney DAO (Sigma-Aldrich, USA) solution (dissolved with 0.2 M Tris–HCl buffer, pH 8.3), 1.1 mL of 15 U/mL peroxidase solution (dissolved with 0.2 M Tris–HCl buffer, pH 8.3), 1.1 mL of 20 mM HPPA solution (dissolved with 0.2 M Tris–HCl buffer, pH 8.3), and 2.2 ml of 2 M Tris–HCl buffer (pH 8.3) for 110 reaction assays.

Fluorescence intensity (Fs) was measured at 405 nm by irradiation excitation at 320 nm. The higher the DAO enzymatic activity was, the higher the fluorescence intensity. The fluorometric inhibition indicator (Fi) was obtained from the following equation: $Fi = (Fs- F_{Drug})/ (F_{DMSO})$, where the fluorescent drug blank (F_{Drug}) was measured in the drug mixture solution (using 0.2 M Tris HCl buffer, pH 8.3, without *D*-alanine). A DMSO blank (F_{DMSO}) was measured under a 100% DMSO solution. Although FAD is generally included in the reaction mixture in the *D*-amino acid oxidase assay since it easily dissociates from the holoenzyme, the present method was performed without FAD. The inhibitory effect of DAO inhibitors was compared by using inhibitory concentrations leading to 50% inhibition of DAO activity (IC_{50}). For inhibitory constant (*Ki*) estimation, seven different final concentrations of

substrate *D*-alanine, ranging from 522.6, 1045.2, 2090.3, 4180.6, 8361.2, 16722.4, and 33444.8 μM, were incubated with 3 drug concentrations of compound. The IC_{50} and inhibitory constant (*Ki*) were calculated by nonlinear regression model using GraphPad Prism, version 5 (GraphPad Software, Inc., La Jolla, CA).[2,3]

In this study, hydantoin fused tetrahydroazepino [4,5-*b*]indoles derivatives were screened for their porcine DAO inhibitory effects. Compound **5** had shown the strongest DAOI effect with IC50 of 4.5 ± 1.04 μM. It showed competitive inhibitory effect with the DAO substrate of D-alanine. Further impact of compound **5** effect on the negative and cognitive symptoms of schizophrenia animal analyses is essential to confirm for its role as a novel antipsychotics.

References:

(1) Oguri, S. *Food chemistry*, **2007**, *100*, 616-622.

(2) Halgren, T. A. *Journal of Computational Chemistry*, **1996**, *17*, 490-519.

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Table 1. The inhibition screening of compounds on porcine kidney DAO (pkDAO).

Compound	M.W.	Screening at final Conc. 20.8 uM	
		DMSO = 1	Na-Benz = 1
7a	381.42	0.89	0.95
7b	435.85	0.91	0.97
7c	447.5	0.72	0.77
7d	413.49	0.76	0.80
7e	446.41	0.63	0.67
7f	462.47	0.68	0.72
7g	383.46	0.76	0.80
7h	431.5	0.84	0.90
7i	582.55	0.65	0.69
7j	393.43	0.62	0.66
7k	355.41	0.88	0.94
7l	409.5	0.67	0.71
7m	431.5	0.77	0.81
7n	417.48	0.71	0.75
7o	407.46	0.93	0.98
7p	431.44	0.88	0.93
7q	383.46	0.58	0.61
7r	445.53	0.88	0.95
7s	423.52	0.59	0.62
5	314.33	0.09	0.10

Table 2. The IC50 of compounds on porcine kidney DAO (pkDAO).

Compound	pkDAO (uM)		
	IC_{50}	±	SEM
7l	20.406	±	1.057
5	4.460	±	1.036
7s	35.519	±	1.050
7i	25.783	±	1.046
7e	30.539	±	1.072
7j	29.029	±	1.045
7q	26.679	±	1.061

Figure 1. **Concentration−inhibition curves.** For compounds with porcine kidney DAO IC_{50} values, inhibition curves are shown. Compound No. are listed on the right. (each data point represented an average ± triplicates of standard error mean; SEM)

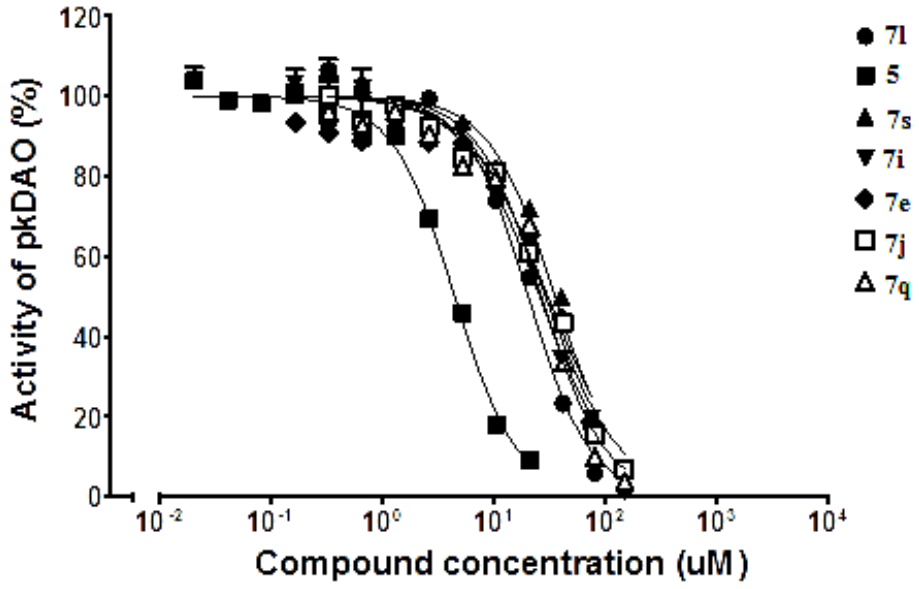
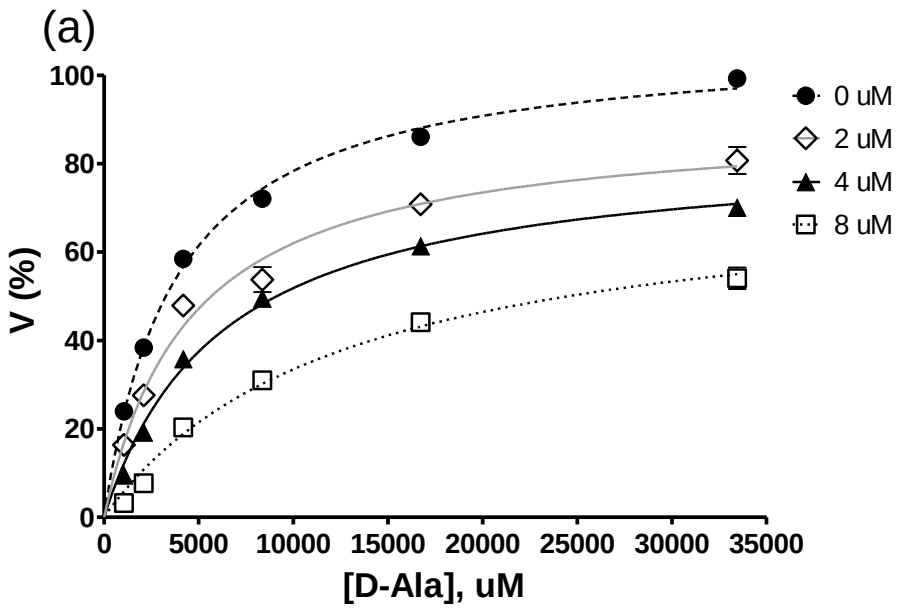
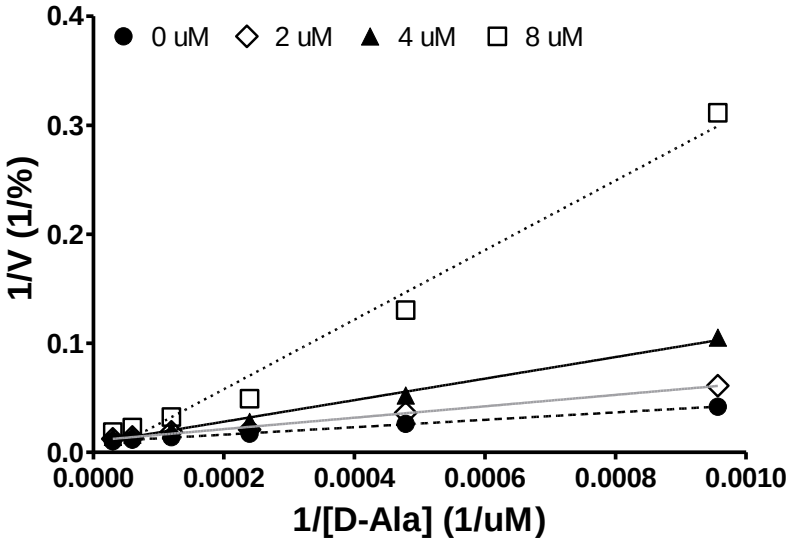


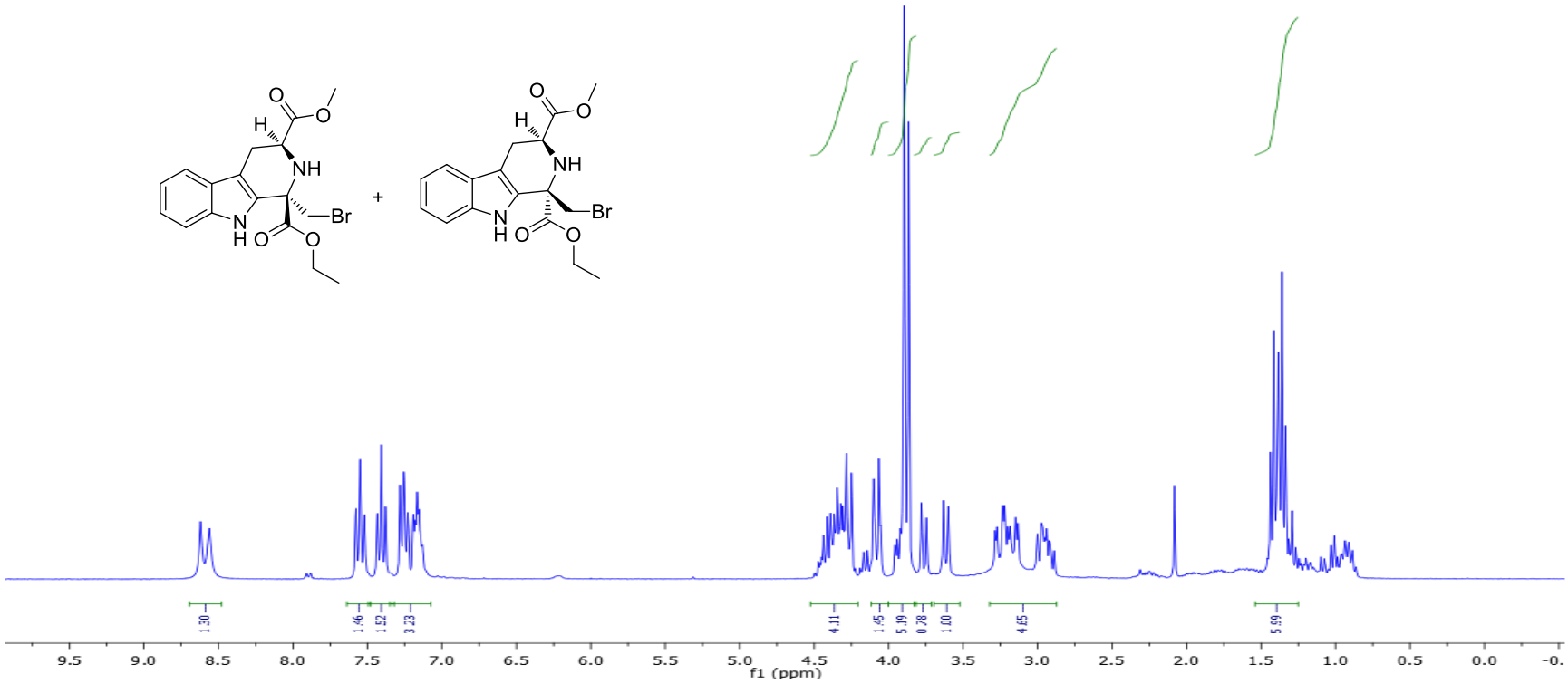
Figure 2. The inhibition mechanism of compound **5** on porcine kidney DAO was determined through **(a)** Michaelis-Menten and **(b)** Lineweaver-Burk diagram of *D*-alanine substrate competition. The absence (dotted line) or presence of compound **5** at concentrations of 2 μM (grey), 4 μM (black) and 8 μM (dotted) are presented. The percentage velocity data were normalized to the highest substrate [*D*-Ala] concentration of 33444.8 μM in the absence of inhibitor. (each data point represented an average ± triplicates of standard error mean; SEM)



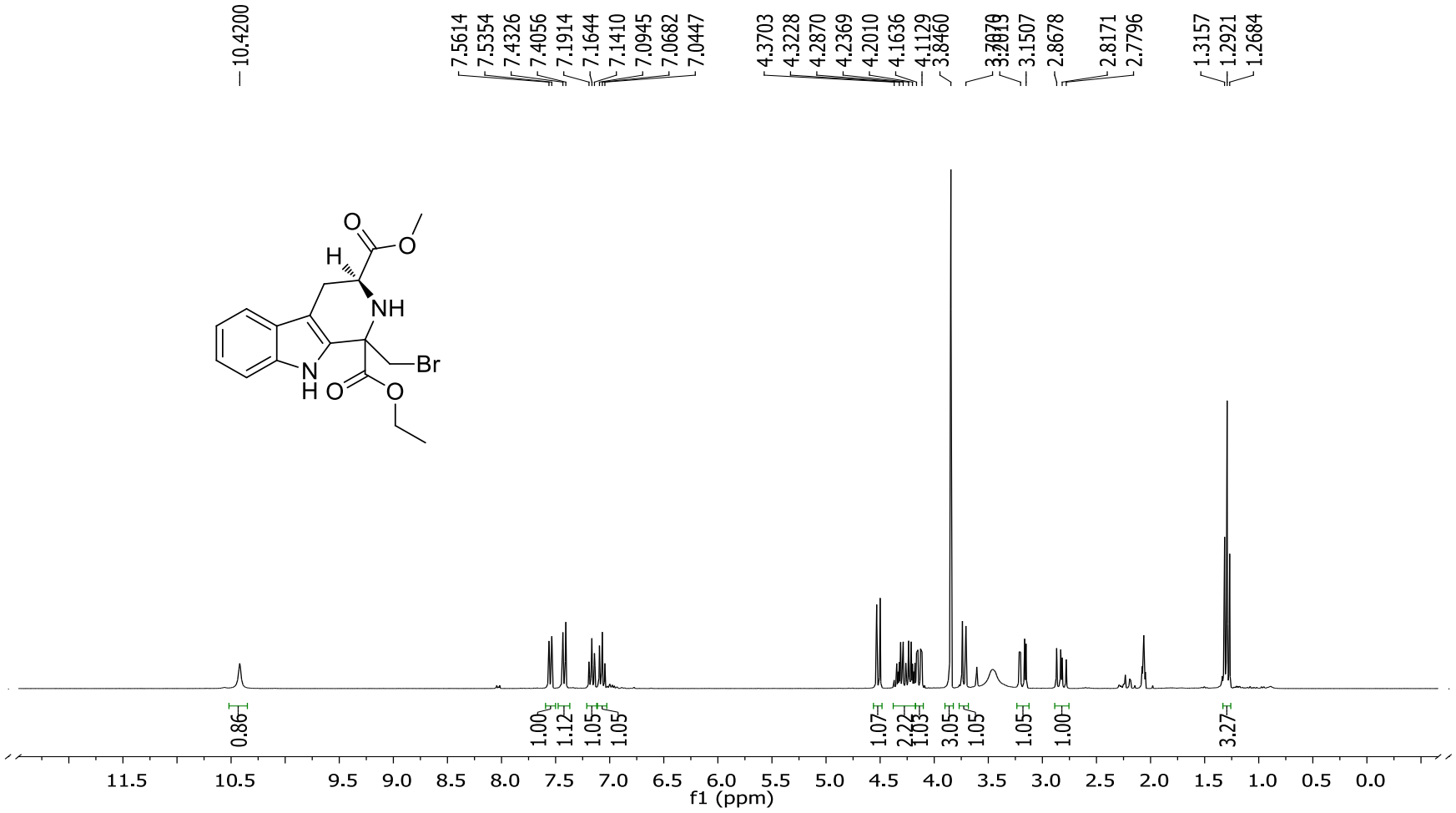
(b)


0 uM
2 uM
4 uM
8 uM
1/V (1/%)
0.4
0.3
0.2
0.1
0.0
0.0000
0.0002
0.0004
0.0006
0.0008
0.0010
1/[D-Ala] (1/uM)

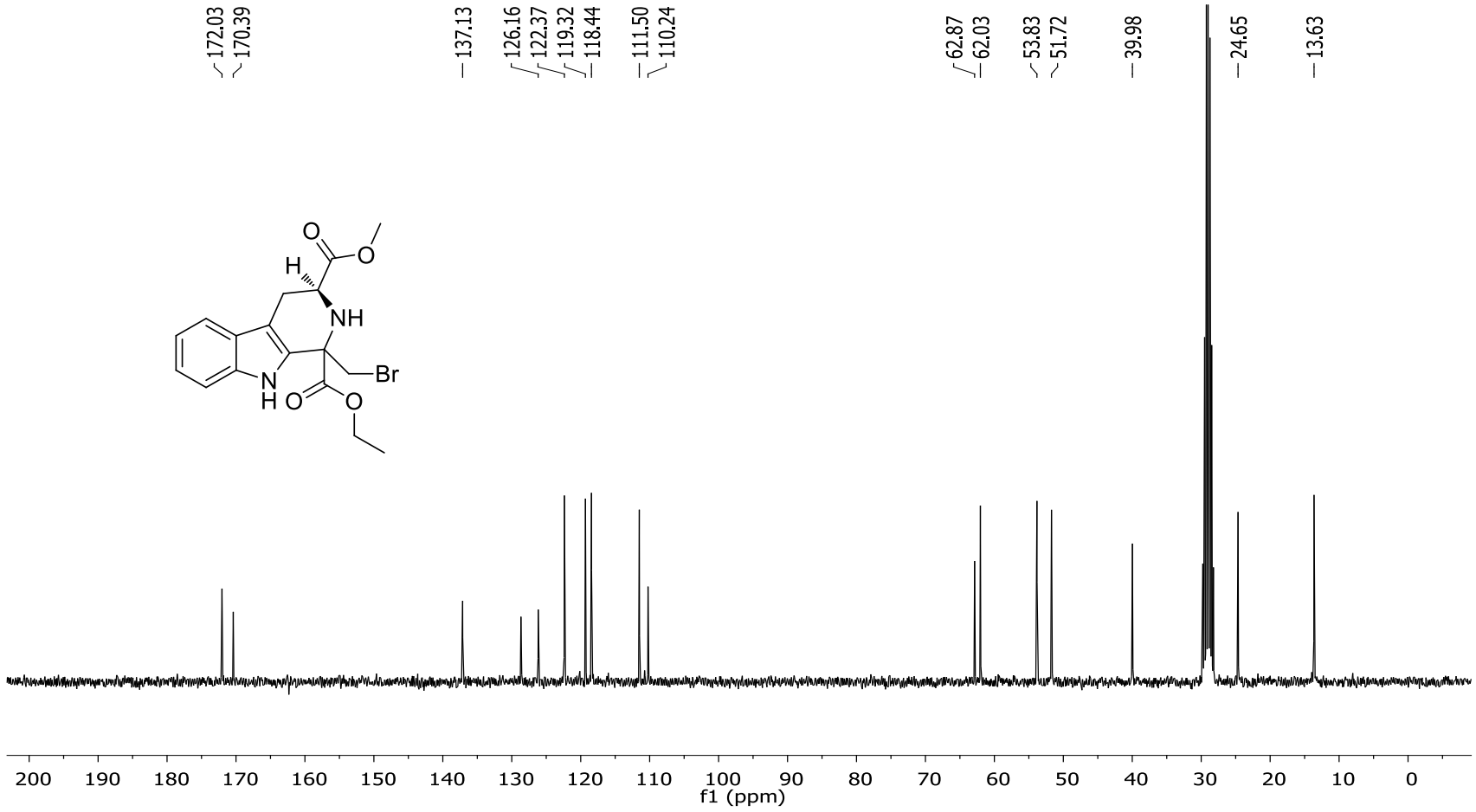
VII. ^{1}H NMR, ^{13}C NMR, LRMS, HRMS, IR spectra and HPLCs



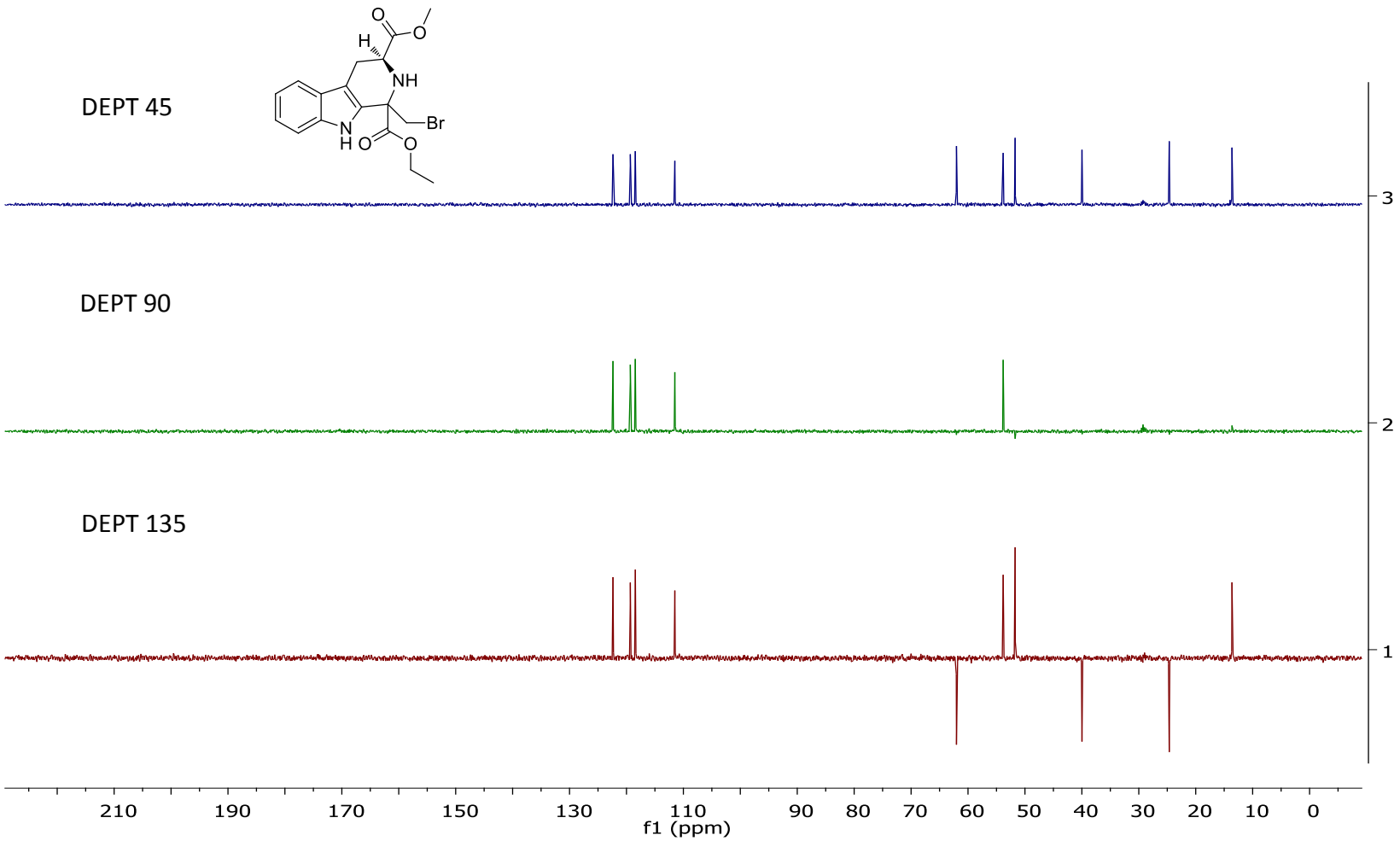
^{1}H NMR spectrum (300 MHz) of compound **4** (Diastereomeric mixture) in Acetone-d_6



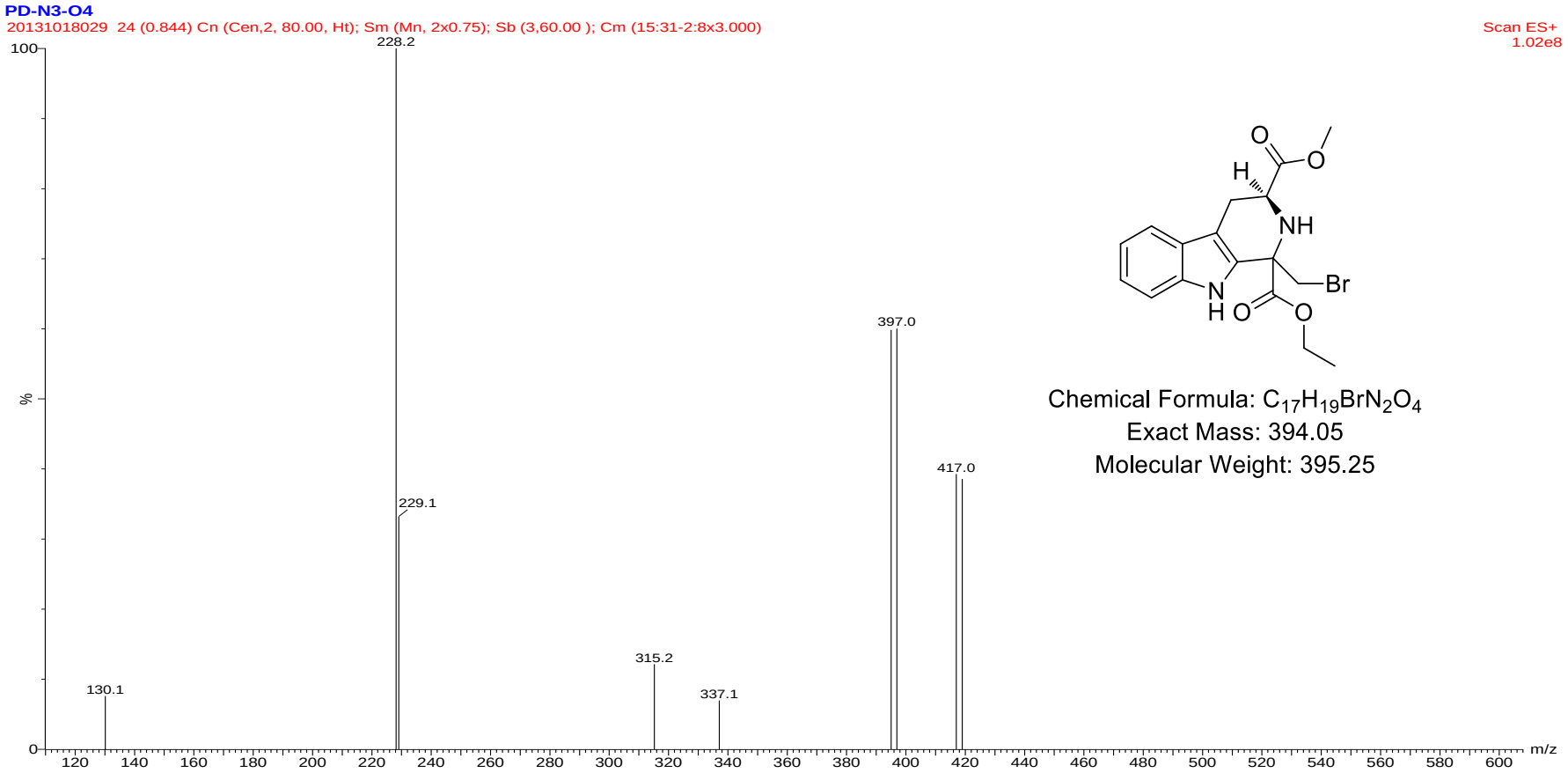
^{1}H NMR spectrum (300 MHz) of compound **4** (Single diastereomer) in Acetone-d_6



^{13}C NMR spectrum (75 MHz) of compound **4** (single diastereomer) in Acetone-d_6



DEPT NMR spectrum (75 MHz) of compound **4** (single diastereomer) in Acetone-d_6



ESI-LRMS of compound **4** (single diastereomer)

Display Report

Analysis Info

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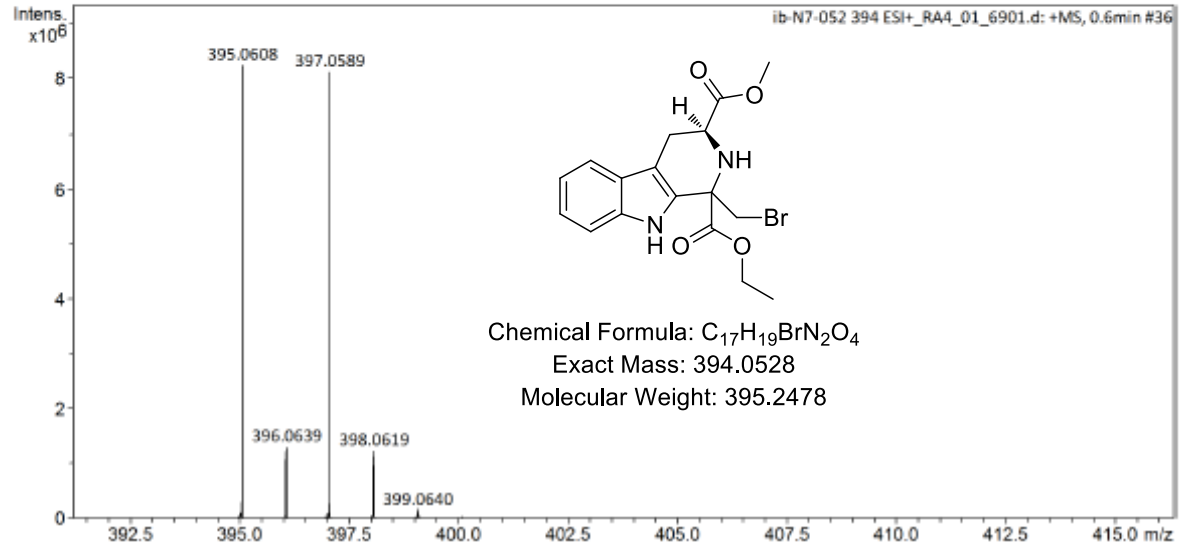
Method	Small molecule.m	Operator	NCTU
Sample Name	ib-N7-052 394 ESI+	Instrument	impact HD 1819696.00164
Comment			

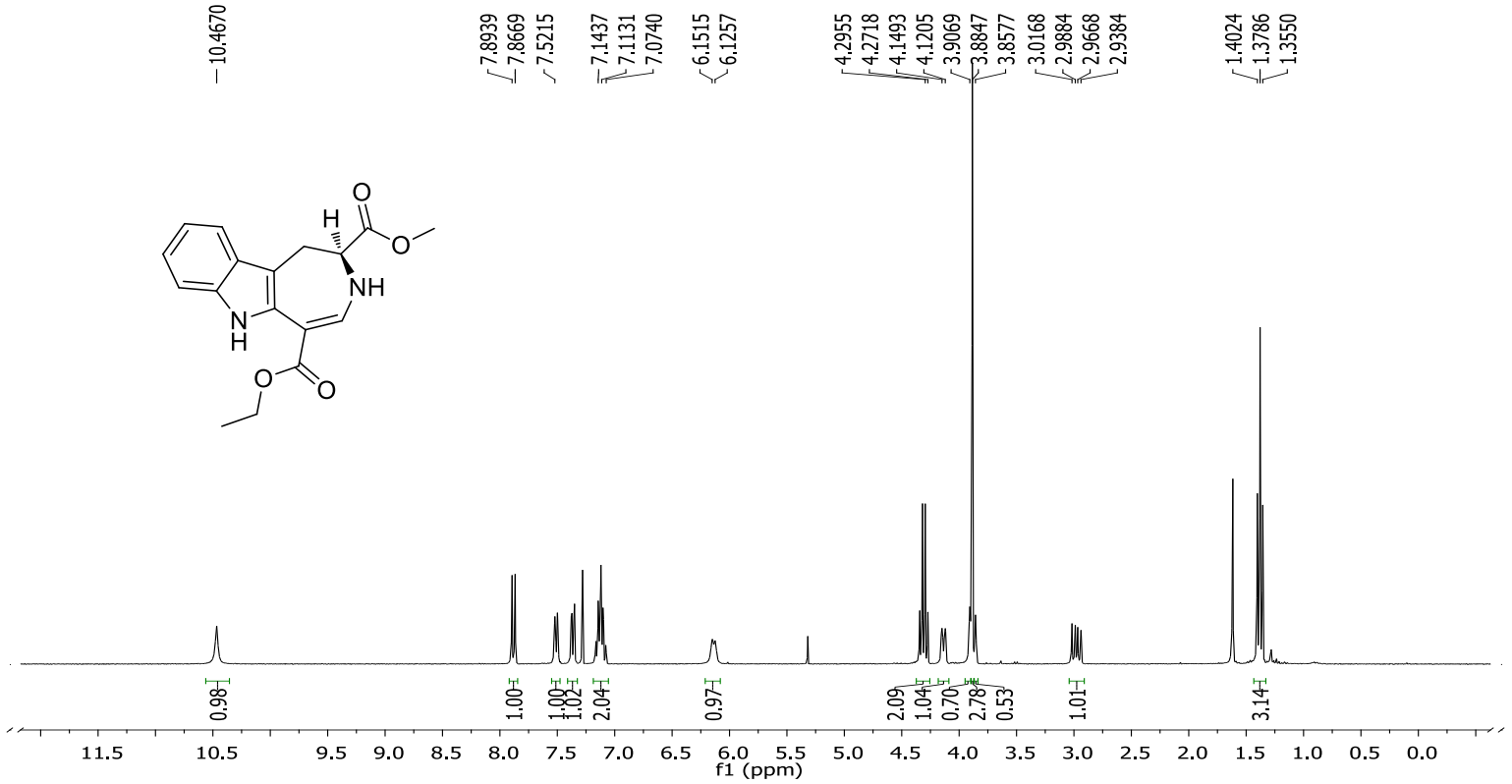
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C

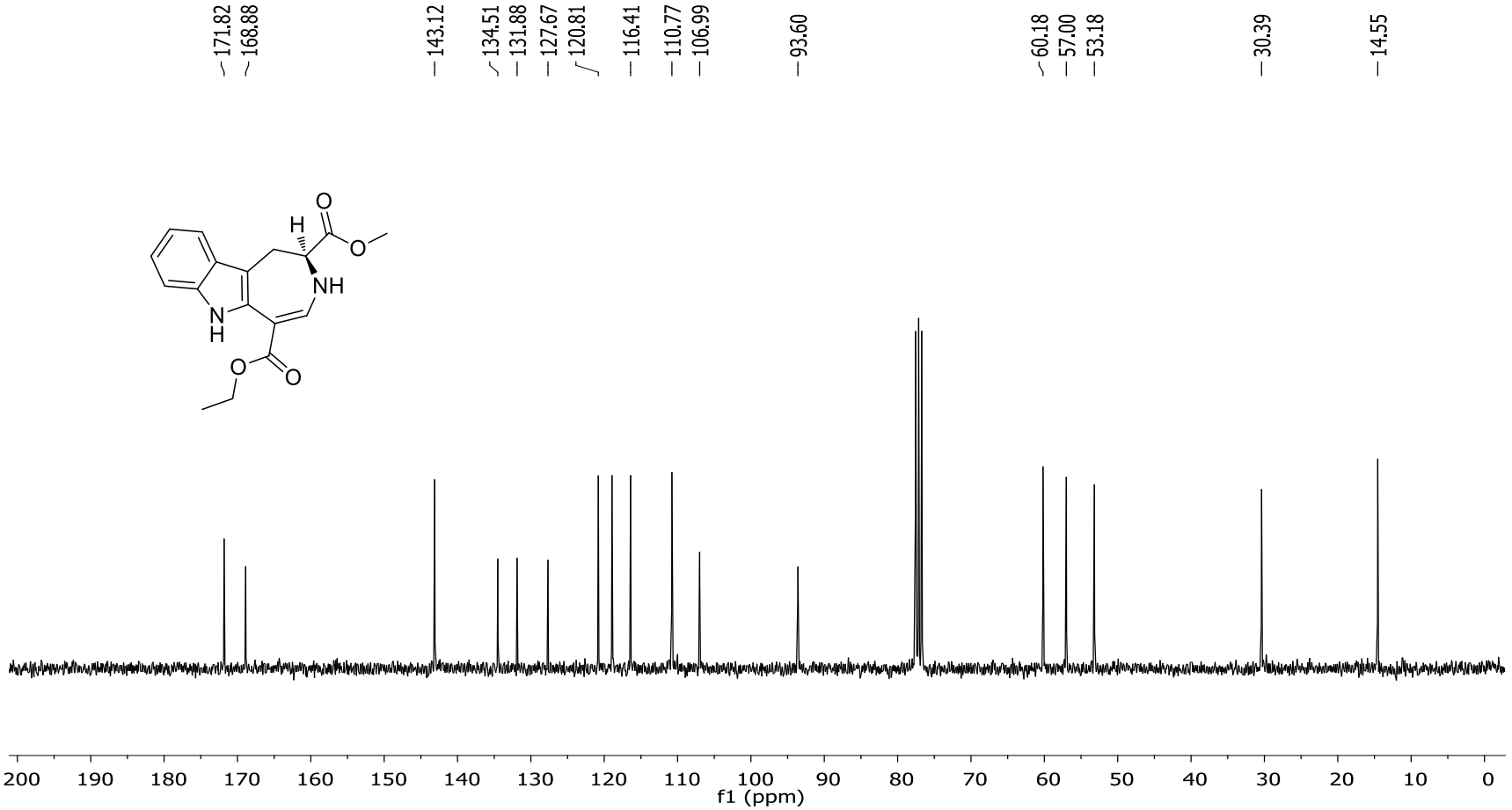


Chemical Formula: $C_{17}H_{19}BrN_2O_4$
Exact Mass: 394.0528
Molecular Weight: 395.2478

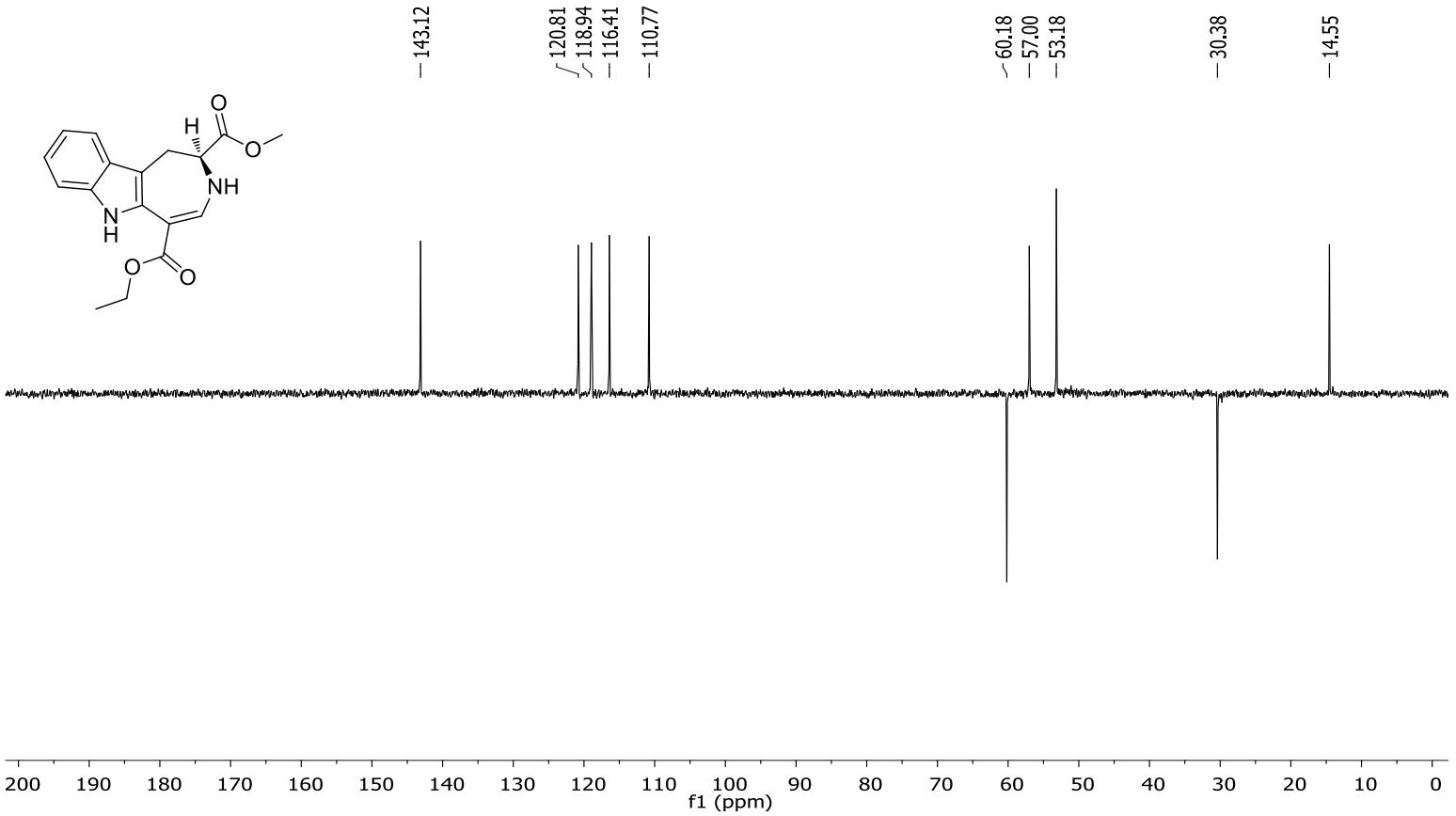
ESI-HRMS of compound **4** (single diastereomer)



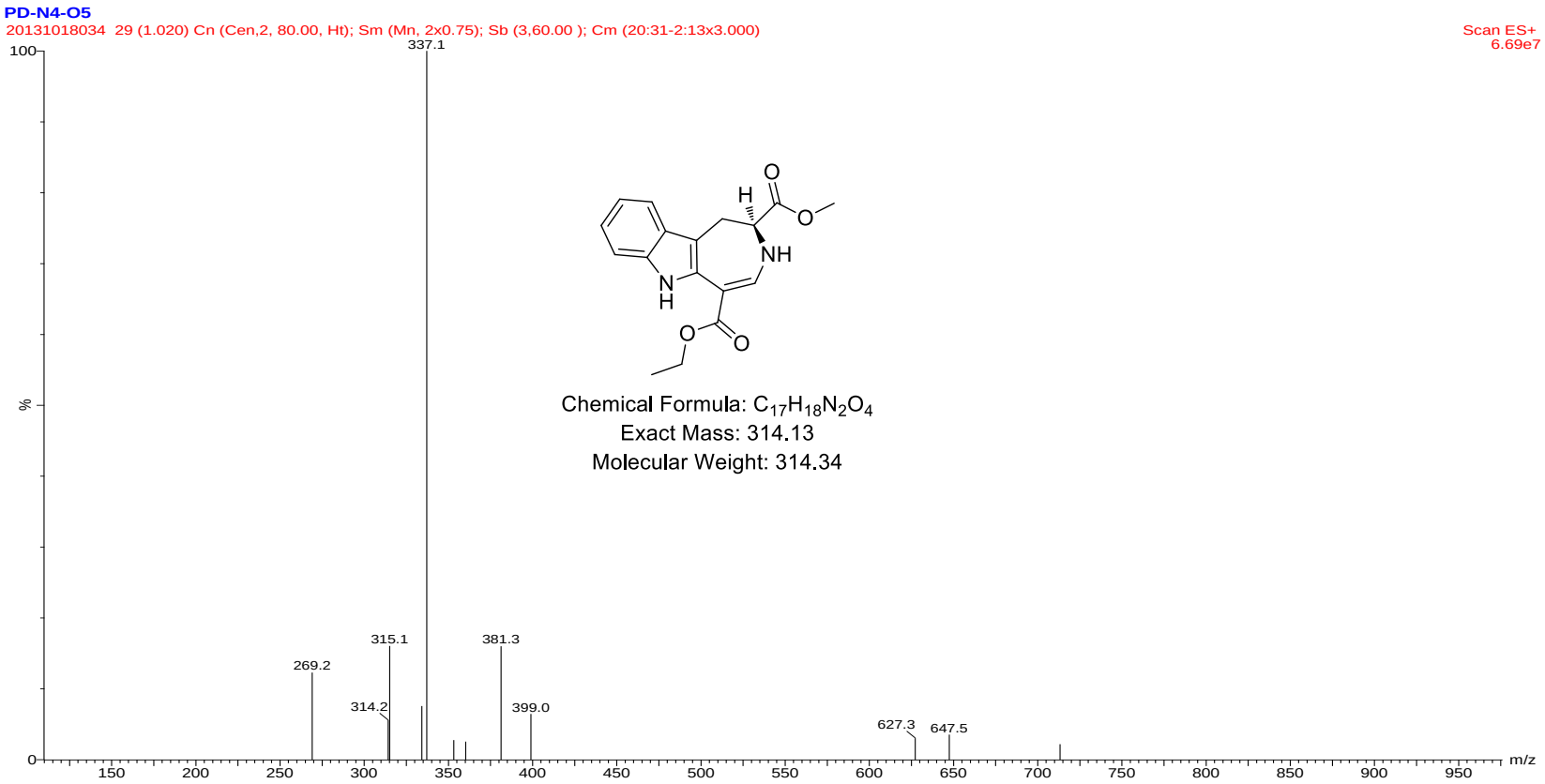
^{1}H NMR spectrum (300 MHz) of compound **5** in $CDCl_3$



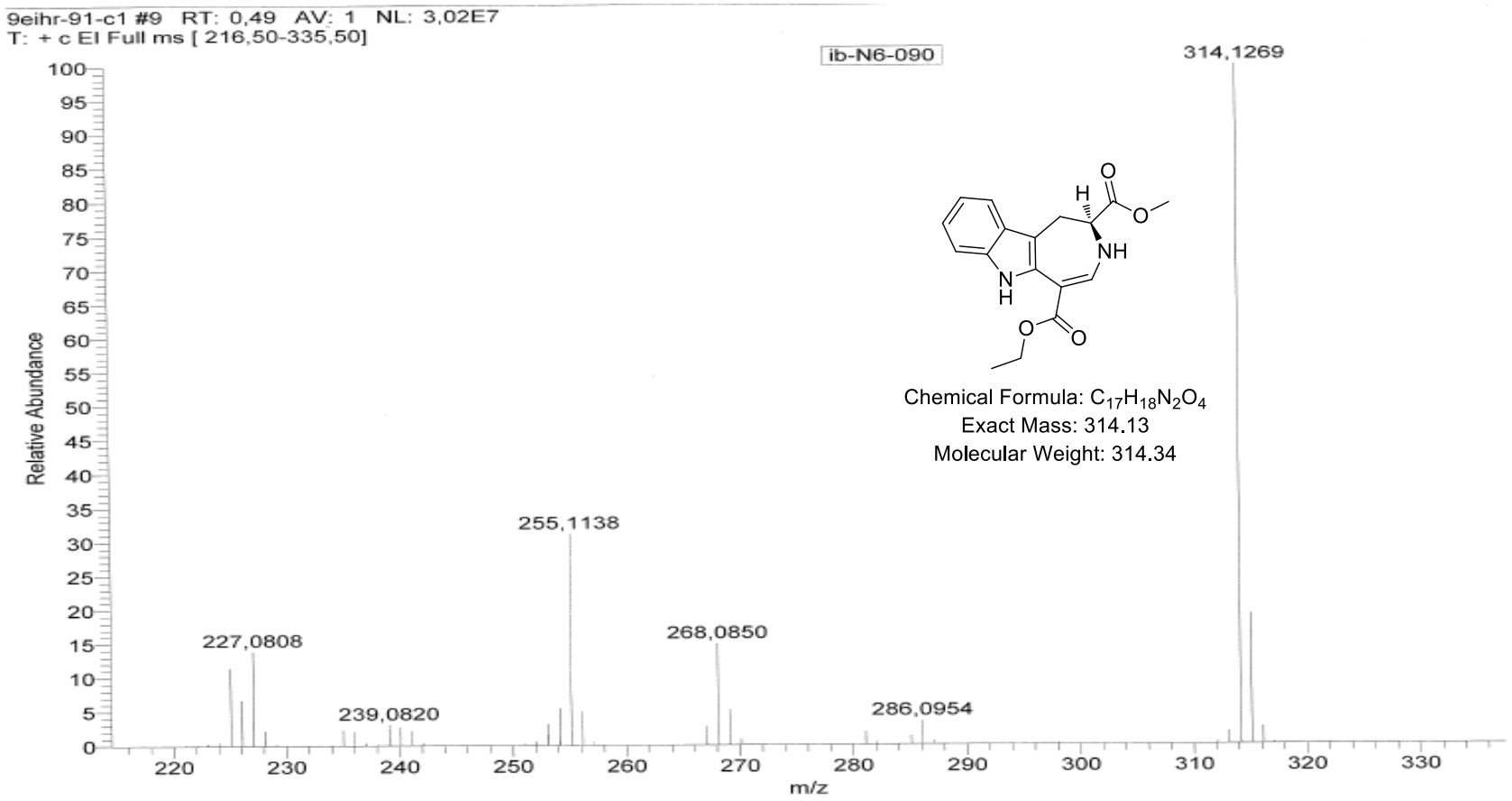
^{13}C NMR spectrum (75 MHz) of compound **5** in $CDCl_3$



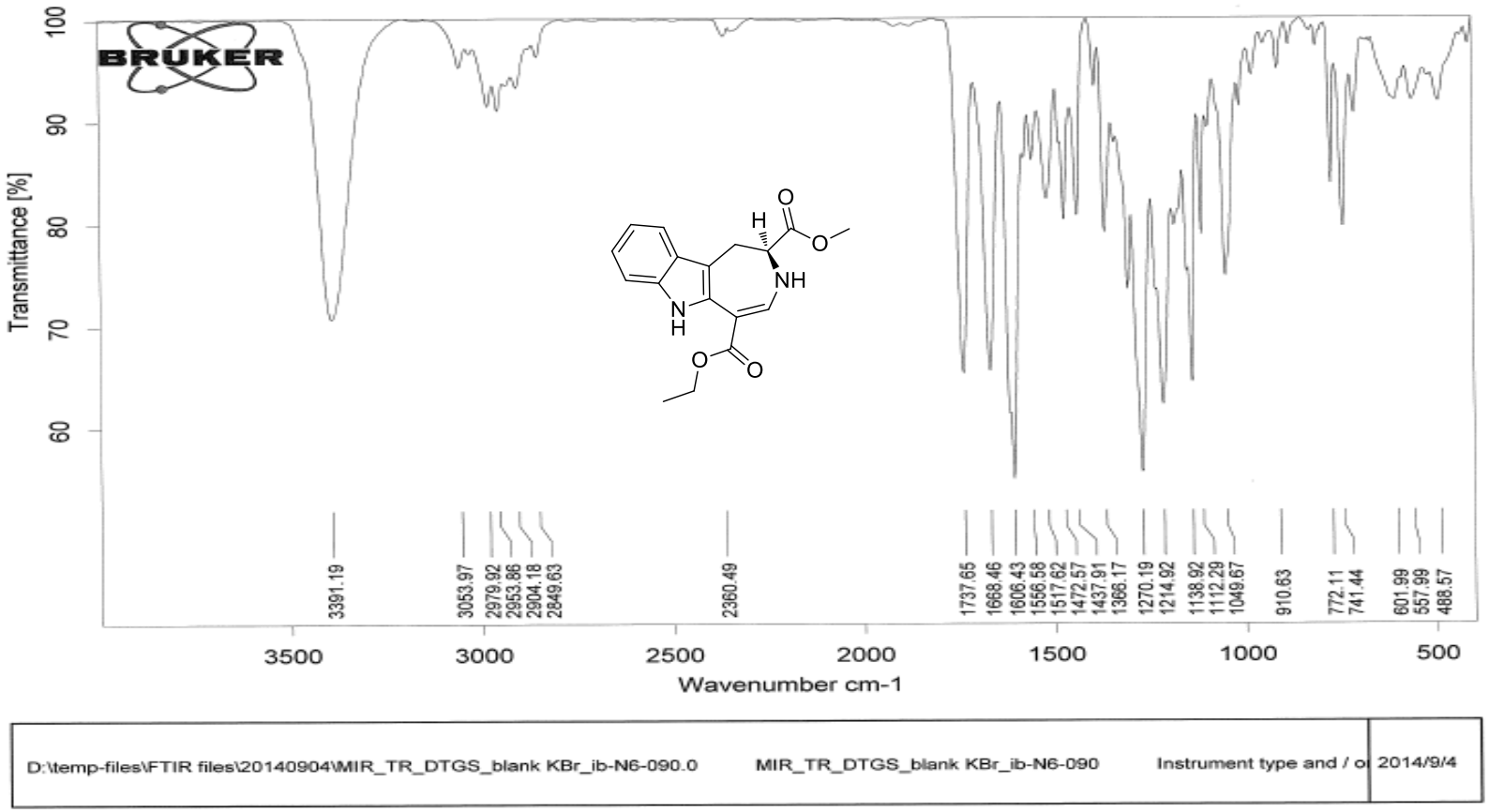
DEPT NMR spectrum (75 MHz) of compound **5** in $CDCl_3$



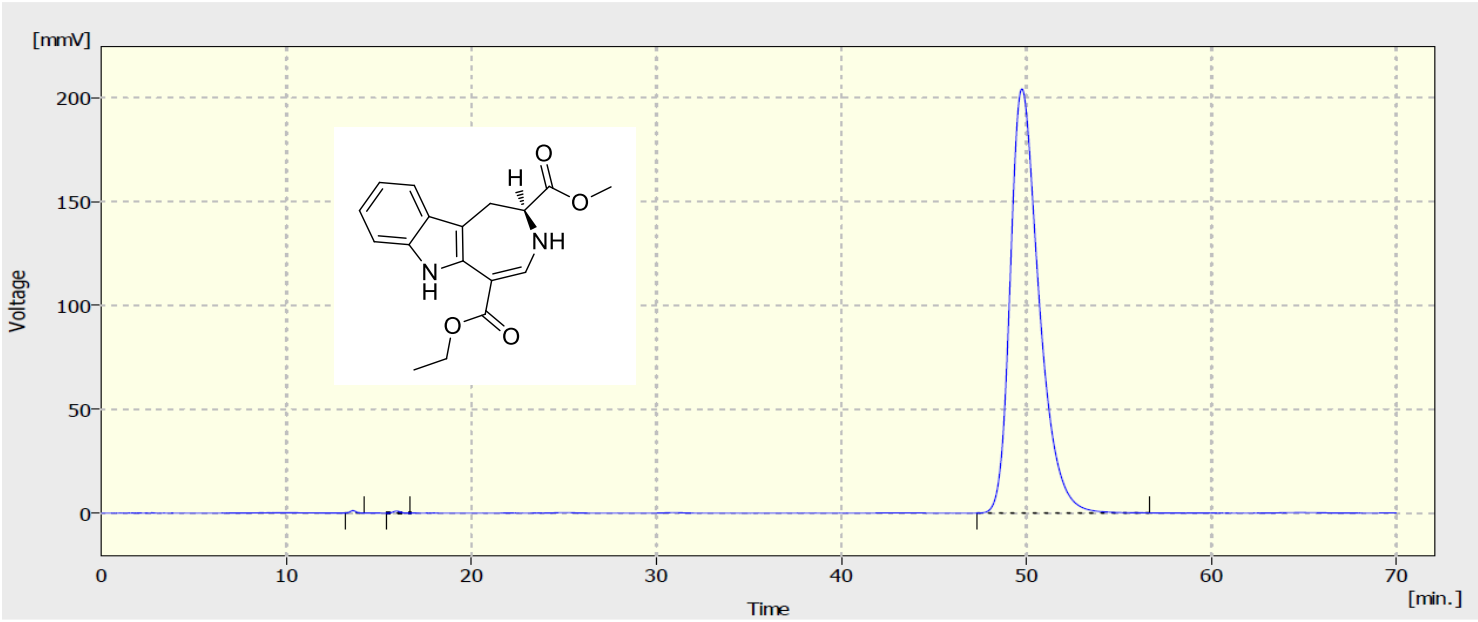
ESI-LRMS of compound **5**



EI-HRMS of compound **5**



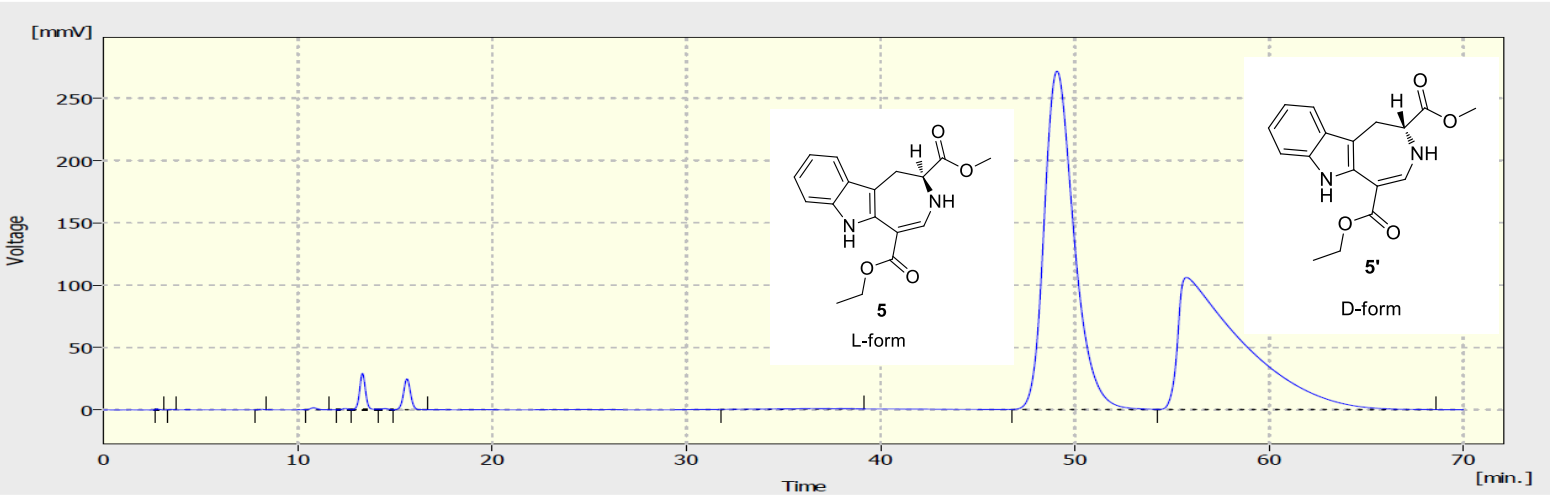
IR spectrum of compound **5**



Result Table (Uncal - D:\PRASHANT HPLC PDF\PD-N4-L-19-02)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	13.600	25.421	1.179	0.1	0.6	0.34
2	15.956	25.240	0.996	0.1	0.5	0.40
3	49.756	22114.626	204.143	99.8	98.9	1.62
	Total	22165.287	206.318	100.0	100.0	

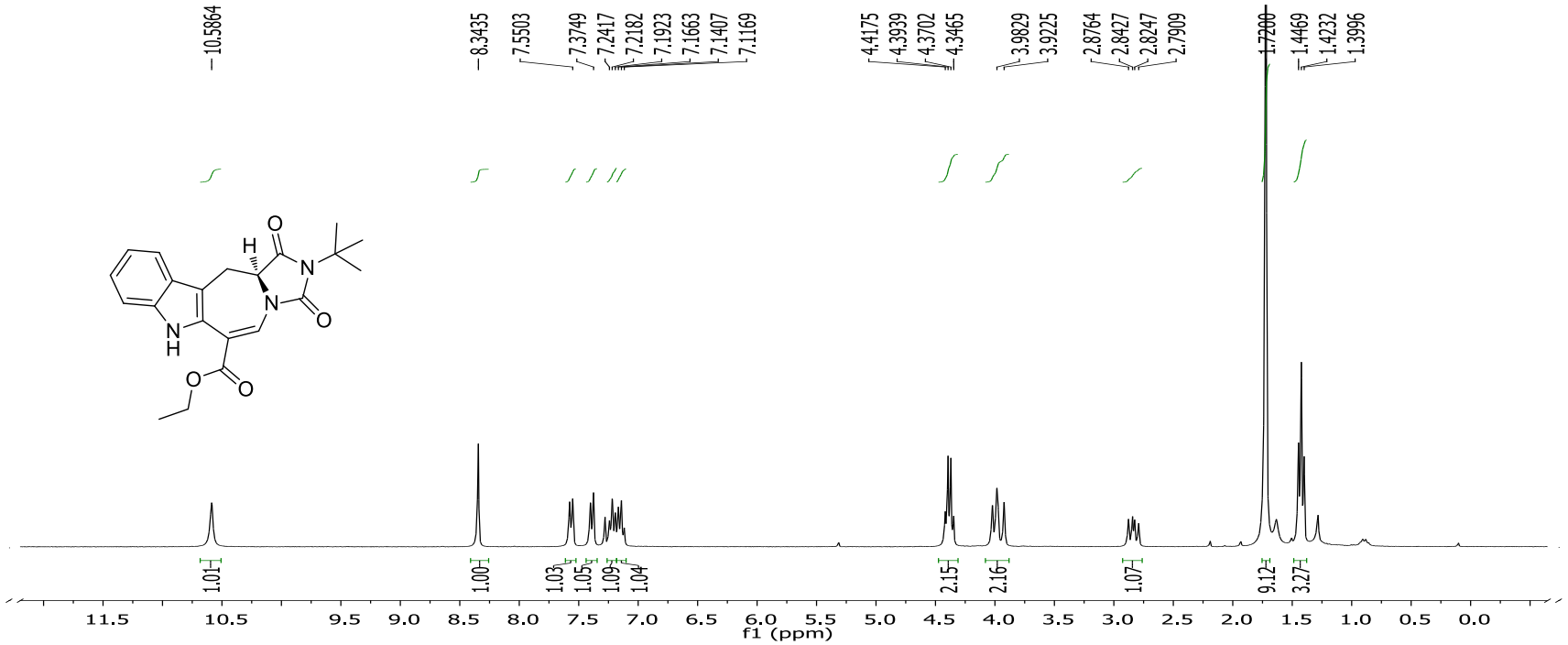
HPLC of compound **5**



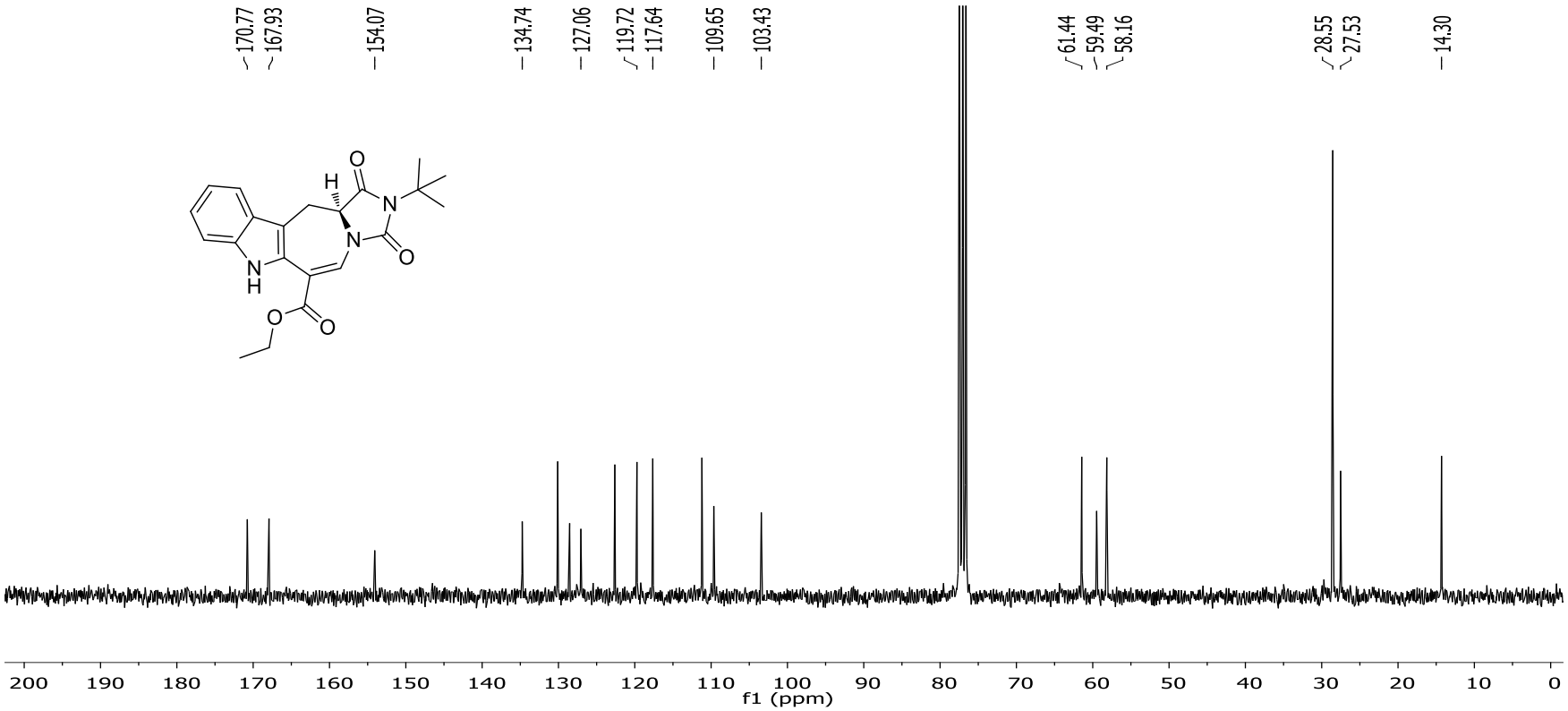
Result Table (Uncal - D:\PRASHANT HPLC PDF\PD-N4-DL-19-02)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	2.748	6.931	0.716	0.0	0.2	0.16
2	3.508	3.477	0.309	0.0	0.1	0.18
3	8.148	5.337	0.384	0.0	0.1	0.22
4	10.804	37.284	1.594	0.1	0.4	0.36
5	12.448	19.945	1.020	0.0	0.2	0.31
6	13.320	624.902	29.212	1.1	6.7	0.32
7	14.488	21.752	0.901	0.0	0.2	0.39
8	15.612	623.842	24.709	1.1	5.7	0.39
9	36.688	99.581	0.409	0.2	0.1	3.32
10	49.076	28951.541	271.332	52.3	62.2	1.61
11	55.736	24919.613	105.959	45.1	24.3	3.42
	Total	55314.204	436.547	100.0	100.0	

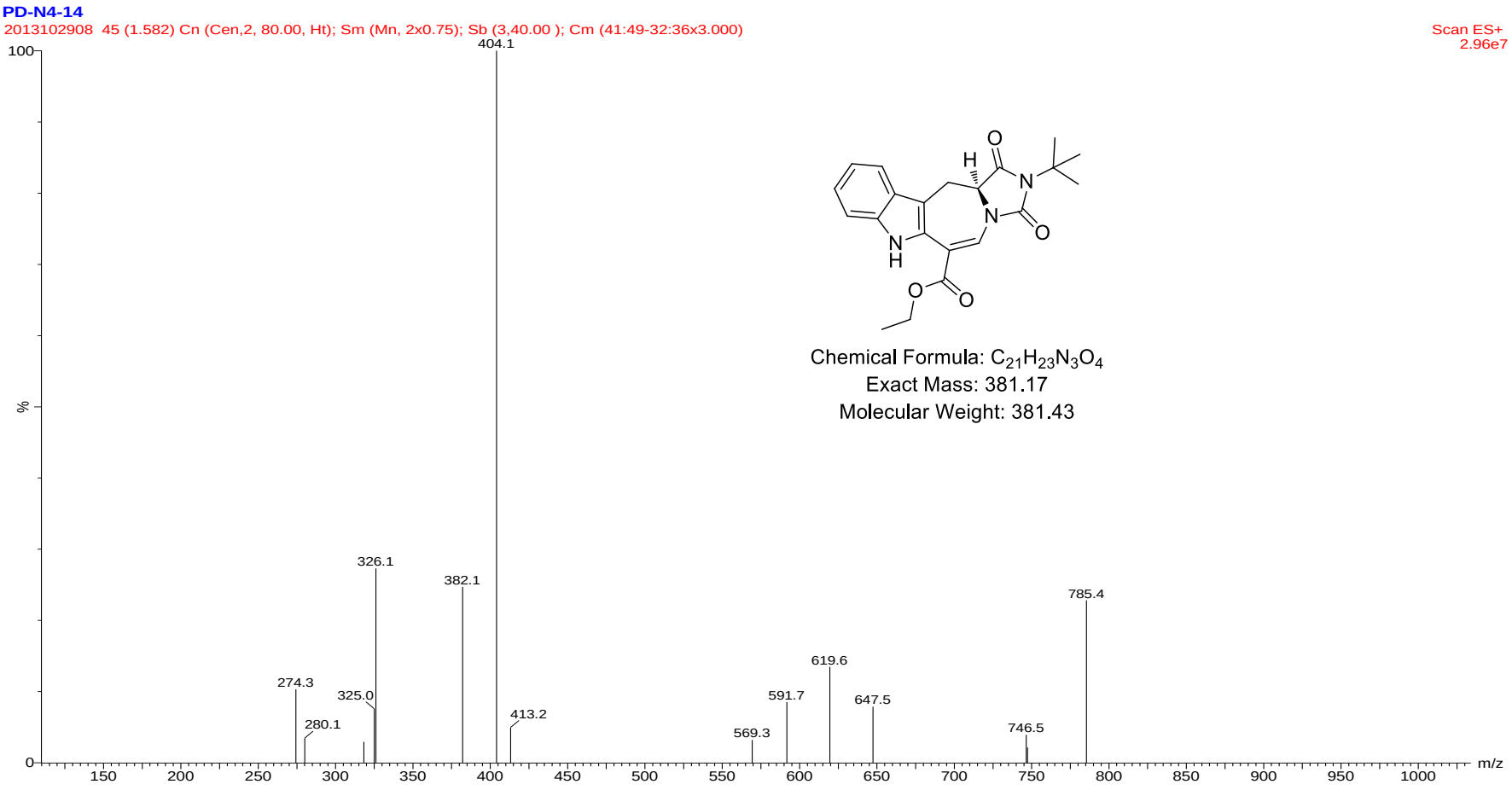
HPLC (co-injection) of compound **5** (L-form) and **5'** (D-form)



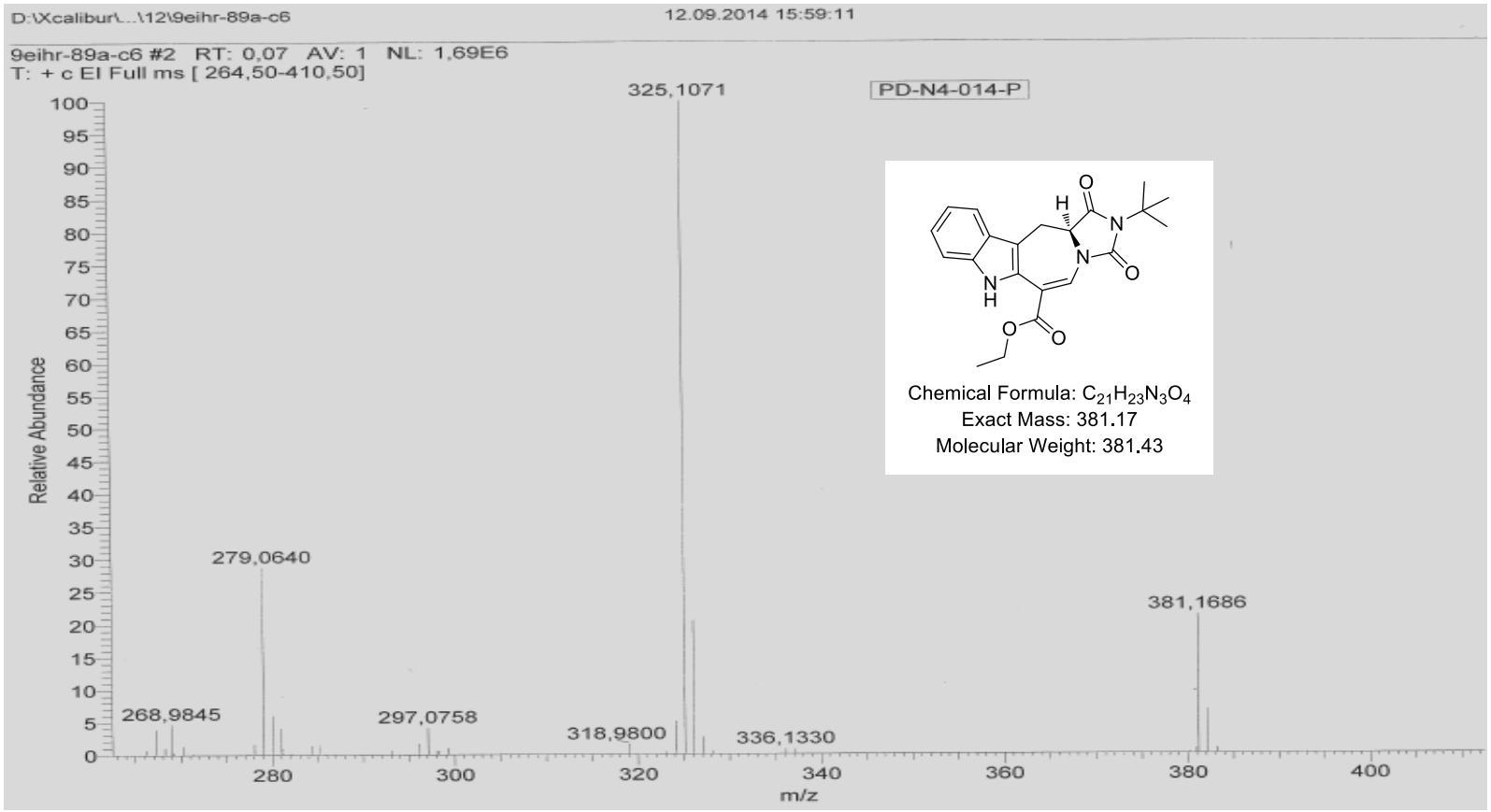
^{1}H NMR spectrum (300 MHz) of compound **7a** in $CDCl_3$



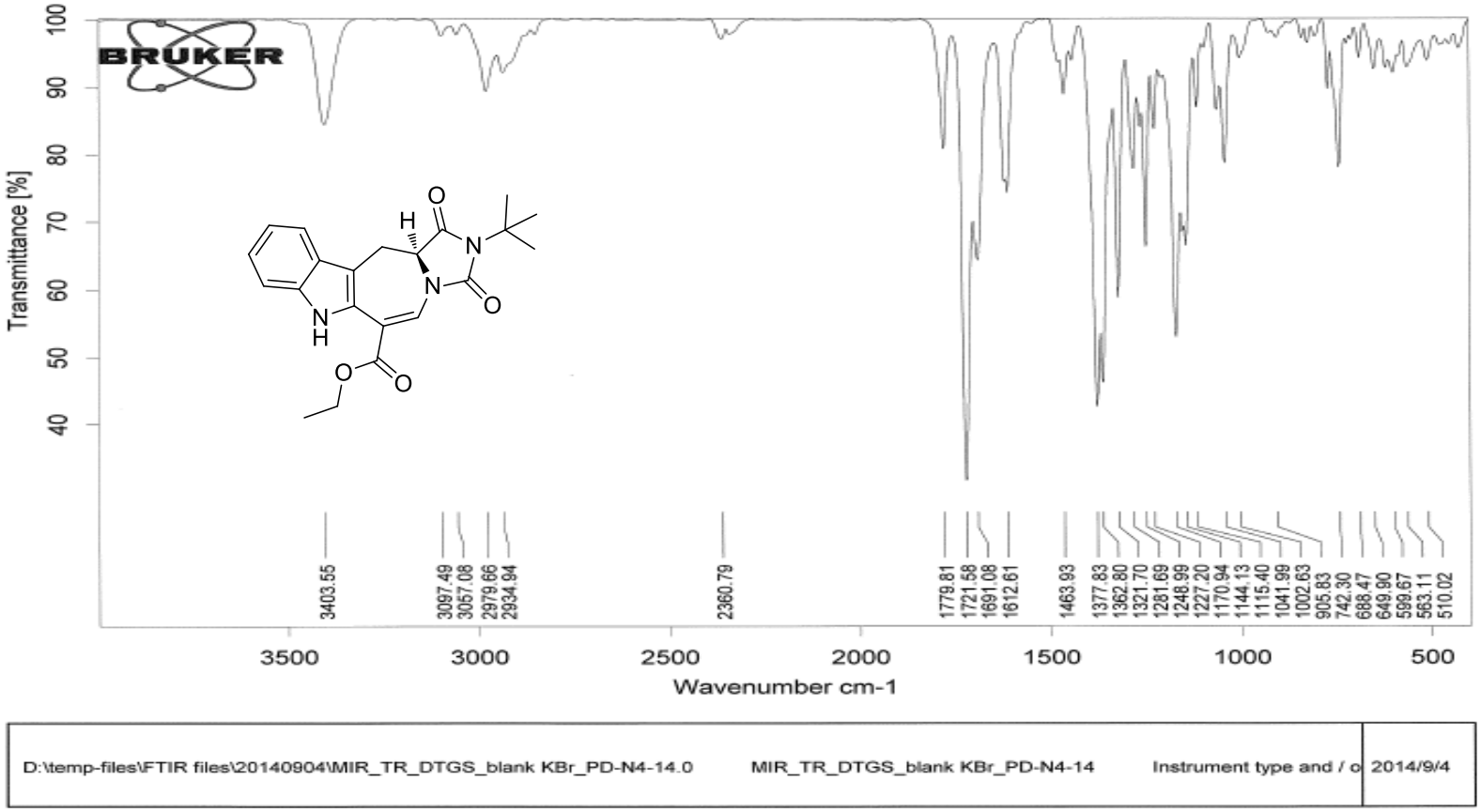
^{13}C NMR spectrum (75 MHz) of compound **7a** in $CDCl_3$



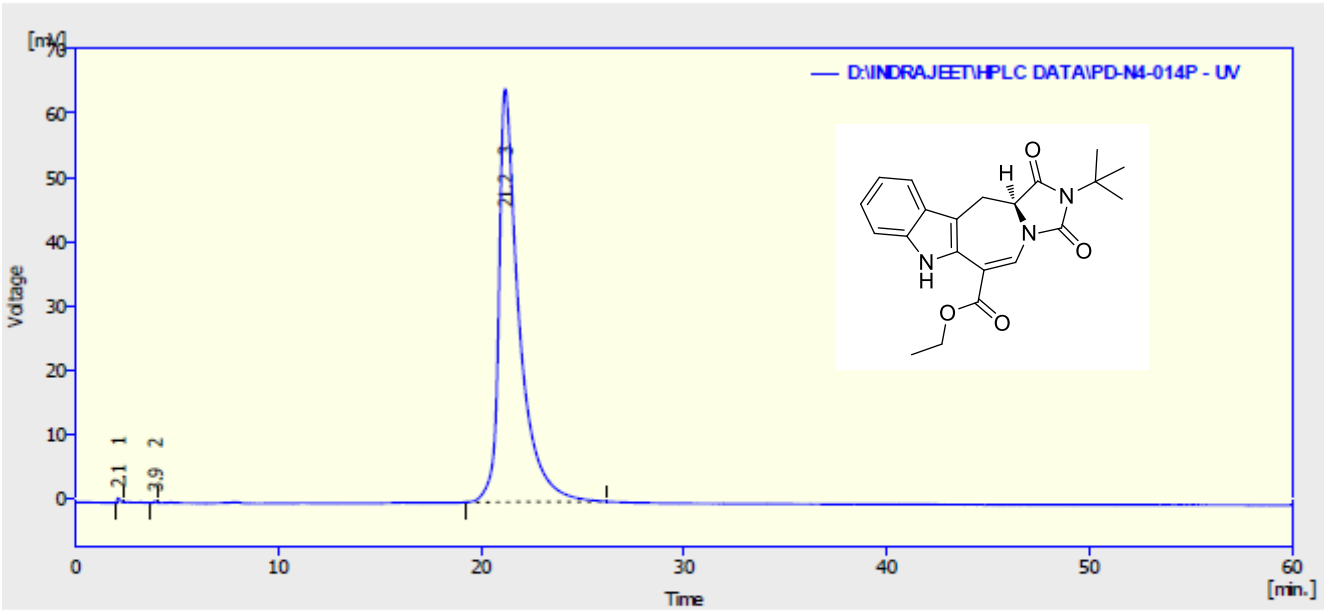
ESI-LRMS of compound **7a**



EI-HRMS of compound **7a**



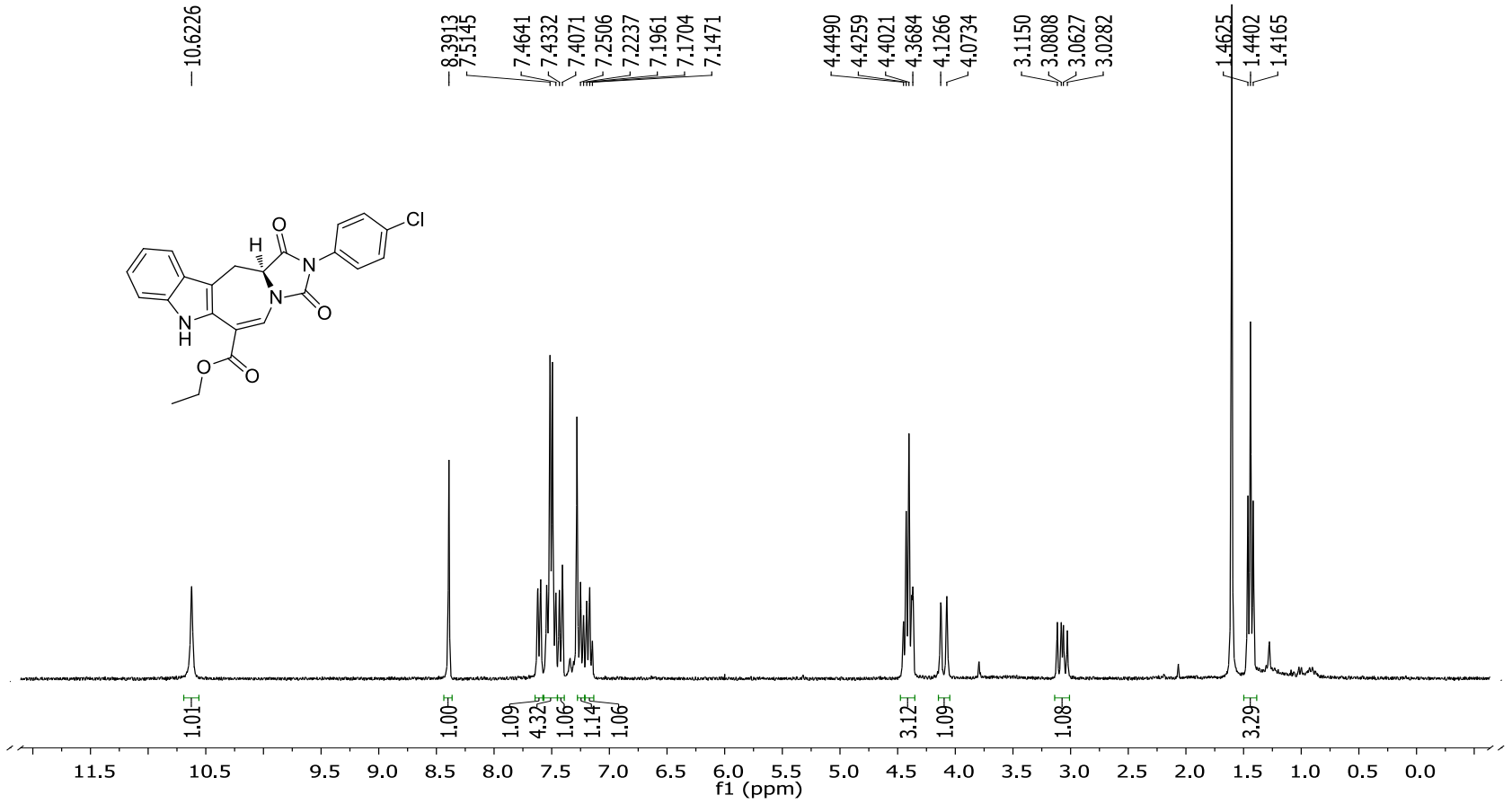
IR spectrum of compound **7a**



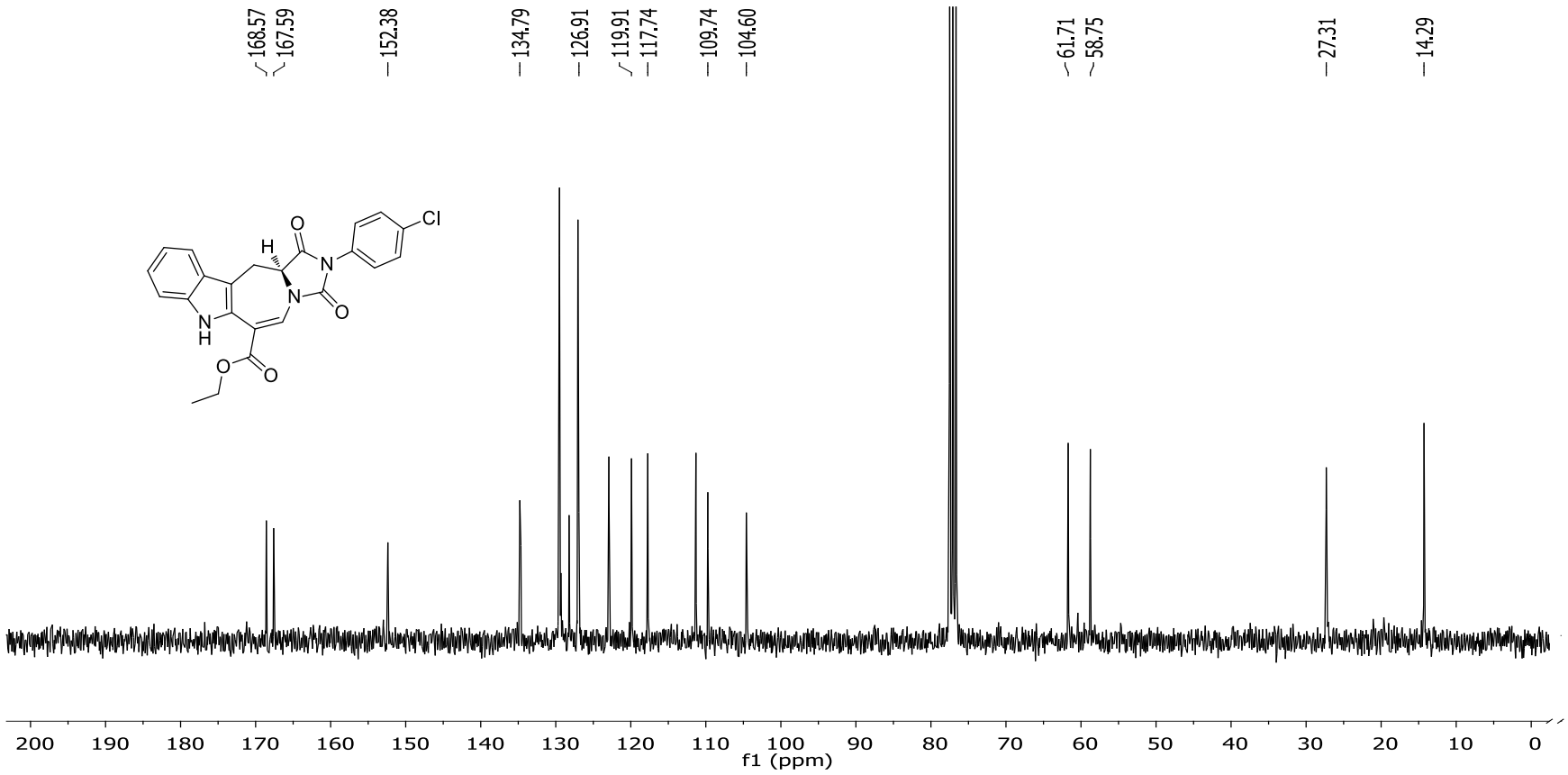
Result Table (Uncal - D:\INDRAJEET\HPLC DATA\PD-N4-014P - UV)

	Retention Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
1	2.096	7.285	0.718	0.2	1.1
2	3.916	1.480	0.251	0.0	0.4
3	21.188	4463.766	64.346	99.8	98.5
	Total	4472.530	65.315	100.0	100.0

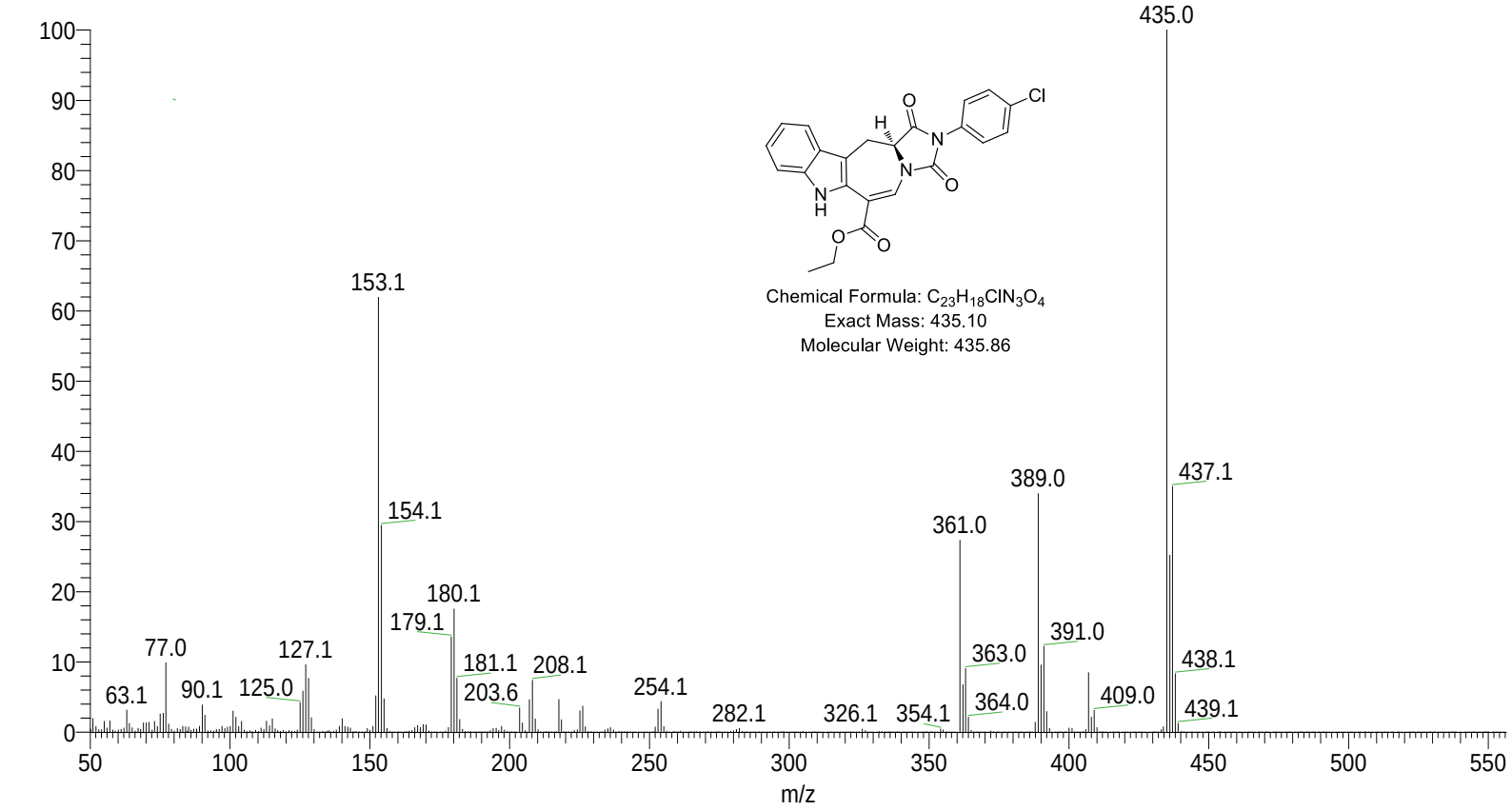
HPLC of compound **7a**



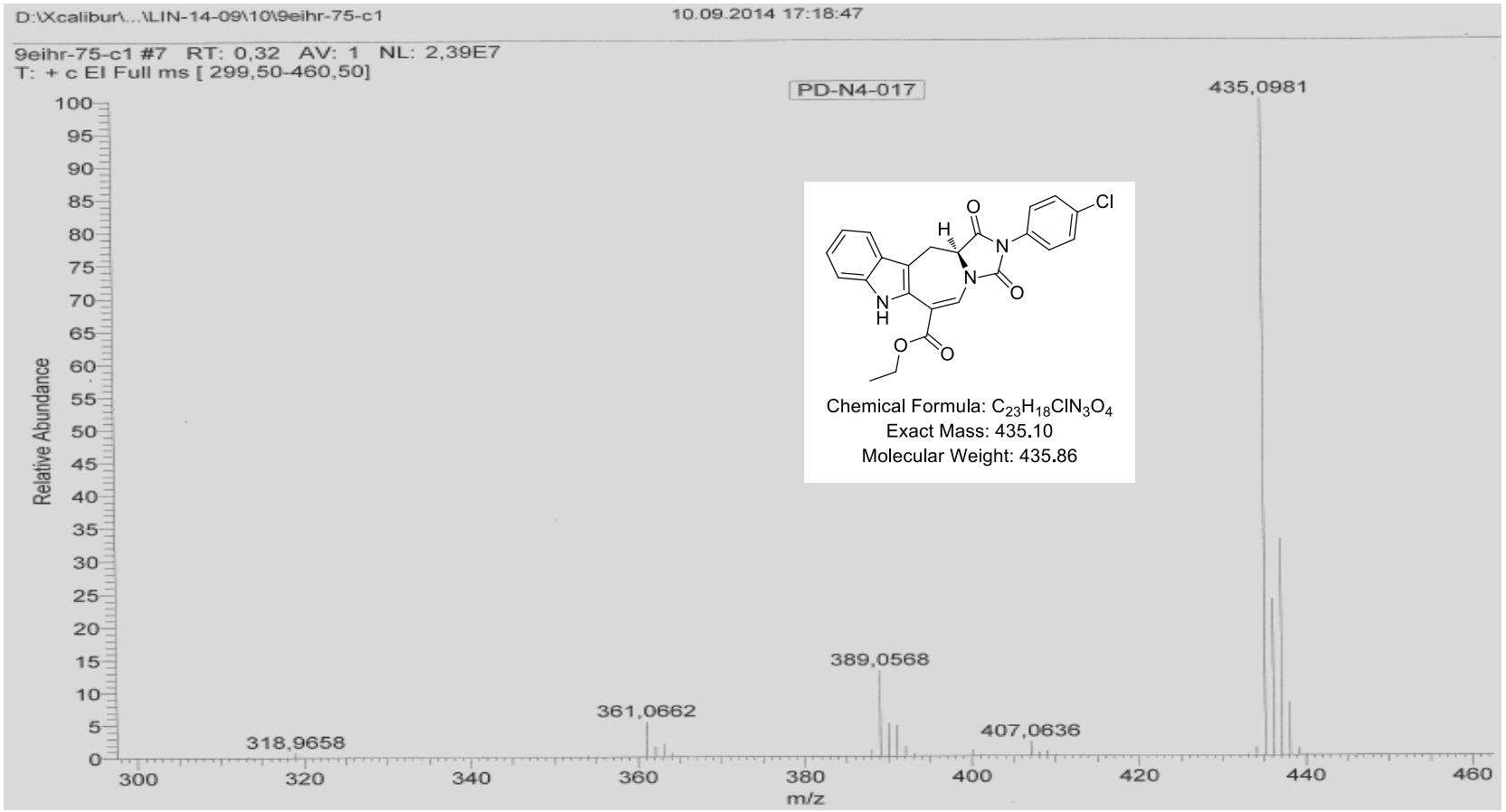
^{1}H NMR spectrum (300 MHz) of compound **7b** in $CDCl_3$



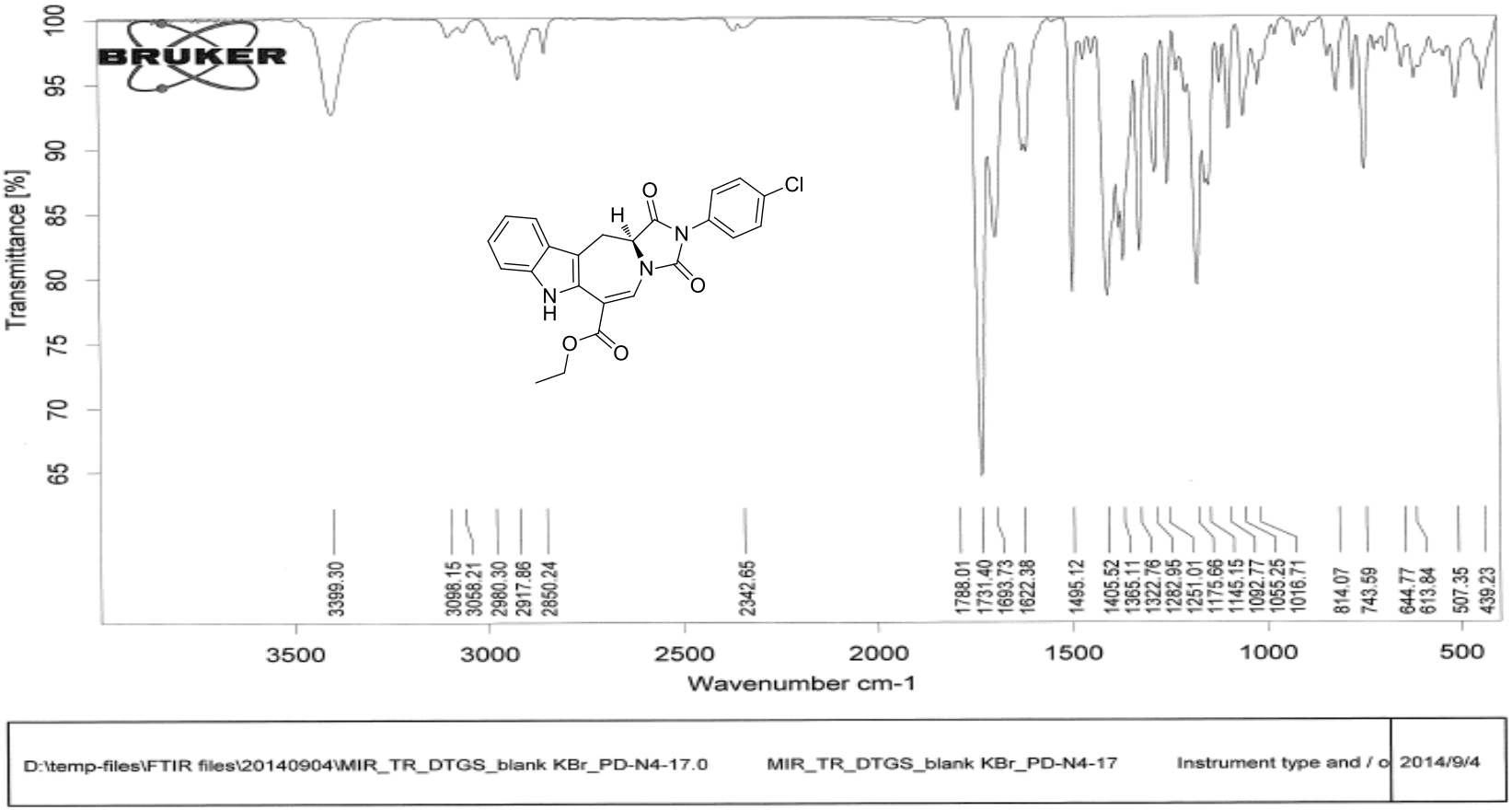
^{13}C NMR spectrum (75 MHz) of compound **7b** in $CDCl_3$



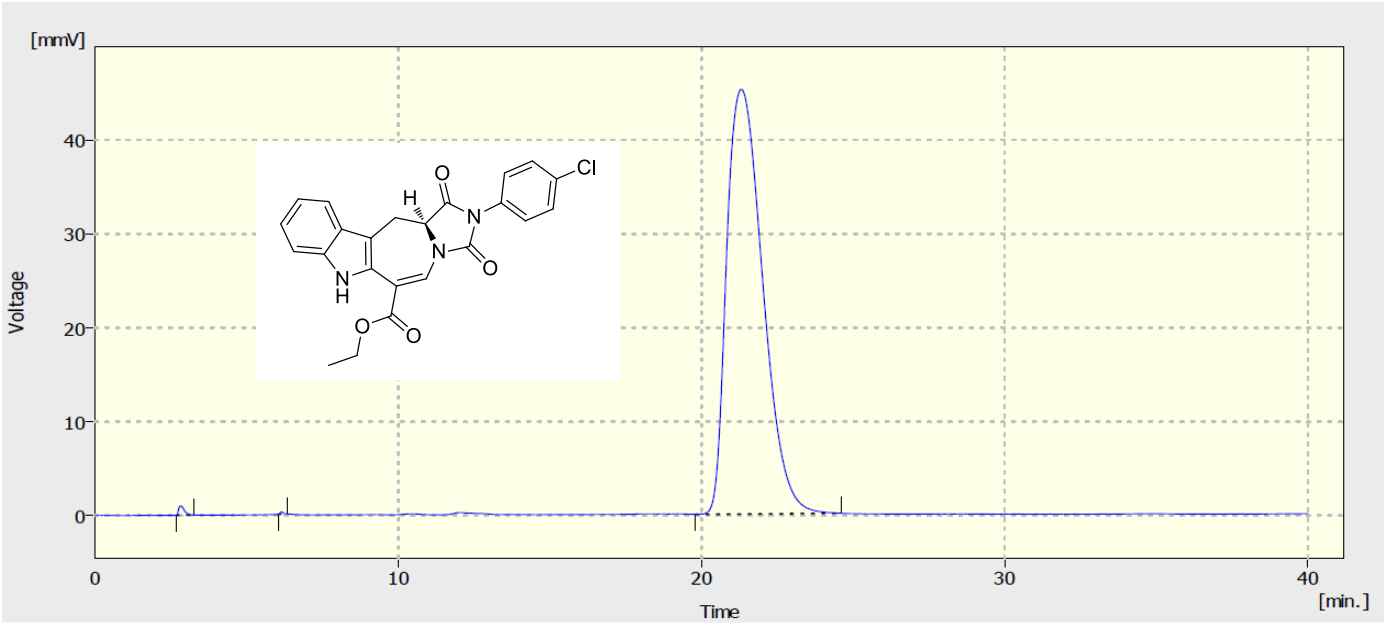
EI-LRMS of compound **7b**



EI-HRMS of compound **7b**



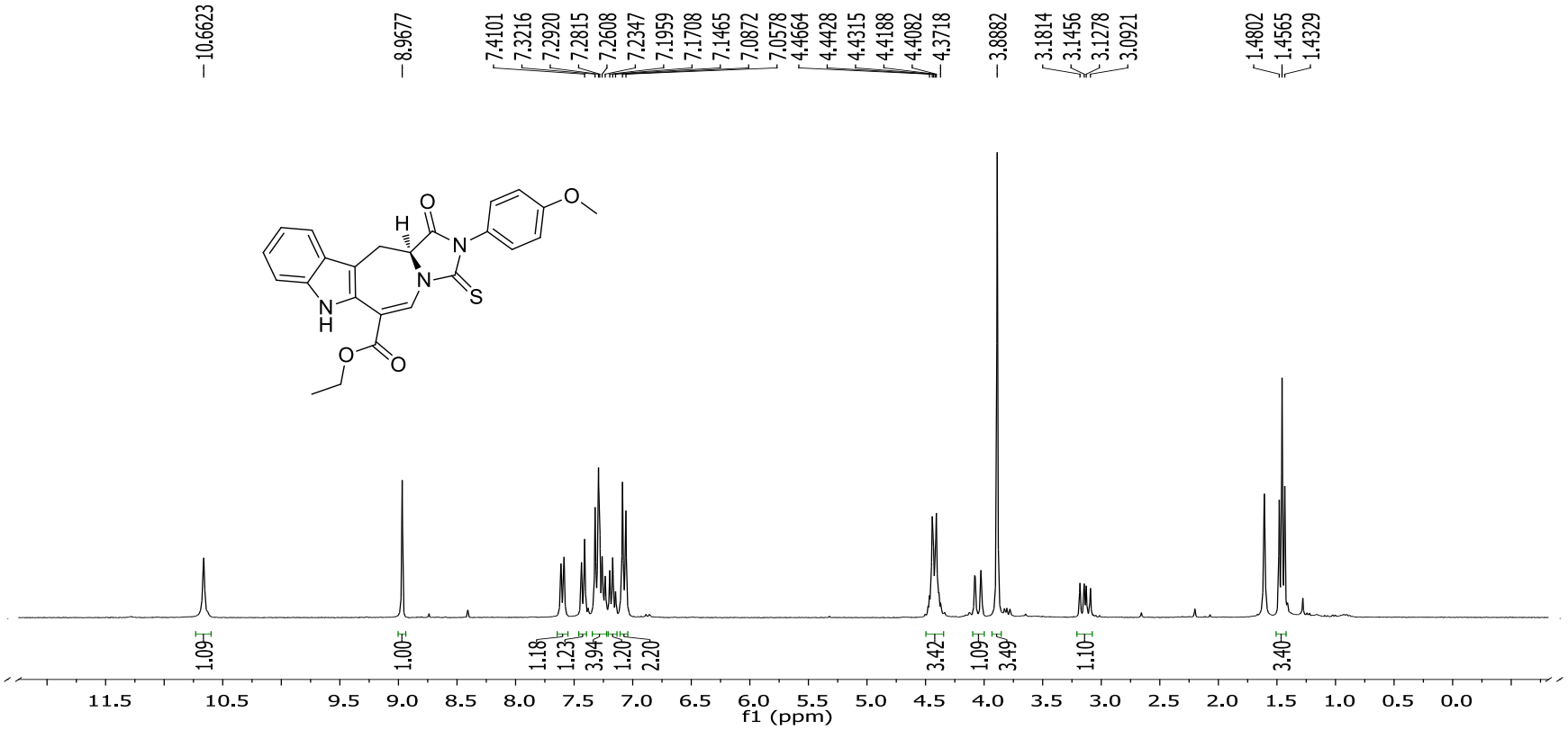
IR spectrum of compound **7b**



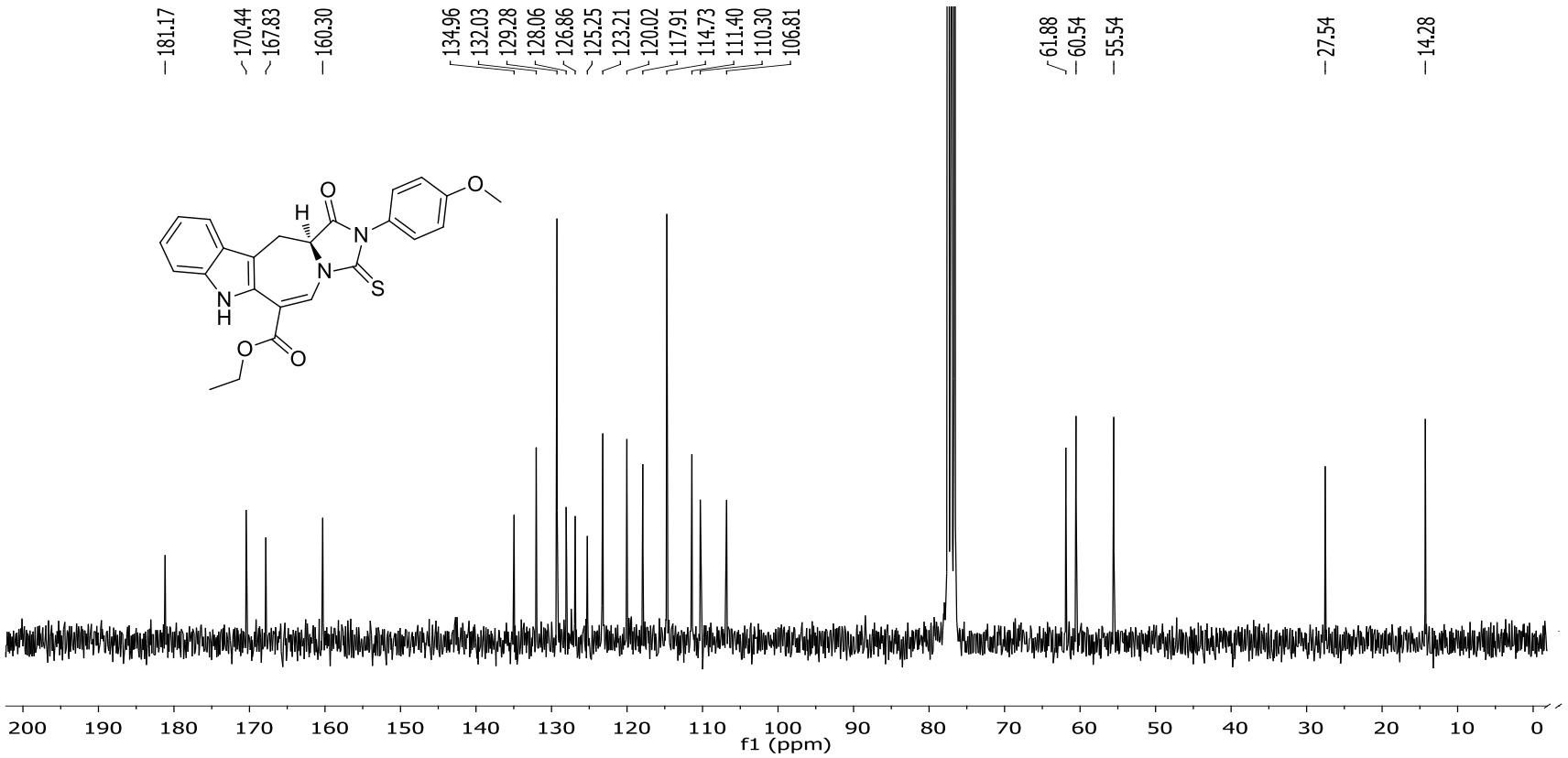
Result Table (Uncal - D:\PRASHANT HPLC PDF\PD-N4-P-CLDMF)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	2.816	13.099	1.000	0.3	2.2	0.20
2	6.148	1.670	0.250	0.0	0.5	0.10
3	21.316	3737.111	45.244	99.6	97.3	1.31
	Total	3751.880	46.494	100.0	100.0	

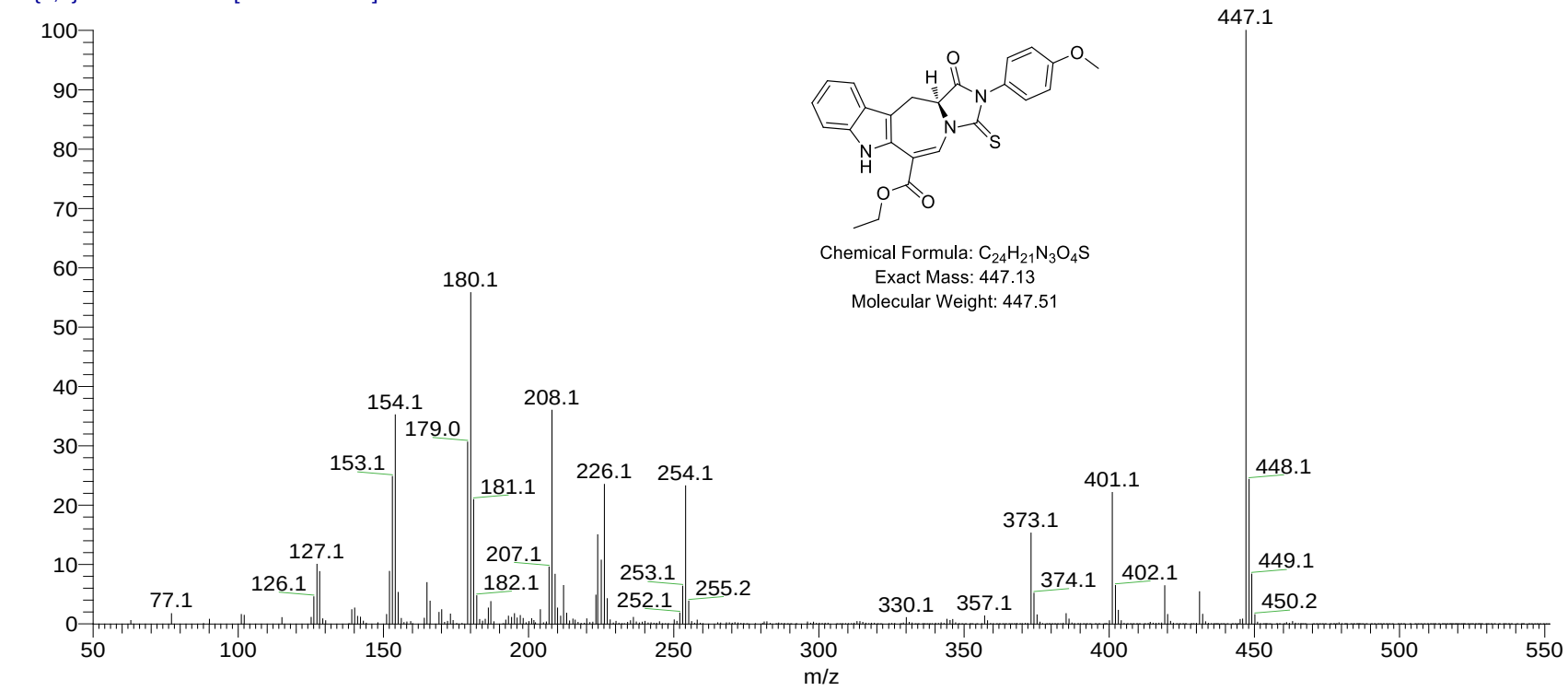
HPLC of compound **7b**



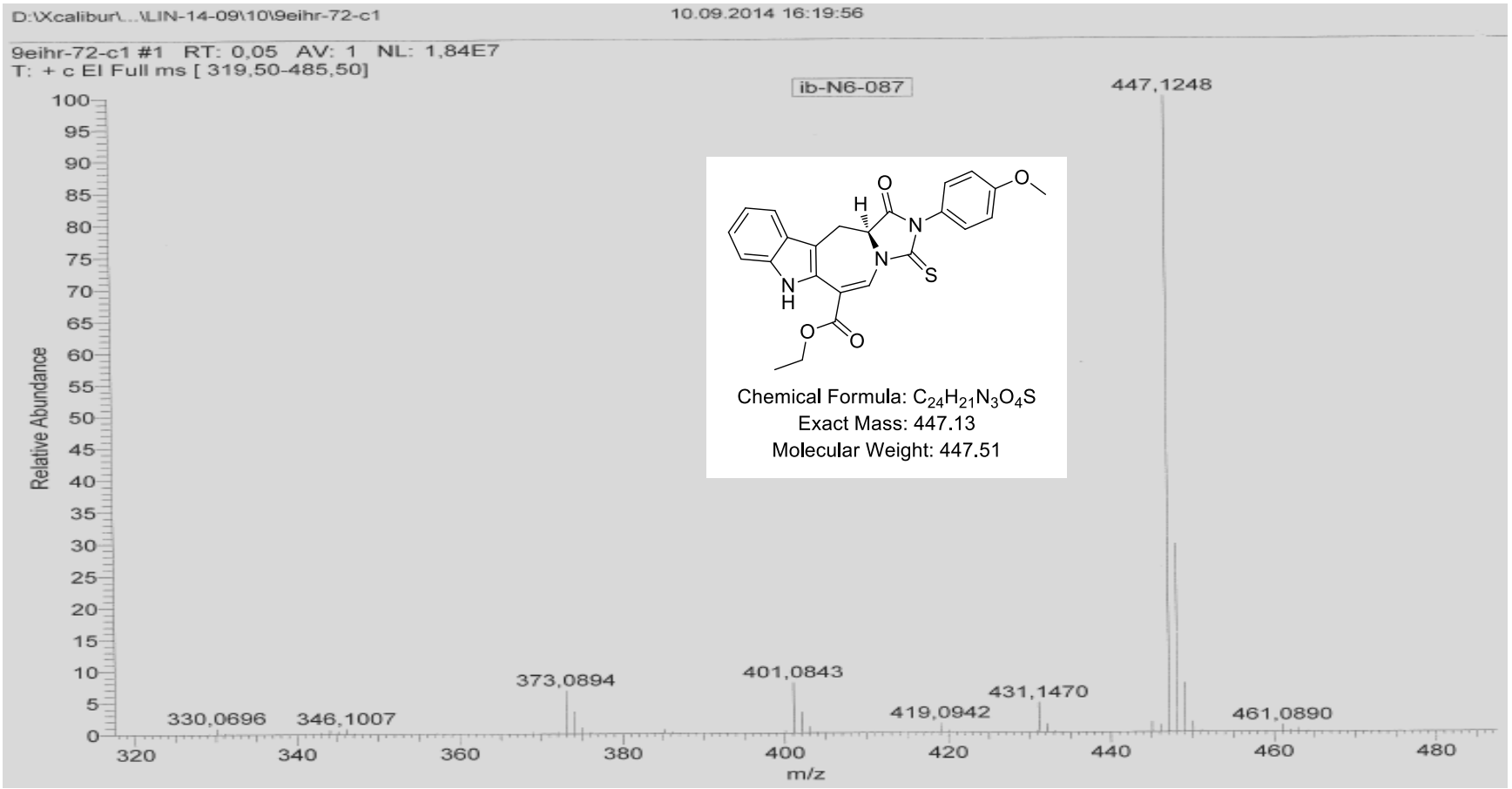
^{1}H NMR spectrum (300 MHz) of compound **7c** in $CDCl_3$



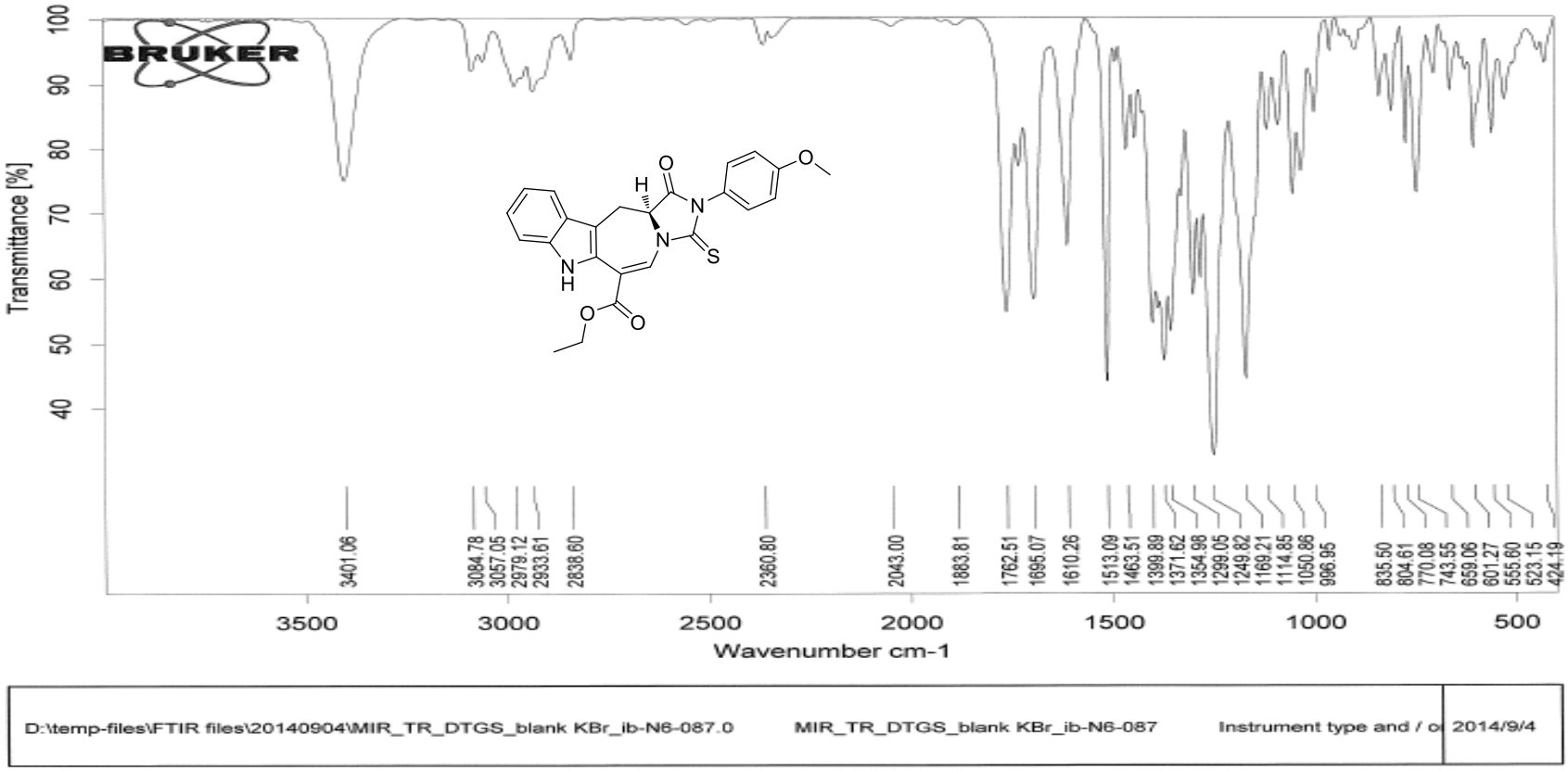
^{13}C NMR spectrum (75 MHz) of compound **7c** in $CDCl_3$



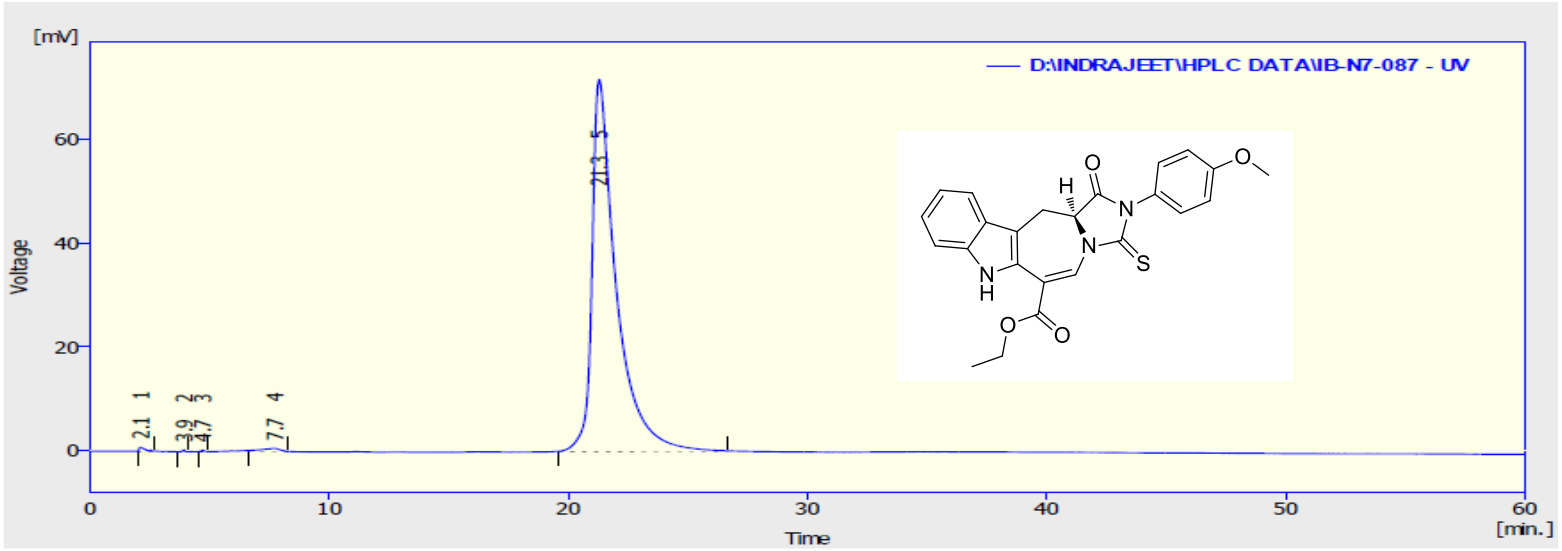
EI-LRMS of compound **7c**



EI-HRMS of compound **7c**



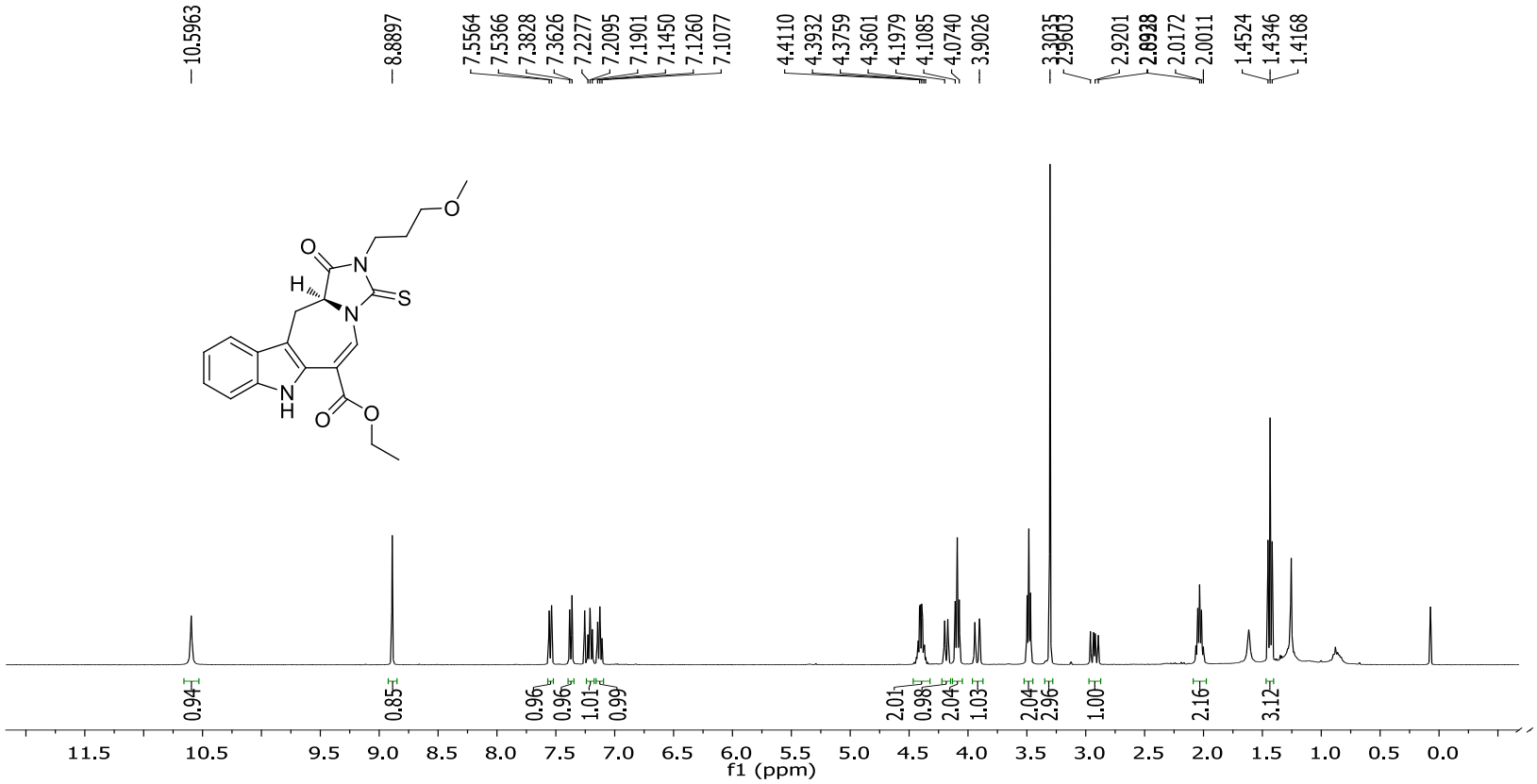
IR spectrum of compound **7c**



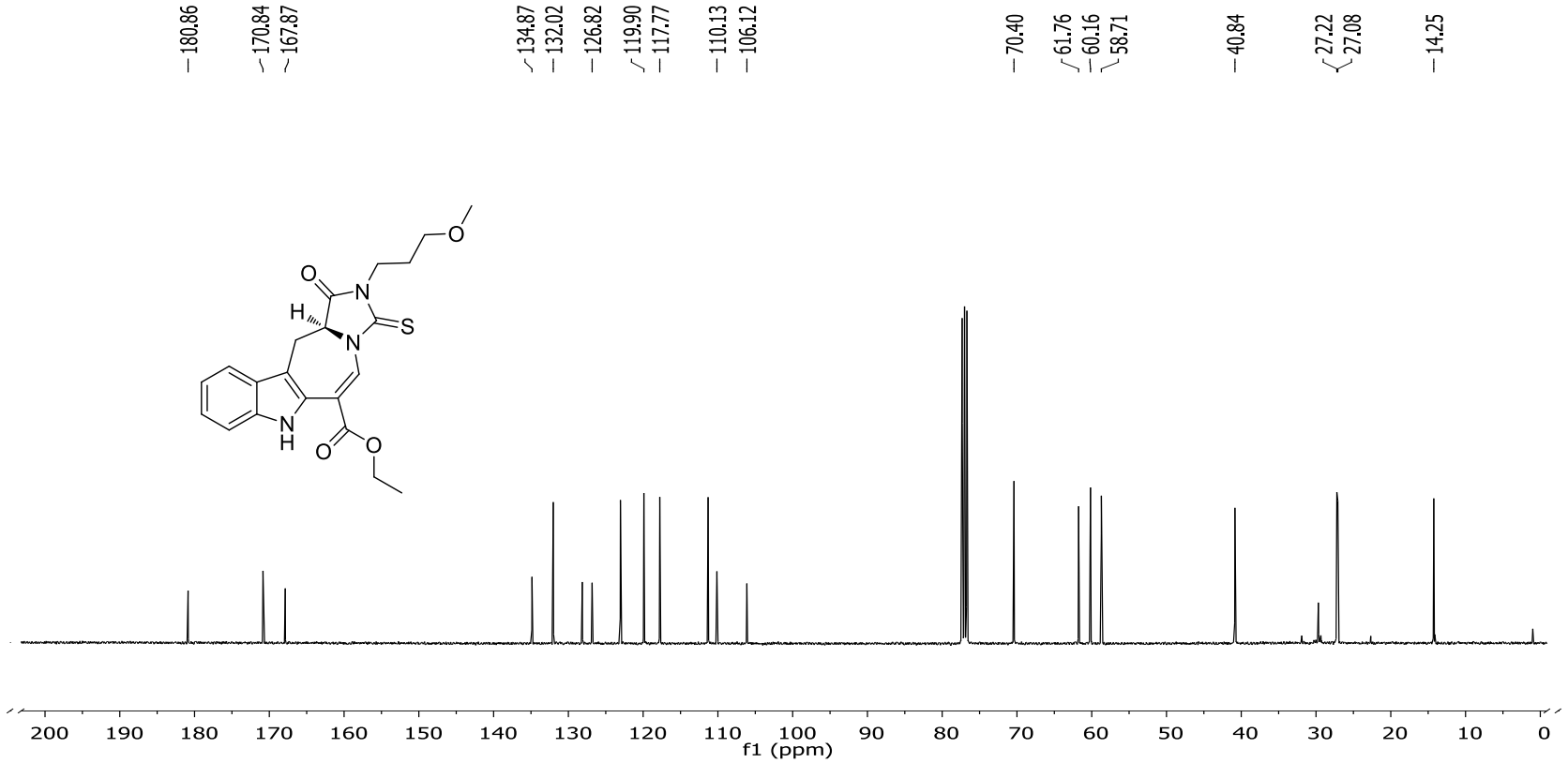
Result Table (Uncal - D:\INDRAJEET\HPLC DATA\IB-N7-087 - UV)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
1	2.124	12.244	0.755	0.2	1.0
2	3.920	2.299	0.365	0.0	0.5
3	4.704	2.333	0.296	0.0	0.4
4	7.728	22.959	0.515	0.5	0.7
5	21.288	5018.974	71.674	99.2	97.4
	Total	5058.809	73.604	100.0	100.0

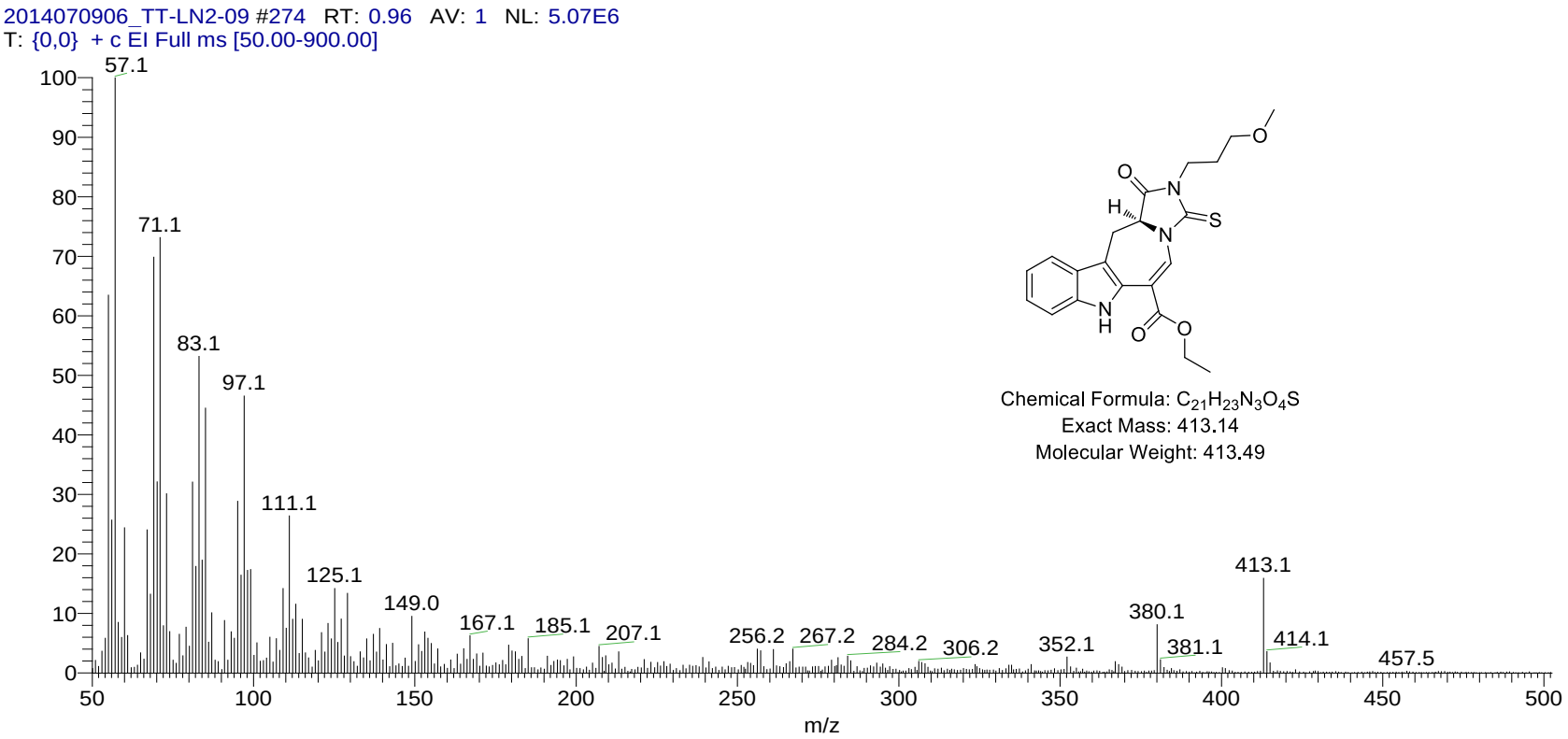
HPLC of compound **7c**



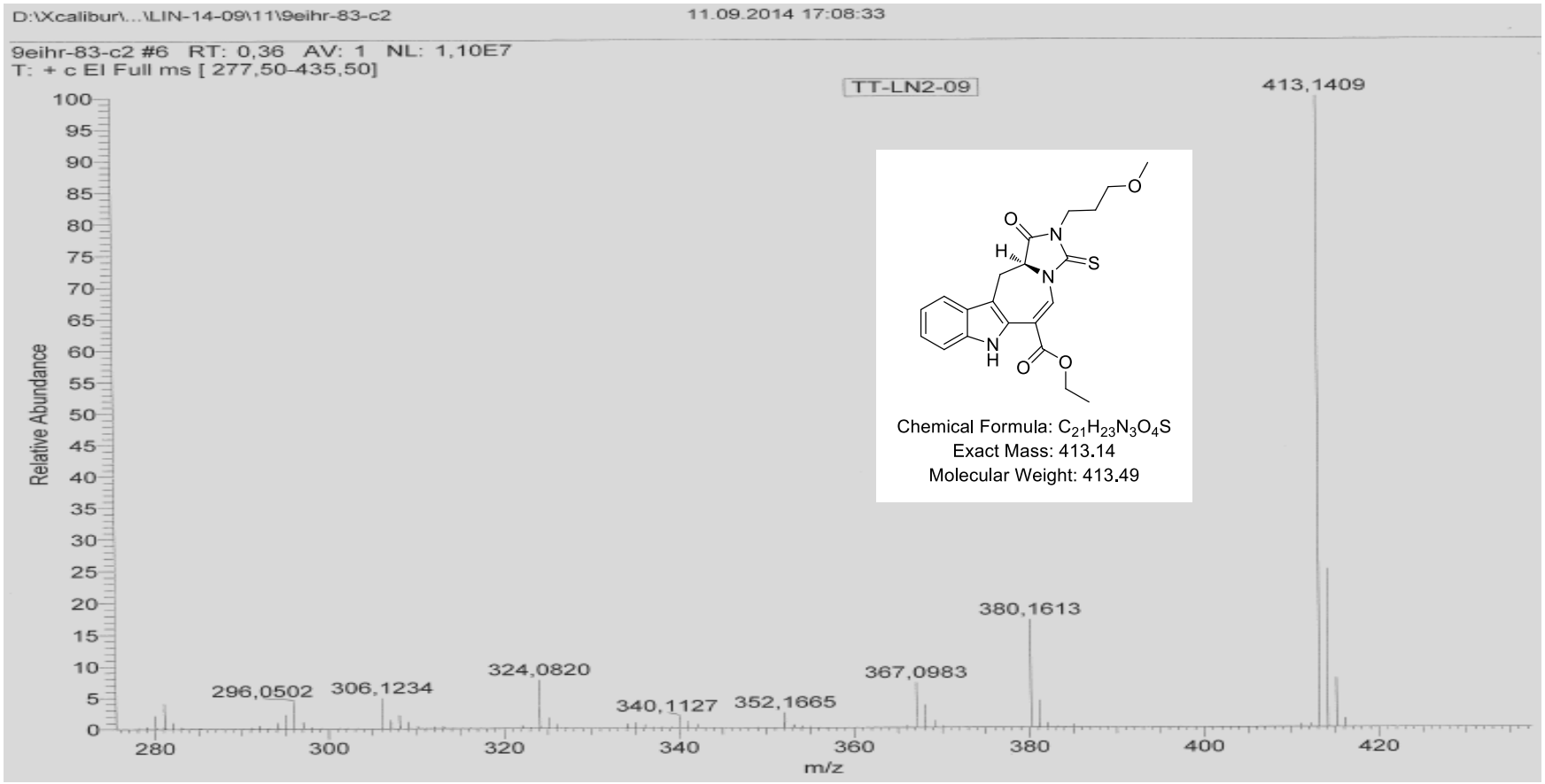
^{1}H NMR spectrum (400 MHz) of compound **7d** in $CDCl_3$



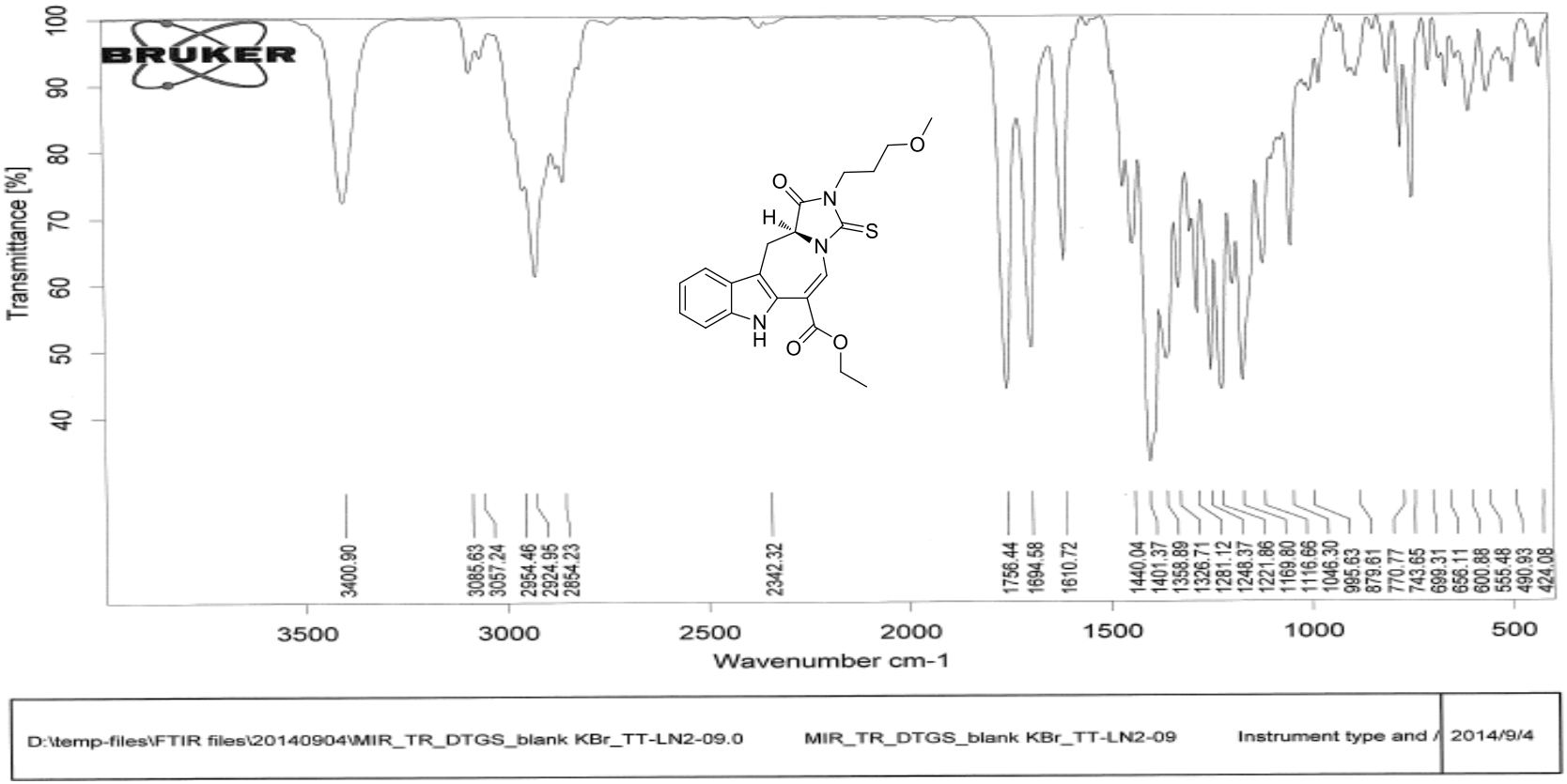
^{13}C NMR spectrum (101 MHz) of compound **7d** in $CDCl_3$



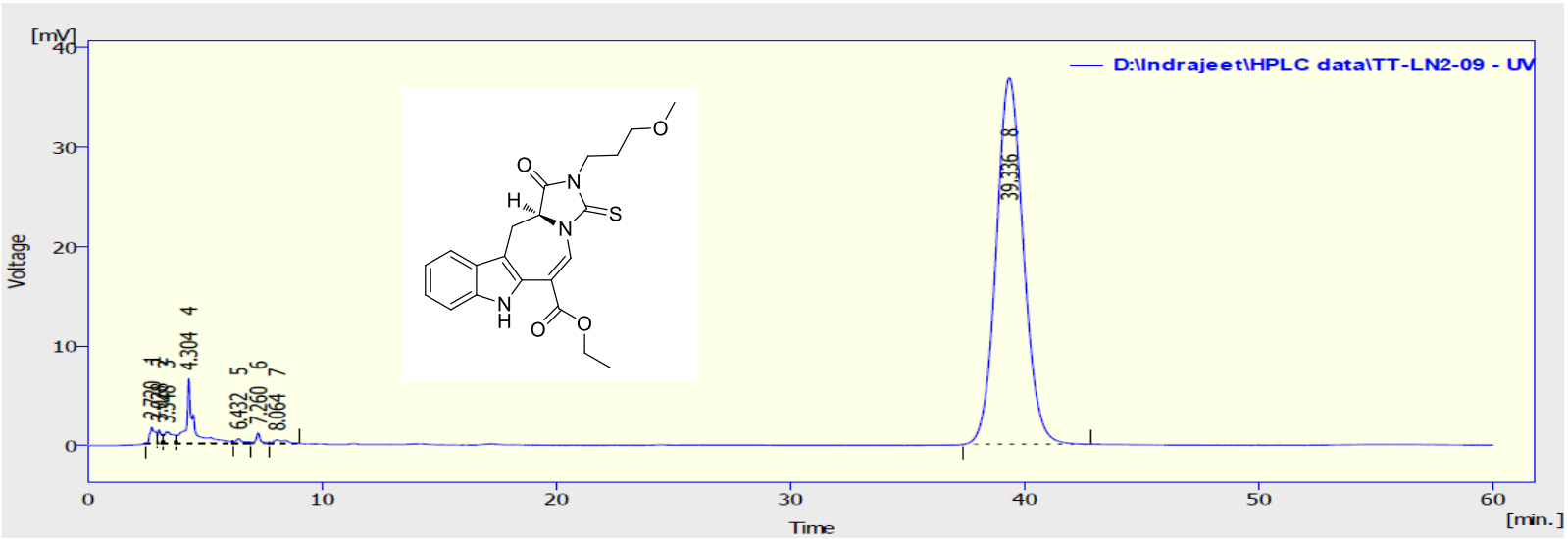
EI-LRMS of compound **7d**



EI-HRMS of compound **7d**



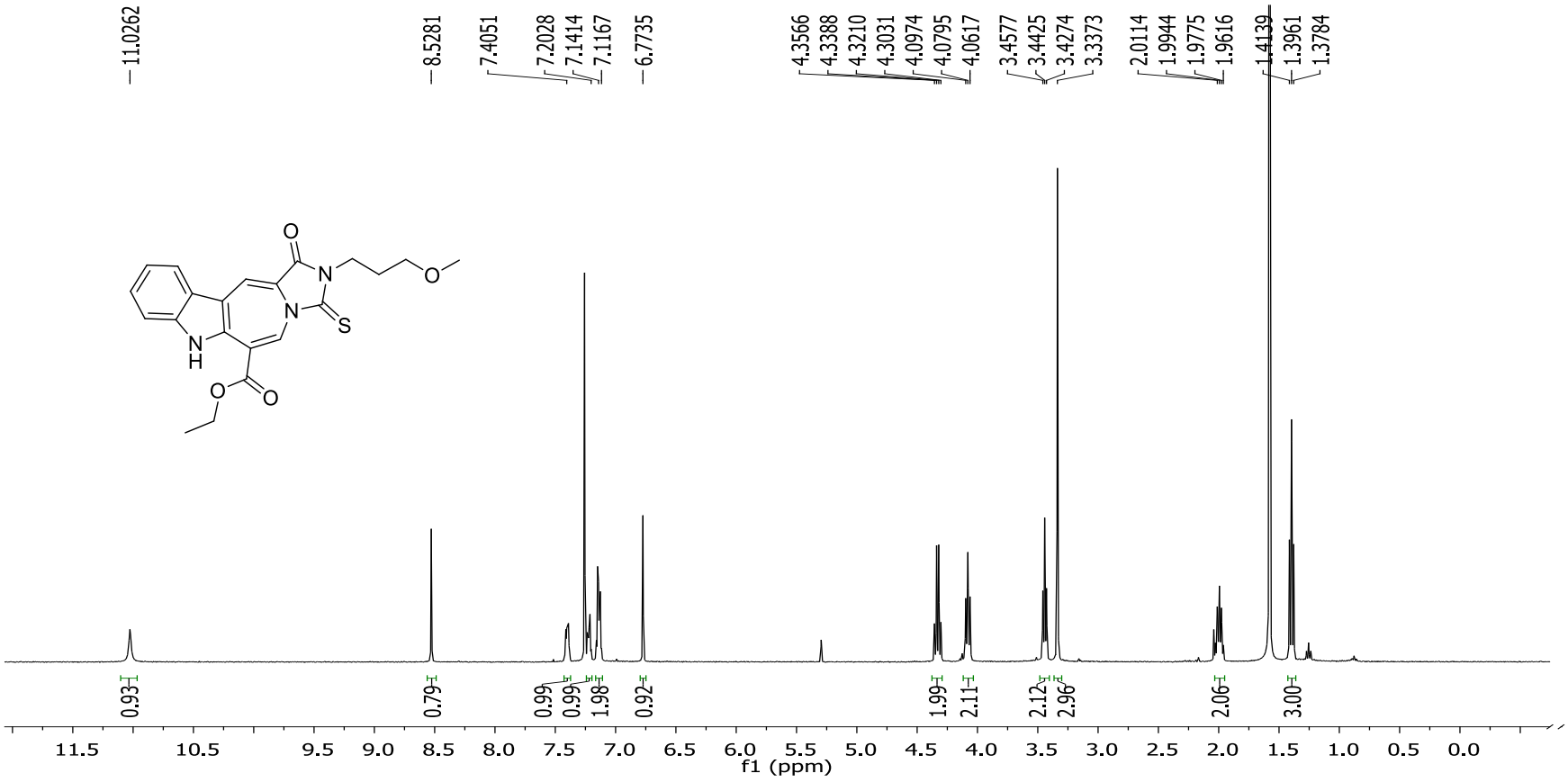
IR spectrum of compound **7d**



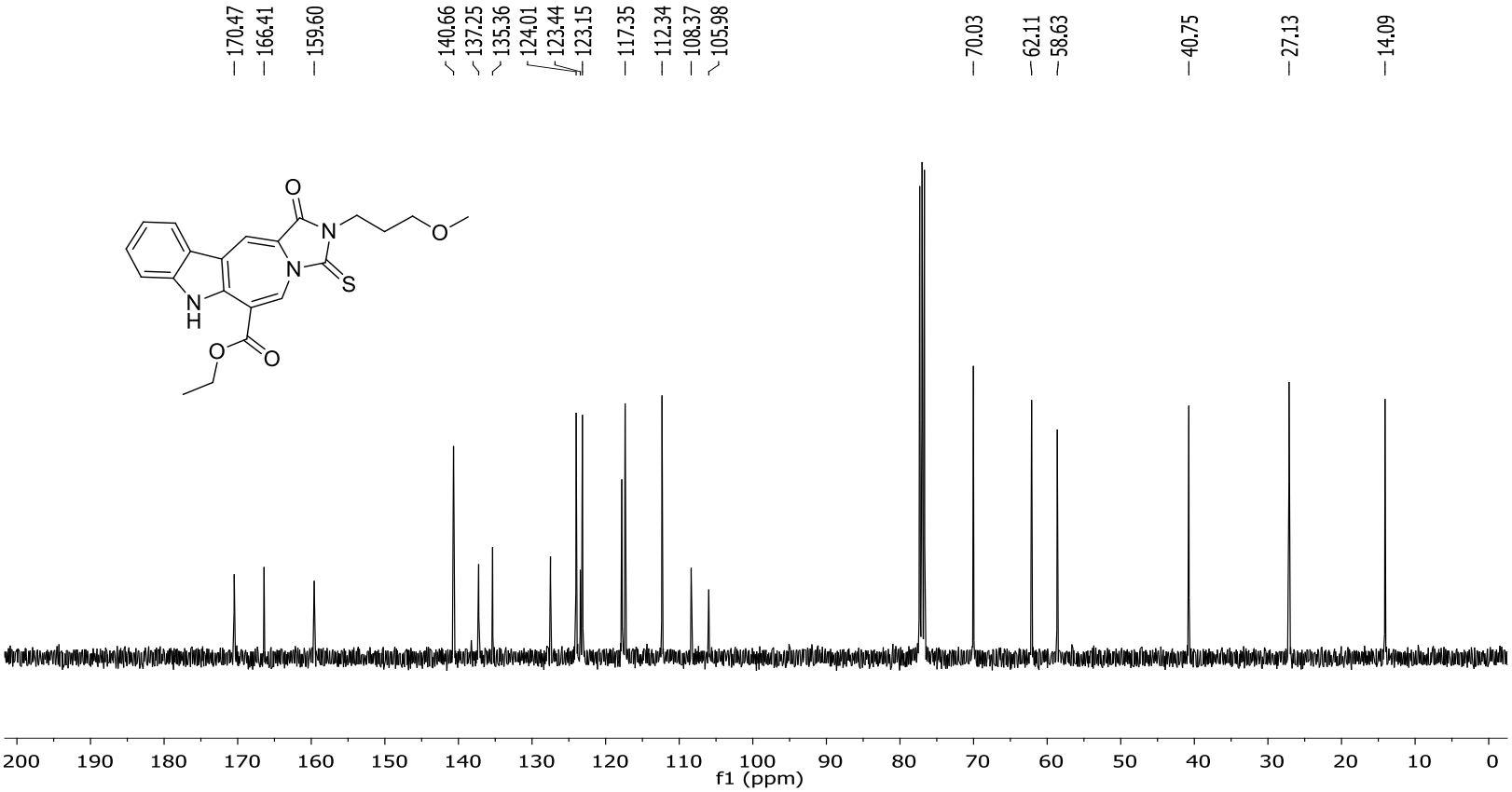
Result Table (Uncal - D:\Indrajeet\HPLC data\TT-LN2-09 - UV)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	2.720	26.642	1.619	0.8	3.3	0.34
2	3.028	15.490	1.343	0.5	2.7	0.24
3	3.348	31.577	1.184	1.0	2.4	0.55
4	4.304	145.225	6.492	4.5	13.2	0.12
5	6.432	10.361	0.473	0.3	1.0	0.23
6	7.260	13.371	1.043	0.4	2.1	0.16
7	8.064	12.908	0.362	0.4	0.7	0.64
8	39.336	2968.802	36.836	92.1	74.6	1.23
	Total	3224.378	49.352	100.0	100.0	

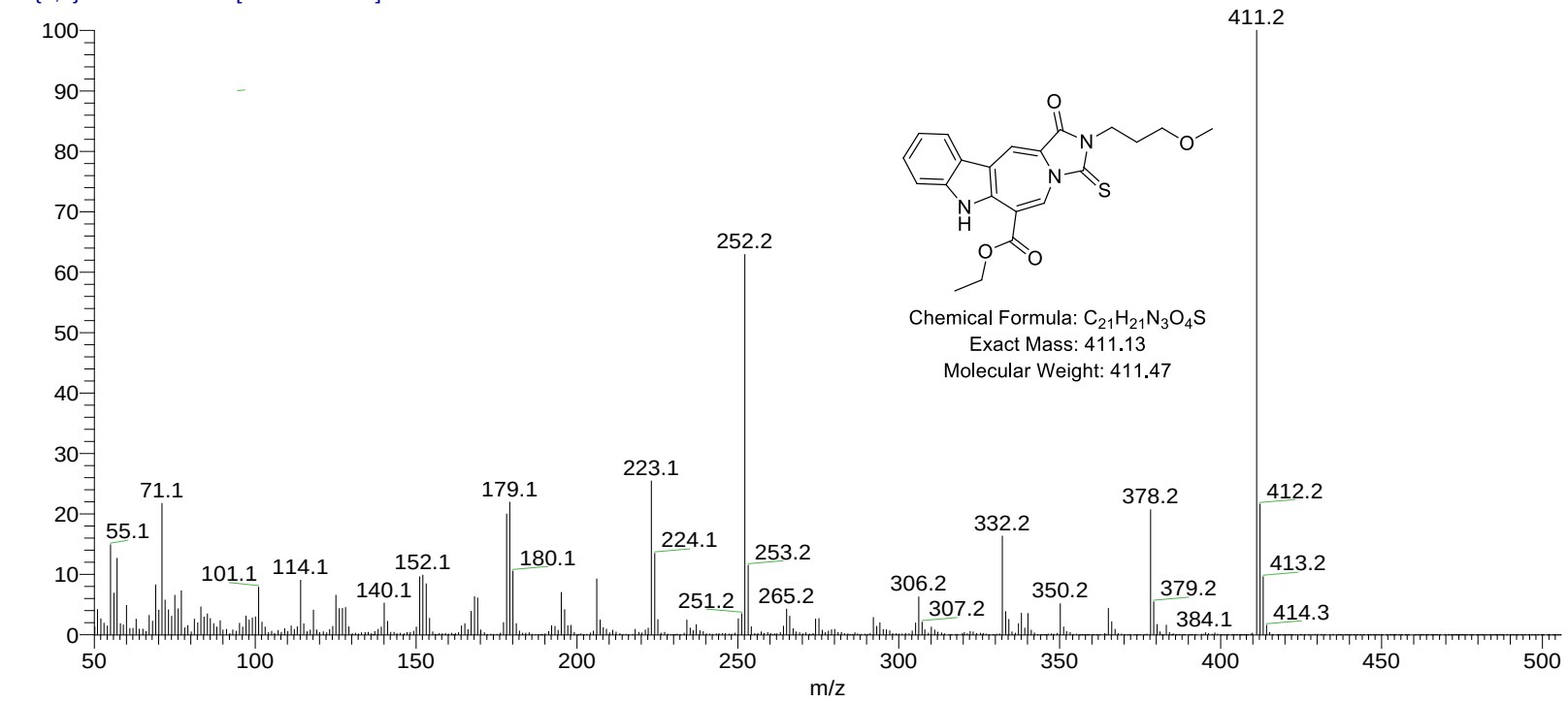
HPLC of compound **7d**



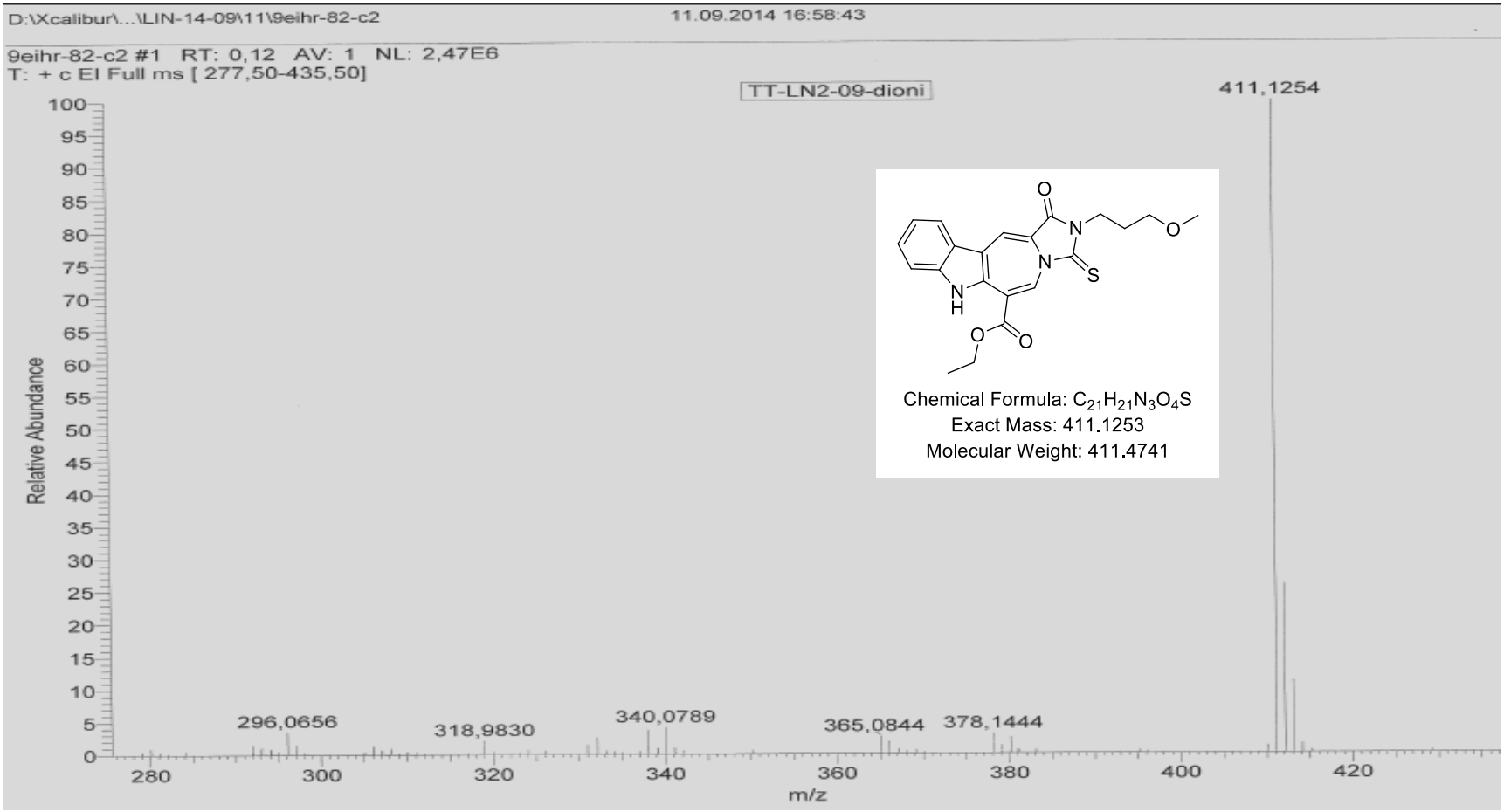
^{1}H NMR spectrum (400 MHz) of compound **7d'** in $CDCl_3$



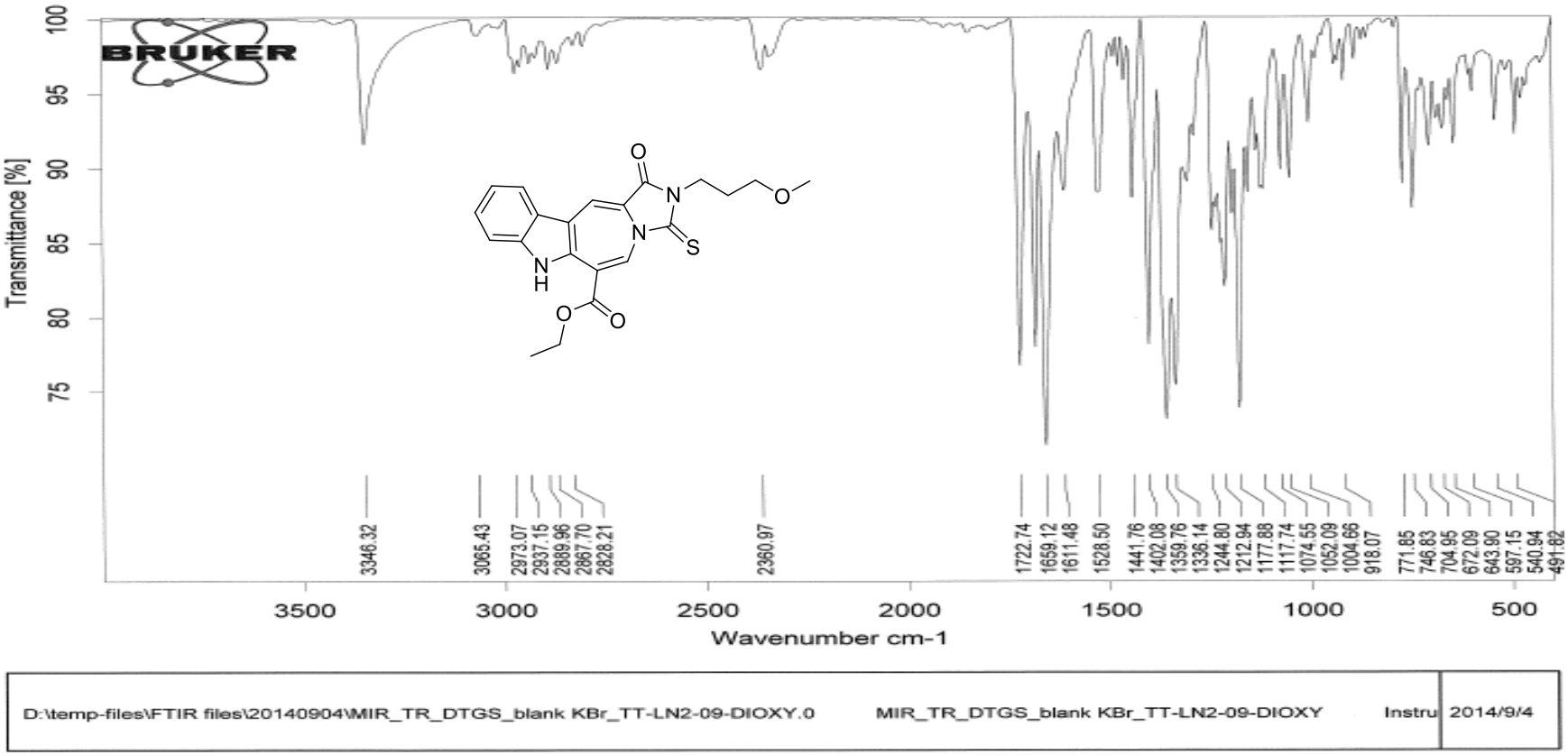
^{13}C NMR spectrum (101 MHz) of compound **7d'** in $CDCl_3$



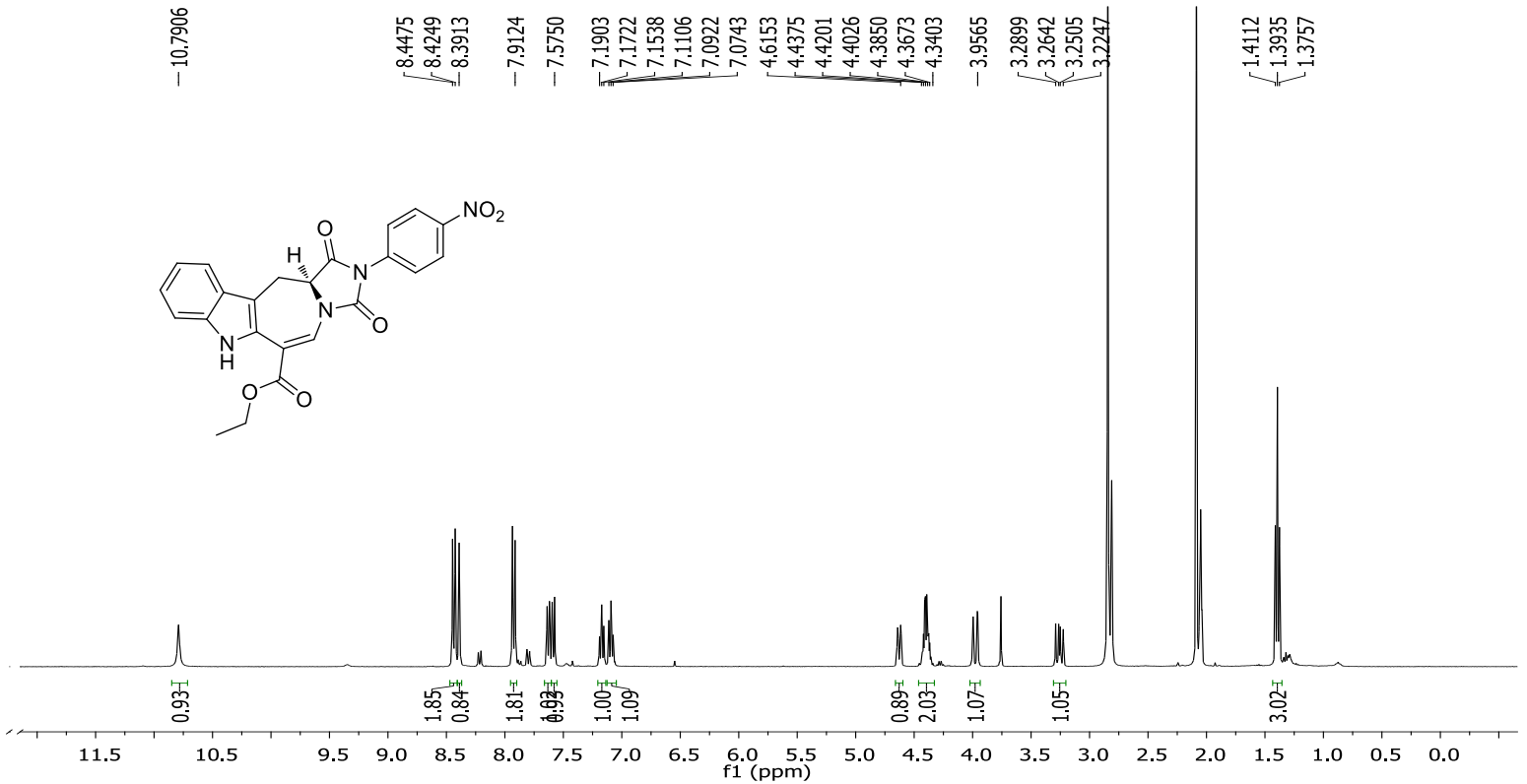
EI-LRMS of compound **7d'**



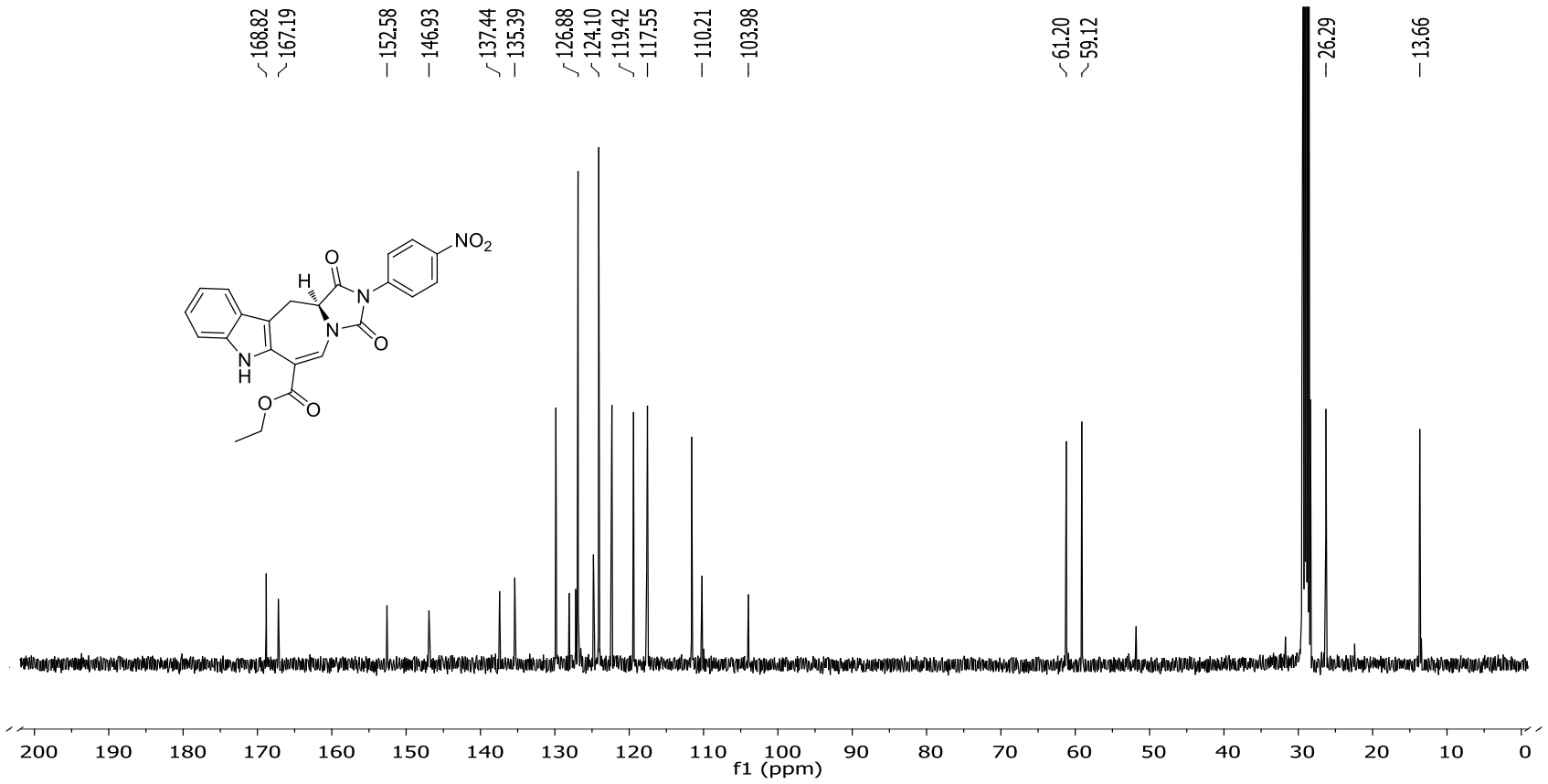
EI-HRMS of compound **7d'**



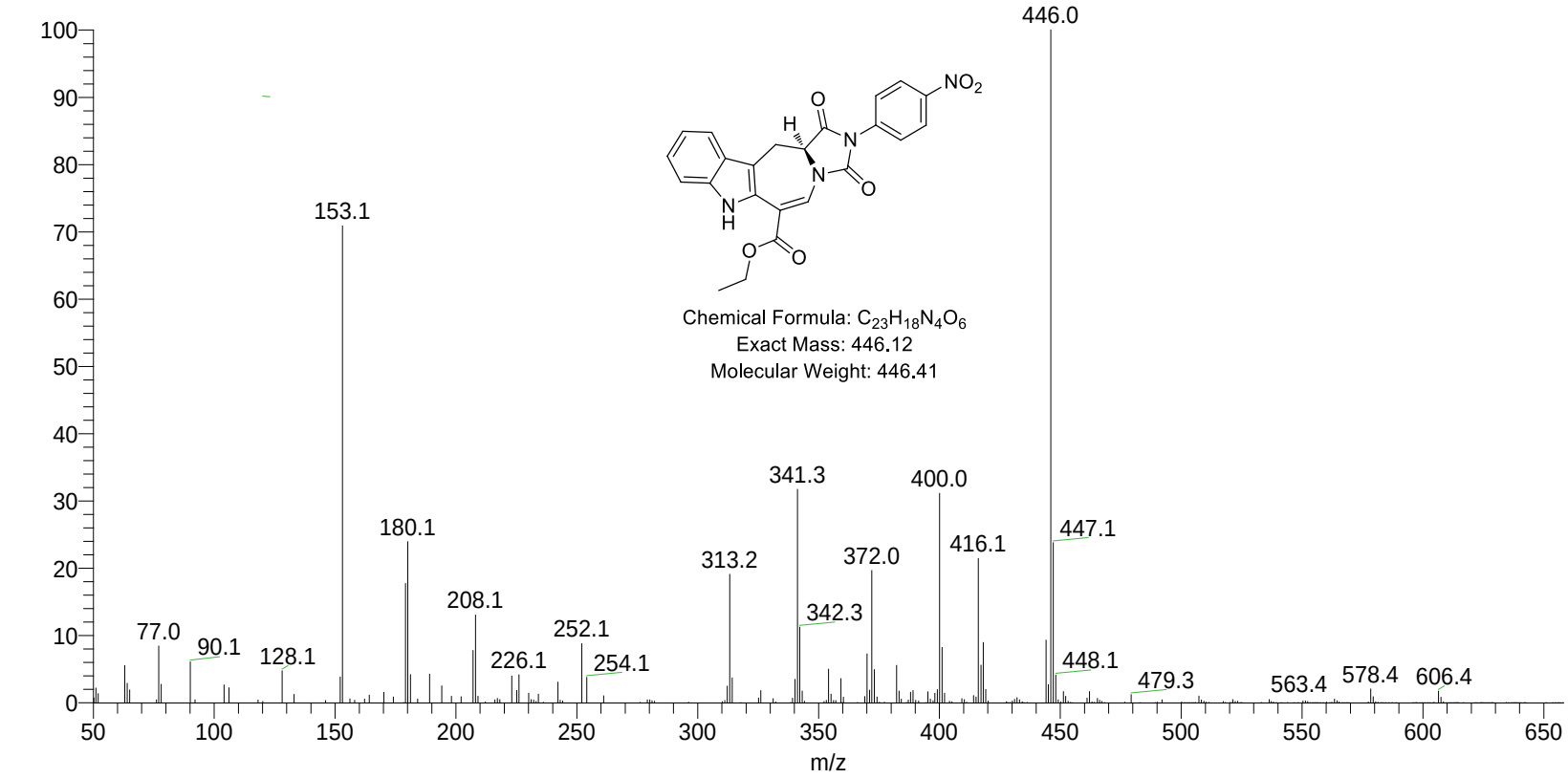
IR spectrum of compound **7d'**



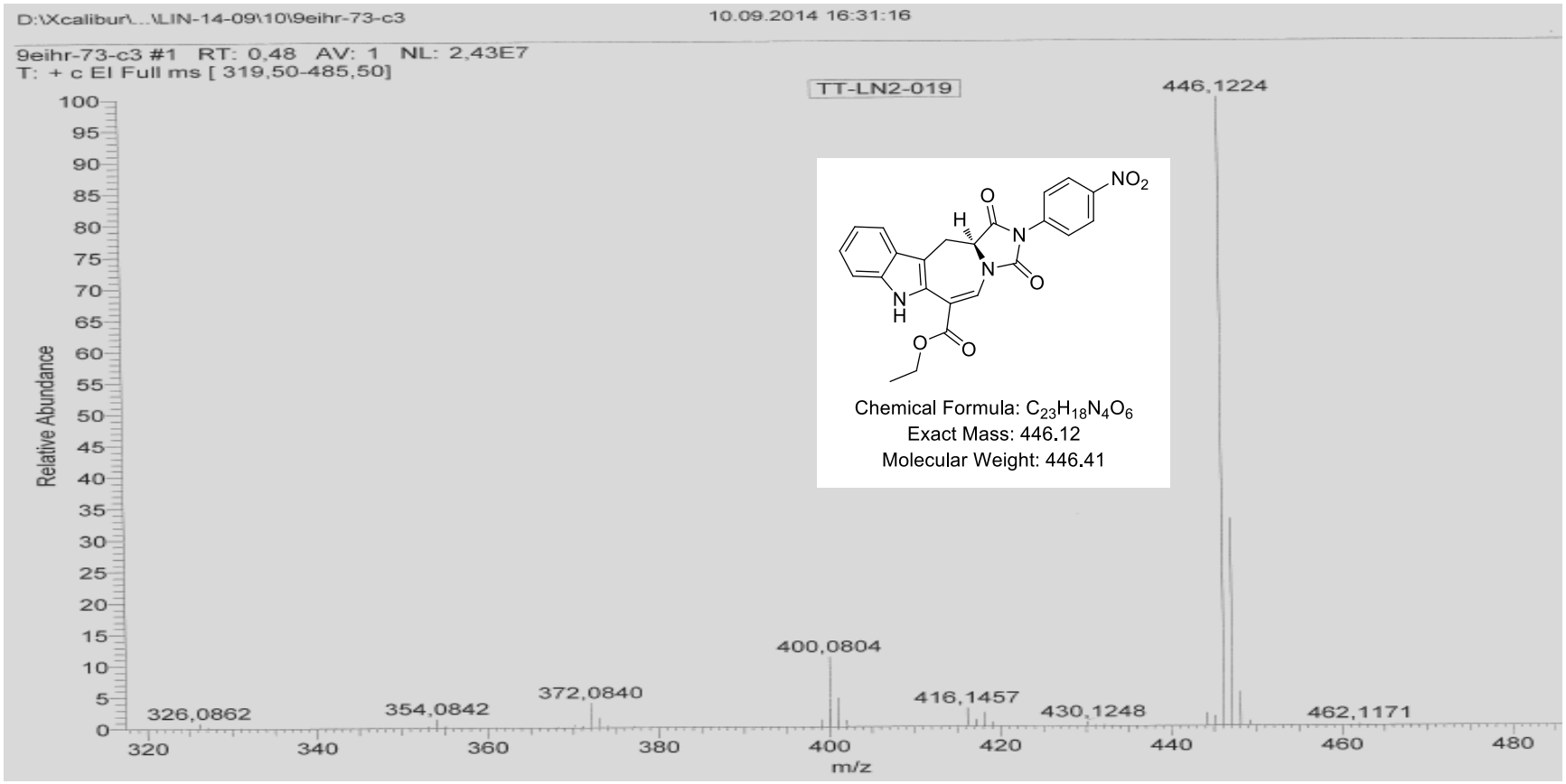
^{1}H NMR spectrum (400 MHz) of compound **7e** in acetone-d_6



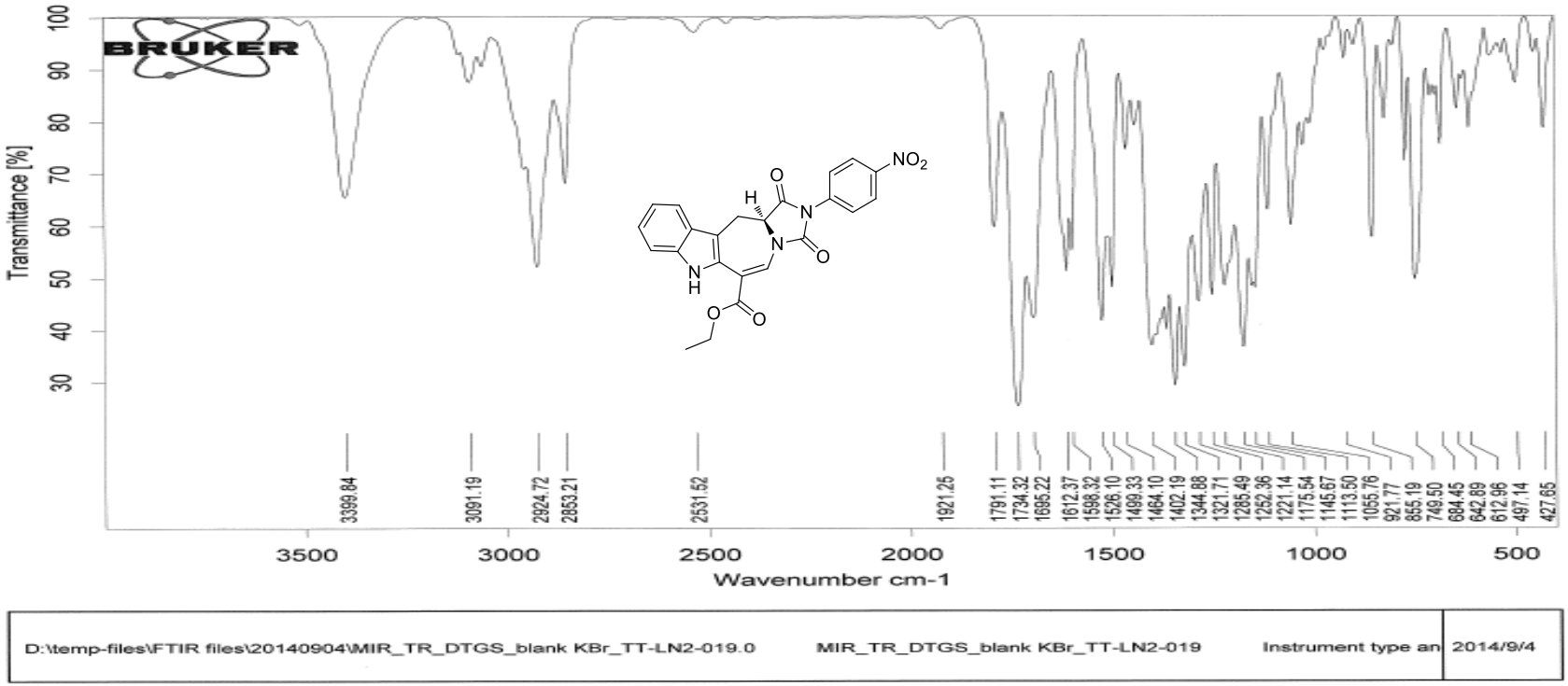
^{13}C NMR spectrum (101 MHz) of compound **7e** in acetone-d_6



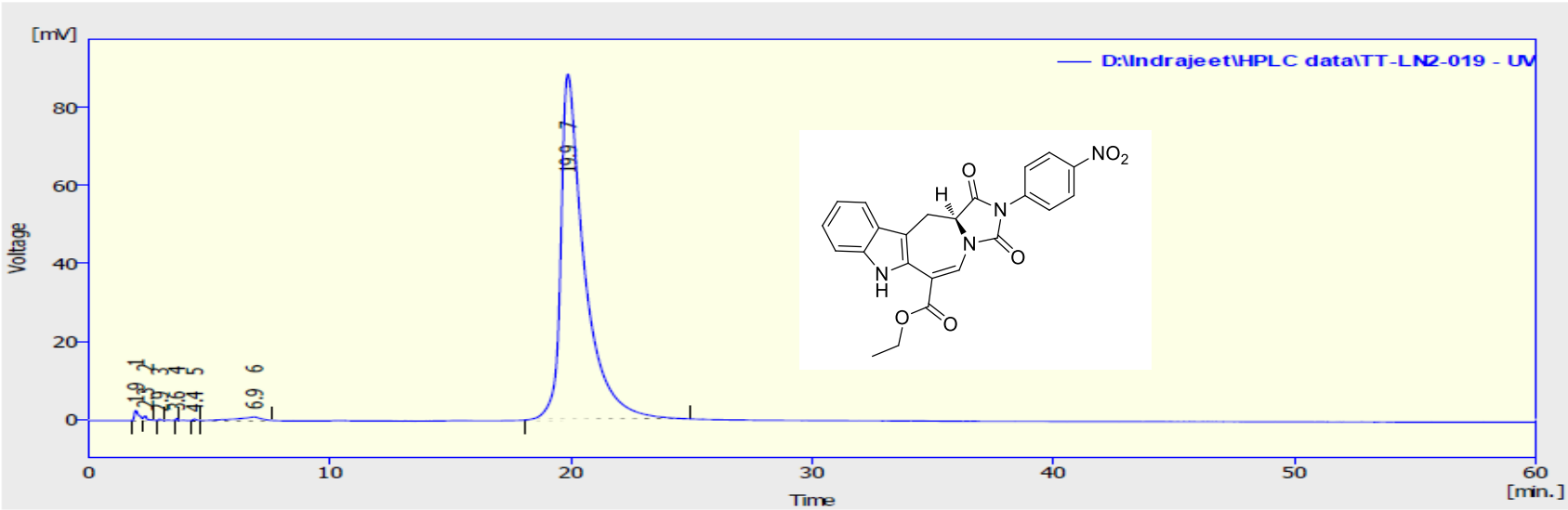
EI-LRMS of compound **7e**



EI-HRMS of compound **7e**



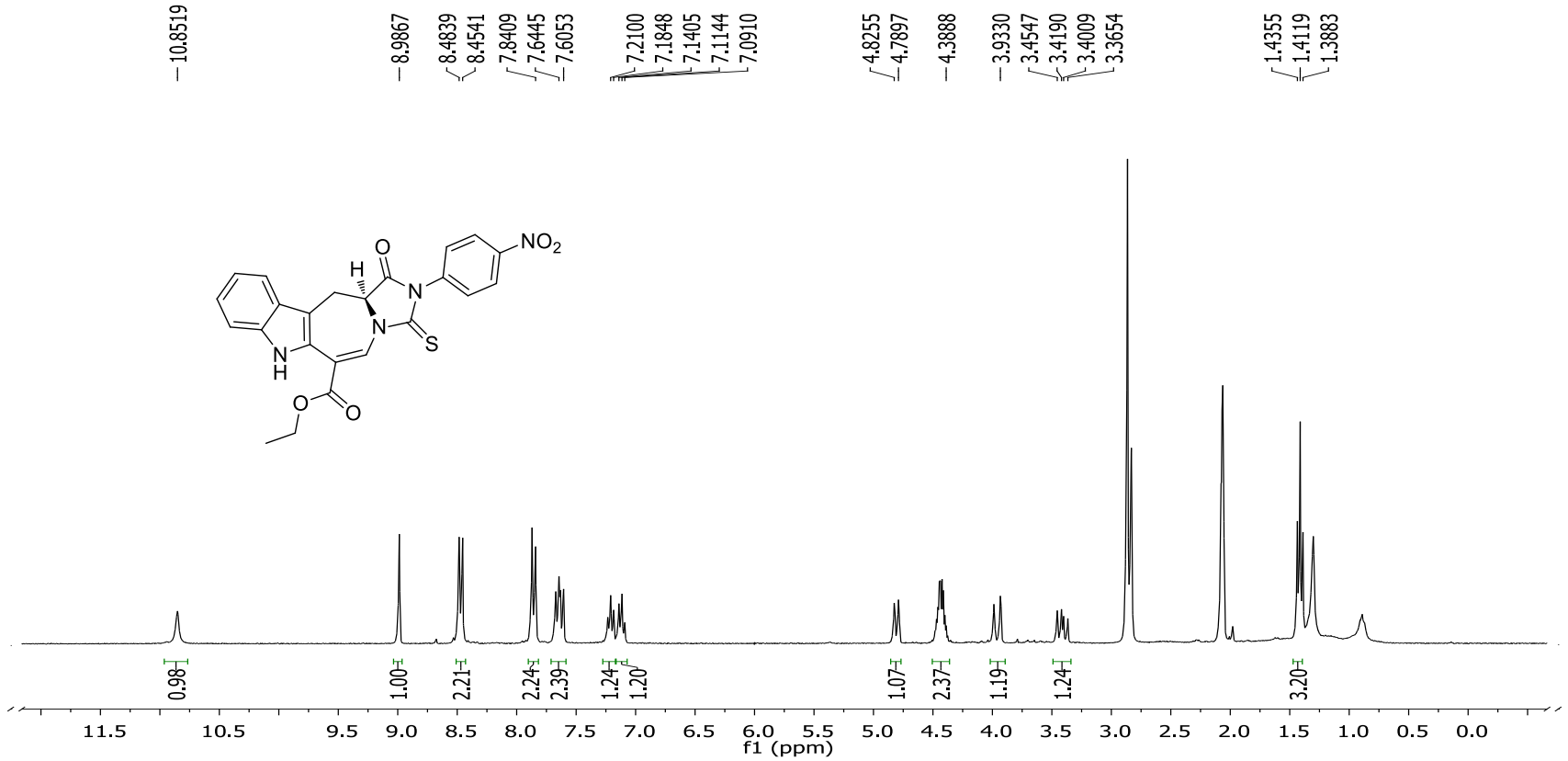
IR spectrum of compound **7e**



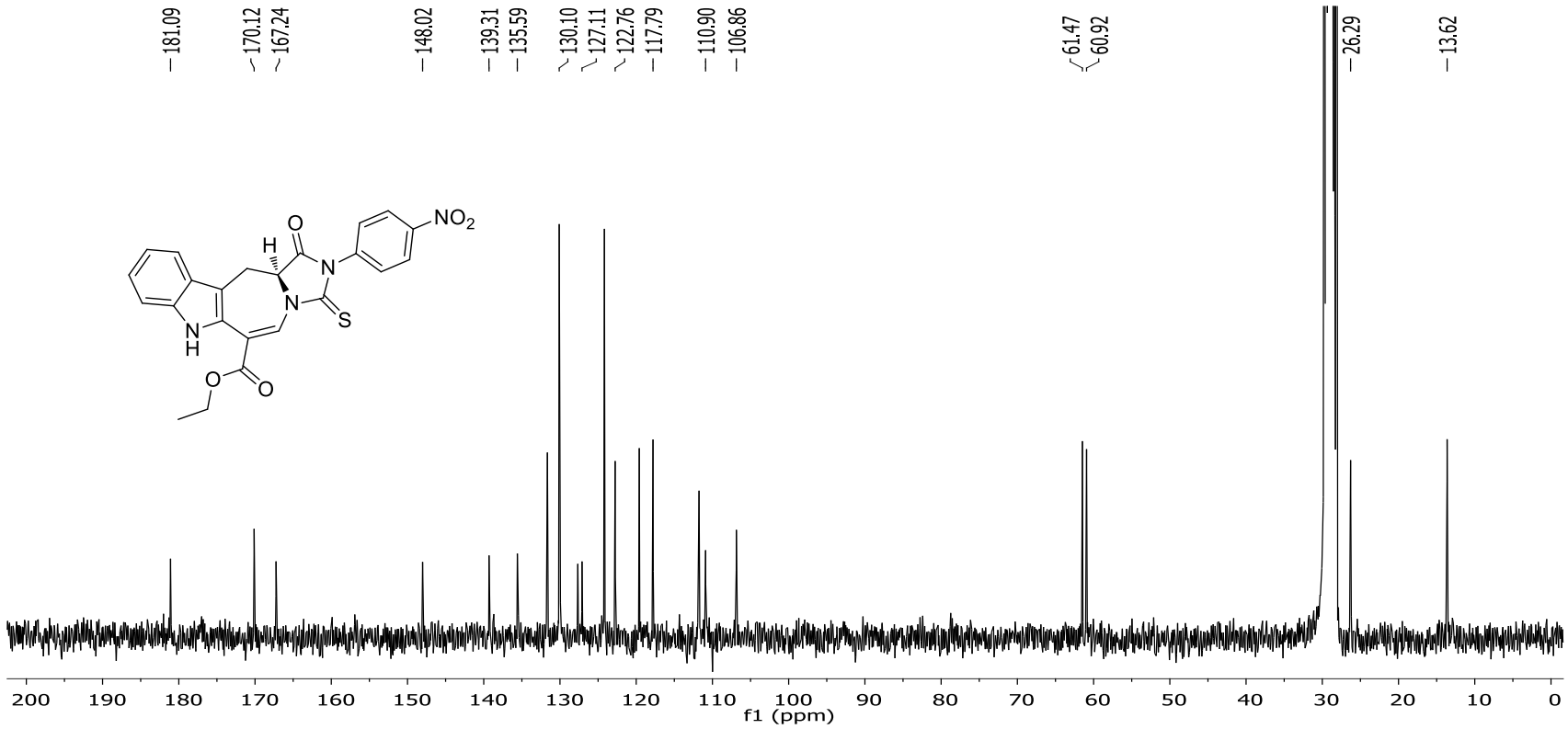
Result Table (Uncal - D:|Indrajeet|HPLC data|TT-LN2-019 - UV)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
1	1.948	33.752	2.553	0.5	2.7
2	2.344	10.666	1.126	0.2	1.2
3	2.936	2.330	0.328	0.0	0.3
4	3.644	2.267	0.447	0.0	0.5
5	4.384	3.304	0.369	0.1	0.4
6	6.864	57.905	0.892	0.9	0.9
7	19.868	6032.542	88.426	98.2	93.9
	Total	6142.768	94.143	100.0	100.0

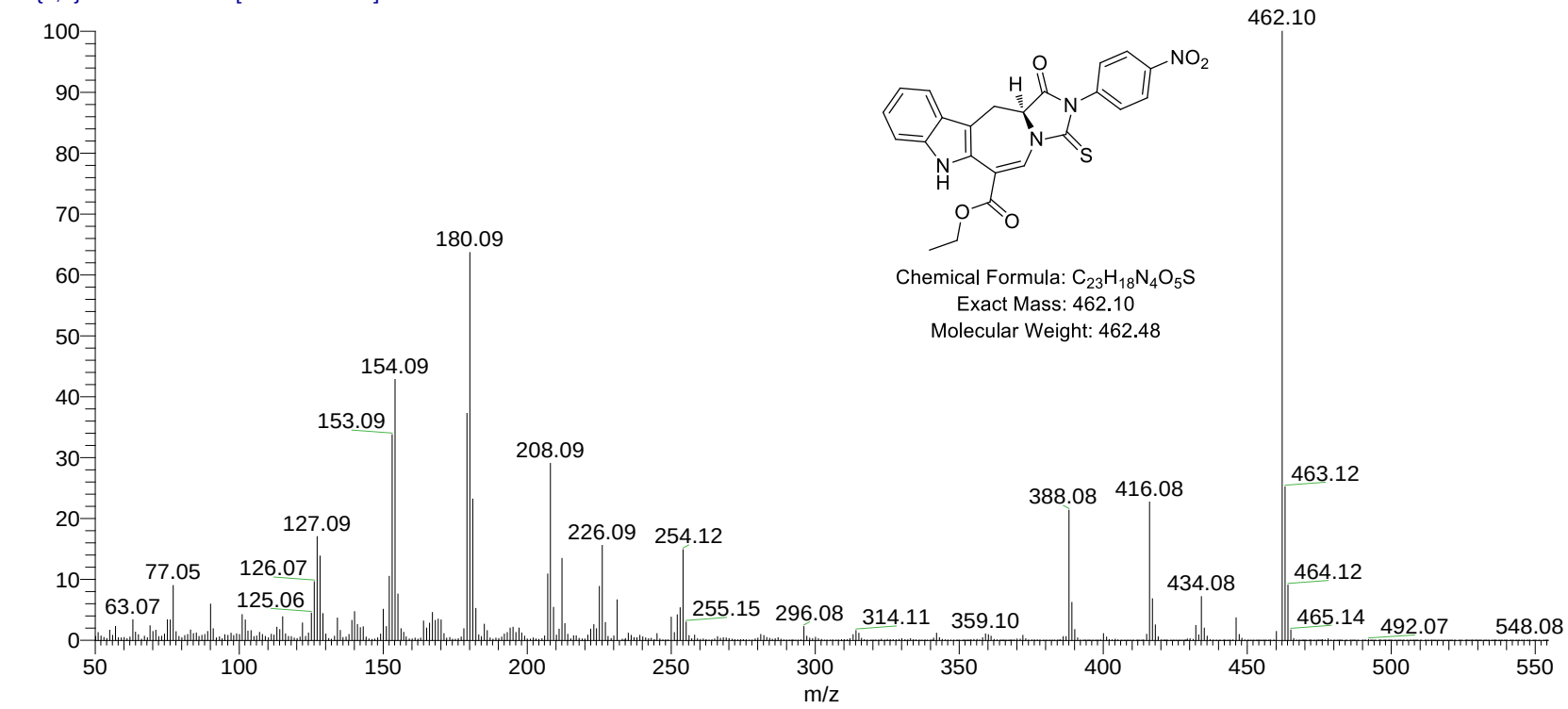
HPLC of compound **7e**



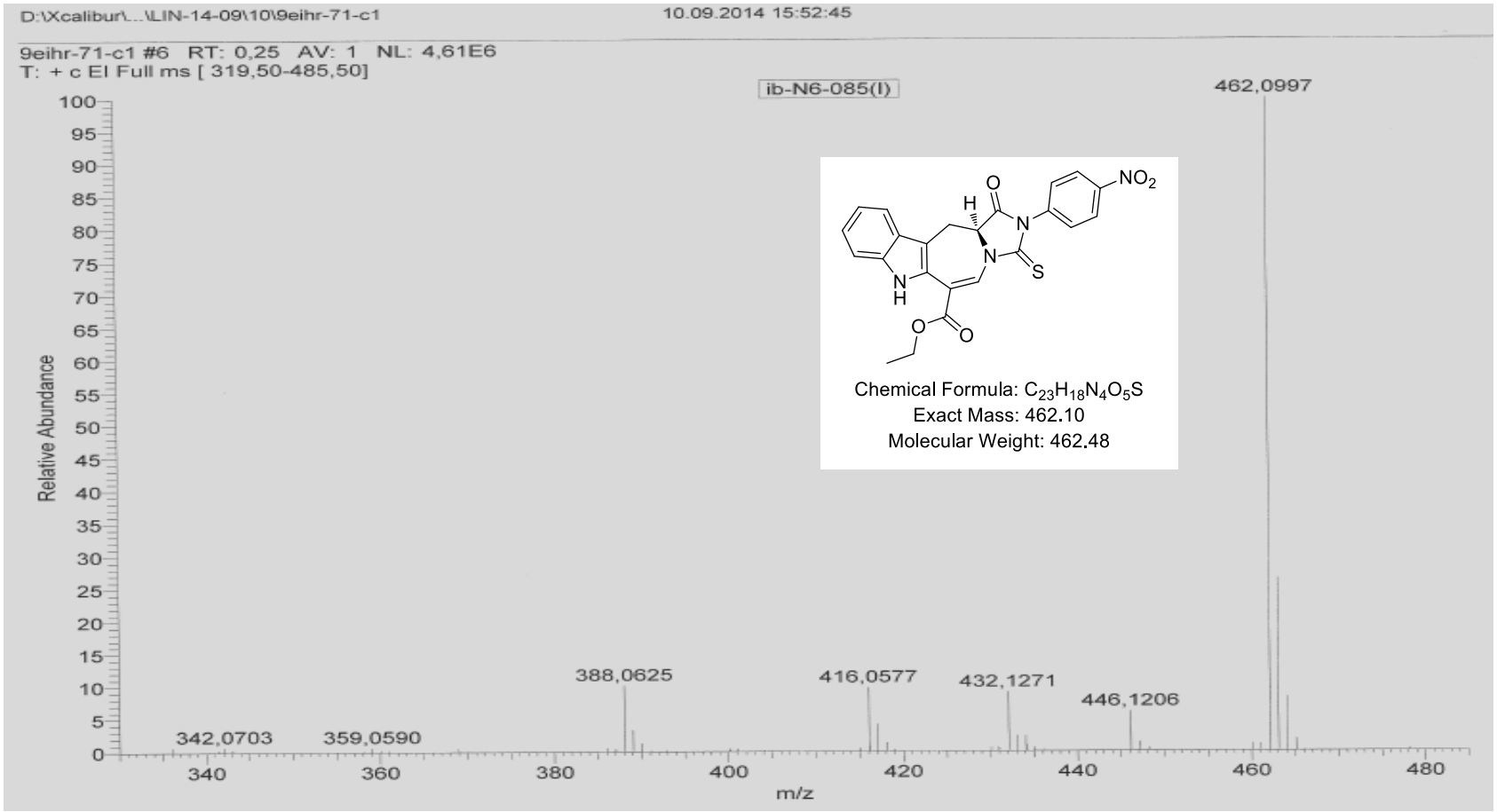
^{1}H NMR spectrum (300 MHz) of compound **7f** in acetone-d_6



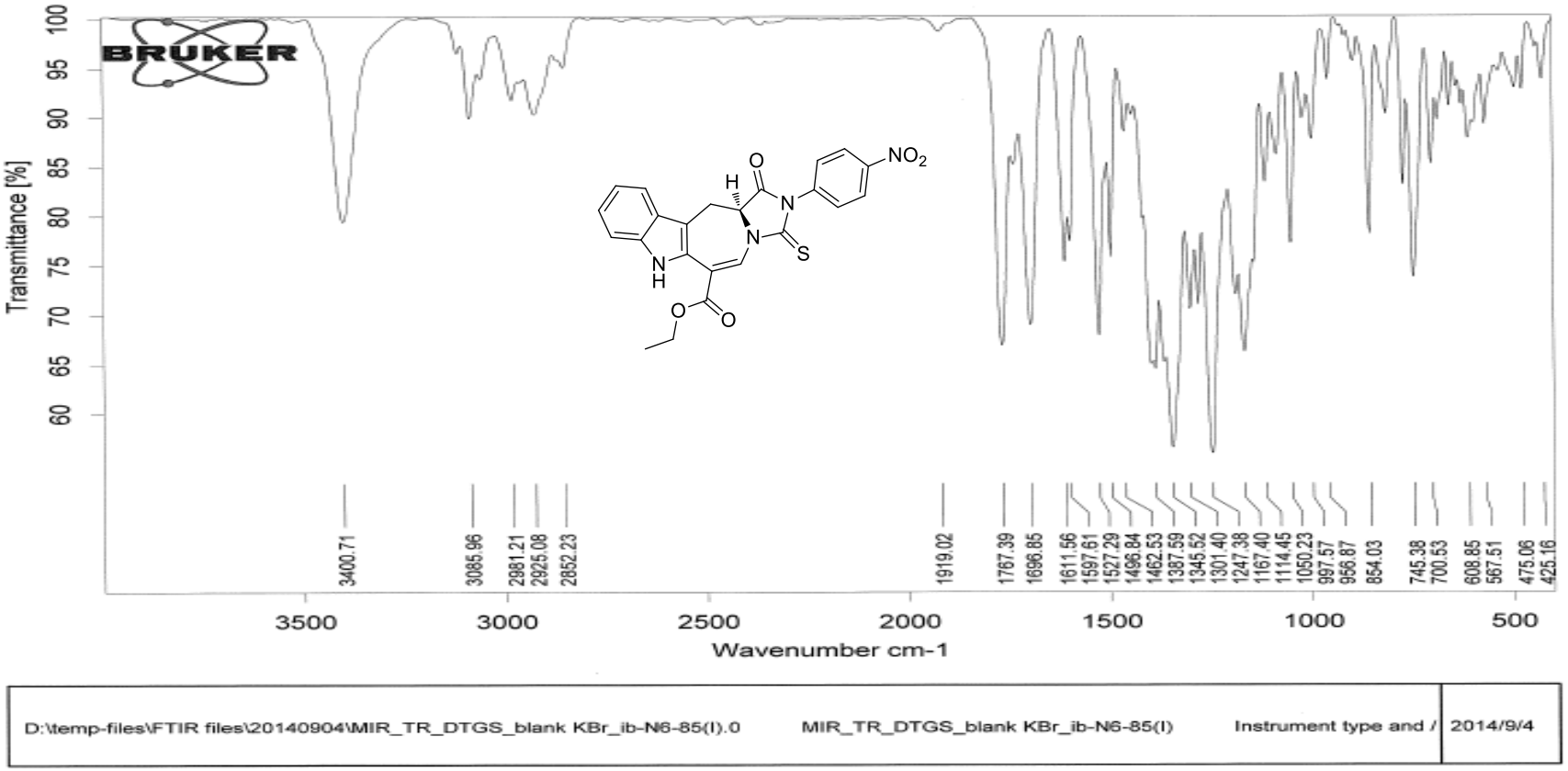
^{13}C NMR spectrum (75 MHz) of compound **7f** in acetone-d_6



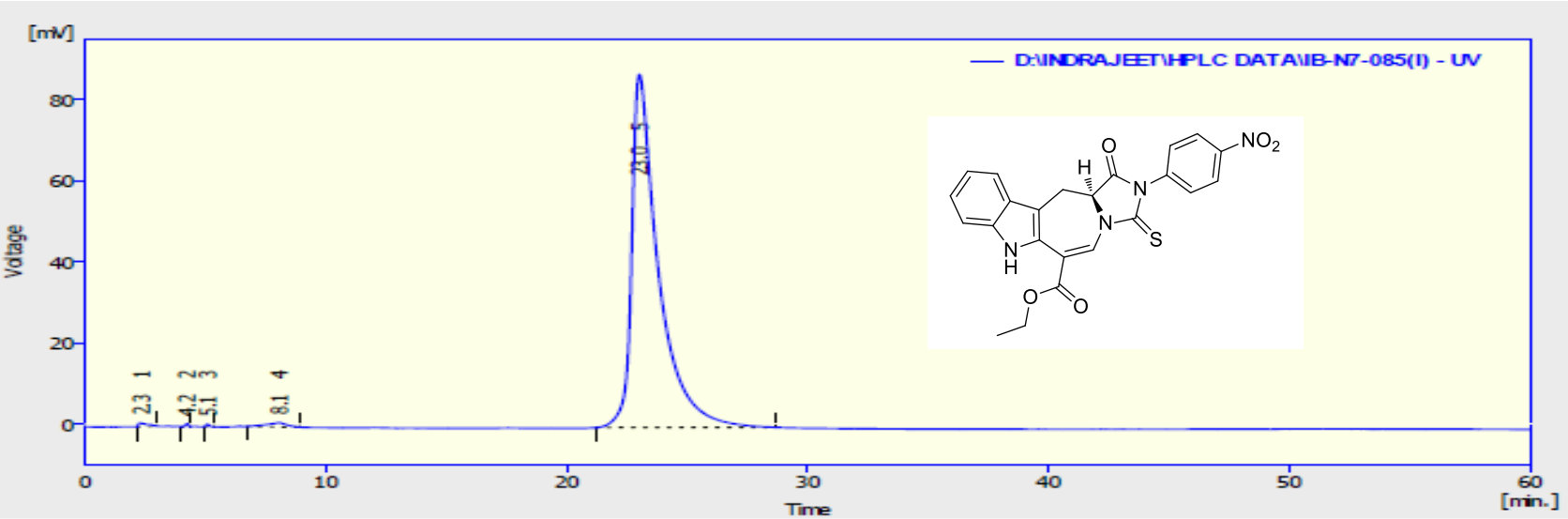
EI-LRMS of compound **7f**



EI-HRMS of compound **7f**



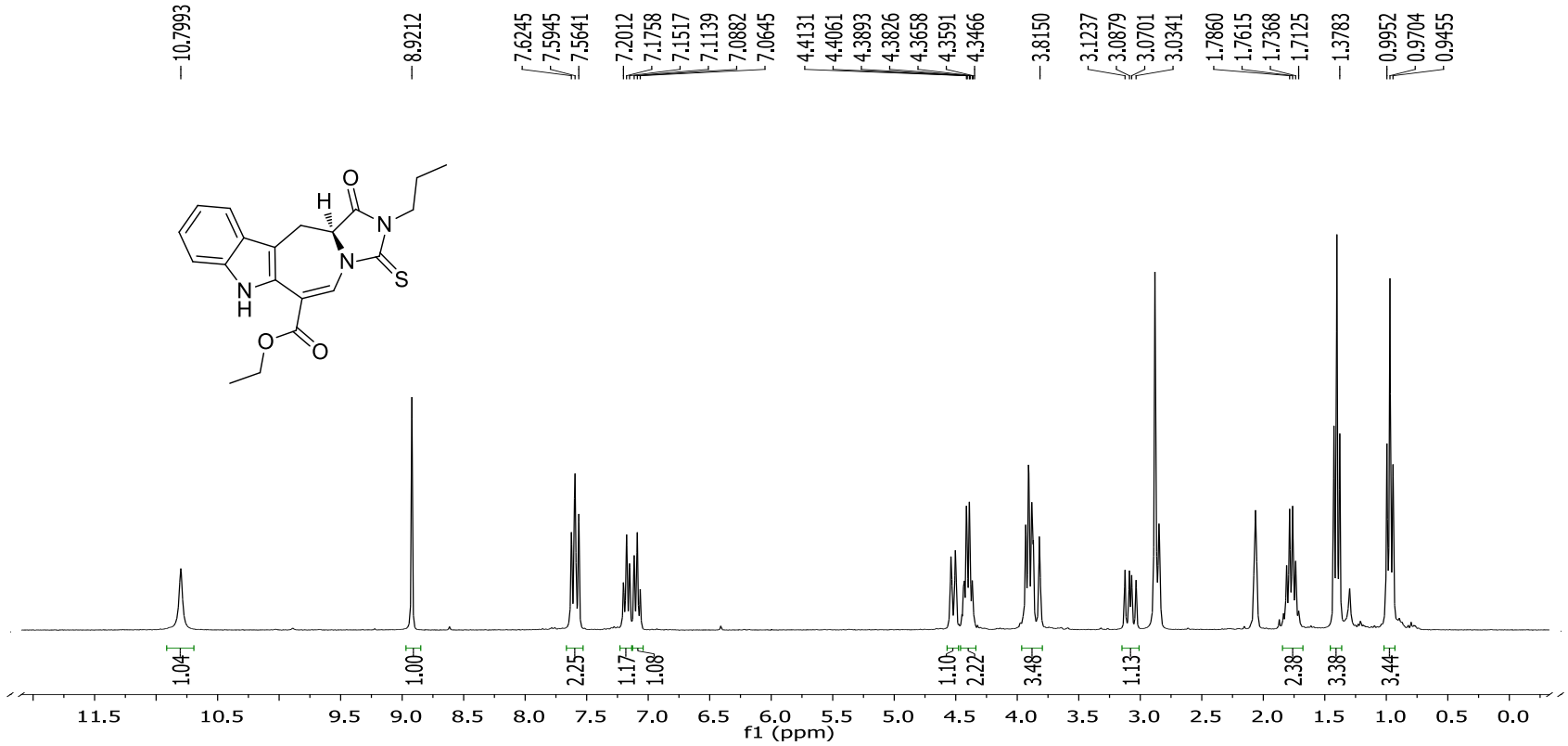
IR spectrum of compound **7f**



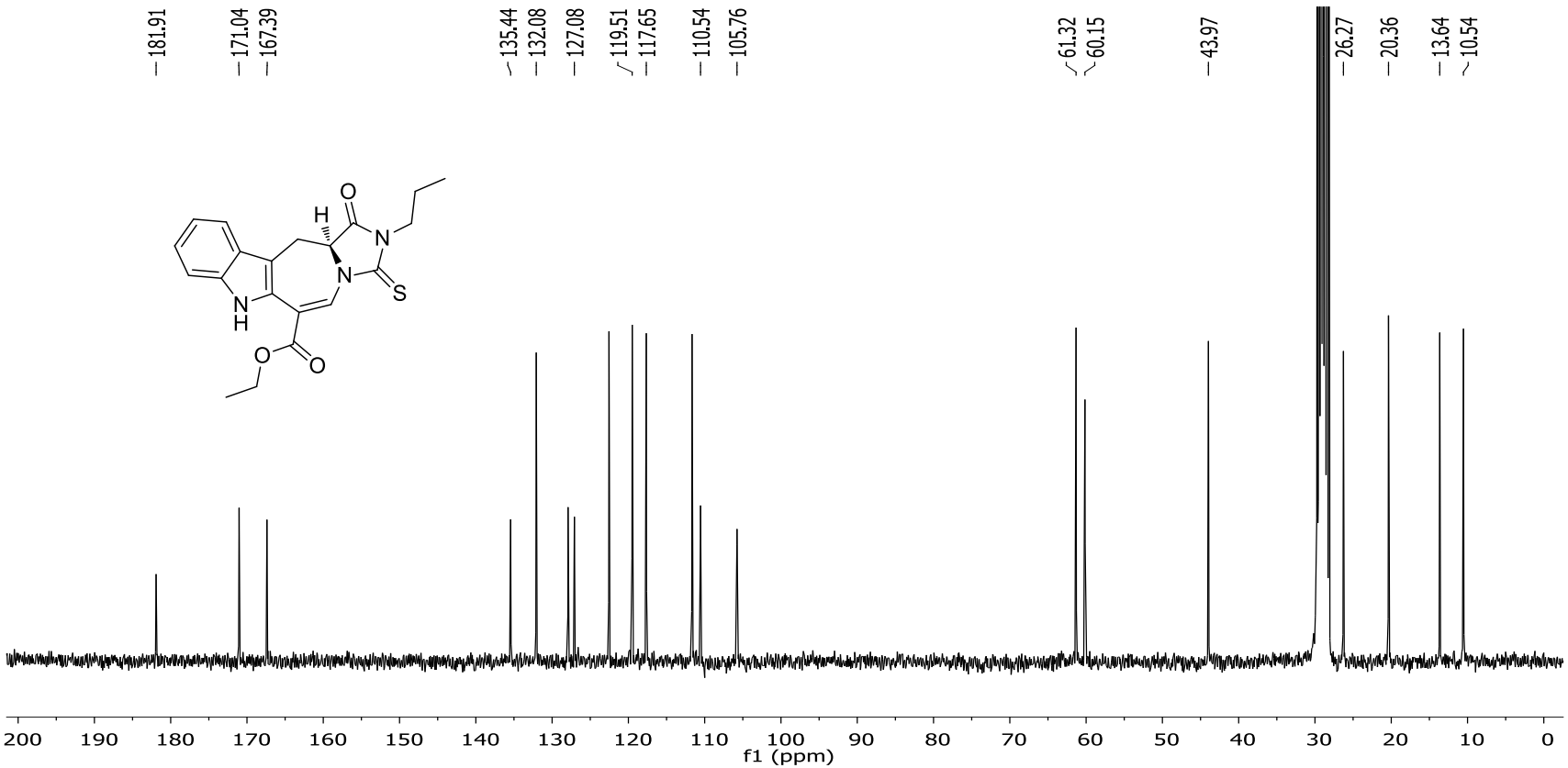
Result Table (Uncal - D:\INDRAJEET\HPLC DATA\IB-N7-085(I) - UV)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
1	2.324	20.669	0.853	0.3	0.9
2	4.232	4.666	0.698	0.1	0.8
3	5.080	5.681	0.634	0.1	0.7
4	8.068	55.355	1.011	0.8	1.1
5	23.020	6710.792	87.037	98.7	96.5
	Total	6797.163	90.233	100.0	100.0

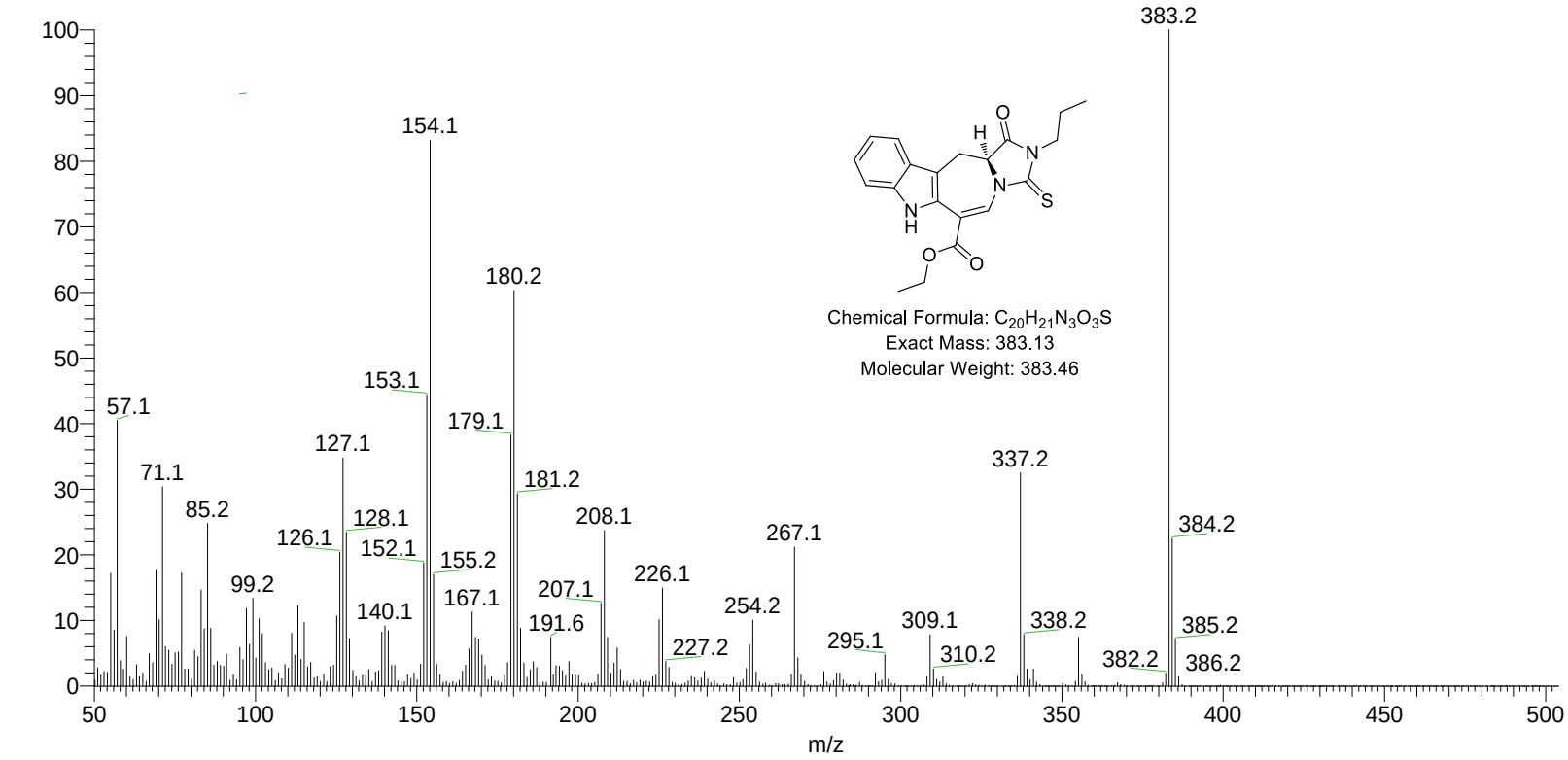
HPLC of compound **7f**



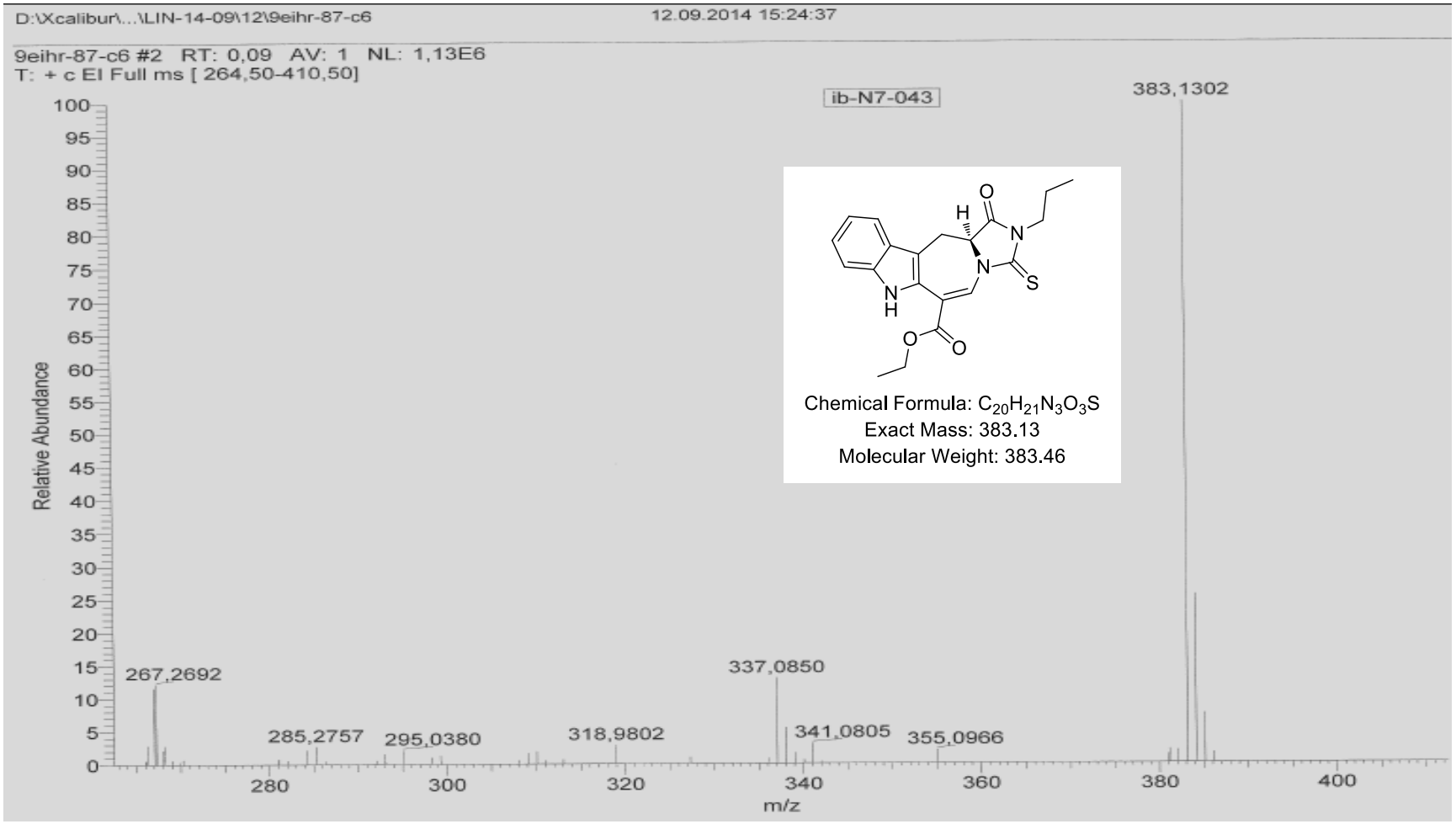
^{1}H NMR spectrum (300 MHz) of compound **7g** in acetone-d_6



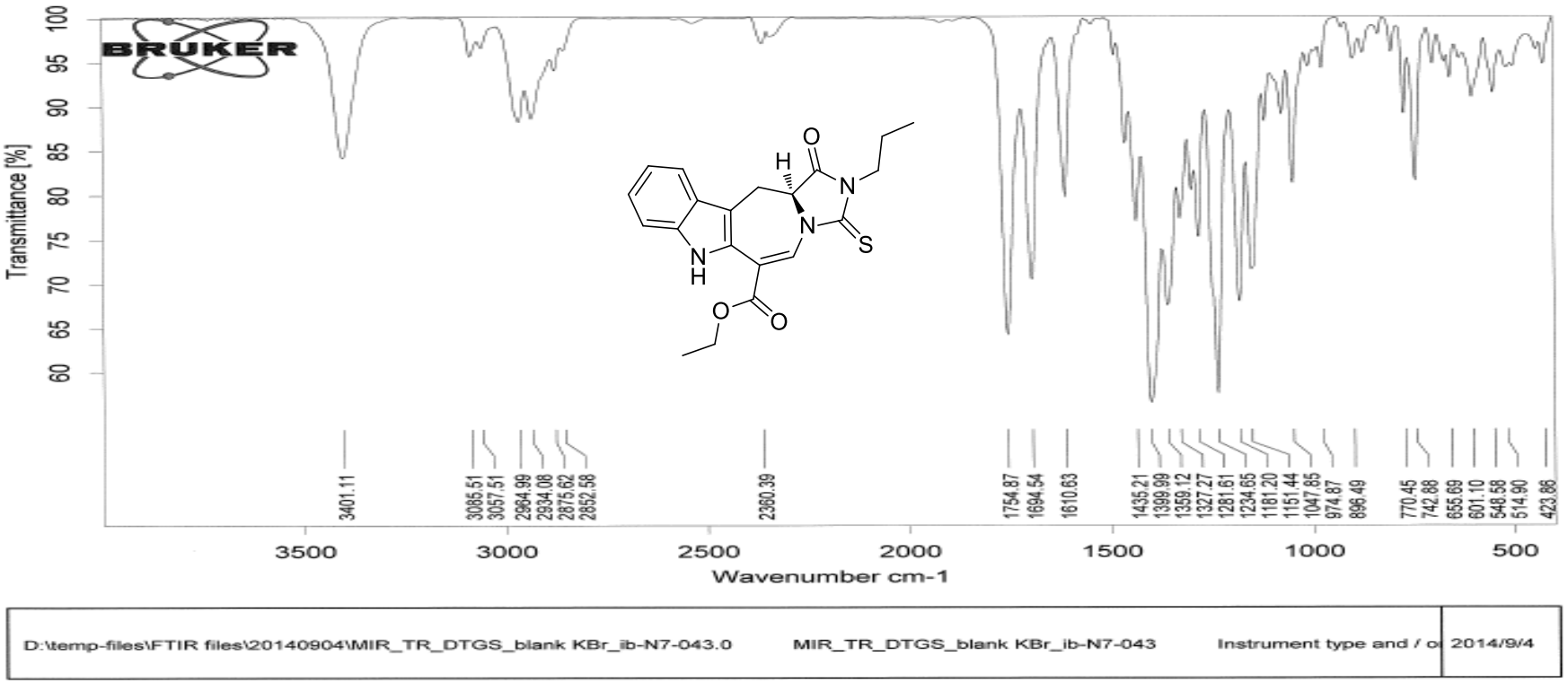
^{13}C NMR spectrum (75 MHz) of compound **7g** in acetone-d_6



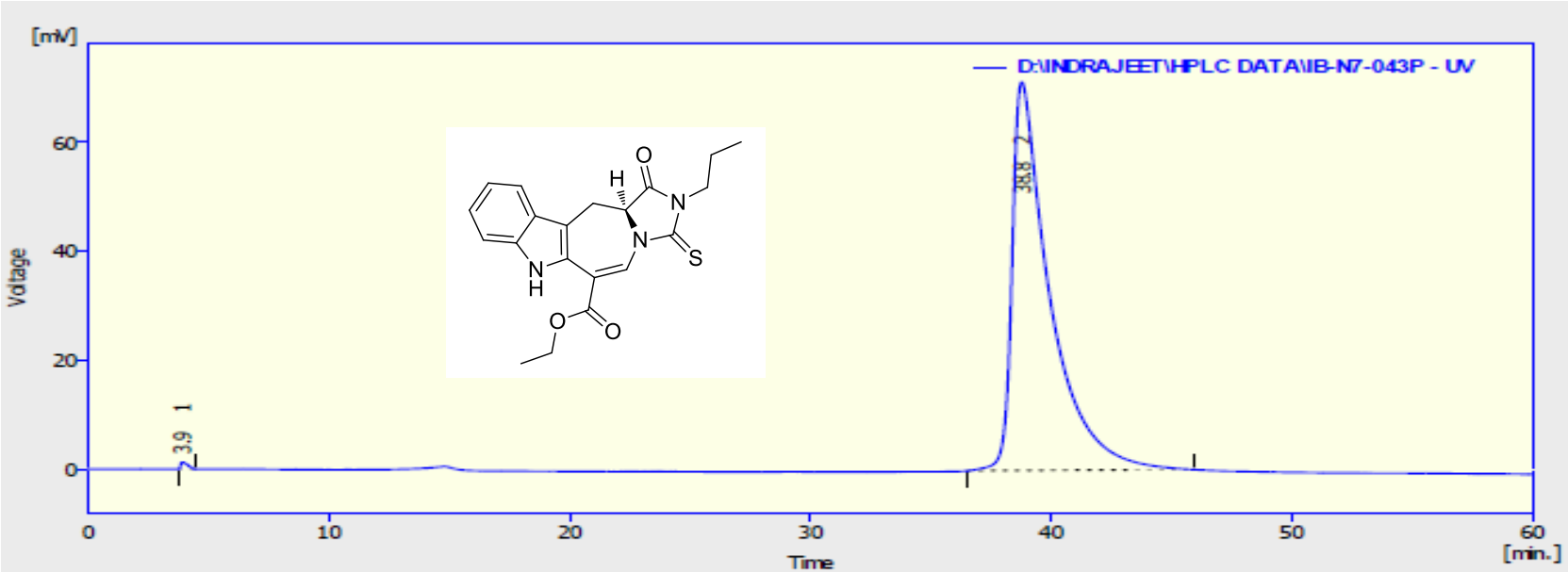
EI-LRMS of compound **7g**



EI-HRMS of compound **7g**



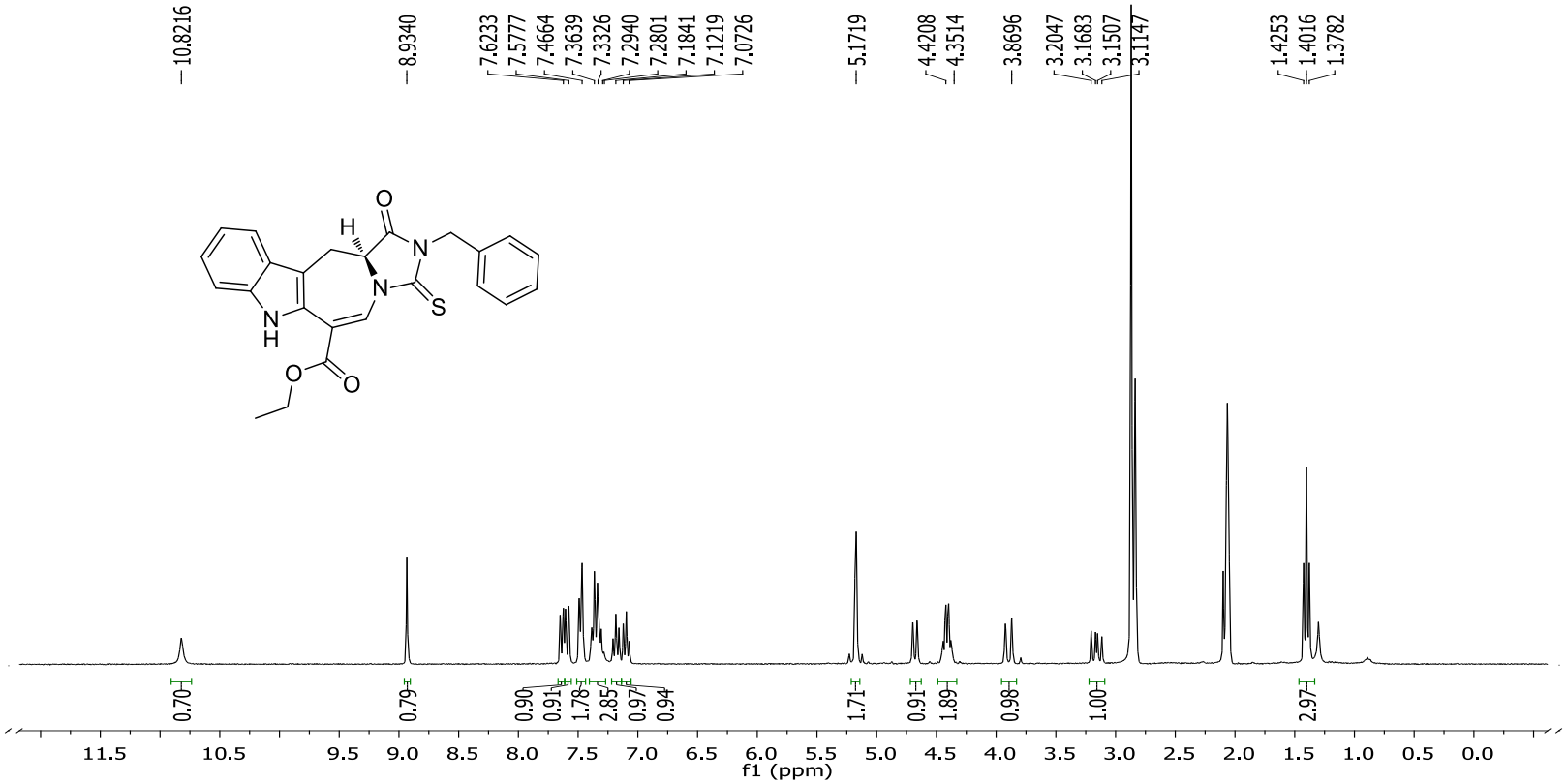
IR spectrum of compound **7g**



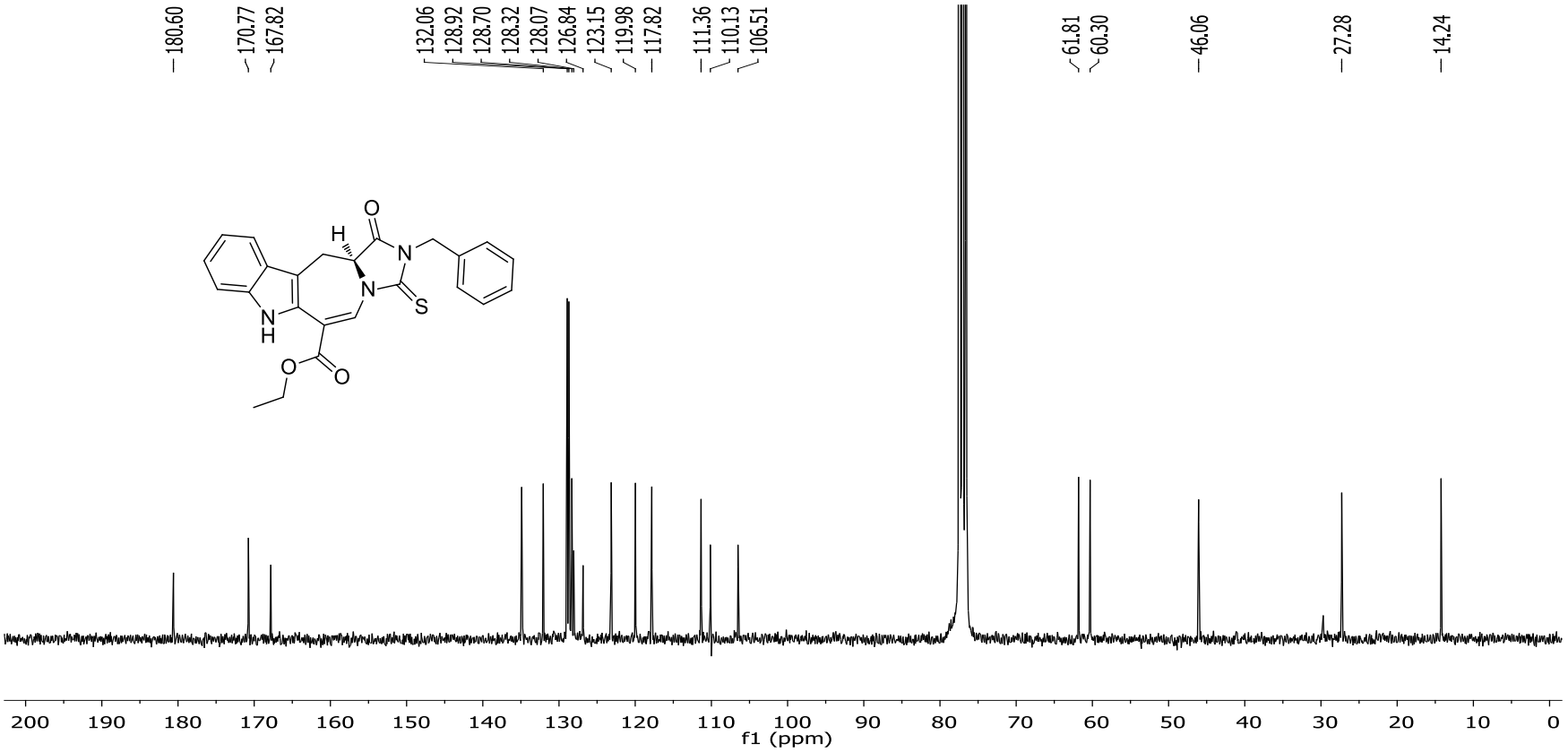
Result Table (Uncal - D:\INDRAJEET\HPLC DATA\IB-N7-043P - UV)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
1	3.884	26.167	1.262	0.3	1.7
2	38.780	7752.907	71.369	99.7	98.3
	Total	7779.074	72.632	100.0	100.0

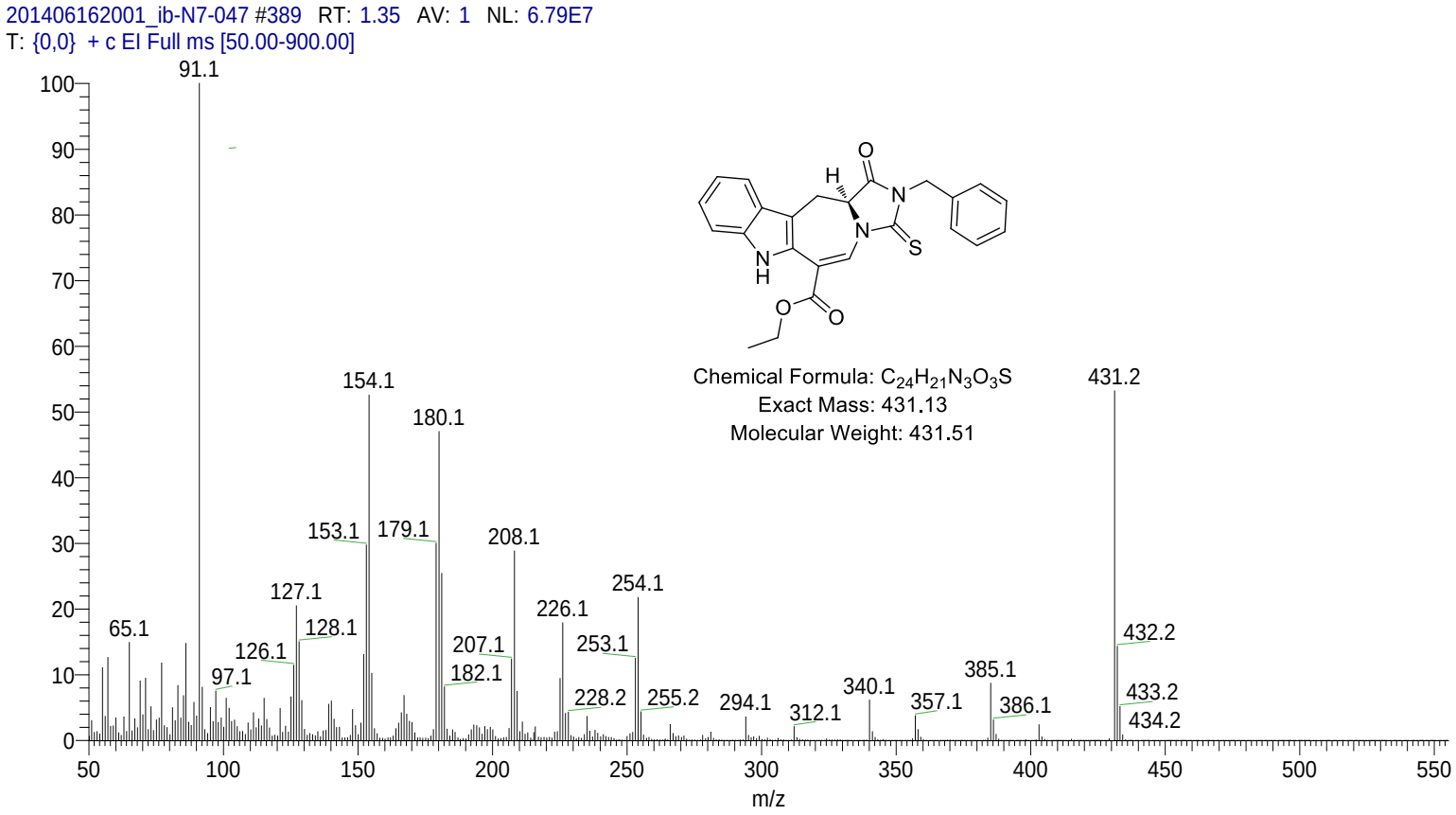
HPLC of compound **7g**



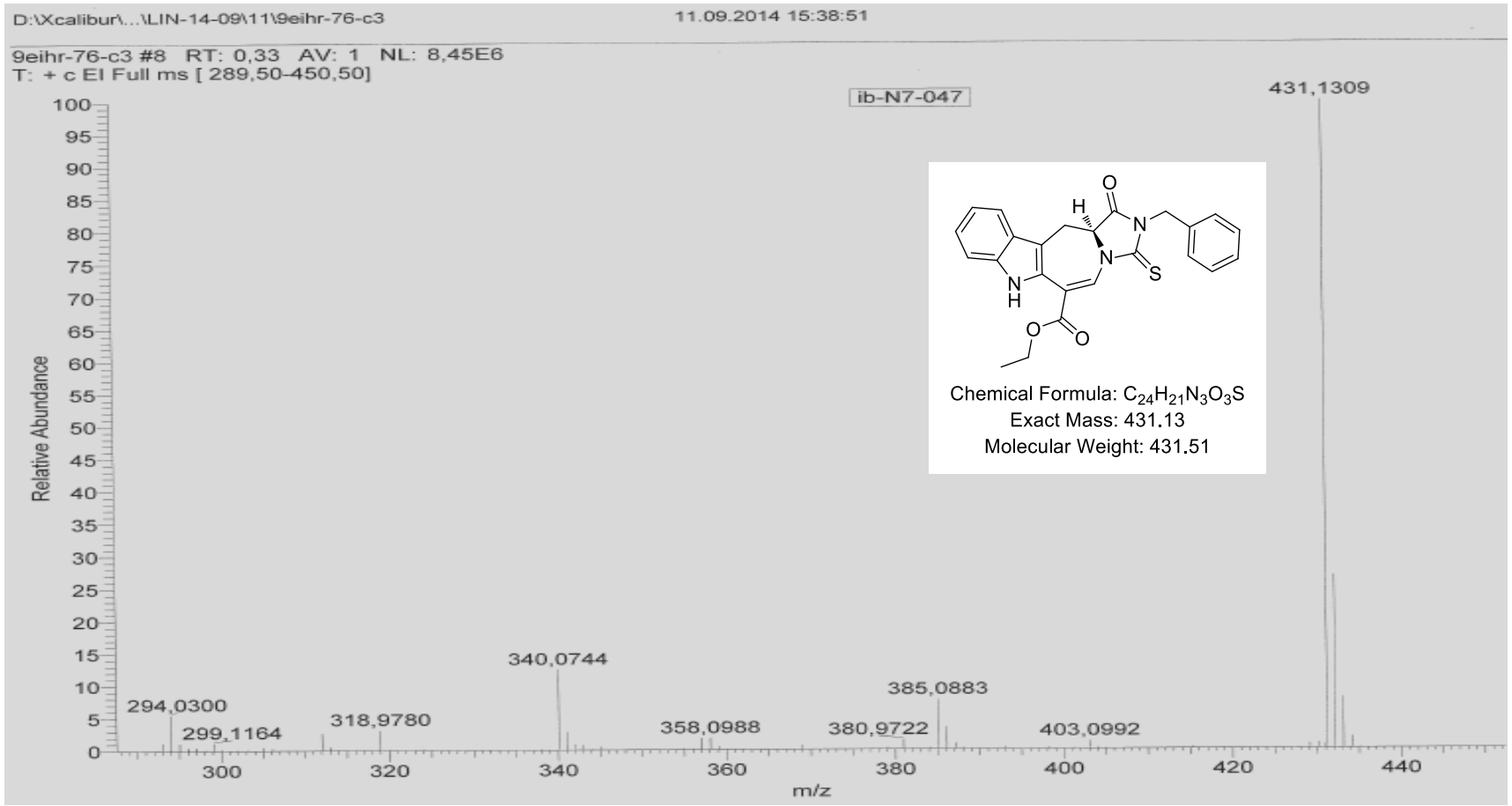
^{1}H NMR spectrum (300 MHz) of compound **7h** in acetone-d_6



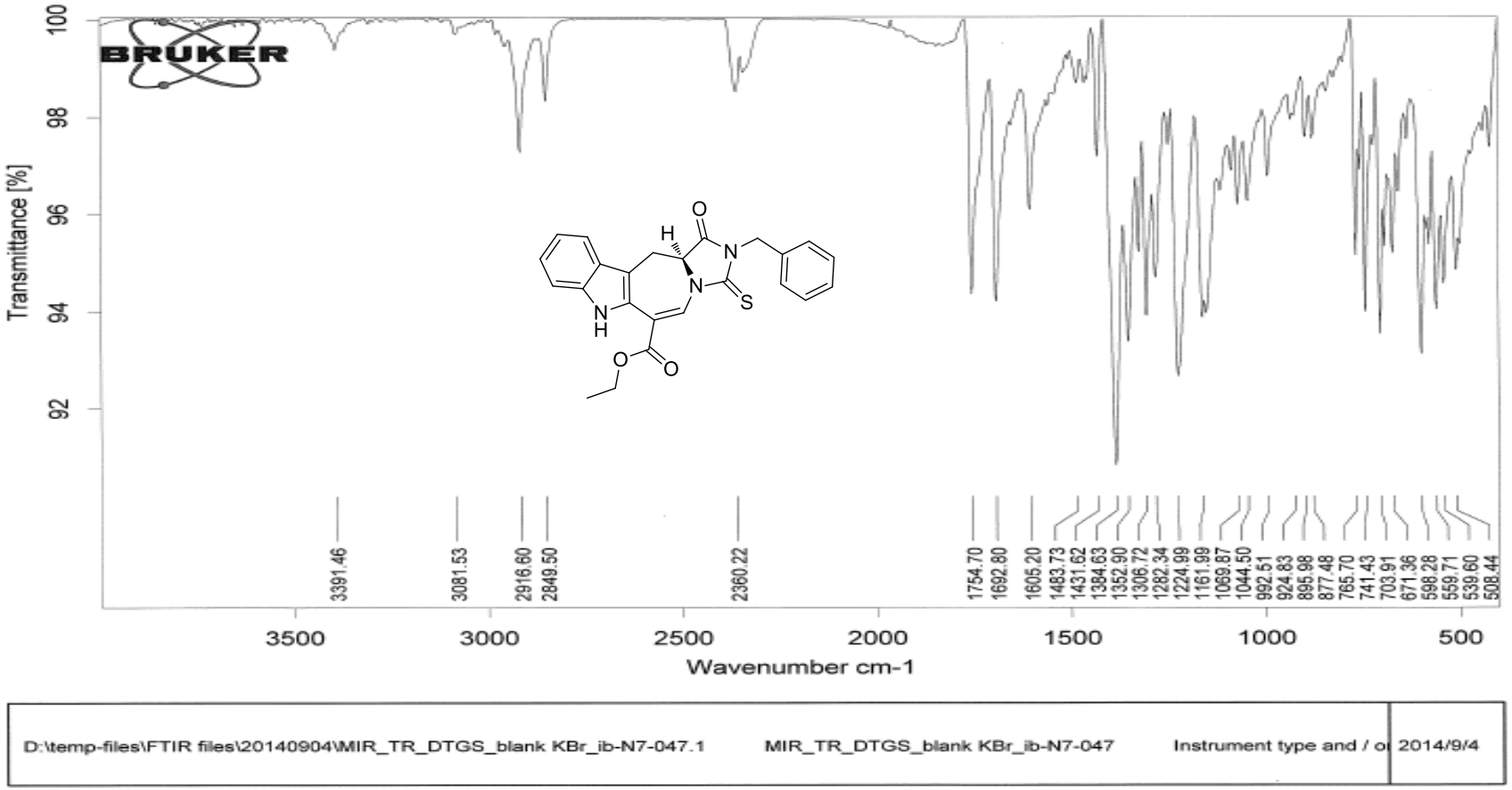
^{13}C NMR spectrum (75 MHz) of compound **7h** in $CDCl_3$



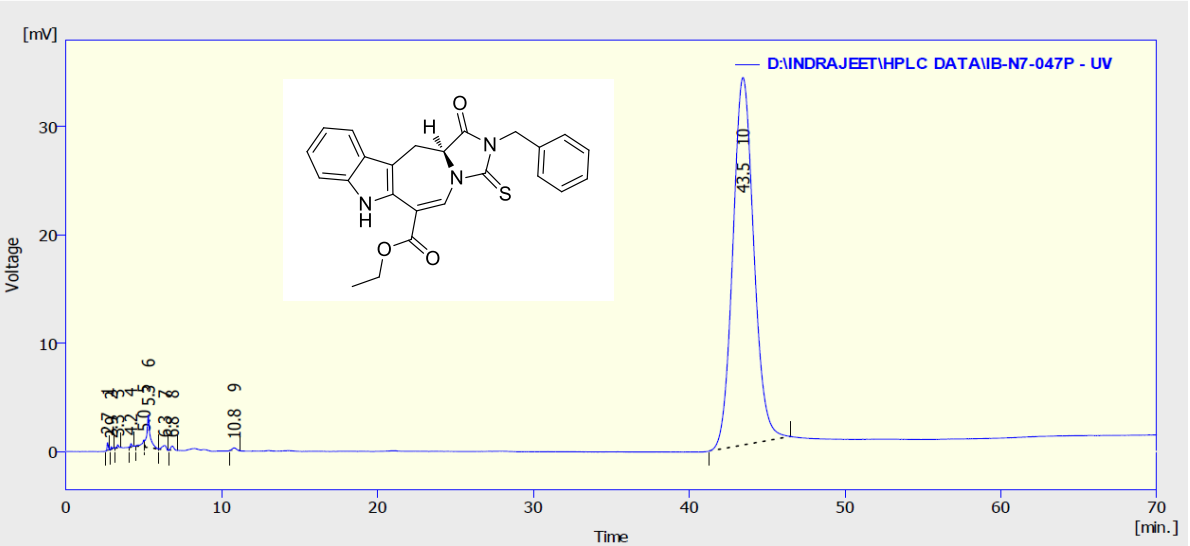
EI-LRMS of compound **7h**



EI-HRMS of compound **7h**



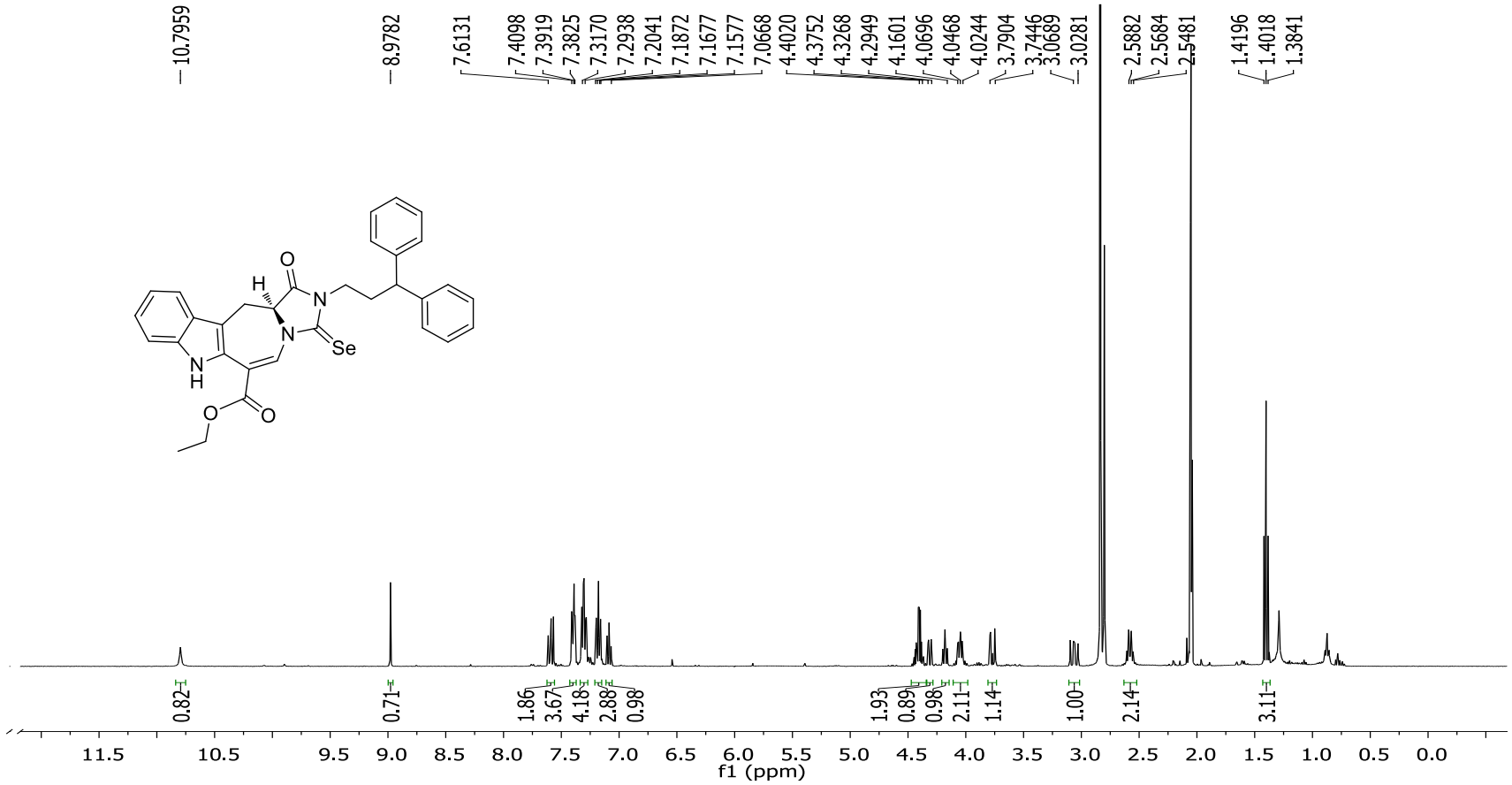
IR spectrum of compound **7h**



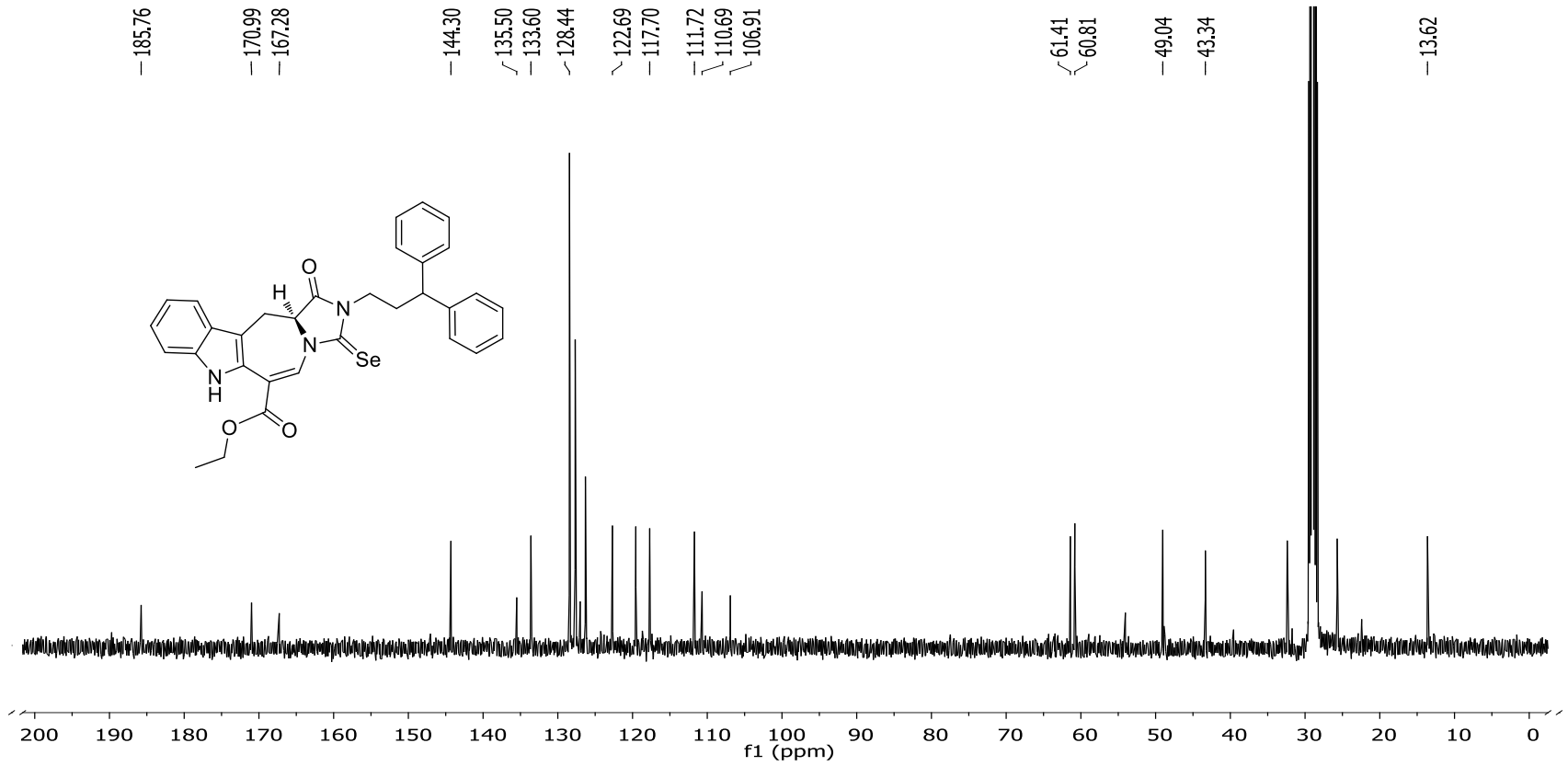
Result Table (Uncal - D:\INDRAJEET\HPLC DATA\IB-N7-047P - UV)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
1	2.676	4.017	0.727	0.1	1.8
2	2.916	1.148	0.226	0.0	0.6
3	3.328	1.929	0.281	0.1	0.7
4	4.180	2.012	0.315	0.1	0.8
5	5.012	8.144	0.652	0.3	1.6
6	5.292	43.362	3.021	1.3	7.5
7	6.348	7.431	0.399	0.2	1.0
8	6.824	5.681	0.398	0.2	1.0
9	10.792	5.000	0.259	0.2	0.6
10	43.456	3164.538	33.889	97.6	84.4
	Total	3243.262	40.169	100.0	100.0

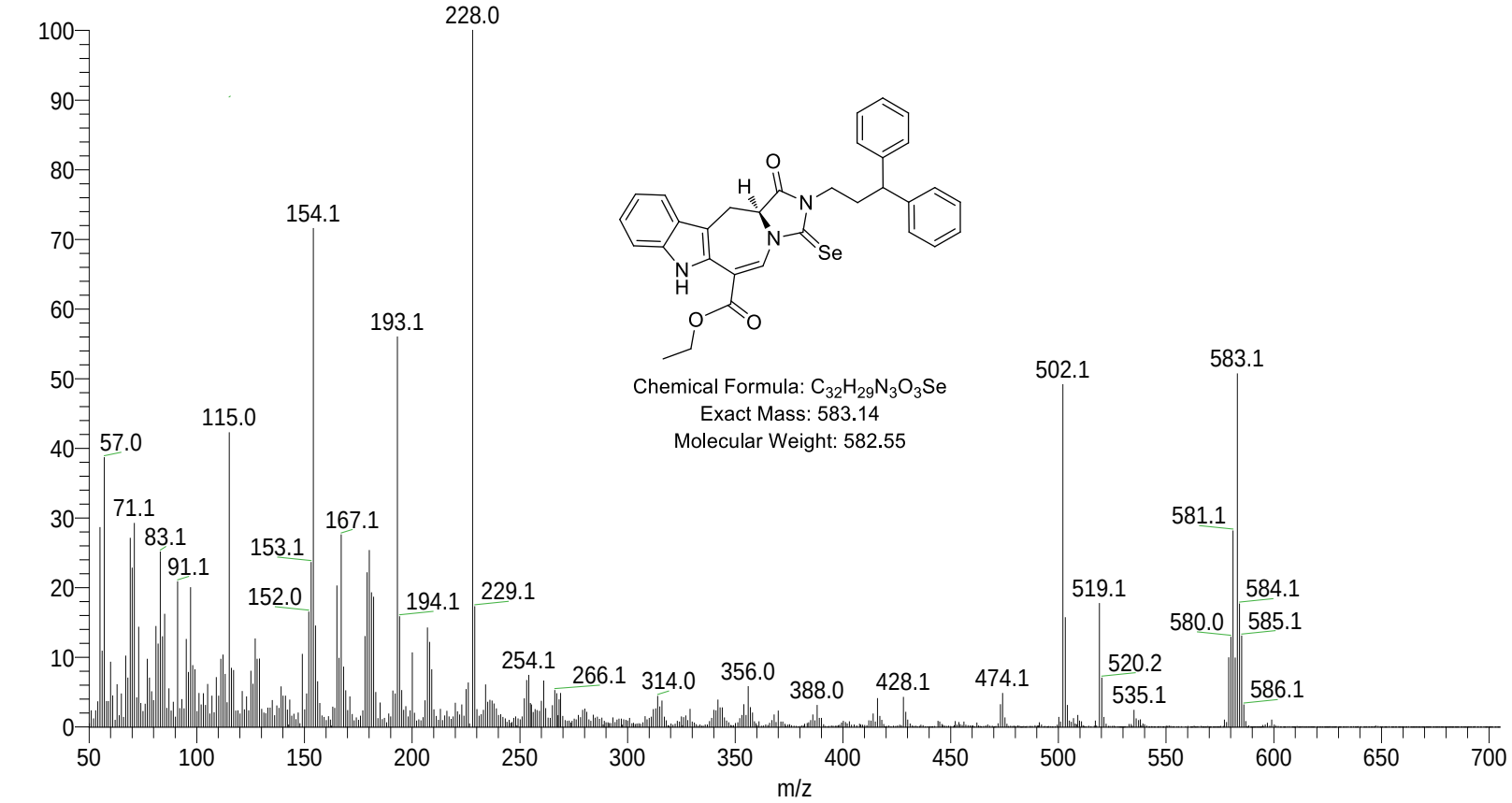
HPLC of compound **7h**



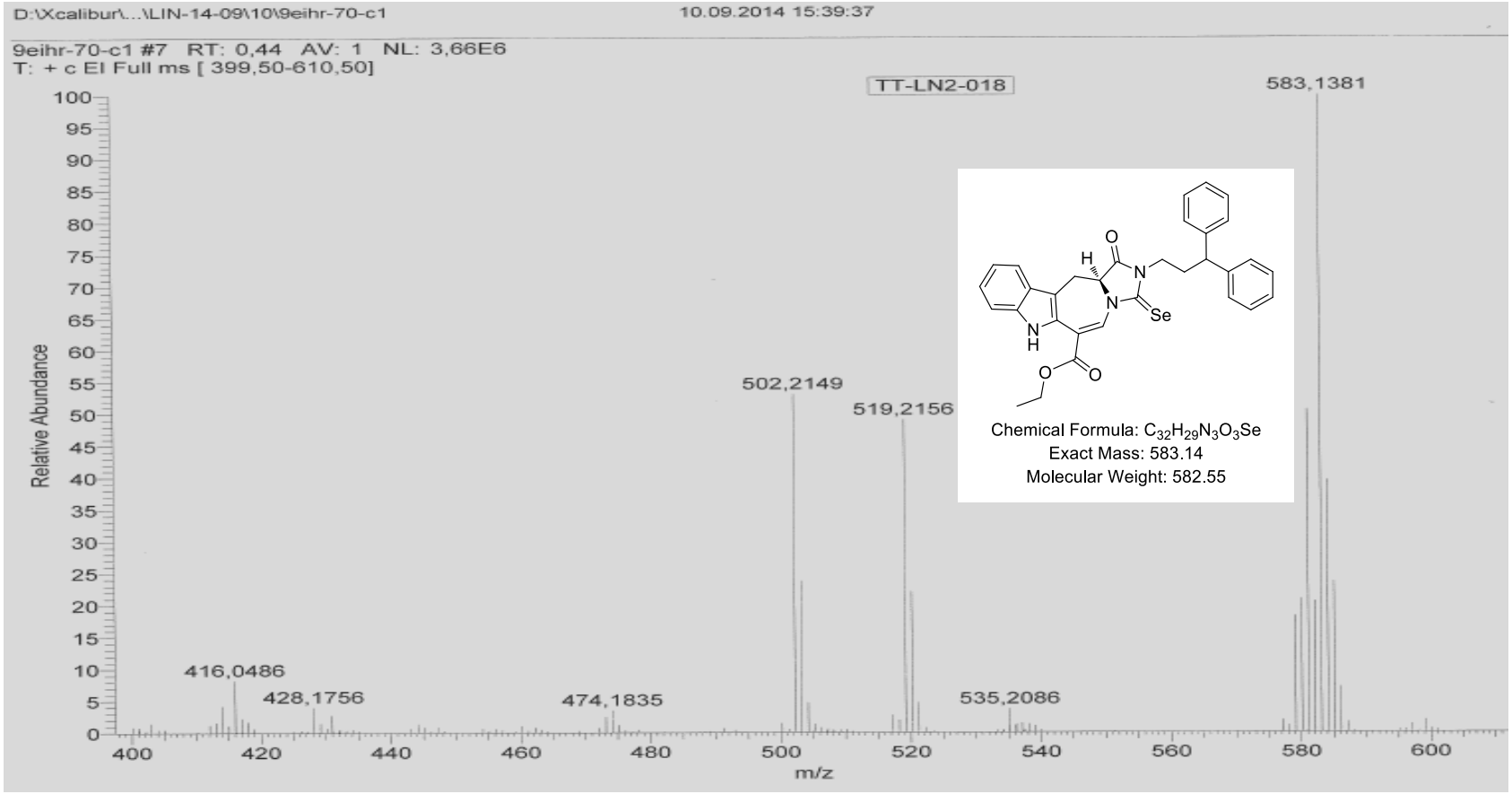
^{1}H NMR spectrum (400 MHz) of compound **7i** in acetone-d_6



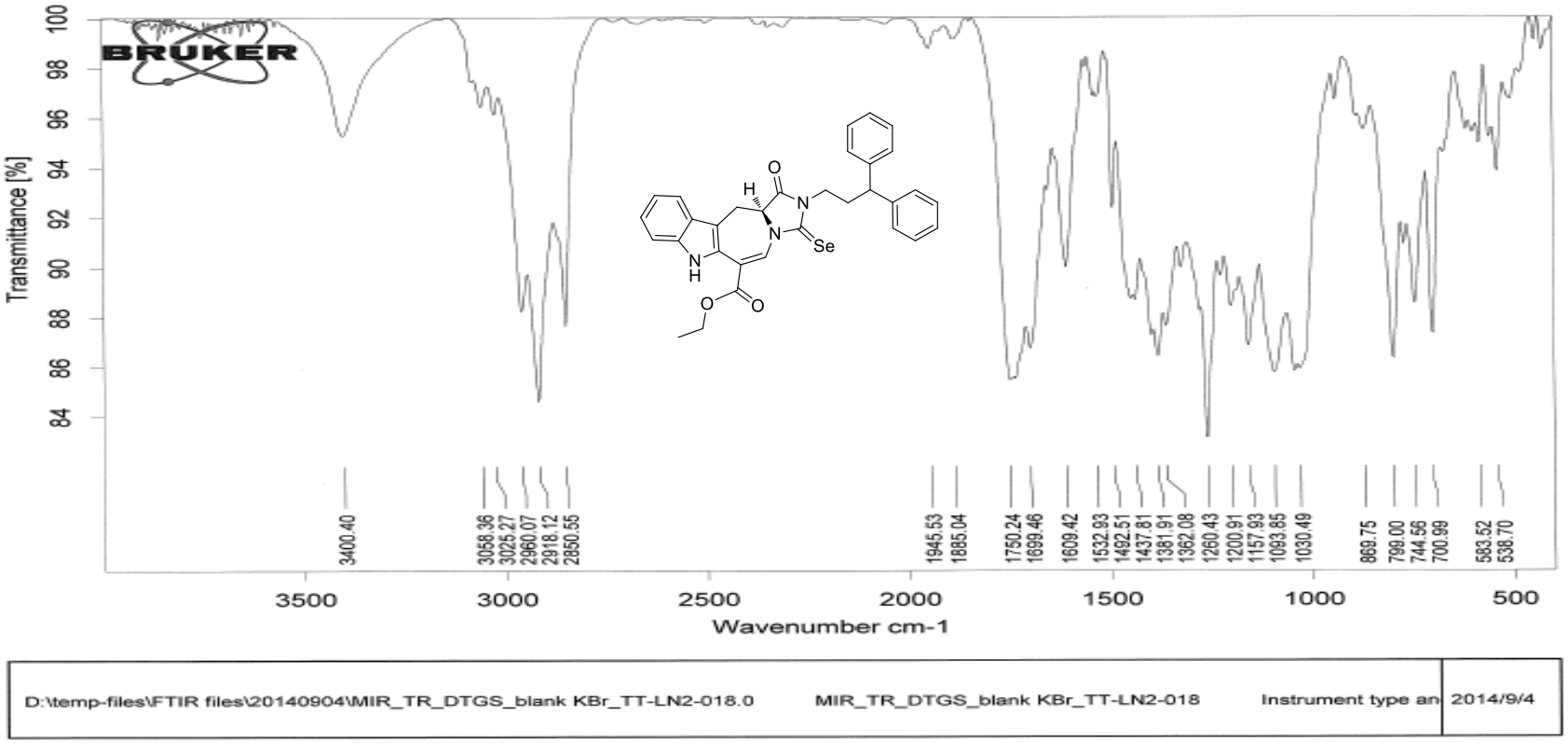
^{13}C NMR spectrum (101 MHz) of compound **7i** in acetone-d_6



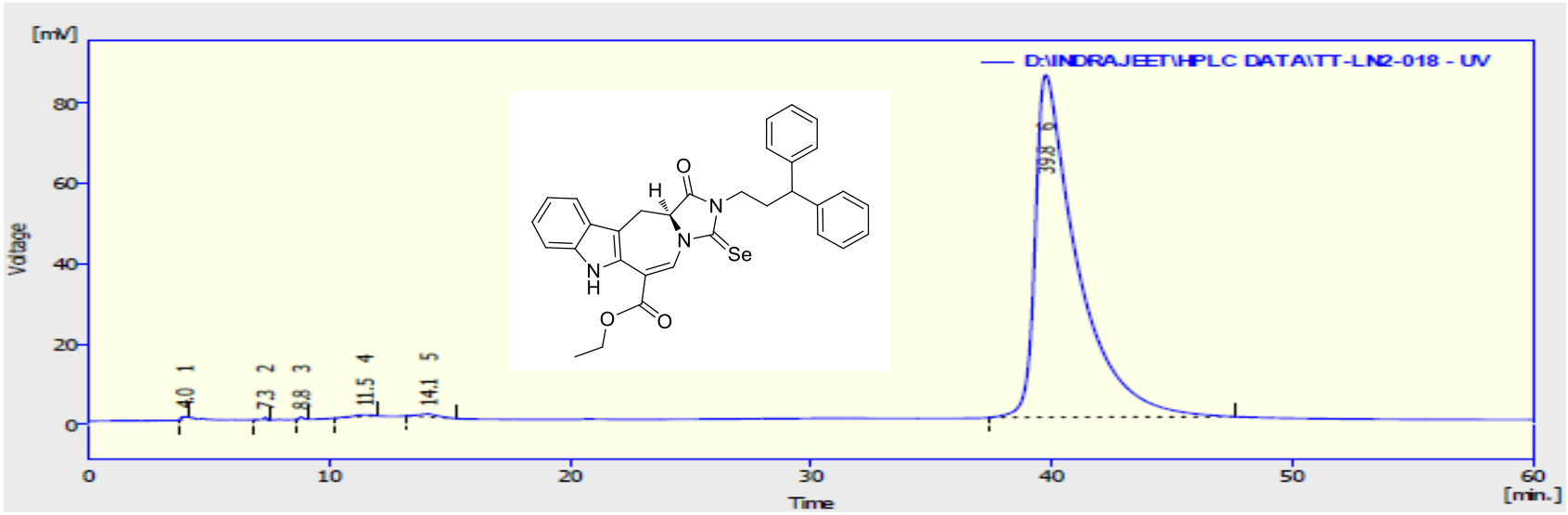
EI-LRMS of compound **7i**



EI-HRMS of compound **7i**



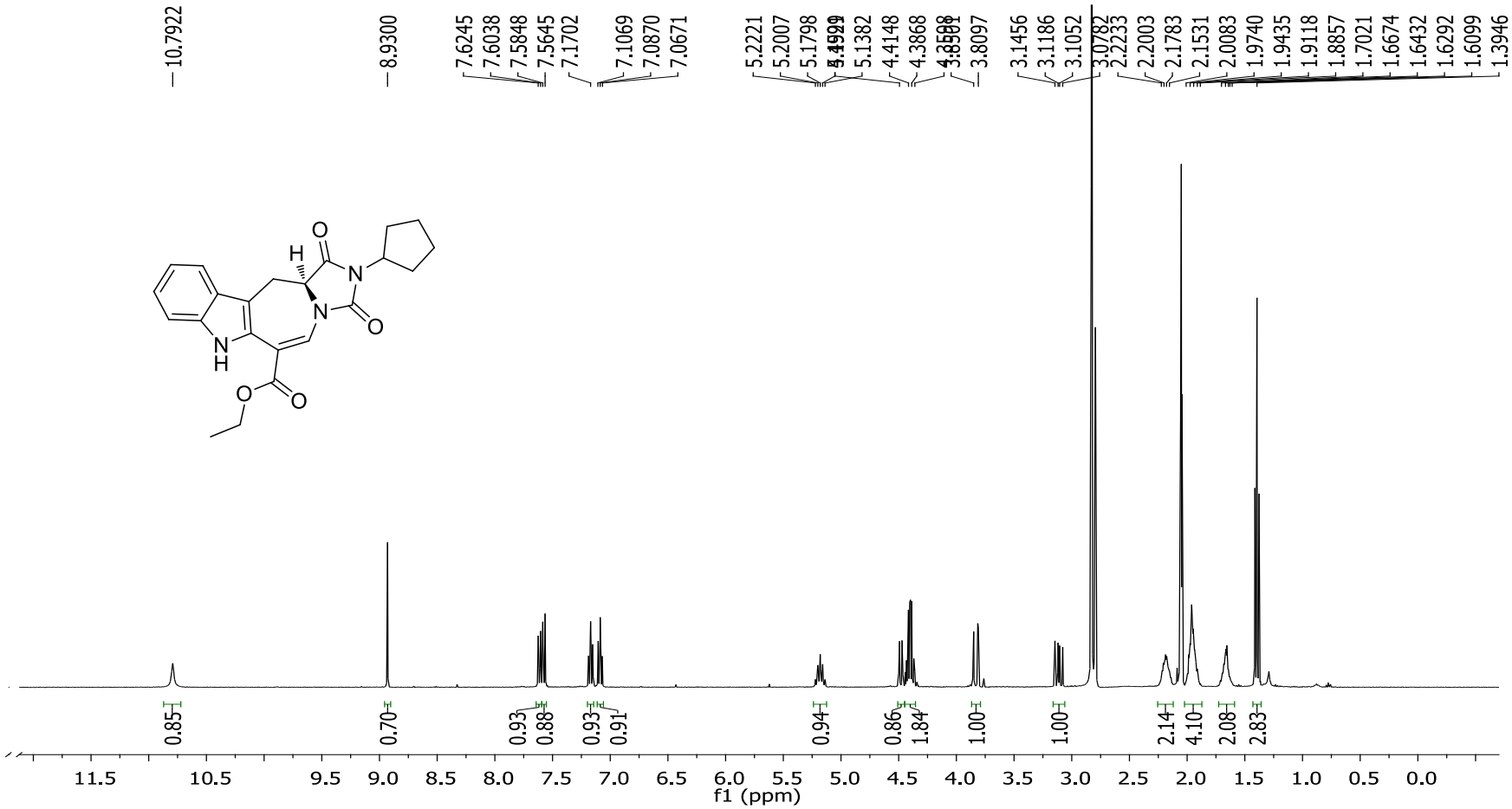
IR spectrum of compound **7i**



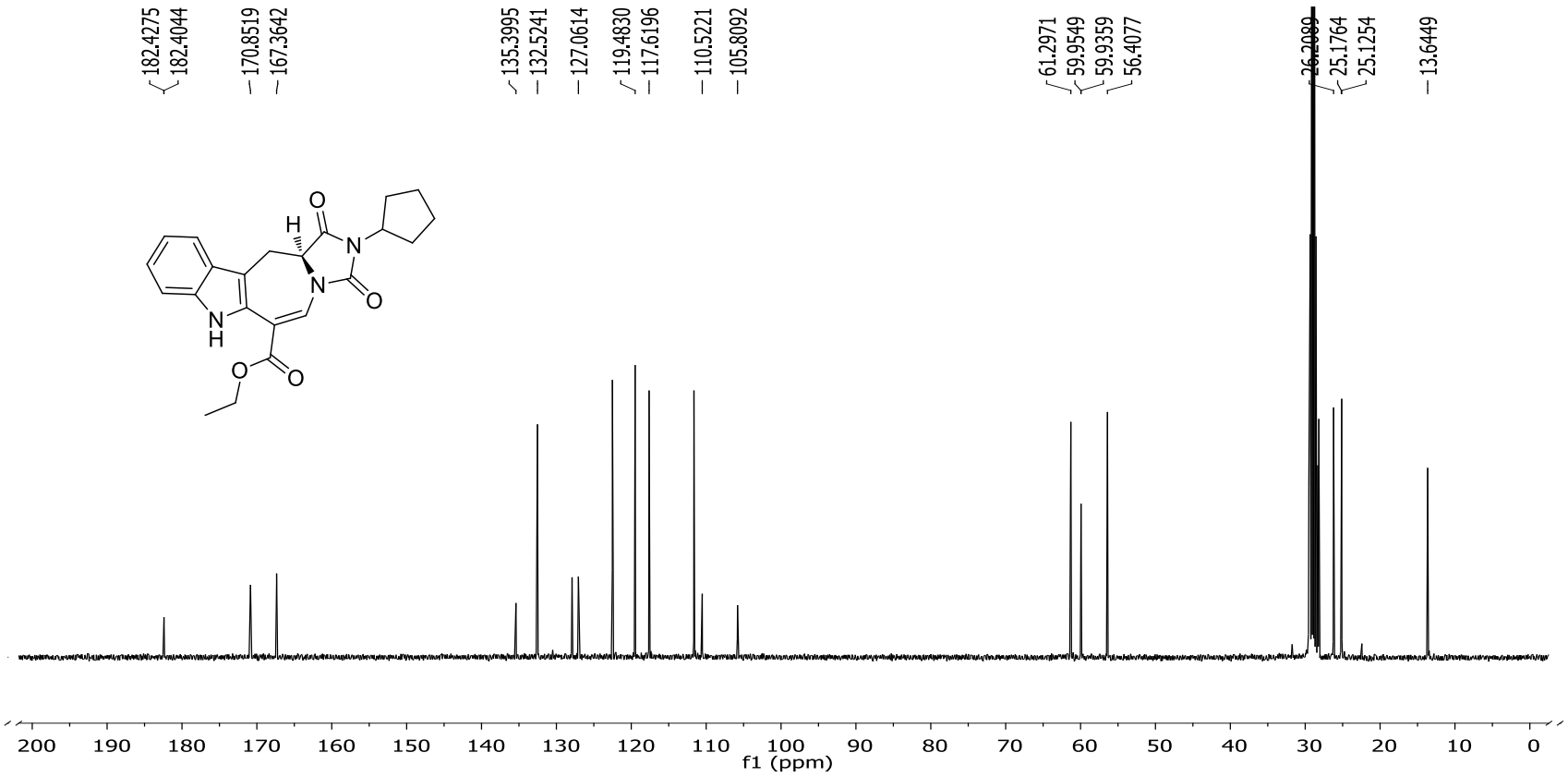
Result Table (Uncal - D:\INDRAJEET\HPLC DATA\TT-LN2-018 - UV)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
1	3.960	6.502	0.398	0.1	0.5
2	7.336	6.736	0.637	0.1	0.7
3	8.820	6.735	0.567	0.1	0.6
4	11.484	19.447	0.346	0.2	0.4
5	14.104	40.795	0.773	0.4	0.9
6	39.764	10348.477	85.279	99.2	96.9
	Total	10428.692	88.001	100.0	100.0

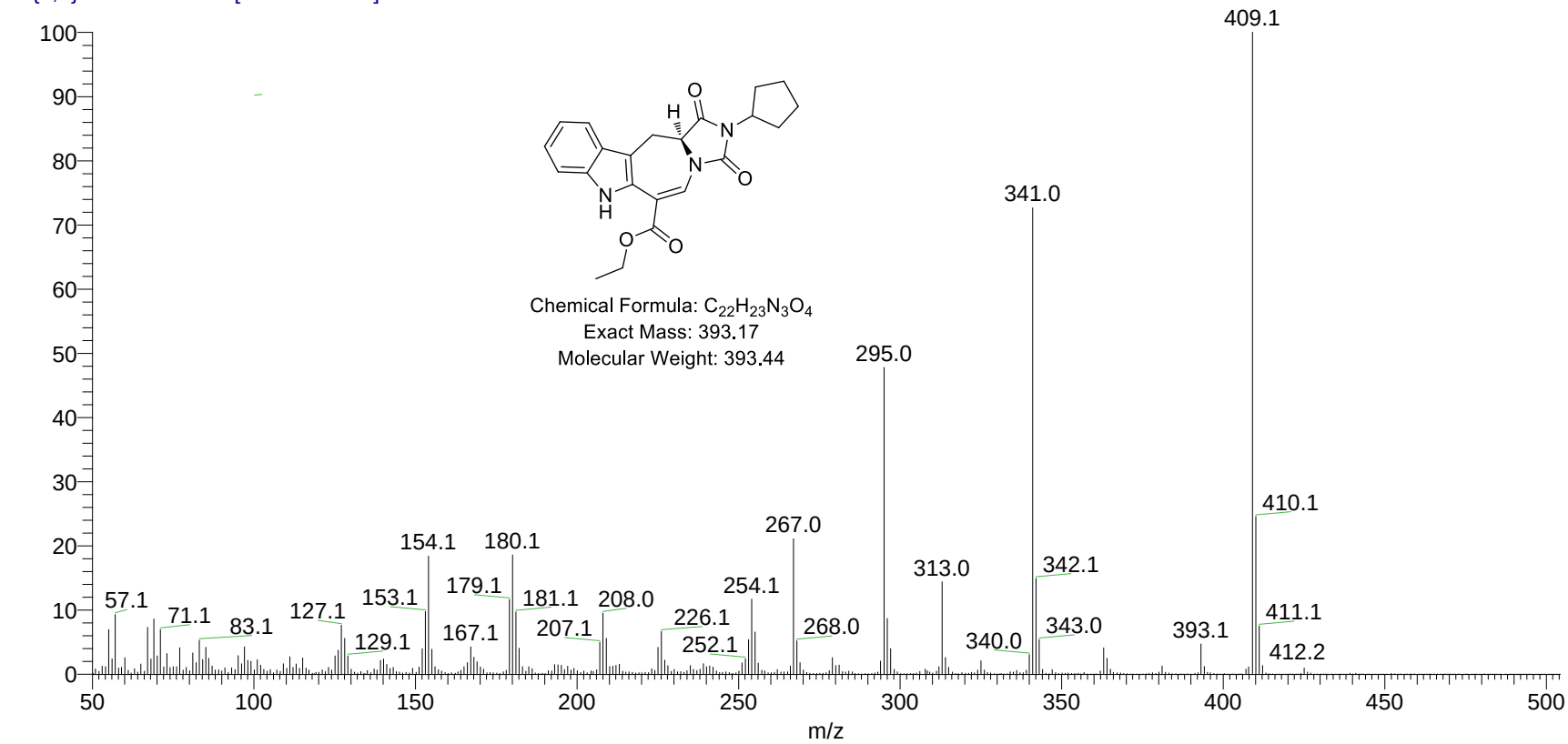
HPLC of compound **7i**



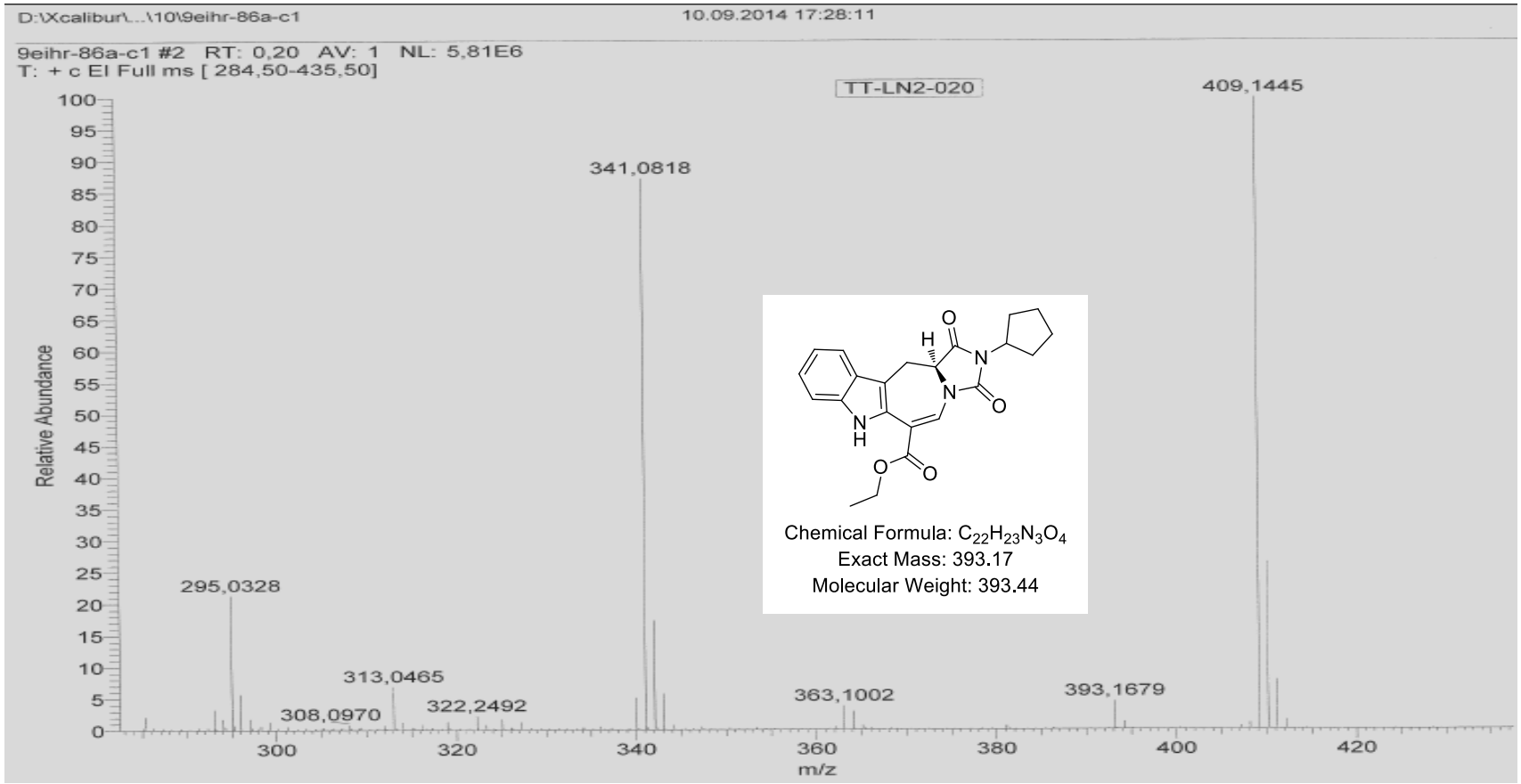
^{1}H NMR spectrum (400 MHz) of compound **7j** in acetone-d_6



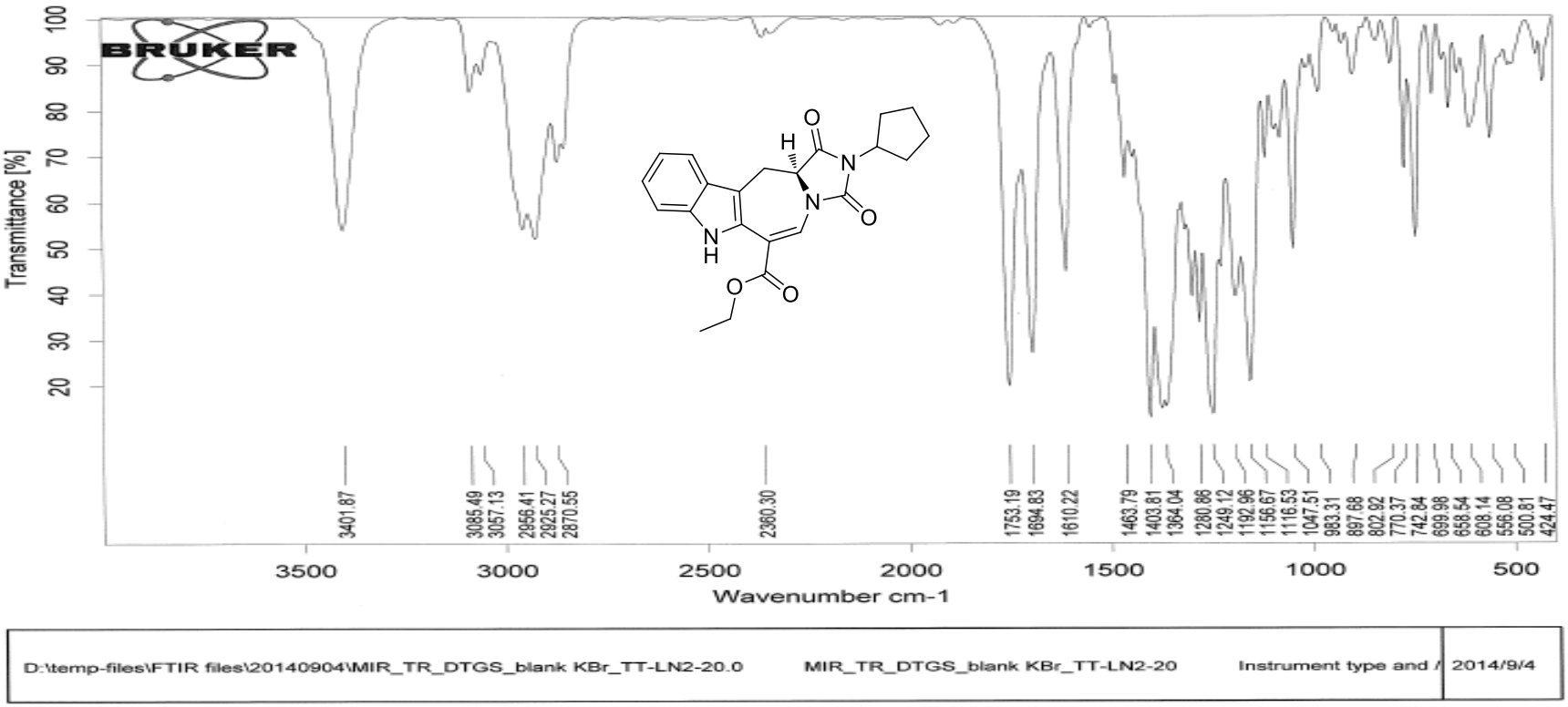
^{13}C NMR spectrum (101 MHz) of compound **7j** in acetone-d_6



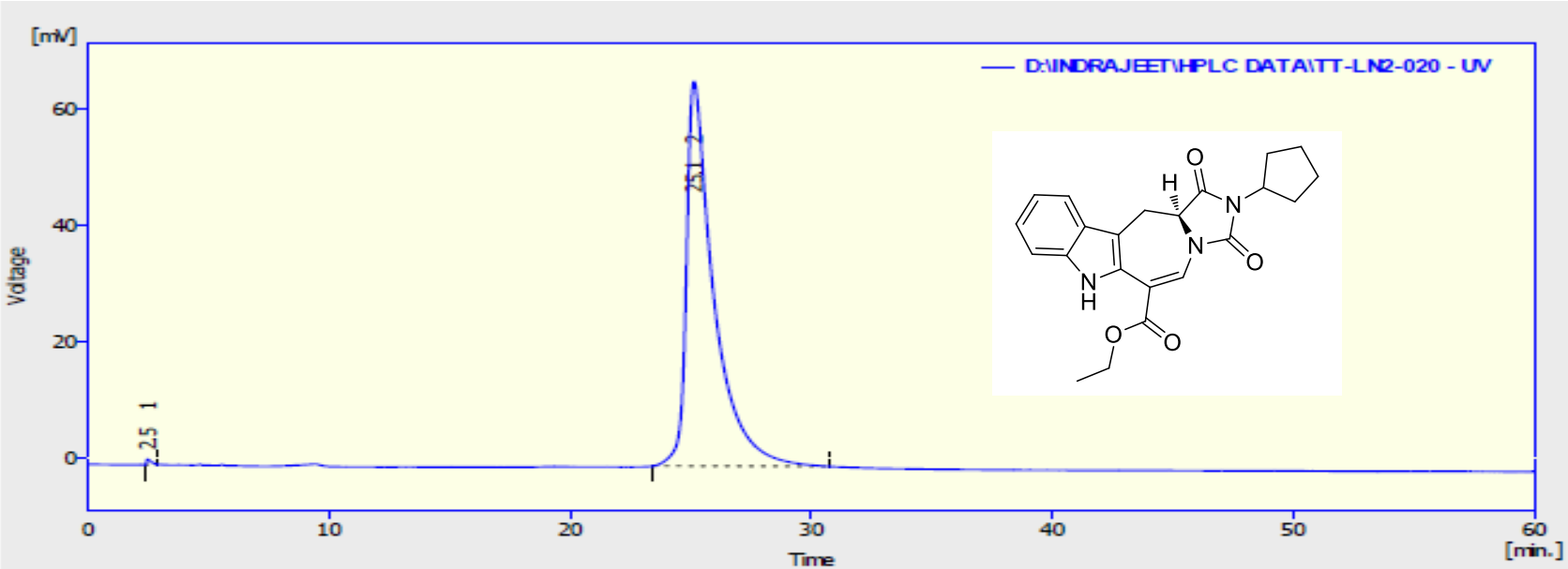
EI-LRMS of compound **7j**



EI-HRMS of compound **7j**



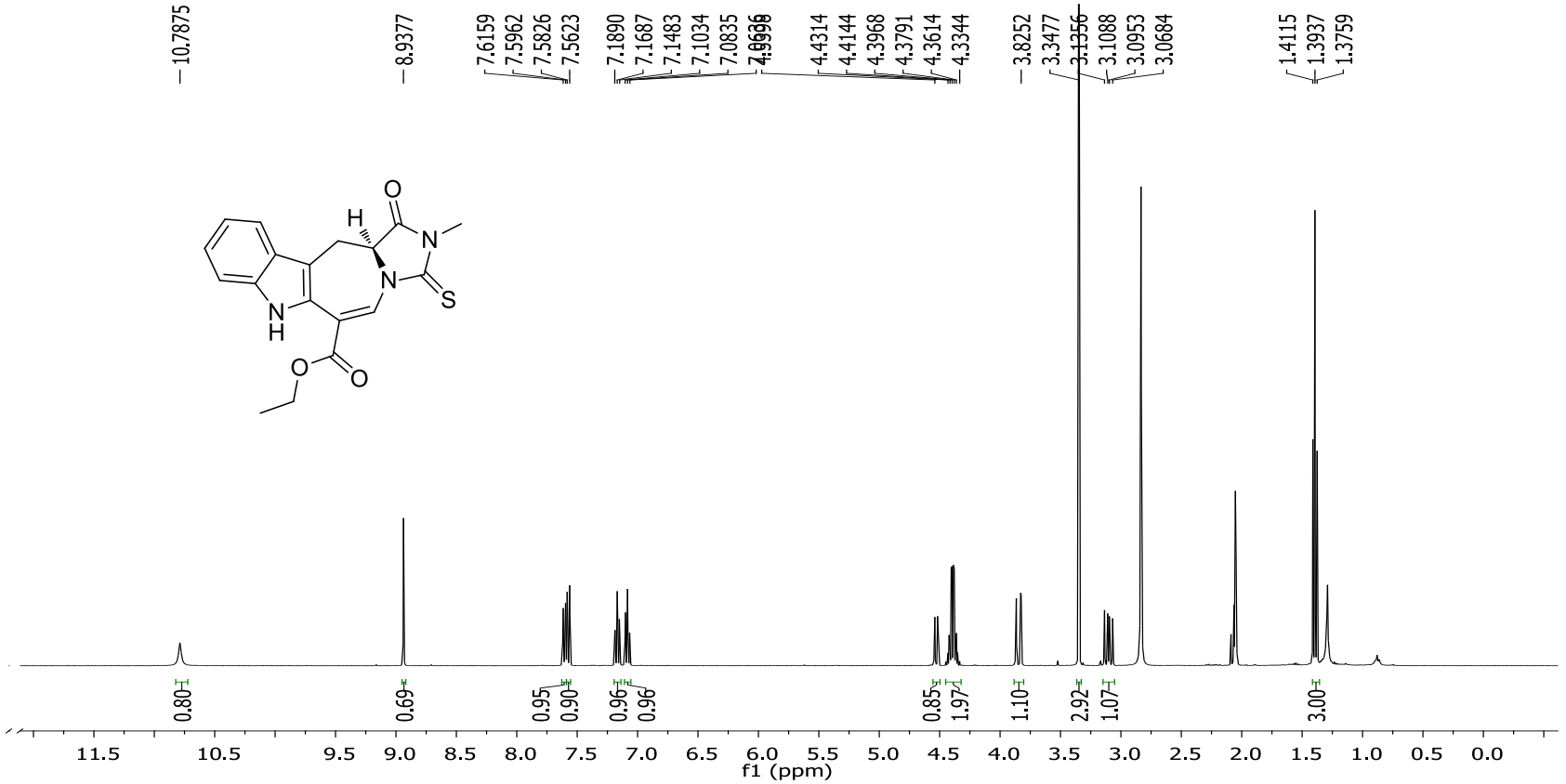
IR spectrum of compound **7j**



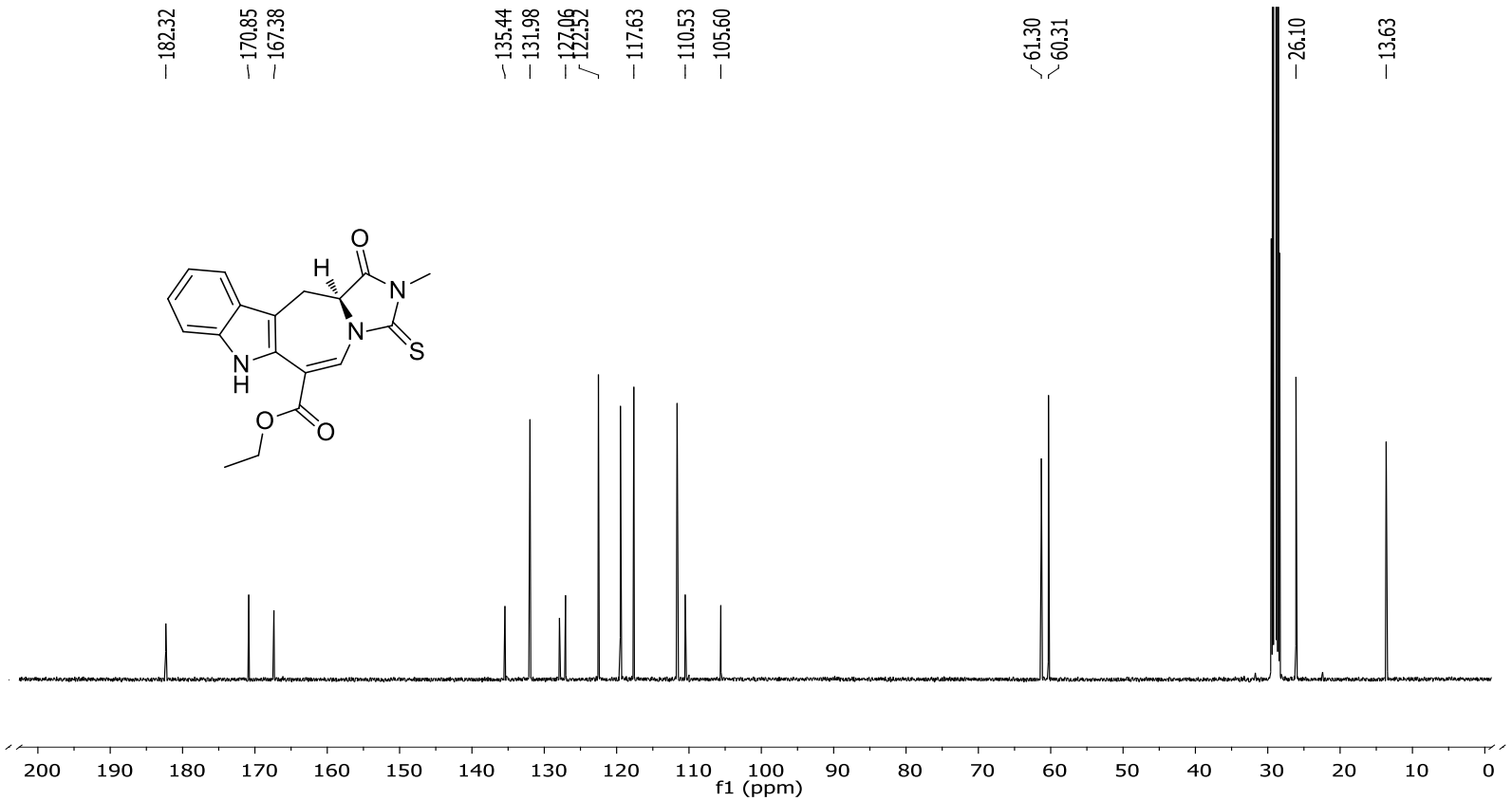
Result Table (Uncal - D:\INDRAJEET\HPLC DATA\TT-LN2-020 - UV)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
1	2.468	13.284	0.969	0.3	1.4
2	25.144	5215.595	66.060	99.7	98.6
	Total	5228.879	67.029	100.0	100.0

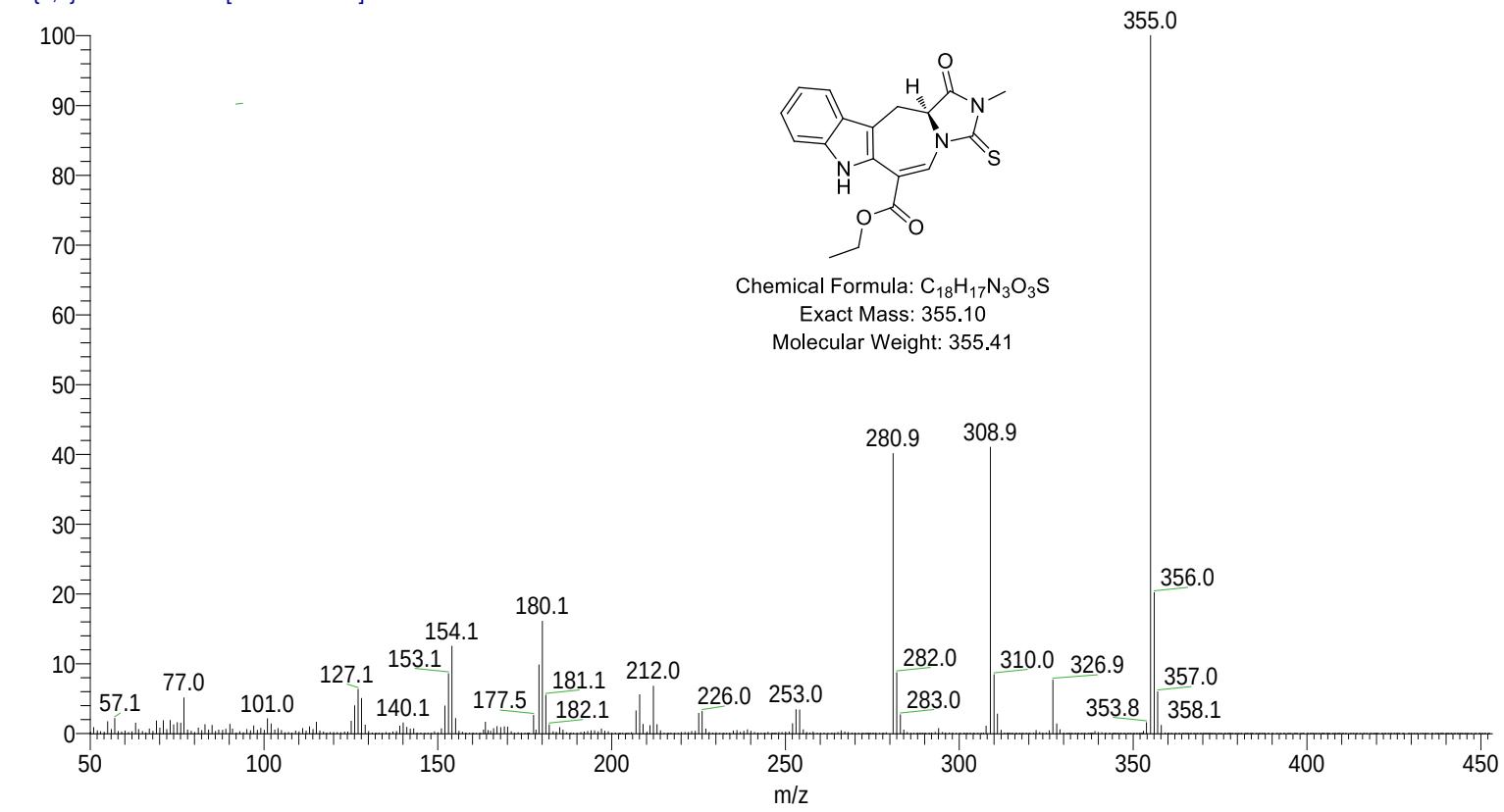
HPLC of compound **7j**



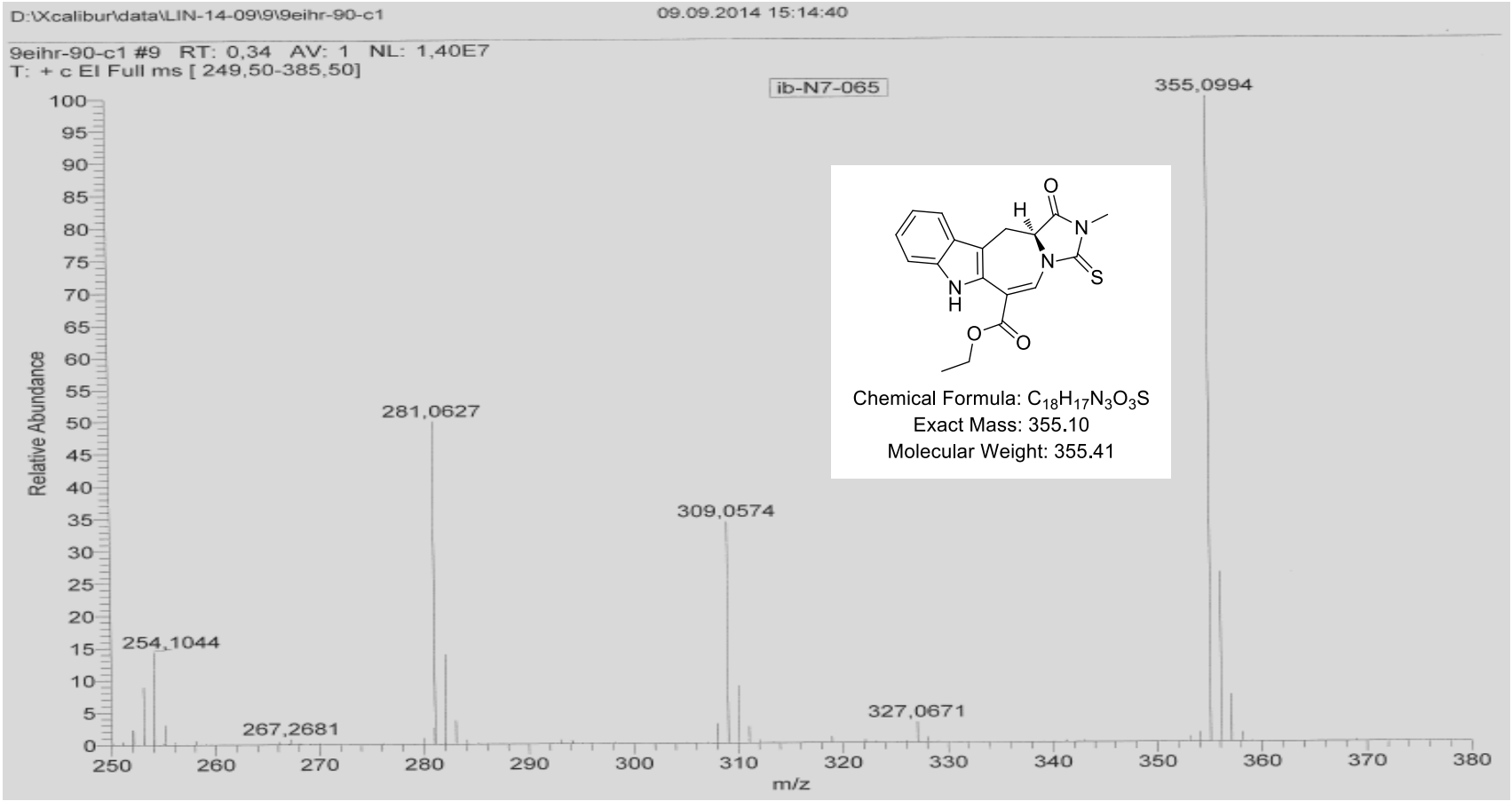
^{1}H NMR spectrum (400 MHz) of compound **7k** in acetone-d_6



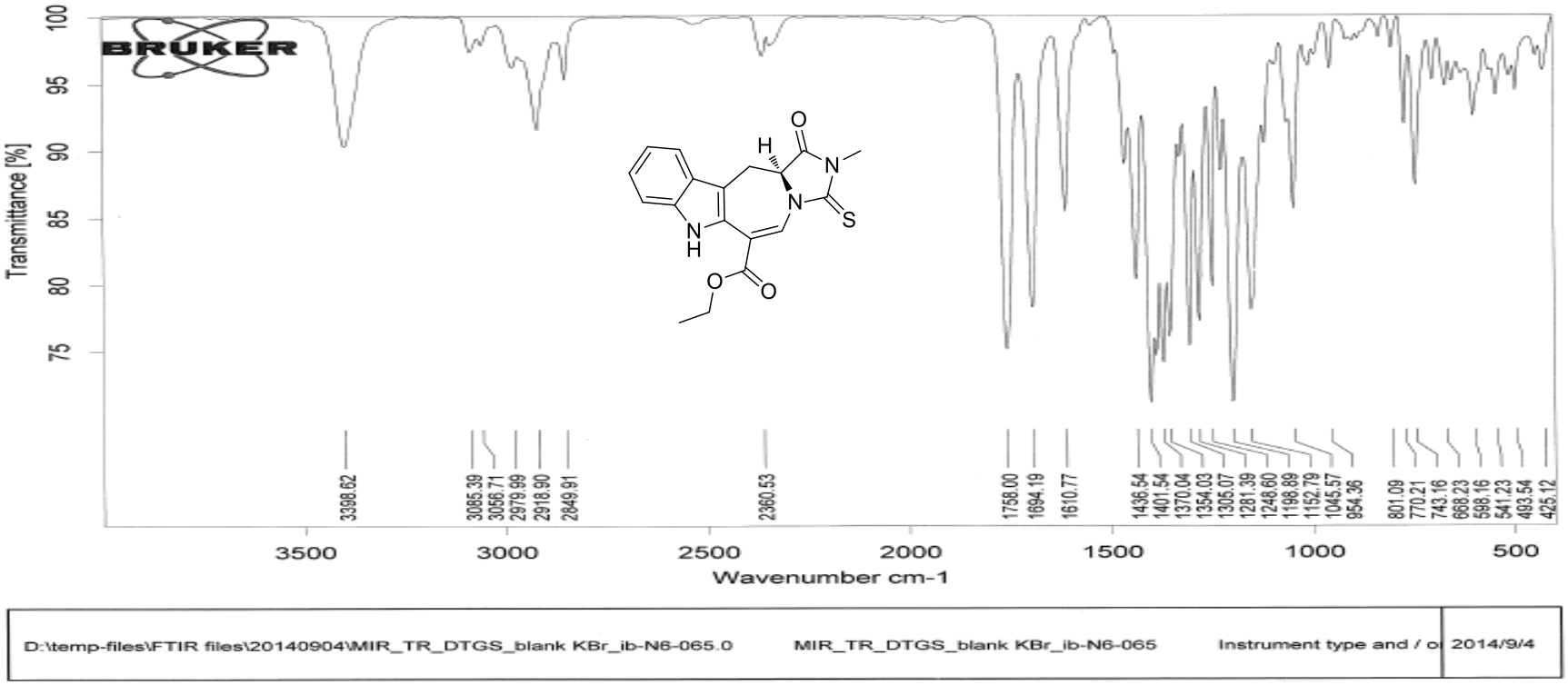
^{13}C NMR spectrum (101 MHz) of compound **7k** in acetone-d_6



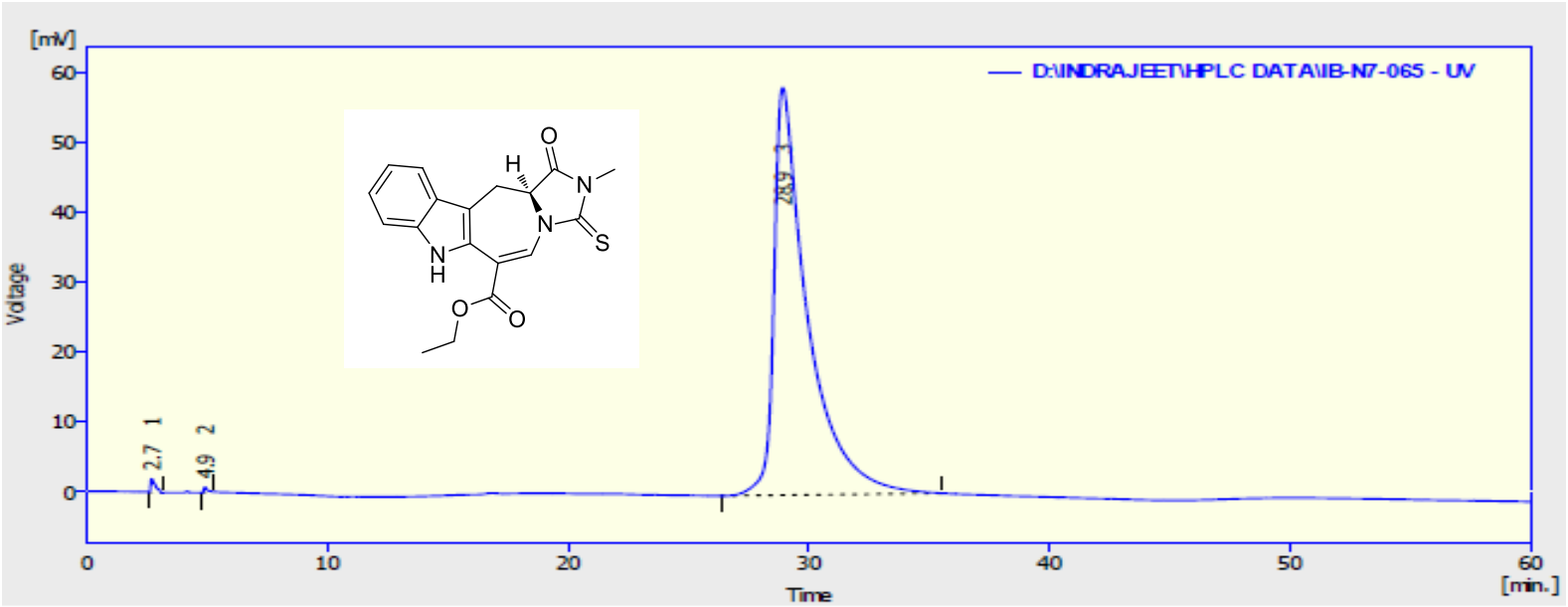
EI-LRMS of compound **7k**



EI-HRMS of compound **7k**



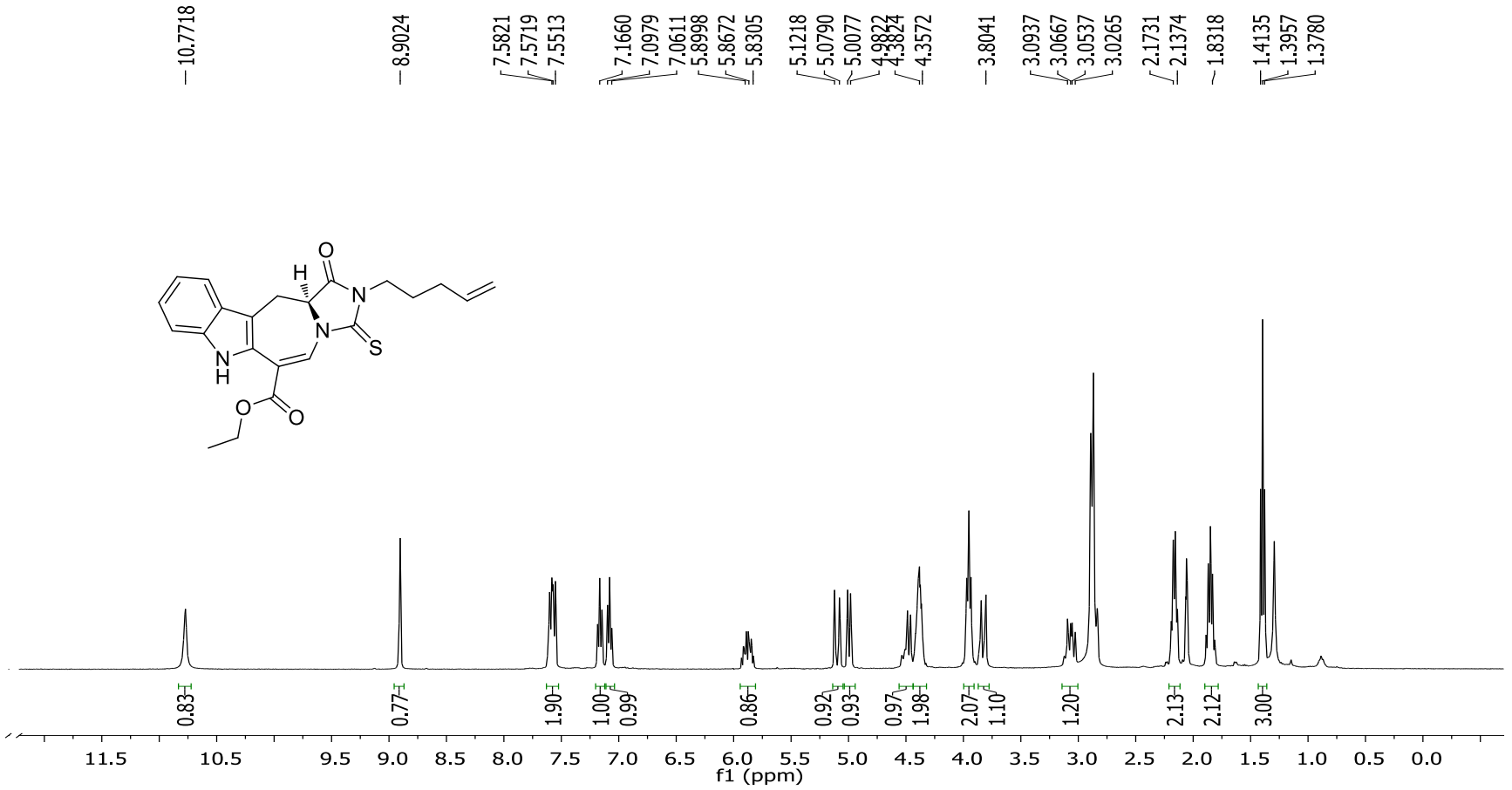
IR spectrum of compound **7k**



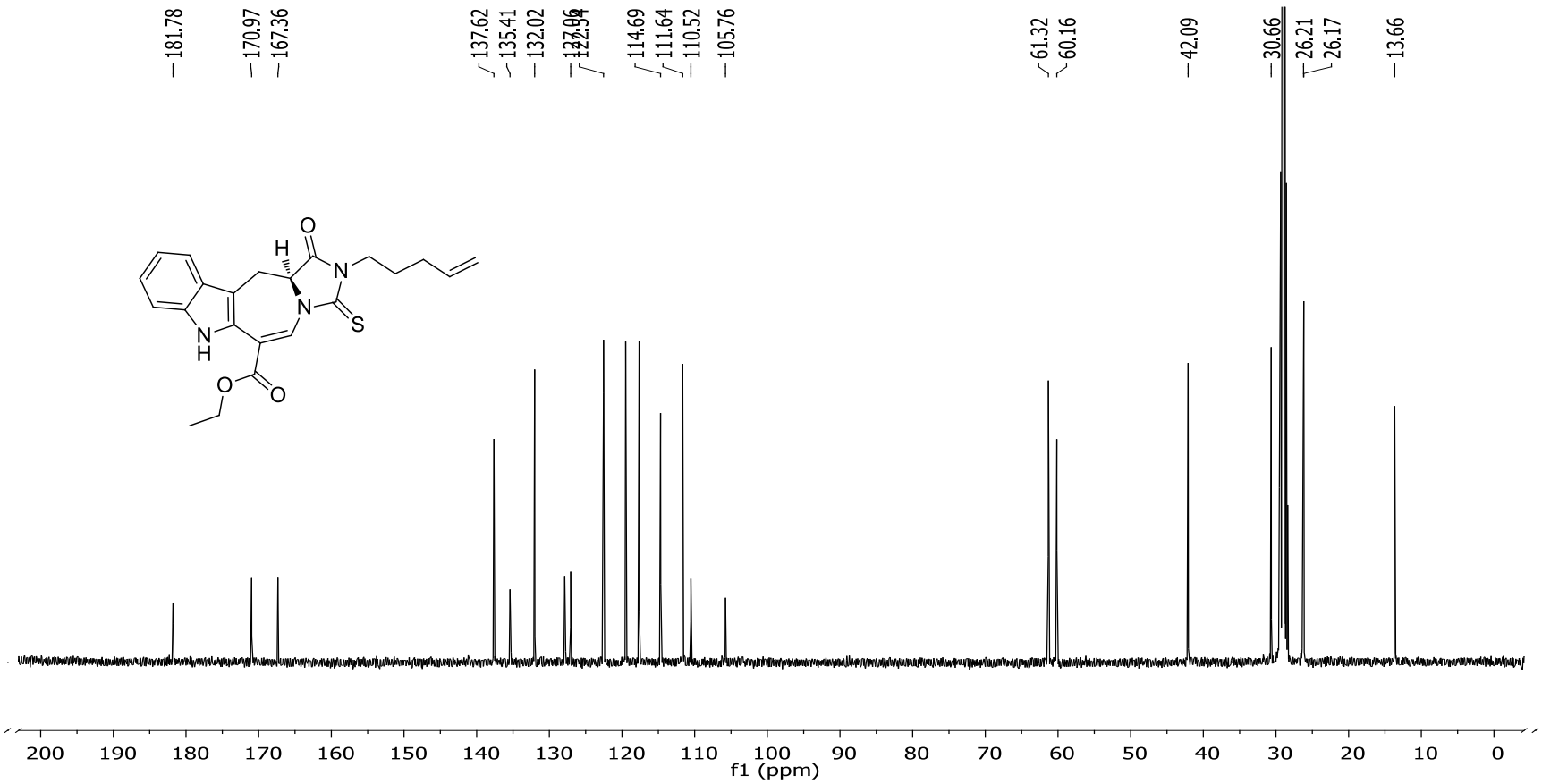
Result Table (Uncal - D:\INDRAJEET\HPLC DATA\IB-N7-065 - UV)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
1	2.692	25.128	1.873	0.4	3.1
2	4.904	7.371	0.759	0.1	1.2
3	28.924	5657.051	58.251	99.4	95.7
	Total	5689.551	60.883	100.0	100.0

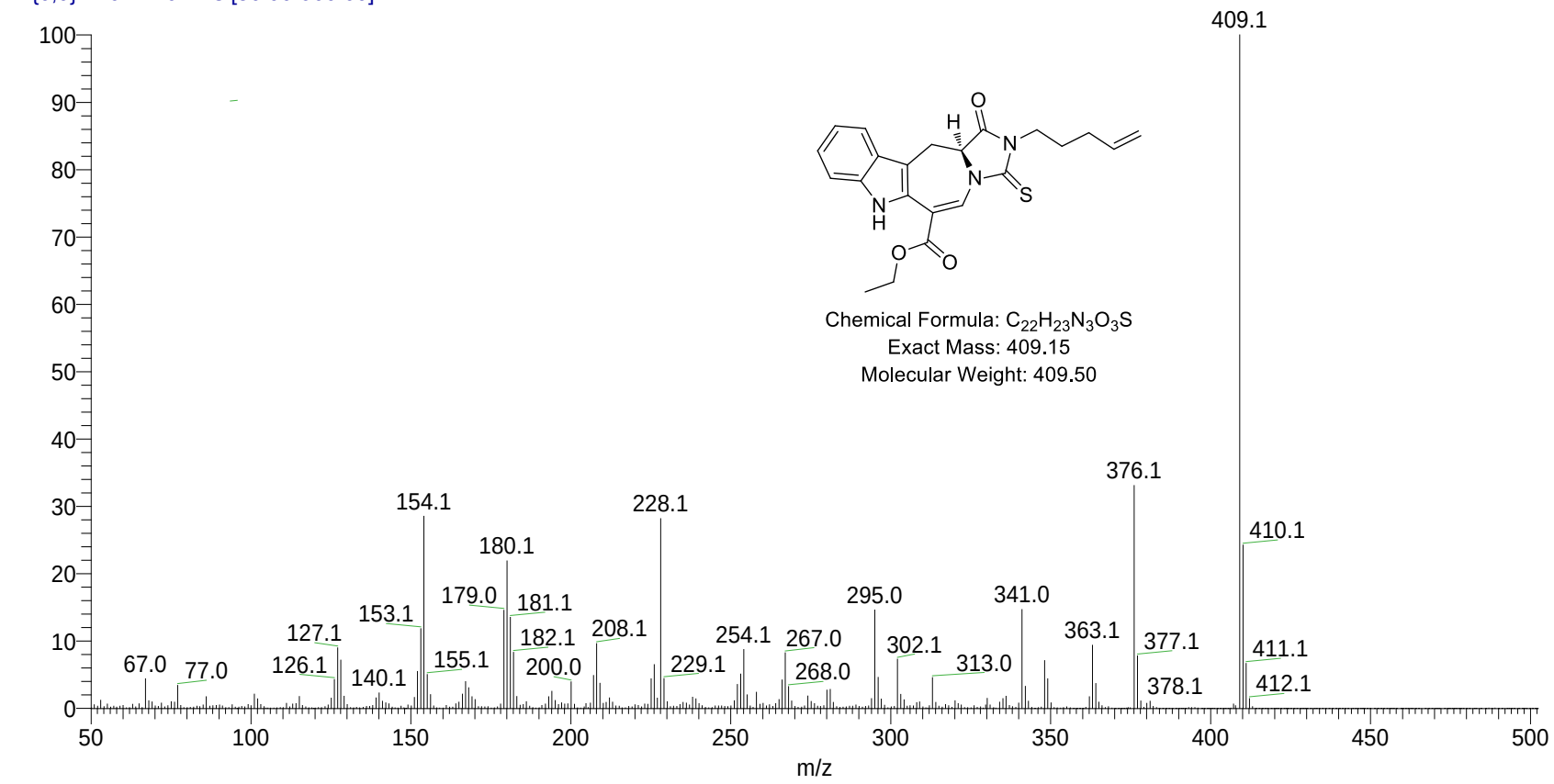
HPLC of compound **7k**



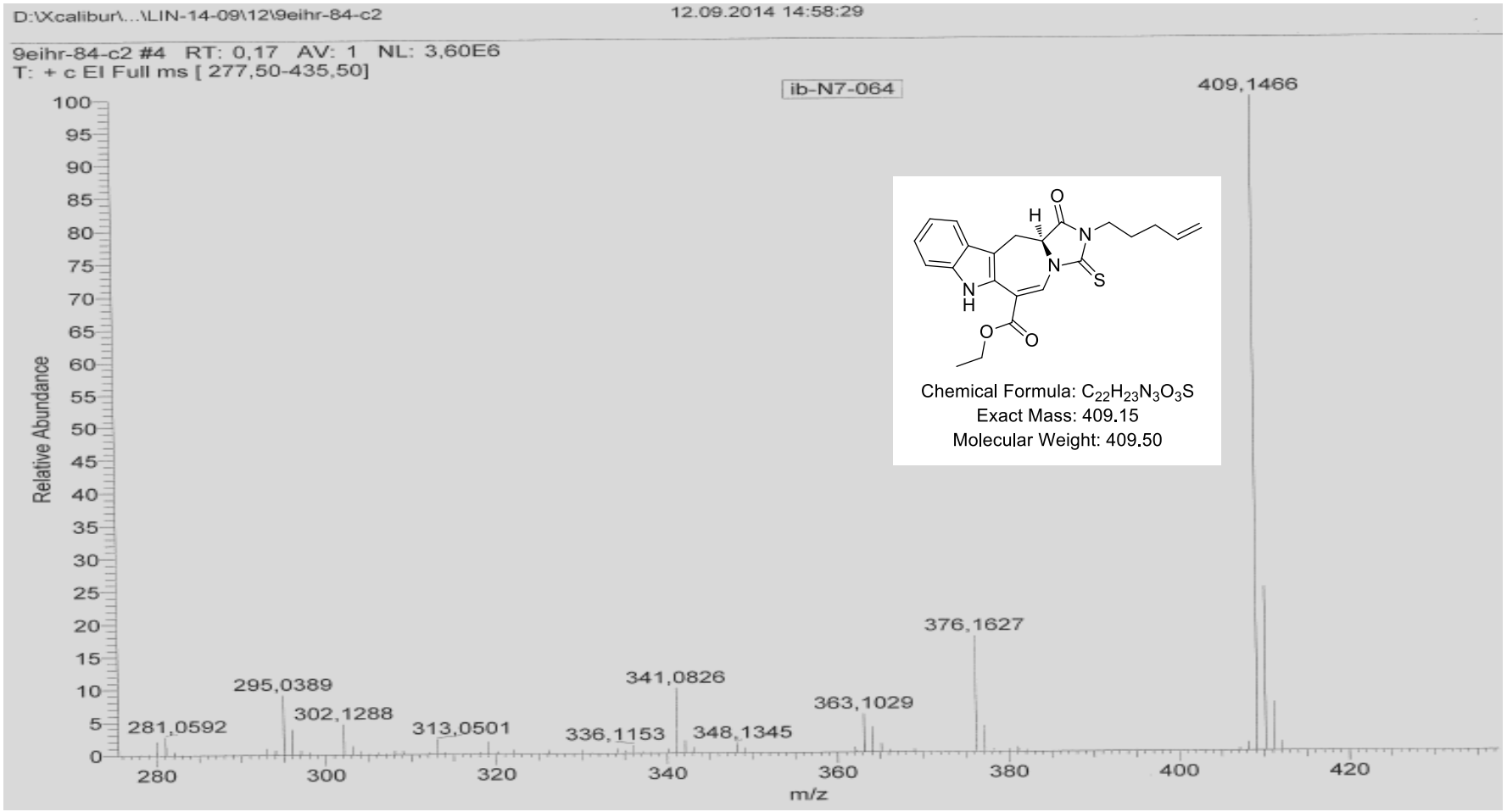
^{1}H NMR spectrum (400 MHz) of compound **7l** in acetone-d_6



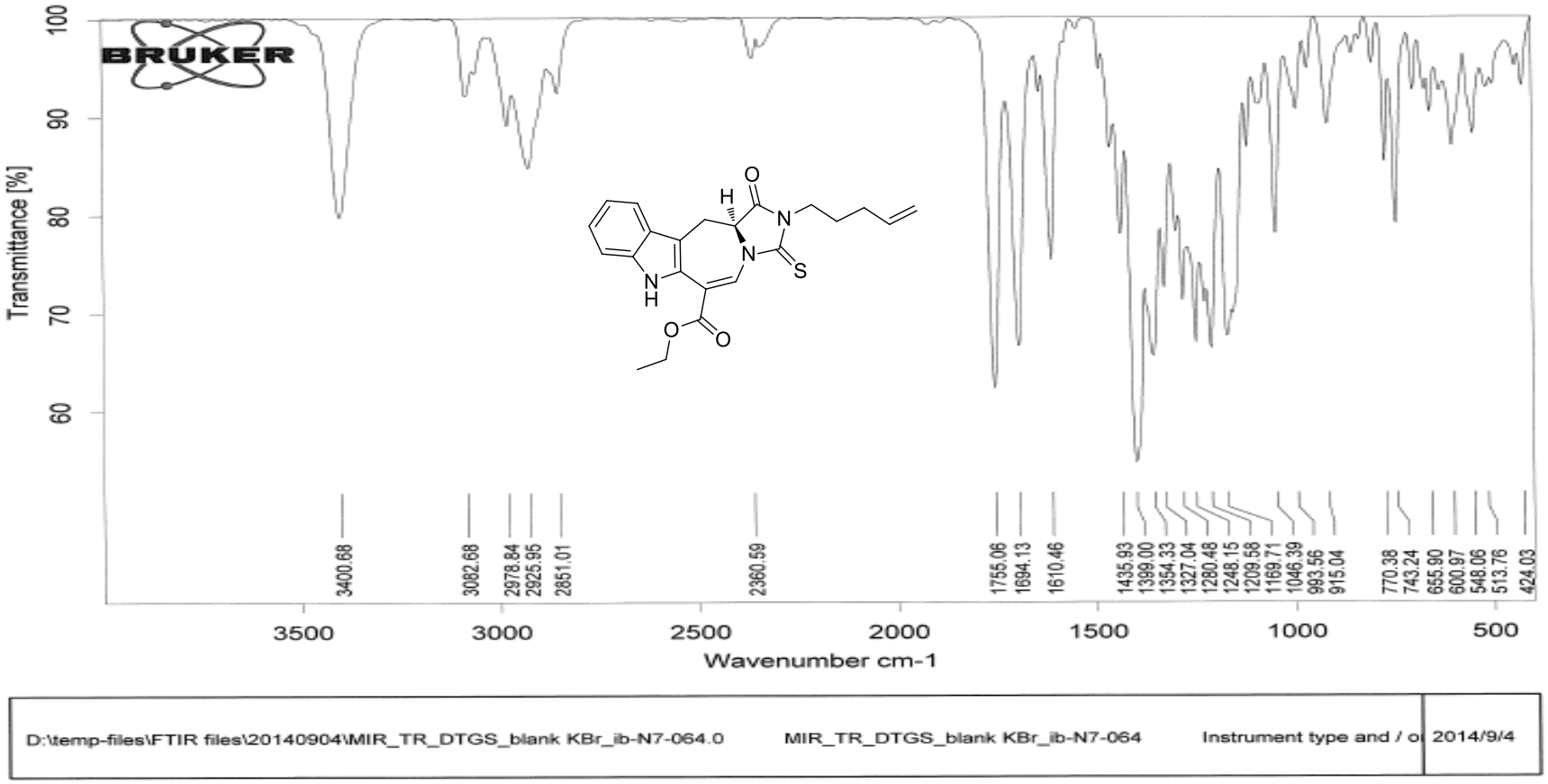
^{13}C NMR spectrum (101 MHz) of compound **7l** in acetone-d_6



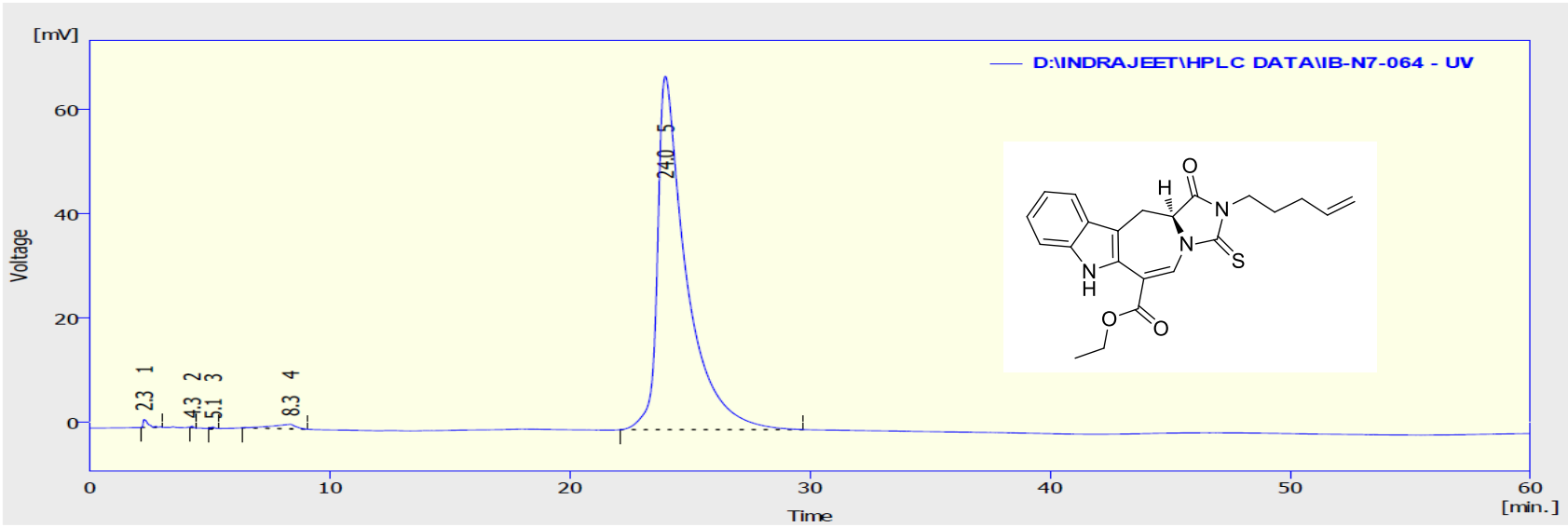
EI-LRMS of compound **7l**



EI-HRMS of compound **7l**



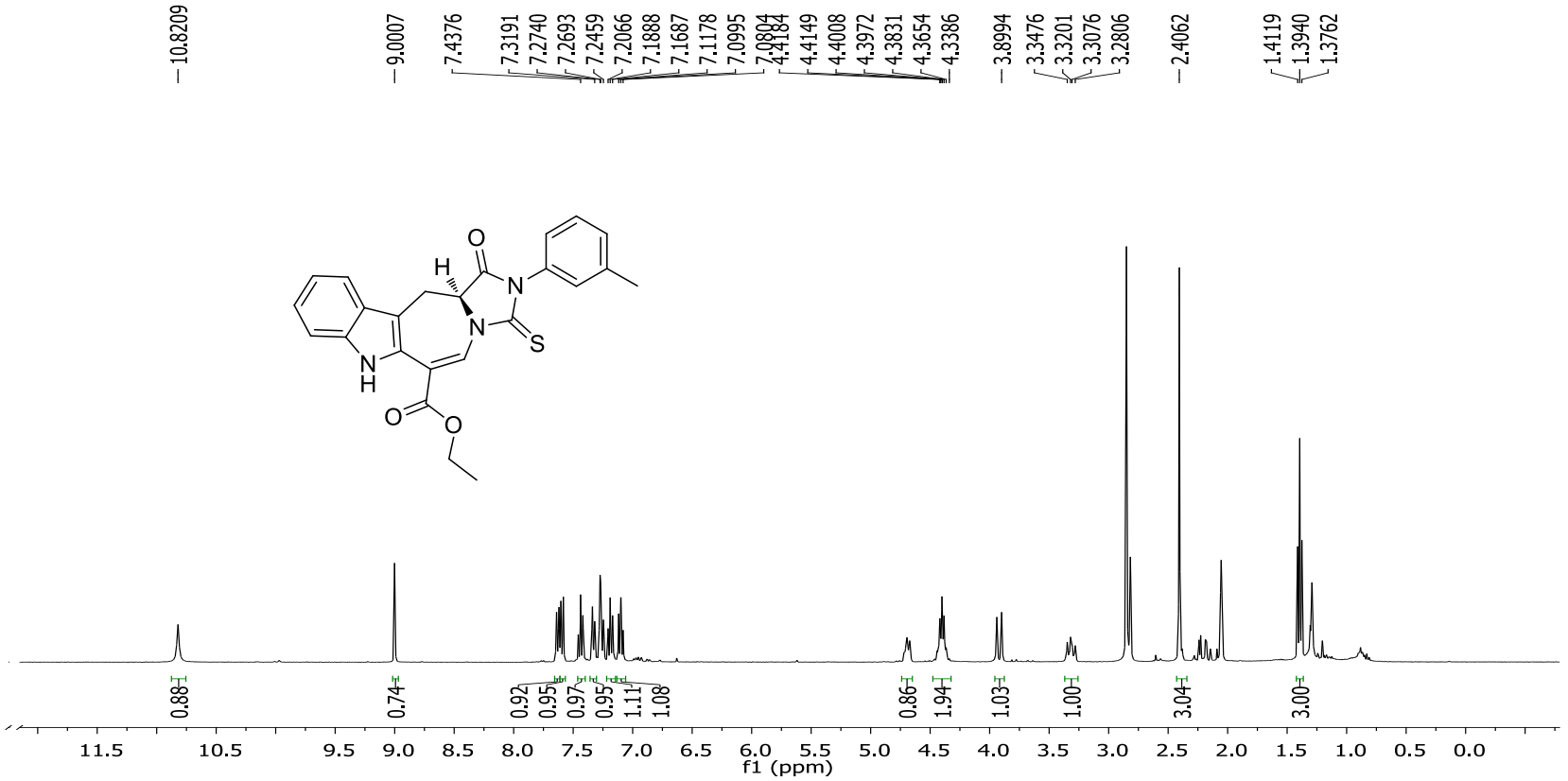
IR spectrum of compound **7l**



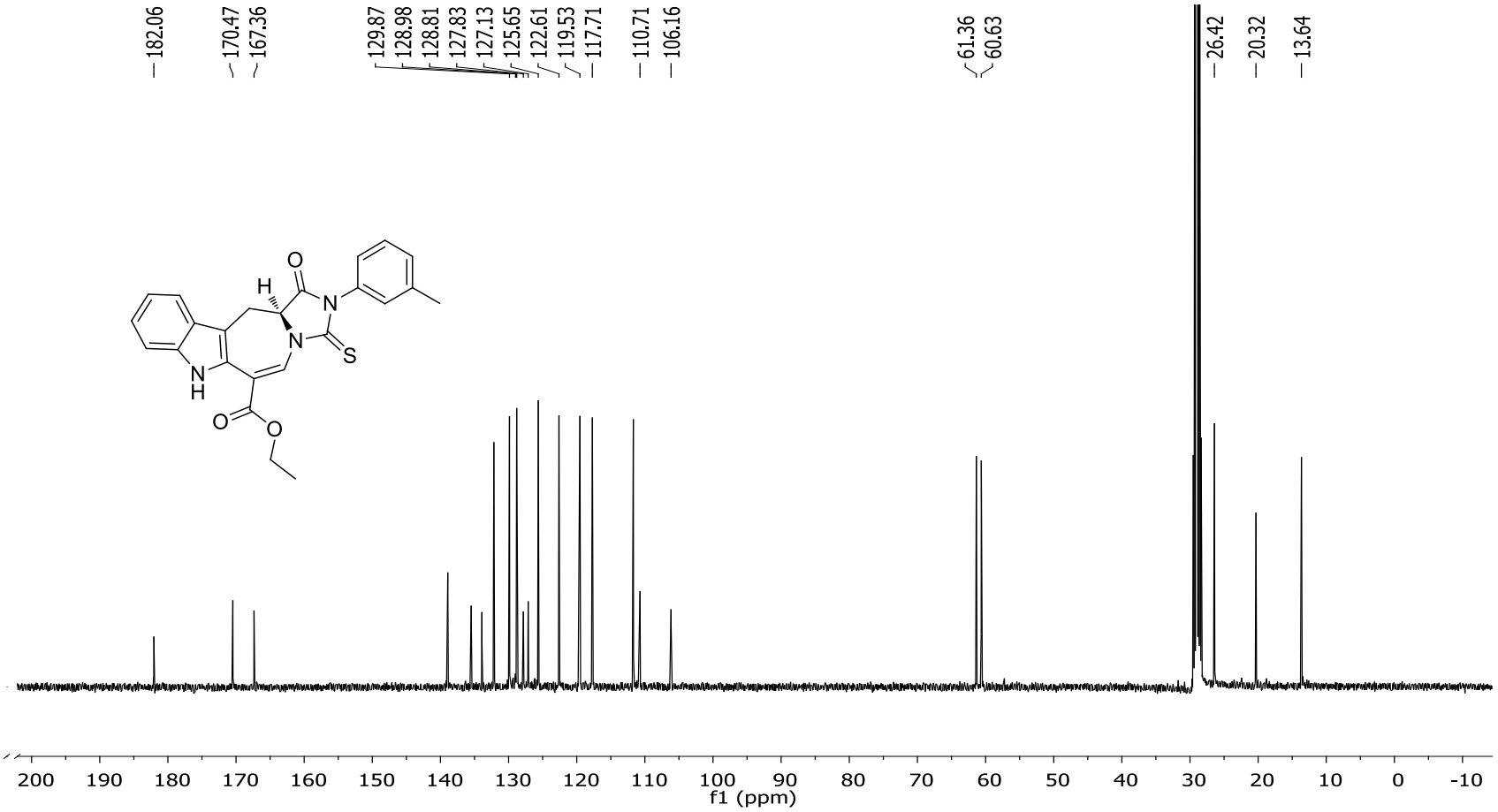
Result Table (Uncal - D:\INDRAJEET\HPLC DATA\IB-N7-064 - UV)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
1	2.260	22.299	1.548	0.4	2.2
2	4.252	1.592	0.297	0.0	0.4
3	5.120	2.388	0.283	0.0	0.4
4	8.344	61.171	0.890	1.1	1.3
5	23.968	5639.881	67.852	98.5	95.7
	Total	5727.331	70.870	100.0	100.0

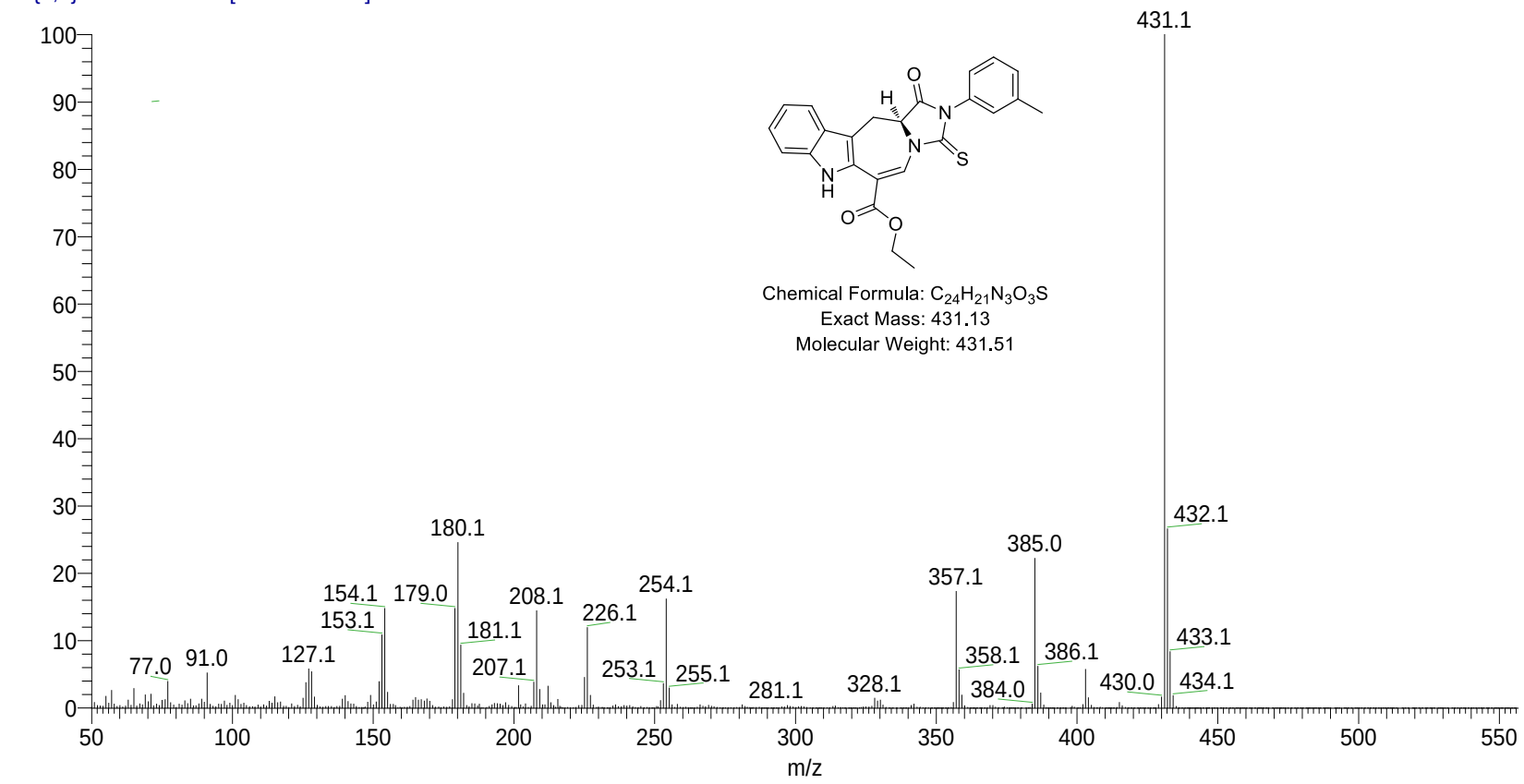
HPLC of compound **7l**



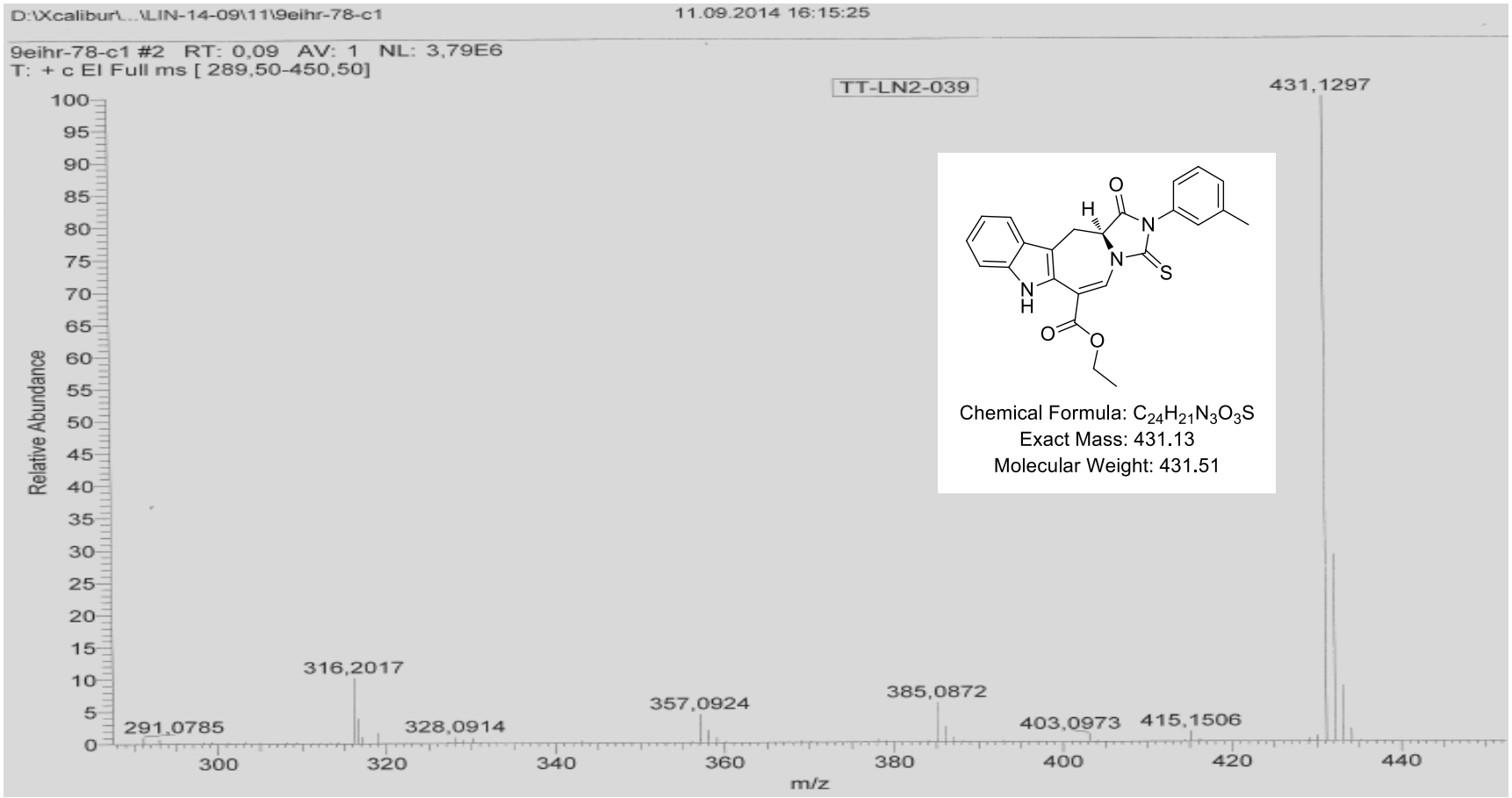
^{1}H NMR spectrum (400 MHz) of compound **7m** in acetone-d_6



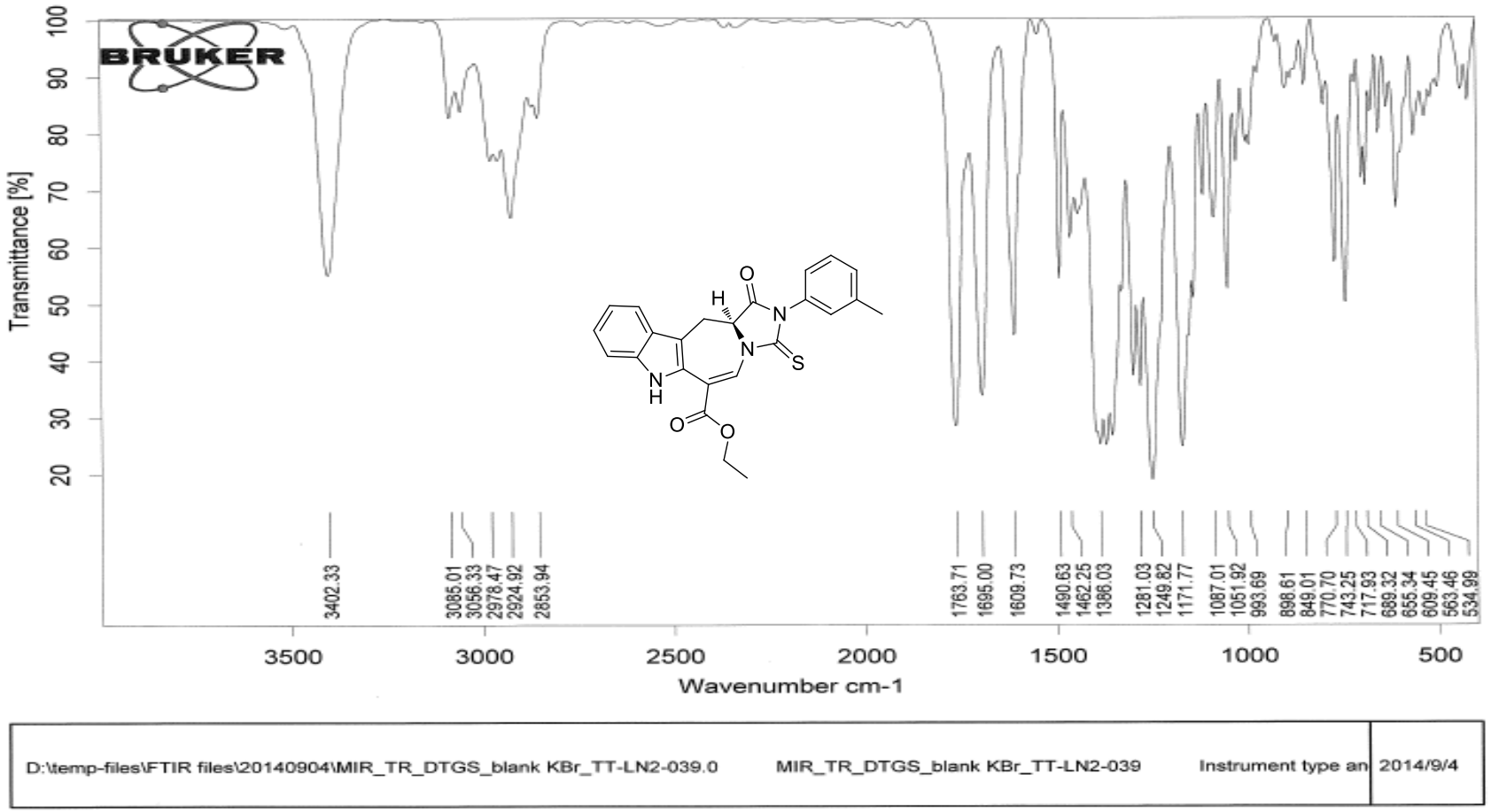
^{13}C NMR spectrum (101 MHz) of compound **7m** in acetone-d_6



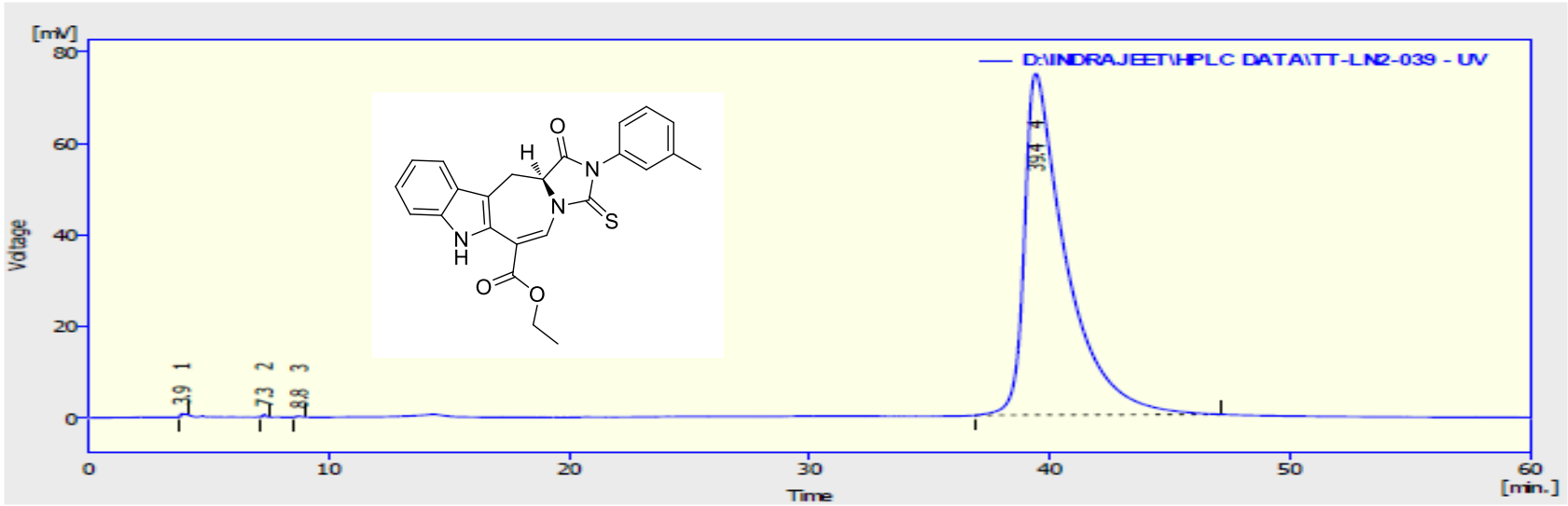
EI-LRMS of compound **7m**



EI-HRMS of compound **7m**



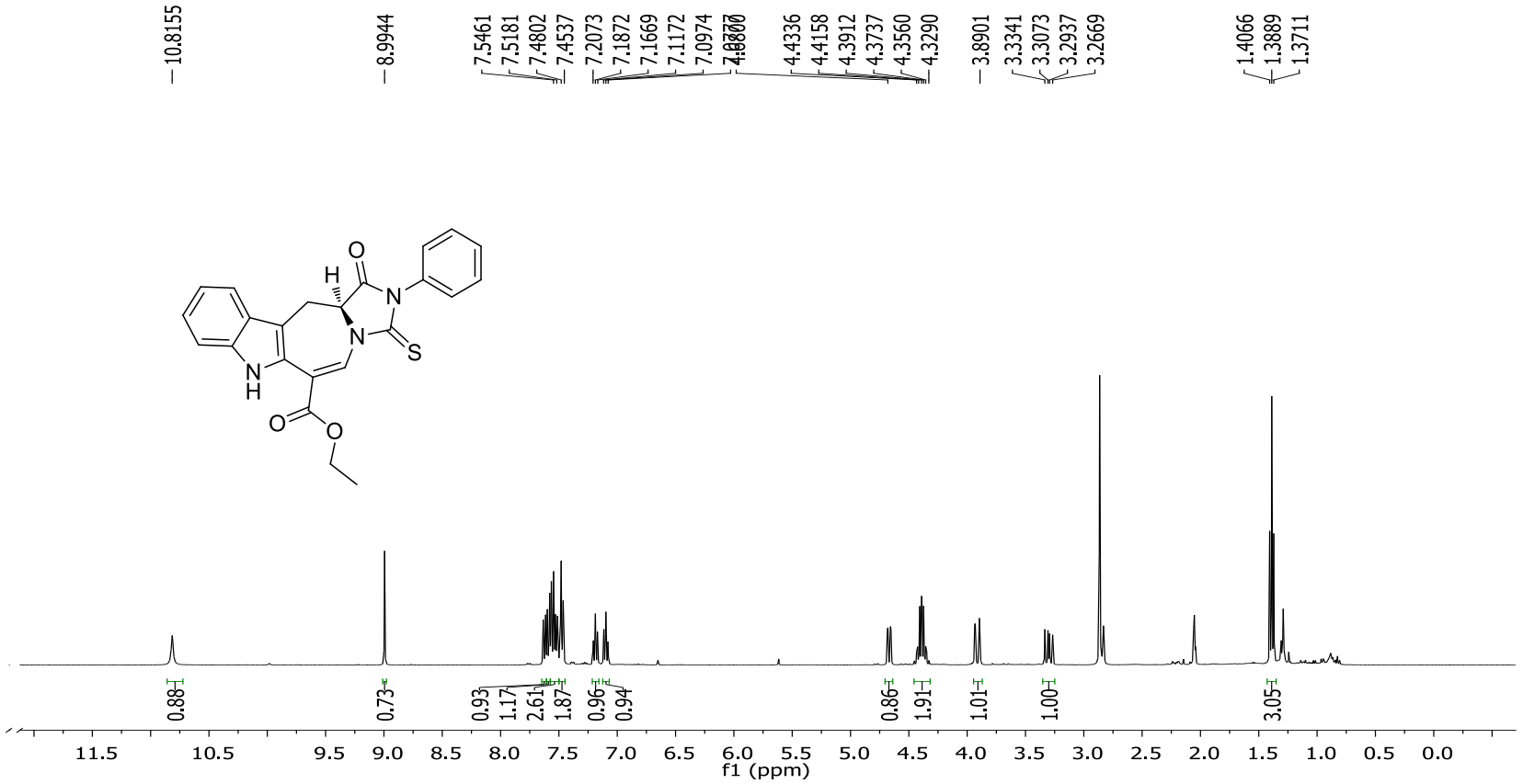
IR spectrum of compound **7m**



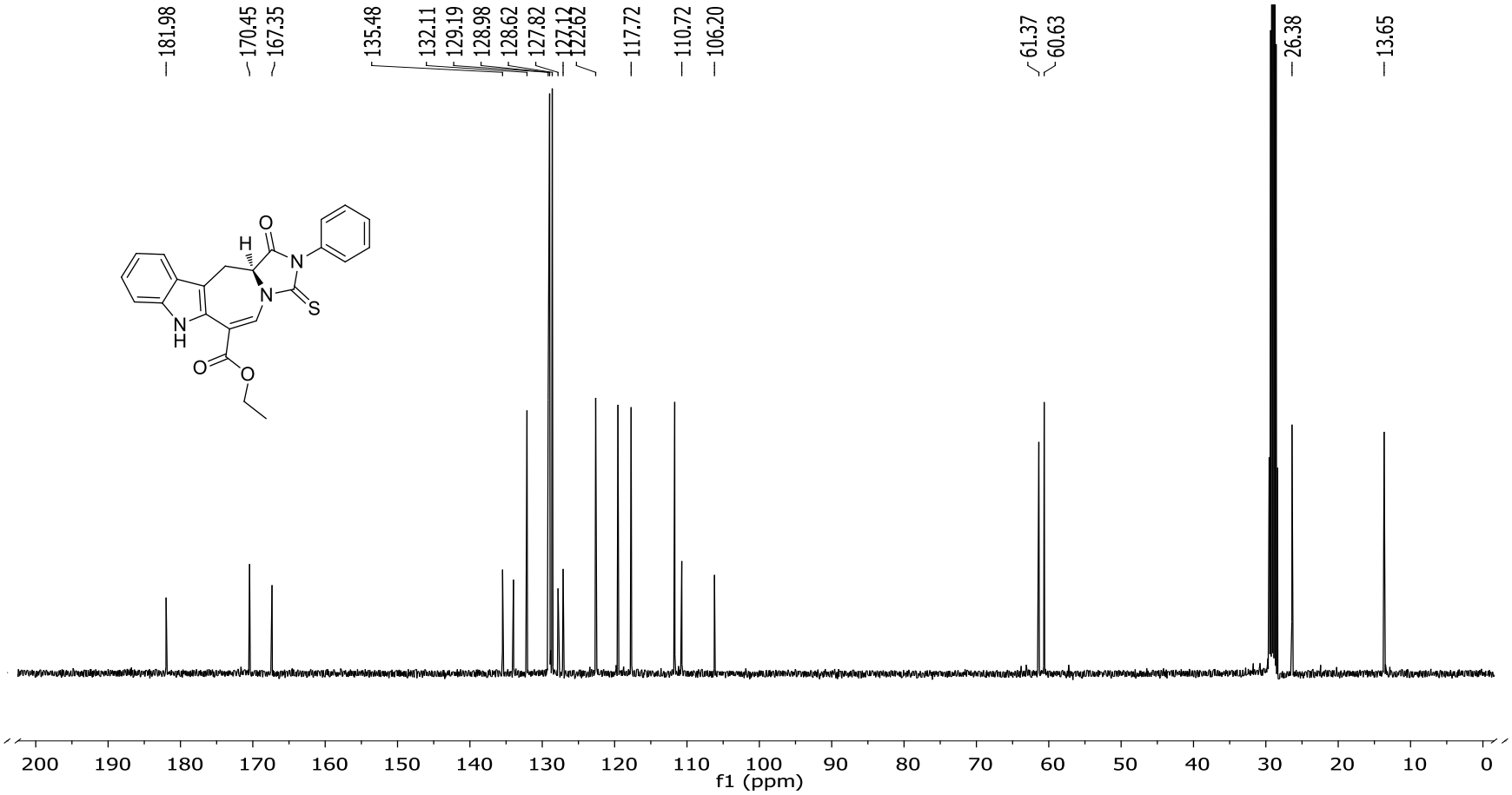
Result Table (Uncal - D:\INDRAJEET\HPLC DATA\TT-LN2-039 - UV)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
1	3.868	6.529	0.578	0.1	0.8
2	7.306	4.854	0.578	0.1	0.8
3	8.760	2.564	0.223	0.0	0.3
4	39.424	8987.213	74.588	99.8	98.2
	Total	9001.160	75.966	100.0	100.0

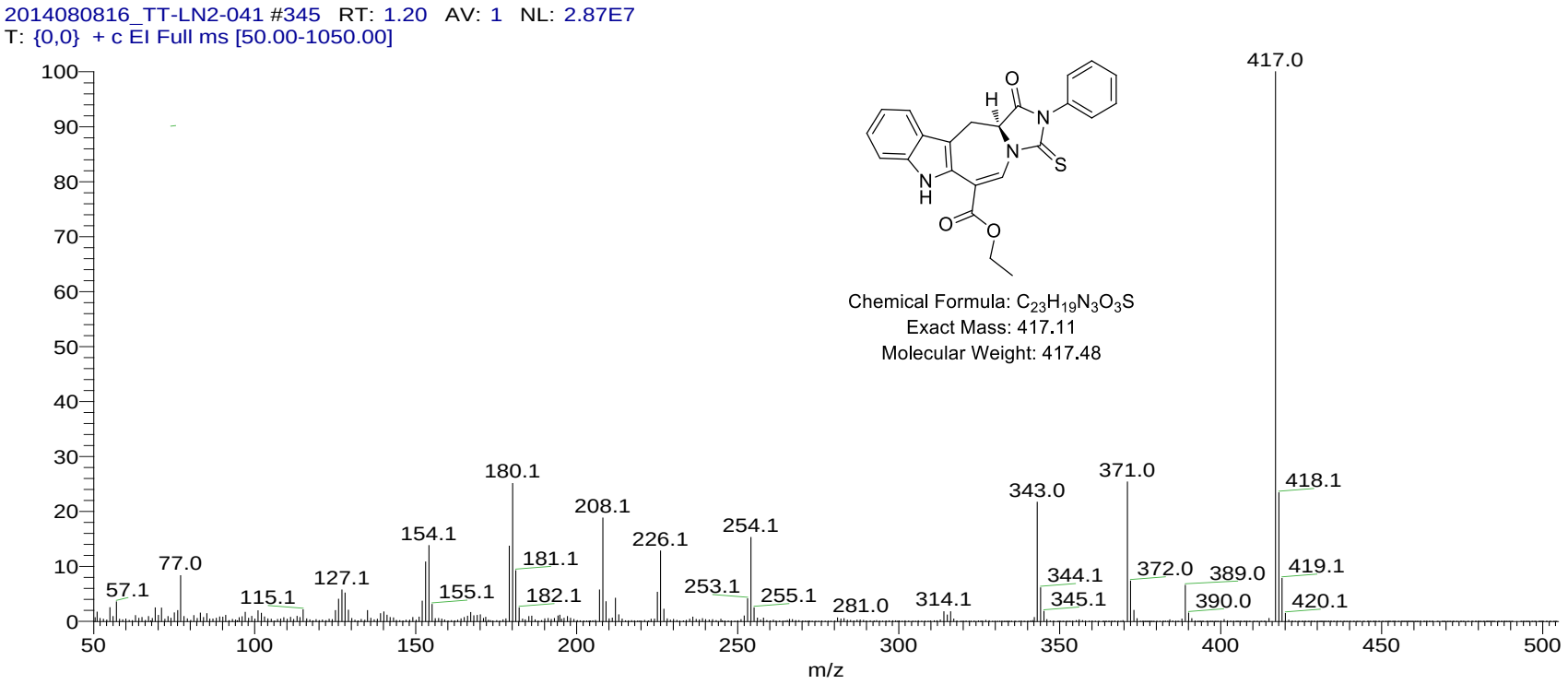
HPLC of compound **7m**



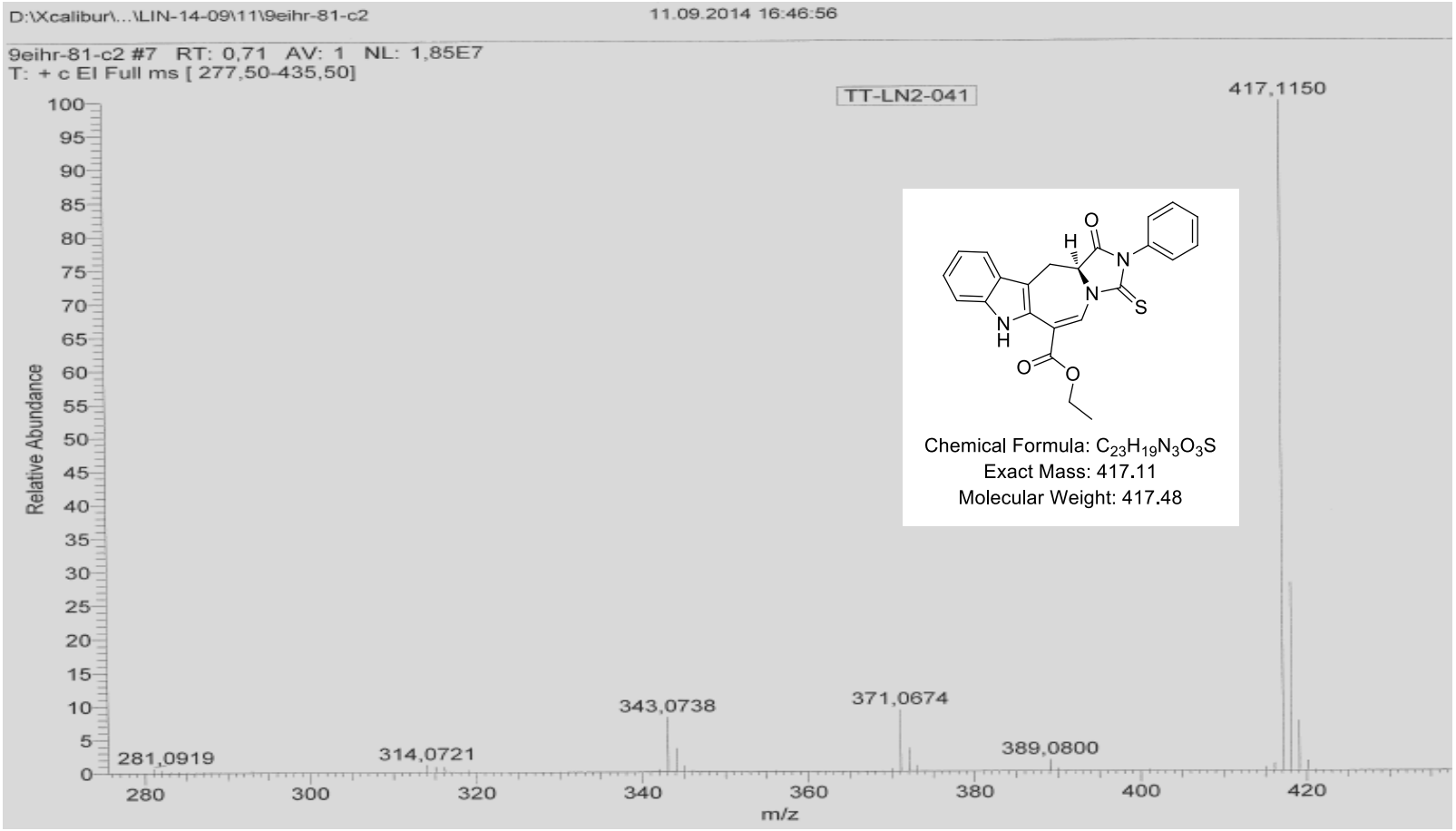
^{1}H NMR spectrum (400 MHz) of compound **7n** in acetone-d_6



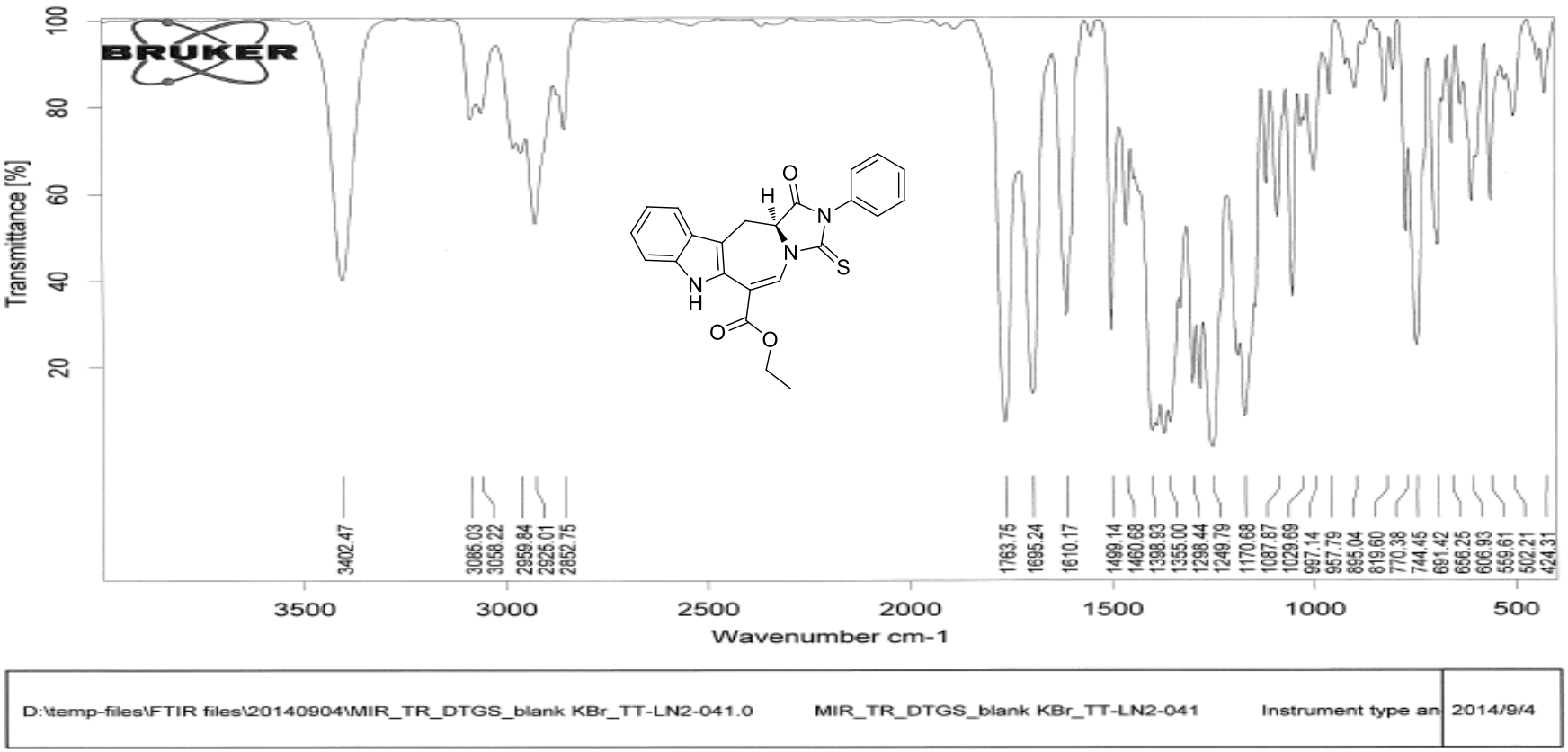
^{13}C NMR spectrum (101 MHz) of compound **7n** in acetone-d_6



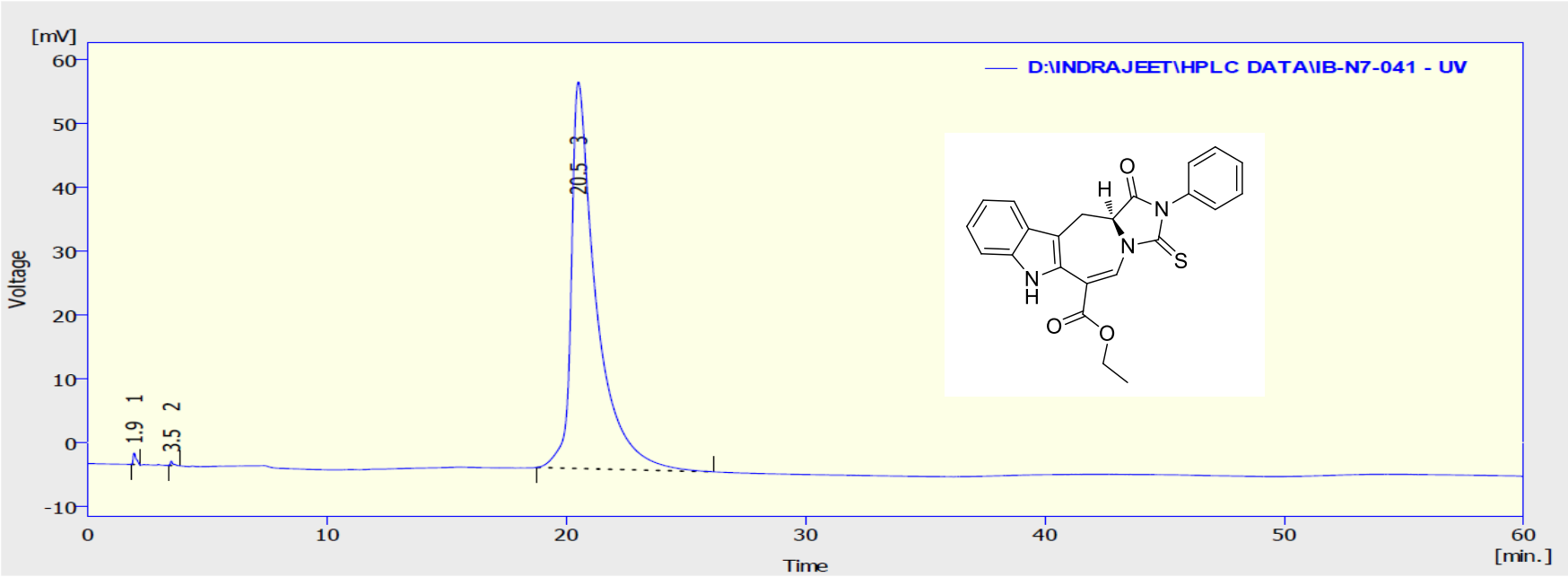
EI-LRMS of compound **7n**



EI-HRMS of compound **7n**



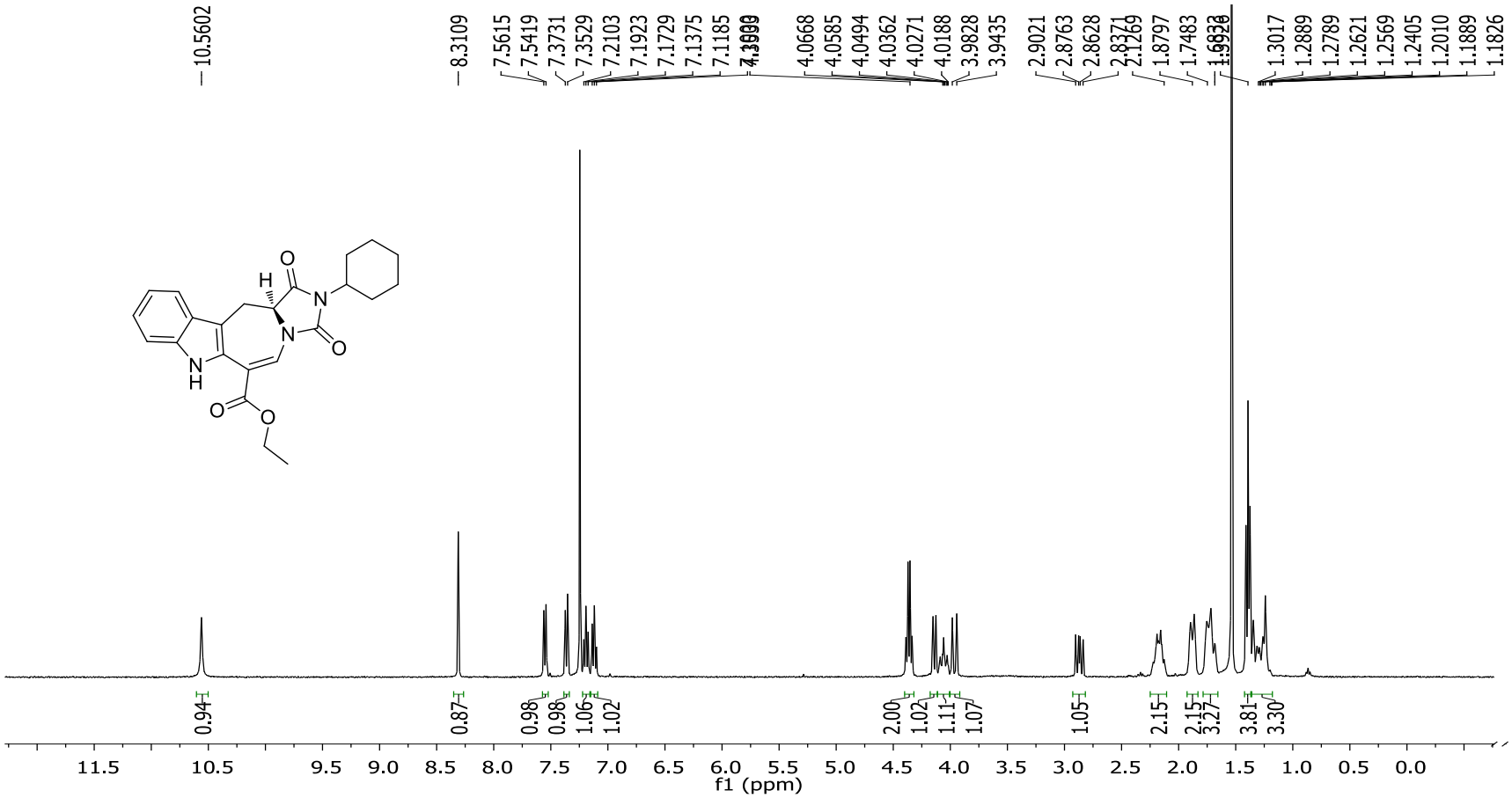
IR spectrum of compound **7n**



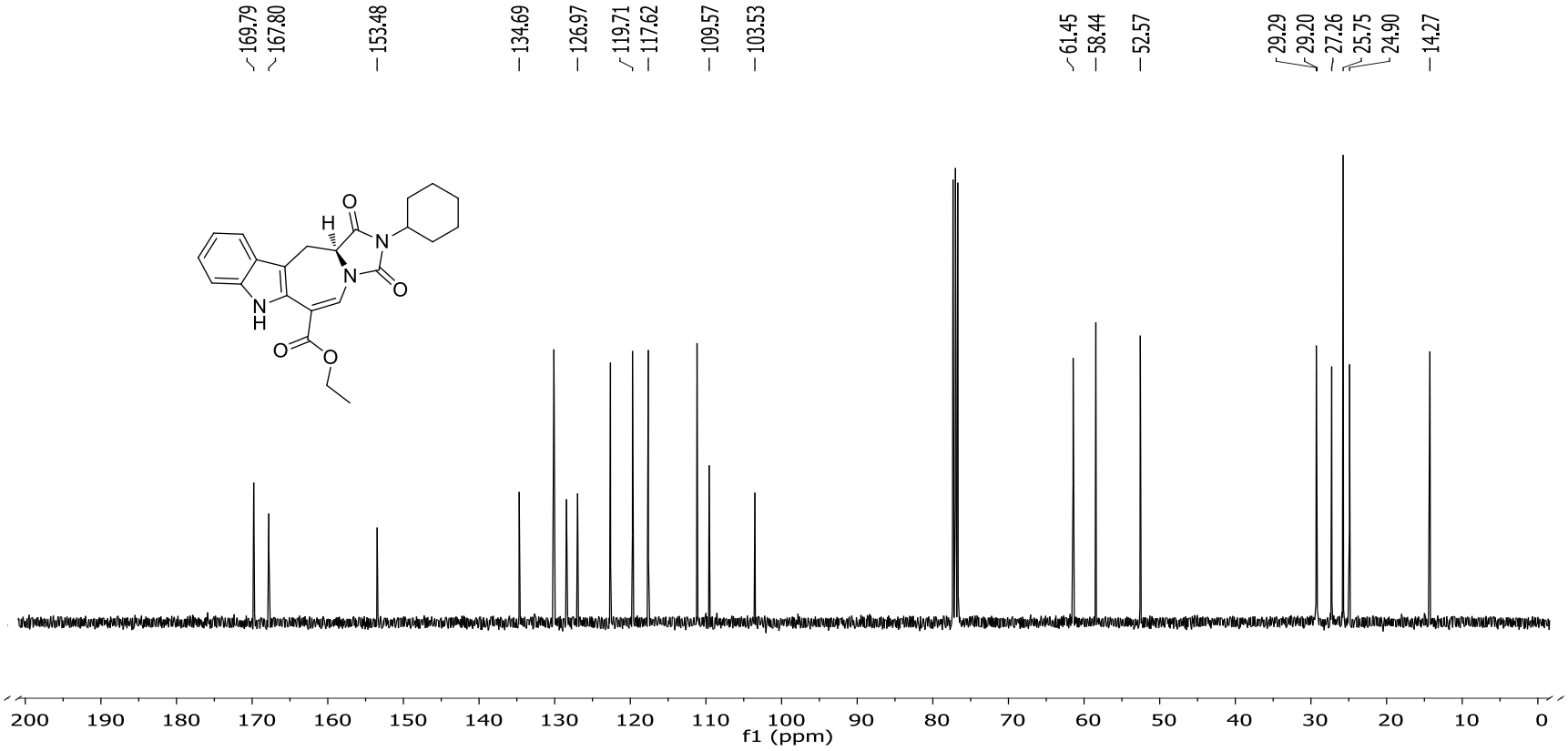
Result Table (Uncal - D:\INDRAJEET\HPLC DATA\IB-N7-041 - UV)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
1	1.932	16.755	1.822	0.4	2.9
2	3.484	5.681	0.709	0.1	1.1
3	20.500	4392.030	60.551	99.5	96.0
	Total	4414.466	63.082	100.0	100.0

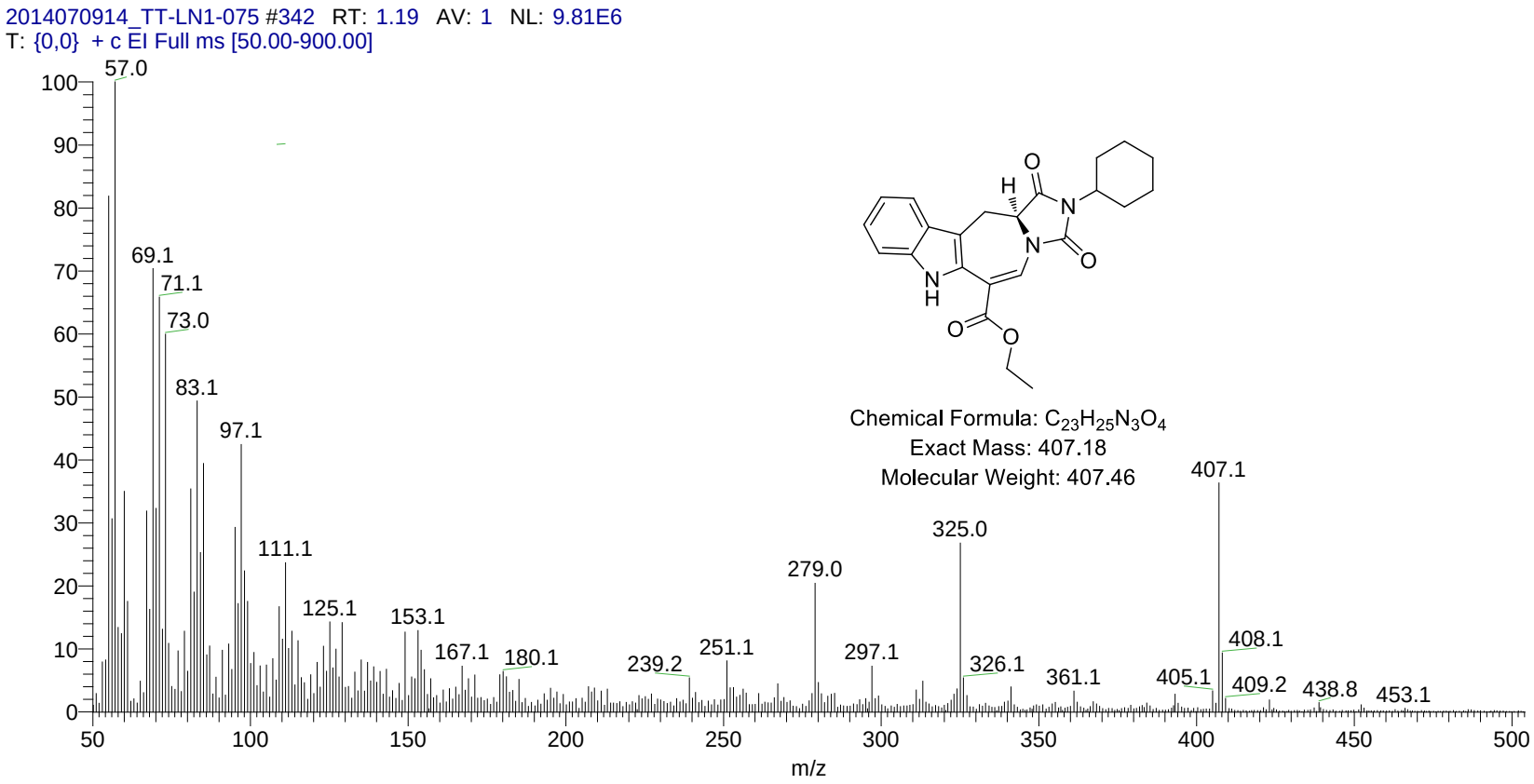
HPLC of compound **7n**



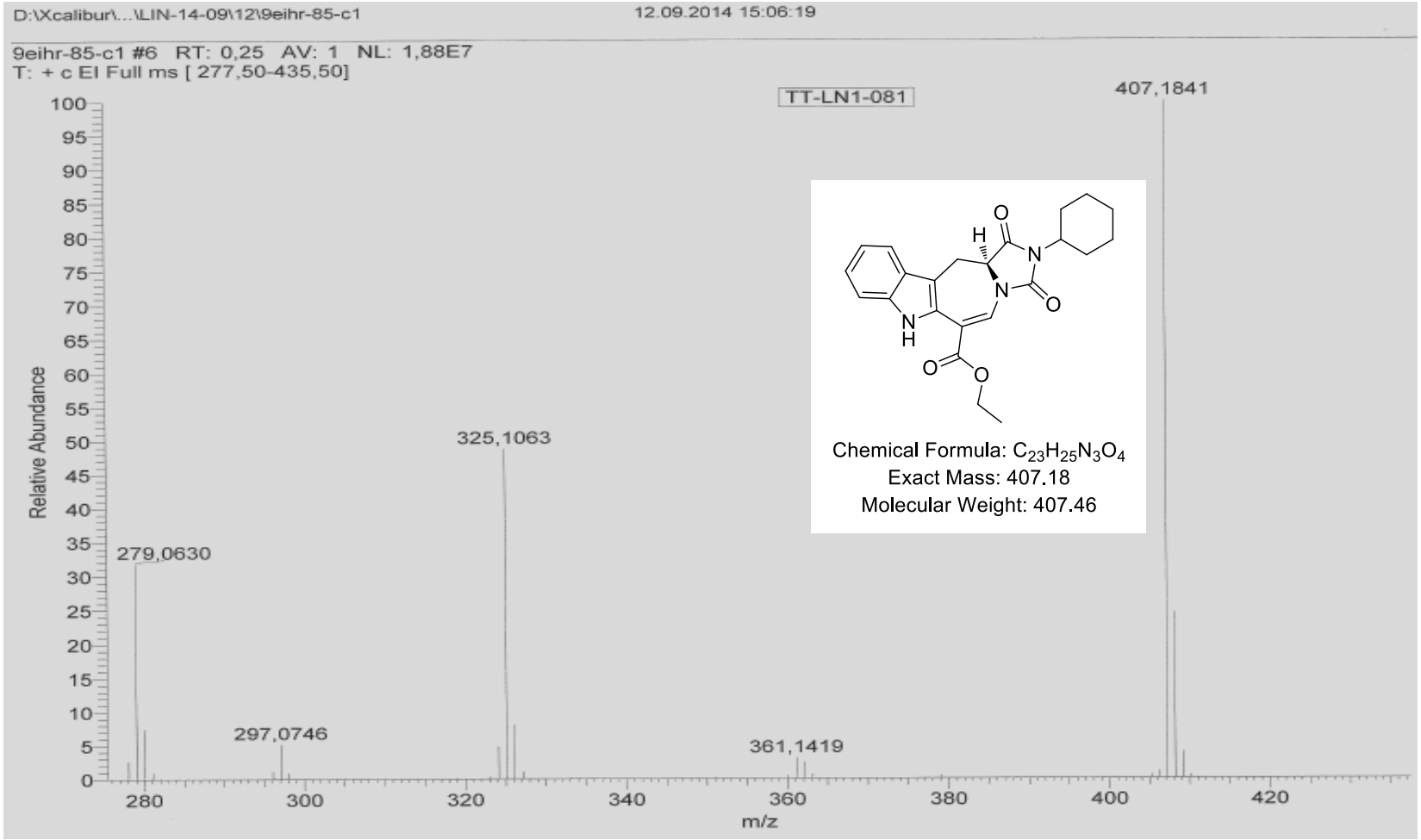
^{1}H NMR spectrum (400 MHz) of compound **7o** in $CDCl_3$



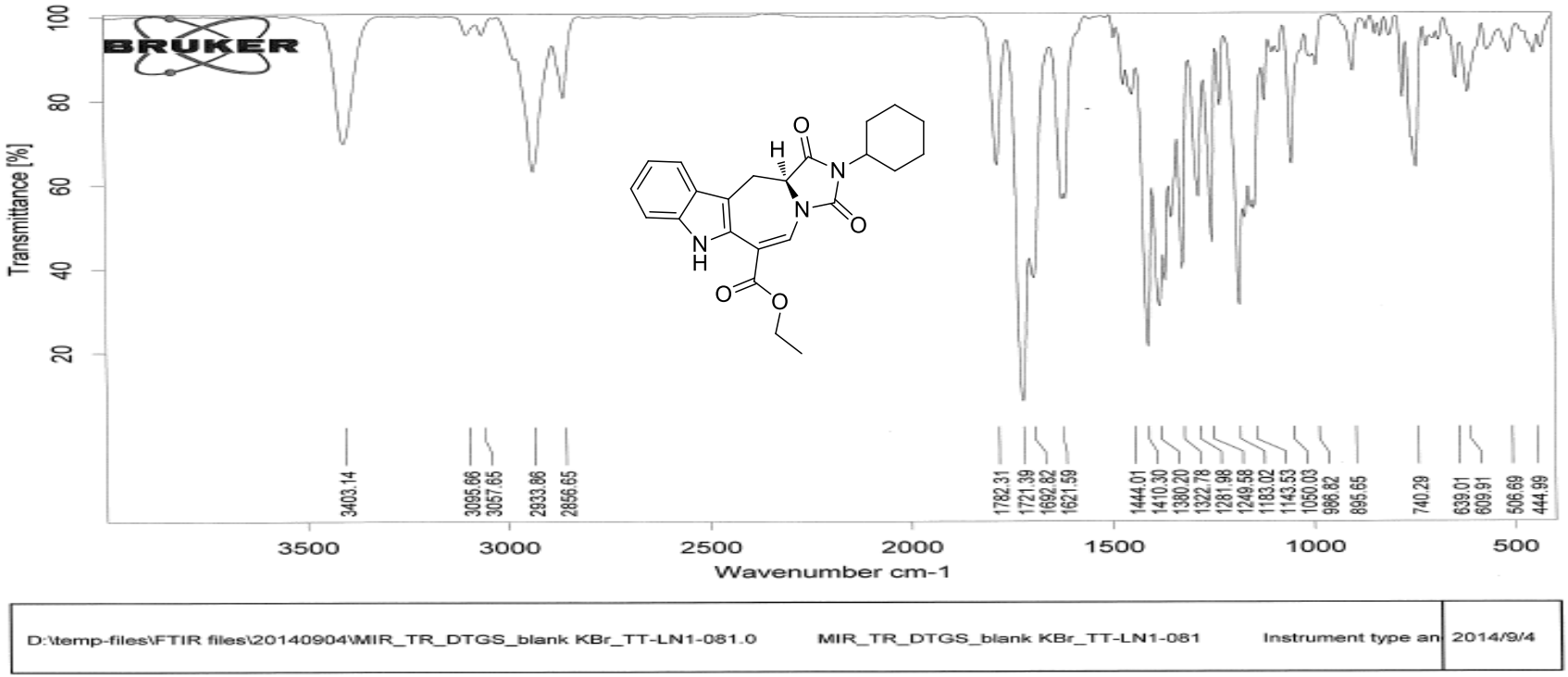
^{13}C NMR spectrum (101 MHz) of compound **7o** in $CDCl_3$



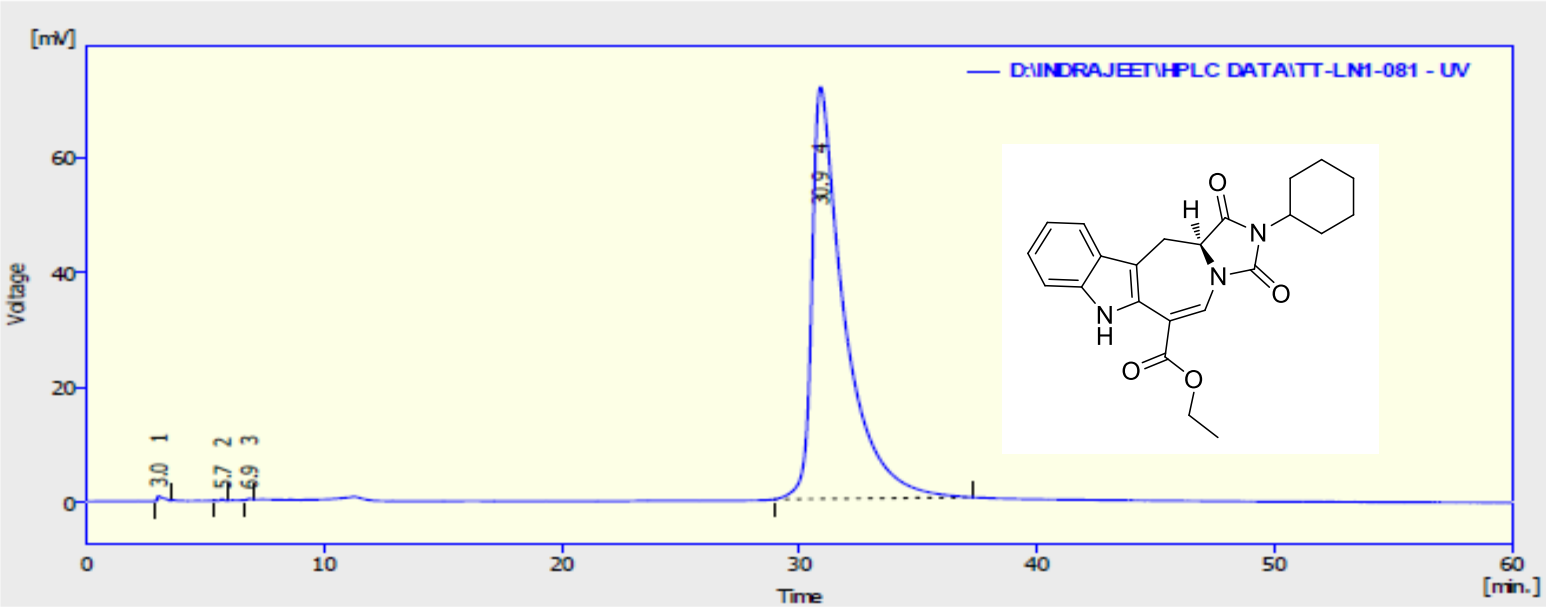
EI-LRMS of compound **7o**



EI-HRMS of compound **7o**



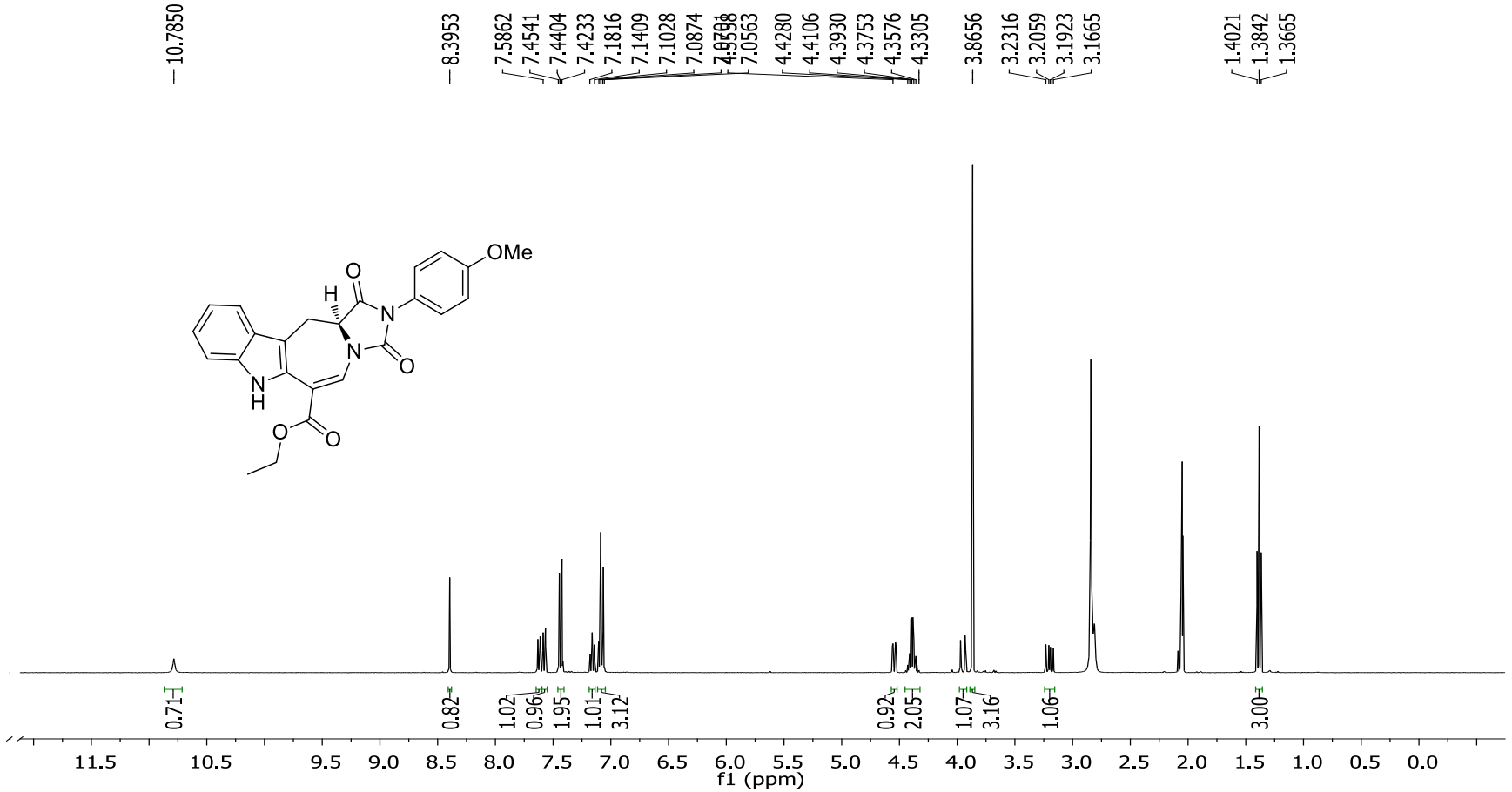
IR spectrum of compound **7o**



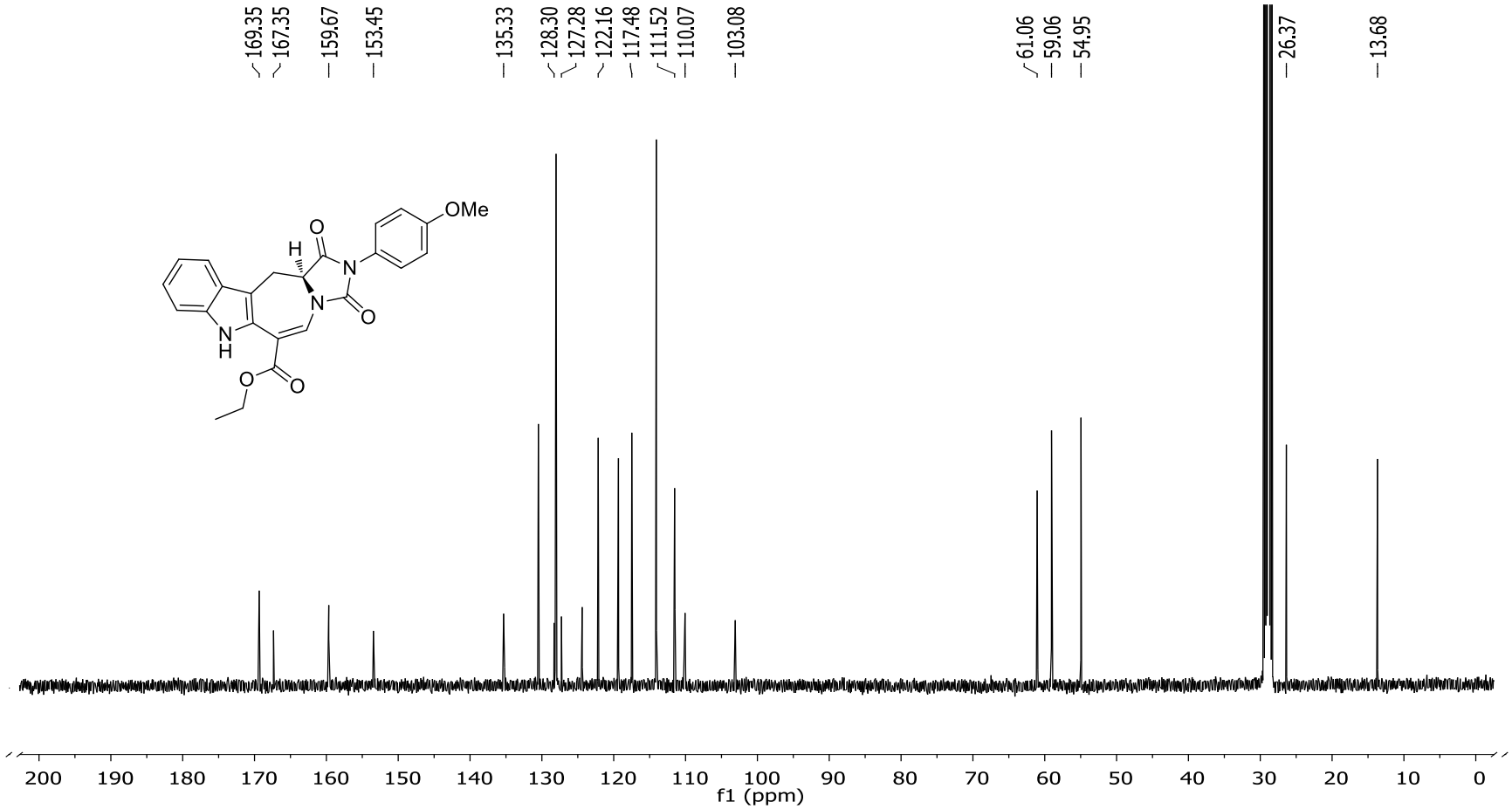
Result Table (Uncal - D:\INDRAJEET\HPLC DATA\TT-LN1-081 - UV)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
1	3.020	15.492	0.878	0.2	1.2
2	5.700	2.603	0.302	0.0	0.4
3	6.852	2.342	0.237	0.0	0.3
4	30.900	6827.827	71.801	99.7	98.1
	Total	6848.264	73.217	100.0	100.0

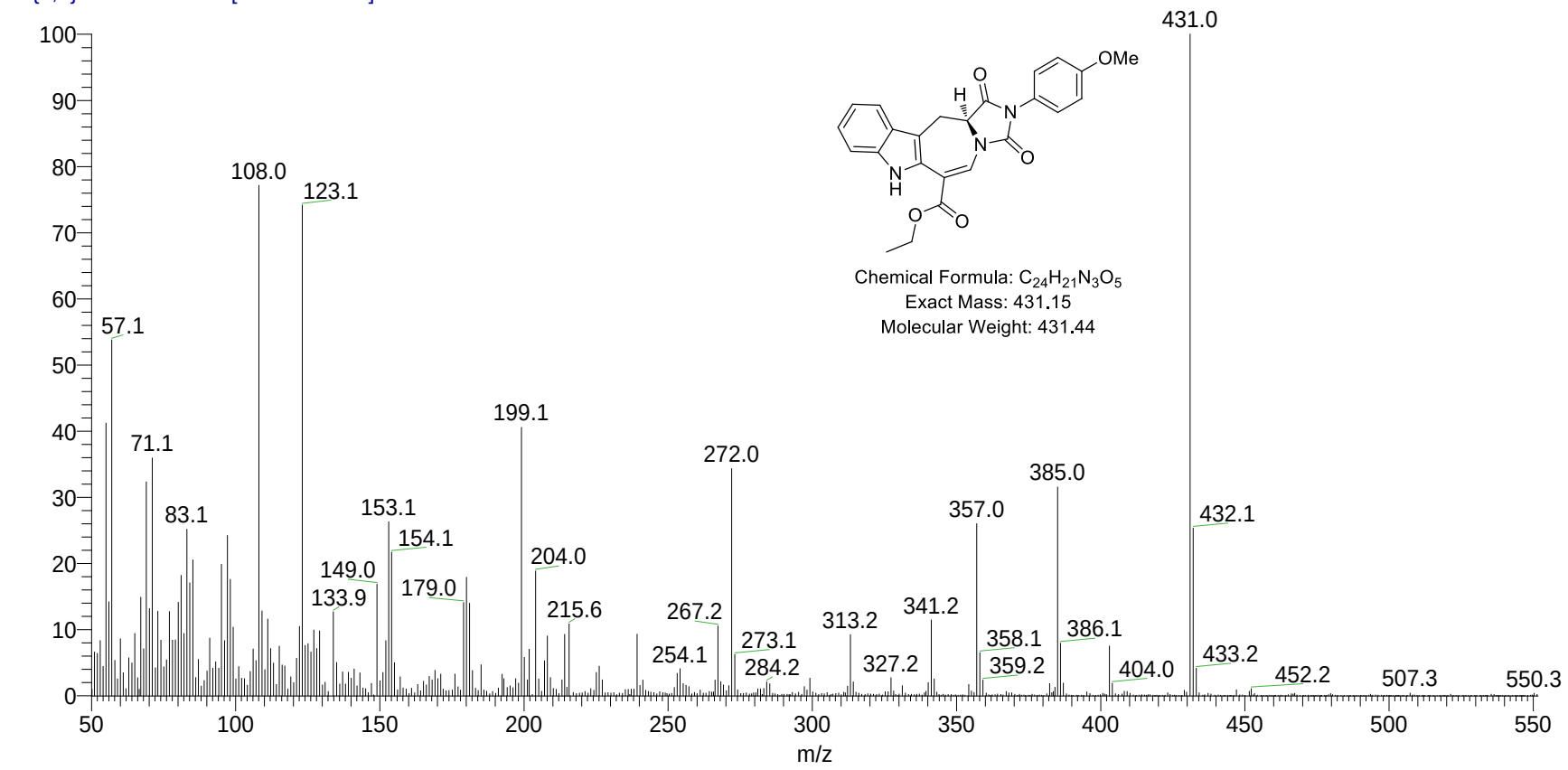
HPLC of compound **7o**



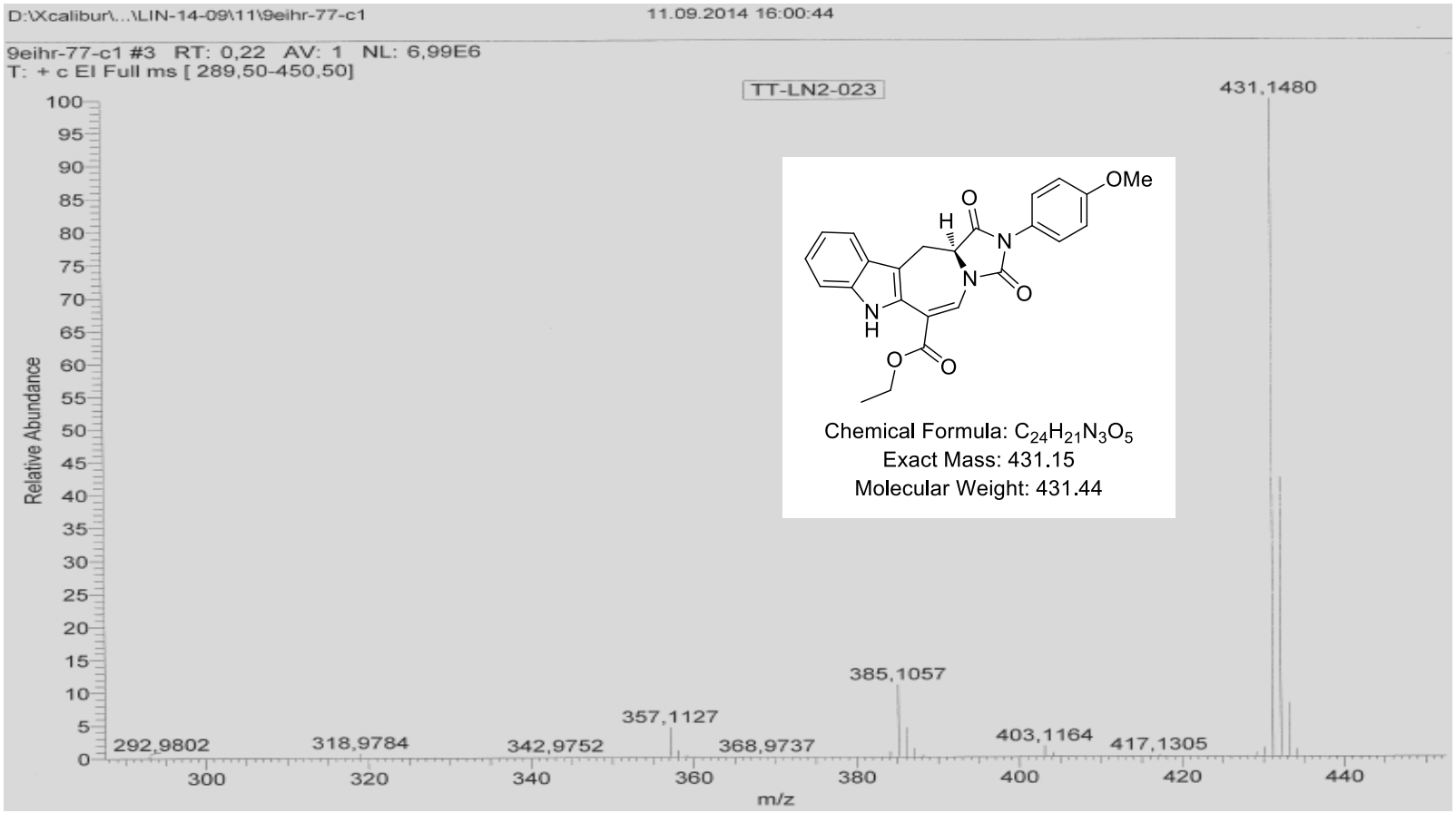
^{1}H NMR spectrum (400 MHz) of compound **7p** in acetone-d_6



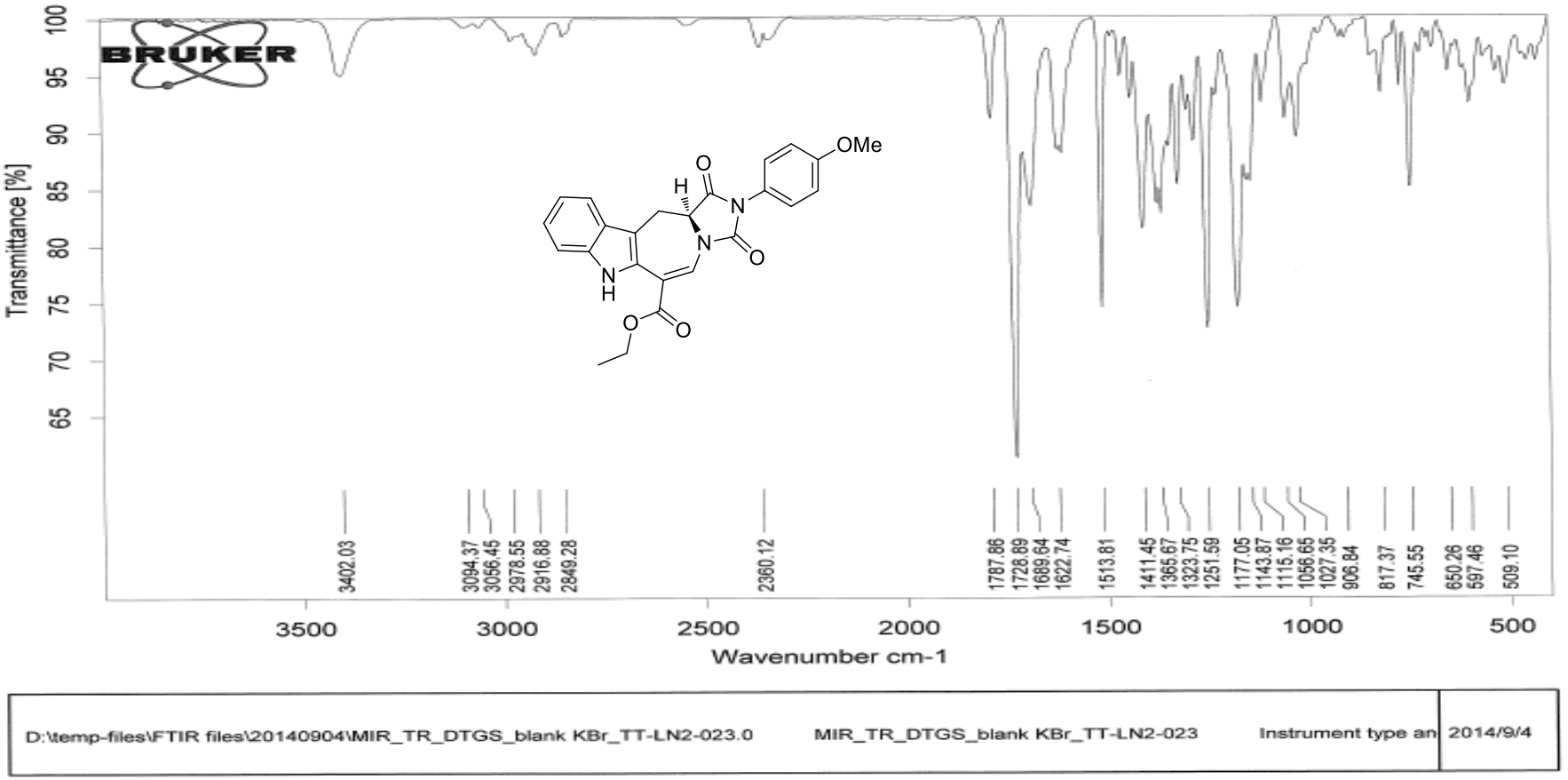
^{13}C NMR spectrum (101 MHz) of compound **7p** in acetone-d_6



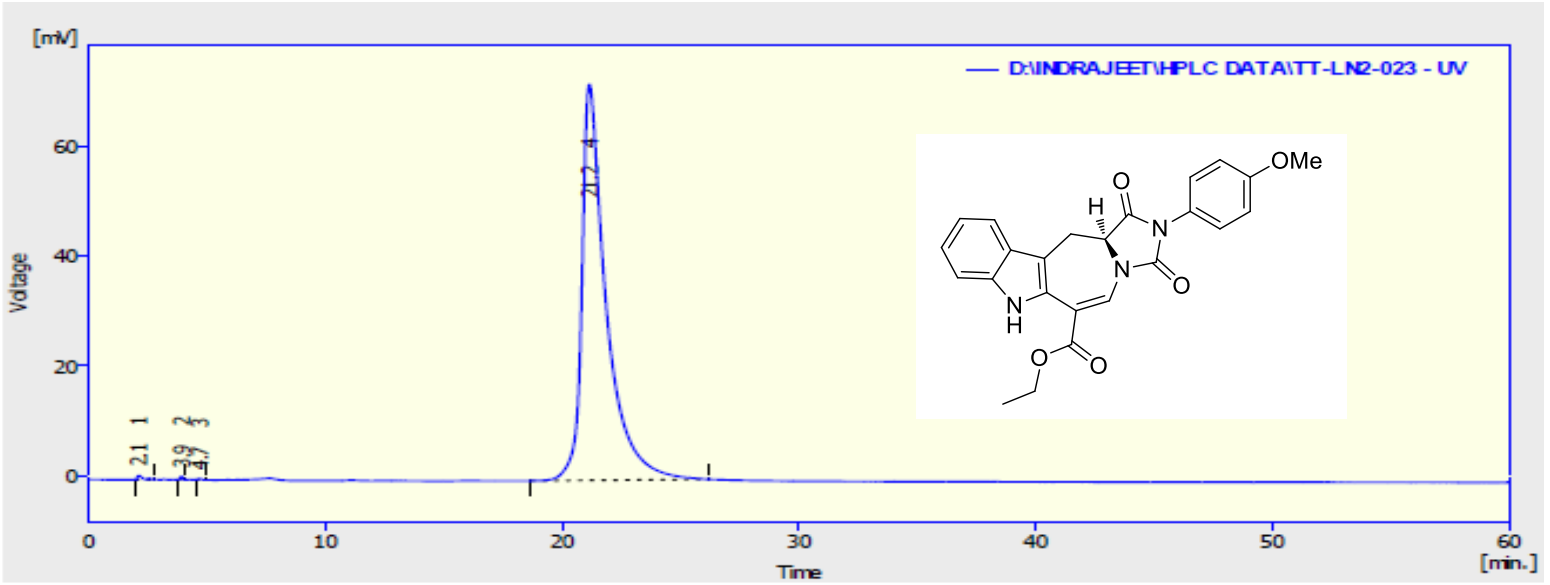
EI-LRMS of compound **7p**



EI-HRMS of compound **7p**



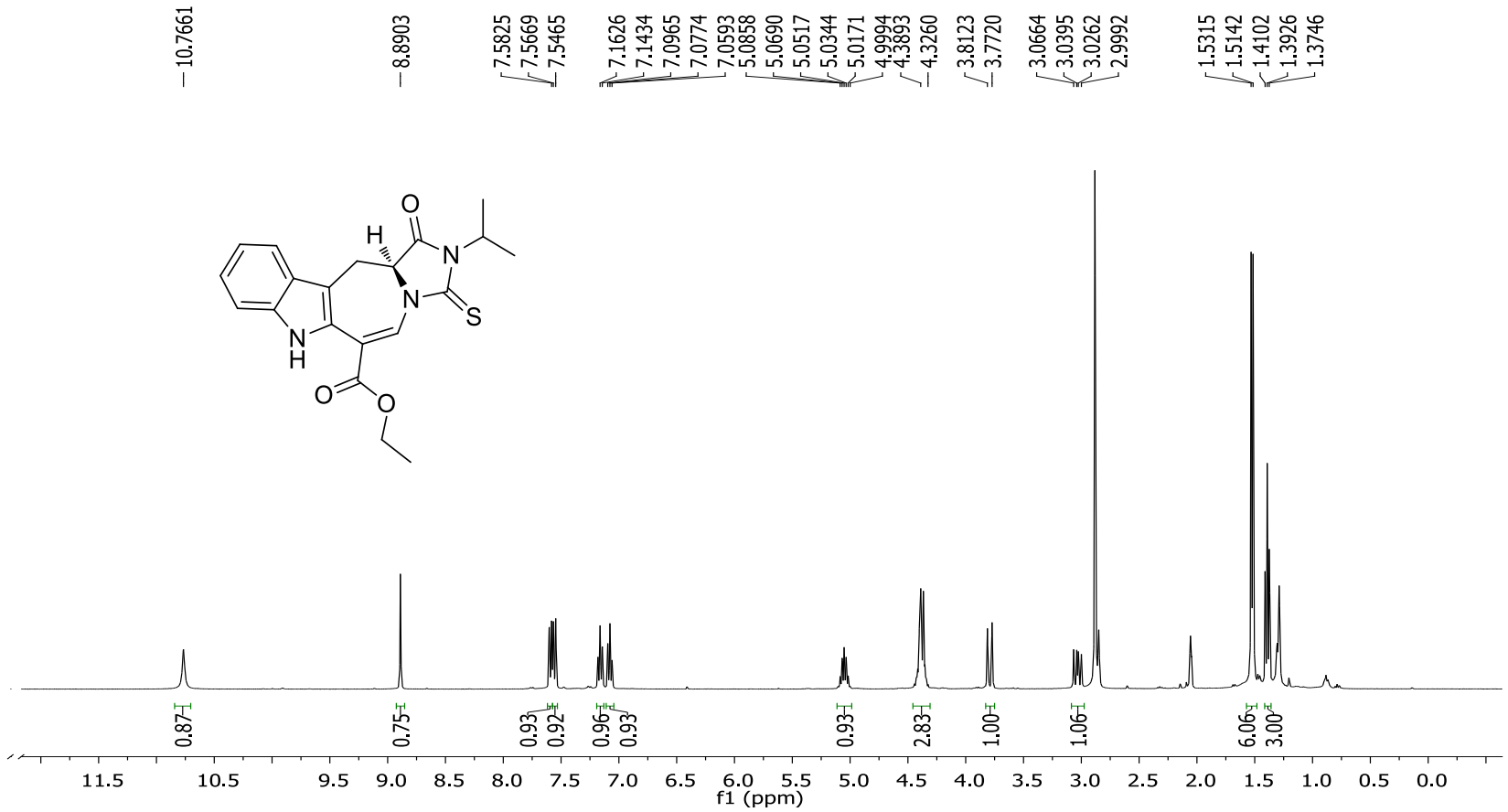
IR spectrum of compound **7p**



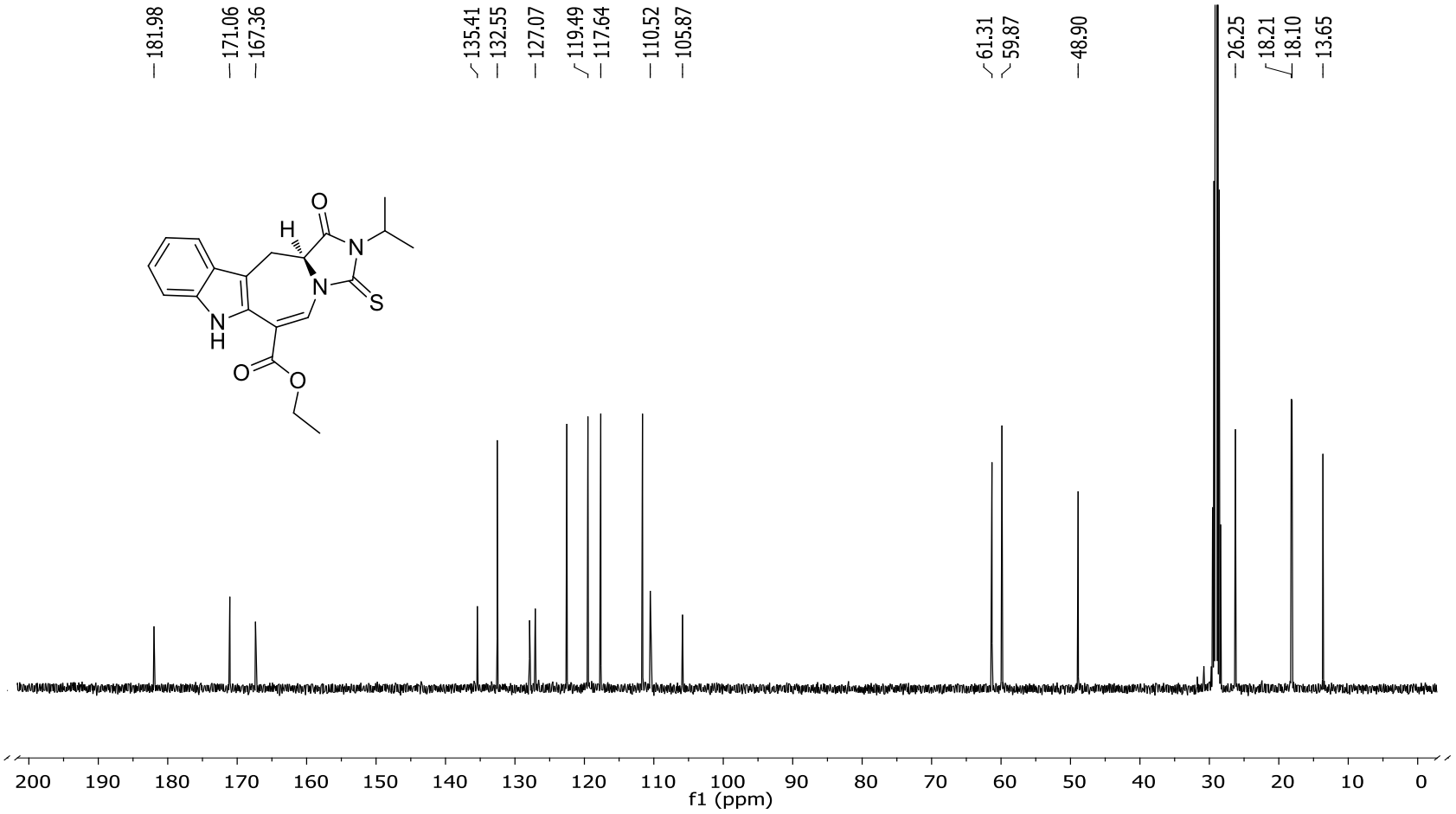
Result Table (Uncal - D:\INDRAJEET\HPLC DATA\TT-LN2-023 - UV)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
1	2.104	11.853	0.746	0.2	1.0
2	3.916	3.168	0.611	0.1	0.8
3	4.688	2.277	0.261	0.0	0.4
4	21.152	5146.503	72.047	99.7	97.8
	Total	5163.802	73.665	100.0	100.0

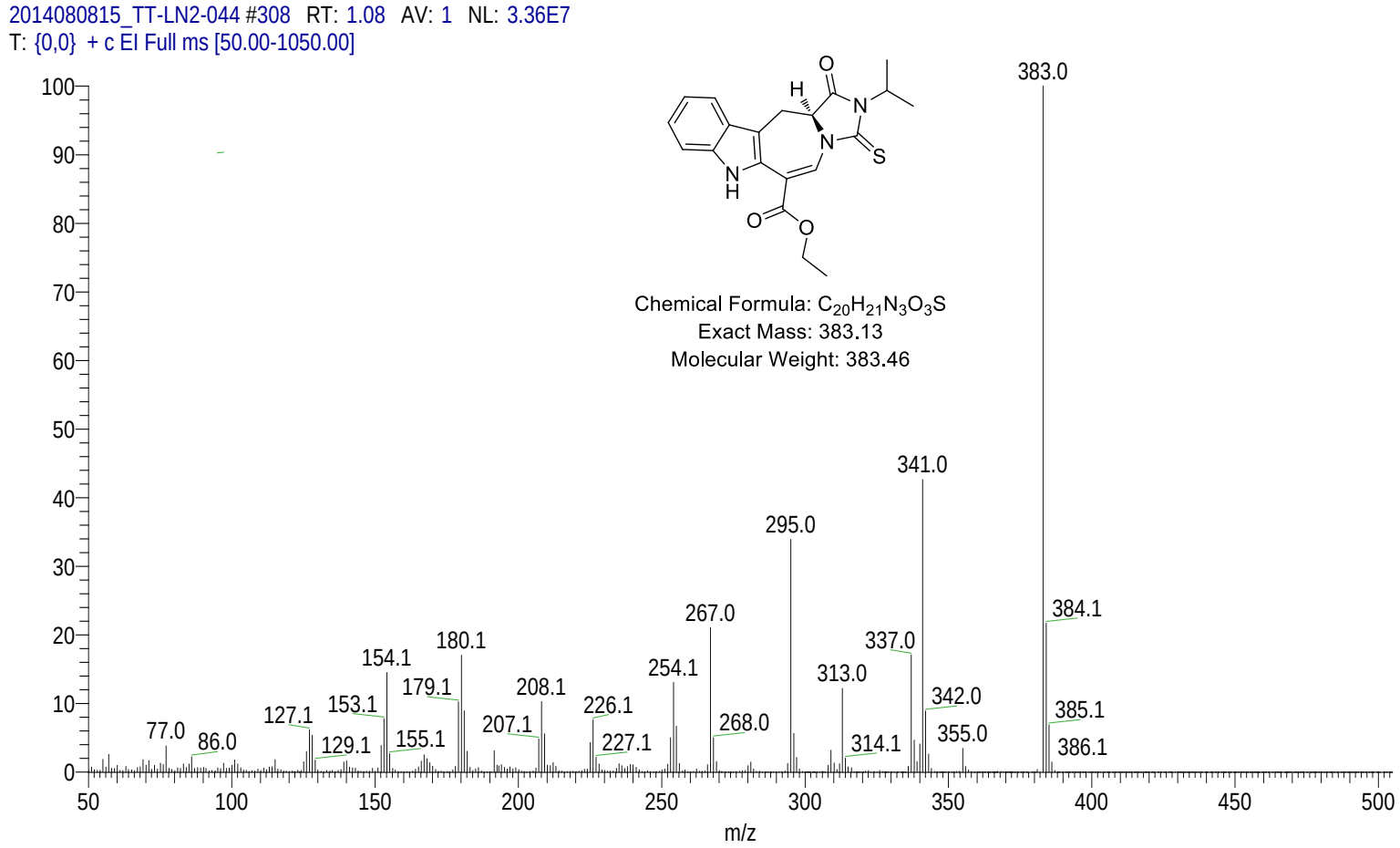
HPLC of compound **7p**



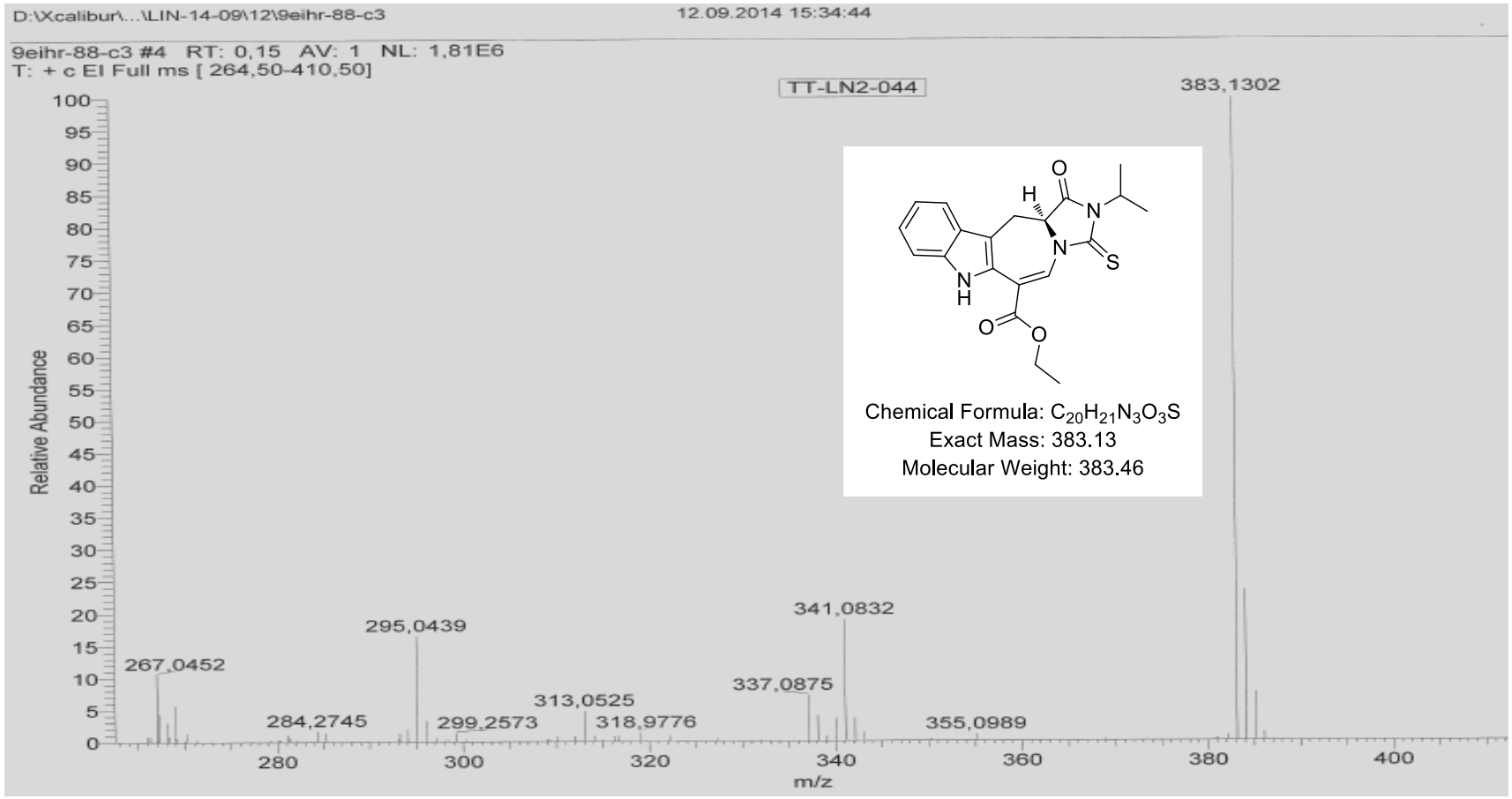
^{1}H NMR spectrum (400 MHz) of compound **7q** in acetone-d_6



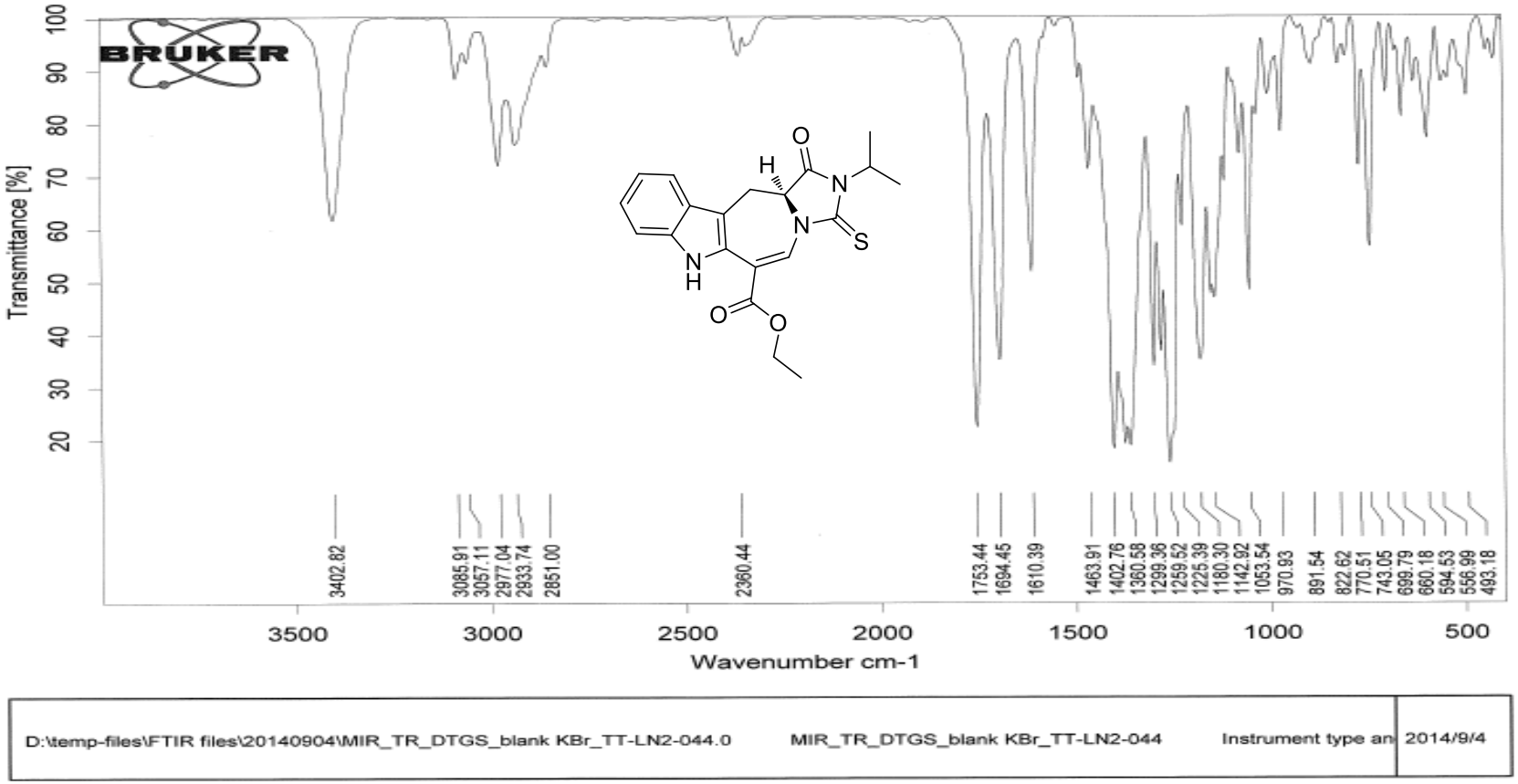
^{13}C NMR spectrum (101 MHz) of compound **7q** in acetone-d_6



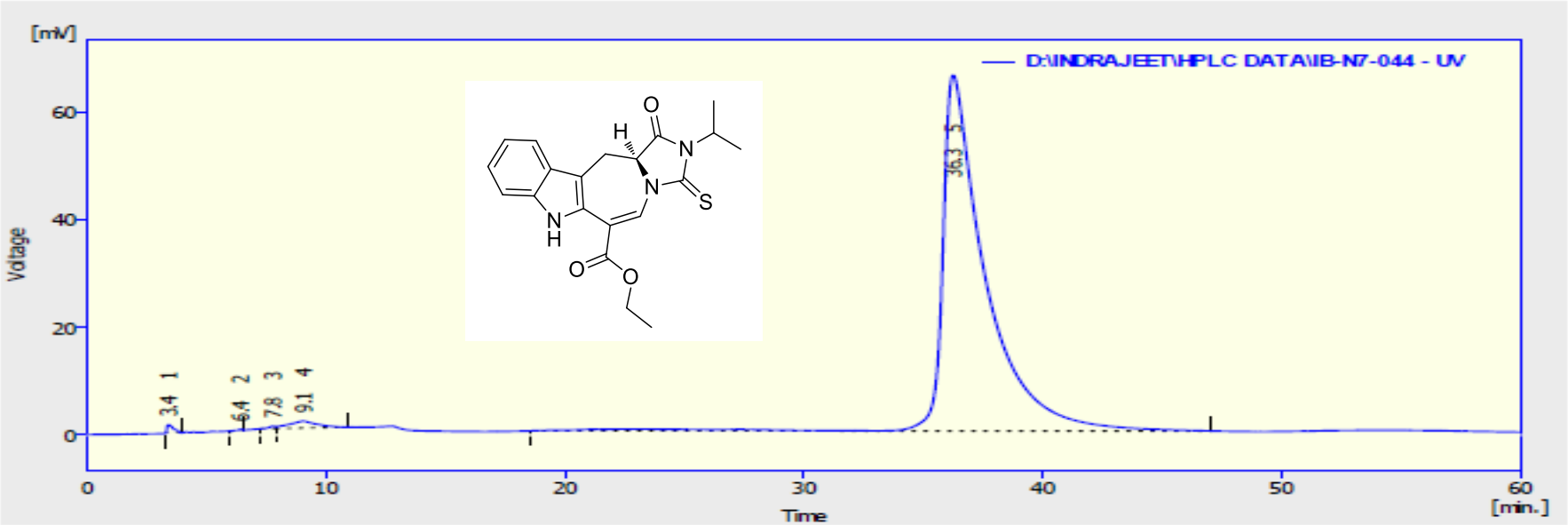
EI-LRMS of compound **7q**



EI-HRMS of compound **7q**



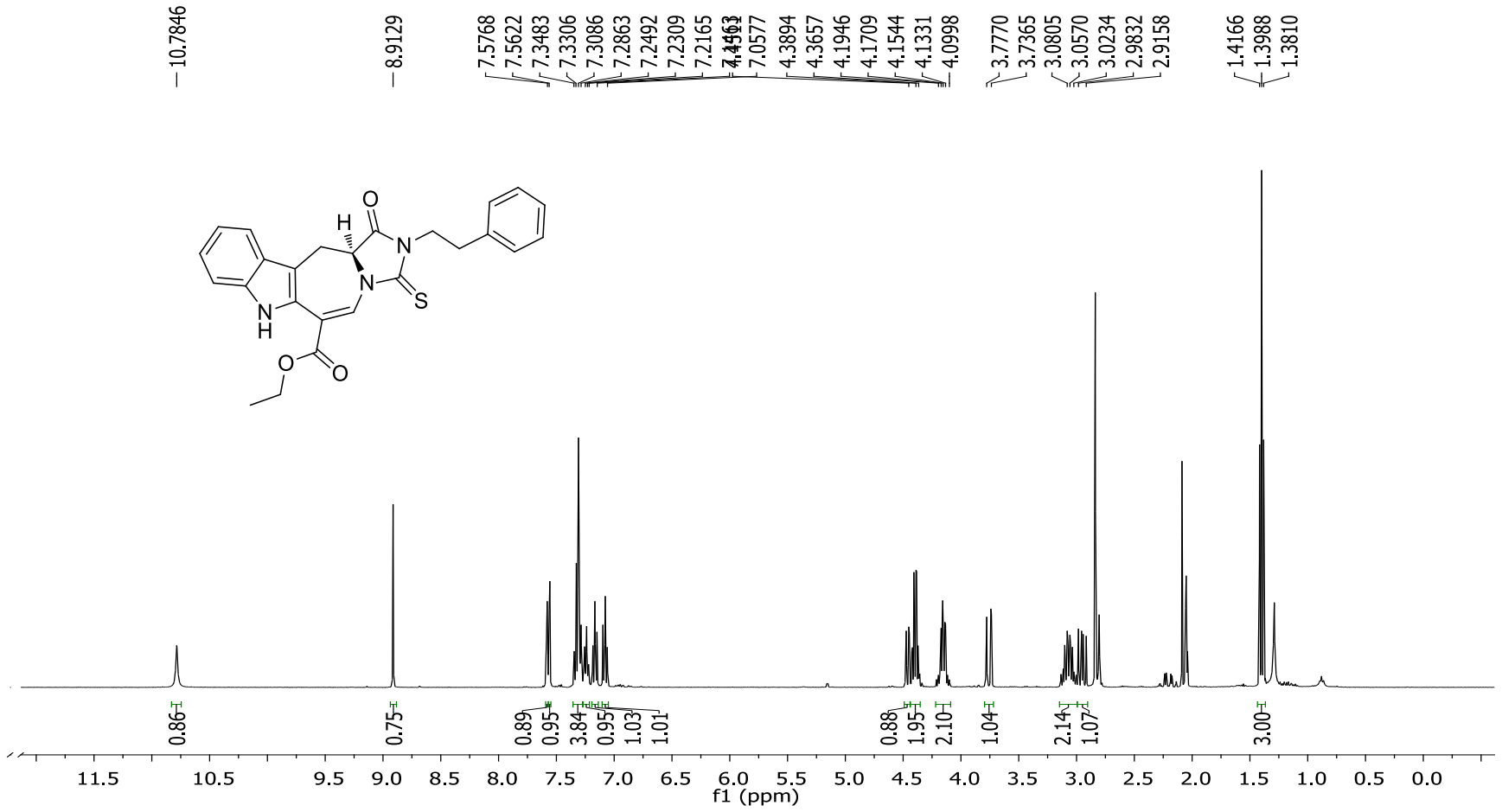
IR spectrum of compound **7q**



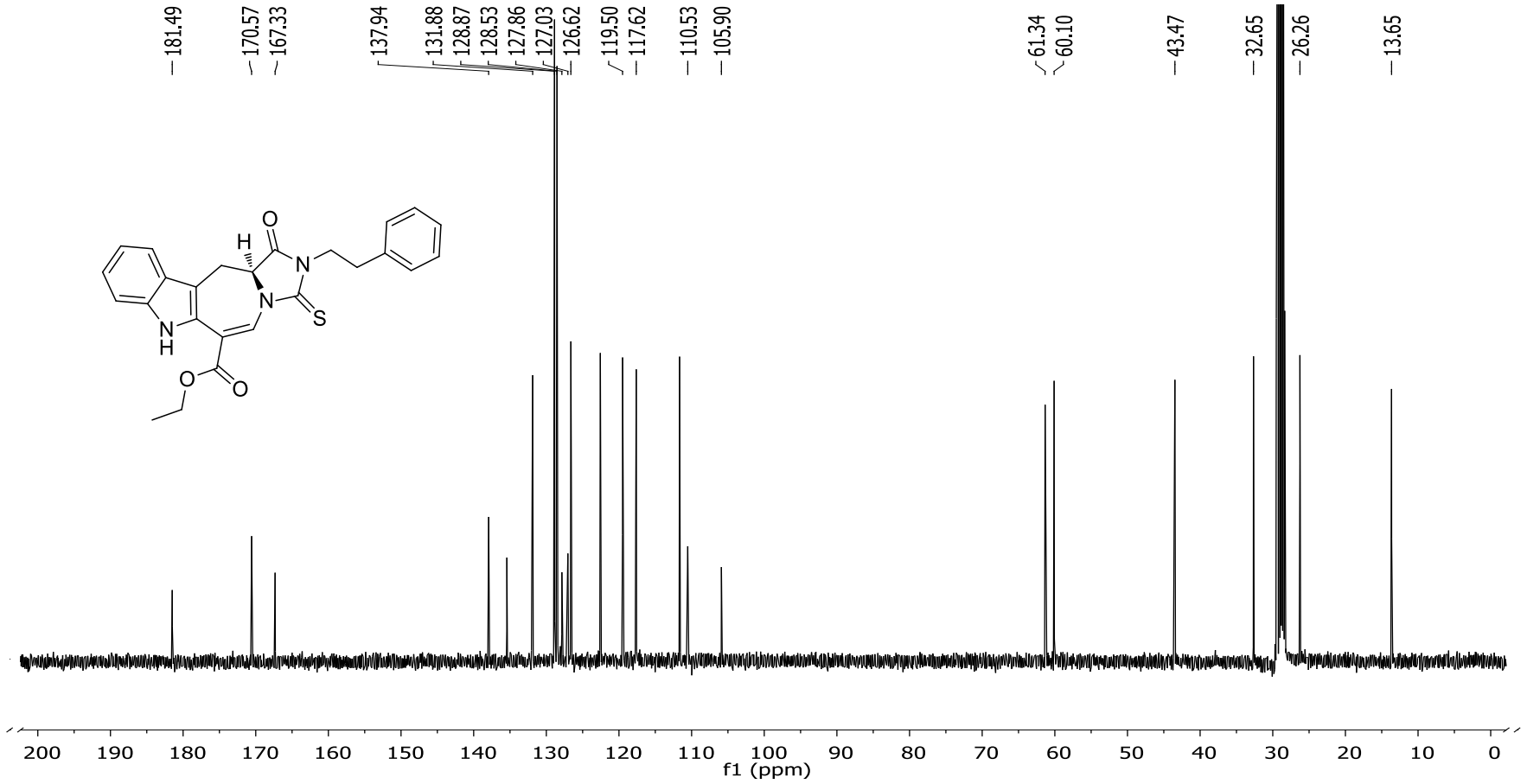
Result Table (Uncal - D:\INDRAJEET\HPLC DATA\IB-N7-044 - UV)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
1	3.384	30.977	1.569	0.4	2.2
2	6.416	2.841	0.246	0.0	0.4
3	7.752	7.524	0.442	0.1	0.6
4	9.052	106.477	1.290	1.3	1.8
5	36.264	8301.639	66.248	98.3	94.9
	Total	8449.459	69.795	100.0	100.0

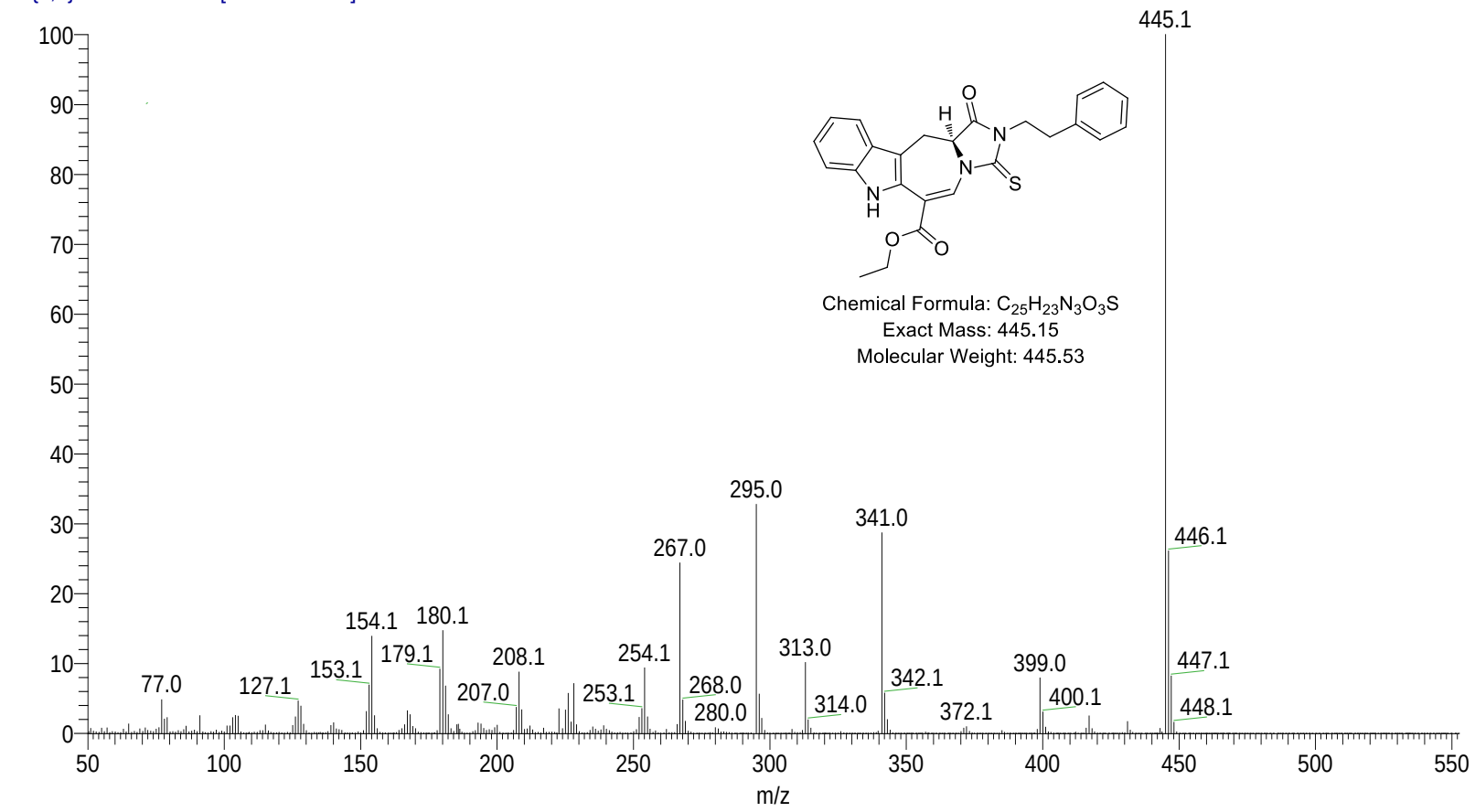
HPLC of compound **7q**



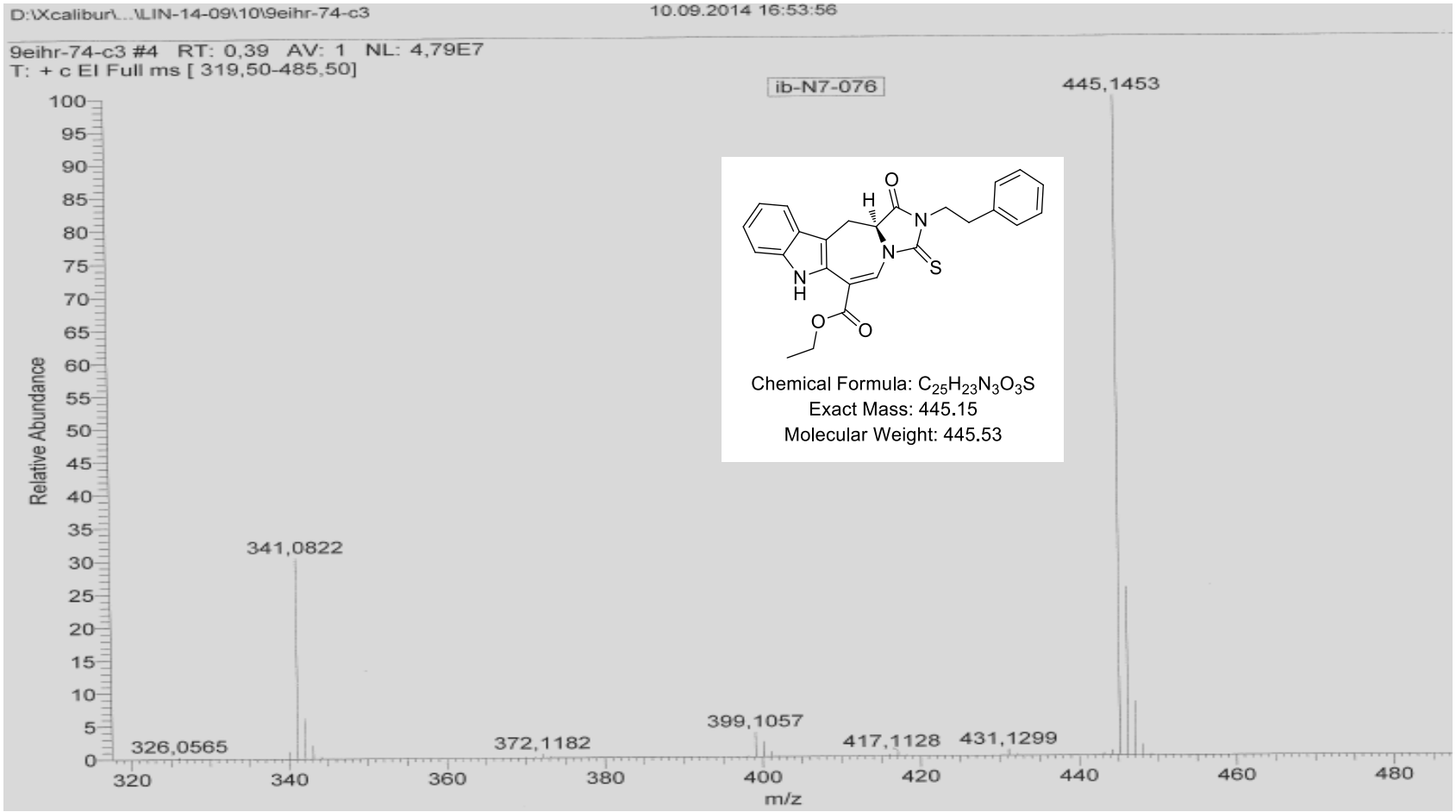
^{1}H NMR spectrum (400 MHz) of compound **7r** in acetone-d_6



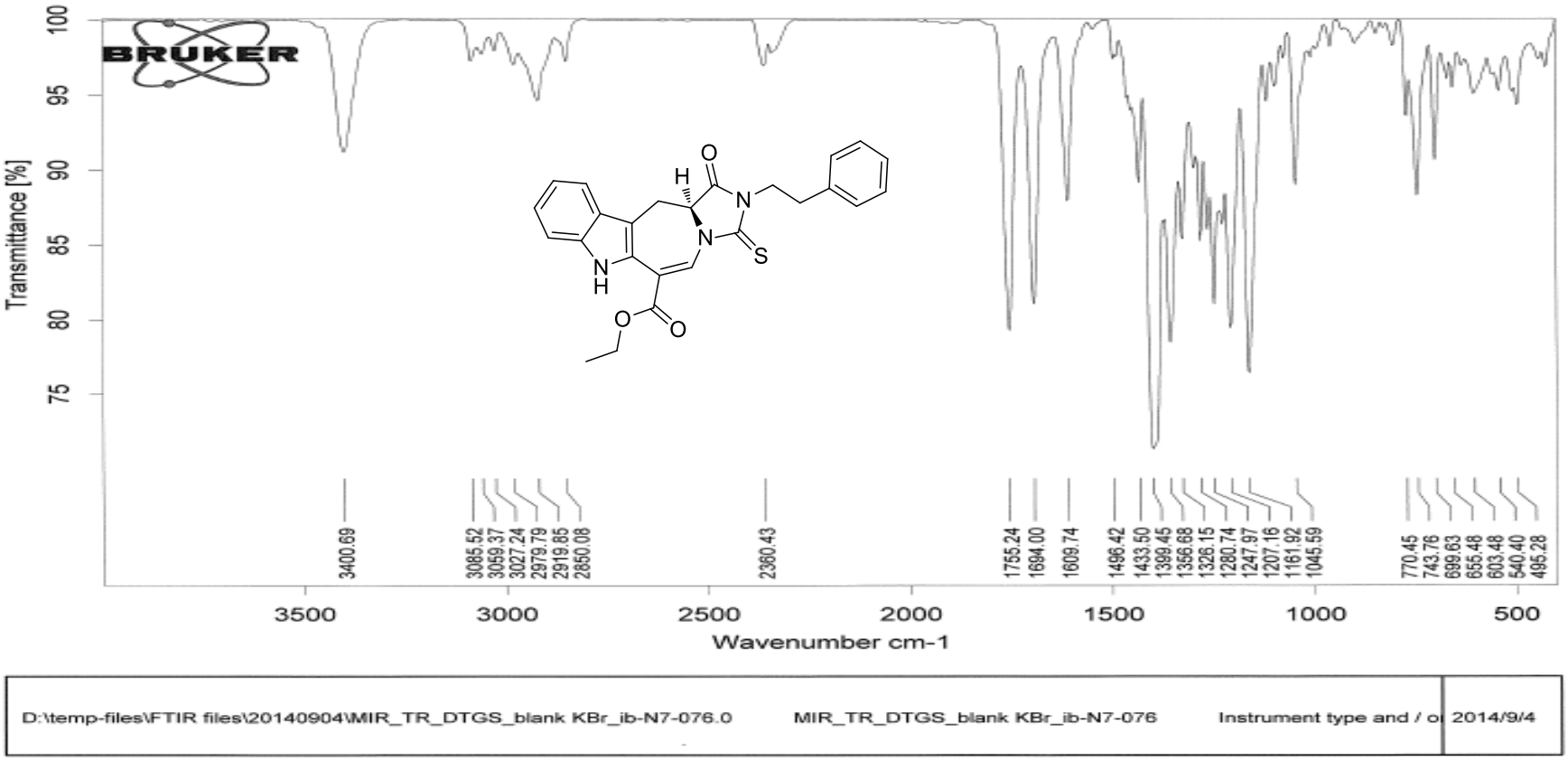
^{13}C NMR spectrum (101 MHz) of compound **7r** in acetone-d_6



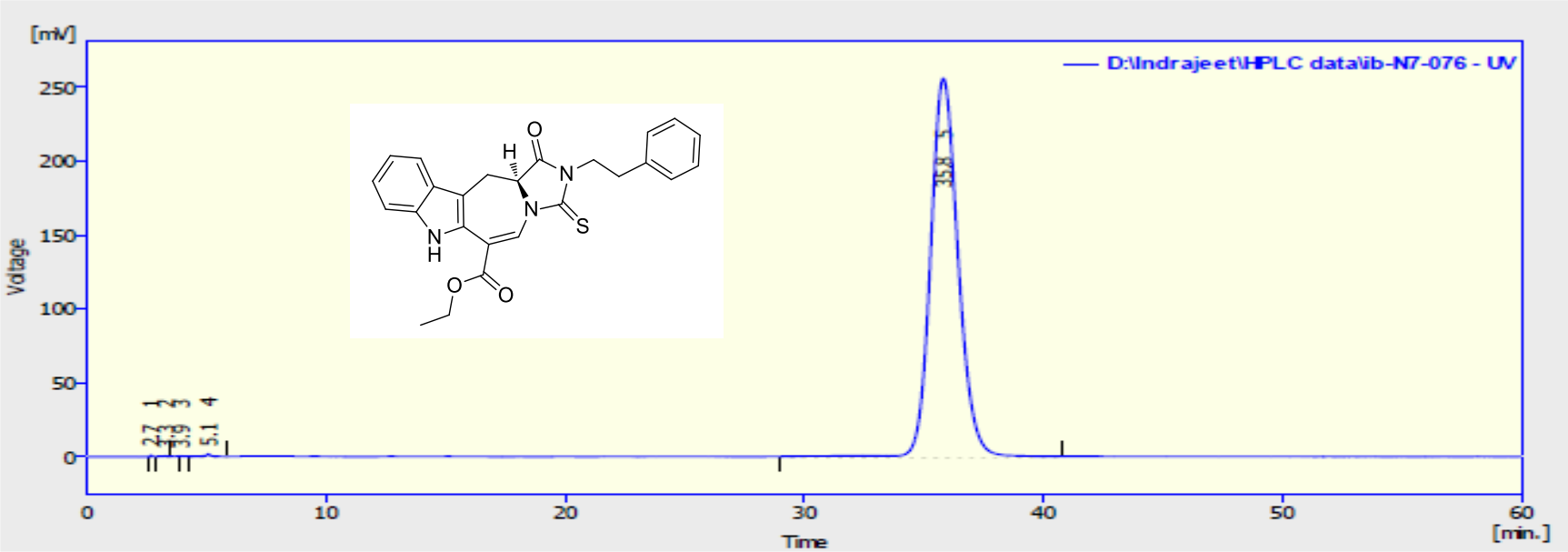
EI-LRMS of compound **7r**



EI-HRMS of compound **7r**



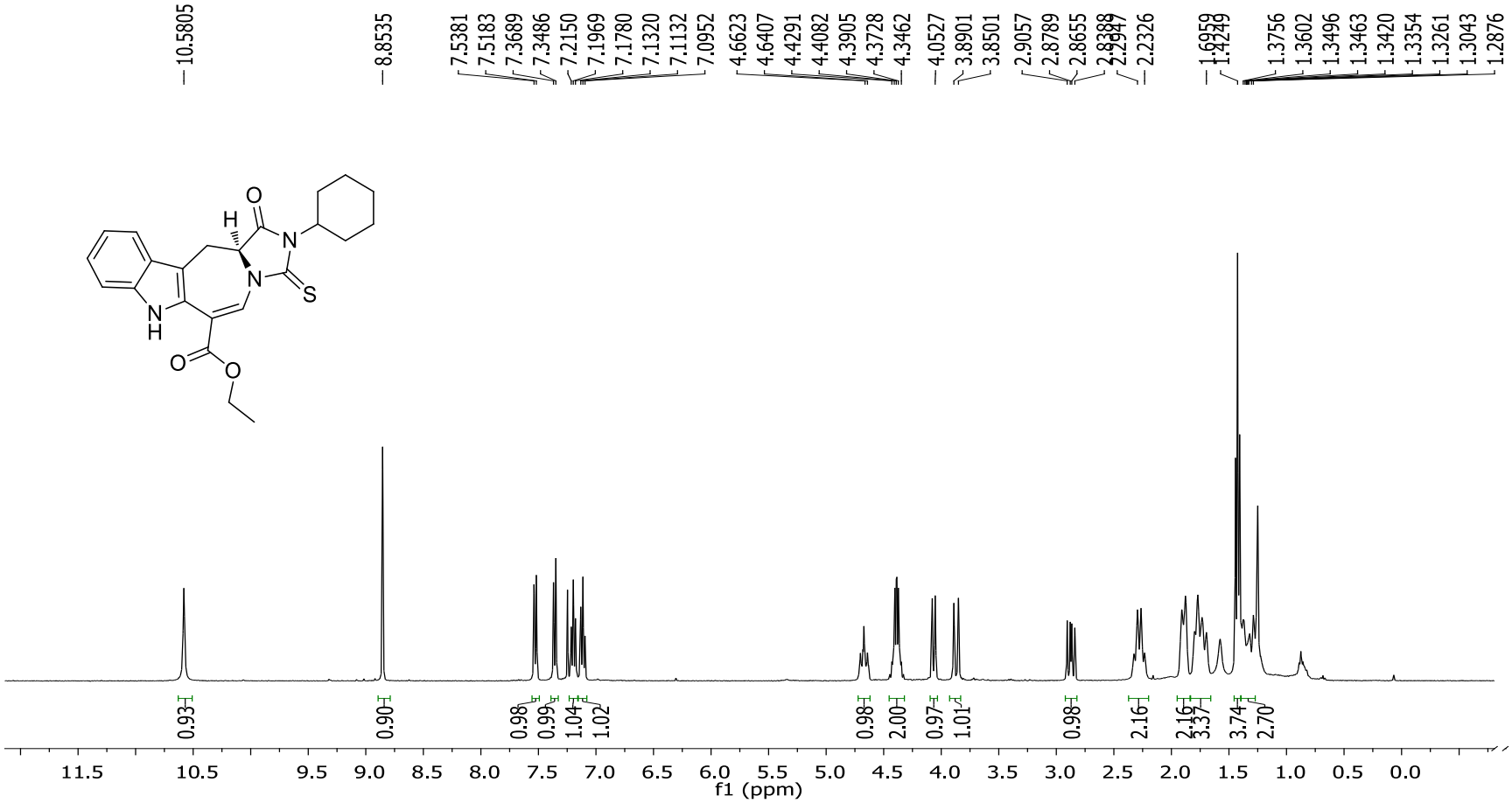
IR spectrum of compound **7r**



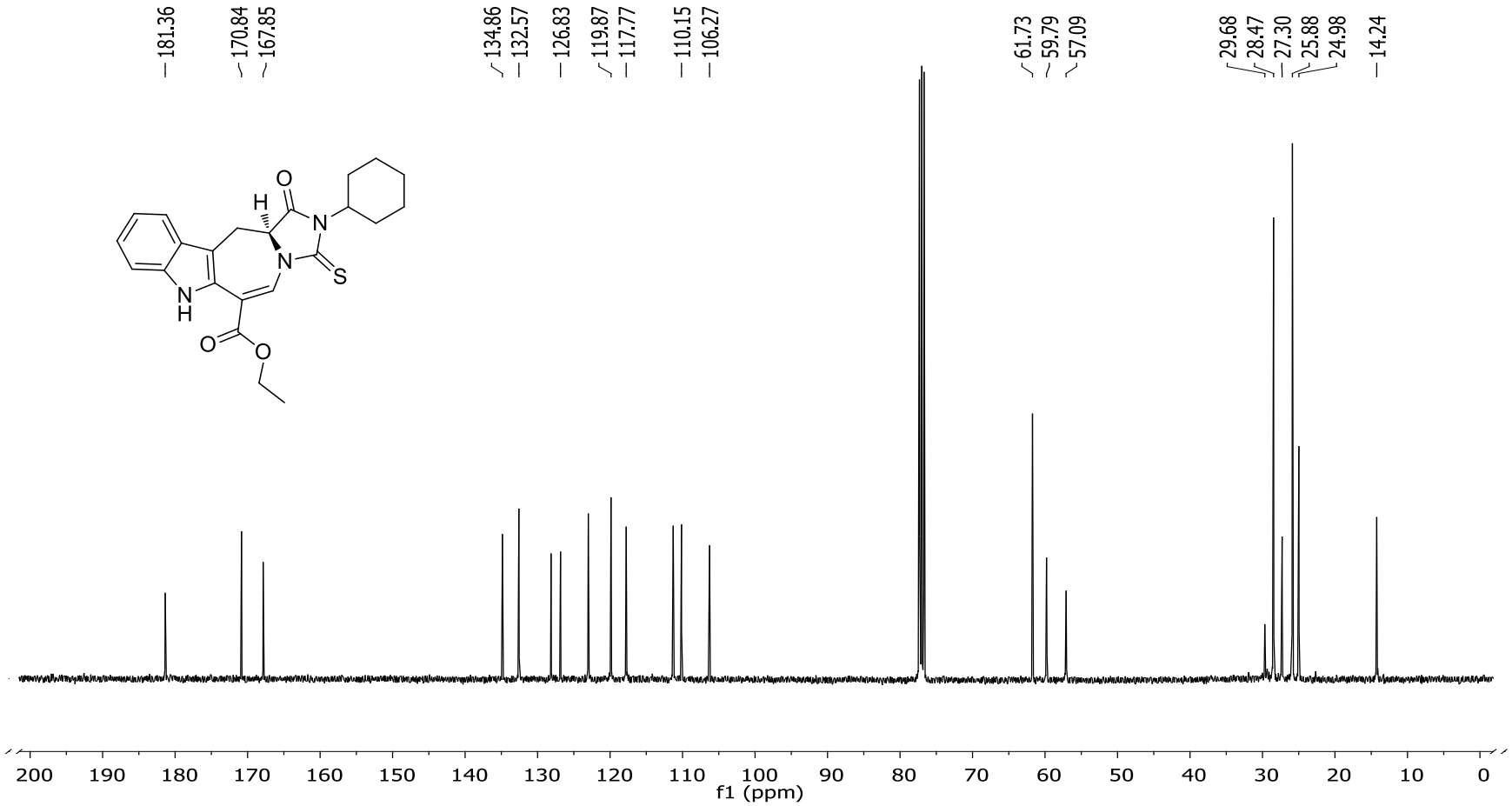
Result Table (Uncal - D:\Indrajeet\HPLC data\ib-N7-076 - UV)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
1	2.676	5.060	0.857	0.0	0.3
2	3.296	3.716	0.362	0.0	0.1
3	3.912	1.814	0.291	0.0	0.1
4	5.076	25.238	1.707	0.1	0.7
5	35.816	20359.697	255.046	99.8	98.8
	Total	20395.525	259.061	100.0	100.0

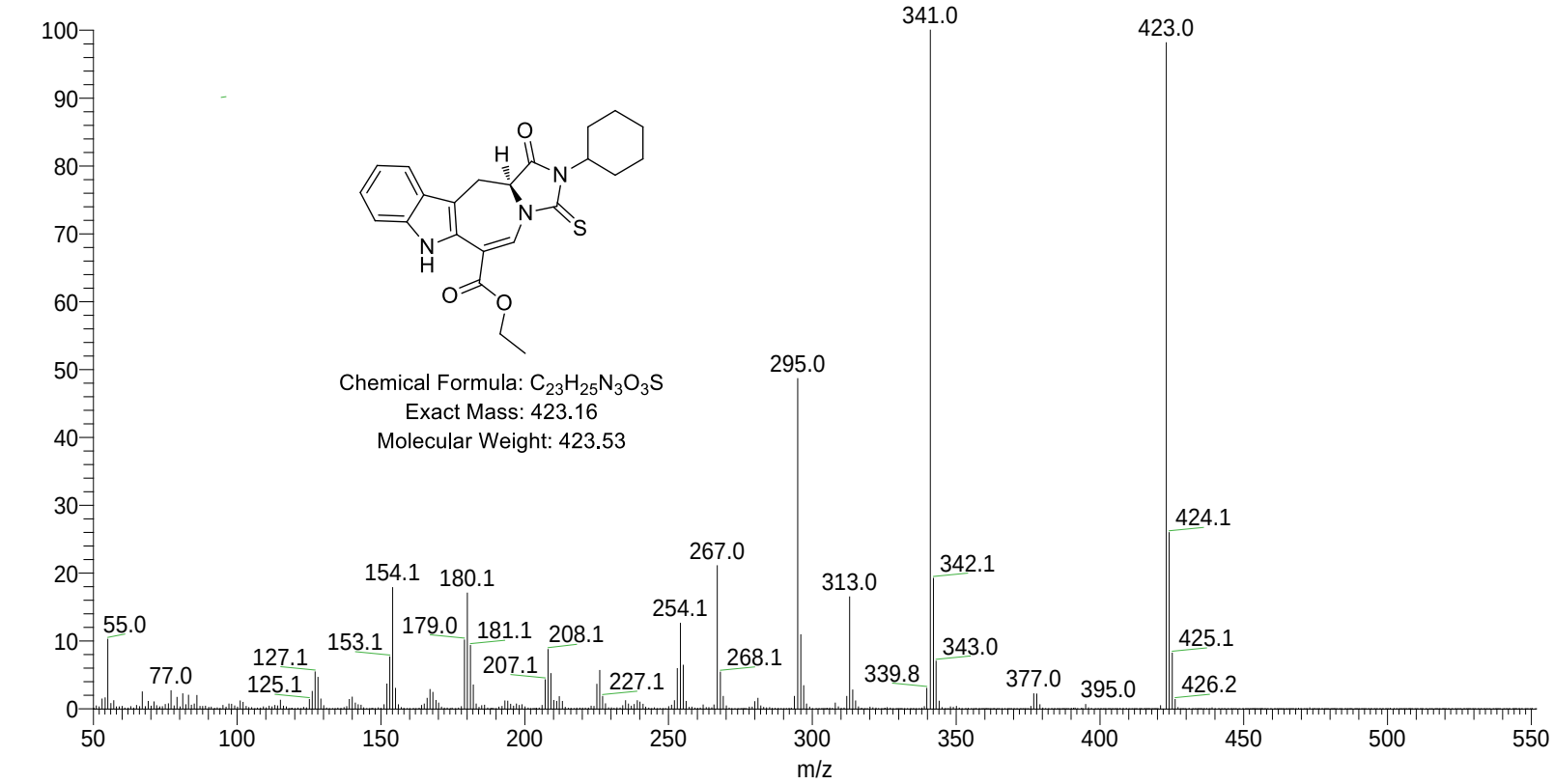
HPLC of compound **7r**



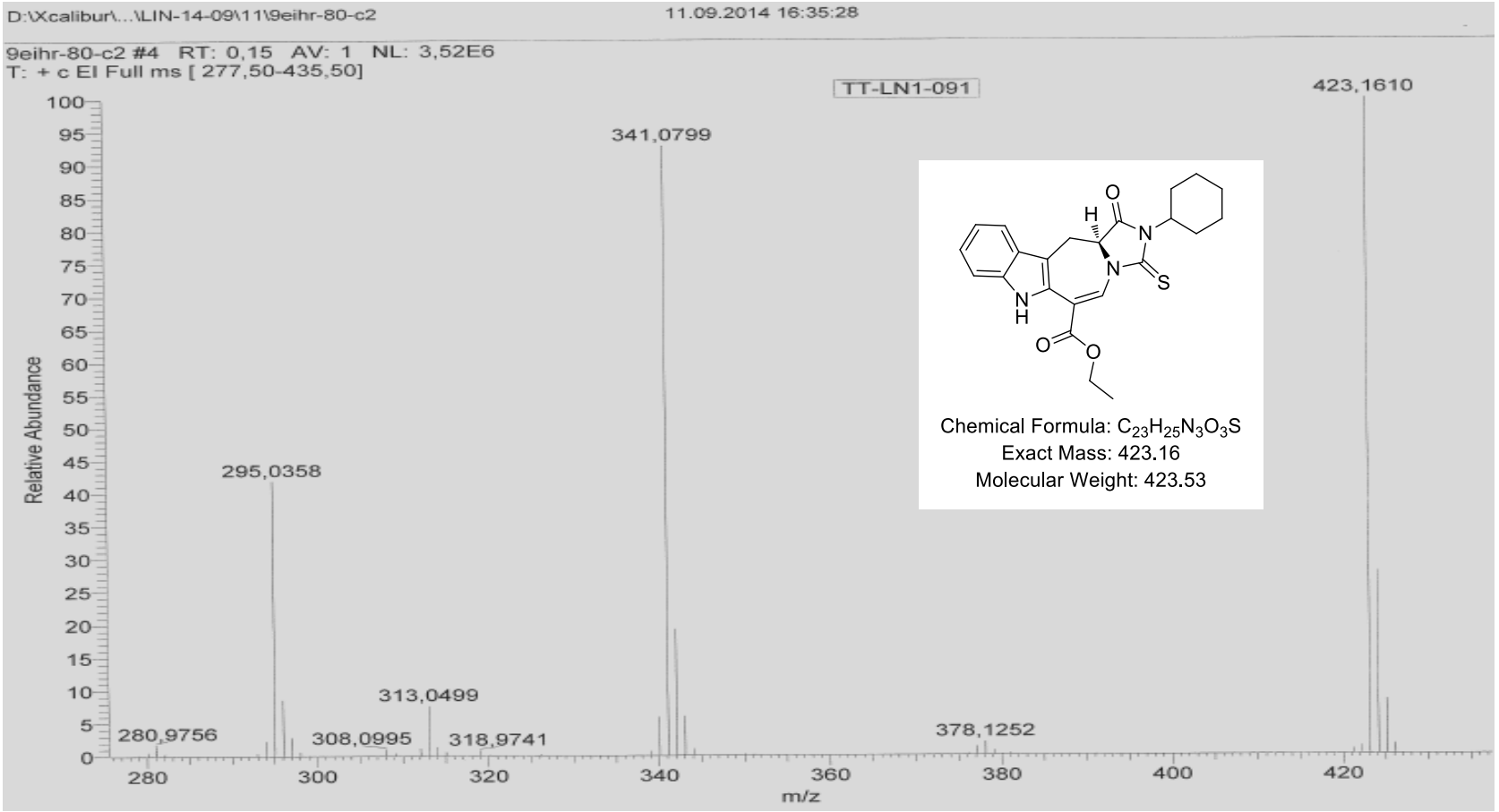
^{1}H NMR spectrum (400 MHz) of compound **7s** in $CDCl_3$



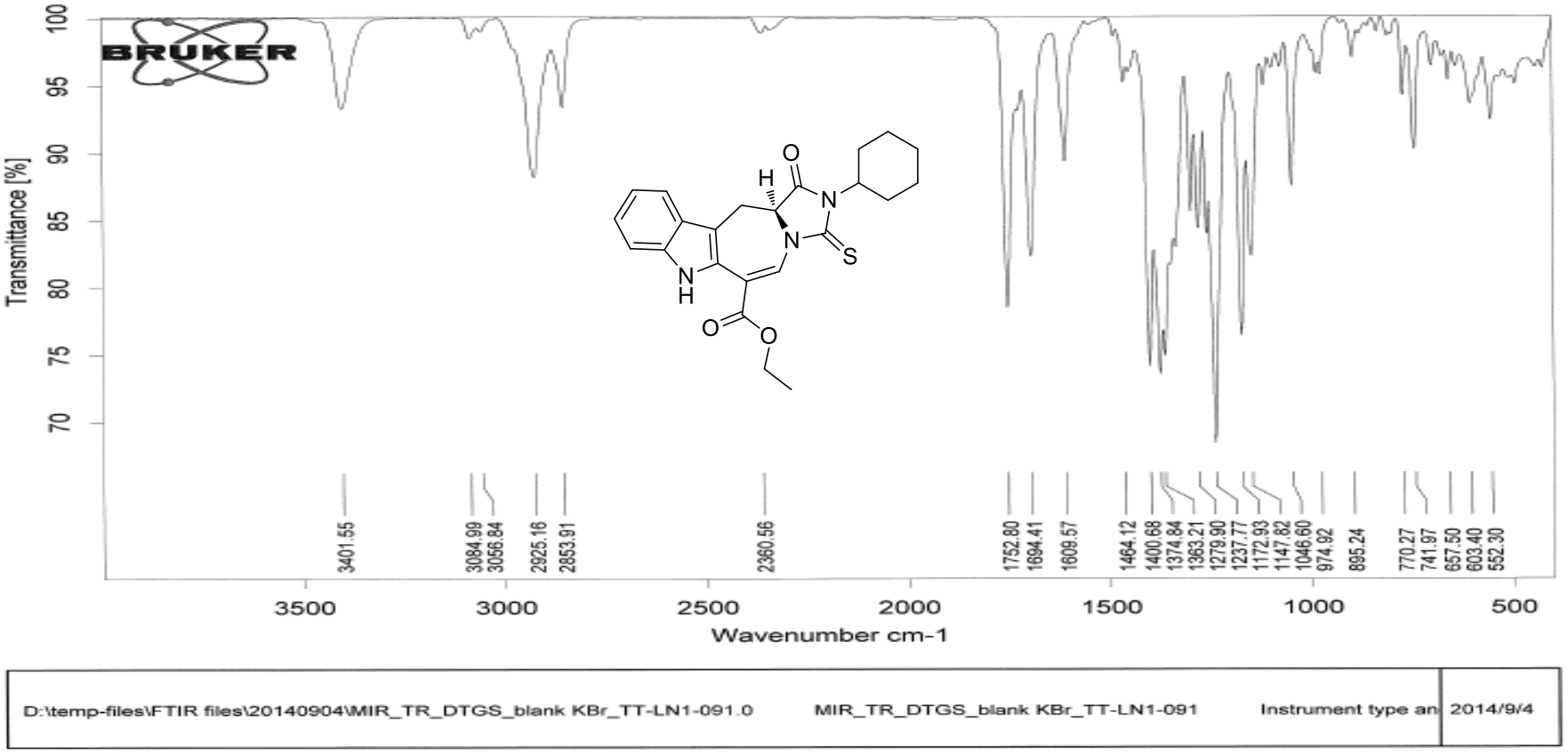
^{13}C NMR spectrum (101 MHz) of compound **7s** in $CDCl_3$



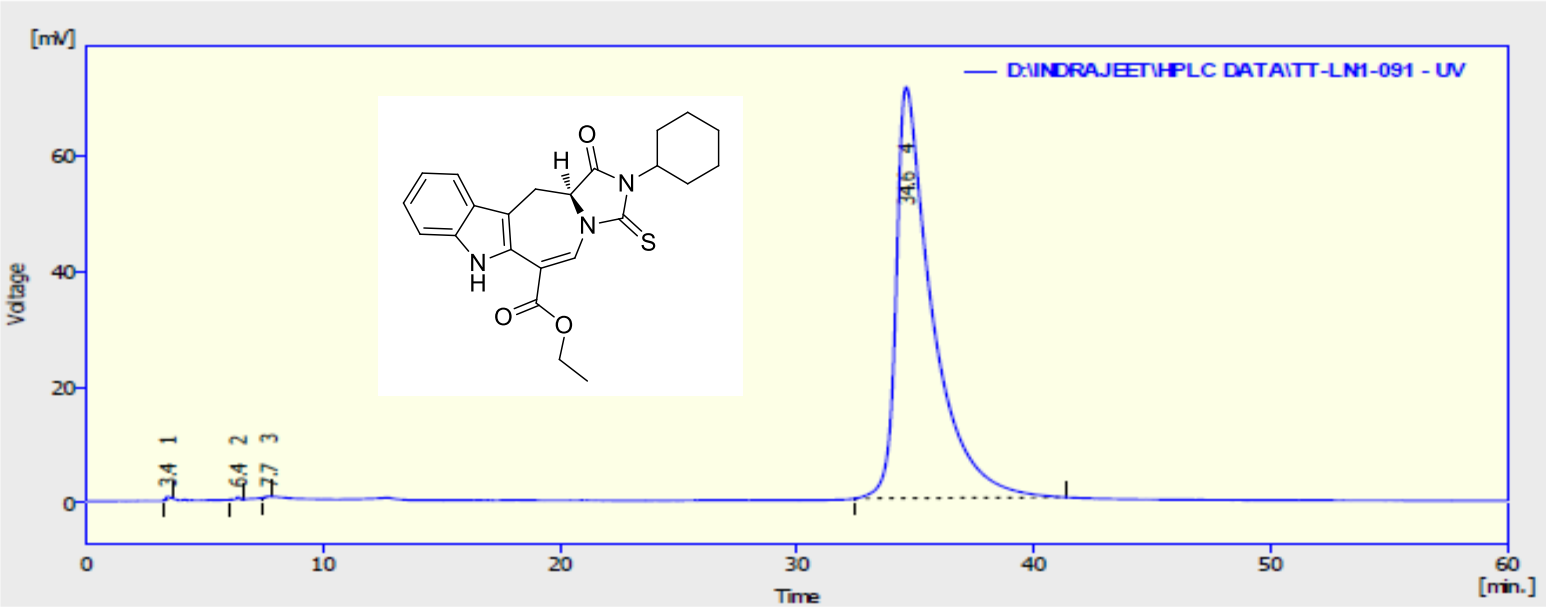
EI-LRMS of compound **7s**



EI-HRMS of compound **7s**



IR spectrum of compound **7s**

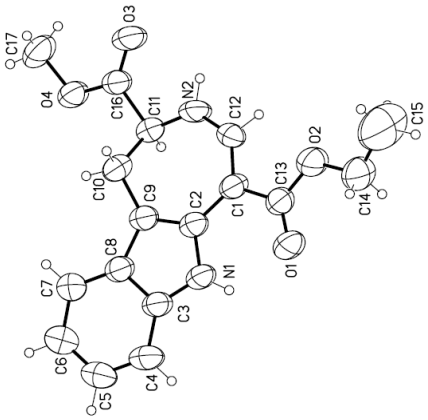


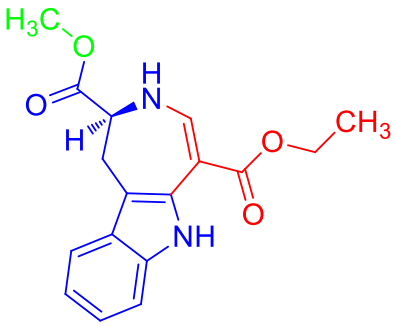
Result Table (Uncal - D:\INDRAJEET\HPLC DATA\TT-LN1-091 - UV)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]
1	3.436	5.502	0.497	0.1	0.7
2	6.400	3.800	0.439	0.1	0.6
3	7.696	1.766	0.152	0.0	0.2
4	34.624	7526.732	71.195	99.9	98.5
	Total	7537.800	72.282	100.0	100.0

HPLC of compound **7s**

VIII. X-ray crystal structure of compound 5





ORTEP diagram of compound 5. Atomic displacement ellipsoids are drawn at the 50% probability level

CCDC no. of **5**: 1033162

Table 1. Crystal data and structure refinement for cu_140110_0m.

Identification code	cu_140110_0m
Empirical formula	C17 H18 N2 O4
Formula weight	314.33
Temperature	296(2) K
Wavelength	1.54178 Å
Crystal system	Triclinic
Space group	P 1
Unit cell dimensions	a = 7.9602(7) Å α= 94.238(5)°. b = 8.6689(7) Å β= 97.302(6)°. c = 11.7774(11) Å γ = 96.834(5)°.
Volume	797.12(12) Å ^{3}
Z	2

Density (calculated)	1.310 Mg/m^3
Absorption coefficient	0.778 mm^{-1}
F(000)	332
Crystal size	0.25 x 0.15 x 0.12 mm^3
Theta range for data collection	5.160 to 66.403°.
Index ranges	-9<=h<=9, -10<=k<=10, -13<=l<=13
Reflections collected	8958
Independent reflections	4412 [R(int) = 0.0458]
Completeness to theta = 67.679°	93.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9492 and 0.6197
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4412 / 3 / 420
Goodness-of-fit on F^2	1.096
Final R indices [I>2sigma(I)]	R1 = 0.0744, wR2 = 0.1953
R indices (all data)	R1 = 0.0779, wR2 = 0.2031
Absolute structure parameter	0.3(4)
Extinction coefficient	n/a
Largest diff. peak and hole	0.350 and -0.345 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for cu_140110_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	9366(6)	470(6)	2219(4)	53(1)
C(2)	9544(5)	1576(5)	3223(4)	52(1)
C(3)	10412(6)	3859(6)	4287(5)	58(1)
C(4)	11152(7)	5370(6)	4723(6)	70(1)
C(5)	10941(8)	5870(7)	5815(6)	75(2)
C(6)	10013(8)	4909(7)	6483(6)	78(2)
C(7)	9264(7)	3421(7)	6055(5)	69(1)
C(8)	9474(6)	2874(6)	4954(5)	56(1)

C(9)	8926(5)	1431(5)	4244(4)	52(1)
C(10)	7800(6)	77(6)	4572(5)	60(1)
C(11)	8308(6)	-1512(5)	4239(4)	54(1)
C(12)	8700(8)	-1065(7)	2192(5)	66(1)
C(13)	9839(6)	1002(7)	1143(5)	59(1)
C(14)	9885(10)	387(10)	-830(6)	89(2)
C(15)	9170(20)	-760(20)	-1698(9)	188(7)
C(16)	7110(6)	-2763(6)	4689(5)	59(1)
C(17)	5895(9)	-3633(10)	6267(6)	89(2)
C(18)	3906(5)	959(6)	3257(4)	51(1)
C(19)	3740(5)	-128(5)	2238(4)	50(1)
C(20)	3222(6)	-2482(6)	1220(5)	58(1)
C(21)	2757(9)	-4050(7)	830(6)	76(2)
C(22)	2842(9)	-4498(7)	-287(6)	79(2)
C(23)	3369(9)	-3437(8)	-1044(6)	78(2)
C(24)	3807(8)	-1863(7)	-670(5)	70(1)
C(25)	3717(6)	-1366(6)	474(5)	57(1)
C(26)	4010(6)	120(6)	1139(4)	53(1)
C(27)	4544(7)	1650(5)	692(4)	58(1)
C(28)	5933(6)	2672(5)	1516(4)	51(1)
C(29)	4603(6)	2493(6)	3314(4)	53(1)
C(30)	3333(6)	406(6)	4310(4)	55(1)
C(31)	3119(9)	1058(9)	6268(6)	80(2)
C(32)	3732(15)	2341(12)	7165(7)	113(3)
C(33)	6764(6)	4066(6)	969(5)	58(1)
C(34)	7849(11)	4945(10)	-648(7)	103(3)
N(1)	10432(5)	3065(5)	3262(4)	60(1)
N(2)	8211(8)	-1894(6)	3019(5)	81(2)
N(3)	3244(6)	-1719(5)	2276(4)	61(1)
N(4)	5358(6)	3281(5)	2533(4)	61(1)
O(1)	10497(6)	2321(5)	1034(4)	74(1)
O(2)	9460(6)	-69(6)	246(4)	78(1)
O(3)	6291(7)	-3859(5)	4091(4)	90(2)
O(4)	7043(5)	-2506(5)	5786(3)	70(1)

O(5)	2721(6)	-915(5)	4421(4)	75(1)
O(6)	3589(5)	1509(5)	5199(4)	70(1)
O(7)	7214(7)	5322(5)	1477(4)	87(1)
O(8)	6999(5)	3702(5)	-88(4)	73(1)

Table 3. Bond lengths [Å] and angles [°] for cu_140110_0m.

C(1)-C(12)	1.369(8)
C(1)-C(2)	1.448(7)
C(1)-C(13)	1.457(7)
C(2)-C(9)	1.365(7)
C(2)-N(1)	1.391(6)
C(3)-N(1)	1.347(7)
C(3)-C(4)	1.403(8)
C(3)-C(8)	1.415(7)
C(4)-C(5)	1.364(9)
C(4)-H(4)	0.9300
C(5)-C(6)	1.399(10)
C(5)-H(5)	0.9300
C(6)-C(7)	1.385(8)
C(6)-H(6)	0.9300
C(7)-C(8)	1.385(8)
C(7)-H(7)	0.9300
C(8)-C(9)	1.441(7)
C(9)-C(10)	1.495(6)
C(10)-C(11)	1.519(6)
C(10)-H(10A)	0.9700
C(10)-H(10B)	0.9700
C(11)-N(2)	1.441(7)
C(11)-C(16)	1.527(6)
C(11)-H(11)	0.9800
C(12)-N(2)	1.322(8)
C(12)-H(12)	0.9300
C(13)-O(1)	1.223(7)

C(13)-O(2)	1.332(8)
C(14)-C(15)	1.385(14)
C(14)-O(2)	1.425(8)
C(14)-H(14A)	0.9700
C(14)-H(14B)	0.9700
C(15)-H(15A)	0.9600
C(15)-H(15B)	0.9600
C(15)-H(15C)	0.9600
C(16)-O(3)	1.209(7)
C(16)-O(4)	1.304(7)
C(17)-O(4)	1.453(7)
C(17)-H(17A)	0.9600
C(17)-H(17B)	0.9600
C(17)-H(17C)	0.9600
C(18)-C(29)	1.372(7)
C(18)-C(19)	1.452(7)
C(18)-C(30)	1.468(7)
C(19)-C(26)	1.367(7)
C(19)-N(3)	1.395(6)
C(20)-N(3)	1.361(8)
C(20)-C(21)	1.393(8)
C(20)-C(25)	1.409(7)
C(21)-C(22)	1.356(10)
C(21)-H(21)	0.9300
C(22)-C(23)	1.393(10)
C(22)-H(22)	0.9300
C(23)-C(24)	1.392(9)
C(23)-H(23)	0.9300
C(24)-C(25)	1.396(8)
C(24)-H(24)	0.9300
C(25)-C(26)	1.436(7)
C(26)-C(27)	1.497(6)
C(27)-C(28)	1.523(6)

C(27)-H(27A)	0.9700
C(27)-H(27B)	0.9700
C(28)-N(4)	1.424(7)
C(28)-C(33)	1.532(6)
C(28)-H(28)	0.9800
C(29)-N(4)	1.344(6)
C(29)-H(29)	0.9300
C(30)-O(5)	1.213(6)
C(30)-O(6)	1.342(7)
C(31)-O(6)	1.427(7)
C(31)-C(32)	1.469(11)
C(31)-H(31A)	0.9700
C(31)-H(31B)	0.9700
C(32)-H(32A)	0.9600
C(32)-H(32B)	0.9600
C(32)-H(32C)	0.9600
C(33)-O(7)	1.195(7)
C(33)-O(8)	1.303(7)
C(34)-O(8)	1.449(7)
C(34)-H(34A)	0.9600
C(34)-H(34B)	0.9600
C(34)-H(34C)	0.9600
N(1)-H(1)	0.8600
N(2)-H(2)	0.8600
N(3)-H(3)	0.8600
N(4)-H(4A)	0.8600
C(12)-C(1)-C(2)	123.9(5)
C(12)-C(1)-C(13)	116.6(5)
C(2)-C(1)-C(13)	119.4(4)
C(9)-C(2)-N(1)	108.1(4)
C(9)-C(2)-C(1)	130.0(4)
N(1)-C(2)-C(1)	122.0(4)
N(1)-C(3)-C(4)	130.8(5)
N(1)-C(3)-C(8)	107.9(4)

C(4)-C(3)-C(8)	121.3(5)
C(5)-C(4)-C(3)	118.0(6)
C(5)-C(4)-H(4)	121.0
C(3)-C(4)-H(4)	121.0
C(4)-C(5)-C(6)	121.2(6)
C(4)-C(5)-H(5)	119.4
C(6)-C(5)-H(5)	119.4
C(7)-C(6)-C(5)	121.1(6)
C(7)-C(6)-H(6)	119.4
C(5)-C(6)-H(6)	119.4
C(8)-C(7)-C(6)	119.0(6)
C(8)-C(7)-H(7)	120.5
C(6)-C(7)-H(7)	120.5
C(7)-C(8)-C(3)	119.3(5)
C(7)-C(8)-C(9)	134.6(5)
C(3)-C(8)-C(9)	106.1(5)
C(2)-C(9)-C(8)	107.6(4)
C(2)-C(9)-C(10)	127.4(5)
C(8)-C(9)-C(10)	124.8(5)
C(9)-C(10)-C(11)	114.8(4)
C(9)-C(10)-H(10A)	108.6
C(11)-C(10)-H(10A)	108.6
C(9)-C(10)-H(10B)	108.6
C(11)-C(10)-H(10B)	108.6
H(10A)-C(10)-H(10B)	107.5
N(2)-C(11)-C(10)	114.5(4)
N(2)-C(11)-C(16)	107.1(4)
C(10)-C(11)-C(16)	108.7(4)
N(2)-C(11)-H(11)	108.8
C(10)-C(11)-H(11)	108.8
C(16)-C(11)-H(11)	108.8
N(2)-C(12)-C(1)	130.8(5)
N(2)-C(12)-H(12)	114.6
C(1)-C(12)-H(12)	114.6

O(1)-C(13)-O(2)	121.0(5)
O(1)-C(13)-C(1)	124.5(5)
O(2)-C(13)-C(1)	114.5(5)
C(15)-C(14)-O(2)	109.8(8)
C(15)-C(14)-H(14A)	109.7
O(2)-C(14)-H(14A)	109.7
C(15)-C(14)-H(14B)	109.7
O(2)-C(14)-H(14B)	109.7
H(14A)-C(14)-H(14B)	108.2
C(14)-C(15)-H(15A)	109.5
C(14)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
C(14)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
O(3)-C(16)-O(4)	123.5(5)
O(3)-C(16)-C(11)	123.8(5)
O(4)-C(16)-C(11)	112.7(4)
O(4)-C(17)-H(17A)	109.5
O(4)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5
O(4)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
C(29)-C(18)-C(19)	124.0(5)
C(29)-C(18)-C(30)	116.8(5)
C(19)-C(18)-C(30)	119.2(4)
C(26)-C(19)-N(3)	108.1(4)
C(26)-C(19)-C(18)	130.6(4)
N(3)-C(19)-C(18)	121.3(4)
N(3)-C(20)-C(21)	130.7(5)
N(3)-C(20)-C(25)	107.8(4)
C(21)-C(20)-C(25)	121.4(6)

C(22)-C(21)-C(20)	118.4(6)
C(22)-C(21)-H(21)	120.8
C(20)-C(21)-H(21)	120.8
C(21)-C(22)-C(23)	121.9(6)
C(21)-C(22)-H(22)	119.1
C(23)-C(22)-H(22)	119.1
C(24)-C(23)-C(22)	120.3(6)
C(24)-C(23)-H(23)	119.9
C(22)-C(23)-H(23)	119.9
C(23)-C(24)-C(25)	119.1(6)
C(23)-C(24)-H(24)	120.5
C(25)-C(24)-H(24)	120.5
C(24)-C(25)-C(20)	118.9(5)
C(24)-C(25)-C(26)	134.5(5)
C(20)-C(25)-C(26)	106.6(5)
C(19)-C(26)-C(25)	107.7(4)
C(19)-C(26)-C(27)	127.0(5)
C(25)-C(26)-C(27)	125.2(5)
C(26)-C(27)-C(28)	112.6(4)
C(26)-C(27)-H(27A)	109.1
C(28)-C(27)-H(27A)	109.1
C(26)-C(27)-H(27B)	109.1
C(28)-C(27)-H(27B)	109.1
H(27A)-C(27)-H(27B)	107.8
N(4)-C(28)-C(27)	113.8(4)
N(4)-C(28)-C(33)	106.9(4)
C(27)-C(28)-C(33)	113.0(4)
N(4)-C(28)-H(28)	107.6
C(27)-C(28)-H(28)	107.6
C(33)-C(28)-H(28)	107.6
N(4)-C(29)-C(18)	130.1(5)
N(4)-C(29)-H(29)	115.0
C(18)-C(29)-H(29)	115.0
O(5)-C(30)-O(6)	120.8(5)

O(5)-C(30)-C(18)	125.7(5)
O(6)-C(30)-C(18)	113.6(4)
O(6)-C(31)-C(32)	109.1(6)
O(6)-C(31)-H(31A)	109.9
C(32)-C(31)-H(31A)	109.9
O(6)-C(31)-H(31B)	109.9
C(32)-C(31)-H(31B)	109.9
H(31A)-C(31)-H(31B)	108.3
C(31)-C(32)-H(32A)	109.5
C(31)-C(32)-H(32B)	109.5
H(32A)-C(32)-H(32B)	109.5
C(31)-C(32)-H(32C)	109.5
H(32A)-C(32)-H(32C)	109.5
H(32B)-C(32)-H(32C)	109.5
O(7)-C(33)-O(8)	124.2(5)
O(7)-C(33)-C(28)	123.4(5)
O(8)-C(33)-C(28)	112.3(4)
O(8)-C(34)-H(34A)	109.5
O(8)-C(34)-H(34B)	109.5
H(34A)-C(34)-H(34B)	109.5
O(8)-C(34)-H(34C)	109.5
H(34A)-C(34)-H(34C)	109.5
H(34B)-C(34)-H(34C)	109.5
C(3)-N(1)-C(2)	110.4(4)
C(3)-N(1)-H(1)	124.8
C(2)-N(1)-H(1)	124.8
C(12)-N(2)-C(11)	131.7(5)
C(12)-N(2)-H(2)	114.1
C(11)-N(2)-H(2)	114.1
C(20)-N(3)-C(19)	109.8(4)
C(20)-N(3)-H(3)	125.1
C(19)-N(3)-H(3)	125.1
C(29)-N(4)-C(28)	128.3(4)
C(29)-N(4)-H(4A)	115.9

C(28)-N(4)-H(4A)	115.9
C(13)-O(2)-C(14)	117.0(5)
C(16)-O(4)-C(17)	115.6(5)
C(30)-O(6)-C(31)	117.3(5)
C(33)-O(8)-C(34)	115.6(5)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for cu_140110_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	51(2)	51(3)	55(3)	9(2)	6(2)	-6(2)
C(2)	48(2)	37(2)	70(3)	9(2)	8(2)	0(2)
C(3)	55(2)	43(2)	72(3)	5(2)	8(2)	-2(2)
C(4)	70(3)	44(3)	91(4)	8(3)	9(3)	-6(2)
C(5)	76(3)	53(3)	90(4)	-4(3)	5(3)	1(2)
C(6)	83(4)	60(3)	85(5)	-11(3)	13(3)	-2(3)
C(7)	73(3)	58(3)	73(4)	0(2)	19(3)	-4(2)
C(8)	51(2)	49(2)	67(3)	6(2)	12(2)	1(2)
C(9)	50(2)	42(2)	63(3)	9(2)	10(2)	1(2)
C(10)	53(2)	53(3)	75(3)	10(2)	20(2)	-3(2)
C(11)	55(2)	43(2)	63(3)	6(2)	12(2)	-7(2)
C(12)	77(3)	59(3)	56(3)	-4(2)	15(2)	-14(2)
C(13)	54(2)	64(3)	59(3)	10(2)	10(2)	-1(2)
C(14)	100(5)	107(5)	55(4)	10(3)	15(3)	-12(4)
C(15)	225(15)	233(17)	73(6)	6(8)	14(7)	-97(12)
C(16)	61(2)	51(2)	62(3)	5(2)	12(2)	-11(2)
C(17)	92(4)	99(5)	74(4)	21(4)	28(3)	-27(4)
C(18)	47(2)	48(2)	58(3)	10(2)	9(2)	-1(2)
C(19)	48(2)	43(2)	55(3)	5(2)	4(2)	-5(2)
C(20)	62(3)	44(2)	67(3)	7(2)	10(2)	-2(2)
C(21)	87(4)	46(3)	91(5)	8(3)	11(3)	-7(2)
C(22)	91(4)	47(3)	94(5)	-6(3)	10(3)	-2(3)
C(23)	87(4)	70(4)	72(4)	-13(3)	14(3)	-5(3)

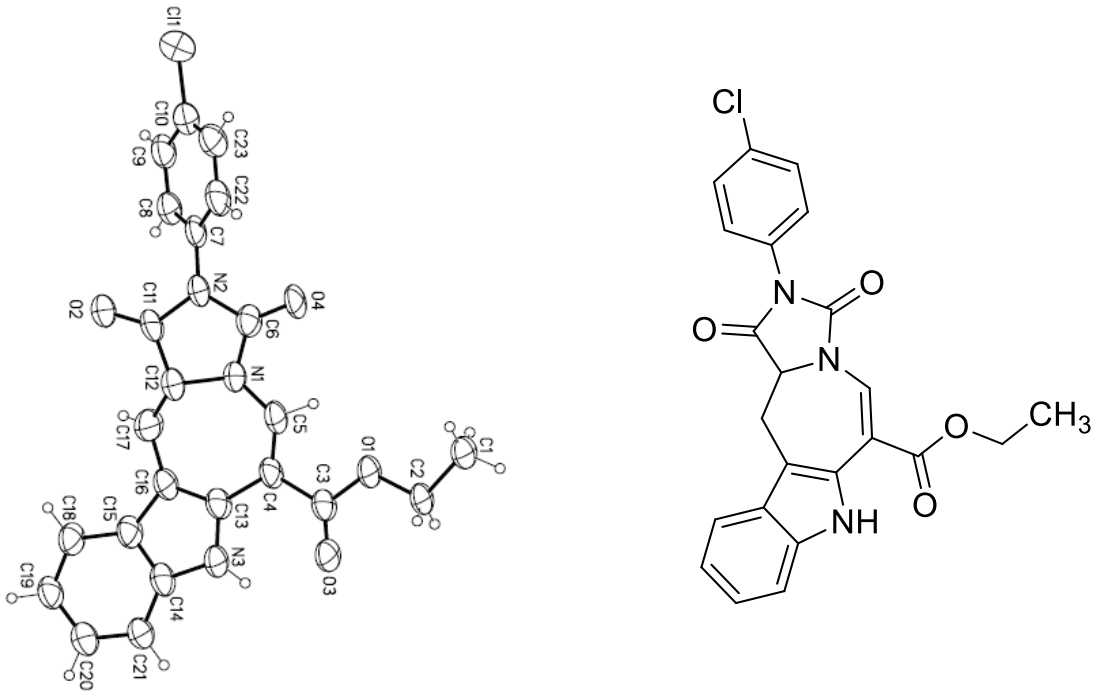
C(24)	76(3)	62(3)	68(4)	0(2)	15(3)	-7(3)
C(25)	52(2)	46(2)	68(3)	4(2)	7(2)	-8(2)
C(26)	56(2)	45(2)	55(3)	7(2)	4(2)	-4(2)
C(27)	63(2)	50(2)	57(3)	7(2)	6(2)	-12(2)
C(28)	51(2)	45(2)	55(3)	8(2)	6(2)	-4(2)
C(29)	55(2)	47(2)	57(3)	4(2)	10(2)	-3(2)
C(30)	53(2)	51(2)	59(3)	6(2)	10(2)	-3(2)
C(31)	93(4)	84(4)	60(4)	11(3)	24(3)	-8(3)
C(32)	158(8)	106(6)	65(5)	-2(4)	19(5)	-15(5)
C(33)	57(2)	54(3)	58(3)	7(2)	9(2)	-14(2)
C(34)	114(5)	104(6)	87(5)	31(4)	35(4)	-41(4)
N(1)	62(2)	45(2)	70(3)	12(2)	15(2)	-9(2)
N(2)	117(4)	46(2)	73(3)	-3(2)	29(3)	-30(2)
N(3)	68(2)	43(2)	70(3)	12(2)	11(2)	-8(2)
N(4)	76(2)	44(2)	59(3)	3(2)	15(2)	-12(2)
O(1)	86(3)	64(2)	72(3)	18(2)	24(2)	-12(2)
O(2)	93(3)	80(3)	56(2)	3(2)	20(2)	-21(2)
O(3)	115(3)	67(3)	74(3)	-2(2)	25(2)	-42(2)
O(4)	76(2)	65(2)	63(2)	12(2)	14(2)	-21(2)
O(5)	91(3)	57(2)	76(3)	12(2)	27(2)	-14(2)
O(6)	84(2)	64(2)	62(2)	8(2)	22(2)	-8(2)
O(7)	117(4)	57(2)	81(3)	0(2)	30(3)	-32(2)
O(8)	80(2)	69(2)	69(3)	11(2)	27(2)	-17(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å ^{2}x 10 ^{3}) for cu_140110_0m.

	x	y	z	U(eq)
H(4)	11768	6013	4281	84
H(5)	11422	6868	6121	90
H(6)	9897	5276	7228	93
H(7)	8631	2799	6500	82

H(10A)	6641	113	4214	72
H(10B)	7803	190	5398	72
H(11)	9478	-1553	4601	65
H(12)	8568	-1618	1472	79
H(14A)	9457	1364	-983	107
H(14B)	11117	544	-806	107
H(15A)	7966	-722	-1855	282
H(15B)	9376	-1762	-1461	282
H(15C)	9686	-578	-2380	282
H(17A)	6219	-4656	6135	134
H(17B)	4746	-3621	5905	134
H(17C)	5960	-3365	7078	134
H(21)	2398	-4772	1323	91
H(22)	2538	-5543	-555	95
H(23)	3429	-3783	-1803	94
H(24)	4155	-1152	-1174	84
H(27A)	3557	2203	558	70
H(27B)	4958	1458	-39	70
H(28)	6827	2028	1748	61
H(29)	4544	3079	3998	64
H(31A)	3619	130	6466	95
H(31B)	1886	822	6205	95
H(32A)	3262	3265	6952	169
H(32B)	4956	2535	7249	169
H(32C)	3379	2062	7880	169
H(34A)	8954	5300	-221	155
H(34B)	7179	5795	-680	155
H(34C)	7978	4563	-1414	155
H(1)	10923	3428	2710	71
H(2)	7752	-2831	2786	97
H(3)	2991	-2154	2876	73
H(4A)	5512	4280	2665	73

IX. X-ray crystal structure of 7b



ORTEP diagram of compound 7b. Atomic displacement ellipsoids are drawn at the 50% probability level

CCDC no. of **7b**: 1028024

Table 1. Crystal data and structure refinement for 7b.

Identification code	cu_140906lt_0m
Empirical formula	C23 H16 Cl N3 O4
Formula weight	433.84
Temperature	100(2) K
Wavelength	1.54178 Å
Crystal system	Monoclinic
Space group	P 21/c

Unit cell dimensions	a = 6.6540(3) Å α= 90°. b = 12.6487(7) Å β= 97.152(3)°. c = 23.3926(13) Å γ = 90°.
Volume	1953.51(18) $Å^3$
Z	4
Density (calculated)	1.475 Mg/m^3
Absorption coefficient	2.058 mm^{-1}
F(000)	896
Crystal size	0.30 x 0.01 x 0.01 mm^3
Theta range for data collection	3.809 to 66.826°.
Index ranges	-7<=h<=4, -13<=k<=15, -27<=l<=27
Reflections collected	12712
Independent reflections	3385 [R(int) = 0.0726]
Completeness to theta = 67.679°	96.2 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9492 and 0.4939
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3385 / 0 / 281
Goodness-of-fit on F^2	1.132
Final R indices [I>2sigma(I)]	R1 = 0.0852, wR2 = 0.2415
R indices (all data)	R1 = 0.1359, wR2 = 0.2862
Extinction coefficient	n/a
Largest diff. peak and hole	0.579 and -0.415 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for CU_140906LT_0M. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	1805(3)	-4503(2)	10614(1)	95(1)
O(1)	2237(4)	1255(3)	6995(1)	54(1)
O(2)	7333(5)	-581(3)	9822(2)	76(1)
O(3)	4614(5)	2442(3)	6855(2)	56(1)
O(4)	1963(4)	-1036(3)	8426(1)	54(1)

N(1)	4702(6)	93(4)	8484(2)	57(1)
N(2)	4454(6)	-1025(3)	9210(2)	53(1)
N(3)	7940(6)	2591(3)	7627(2)	53(1)
C(1)	-972(7)	1051(4)	6451(2)	55(1)
C(2)	968(6)	1676(4)	6498(2)	53(1)
C(3)	4037(7)	1741(4)	7143(2)	47(1)
C(4)	5136(6)	1312(4)	7688(2)	47(1)
C(5)	4160(7)	574(4)	7958(2)	50(1)
C(6)	3505(7)	-694(4)	8673(2)	50(1)
C(7)	3752(7)	-1874(4)	9533(2)	49(1)
C(8)	5045(7)	-2713(4)	9682(2)	53(1)
C(9)	4464(8)	-3529(4)	10013(2)	60(1)
C(10)	2528(8)	-3485(5)	10179(2)	66(2)
C(11)	6196(8)	-447(5)	9385(3)	72(2)
C(12)	6317(9)	412(6)	8941(3)	91(2)
C(13)	7127(6)	1756(4)	7900(2)	48(1)
C(14)	9819(7)	2840(4)	7904(2)	50(1)
C(15)	10174(7)	2157(4)	8379(2)	52(1)
C(16)	8454(7)	1468(5)	8368(2)	57(1)
C(17)	8276(9)	567(7)	8774(3)	98(3)
C(18)	12008(7)	2263(5)	8751(2)	57(1)
C(19)	13405(7)	3001(4)	8616(2)	54(1)
C(20)	13033(7)	3638(4)	8132(2)	56(1)
C(21)	11229(7)	3583(4)	7776(2)	56(1)
C(22)	1821(7)	-1845(5)	9703(2)	59(1)
C(23)	1210(8)	-2667(5)	10029(2)	64(2)

Table 3. Bond lengths [Å] and angles [°] for CU_140906LT_0M.

O(1)-C(3)	1.352(6)
O(1)-C(2)	1.451(5)
O(2)-C(11)	1.205(6)
O(3)-C(3)	1.206(6)
O(4)-C(6)	1.193(6)
N(1)-C(5)	1.380(6)
N(1)-C(6)	1.382(7)
N(1)-C(12)	1.475(6)
N(2)-C(11)	1.389(6)
N(2)-C(6)	1.398(6)
N(2)-C(7)	1.426(7)
N(3)-C(14)	1.372(6)
N(3)-C(13)	1.379(6)
N(3)-H(5)	0.8800
C(1)-C(2)	1.506(7)
C(1)-H(1)	0.9800
C(1)-H(13)	0.9800
C(1)-H(14)	0.9800
C(2)-H(11)	0.9900
C(2)-H(12)	0.9900
C(3)-C(4)	1.490(7)
C(4)-C(5)	1.339(7)
C(4)-C(13)	1.467(6)
C(5)-H(4)	0.9500
C(7)-C(8)	1.383(7)
C(7)-C(22)	1.391(6)
C(8)-C(9)	1.374(7)
C(8)-H(2)	0.9500
C(9)-C(10)	1.392(7)
C(9)-H(3)	0.9500
C(10)-C(23)	1.373(8)
C(11)-C(12)	1.511(8)
C(12)-C(17)	1.421(9)

C(13)-C(16)	1.369(6)
C(14)-C(21)	1.387(7)
C(14)-C(15)	1.404(7)
C(15)-C(18)	1.413(6)
C(15)-C(16)	1.436(7)
C(16)-C(17)	1.497(8)
C(17)-H(6)	0.9500
C(18)-C(19)	1.381(7)
C(18)-H(10)	0.9500
C(19)-C(20)	1.386(8)
C(19)-H(9)	0.9500
C(20)-C(21)	1.374(7)
C(20)-H(8)	0.9500
C(21)-H(7)	0.9500
C(22)-C(23)	1.381(8)
C(22)-H(16)	0.9500
C(23)-H(15)	0.9500
C(3)-O(1)-C(2)	116.6(4)
C(5)-N(1)-C(6)	120.4(4)
C(5)-N(1)-C(12)	127.8(4)
C(6)-N(1)-C(12)	111.2(4)
C(11)-N(2)-C(6)	112.0(4)
C(11)-N(2)-C(7)	123.8(4)
C(6)-N(2)-C(7)	124.2(4)
C(14)-N(3)-C(13)	110.1(4)
C(14)-N(3)-H(5)	124.9
C(13)-N(3)-H(5)	124.9
C(2)-C(1)-H(1)	109.5
C(2)-C(1)-H(13)	109.5
H(1)-C(1)-H(13)	109.5
C(2)-C(1)-H(14)	109.5
H(1)-C(1)-H(14)	109.5
H(13)-C(1)-H(14)	109.5

O(1)-C(2)-C(1)	106.0(4)
O(1)-C(2)-H(11)	110.5
C(1)-C(2)-H(11)	110.5
O(1)-C(2)-H(12)	110.5
C(1)-C(2)-H(12)	110.5
H(11)-C(2)-H(12)	108.7
O(3)-C(3)-O(1)	121.6(4)
O(3)-C(3)-C(4)	126.1(4)
O(1)-C(3)-C(4)	112.2(4)
C(5)-C(4)-C(13)	125.3(4)
C(5)-C(4)-C(3)	116.2(4)
C(13)-C(4)-C(3)	118.4(4)
C(4)-C(5)-N(1)	129.7(4)
C(4)-C(5)-H(4)	115.1
N(1)-C(5)-H(4)	115.1
O(4)-C(6)-N(1)	127.0(5)
O(4)-C(6)-N(2)	126.2(5)
N(1)-C(6)-N(2)	106.8(4)
C(8)-C(7)-C(22)	121.1(5)
C(8)-C(7)-N(2)	118.5(4)
C(22)-C(7)-N(2)	120.4(5)
C(9)-C(8)-C(7)	120.5(5)
C(9)-C(8)-H(2)	119.7
C(7)-C(8)-H(2)	119.7
C(8)-C(9)-C(10)	117.5(5)
C(8)-C(9)-H(3)	121.2
C(10)-C(9)-H(3)	121.2
C(23)-C(10)-C(9)	122.8(5)
C(23)-C(10)-Cl(1)	119.4(5)
C(9)-C(10)-Cl(1)	117.7(5)
O(2)-C(11)-N(2)	126.1(5)
O(2)-C(11)-C(12)	127.0(5)
N(2)-C(11)-C(12)	106.8(4)
C(17)-C(12)-N(1)	116.7(6)
C(17)-C(12)-C(11)	114.7(6)

N(1)-C(12)-C(11)	101.9(4)
C(16)-C(13)-N(3)	108.9(4)
C(16)-C(13)-C(4)	129.3(5)
N(3)-C(13)-C(4)	121.8(4)
N(3)-C(14)-C(21)	130.9(5)
N(3)-C(14)-C(15)	106.7(4)
C(21)-C(14)-C(15)	122.4(4)
C(14)-C(15)-C(18)	118.4(5)
C(14)-C(15)-C(16)	107.7(4)
C(18)-C(15)-C(16)	133.9(5)
C(13)-C(16)-C(15)	106.6(5)
C(13)-C(16)-C(17)	128.0(5)
C(15)-C(16)-C(17)	125.2(4)
C(12)-C(17)-C(16)	115.3(6)
C(12)-C(17)-H(6)	122.3
C(16)-C(17)-H(6)	122.3
C(19)-C(18)-C(15)	118.6(5)
C(19)-C(18)-H(10)	120.7
C(15)-C(18)-H(10)	120.7
C(18)-C(19)-C(20)	121.5(5)
C(18)-C(19)-H(9)	119.3
C(20)-C(19)-H(9)	119.3
C(21)-C(20)-C(19)	121.3(5)
C(21)-C(20)-H(8)	119.4
C(19)-C(20)-H(8)	119.4
C(20)-C(21)-C(14)	117.8(5)
C(20)-C(21)-H(7)	121.1
C(14)-C(21)-H(7)	121.1
C(23)-C(22)-C(7)	118.9(5)
C(23)-C(22)-H(16)	120.6
C(7)-C(22)-H(16)	120.6
C(10)-C(23)-C(22)	119.1(5)
C(10)-C(23)-H(15)	120.5
C(22)-C(23)-H(15)	120.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for CU_140906LT_0M. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

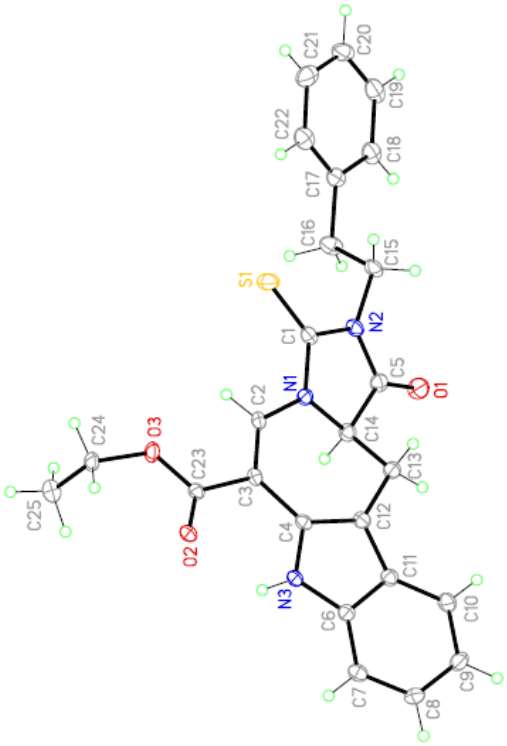
	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	86(1)	108(1)	89(1)	25(1)	-3(1)	-41(1)
O(1)	26(2)	73(2)	58(2)	7(2)	-8(1)	1(2)
O(2)	44(2)	99(3)	76(3)	27(2)	-24(2)	-21(2)
O(3)	28(2)	81(2)	57(2)	8(2)	-4(1)	2(2)
O(4)	24(2)	76(2)	59(2)	-1(2)	-12(1)	-3(2)
N(1)	37(2)	70(3)	58(3)	15(2)	-17(2)	-9(2)
N(2)	29(2)	69(3)	56(2)	4(2)	-12(2)	-6(2)
N(3)	32(2)	69(3)	55(3)	4(2)	-8(2)	1(2)
C(1)	30(2)	73(3)	60(3)	7(3)	-5(2)	3(2)
C(2)	24(2)	74(3)	58(3)	7(3)	-8(2)	-3(2)
C(3)	27(2)	59(3)	54(3)	-1(2)	-2(2)	3(2)
C(4)	24(2)	62(3)	54(3)	-1(2)	-4(2)	0(2)
C(5)	32(2)	58(3)	56(3)	-3(2)	-11(2)	1(2)
C(6)	34(2)	64(3)	51(3)	0(2)	-2(2)	7(2)
C(7)	26(2)	66(3)	51(3)	-2(2)	-10(2)	-4(2)
C(8)	32(2)	65(3)	59(3)	-1(3)	-7(2)	-1(2)
C(9)	40(3)	66(3)	66(3)	2(3)	-15(2)	-3(3)
C(10)	52(3)	88(4)	54(3)	5(3)	-8(2)	-20(3)
C(11)	35(3)	89(4)	82(4)	24(3)	-27(3)	-21(3)
C(12)	53(3)	132(6)	78(4)	41(4)	-34(3)	-42(4)
C(13)	27(2)	59(3)	55(3)	-1(2)	1(2)	3(2)
C(14)	28(2)	66(3)	54(3)	-8(2)	0(2)	1(2)
C(15)	28(2)	75(3)	54(3)	0(3)	1(2)	3(2)
C(16)	26(2)	82(4)	61(3)	7(3)	-4(2)	-7(3)
C(17)	45(3)	151(7)	89(5)	59(5)	-24(3)	-29(4)
C(18)	32(2)	87(4)	52(3)	7(3)	0(2)	2(3)
C(19)	31(2)	73(3)	56(3)	-4(3)	0(2)	-4(2)
C(20)	38(3)	65(3)	62(3)	-5(3)	0(2)	-1(2)

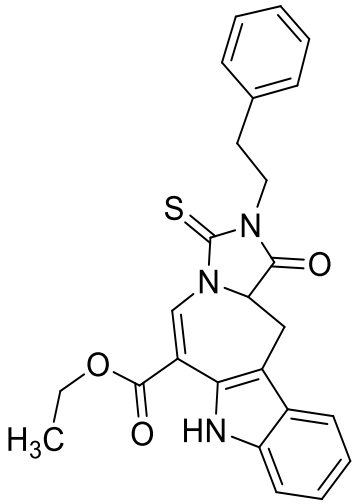
C(21)	33(2)	72(3)	60(3)	6(3)	-2(2)	-4(3)
C(22)	29(2)	82(4)	63(3)	-7(3)	-4(2)	0(3)
C(23)	35(3)	88(4)	67(4)	4(3)	1(2)	-9(3)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å ^{2}x 10 ^{3}) for CU_140906LT_0M.

	x	y	z	U(eq)
H(5)	7341	2916	7319	64
H(1)	-686	307	6376	83
H(13)	-1924	1331	6133	83
H(14)	-1569	1107	6812	83
H(11)	1636	1592	6146	64
H(12)	696	2437	6553	64
H(4)	2905	347	7757	60
H(2)	6345	-2726	9554	64
H(3)	5352	-4101	10125	71
H(6)	9395	130	8910	117
H(10)	12274	1836	9085	69
H(9)	14649	3073	8860	64
H(8)	14044	4121	8045	67
H(7)	10958	4040	7453	67
H(16)	938	-1268	9596	71
H(15)	-103	-2667	10148	77

X. X-ray crystal structure of 7r





ORTEP diagram of compound 7r. Atomic displacement ellipsoids are drawn at the 50% probability level

CCDC no. of **7r**: 1027211

Table 1. Crystal data and structure refinement for 7r.

Identification code	140905LT_a
Empirical formula	C25 H23 N3 O3 S
Formula weight	445.52
Temperature	100(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P 21/c

Unit cell dimensions	a = 12.6842(5) Å α= 90°. b = 20.3035(8) Å β= 99.772(2)°. c = 8.2806(3) Å γ = 90°.
Volume	2101.59(14) $Å^3$
Z	4
Density (calculated)	1.408 Mg/m^3
Absorption coefficient	0.188 mm^{-1}
F(000)	936
Crystal size	0.30 x 0.07 x 0.07 mm^3
Theta range for data collection	1.629 to 26.363°.
Index ranges	-15<=h<=15, -25<=k<=25, -10<=l<=5
Reflections collected	15564
Independent reflections	4291 [R(int) = 0.0368]
Completeness to theta = 25.242°	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9485 and 0.8830
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4291 / 0 / 290
Goodness-of-fit on F^2	1.157
Final R indices [I>2sigma(I)]	R1 = 0.0397, wR2 = 0.1212
R indices (all data)	R1 = 0.0550, wR2 = 0.1514
Extinction coefficient	n/a
Largest diff. peak and hole	0.530 and -0.433 $e.Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å ^{2}x 10^3) for 140905lt_A. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	2846(2)	4271(1)	3163(2)	15(1)
C(2)	3707(2)	4270(1)	790(2)	13(1)
C(3)	3949(1)	4510(1)	-626(2)	13(1)
C(4)	3453(2)	5090(1)	-1509(2)	12(1)
C(5)	1224(2)	4753(1)	2164(2)	16(1)
C(6)	3192(2)	5840(1)	-3535(2)	13(1)
C(7)	3225(2)	6204(1)	-4970(2)	16(1)
C(8)	2530(2)	6729(1)	-5269(2)	17(1)
C(9)	1832(2)	6898(1)	-4186(2)	19(1)
C(10)	1800(2)	6532(1)	-2784(2)	19(1)
C(11)	2486(2)	5986(1)	-2448(2)	15(1)
C(12)	2650(2)	5505(1)	-1168(2)	15(1)
C(13)	2031(2)	5475(1)	209(2)	16(1)
C(14)	1880(2)	4776(1)	790(2)	16(1)
C(15)	1550(2)	4365(1)	5108(2)	17(1)
C(16)	1128(2)	3663(1)	5268(2)	21(1)
C(17)	1144(2)	3468(1)	7038(2)	17(1)
C(18)	227(2)	3505(1)	7745(2)	21(1)
C(19)	258(2)	3321(1)	9371(3)	27(1)
C(20)	1203(2)	3099(1)	10301(2)	28(1)
C(21)	2117(2)	3058(1)	9605(2)	27(1)
C(22)	2090(2)	3248(1)	7988(2)	21(1)
C(23)	4822(2)	4159(1)	-1266(2)	13(1)
C(24)	5832(2)	3155(1)	-1116(2)	18(1)
C(25)	5297(2)	2639(1)	-2284(2)	25(1)
N(1)	2886(1)	4464(1)	1575(2)	14(1)
N(2)	1869(1)	4468(1)	3512(2)	16(1)
N(3)	3774(1)	5297(1)	-2941(2)	14(1)

O(1)	320(1)	4950(1)	2132(2)	22(1)
O(2)	5306(1)	4373(1)	-2301(2)	15(1)
O(3)	5018(1)	3564(1)	-573(2)	19(1)
S(1)	3787(1)	3887(1)	4412(1)	21(1)

Table 3. Bond lengths [Å] and angles [°] for 140905lt_A.

C(1)-N(2)	1.378(2)
C(1)-N(1)	1.381(2)
C(1)-S(1)	1.6391(19)
C(2)-C(3)	1.352(3)
C(2)-N(1)	1.376(2)
C(2)-H(2)	0.9500
C(3)-C(4)	1.470(2)
C(3)-C(23)	1.489(3)
C(4)-N(3)	1.383(2)
C(4)-C(12)	1.388(3)
C(5)-O(1)	1.210(2)
C(5)-N(2)	1.393(2)
C(5)-C(14)	1.521(3)
C(6)-N(3)	1.370(2)
C(6)-C(11)	1.405(3)
C(6)-C(7)	1.406(3)
C(7)-C(8)	1.379(3)
C(7)-H(7)	0.9500
C(8)-C(9)	1.405(3)
C(8)-H(8)	0.9500
C(9)-C(10)	1.385(3)
C(9)-H(9)	0.9500
C(10)-C(11)	1.408(3)
C(10)-H(10)	0.9500
C(11)-C(12)	1.429(3)
C(12)-C(13)	1.492(3)
C(13)-C(14)	1.521(3)
C(13)-H(13A)	0.9900
C(13)-H(13B)	0.9900
C(14)-N(1)	1.473(2)
C(14)-H(14)	1.0000
C(15)-N(2)	1.462(2)

C(15)-C(16)	1.535(3)
C(15)-H(15A)	0.9900
C(15)-H(15B)	0.9900
C(16)-C(17)	1.516(3)
C(16)-H(16A)	0.9900
C(16)-H(16B)	0.9900
C(17)-C(18)	1.389(3)
C(17)-C(22)	1.392(3)
C(18)-C(19)	1.392(3)
C(18)-H(18)	0.9500
C(19)-C(20)	1.386(3)
C(19)-H(19)	0.9500
C(20)-C(21)	1.382(3)
C(20)-H(20)	0.9500
C(21)-C(22)	1.388(3)
C(21)-H(21)	0.9500
C(22)-H(22)	0.9500
C(23)-O(2)	1.216(2)
C(23)-O(3)	1.342(2)
C(24)-O(3)	1.454(2)
C(24)-C(25)	1.507(3)
C(24)-H(24A)	0.9900
C(24)-H(24B)	0.9900
C(25)-H(25A)	0.9800
C(25)-H(25B)	0.9800
C(25)-H(25C)	0.9800
N(3)-H(3)	0.8800
N(2)-C(1)-N(1)	107.29(15)
N(2)-C(1)-S(1)	126.18(14)
N(1)-C(1)-S(1)	126.53(15)
C(3)-C(2)-N(1)	127.50(17)
C(3)-C(2)-H(2)	116.2
N(1)-C(2)-H(2)	116.2

C(2)-C(3)-C(4)	125.37(17)
C(2)-C(3)-C(23)	115.38(16)
C(4)-C(3)-C(23)	119.21(16)
N(3)-C(4)-C(12)	108.81(16)
N(3)-C(4)-C(3)	120.46(17)
C(12)-C(4)-C(3)	130.72(17)
O(1)-C(5)-N(2)	125.49(18)
O(1)-C(5)-C(14)	127.64(17)
N(2)-C(5)-C(14)	106.86(16)
N(3)-C(6)-C(11)	107.60(16)
N(3)-C(6)-C(7)	129.41(18)
C(11)-C(6)-C(7)	122.98(18)
C(8)-C(7)-C(6)	116.66(18)
C(8)-C(7)-H(7)	121.7
C(6)-C(7)-H(7)	121.7
C(7)-C(8)-C(9)	121.78(17)
C(7)-C(8)-H(8)	119.1
C(9)-C(8)-H(8)	119.1
C(10)-C(9)-C(8)	121.02(18)
C(10)-C(9)-H(9)	119.5
C(8)-C(9)-H(9)	119.5
C(9)-C(10)-C(11)	118.93(19)
C(9)-C(10)-H(10)	120.5
C(11)-C(10)-H(10)	120.5
C(6)-C(11)-C(10)	118.61(17)
C(6)-C(11)-C(12)	107.63(17)
C(10)-C(11)-C(12)	133.76(19)
C(4)-C(12)-C(11)	106.47(16)
C(4)-C(12)-C(13)	129.08(17)
C(11)-C(12)-C(13)	124.43(17)
C(12)-C(13)-C(14)	113.00(16)
C(12)-C(13)-H(13A)	109.0
C(14)-C(13)-H(13A)	109.0
C(12)-C(13)-H(13B)	109.0

C(14)-C(13)-H(13B)	109.0
H(13A)-C(13)-H(13B)	107.8
N(1)-C(14)-C(13)	113.26(16)
N(1)-C(14)-C(5)	101.52(14)
C(13)-C(14)-C(5)	112.27(16)
N(1)-C(14)-H(14)	109.8
C(13)-C(14)-H(14)	109.8
C(5)-C(14)-H(14)	109.8
N(2)-C(15)-C(16)	111.36(15)
N(2)-C(15)-H(15A)	109.4
C(16)-C(15)-H(15A)	109.4
N(2)-C(15)-H(15B)	109.4
C(16)-C(15)-H(15B)	109.4
H(15A)-C(15)-H(15B)	108.0
C(17)-C(16)-C(15)	112.23(15)
C(17)-C(16)-H(16A)	109.2
C(15)-C(16)-H(16A)	109.2
C(17)-C(16)-H(16B)	109.2
C(15)-C(16)-H(16B)	109.2
H(16A)-C(16)-H(16B)	107.9
C(18)-C(17)-C(22)	118.78(18)
C(18)-C(17)-C(16)	121.26(19)
C(22)-C(17)-C(16)	119.96(19)
C(17)-C(18)-C(19)	120.4(2)
C(17)-C(18)-H(18)	119.8
C(19)-C(18)-H(18)	119.8
C(20)-C(19)-C(18)	120.3(2)
C(20)-C(19)-H(19)	119.9
C(18)-C(19)-H(19)	119.9
C(21)-C(20)-C(19)	119.72(19)
C(21)-C(20)-H(20)	120.1
C(19)-C(20)-H(20)	120.1
C(20)-C(21)-C(22)	120.0(2)
C(20)-C(21)-H(21)	120.0

C(22)-C(21)-H(21)	120.0
C(21)-C(22)-C(17)	120.9(2)
C(21)-C(22)-H(22)	119.6
C(17)-C(22)-H(22)	119.6
O(2)-C(23)-O(3)	122.93(18)
O(2)-C(23)-C(3)	125.12(17)
O(3)-C(23)-C(3)	111.95(16)
O(3)-C(24)-C(25)	109.26(17)
O(3)-C(24)-H(24A)	109.8
C(25)-C(24)-H(24A)	109.8
O(3)-C(24)-H(24B)	109.8
C(25)-C(24)-H(24B)	109.8
H(24A)-C(24)-H(24B)	108.3
C(24)-C(25)-H(25A)	109.5
C(24)-C(25)-H(25B)	109.5
H(25A)-C(25)-H(25B)	109.5
C(24)-C(25)-H(25C)	109.5
H(25A)-C(25)-H(25C)	109.5
H(25B)-C(25)-H(25C)	109.5
C(2)-N(1)-C(1)	121.51(16)
C(2)-N(1)-C(14)	125.61(15)
C(1)-N(1)-C(14)	111.91(15)
C(1)-N(2)-C(5)	112.31(15)
C(1)-N(2)-C(15)	123.02(16)
C(5)-N(2)-C(15)	124.64(17)
C(6)-N(3)-C(4)	109.49(16)
C(6)-N(3)-H(3)	125.3
C(4)-N(3)-H(3)	125.3
C(23)-O(3)-C(24)	118.12(15)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (Å ^{2}x 10^3) for 140905lt_A. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	16(1)	14(1)	14(1)	-2(1)	6(1)	-2(1)
C(2)	13(1)	13(1)	14(1)	-3(1)	3(1)	2(1)
C(3)	12(1)	12(1)	14(1)	-3(1)	3(1)	0(1)
C(4)	14(1)	13(1)	11(1)	-2(1)	2(1)	-3(1)
C(5)	17(1)	14(1)	18(1)	1(1)	5(1)	-1(1)
C(6)	13(1)	13(1)	13(1)	-1(1)	1(1)	-1(1)
C(7)	15(1)	20(1)	15(1)	0(1)	6(1)	-2(1)
C(8)	16(1)	19(1)	15(1)	4(1)	2(1)	0(1)
C(9)	19(1)	18(1)	20(1)	4(1)	2(1)	4(1)
C(10)	16(1)	23(1)	18(1)	0(1)	6(1)	4(1)
C(11)	15(1)	16(1)	14(1)	-1(1)	2(1)	0(1)
C(12)	15(1)	16(1)	14(1)	-1(1)	3(1)	0(1)
C(13)	17(1)	17(1)	16(1)	2(1)	7(1)	3(1)
C(14)	14(1)	17(1)	16(1)	-1(1)	3(1)	1(1)
C(15)	20(1)	19(1)	15(1)	-1(1)	10(1)	-1(1)
C(16)	24(1)	22(1)	17(1)	-1(1)	6(1)	-8(1)
C(17)	23(1)	12(1)	18(1)	-1(1)	6(1)	-6(1)
C(18)	24(1)	15(1)	24(1)	-2(1)	9(1)	-4(1)
C(19)	41(1)	15(1)	29(1)	-3(1)	21(1)	-5(1)
C(20)	56(2)	13(1)	17(1)	1(1)	10(1)	-5(1)
C(21)	38(1)	15(1)	24(1)	0(1)	-5(1)	-4(1)
C(22)	22(1)	18(1)	24(1)	-4(1)	6(1)	-5(1)
C(23)	14(1)	14(1)	11(1)	-2(1)	1(1)	-1(1)
C(24)	20(1)	16(1)	20(1)	2(1)	10(1)	7(1)
C(25)	31(1)	22(1)	24(1)	-3(1)	8(1)	6(1)
N(1)	14(1)	14(1)	14(1)	2(1)	5(1)	2(1)
N(2)	17(1)	17(1)	15(1)	0(1)	8(1)	1(1)
N(3)	14(1)	16(1)	13(1)	1(1)	6(1)	2(1)
O(1)	17(1)	27(1)	24(1)	5(1)	9(1)	4(1)
O(2)	17(1)	17(1)	13(1)	0(1)	7(1)	2(1)

O(3)	22(1)	14(1)	23(1)	3(1)	13(1)	6(1)
S(1)	18(1)	31(1)	15(1)	4(1)	3(1)	6(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å ^{2}x 10 ^{3}) for 140905lt_A.

	x	y	z	U(eq)
H(2)	4156	3929	1300	16
H(7)	3702	6094	-5697	20
H(8)	2522	6984	-6233	20
H(9)	1375	7269	-4419	23
H(10)	1323	6648	-2061	22
H(13A)	1320	5677	-149	19
H(13B)	2408	5737	1140	19
H(14)	1531	4500	-151	19
H(15A)	986	4686	5252	21
H(15B)	2172	4443	5984	21
H(16A)	386	3634	4664	25
H(16B)	1572	3350	4759	25
H(18)	-423	3656	7115	25
H(19)	-372	3347	9846	32
H(20)	1223	2975	11412	34
H(21)	2765	2900	10234	32
H(22)	2724	3227	7524	25
H(24A)	6274	2940	-160	22
H(24B)	6307	3431	-1670	22
H(25A)	4755	2408	-1784	38
H(25B)	5833	2323	-2525	38
H(25C)	4955	2851	-3303	38
H(3)	4274	5109	-3400	16