Chemistry of Heterocyclic Compounds **2024**, *60*(3/4), 150–155

The synthesis of ethyl 2-amino-1-(aryl)-5-(arylcarbamoyl)-6-oxo-1,6-dihydropyridine-3-carboxylates

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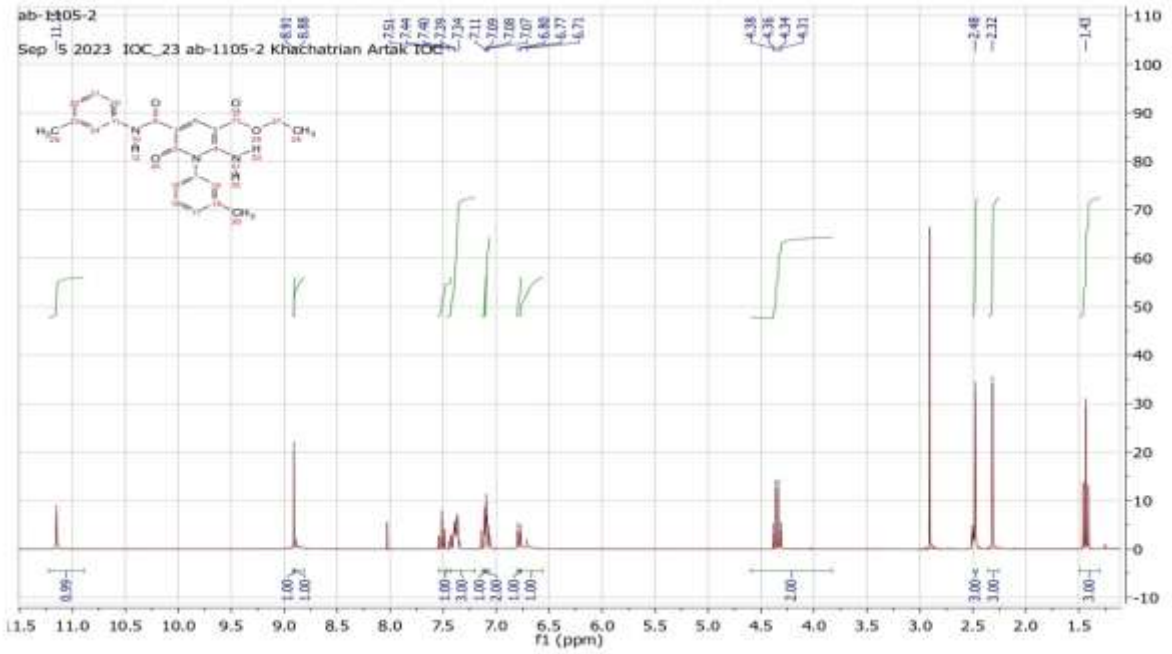
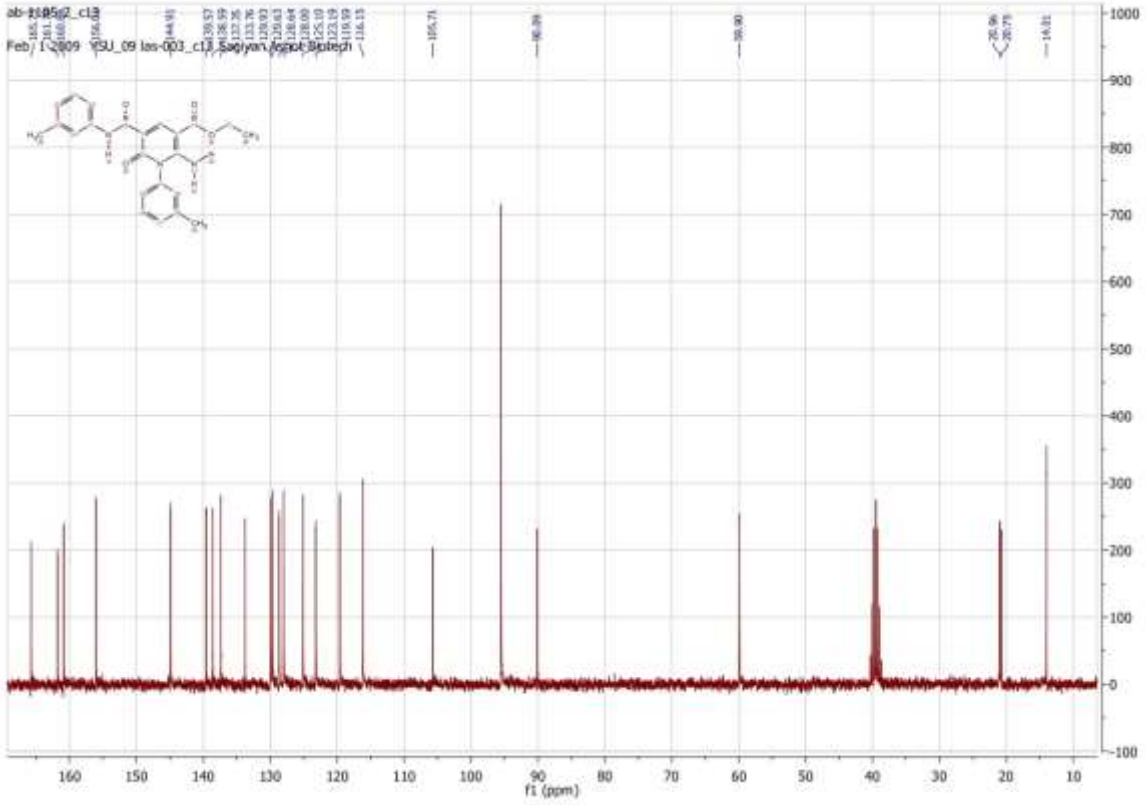
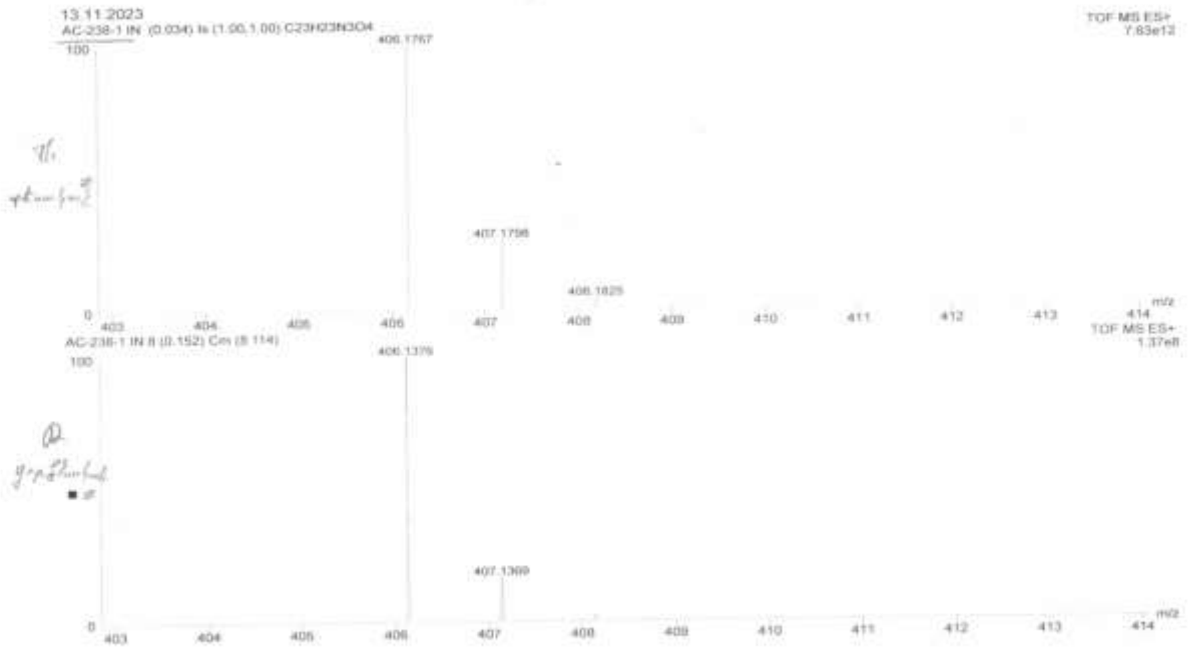
SUPPLEMENTARY INFORMATION

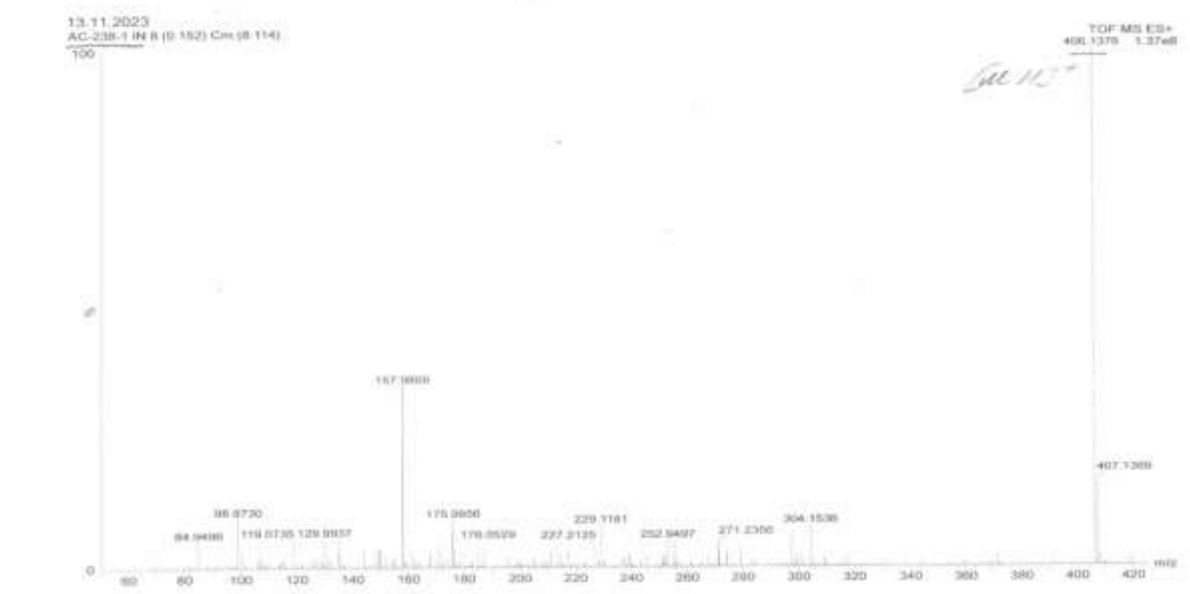
Copies of ^{1}H, ^{13}C NMR spectra, HRMS of new compounds, X-ray compound 5d

Copies of ^{1}H (300 MHz, DMSO-d_6/CCl_4 (1:3)) and ^{13}C (300 MHz, DMSO-d_6/CCl_4 (1:3)) spectra, HRMS of **5a**

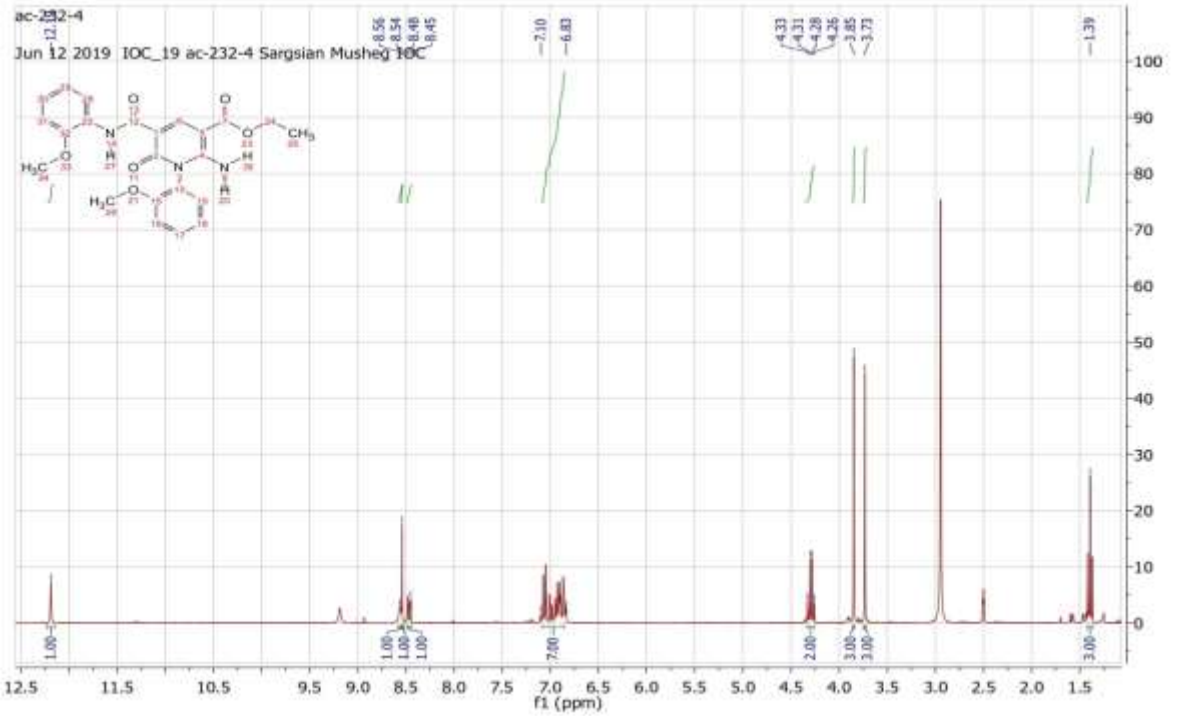


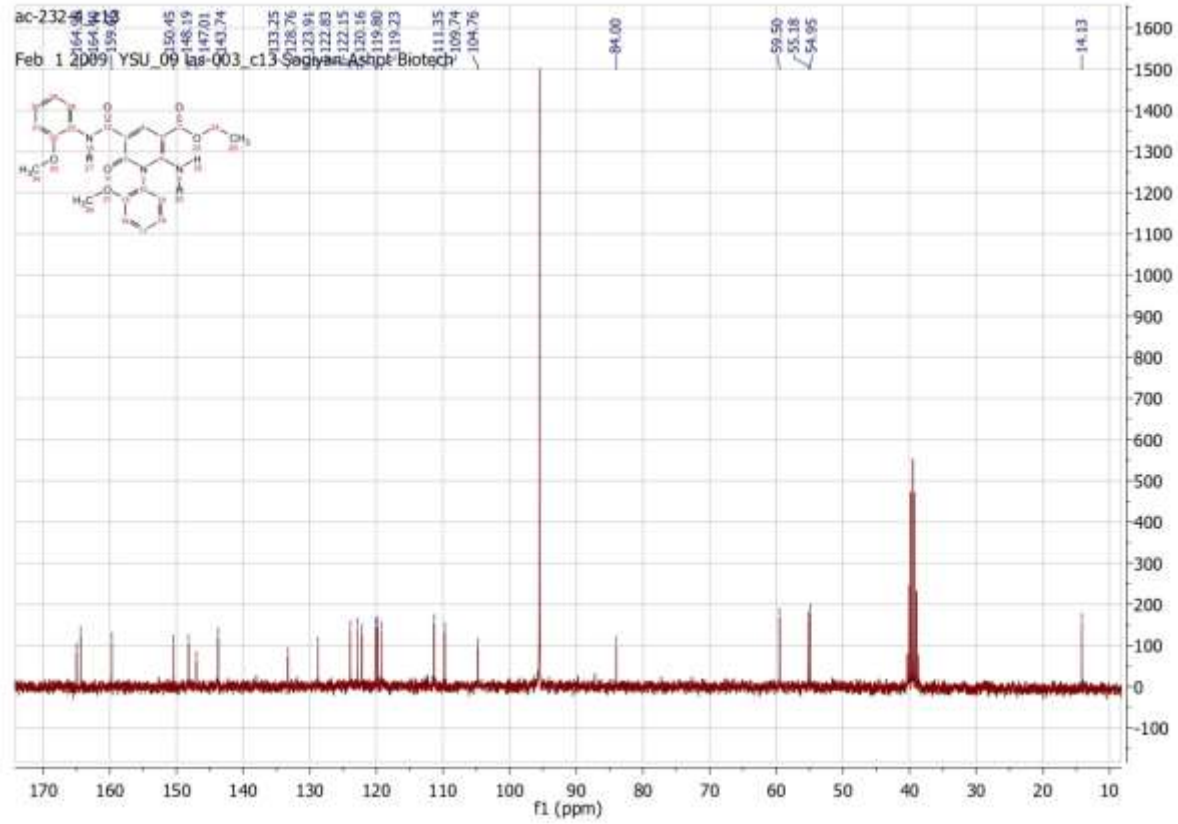


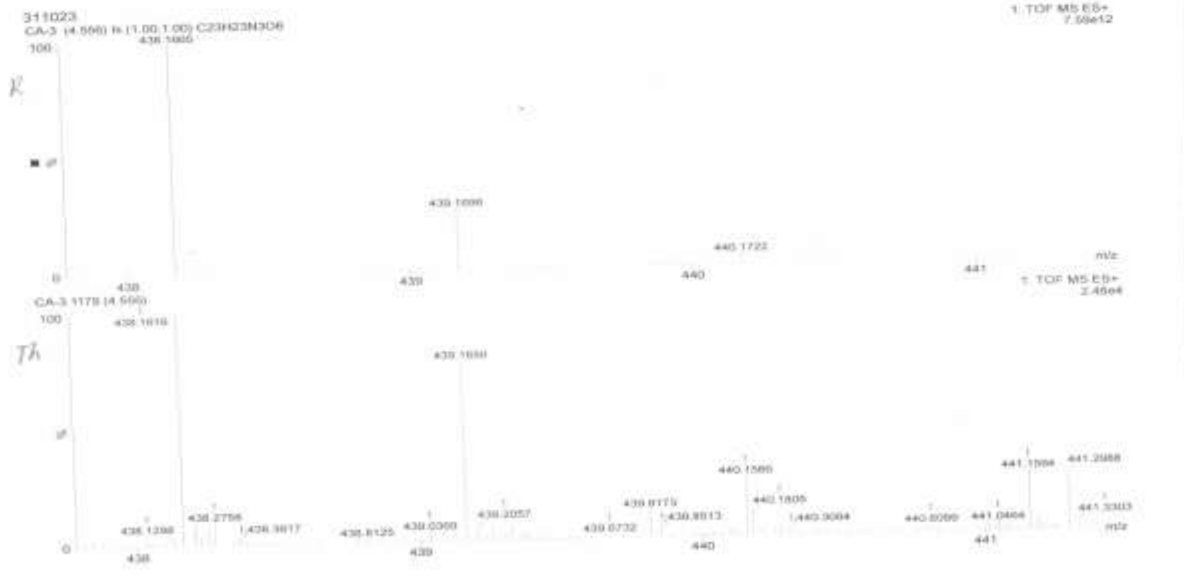

13.11.2023
AC-238-1 IN (0.034) Is (1.00,1.00) C23H23N3O4
TOF MS ES+
7.83e12
406.1767
407.1796
408.1825
AC-238-1 IN 8 (0.152) Cm (8:114)
TOF MS ES+
1.37e8
m/z

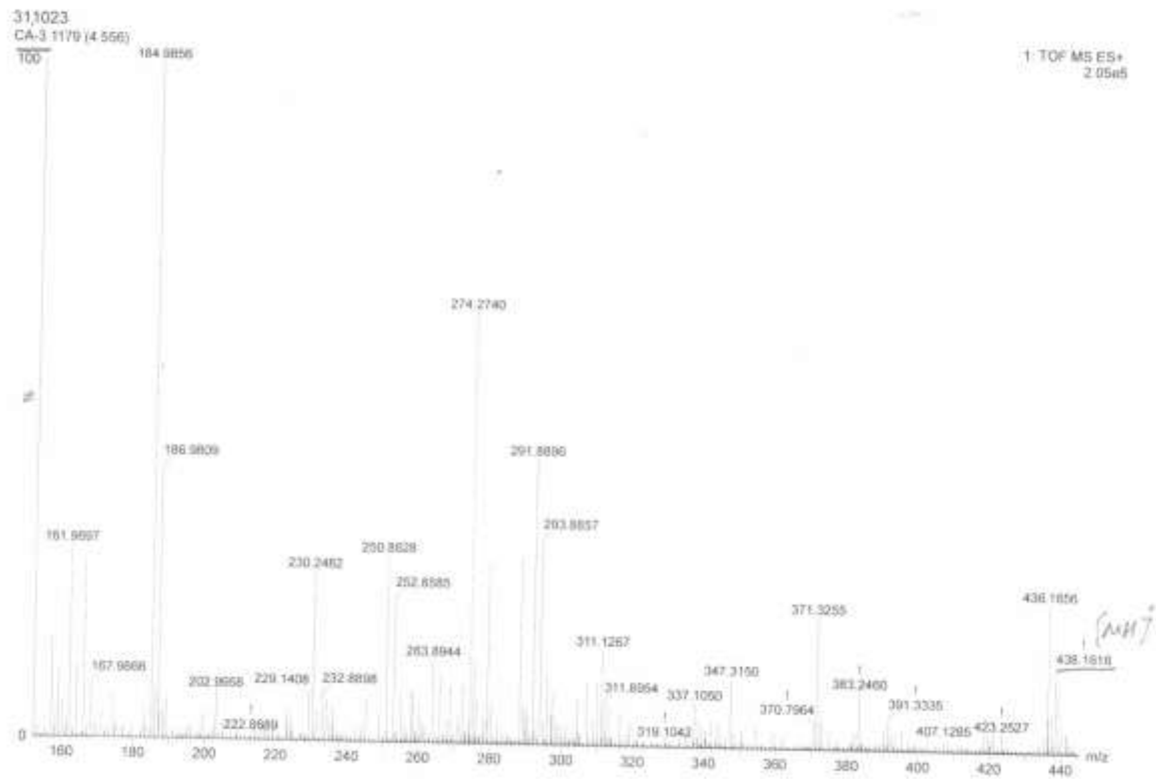

13.11.2023
AC-238-1 IN 8 (0.152) Cm (8:114)
TOF MS ES+
1.37e8
m/z

Copies of ^{1}H (300 MHz, DMSO-d_6/CCl_4 (1:3)) and ^{13}C (300 MHz, DMSO-d_6/CCl_4 (1:3)) spectra, HRMS of **5b**

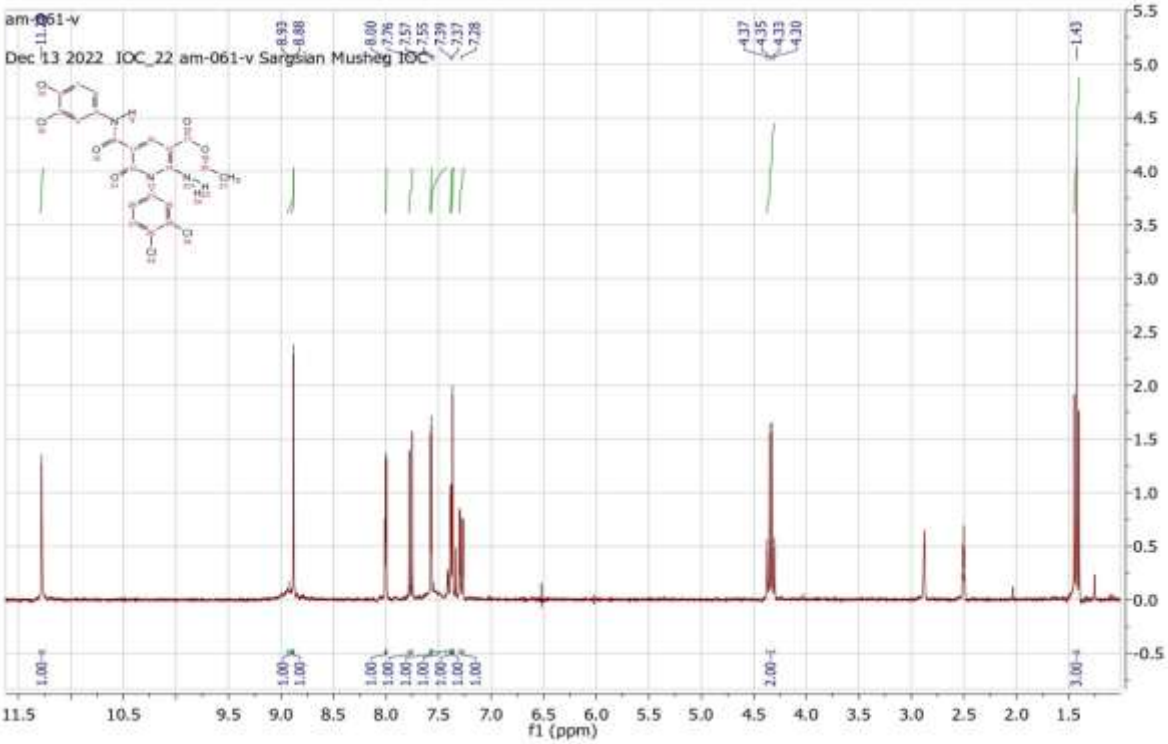


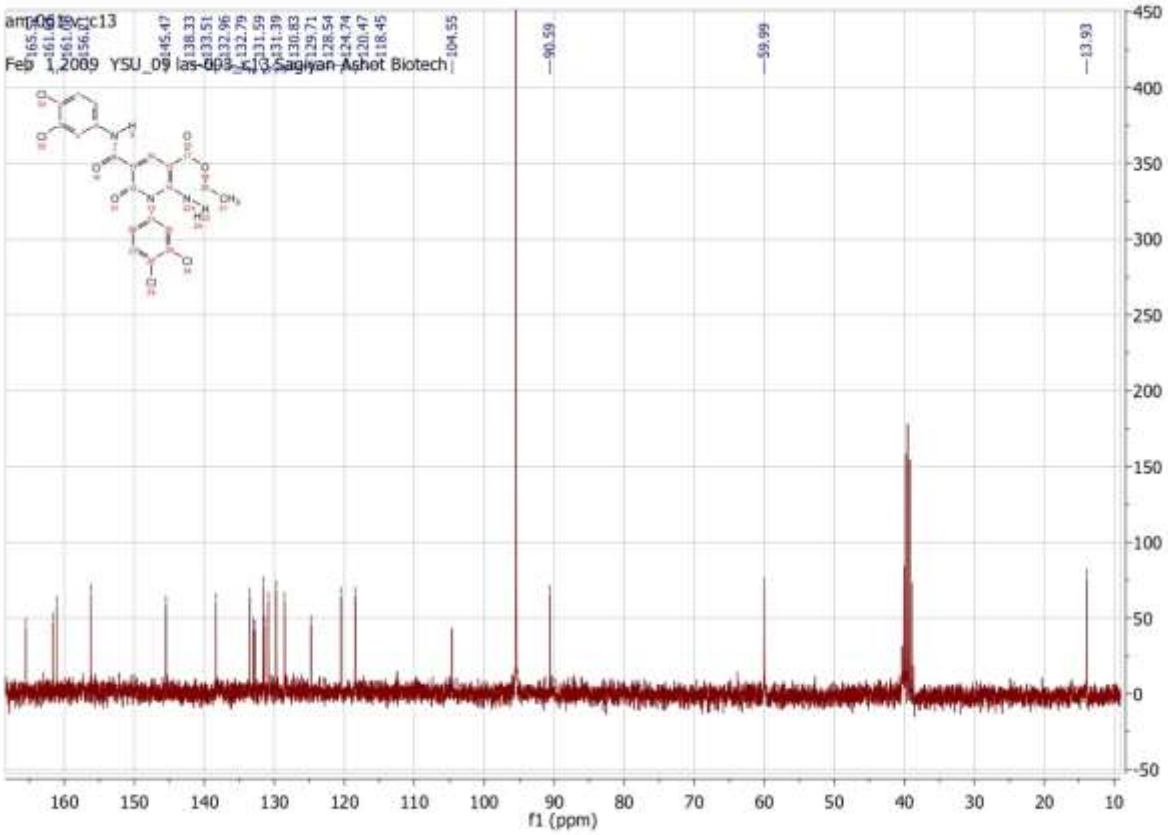


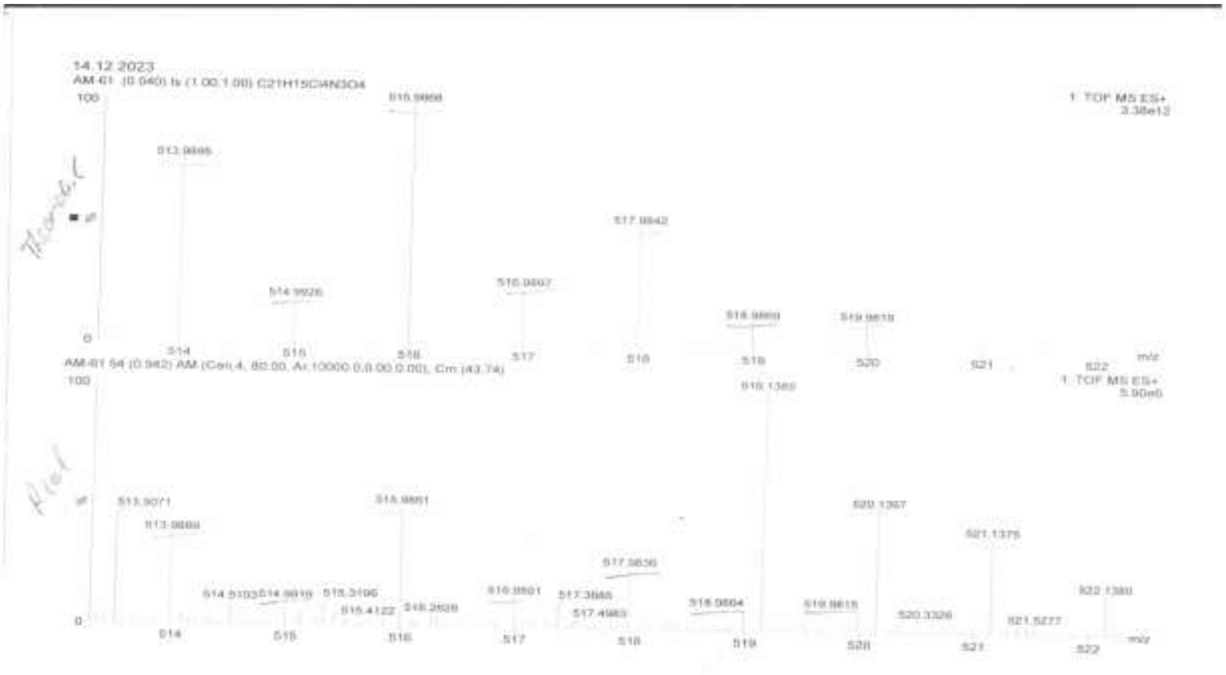

311023
CA-3 1179 (4.556)
1: TOF MS ES+
438.1616
439.1650
m/z

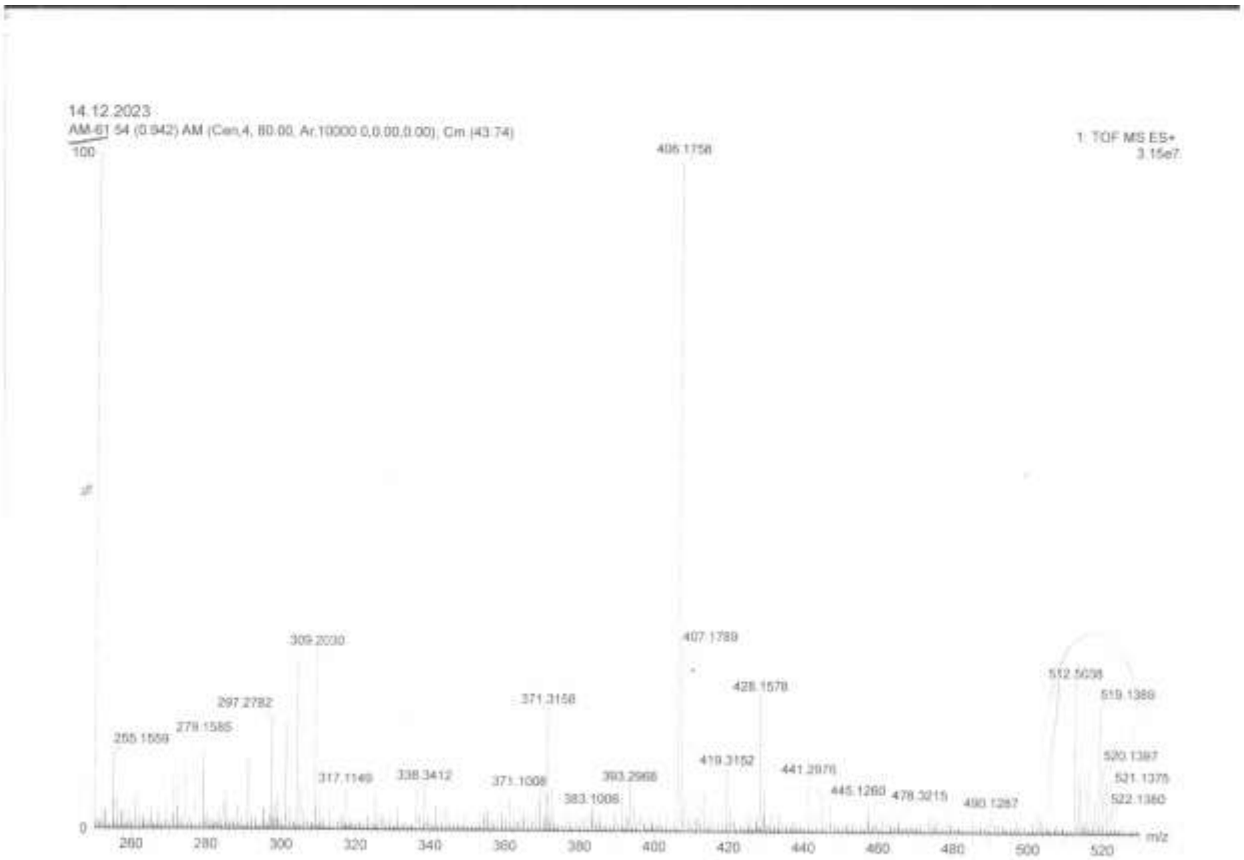

311023
CA-3 1179 (4.556)
1: TOF MS ES+
2.05e5
184.9856
274.2740
186.9809
291.8896
161.9897
293.8857
230.2482
250.8628
252.8585
436.1656
371.3255
263.8944
311.1267
167.9866
347.3150
438.1616
202.9958
229.1408
232.8898
311.8954
337.1050
383.2460
370.7964
391.3335
222.8689
319.1042
407.1285
423.2527
[MH]+
160
180
200
220
240
260
280
300
320
340
360
380
400
420
440
m/z

Copies of ^{1}H (300 MHz, DMSO-d_6/CCl_4 (1:3)) and ^{13}C (300 MHz, DMSO-d_6/CCl_4 (1:3)) spectra, HRMS of **5c**

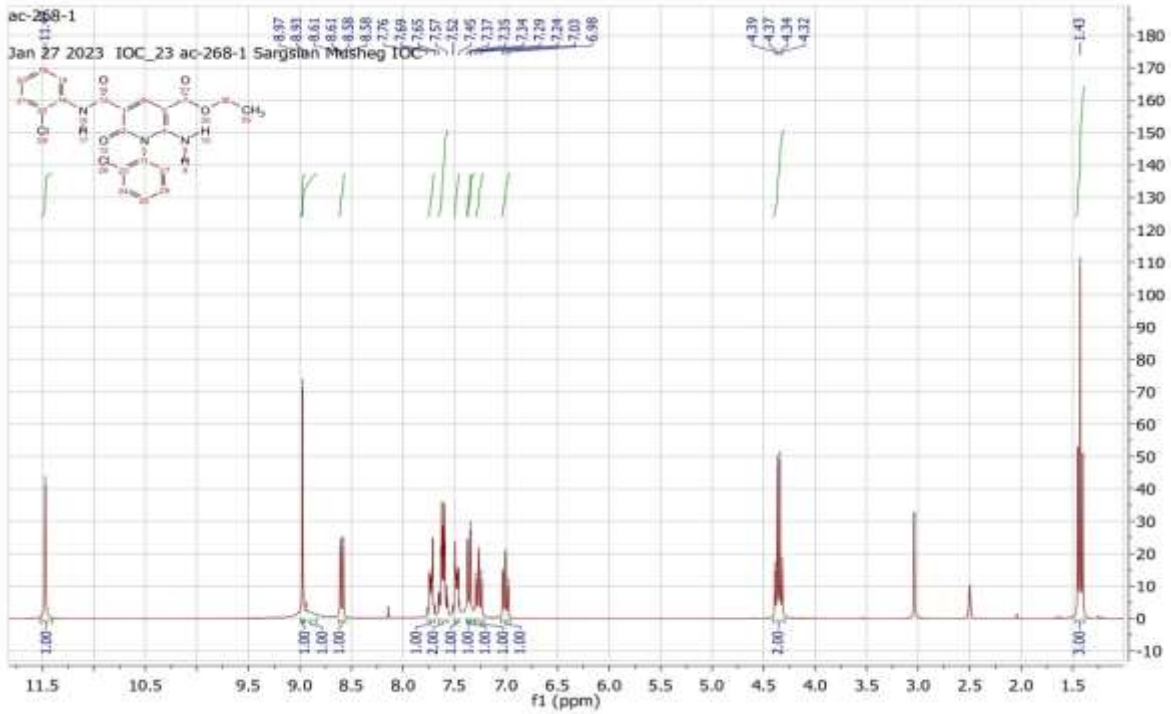


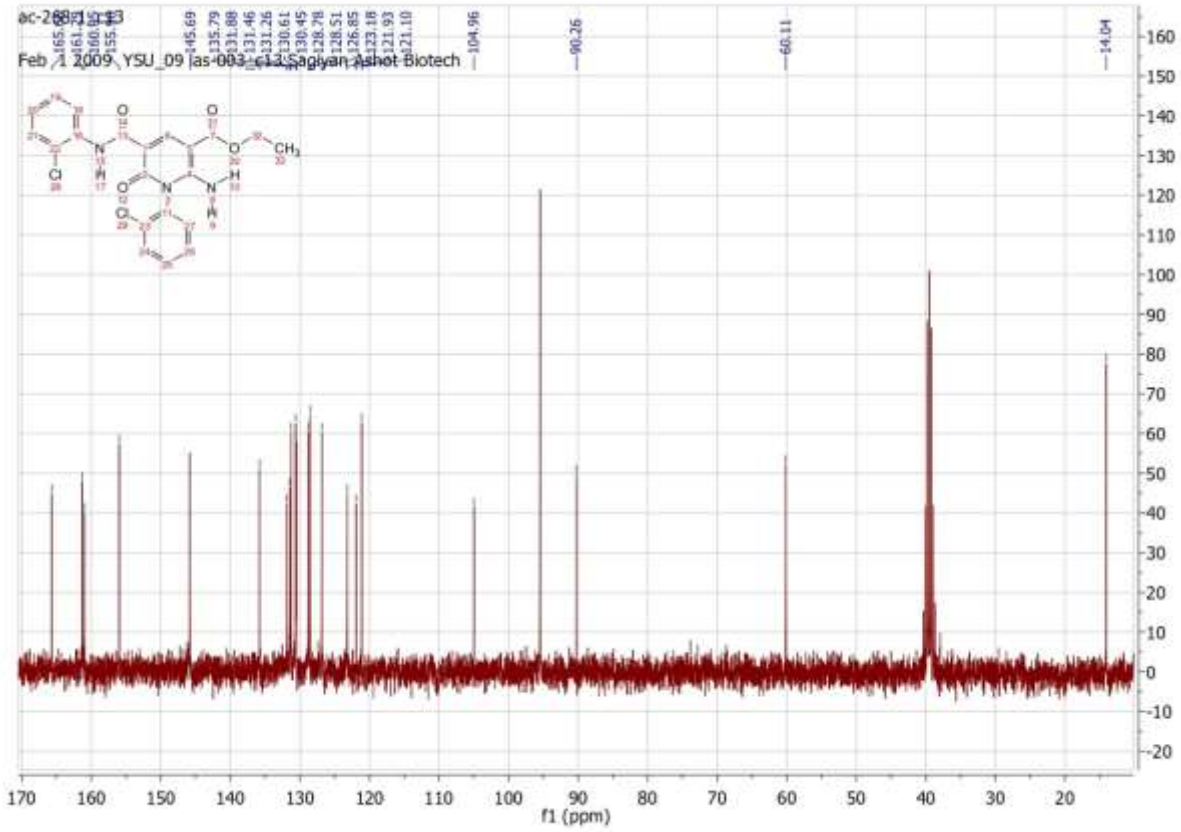


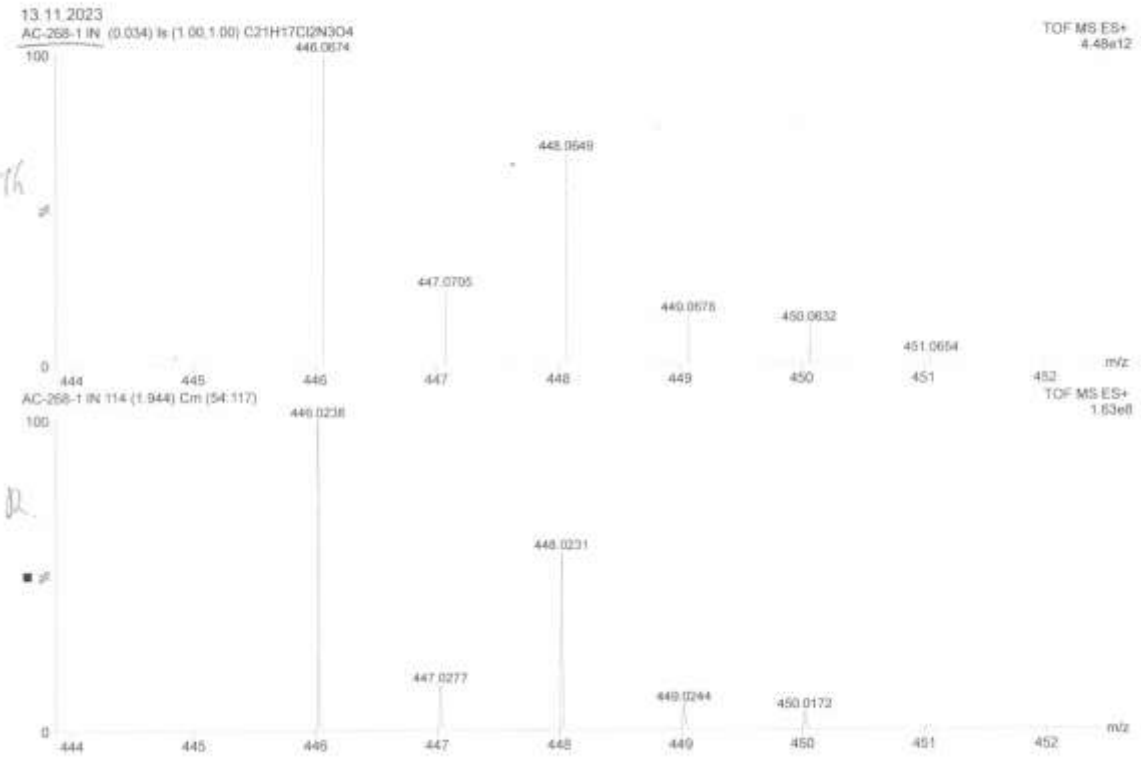

14.12.2023
AM-61 (0.040) Is (1.00,1.00) C21H15ClN3O4
1: TOF MS ES+
3.38e12
AM-61 54 (0.942) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.00); Cm (43.74)
1: TOF MS ES+
5.90e6
m/z

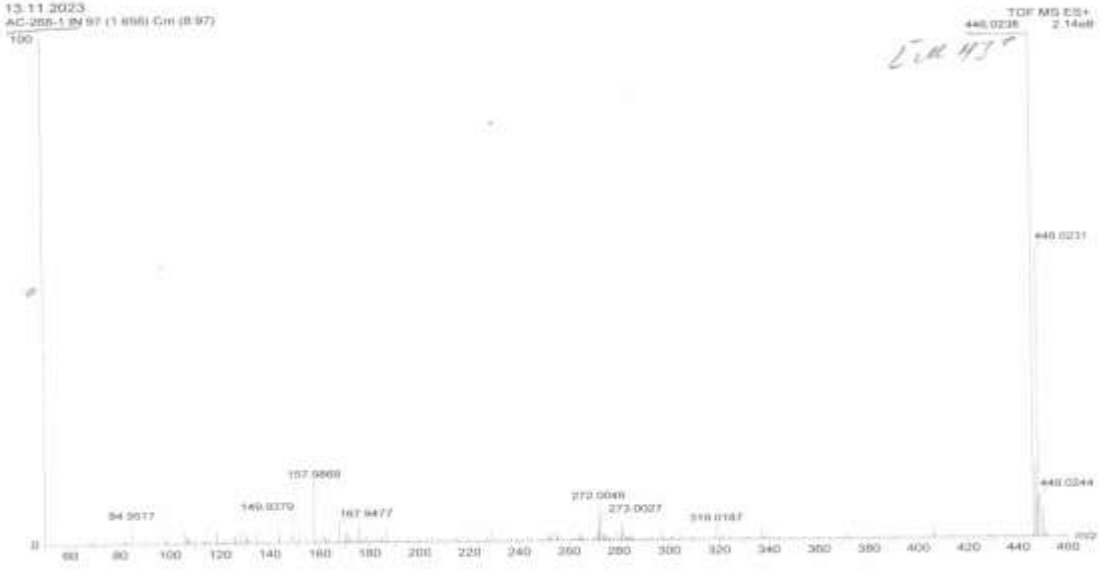

14.12.2023
AM-61 54 (0.942) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.00); Cm (43.74)
1: TOF MS ES+
3.15e7
406.1756
407.1789
309.2030
428.1578
371.3158
297.2782
279.1585
255.1559
512.5038
519.1389
m/z

Copies of ^{1}H (300 MHz, DMSO-d_6/CCl_4 (1:3)) and ^{13}C (300 MHz, DMSO-d_6/CCl_4 (1:3)) spectra, HRMS of **5d**

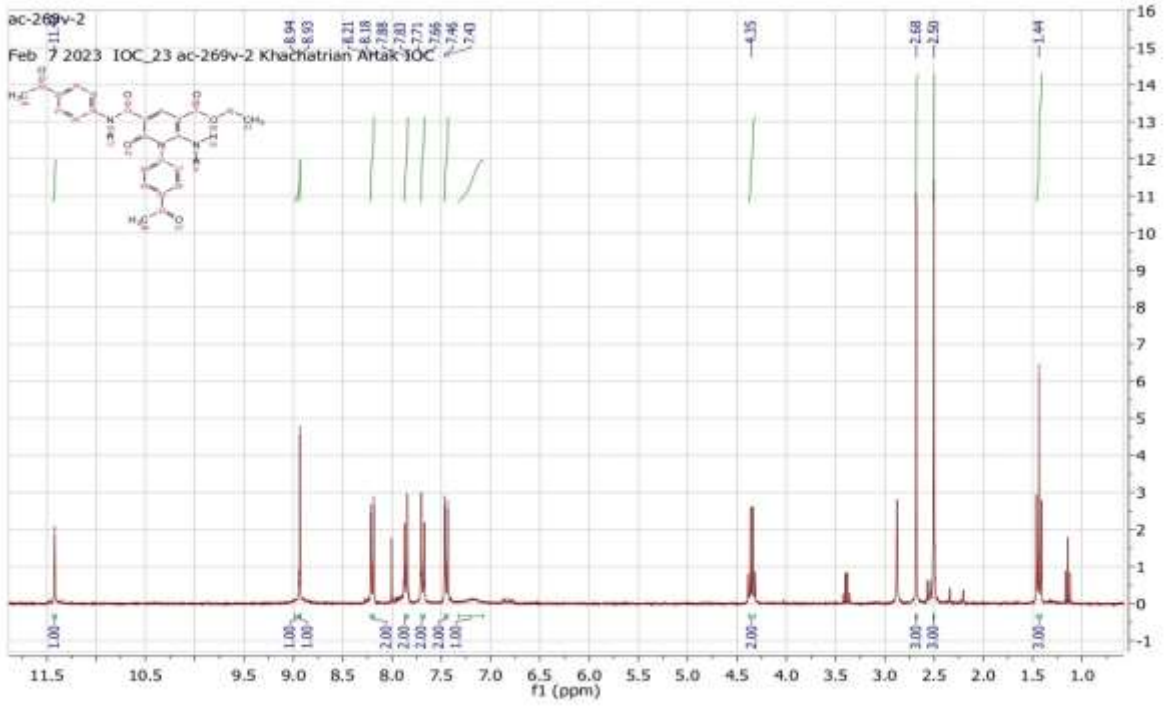


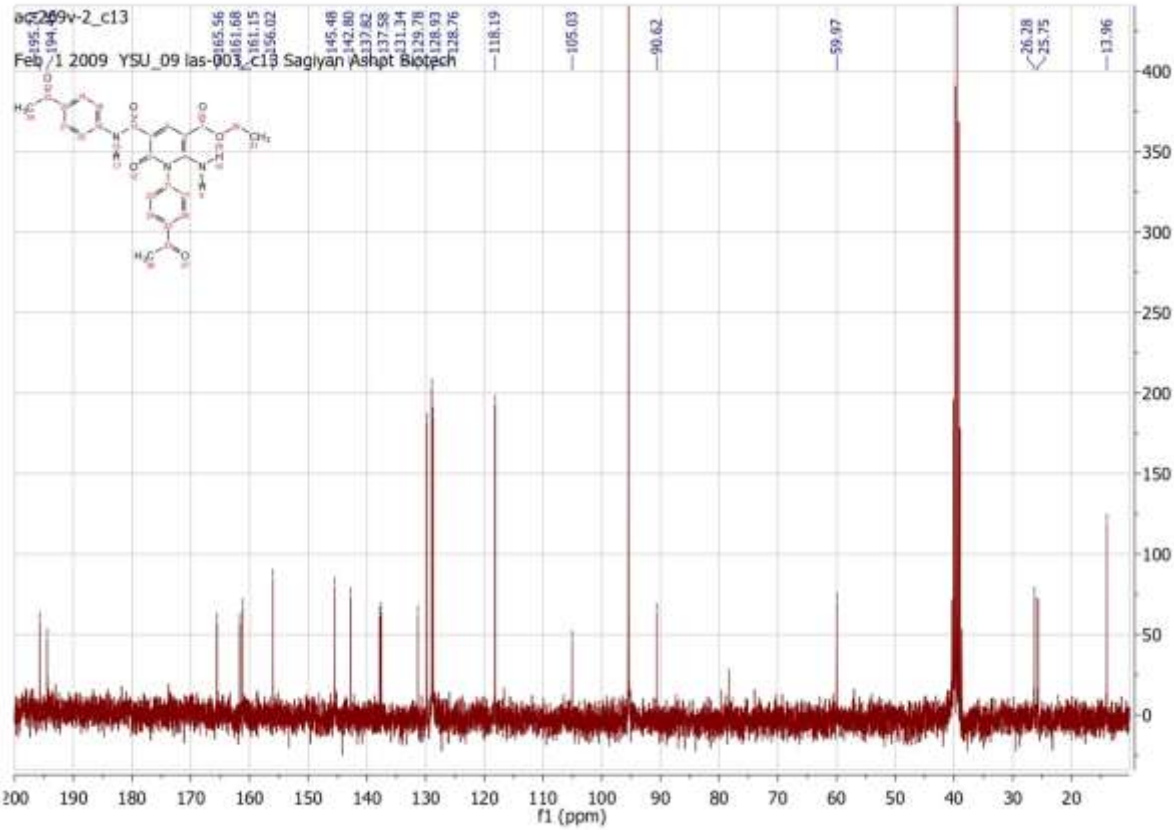


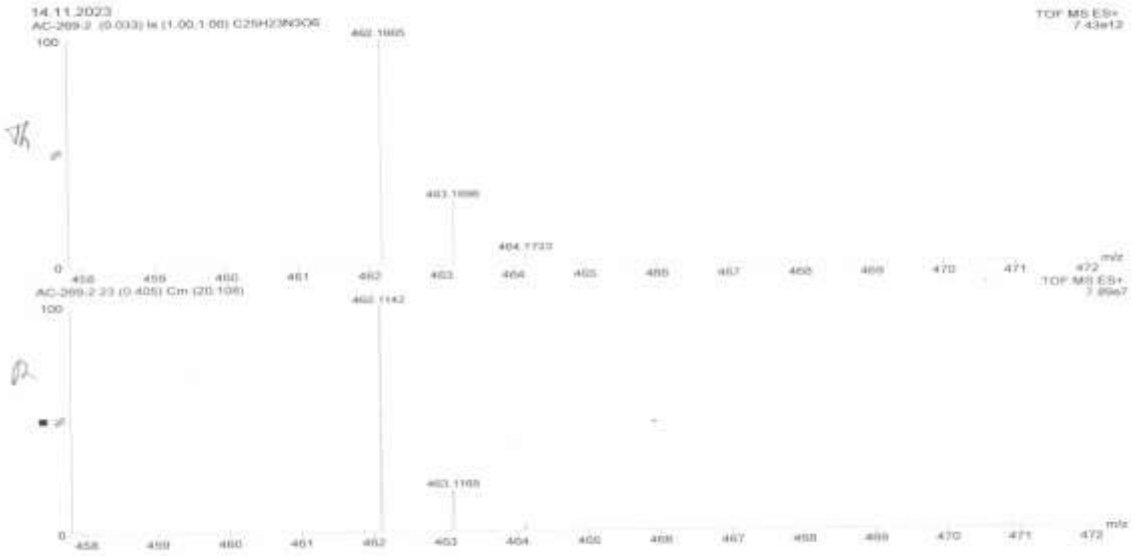

13.11.2023
AC-268-1 IN (0.034) Is (1.00,1.00) C21H17Cl2N3O4
TOF MS ES+
4.48e12
446.0674
448.0649
447.0705
449.0678
450.0632
451.0654
AC-268-1 IN 114 (1.944) Cm (54:117)
TOF MS ES+
1.63e8
446.0238
448.0231
447.0277
449.0244
450.0172
m/z

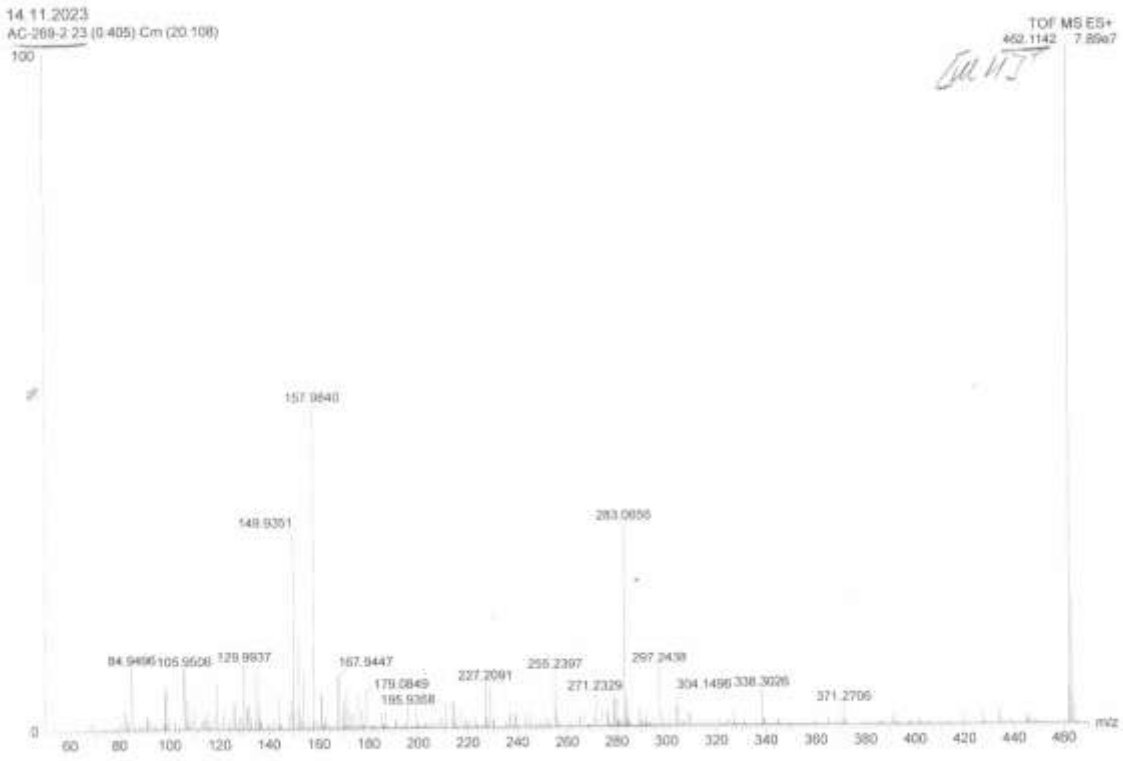

13.11.2023
TOF MS ES+
446.0238
2.14e8
448.0231
448.0244
157.9868
149.0379
94.9577
167.9477
272.0049
273.0027
318.0187

Copies of ^{1}H (300 MHz, DMSO-d_6/CCl_4 (1:3)) and ^{13}C (300 MHz, DMSO-d_6/CCl_4 (1:3)) spectra, HRMS of **5e**

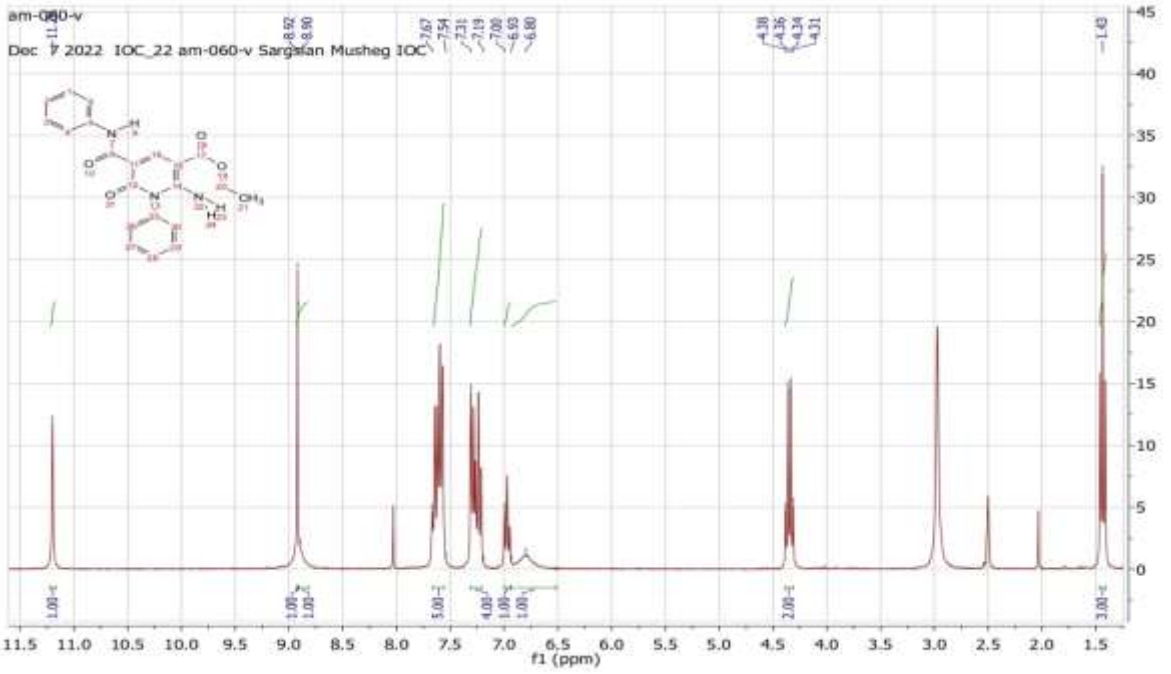


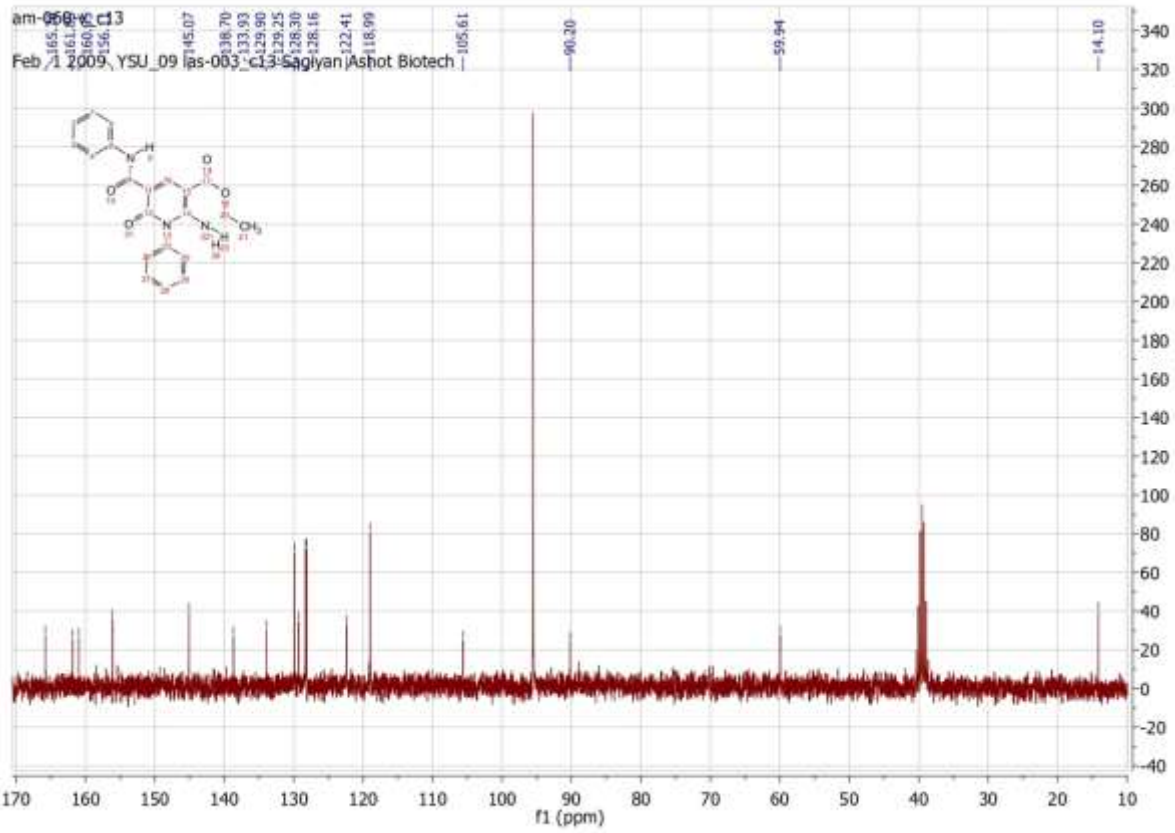


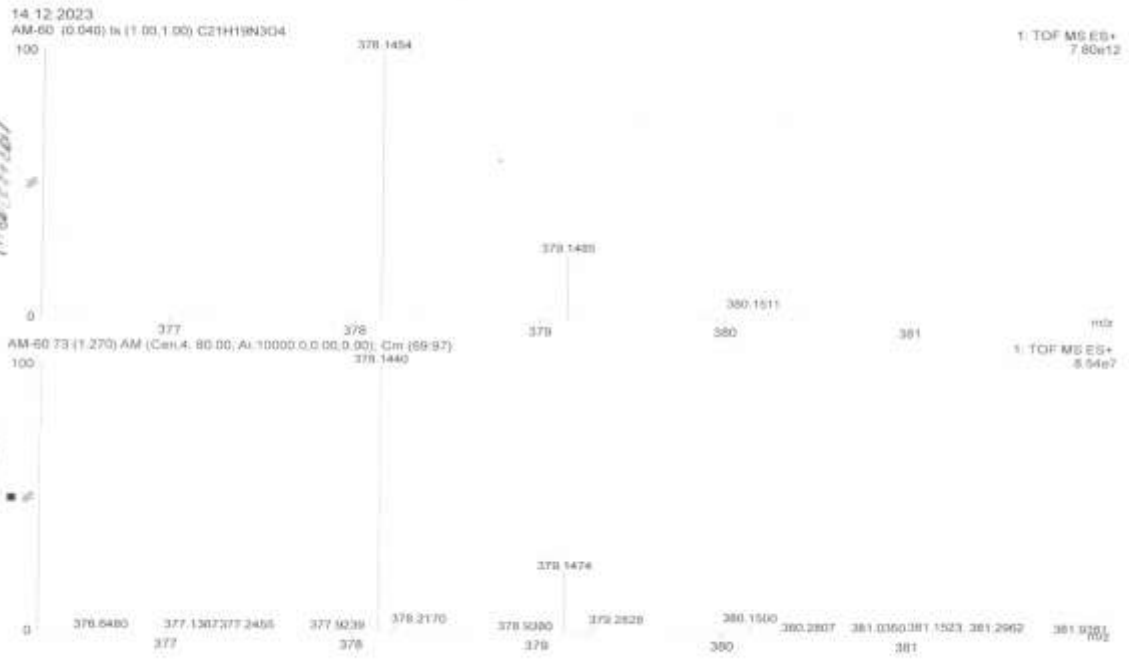

14.11.2023
AC-269-2 (0.033) Is (1.00,1.00) C25H23N3O6
462.1665
463.1698
464.1733
TOF MS ES+
7.43e12
AC-269-2 23 (0.405) Cm (20:108)
462.1142
463.1165
TOF MS ES+
7.89e7
m/z

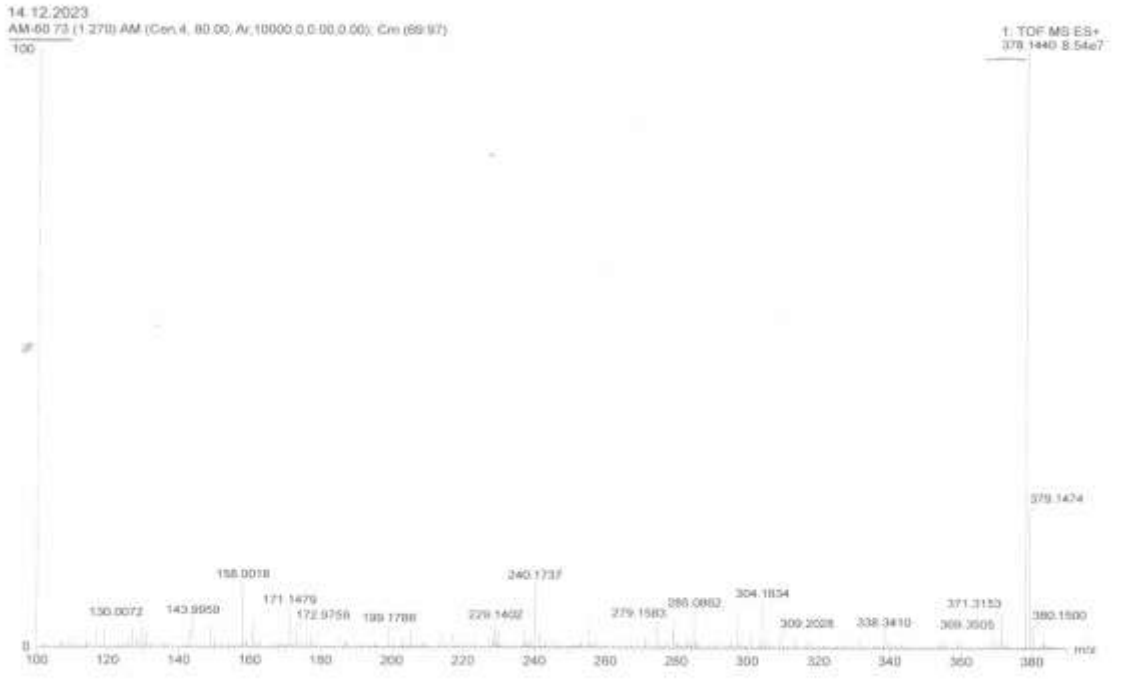

14.11.2023
AC-269-2 23 (0.405) Cm (20:108)
TOF MS ES+
462.1142 7.89e7
157.9840
148.9351
283.0656
84.9496
105.9508
129.9837
167.9447
179.0849
185.9358
227.2091
255.2397
271.2329
297.2438
304.1496
338.3026
371.2706
m/z

Copies of ^{1}H (300 MHz, DMSO-d_6/CCl_4 (1:3)) and ^{13}C (300 MHz, DMSO-d_6/CCl_4 (1:3)) spectra, HRMS of **5f**

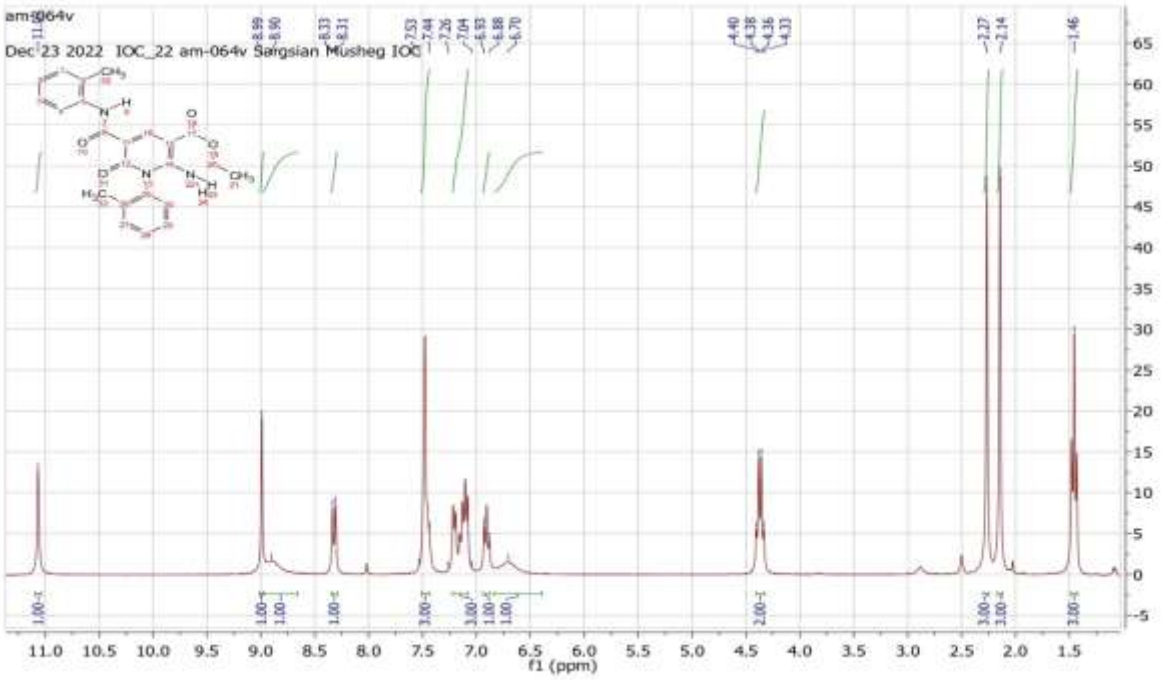


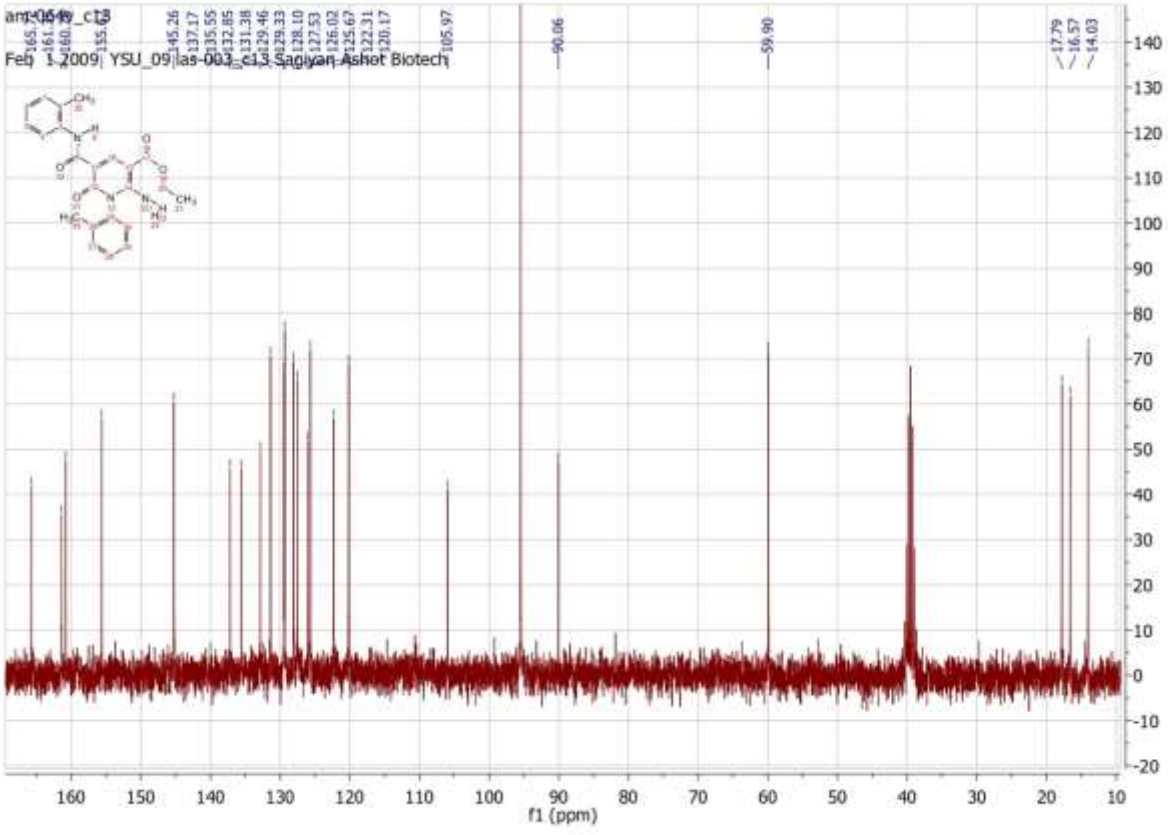


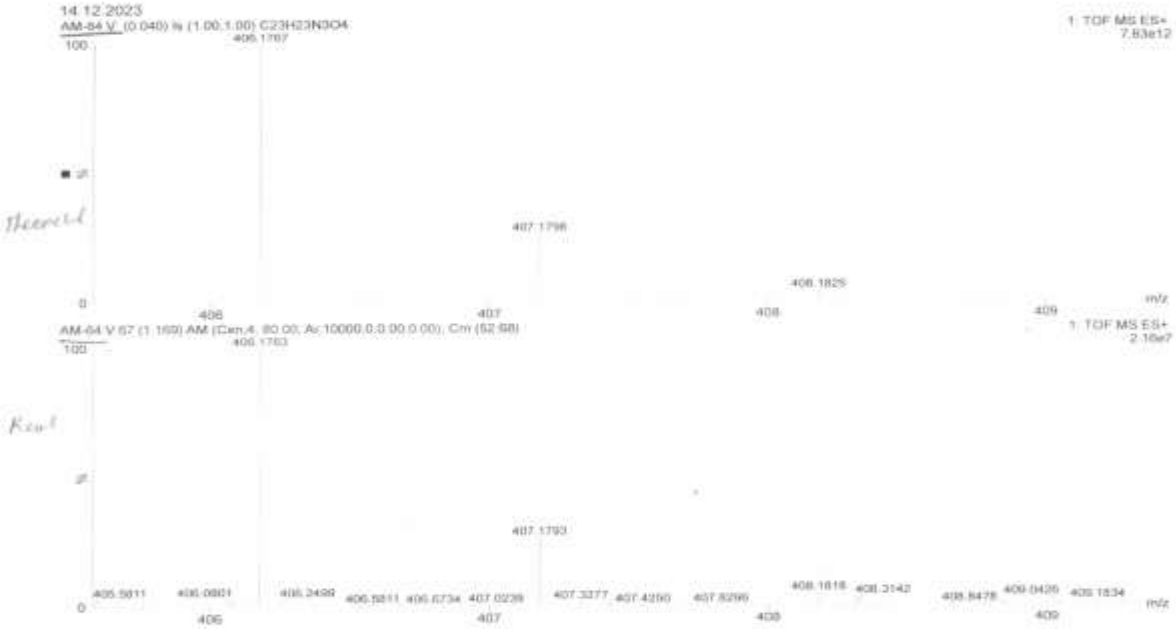

14.12.2023
AM-60 (0.040) Is (1.00,1.00) C21H19N3O4
1: TOF MS ES+
7.80e12
378.1454
379.1485
380.1511
AM-60 73 (1.270) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.00); Cm (69:97)
1: TOF MS ES+
8.54e7
378.1440
379.1474
376.6480
377.1387
377.2455
377.9039
378.2170
378.9380
379.2829
380.1500
380.2807
381.0350
381.1523
381.2962
381.9381
m/z

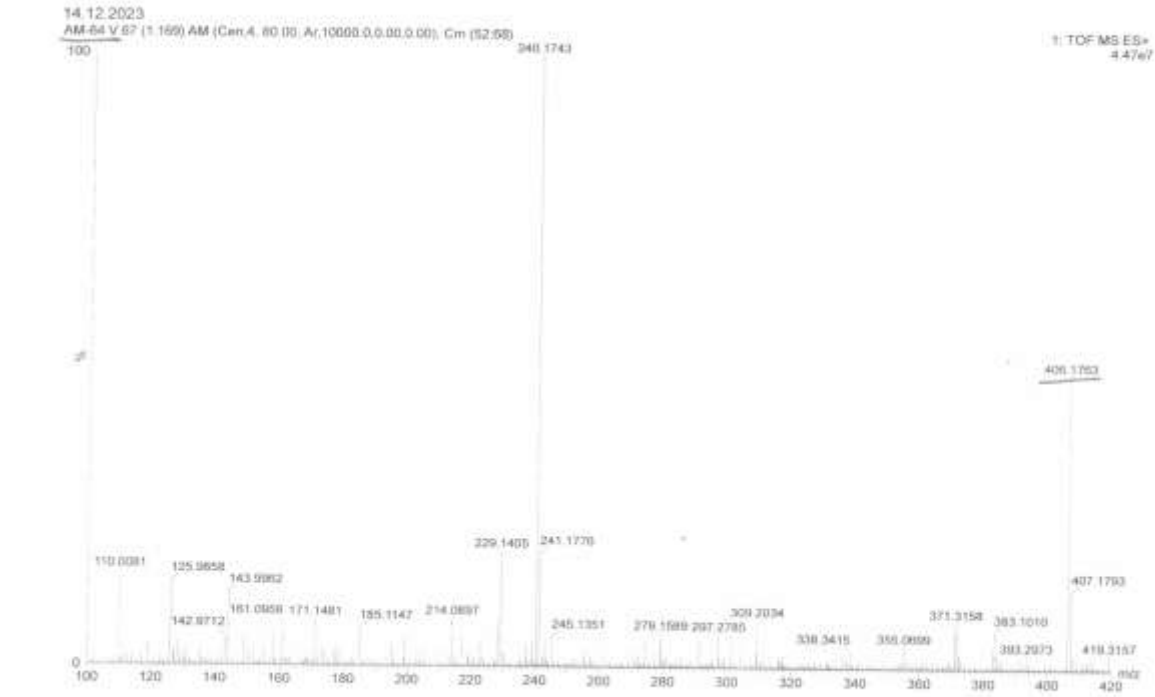

14.12.2023
AM-60 73 (1.270) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.00); Cm (69:97)
1: TOF MS ES+
378.1440 8.54e7
379.1474
158.0018
240.1737
150.0072
143.9959
171.1479
172.9758
199.1788
229.1402
279.1583
286.0862
304.1854
309.2028
338.3410
369.3505
371.3153
380.1500
m/z

Copies of ^{1}H (300 MHz, DMSO-d_6/CCl_4 (1:3)) and ^{13}C (300 MHz, DMSO-d_6/CCl_4 (1:3)) spectra, HRMS of **5g**

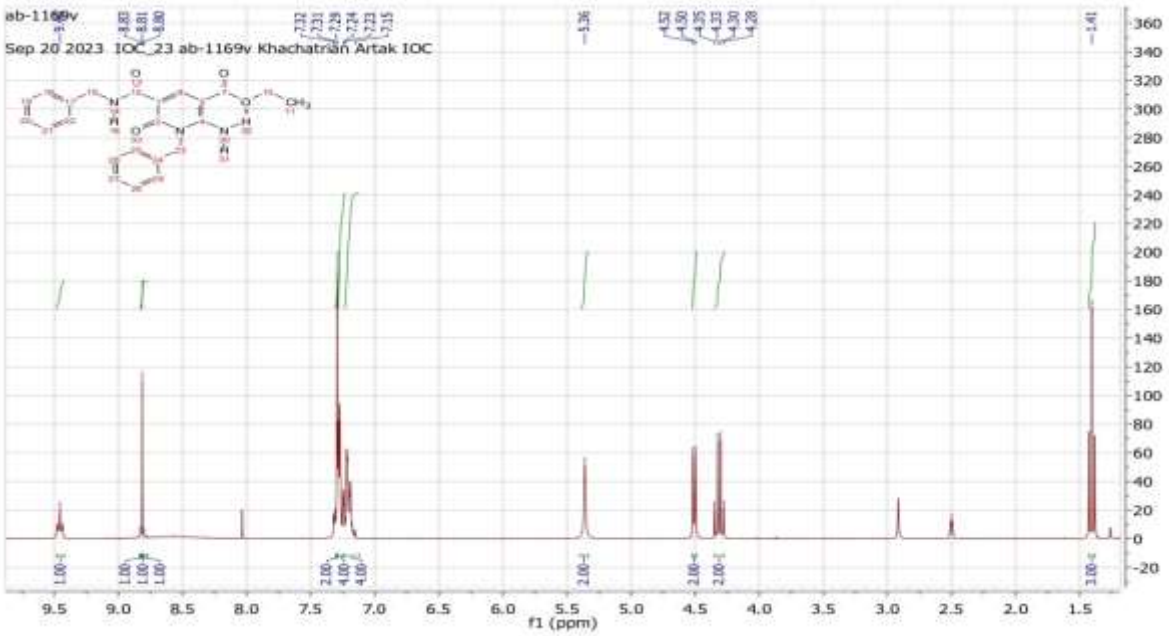


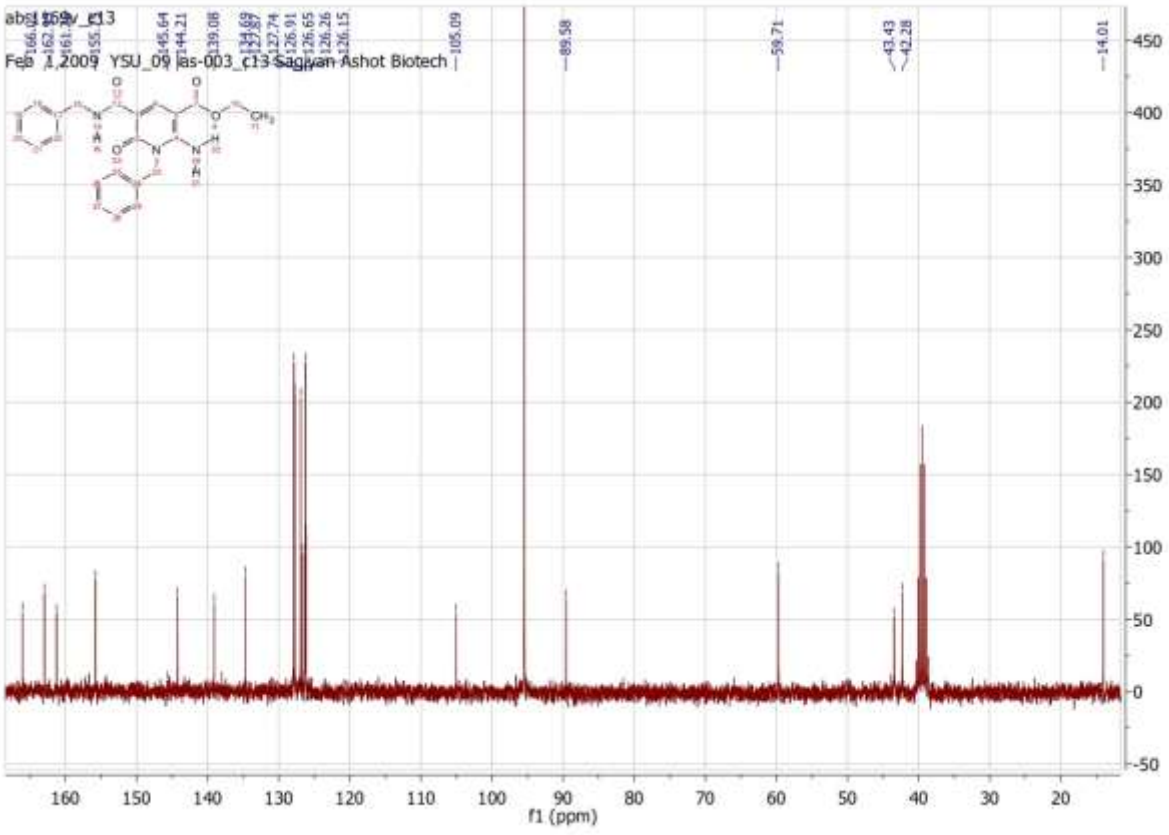


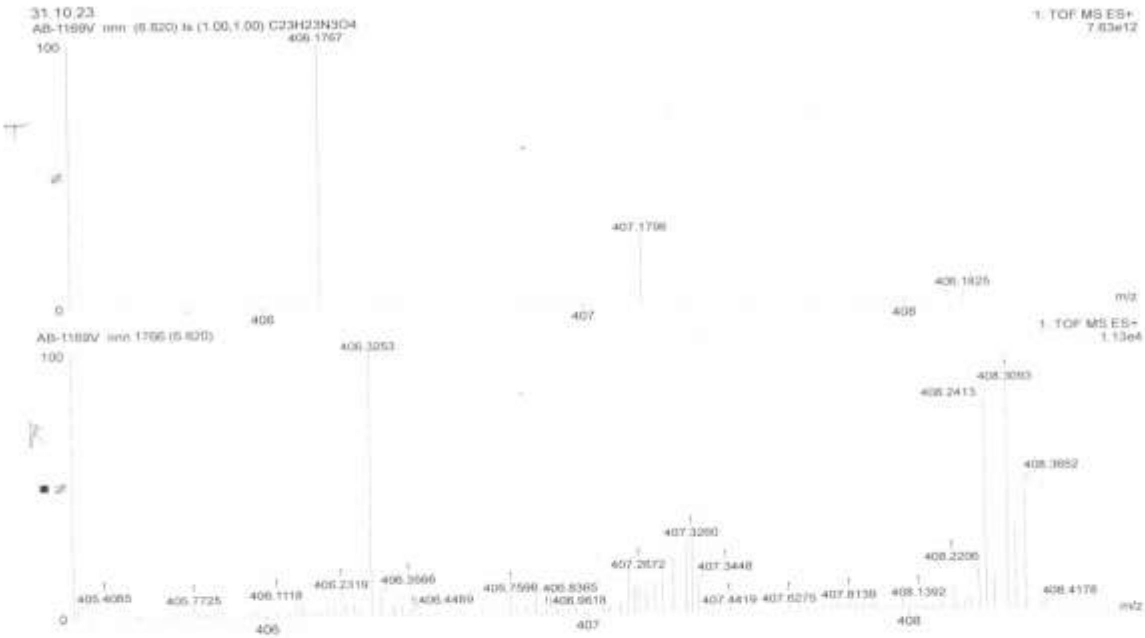

14.12.2023
AM-64 V_(0.040) is (1.00,1.00) C23H23N3O4
406.1767
1: TOF MS ES+
7.83e12
100
%
0
407.1798
406.1825
406
407
408
409
m/z
AM-64 V 67 (1.169) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.00); Cm (52:68)
406.1763
1: TOF MS ES+
2.16e7
100
%
0
405.5811
406.0801
406.2499
406.5811
406.6734
407.0239
407.1793
407.3377
407.4260
407.8266
408.1618
408.3142
408.8478
409.0426
409.1834
406
407
408
409
m/z

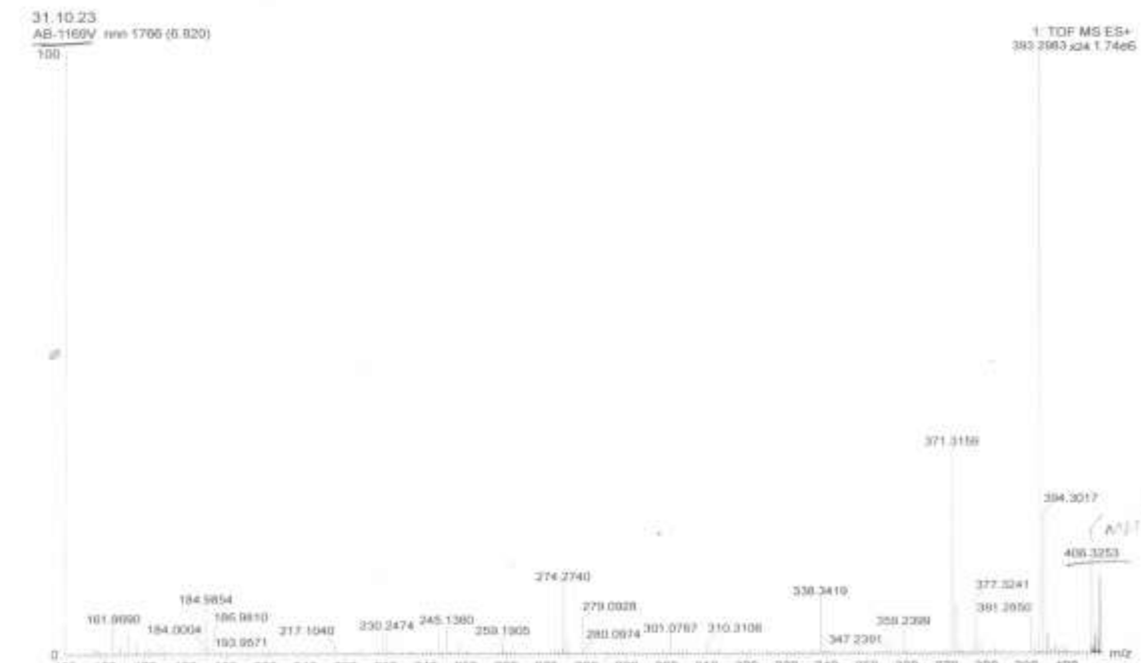

14.12.2023
AM-64 V 67 (1.169) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.00); Cm (52:68)
100
%
0
246.1743
1: TOF MS ES+
4.47e7
406.1763
229.1405
241.1770
110.0081
125.0858
143.0982
142.9712
161.0958
171.1481
185.1147
214.0897
245.1351
279.1589
297.2780
309.2034
338.3415
355.0699
371.3158
383.1010
383.2973
407.1793
419.3157
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
400
420
m/z

Copies of ^{1}H (300 MHz, DMSO-d_6/CCl_4 (1:3)) and ^{13}C (300 MHz, DMSO-d_6/CCl_4 (1:3)) spectra, HRMS of **5h**

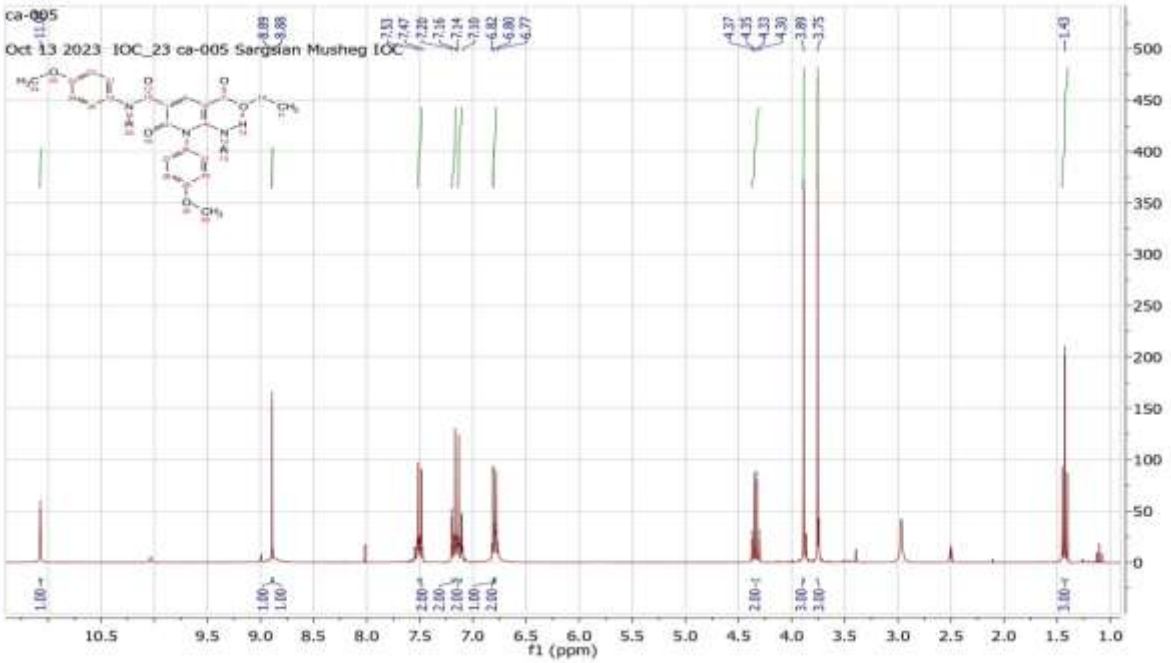


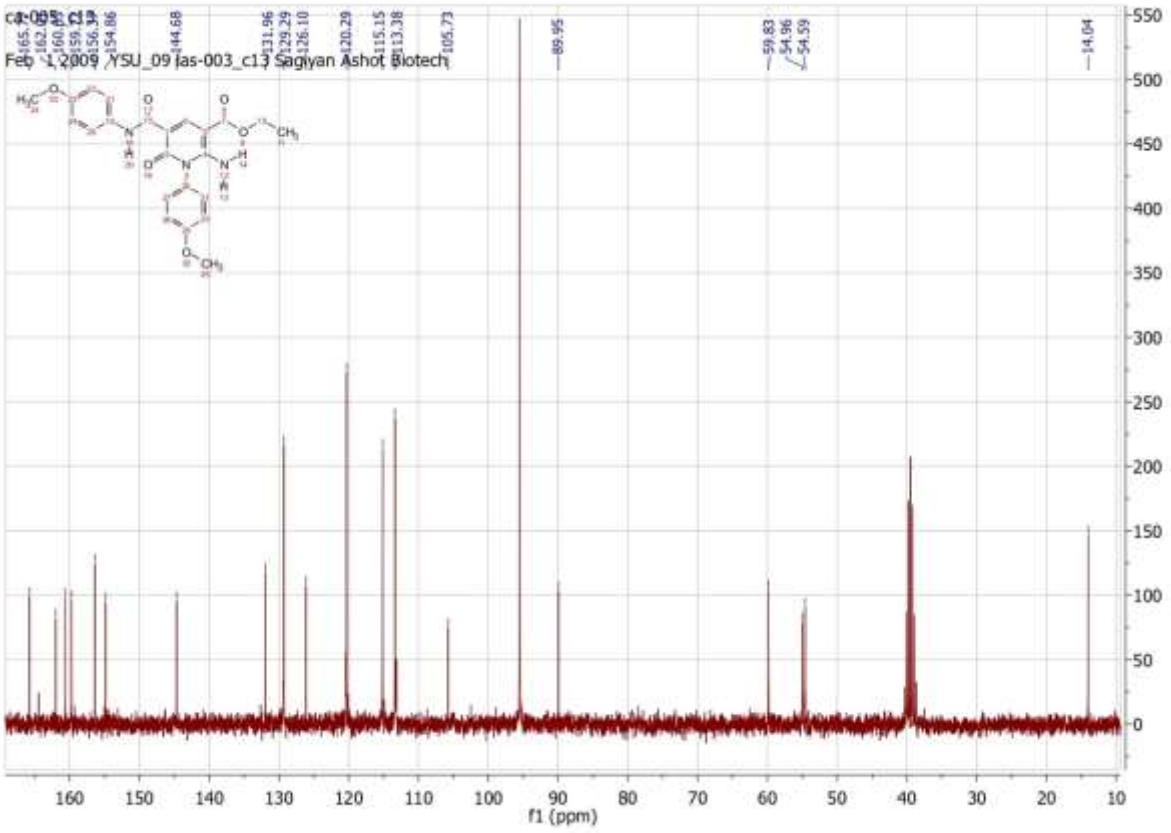


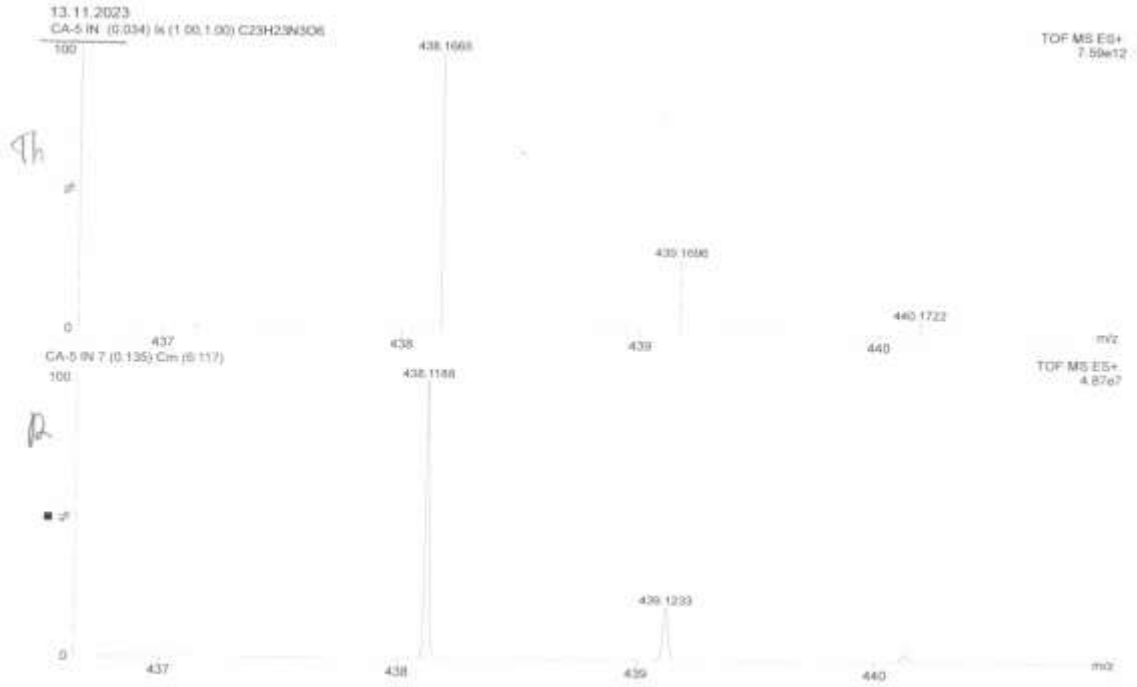

31.10.23
AB-1169V
1: TOF MS ES+
406.1767
407.1796
406.3253
m/z

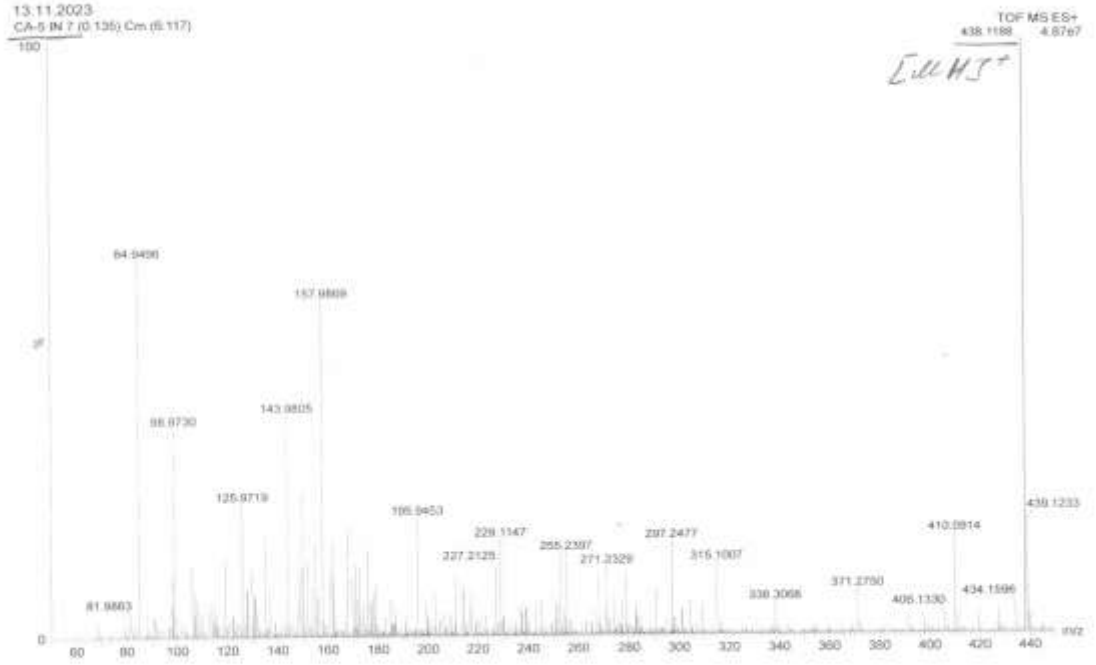

31.10.23
AB-1169V
1: TOF MS ES+
371.3158
394.3017
406.3253
m/z

Copies of ^{1}H (300 MHz, DMSO-d_6/CCl_4 (1:3)) and ^{13}C (300 MHz, DMSO-d_6/CCl_4 (1:3)) spectra, HRMS of **5i**

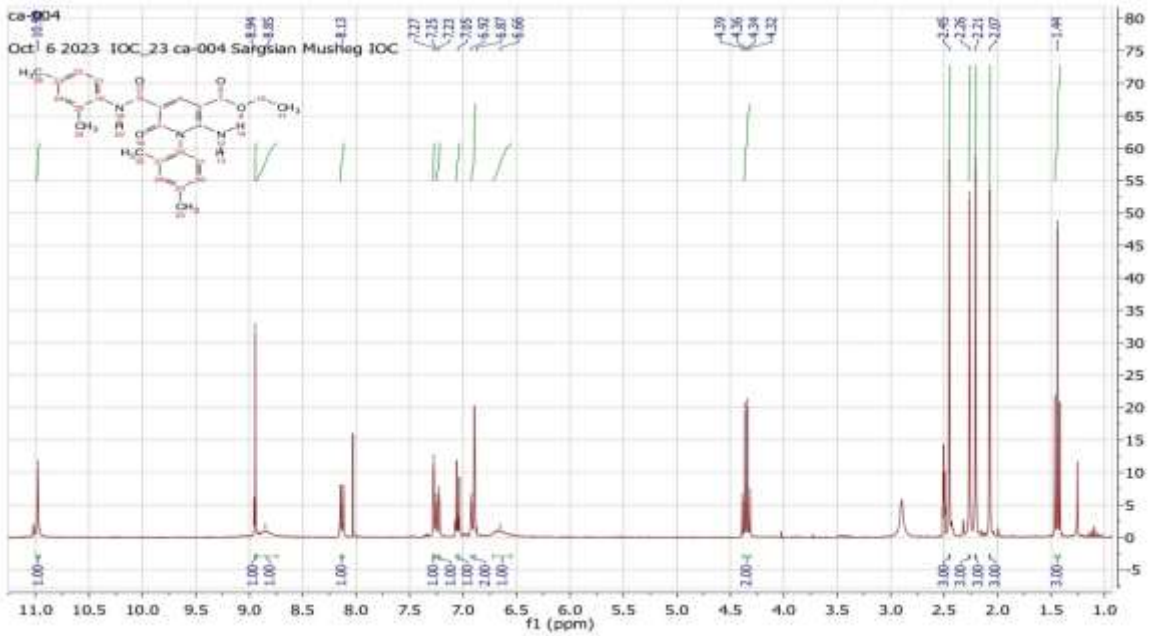


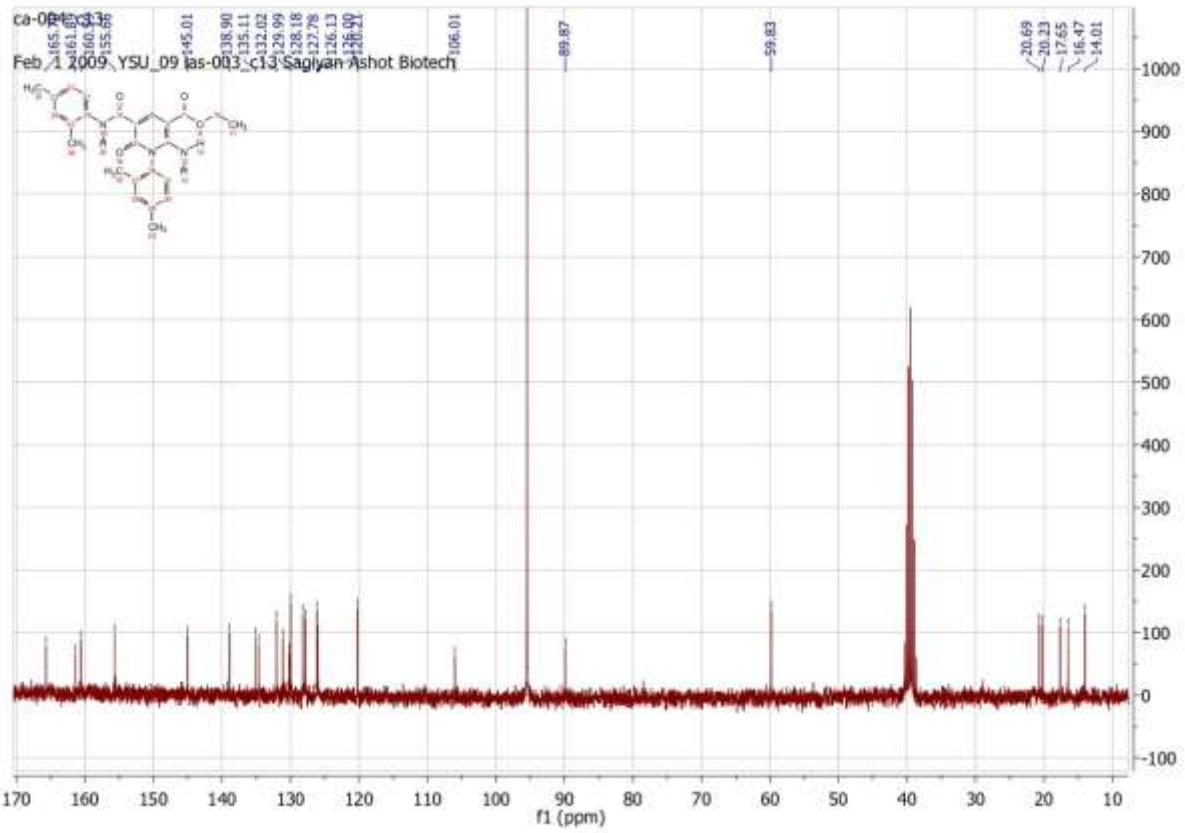


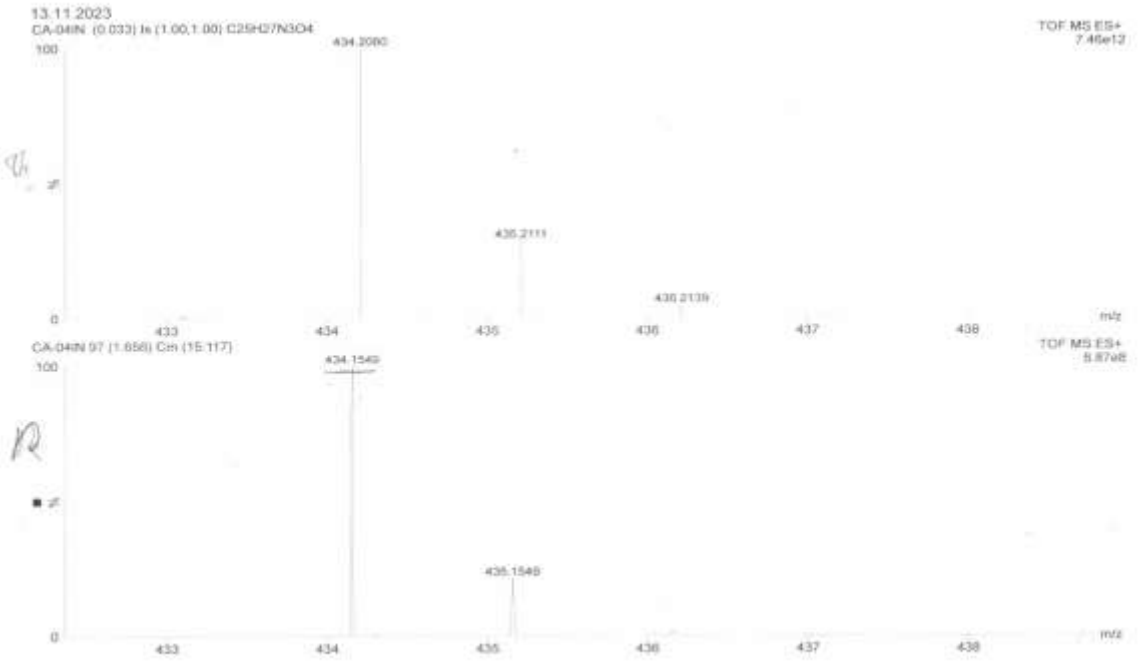

13.11.2023
CA-5 IN (0.034) Is (1.00,1.00) C23H23N3O6
TOF MS ES+
7.59e12
438.1663
439.1696
440.1722
CA-5 IN 7 (0.135) Cm (6:117)
TOF MS ES+
4.87e7
438.1188
439.1233
m/z

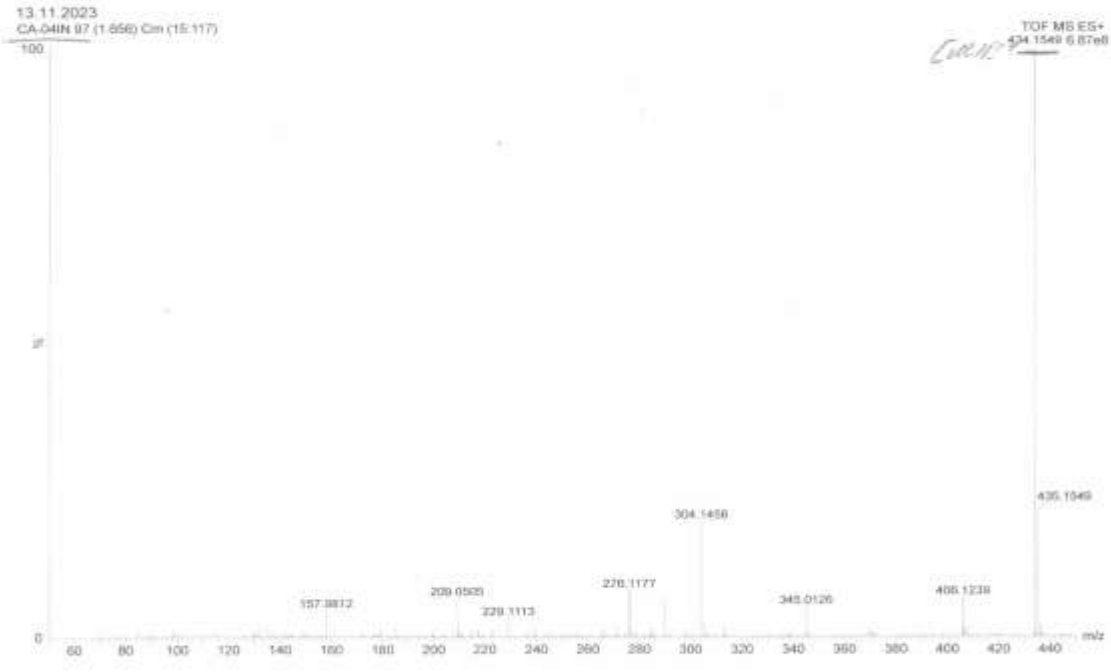

13.11.2023
CA-5 IN 7 (0.135) Cm (6:117)
TOF MS ES+
438.1188
4.87e7
64.9496
157.9869
143.9805
88.9730
125.9719
166.9453
228.1147
227.2125
255.2307
271.2329
297.2477
315.1007
338.3068
371.2750
406.1330
410.0914
434.1596
439.1233

Copies of ^{1}H (300 MHz, DMSO-d_6/CCl_4 (1:3)) and ^{13}C (300 MHz, DMSO-d_6/CCl_4 (1:3)) spectra, HRMS of **5j**

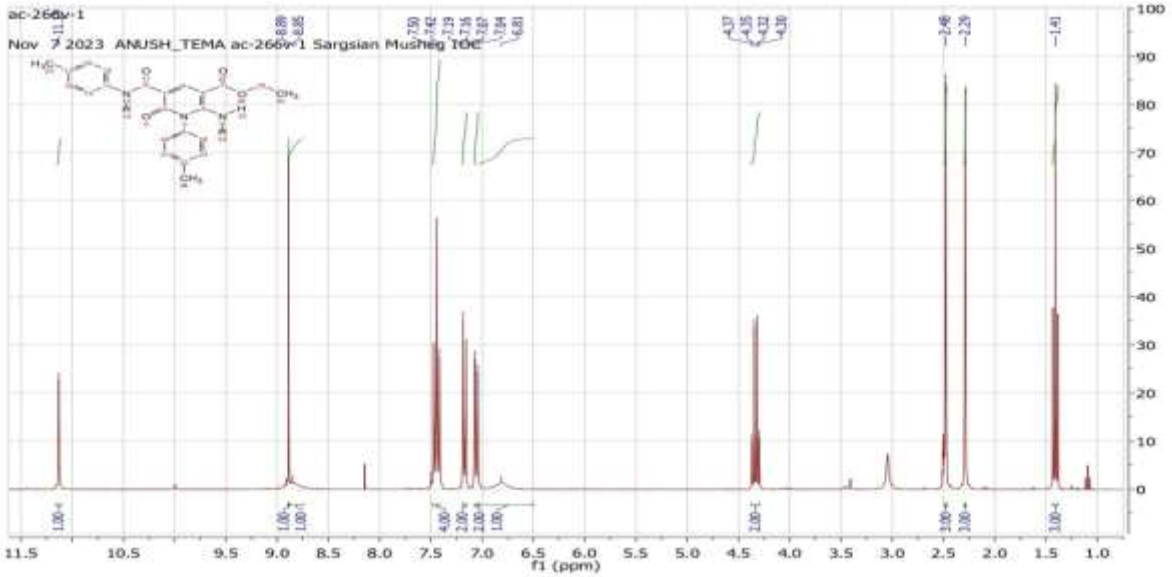


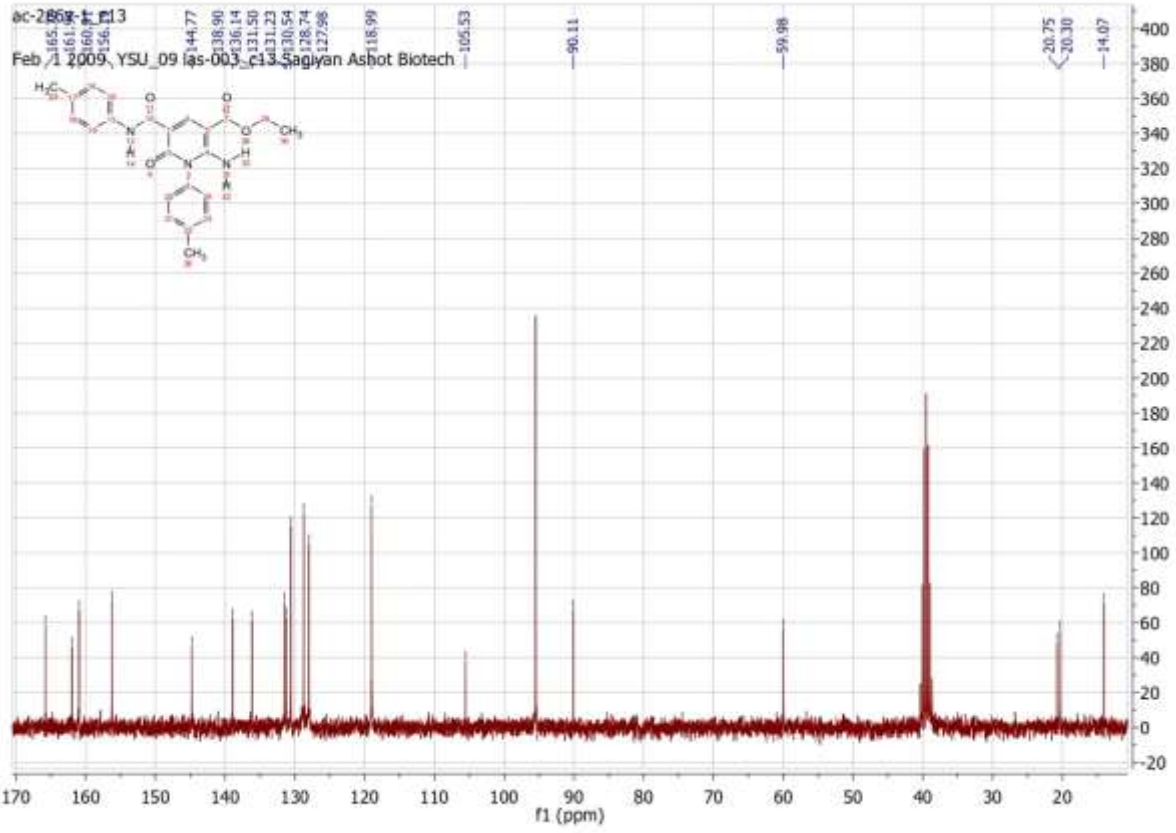


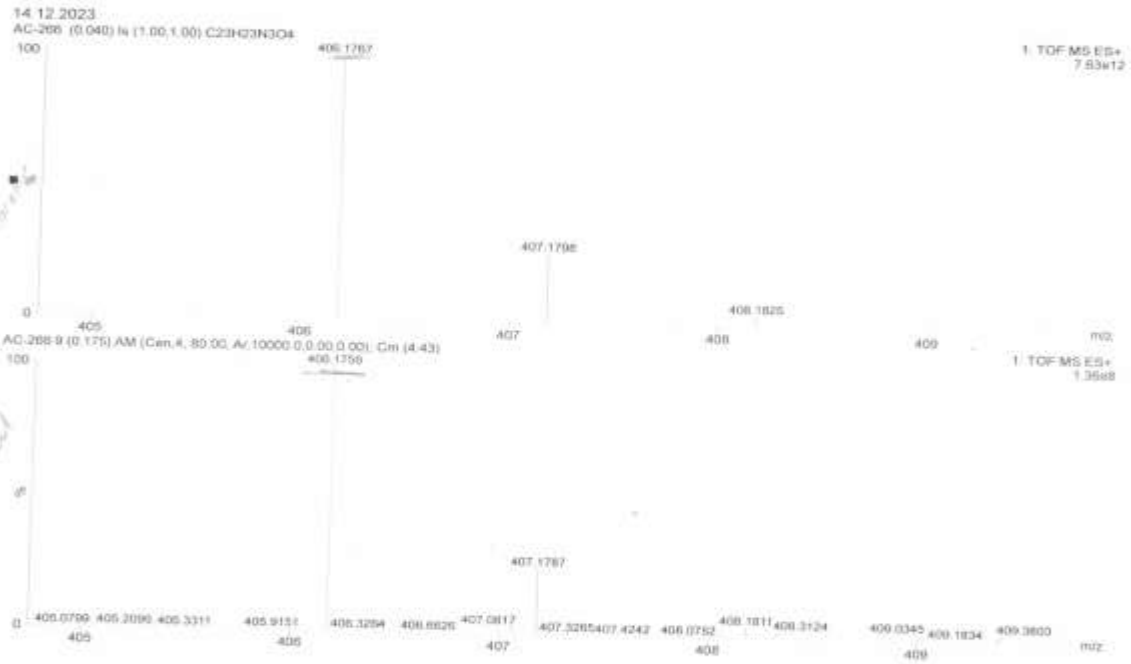

13.11.2023
CA-04IN (0.033) Is (1.00,1.00) C25H27N3O4
TOF MS ES+
7.46e12
434.2080
435.2111
436.2139
CA-04IN 97 (1.656) Cm (15:117)
TOF MS ES+
6.87e8
434.1549
435.1548
m/z

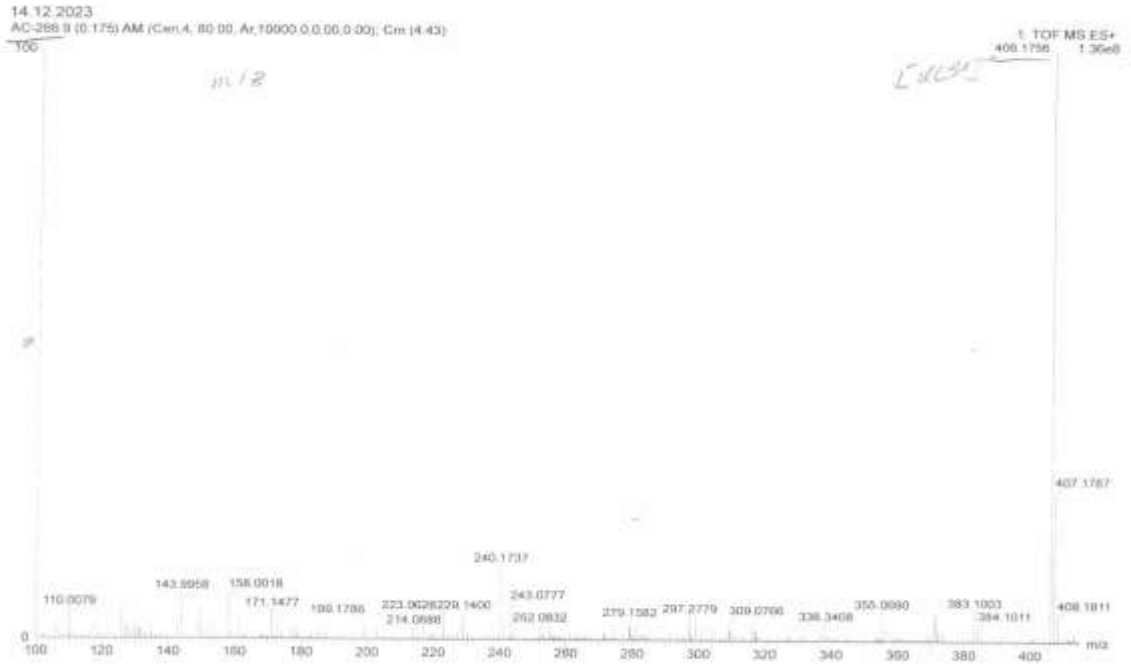

13.11.2023
CA-04IN 97 (1.656) Cm (15:117)
TOF MS ES+
434.1549 6.87e8
435.1549
304.1458
276.1177
209.0505
229.1113
157.9812
348.0126
406.1239
m/z

Copies of ^{1}H (300 MHz, DMSO-d_6/CCl_4 (1:3)) and ^{13}C (300 MHz, DMSO-d_6/CCl_4 (1:3)) spectra, HRMS of **5k**

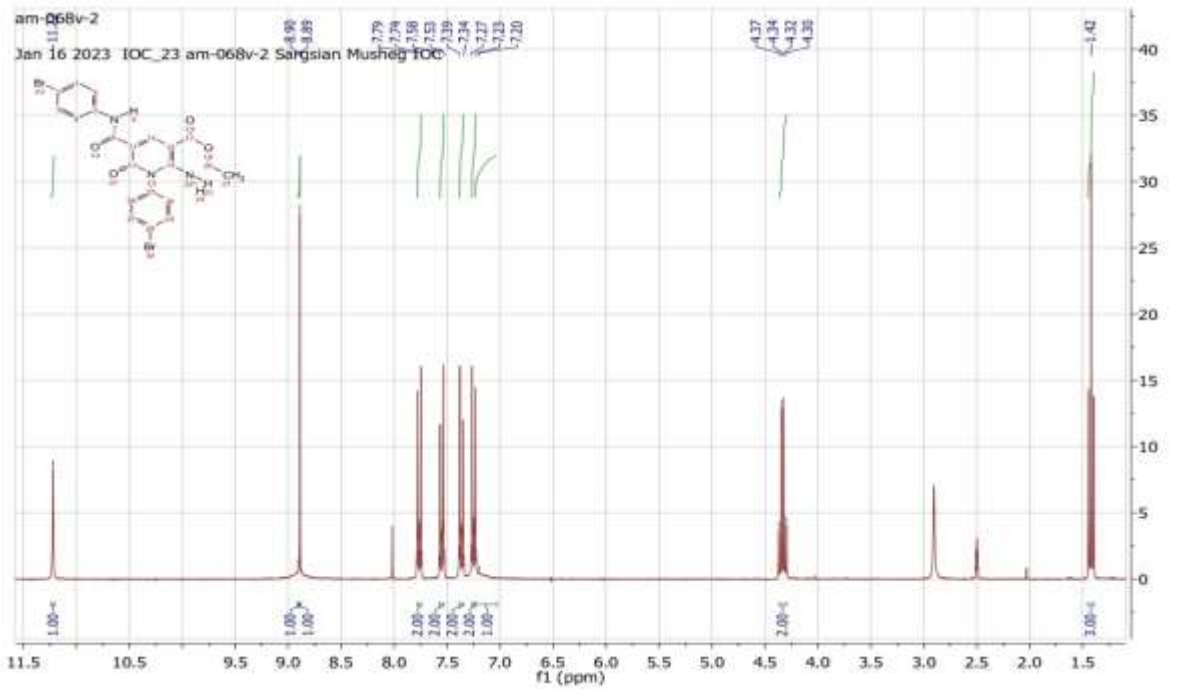


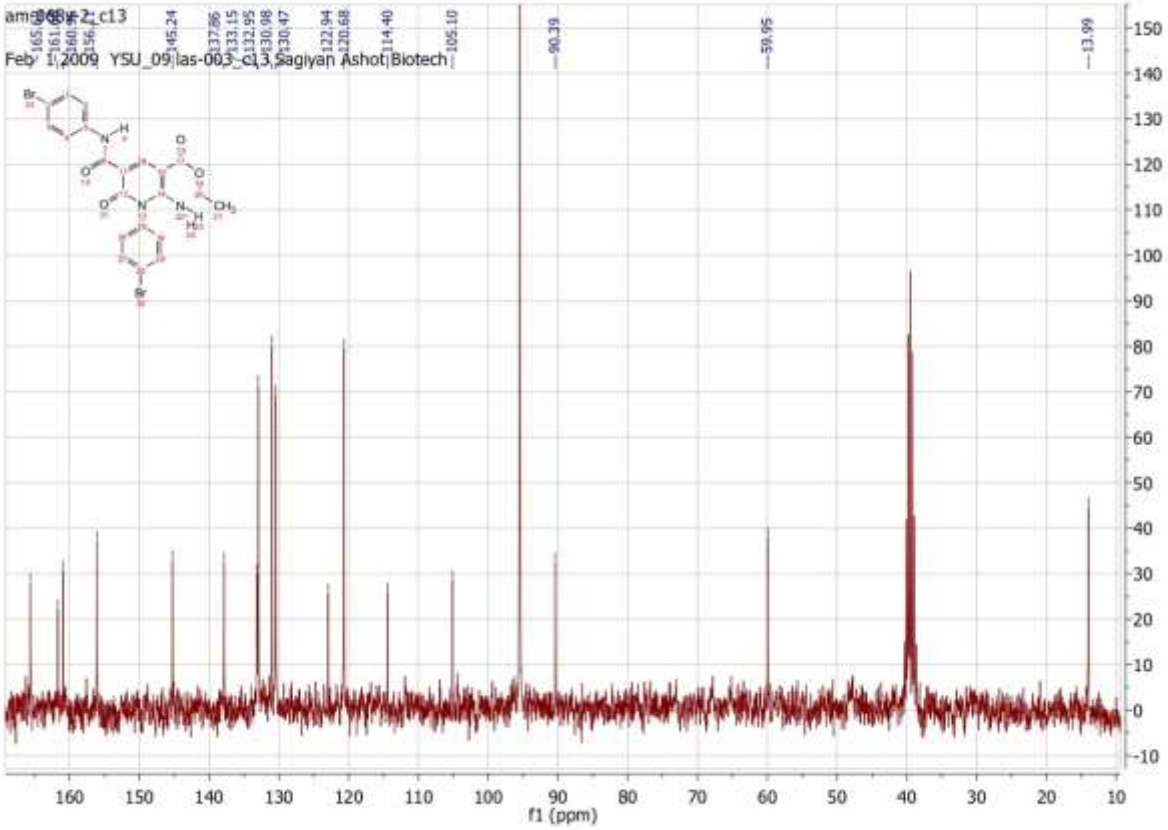


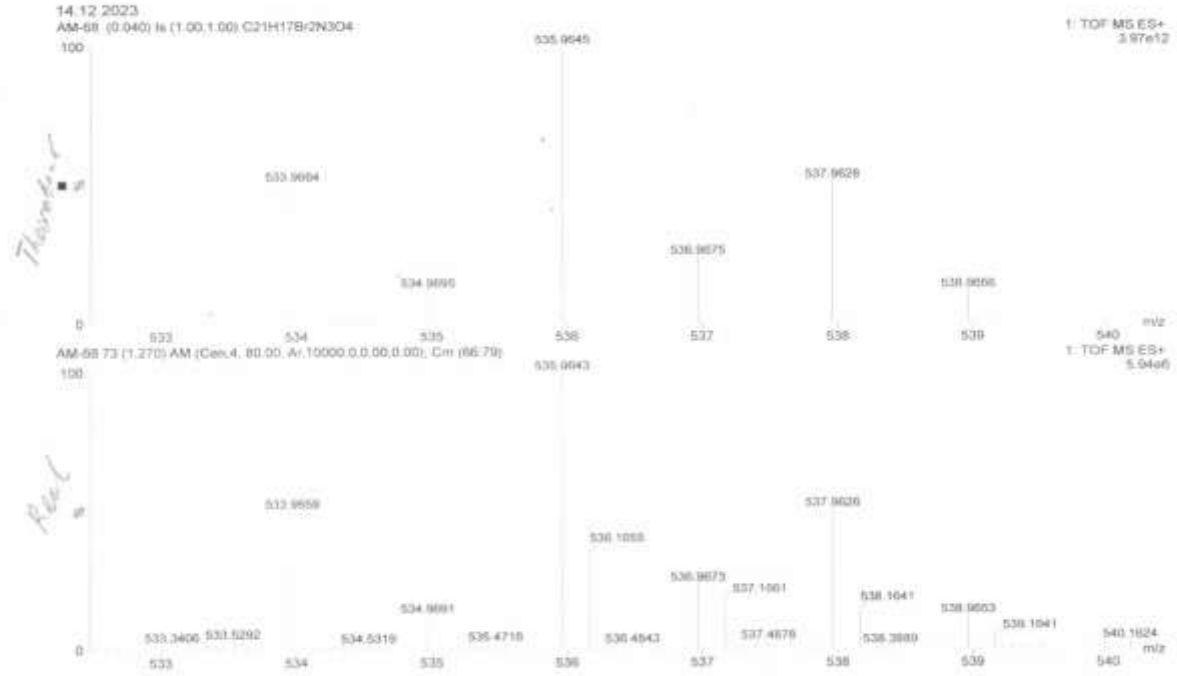

14.12.2023
AC-266 (0.040) Is (1.00,1.00) C23H23N3O4
100
406.1767
1: TOF MS ES+
7.83e12
407.1798
408.1825
405
406
407
408
409
m/z
AC-266 9 (0.175) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.00); Cm (4.43)
406.1756
1: TOF MS ES+
1.36e8
407.1787
405.0799
405.2090
405.3311
405.9151
406.3259
406.6626
407.0817
407.3265
407.4242
408.0752
408.1811
408.3124
409.0345
409.1834
409.3803

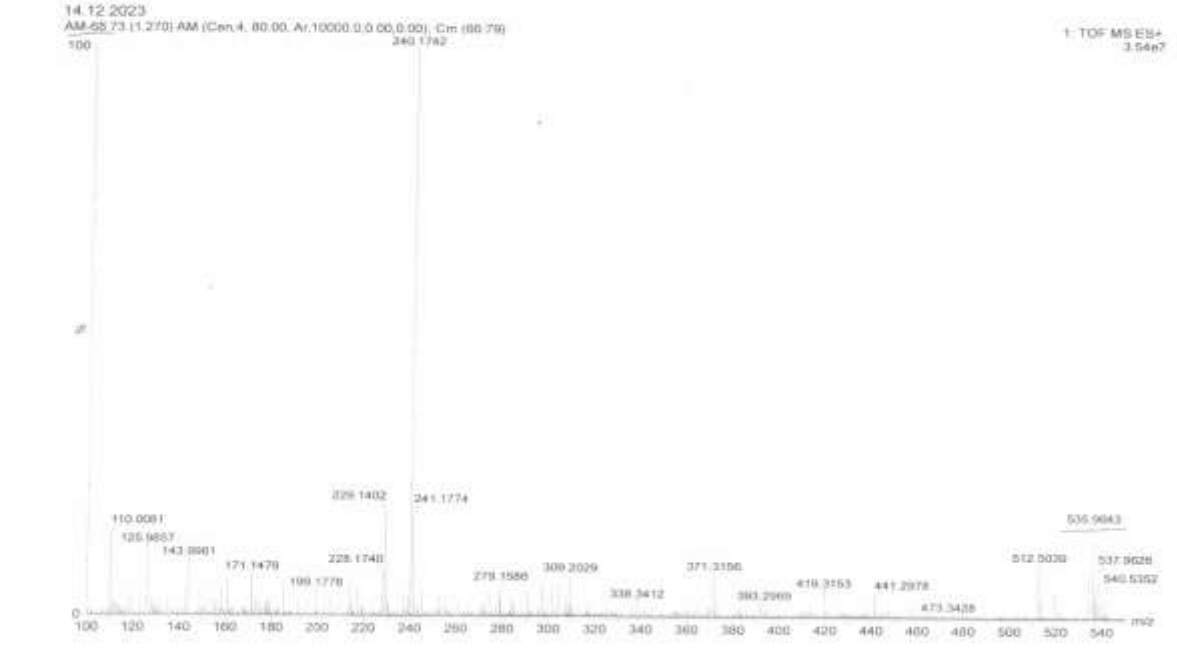

14.12.2023
AC-266 9 (0.175) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.00); Cm (4.43)
100
m/z
1: TOF MS ES+
406.1756
1.36e8
407.1787
240.1737
143.9958
158.0018
110.0079
171.1477
199.1786
223.0028
229.1400
214.0688
243.0777
252.0832
279.1582
297.2779
309.0766
338.3408
355.0990
383.1003
384.1011
408.1811
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
400
m/z

Copies of ^{1}H (300 MHz, DMSO-d_6/CCl_4 (1:3)) and ^{13}C (300 MHz, DMSO-d_6/CCl_4 (1:3)) spectra, HRMS of **5l**






14.12.2023
AM-68 (0.040) Is (1.00,1.00) C21H17Br2N3O4
1: TOF MS ES+
3.97e12
535.9645
533.9664
537.9629
536.9675
534.9695
538.9656
AM-68 73 (1.270) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.00); Cm (66:79)
1: TOF MS ES+
5.94e6
535.9643
533.9659
537.9626
536.1055
536.9673
537.1061
538.1041
534.9691
538.9653
539.1041
533.3406
533.5292
534.5319
535.4715
536.4843
537.4878
538.3889
540.1624
m/z


14.12.2023
AM-68 73 (1.270) AM (Cen,4, 80.00, Ar,10000.0,0.00,0.00); Cm (66:79)
1: TOF MS ES+
3.54e7
240.1742
229.1402
241.1774
110.0081
125.9857
143.9981
171.1479
228.1740
199.1778
279.1586
309.2029
338.3412
371.3156
393.2969
419.3153
441.2978
473.3428
512.5030
535.9643
537.9626
540.5352
m/z

Diffraction measurements of a single crystal of compound **5d** were carried out at room temperature on an Enraf-Nonius CAD-4 autodiffractometer (MoKα radiation, graphite monochromator, θ/2θ scanning). The parameters of the triclinic unit cell were determined and refined based on 24 reflections with 11.97<θ<13.86. The absorption of X-rays was taken into account using the "psi-scan" method.[1] The structure was solved using the direct method. The coordinates of hydrogen atoms were determined from difference Fourier syntheses and refined independently. The structure was refined by full-matrix least squares in the anisotropic approximation for non-hydrogen atoms and the isotropic approximation for hydrogen atoms. All structural calculations were carried out using the SHELXTL software package.[2]

Crystallographic data in CIF format have been deposited at the Cambridge Crystallographic Data Center, deposit number CCDC 2283418.

Main crystallographic and experimental data are shown in Table 1.

Table 1. Main crystallographic characteristics and experimental data

Crystallographic characteristics	
Compound	5d
Brutto formula	$C_{21}H_{17}N_3O_4Cl_2$
Molecular weight	446.27
Singony	Triclinic
Space group	P-1
a, b, c [Å]	9.2945(19), 10.567(2), 11.529(2)
α, β, γ [deg.]	109.94(3), 107.01(3), 90.60(3)
V [Å^3]	1010.2(4)
Z	2
Density (calc.) [g/cm^3]	1.467
μ(MoKα) [mm^{-1}], T_{min}, T_{max}	0.356, 0.84889, 0.87066
F(000)	460
Crystal size [mm]	0.36×0.30×0.24
Experimental data	
Temperature (K)	288
Radiation [Å]	0.71073
θ_{min}, θ_{max} [deg.]	2.0; 30.0
Scan area	0≤h≤13; -14≤k≤14; -16≤l≤15
Number of measured reflections	6207
Number of observed reflections with [I > 2.0 σ(I)]	3701
Calculated data	
Nref, Npar	5869, 328
R, wR2, S	0.0510, 0.1343, 1.02

The molecular structure of compound **5d** is shown in Figure 1.

Conformational calculations showed that all cyclic fragments are planar, the maximum deviation of atoms does not exceed 0.0295(4)Å.

In the molecule of compound **5d**, intramolecular hydrogen bonds are observed between the N8–H8A···O10, N23–H23···O7, N23–H23···Cl30 atoms (Fig. 1). The geometry of hydrogen bonds is shown in Table 2. In the three-dimensional packing of molecules, intermolecular interactions are mainly due to Van der Waalsian forces

Table 2. Geometry of hydrogen bonds.

Atoms	D-H(Å)	H····A(Å)	D····A(Å)	D-H····A(deg.)
N8-H8A···O10	0.85(3)	1.99(3)	2.661(4)	135(3)
N23-H23···O7	0.85(2)	1.94(2)	2.670(2)	143(2)
N23-H23···Cl30	0.85(2)	2.48(3)	2.936(2)	115(2)

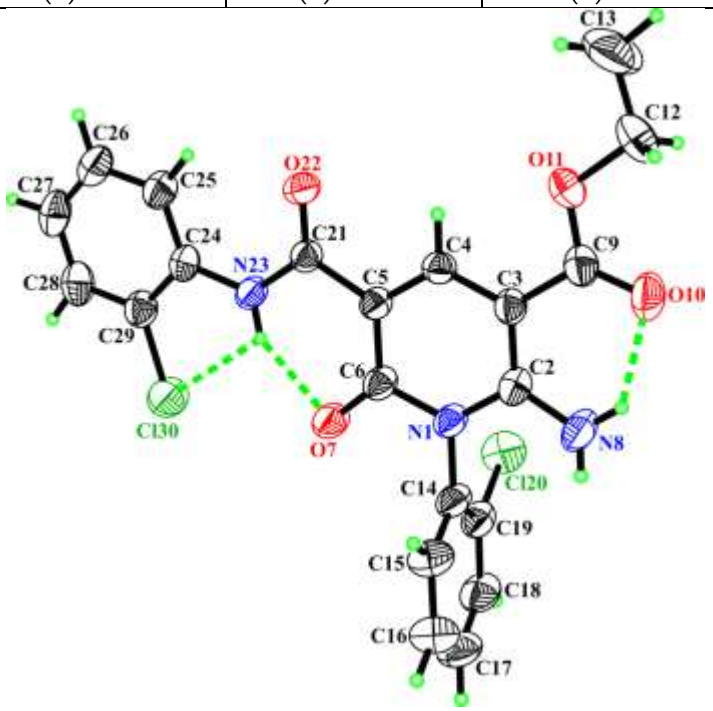


Figure 1. The molecular structure of compound **5d** with conditionally accepted numbering of atoms. Ellipsoids of anisotropic thermal vibrations are depicted at the 50% probability level.

1. A. C. T. North, D. C. Phillips and F. S. Mathews, A Acta Cryst. (1968), A24, 351-359.

2. G.M. Sheldrick (2015) "Crystal structure refinement with SHELXL", Acta Cryst., C71, 3-8