Supporting Information

α-Haloacrylates as acceptors in the [3+2] cycloaddition reaction with NaN_3: an expedient approach to *N*-unsubstituted 1,2,3-triazole-4-carboxylates

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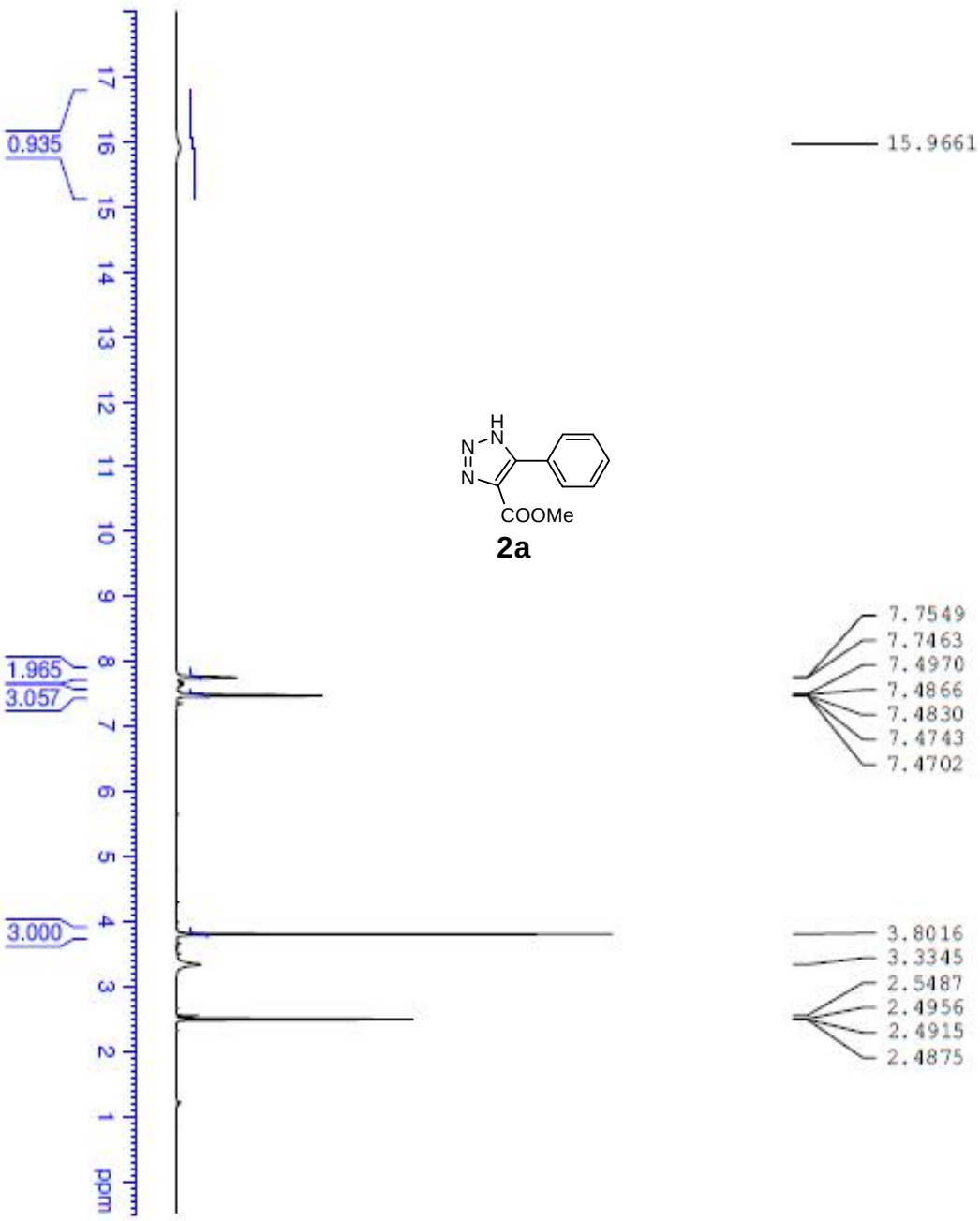
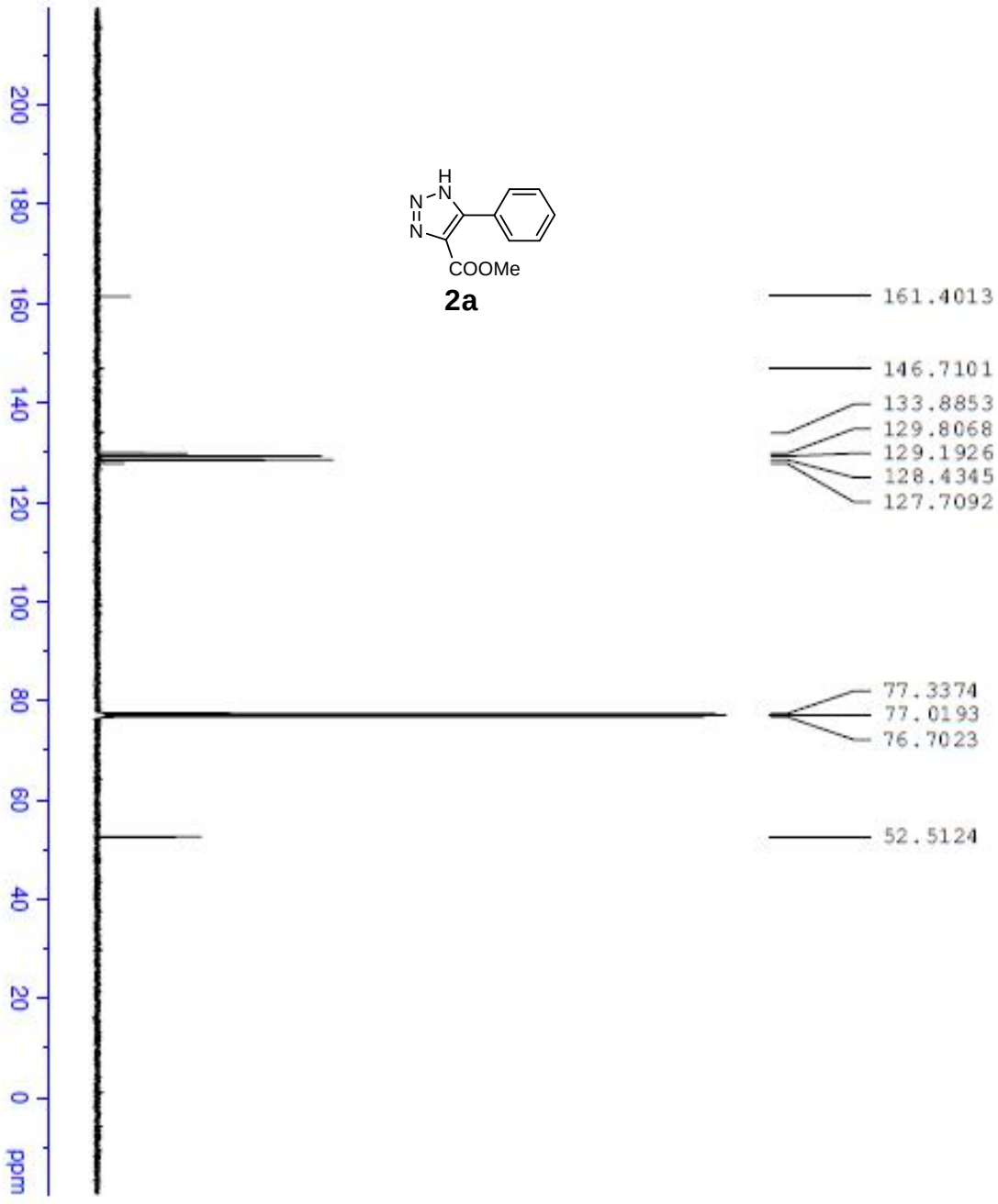


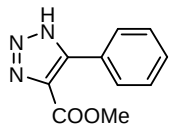
^{1}H NMR (400 MHz) in DMSO-d_6 (**2a**)



^{13}C NMR (100 MHz) in $CDCl_3$ (**2a**)

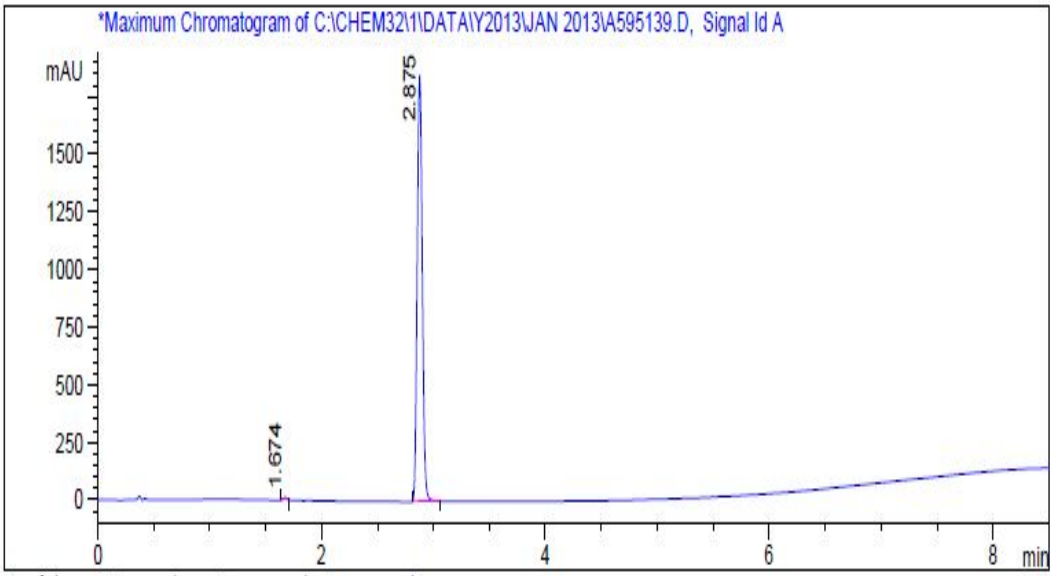
Method info : A: 0.1% TFA IN H2O , B:0.1% TFA IN ACN ; Flow Rate:2.0 ml/min
COLUMN:XBridge C8 (50x4.6mm, 3.5µm), +ve mode

TIME	%B
0	05
8.0	100
8.1	100
8.5	05
10	05



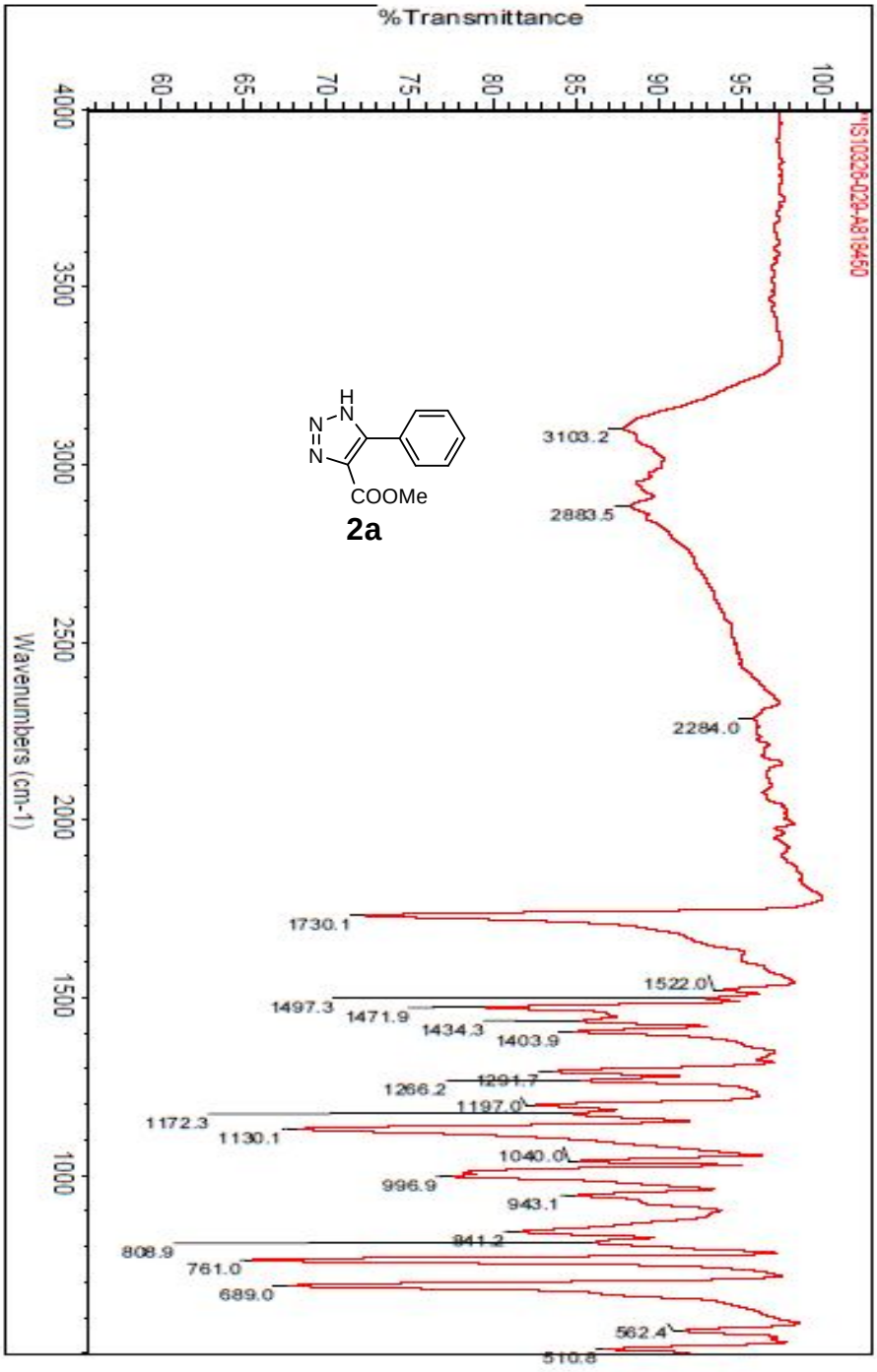
2a

->

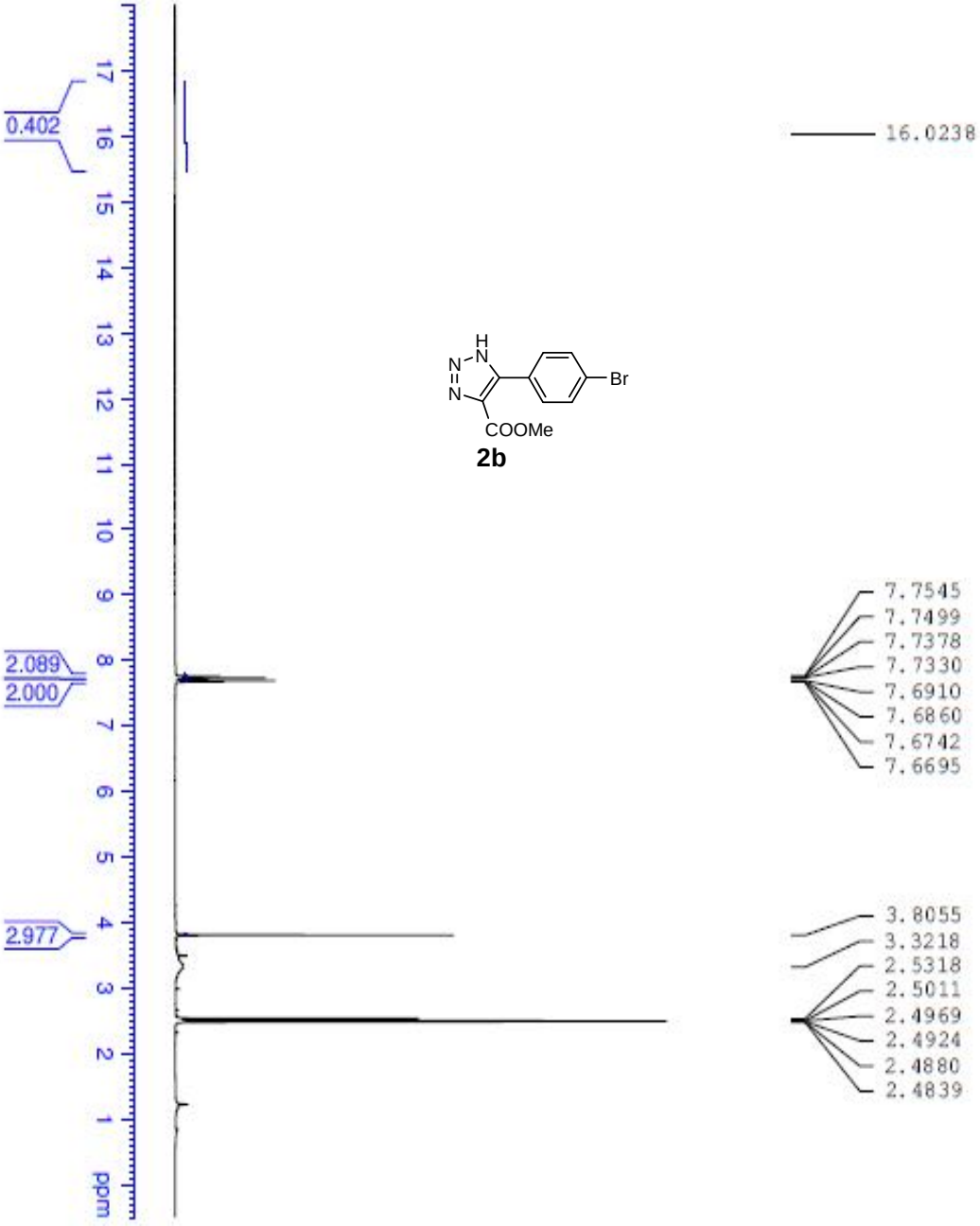


Peak No	RT min	Area	Area %
1	1.674	2.589e+001	0.440
2	2.875	5.853e+003	99.560

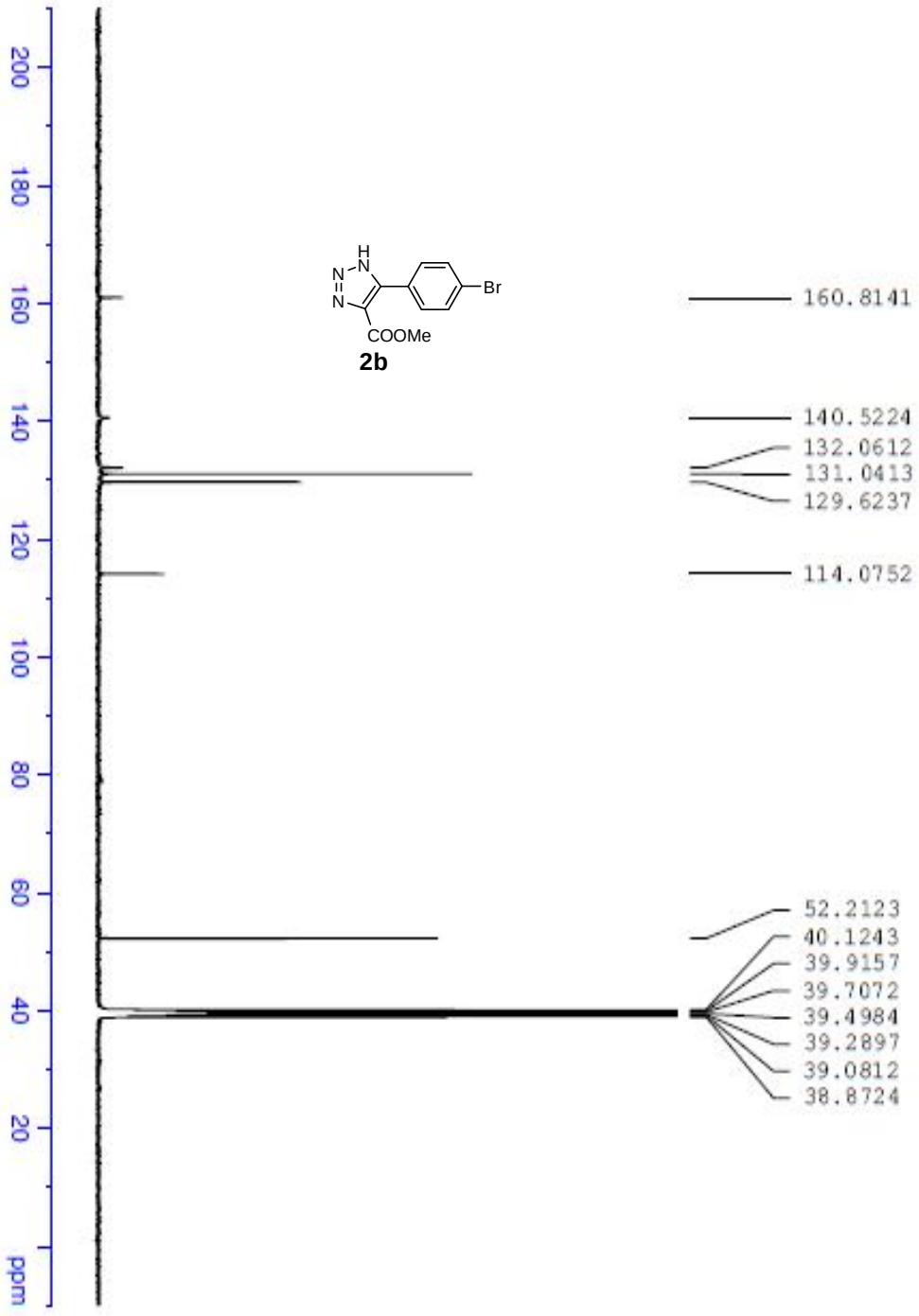
HPLC (**2a**)


%Transmittance
Wavenumbers (cm-1)
4000
3500
3000
2500
2000
1500
1000
60
65
70
75
80
85
90
95
100
3103.2
2883.5
2284.0
1730.1
1522.0
1497.3
1471.9
1434.3
1403.9
1291.7
1266.2
1197.0
1172.3
1130.1
1040.0
996.9
943.1
841.2
808.9
761.0
689.0
562.4
510.8
COOMe
2a

IR (**2a**)



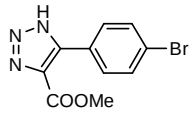
^{1}H NMR (400 MHz) in DMSO-d_6 (**2b**)



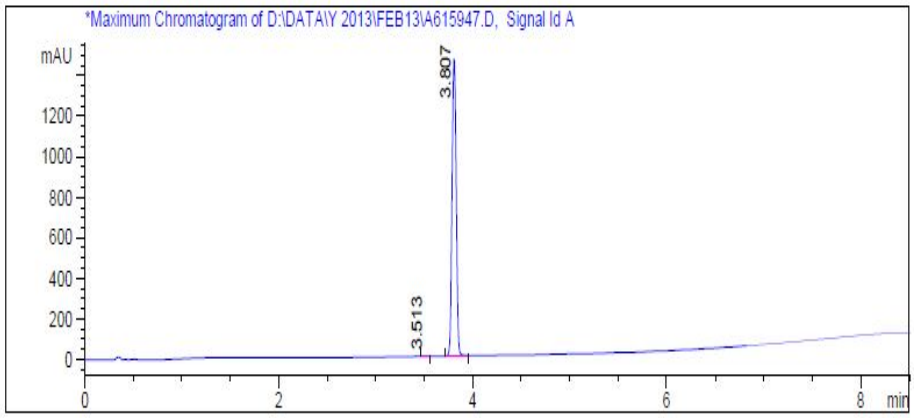
^{13}C NMR (100 MHz) in DMSO-d_6 (**2b**)

Method info : A : 0.1%TFA IN H2O B: 0.1%TFA IN ACN Flow = 2.0 mL/min
COLUMN:XBridge C8 (50X4.6)mm,3.5µm , +ve mode

Time	% of B
0	5
8.0	100
8.1	100
8.5	5
10.0	5

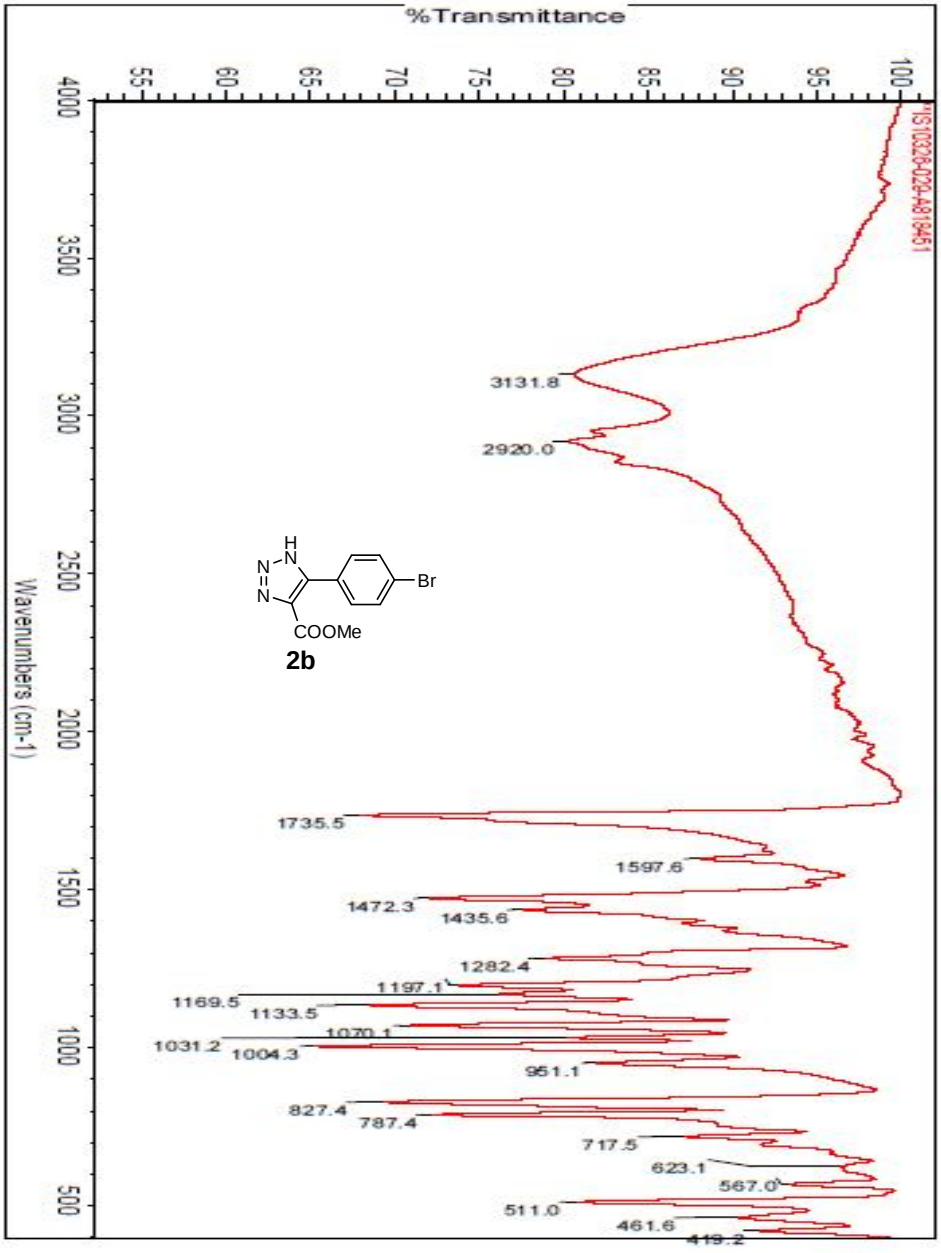


2b

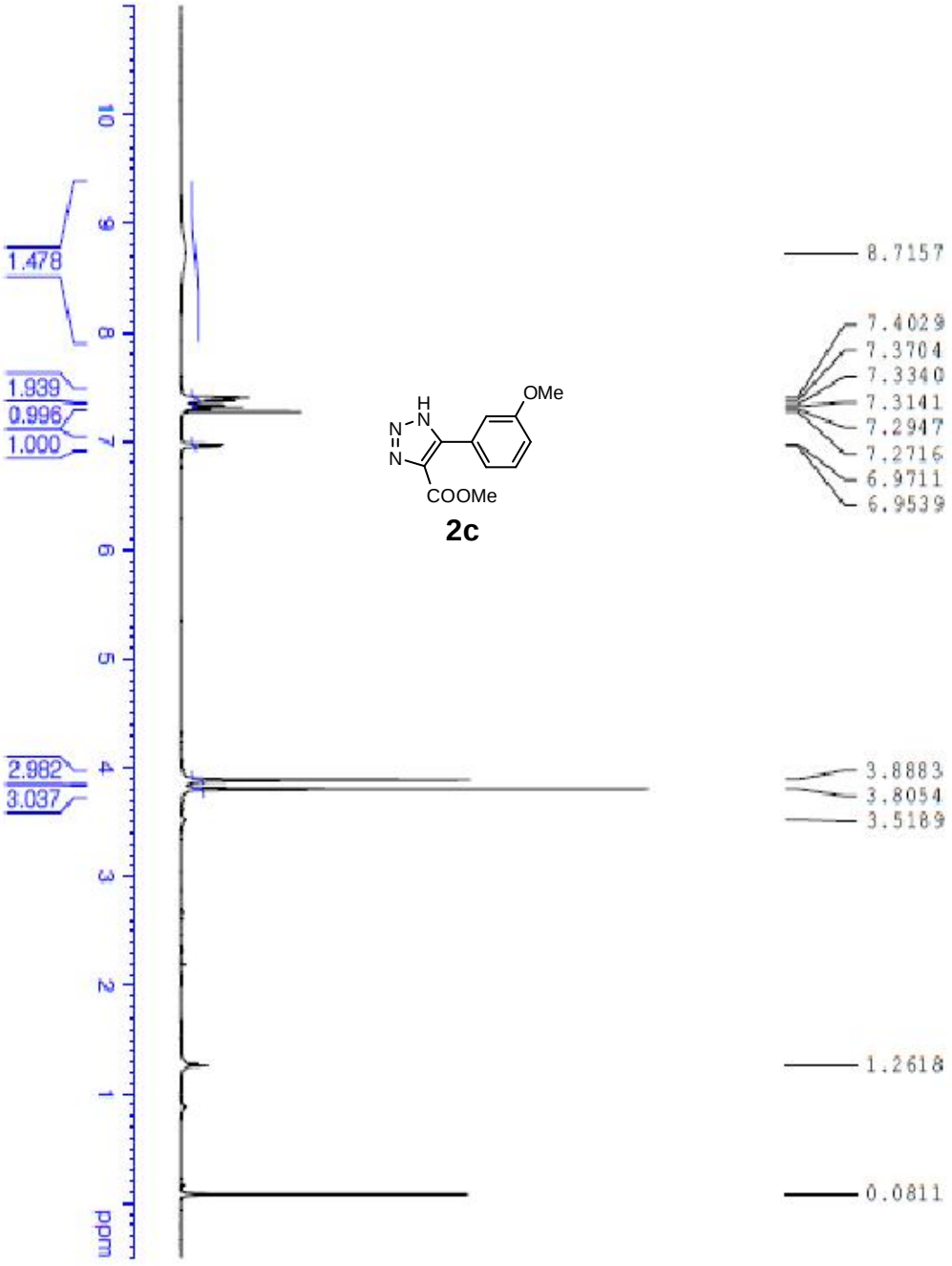


Peak No	RT min	Area	Area %
1	3.513	9.212e+000	0.209
2	3.807	4.395e+003	99.791

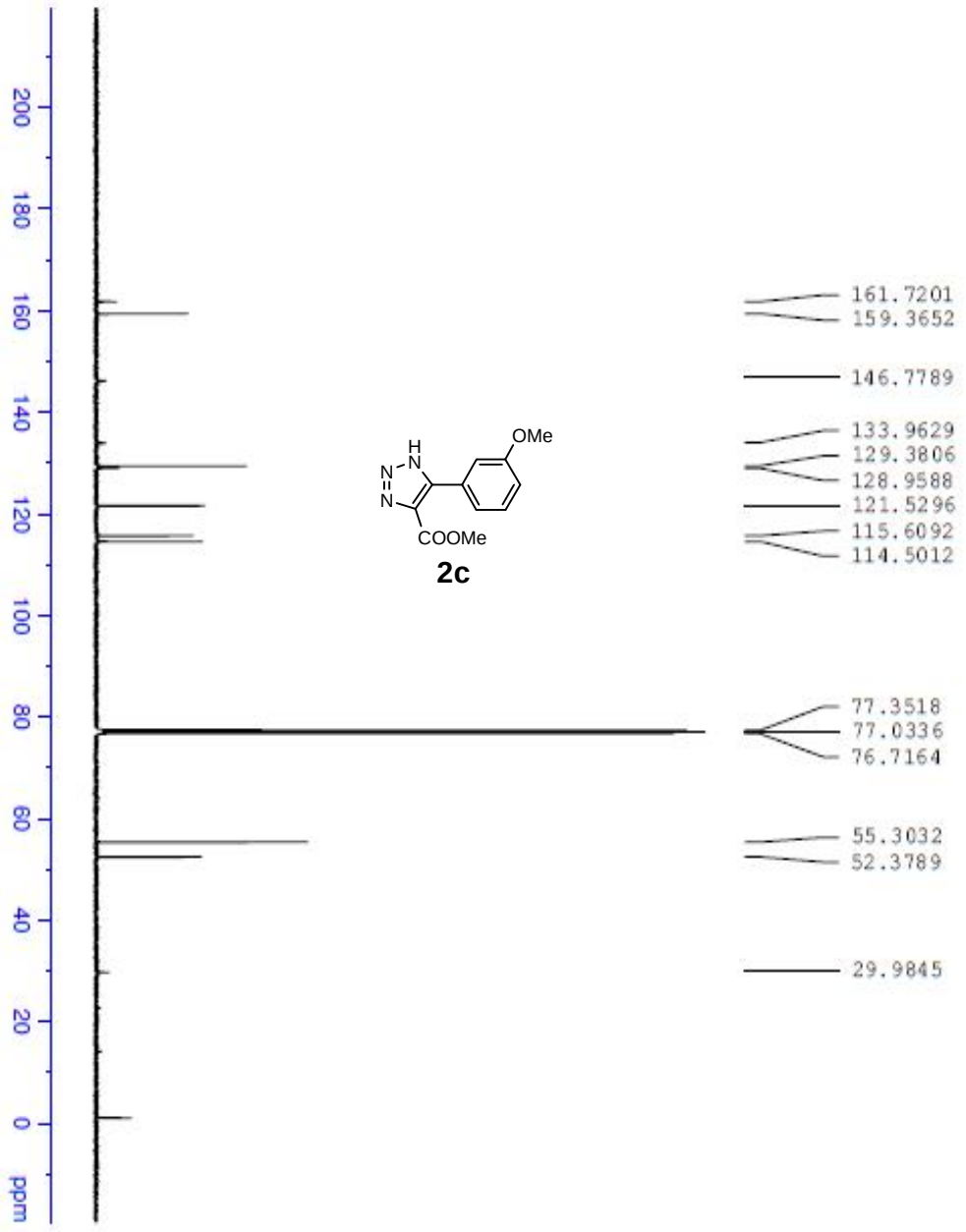
HPLC (**2b**)


%Transmittance
Wavenumbers (cm-1)
3131.8
2920.0
1735.5
1597.6
1472.3
1435.6
1282.4
1197.1
1169.5
1133.5
1070.1
1031.2
1004.3
951.1
827.4
787.4
717.5
623.1
567.0
511.0
461.6
419.2
Br
COOMe
2b

IR (**2b)**



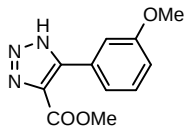
^{1}H NMR (400 MHz) in $CDCl_3$ (**2c**)



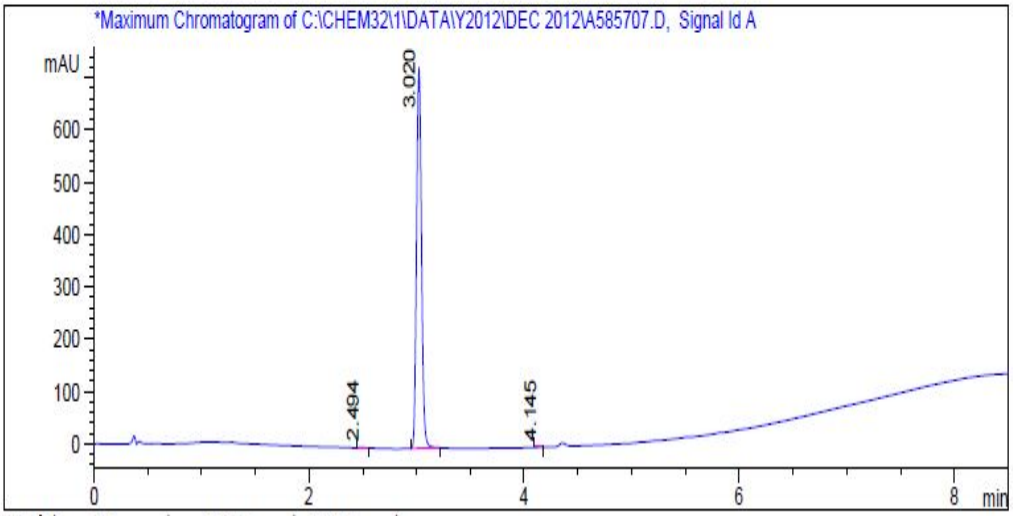
^{13}C NMR (100 MHz) in $CDCl_3$ (**2c**)

Method info : A: 0.1% TFA IN H2O , B:0.1% TFA IN ACN ; Flow Rate:2.0 ml/min
COLUMN:XBridge C8 (50x4.6mm, 3.5µm), +ve mode

TIME	%B
0	05
8.0	100
8.1	100
8.5	05
10	05

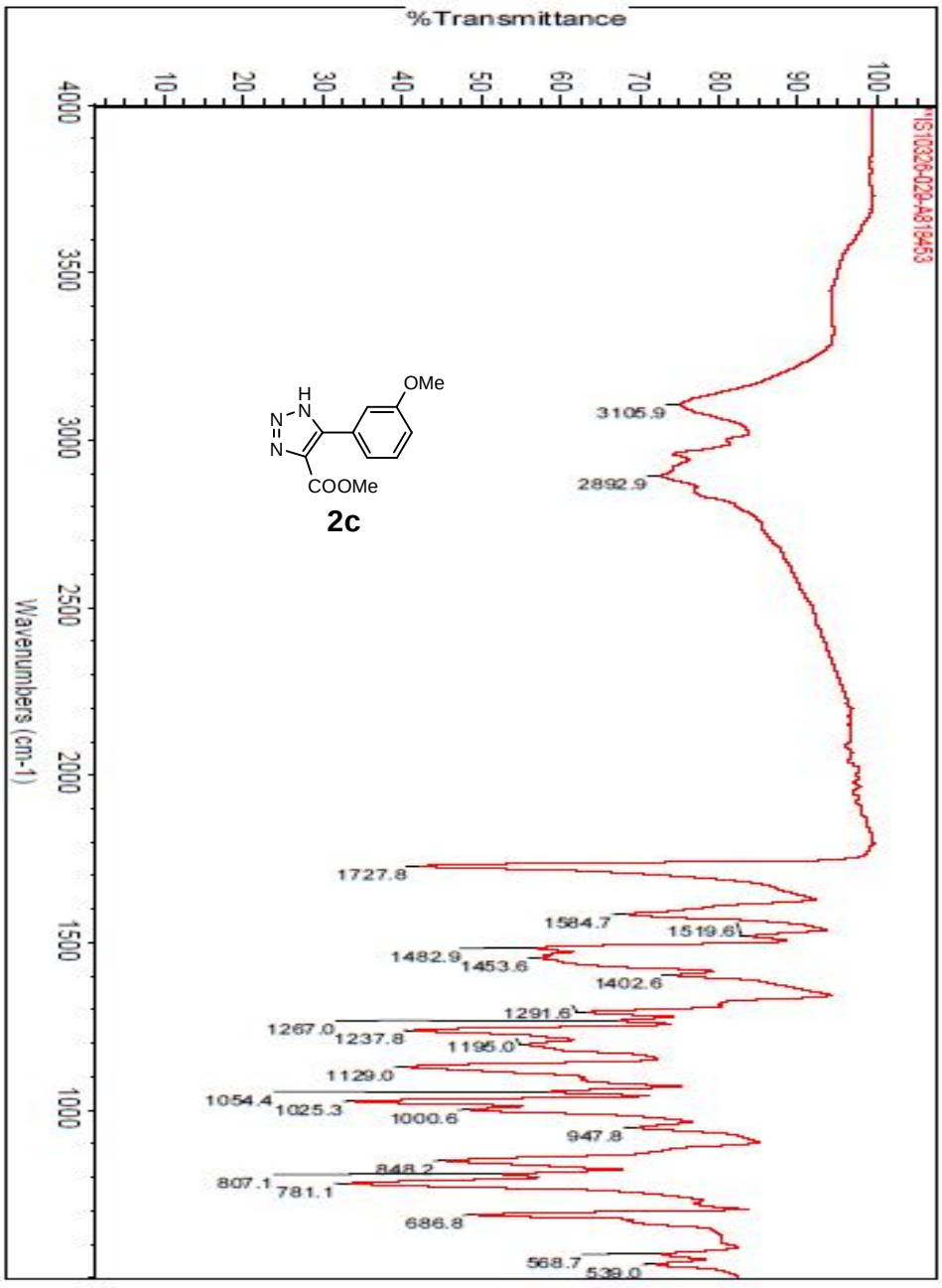


2c ->

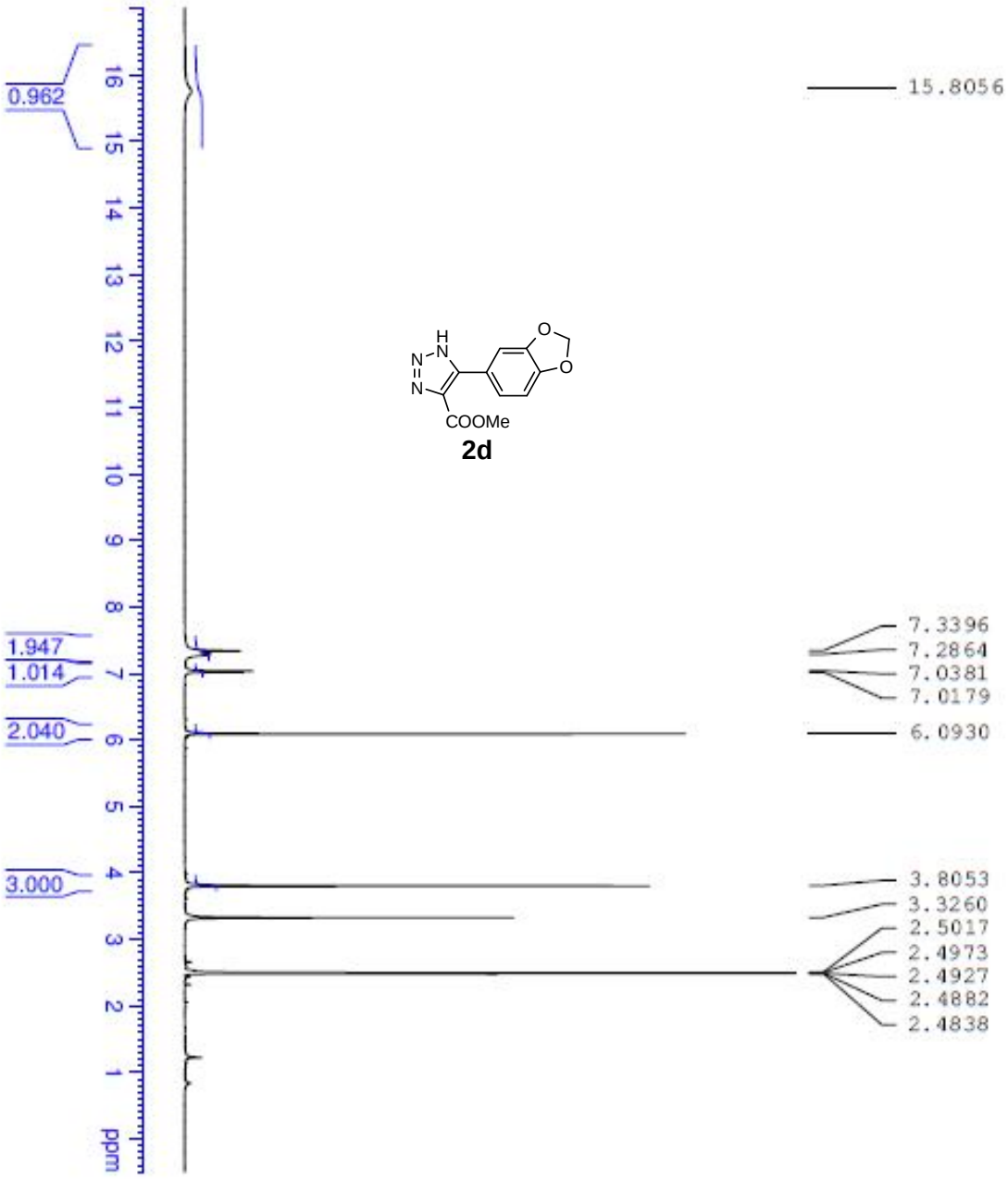


Peak No	RT min	Area	Area %
1	2.494	4.465e+000	0.190
2	3.020	2.340e+003	99.746
3	4.145	1.500e+000	0.064

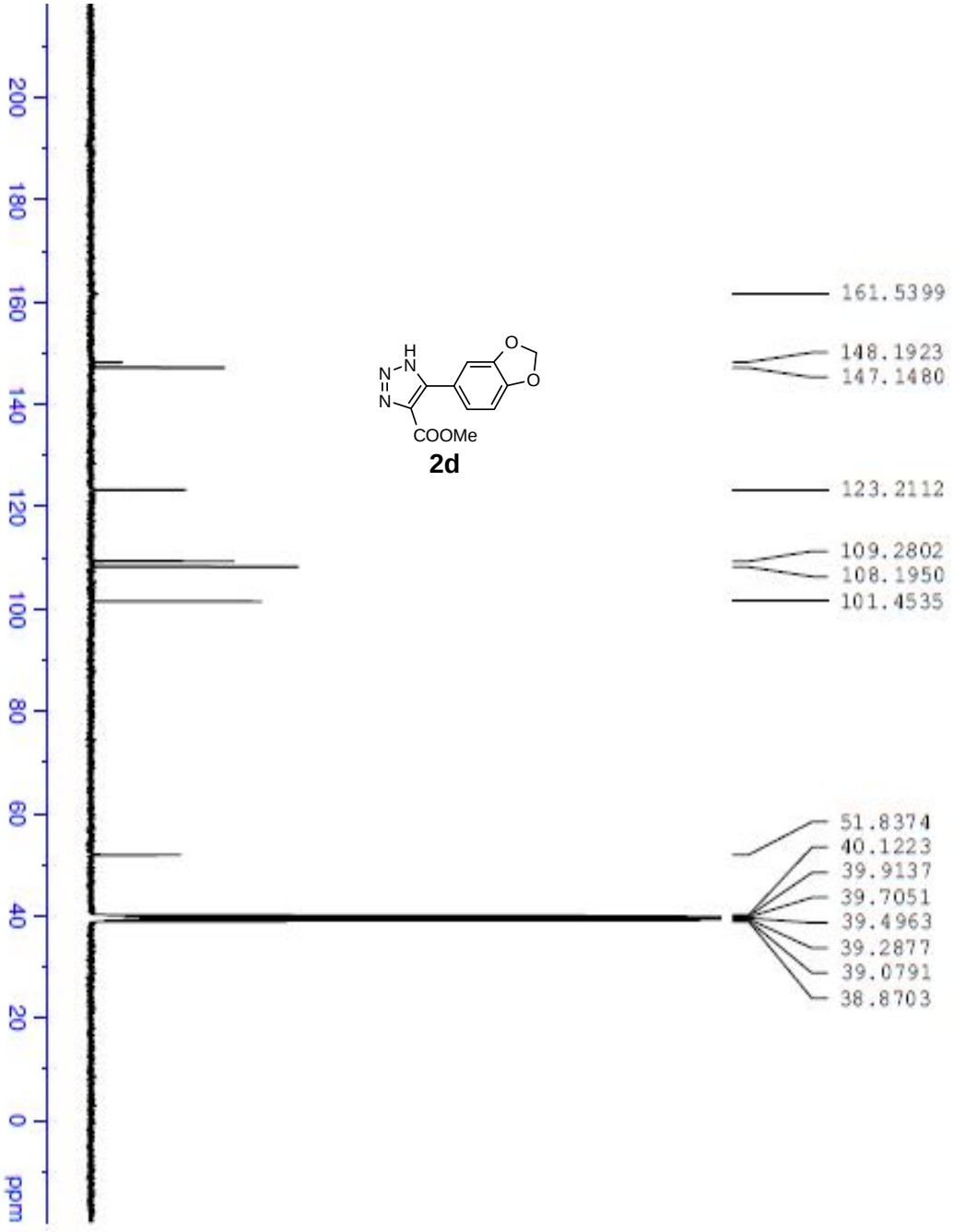
HPLC (2c)


%Transmittance
Wavenumbers (cm-1)
OMe
COOMe
2c
3105.9
2892.9
1727.8
1584.7
1519.6
1482.9
1453.6
1402.6
1291.6
1267.0
1237.8
1196.0
1129.0
1054.4
1025.3
1000.6
947.8
848.2
807.1
781.1
686.8
568.7
539.0

IR (**2c**)



^{1}H NMR (400 MHz) in DMSO-d_6 (**2d**)



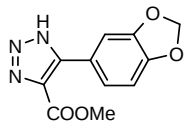
^{13}C NMR (100 MHz) in DMSO-d_6 (**2d**)

Method info :

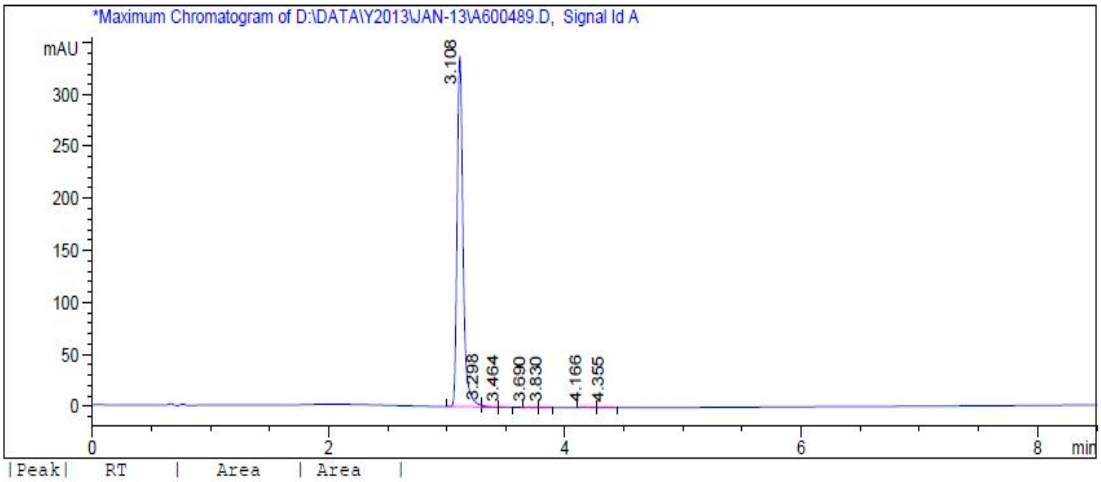
A-10mM NH4HCO3 IN H2O , B- ACN Flow: 1.0 ml/min

COLUMN: XBridge C8 (50X4.6mm, 3.5µm), -ve mode

TIME	%B
0	05
8.0	100
8.1	100
8.5	05
10	05

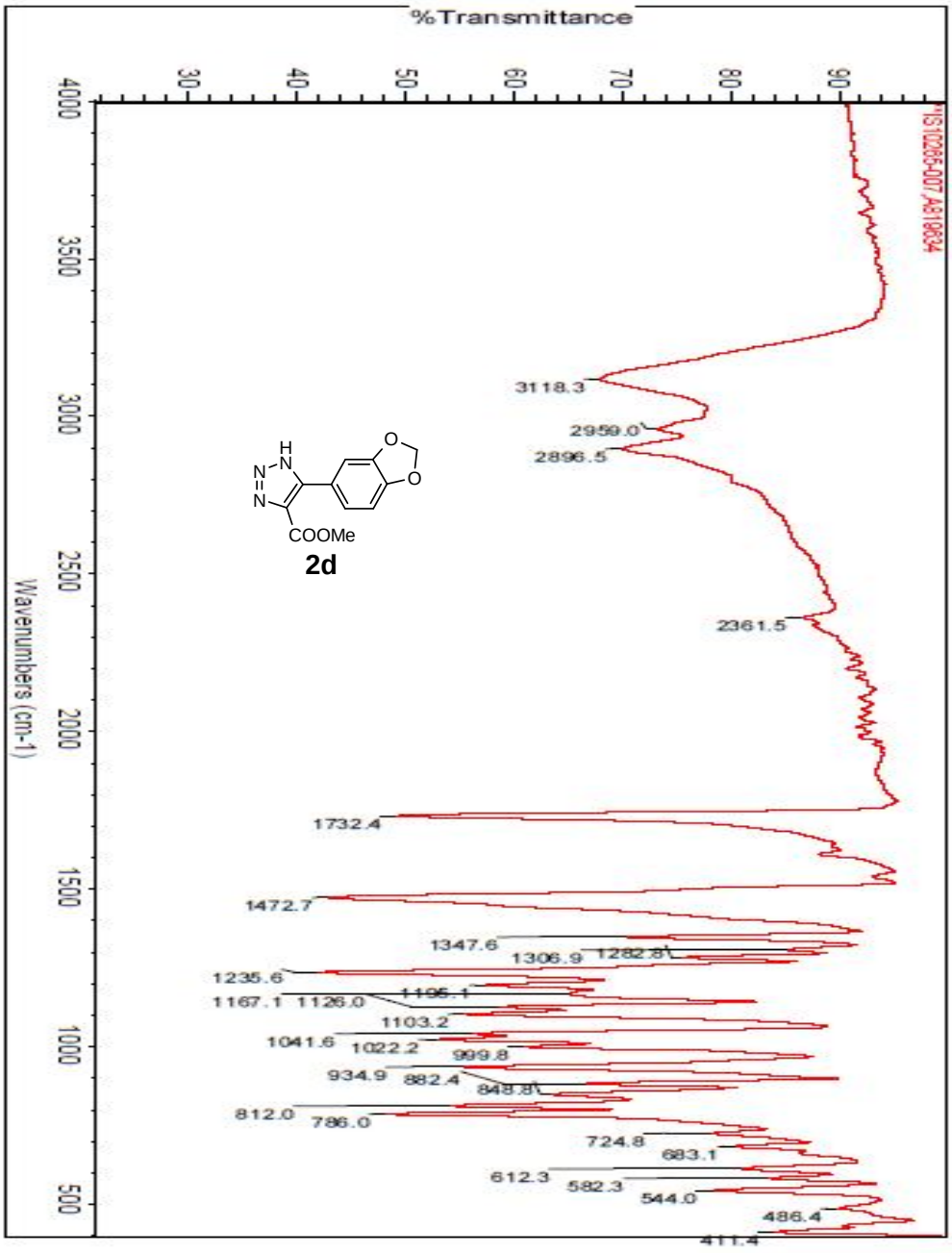


2d

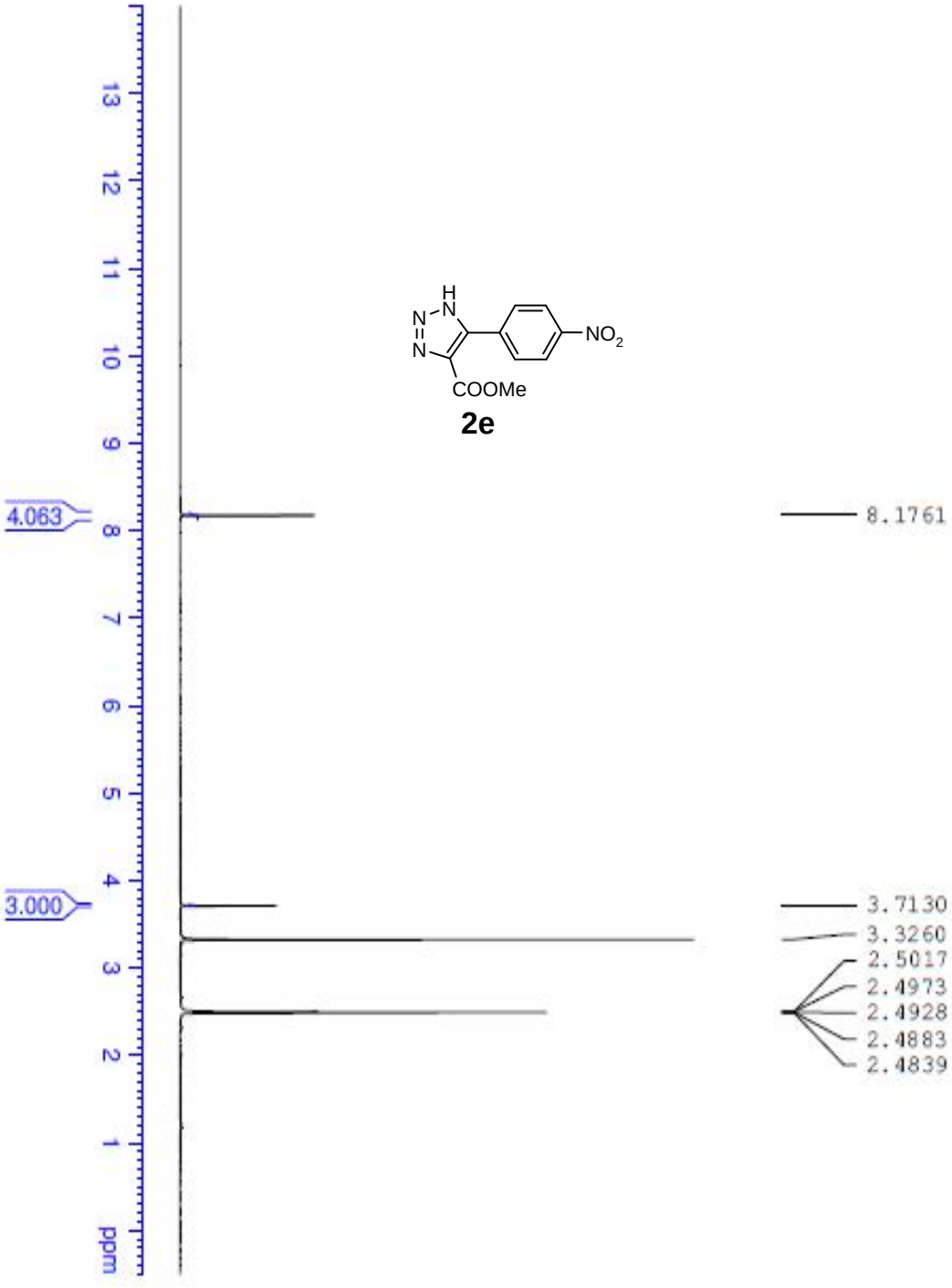


Peak No	RT min	Area	Area %
1	3.108	1.174e+003	99.060
2	3.298	4.422e+000	0.373
3	3.464	7.986e-001	0.067
4	3.690	9.200e-001	0.078
5	3.830	7.689e-001	0.065
6	4.166	3.010e+000	0.254
7	4.355	1.219e+000	0.103

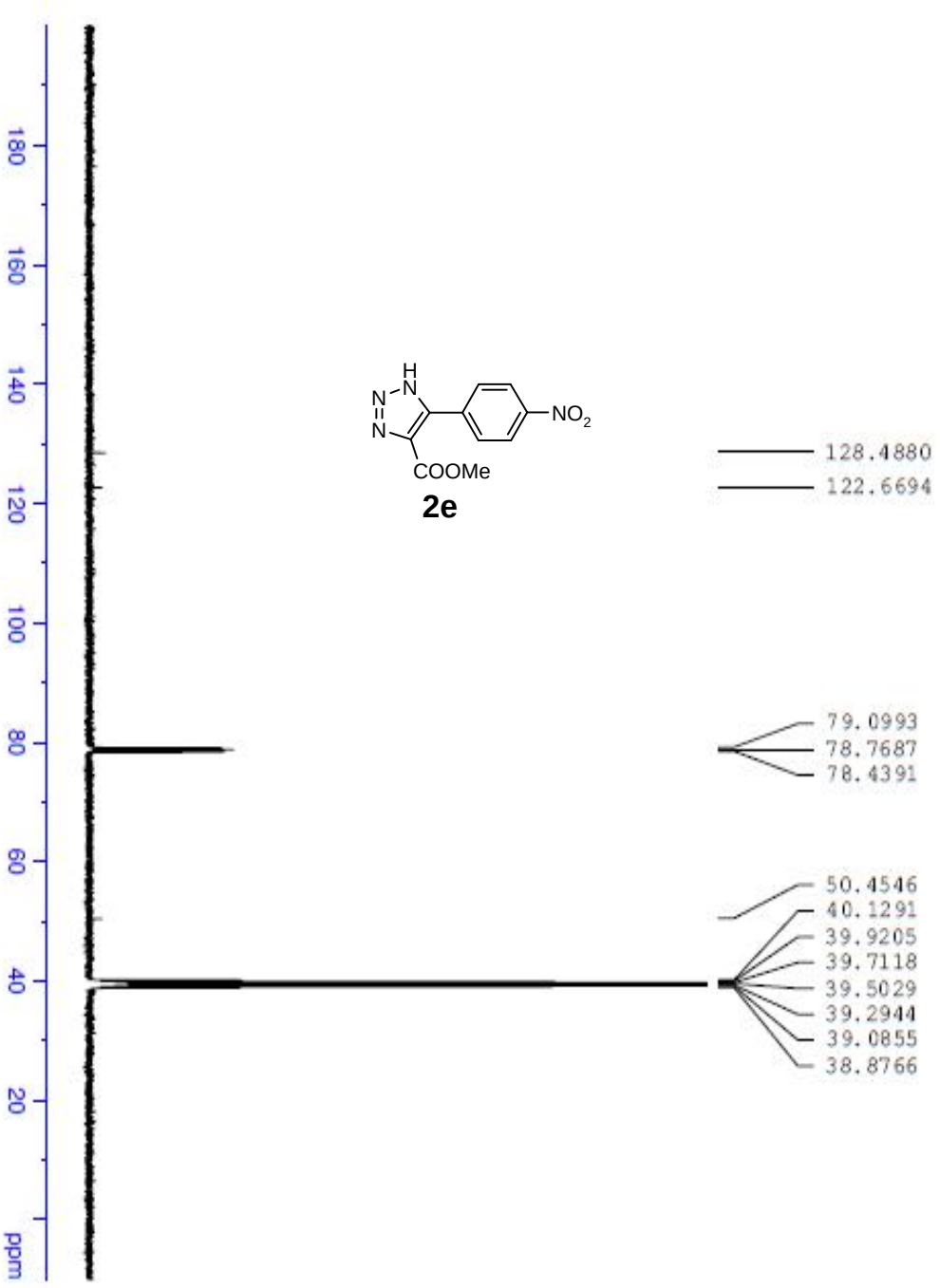
HPLC (**2d**)


%Transmittance
Wavenumbers (cm-1)
3118.3
2959.0
2896.5
2361.5
1732.4
1472.7
1347.6
1306.9
1282.8
1235.6
1196.1
1167.1
1126.0
1103.2
1041.6
1022.2
999.8
934.9
882.4
848.8
812.0
786.0
724.8
683.1
612.3
582.3
544.0
486.4
411.4
COOMe
2d

IR (**2d**)



^{1}H NMR (400 MHz) in DMSO-d_6 (**2e**)



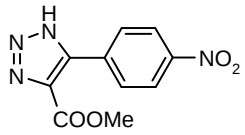
^{13}C NMR (100 MHz) in DMSO-d_6 (**2e**)

Method info :

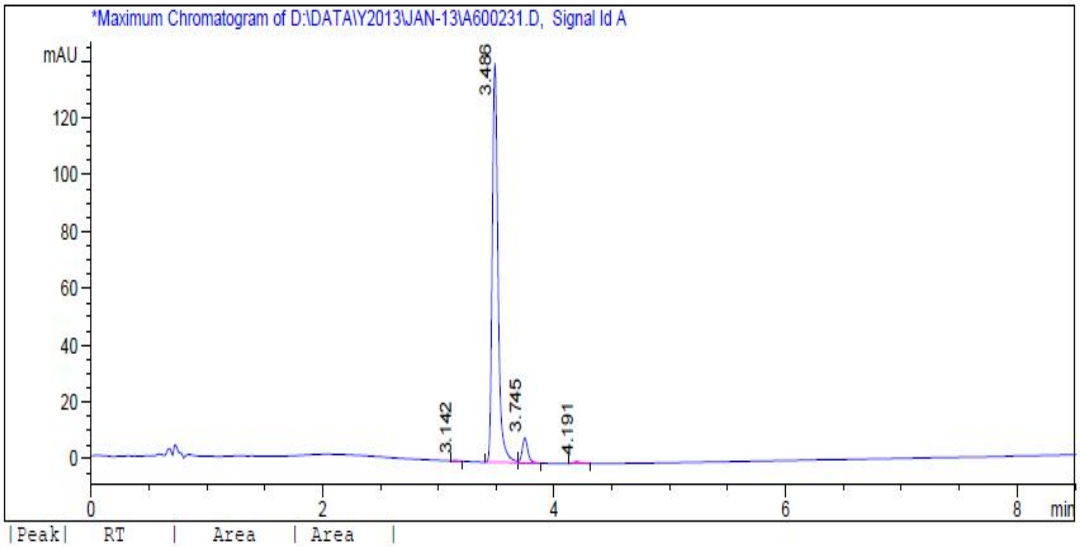
A-10mM NH4HCO3 IN H2O , B- ACN Flow: 1.0 ml/min

COLUMN: XBridge C8 (50X4.6mm, 3.5µm), -ve mode

TIME	%B
0	05
8.0	100
8.1	100
8.5	05
10	05

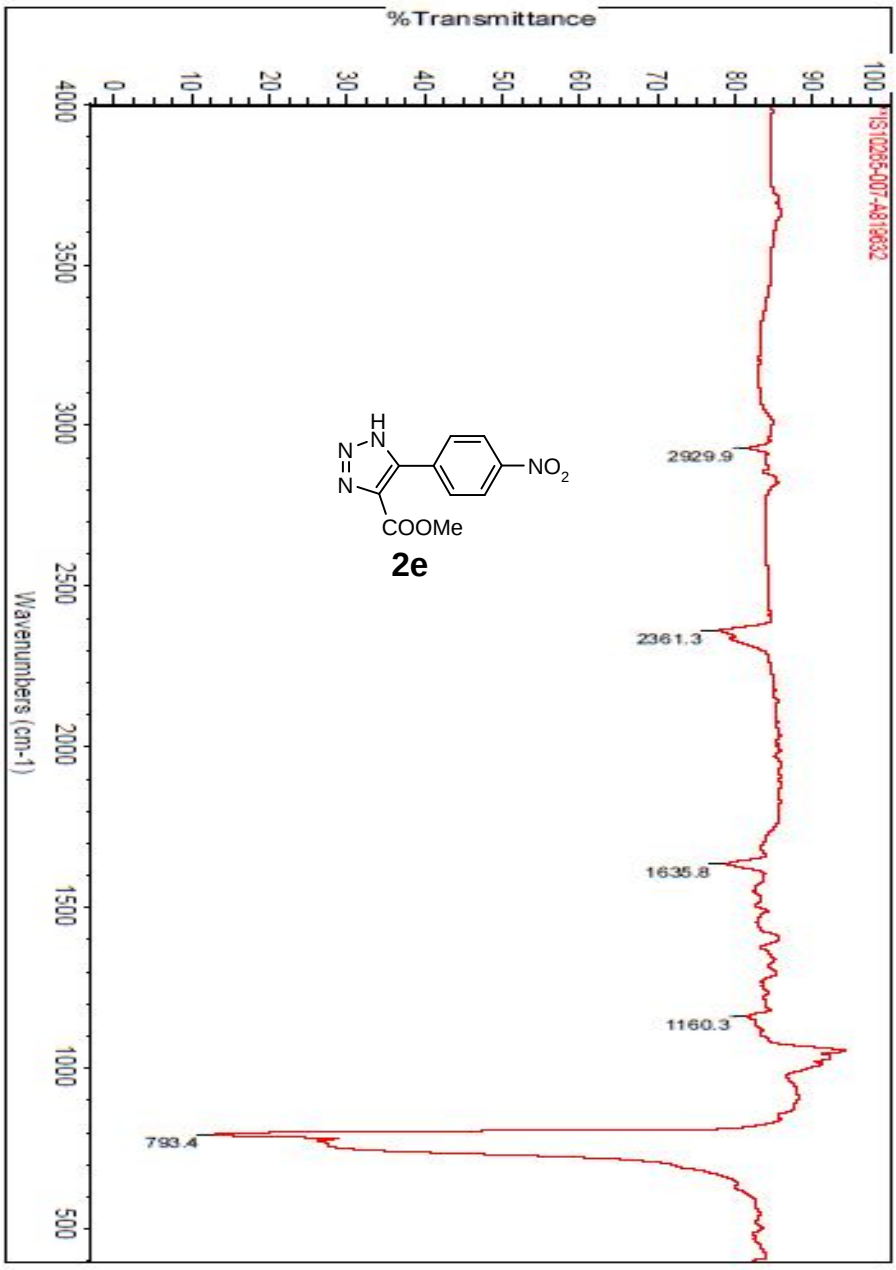


2e



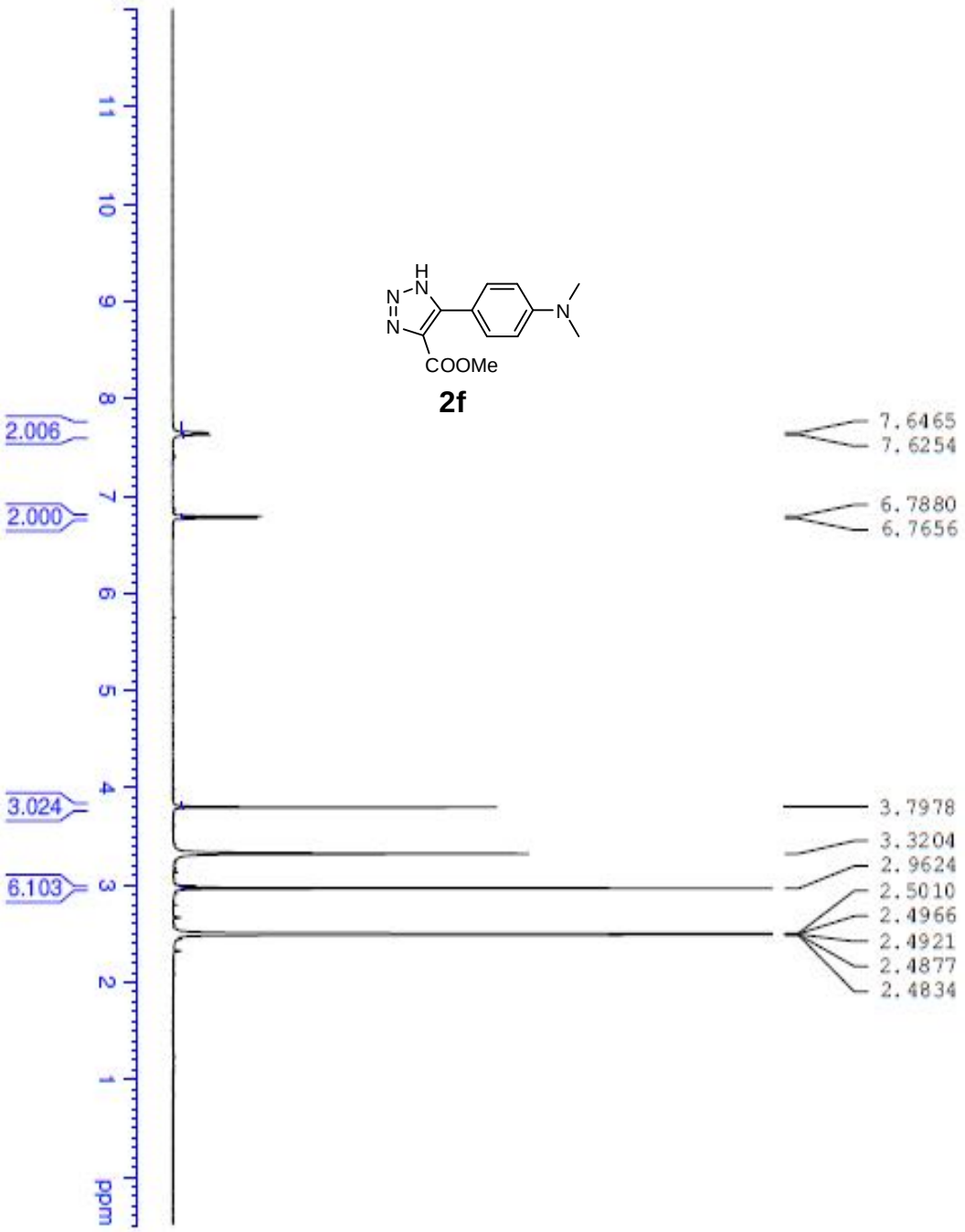
Peak No	RT min	Area	Area %
1	3.142	8.734e-001	0.168
2	3.486	4.870e+002	93.408
3	3.745	3.046e+001	5.842
4	4.191	3.042e+000	0.583

HPLC (**2e**)

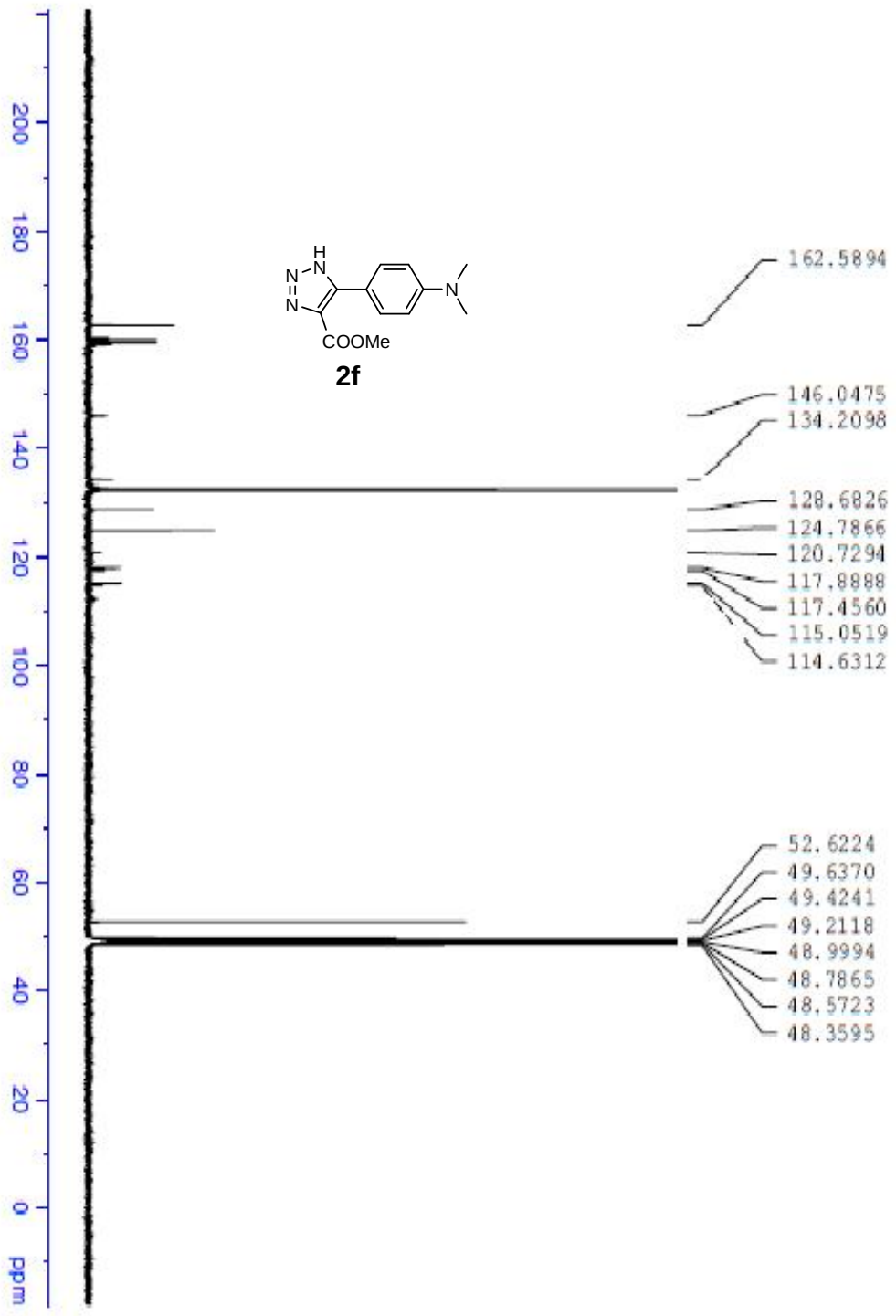

%Transmittance
Wavenumbers (cm-1)
2929.9
2361.3
1635.8
1160.3
793.4
COOMe
NO2

2e

IR (**2e**)



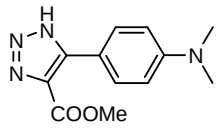
^{1}H NMR (400 MHz) in DMSO-d_6 (**2f**)



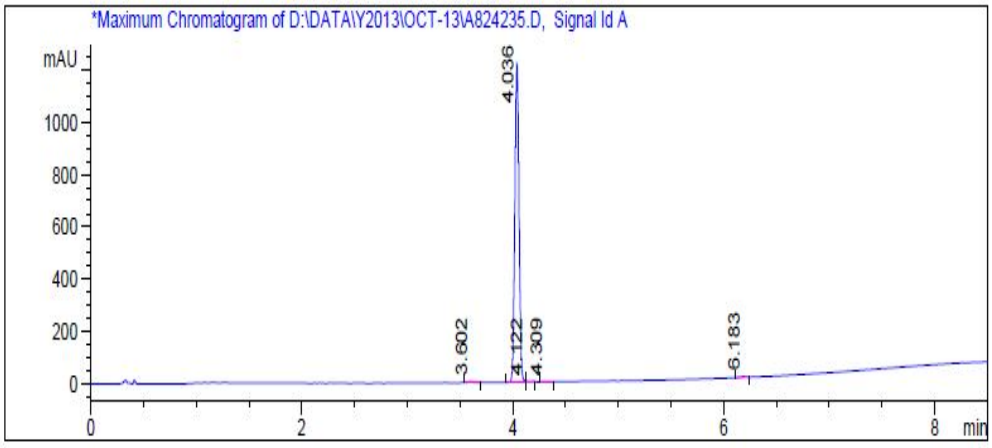
^{13}C NMR (100 MHz) in DMSO-d_6 + CF_3COOD (**2f**)

Method info : A: 0.1% TFA IN H2O , B:0.1% TFA IN ACN ; Flow Rate:2.0 ml/min
COLUMN:XBridge C8 (50x4.6mm, 3.5µ), +ve mode

TIME	%B
0	05
8.0	100
8.1	100
8.5	05
10.0	05

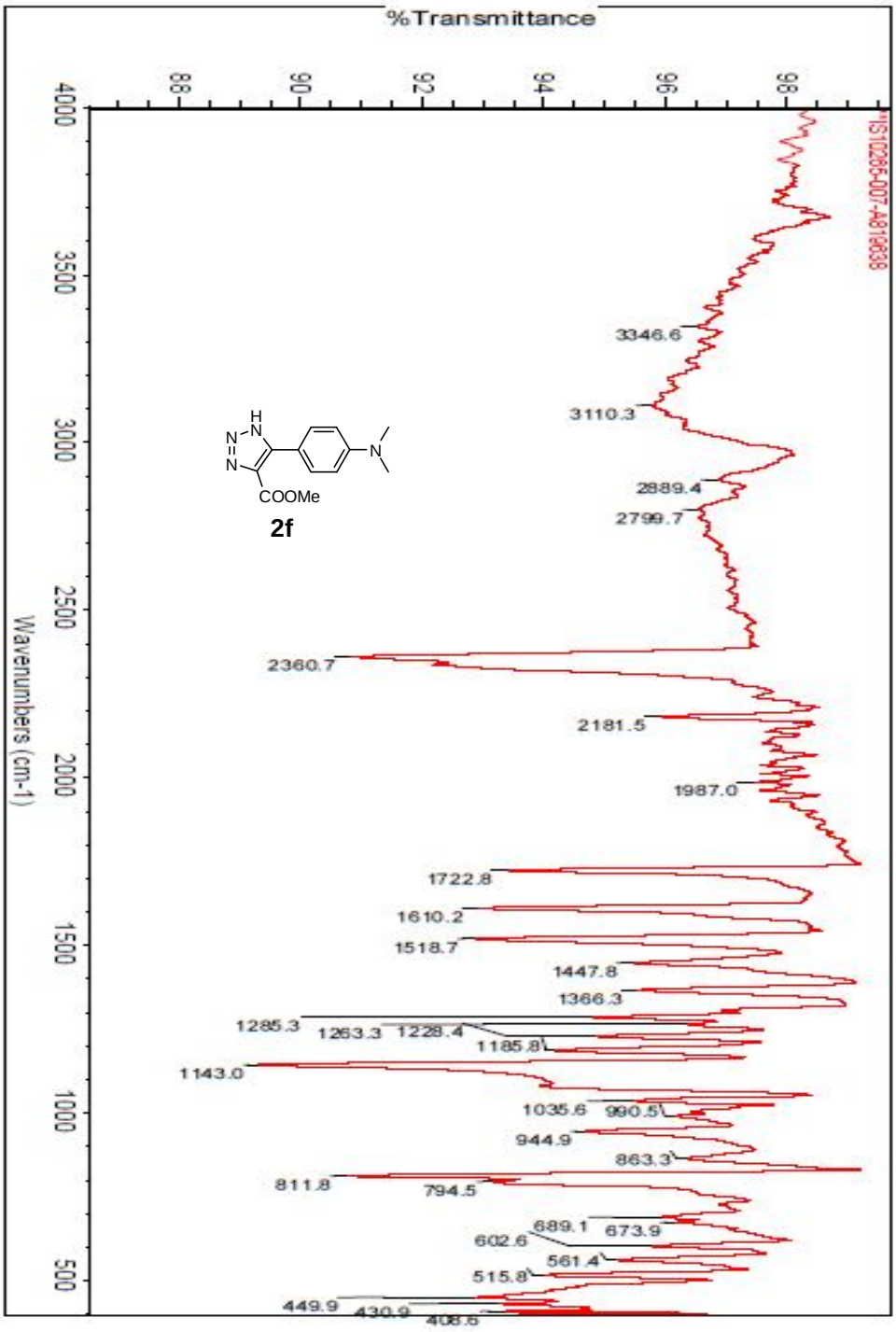


2f

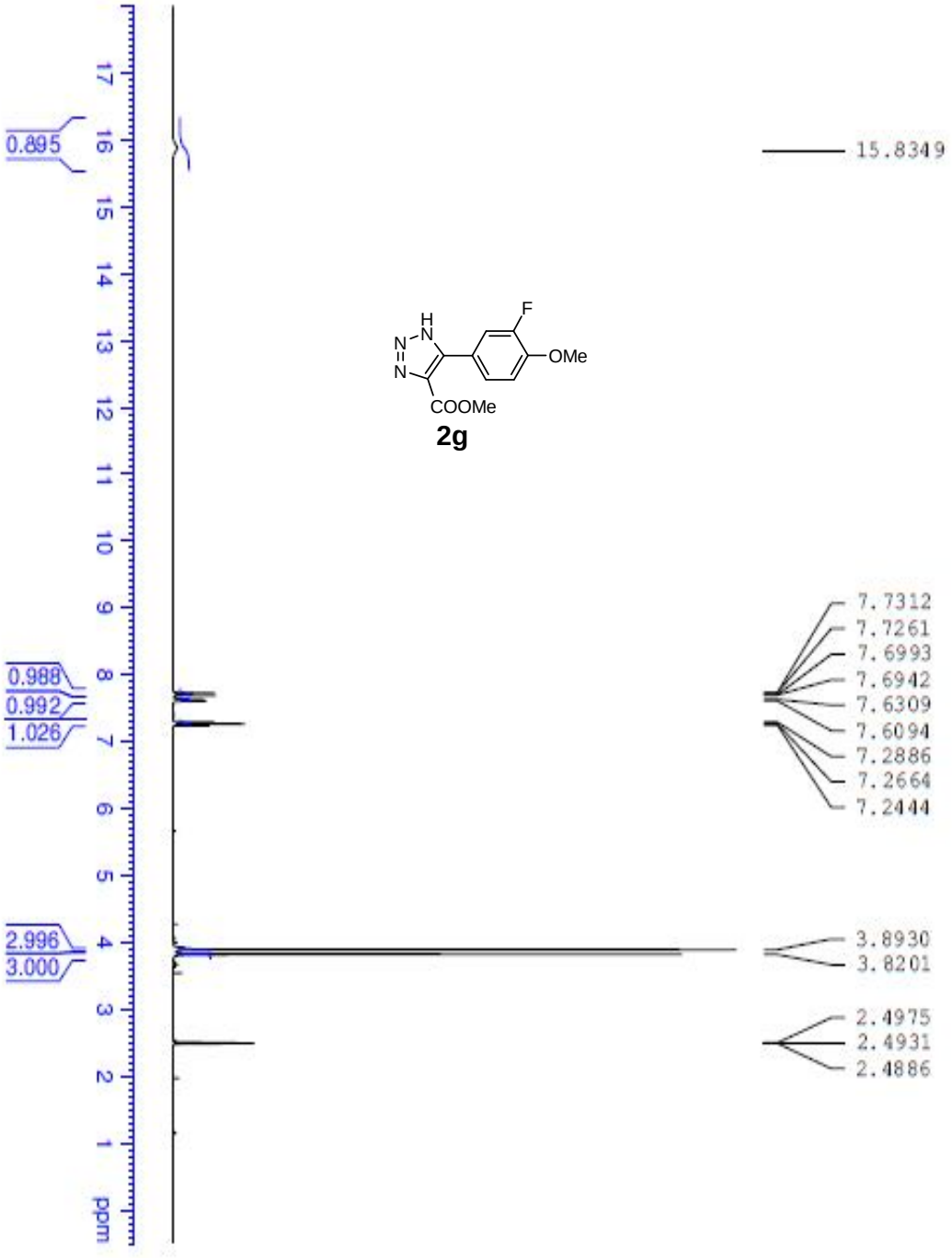


Peak No	RT min	Area	Area %
1	3.602	1.739e+001	0.511
2	4.036	3.359e+003	98.680
3	4.122	4.808e+000	0.141
4	4.309	9.103e+000	0.267
5	6.183	1.361e+001	0.400

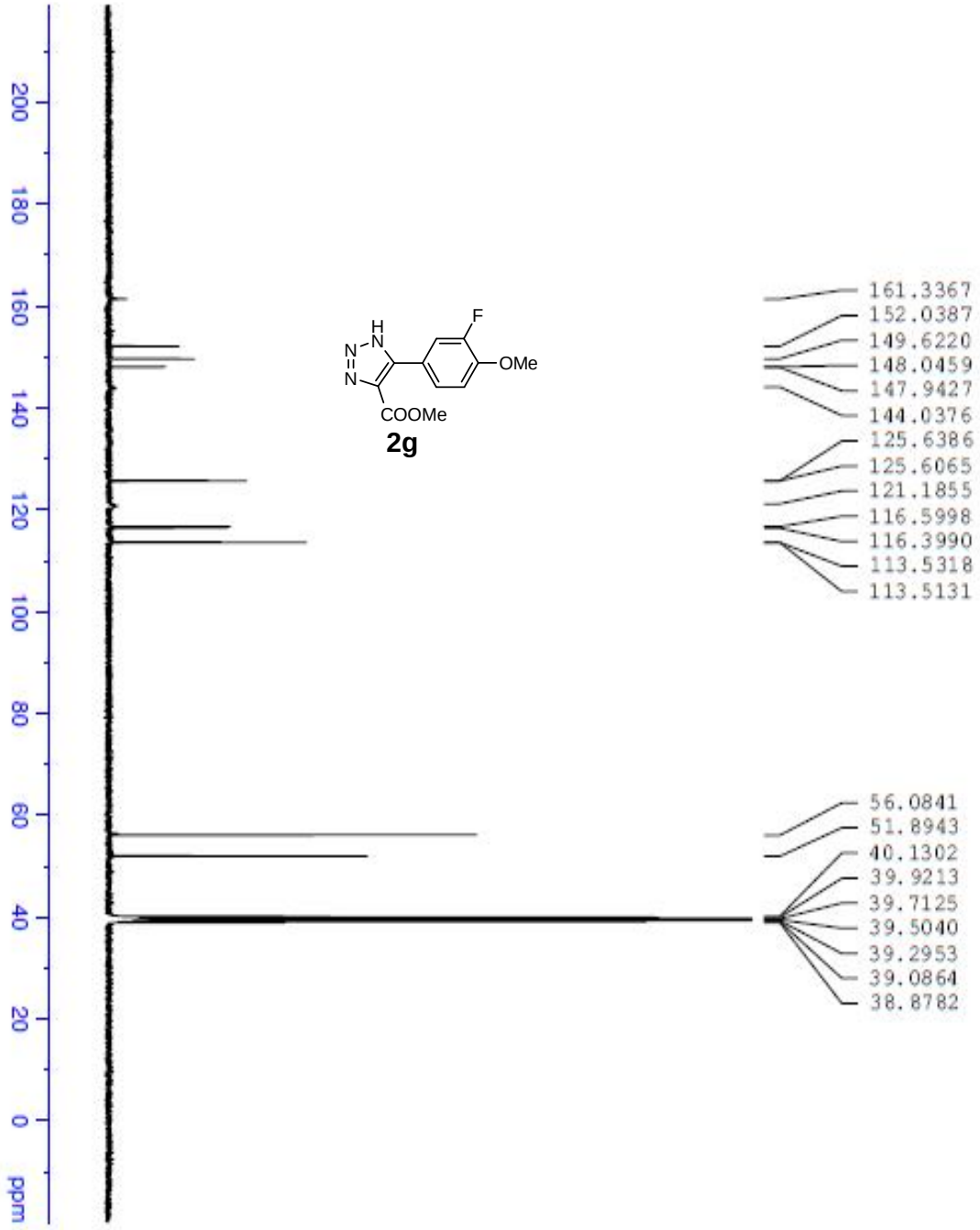
HPLC (**2f**)


%Transmittance
Wavenumbers (cm-1)
4000
3500
3000
2500
2000
1500
1000
500
88
90
92
94
96
98
3346.6
3110.3
2889.4
2799.7
2360.7
2181.5
1987.0
1722.8
1610.2
1518.7
1447.8
1366.3
1285.3
1263.3
1228.4
1185.8
1143.0
1035.6
990.5
944.9
863.3
811.8
794.5
689.1
673.9
602.6
561.4
515.8
449.9
430.9
408.6
COOMe
2f

IR (**2f**)



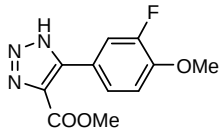
^{1}H NMR (400 MHz) in DMSO-d_6 (**2g**)



^{13}C NMR (100 MHz) in DMSO-d_6 (**2g**)

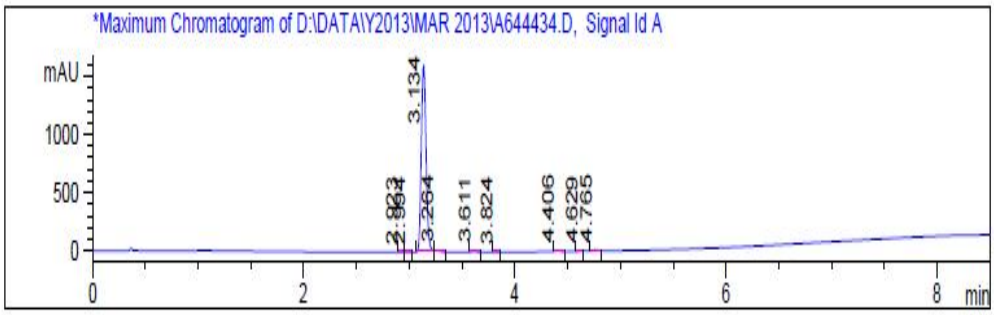
Method info : A: 0.1% TFA IN H2O , B:0.1% TFA IN ACN ; Flow Rate:2.0 ml/min
COLUMN:XBridge C8 (50x4.6mm, 3.5µm), +ve mode

TIME	%B
0	05
8.0	100
8.1	100
8.5	05
10	05



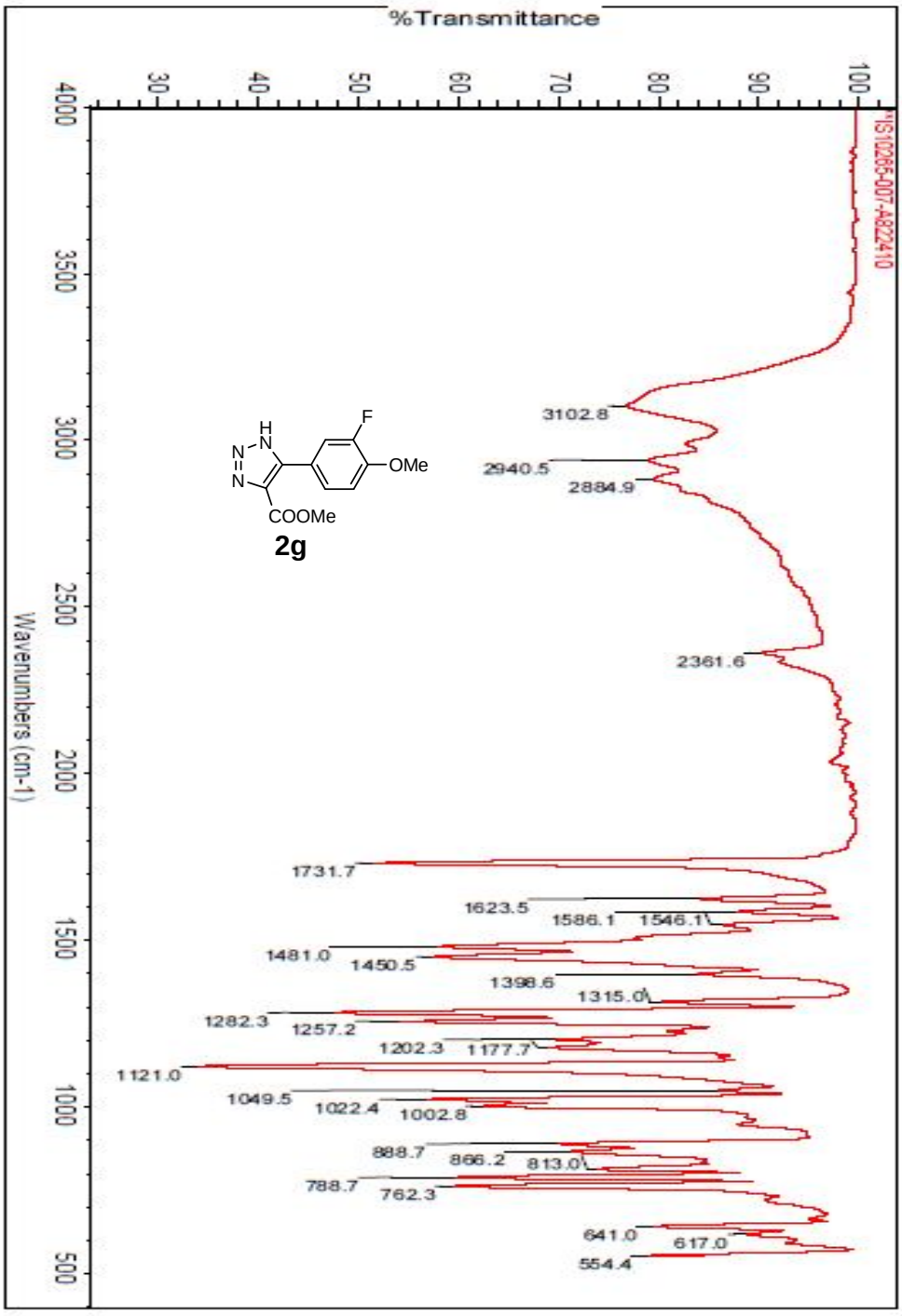
2g

->

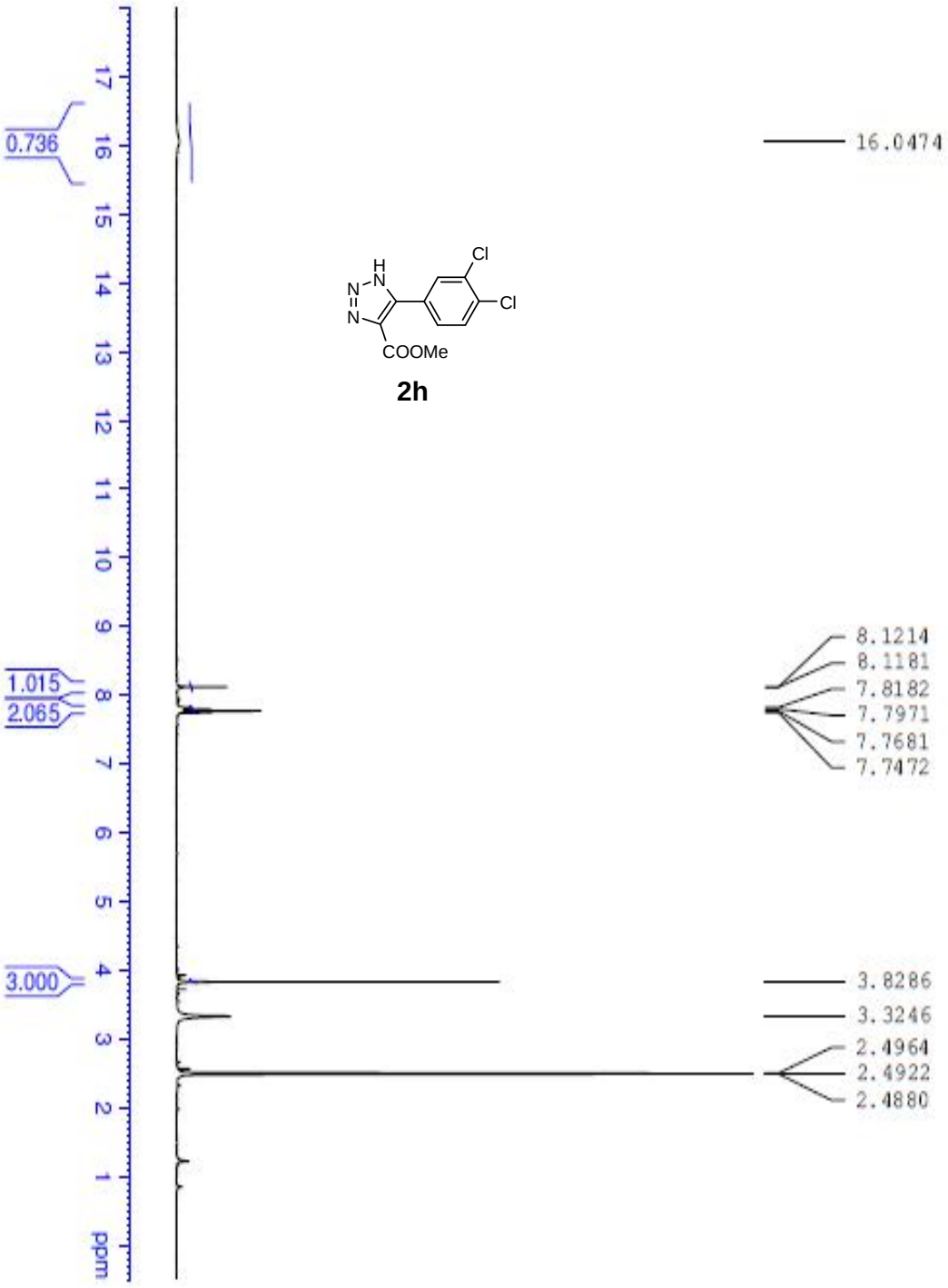


Peak No	RT min	Area	Area %
1	2.923	5.059e+000	0.096
2	2.994	3.337e+000	0.064
3	3.134	5.083e+003	96.763
4	3.264	5.741e+001	1.093
5	3.611	2.572e+001	0.490
6	3.824	3.314e+000	0.063
7	4.406	3.671e+001	0.699
8	4.629	4.127e+000	0.079
9	4.765	3.433e+001	0.654

HPLC (**2g**)

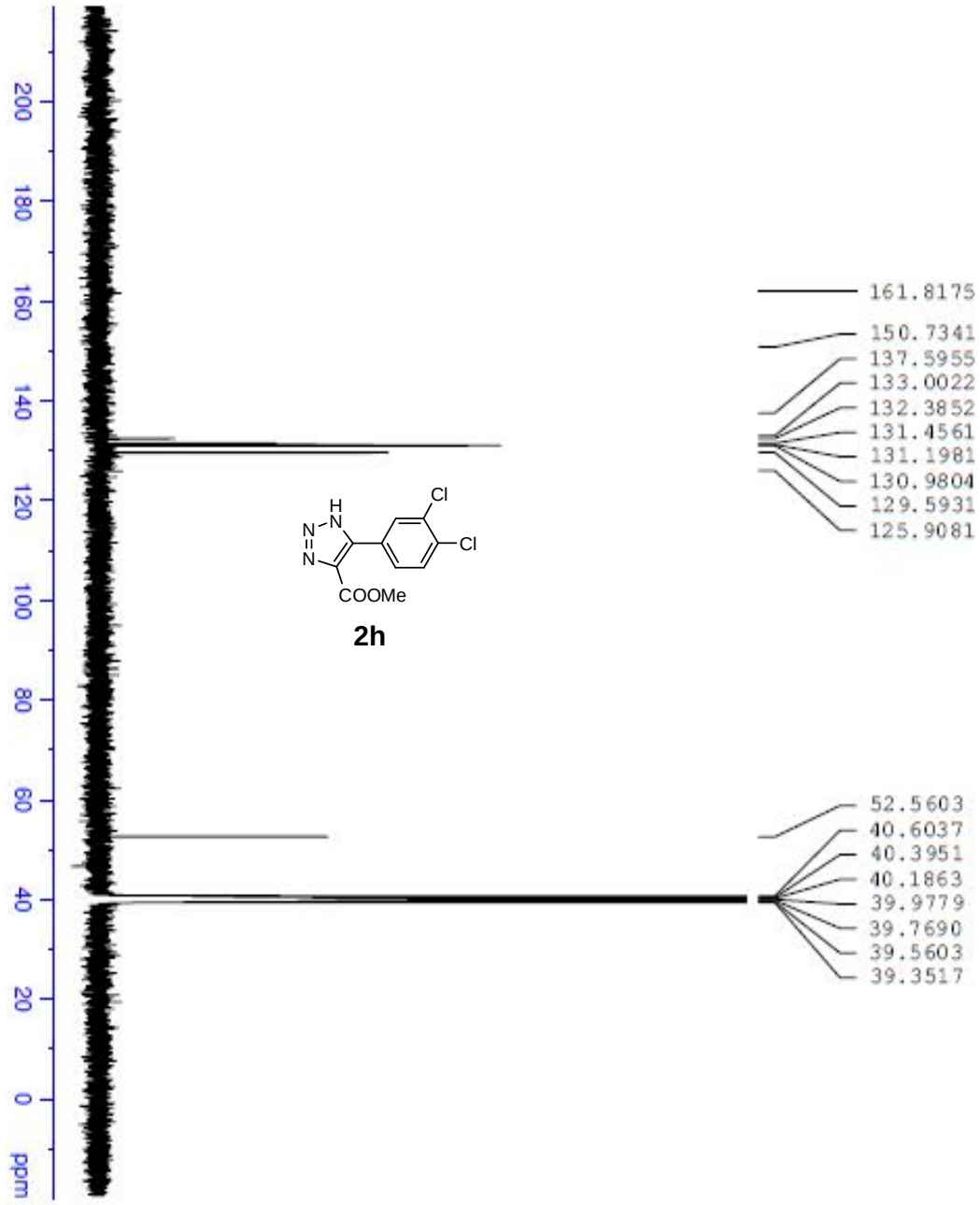

%Transmittance
Wavenumbers (cm-1)
4000
3500
3000
2500
2000
1500
1000
500
30
40
50
60
70
80
90
100
3102.8
2940.5
2884.9
2361.6
1731.7
1623.5
1586.1
1546.1
1481.0
1450.5
1398.6
1315.0
1282.3
1257.2
1202.3
1177.7
1121.0
1049.5
1022.4
1002.8
888.7
866.2
813.0
788.7
762.3
641.0
617.0
554.4
F
OMe
COOMe
2g

IR (**2g**)



2h

^{1}H NMR (400 MHz) in DMSO-d_6 (**2h**)



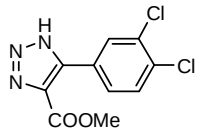
2h

^{13}C NMR (100 MHz) in DMSO-d_6 (**2h**)

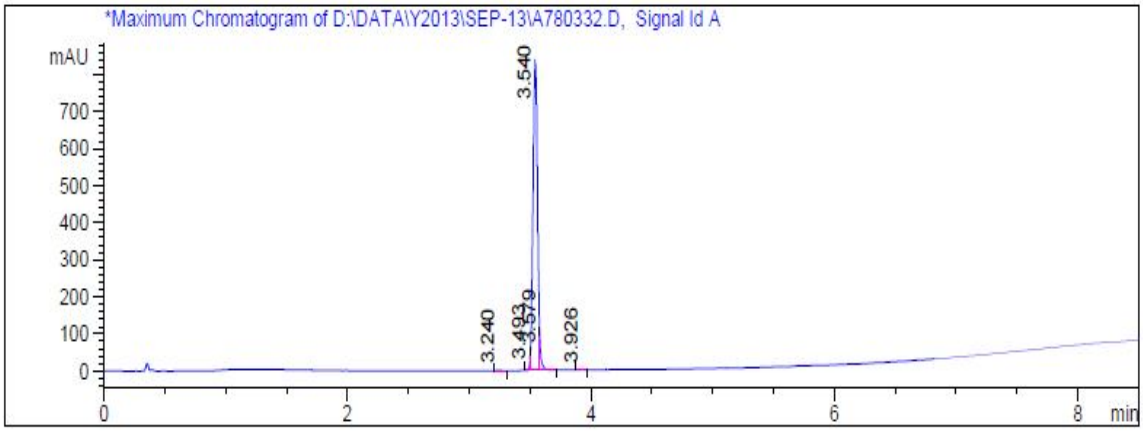
Method info : A: 0.1% TFA IN H2O , B:0.1% TFA IN ACN ; Flow Rate:2.0 ml/min

COLUMN:XBridge C8 (50x4.6mm, 3.5µ), +ve mode

TIME	%B
0	05
8.0	100
8.1	100
8.5	05
10.0	05

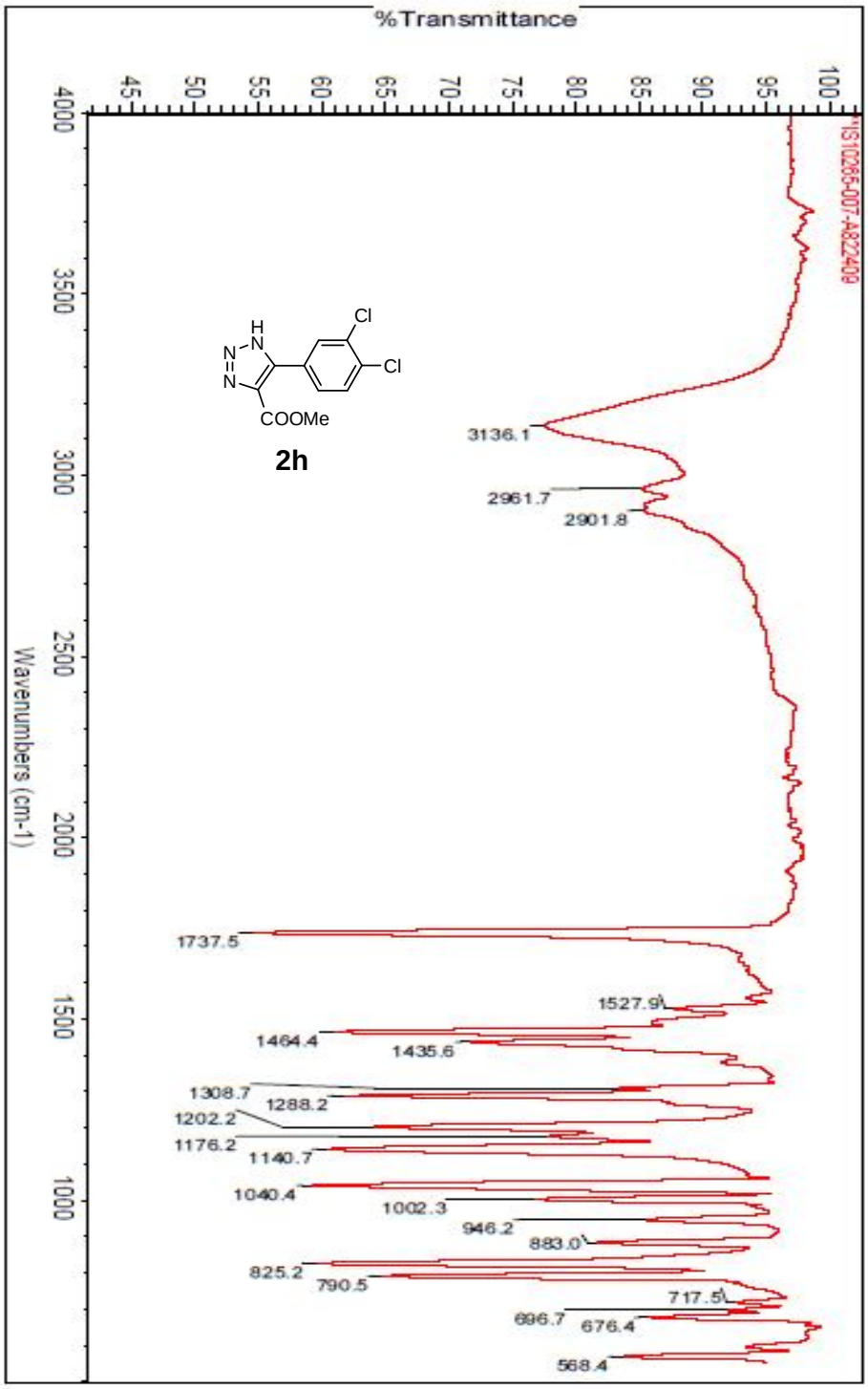


2h

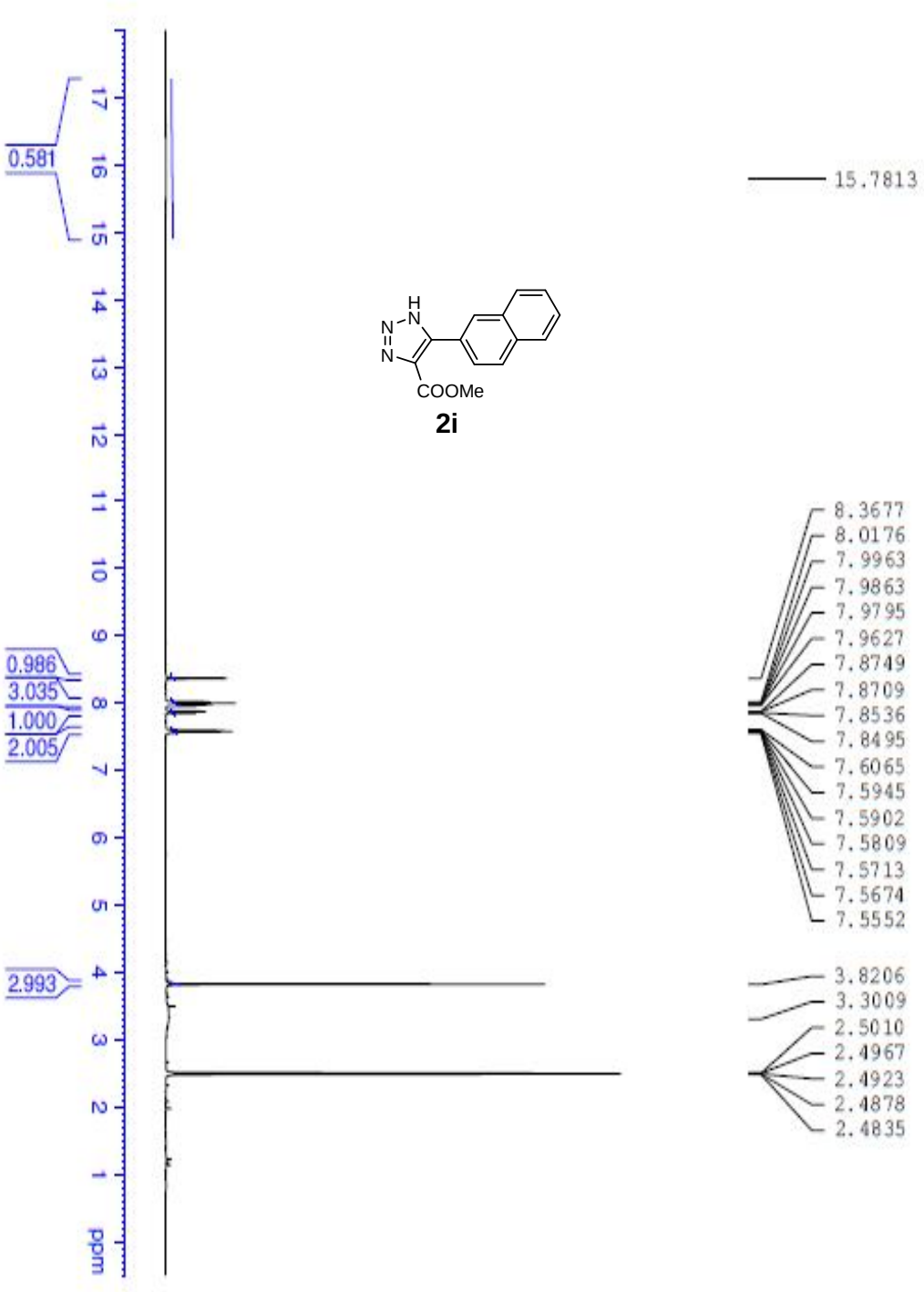


Peak No	RT min	Area	Area %
1	3.240	3.498e+000	0.157
2	3.493	1.341e+001	0.603
3	3.540	2.147e+003	96.483
4	3.579	5.926e+001	2.663
5	3.926	2.092e+000	0.094

HPLC (**2h**)

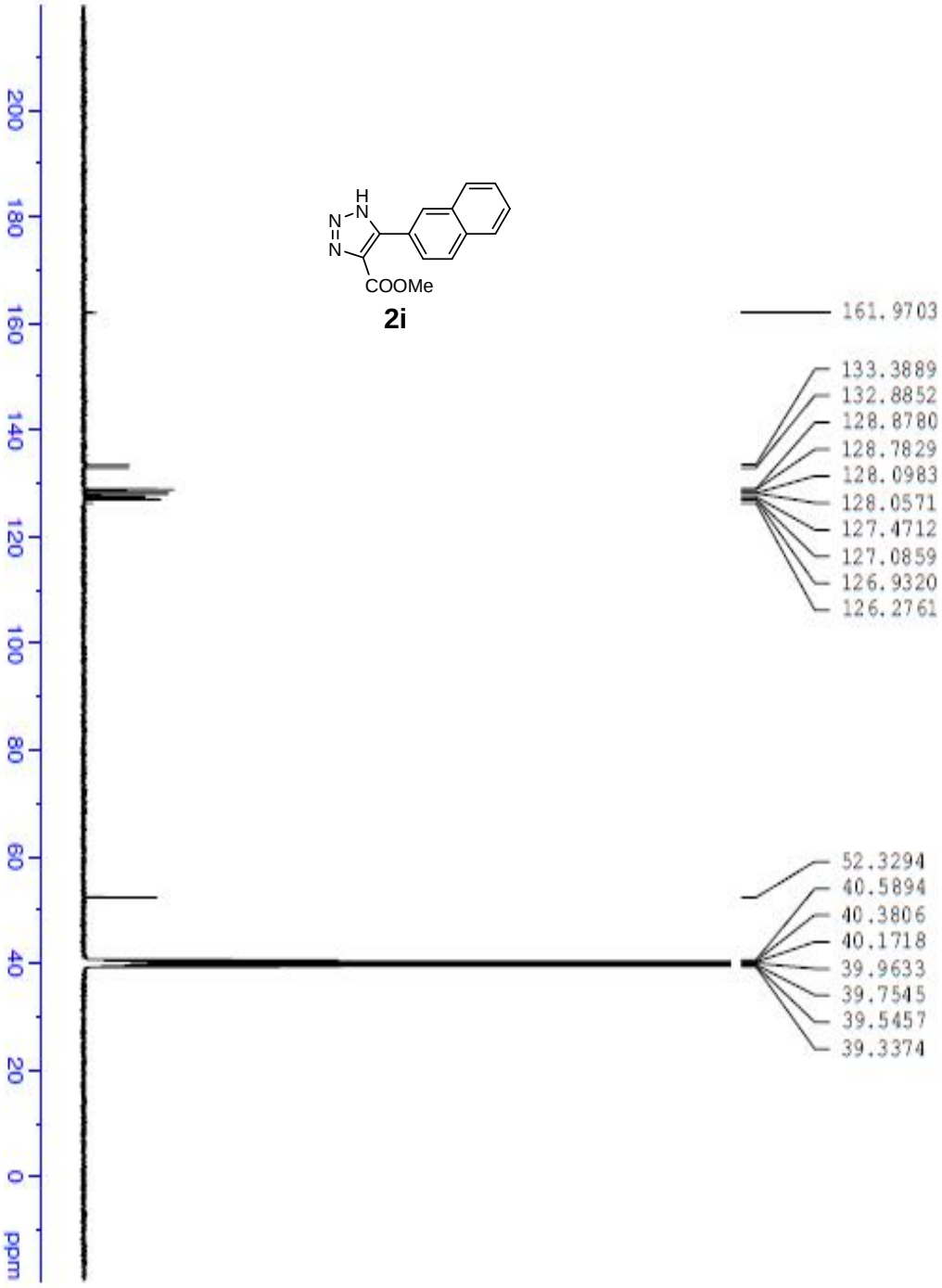

%Transmittance
Wavenumbers (cm-1)
4000
3500
3000
2500
2000
1500
1000
45
50
55
60
65
70
75
80
85
90
95
100
IIS10266-007-A822409
Cl
Cl
H
N
N
N
COOMe
2h
3136.1
2961.7
2901.8
1737.5
1527.9
1464.4
1435.6
1308.7
1288.2
1202.2
1176.2
1140.7
1040.4
1002.3
946.2
883.0
825.2
790.5
717.5
696.7
676.4
568.4

IR (**2h**)



2i

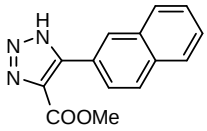
[1]H NMR (400 MHz) in DMSO-d_6 (**2i**)



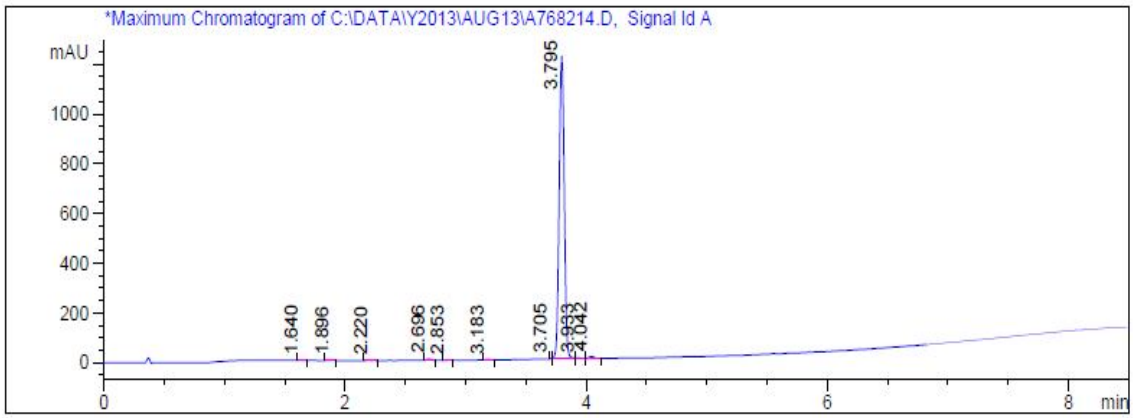
^{13}C NMR (100 MHz) in DMSO-d_6 (**2i**)

Method info : A:0.1%TFA in H2O, B:0.1%TFA in ACN, Flow Rate:2.0ml/min
COLUMN: XBridge C8(50X4.6)mm,3.5µm

Time	% of B
0	5
8.0	100
8.1	100
8.5	5
10.0	5

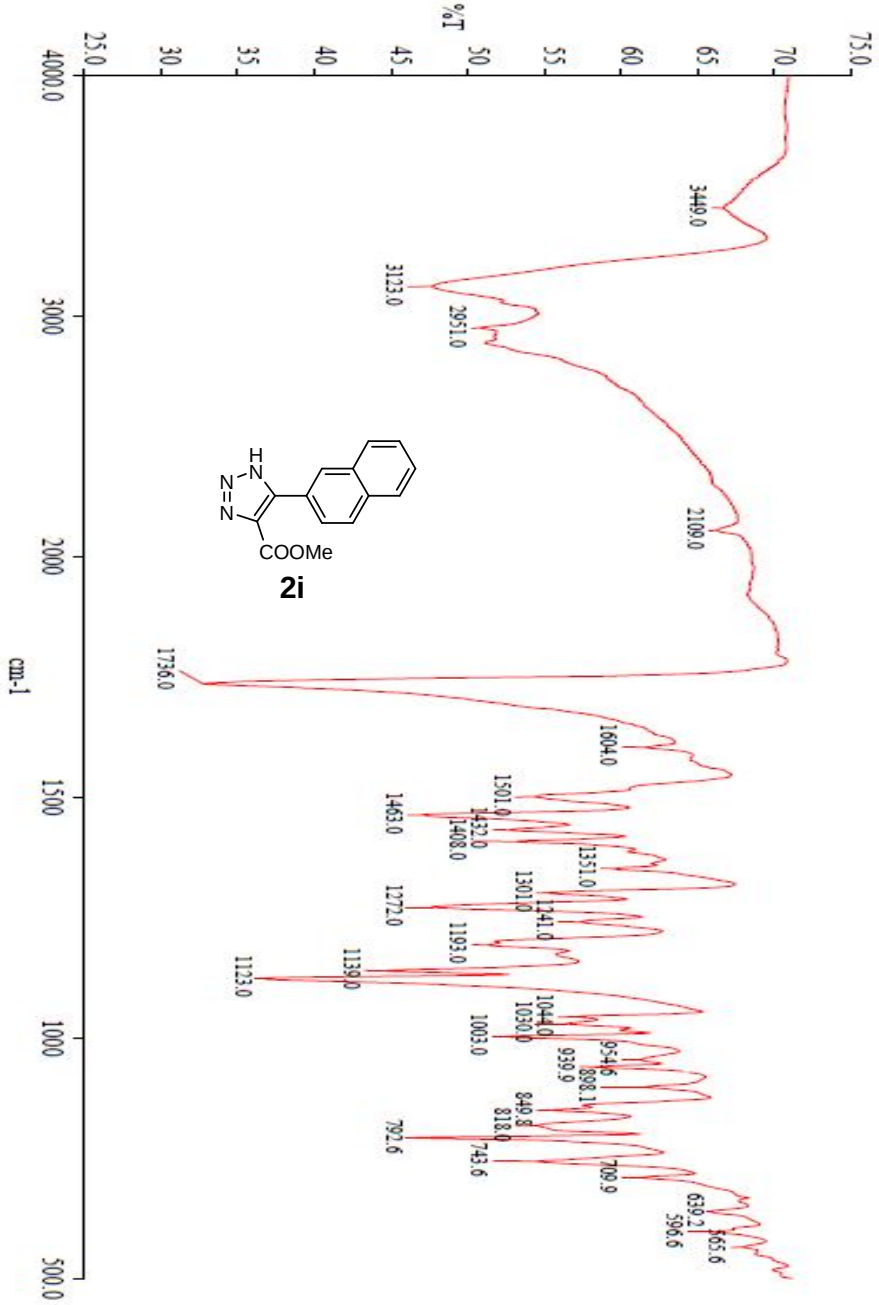


2i

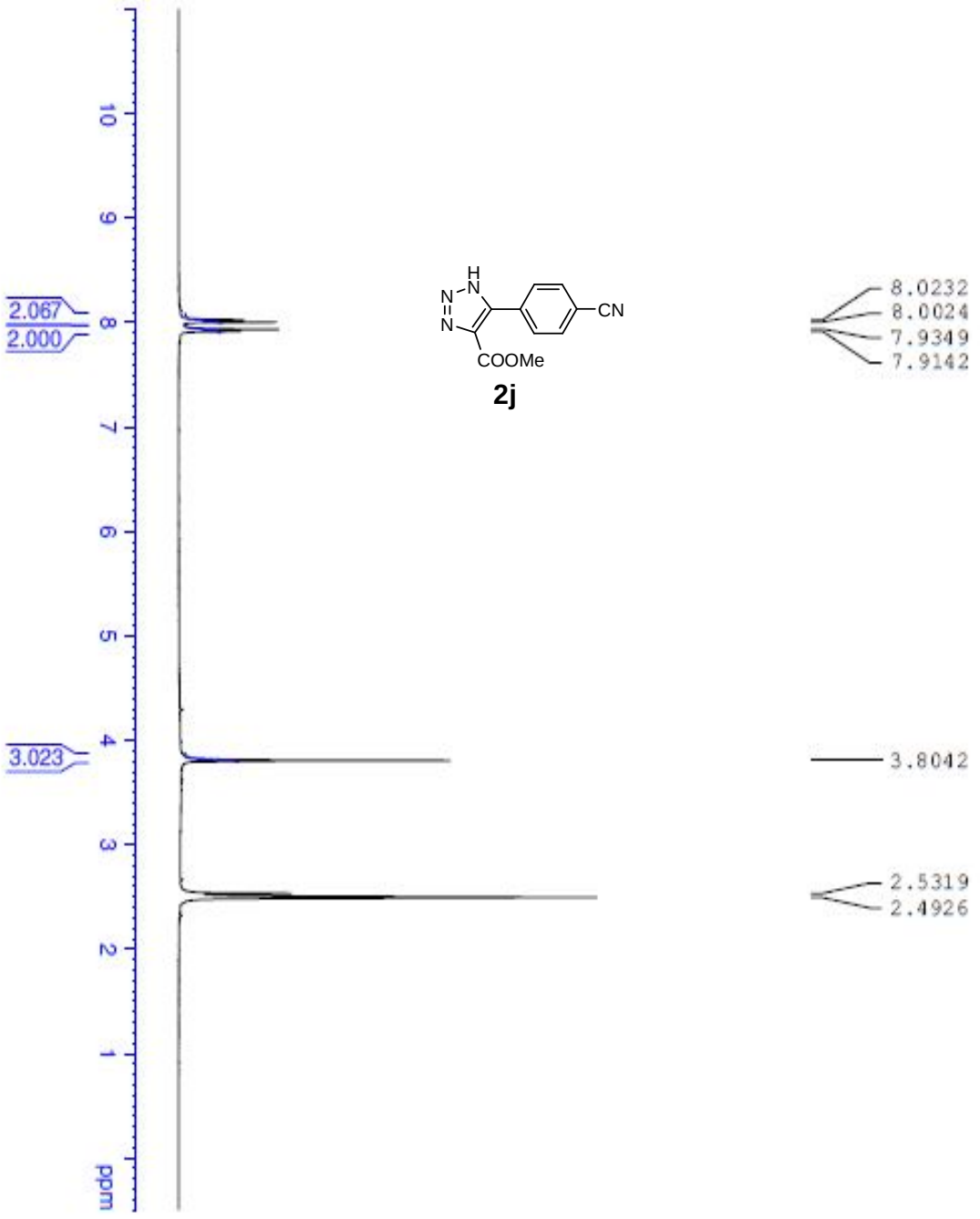


Peak No	RT min	Area	Area %
1	1.640	3.772e+000	0.10
2	1.896	1.726e+000	0.05
3	2.220	3.651e+000	0.10
4	2.696	1.596e+001	0.44
5	2.853	6.188e+000	0.17
6	3.183	6.152e+000	0.17
7	3.705	3.436e+000	0.09
8	3.795	3.554e+003	97.82
9	3.933	9.567e+000	0.26
10	4.042	2.888e+001	0.79

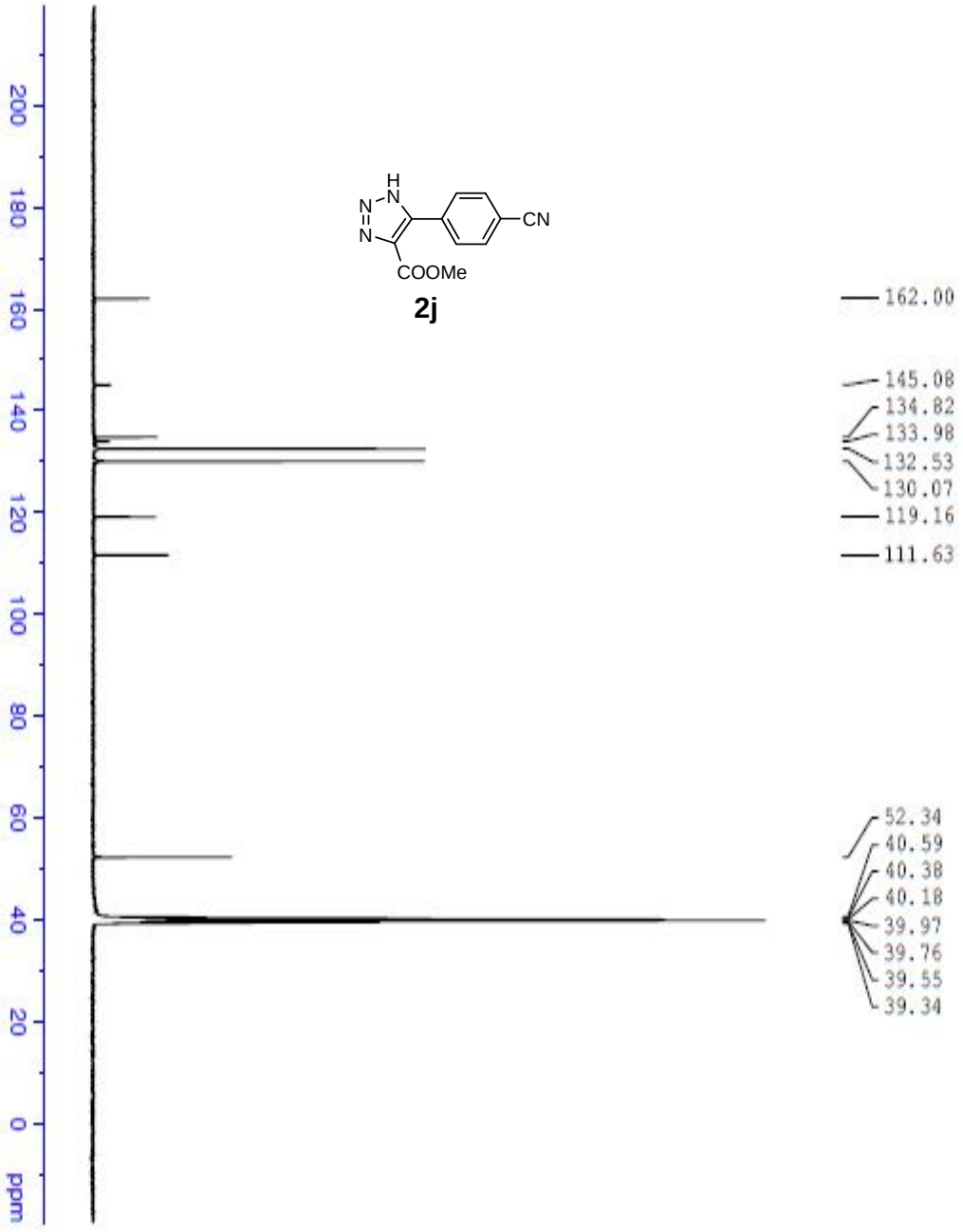
HPLC (**2i**)


%T
cm-1
3449.0
3123.0
2951.0
2109.0
1736.0
1604.0
1501.0
1463.0
1432.0
1408.0
1351.0
1301.0
1272.0
1241.0
1193.0
1139.0
1123.0
1044.0
1030.0
1003.0
954.6
939.9
898.1
849.8
818.0
792.6
743.6
709.9
639.2
596.6
565.6
COOMe
2i

IR (**2i)**



^{1}H NMR (400 MHz) in DMSO-d_6 (**2j**)



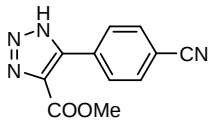
^{13}C NMR (100 MHz) in DMSO-d_6 (**2j**)

Method info :

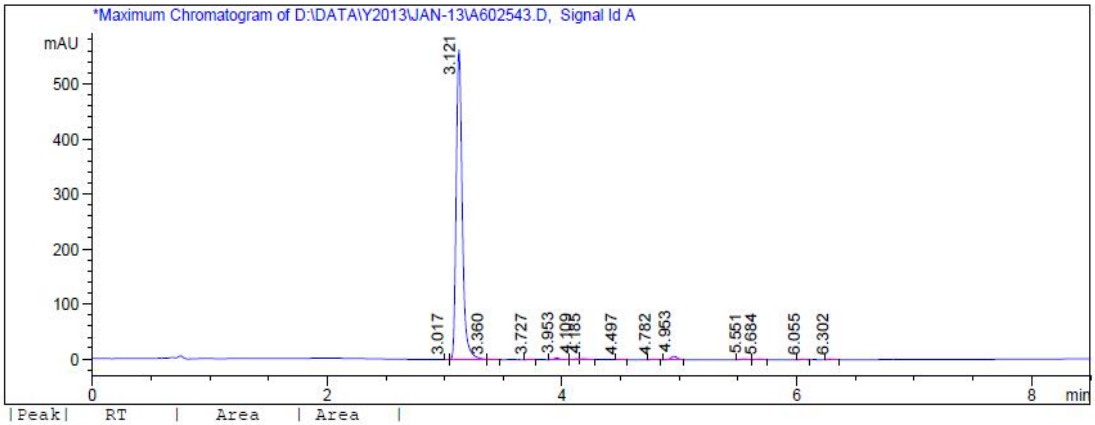
A-10mM NH4HCO3 IN H2O , B- ACN Flow: 1.0 ml/min

COLUMN: XBridge C8 (50X4.6mm, 3.5µm), -ve mode

TIME	%B
0	05
8.0	100
8.1	100
8.5	05
10	05

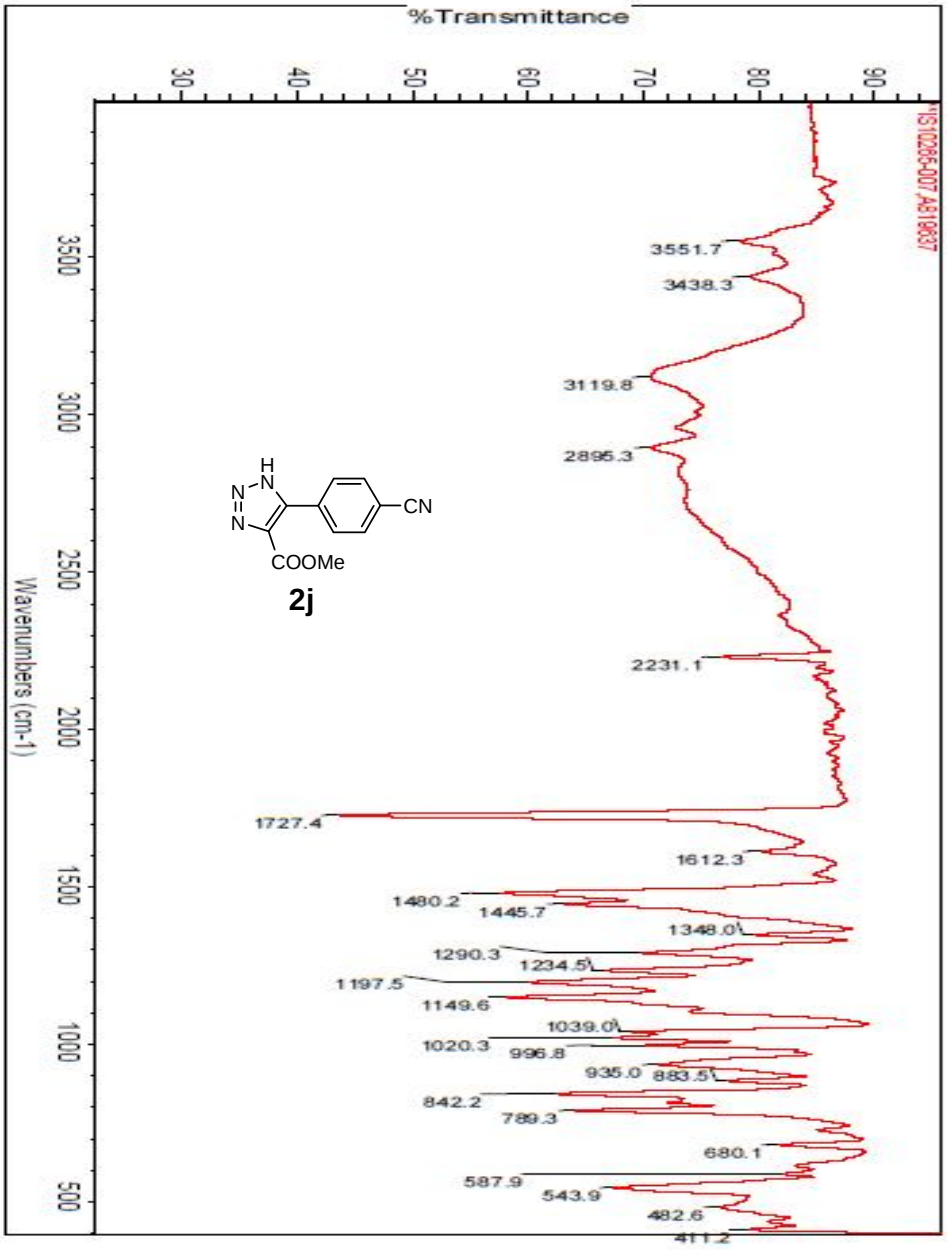


2j

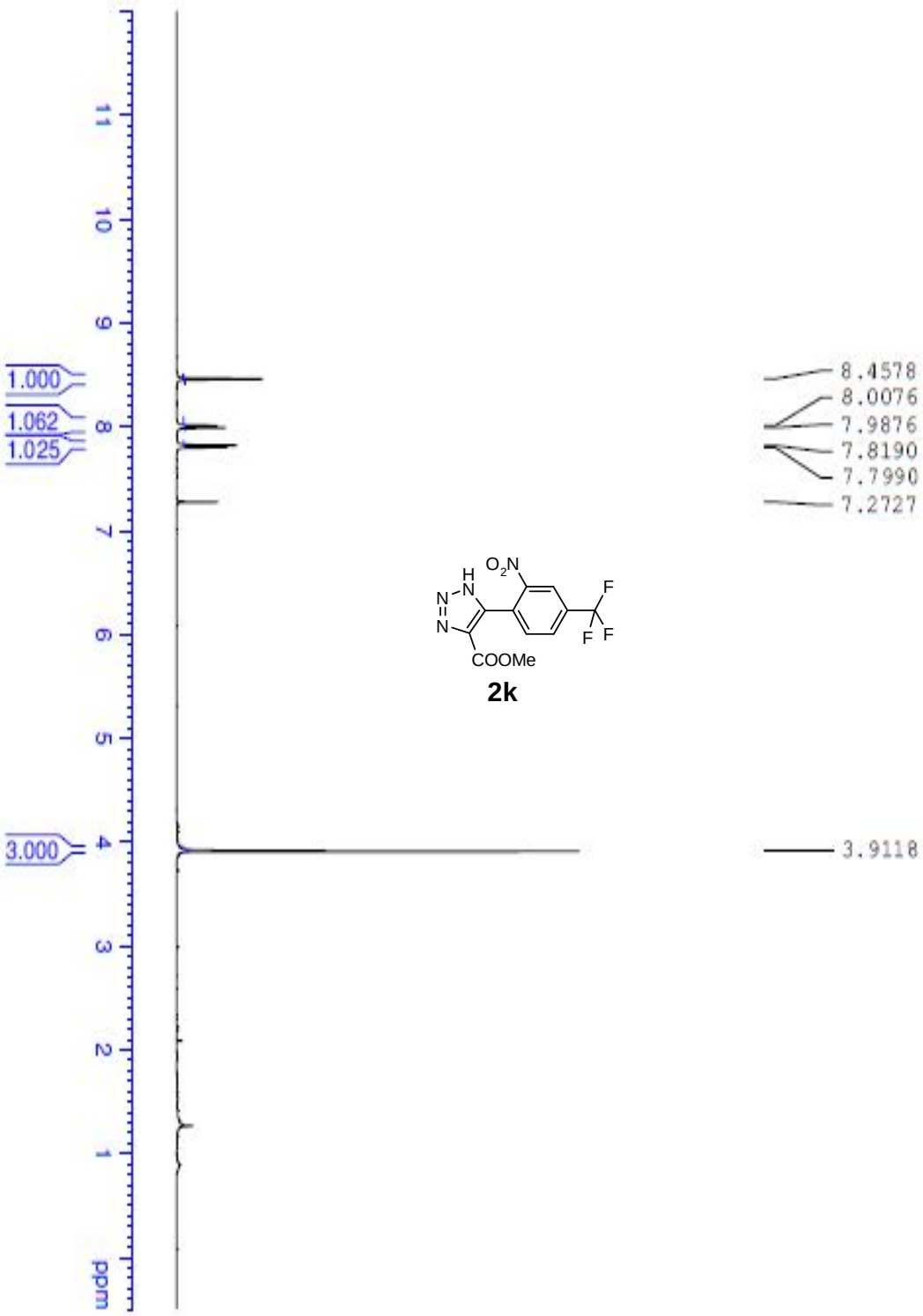


Peak No	RT min	Area	Area %
1	3.017	1.255e+000	0.059
2	3.121	2.022e+003	95.798
3	3.360	4.956e+000	0.235
4	3.727	1.645e+000	0.078
5	3.953	1.118e+001	0.530
6	4.109	5.492e+000	0.260
7	4.185	6.648e+000	0.315
8	4.497	6.456e+000	0.306
9	4.782	4.212e+000	0.200
10	4.953	2.491e+001	1.180
11	5.551	8.819e+000	0.418
12	5.684	7.305e+000	0.346
13	6.055	3.662e+000	0.174
14	6.302	2.153e+000	0.102

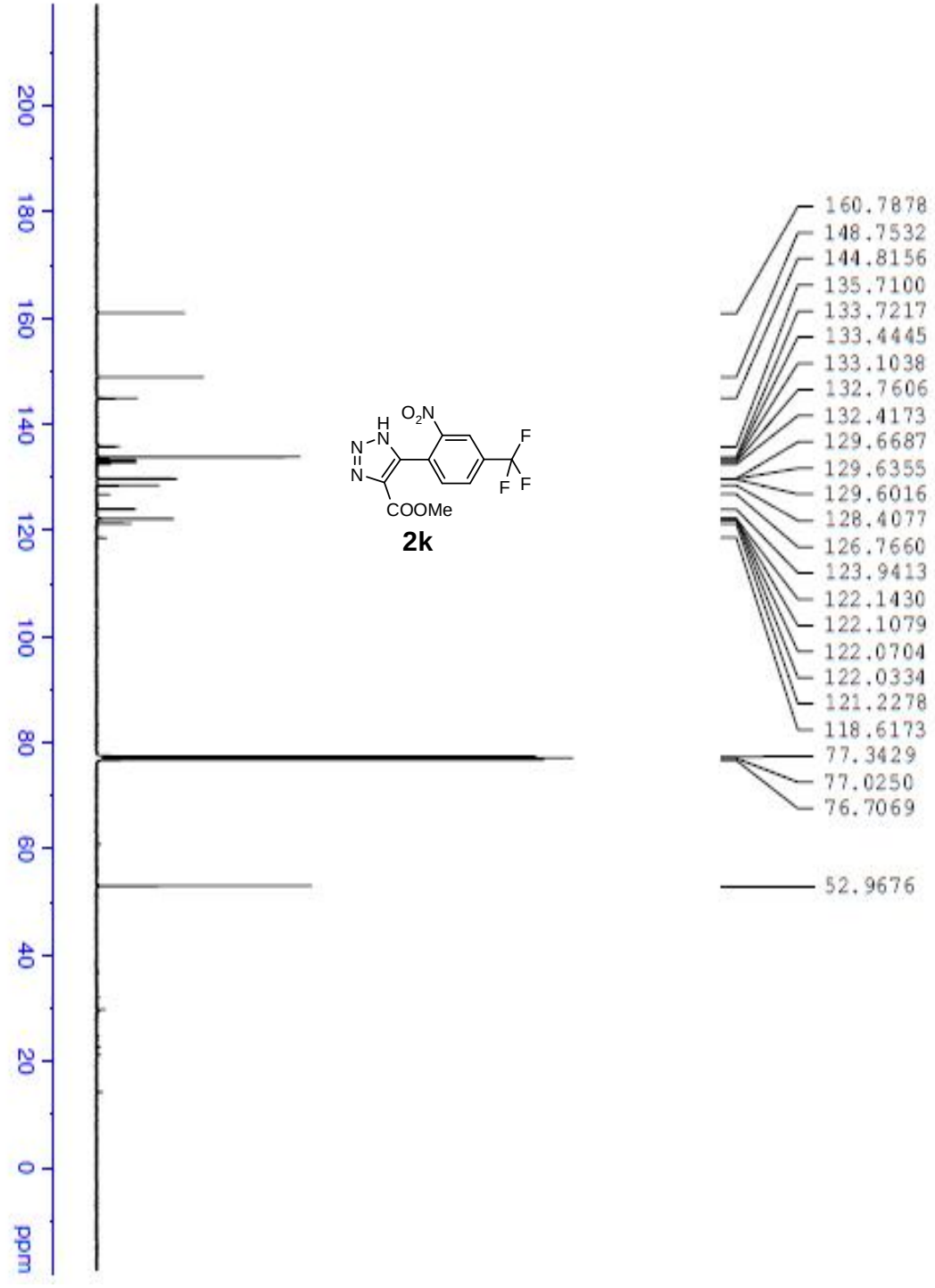
HPLC (**2j**)


%Transmittance
30
40
50
60
70
80
90
Wavenumbers (cm-1)
3500
3000
2500
2000
1500
1000
500
3551.7
3438.3
3119.8
2895.3
2231.1
1727.4
1612.3
1480.2
1445.7
1348.0
1290.3
1234.5
1197.5
1149.6
1039.0
1020.3
996.8
935.0
883.5
842.2
789.3
680.1
587.9
543.9
482.6
411.2
H
N
N
N
CN
COOMe
2j

IR (**2j**)



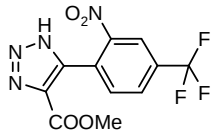
^{1}H NMR (400 MHz) in $CDCl_3$ (**2k**)



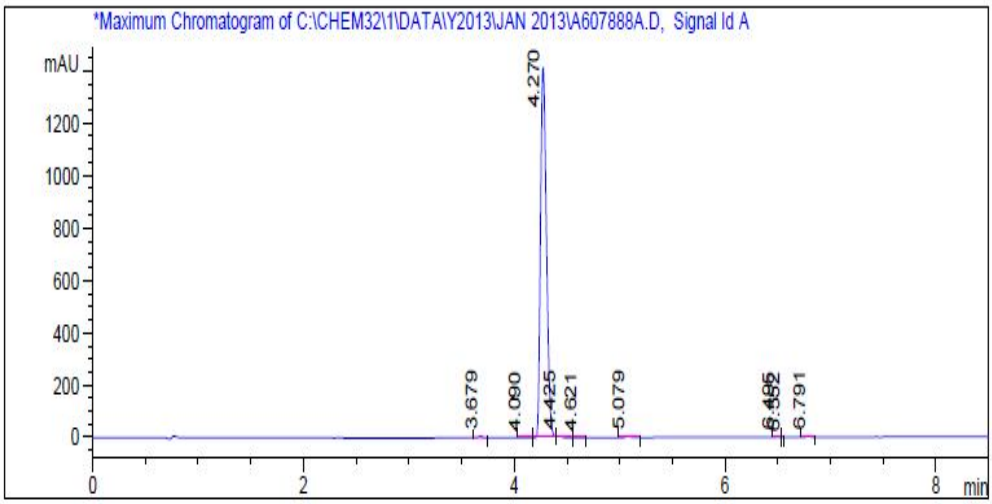
^{13}C NMR (100 MHz) in $CDCl_3$ (**2k**)

Method info : B: 10mM NH4HCO3 , C: ACN ; Flow Rate:1.0 ml/min
COLUMN:XBridge C8 (50x4.6mm, 3.5µ), -ve mode

TIME	%C
0	05
8.0	100
8.1	100
8.5	05
10.0	05

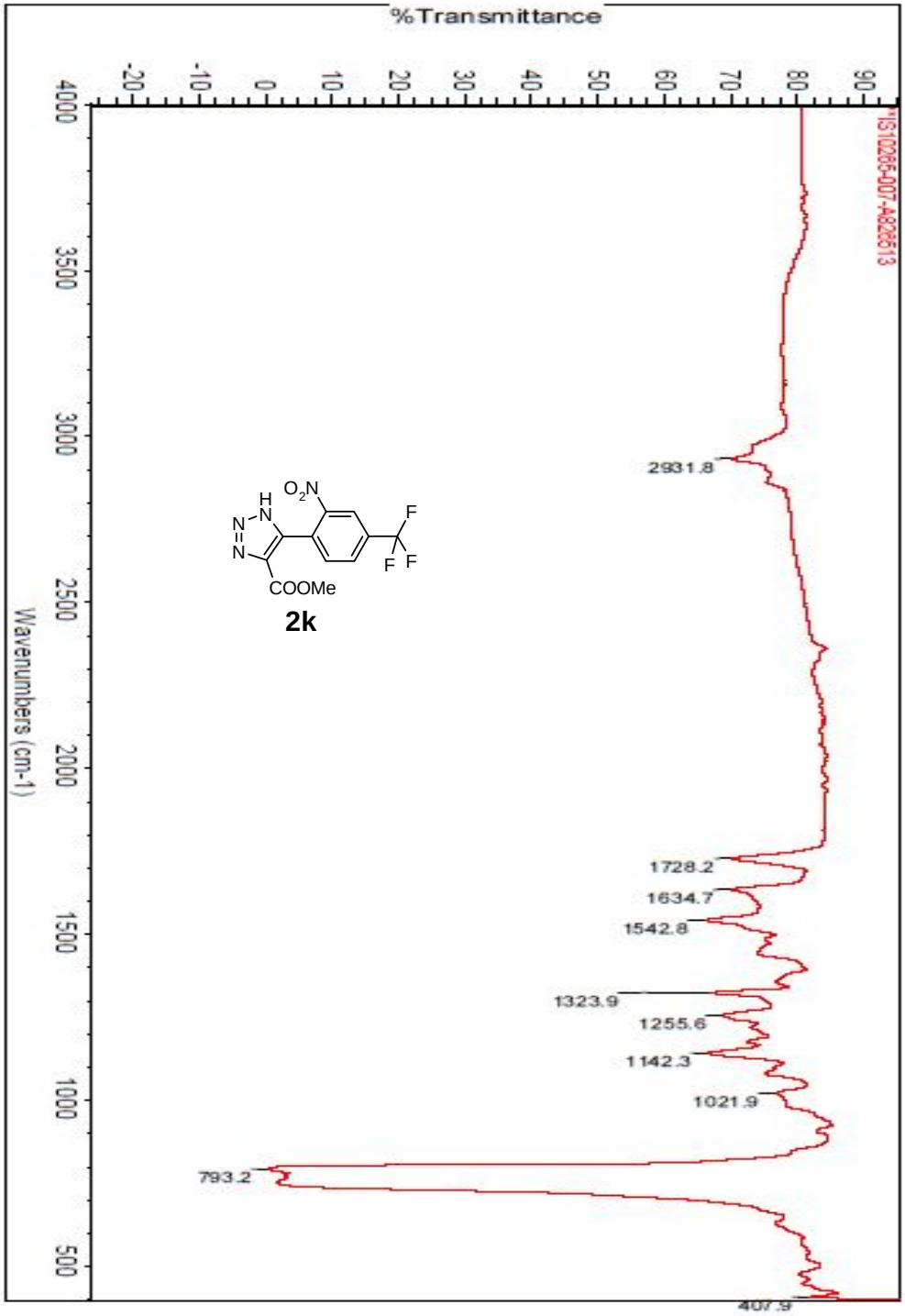


2k

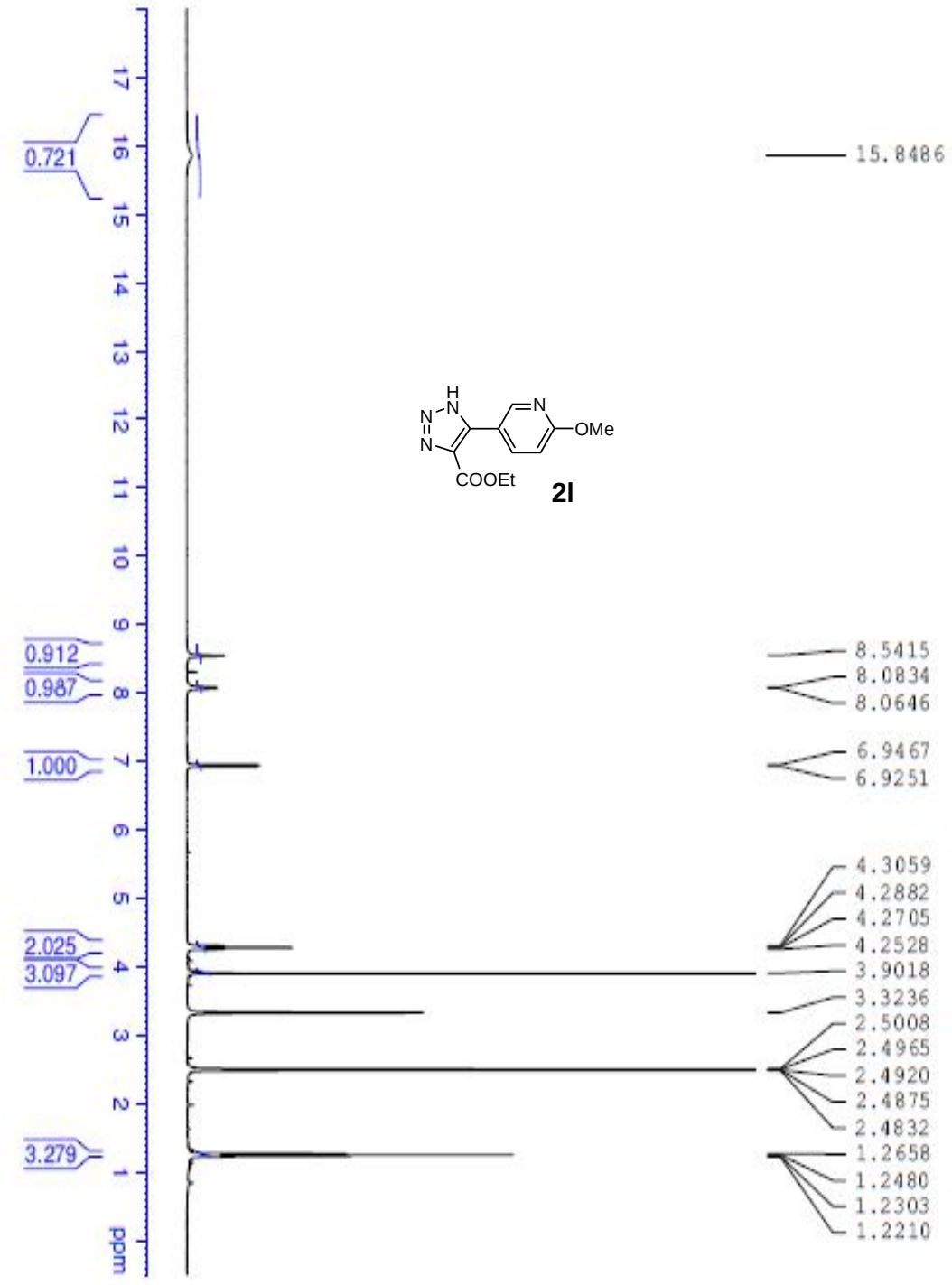


Peak No	RT min	Area	Area %
1	3.679	1.341e+001	0.240
2	4.090	2.727e+000	0.049
3	4.270	5.522e+003	98.723
4	4.425	2.179e+001	0.390
5	4.621	3.663e+000	0.065
6	5.079	1.611e+001	0.288
7	6.495	3.362e+000	0.060
8	6.552	4.135e-001	0.007
9	6.791	9.950e+000	0.178

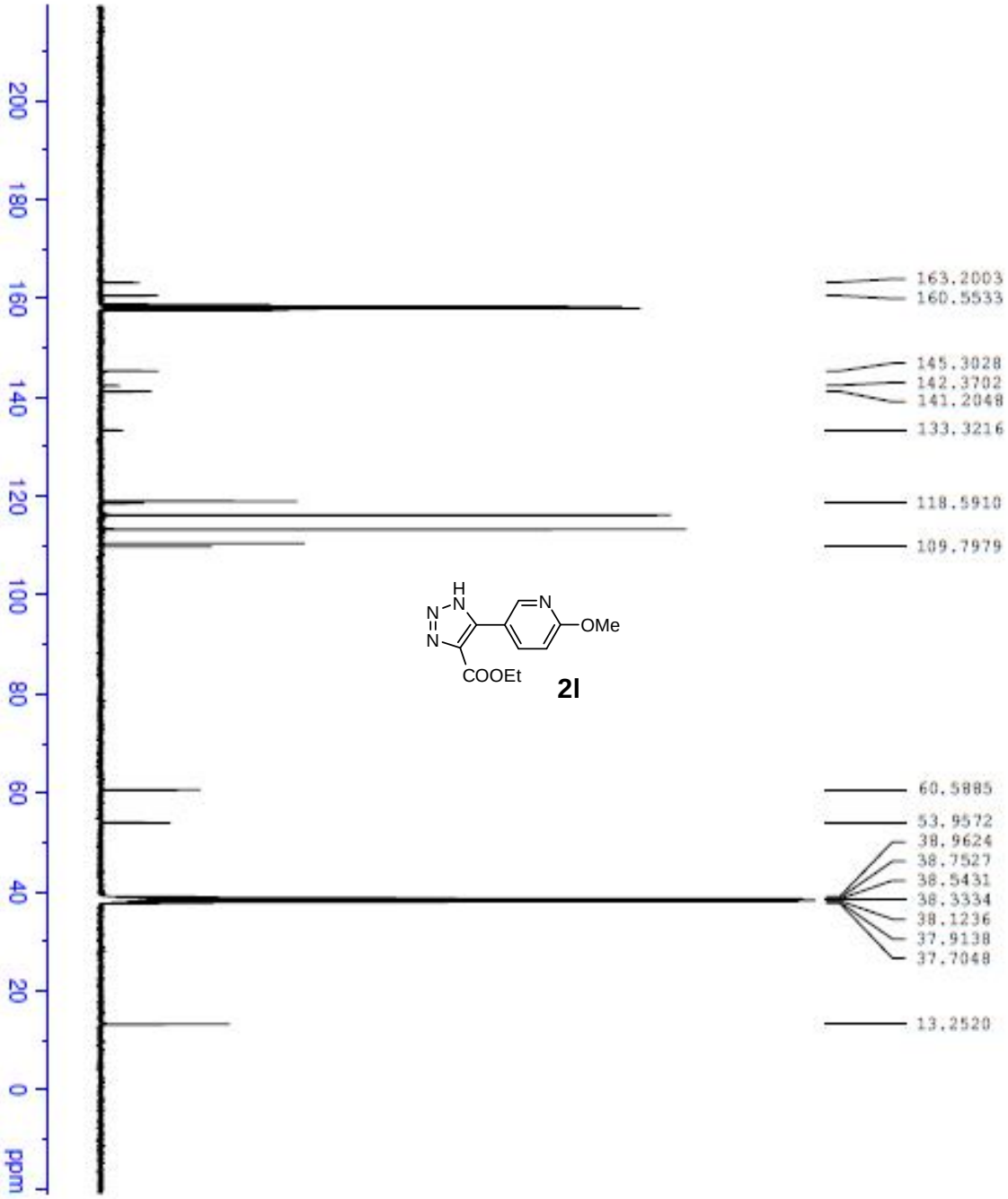
HPLC (**2k**)


%Transmittance
Wavenumbers (cm-1)
2931.8
1728.2
1634.7
1542.8
1323.9
1255.6
1142.3
1021.9
793.2
407.9
COOMe
2k

IR (**2k**)



^{1}H NMR (400 MHz) in DMSO-d_6 (**2l**)



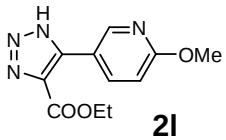
^{13}C NMR (100 MHz) in DMSO-d_6 (**2l**)

Method info :

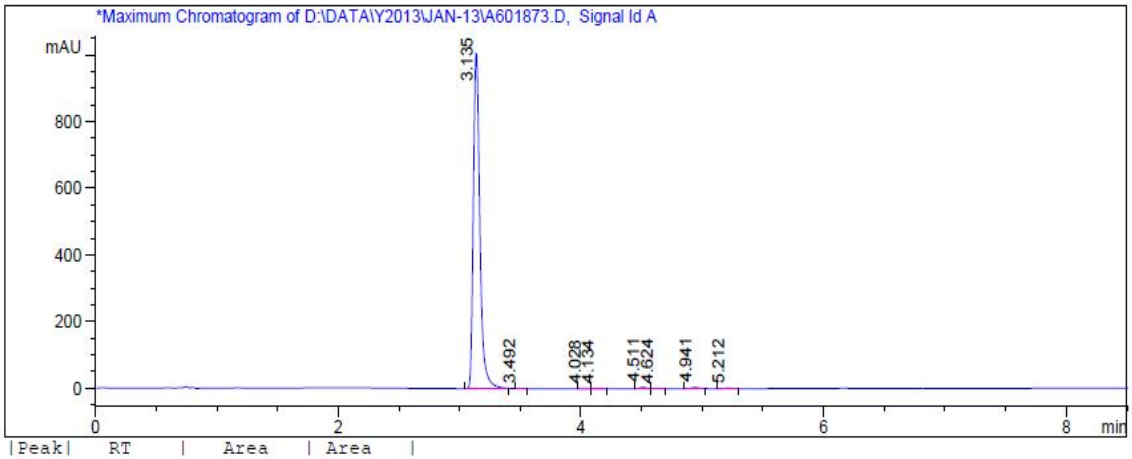
A-10mM NH4HCO3 IN H2O , B- ACN Flow: 1.0 ml/min

COLUMN: XBridge C8 (50X4.6mm, 3.5µm), +ve mode

TIME	%B
0	05
8.0	100
8.1	100
8.5	05
10	05

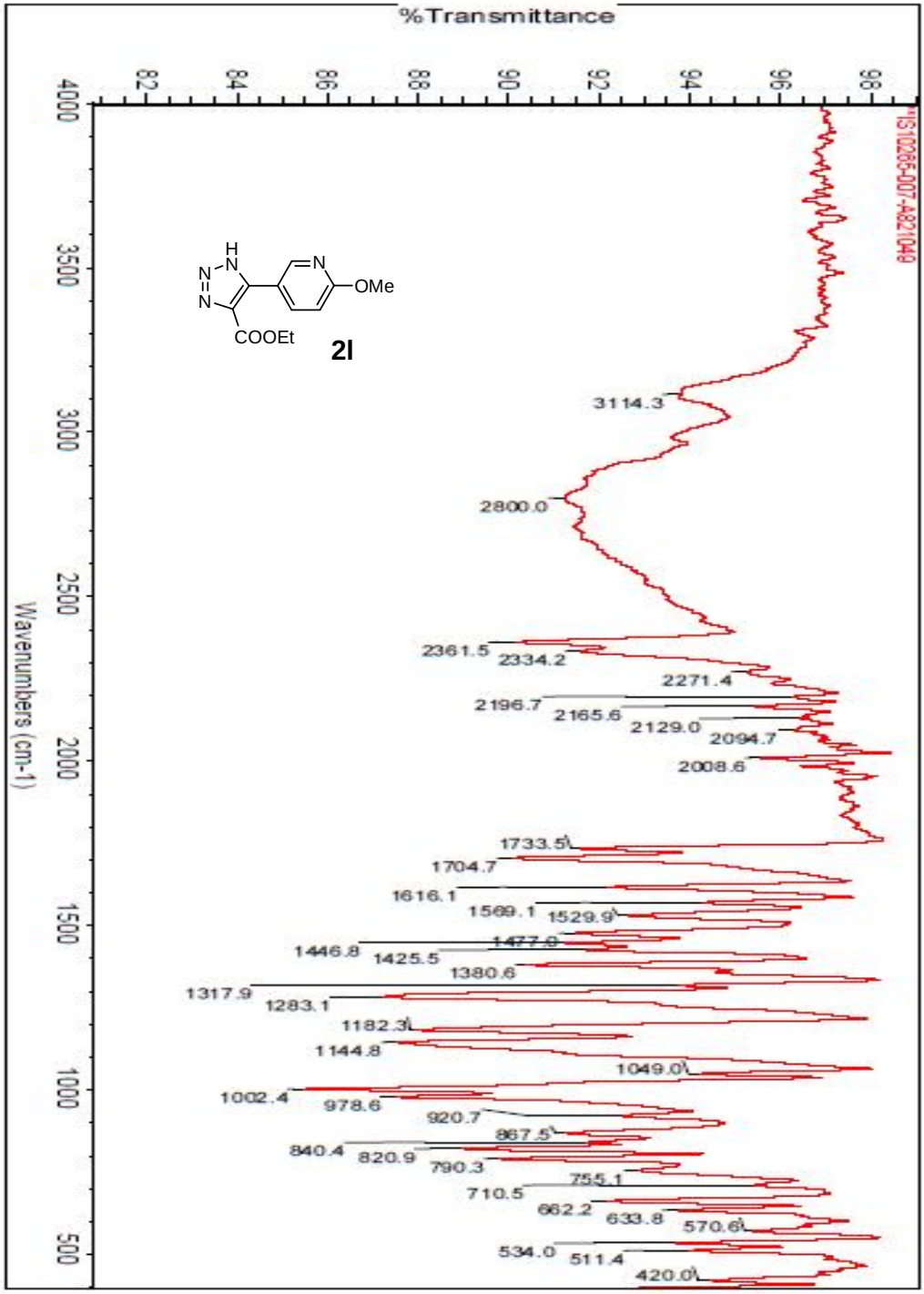


2l

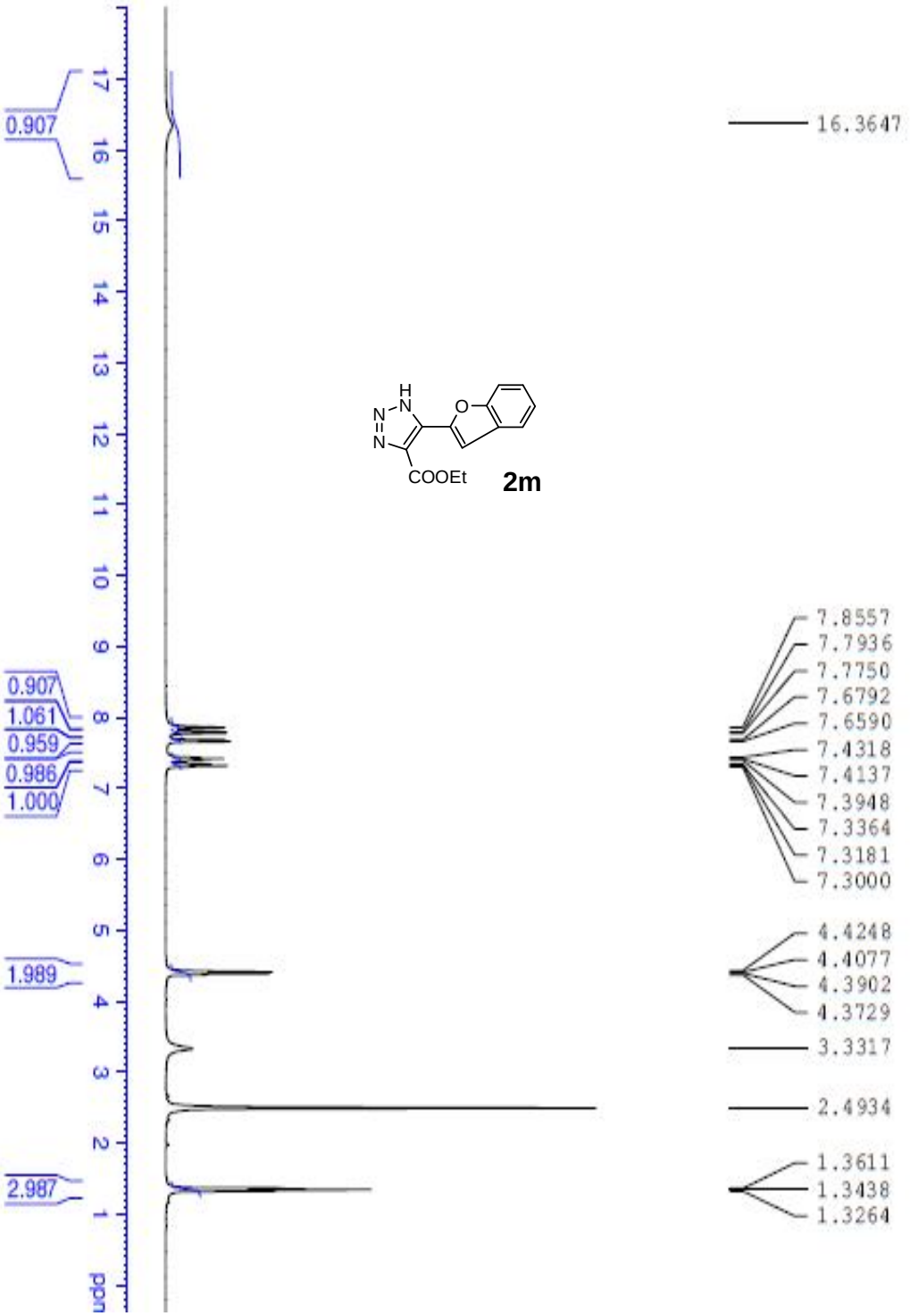


Peak No	RT min	Area	Area %
1	3.135	3.775e+003	97.769
2	3.492	4.428e+000	0.115
3	4.028	6.973e+000	0.181
4	4.134	1.052e+001	0.273
5	4.511	2.093e+001	0.542
6	4.624	1.029e+001	0.266
7	4.941	1.760e+001	0.456
8	5.212	1.539e+001	0.399

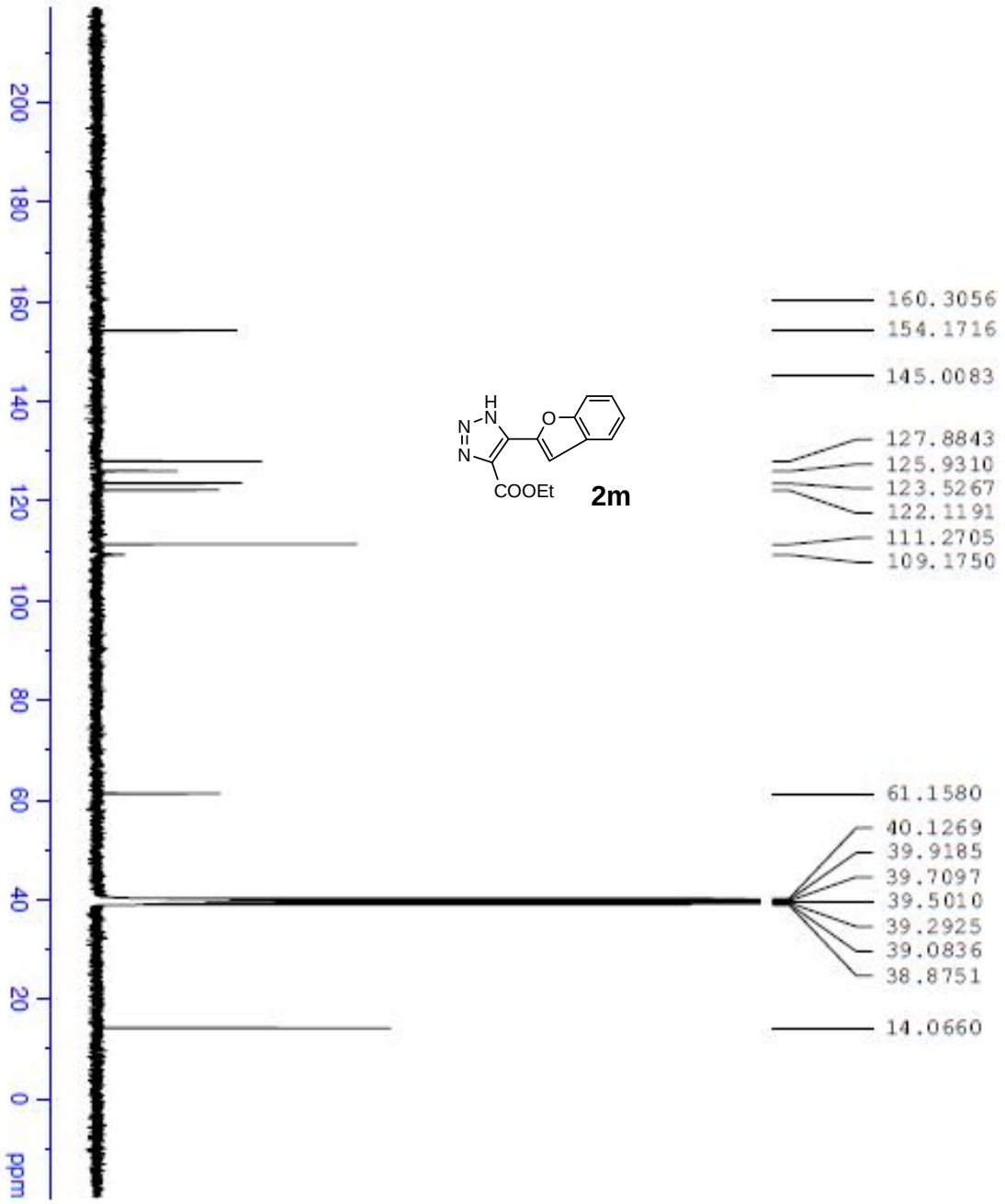
HPLC (**2l**)


%Transmittance
Wavenumbers (cm-1)
*IS10265-007-A8210419
OMe
COOEt
2l
3114.3
2800.0
2361.5
2334.2
2271.4
2196.7
2165.6
2129.0
2094.7
2008.6
1733.5
1704.7
1616.1
1569.1
1529.9
1446.8
1425.5
1380.6
1317.9
1283.1
1182.3
1144.8
1049.0
1002.4
978.6
920.7
867.5
840.4
820.9
790.3
755.1
710.5
662.2
633.8
570.6
534.0
511.4
420.0

IR (**2l**)



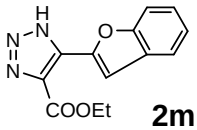
^{1}H NMR (400 MHz) in DMSO-d_6 (**2m**)



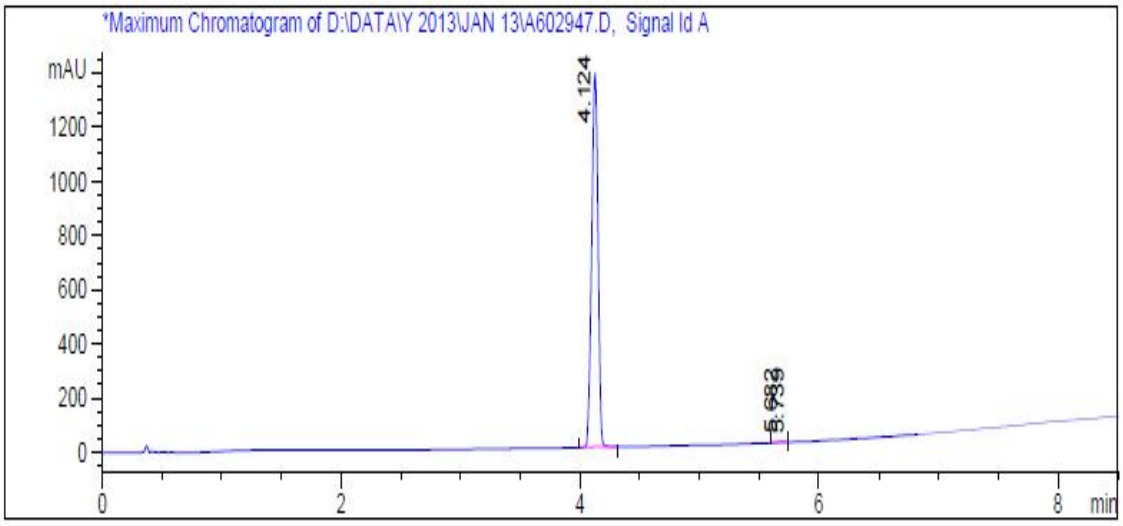
^{13}C NMR (100 MHz) in DMSO-d_6 (**2m**)

Method info : A : 0.1%TFA IN H2O B: 0.1%TFA IN ACN Flow = 2.0 mL/min
COLUMN:XBridge C8 (50X4.6)mm,3.5µm , +ve mode

Time	% of B
0	5
8.0	100
8.1	100
8.5	5
10.0	5

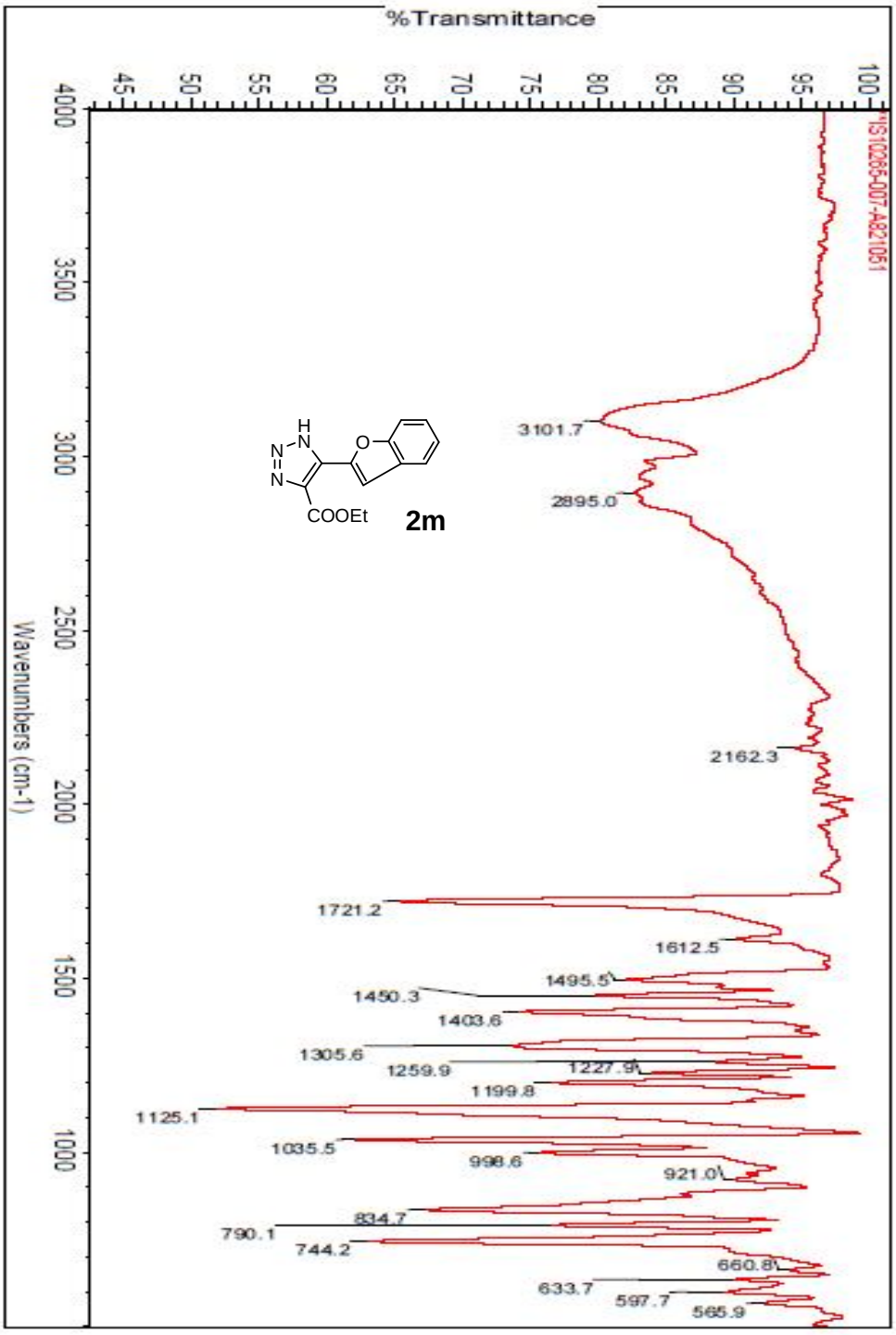


2m

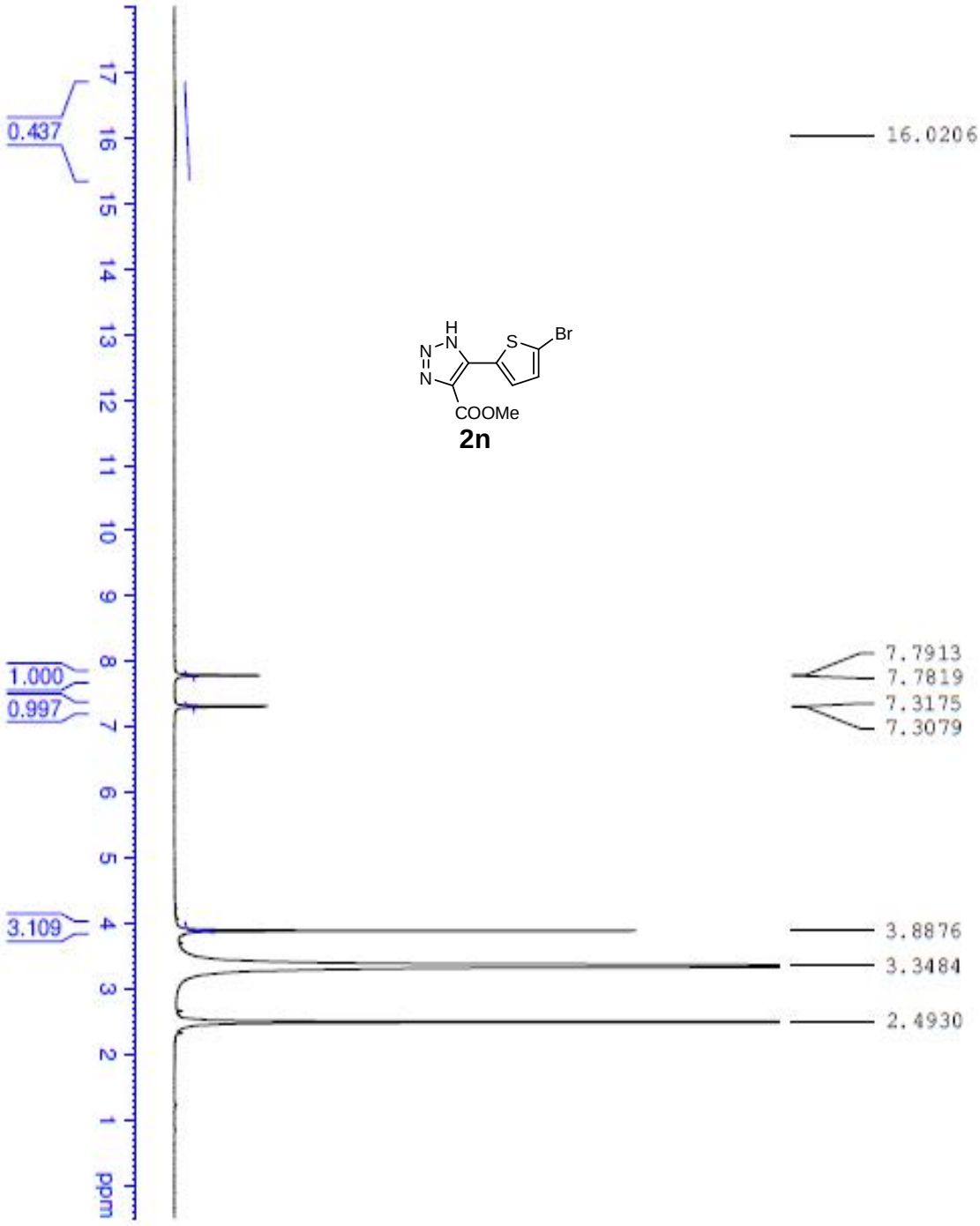


Peak No	RT min	Area	Area %
1	4.124	5.043e+003	99.830
2	5.682	8.387e+000	0.166
3	5.739	1.756e-001	0.003

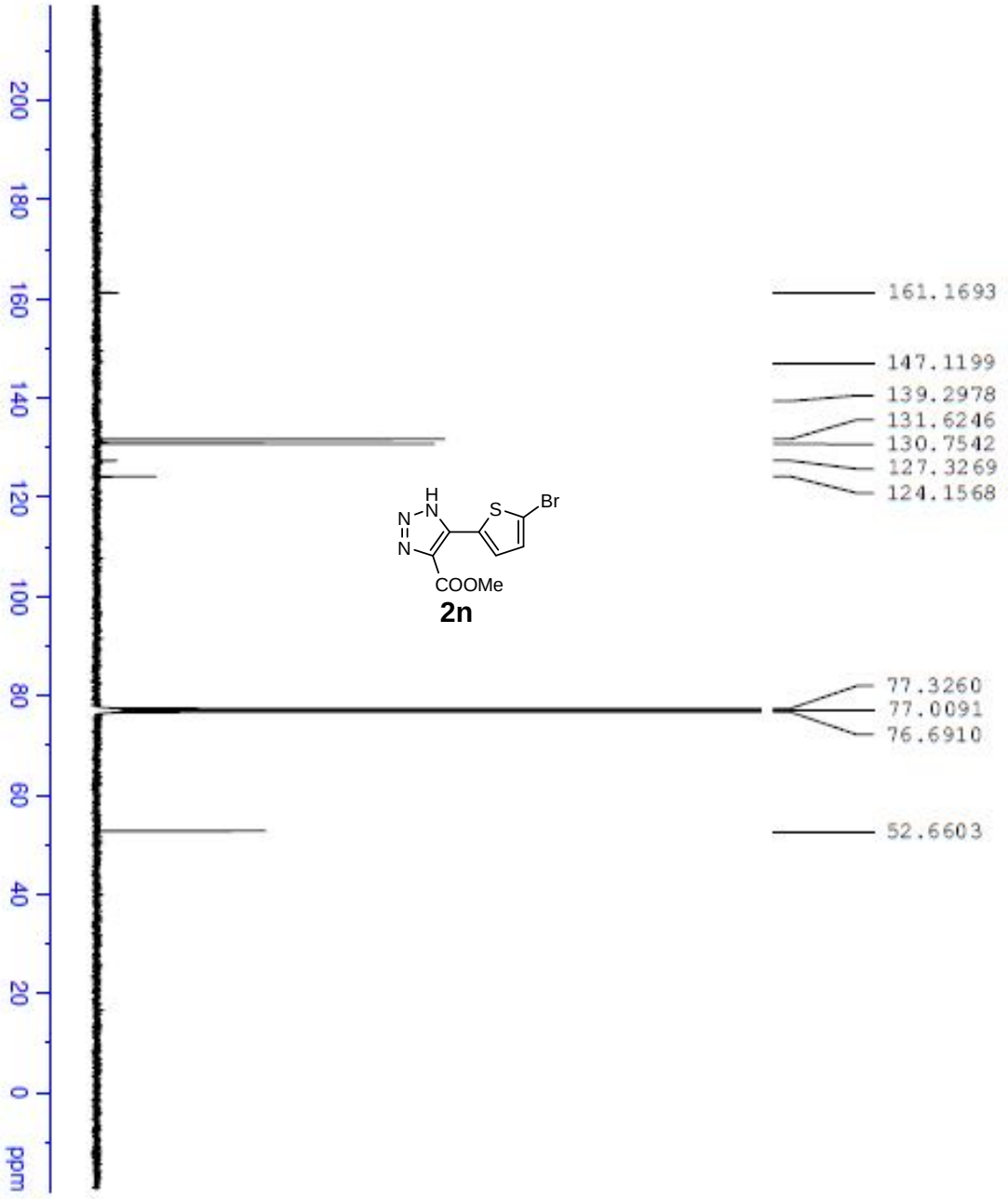
HPLC (**2m**)


%Transmittance
Wavenumbers (cm-1)
*IS10265-007-A821051
3101.7
2895.0
2162.3
1721.2
1612.5
1495.5
1450.3
1403.6
1305.6
1259.9
1227.9
1199.8
1125.1
1035.5
998.6
921.0
834.7
790.1
744.2
660.8
633.7
597.7
565.9
COOEt
2m

IR (**2m**)



^{1}H NMR (400 MHz) in DMSO-d_6 (**2n**)



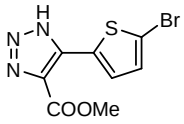
^{13}C NMR (100 MHz) in DMSO-d_6 (**2n**)

Method info :

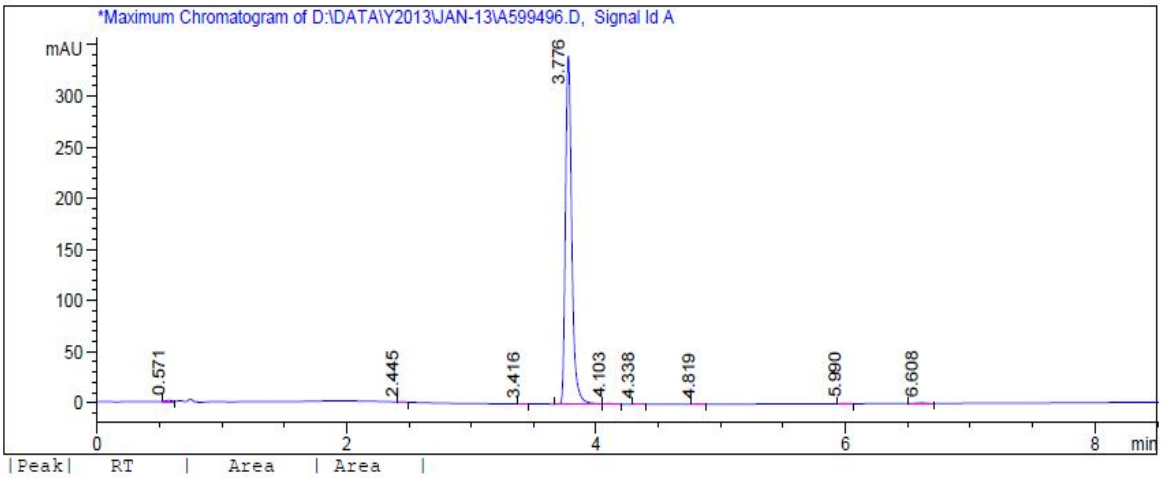
A-10mM NH4HCO3 IN H2O , B- ACN Flow: 1.0 ml/min

COLUMN: XBridge C8 (50X4.6mm, 3.5µm), -ve mode

TIME	%B
0	05
8.0	100
8.1	100
8.5	05
10	05

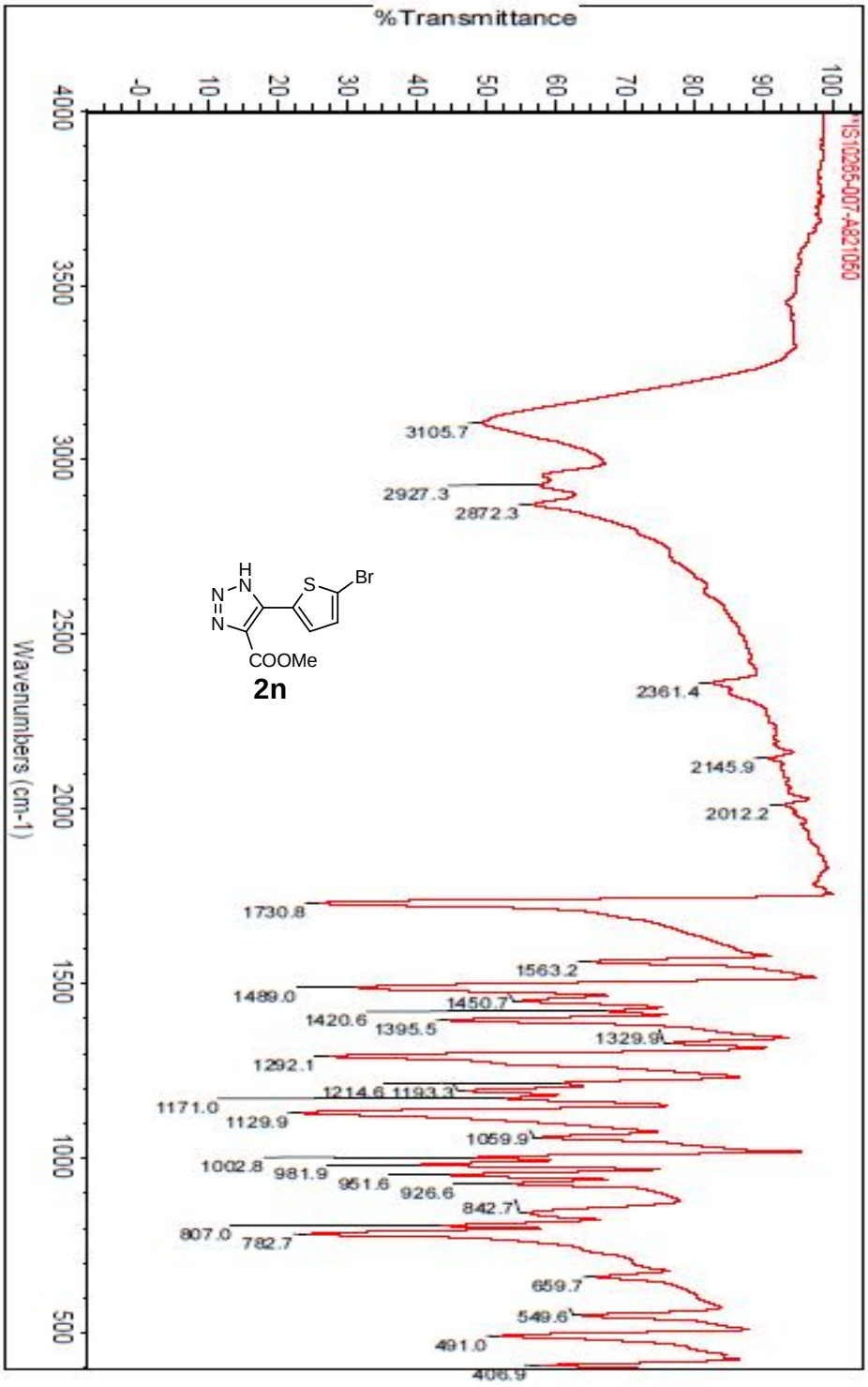


2n

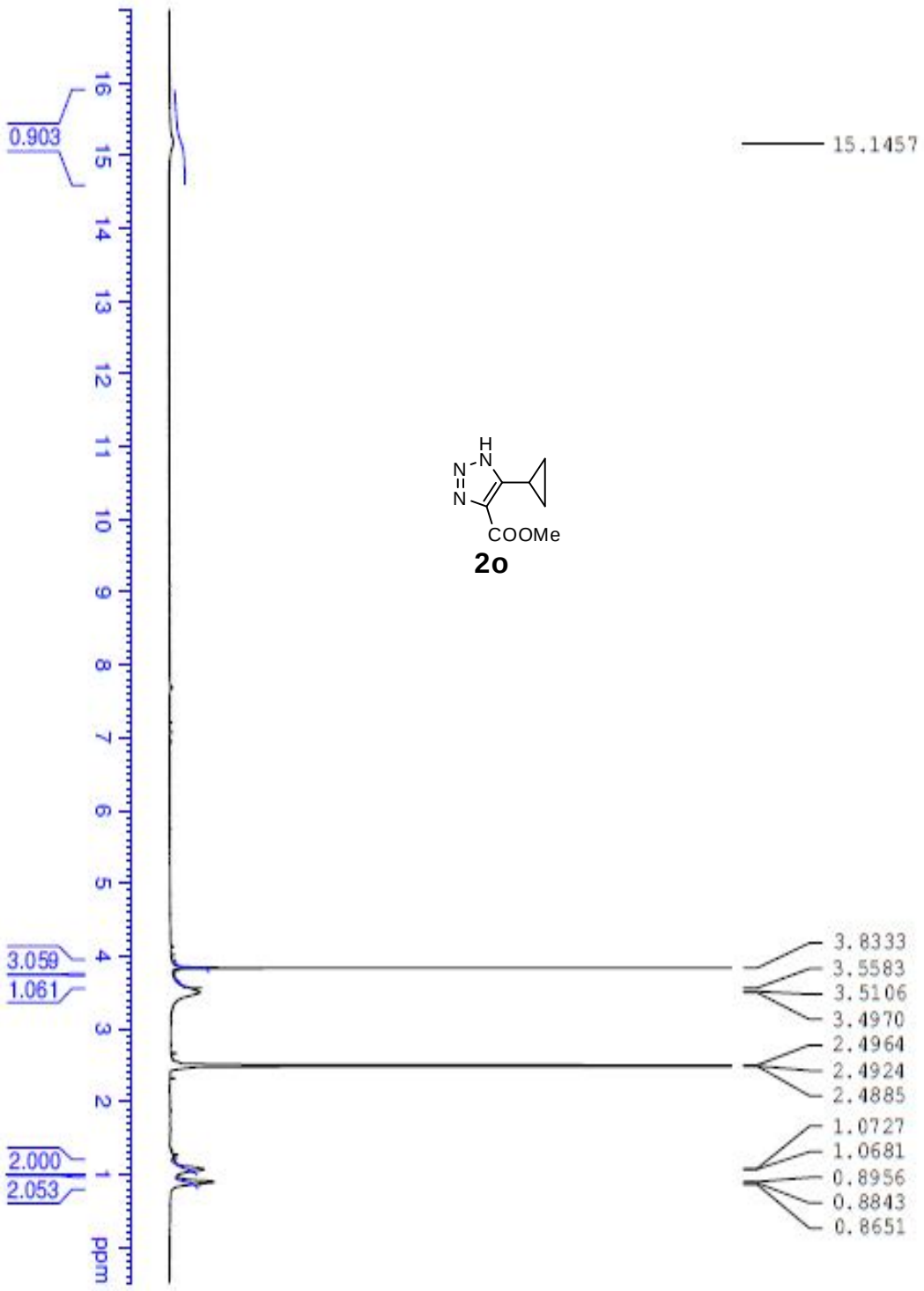


Peak No	RT min	Area	Area %
1	0.571	4.722e+000	0.389
2	2.445	6.176e-001	0.051
3	3.416	2.909e-001	0.024
4	3.776	1.197e+003	98.729
5	4.103	2.774e+000	0.229
6	4.338	1.621e+000	0.134
7	4.819	5.845e-001	0.048
8	5.990	1.409e+000	0.116
9	6.608	3.394e+000	0.280

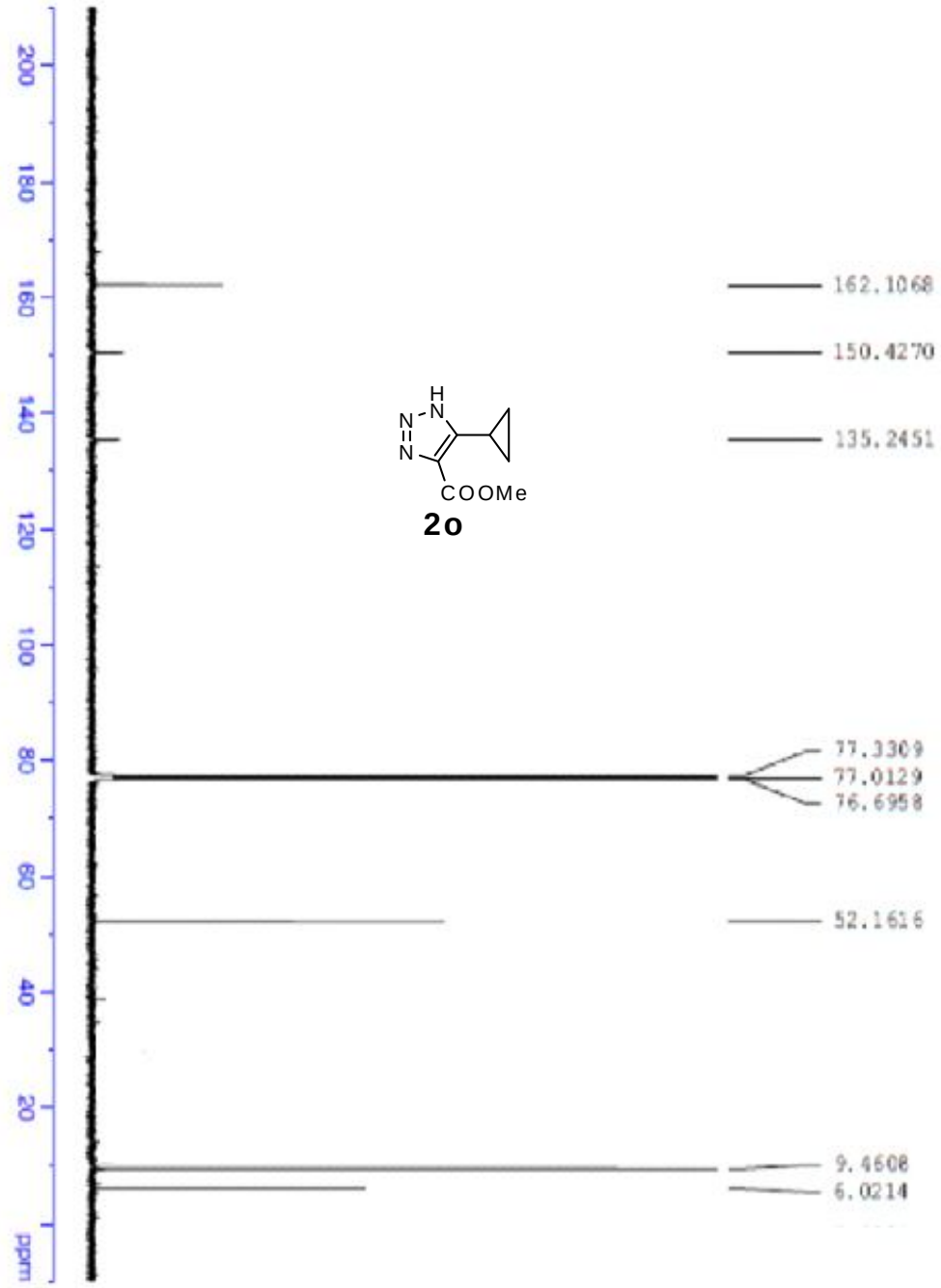
HPLC (**2n**)


%Transmittance
Wavenumbers (cm-1)
IS10265-007-A821050
3105.7
2927.3
2872.3
2361.4
2145.9
2012.2
1730.8
1563.2
1489.0
1450.7
1420.6
1395.5
1329.9
1292.1
1214.6
1193.3
1171.0
1129.9
1059.9
1002.8
981.9
951.6
926.6
842.7
807.0
782.7
659.7
549.6
491.0
406.9
COOMe
2n

IR (**2n**)



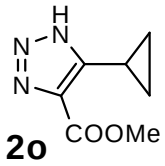
^{1}H NMR (400 MHz) in DMSO-d_6 (**2o**)



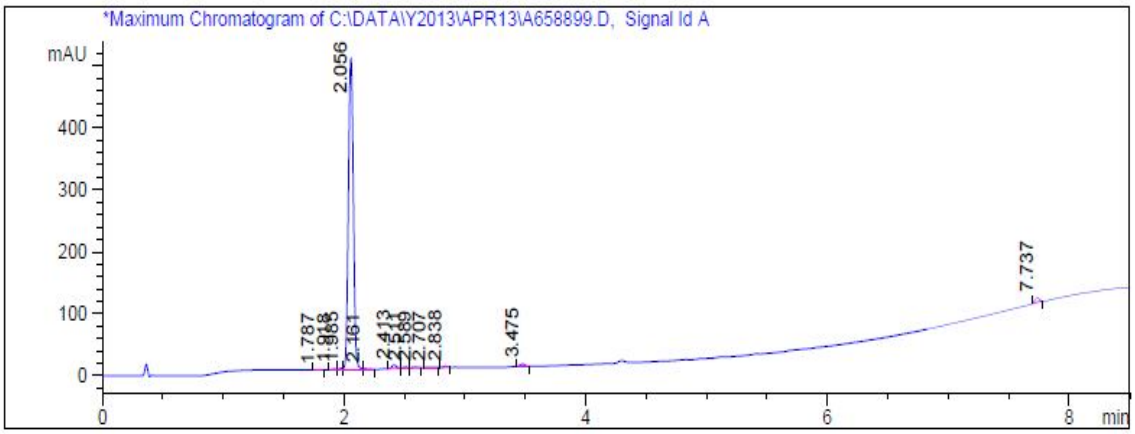
^{13}C NMR (100 MHz) in $CDCl_3$ (**2o**)

Method info : A:0.1%TFA in H2O, B:0.1%TFA in ACN, Flow Rate:2.0ml/min
COLUMN: XBridge C8(50X4.6)mm,3.5µm

Time	% of B
0	5
8.0	100
8.1	100
8.5	5
10.0	5

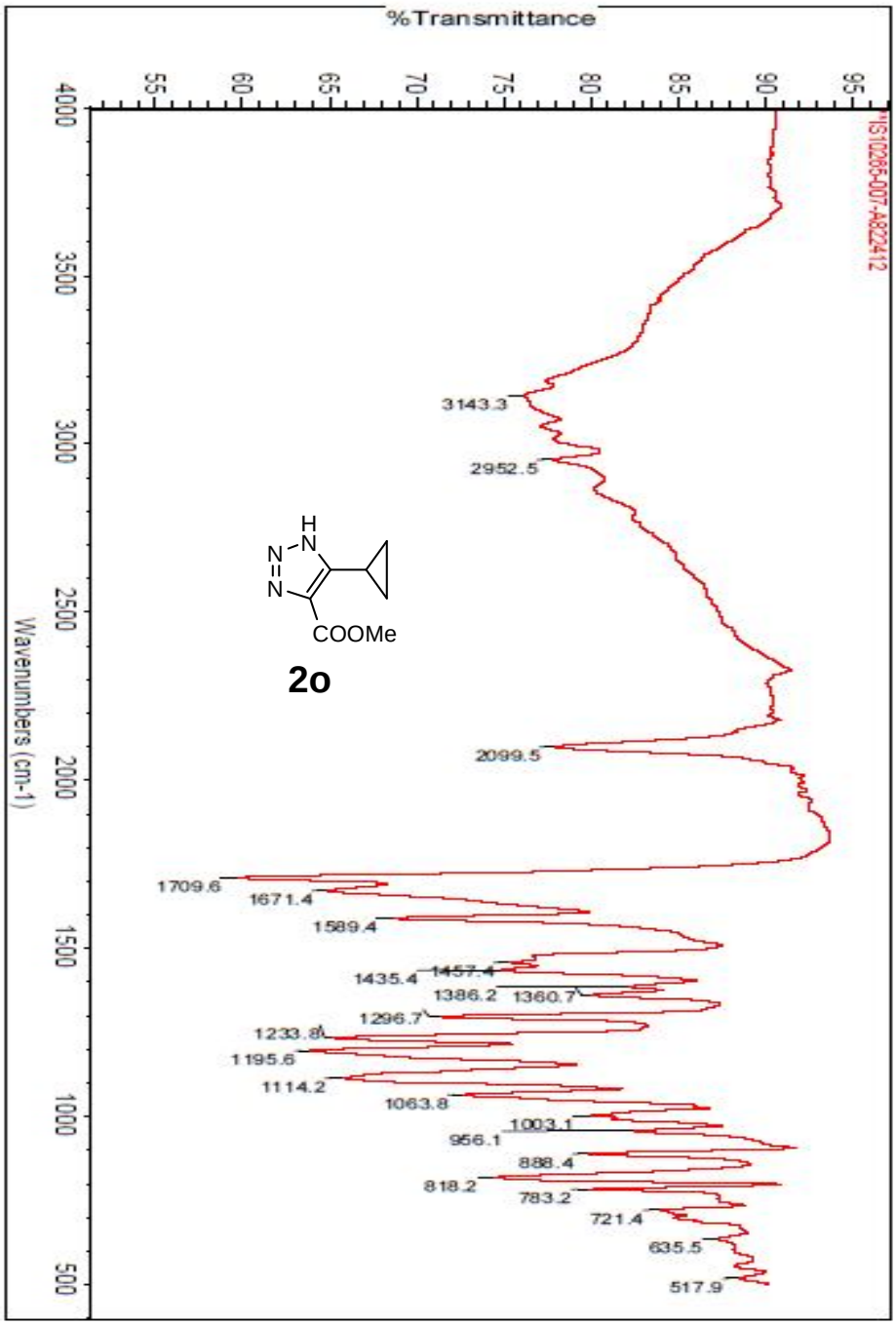


2o

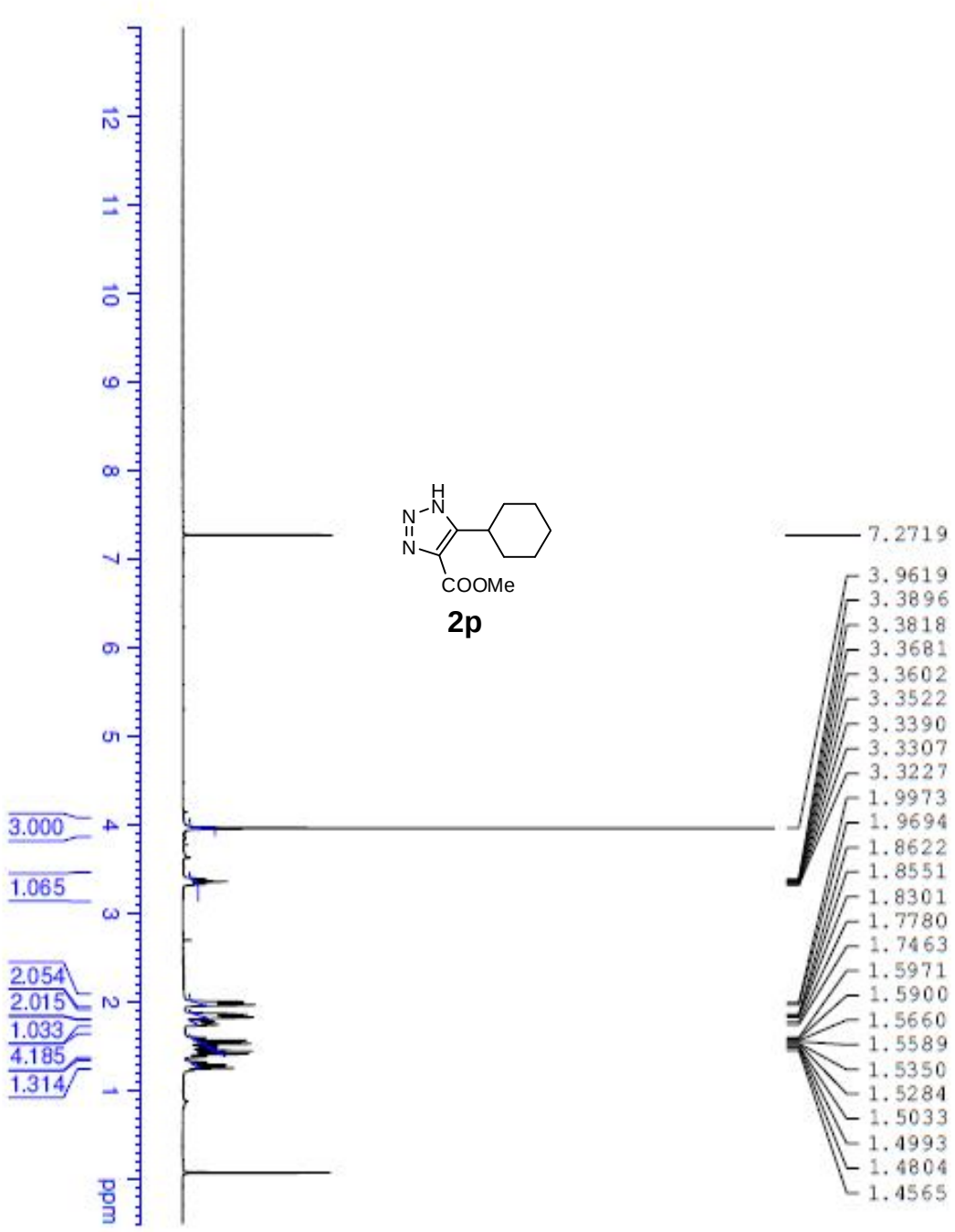


Peak No	RT min	Area	Area %
1	1.787	1.368e+000	0.09
2	1.918	4.936e+000	0.33
3	1.985	5.626e+000	0.37
4	2.056	1.422e+003	94.20
5	2.161	3.200e+000	0.21
6	2.413	1.926e+001	1.28
7	2.511	7.018e+000	0.46
8	2.589	6.261e+000	0.41
9	2.707	4.404e+000	0.29
10	2.838	5.384e+000	0.36
11	3.475	1.049e+001	0.69
12	7.737	1.953e+001	1.29

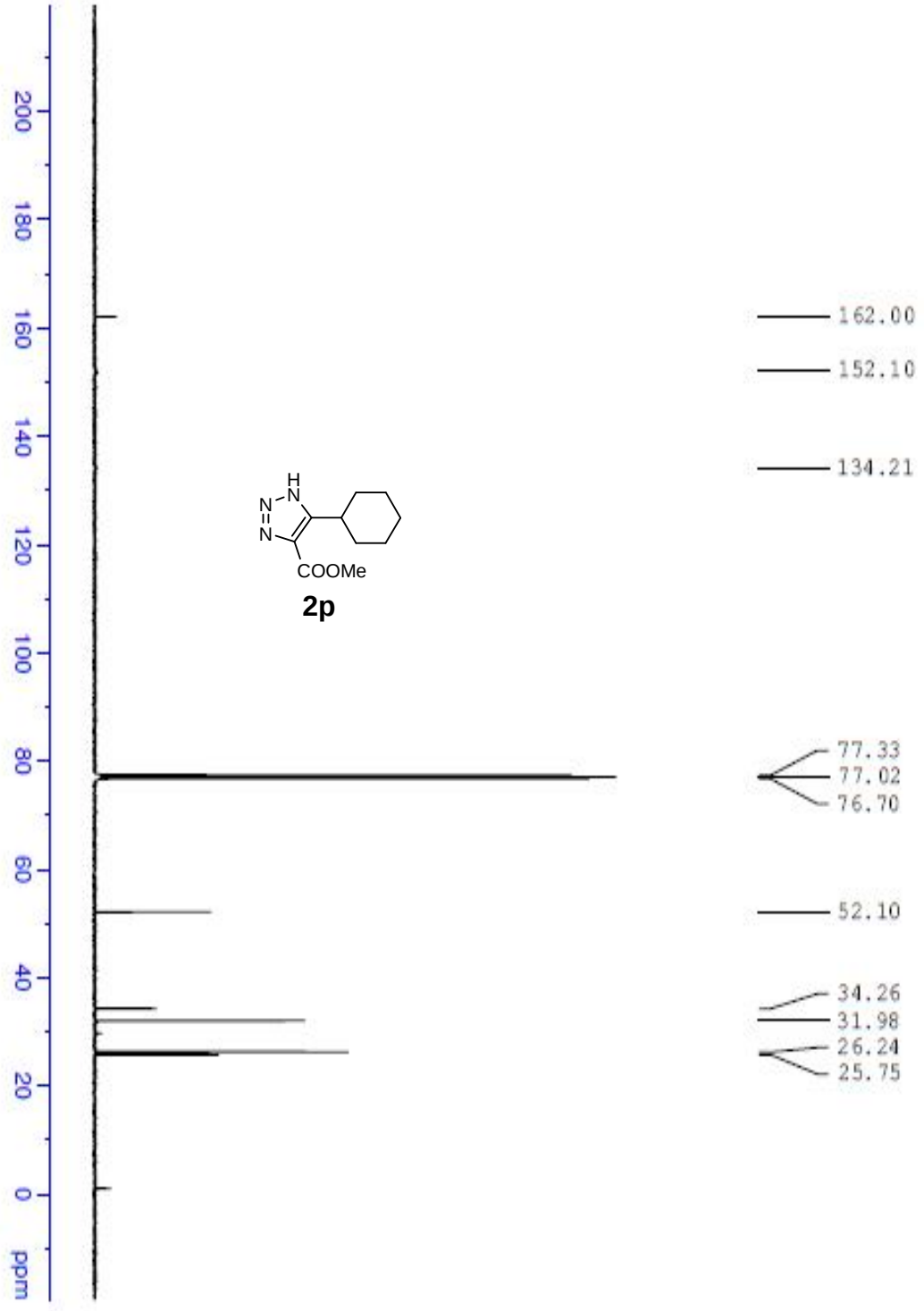
HPLC (**2o**)


%Transmittance
Wavenumbers (cm-1)
4000
3500
3000
2500
2000
1500
1000
500
55
60
65
70
75
80
85
90
95
3143.3
2952.5
2099.5
1709.6
1671.4
1589.4
1435.4
1457.4
1386.2
1360.7
1296.7
1233.8
1195.6
1114.2
1063.8
1003.1
956.1
888.4
818.2
783.2
721.4
635.5
517.9
COOMe
2o

IR (**2o**)



^{1}H NMR (400 MHz) in DMSO-d_6 (**2p**)

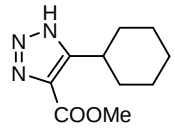


^{13}C NMR (100 MHz) in DMSO-d_6 (**2p**)

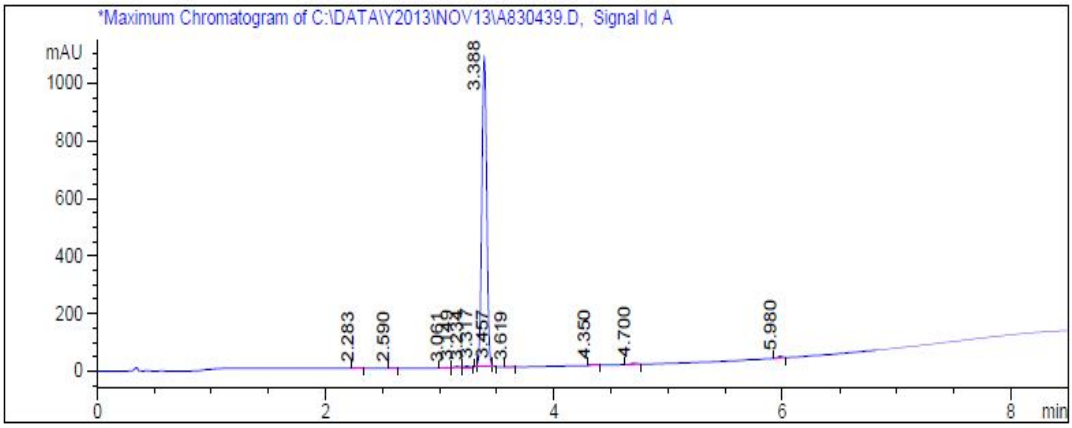
Method info : A:0.1%TFA in H2O, B:0.1%TFA in ACN, Flow Rate:2.0ml/min

COLUMN: XBridge C8(50X4.6)mm,3.5µm

Time	% of B
0	5
8.0	100
8.1	100
8.5	5
10.0	5

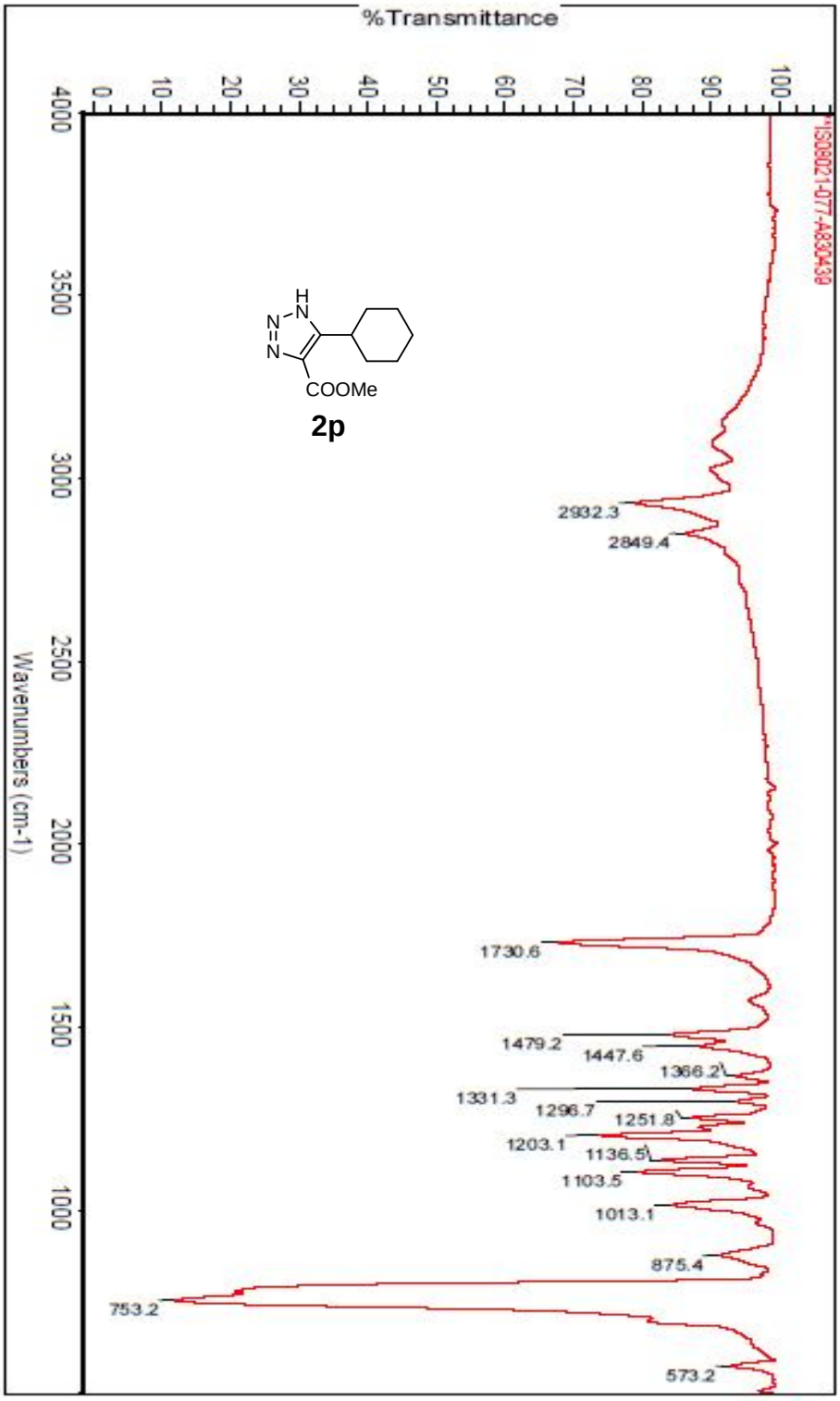


2p

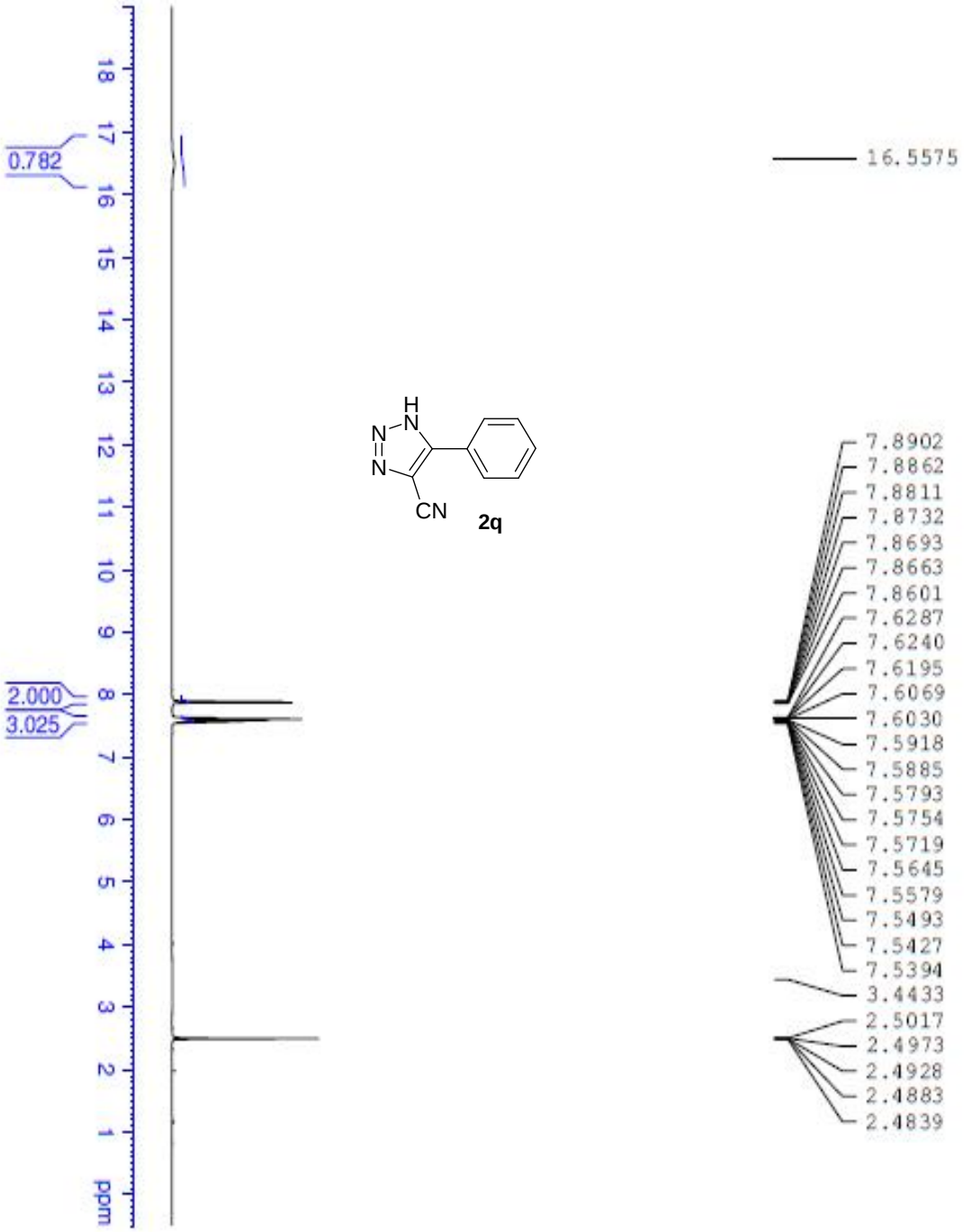


Peak No	RT min	Area	Area %
1	2.283	1.158e+001	0.36
2	2.590	1.994e+000	0.06
3	3.061	3.543e+000	0.11
4	3.149	1.483e+001	0.46
5	3.234	1.857e+001	0.57
6	3.317	7.646e+000	0.24
7	3.388	3.145e+003	96.70
8	3.457	2.277e+000	0.07
9	3.619	3.762e+000	0.12
10	4.350	7.162e+000	0.22
11	4.700	1.958e+001	0.60
12	5.980	1.627e+001	0.50

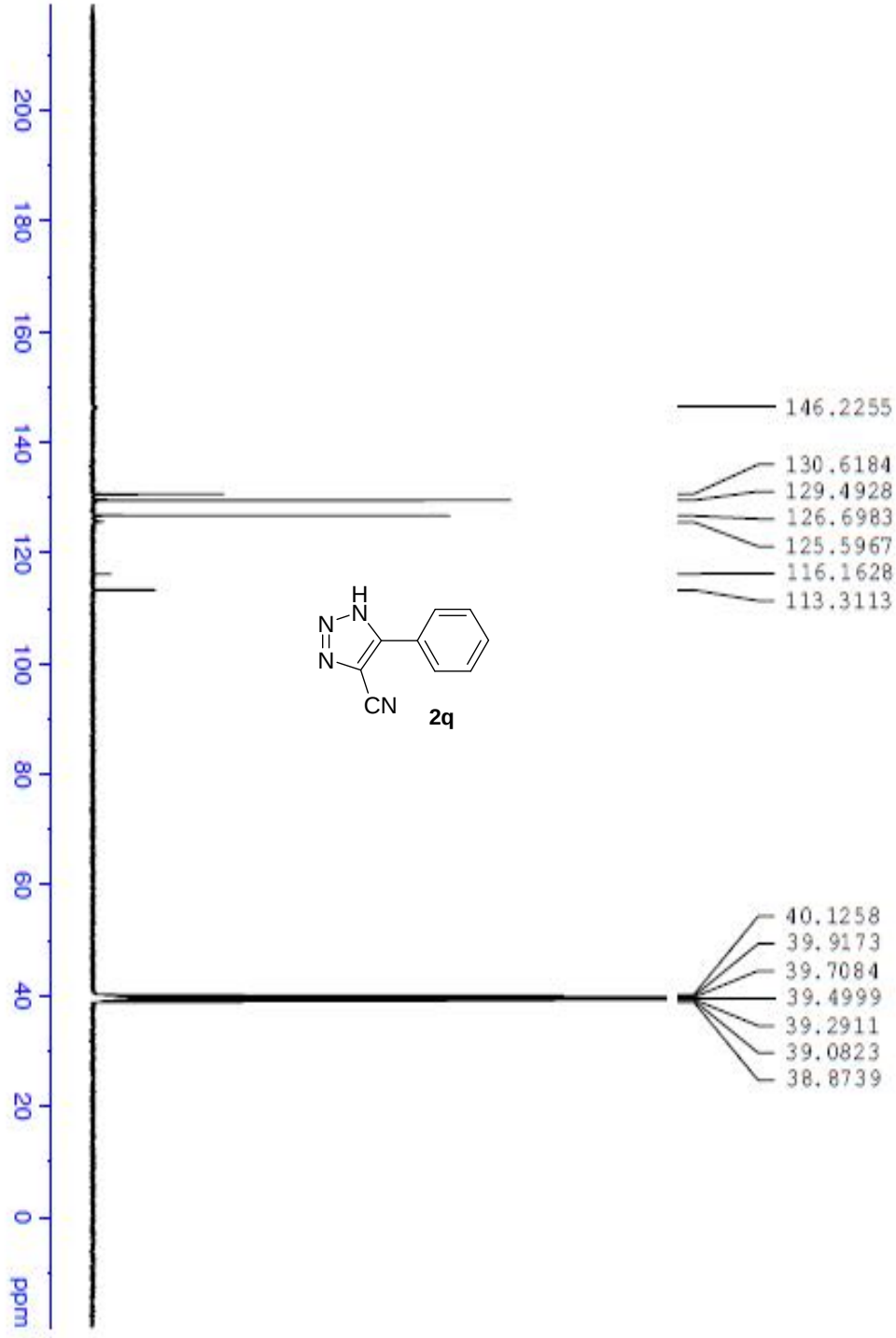
HPLC (**2p**)


%Transmittance
Wavenumbers (cm-1)
2932.3
2849.4
1730.6
1479.2
1447.6
1366.2
1331.3
1296.7
1251.8
1203.1
1136.5
1103.5
1013.1
875.4
753.2
573.2
COOMe
2p

IR (**2p**)



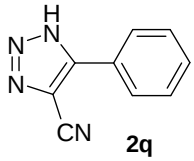
^{1}H NMR (400 MHz) in DMSO-d_6 (**2q**)



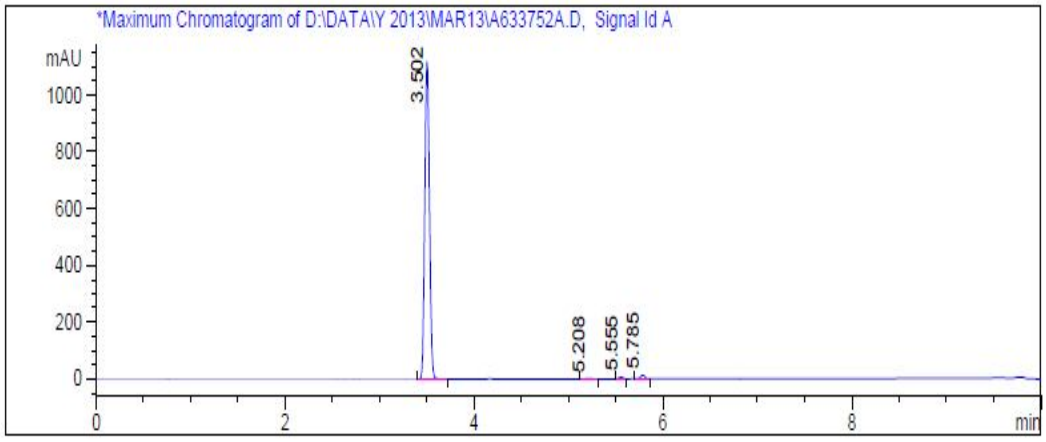
^{13}C NMR (100 MHz) in DMSO-d_6 (**2q**)

Method info : A : 10 mM NH4HCo3 H2O B: ACN Flow = 1.0 mL/min
COLUMN:XBridge C8 (50X4.6)mm,3.5µm , -ve mode

Time	% of B
0	5
8.0	100
8.1	100
8.5	5
10.0	5

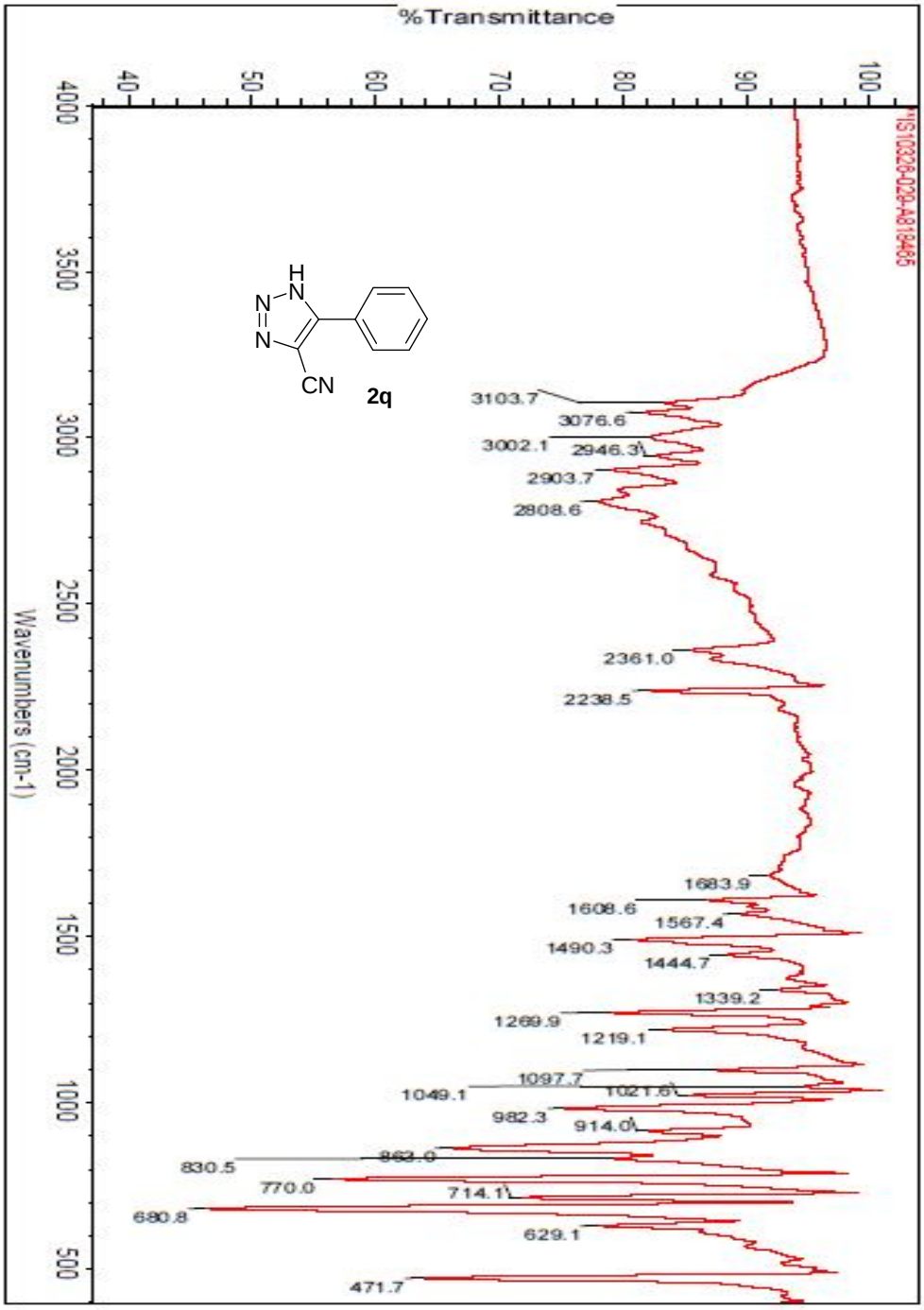


2q

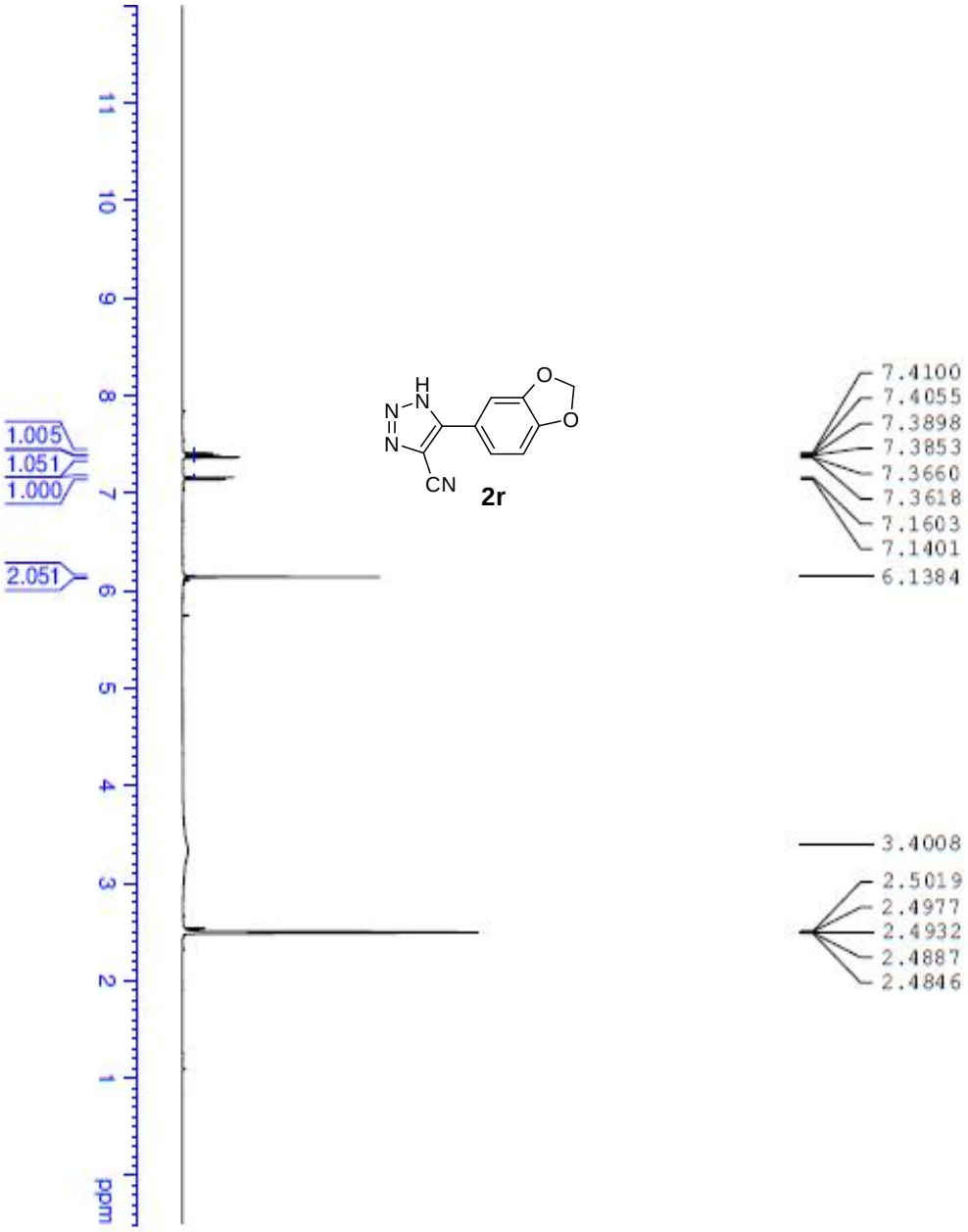


Peak No	RT min	Area	Area %
1	3.502	3.908e+003	98.348
2	5.208	3.817e+000	0.096
3	5.555	1.995e+001	0.502
4	5.785	4.189e+001	1.054

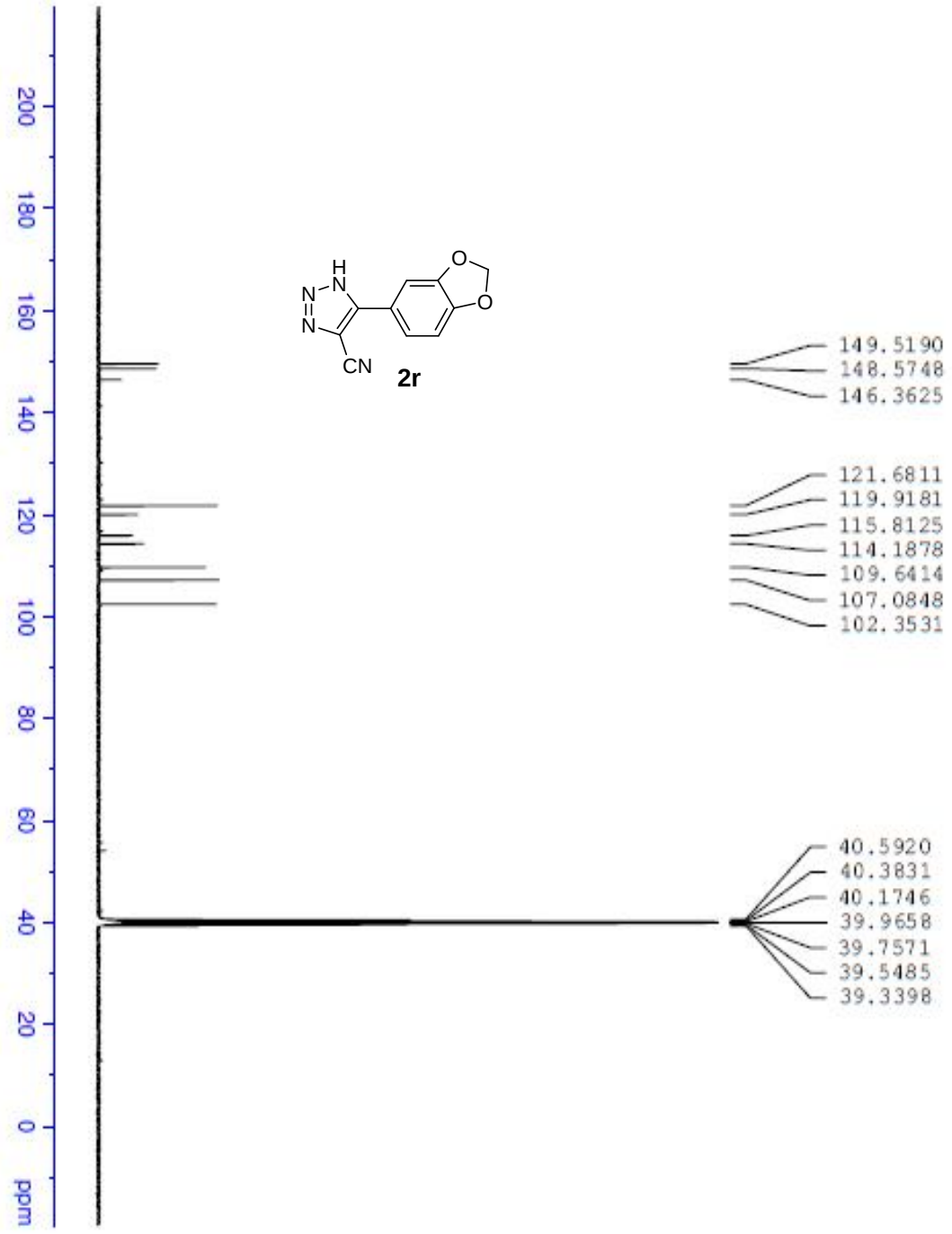
HPLC (**2q**)


%Transmittance
Wavenumbers (cm-1)
2q
3103.7
3076.6
3002.1
2946.3
2903.7
2808.6
2361.0
2238.5
1683.9
1608.6
1567.4
1490.3
1444.7
1339.2
1269.9
1219.1
1097.7
1049.1
1021.6
982.3
914.0
863.0
830.5
770.0
714.1
680.8
629.1
471.7

IR (**2q**)



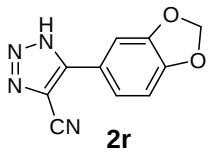
^{1}H NMR (400 MHz) in DMSO-d_6 (**2r**)



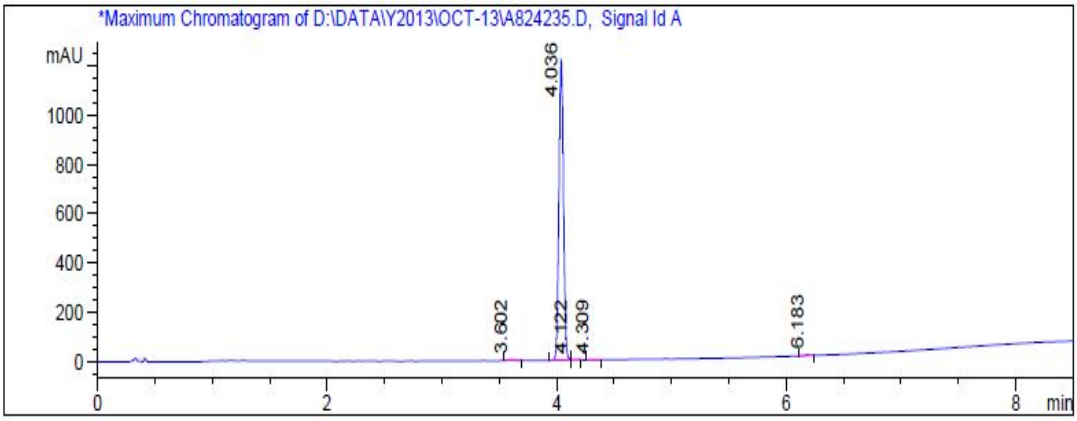
^{13}C NMR (100 MHz) in DMSO-d_6 (**2r**)

Method info : A: 0.1% TFA IN H2O , B:0.1% TFA IN ACN ; Flow Rate:2.0 ml/min
COLUMN:XBridge C8 (50x4.6mm, 3.5µ), +ve mode

TIME	%B
0	05
8.0	100
8.1	100
8.5	05
10.0	05

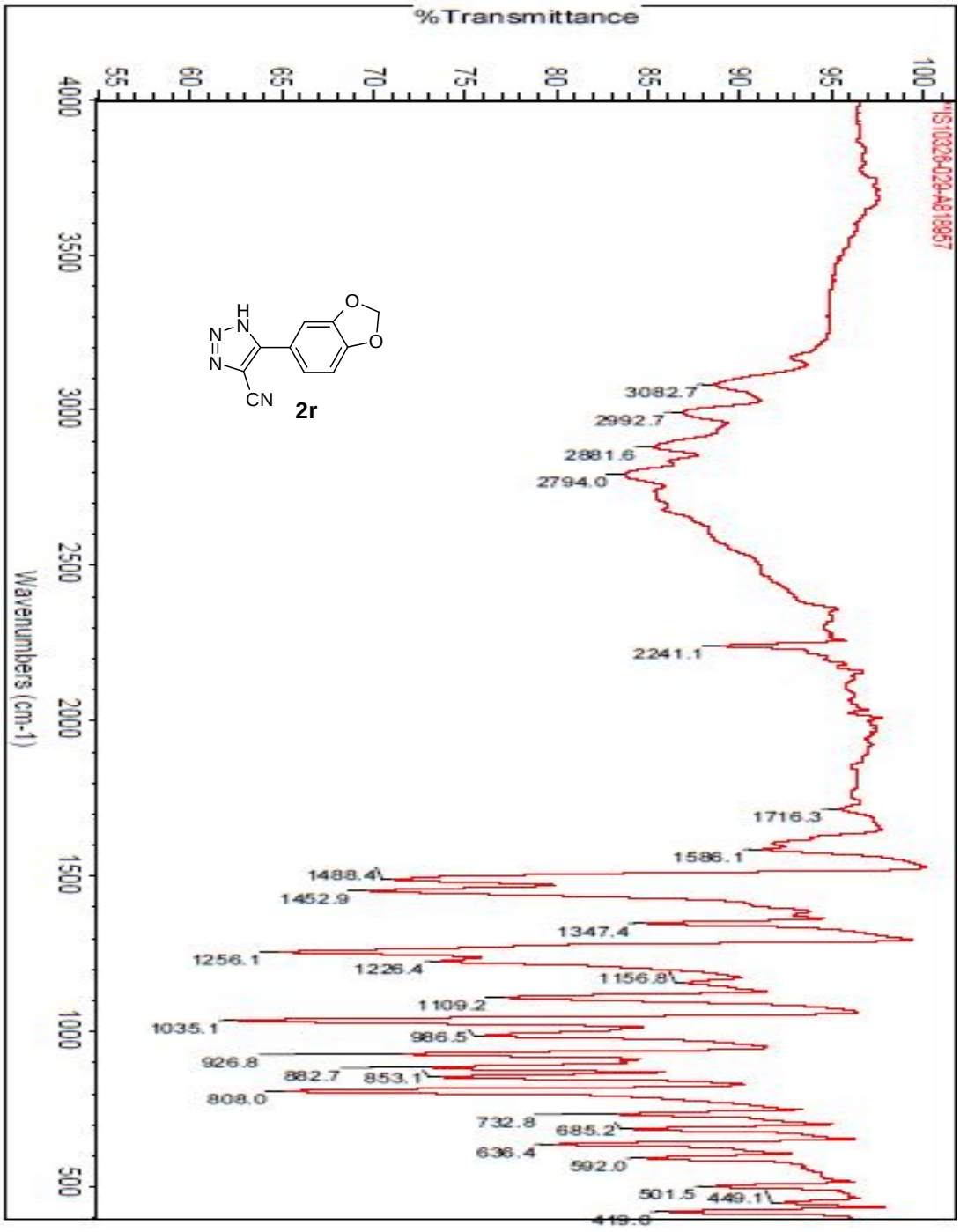


2r

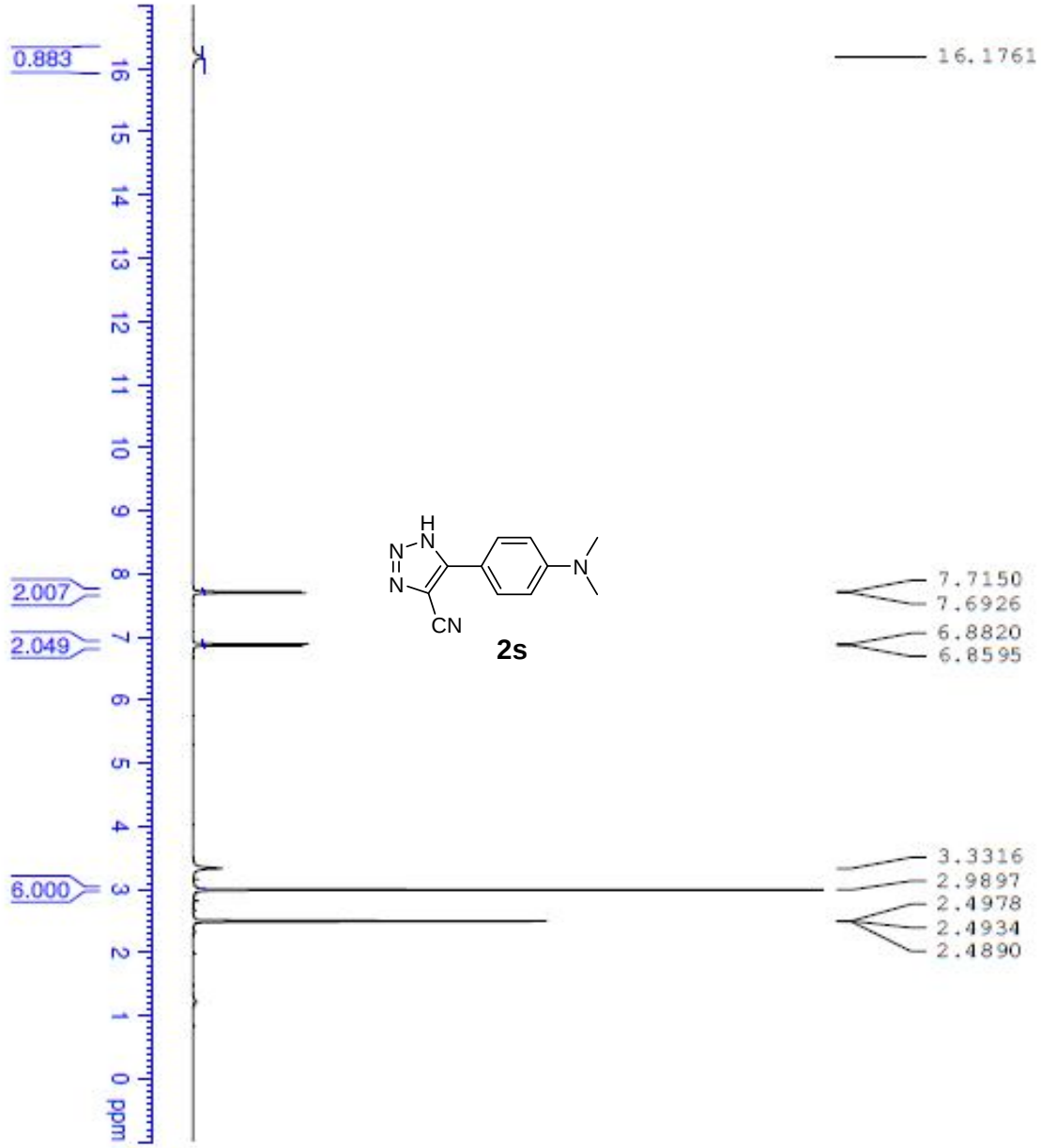


Peak No	RT min	Area	Area %
1	3.602	1.739e+001	0.511
2	4.036	3.359e+003	98.680
3	4.122	4.808e+000	0.141
4	4.309	9.103e+000	0.267
5	6.183	1.361e+001	0.400

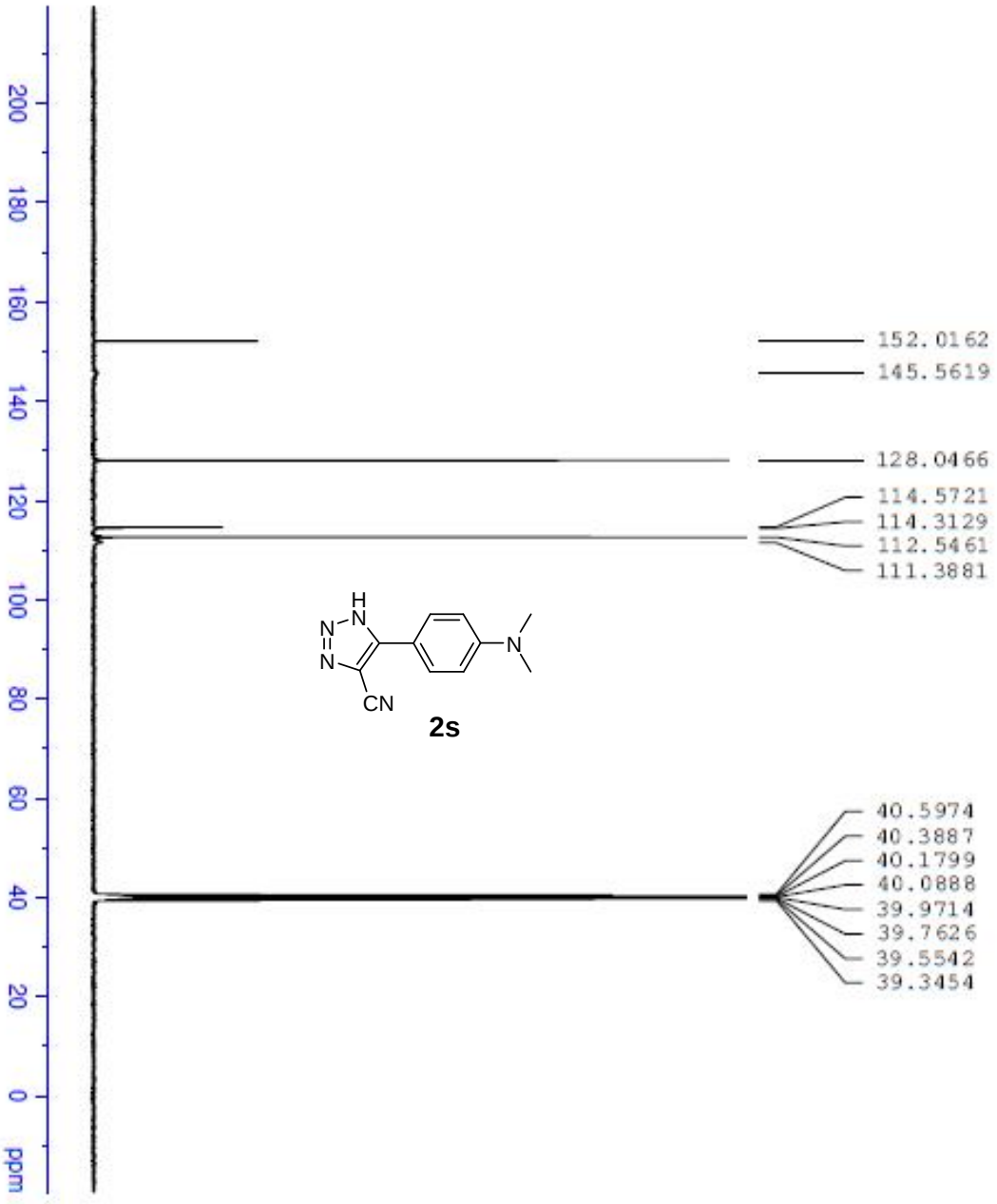
HPLC (**2r**)


%Transmittance
55
60
65
70
75
80
85
90
95
100
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)
*IS10326-029-A819857
H
N
N
N
O
O
CN
2r
3082.7
2992.7
2881.6
2794.0
2241.1
1716.3
1586.1
1488.4
1452.9
1347.4
1256.1
1226.4
1156.8
1109.2
1035.1
986.5
926.8
882.7
853.1
808.0
732.8
685.2
636.4
592.0
501.5
449.1
419.0

IR (**2r**)



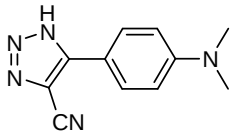
^{1}H NMR (400 MHz) in DMSO-d_6 (**2s**)



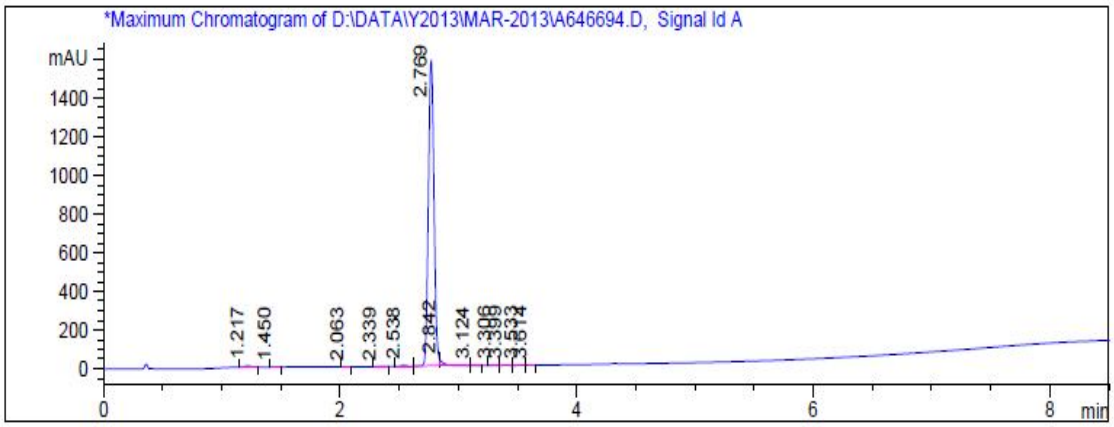
^{13}C NMR (100 MHz) in DMSO-d_6 (**2s**)

Method info : A: 0.1% TFA IN H2O , B:0.1% TFA IN ACN ; Flow Rate:2.0 ml/min
COLUMN:XBridge C8 (50x4.6mm, 3.5µ), +ve mode

TIME	%B
0	05
8.0	100
8.1	100
8.5	05
10.0	05

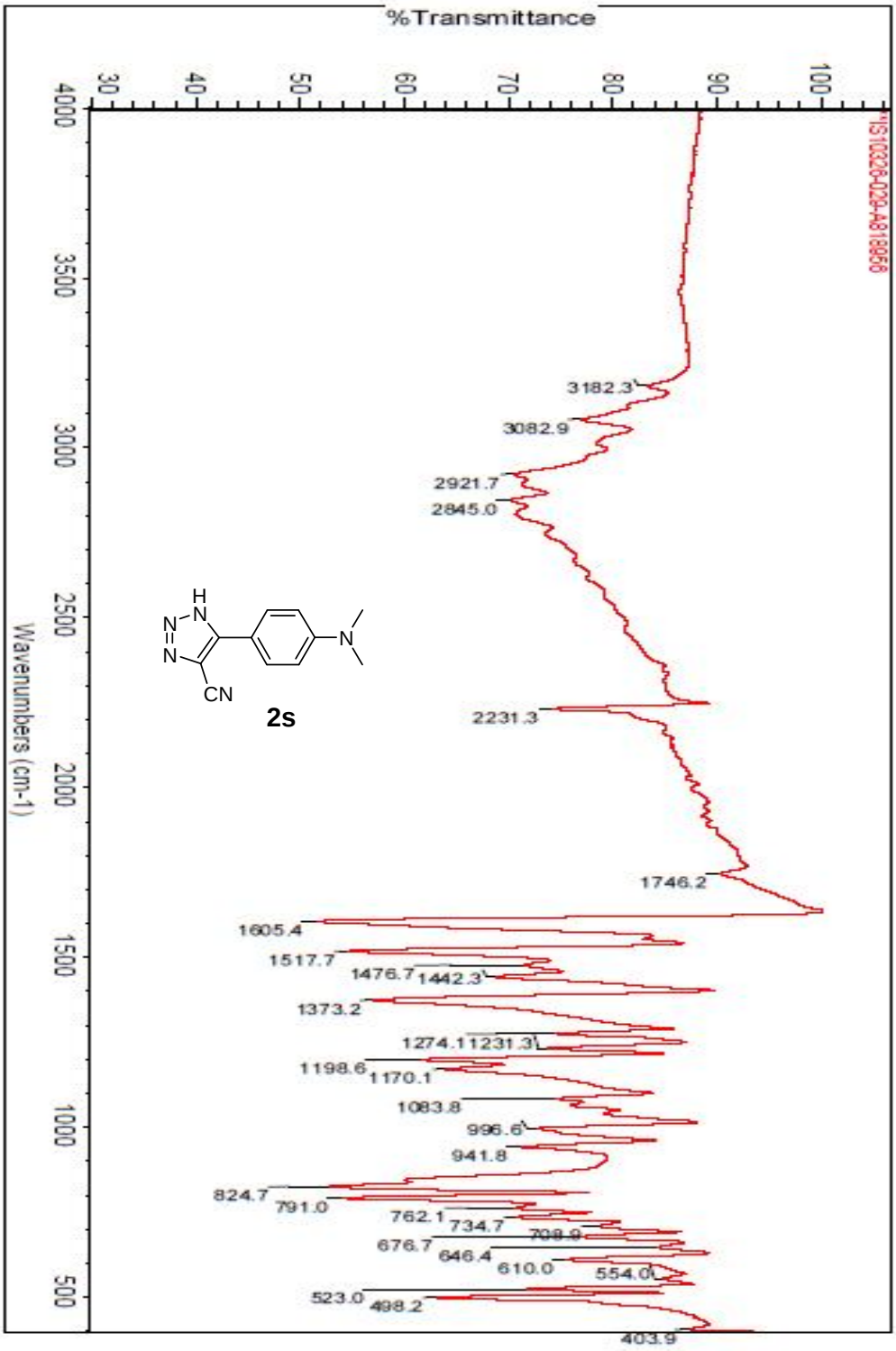


2s

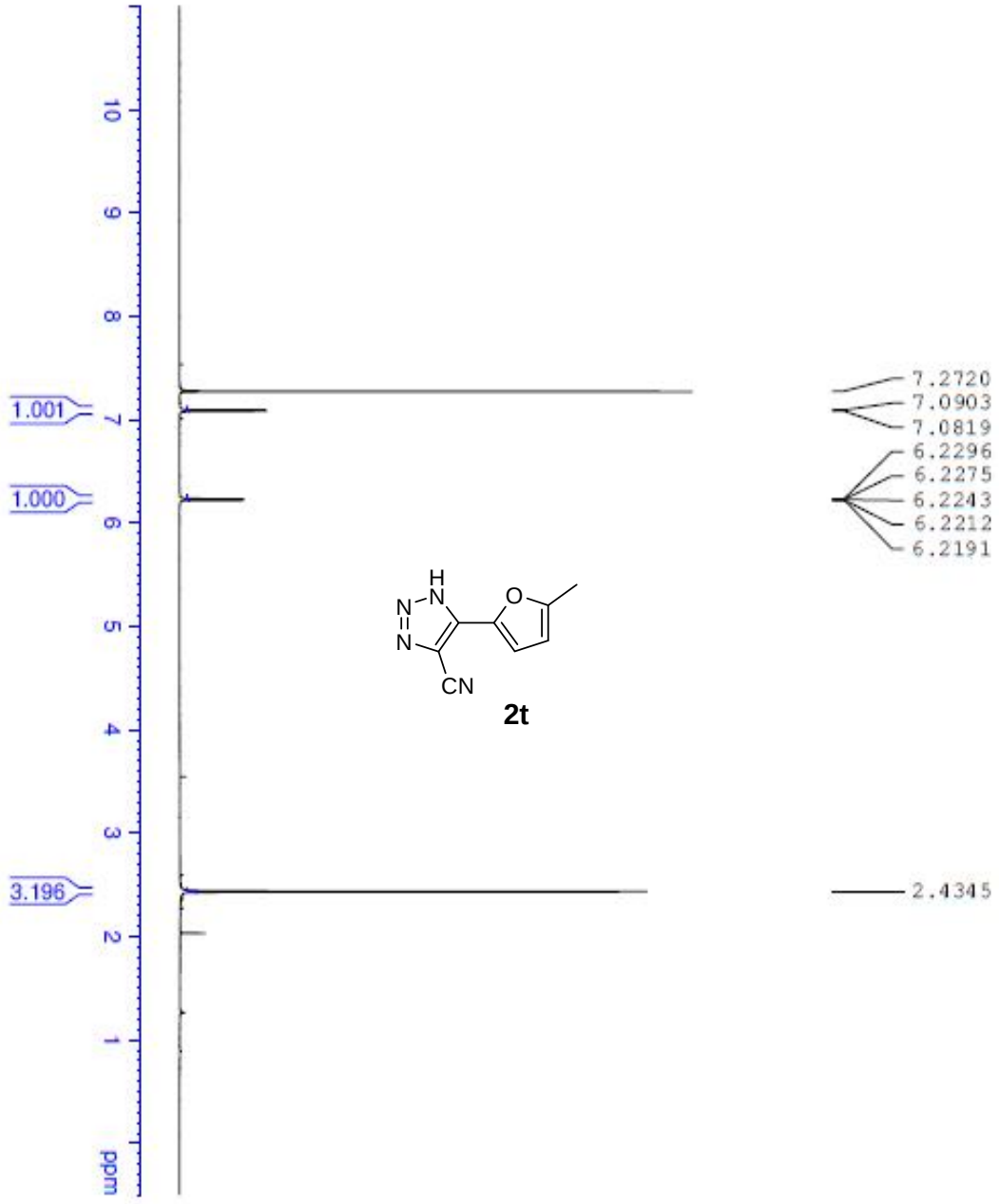


Peak No	RT min	Area	Area %
1	1.217	2.648e+001	0.492
2	1.450	1.461e+000	0.027
3	2.063	1.535e+000	0.029
4	2.339	6.883e+000	0.128
5	2.538	1.539e+001	0.286
6	2.769	5.227e+003	97.188
7	2.842	7.976e+001	1.483
8	3.124	2.635e+000	0.049
9	3.306	4.325e+000	0.080
10	3.399	6.432e+000	0.120
11	3.533	4.157e+000	0.077
12	3.614	2.185e+000	0.041

HPLC (**2s**)

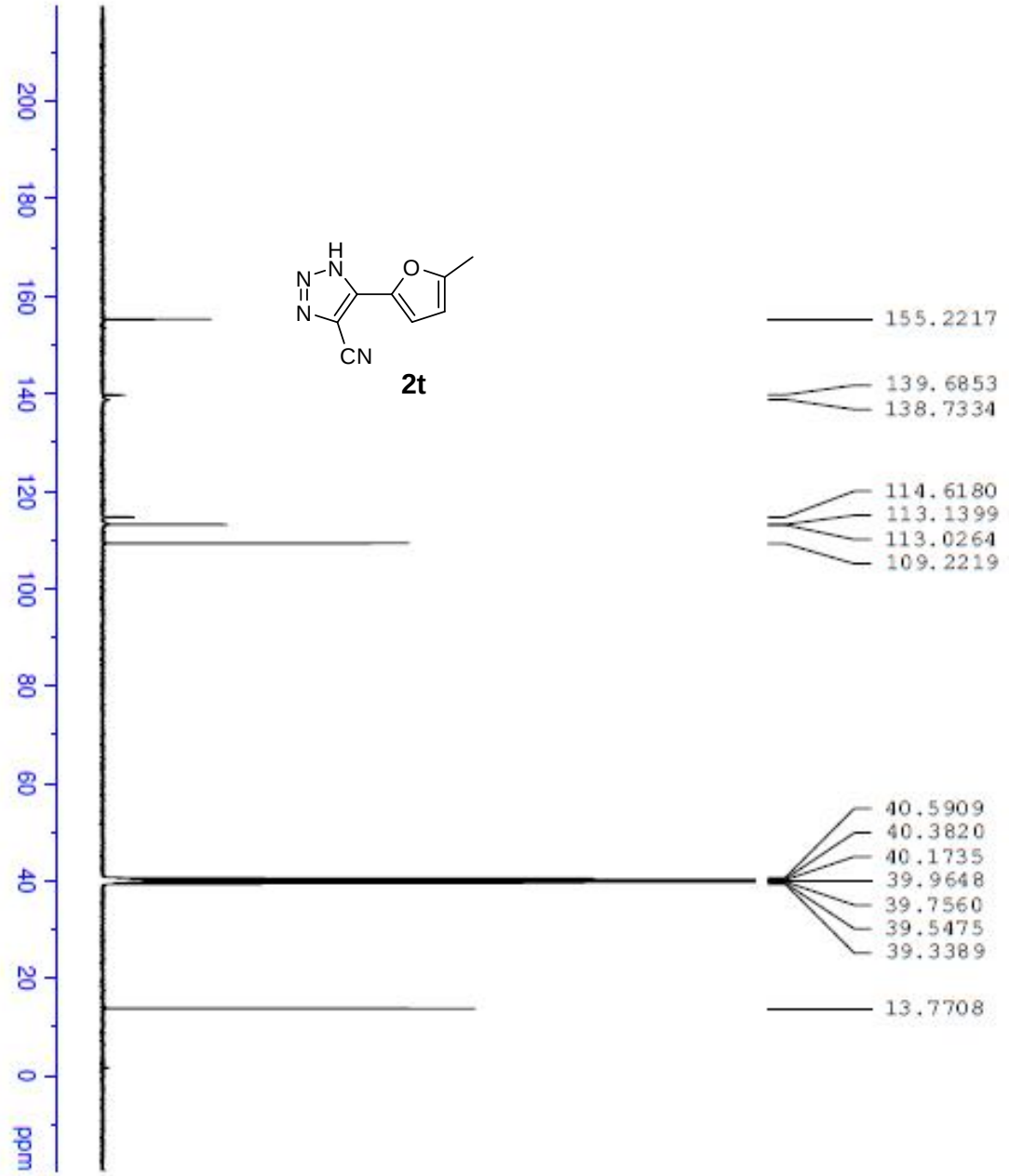

%Transmittance
30
40
50
60
70
80
90
100
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)
3182.3
3082.9
2921.7
2845.0
2231.3
1746.2
1605.4
1517.7
1476.7
1442.3
1373.2
1274.1
1231.3
1198.6
1170.1
1083.8
996.6
941.8
824.7
791.0
762.1
734.7
708.9
676.7
646.4
610.0
554.0
523.0
498.2
403.9
H
N
N
N
CN
N
2s

IR (**2s**)



2t

^{1}H NMR (400 MHz) in $CDCl_3$ (**2t**)



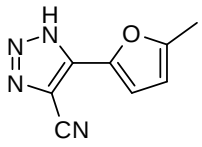
2t

^{13}C NMR (100 MHz) in DMSO-d_6 (**2t**)

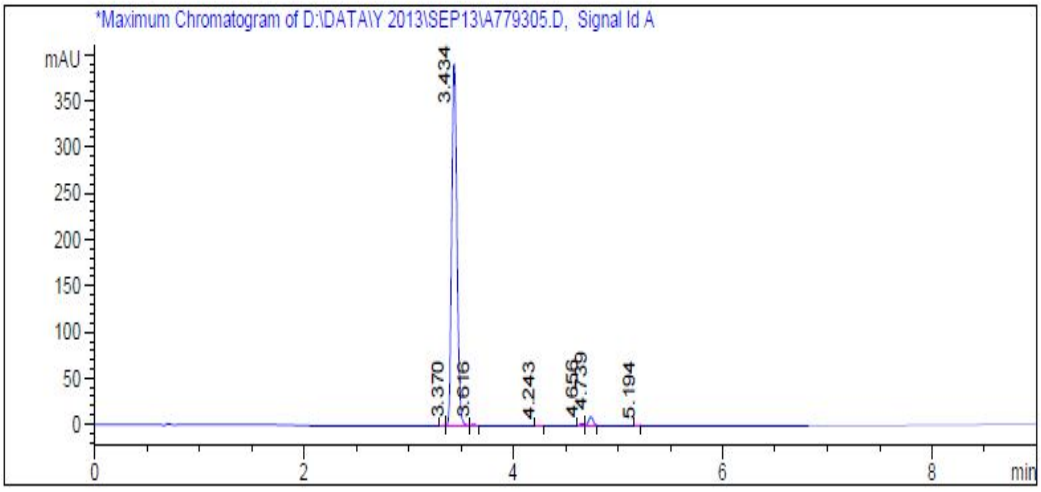
A : 10 mM NH4HCo3 H2O B: ACN Flow = 1.0 mL/min

COLUMN:XBridge C8 (50X4.6)mm,3.5µm , -ve mode

Time	% of B
0	5
8.0	100
8.1	100
8.5	5
10.0	5

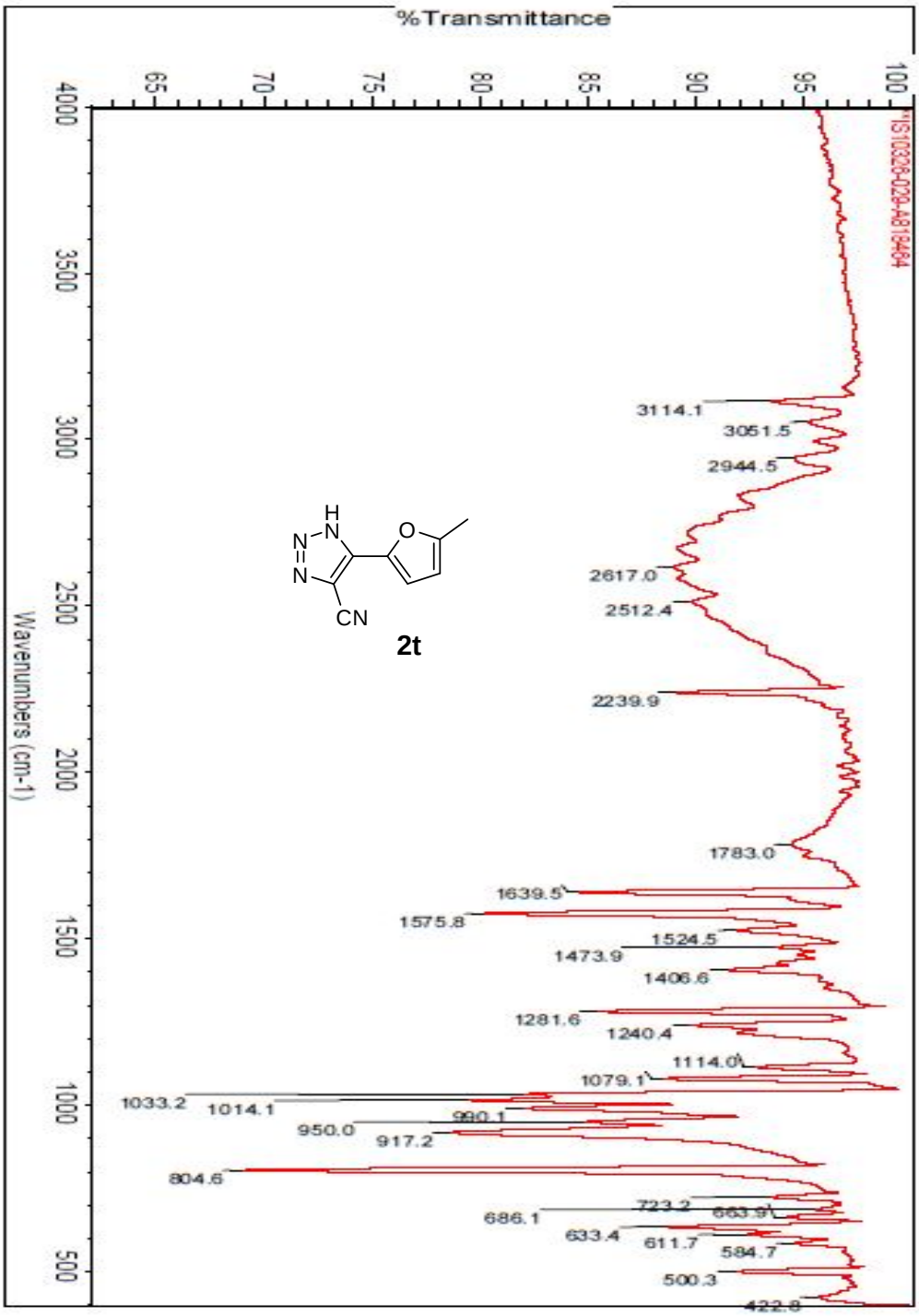


2t

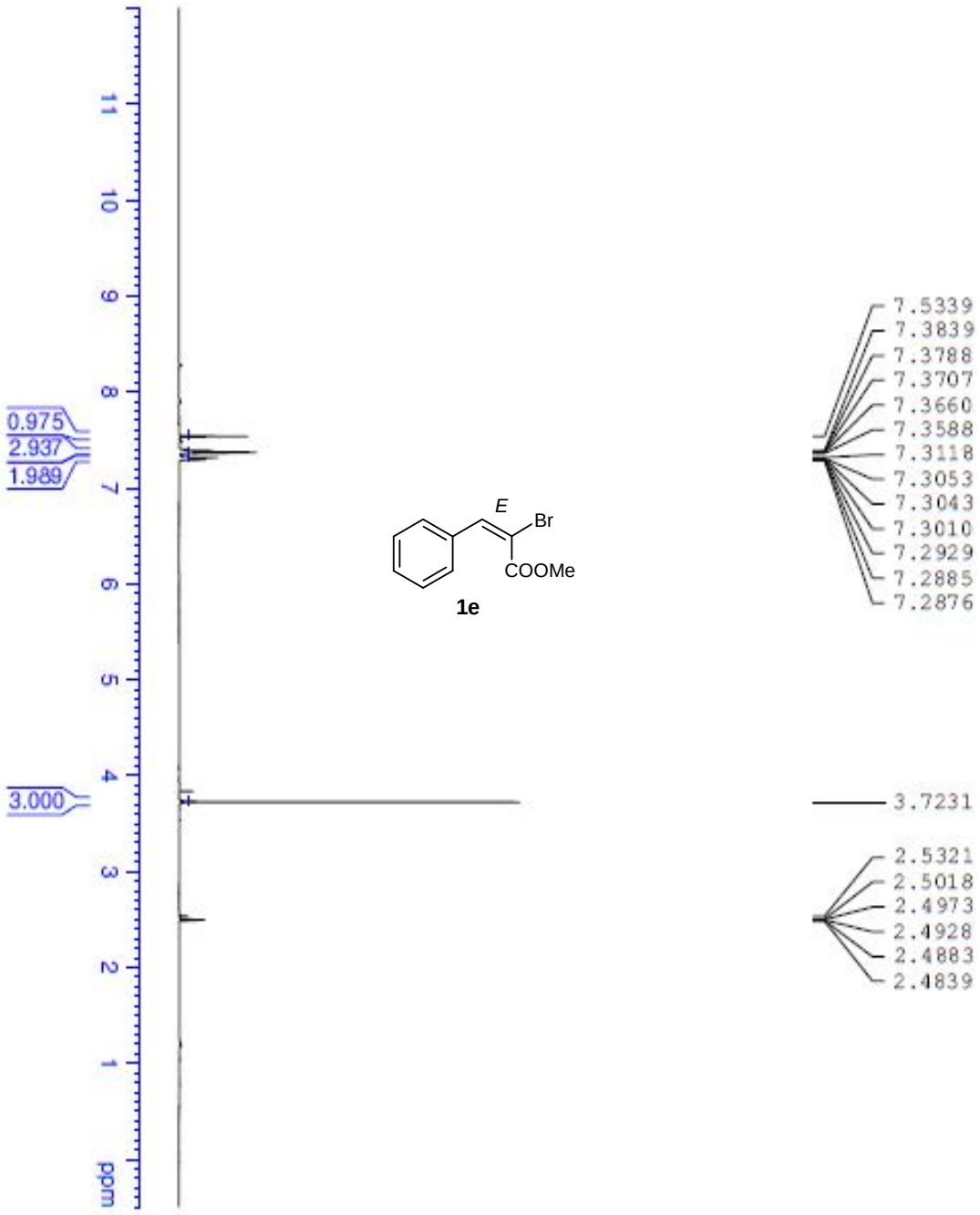


Peak No	RT min	Area	Area %
1	3.370	1.756	0.124
2	3.434	1367.868	96.670
3	3.616	5.558	0.393
4	4.243	1.150	0.081
5	4.656	5.863	0.414
6	4.739	31.318	2.213
7	5.194	1.478	0.104

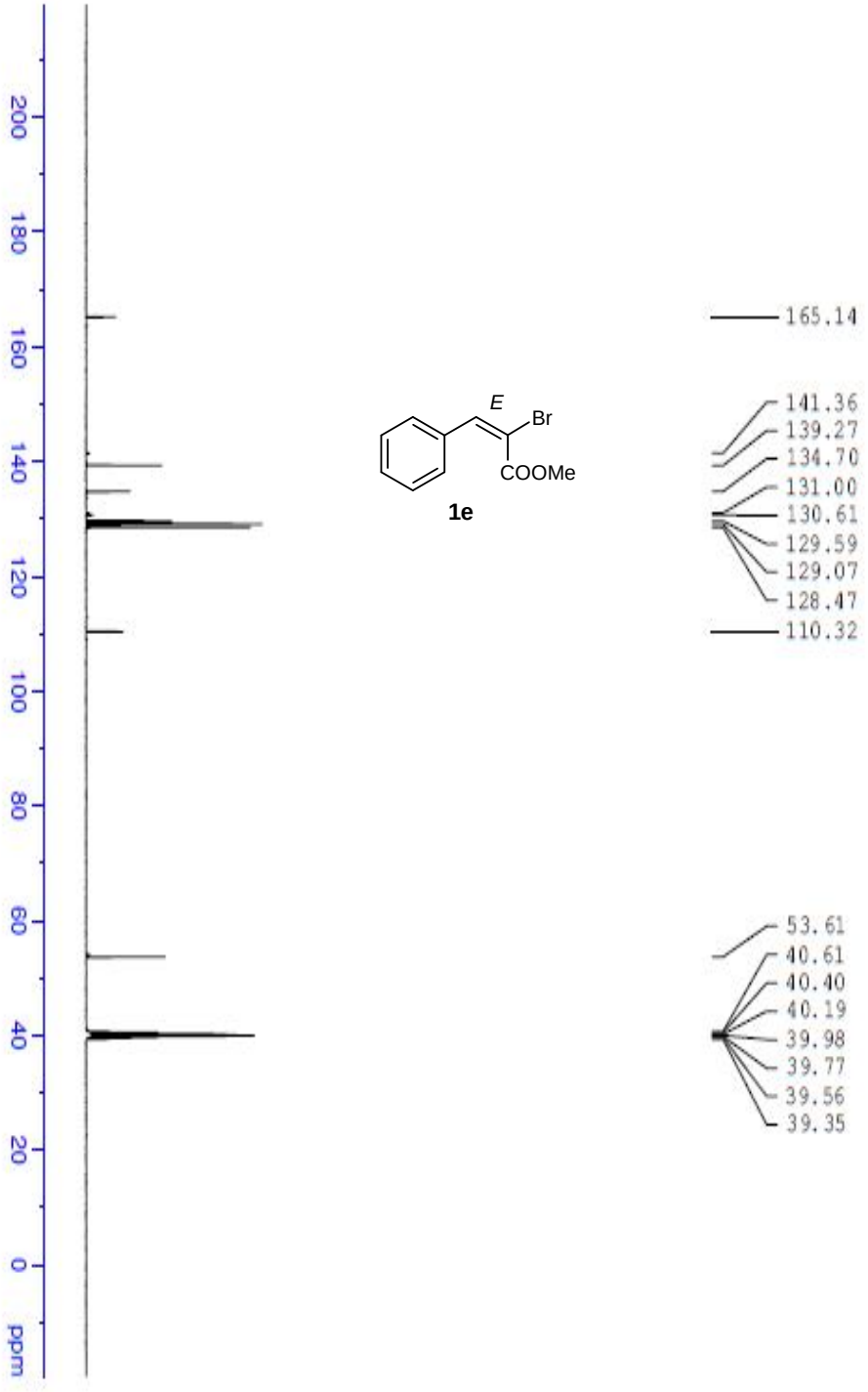
HPLC (**2t**)


%Transmittance
65
70
75
80
85
90
95
100
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)
3114.1
3051.5
2944.5
2617.0
2512.4
2239.9
1783.0
1639.5
1575.8
1524.5
1473.9
1406.6
1281.6
1240.4
1114.0
1079.1
1033.2
1014.1
990.1
950.0
917.2
804.6
723.2
686.1
663.9
633.4
611.7
584.7
500.3
422.8
H
N
N
N
O
CN
2t

IR (**2t**)



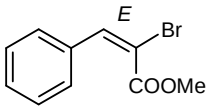
^{1}H NMR (400 MHz) in DMSO-d_6 (**1e**)



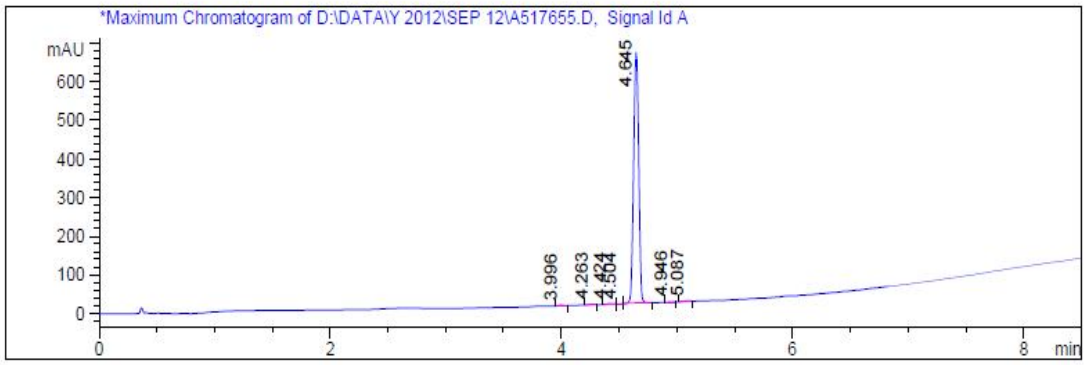
^{13}C NMR (100 MHz) in DMSO-d_6 (**1e**)

Method info : A : 0.1%TFA IN H2O B: 0.1%TFA IN ACN Flow = 2.0 mL/min
COLUMN:XBridge C8 (50X4.6)mm,3.5µm , +ve mode

Time	% of B
0	5
8.0	100
8.1	100
8.5	5
10.0	5

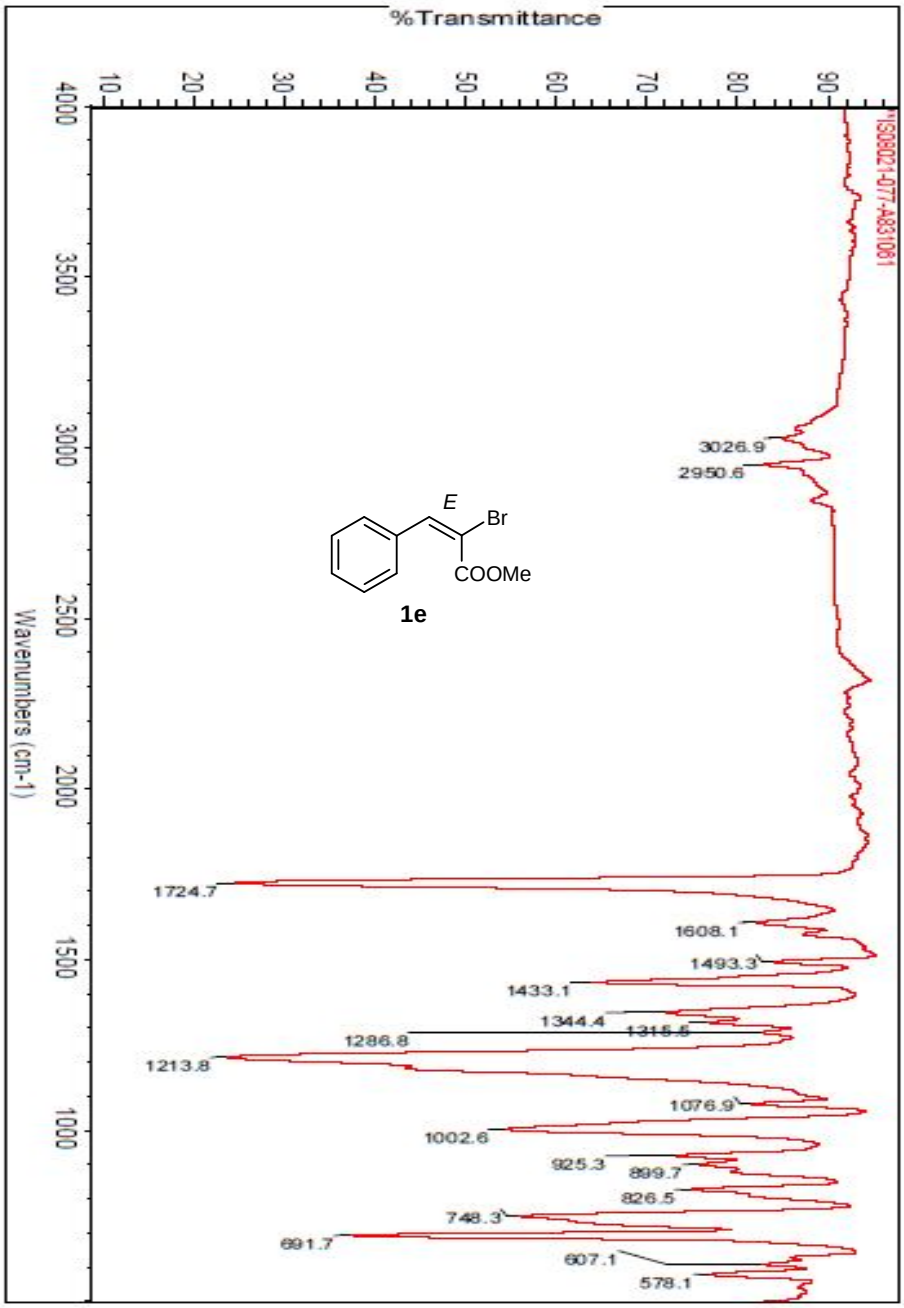


1e

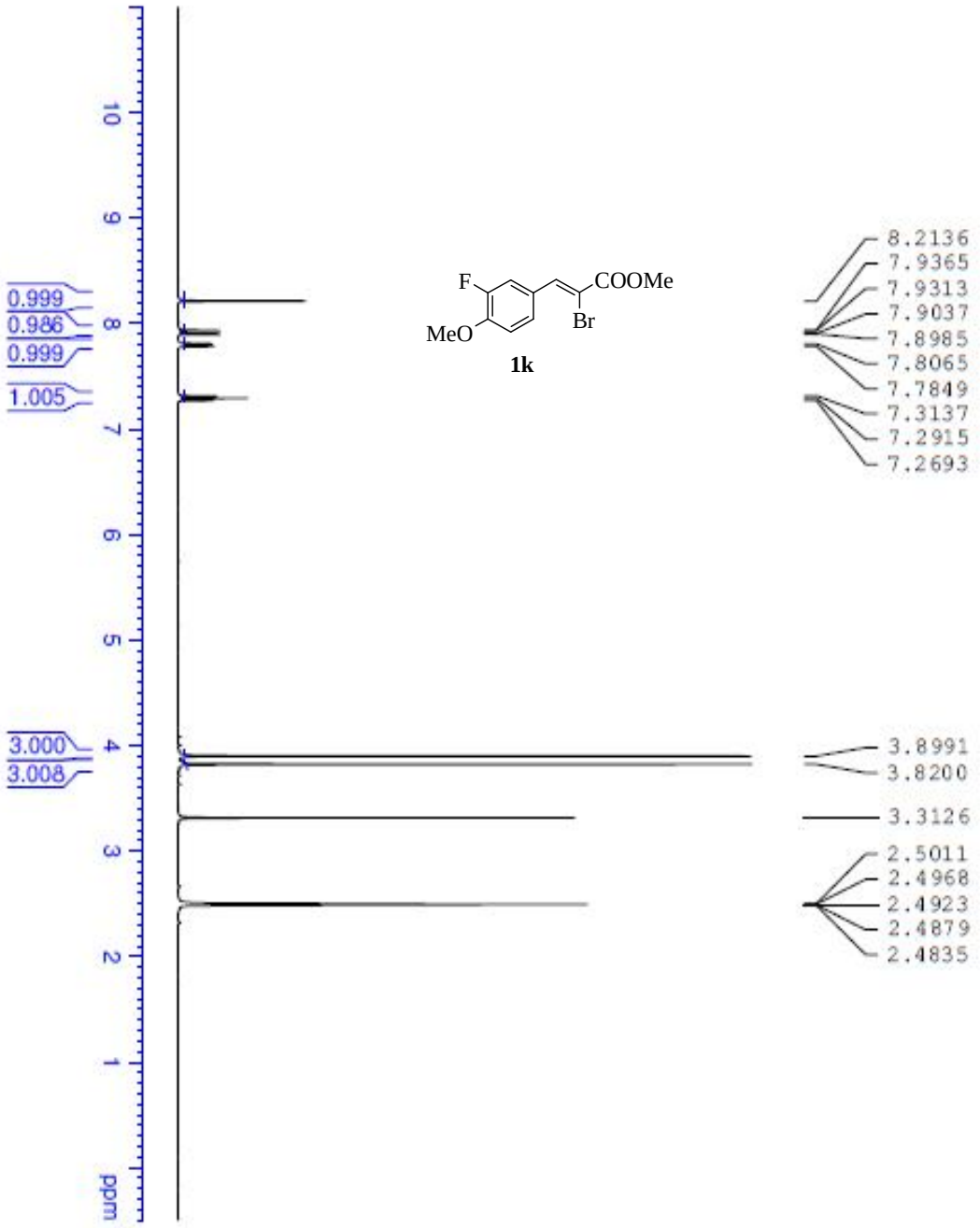


Peak No	RT min	Area	Area %
1	3.996	3.698e+000	0.186
2	4.263	5.120e+000	0.257
3	4.424	5.185e+000	0.261
4	4.504	1.187e+000	0.060
5	4.645	1.967e+003	98.820
6	4.946	2.314e+000	0.116
7	5.087	5.975e+000	0.300

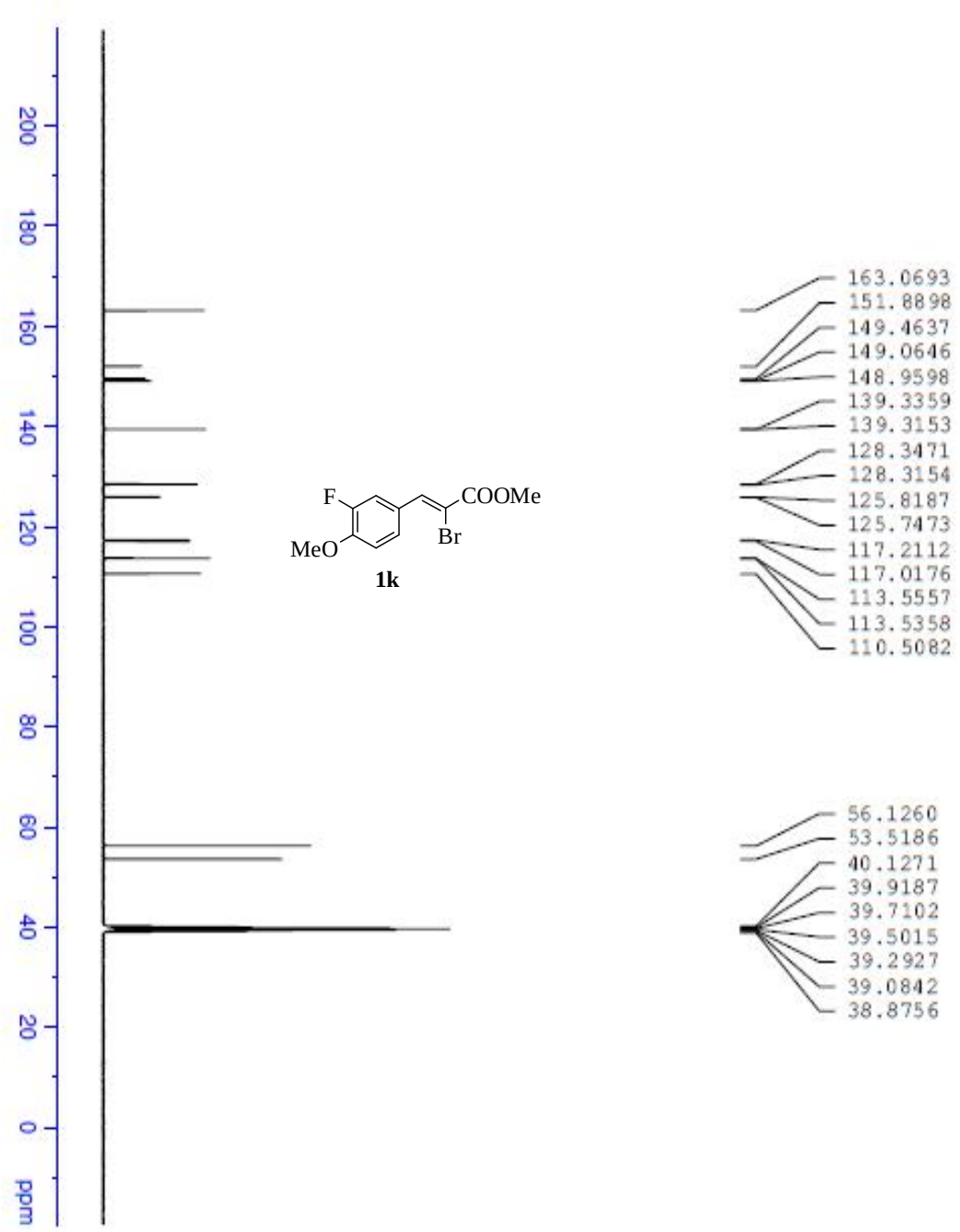
HPLC (**1e**)


%Transmittance
Wavenumbers (cm-1)
IS09021-077-A831061
3026.9
2950.6
1724.7
1608.1
1493.3
1433.1
1344.4
1315.5
1286.8
1213.8
1076.9
1002.6
925.3
899.7
826.5
748.3
691.7
607.1
578.1
E
Br
COOMe
1e

IR (**1e**)



^{1}H NMR (400 MHz) in DMSO-d_6 (**1k**)

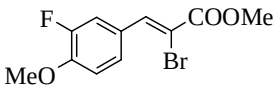


^{13}C NMR (100 MHz) in DMSO-d_6 (**1k**)

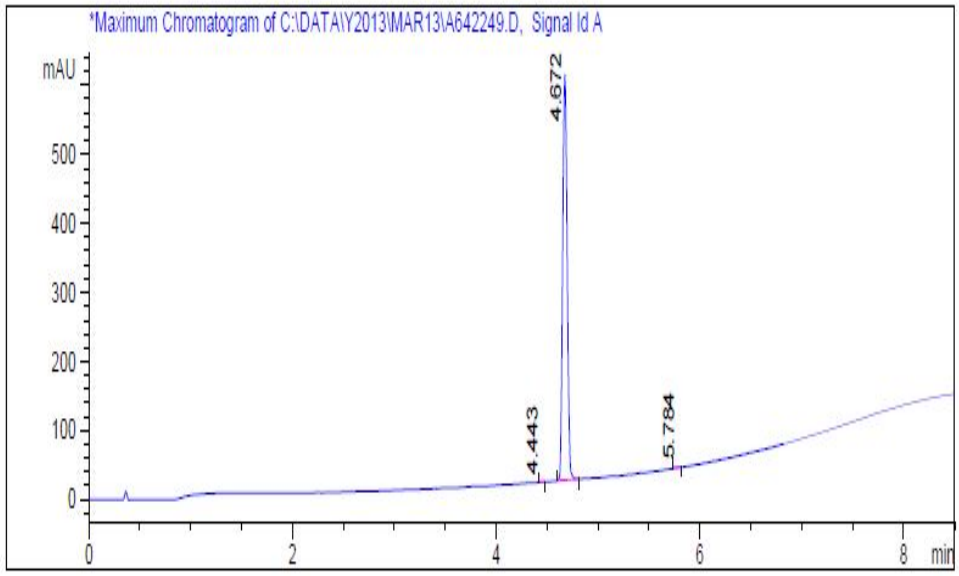
Method info : A:0.1%TFA in H2O, B:0.1%TFA in ACN, Flow Rate:2.0ml/min

COLUMN: XBridge C8(50X4.6)mm,3.5µm

Time	% of B
0	5
8.0	100
8.1	100
8.5	5
10.0	5

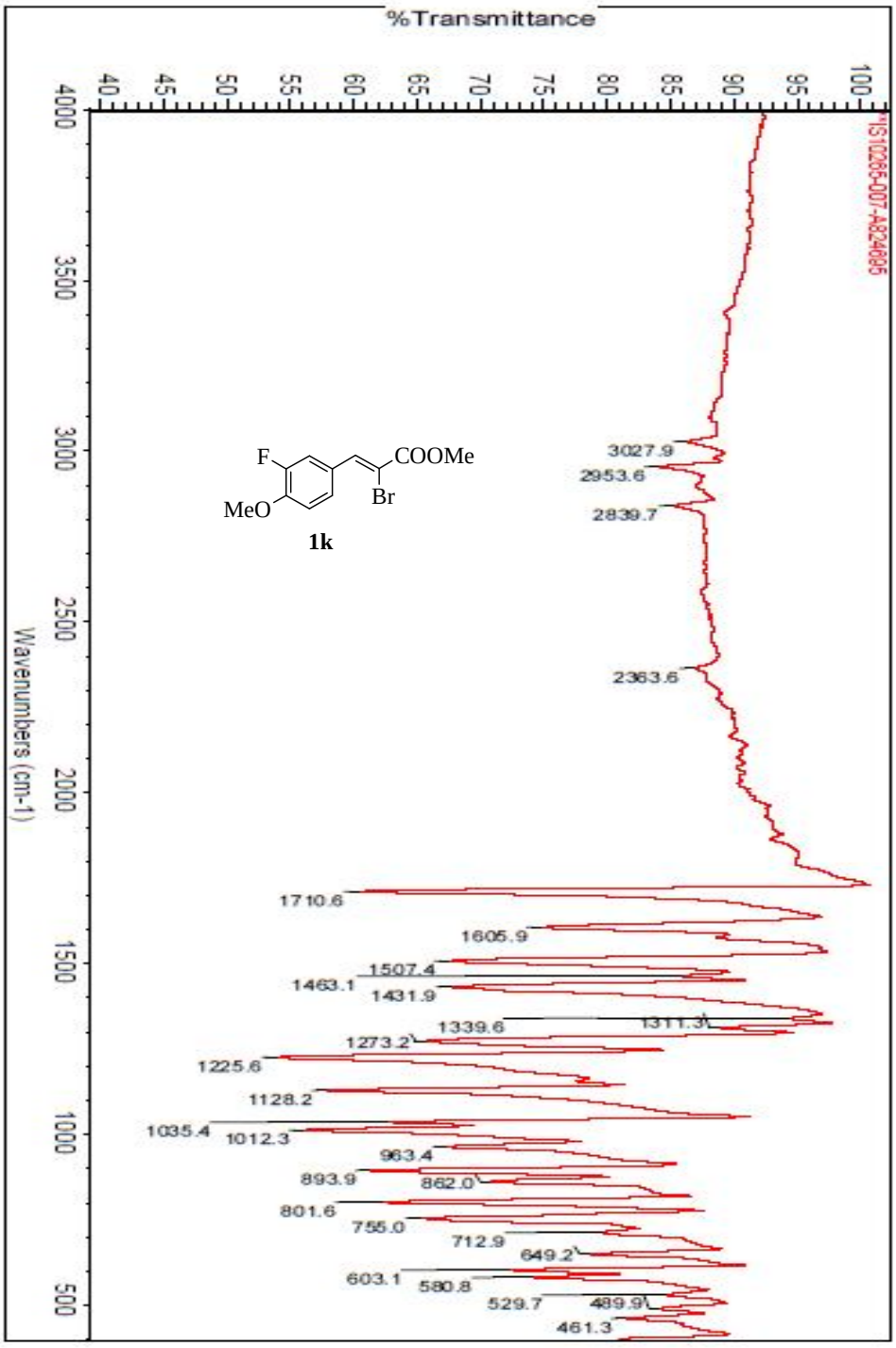


1k

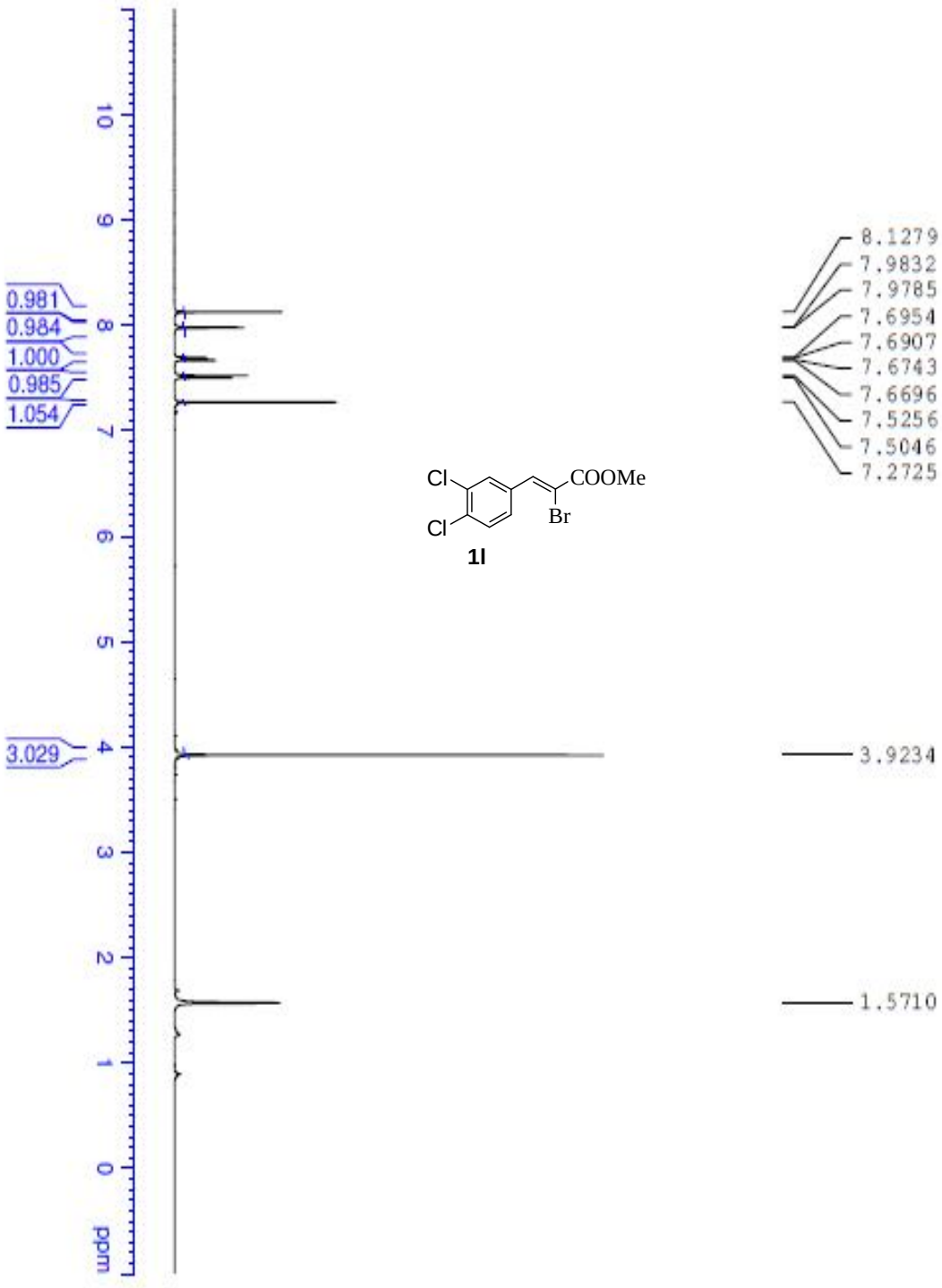


Peak No	RT min	Area	Area %
1	4.443	1.661e+000	0.09
2	4.672	1.791e+003	99.71
3	5.784	3.478e+000	0.19

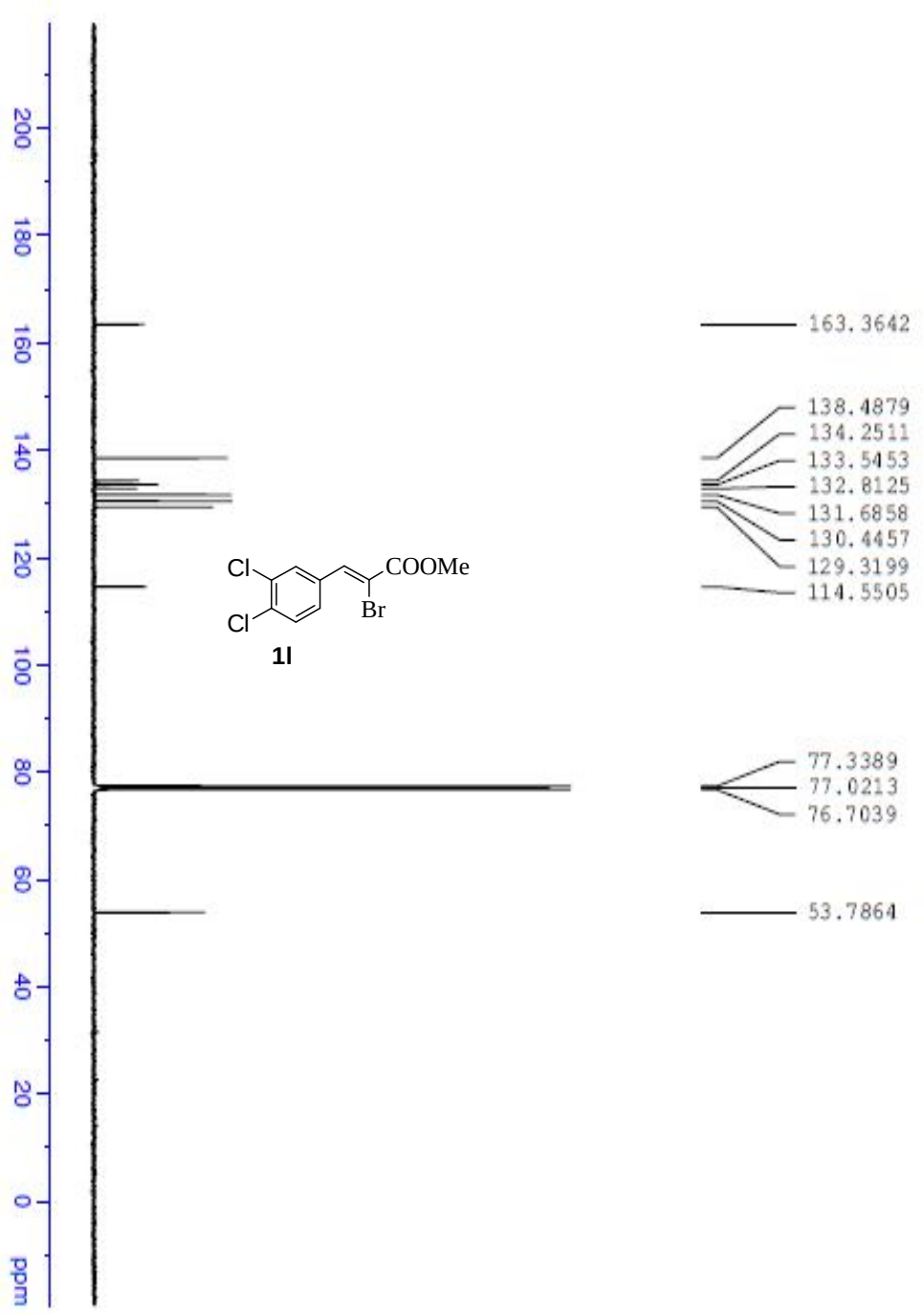
HPLC (**1k**)


%Transmittance
Wavenumbers (cm-1)
*IS10266-007-A824685
F
COOMe
MeO
Br
1k
3027.9
2953.6
2839.7
2363.6
1710.6
1605.9
1507.4
1463.1
1431.9
1339.6
1311.3
1273.2
1225.6
1128.2
1035.4
1012.3
963.4
893.9
862.0
801.6
755.0
712.9
649.2
603.1
580.8
529.7
489.9
461.3

IR (**1k**)



^{1}H NMR (400 MHz) in $CDCl_3$ (**1l**)

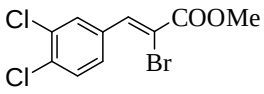


^{13}C NMR (100 MHz) in $CDCl_3$ (**1l**)

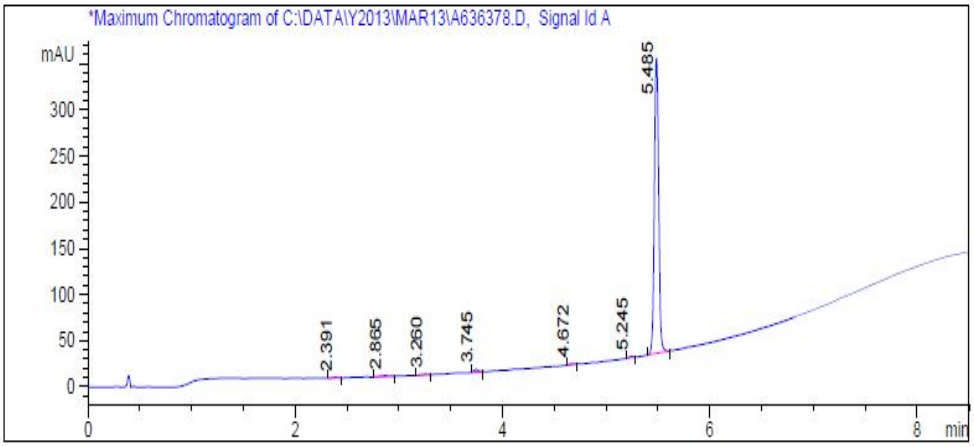
Method info : A:0.1%TFA in H2O, B:0.1%TFA in ACN, Flow Rate:2.0ml/min

COLUMN: XBridge C8(50X4.6)mm,3.5µm

Time	% of B
0	5
8.0	100
8.1	100
8.5	5
10.0	5

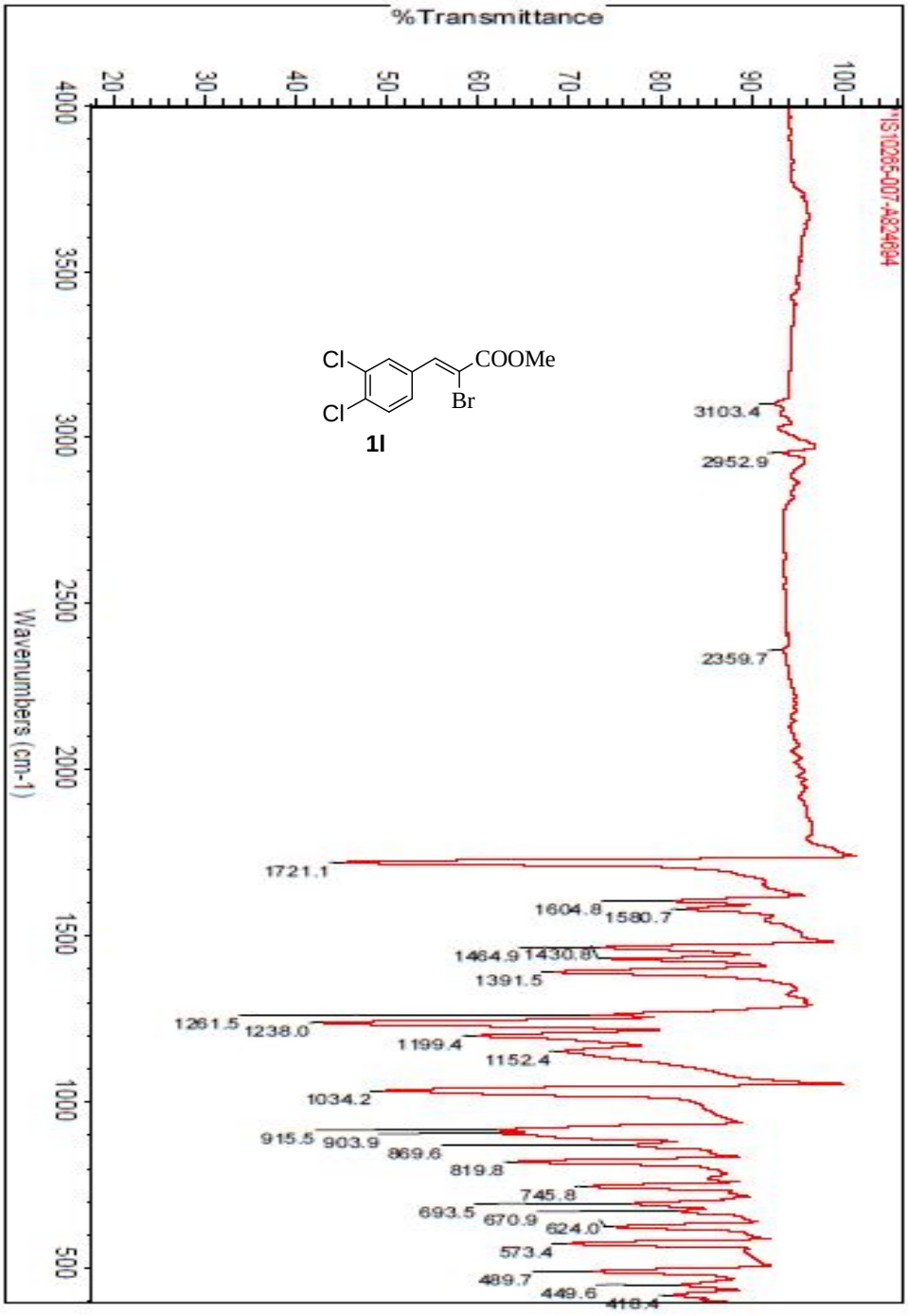


1l

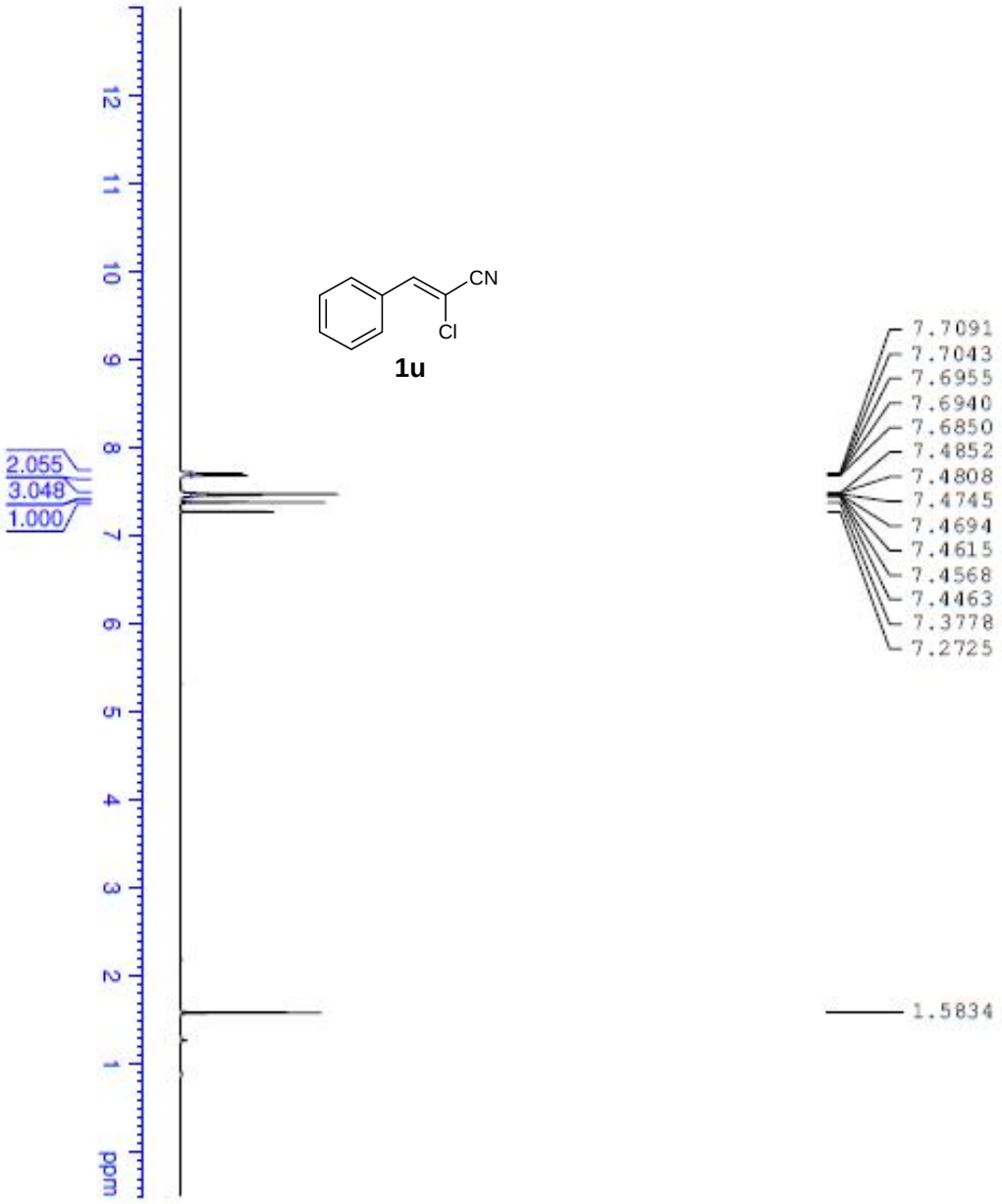


Peak No	RT min	Area	Area %
1	2.391	3.954e+000	0.40
2	2.865	7.417e+000	0.76
3	3.260	3.990e+000	0.41
4	3.745	8.718e+000	0.89
5	4.672	3.873e+000	0.40
6	5.245	4.446e+000	0.45
7	5.485	9.456e+002	96.69

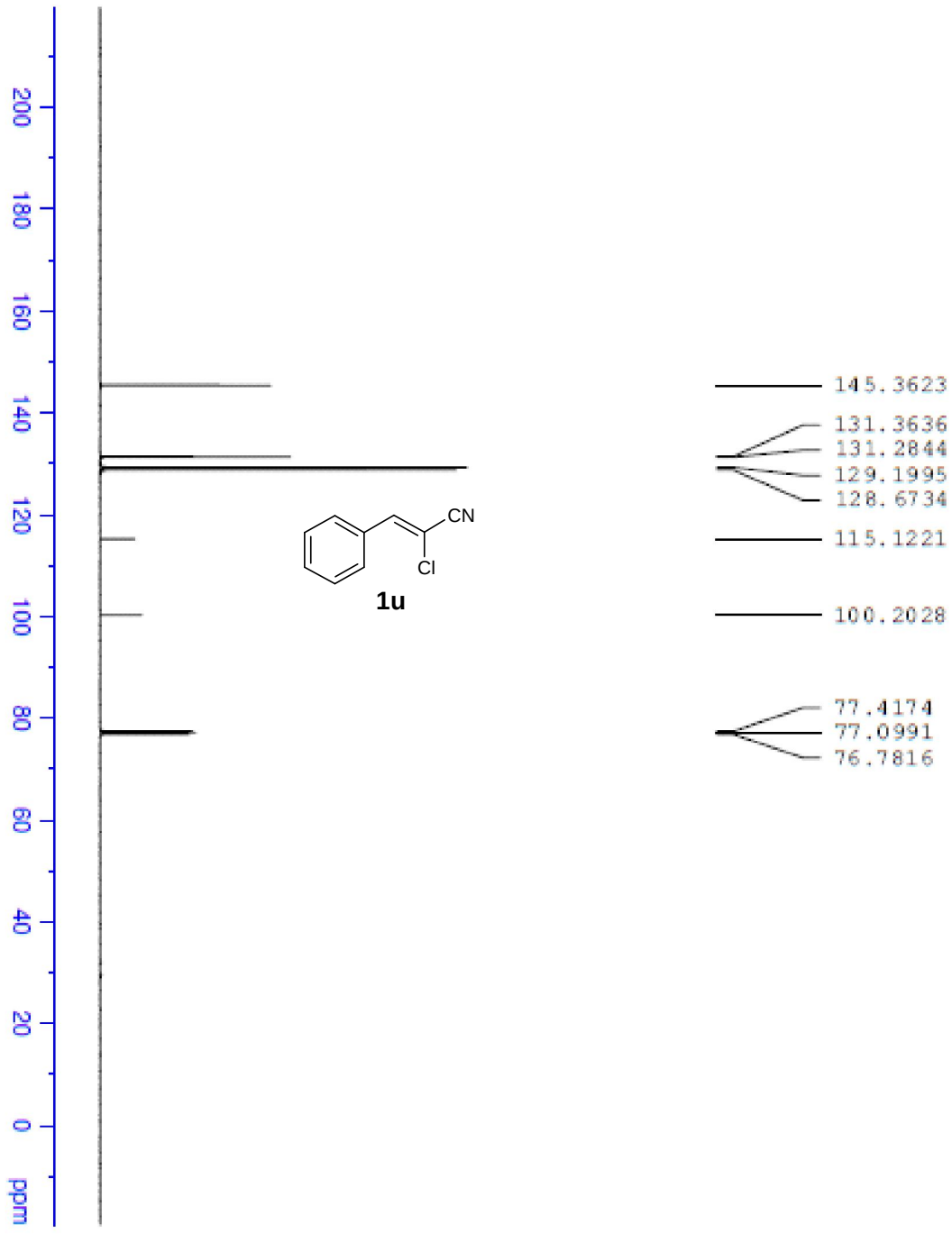
HPLC (**1l**)


%Transmittance
Wavenumbers (cm-1)
Cl
Cl
COOMe
Br
1l
3103.4
2952.9
2359.7
1721.1
1604.8
1580.7
1464.9
1430.8
1391.5
1261.5
1238.0
1199.4
1152.4
1034.2
915.5
903.9
869.6
819.8
745.8
693.5
670.9
624.0
573.4
489.7
449.6
416.4

IR (**1l**)



^{1}H NMR (400 MHz) in $CDCl_3$ (**1u**)

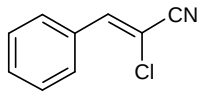


^{13}C NMR (100 MHz) in $CDCl_3$ (**1u**)

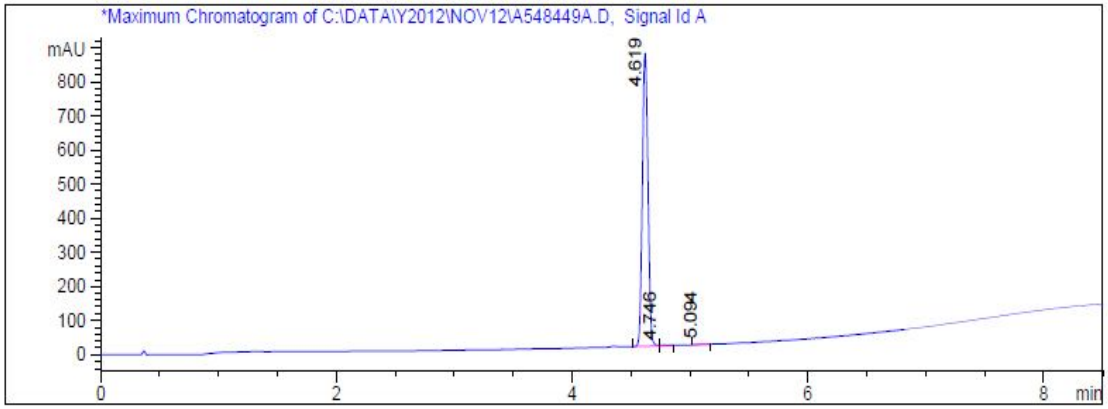
Method info : A:0.1%TFA in H2O, B:0.1%TFA in ACN, Flow Rate:2.0ml/min

COLUMN: XBridge C8(50X4.6)mm,3.5µm

Time	% of B
0	5
8.0	100
8.1	100
8.5	5
10.0	5

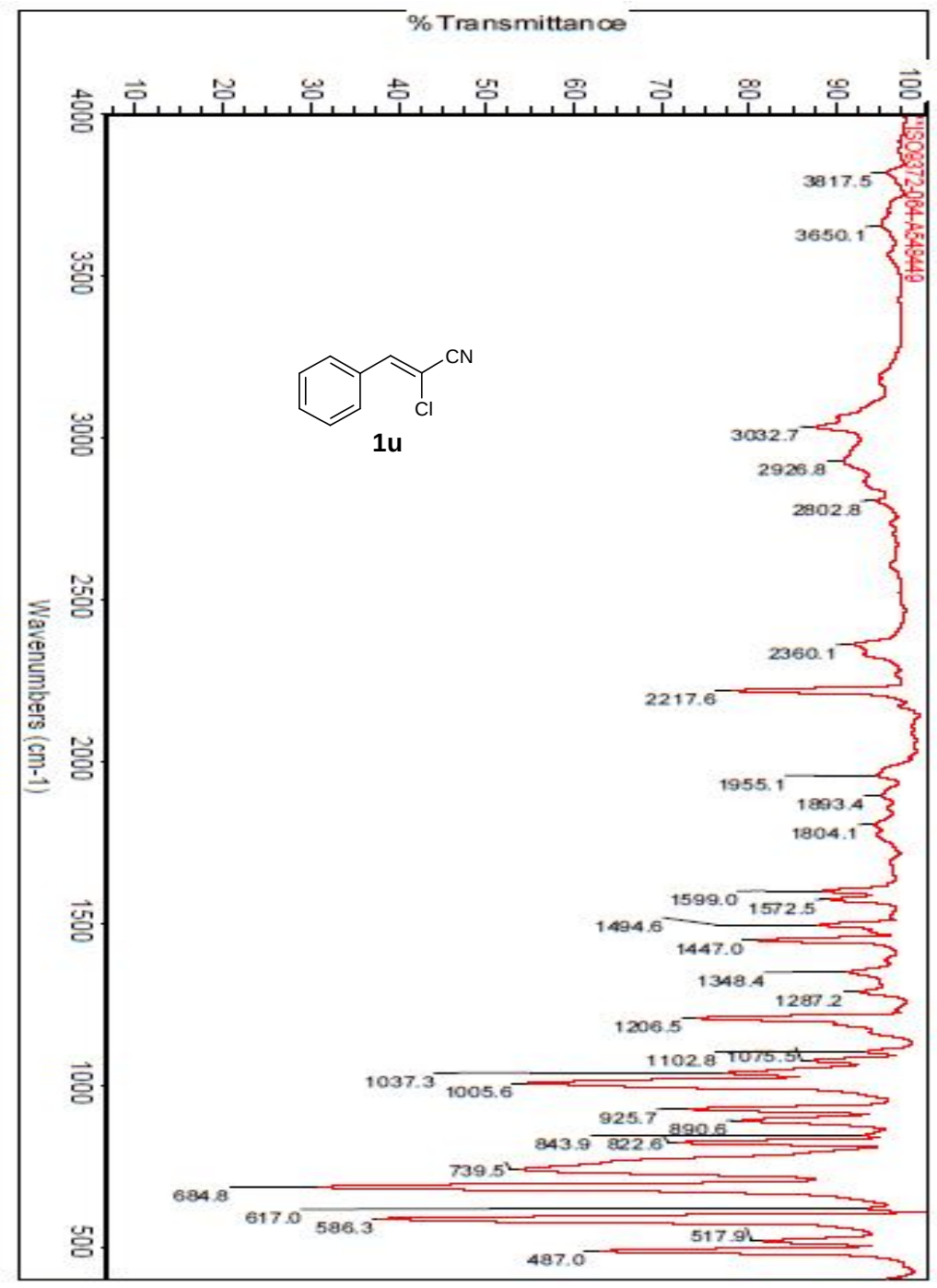


1u

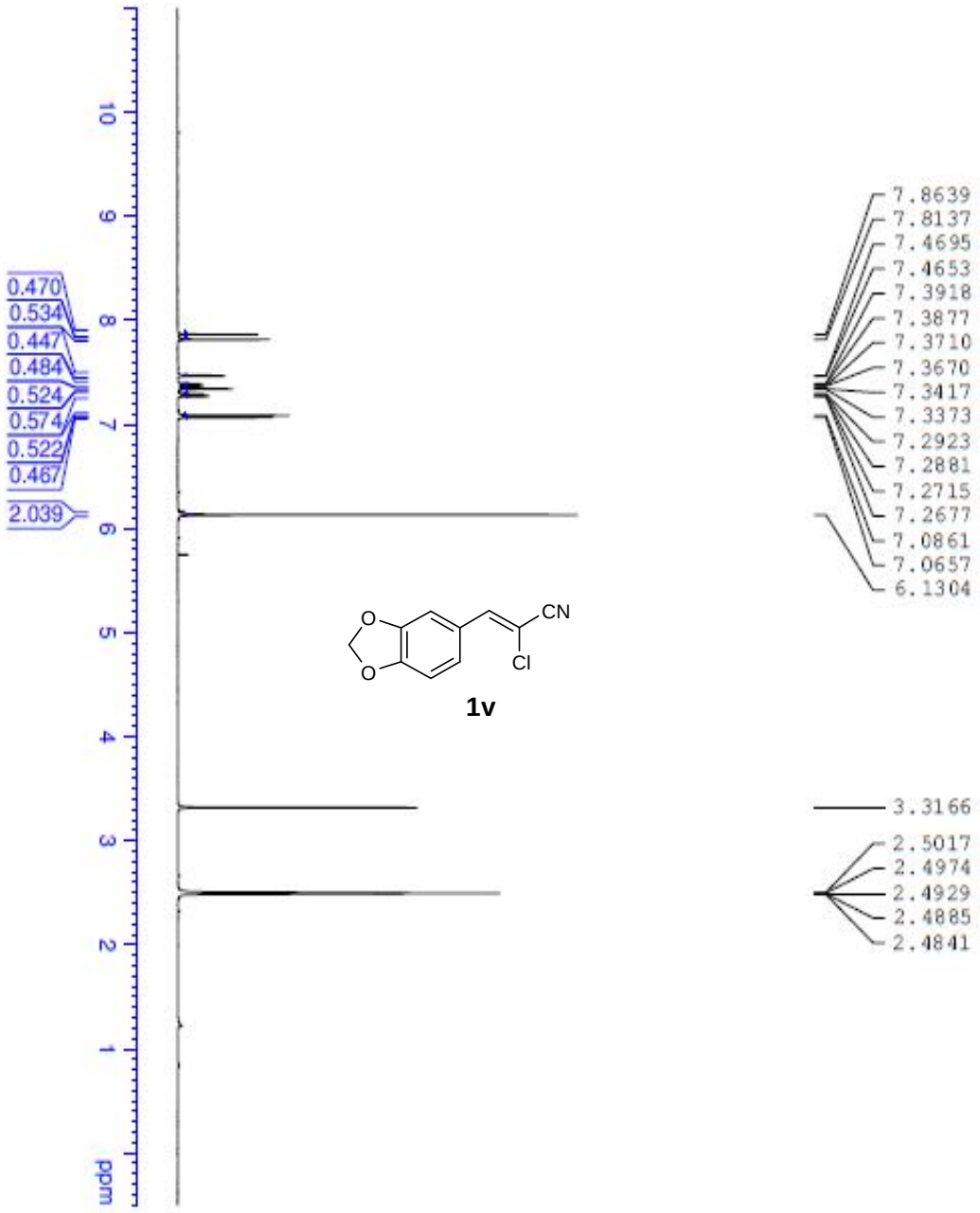


Peak No	RT min	Area	Area %
1	4.619	2.922e+003	99.73
2	4.746	4.383e+000	0.15
3	5.094	3.465e+000	0.12

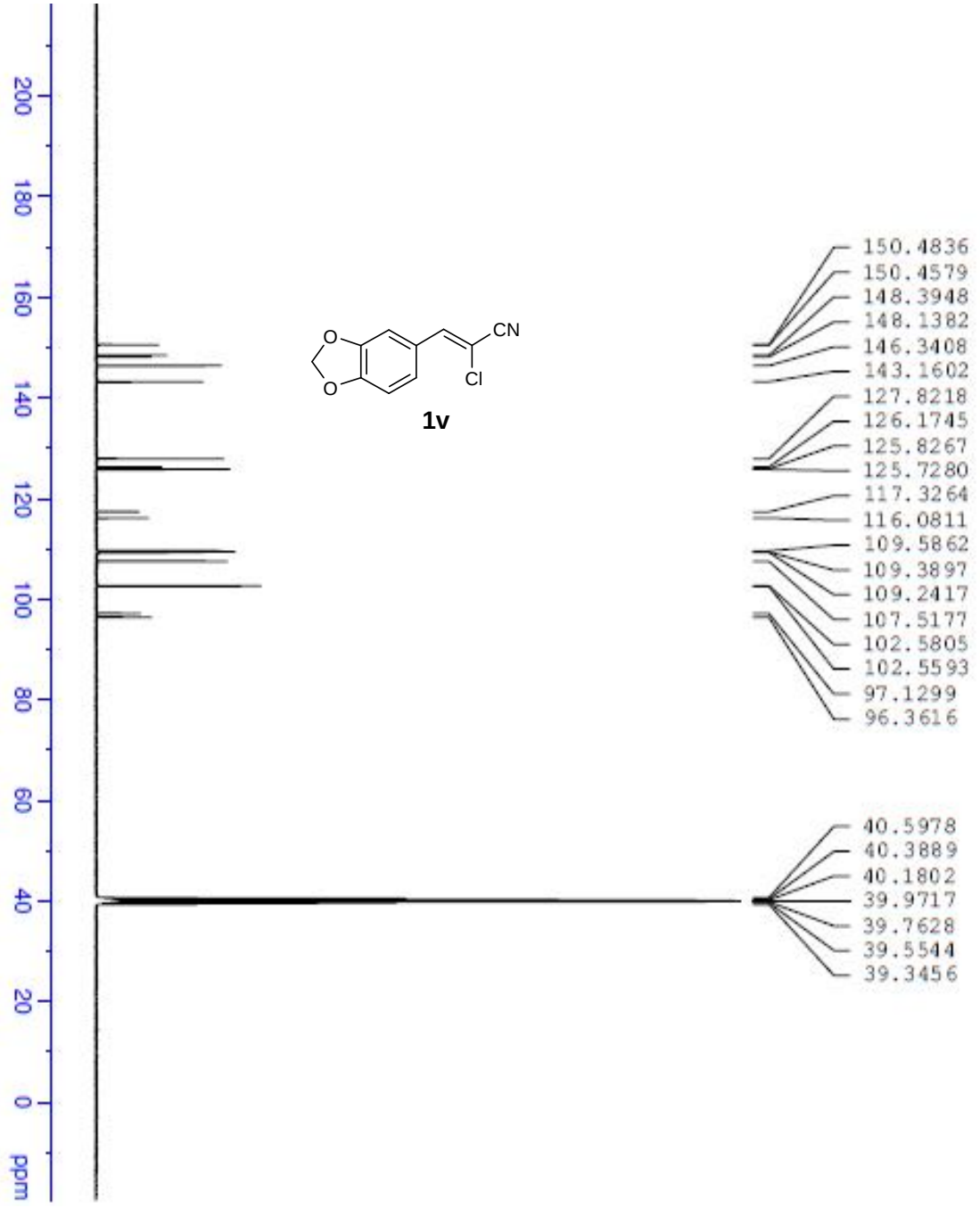
HPLC (**1u**)


% Transmittance
Wavenumbers (cm-1)
CN
Cl
1u
3817.5
3650.1
3032.7
2926.8
2802.8
2360.1
2217.6
1955.1
1893.4
1804.1
1599.0
1572.5
1494.6
1447.0
1348.4
1287.2
1206.5
1102.8
1075.5
1037.3
1005.6
925.7
890.6
843.9
822.6
739.5
684.8
617.0
586.3
517.9
487.0

IR (**1u**)



^{1}H NMR (400 MHz) in DMSO-d_6 (**1v**)



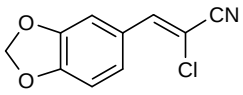
^{13}C NMR (100 MHz) in DMSO-d_6 (**1v**)

Method info :

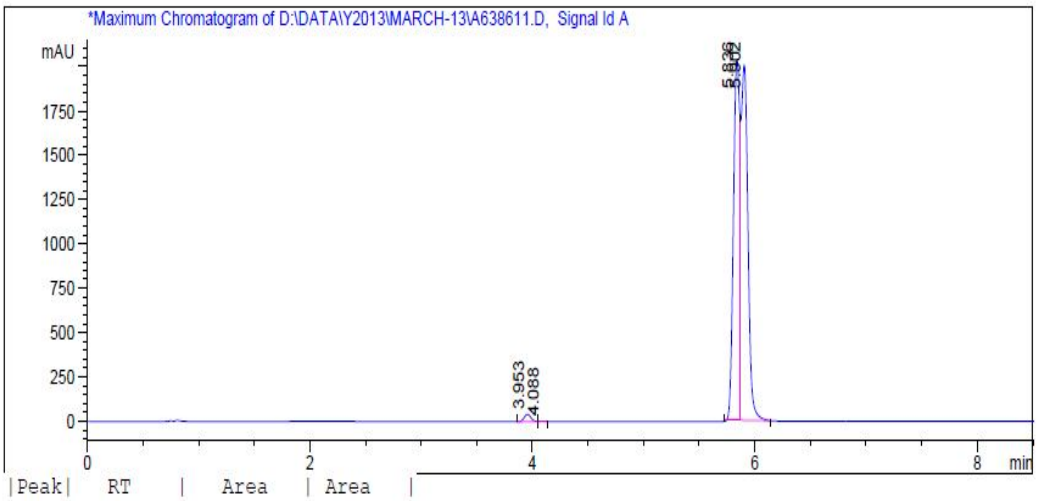
A-10mM NH4HCO3 IN H2O , B- ACN Flow: 1.0 ml/min

COLUMN: XBridge C8 (50X4.6mm, 3.5µm),

TIME	%B
0	05
8.0	100
8.1	100
8.5	05
10	05

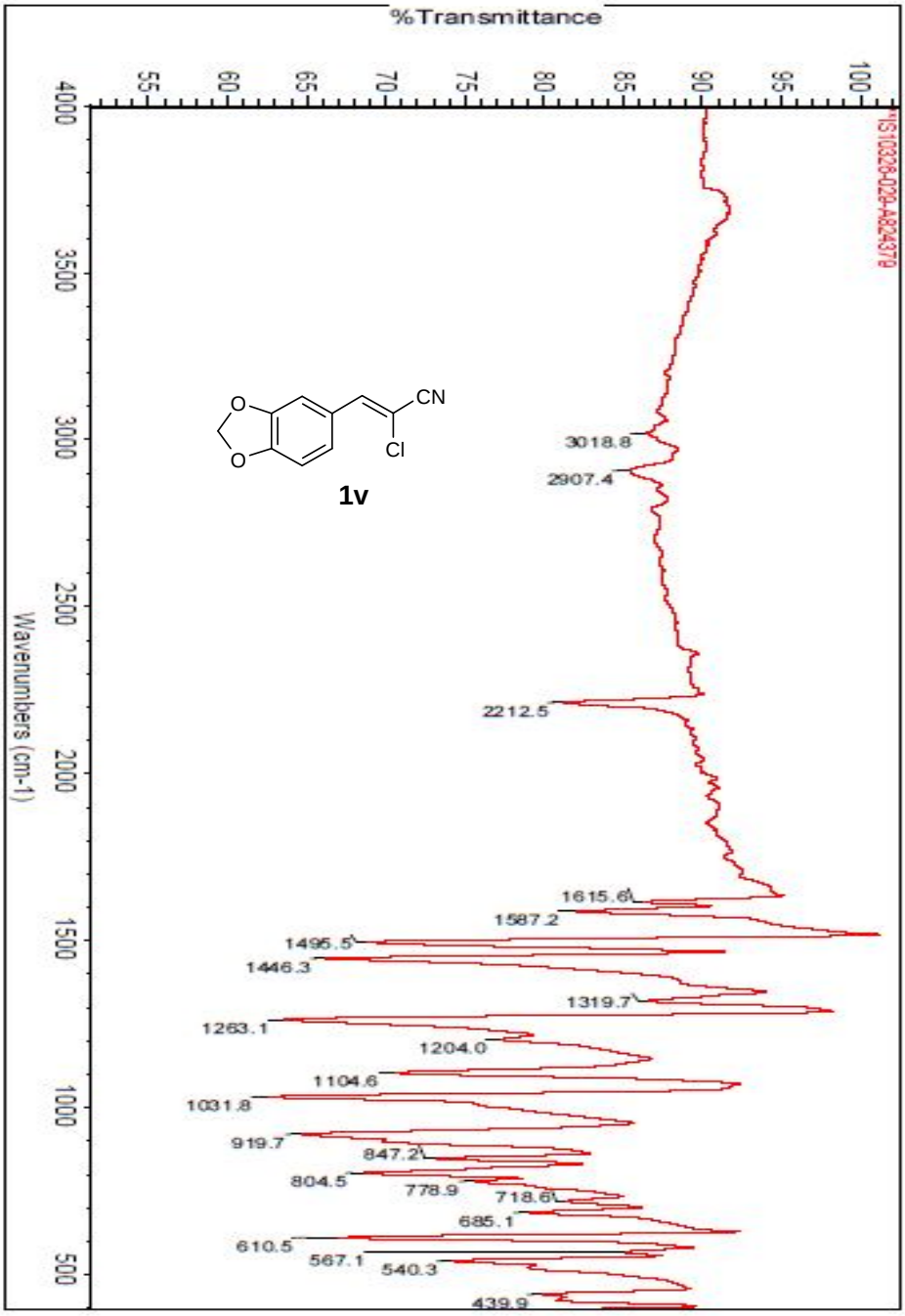


1v

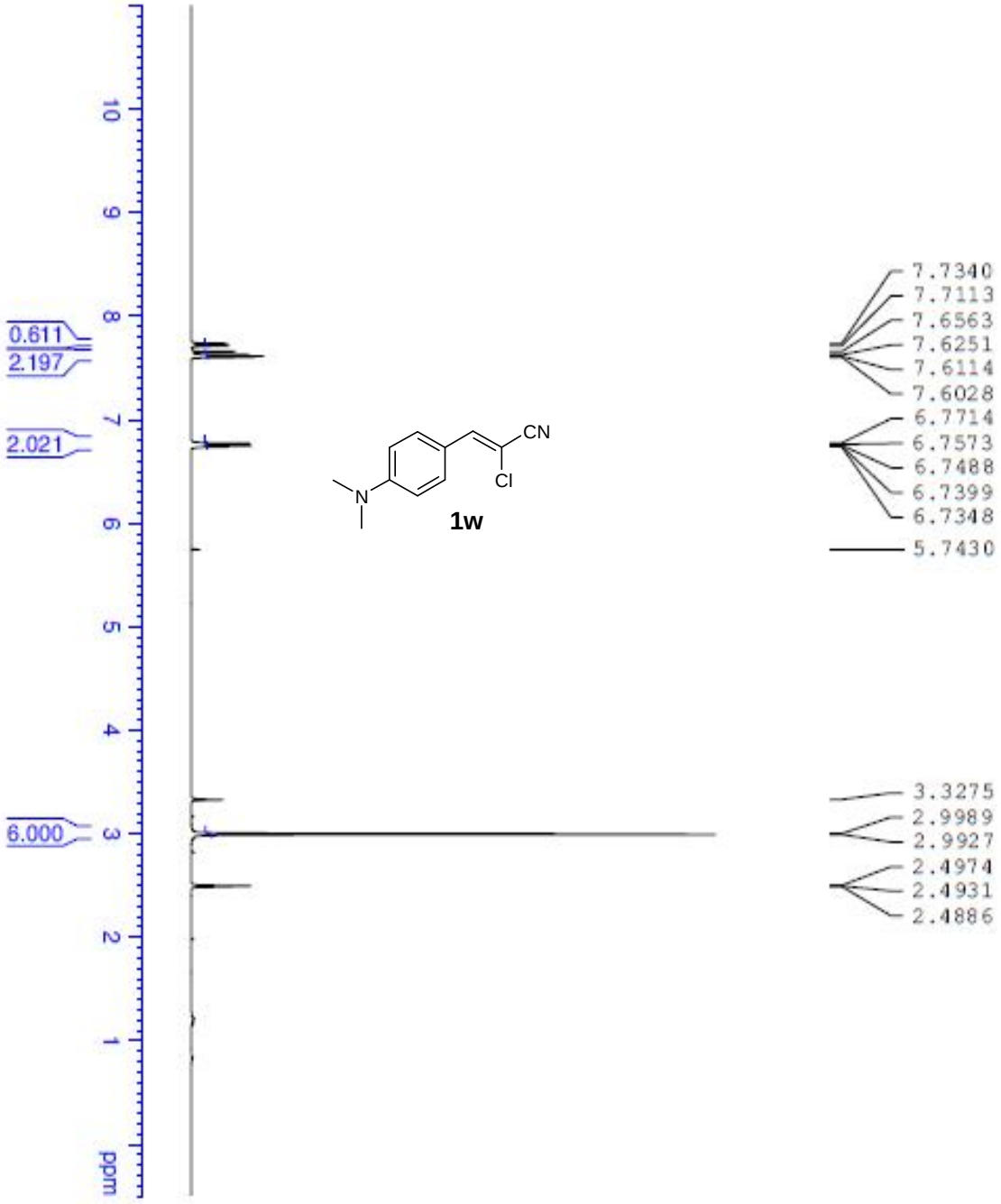


Peak No	RT min	Area	Area %
1	3.953	1.919e+002	1.144
2	4.088	1.194e+001	0.071
3	5.836	8.192e+003	48.817
4	5.902	8.385e+003	49.968

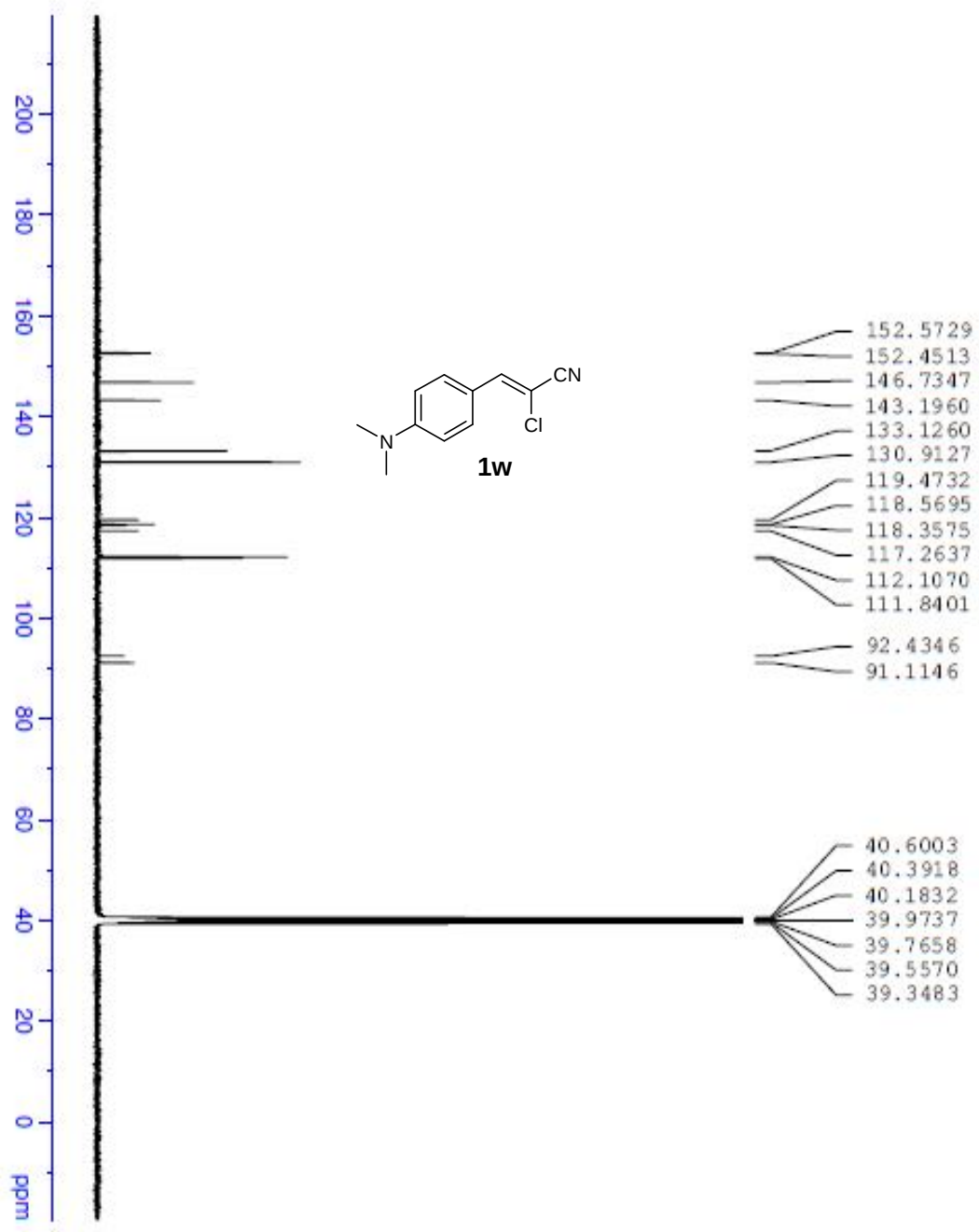
HPLC (**1v**)


%Transmittance
Wavenumbers (cm-1)
IS10326-029-AB24379
O
O
CN
Cl
1v
3018.8
2907.4
2212.5
1615.6
1587.2
1496.5
1446.3
1319.7
1263.1
1204.0
1104.6
1031.8
919.7
847.2
804.5
778.9
718.6
685.1
610.5
567.1
540.3
439.9

IR (**1v**)



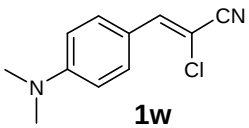
^{1}H NMR (400 MHz) in DMSO-d_6 (**1w**)



^{13}C NMR (100 MHz) in DMSO-d_6 (**1w**)

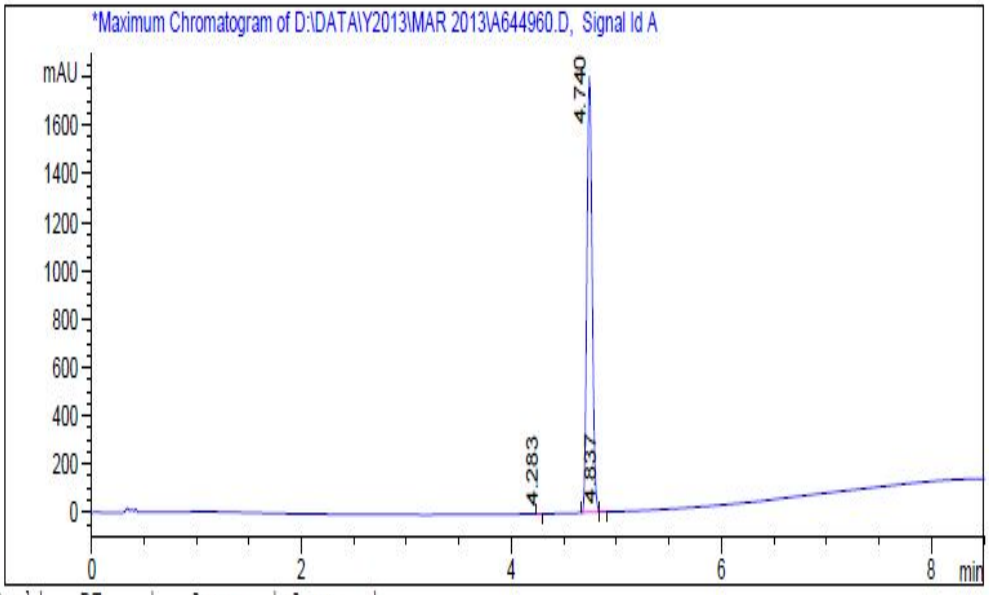
Method info : A: 0.1% TFA IN H2O , B:0.1% TFA IN ACN ; Flow Rate:2.0 ml/min
COLUMN:XBridge C8 (50x4.6mm, 3.5µm), +ve mode

TIME	%B
0	05
8.0	100
8.1	100
8.5	05
10	05



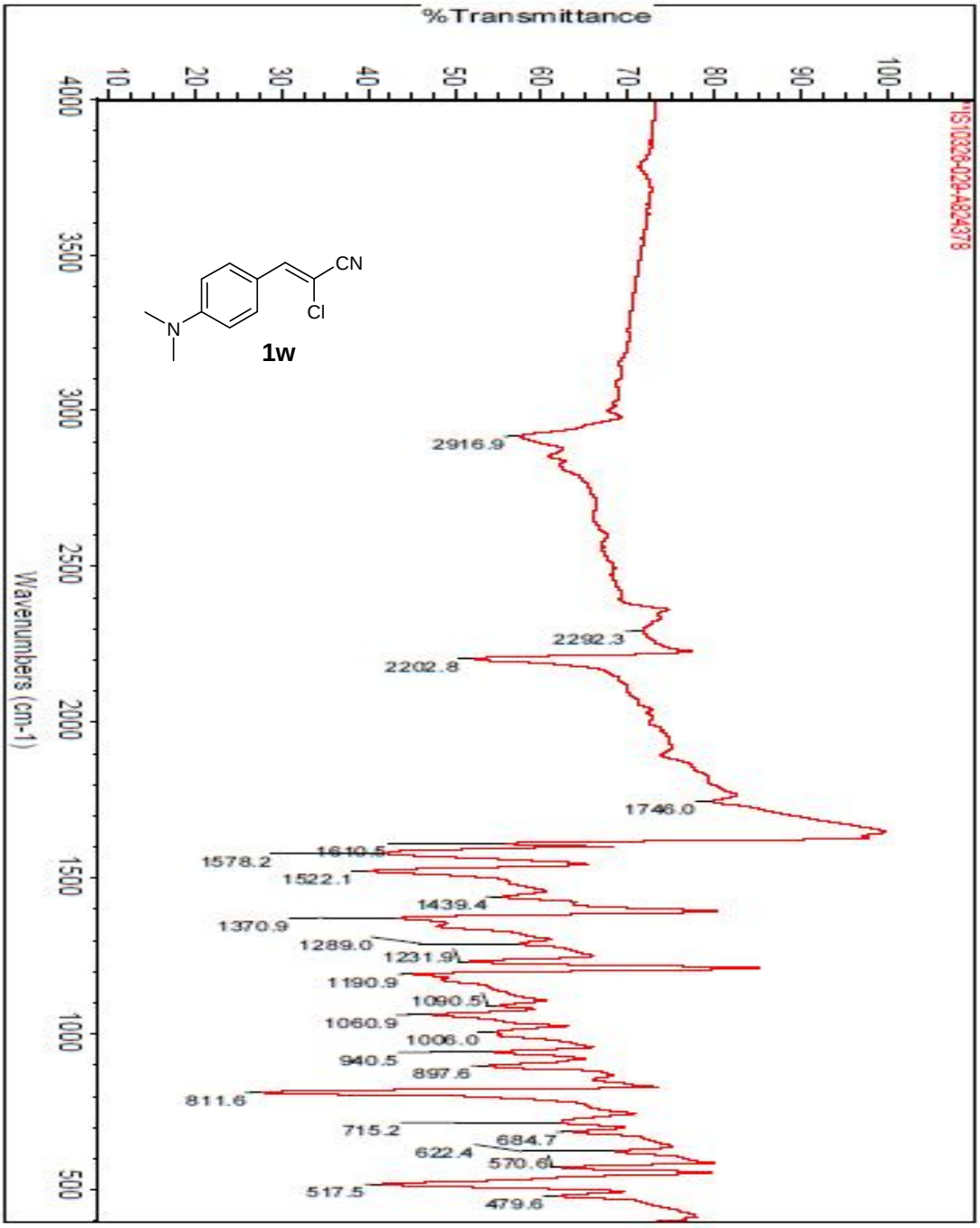
1w

->



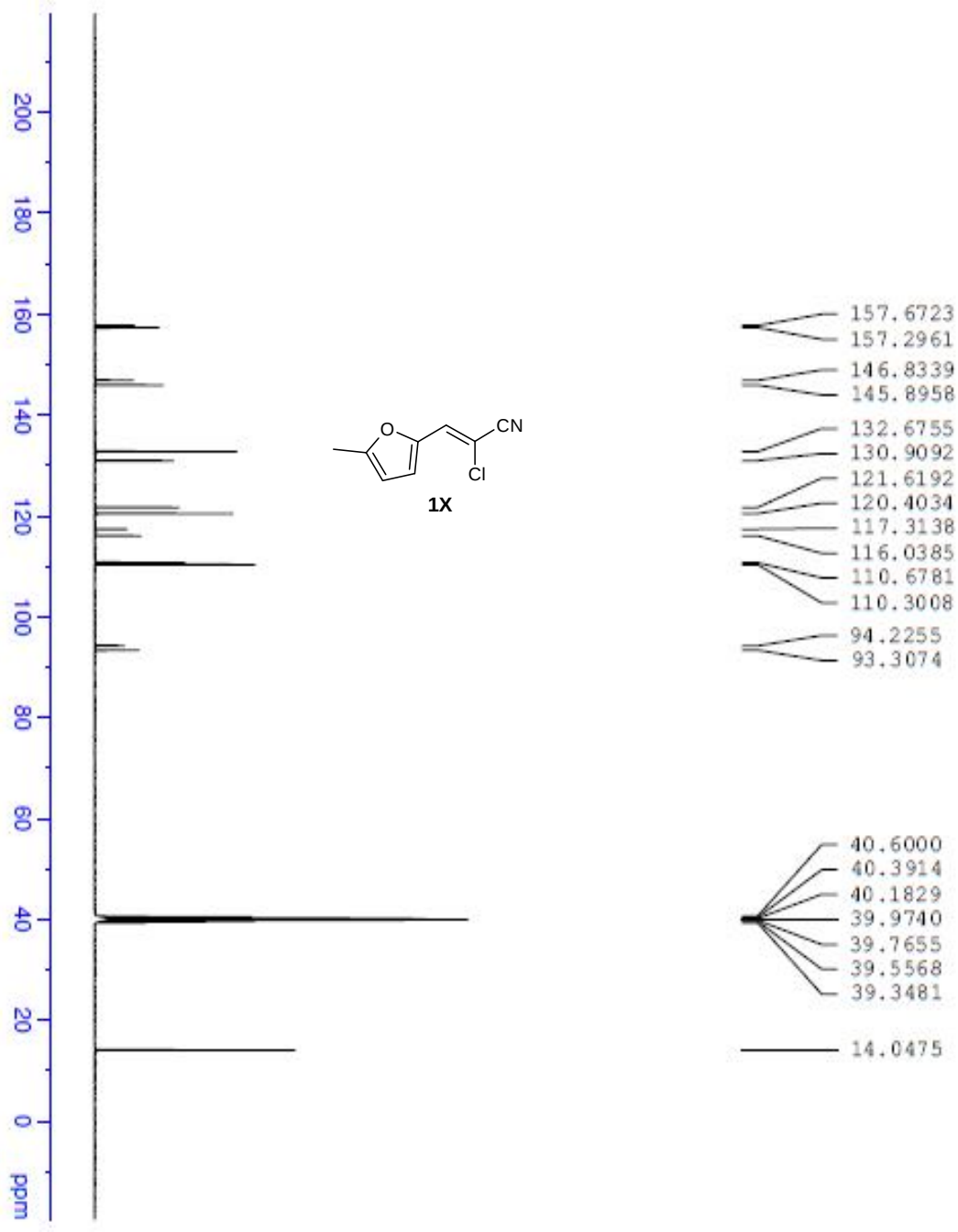
Peak No	RT min	Area	Area %
1	4.283	2.539e+000	0.040
2	4.740	6.281e+003	99.886
3	4.837	4.603e+000	0.073

HPLC (**1w**)


%Transmittance
Wavenumbers (cm-1)
CN
Cl
N
1w
2916.9
2292.3
2202.8
1746.0
1610.5
1578.2
1522.1
1439.4
1370.9
1289.0
1231.9
1190.9
1090.5
1060.9
1006.0
940.5
897.6
811.6
715.2
684.7
622.4
570.6
517.5
479.6

IR (**1w**)

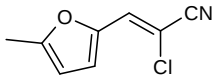
^{1}H NMR (400 MHz) in DMSO-d_6 (**1x**)



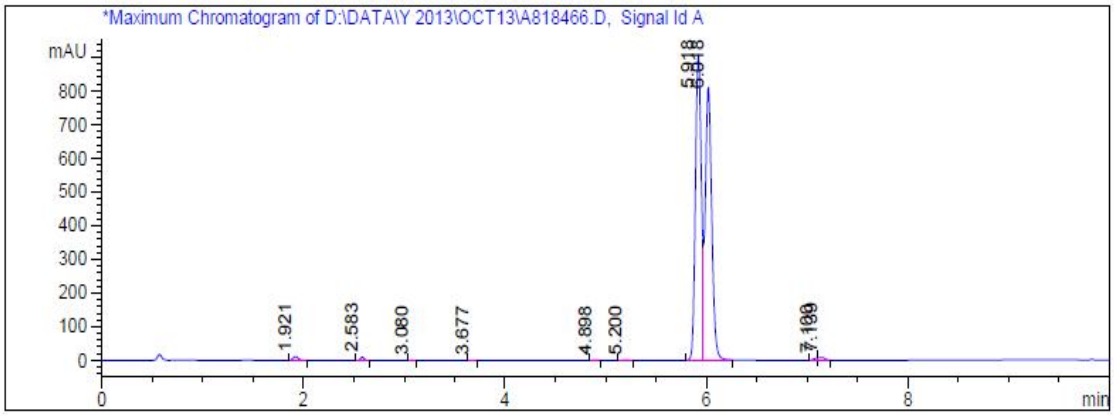
^{13}C NMR (100 MHz) in DMSO-d_6 (**1x**)

Method Info : A : 10 mM NH4HCo3 H2O B: ACN Flow = 1.0 mL/min
COLUMN:XBridge C8 (50X4.6)mm,3.5µm , -ve mode

Time	% of B
0	5
8.0	100
8.1	100
8.5	5
10.0	5

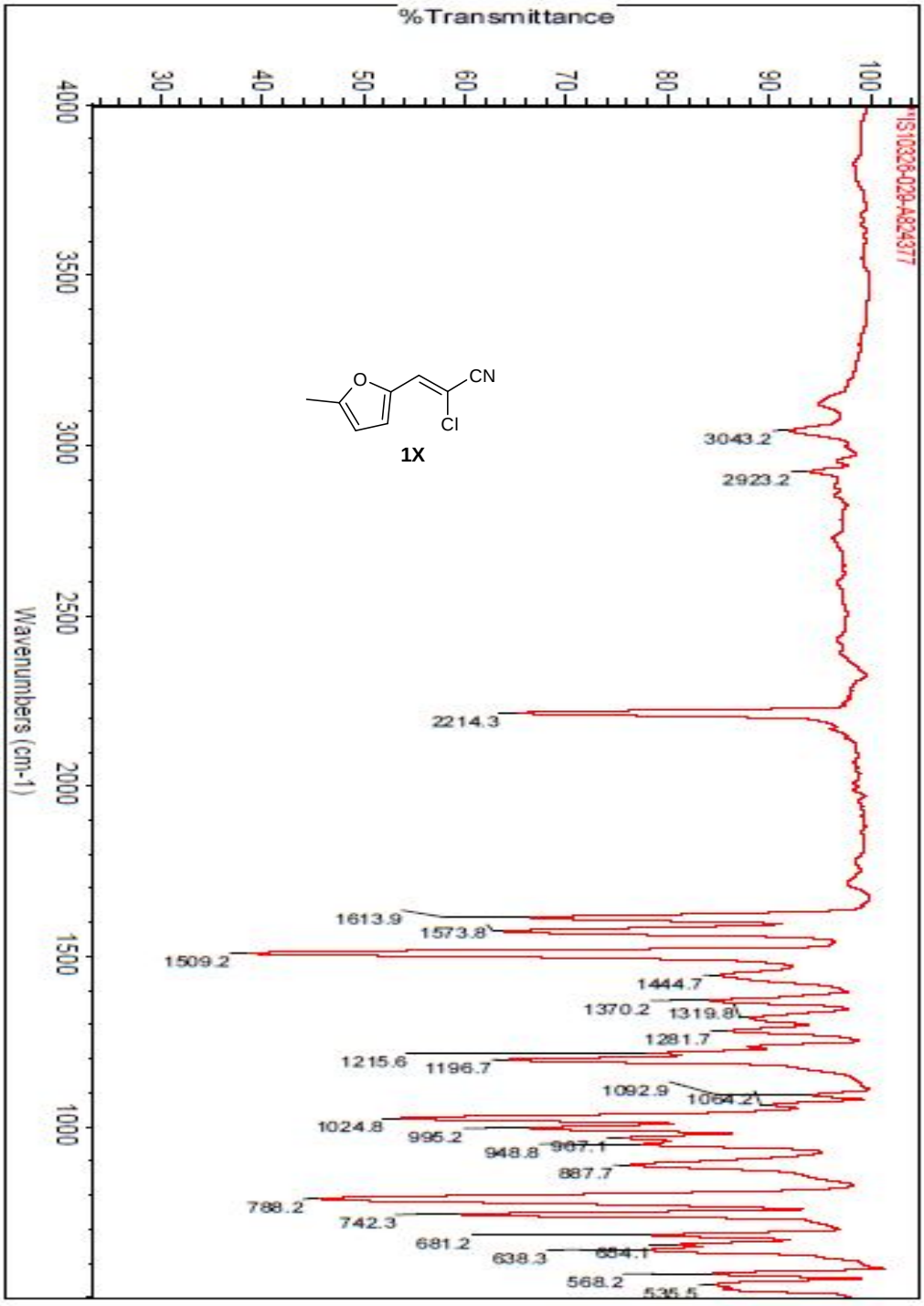


1X

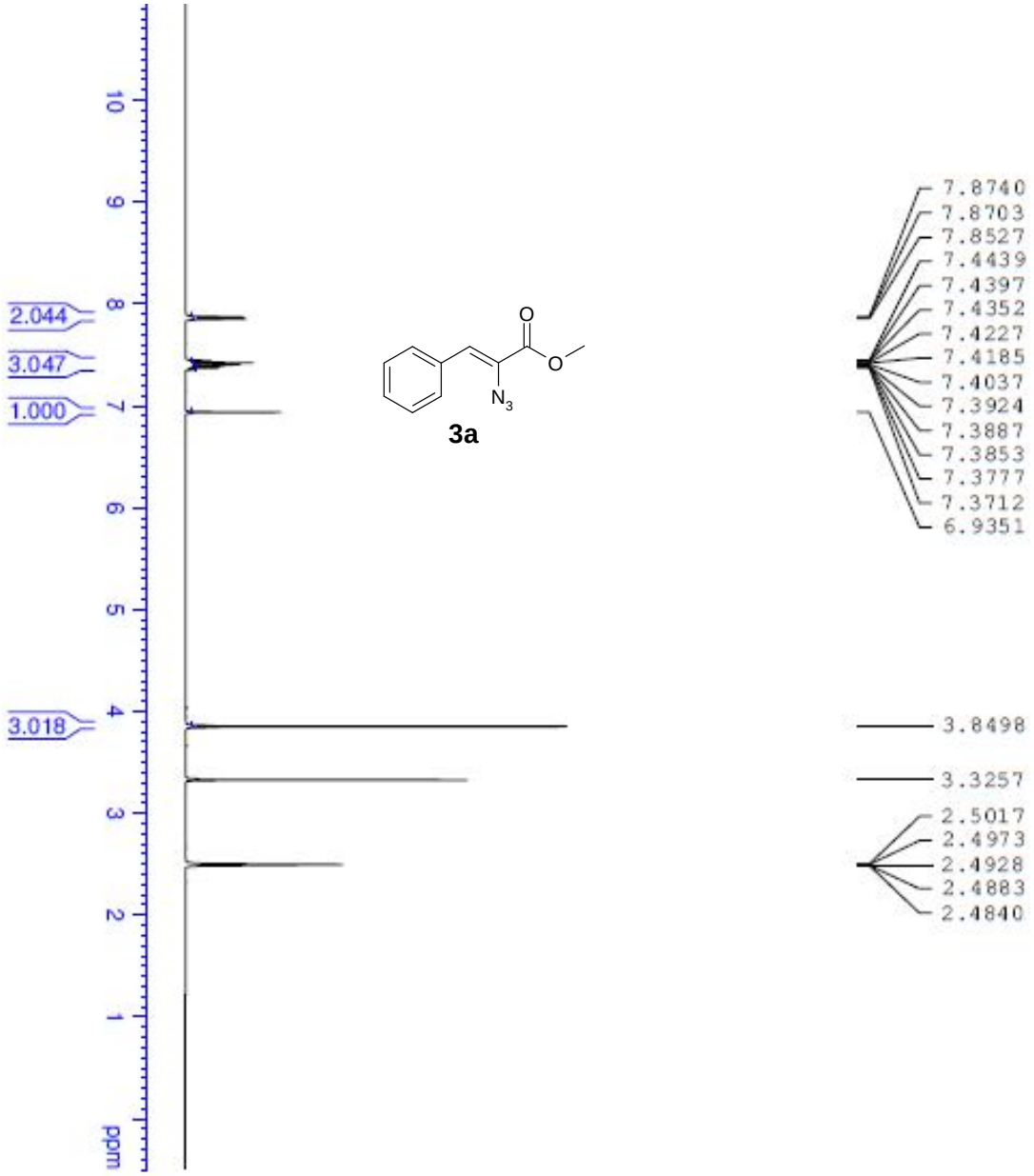


Peak No	RT min	Area	Area %
1	1.921	41.158	0.554
2	2.583	26.143	0.352
3	3.080	2.859	0.038
4	3.677	2.271	0.031
5	4.898	9.259	0.125
6	5.200	10.507	0.141
7	5.918	3692.588	49.680
8	6.018	3587.146	48.262
9	7.100	20.121	0.271
10	7.139	40.653	0.547

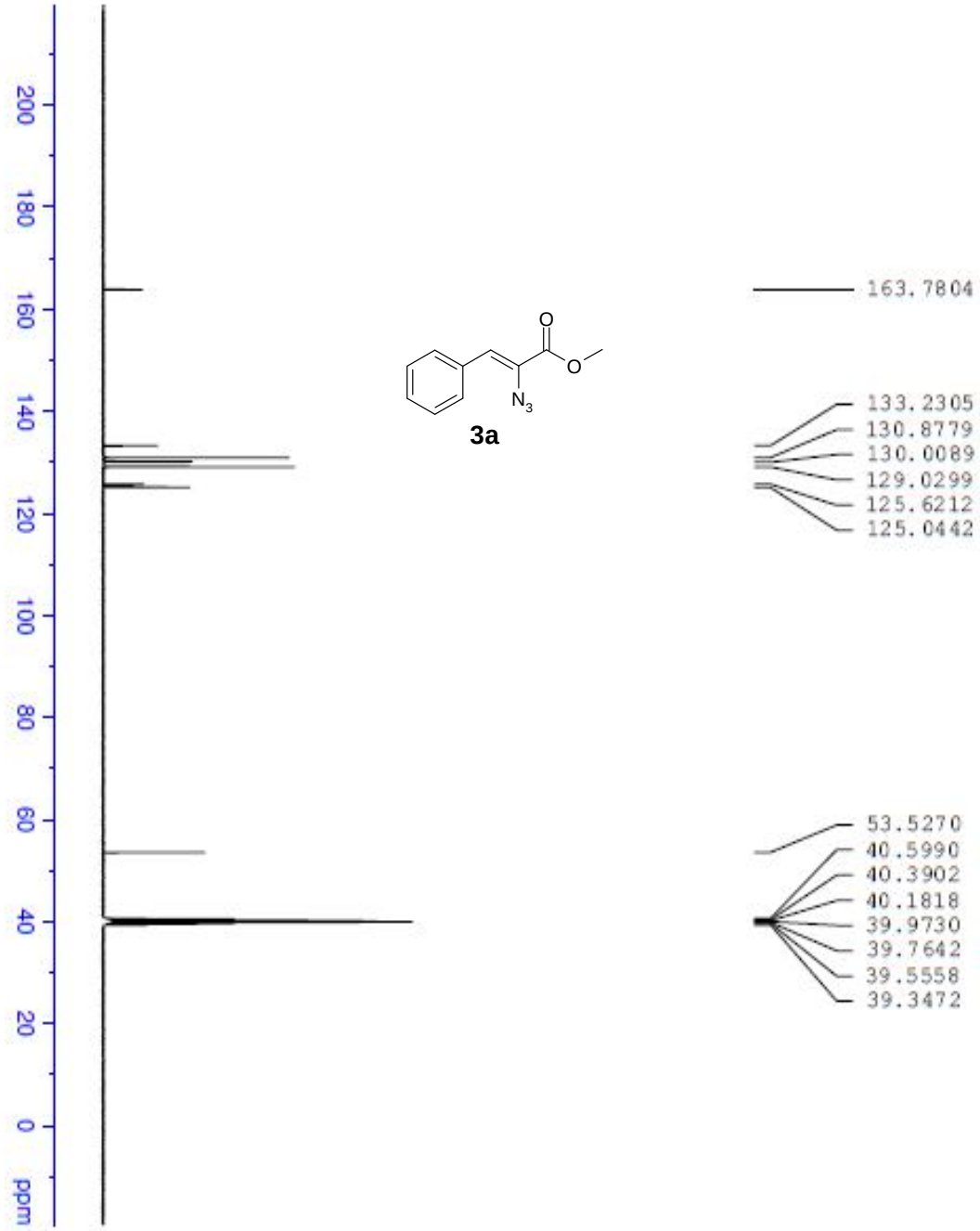
HPLC (**1x**)


%Transmittance
Wavenumbers (cm-1)
TIS10226-028-A824377
O
CN
Cl
1X
3043.2
2923.2
2214.3
1613.9
1573.8
1509.2
1444.7
1370.2
1319.8
1281.7
1215.6
1196.7
1092.9
1064.2
1024.8
995.2
948.8
967.1
887.7
788.2
742.3
681.2
638.3
694.1
568.2
535.5

IR (**1x**)



^{1}H NMR (400 MHz) in DMSO-d_6 (**3a**)



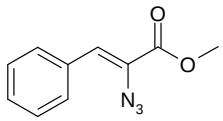
^{13}C NMR (100 MHz) in DMSO-d_6 (**3a**)

Method info :

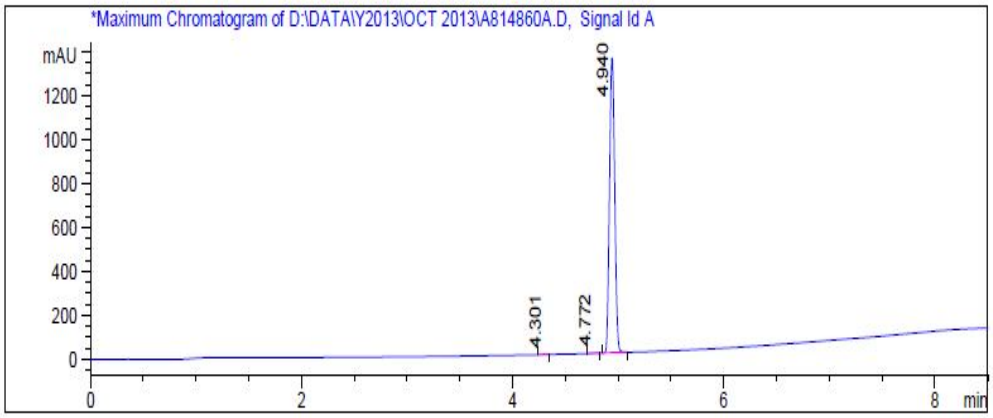
A: 0.1% TFA IN H2O , B:0.1% TFA IN ACN ; Flow Rate:2.0 ml/min

COLUMN:XBridge C8 (50x4.6mm, 3.5µm), +ve mode

TIME	%B
0	05
8.0	100
8.1	100
8.5	05
10	05

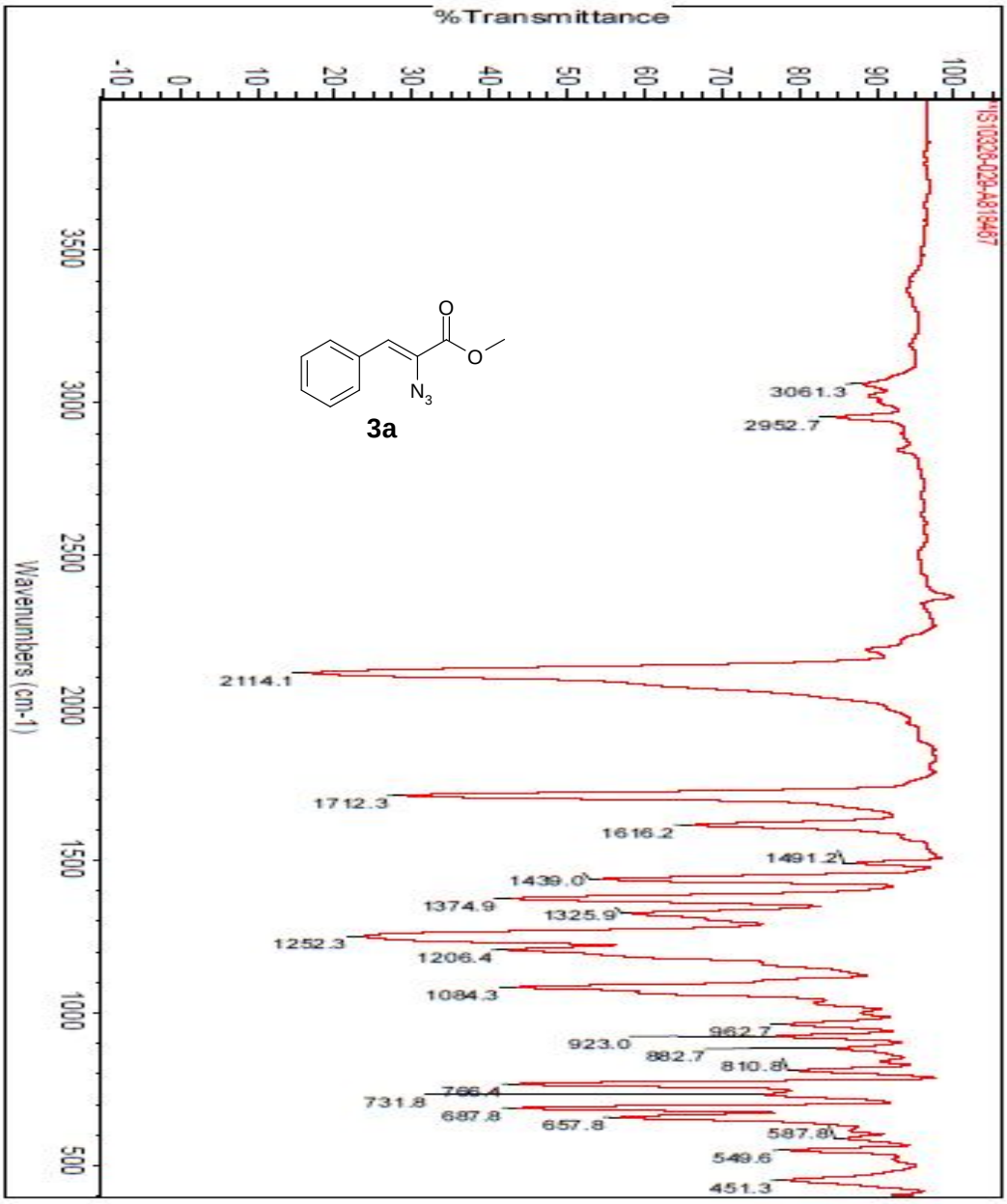


3a

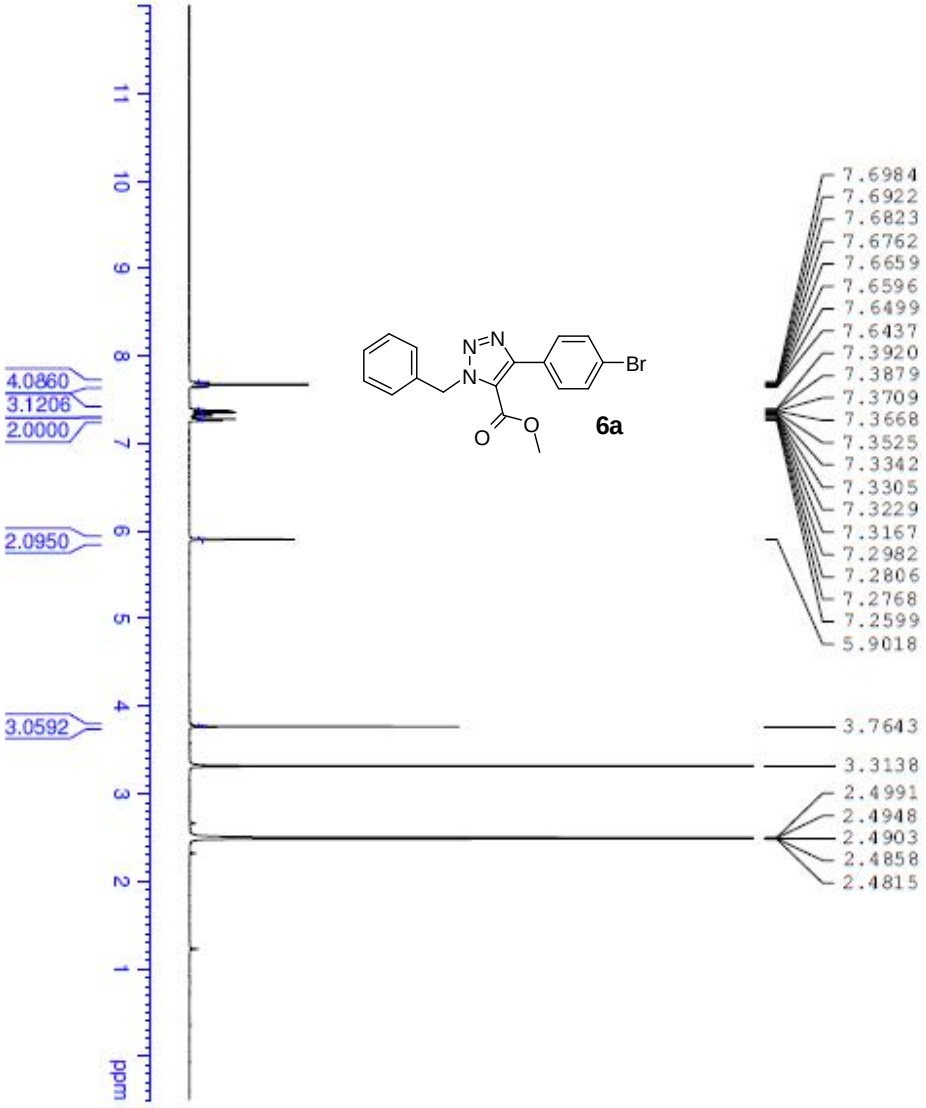


Peak No	RT min	Area	Area %
1	4.301	4.847e+000	0.105
2	4.772	7.952e+000	0.172
3	4.940	4.623e+003	99.724

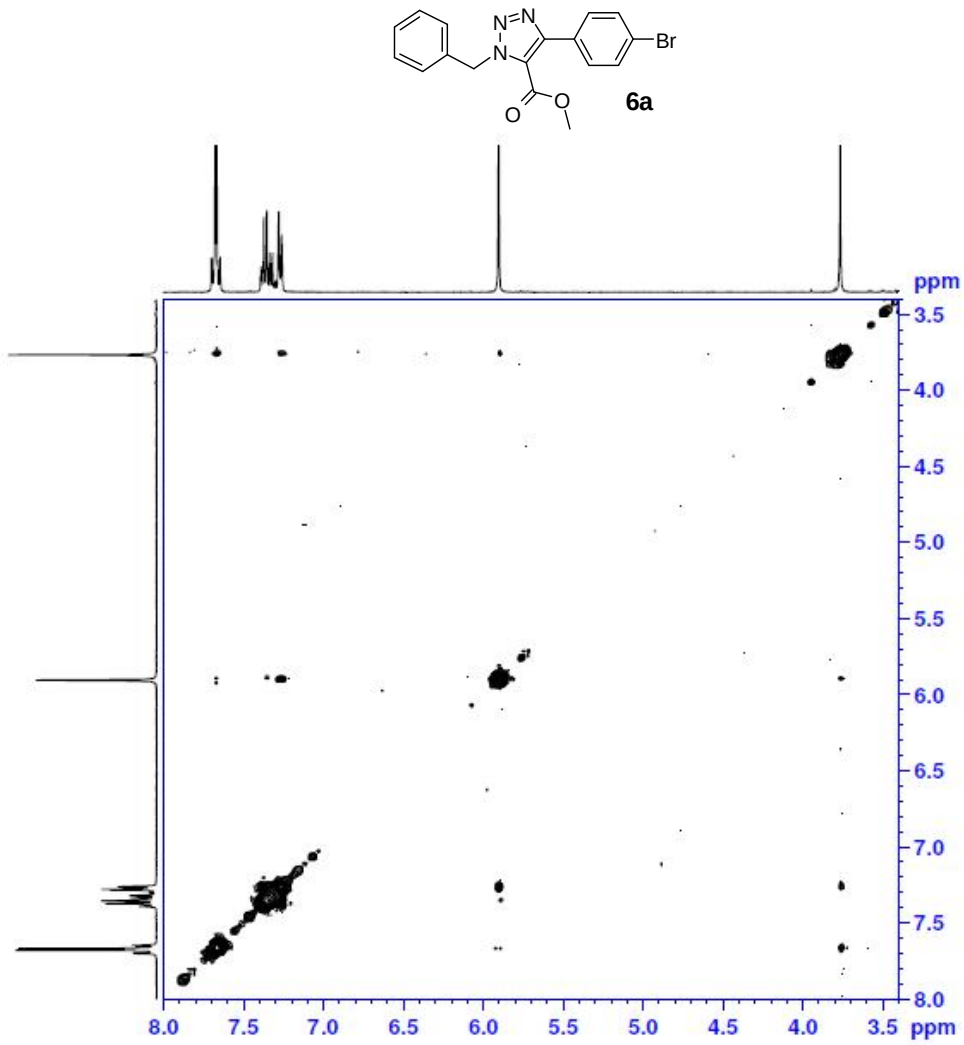
HPLC (**3a**)


%Transmittance
Wavenumbers (cm-1)
3061.3
2952.7
2114.1
1712.3
1616.2
1491.2
1439.0
1374.9
1325.9
1252.3
1206.4
1084.3
962.7
923.0
882.7
810.8
786.4
731.8
687.8
657.8
587.8
549.6
451.3
3a

IR (**3a**)



^{1}H NMR (400 MHz) in DMSO-d_6 (**6a**)



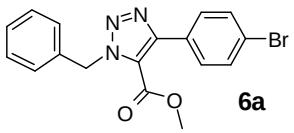
NOESY correlation (**6a**)

Method info :

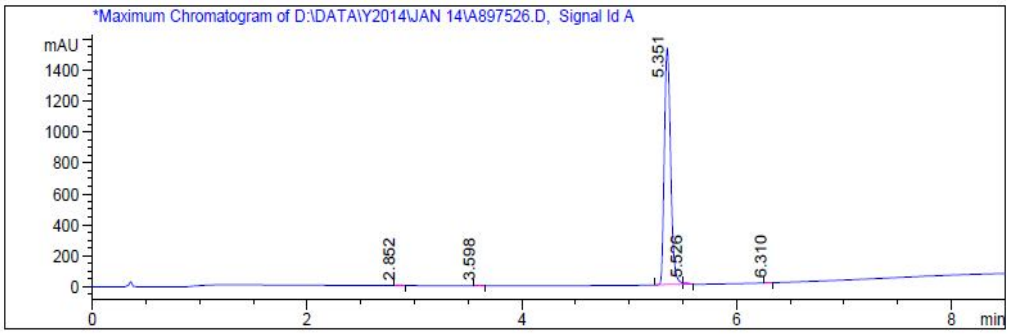
A: 0.1% TFA IN H2O , B:0.1% TFA IN ACN ; Flow Rate:2.0 ml/min

COLUMN:XBridge C8 (50x4.6mm, 3.5µ), +ve mode

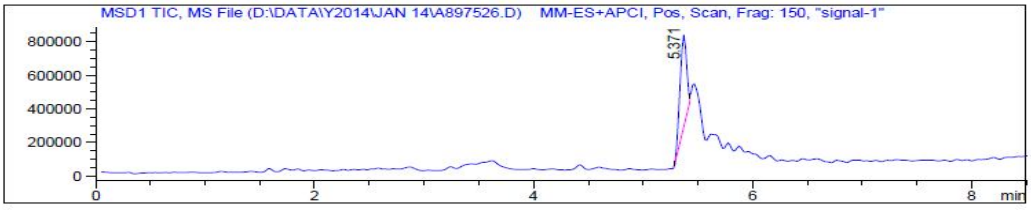
TIME	%B
0	05
8.0	100
8.1	100
8.5	05
10.0	05

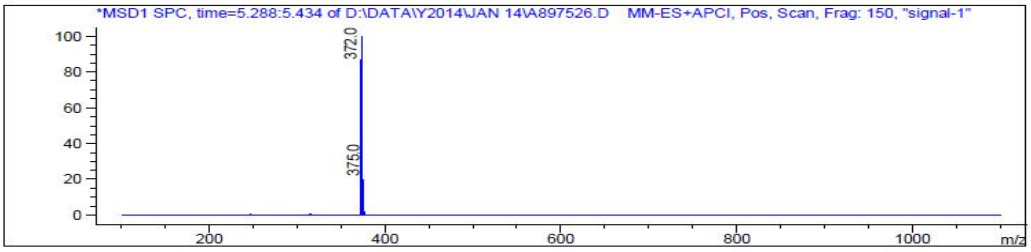


6a

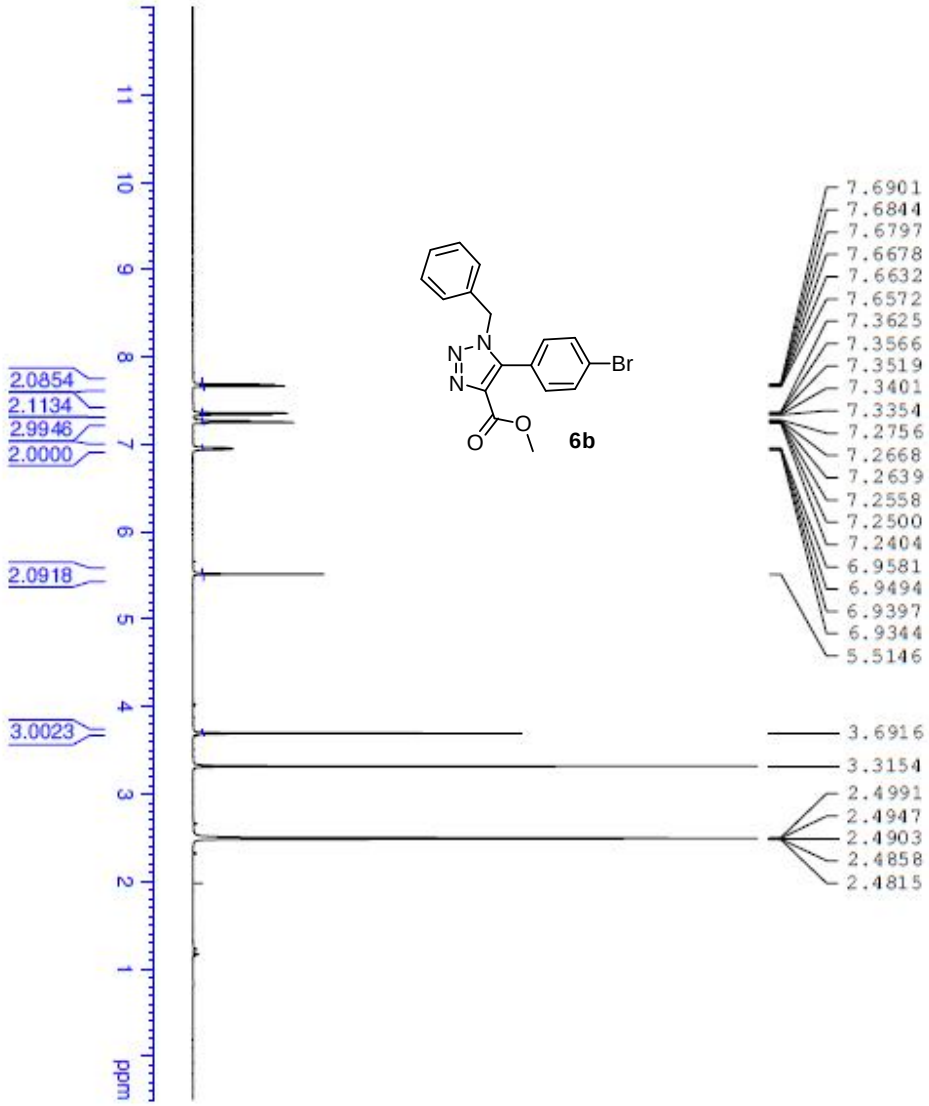


Peak No	RT min	Area	Area %
1	2.852	1.135e+001	0.179
2	3.598	8.230e+000	0.130
3	5.351	6.257e+003	98.916
4	5.526	3.948e+001	0.624
5	6.310	9.498e+000	0.150

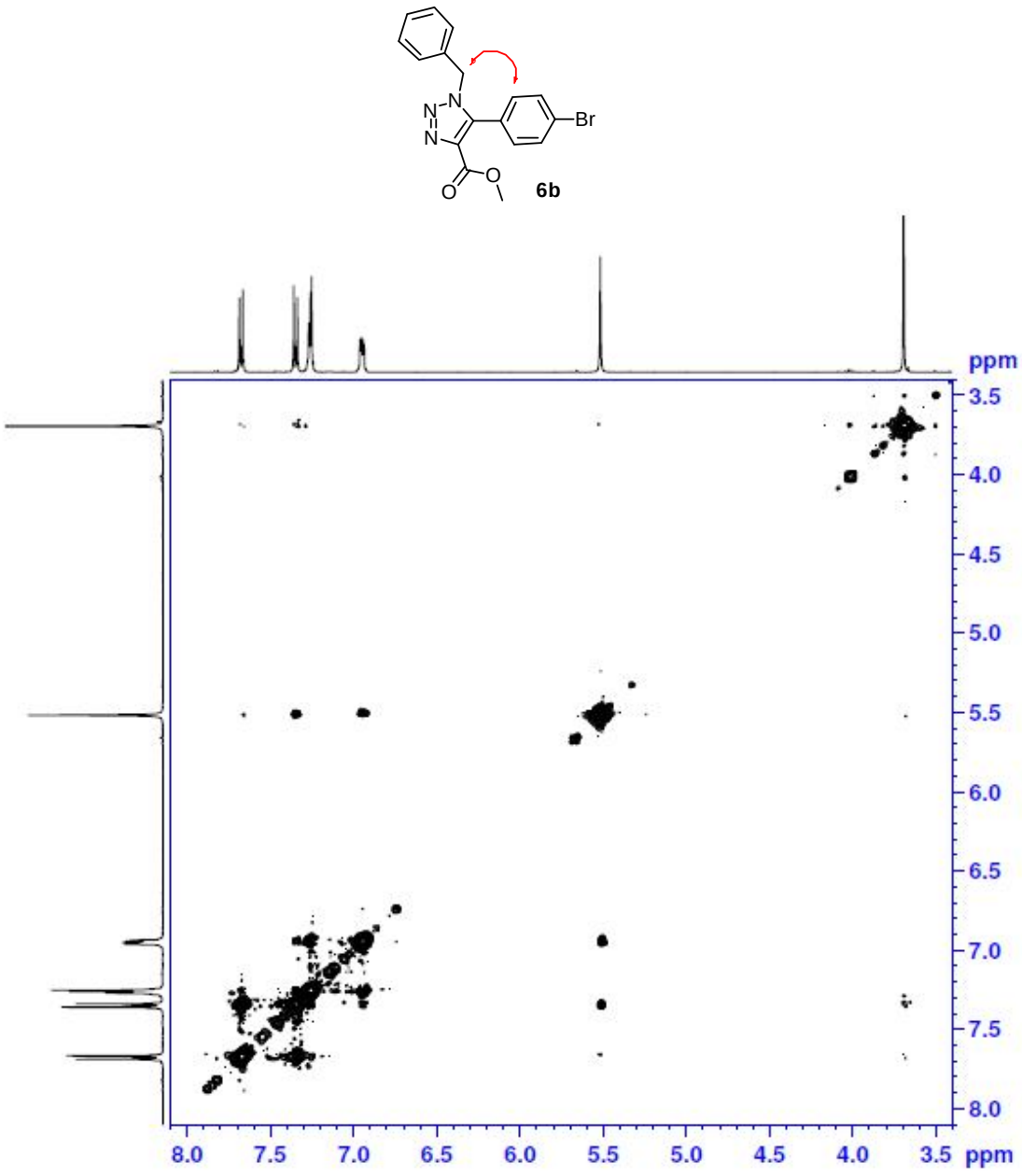




LCMS (**6a**)



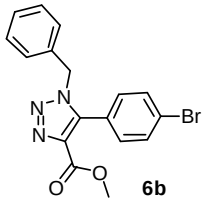
^{1}H NMR (400 MHz) in DMSO-d_6 (**6b**)



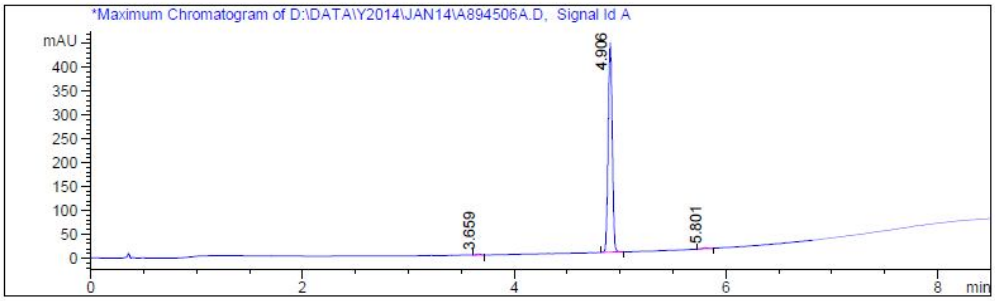
NOESY correlation (**6b**)

Method info : A: 0.1% TFA IN H2O , B:0.1% TFA IN ACN ; Flow Rate:2.0 ml/min
COLUMN:XBridge C8 (50x4.6mm, 3.5µ), +ve mode

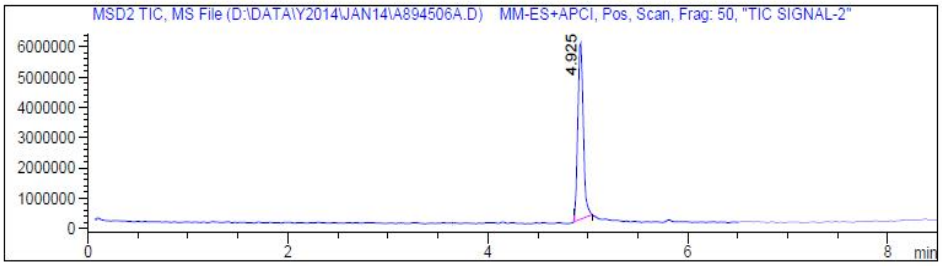
TIME	%B
0	05
8.0	100
8.1	100
8.5	05
10.0	05

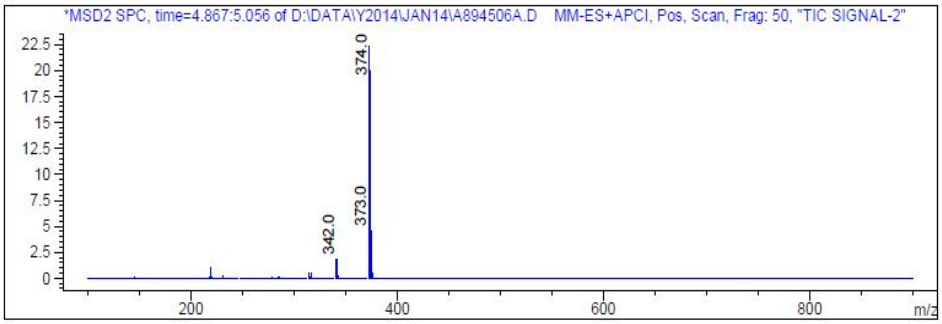


6b

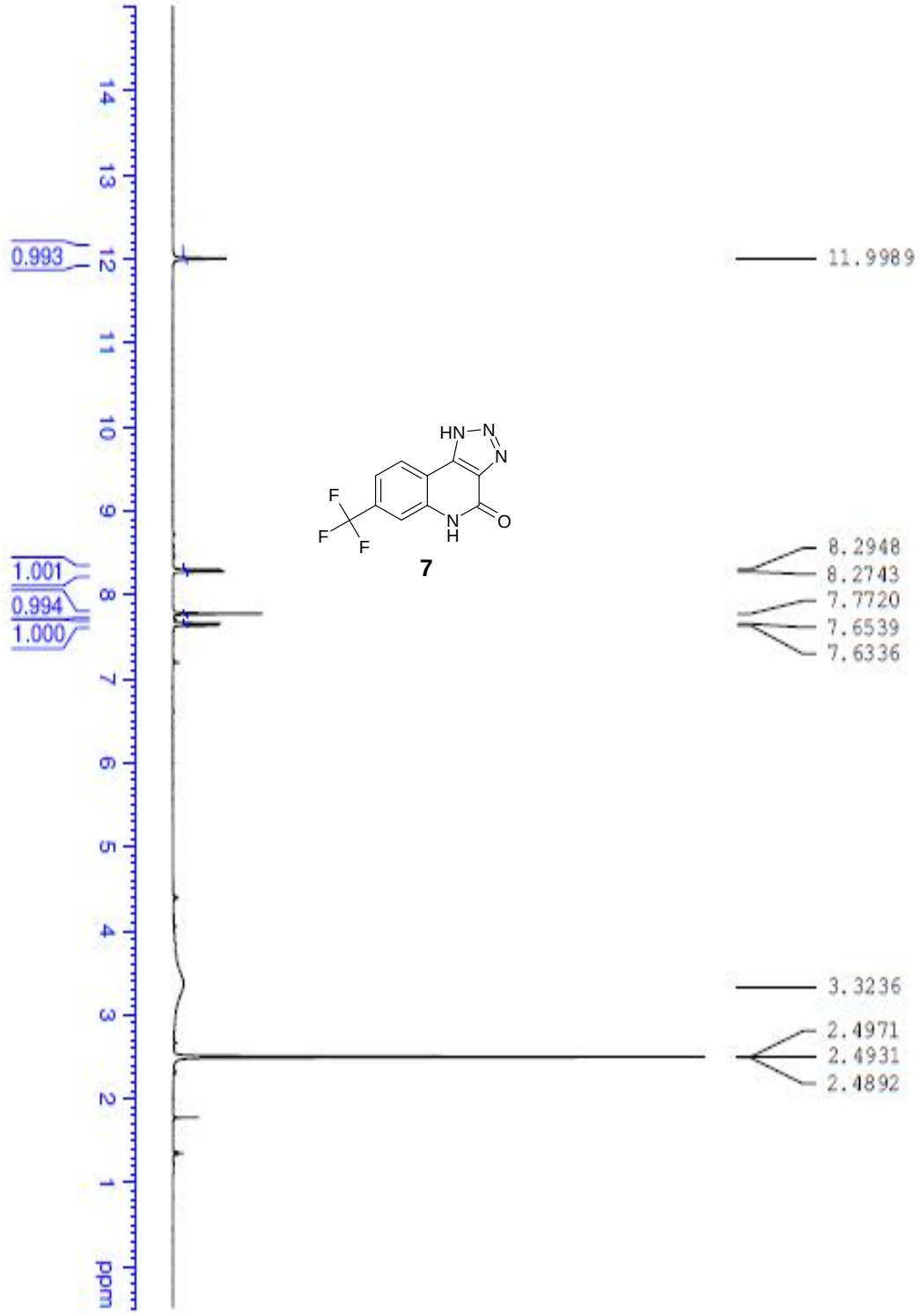


Peak No	RT min	Area	Area %
1	3.659	7.276e+000	0.614
2	4.906	1.170e+003	98.765
3	5.801	7.358e+000	0.621

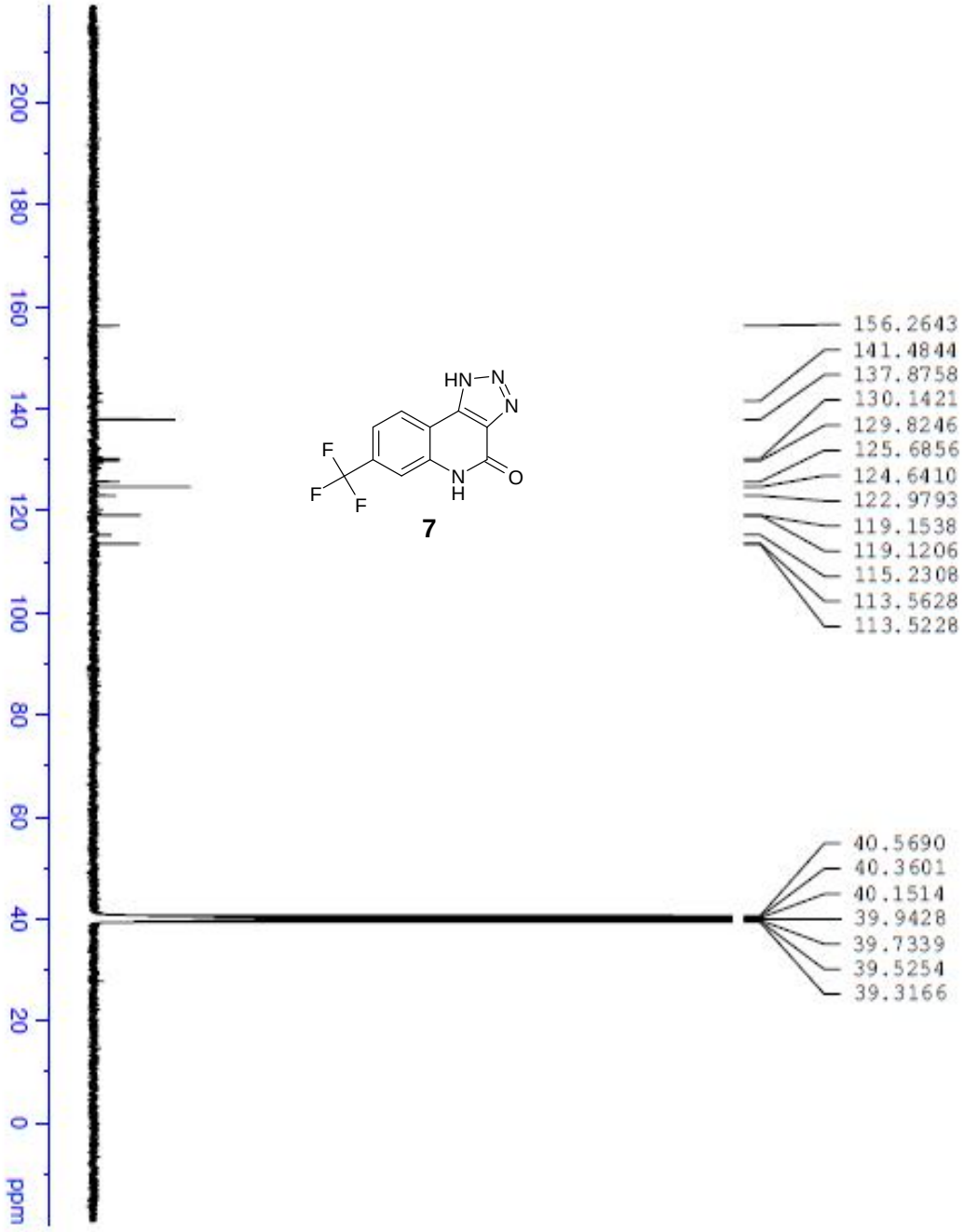




LC MS (**6b**)



^{1}H NMR (400 MHz) in DMSO-d_6 (**7**)

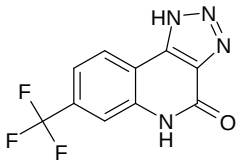


^{13}C NMR (100 MHz) in DMSO-d_6 (**7**)

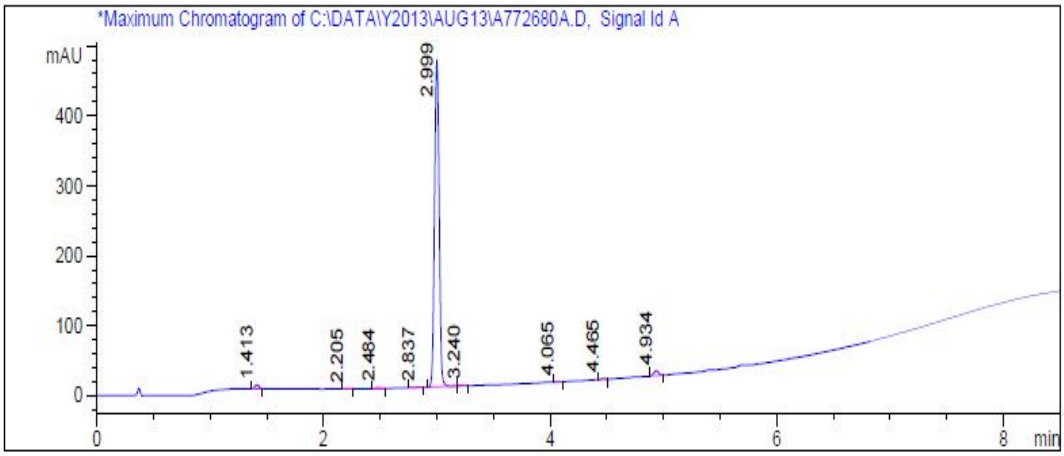
Method info : A:0.1%TFA in H2O, B:0.1%TFA in ACN, Flow Rate:2.0ml/min

COLUMN: XBridge C8(50X4.6)mm,3.5µm

Time	% of B
0	5
8.0	100
8.1	100
8.5	5
10.0	5

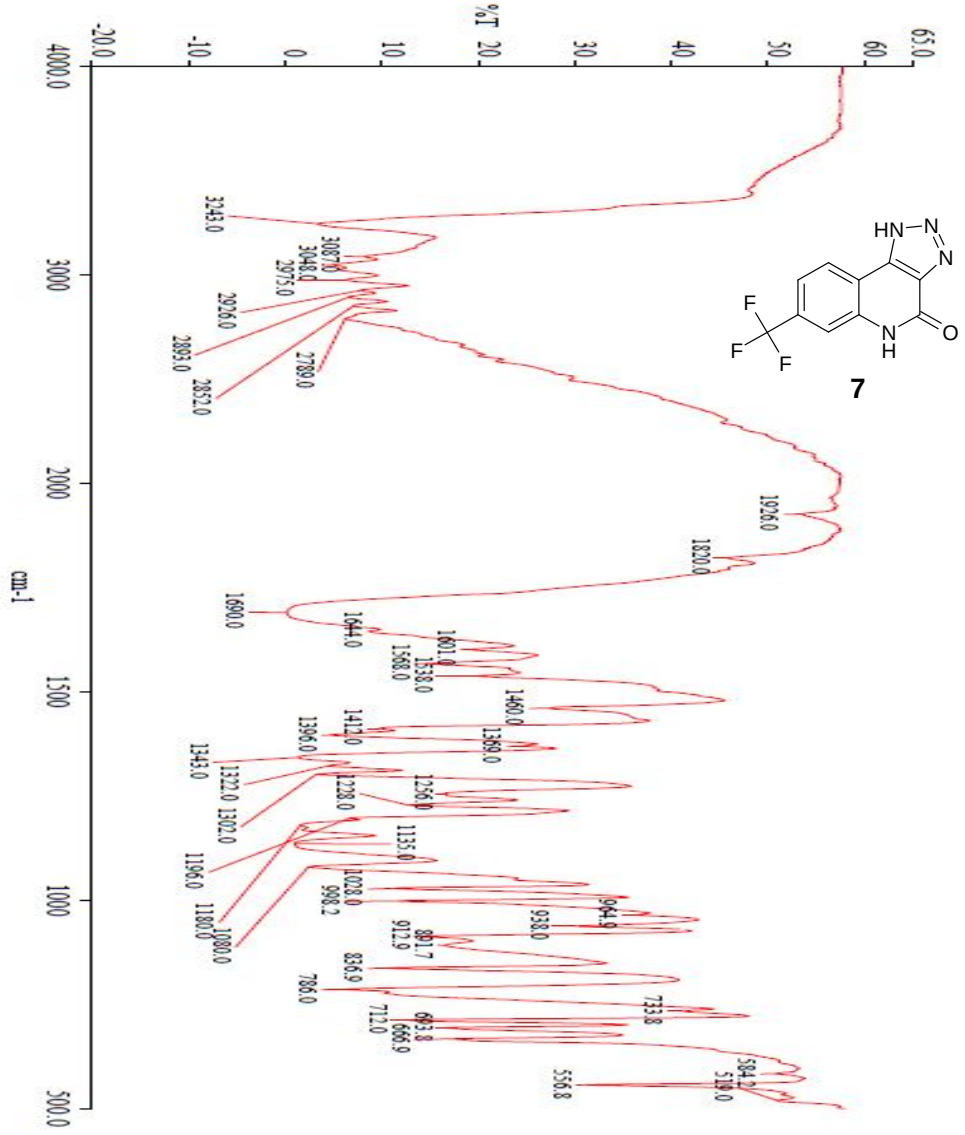


7



Peak No	RT min	Area	Area %
1	1.413	1.451e+001	1.06
2	2.205	2.060e+000	0.15
3	2.484	3.556e+000	0.26
4	2.837	3.028e+000	0.22
5	2.999	1.317e+003	96.00
6	3.240	1.803e+000	0.13
7	4.065	2.940e+000	0.21
8	4.465	3.522e+000	0.26
9	4.934	2.350e+001	1.71

HPLC (**7**)



IR (**7**)

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