Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Metal-assisted Addition of Nitrate Aanion to Bis(oxy)enamines. A General Approach to the Synthesis of α-Nitroxy-oxime Ethers from Nitronates

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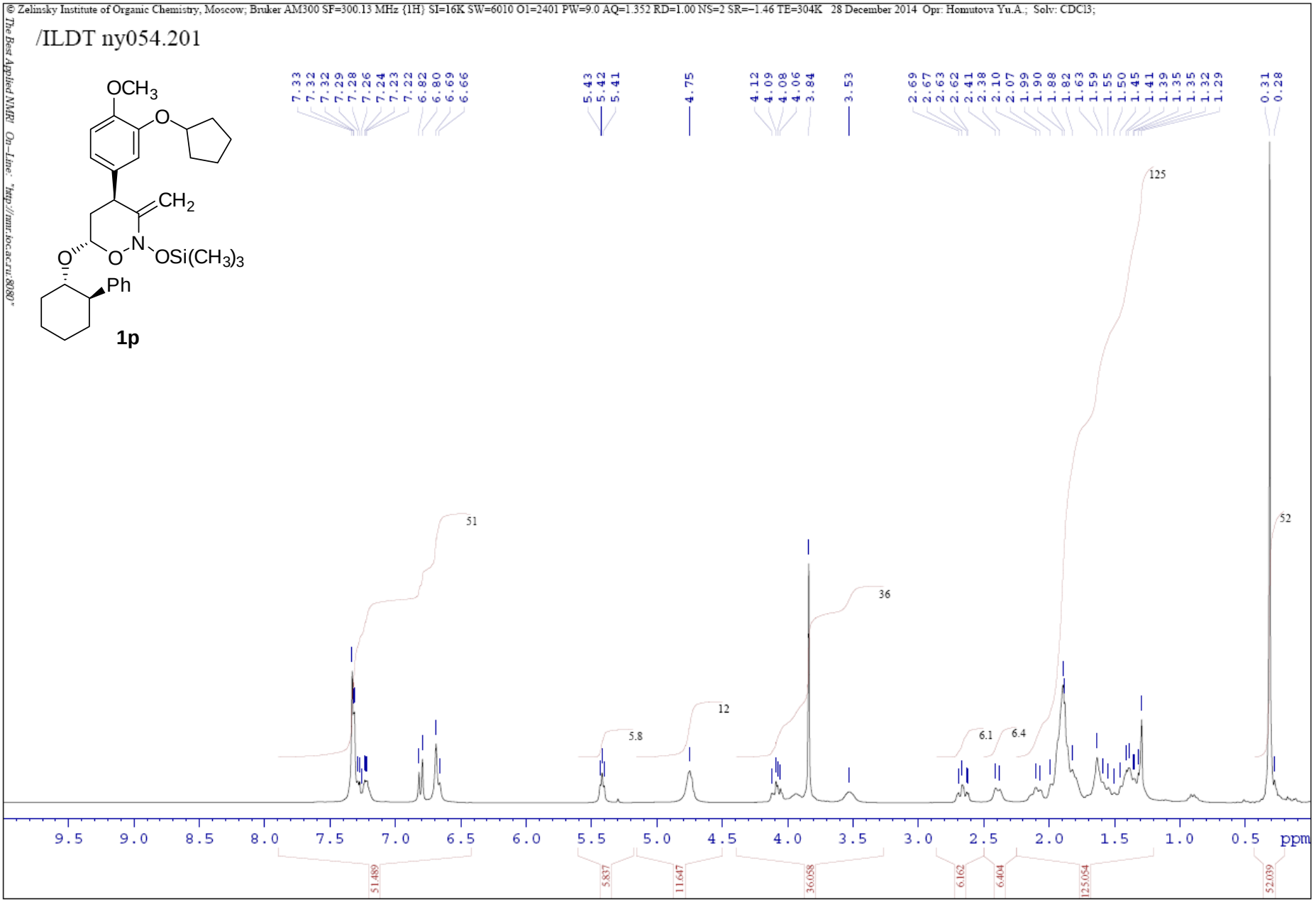
[d] *A.N. Nesmeyanov Institute of Organoelement Compounds, Vavilov str. 28, 119991, Moscow, Russia.*

[*] *Corresponding author: E-mail: sukhorukov@ioc.ac.ru; Tel: +7 499 1355329; Fax: +7 499 1355328*

Contents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O
CH2
N
O
O
OSi(CH3)3
Ph
1p
7.33 7.32 7.32 7.29 7.28 7.26 7.24 7.23 7.22 6.82 6.80 6.69 6.66
5.43 5.42 5.41
4.75
4.12 4.09 4.08 4.06 3.84
3.53
2.69 2.67 2.63 2.62 2.41 2.38 2.10 2.07 1.99 1.90 1.88 1.82 1.63 1.59 1.55 1.50 1.45 1.41 1.39 1.35 1.35 1.32 1.29
0.31 0.28
125
51
52
36
12
5.8
6.1
6.4
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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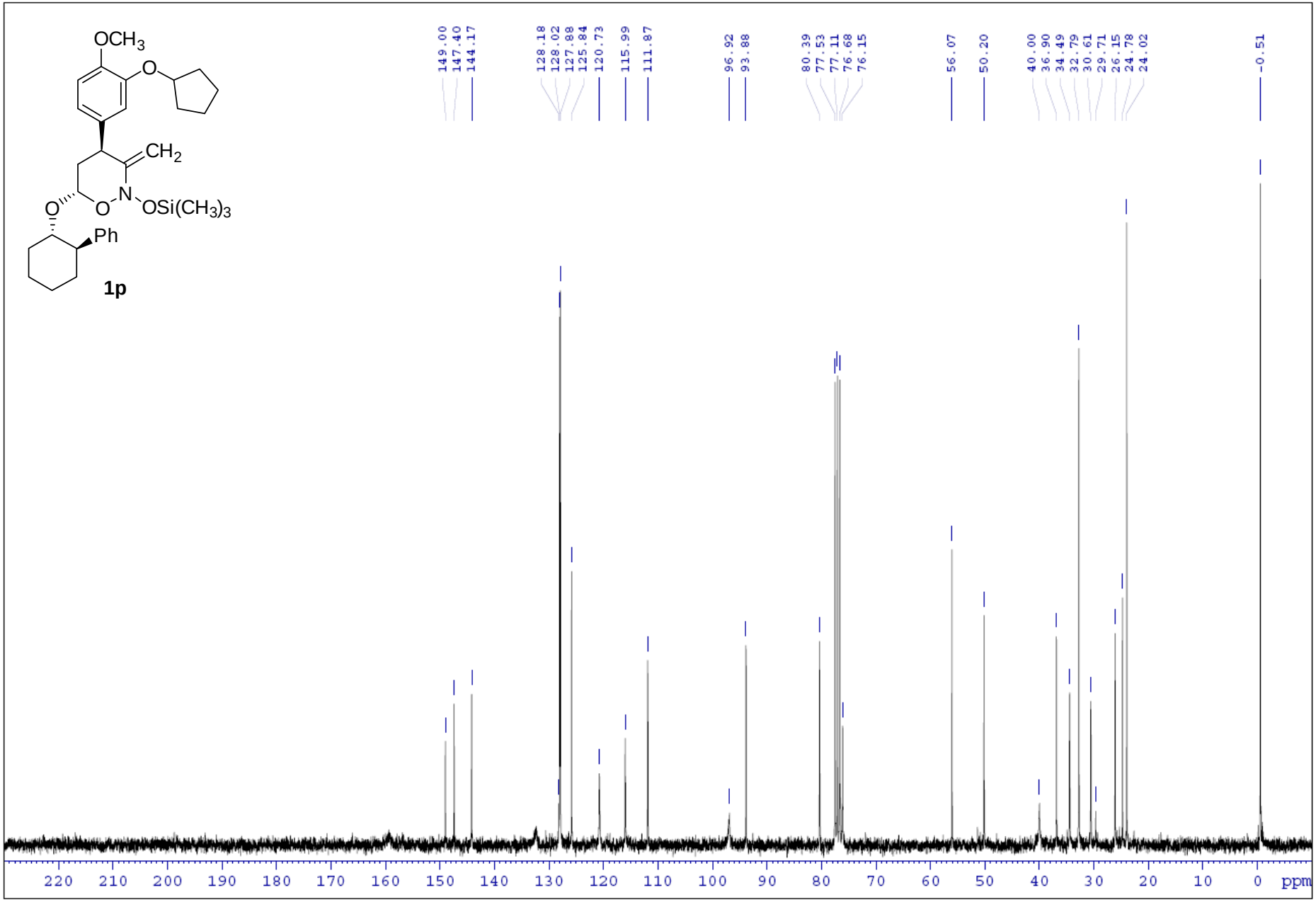
11.647
36.058
6.162
6.404
125.054
52.039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O
CH2
N
O
O
OSi(CH3)3
Ph
1p
149.00
147.40
144.17
128.18
128.02
127.88
125.84
120.73
115.99
111.87
96.92
93.88
80.39
77.53
77.11
76.68
76.15
56.07
50.20
40.00
36.90
34.49
32.79
30.61
29.71
26.15
24.78
24.02
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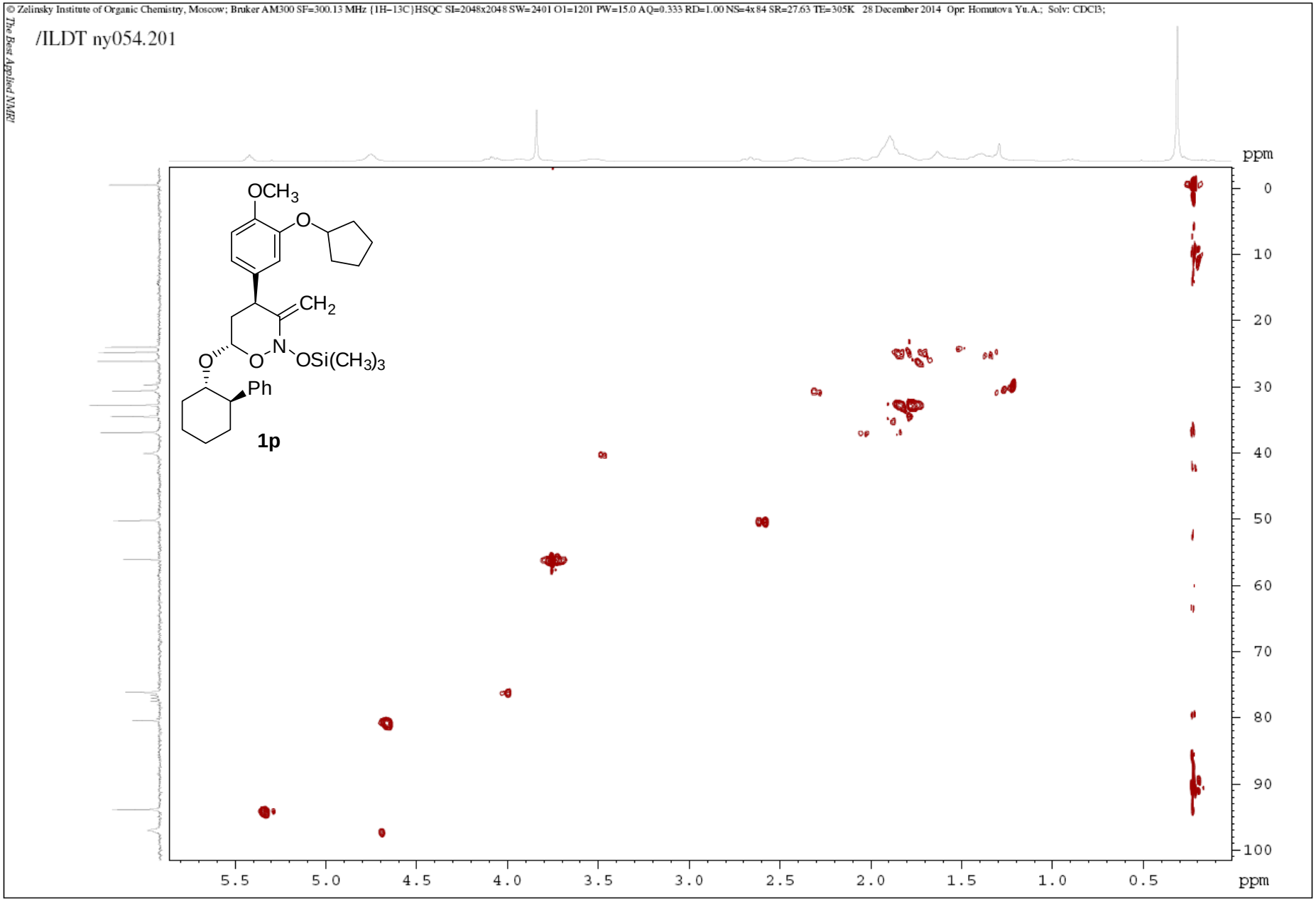
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The Best Applied NMR!
/ILDT ny054.201
OCH3
O
CH2
N
O
OSi(CH3)3
O
Ph
1p
ppm
0
10
20
30
40
50
60
70
80
90
100
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

The Best Applied NMR!

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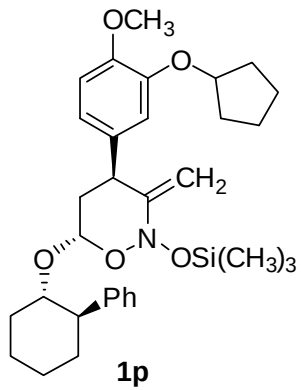

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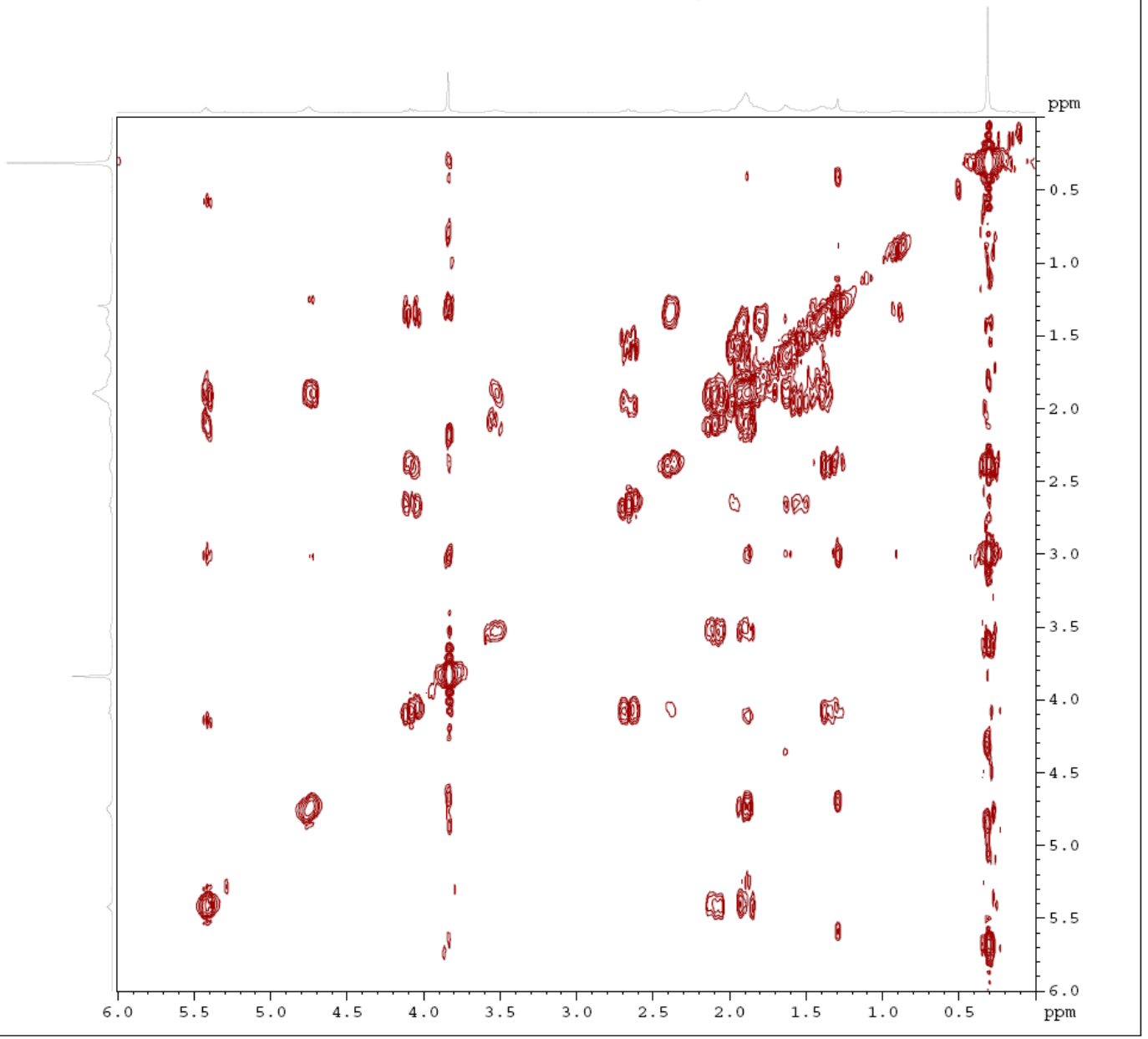

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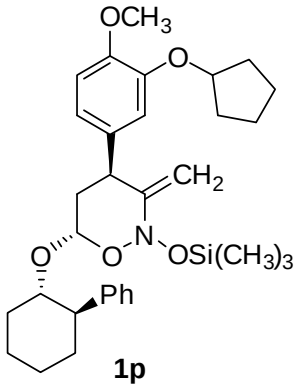

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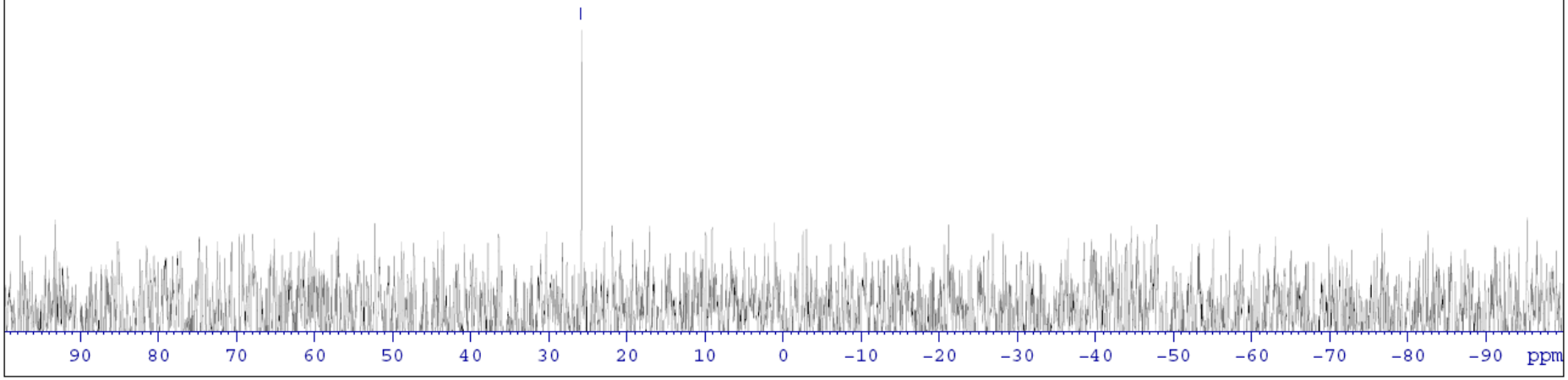
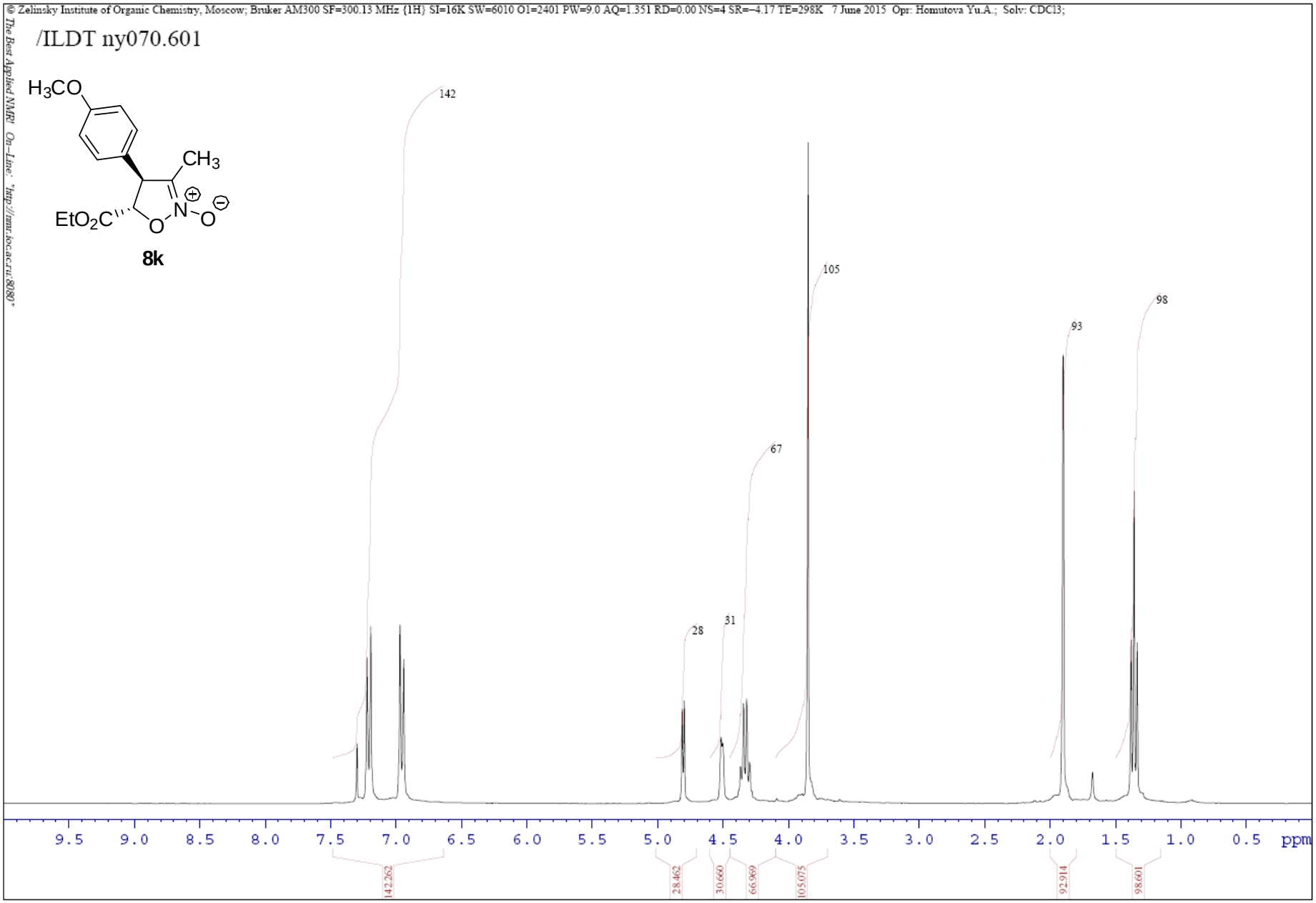
25.86

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/ILDT ny070.601
H3CO
CH3
EtO2C
N
O
8k
142
105
98
93
67
31
28
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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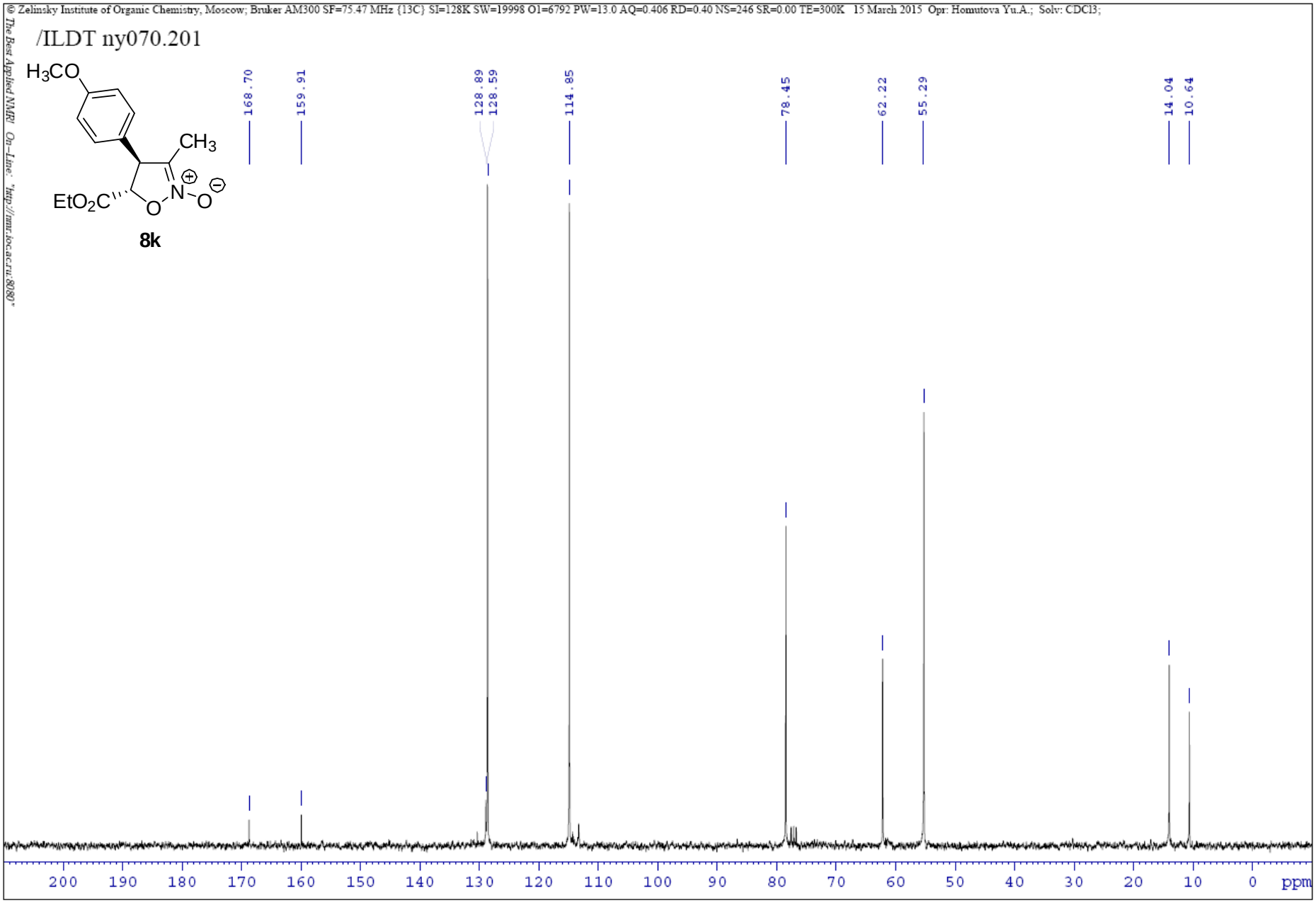
28.462
30.660
66.969
105.075
92.914
98.601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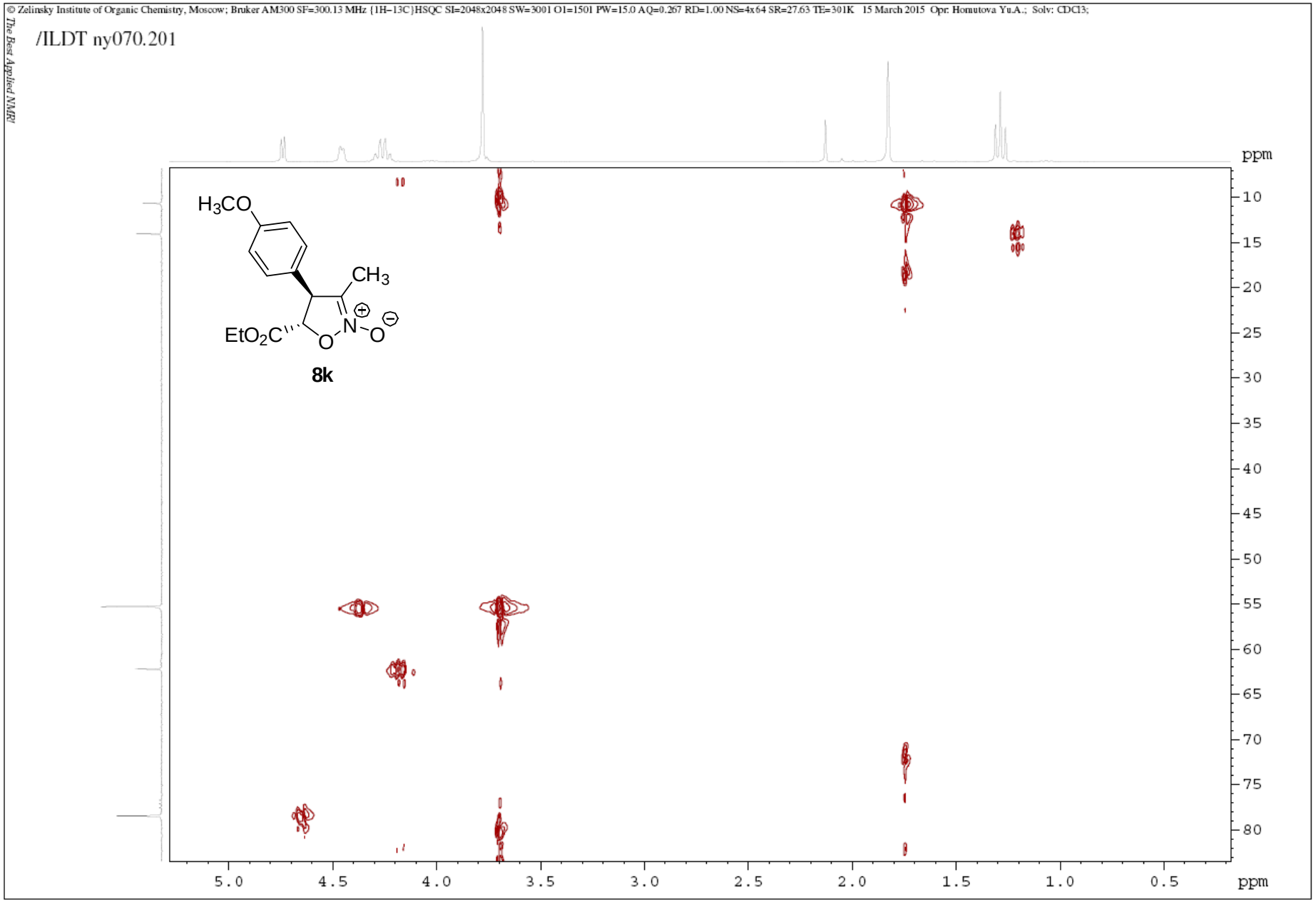
H3CO
CH3
EtO2C
N
O
O
8k
168.70
159.91
128.89
128.59
114.85
78.45
62.22
55.29
14.04
10.64
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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H3CO
CH3
EtO2C
N
O
O
8k
ppm
10
15
20
25
30
35
40
45
50
55
60
65
70
75
80
5.0
4.5
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2.5
2.0
1.5
1.0
0.5
ppm

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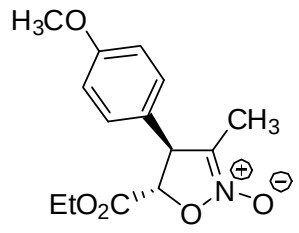

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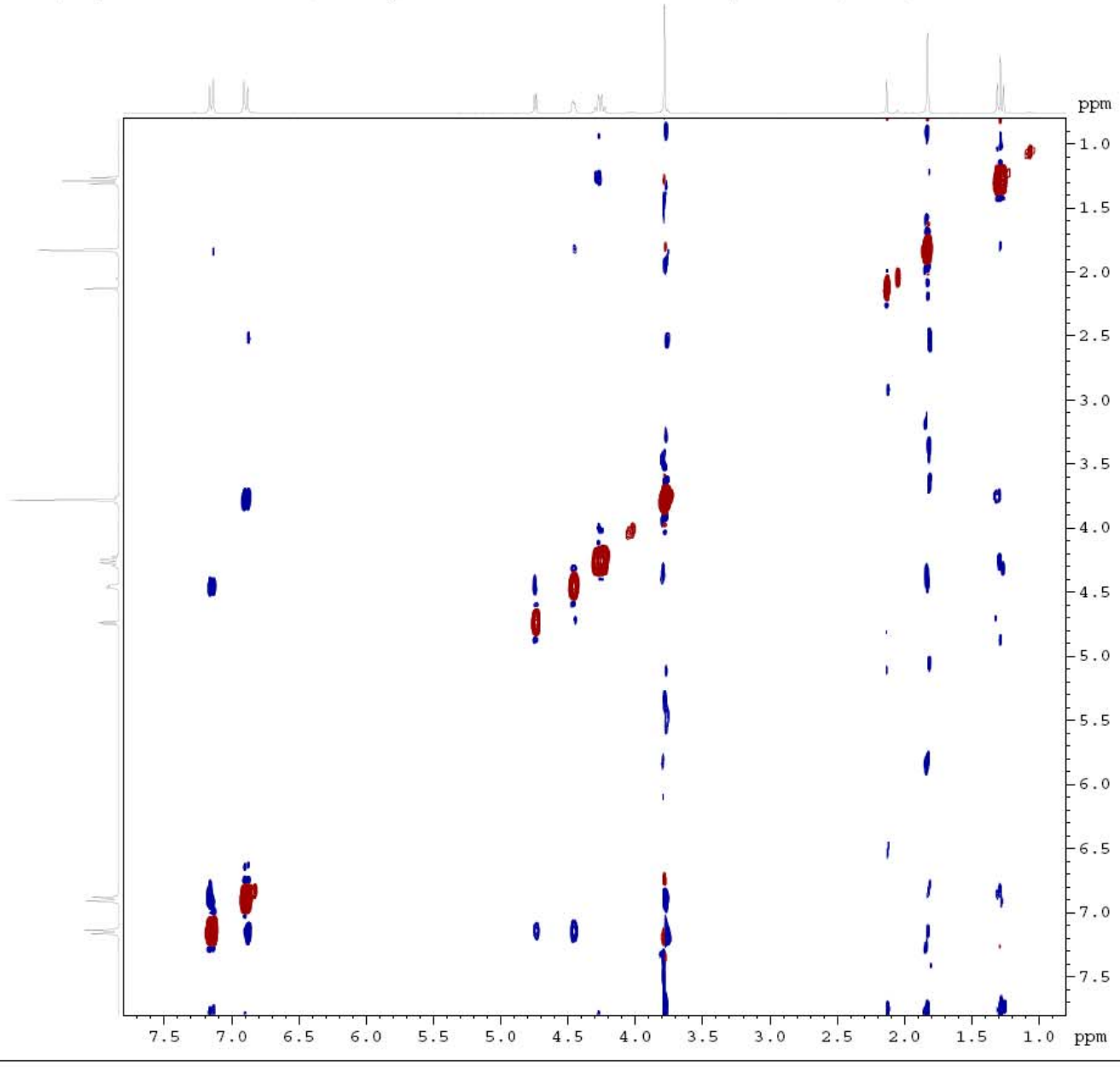

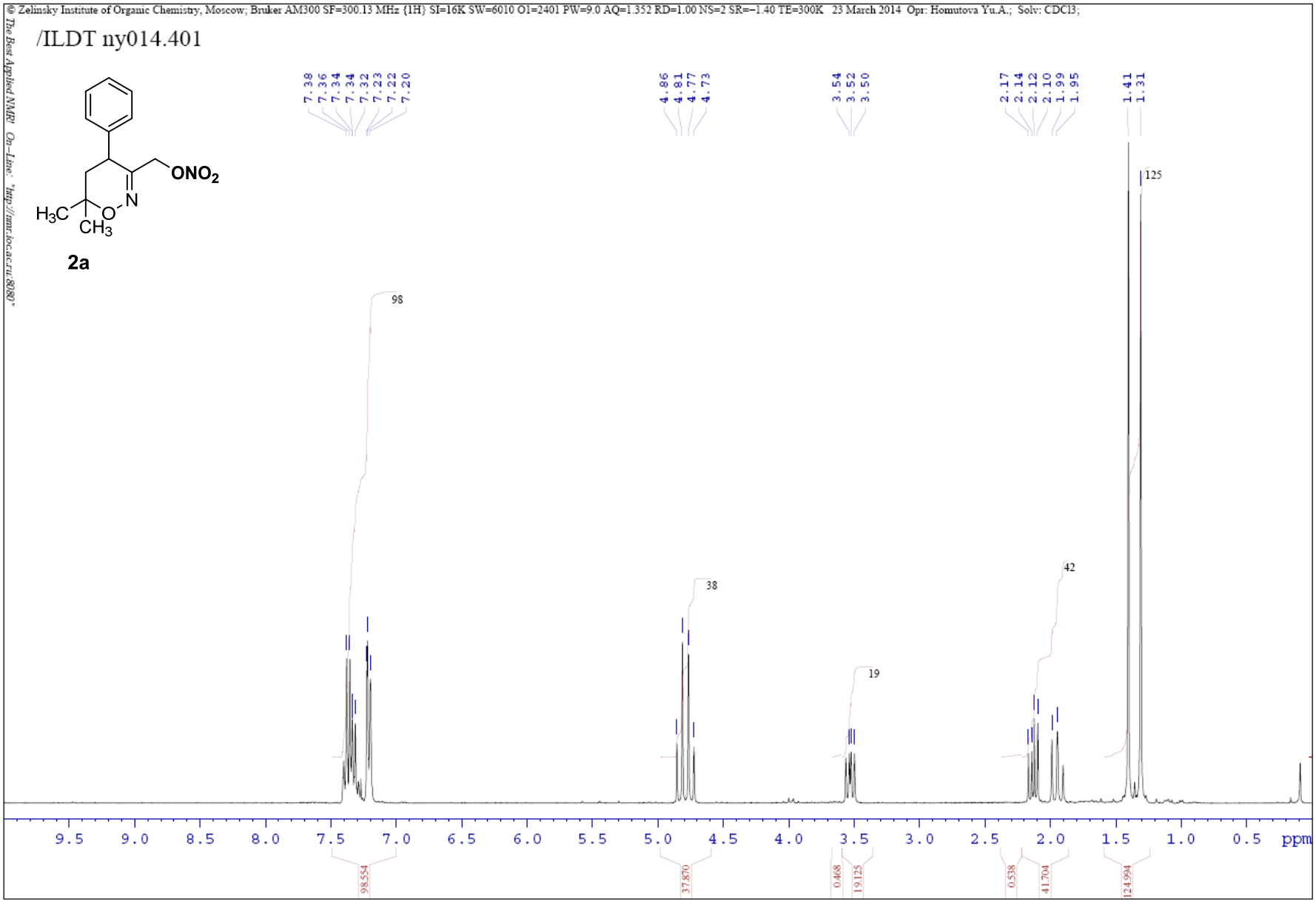
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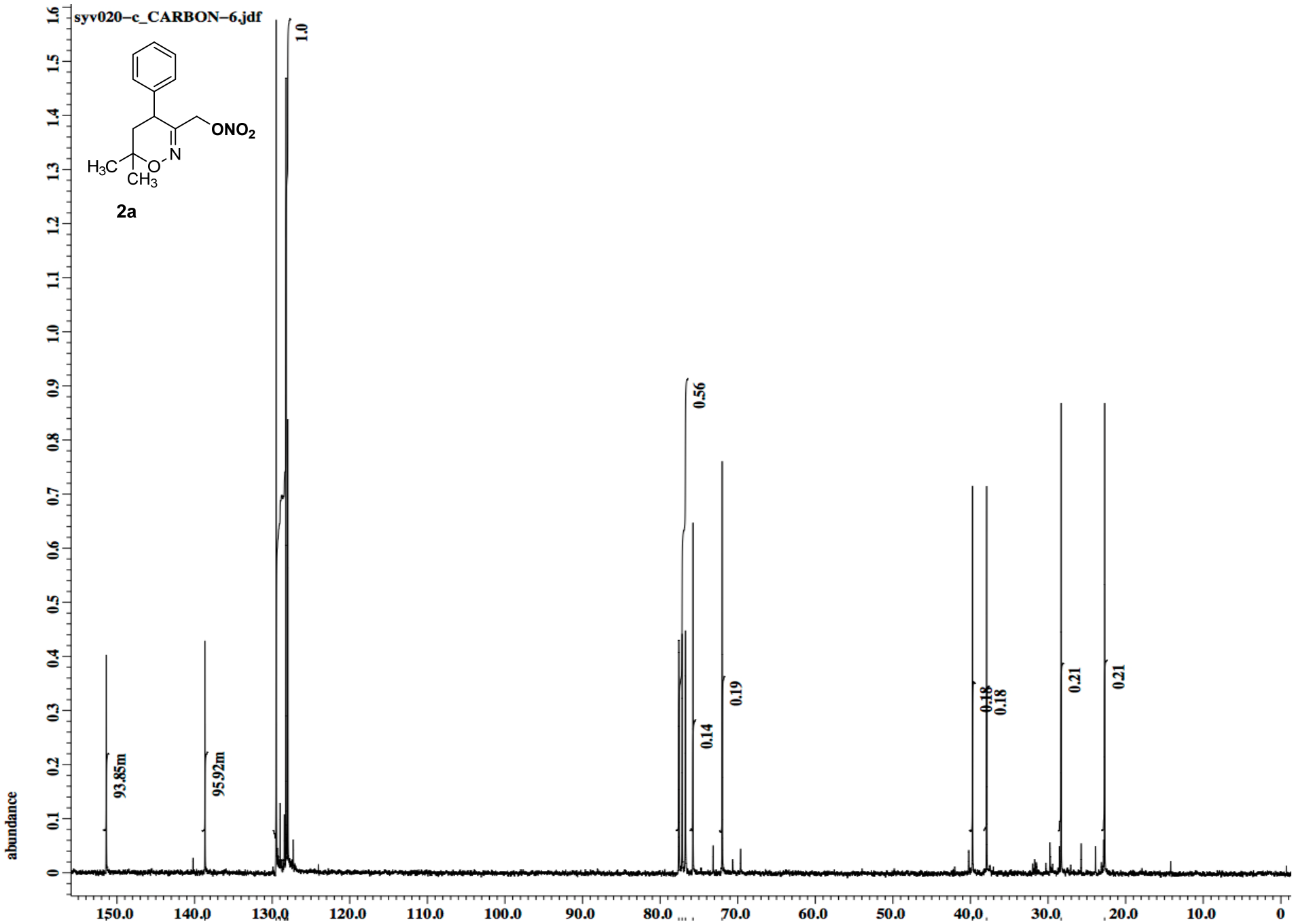
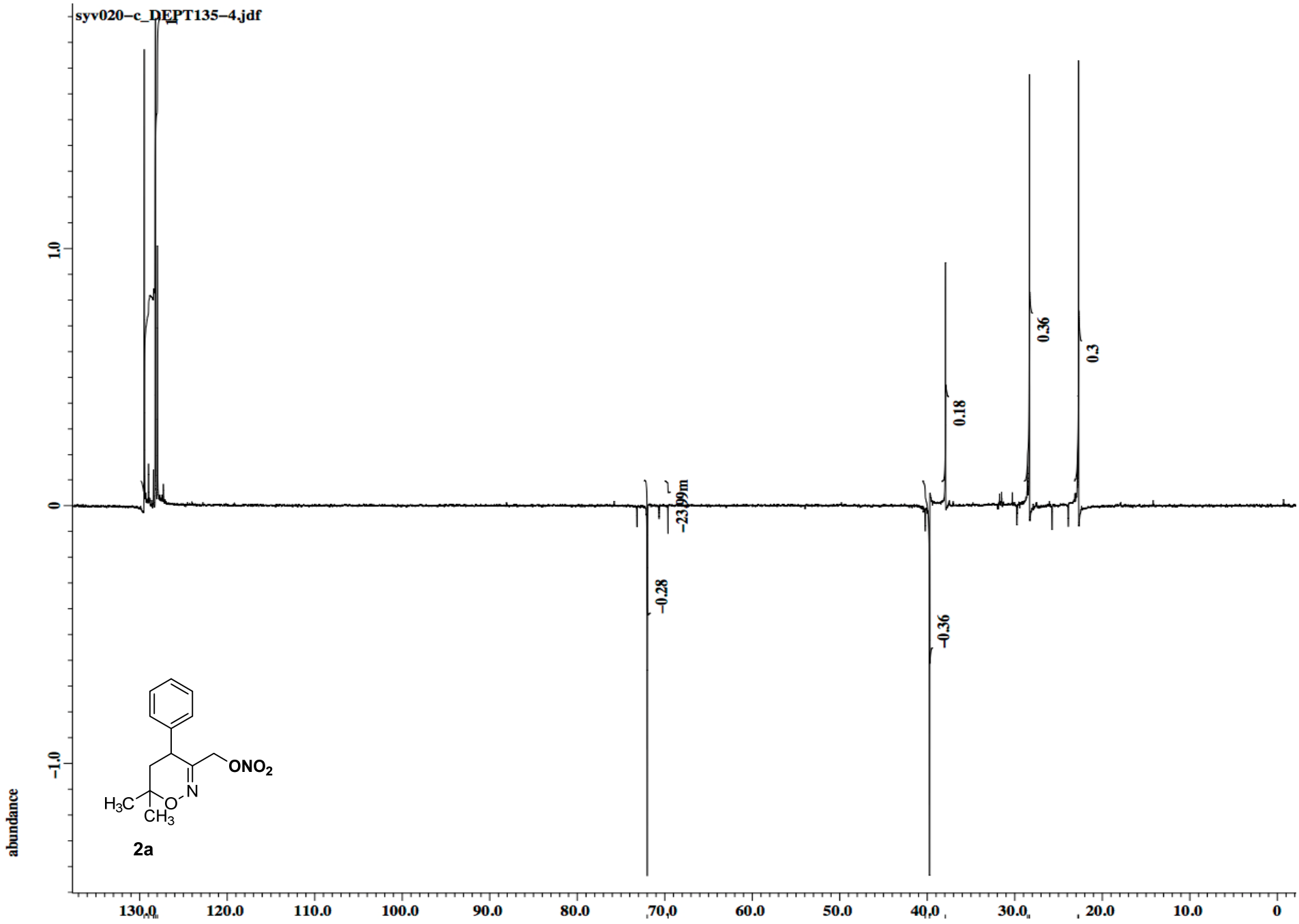
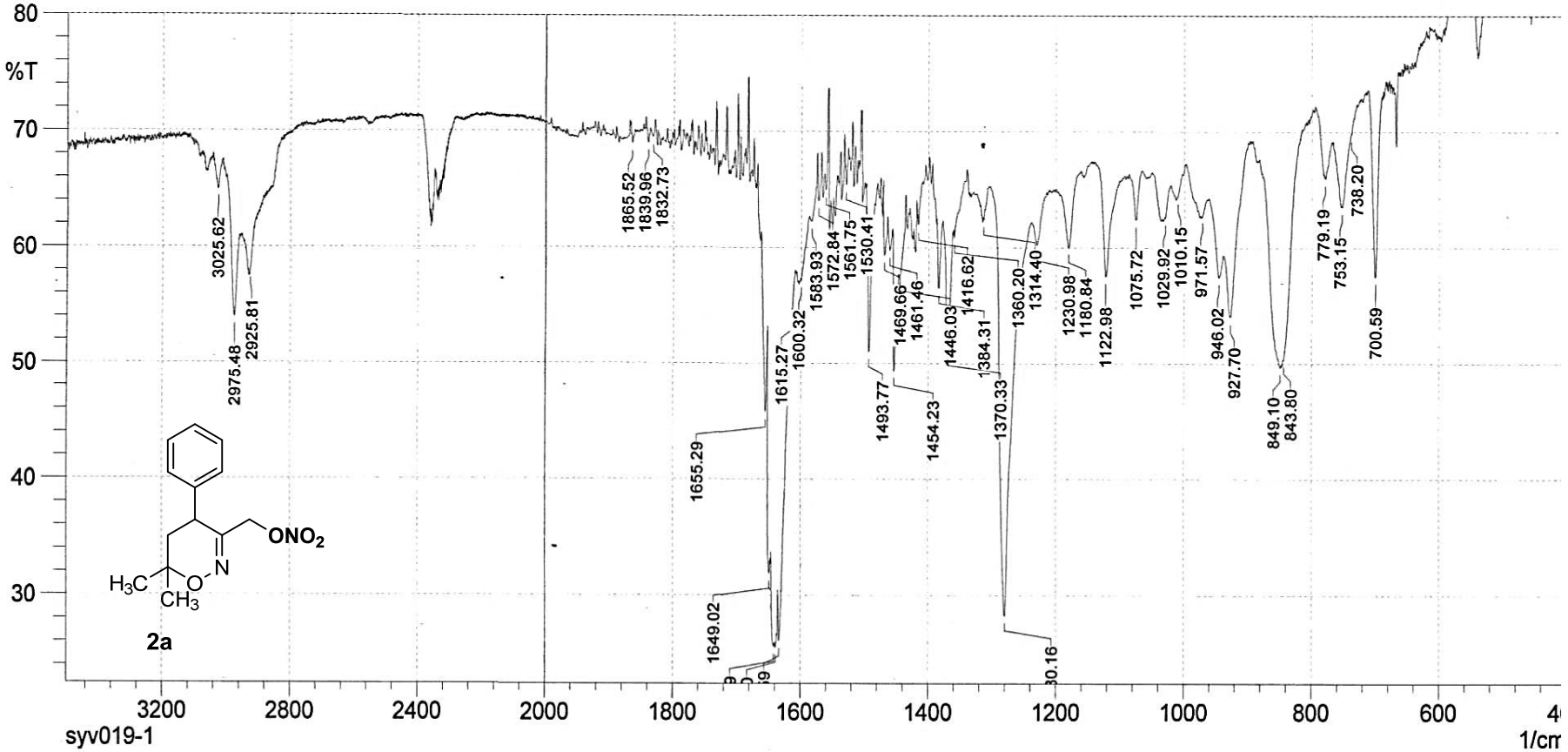
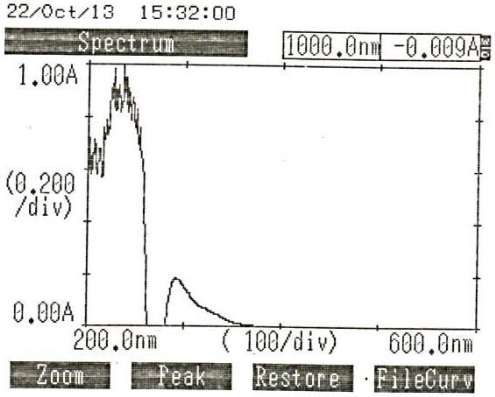
ONO2
H3C
CH3
O
N
2a
7.38
7.36
7.34
7.34
7.32
7.23
7.22
7.20
4.86
4.81
4.77
4.73
3.54
3.52
3.50
2.17
2.14
2.12
2.10
1.99
1.95
1.41
1.31
98
38
19
42
125
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
98.554
37.870
0.468
19.125
0.538
41.704
124.994


syv020-c_CARBON-6.jdf
ONO2
H3C
CH3
O
N
2a
abundance
93.85m
95.92m
1.0
0.56
0.14
0.19
0.18
0.18
0.21
0.21
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0


syv020-c_DEPT135-4.jdf
1.0
0
-1.0
abundance
0.18
0.36
0.3
-0.28
-0.36
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
ONO2
H3C
CH3
O
N
2a


%T
80
70
60
50
40
30
3025.62
2975.48
2925.81
1865.52
1839.96
1832.73
1655.29
1649.02
1615.27
1600.32
1583.93
1572.84
1561.75
1530.41
1493.77
1469.66
1461.46
1454.23
1446.03
1416.62
1384.31
1370.33
1360.20
1314.40
1230.98
1180.84
1122.98
1075.72
1029.92
1010.15
971.57
946.02
927.70
849.10
843.80
779.19
753.15
738.20
700.59
ONO2
H3C
CH3
O
N
2a
3200
2800
2400
2000
1800
1600
1400
1200
1000
800
600
syv019-1
1/cm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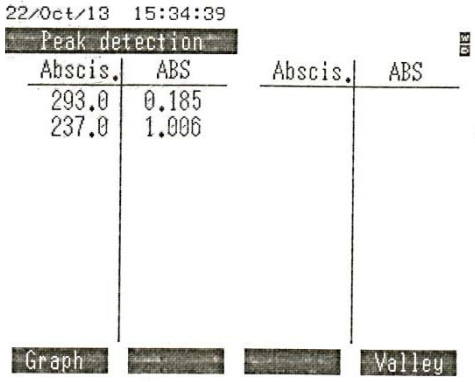
1.00A
(0.200 /div)
0.00A
200.0nm
(100/div)
600.0nm
Zoom
Peak
Restore
FileCurv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Peak detection
Abscis. ABS
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237.0 1.006
Abscis. ABS
Graph
Valley

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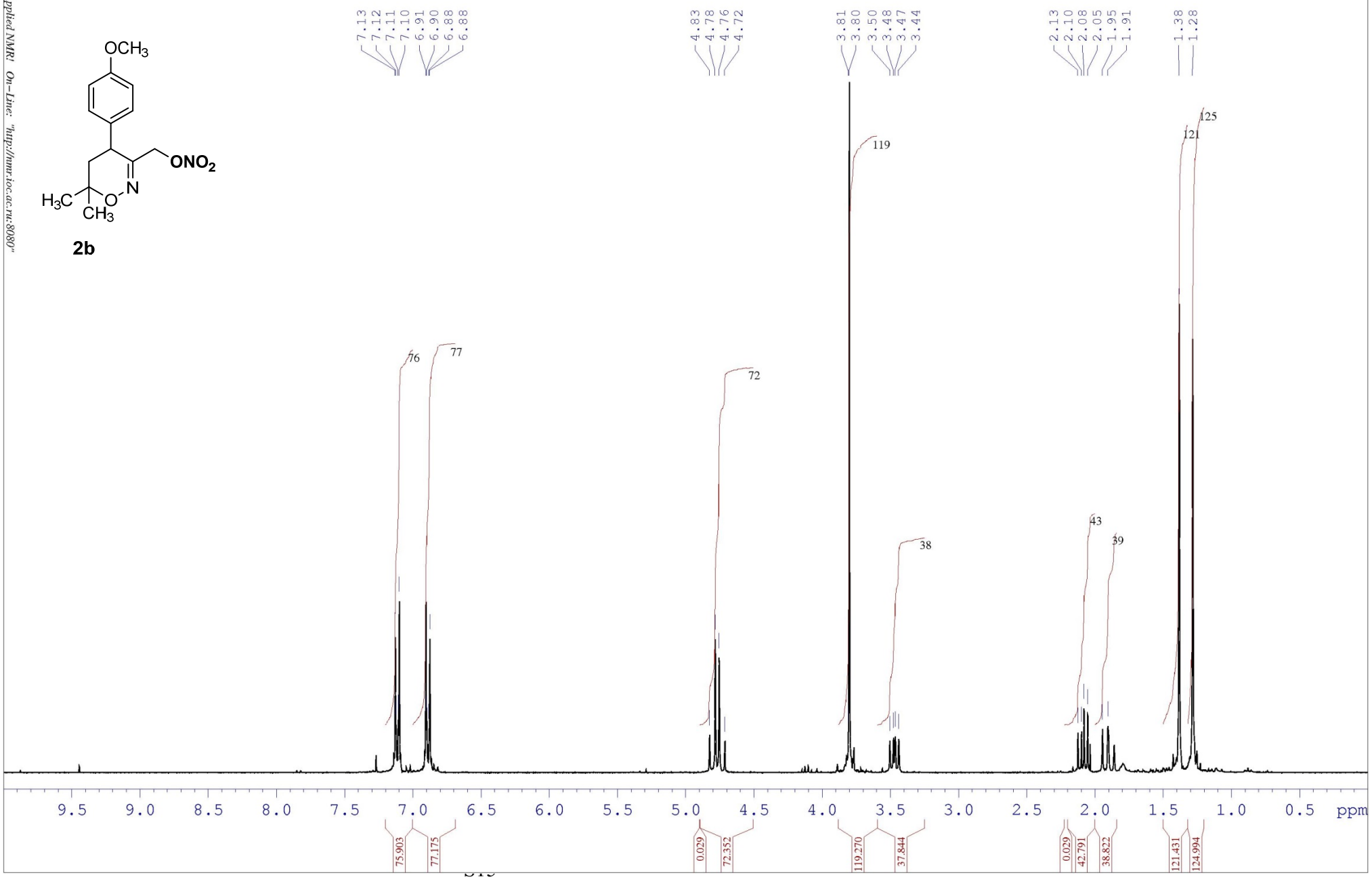

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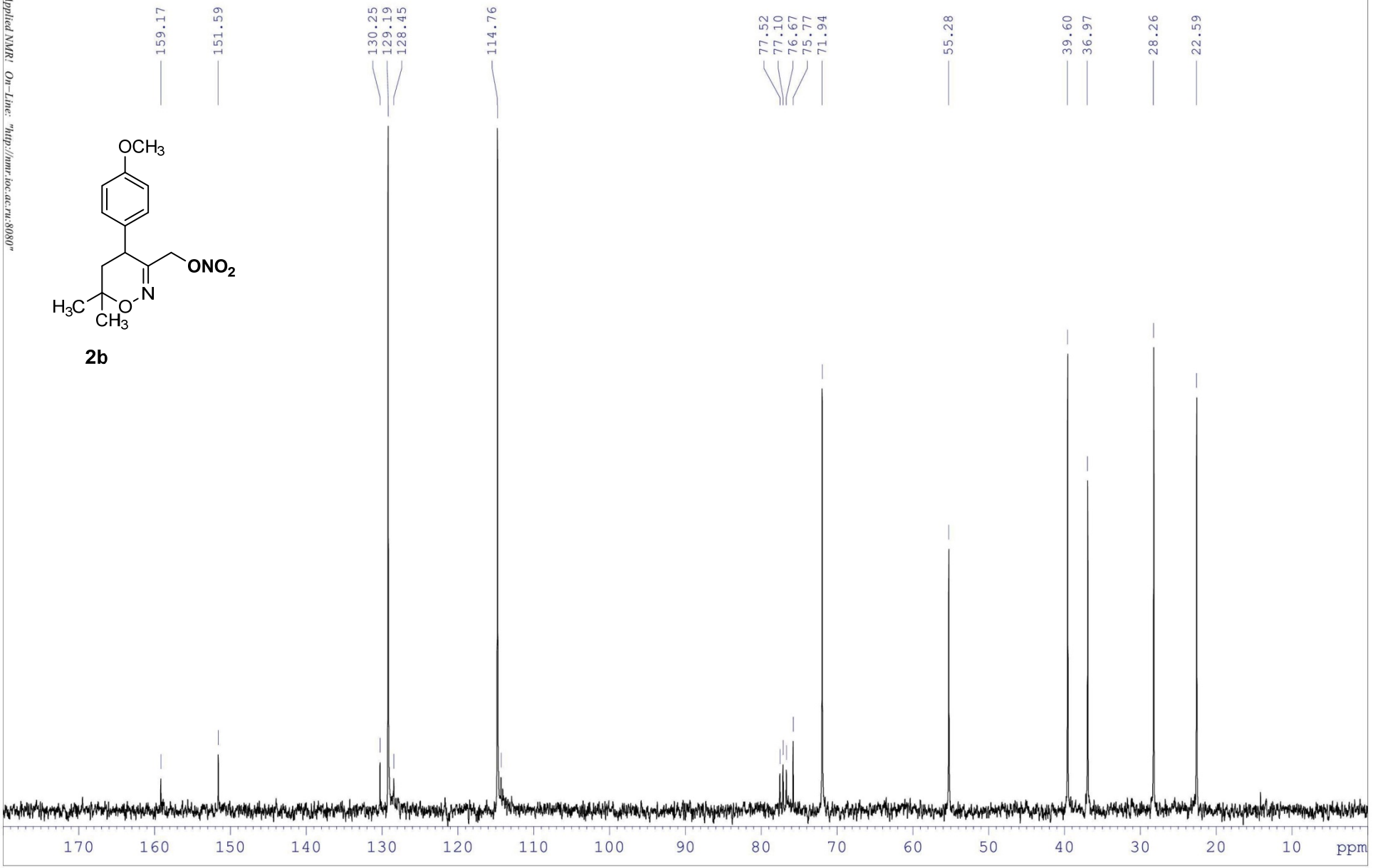

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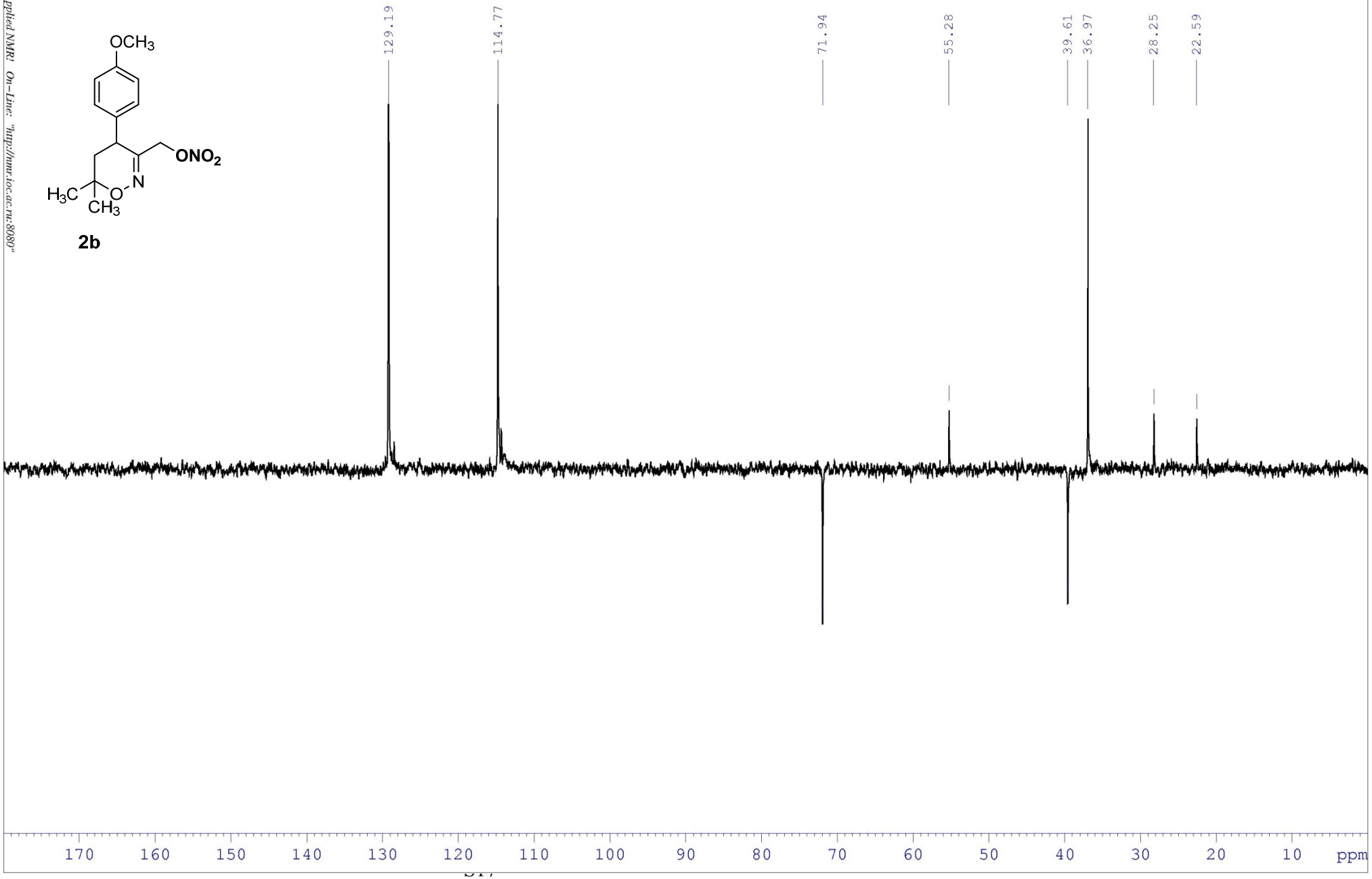

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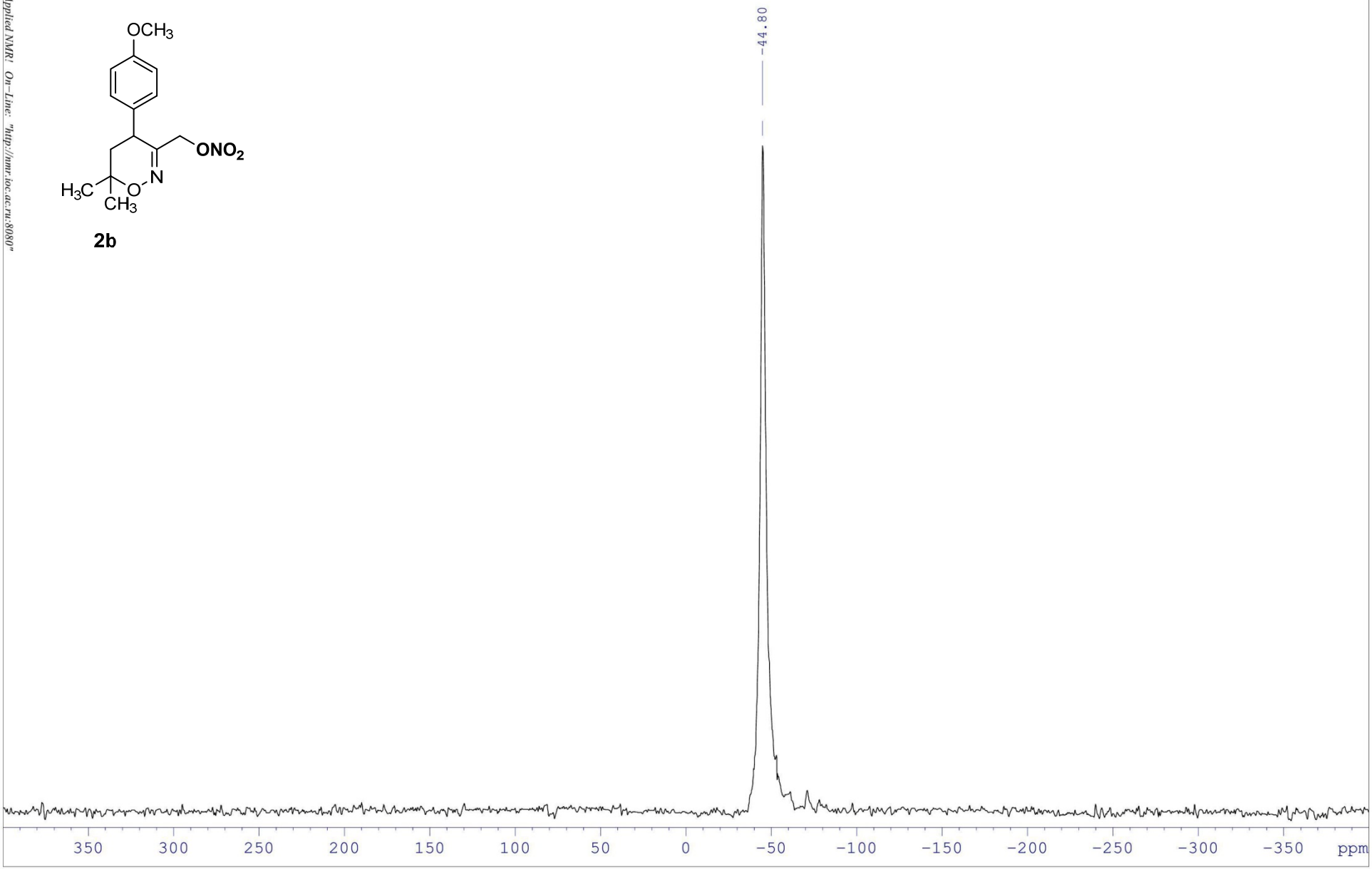

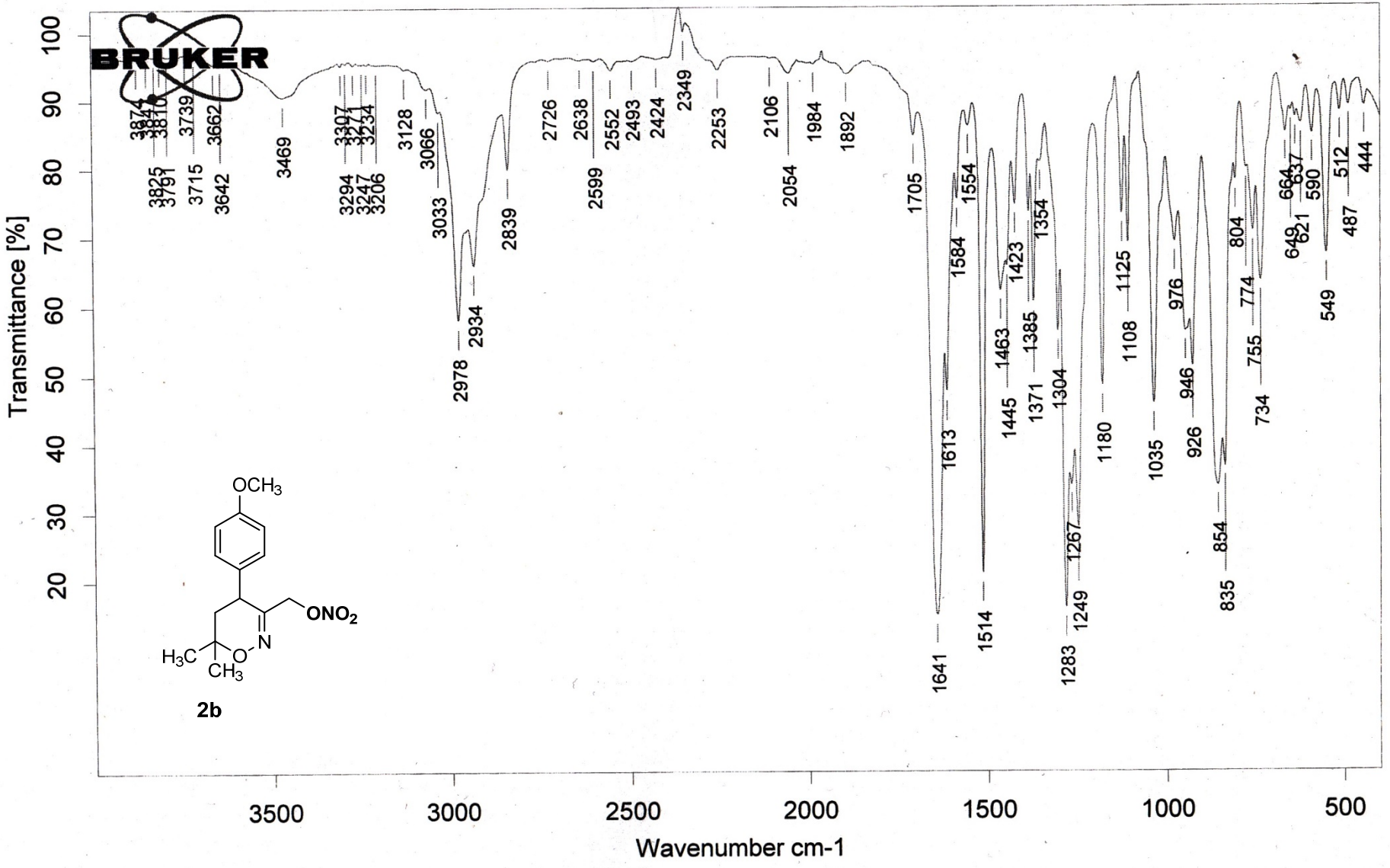

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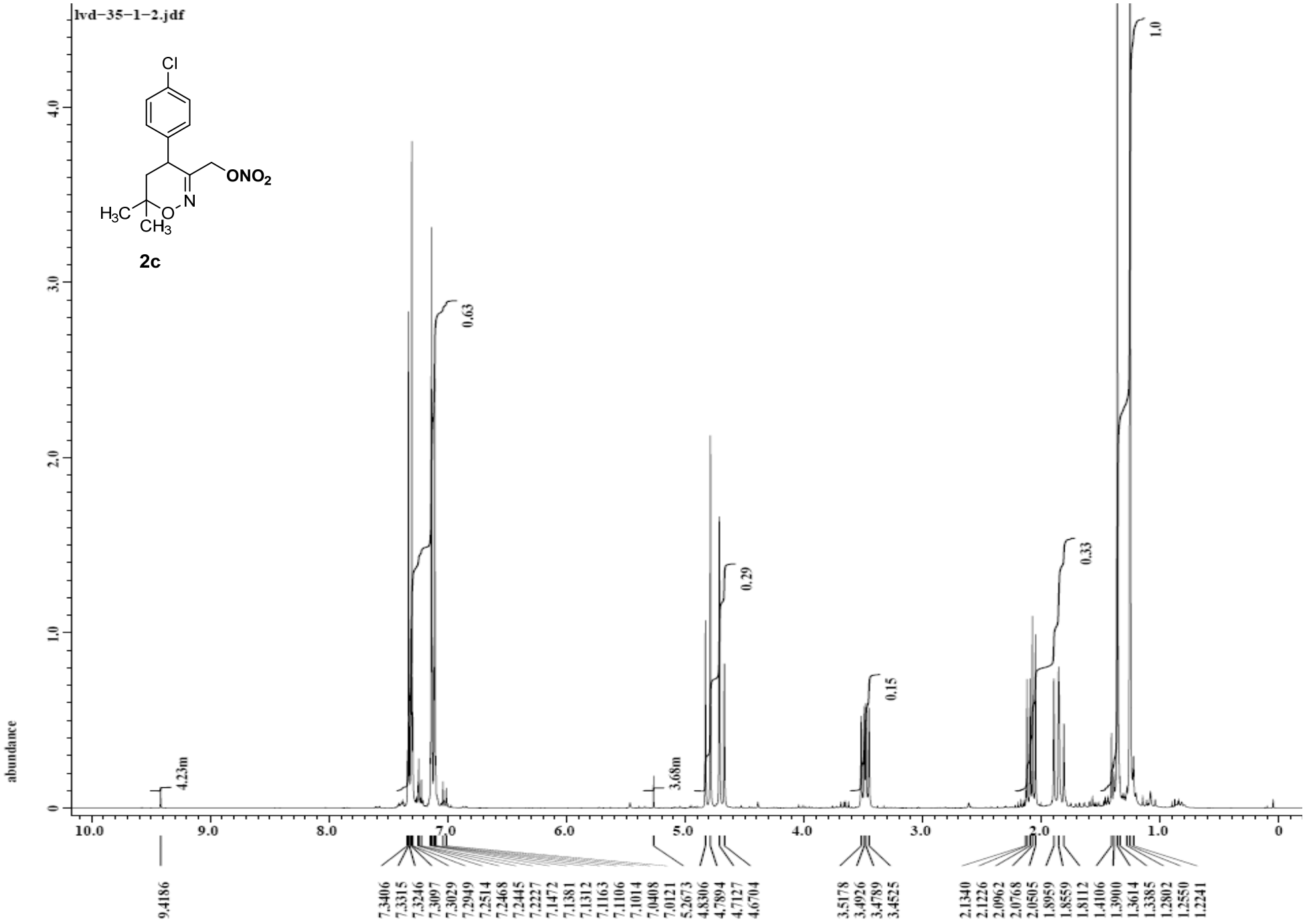
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ONO2
H3C
CH3
O
N
2c
abundance
4.23m
0.63
3.68m
0.29
0.15
0.33
1.0
9.4186
7.3406
7.3315
7.3246
7.3097
7.3029
7.2949
7.2514
7.2468
7.2445
7.2227
7.1472
7.1381
7.1312
7.1163
7.1106
7.1014
7.0408
7.0121
5.2673
4.8306
4.7894
4.7127
4.6704
3.5178
3.4926
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3.4525
2.1340
2.1226
2.0962
2.0768
2.0505
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1.3900
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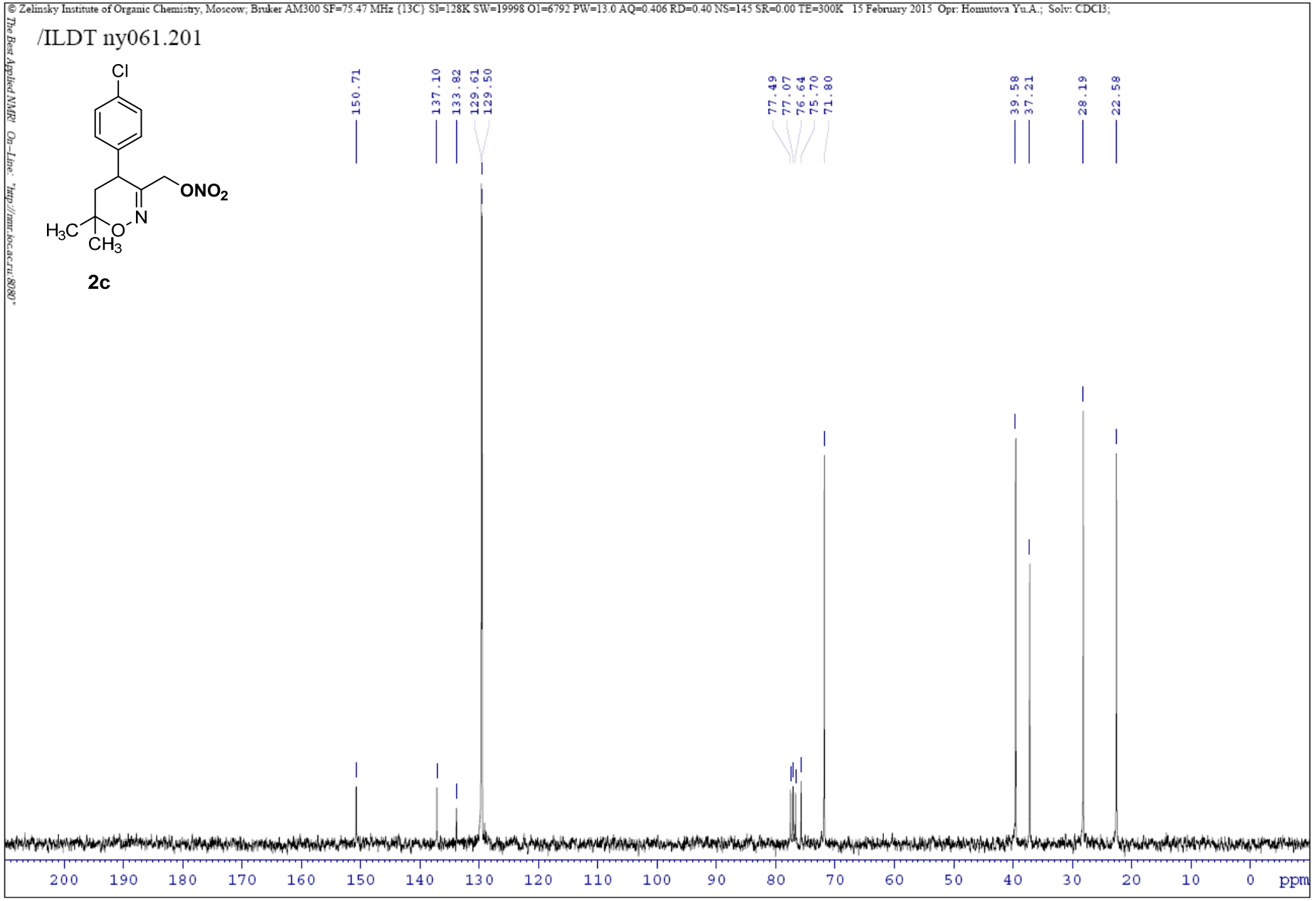
1.2550
1.2241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/ILDT ny061.201
Cl
ONO2
N
H3C
O
CH3
2c
150.71
137.10
133.82
129.61
129.50
77.49
77.07
76.64
75.70
71.80
39.58
37.21
28.19
22.58
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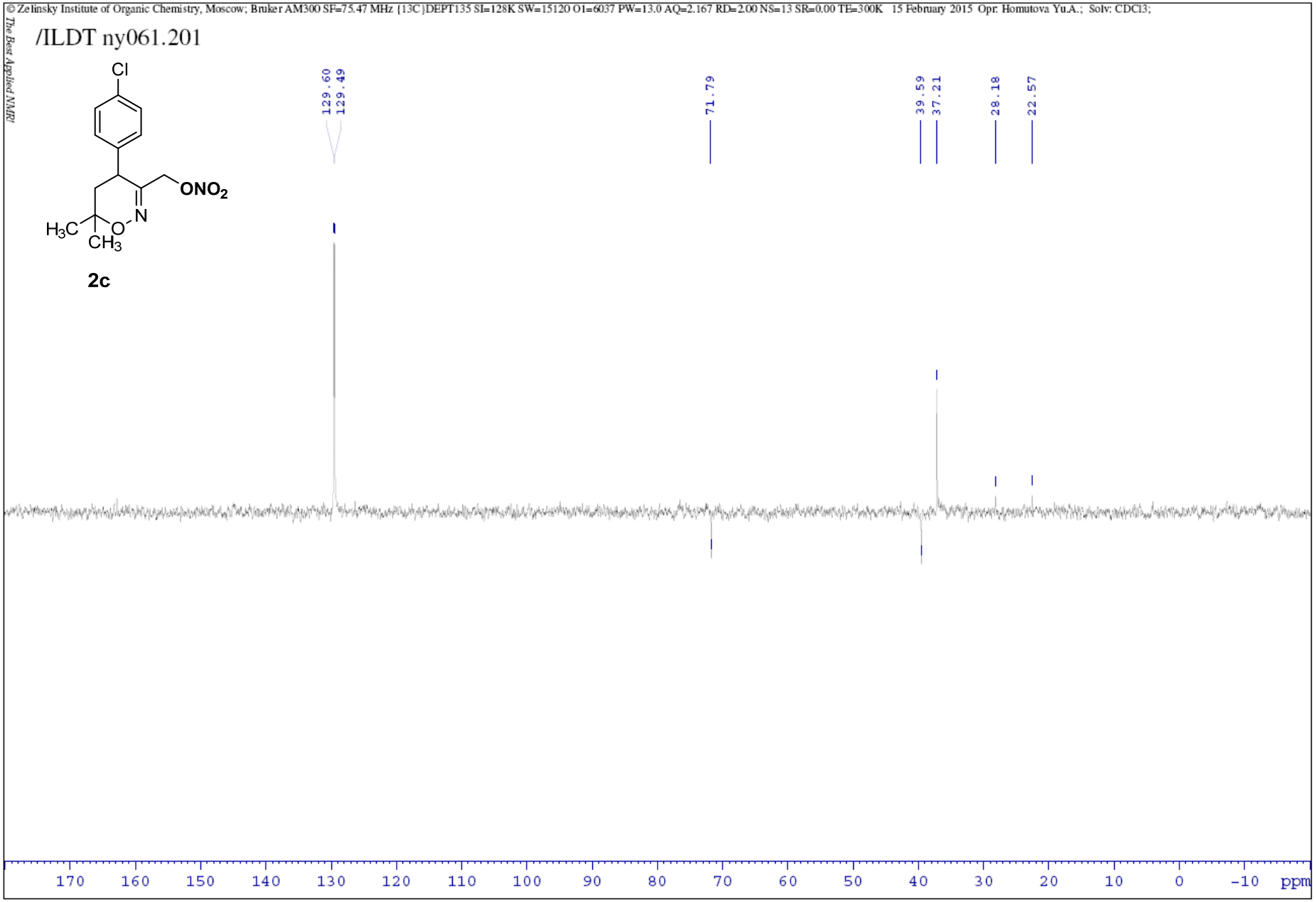
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Cl
ONO2
H3C
O
N
CH3
2c
129.60
129.49
71.79
39.59
37.21
28.18
22.57
170
160
150
140
130
120
110
100
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80
70
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40
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20
10
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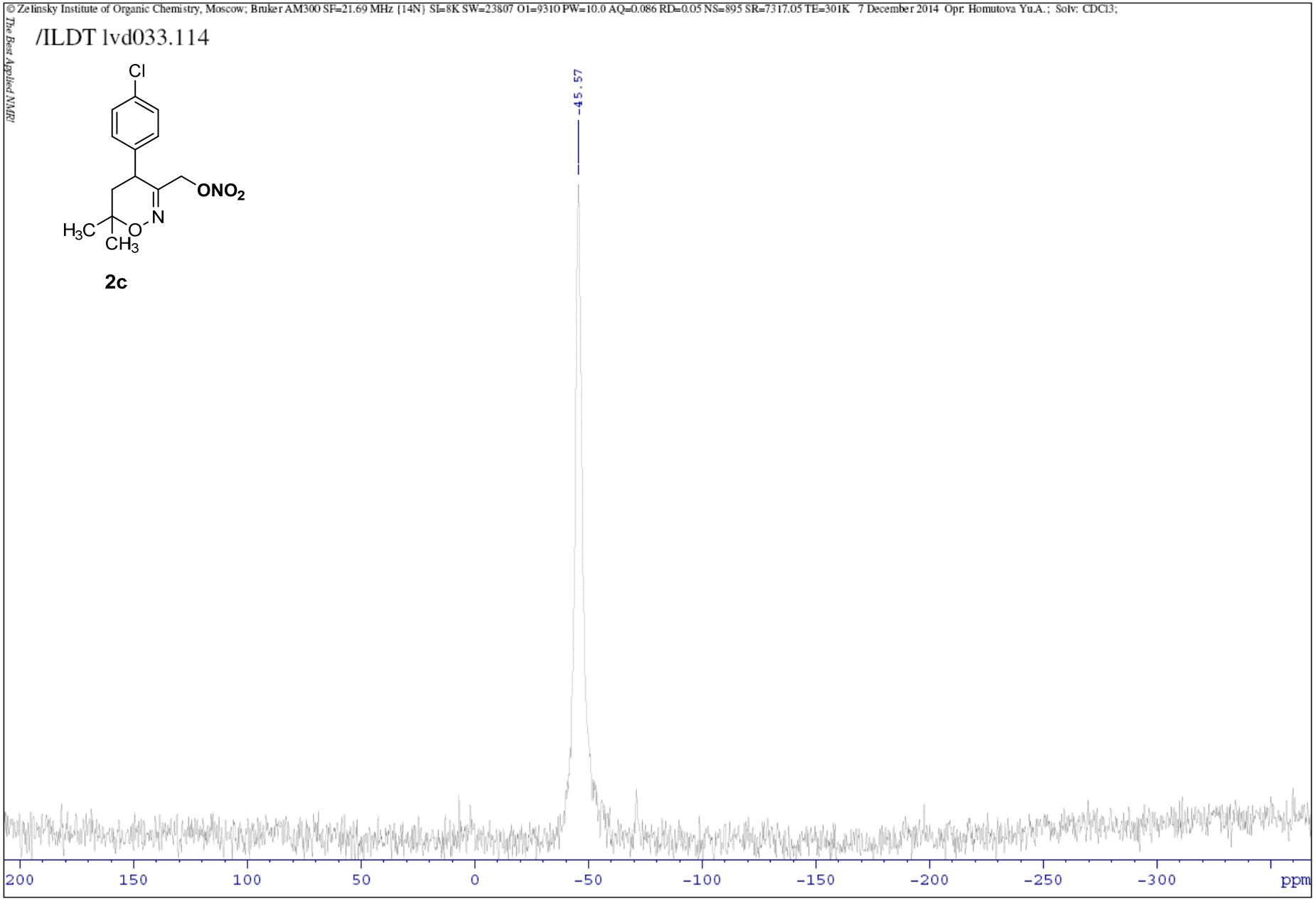
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H3C
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CH3
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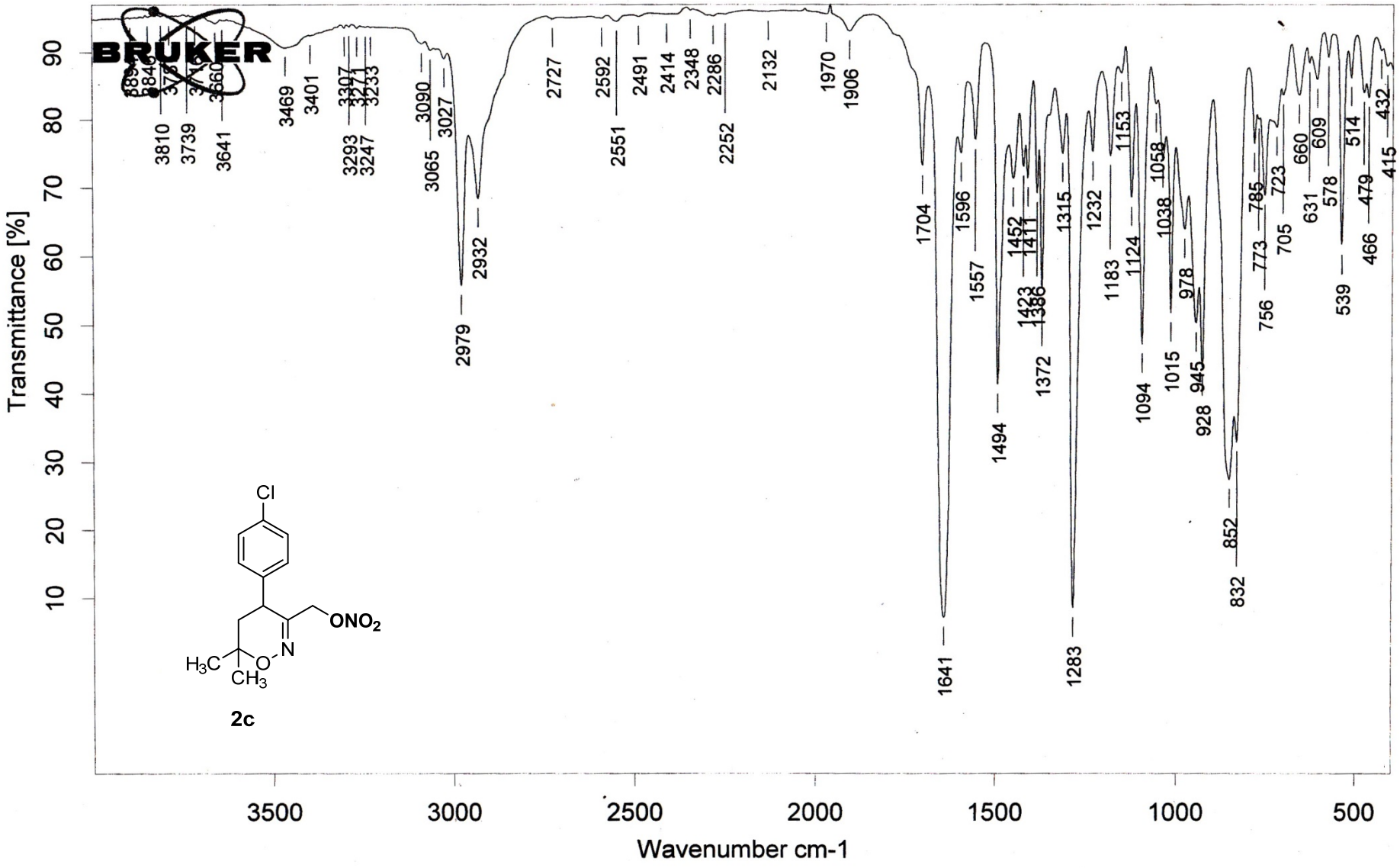
-200
-250
-300
ppm


BRUKER
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2500
2000
1500
1000
500
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50
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70
80
90
3810
3739
3641
3469
3401
3307
3293
3271
3247
3233
3090
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832
785
773
756
723
705
660
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609
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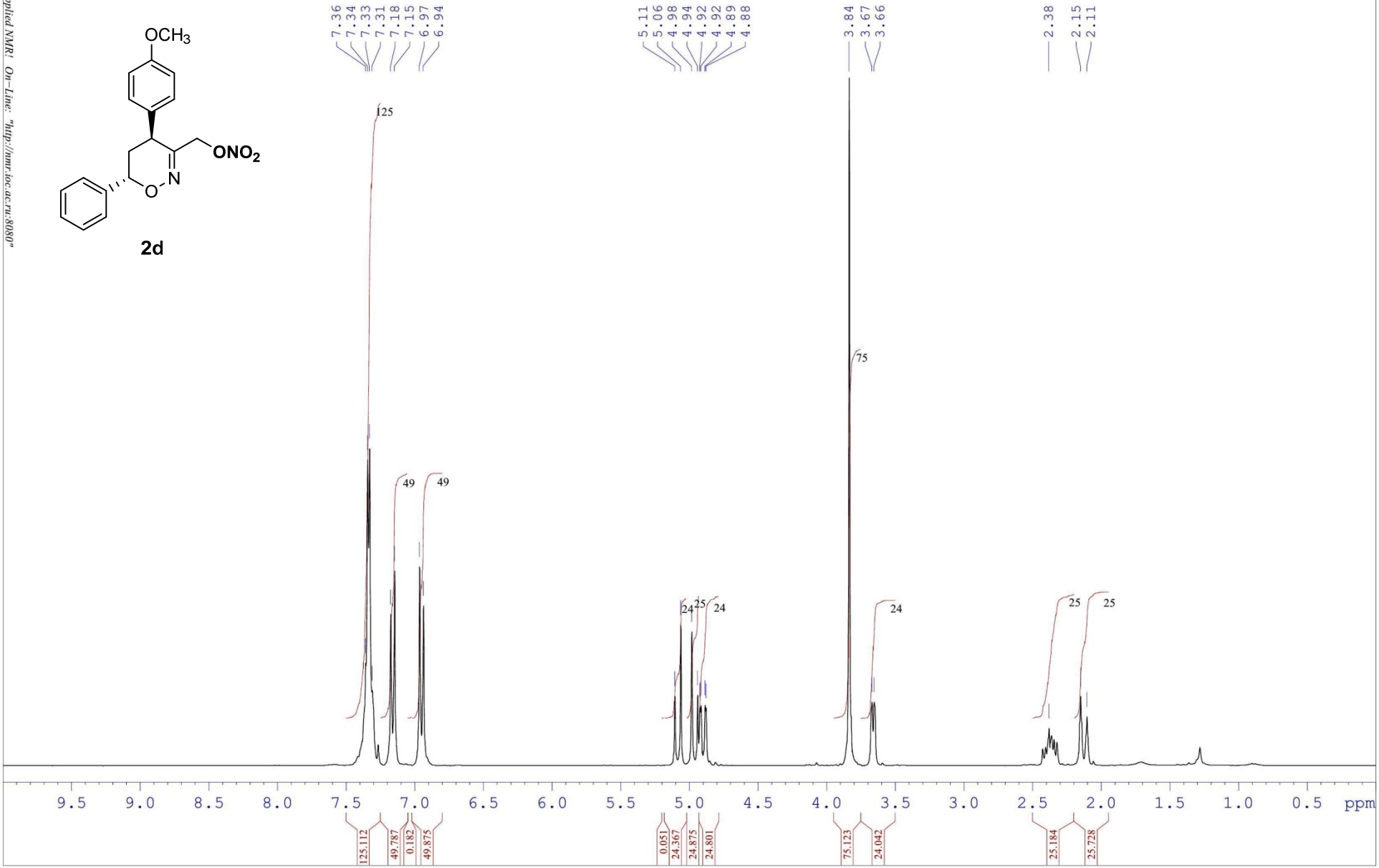
2d



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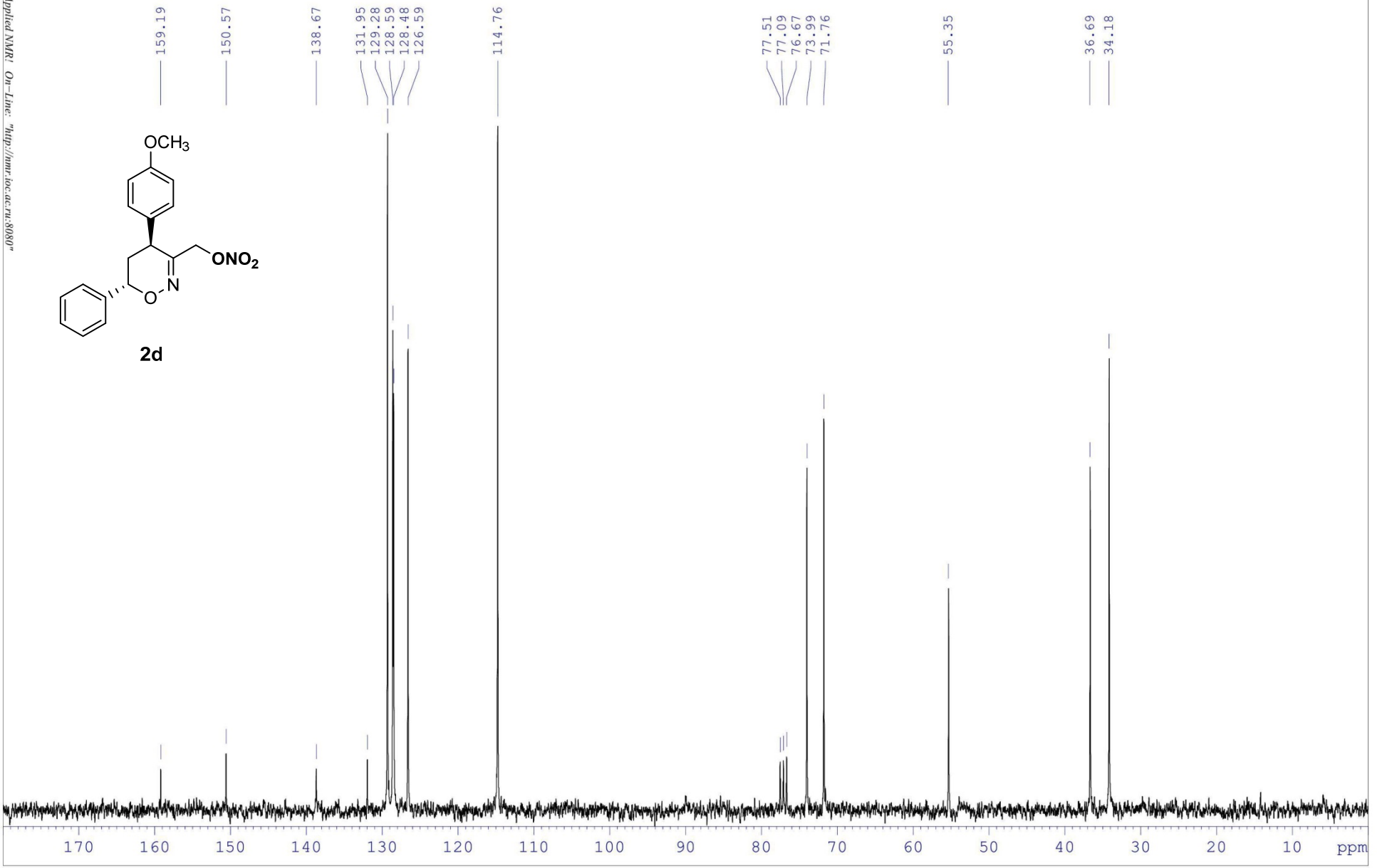

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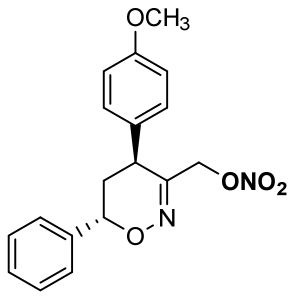


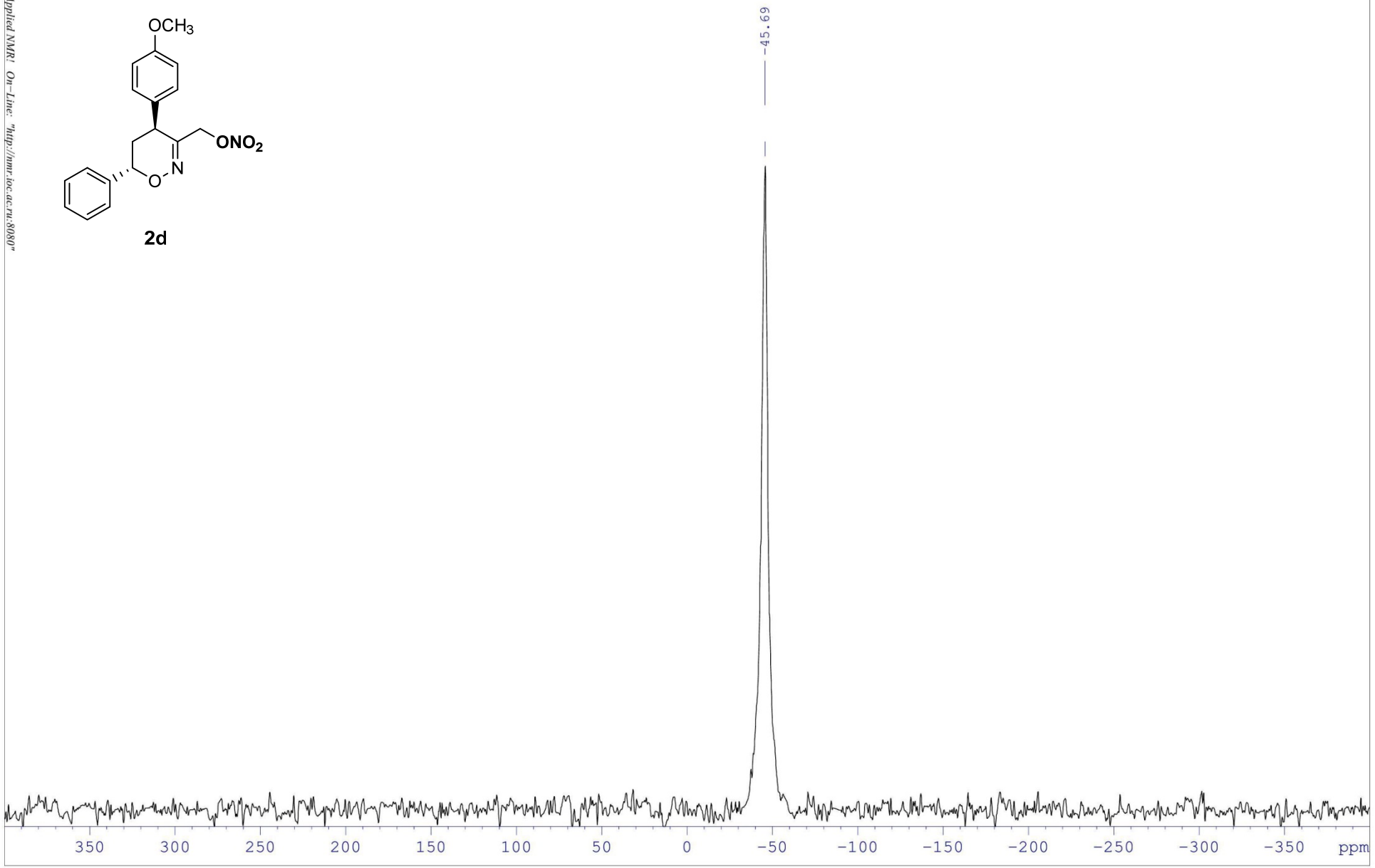
2d

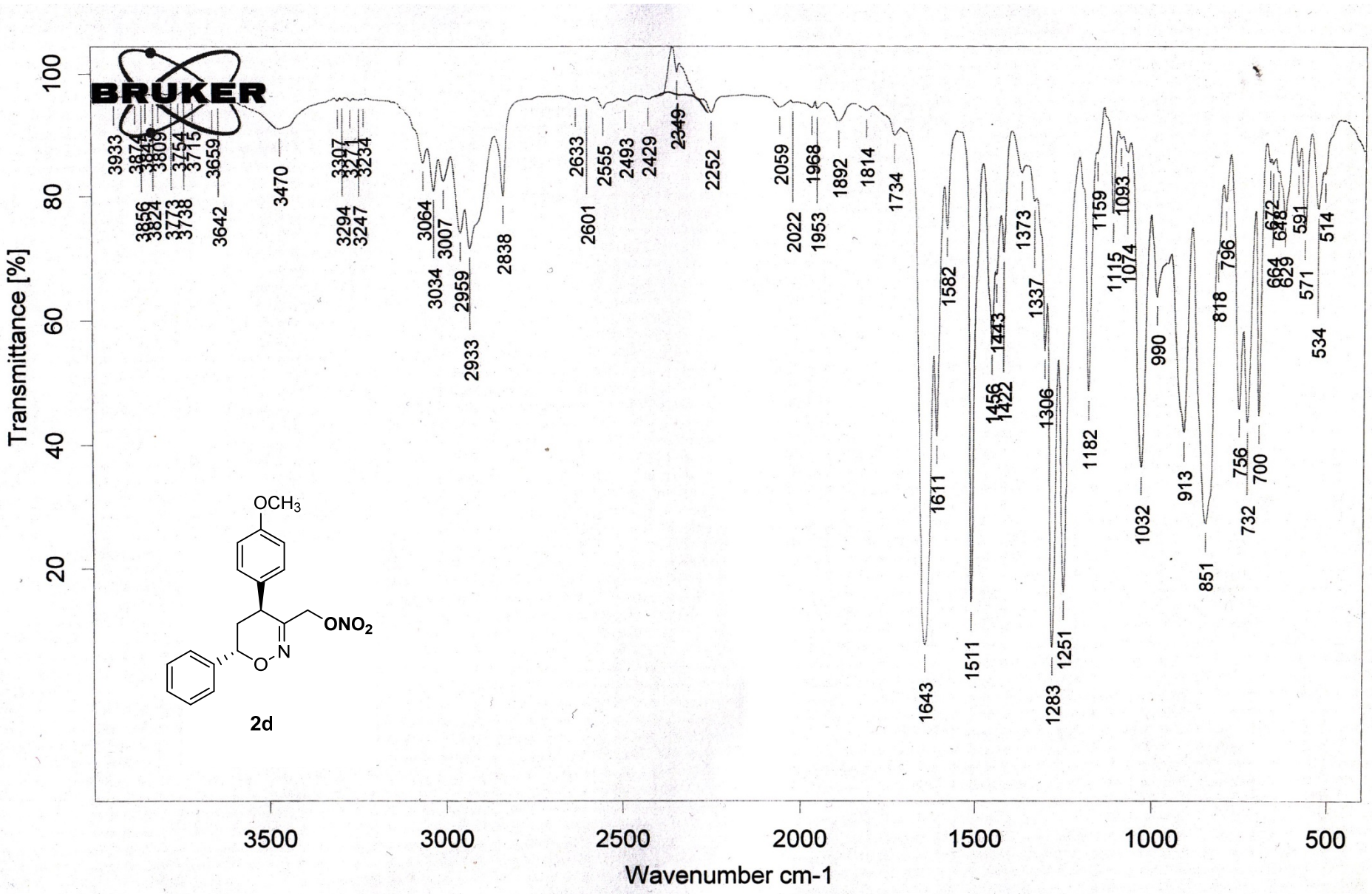
129.28
128.59
128.47
126.58
114.76
73.99
71.76
55.35
36.69
34.19

170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

/ILDT ny040.601

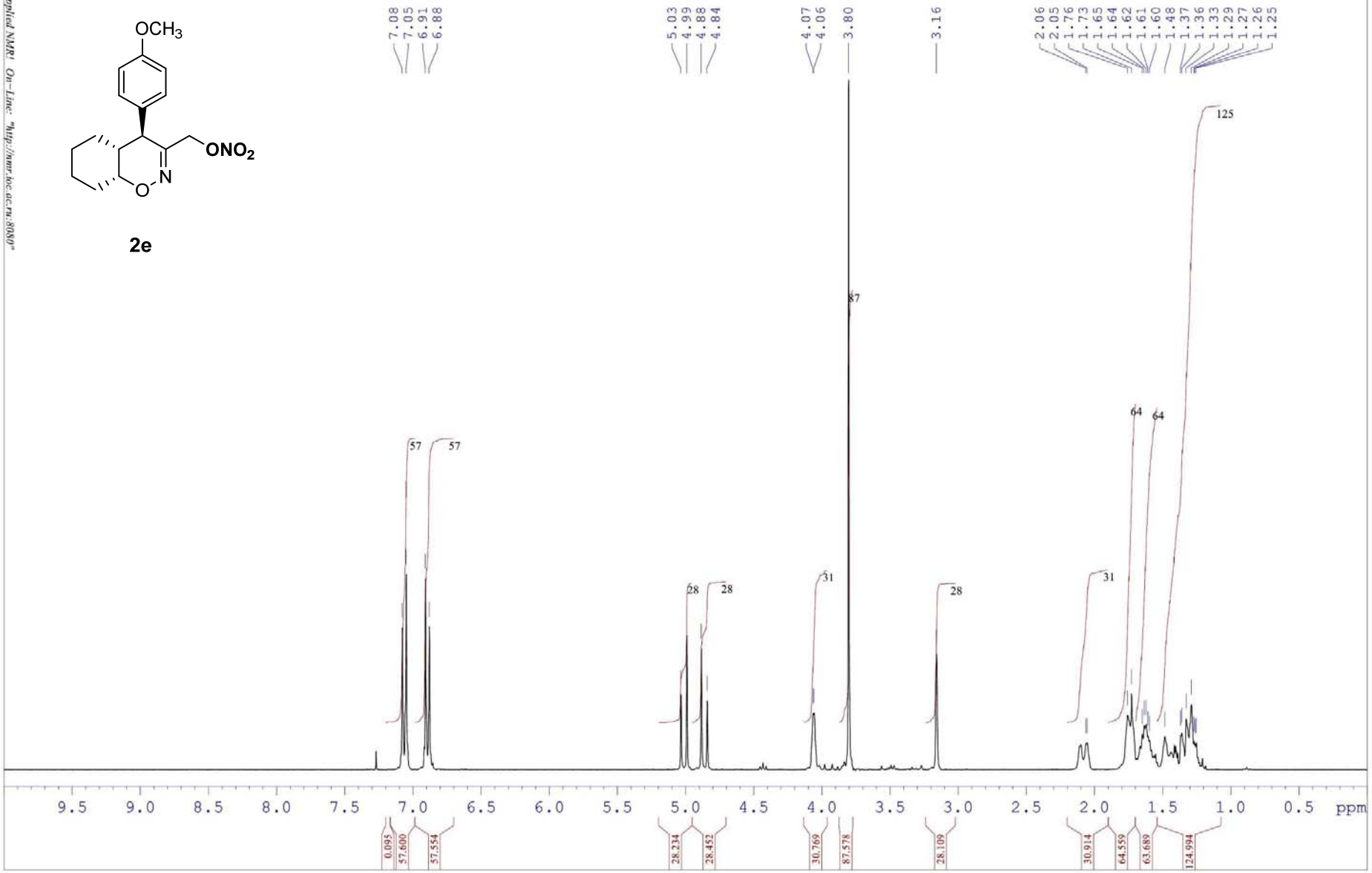
2d




BRUKER
Transmittance [%]
Wavenumber cm-1
100
80
60
40
20
3500
3000
2500
2000
1500
1000
500
3933
3874
3856
3824
3809
3773
3754
3738
3715
3659
3642
3470
3307
3294
3271
3247
3234
3064
3034
3007
2959
2933
2838
2633
2601
2555
2493
2429
2349
2252
2059
2022
1968
1953
1892
1814
1734
1643
1611
1582
1511
1456
1443
1422
1373
1337
1306
1283
1251
1182
1159
1115
1093
1074
1032
990
913
851
818
796
756
732
700
672
664
648
629
591
571
534
514
OCH3
ONO2
N
O
2d
D:\EDL\NY-040.0
NY-040 ,
KBr.
14.10.2014

/ILDT ny036.302

2e



/ILDT ny036.301

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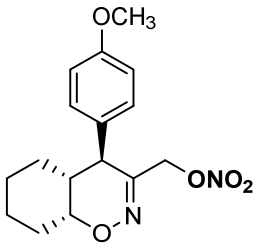

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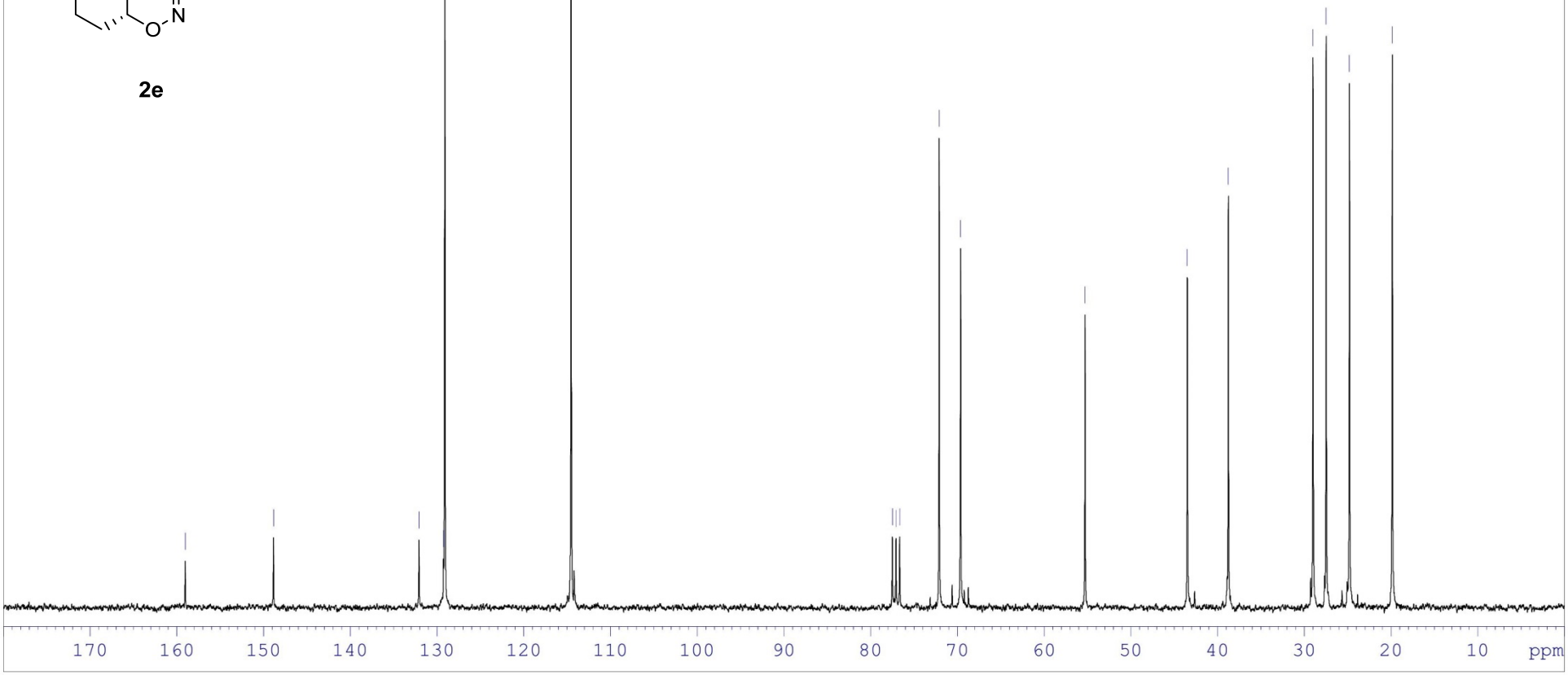

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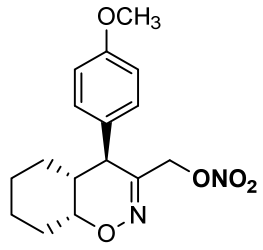


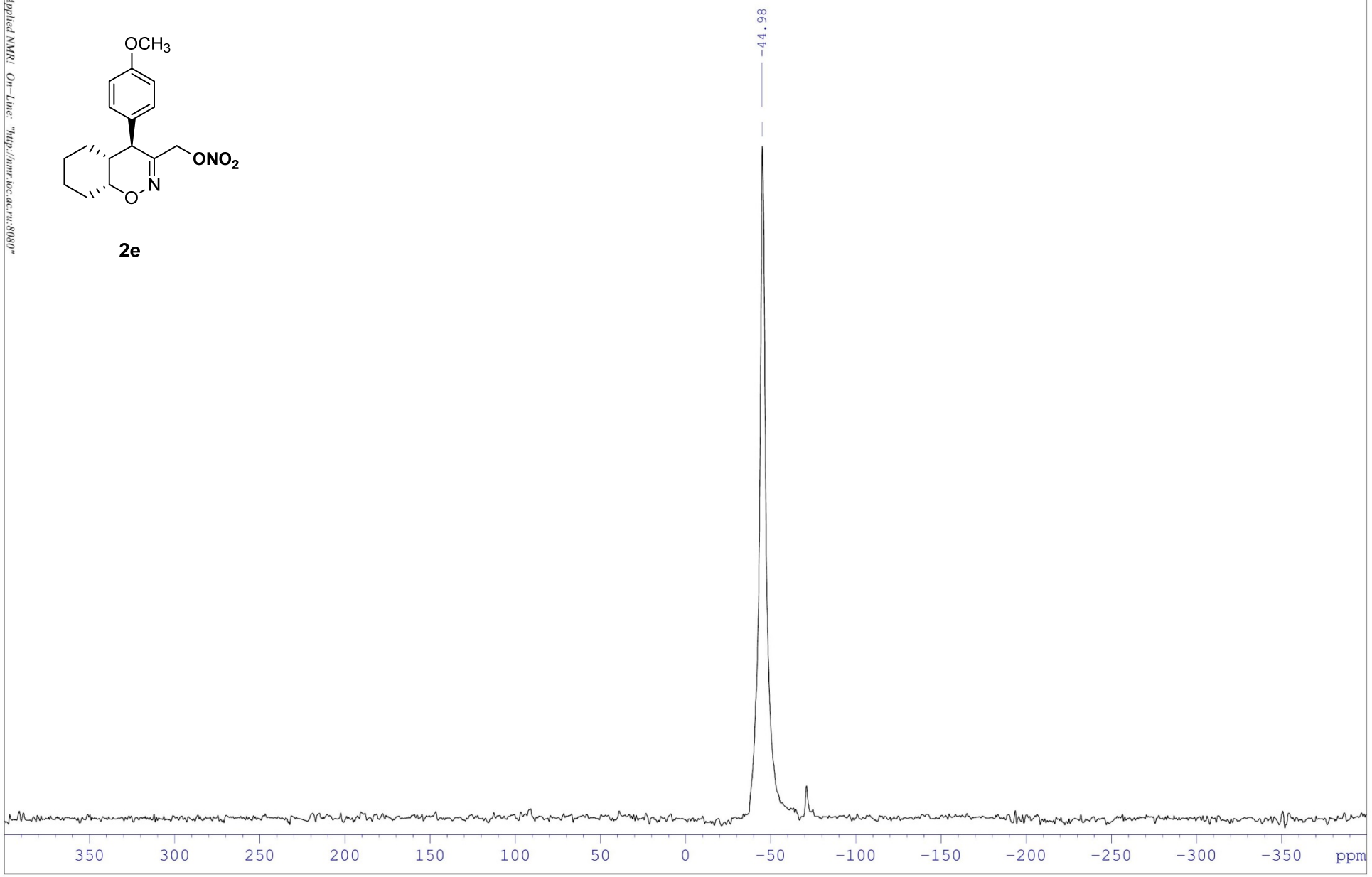
2e

129.08 114.52 72.08 69.66 55.31 43.51 38.78 29.02 27.50 24.81 19.87

170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

/ILDT ny036.301

2e



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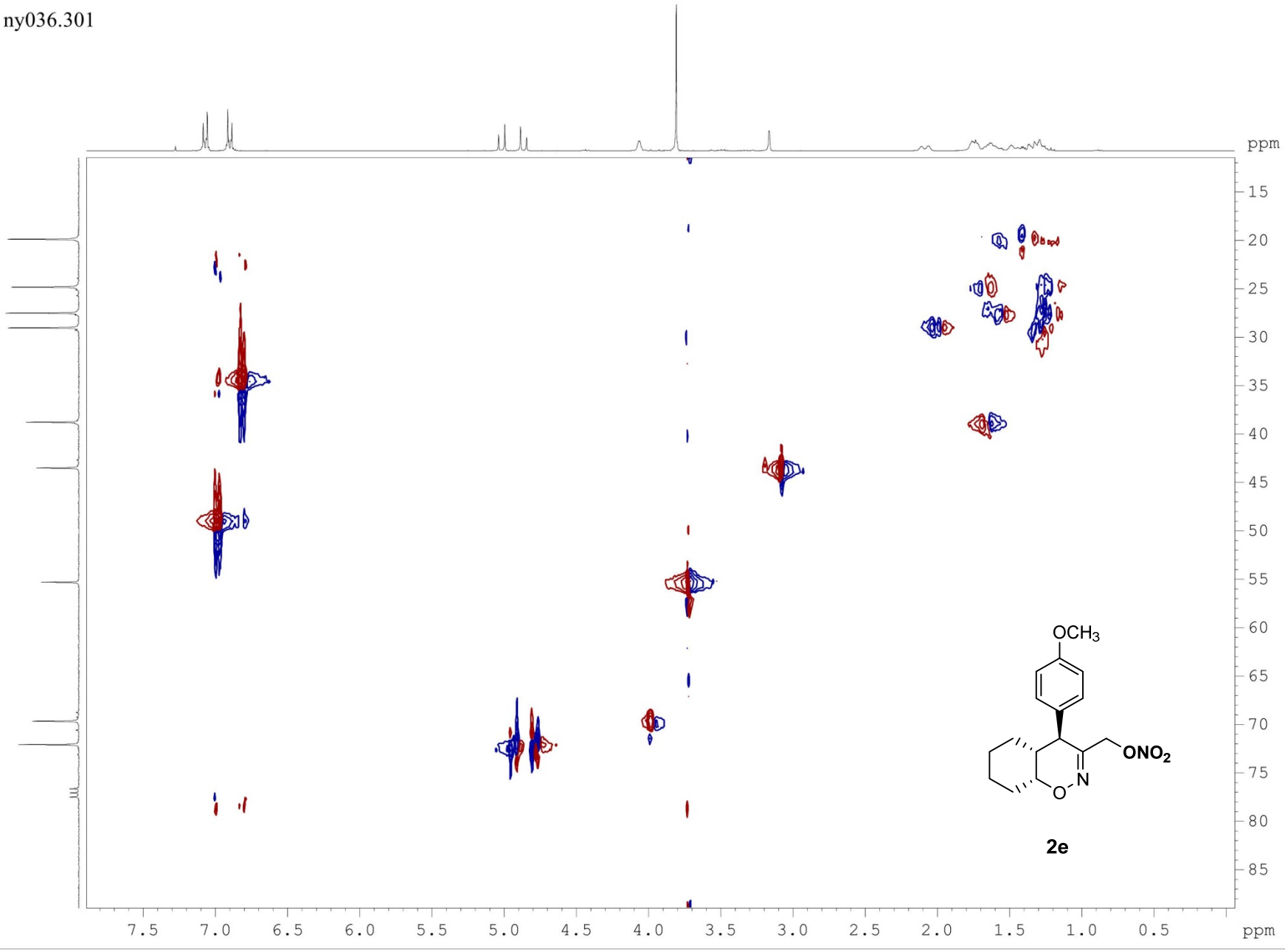

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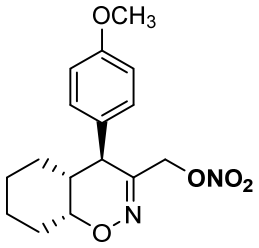

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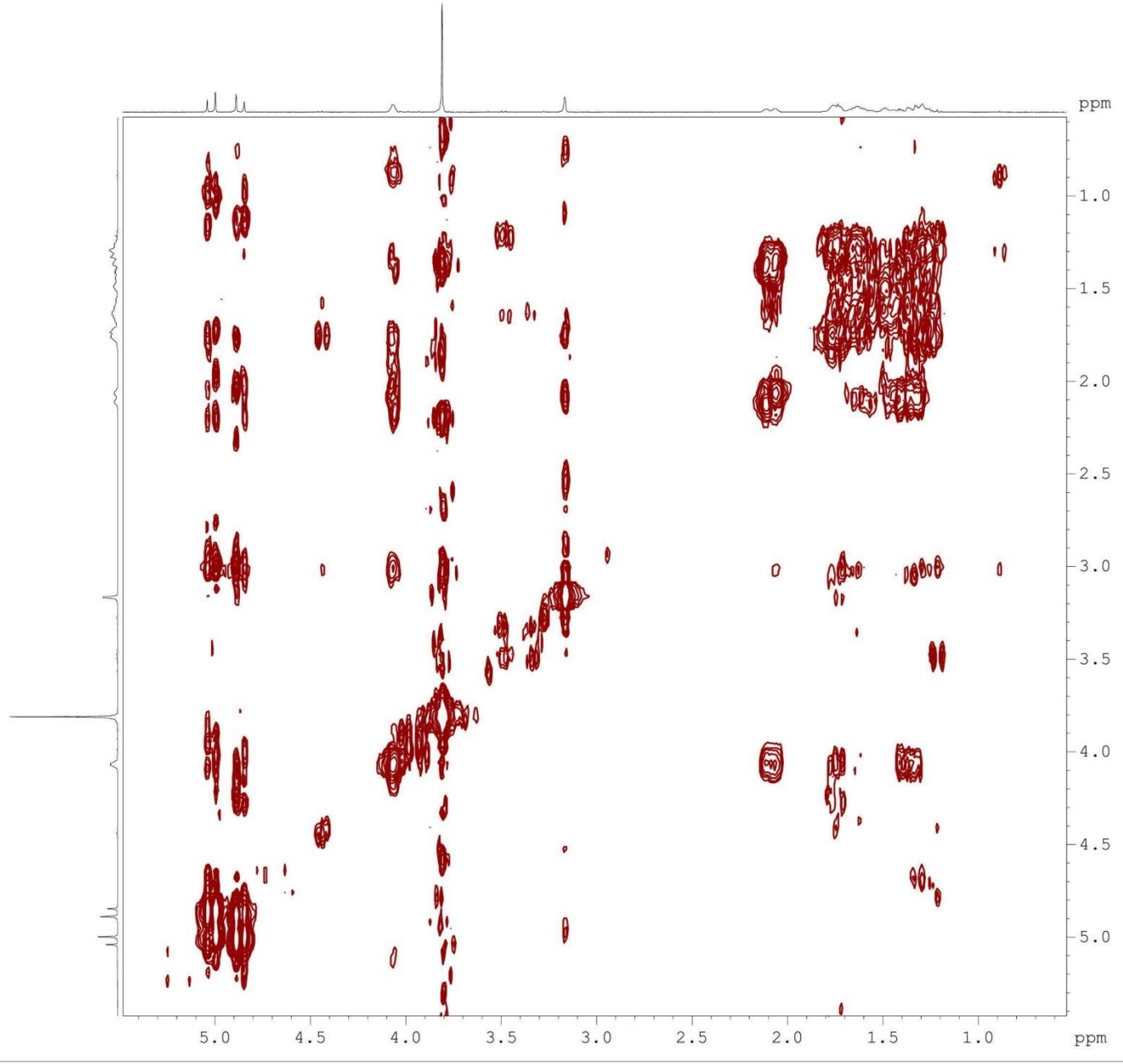
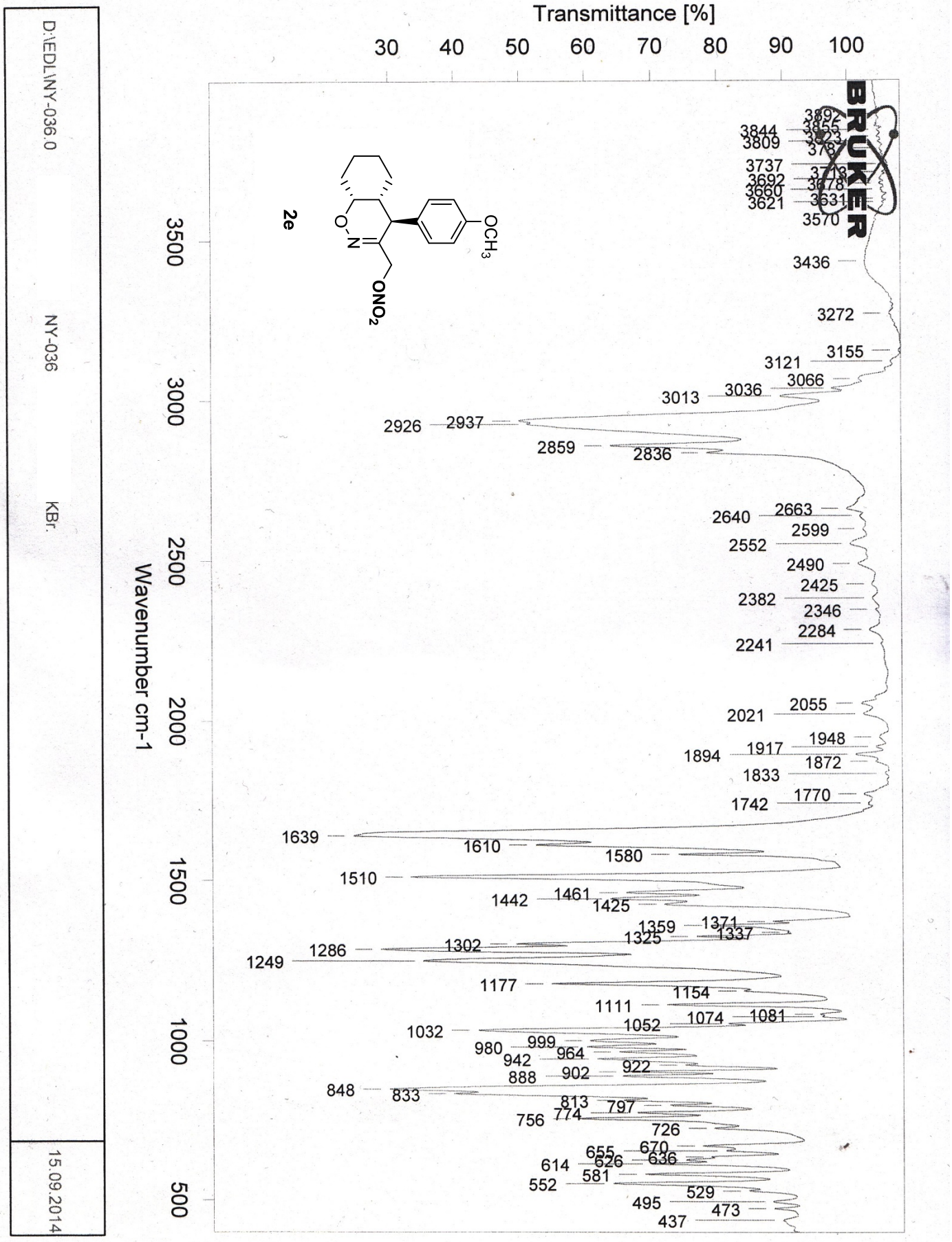
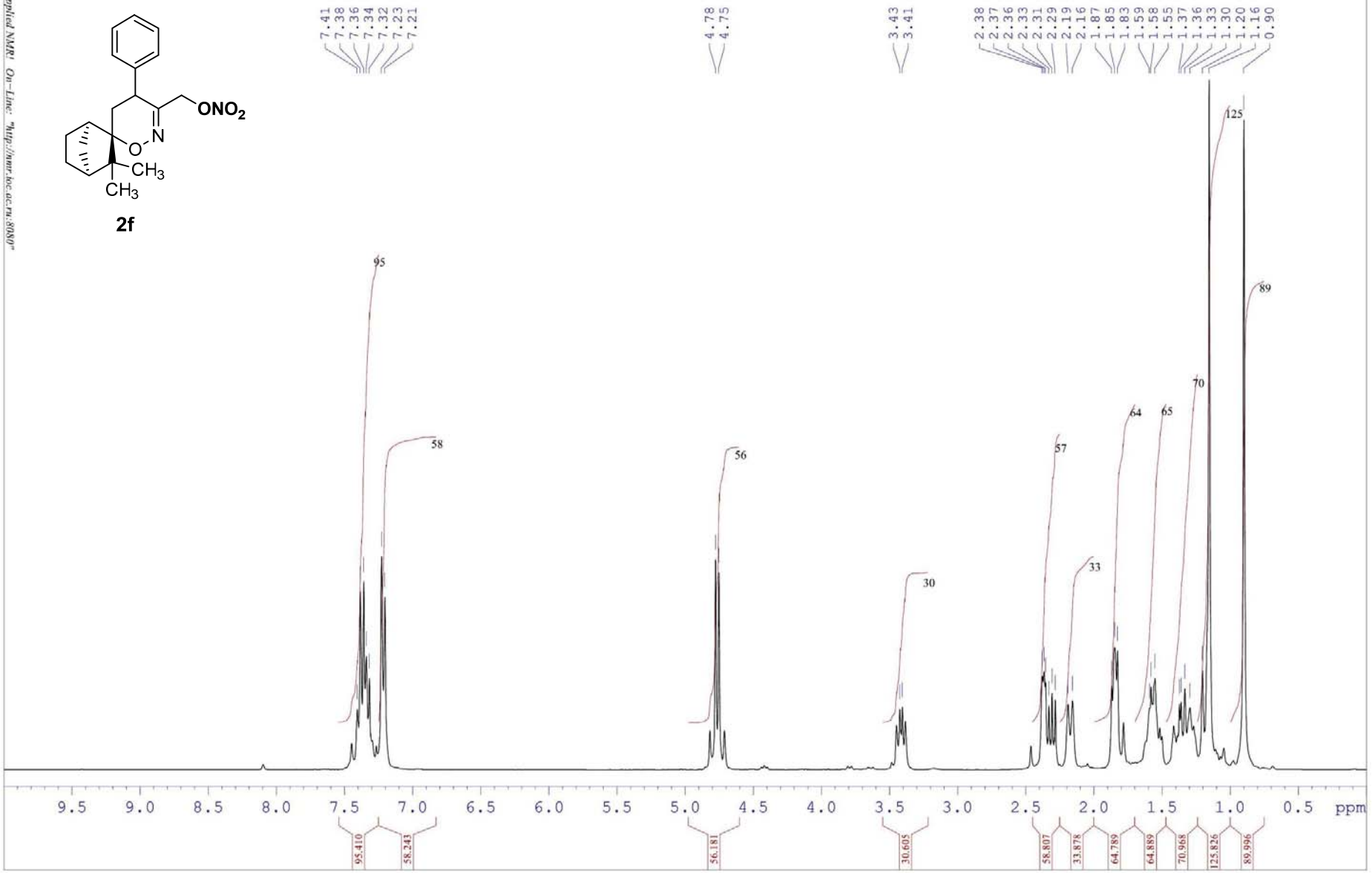



D:\EDL\NY-036.0
NY-036
KBr
15.09.2014
Transmittance [%]
30 40 50 60 70 80 90 100
Wavenumber cm-1
3500 3000 2500 2000 1500 1000 500
2e
OCH3
ONO2
BRUKER
3892
3844
3809
3737
3692
3660
3621
3570
3436
3272
3155
3121
3066
3036
3013
2937
2926
2859
2836
2663
2640
2599
2552
2490
2425
2382
2346
2284
2241
2055
2021
1948
1917
1894
1872
1833
1770
1742
1639
1610
1580
1510
1461
1442
1425
1371
1359
1337
1325
1302
1286
1249
1177
1154
1111
1081
1074
1052
1032
999
980
964
942
922
902
888
848
833
813
797
774
756
726
670
655
636
626
614
581
552
529
495
473
437

/ILDT NY042.501

2f



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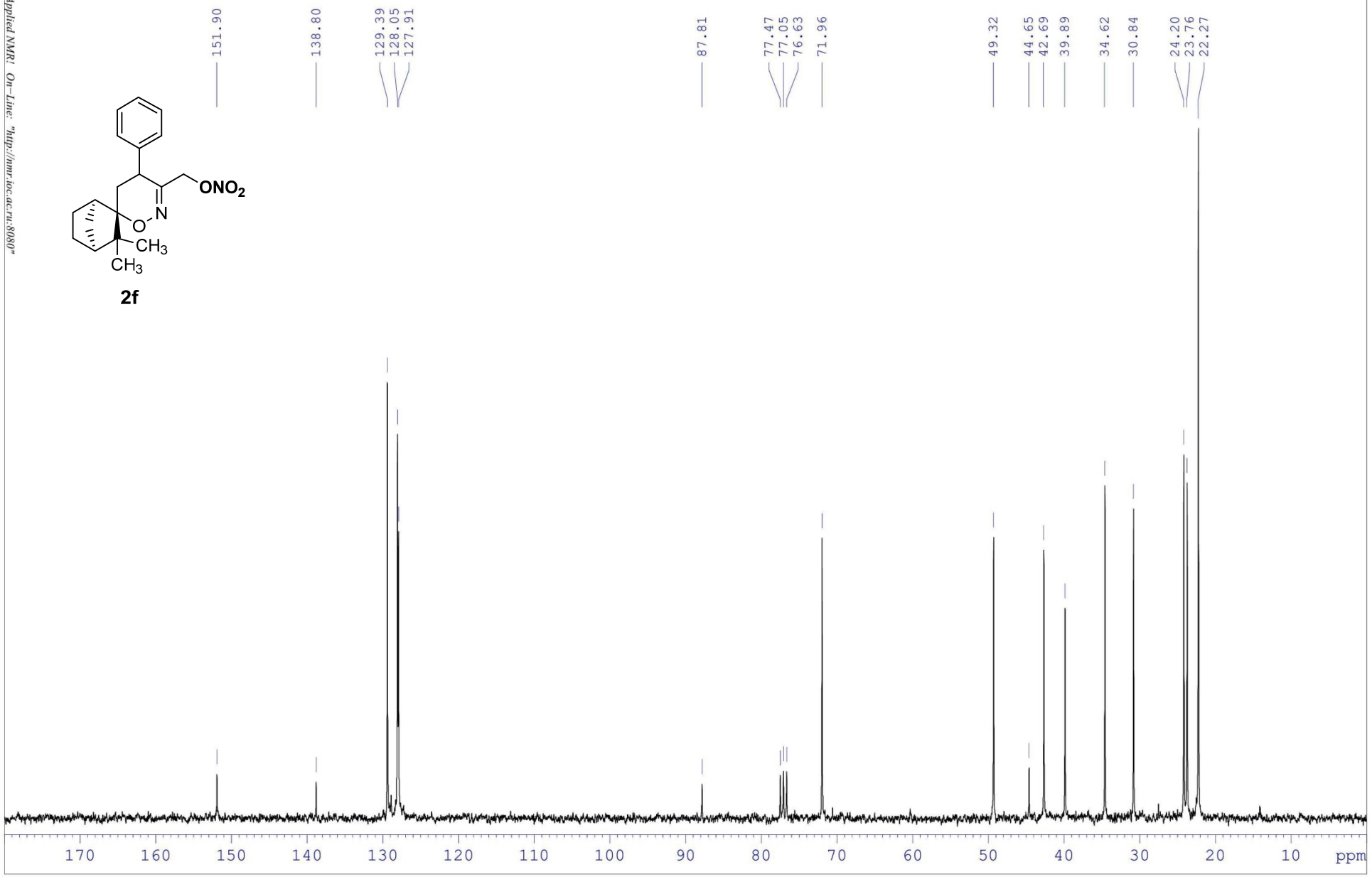
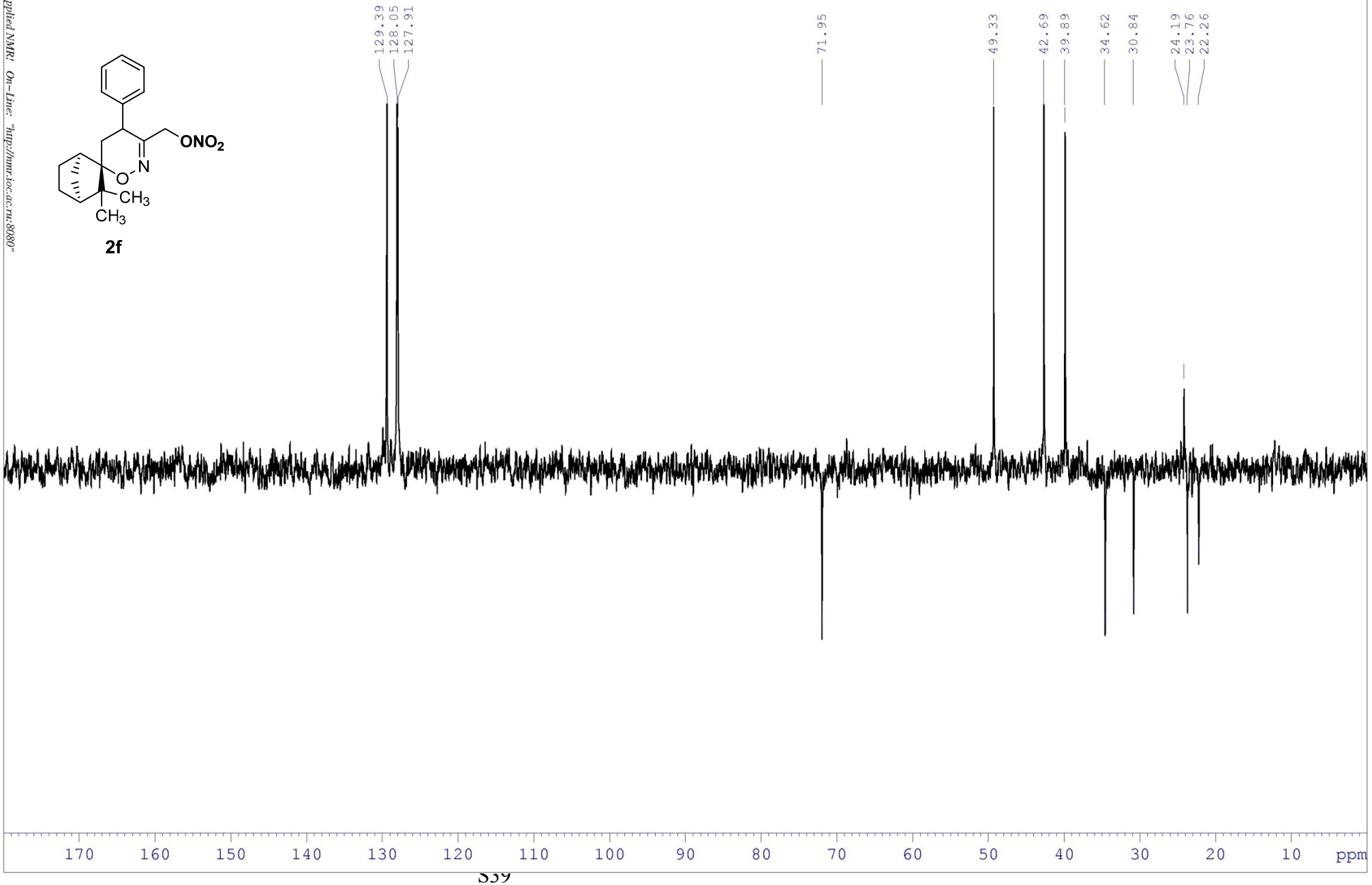


/ILDT ny042.201

2f



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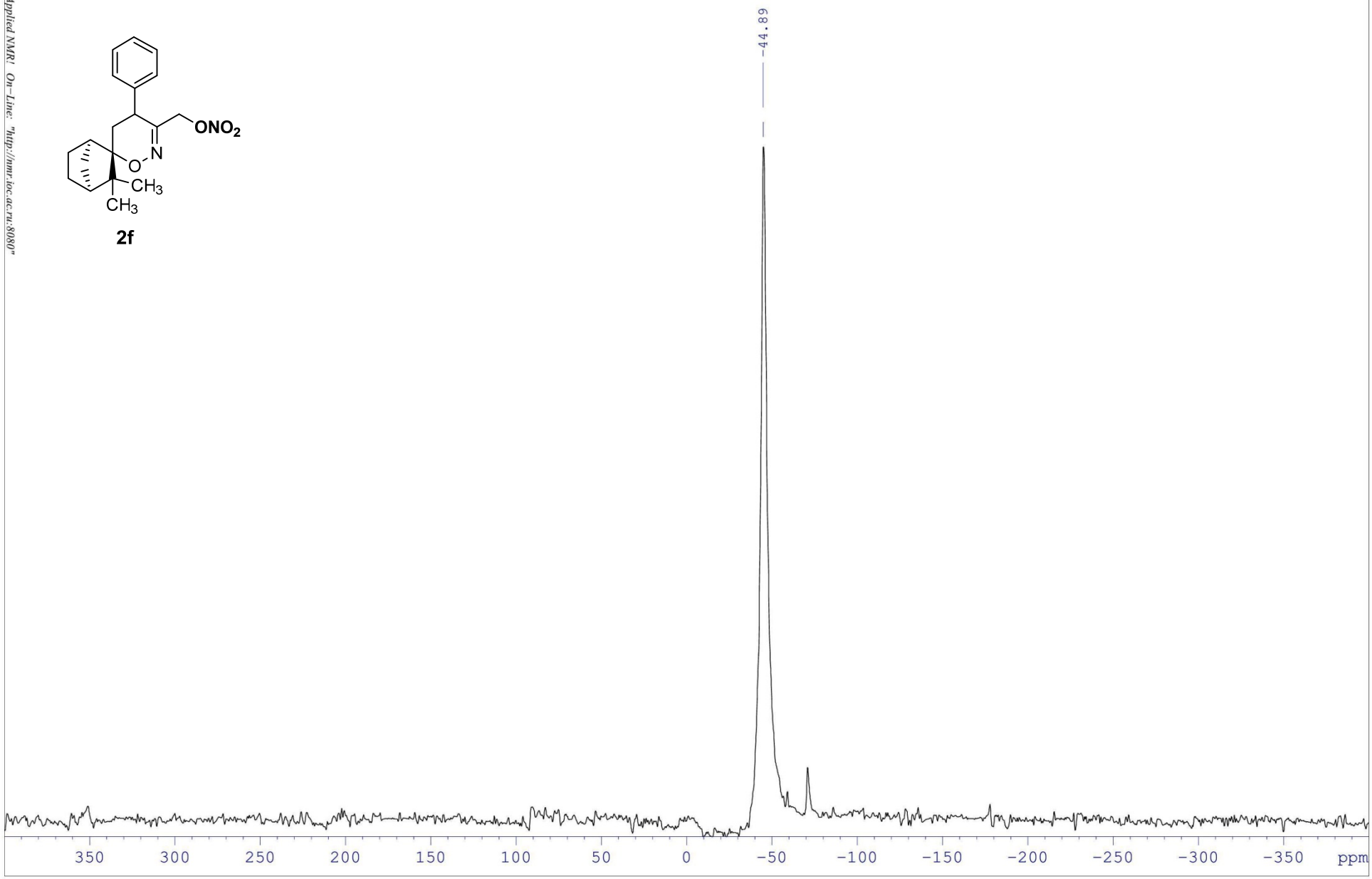
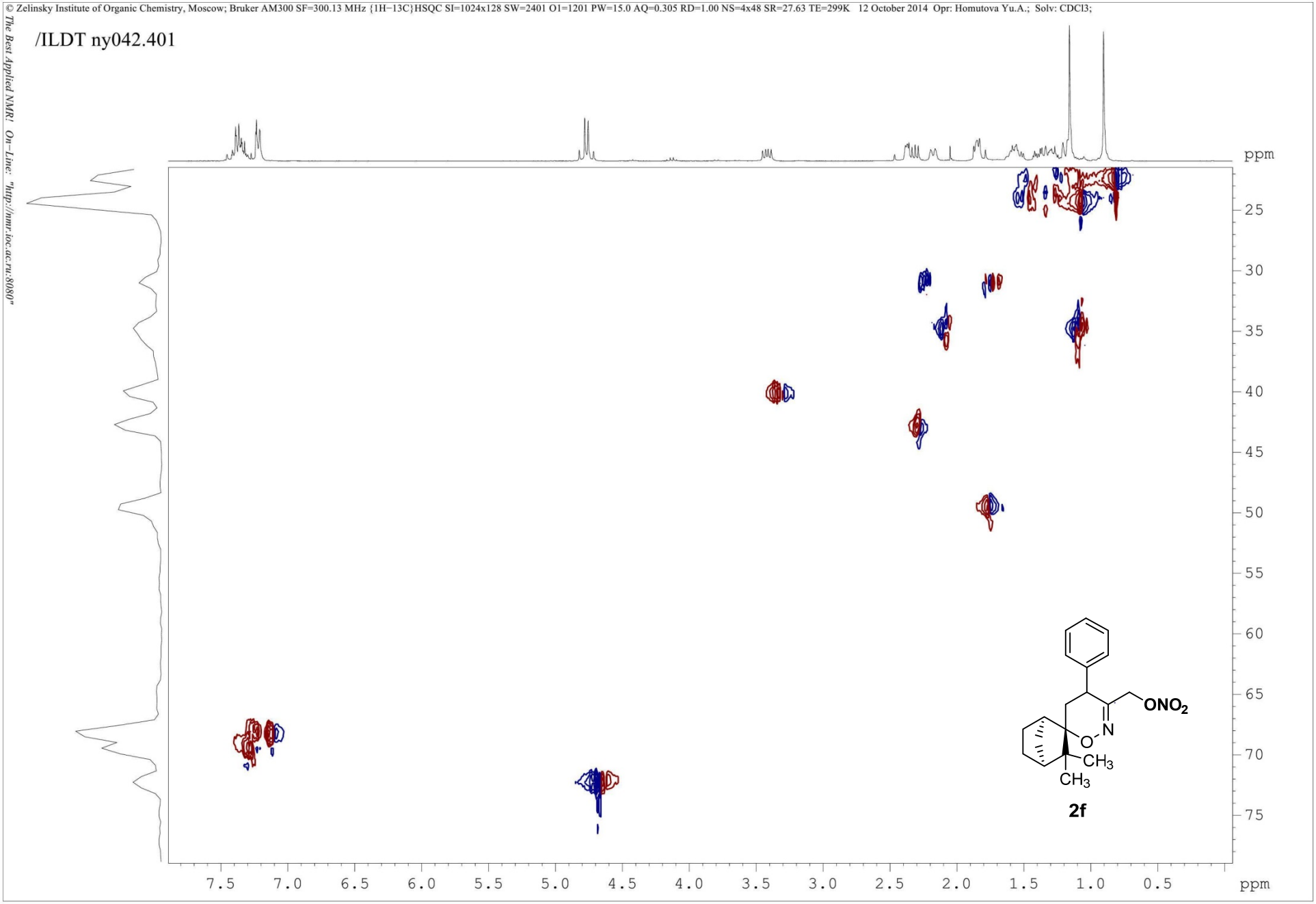
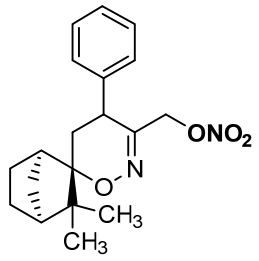



© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H−13C}HSQC SI=1024x128 SW=2401 O1=1201 PW=15.0 AQ=0.305 RD=1.00 NS=4x48 SR=27.63 TE=299K 12 October 2014 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR! On−Line: "http://nmr.ioc.ac.ru:8080"
/ILDT ny042.401
ppm
25
30
35
40
45
50
55
60
65
70
75
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
ONO2
N
O
CH3
CH3
2f

/ILDT ny042.401



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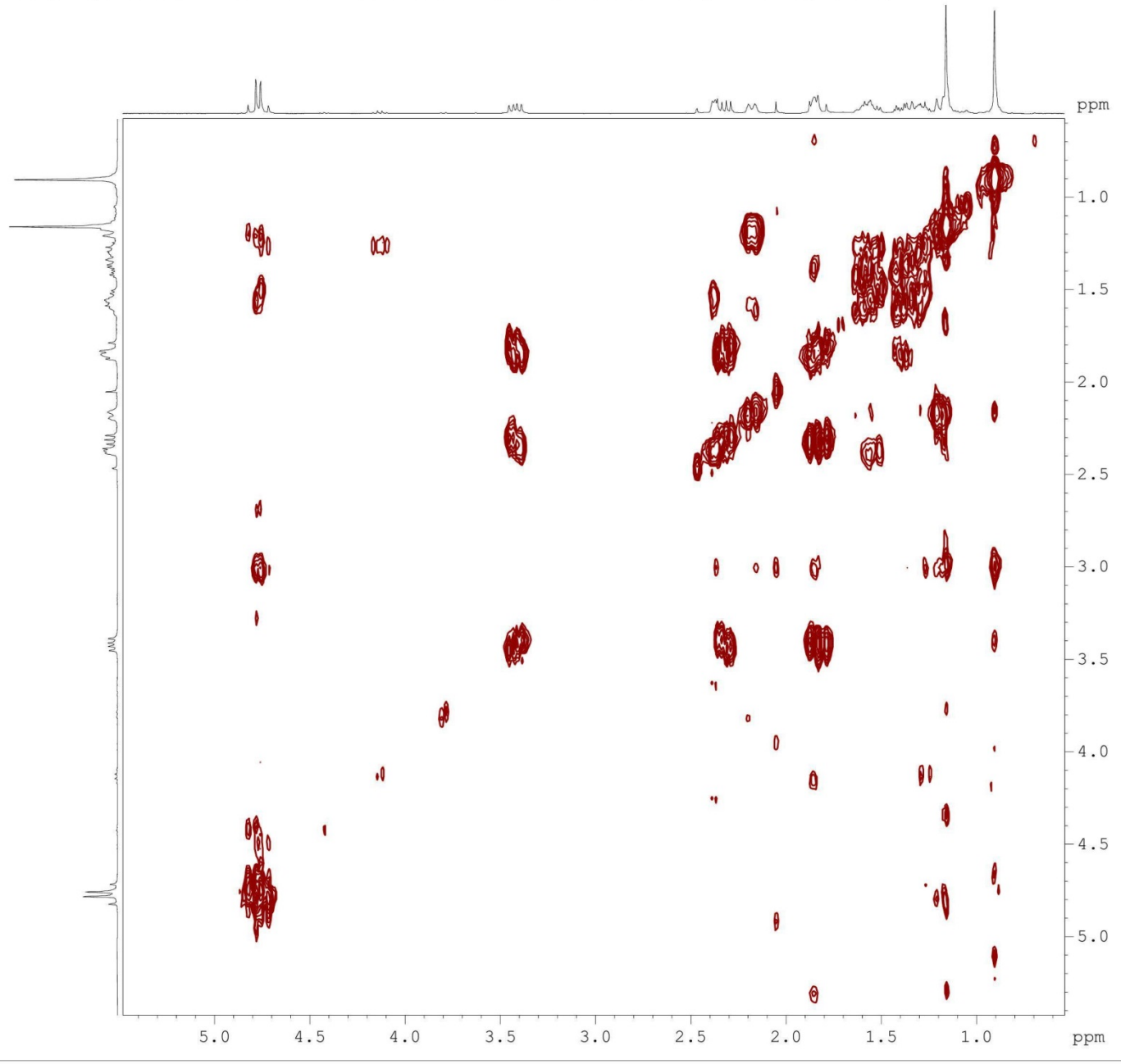

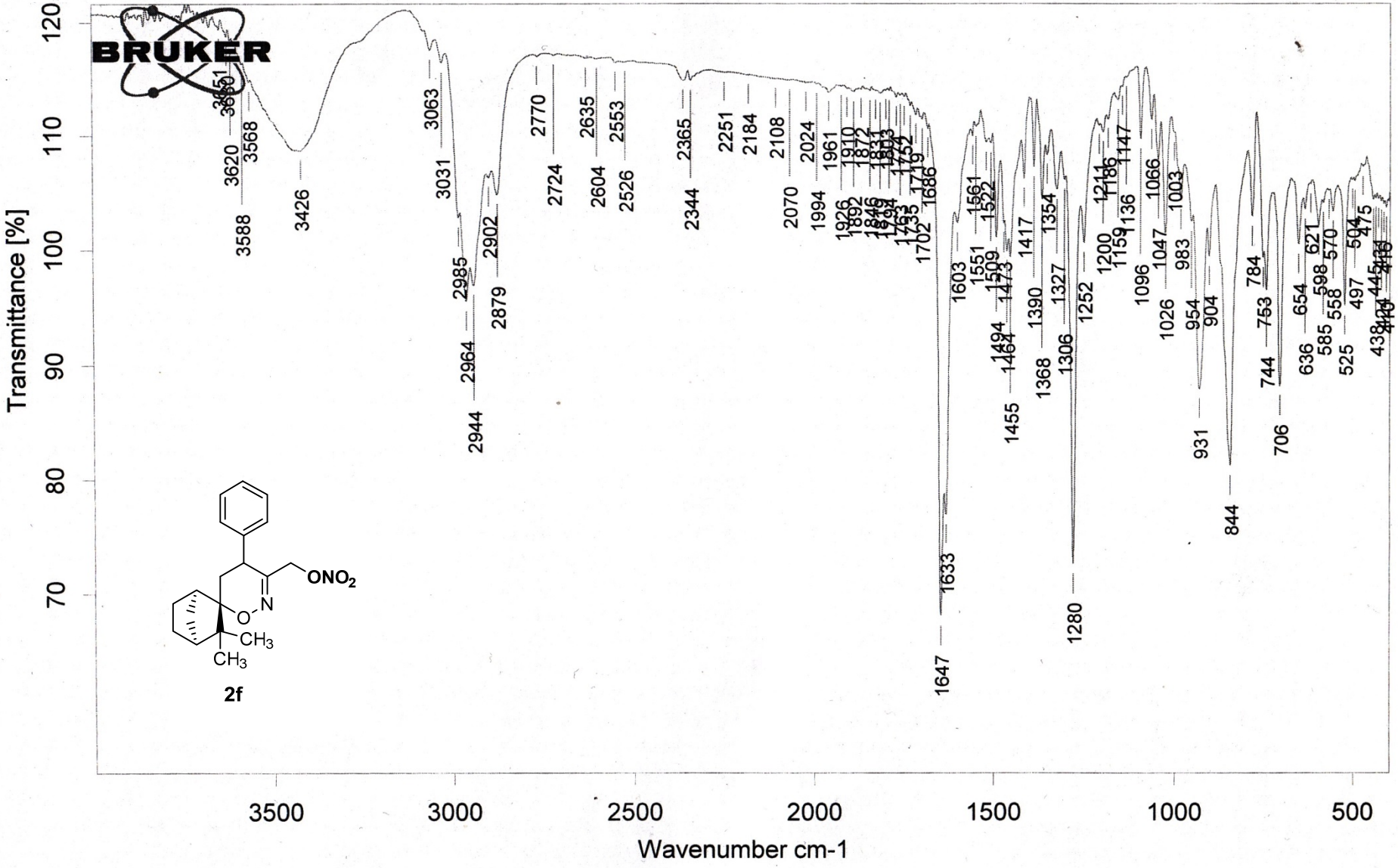

2f

D:\EDL\NY-042.0	NY-042 ,	KBr.	08.10.2014

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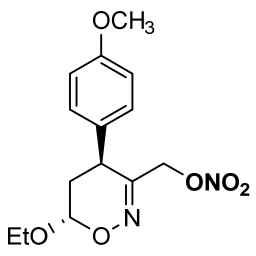

2g

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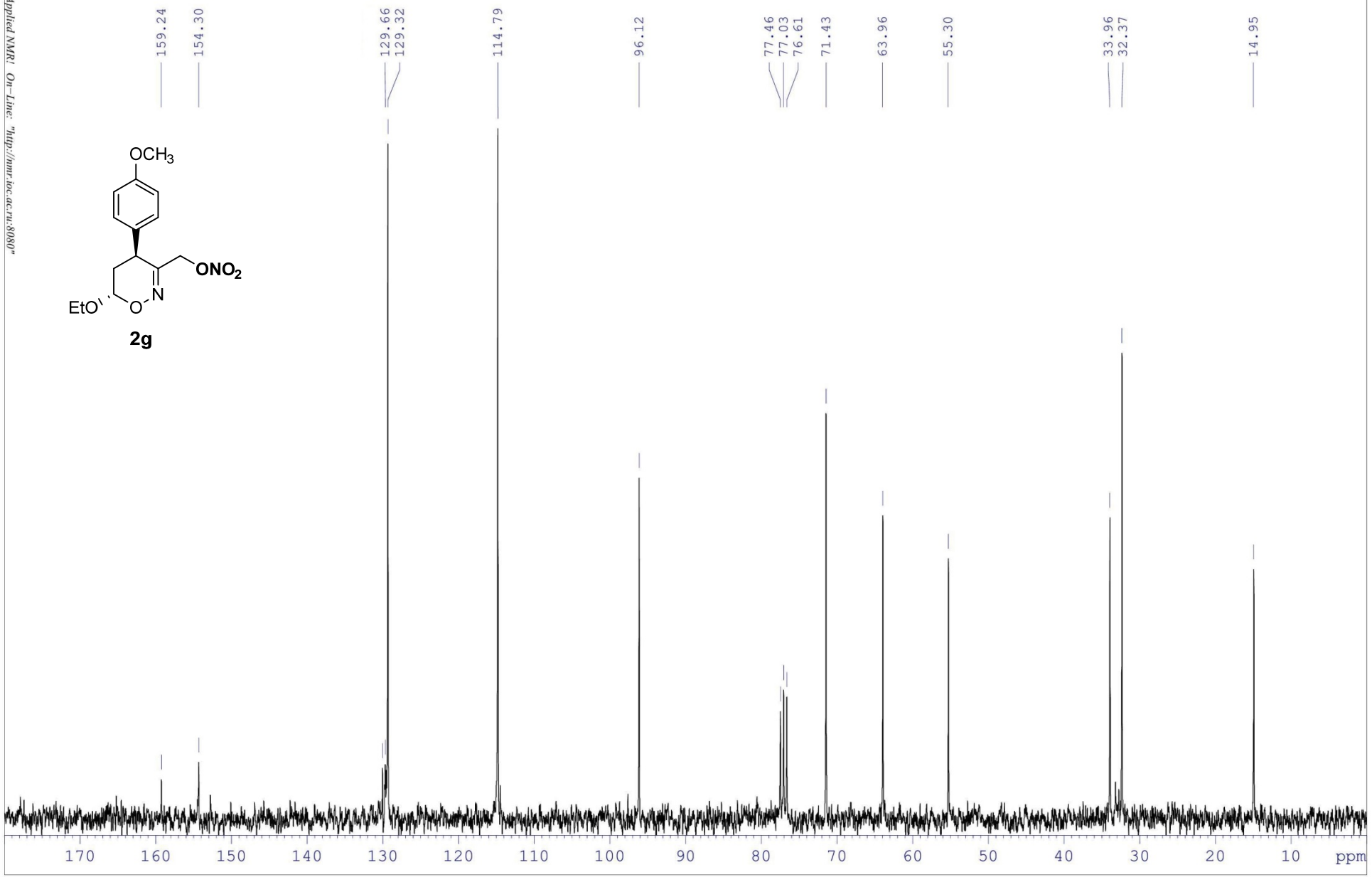
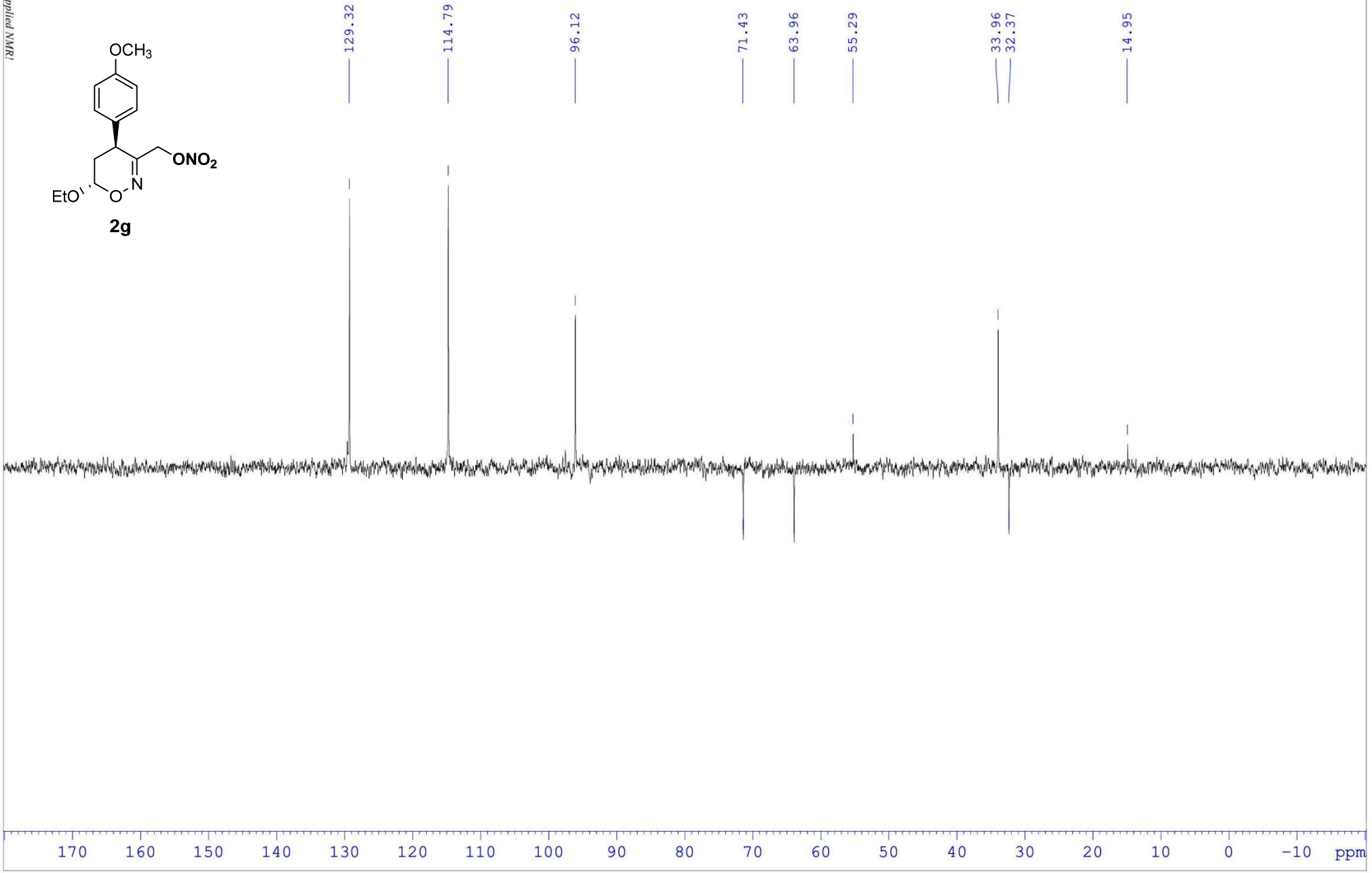


2g

/ILDT ny041.201

2g



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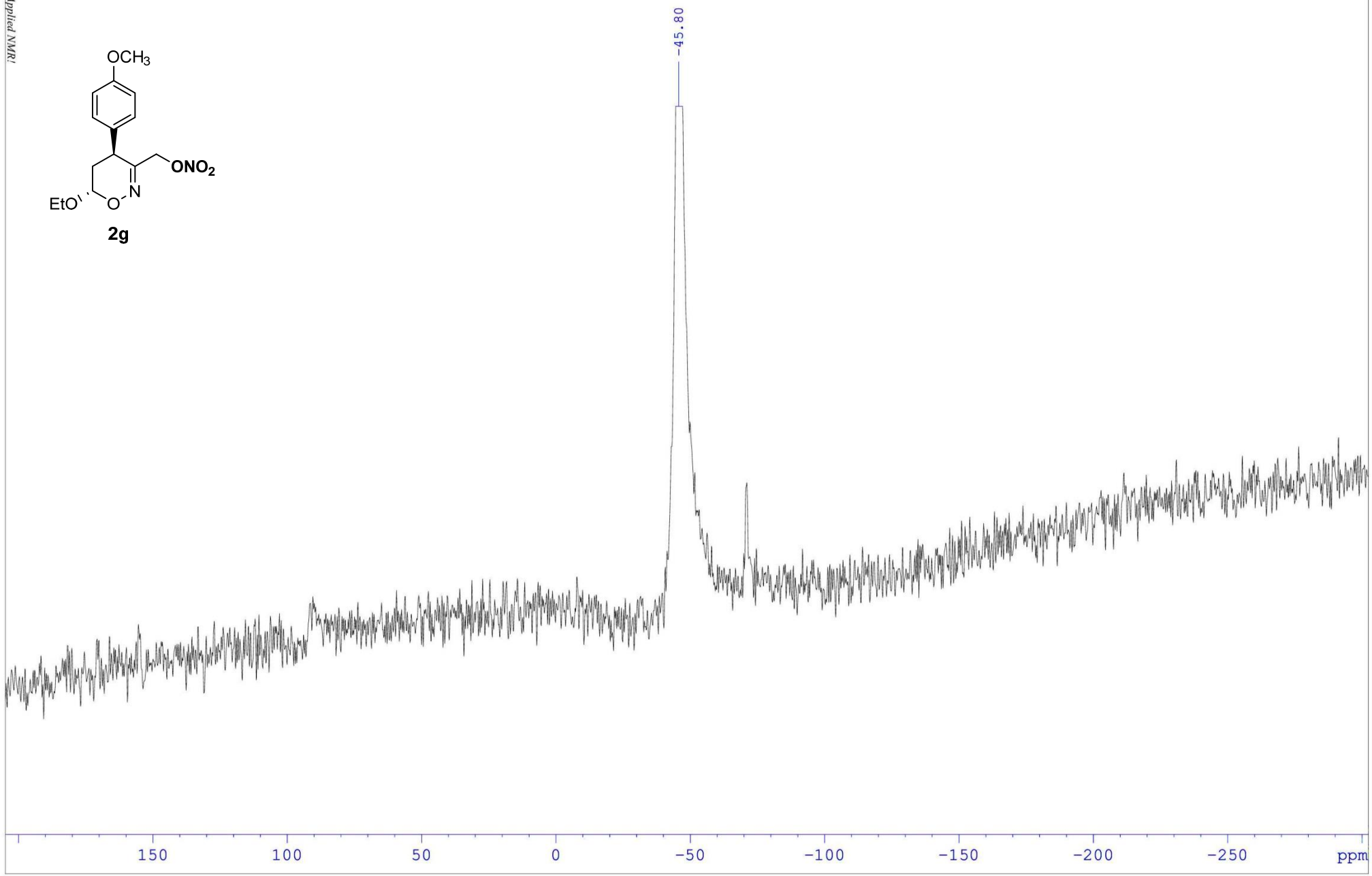
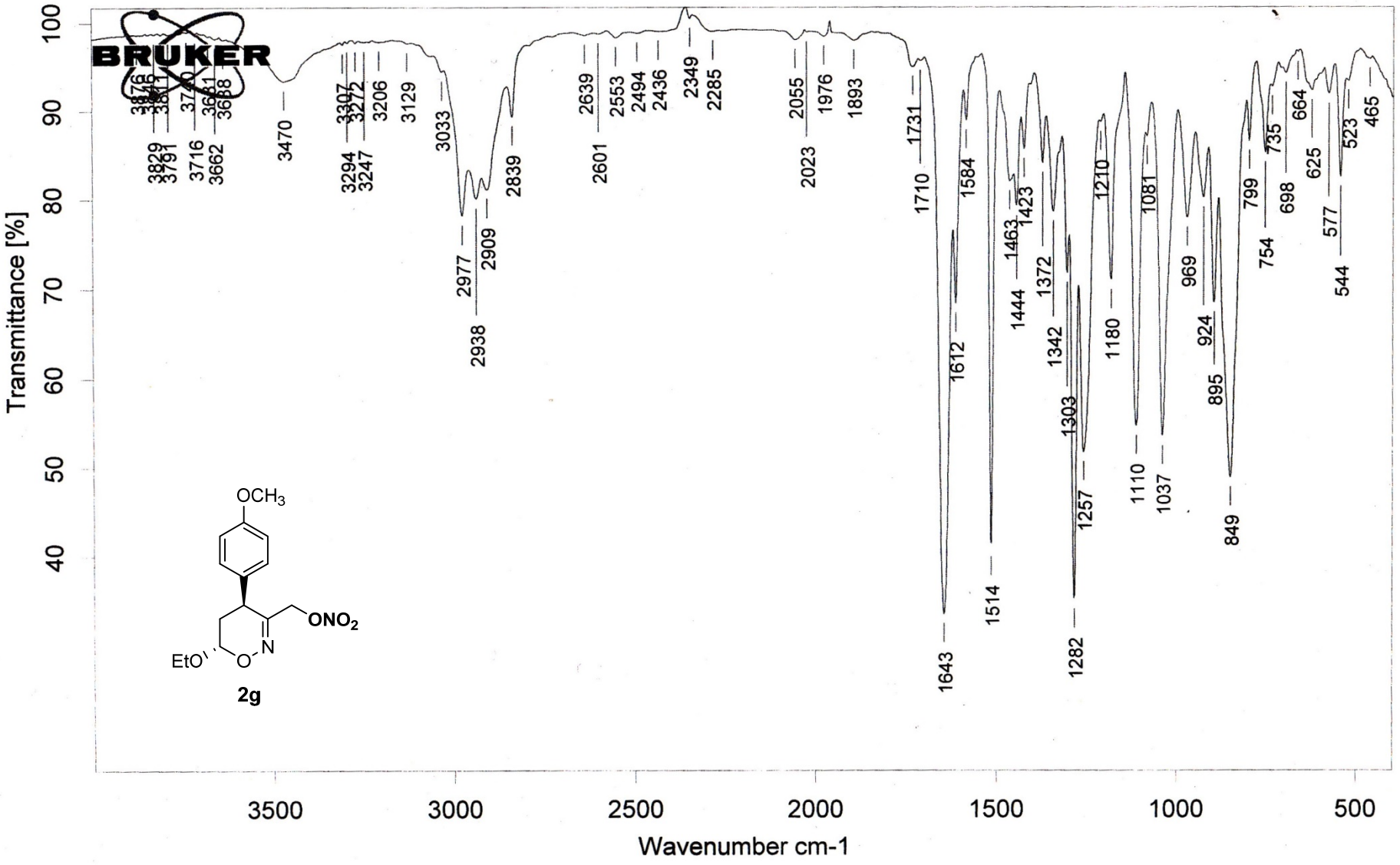
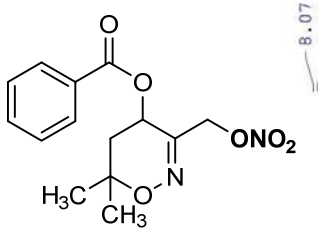
2g





D:\EDL\NY-041.0	NY-041	KBr.	08.10.2014

/ILDT ny080.501



2h

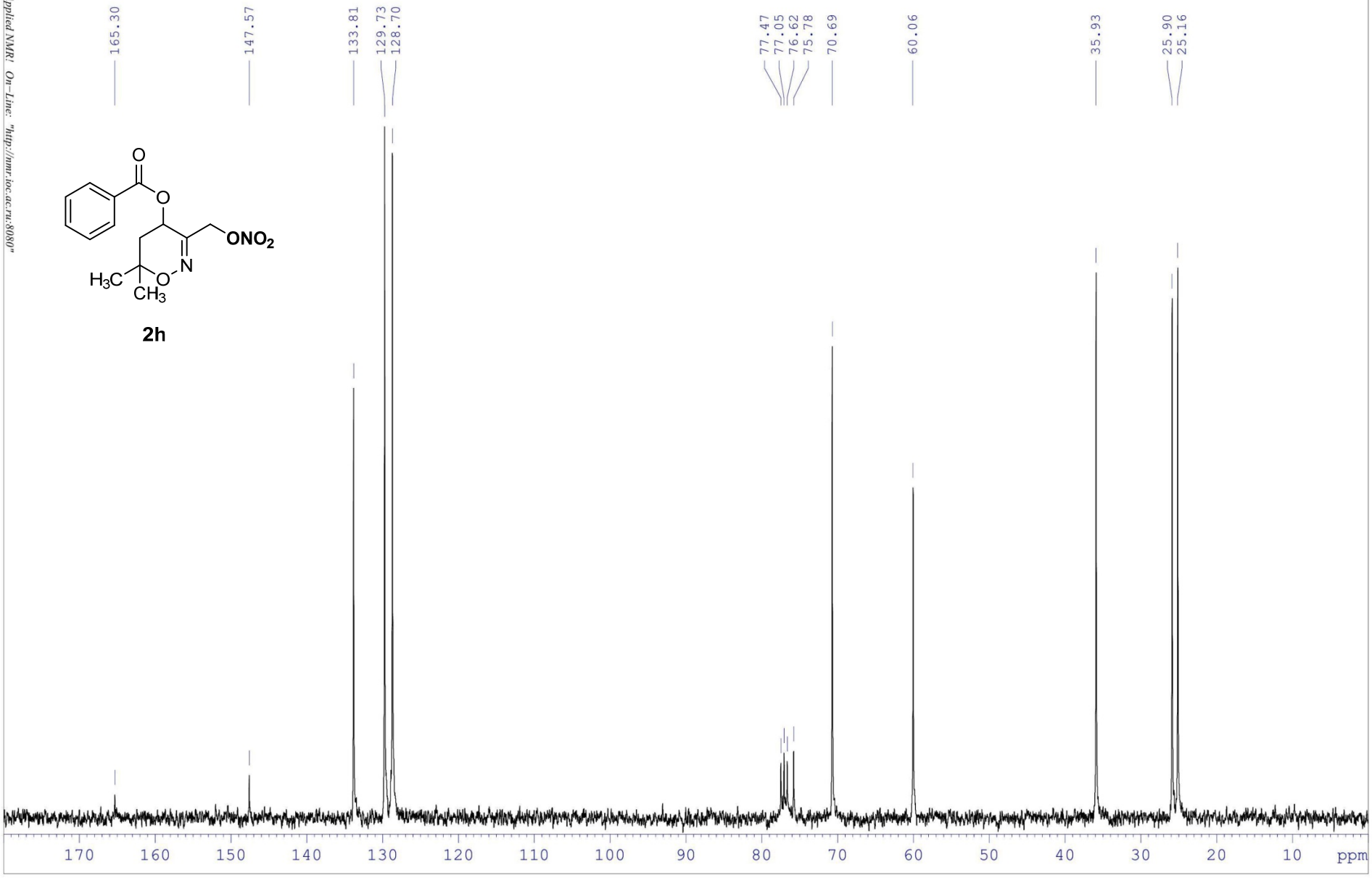
8.07 8.04 7.66 7.63 7.54 7.51 7.49 5.64 5.62 5.60 5.32 5.27 5.18 5.14 2.37 2.34 2.32 2.30 2.11 2.09 2.06 2.04 1.44 1.43

45 43 25 22 23 21 24 25 125

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

45.709 25.126 43.079 22.246 23.613 21.520 24.639 25.239 125.263

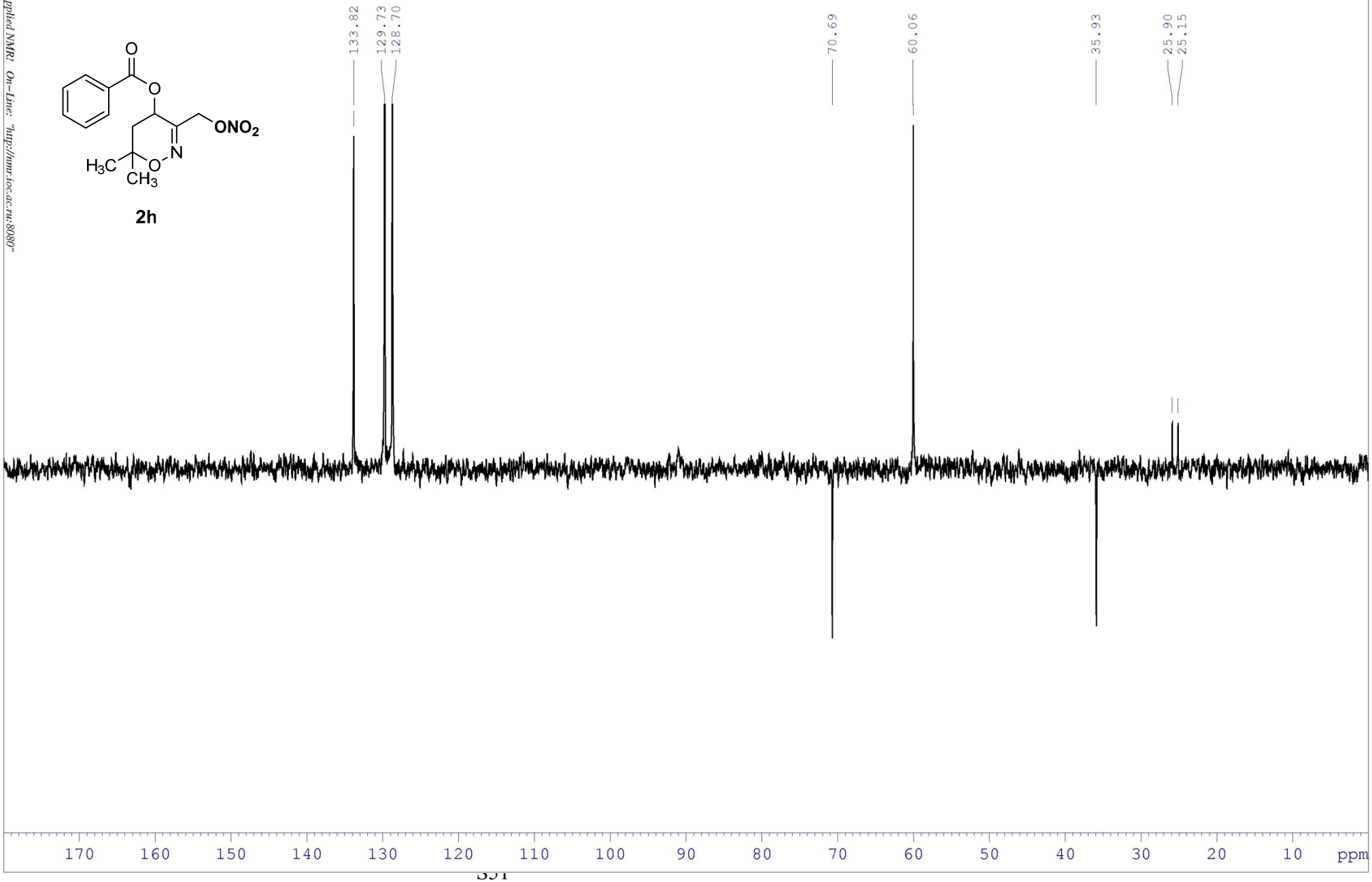
/ILDT ny080.501



2h

/ILDT ny080.501

2h



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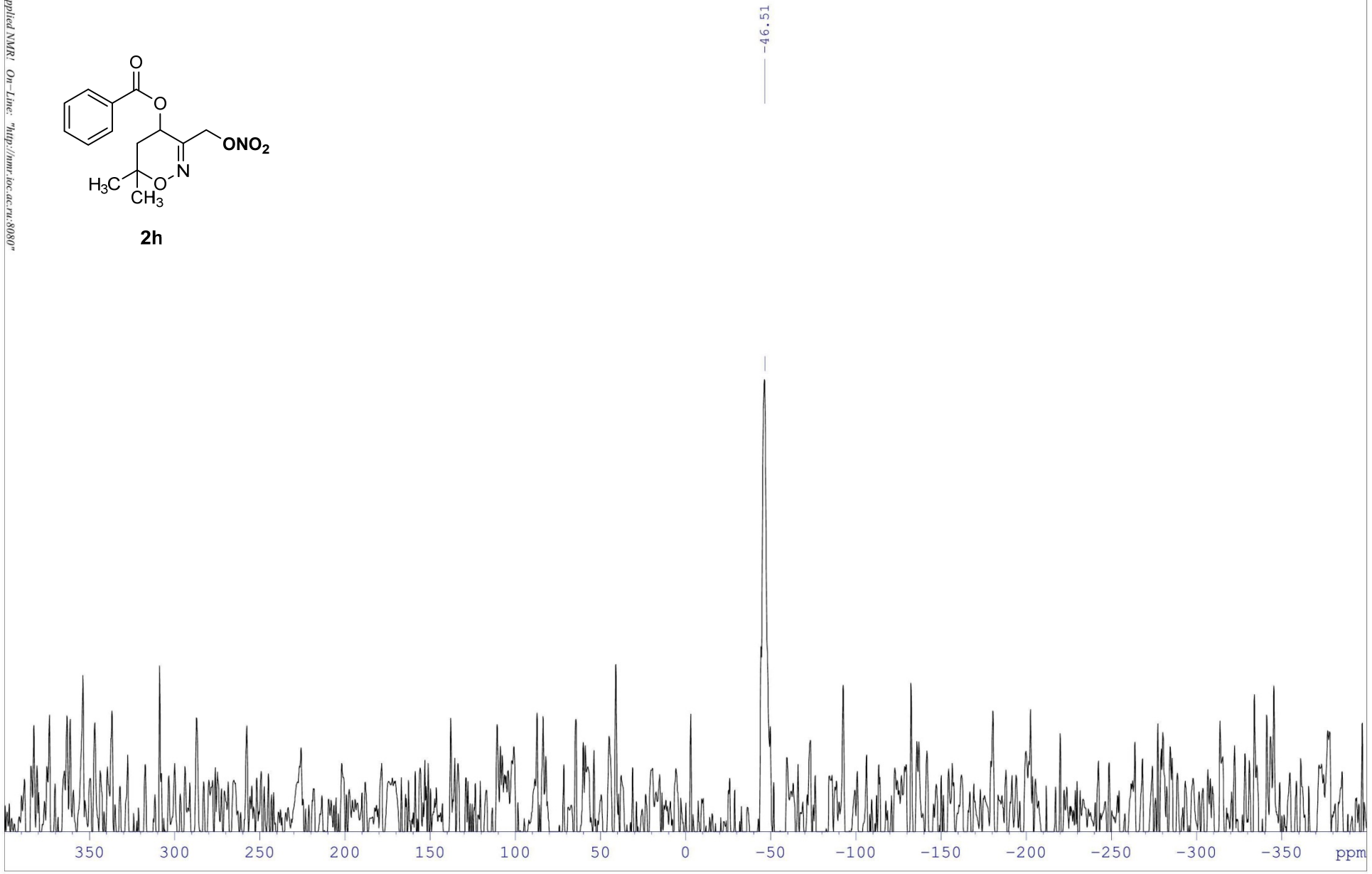
2h



BRUKER
Transmittance [%]
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
10
20
30
40
50
60
70
80
90
3859
3827
3775
3729
3661
3624
3571
3468
3436
3351
3340
3307
3294
3271
3247
3233
3167
3066
3035
2981
2938
2774
2729
2659
2601
2559
2522
2360
2341
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1915
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1602
1585
1536
1512
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1425
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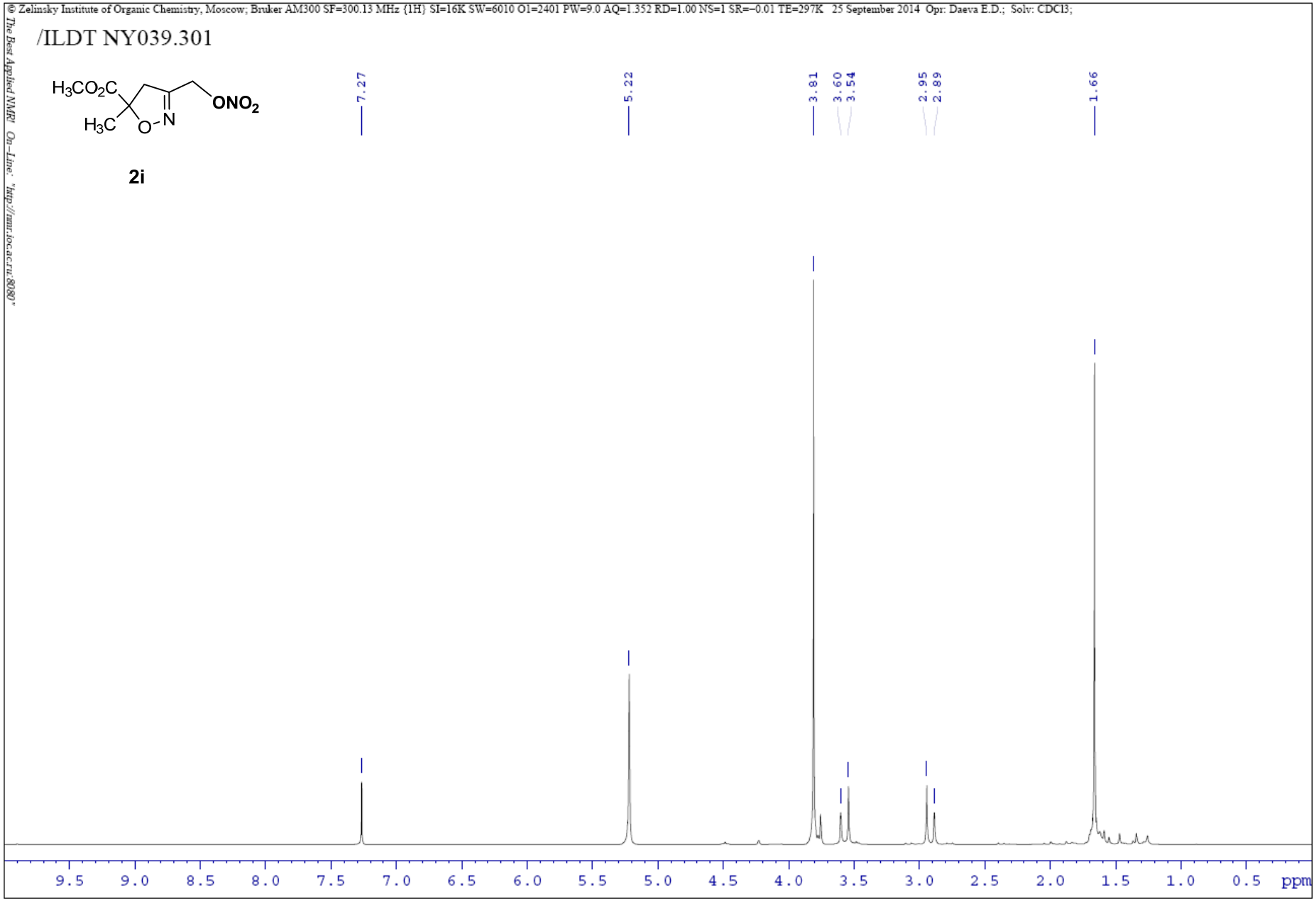
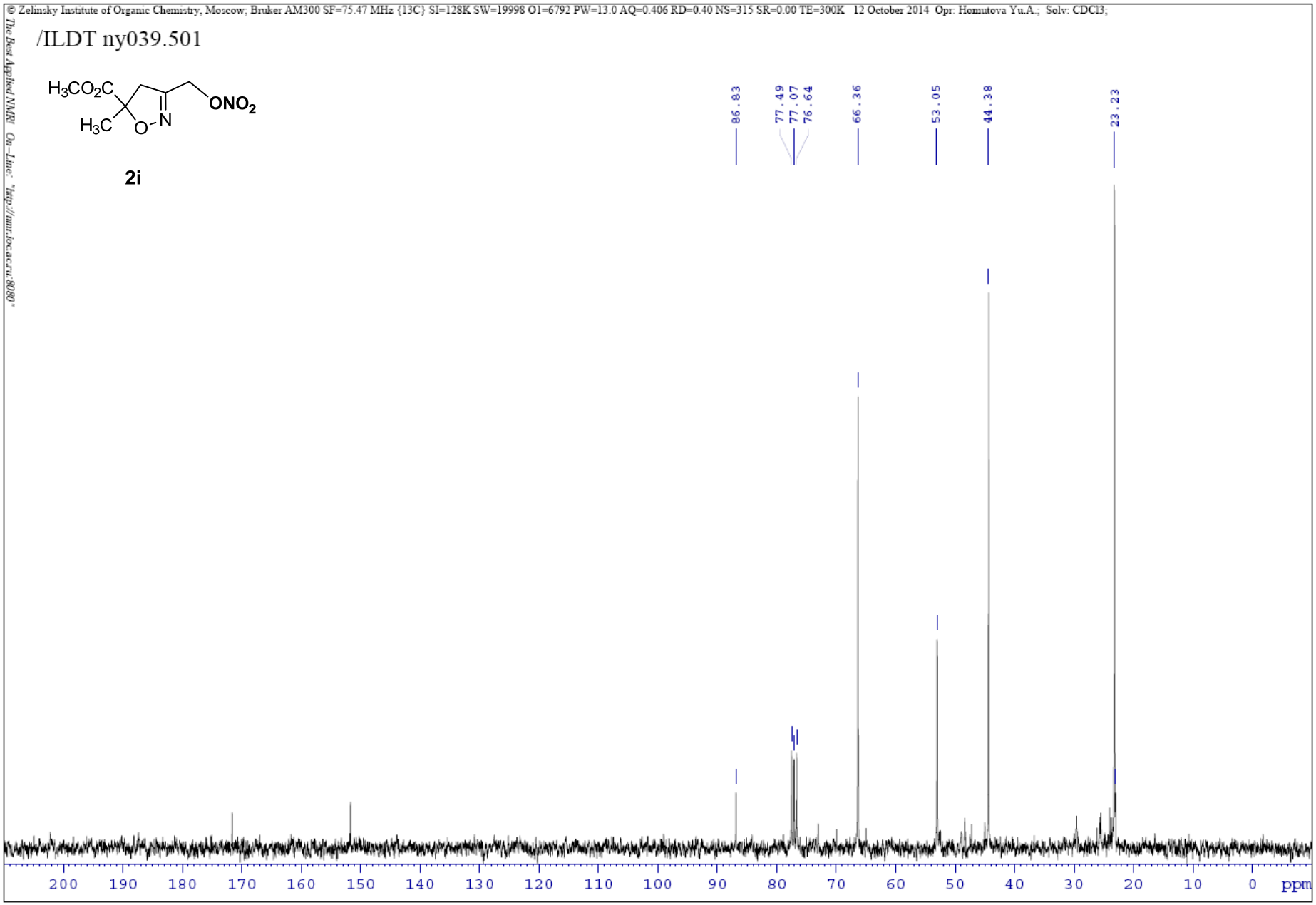
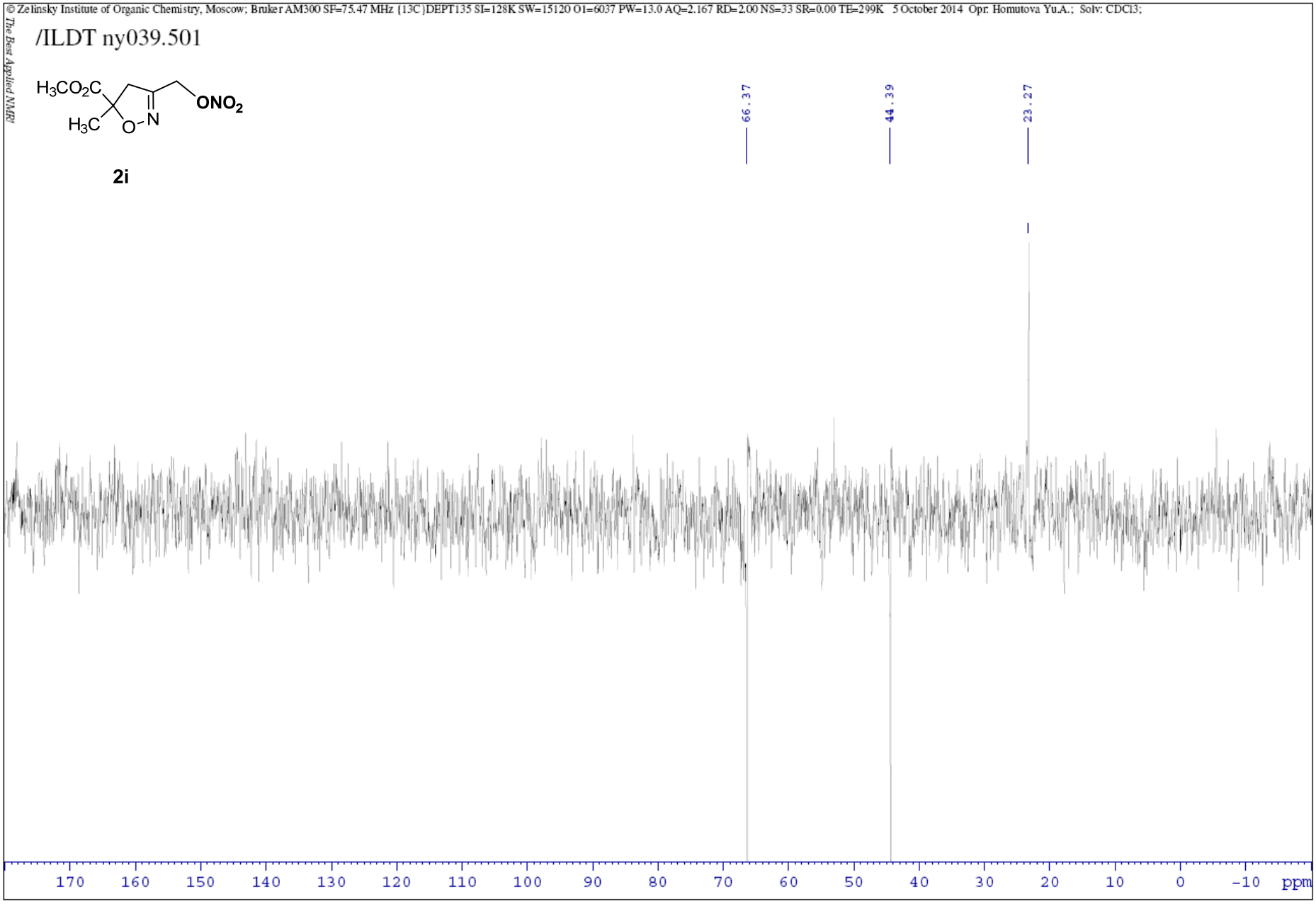
1373
1344
1317
1283
1263
1194
1177
1161
1134
1106
1097
1070
1050
1026
989
953
928
851
805
774
756
712
687
670
643
617
577
544
506
483
445
422
O
O
O
ONO2
N
H3C
CH3
2h
D:\EDL\NY-80.0
NY-80
KBr.

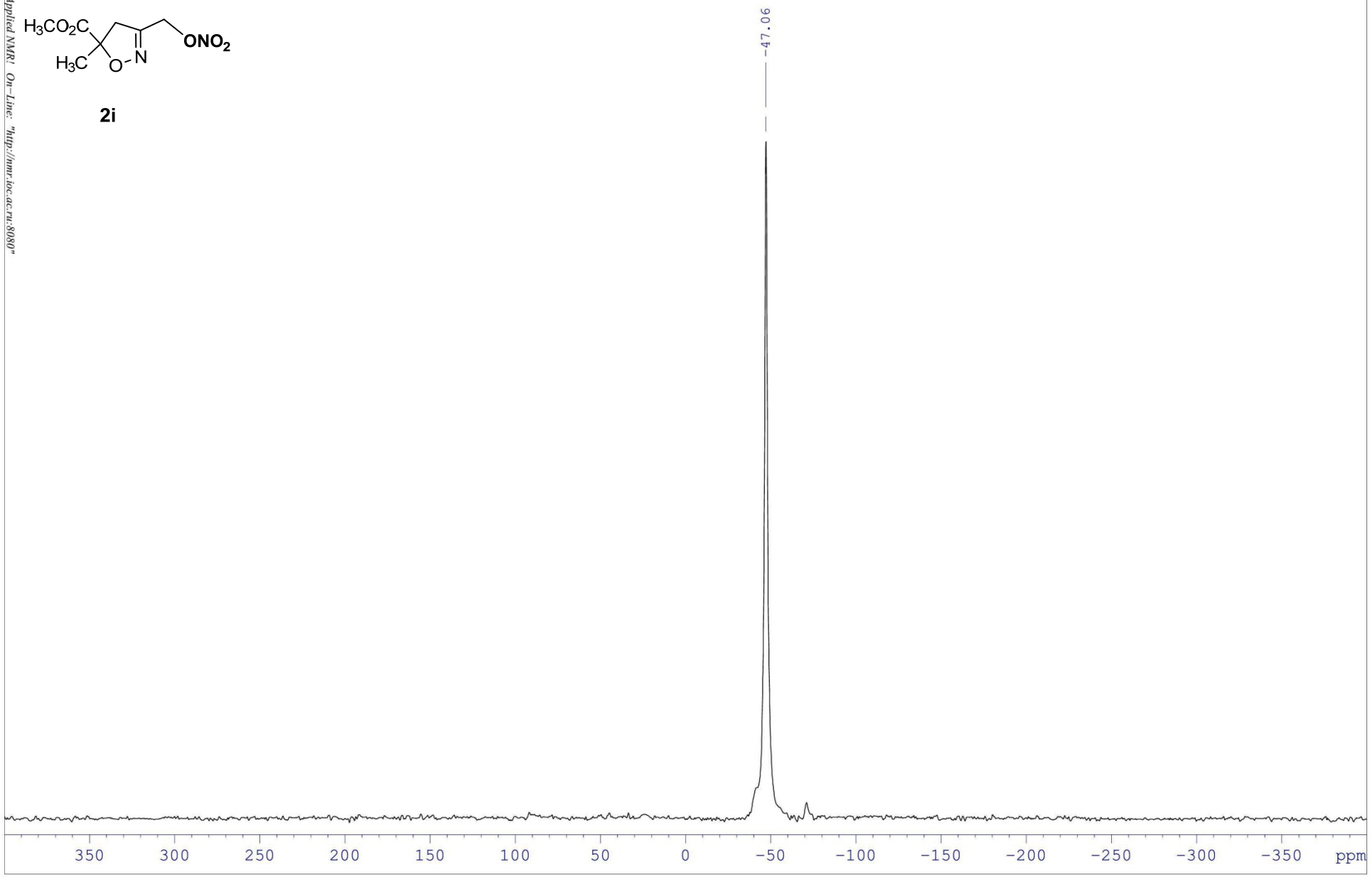

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=1.00 NS=1 SR=–0.01 TE=297K 25 September 2014 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT NY039.301
H3CO2C
H3C
O
N
ONO2
2i
7.27
5.22
3.81
3.60
3.54
2.95
2.89
1.66
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

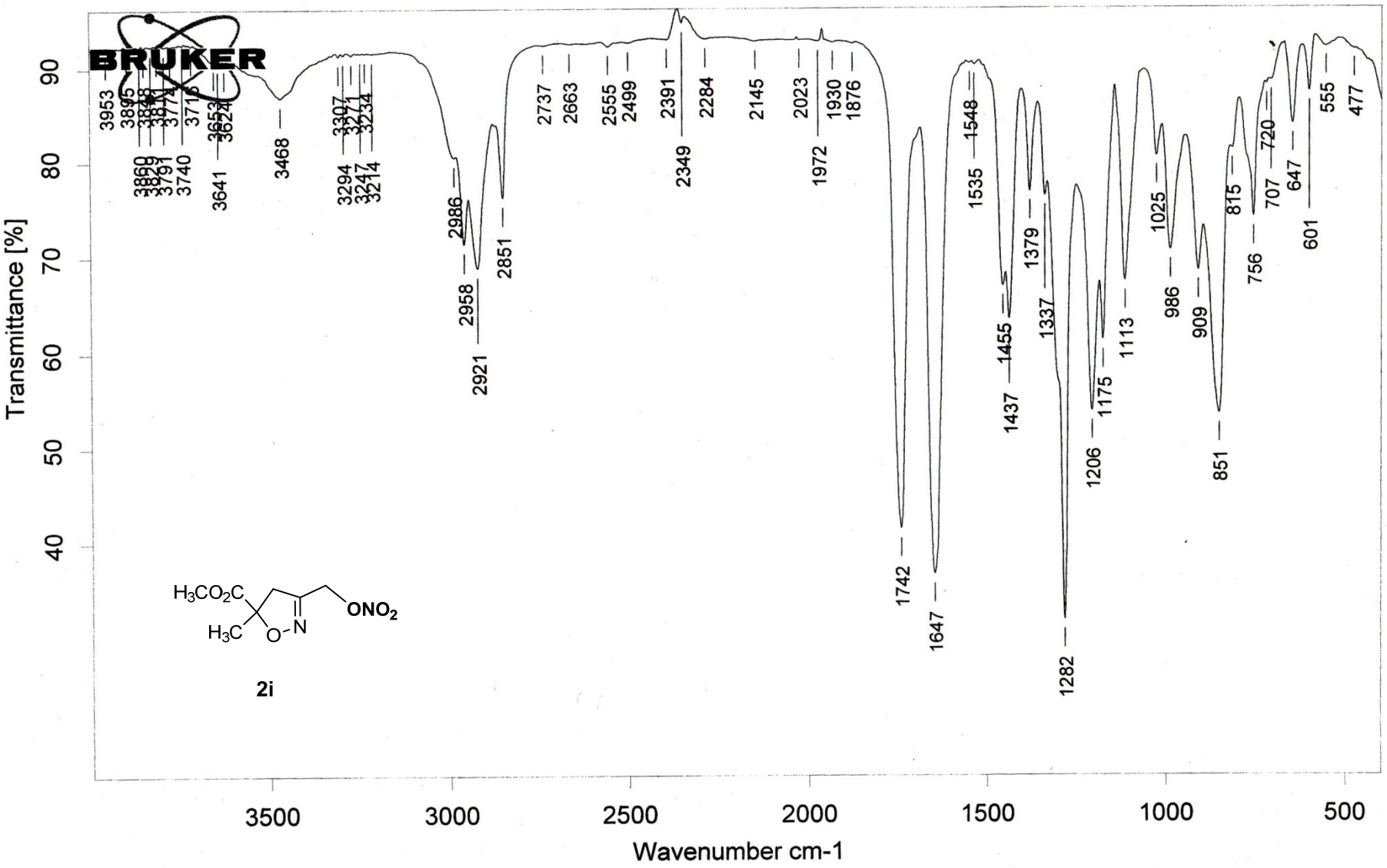

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=128K SW=19998 O1=6792 PW=13.0 AQ=0.406 RD=0.40 NS=315 SR=0.00 TE=300K 12 October 2014 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT ny039.501
H3CO2C
H3C
O
N
ONO2
2i
86.83
77.49
77.07
76.64
66.36
53.05
44.38
23.23
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C}DEPT135 SI=128K SW=15120 O1=6037 PW=13.0 AQ=2.167 RD=2.00 NS=33 SR=0.00 TE=299K 5 October 2014 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR
/ILDT ny039.501
H_3CO_2C
H_3C
O
N
ONO_2
2i
66.37
44.39
23.27
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm

/ILDT ny039.501

2i

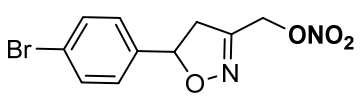




2i

D:\EDL\NY-039.0	NY-039 , .KBr.	13.10.2014

/ILDT ny049.601



2j

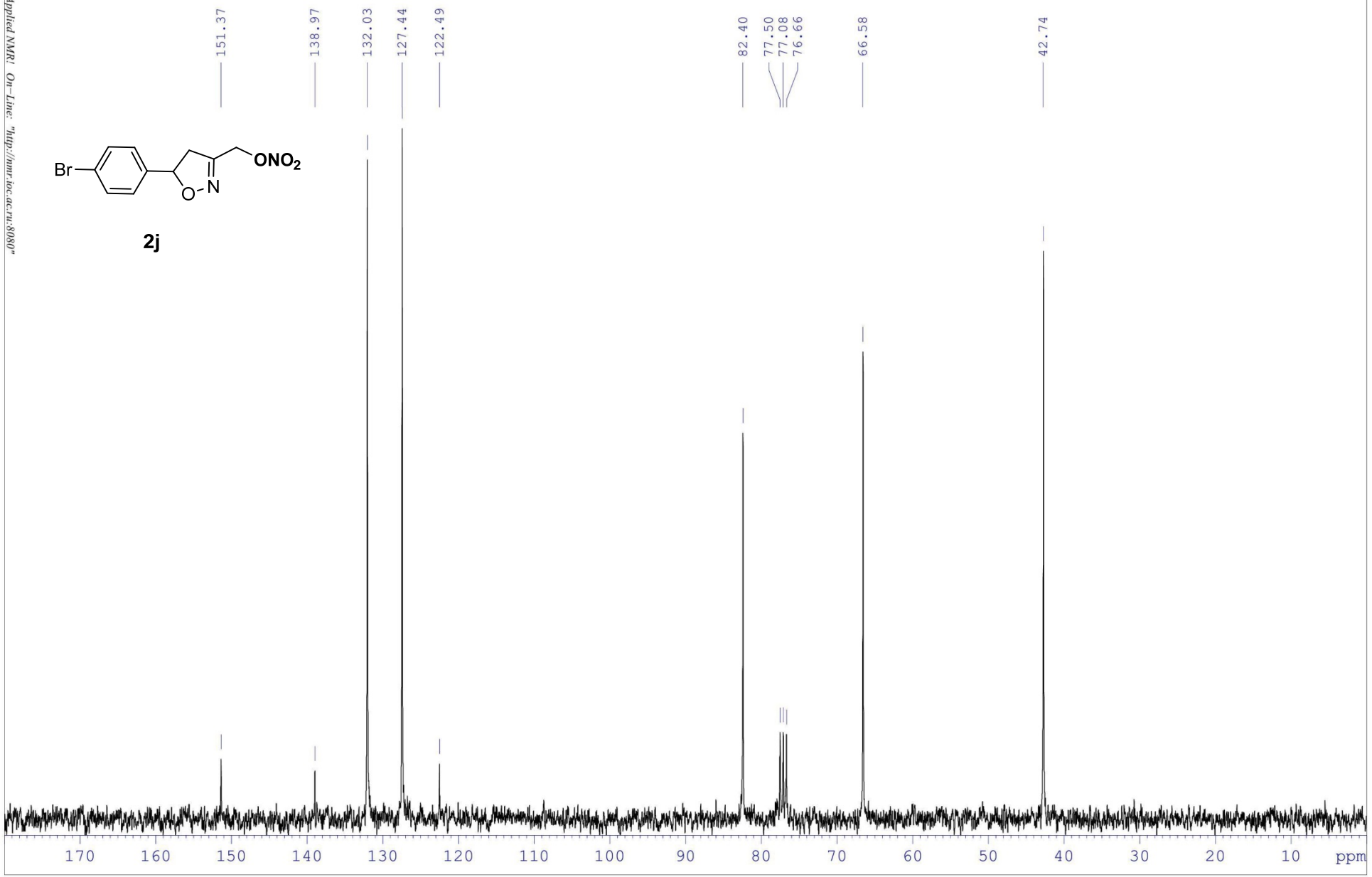
7.53 7.51 7.21 7.18 5.69 5.66 5.65 5.62 5.27 3.54 3.50 3.48 3.44 3.02 3.00 2.97 2.94

124 125 61 68 66

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

124.747 125.373 61.769 68.526 66.922

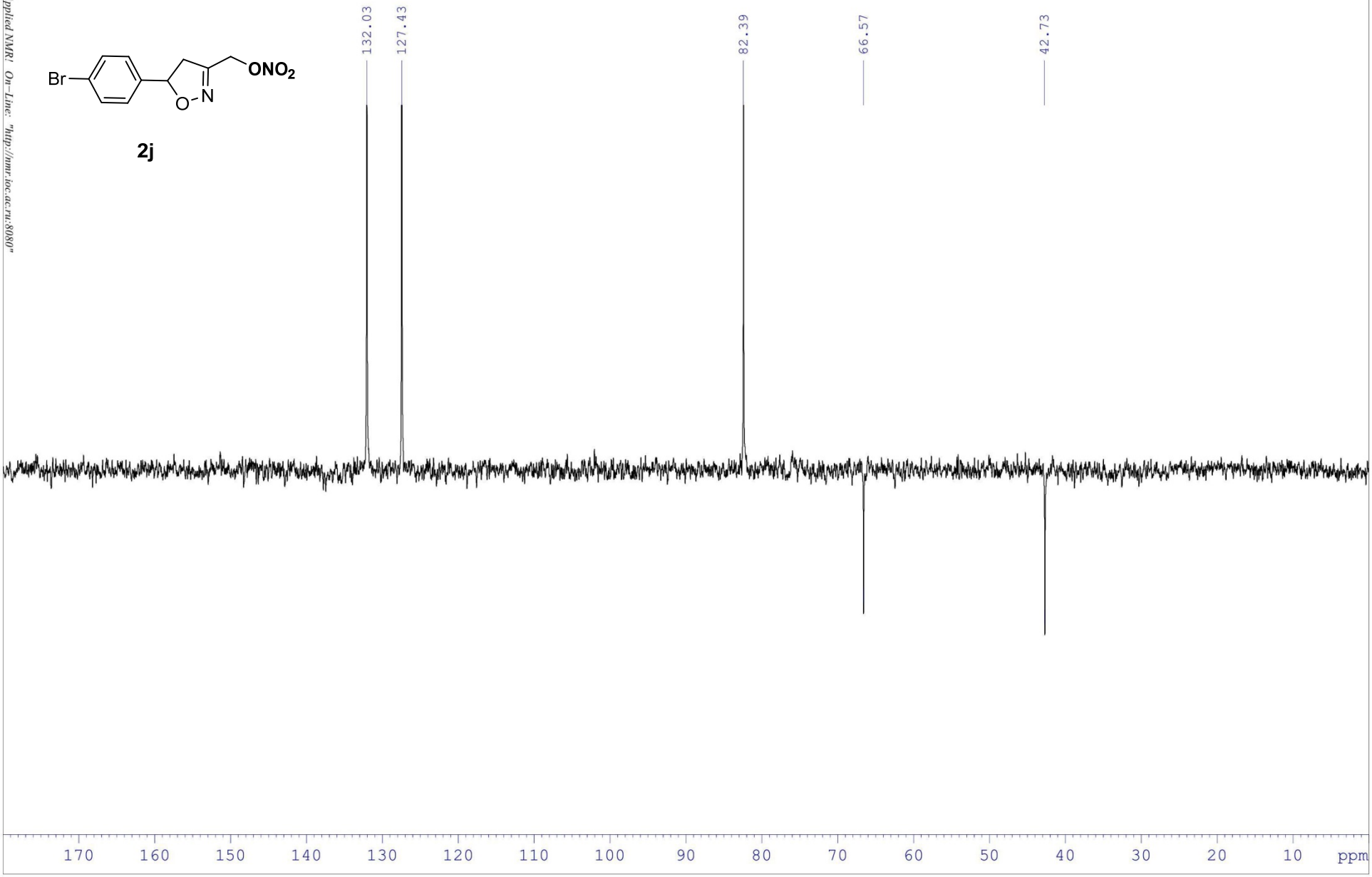
/ILDT ny049.601



2j

/ILDT ny049.601

2j



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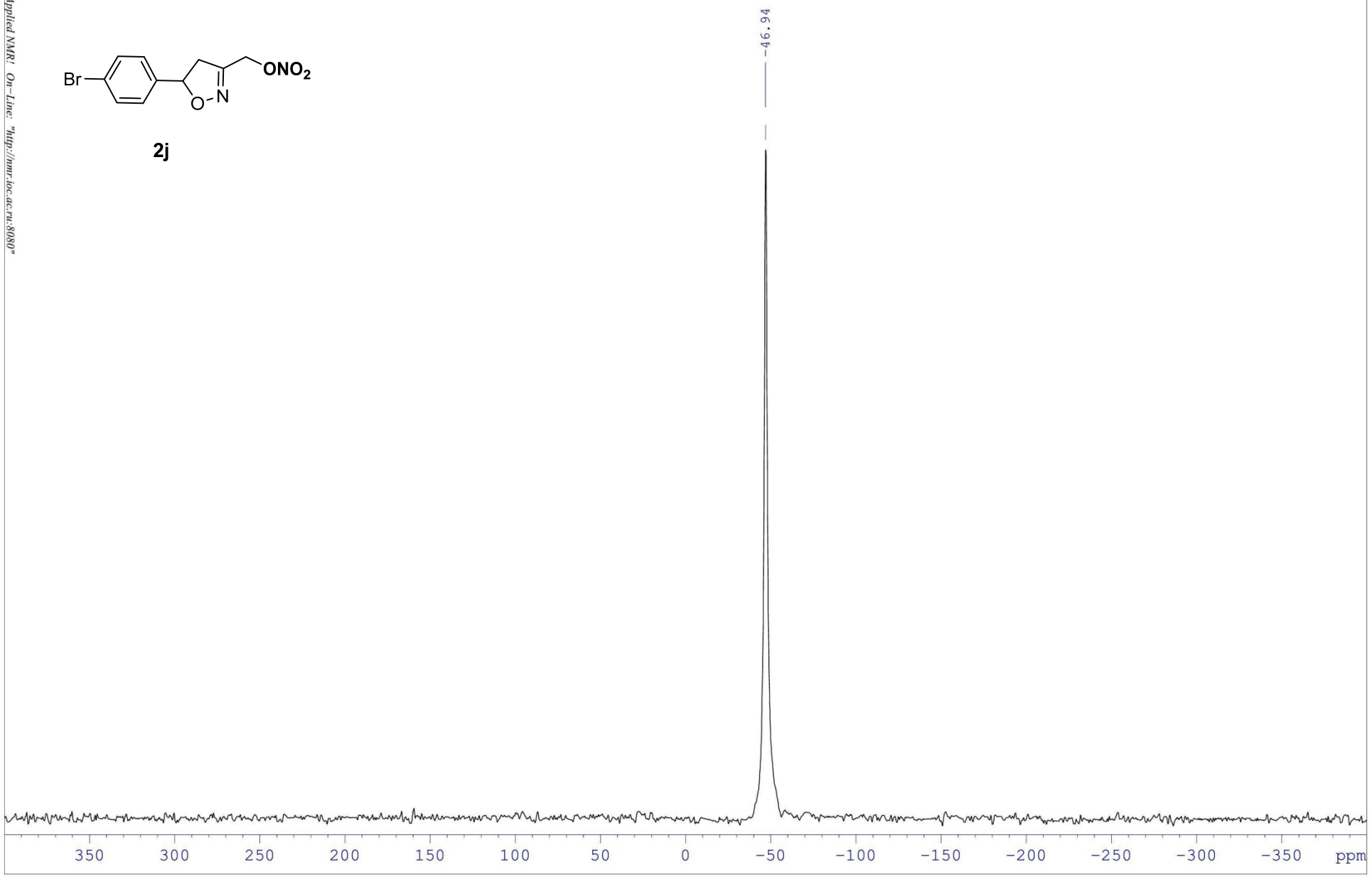
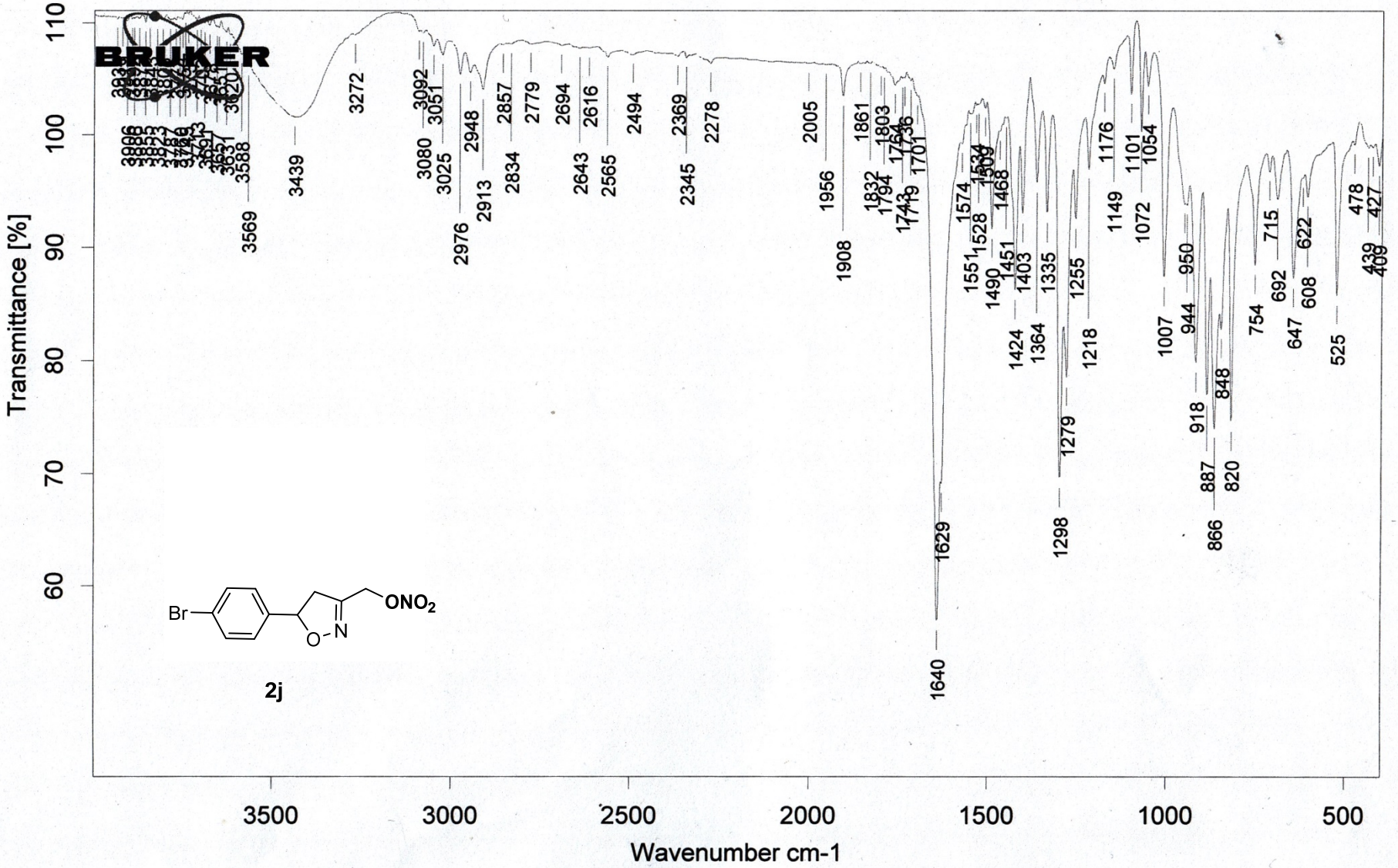
2j

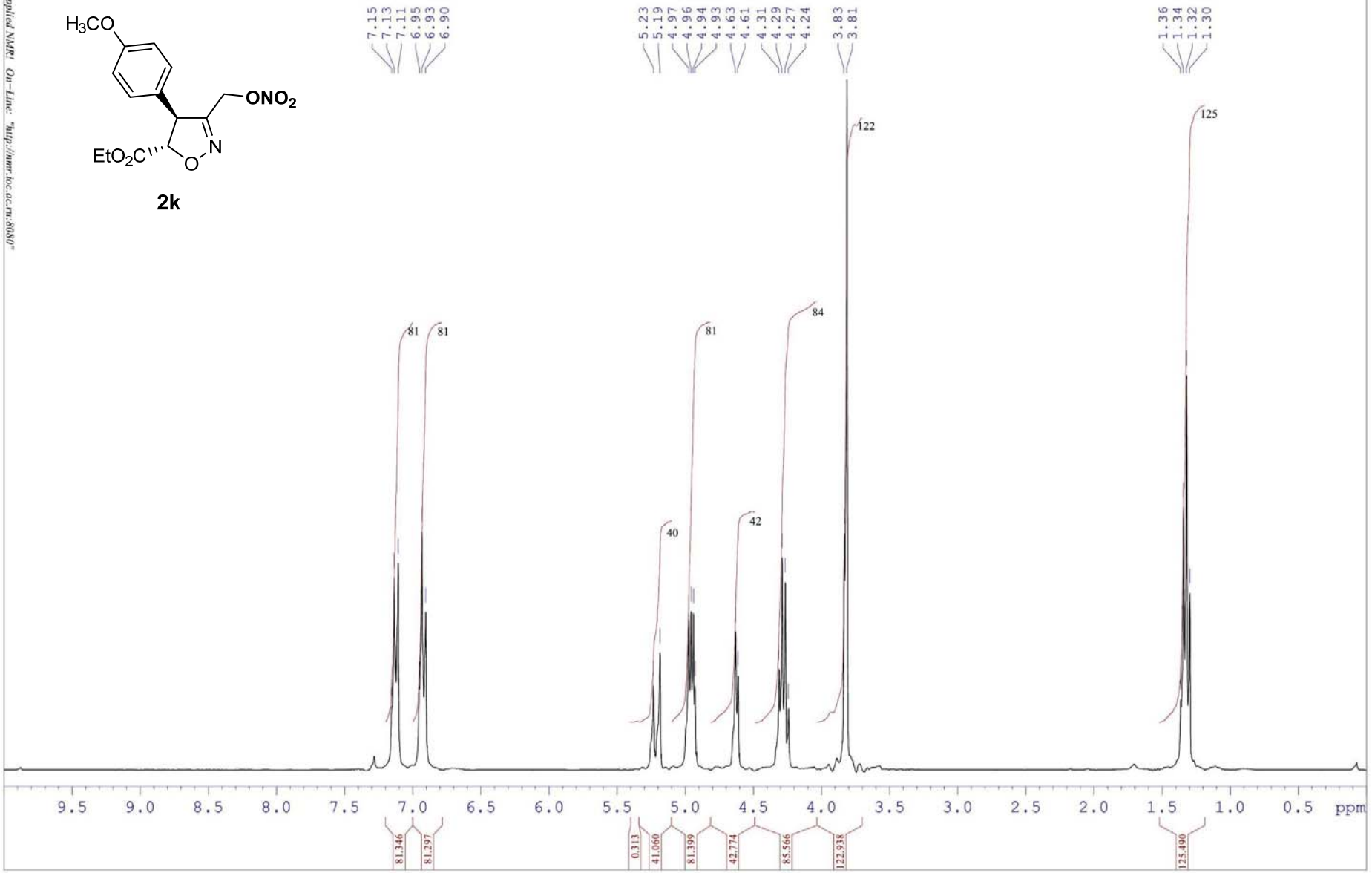




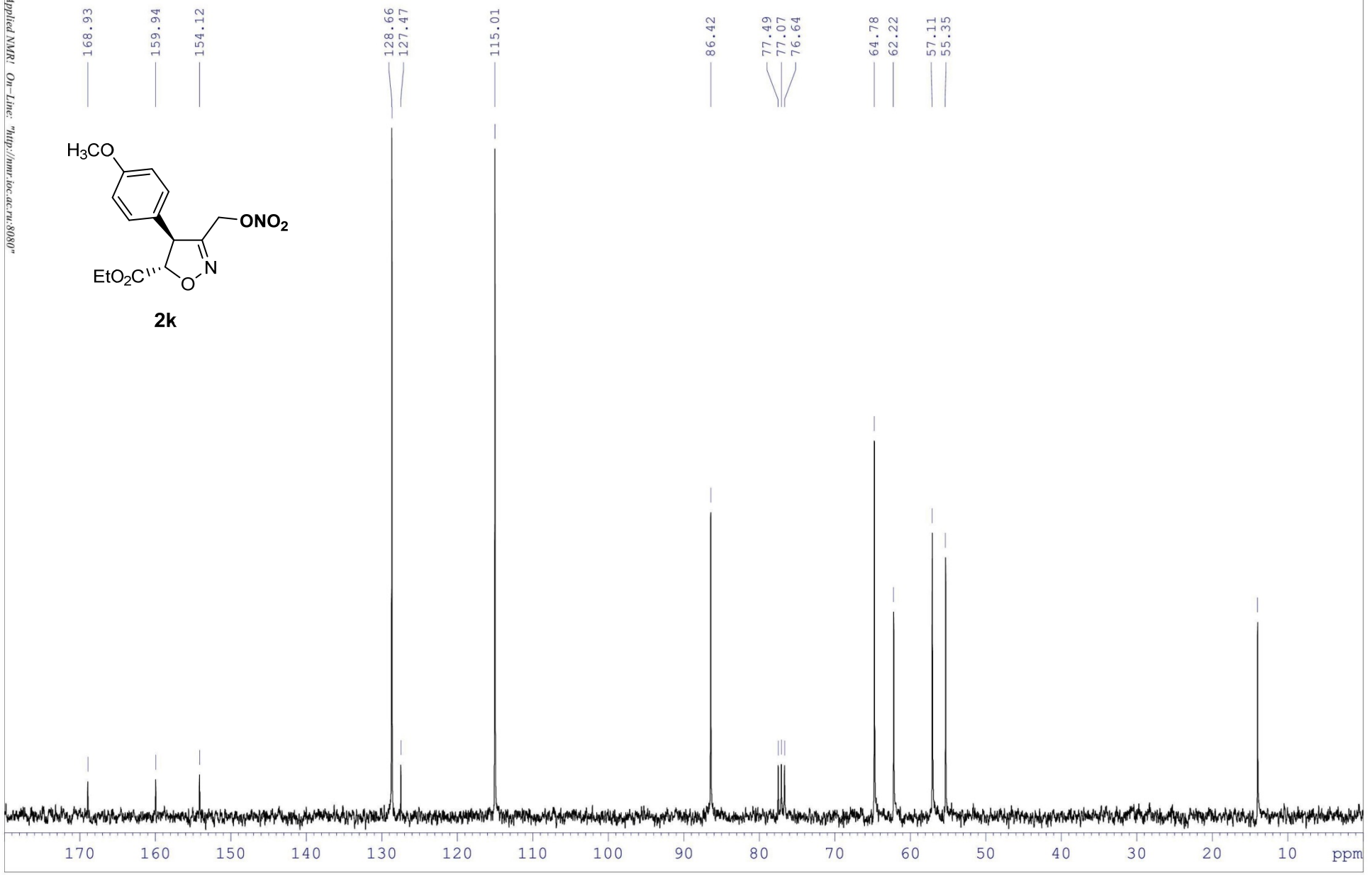
D:\EDL\NY-049.0	NY-049 ,	KBr,1/100.	09.06.2015

/ILDT NY074.601

2k

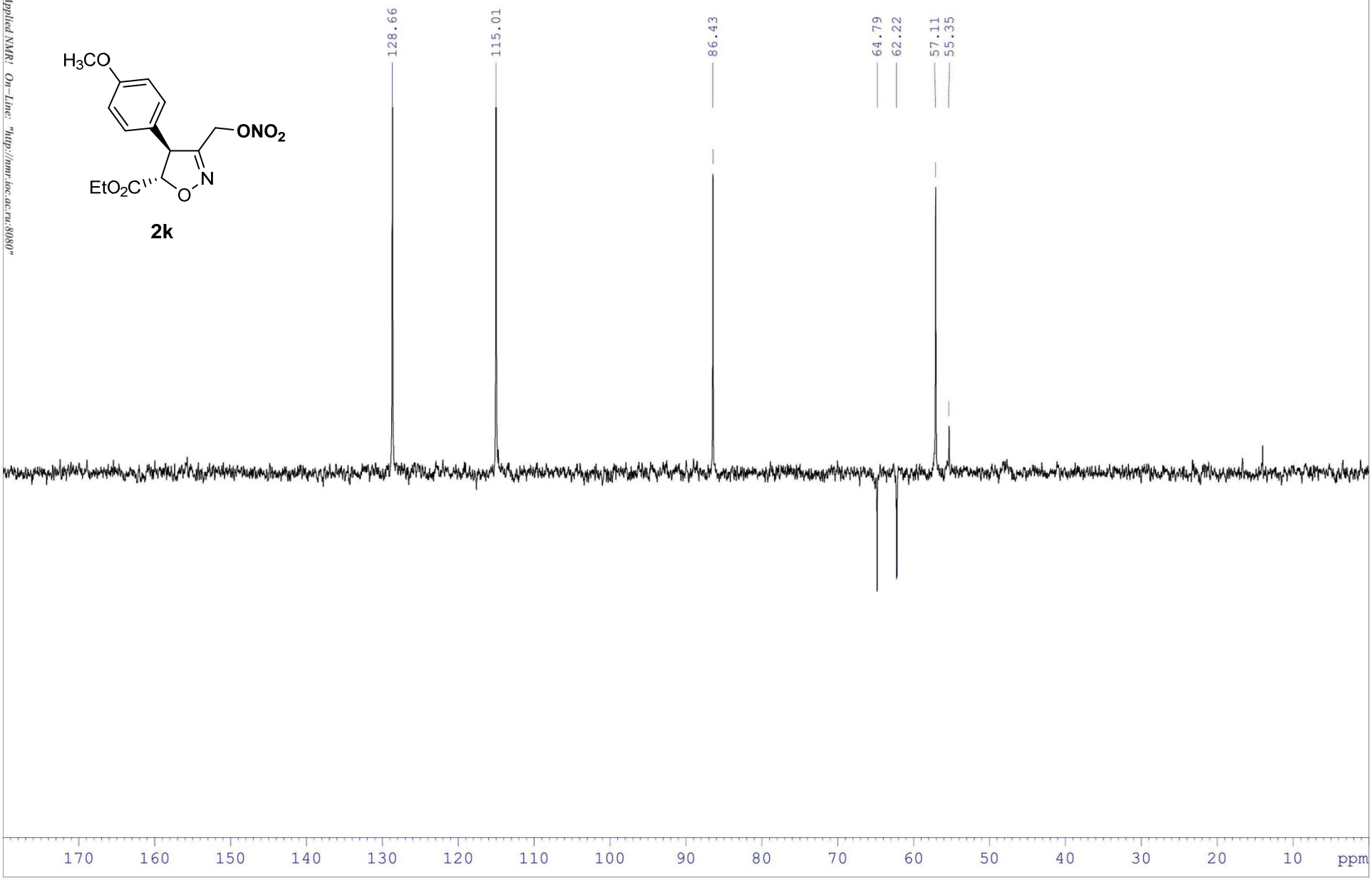


/ILDT ny074.601



/ILDT ny074.601

2k



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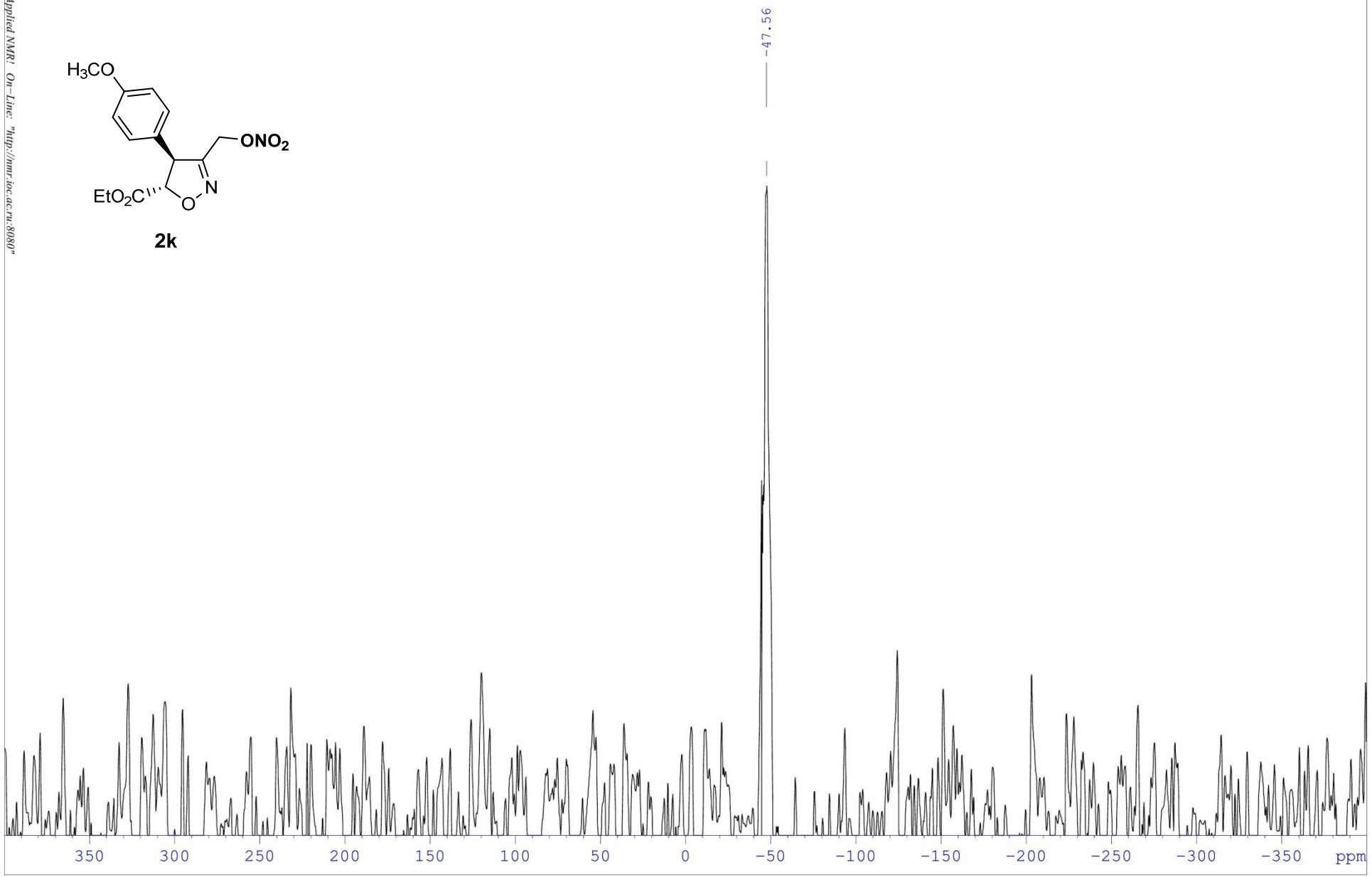
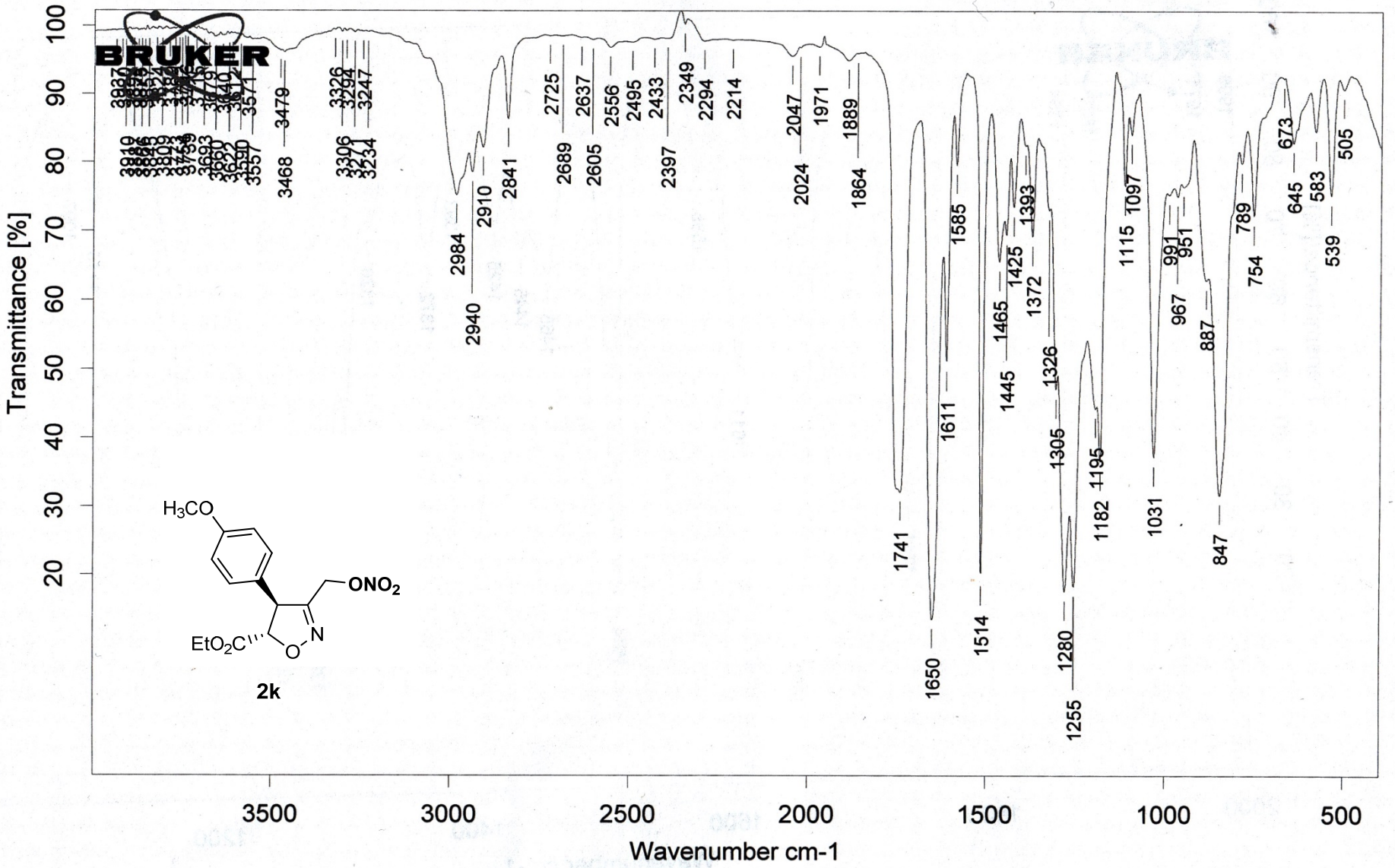
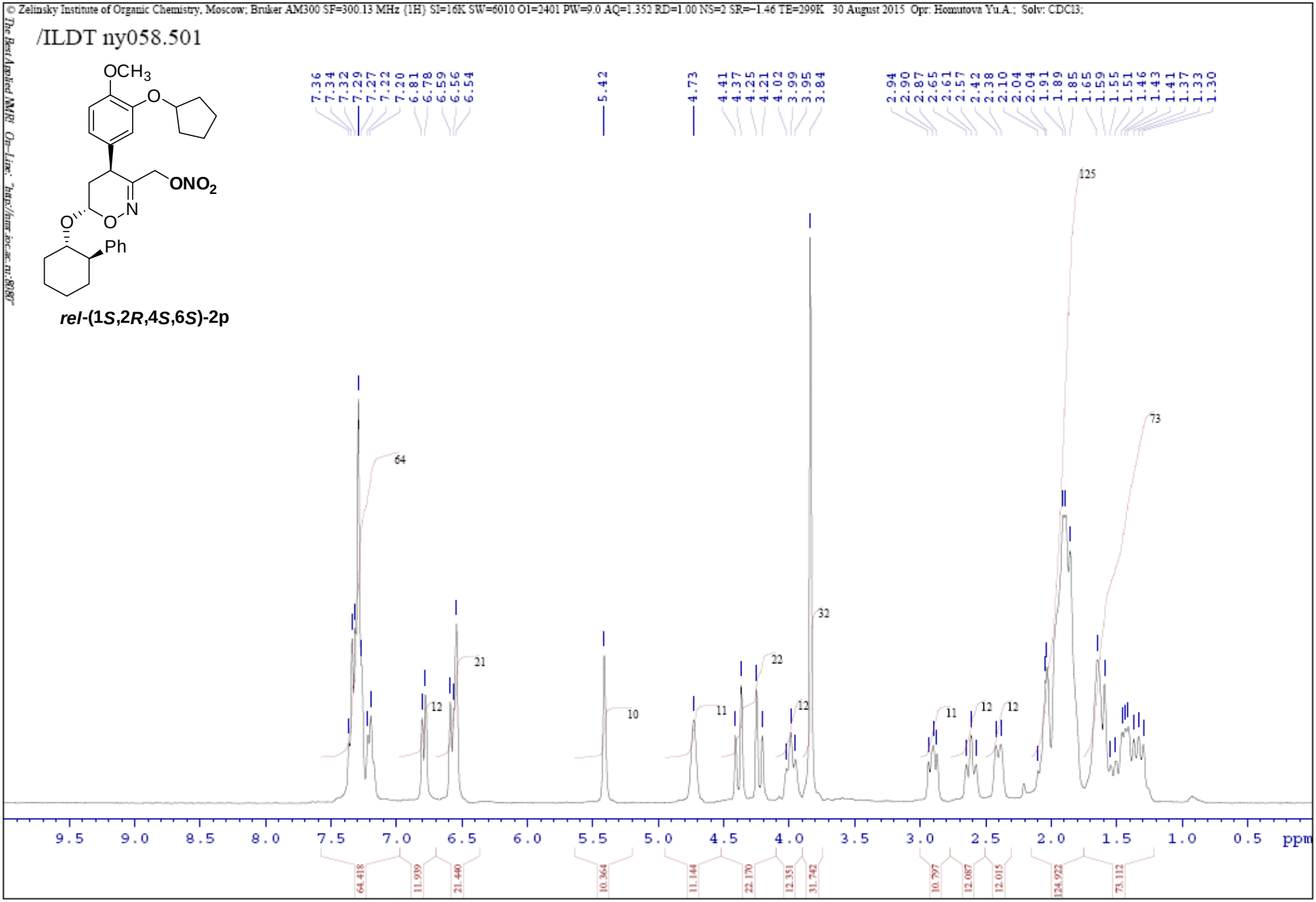




D:\EDL\NY-74.0	NY-74	.KBr.	08.06.2015

/ILDT ny058.501

***rel*-(1*S*,2*R*,4*S*,6*S*)-2p**



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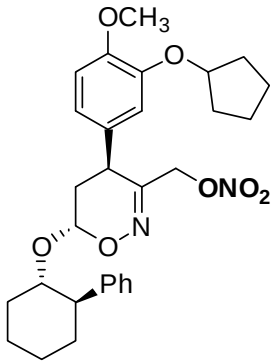
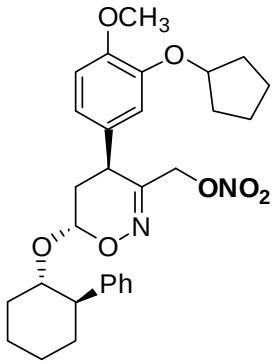


***rel*-(1*S*,2*R*,4*S*,6*S*)-2p**

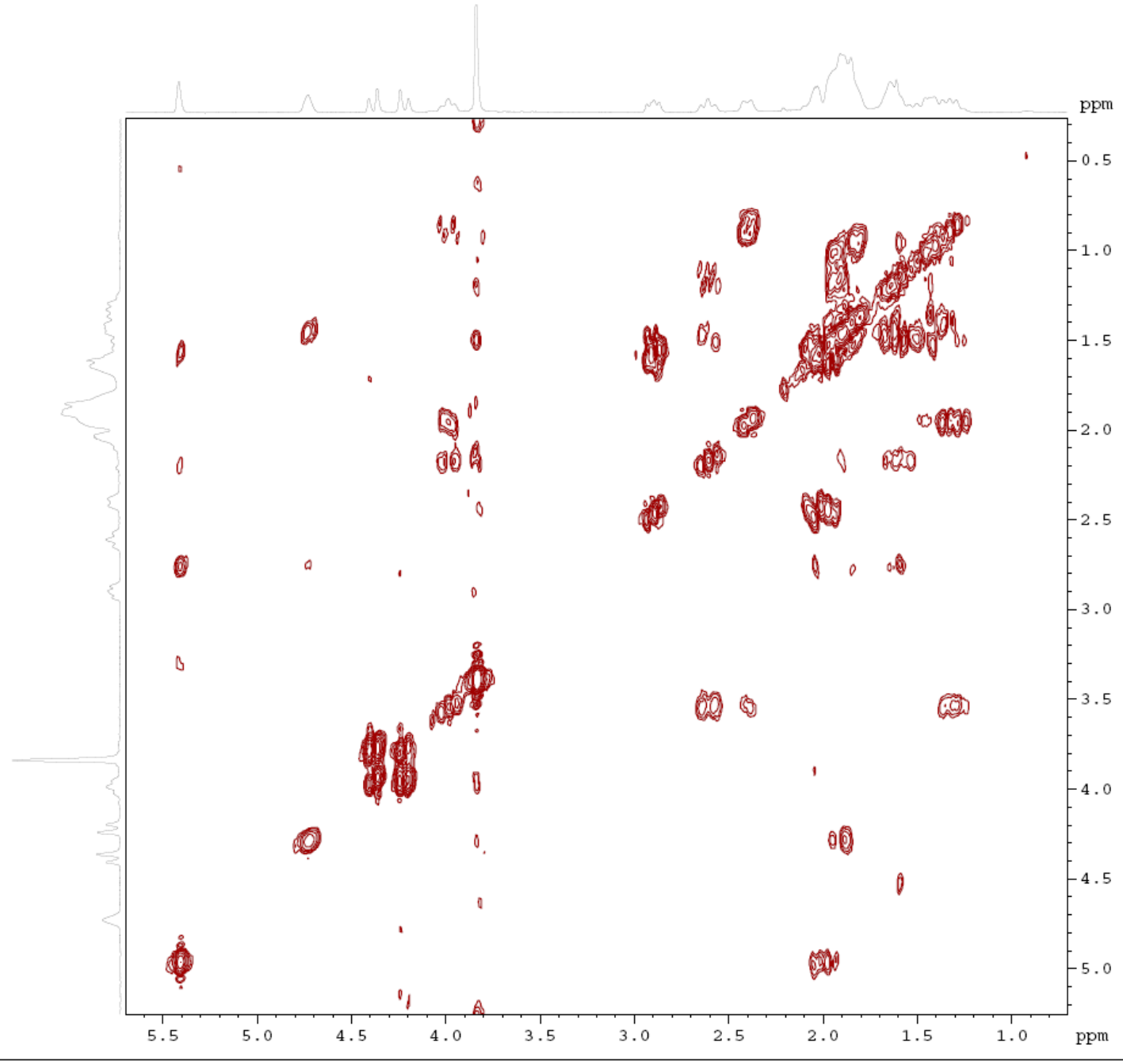
153.06 149.70 148.16 144.45 130.08 128.06 127.88 125.86 120.63 114.79 112.39 91.06 80.57 77.44 77.22 77.02 76.60 76.30 70.63 56.09 50.88 33.99 33.64 32.77 32.73 31.81 30.52 26.11 24.64 24.02

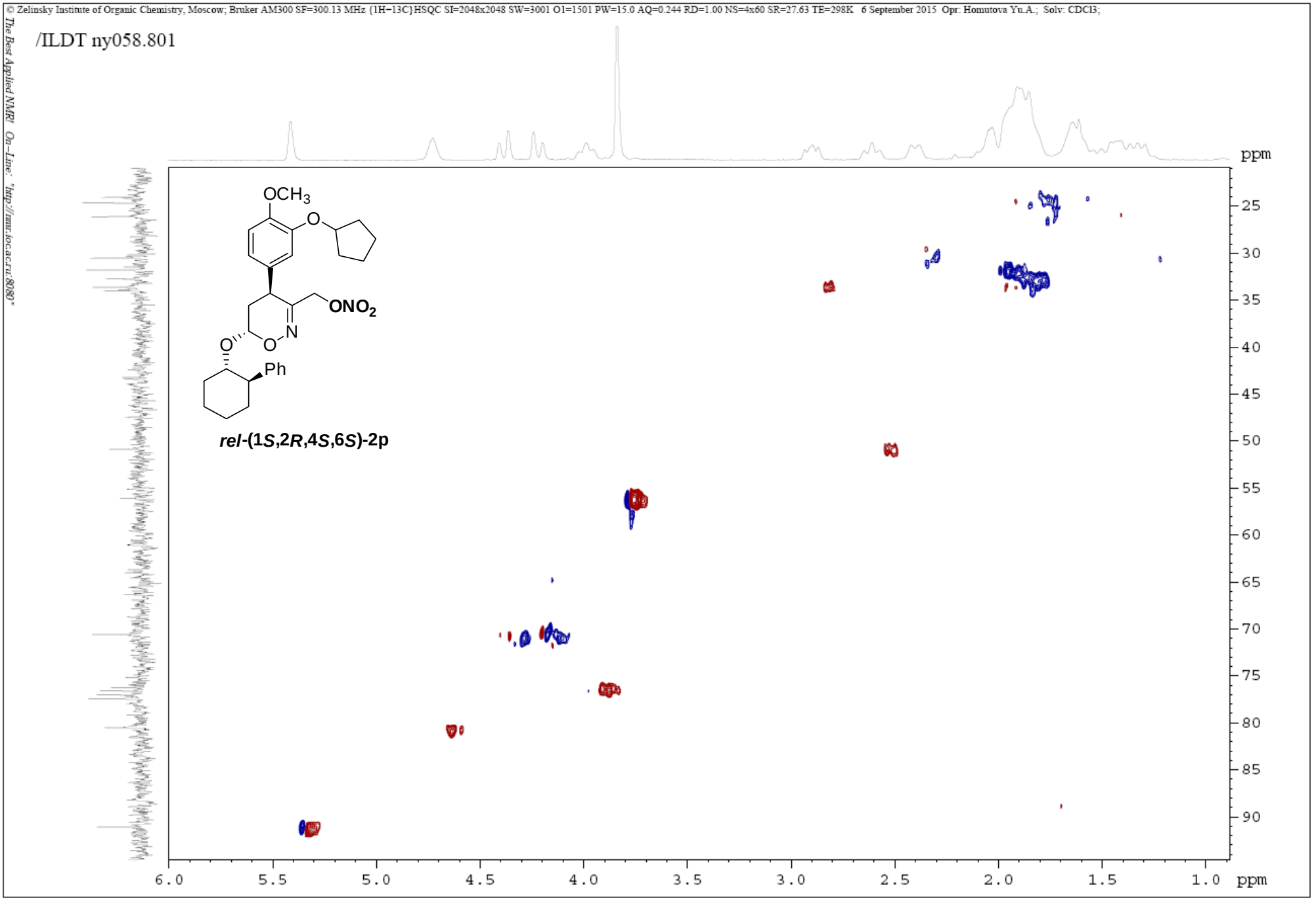
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

/ILDT ny058.801



***rel*-(1*S*,2*R*,4*S*,6*S*)-2p**

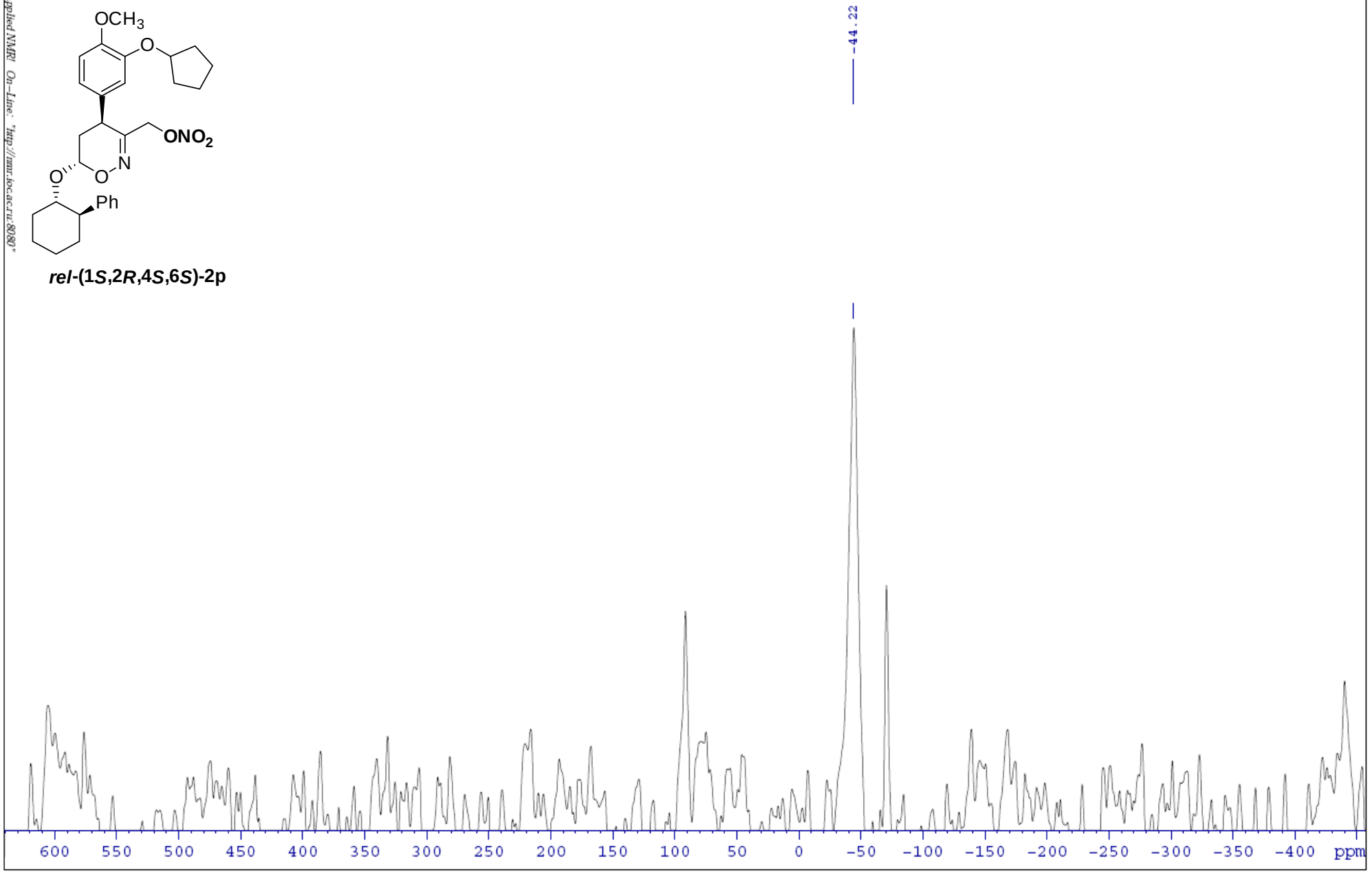



© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H-13C}HSQC SI=2048x2048 SW=3001 O1=1501 PW=15.0 AQ=0.244 RD=1.00 NS=4x60 SR=27.63 TE=298K 6 September 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
© The Best Applied NMR' On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT ny058.801
OCH3
O
ONO2
N
O
O
Ph
rel-(1S,2R,4S,6S)-2p
ppm
25
30
35
40
45
50
55
60
65
70
75
80
85
90
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=21.69 MHz {14N} SI=8K SW=23807 O1=9310 PW=10.0 AQ=0.083 RD=0.05 NS=2553 SR=7317.05 TE=297K 6 September 2015 Opr: Homutova Yu.A.; Solv: CDCl3;

/ILDT ny058.801

***rel*-(1*S*,2*R*,4*S*,6*S*)-2p**

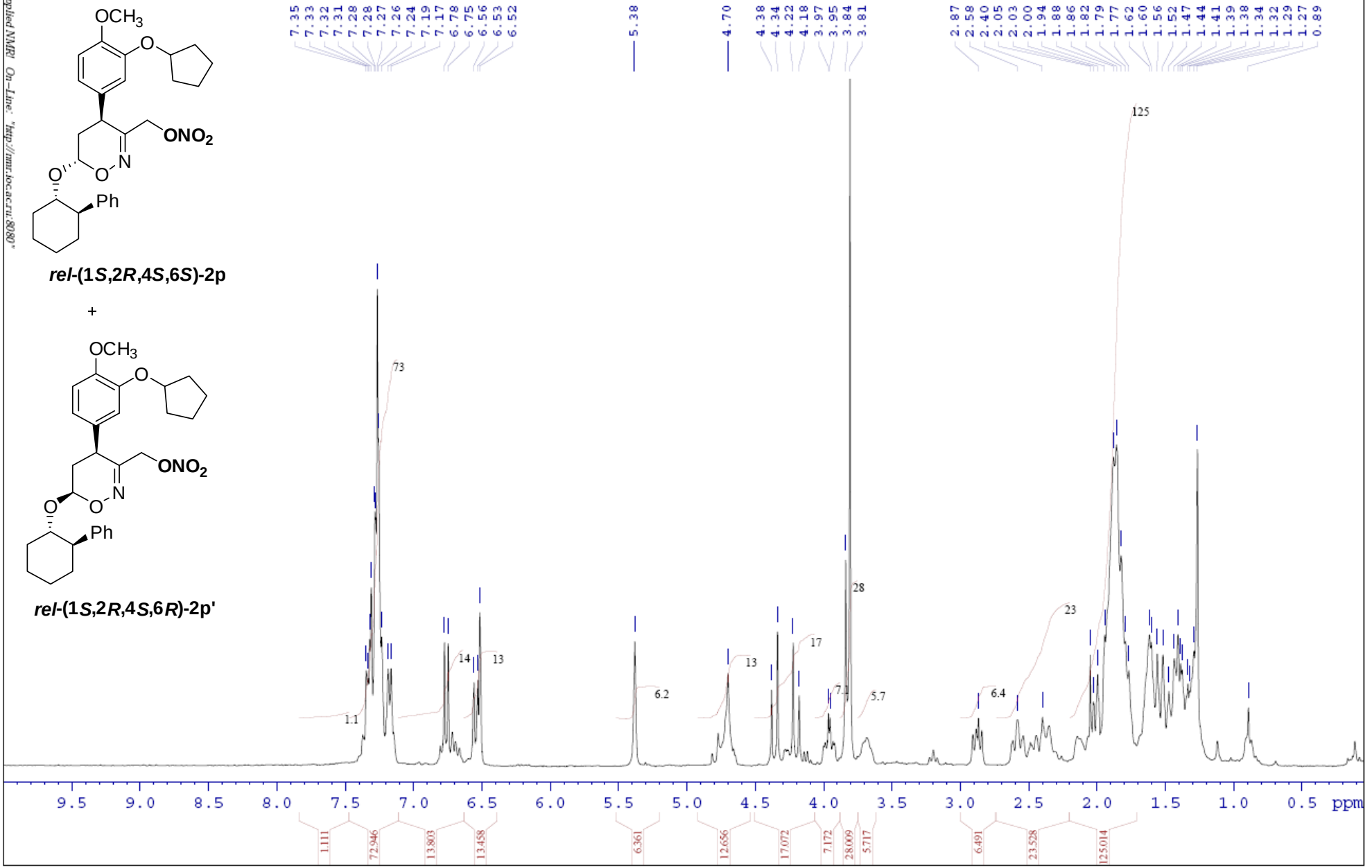


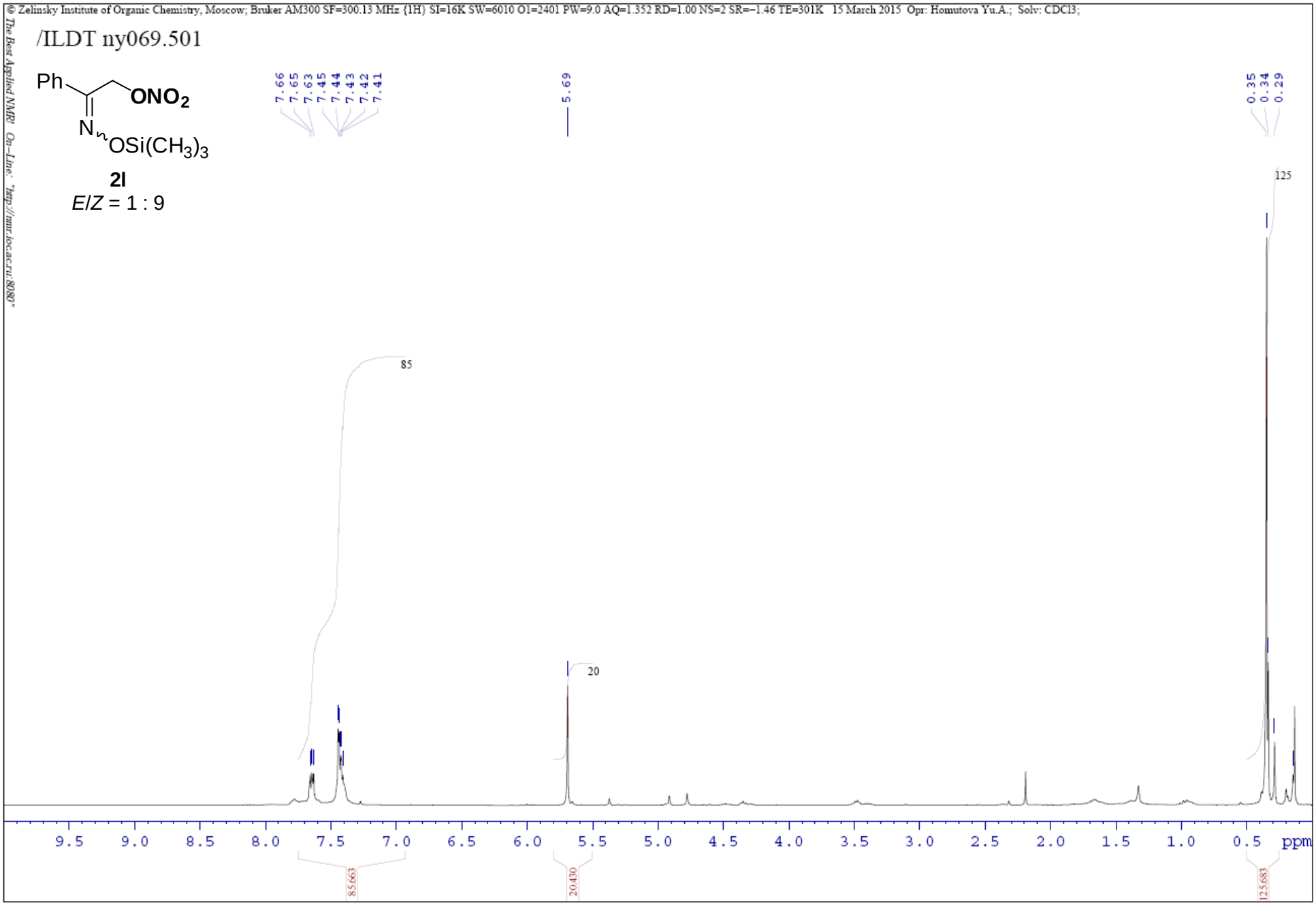
/ILDT NY058.201

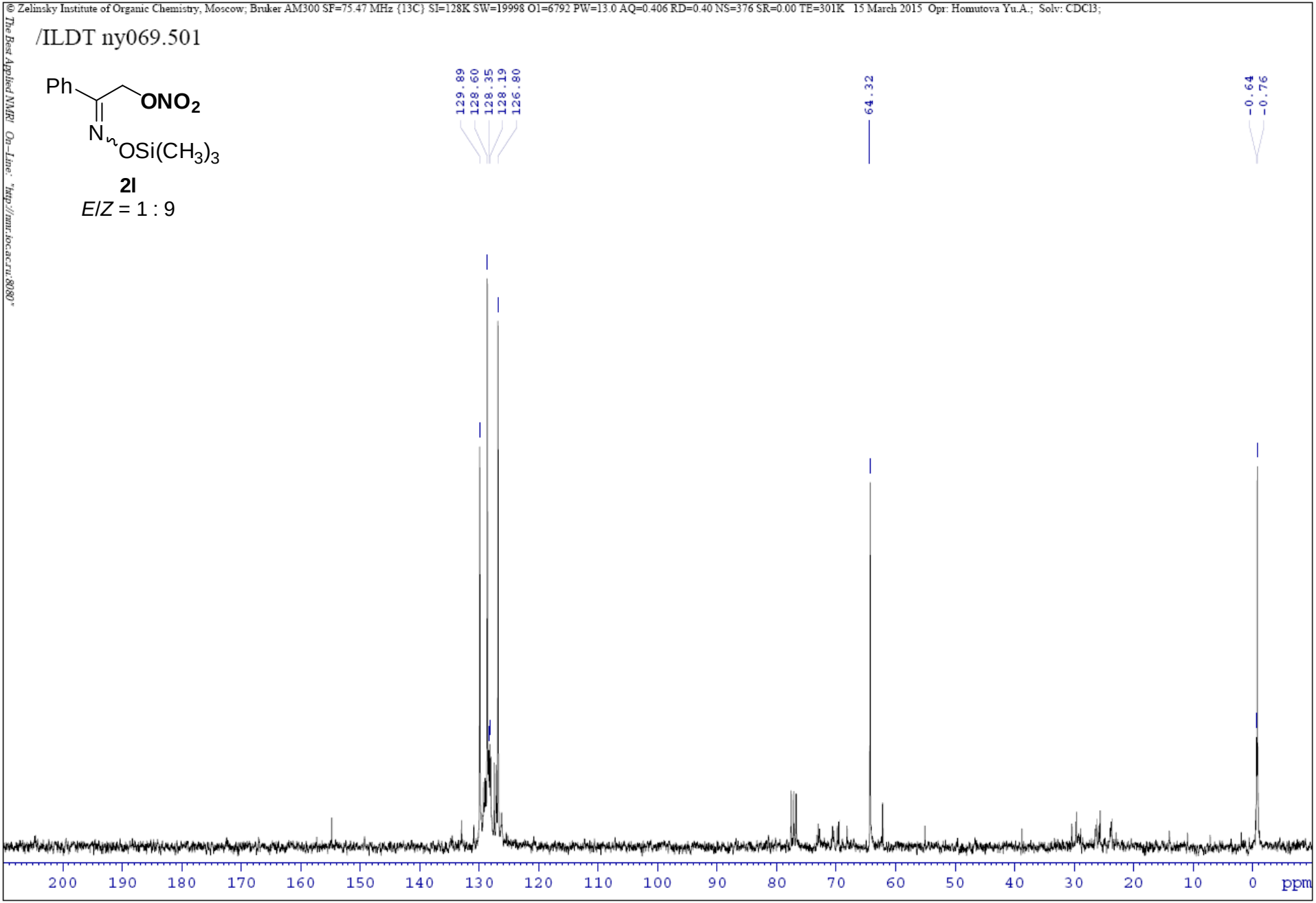
***rel*-(1*S*,2*R*,4*S*,6*S*)-2p**

+

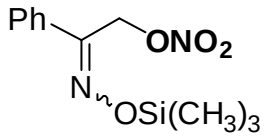
***rel*-(1*S*,2*R*,4*S*,6*R*)-2p'**




© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=1.00 NS=2 SR=-1.46 TE=301K 15 March 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT ny069.501
Ph
ONO2
N
OSi(CH3)3
2l
E/Z = 1 : 9
7.66
7.65
7.63
7.45
7.44
7.43
7.42
7.41
5.69
0.35
0.34
0.29
125
85
20
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
85.663
20.430
125.683

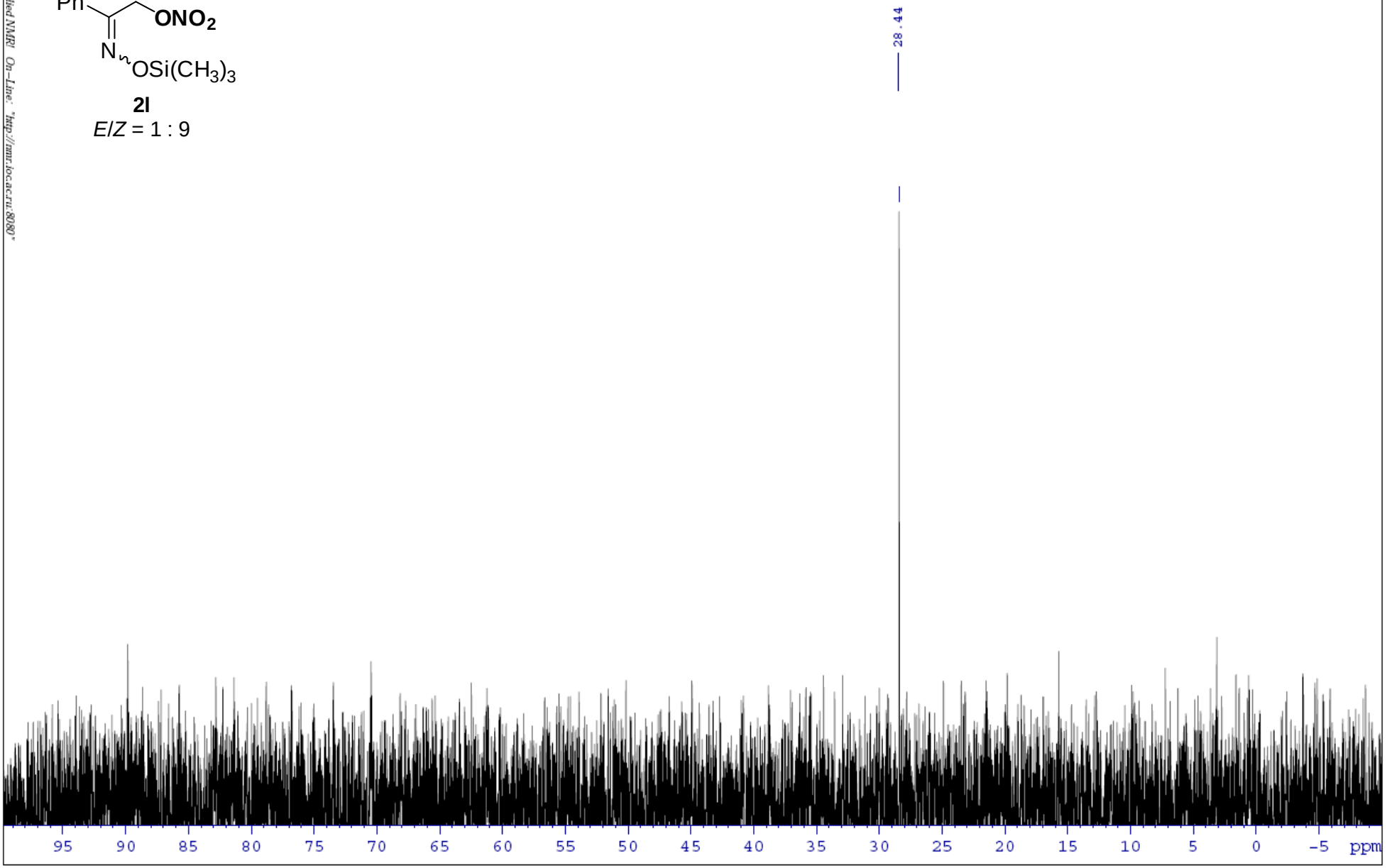

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=128K SW=19998 O1=6792 PW=13.0 AQ=0.406 RD=0.40 NS=376 SR=0.00 TE=301K 15 March 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR On-Line" "http://nmr.ioc.ac.ru:8080"
/ILDT ny069.501
Ph
ONO2
N
OSi(CH3)3
2l
E/Z = 1 : 9
129.89
128.60
128.35
128.19
126.80
64.32
-0.64
-0.76
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

/ILDT ny069.501



2l

E/*Z* = 1 : 9

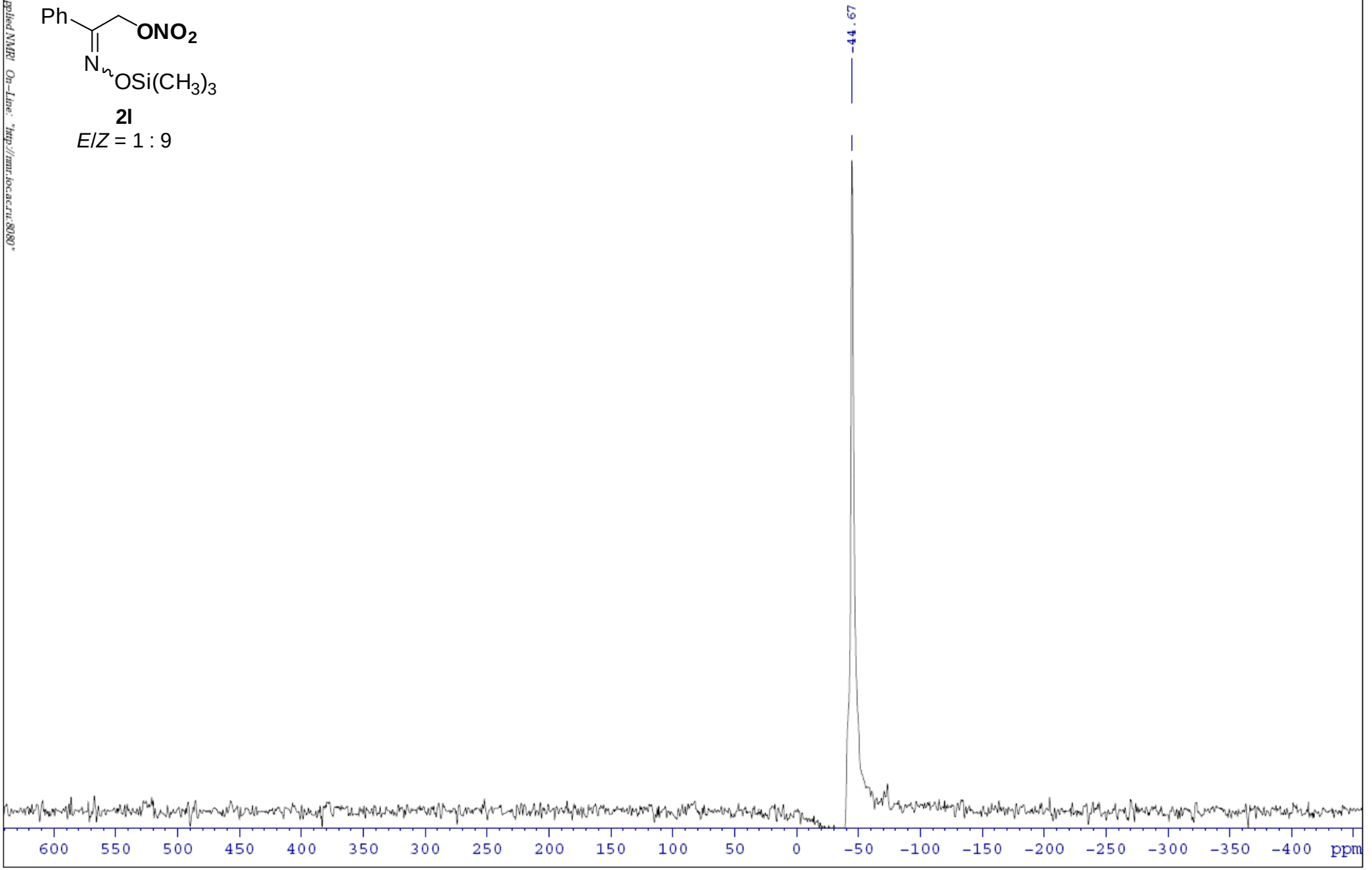


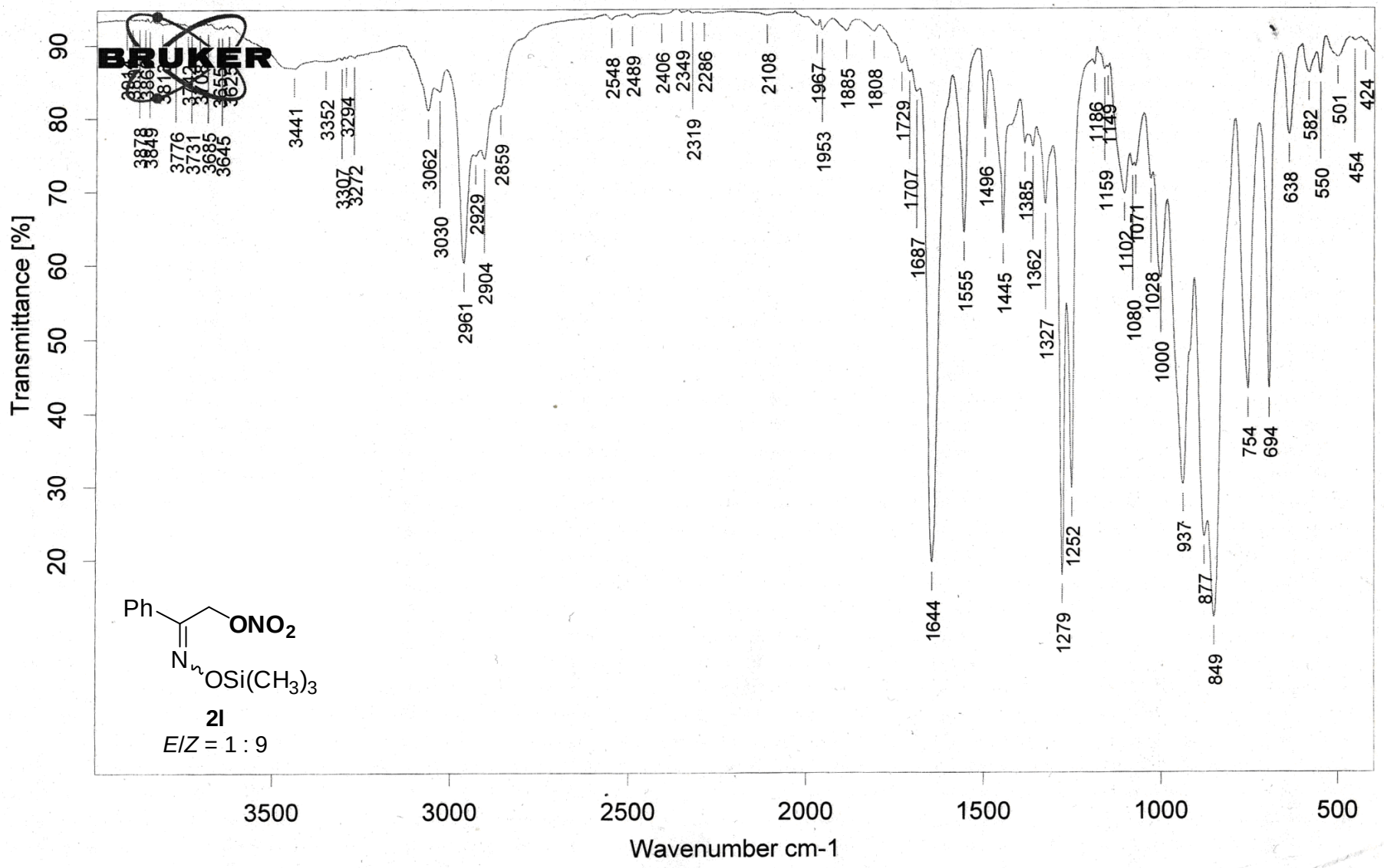
© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=21.69 MHz {14N} SI=8K SW=23807 O1=9310 PW=10.0 AQ=0.083 RD=0.05 NS=272 SR=7317.05 TE=301K 15 March 2015 Opr: Homutova Yu.A.; Solv: CDCl3;

/ILDT ny069.501

2l

E/*Z* = 1 : 9





2l

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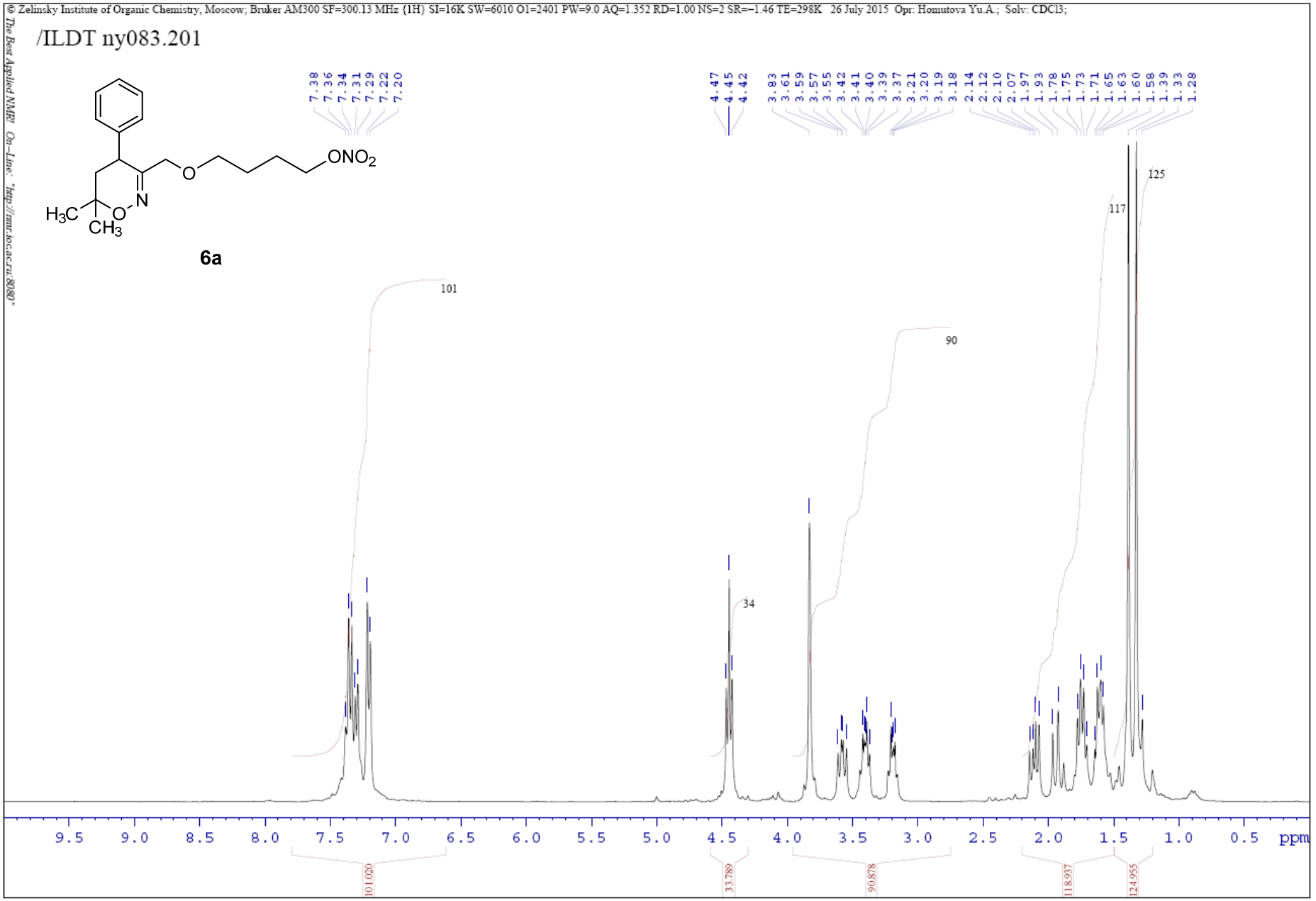
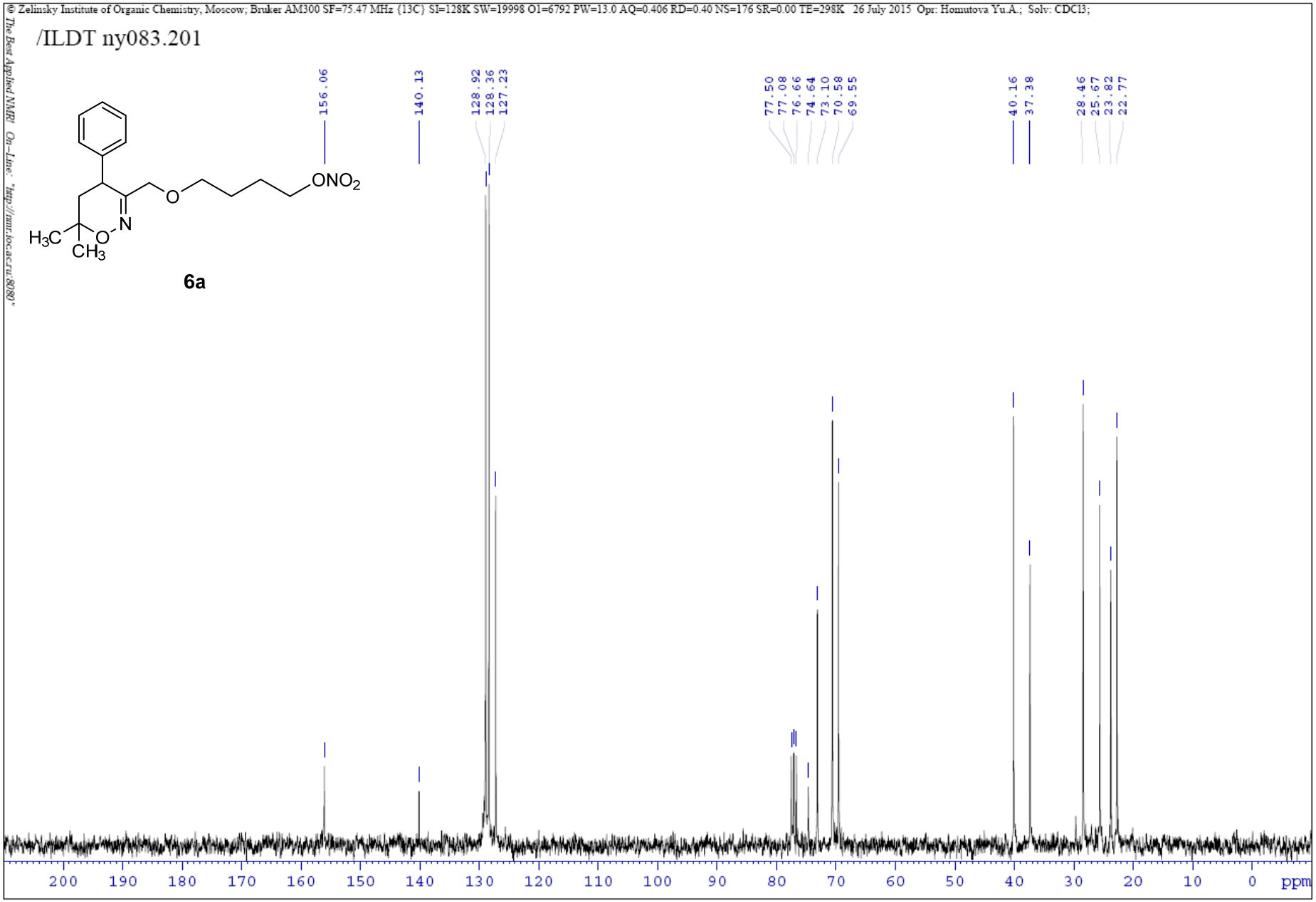
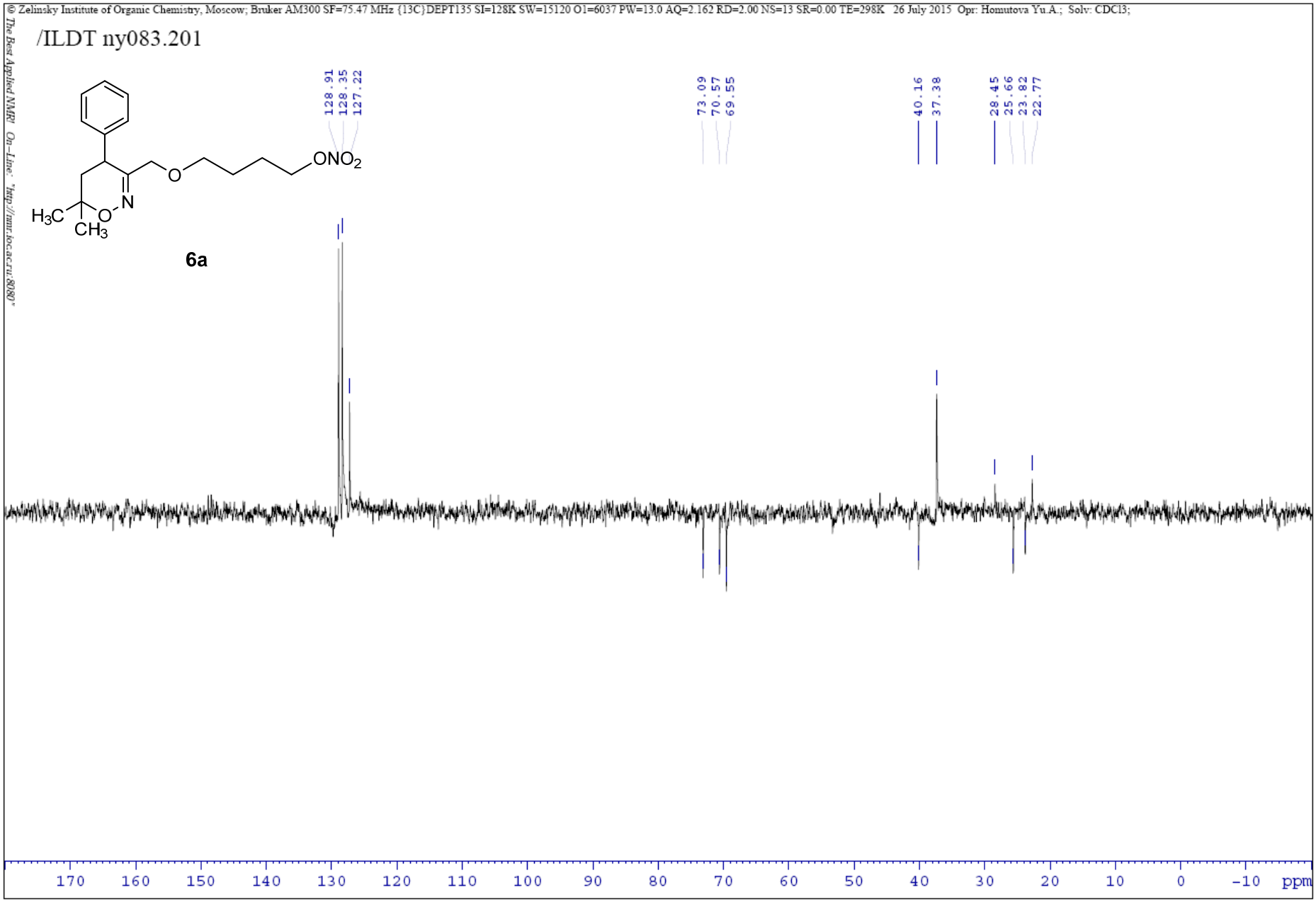
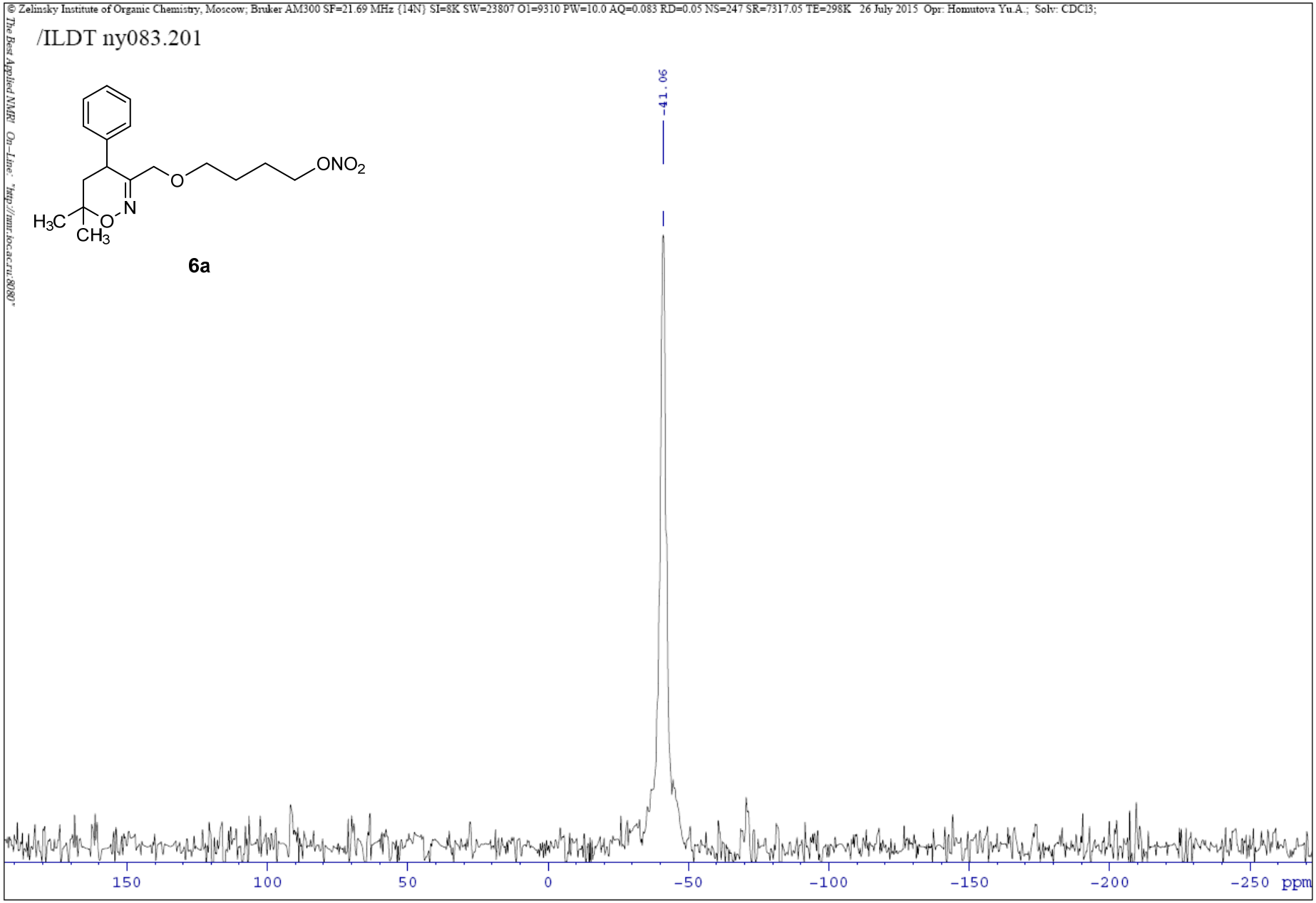
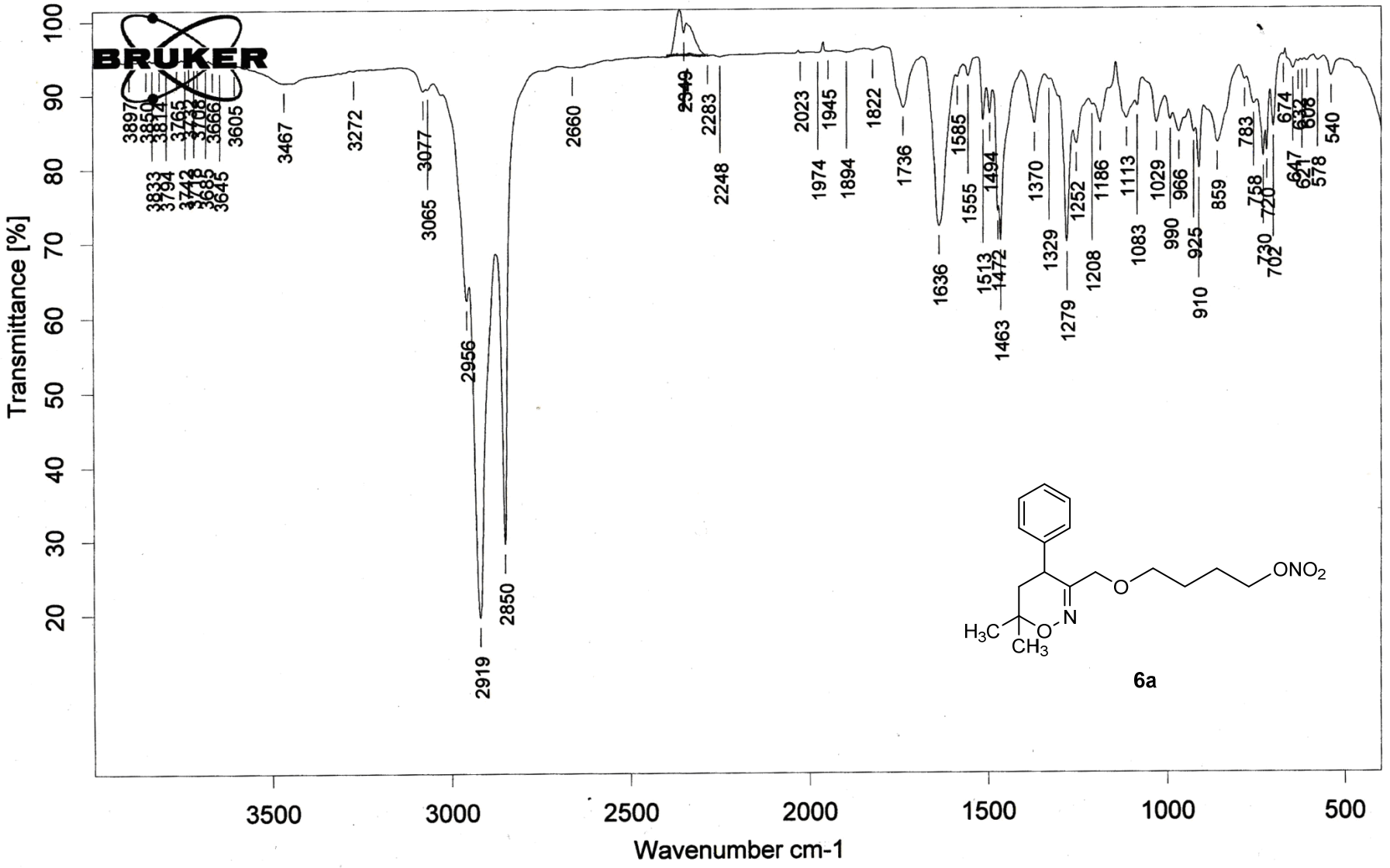
D:\EDL\NY-044.0	NY-044	20.10.2014

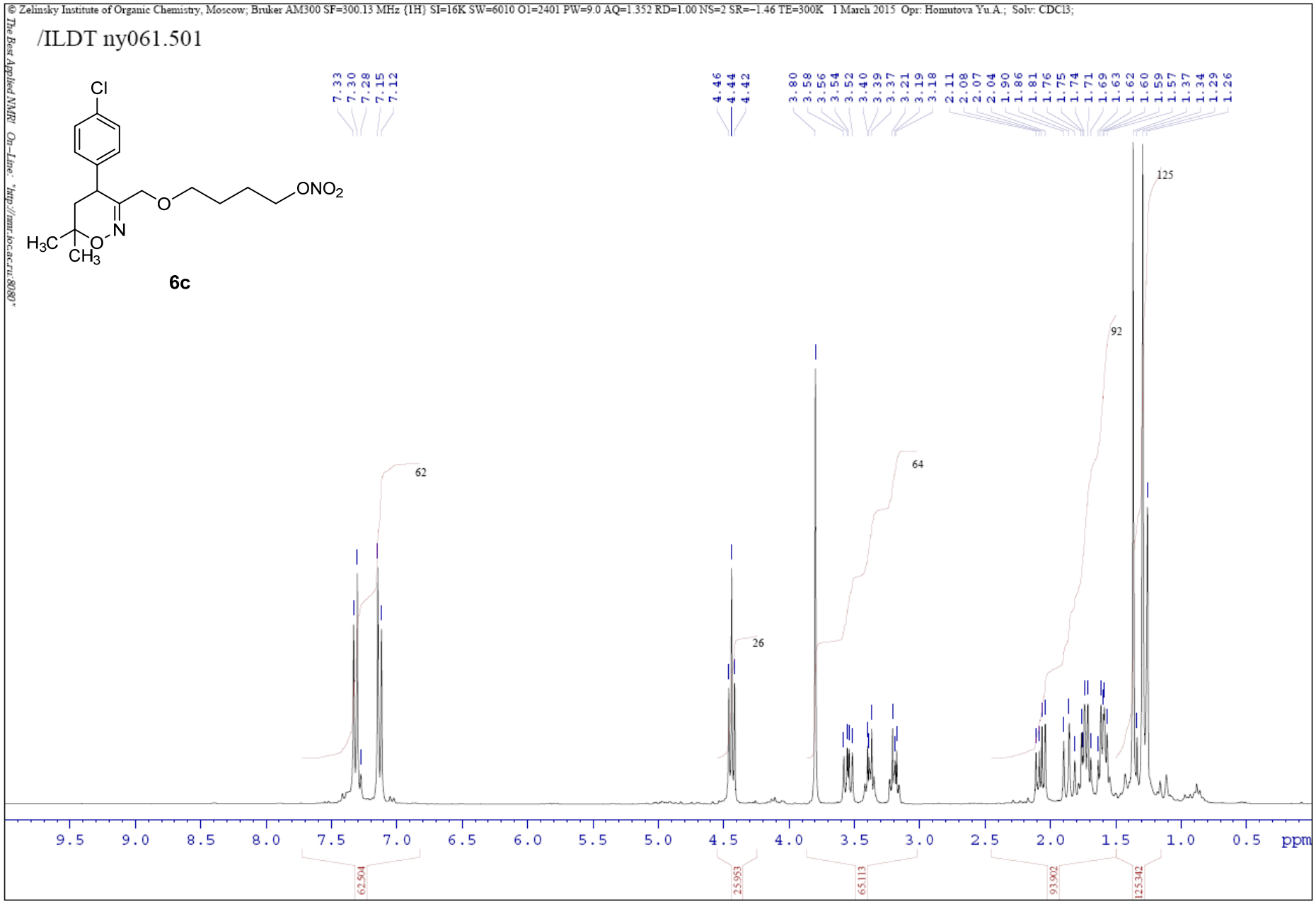

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=1.00 NS=2 SR=-1.46 TE=298K 26 July 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT ny083.201
7.38 7.36 7.34 7.31 7.29 7.22 7.20
4.47 4.45 4.42 3.83 3.61 3.59 3.57 3.55 3.42 3.41 3.40 3.39 3.37 3.21 3.20 3.19 3.18 2.14 2.12 2.10 2.07 1.97 1.93 1.78 1.75 1.73 1.71 1.65 1.63 1.60 1.58 1.39 1.33 1.28
ONO2
O
N
H3C
O
CH3
6a
101
90
34
117
125
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
101.020
33.789
90.878
118.937
124.955

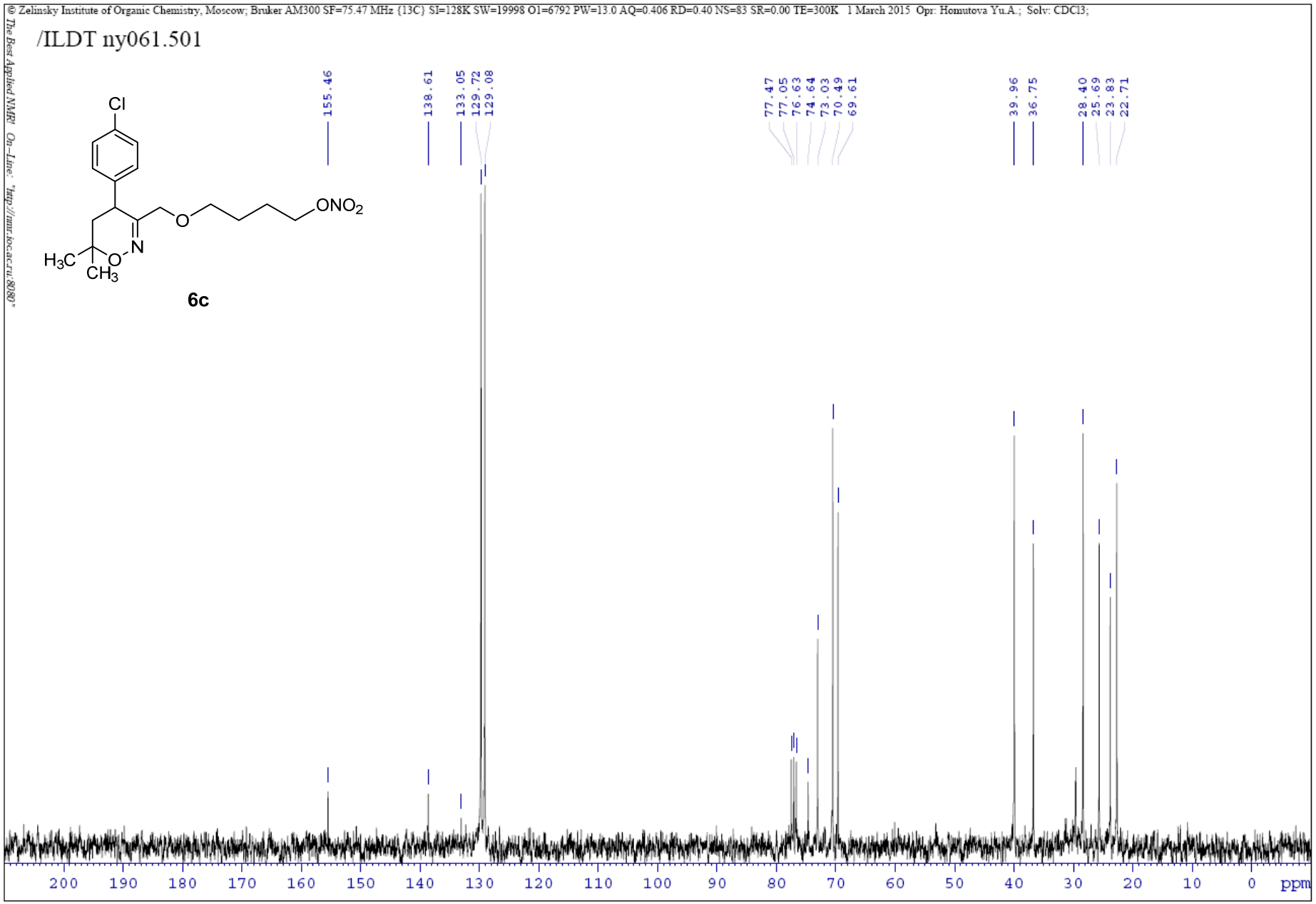

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=128K SW=19998 O1=6792 PW=13.0 AQ=0.406 RD=0.40 NS=176 SR=0.00 TE=298K 26 July 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT ny083.201
156.06
140.13
128.92
128.36
127.23
77.50
77.08
76.66
74.64
73.10
70.58
69.55
40.16
37.38
28.46
25.67
23.82
22.77
ONO2
O
N
H3C
O
CH3
6a
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

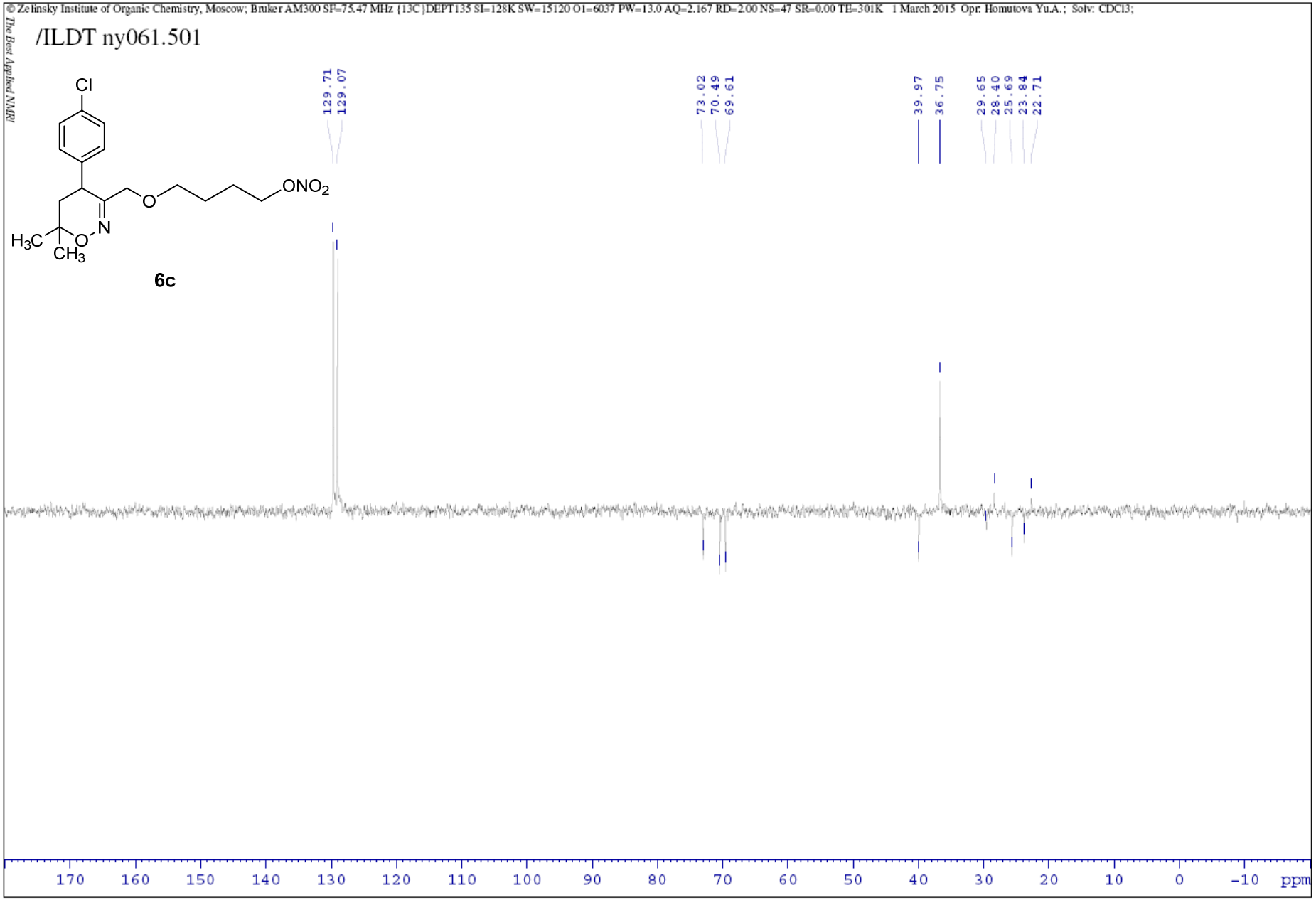

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C}DEPT135 SI=128K SW=15120 O1=6037 PW=13.0 AQ=2.162 RD=2.00 NS=13 SR=0.00 TE=298K 26 July 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT ny083.201
128.91
128.35
127.22
73.09
70.57
69.55
40.16
37.38
28.45
25.66
23.82
22.77
ONO2
O
N
H3C
O
CH3
6a
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm

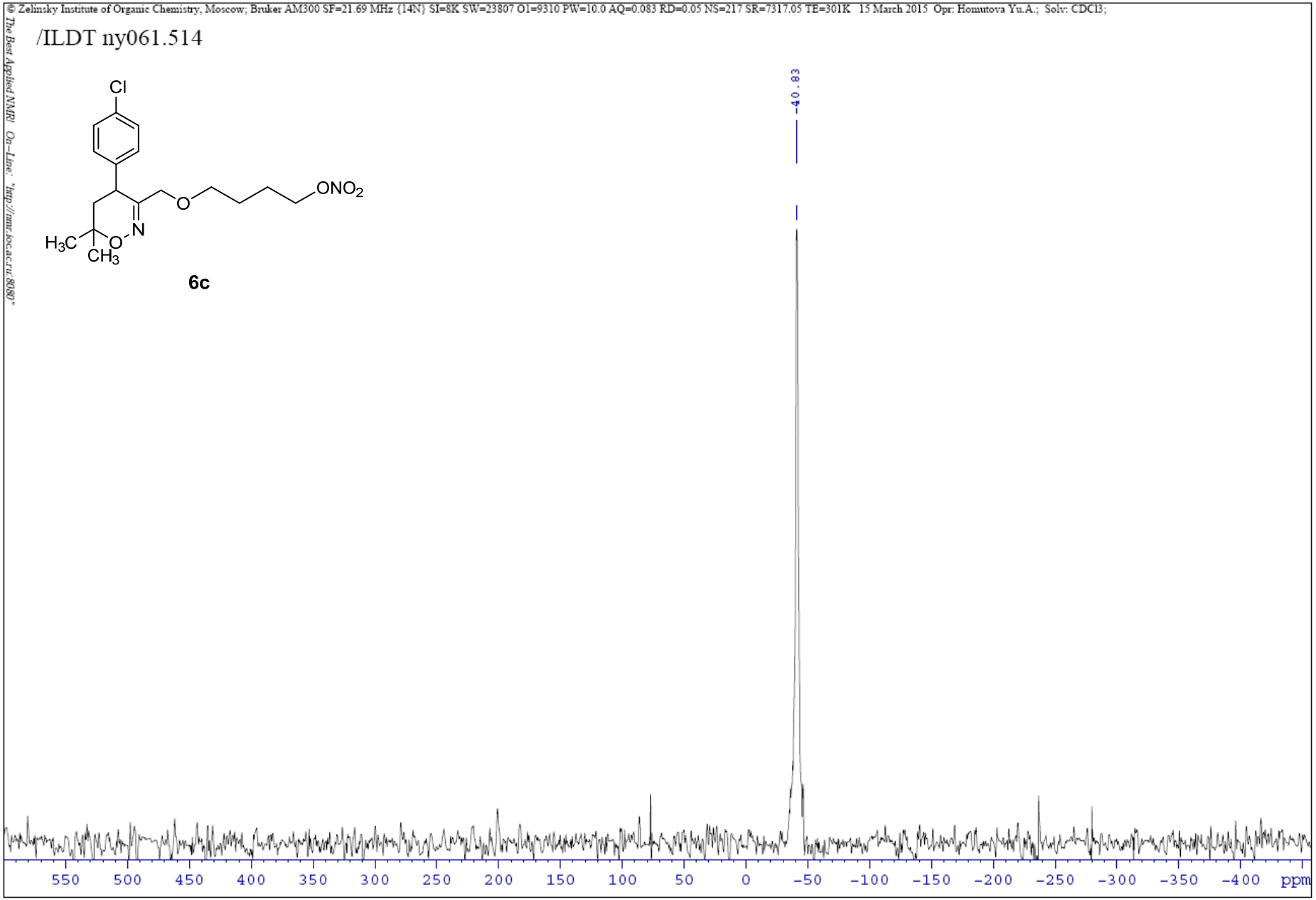

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=21.69 MHz {14N} SI=8K SW=23807 O1=9310 PW=10.0 AQ=0.083 RD=0.05 NS=247 SR=7317.05 TE=298K 26 July 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT ny083.201
-41.06
ONO2
O
N
H3C
O
CH3
6a
150
100
50
0
-50
-100
-150
-200
-250 ppm

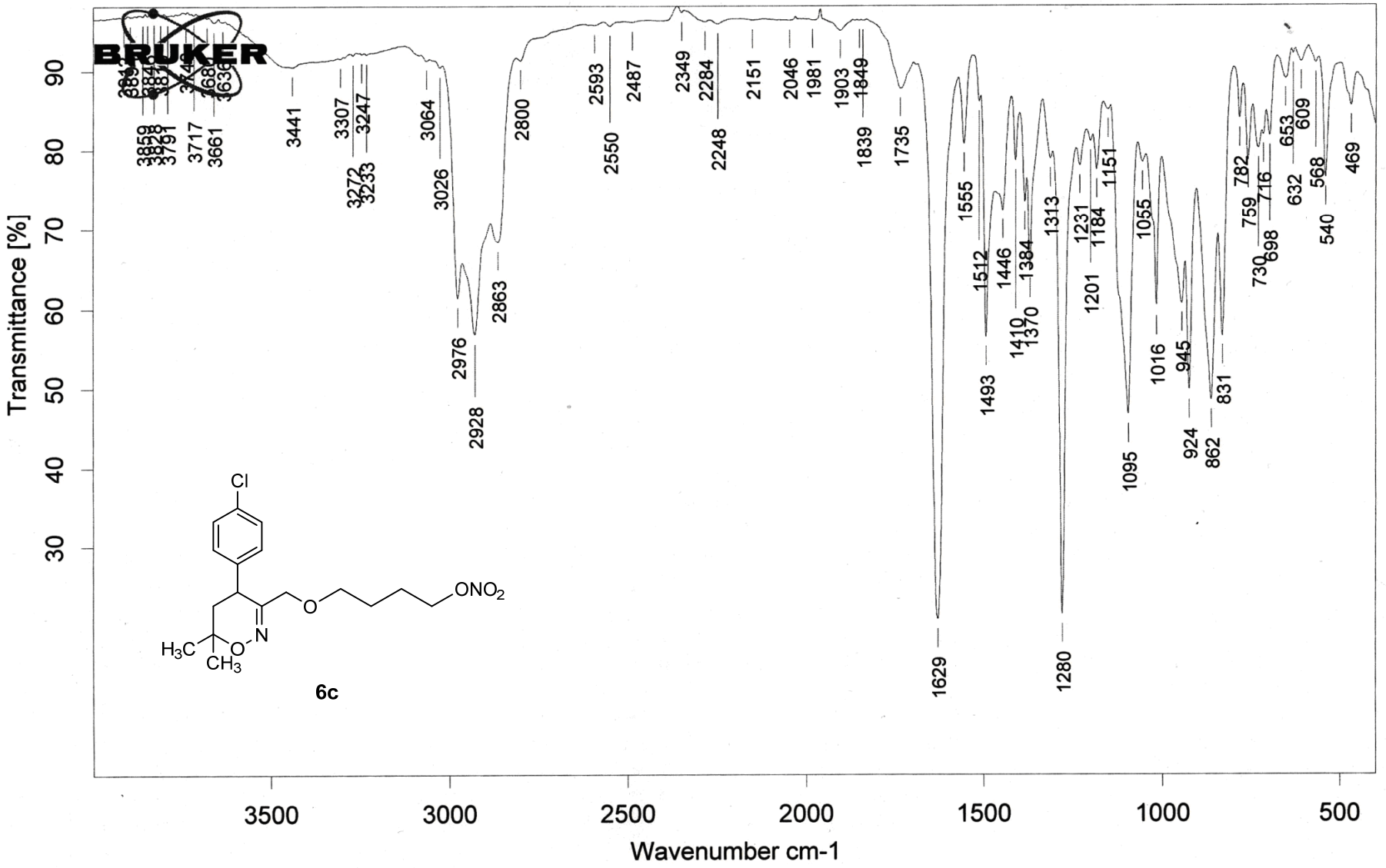

BRUKER
Transmittance [%]
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
20
30
40
50
60
70
80
90
100
3897
3850
3814
3833
3794
3742
3718
3685
3645
3605
3467
3272
3077
3065
2956
2919
2850
2660
2346
2283
2248
2023
1974
1945
1894
1822
1736
1636
1585
1555
1513
1494
1472
1463
1370
1329
1279
1252
1208
1186
1113
1083
1029
990
966
925
910
859
783
758
730
720
702
674
647
621
608
578
540
ONO2
O
N
O
H3C
CH3
6a
D:\EDL\NY-046.0


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=1.00 NS=2 SR=-1.46 TE=300K 1 March 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
/ILDT ny061.501
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
Cl
ONO2
O
N
H3C
O
CH3
6c
7.33 7.30 7.28 7.15 7.12
4.46 4.44 4.42
3.80 3.58 3.56 3.54 3.52 3.40 3.39 3.37 3.21 3.19 3.18
2.11 2.08 2.07 2.04 1.90 1.86 1.81 1.76 1.75 1.74 1.71 1.69 1.63 1.62 1.60 1.59 1.57 1.37 1.34 1.29 1.26
62
26
64
92
125
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
62.504
25.953
65.113
93.902
125.342


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=128K SW=19998 O1=6792 PW=13.0 AQ=0.406 RD=0.40 NS=83 SR=0.00 TE=300K 1 March 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT ny061.501
155.46
138.61
133.05
129.72
129.08
77.47
77.05
76.63
74.64
73.03
70.49
69.61
39.96
36.75
28.40
25.69
23.83
22.71
Cl
ONO2
O
N
H3C
O
CH3
6c
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

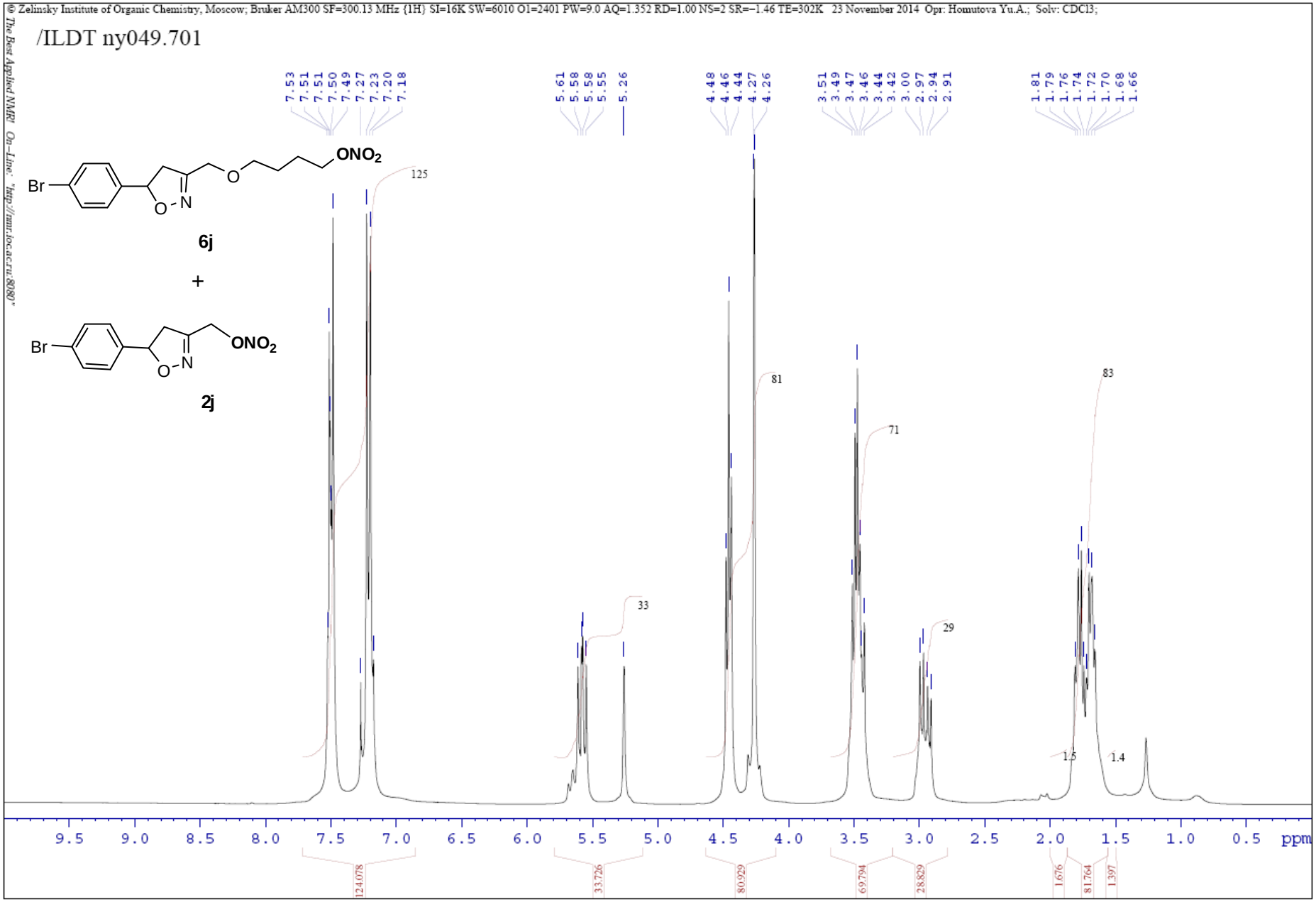

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C}DEPT135 SI=128K SW=15120 O1=6037 PW=13.0 AQ=2.167 RD=2.00 NS=47 SR=0.00 TE=301K 1 March 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR!
/ILDT ny061.501
129.71
129.07
73.02
70.49
69.61
39.97
36.75
29.65
28.40
25.69
23.84
22.71
Cl
ONO2
O
N
H3C
O
CH3
6c
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm

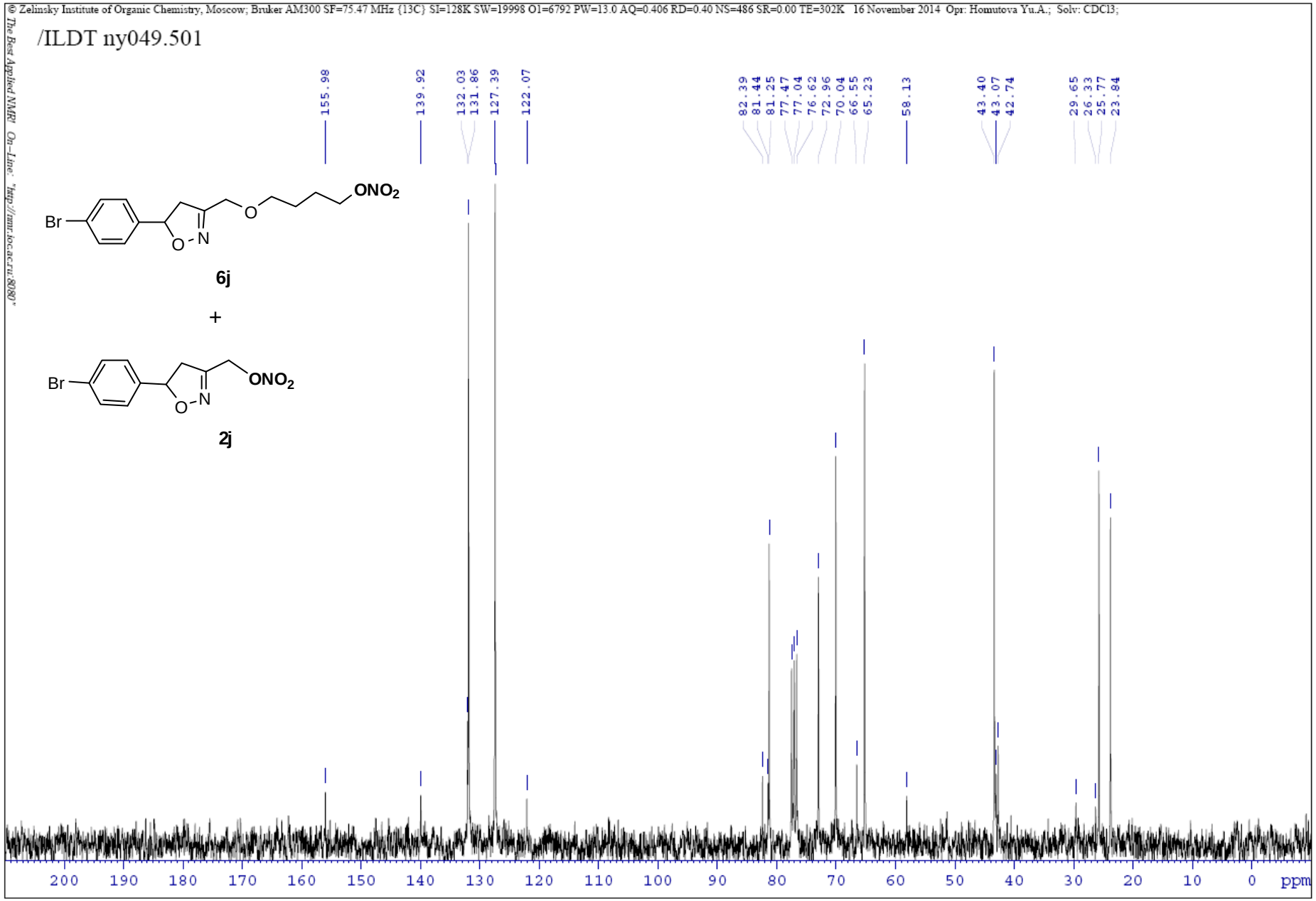

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=21.69 MHz {14N} SI=8K SW=23807 O1=9310 PW=10.0 AQ=0.083 RD=0.05 NS=217 SR=7317.05 TE=301K 15 March 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT ny061.514
Cl
ONO2
O
N
H3C
O
CH3
6c
-40.83
550 500 450 400 350 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -350 -400 ppm



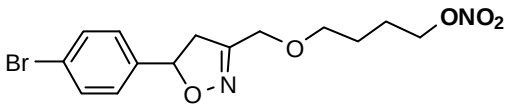
6c

D:\EDL\NY-061.1	NY-061	KBr.	10.06.2015


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=1.00 NS=2 SR=-1.46 TE=302K 23 November 2014 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT ny049.701
7.53 7.51 7.51 7.50 7.49 7.27 7.23 7.20 7.18
5.61 5.58 5.58 5.55 5.26
4.48 4.46 4.44 4.27 4.26
3.51 3.49 3.47 3.46 3.44 3.42 3.00 2.97 2.94 2.91
1.81 1.79 1.76 1.74 1.72 1.70 1.68 1.66
Br
O
ONO2
O
N
6j
+
Br
ONO2
O
N
2j
125
33
81
71
29
83
1.5
1.4
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
124.078
33.726
80.929
69.794
28.829
1.676
81.764
1.397


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=128K SW=19998 O1=6792 PW=13.0 AQ=0.406 RD=0.40 NS=486 SR=0.00 TE=302K 16 November 2014 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT ny049.501
155.98
139.92
132.03
131.86
127.39
122.07
82.39
81.44
81.25
77.47
77.04
76.62
72.96
70.04
66.55
65.23
58.13
43.40
43.07
42.74
29.65
26.33
25.77
23.84
Br
O
N
ONO2
6j
+
Br
ONO2
2j
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

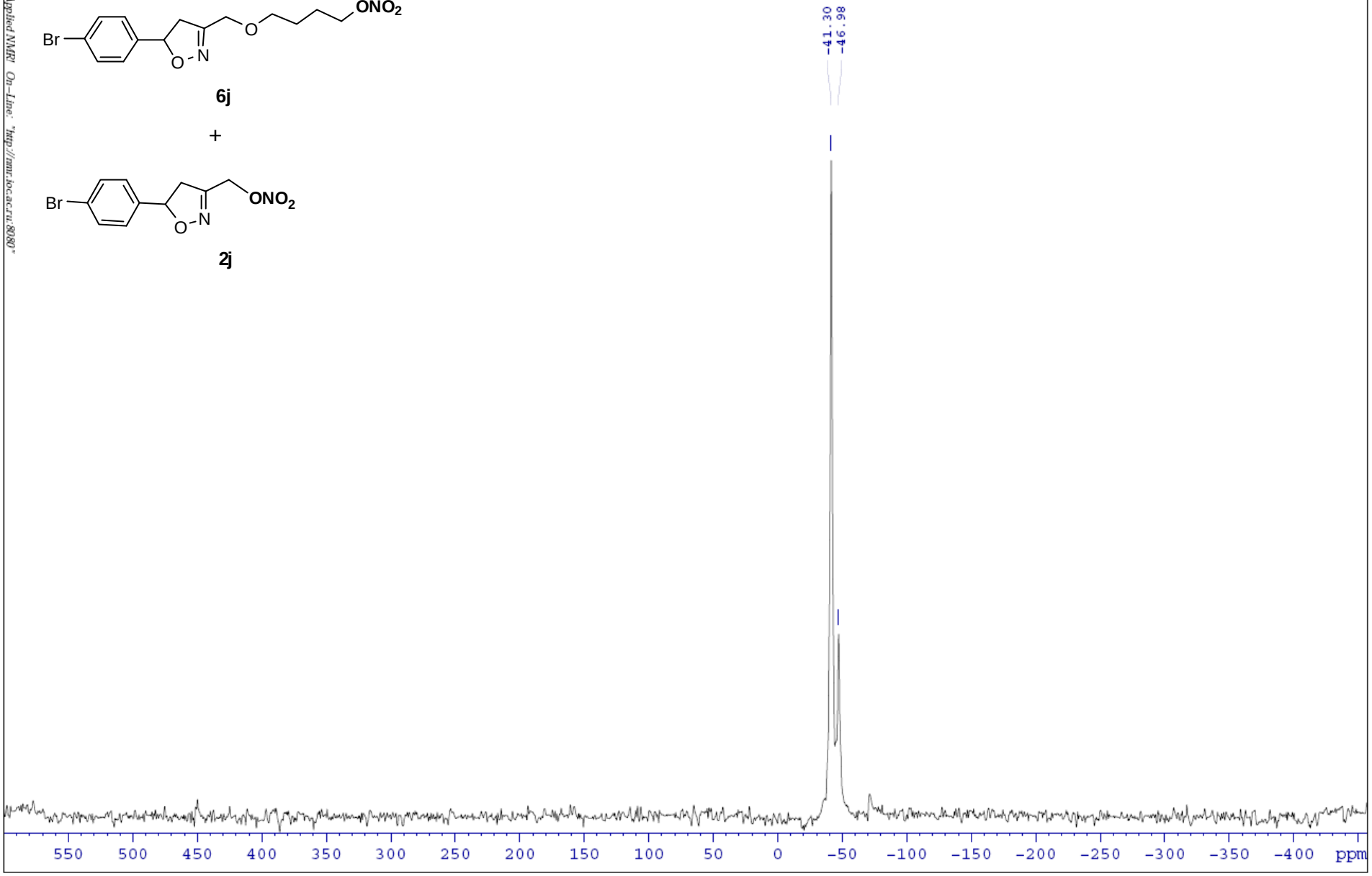
/ILDT ny049.701

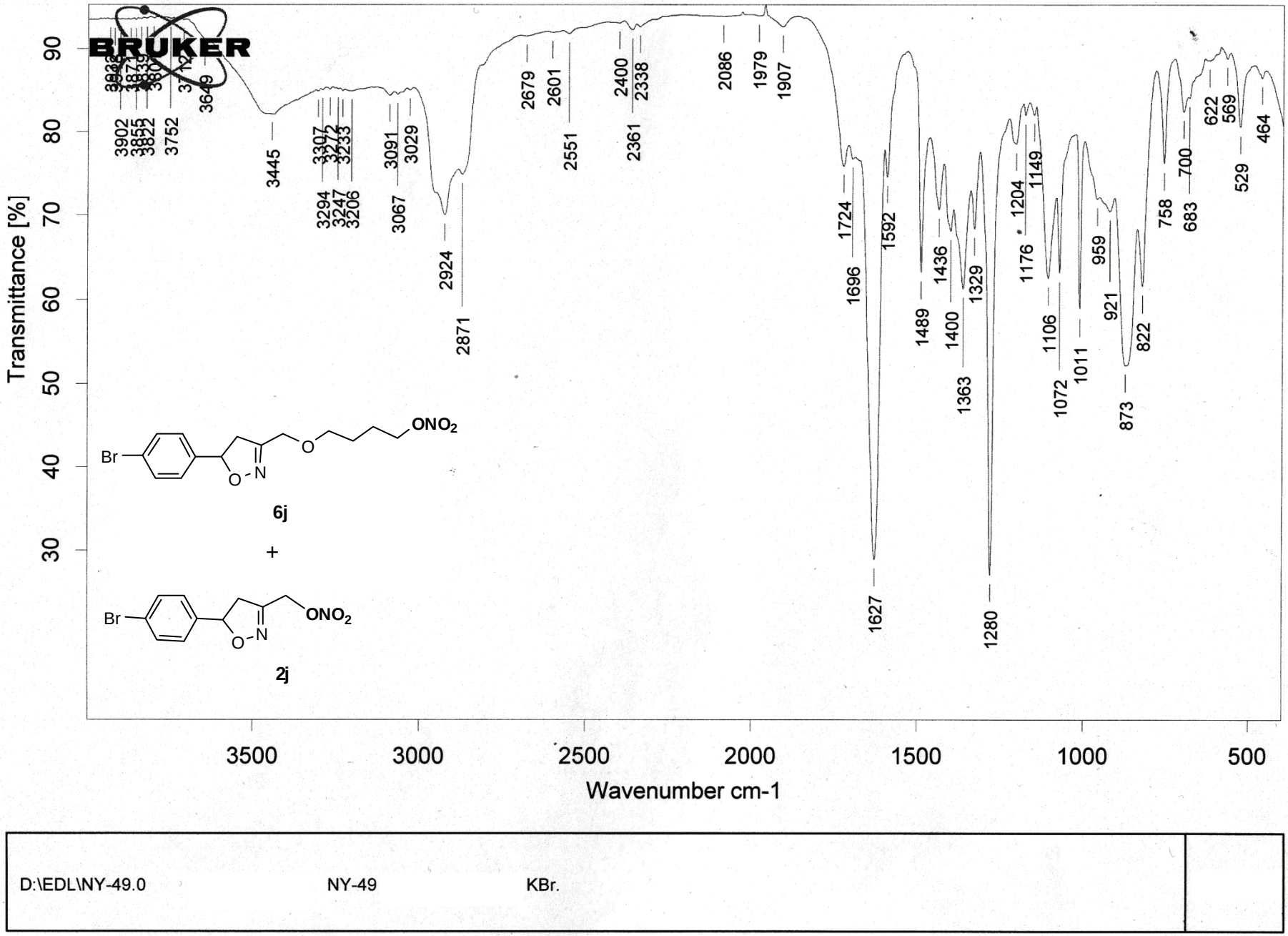


6j

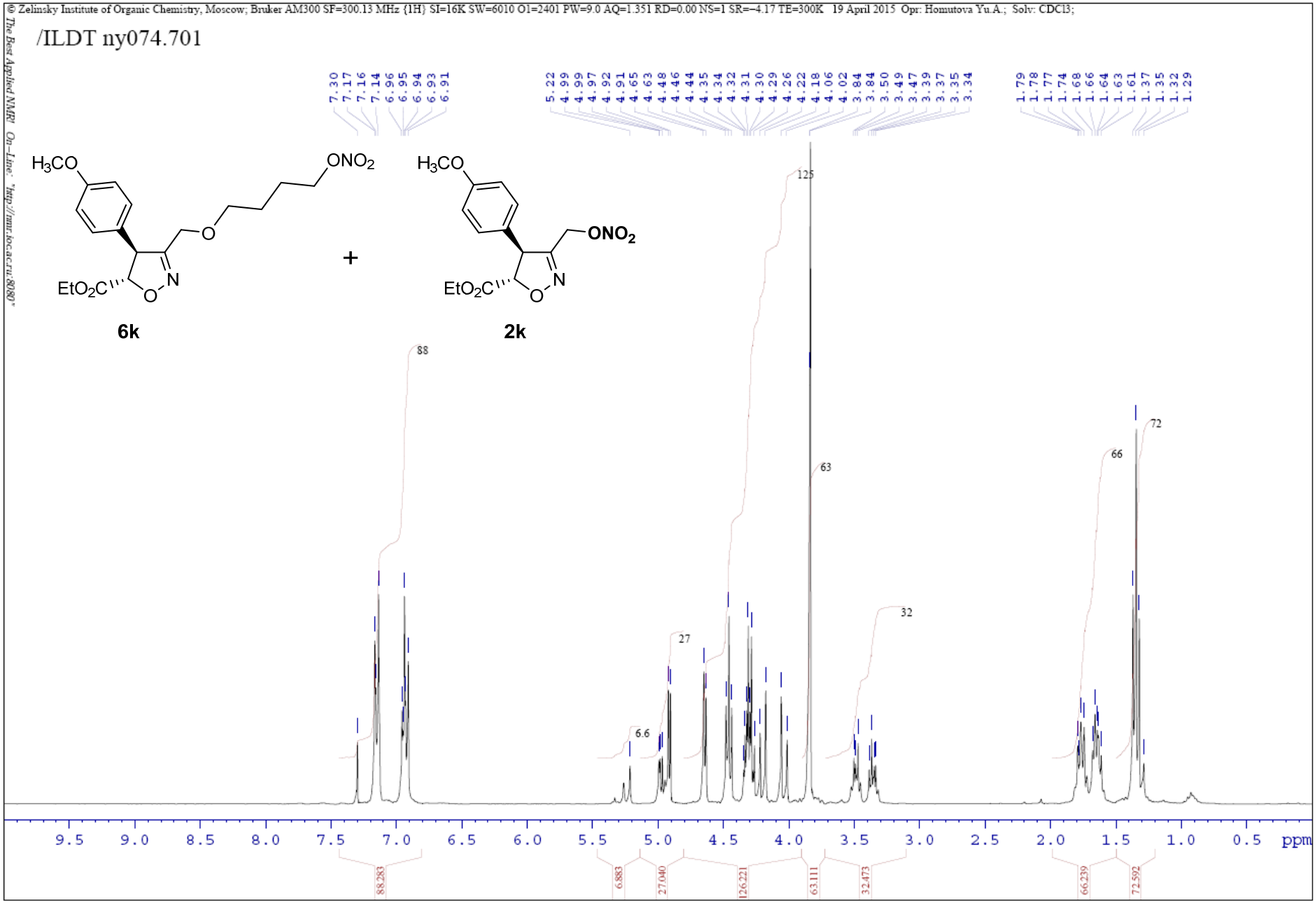
+

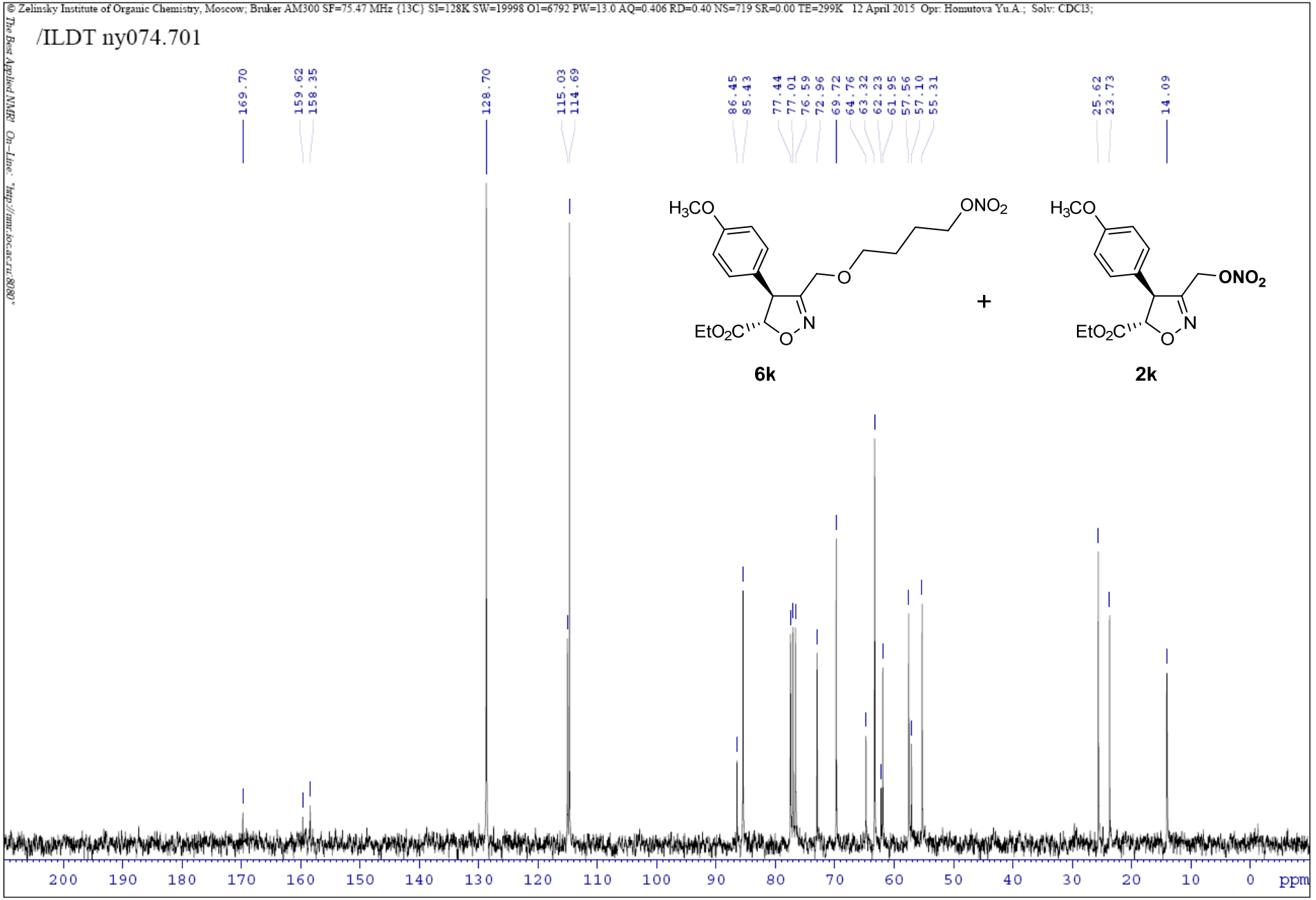
2j

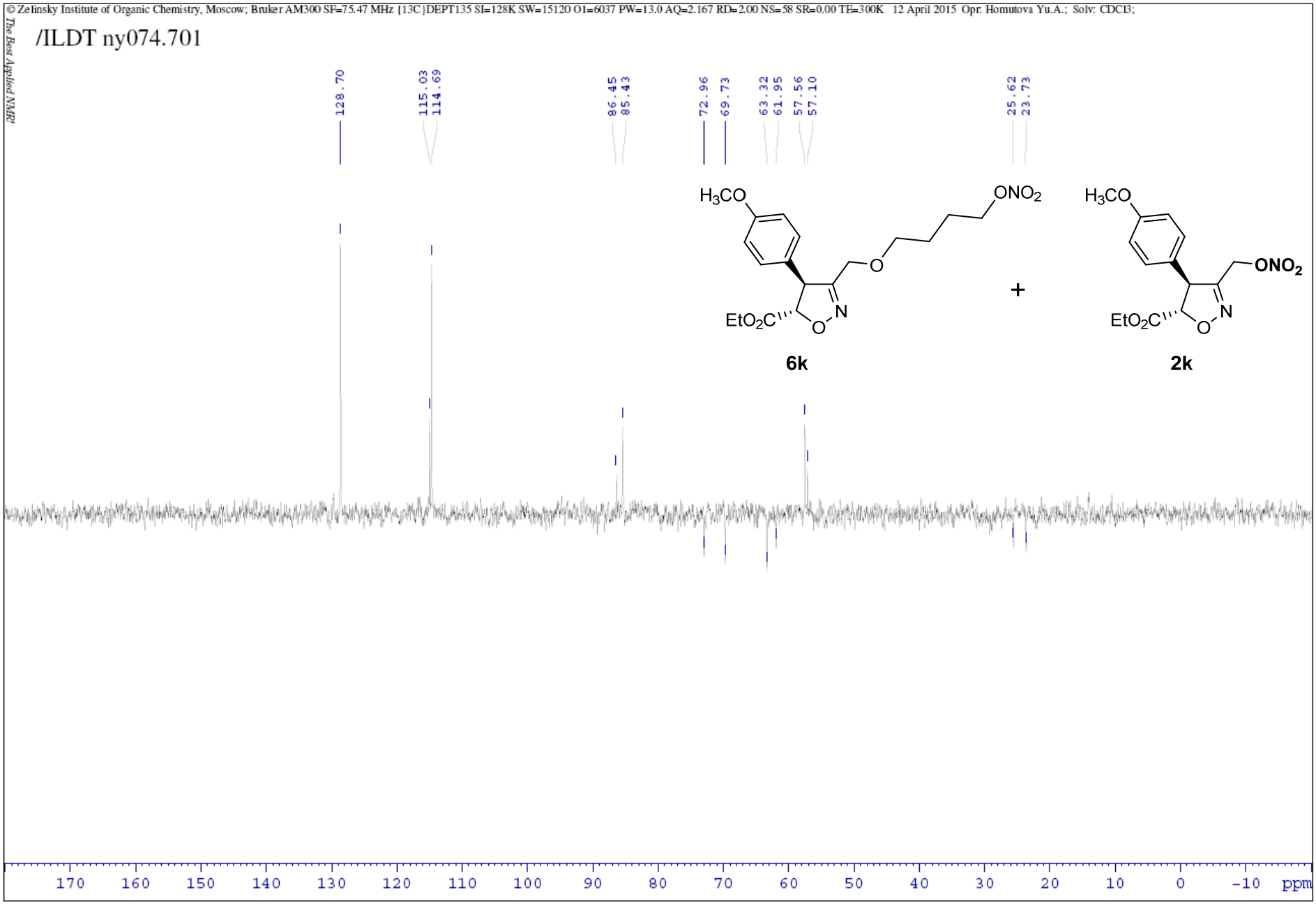




D:\EDL\NY-49.0	NY-49	KBr.	


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.351 RD=0.00 NS=1 SR=-4.17 TE=300K 19 April 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT ny074.701
7.30 7.17 7.16 7.14 6.96 6.95 6.94 6.93 6.91
5.22 4.99 4.99 4.97 4.92 4.91 4.65 4.63 4.48 4.46 4.44 4.35 4.34 4.32 4.31 4.30 4.29 4.26 4.22 4.18 4.06 4.02 3.84 3.84 3.50 3.49 3.47 3.39 3.37 3.35 3.34
1.79 1.78 1.77 1.74 1.68 1.66 1.64 1.63 1.61 1.37 1.35 1.32 1.29
H3CO
ONO2
EtO2C
O
N
6k
+
2k
125
88
63
72
66
32
27
6.6
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
88.283
6.883
27.040
126.221
63.111
32.473
66.239
72.592


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=128K SW=19998 O1=6792 PW=13.0 AQ=0.406 RD=0.40 NS=719 SR=0.00 TE=299K 12 April 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT ny074.701
169.70
159.62
158.35
128.70
115.03
114.69
86.45
85.43
77.44
77.01
76.59
72.96
69.72
64.76
63.32
62.23
61.95
57.56
57.10
55.31
25.62
23.73
14.09
H3CO
ONO2
O
N
O
EtO2C
6k
+
H3CO
ONO2
EtO2C
N
O
2k
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C}DEPT135 SI=128K SW=15120 O1=6037 PW=13.0 AQ=2.167 RD=2.00 NS=58 SR=0.00 TE=300K 12 April 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR!
/ILDT ny074.701
128.70
115.03
114.69
86.45
85.43
72.96
69.73
63.32
61.95
57.56
57.10
25.62
23.73
H3CO
ONO2
O
N
O
EtO2C
6k
+
H3CO
ONO2
N
O
EtO2C
2k
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm

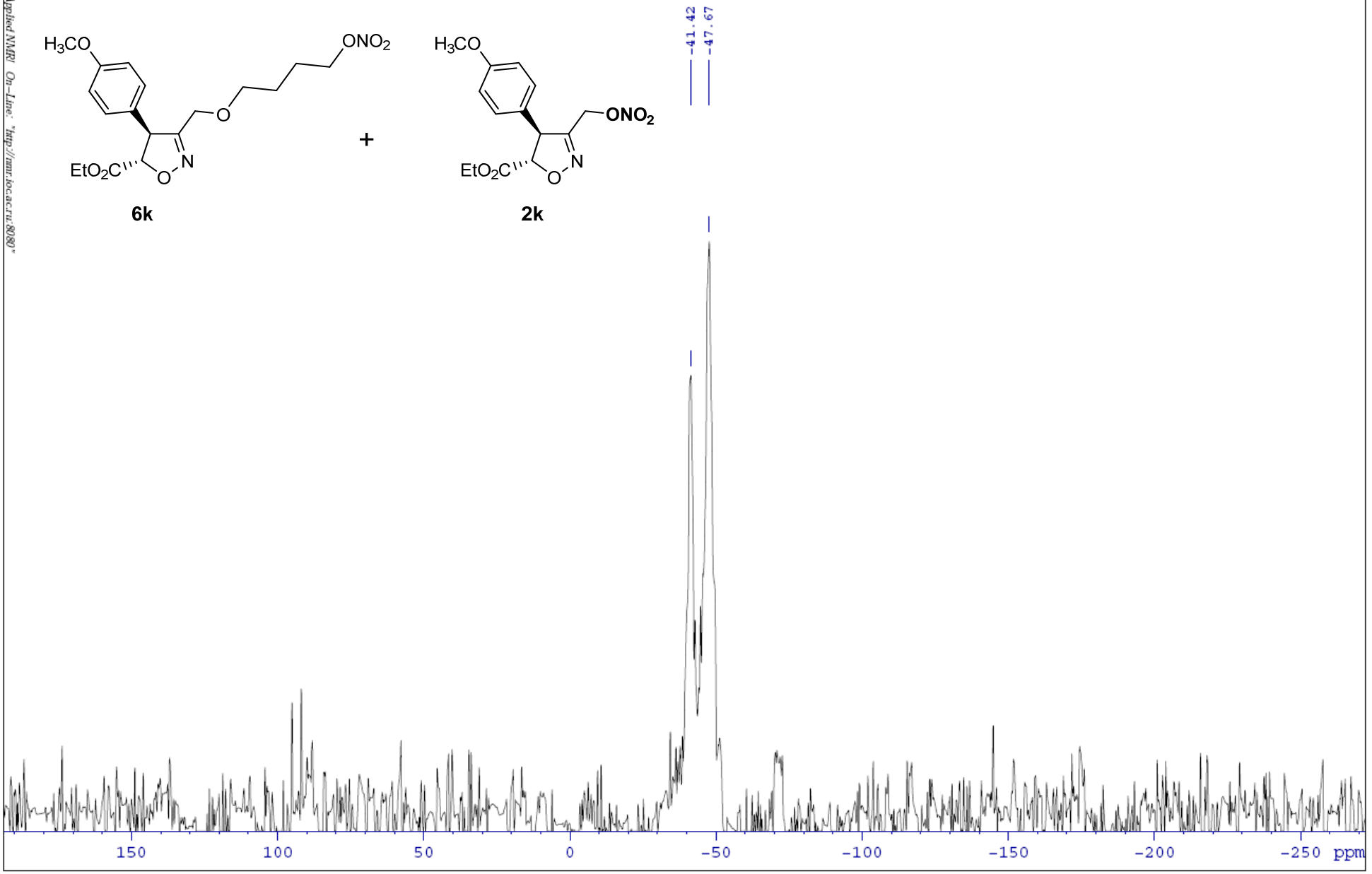
© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=21.69 MHz {14N} SI=8K SW=23807 O1=9310 PW=10.0 AQ=0.083 RD=0.05 NS=775 SR=7317.05 TE=298K 26 July 2015 Opr: Homutova Yu.A.; Solv: CDCl3;

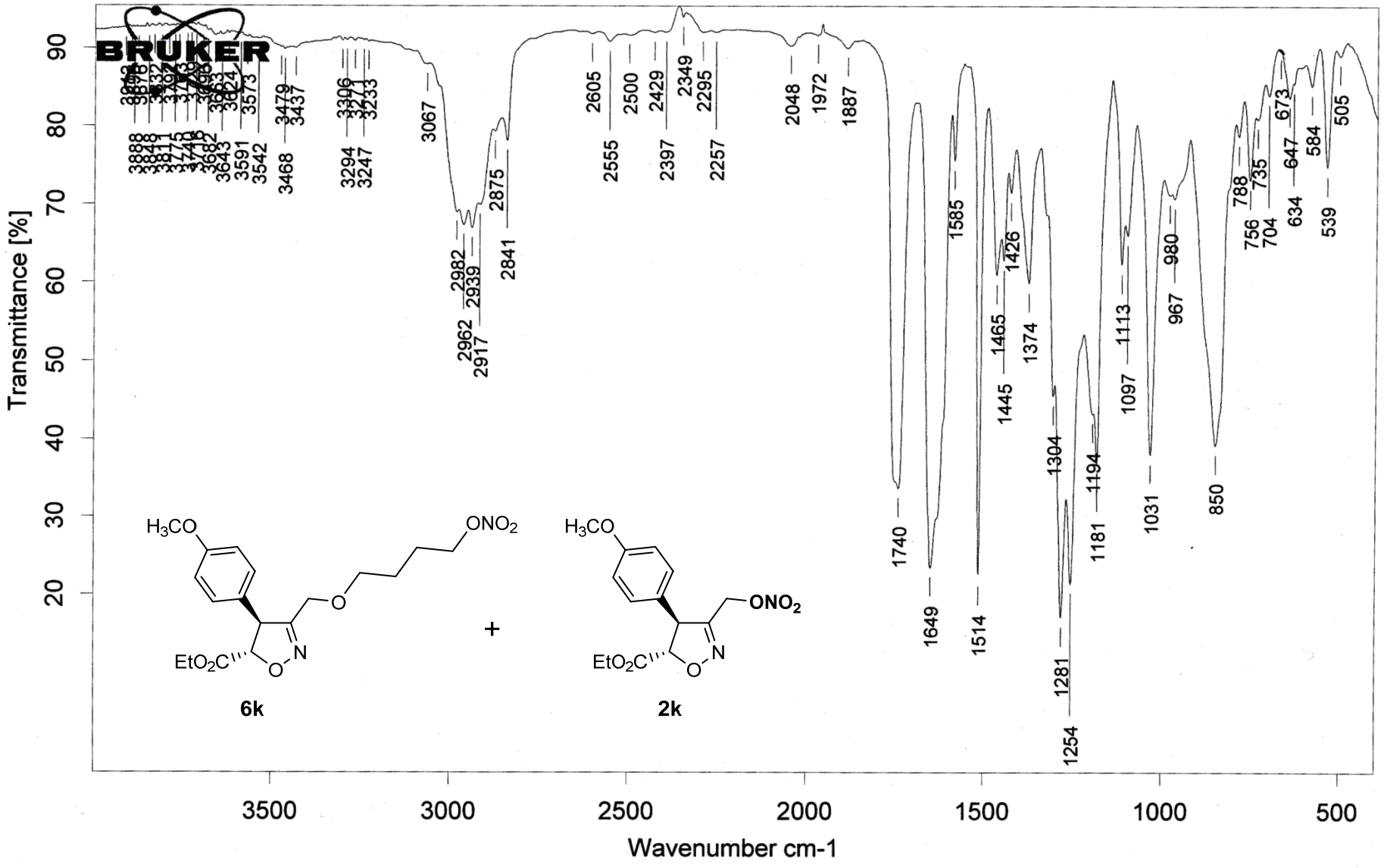
/ILDT ny074.711

6k

+

2k

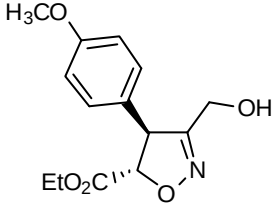




D:\EDL\NY-74-2.0	NY-74-2	KBr.

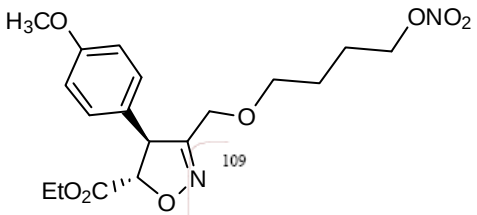
© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=1.00 NS=2 SR=-1.46 TE=300K 5 April 2015 Opr: Homutova Yu.A.; Solv: CDCl3;

/ILDT ny074.501



3k

+



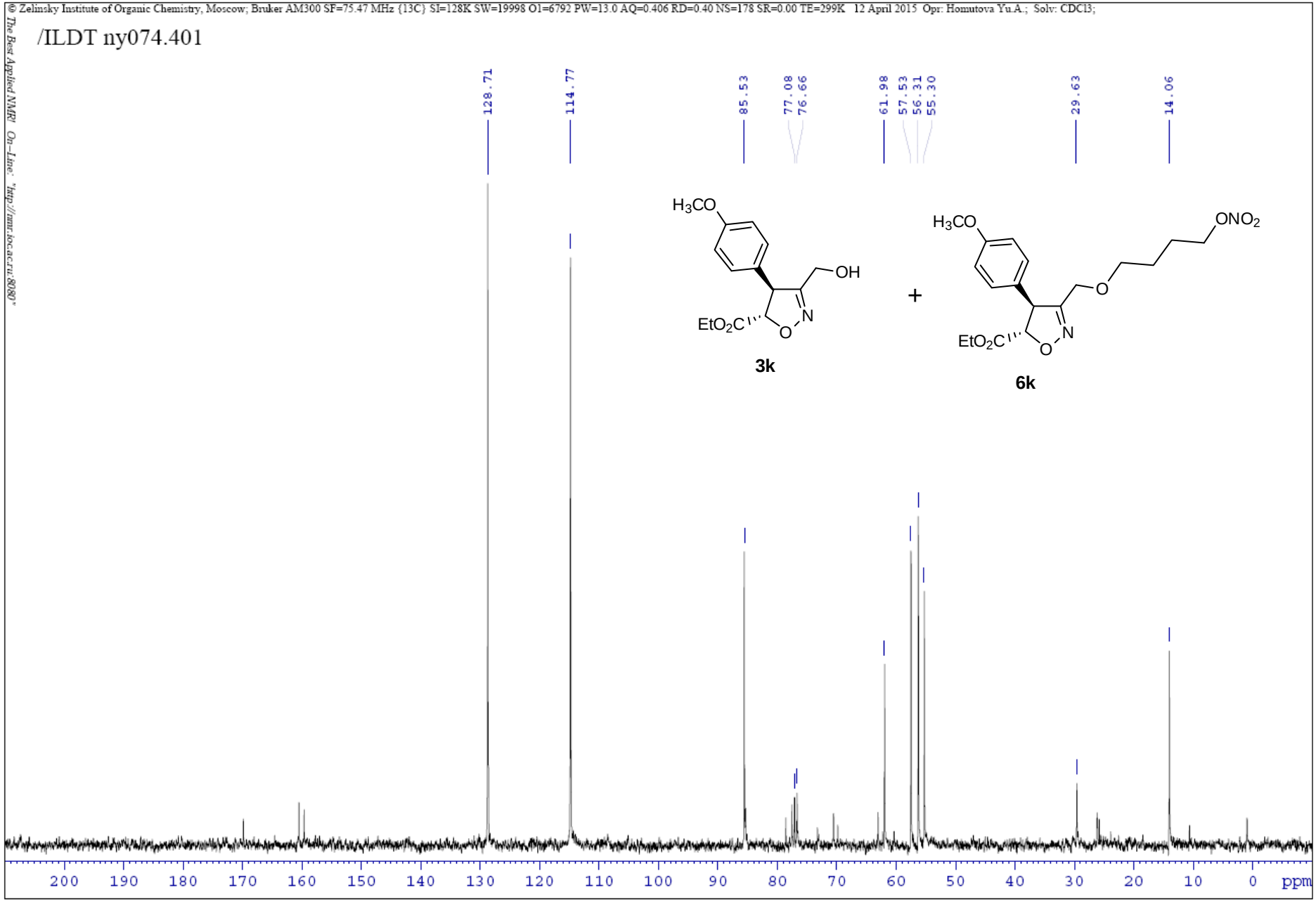
6k

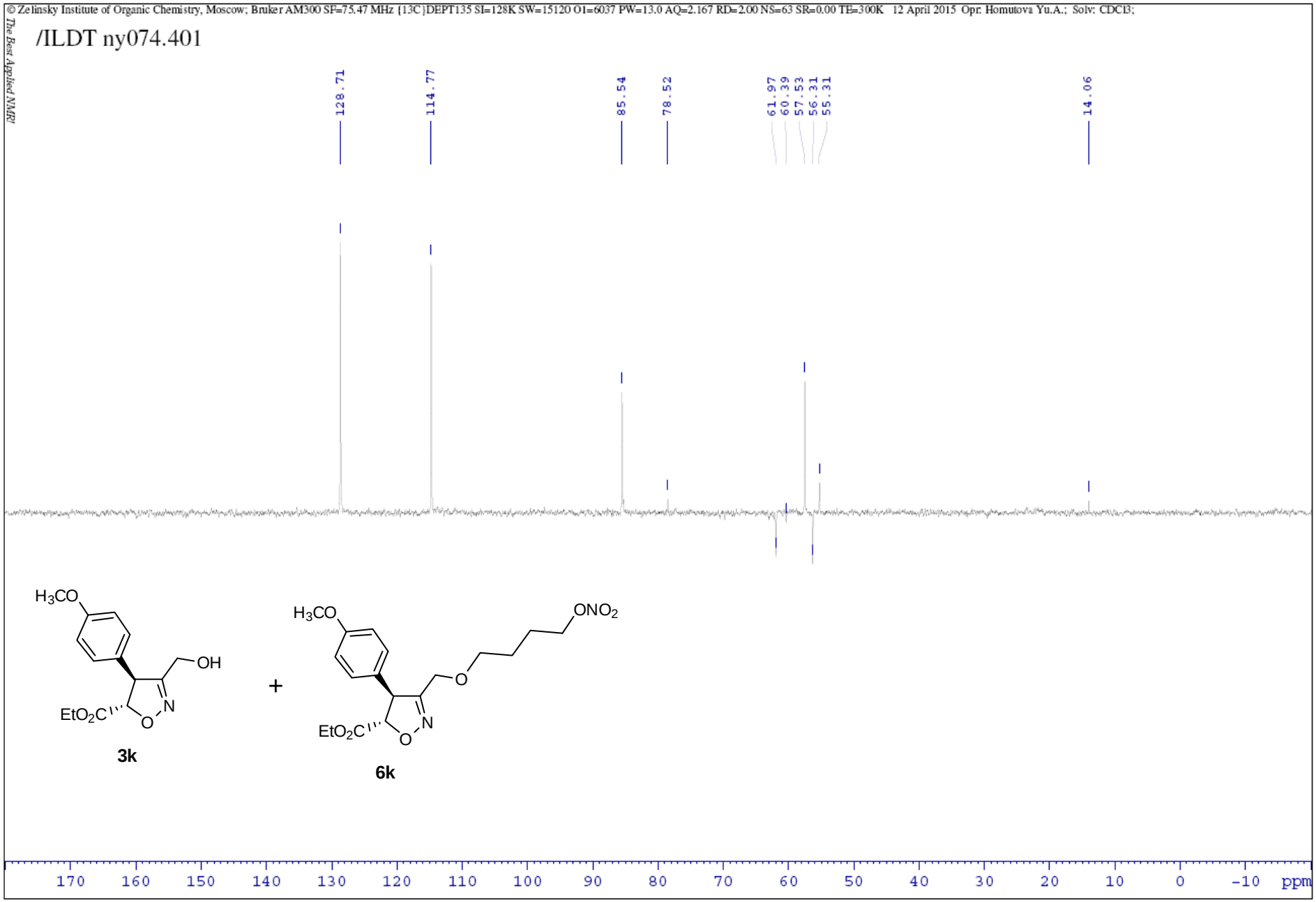
7.15 7.12 6.90 6.87 4.88 4.86 4.69 4.67 4.26 4.24 4.09 3.81 3.79 2.03 1.32 1.30 1.28 1.25

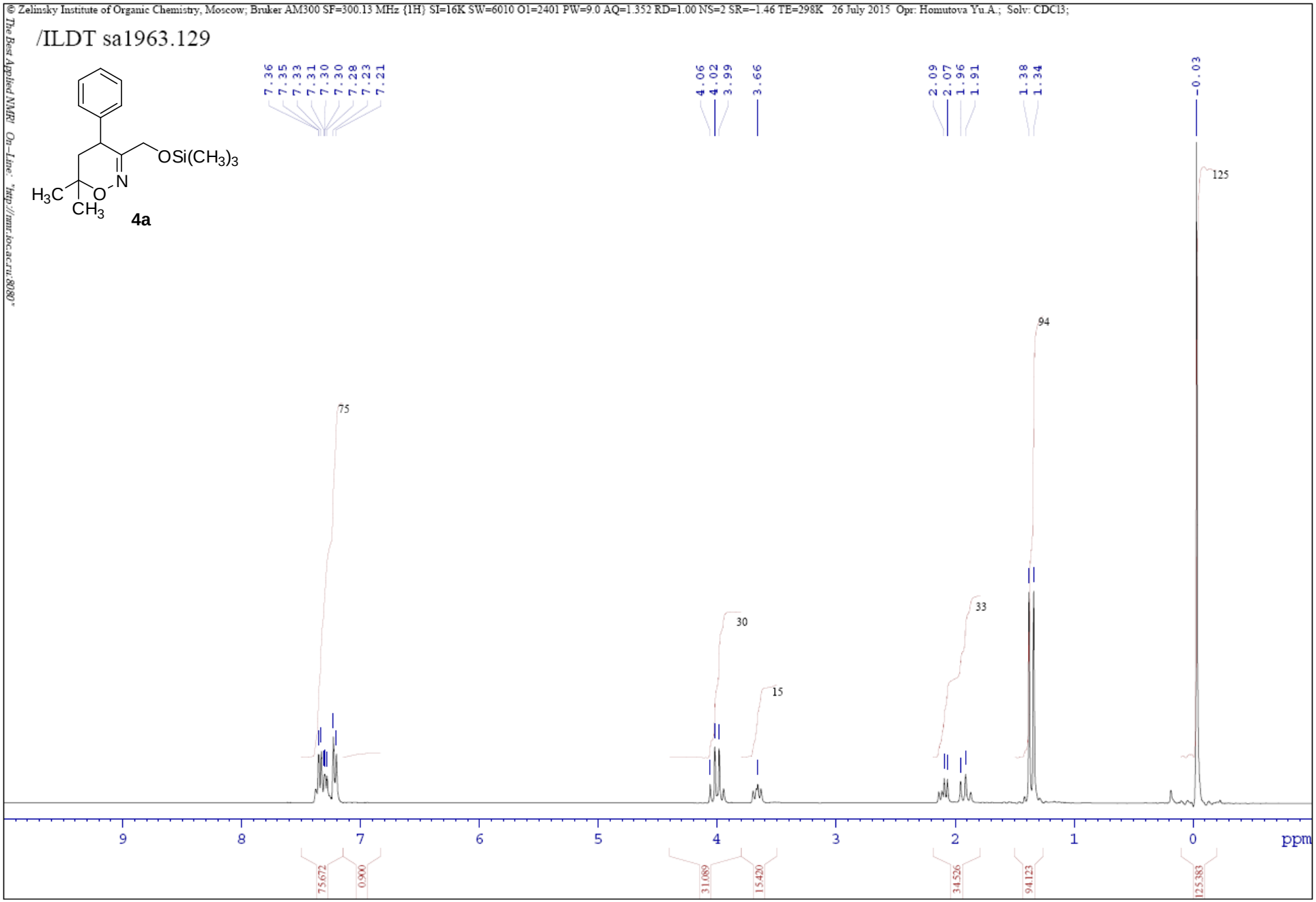
125 118 109 84 48 20 10

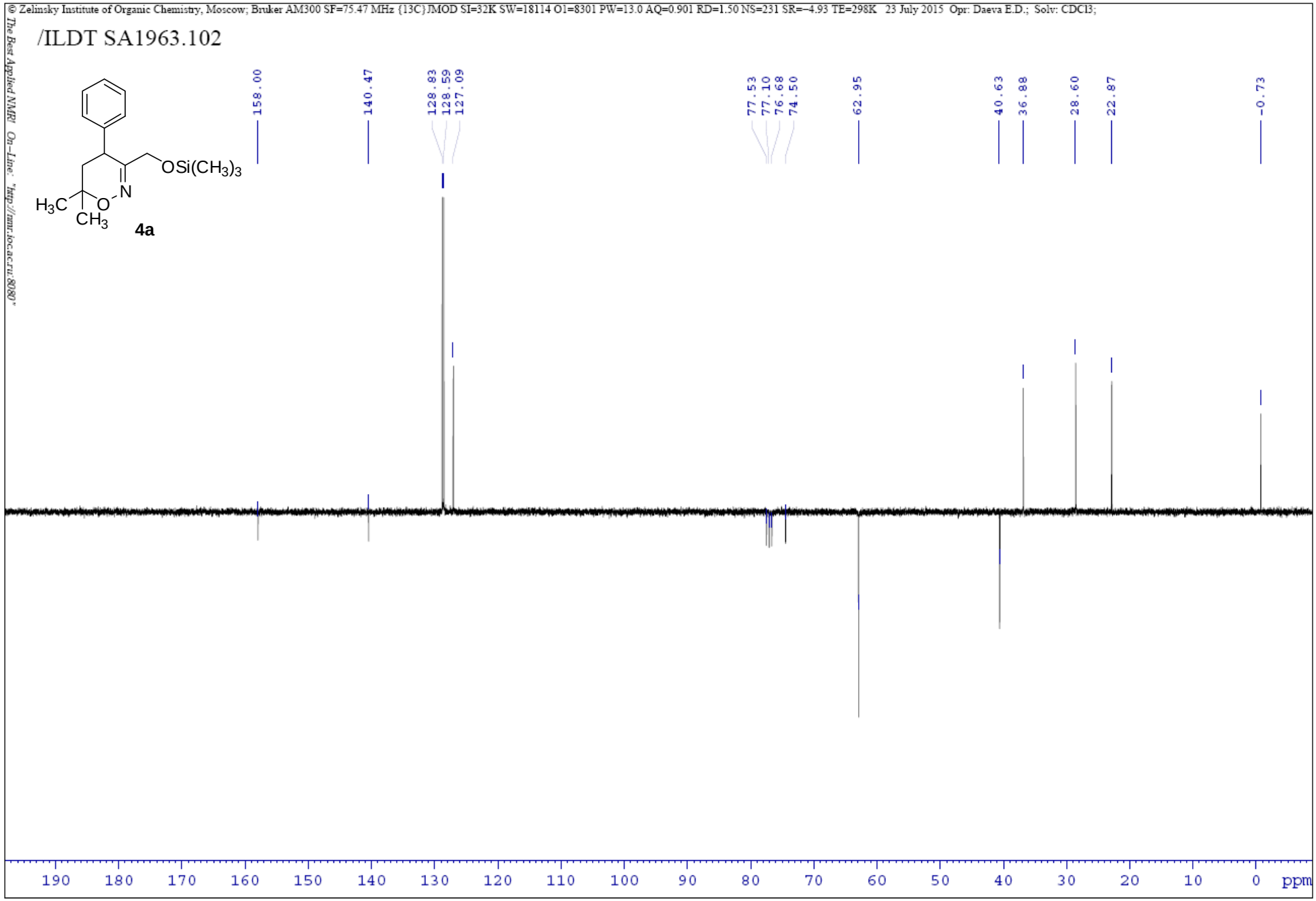
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

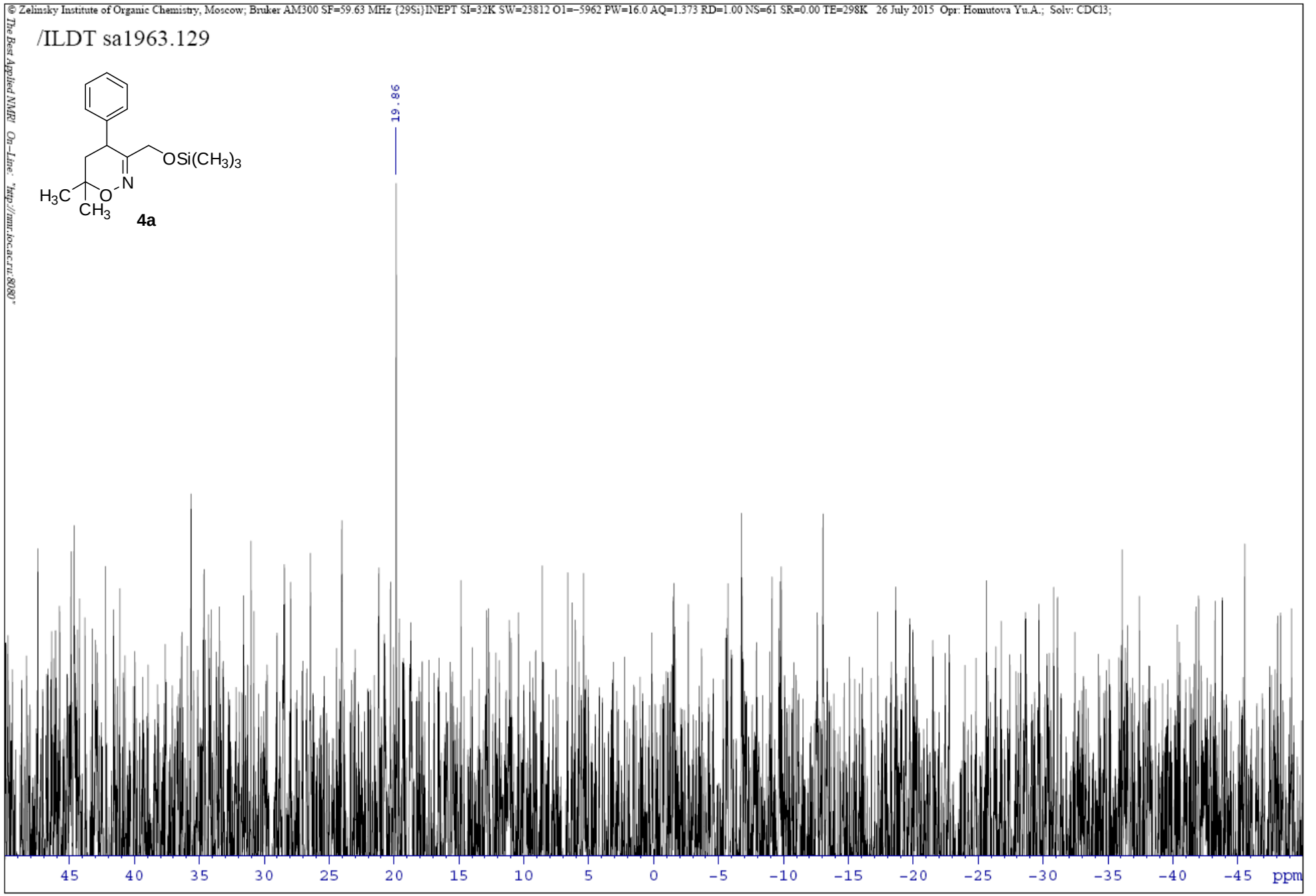
110.749 50.618 120.352 84.832 10.008 19.973 125.852

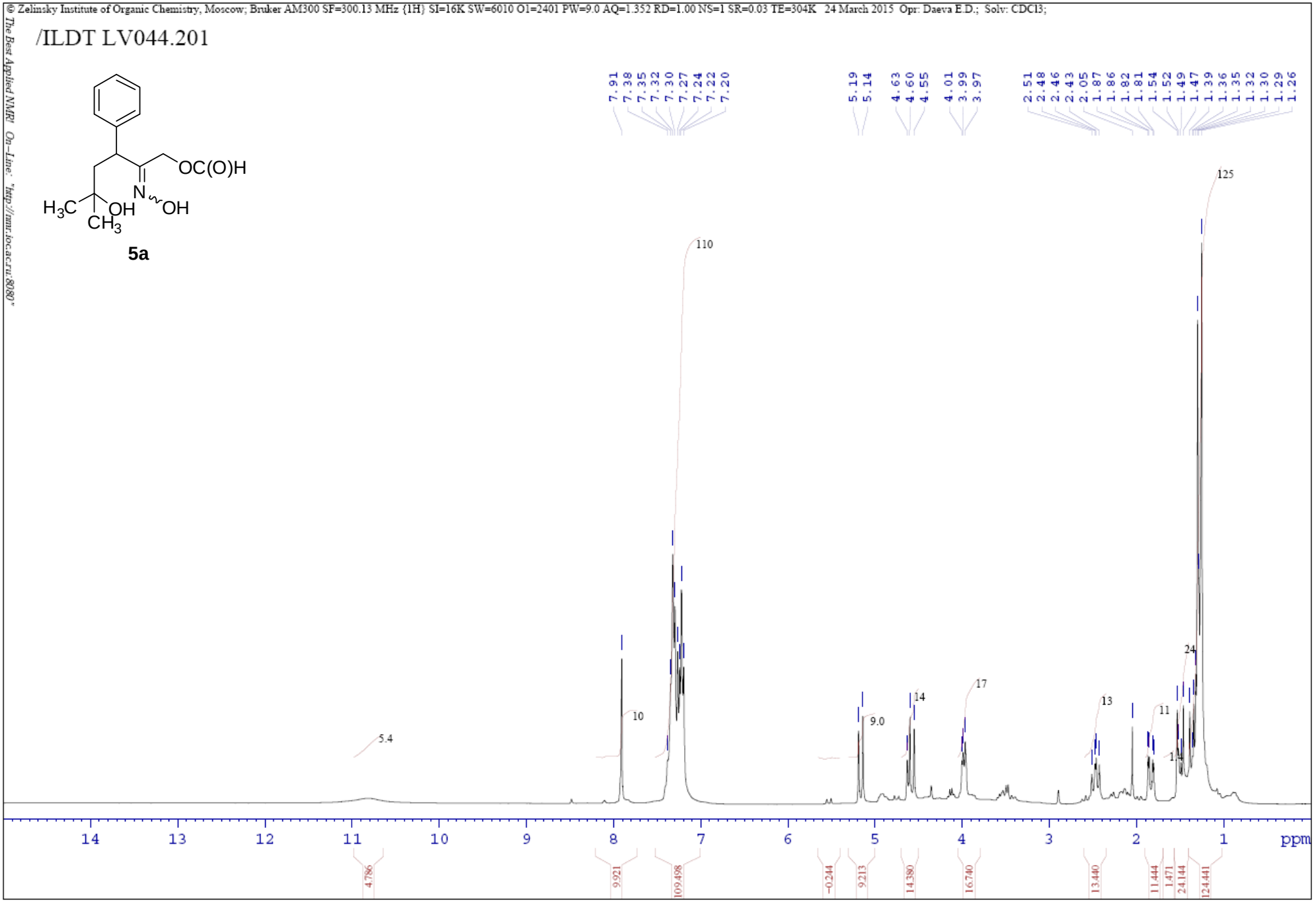

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=128K SW=19998 O1=6792 PW=13.0 AQ=0.406 RD=0.40 NS=178 SR=0.00 TE=299K 12 April 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT ny074.401
129.71
114.77
85.53
77.08
76.66
61.98
57.53
56.31
55.30
29.63
14.06
H3CO
OH
EtO2C
O
N
3k
+
H3CO
ONO2
O
EtO2C
O
N
6k
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

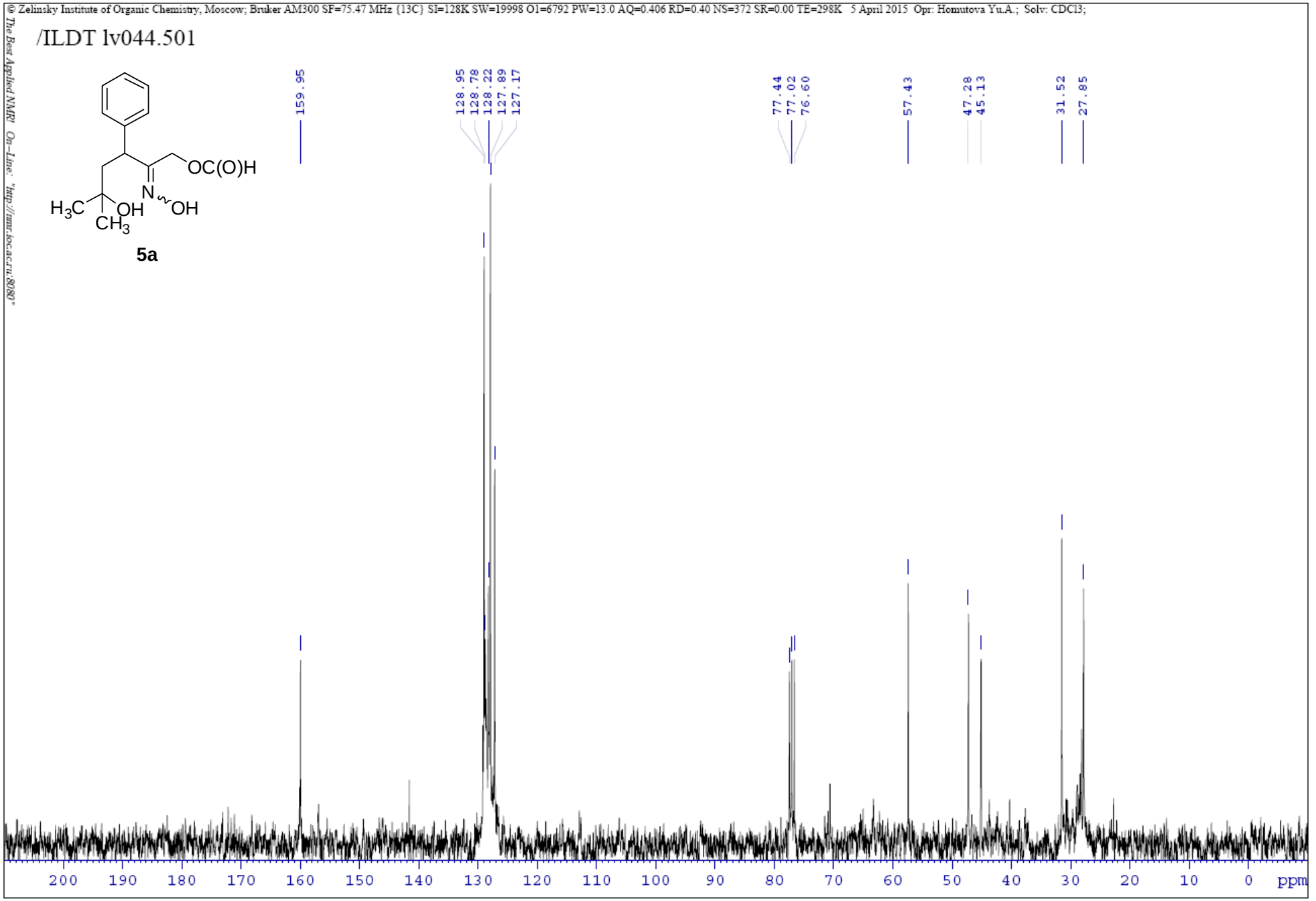

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C}DEPT135 SI=128K SW=15120 O1=6037 PW=13.0 AQ=2.167 RD=2.00 NS=63 SR=0.00 TE=300K 12 April 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR!
/ILDT ny074.401
128.71
114.77
85.54
78.52
61.97
60.39
57.53
56.31
55.31
14.06
H3CO
OH
EtO2C
O
N
3k
+
H3CO
ONO2
O
EtO2C
O
N
6k
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm

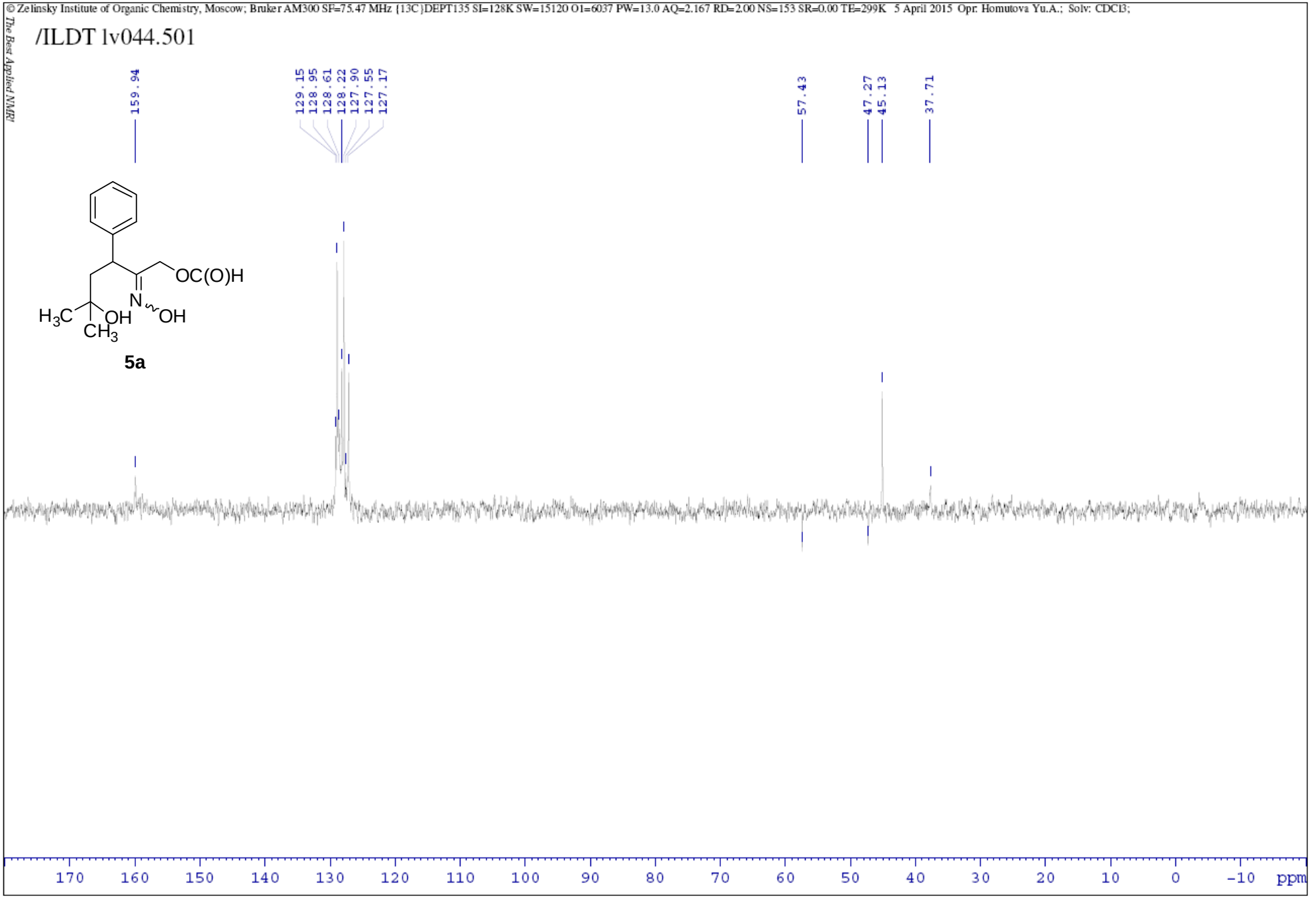

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=1.00 NS=2 SR=-1.46 TE=298K 26 July 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT sa1963.129
OSi(CH3)3
H3C
CH3
4a
7.36
7.35
7.33
7.31
7.30
7.30
7.28
7.23
7.21
4.06
4.02
3.99
3.66
2.09
2.07
1.96
1.91
1.38
1.34
-0.03
125
94
75
30
33
15
9
8
7
6
5
4
3
2
1
0
ppm
75.672
0.900
31.089
15.420
34.526
94.123
125.383


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} JMOD SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.901 RD=1.50 NS=231 SR=-4.93 TE=298K 23 July 2015 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT SA1963.102
158.00
140.47
128.83
128.59
127.09
77.53
77.10
76.68
74.50
62.95
40.63
36.88
28.60
22.87
-0.73
OSi(CH3)3
H3C
CH3
O
N
4a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

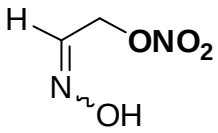

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=59.63 MHz {29Si}INEPT SI=32K SW=23812 O1=-5962 PW=16.0 AQ=1.373 RD=1.00 NS=61 SR=0.00 TE=298K 26 July 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT sa1963.129
19.86
OSi(CH3)3
H3C
CH3
O
N
4a
45 40 35 30 25 20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 ppm


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=1.00 NS=1 SR=0.03 TE=304K 24 March 2015 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT LV044.201
OC(O)H
N
OH
H3C
OH
CH3
5a
7.91 7.38 7.35 7.32 7.30 7.27 7.24 7.22 7.20
5.19 5.14
4.63 4.60 4.55
4.01 3.99 3.97
2.51 2.48 2.46 2.43 2.05 1.87 1.86 1.82 1.81 1.54 1.52 1.49 1.47 1.39 1.36 1.35 1.32 1.30 1.29 1.26
125
110
24
17
14
13
11
10
9.0
5.4
14 13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
4.786
9.921
109.498
-0.244
9.213
14.380
16.740
13.440
11.444
1.471
24.144
124.441


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=128K SW=19998 O1=6792 PW=13.0 AQ=0.406 RD=0.40 NS=372 SR=0.00 TE=298K 5 April 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT lv044.501
159.95
128.95
128.78
128.22
127.89
127.17
77.44
77.02
76.60
57.43
47.28
45.13
31.52
27.85
OC(O)H
N
OH
H3C
OH
CH3
5a
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C}DEPT135 SI=128K SW=15120 O1=6037 PW=13.0 AQ=2.167 RD=2.00 NS=153 SR=0.00 TE=299K 5 April 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR!
/ILDT lv044.501
159.94
129.15
128.95
128.61
128.22
127.90
127.55
127.17
57.43
47.27
45.13
37.71
OC(O)H
N
OH
H3C
OH
CH3
5a
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm

/ILDT sa1949.101



9m

E/*Z* = 1.4 : 1

7.56 7.54 7.52 7.27 6.91 5.29 5.28 5.06 5.04

99 122 125 98 2.0

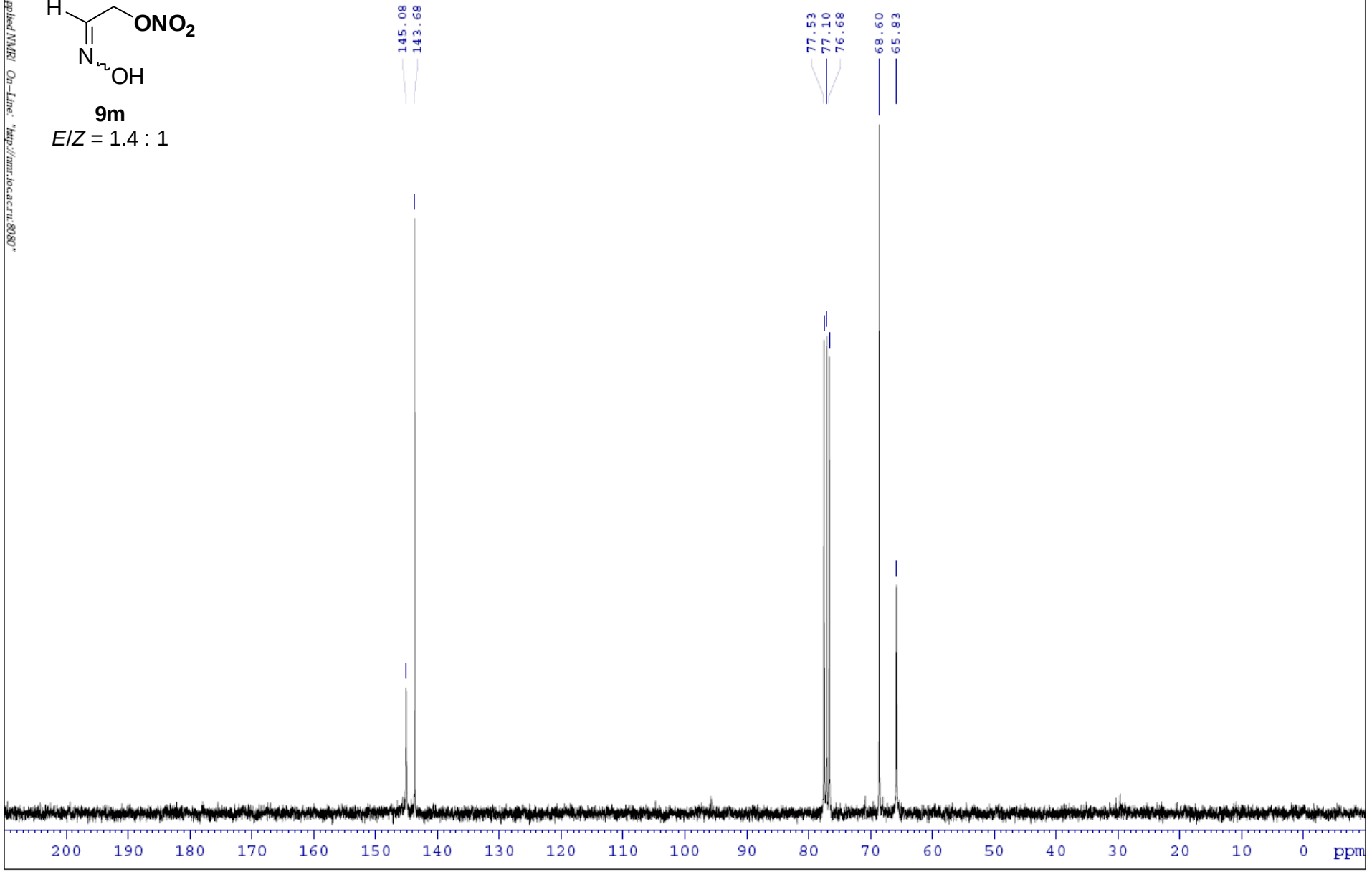
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

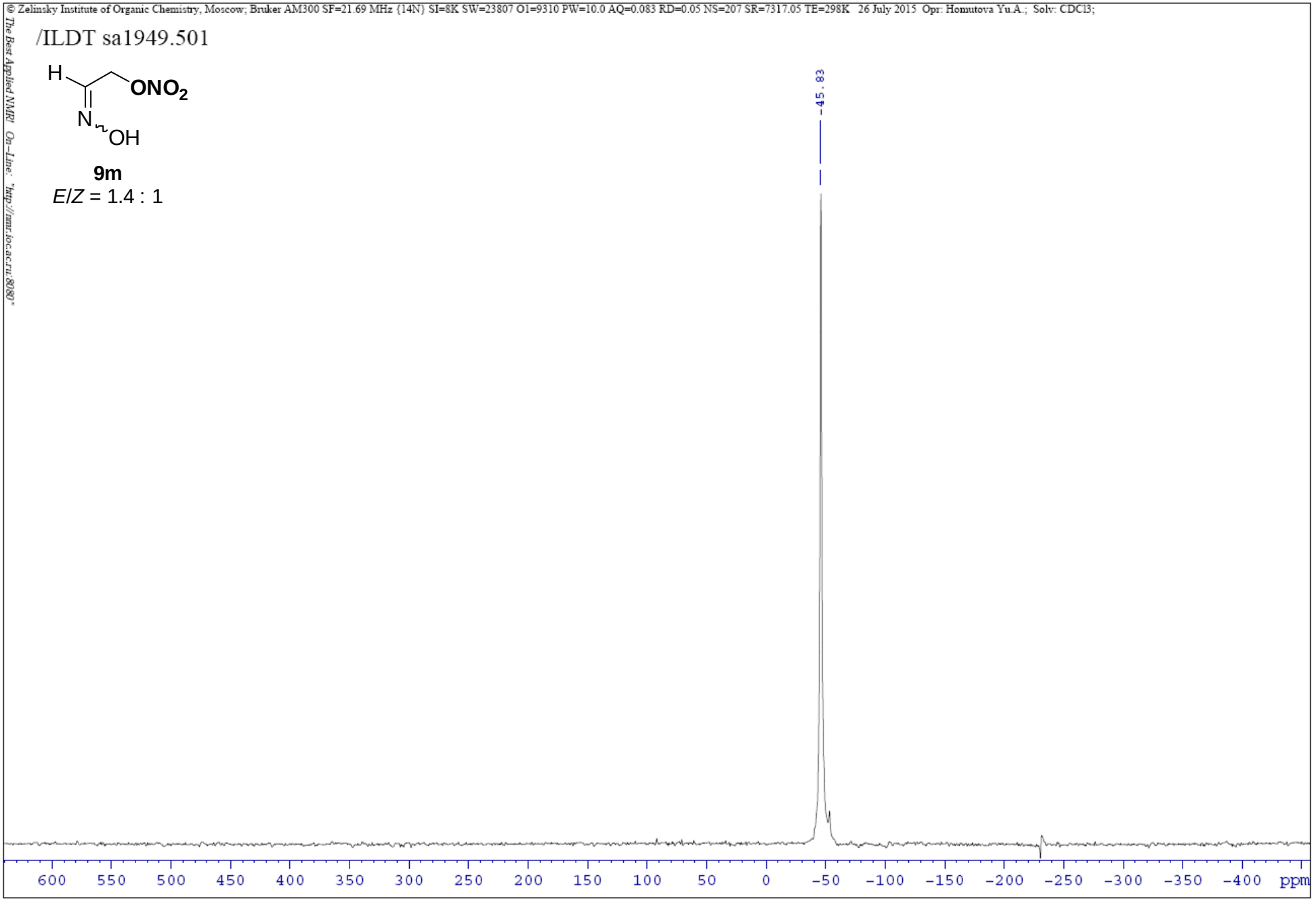
99.520 122.250 1.984 98.134 124.999

/ILDT SA1949.202

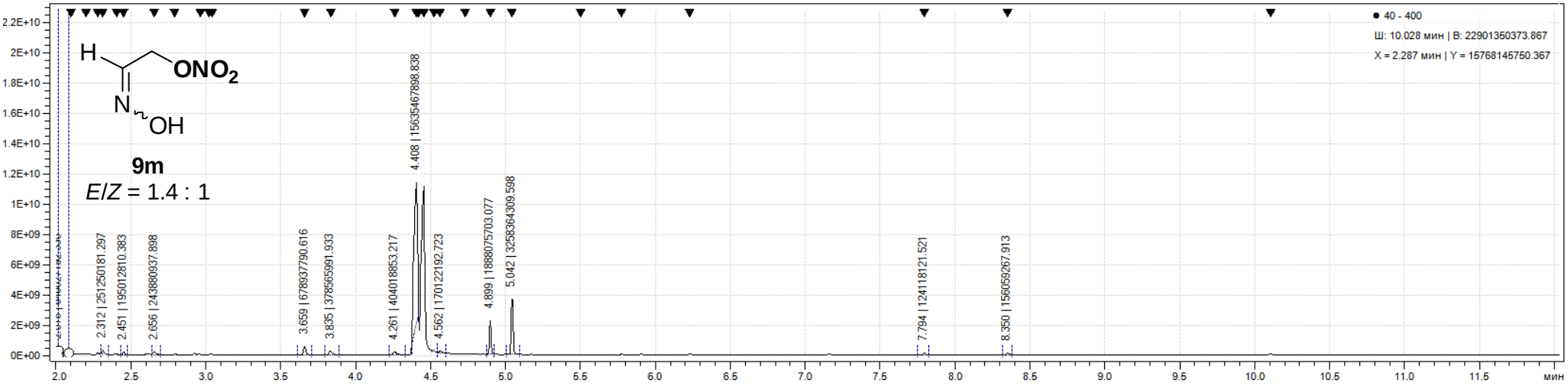
9m

E/*Z* = 1.4 : 1




© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=21.69 MHz {14N} SI=8K SW=23807 O1=9310 PW=10.0 AQ=0.083 RD=0.05 NS=207 SR=7317.05 TE=298K 26 July 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT sa1949.501
H
ONO2
N
OH
9m
E/Z = 1.4 : 1
-45.83
600 550 500 450 400 350 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -350 -400 ppm

GC-MS Chromatogram



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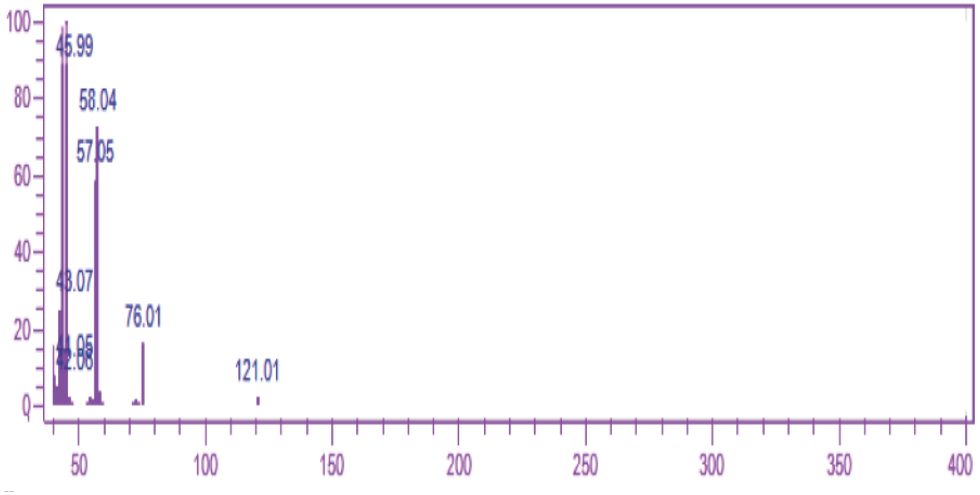
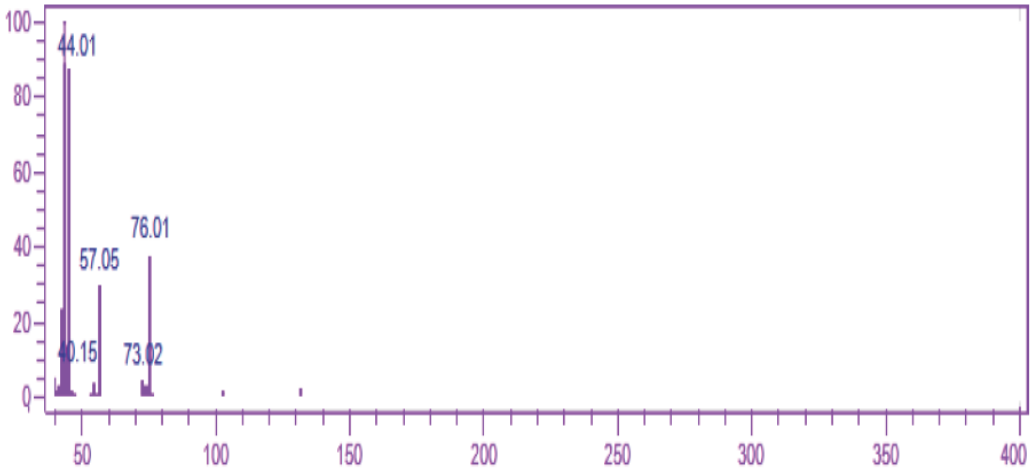
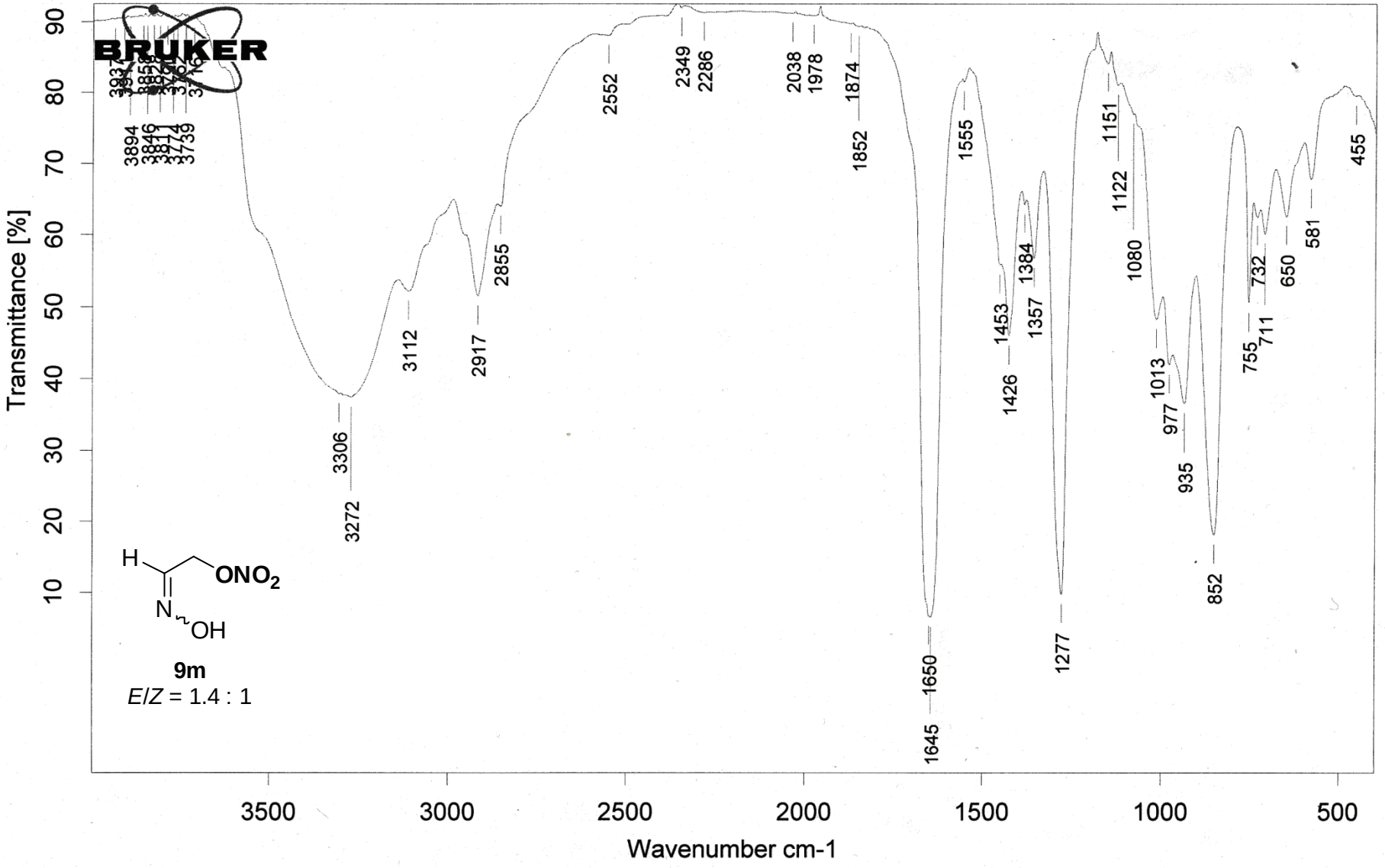
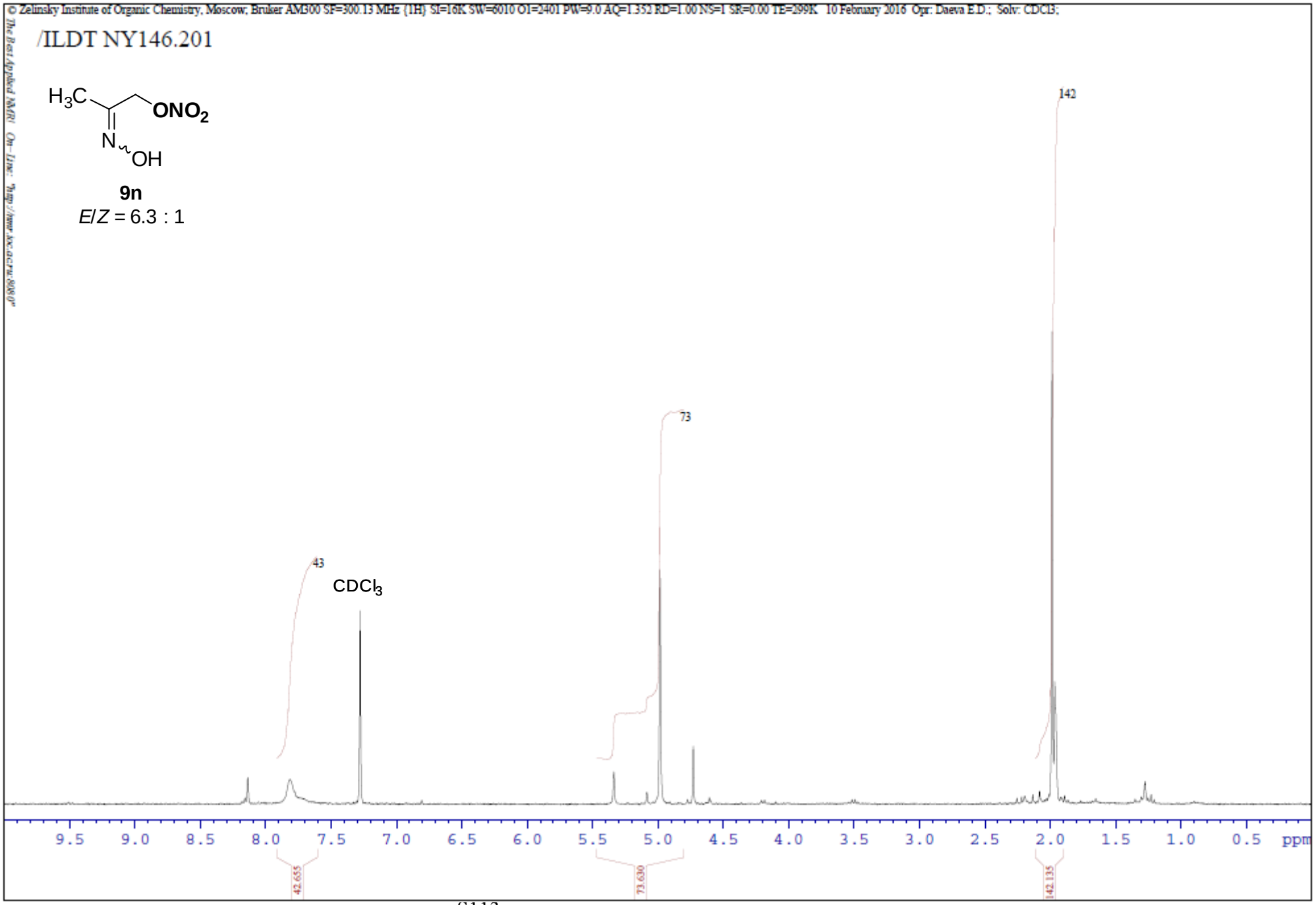


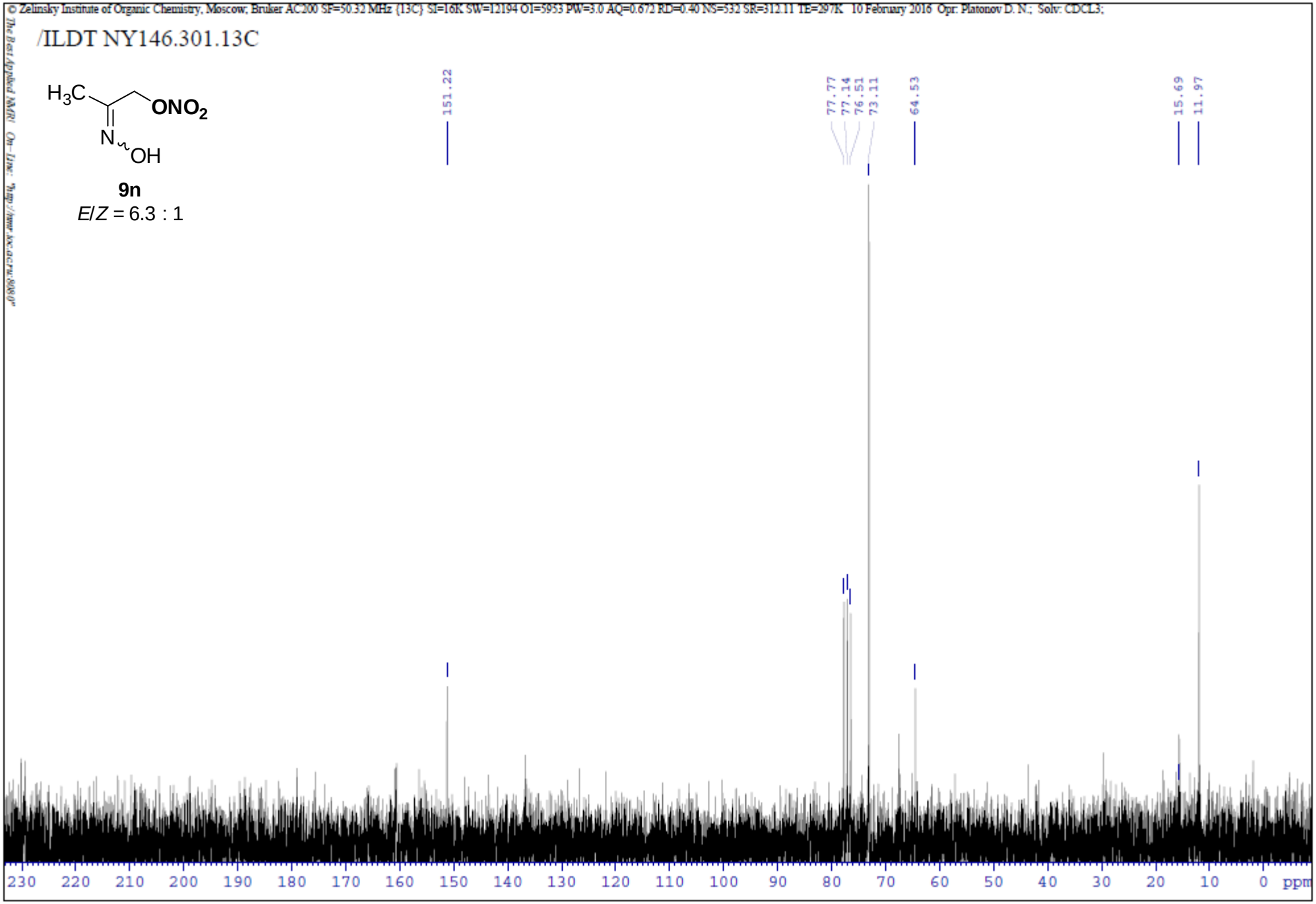
Time: 4.45 min

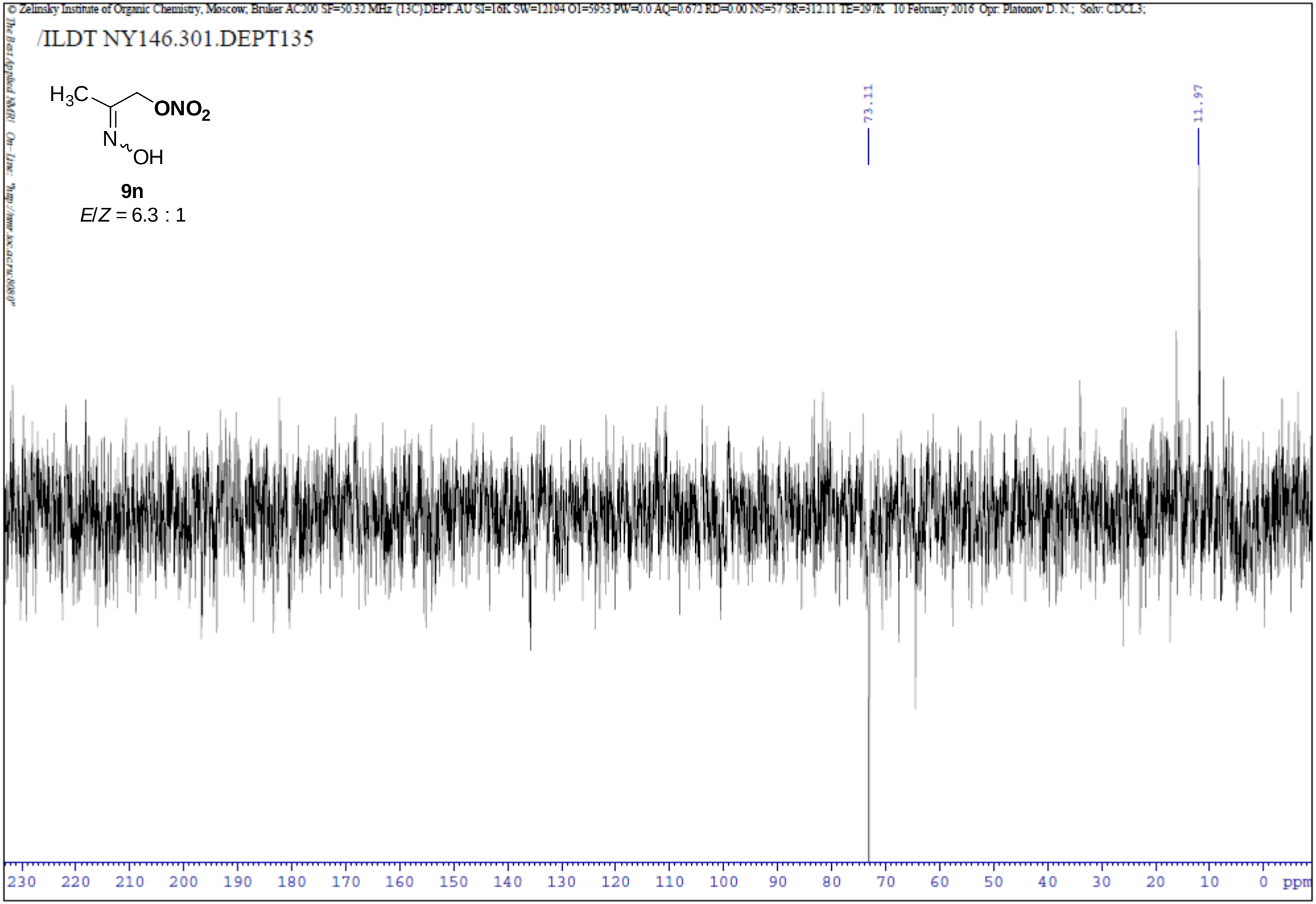


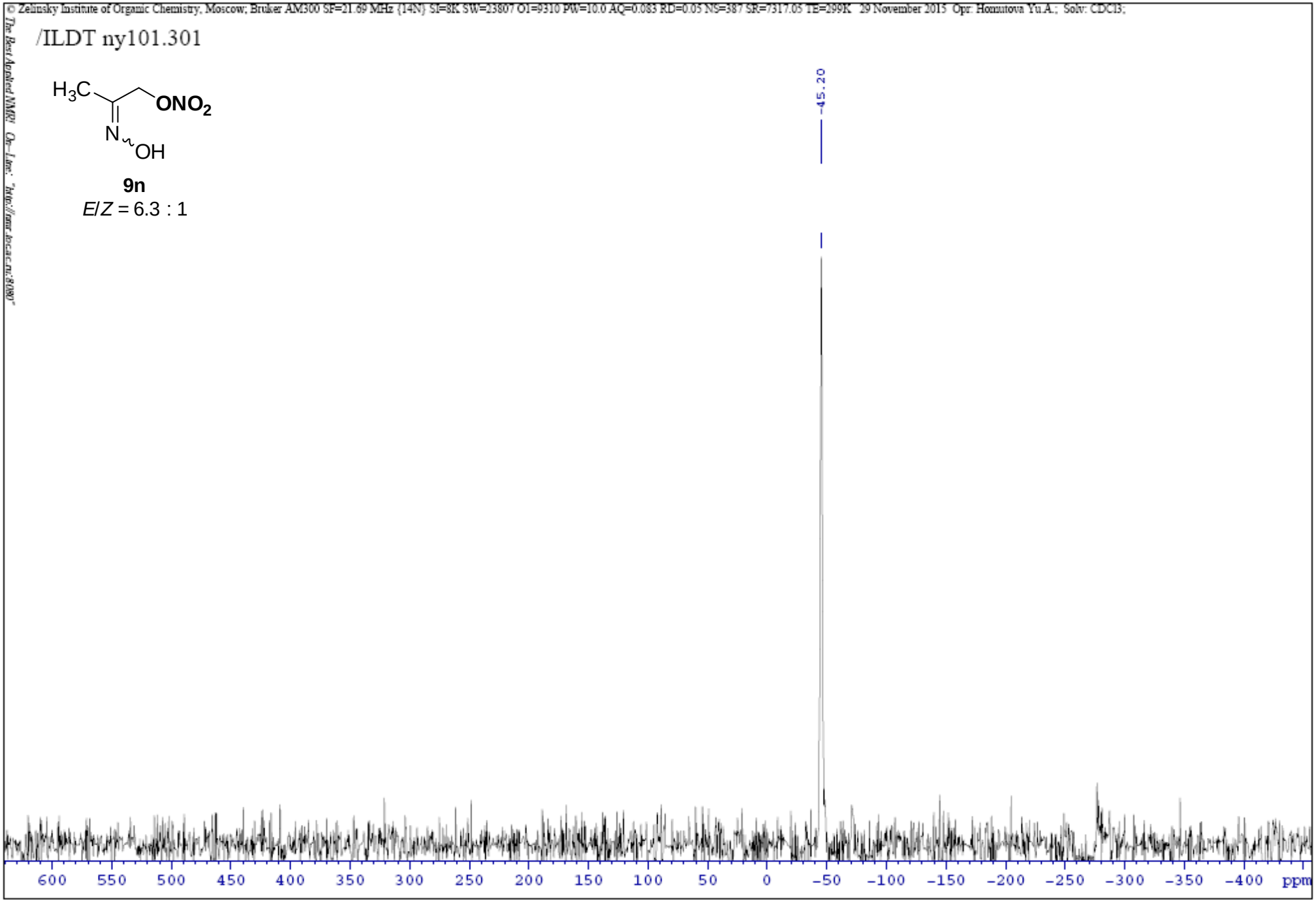


D:\EDL\SA-1949.0	SA-1949	KBr.

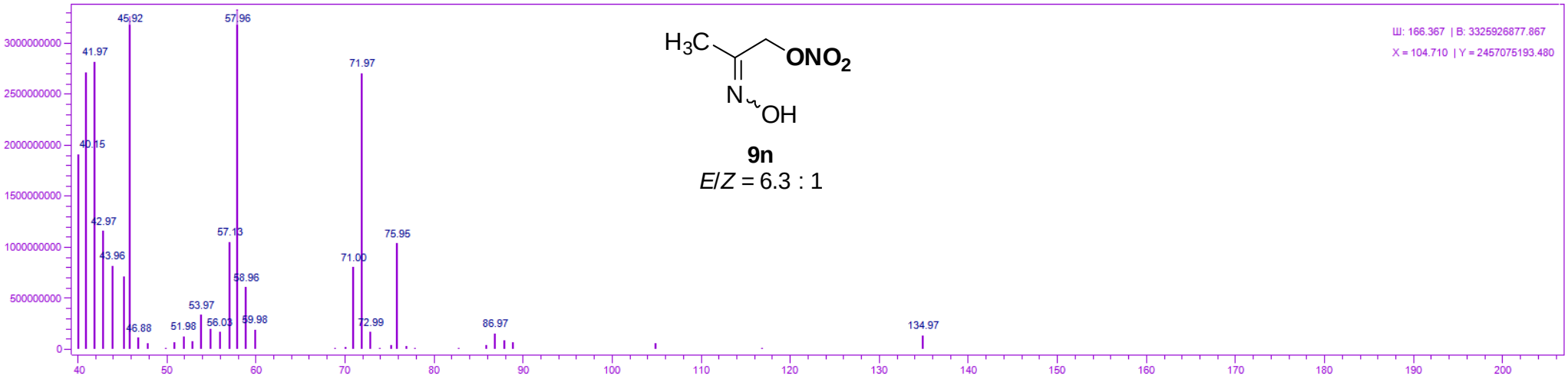

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=1.00 NS=1 SR=0.00 TE=299K 10 February 2016 Opr: Daeva E.D.; Solv: CDCl3;
/ILDT NY146.201
H_3C
ONO_2
N
OH
9n
E/Z = 6.3 : 1
142
73
43
$CDCl_3$
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
42.655
73.630
142.135


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AC200 SF=50.32 MHz {13C} SI=16K SW=12194 O1=5953 PW=3.0 AQ=0.672 RD=0.40 NS=532 SR=312.11 TE=297K 10 February 2016 Opr: Platonov D. N.; Solv: CDCL3;
/ILDT NY146.301.13C
H3C
ONO2
N
OH
9n
E/Z = 6.3 : 1
151.22
77.77
77.14
76.51
73.11
64.53
15.69
11.97
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AC200 SF=50.32 MHz {13C}DEPT.AU SI=16K SW=12194 O1=5953 PW=0.0 AQ=0.672 RD=0.00 NS=57 SR=312.11 TE=297K 10 February 2016 Opr: Platonov D. N.; Solv: CDCL3;
/ILDT NY146.301.DEPT135
H_3C
ONO_2
N
OH
9n
E/Z = 6.3 : 1
73.11
11.97
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

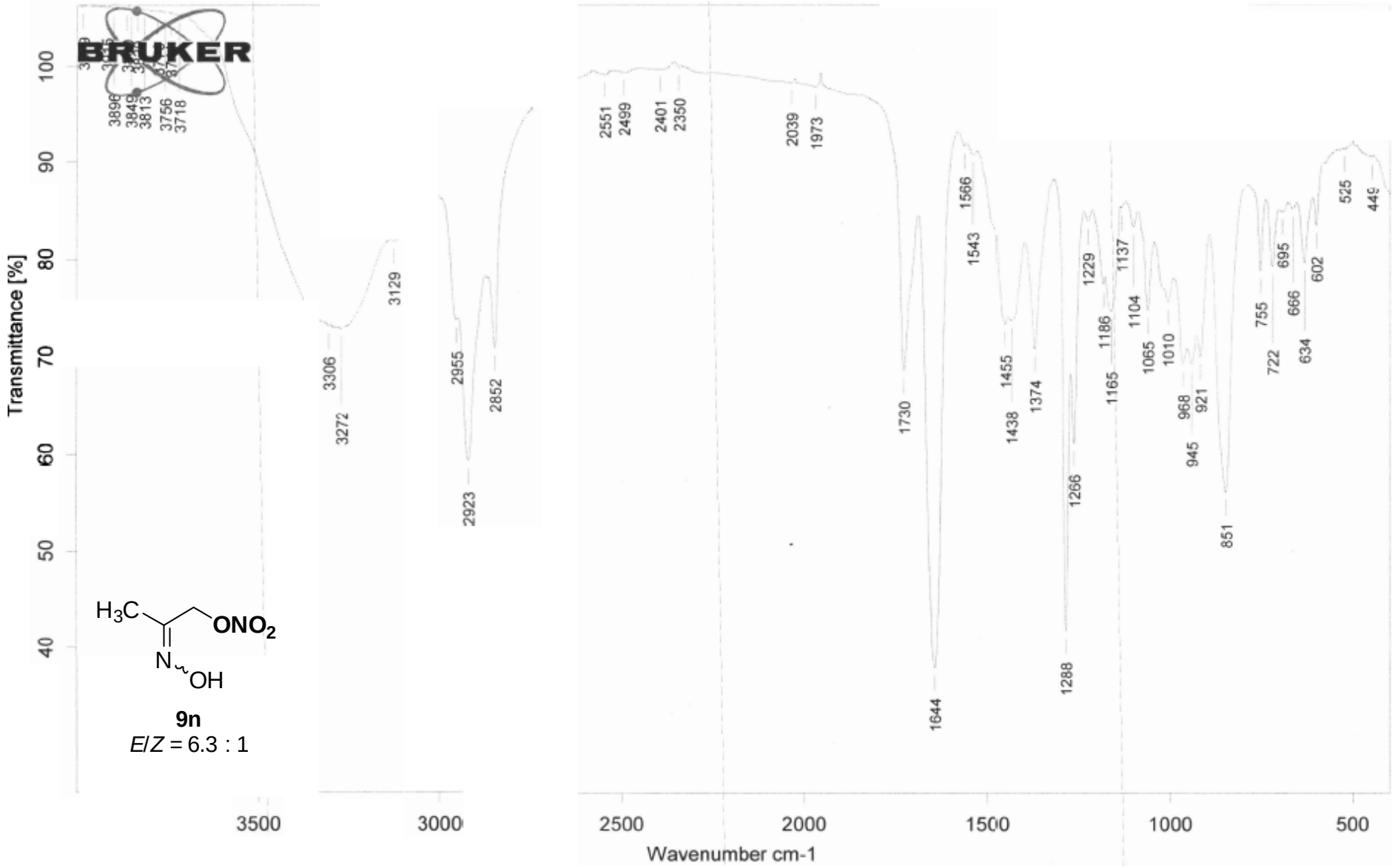

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=21.69 MHz {14N} SI=8K SW=23807 O1=9310 PW=10.0 AQ=0.083 RD=0.05 NS=387 SR=7317.05 TE=299K 29 November 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR; On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT ny101.301
H3C
ONO2
N
OH
9n
E/Z = 6.3 : 1
45.20
600 550 500 450 400 350 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -350 -400 ppm

EI-MS



9n

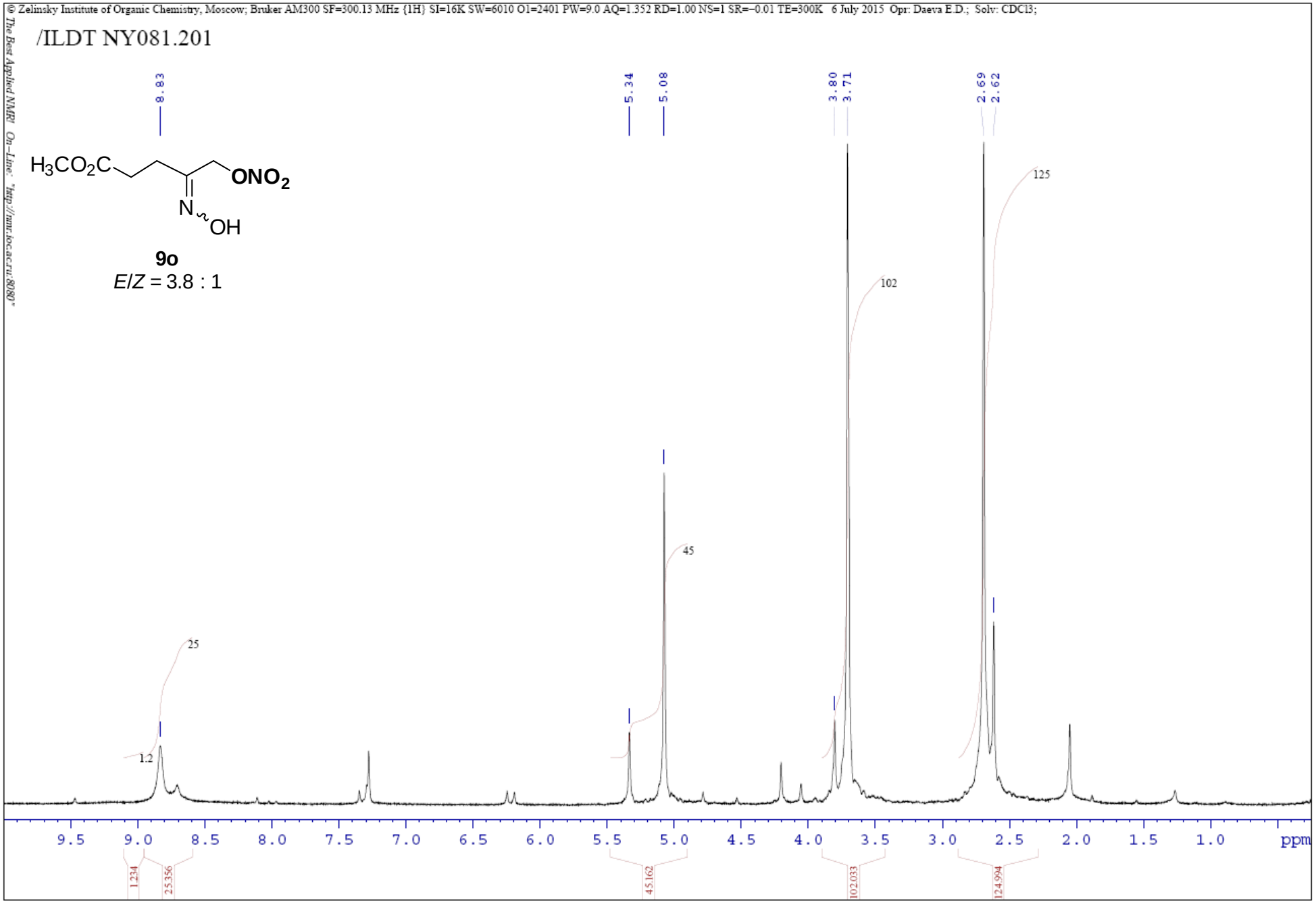
E/*Z* = 6.3 : 1

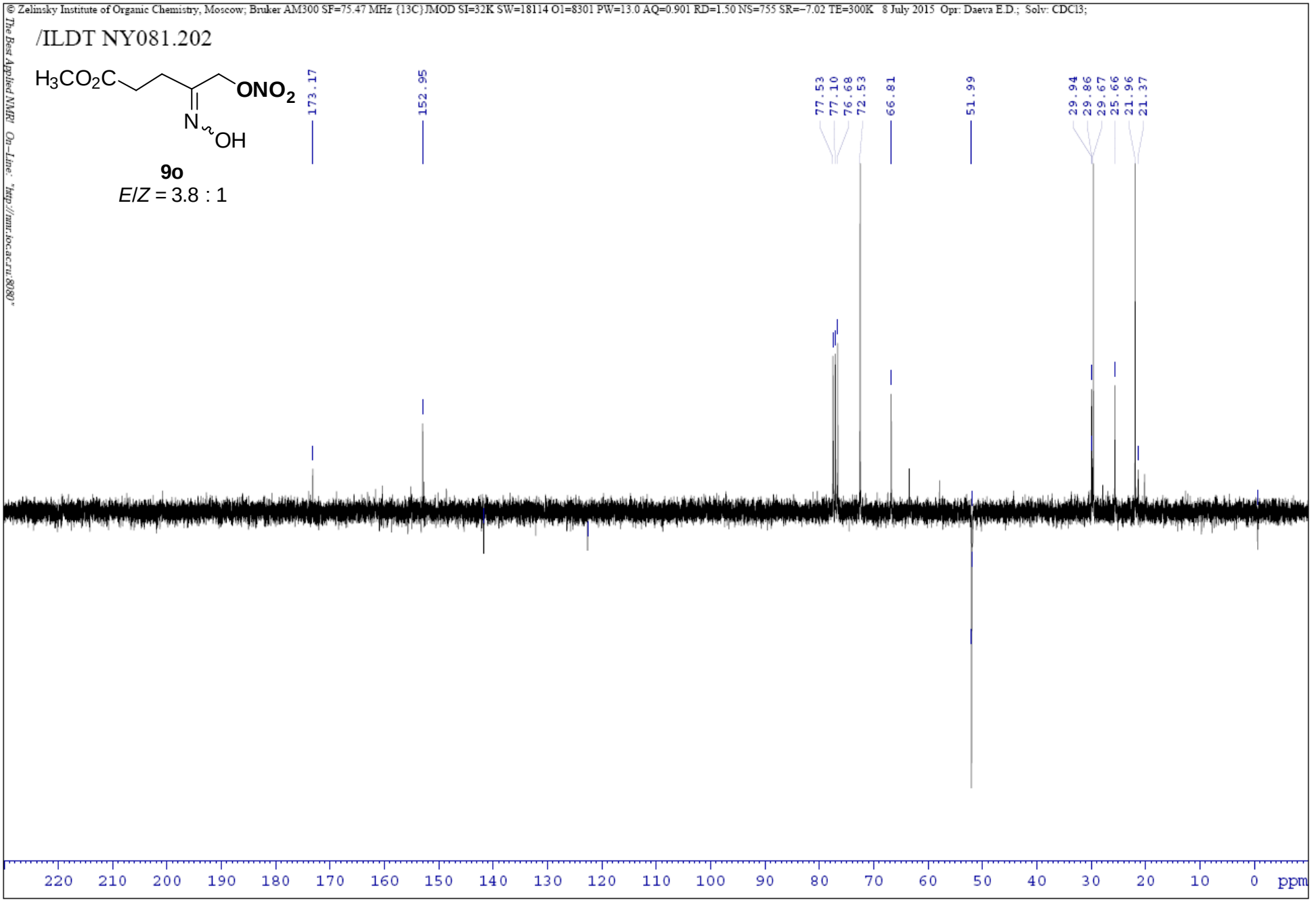


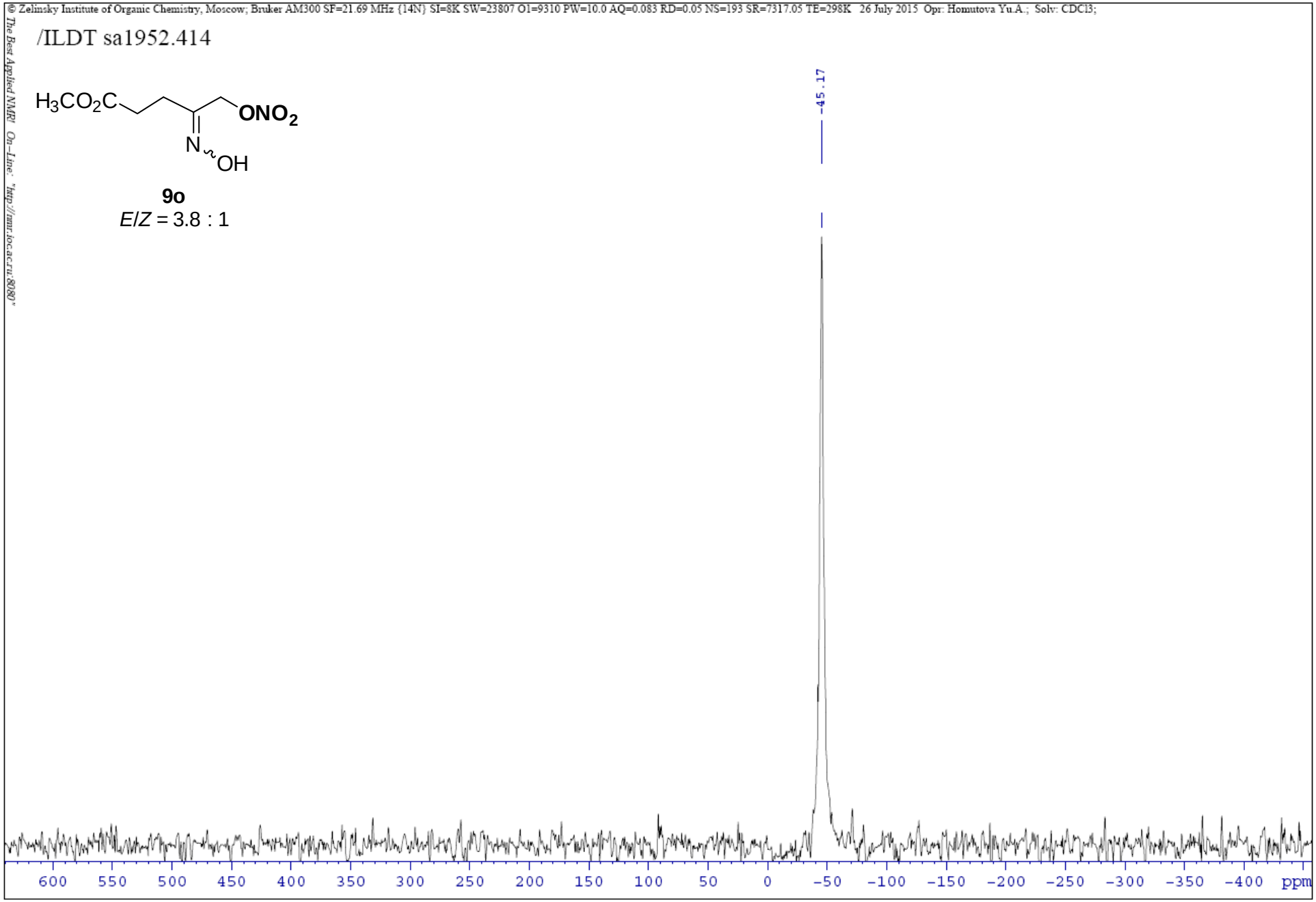
9n

E/*Z* = 6.3 : 1

C:\OPUS_7.0.122\EDL1\NY-146.0	НАУМОВИЧ. NY-146 , тонкий слой , пл.KBr.	10.02.2016

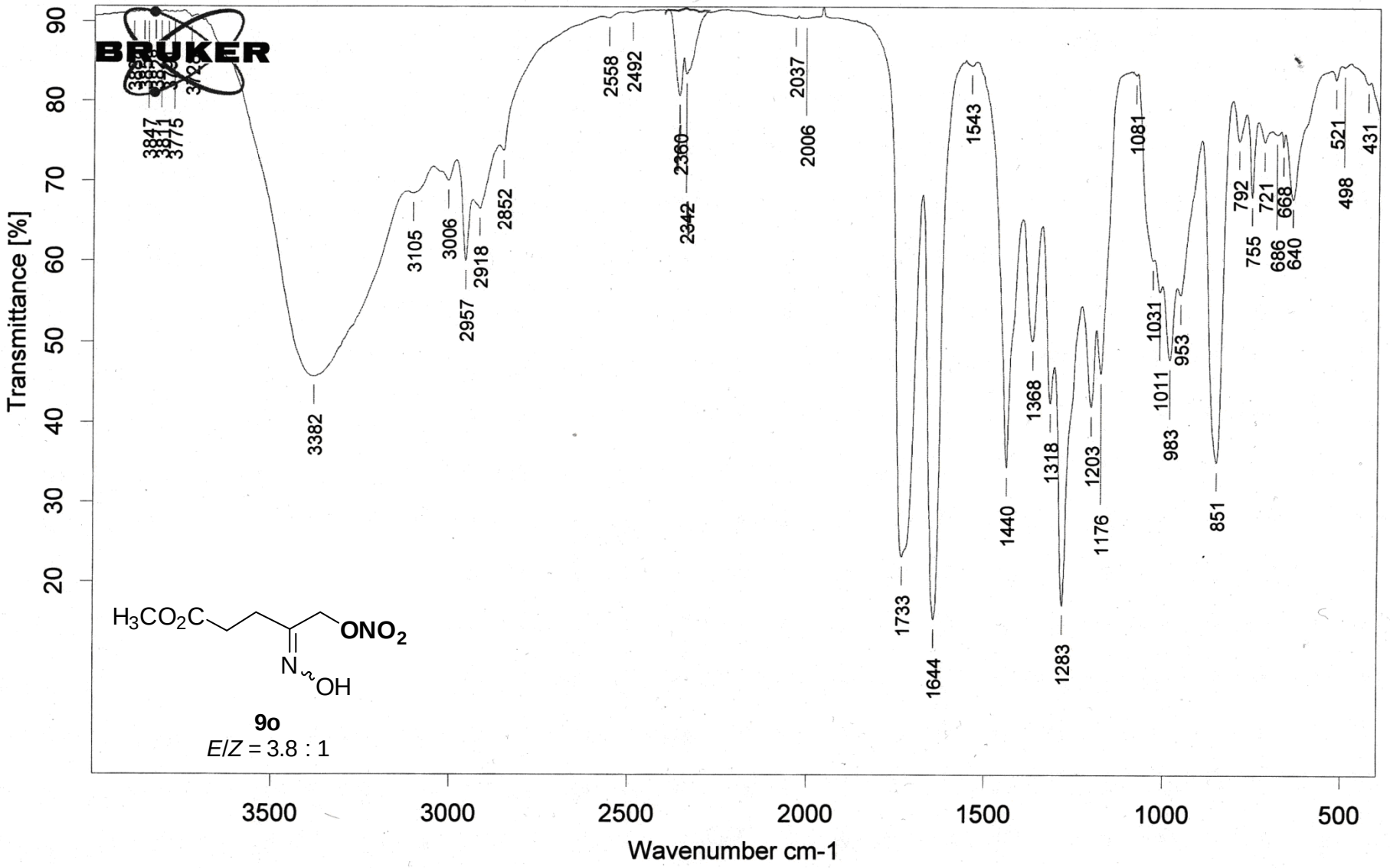

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=1.00 NS=1 SR=-0.01 TE=300K 6 July 2015 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT NY081.201
8.83
5.34
5.08
3.80
3.71
2.69
2.62
H3CO2C
ONO2
N
OH
9o
E/Z = 3.8 : 1
125
102
45
25
1.2
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.234
25.356
45.162
102.033
124.994


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C}JMOD SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.901 RD=1.50 NS=755 SR=-7.02 TE=300K 8 July 2015 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT NY081.202
H3CO2C
ONO2
N
OH
9o
E/Z = 3.8 : 1
173.17
152.95
77.53
77.10
76.68
72.53
66.81
51.99
29.94
29.86
29.67
25.66
21.96
21.37
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=21.69 MHz {14N} SI=8K SW=23807 O1=9310 PW=10.0 AQ=0.083 RD=0.05 NS=193 SR=7317.05 TE=298K 26 July 2015 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR On-Line: "http://nmr.ioca.ru:8080"
/ILDT sa1952.414
H3CO2C
ONO2
N
OH
-45.17
600 550 500 450 400 350 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -350 -400 ppm

9o

E/*Z* = 3.8 : 1

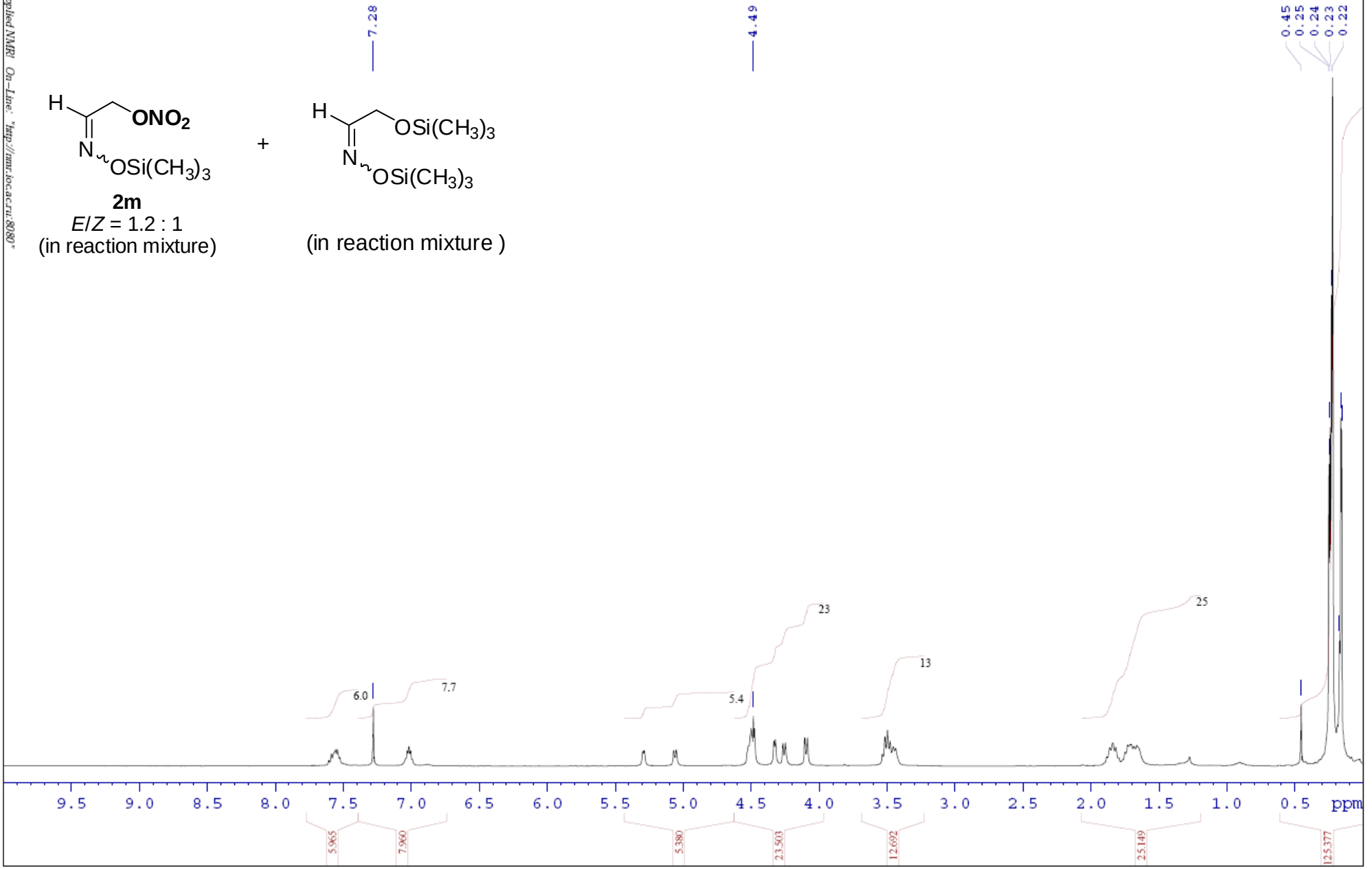

BRUKER
Transmittance [%]
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
20
30
40
50
60
70
80
90
3847
3775
3382
3105
3006
2957
2918
2852
2558
2492
2360
2342
2037
2006
1733
1644
1543
1440
1368
1318
1283
1203
1176
1081
1031
1011
983
953
851
792
755
721
686
668
640
521
498
431
H3CO2C
ONO2
N
OH
9o
E/Z = 3.8 : 1
D:\EDL\SA1952.0
SA1952
KBr.

/ILDT SA1941.101

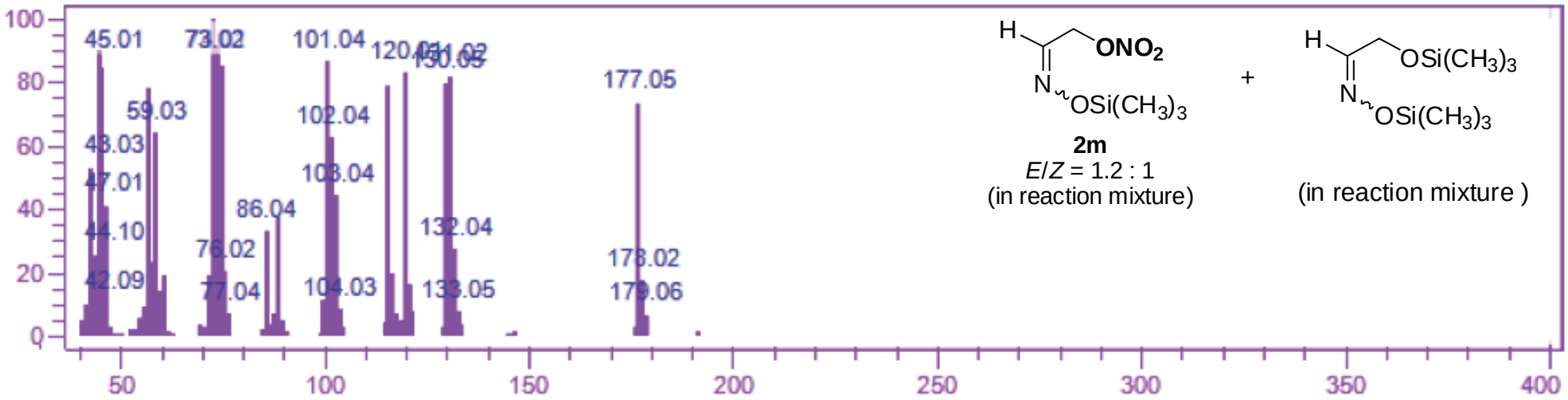
2m
E/*Z* = 1.2 : 1
(in reaction mixture)

+

(in reaction mixture)

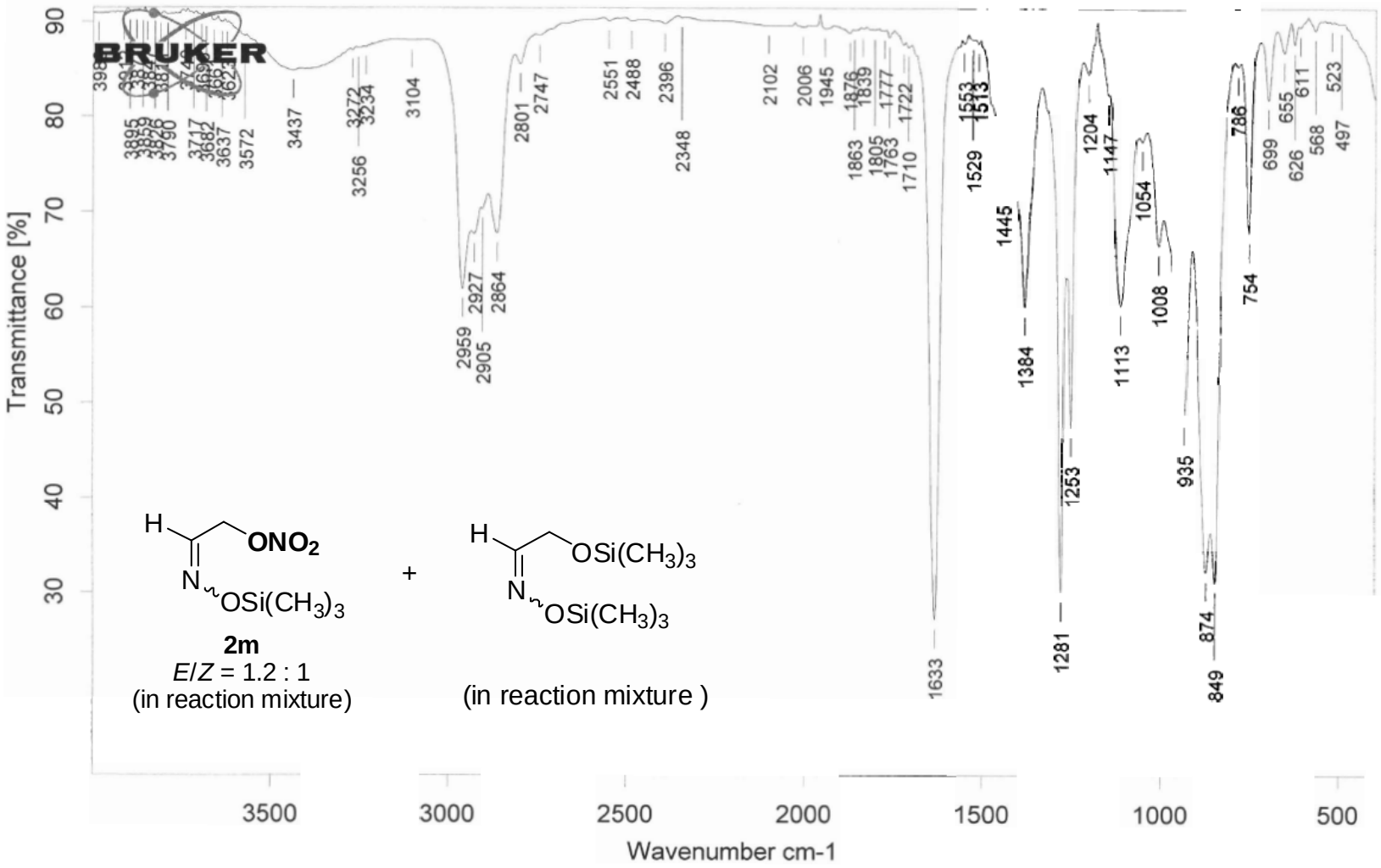


MS-spectra (EI)

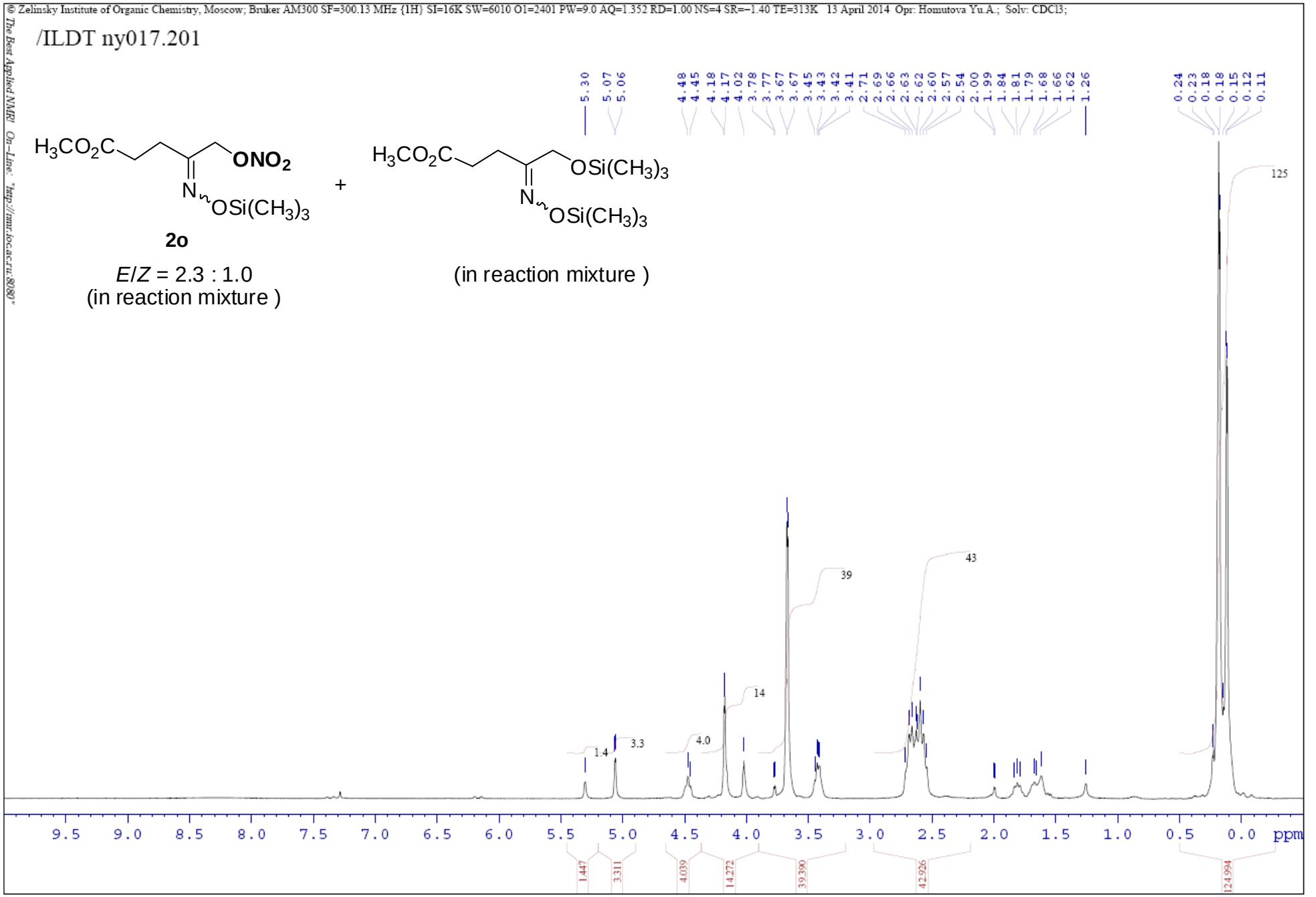


2-(Trimethylsilyloxyimino)ethyl nitrate (2m). Yield: c.a. 15% (procedure *iii*, determined by ^{1}H NMR with internal standard). Characterized in reaction mixture containing **2m** (18%), [(trimethylsilyl)oxy]acetaldehyde *O*-(trimethylsilyl)oxime* (23%) and unidentified products by ^{1}H NMR and GC-MS. Mixture of E/Z-isomers, ratio 1.2 : 1. ^{1}H NMR ($CDCl_3$, 300.13 MHz, E-isomer): 5.06 (d, J = 5.5 Hz, 2 H, C*H*$_2$ONO$_2$), 7.56 (t, J = 5.5 Hz, 1 H, C*H*). ^{1}H NMR ($CDCl_3$, 300.13 MHz, Z-isomer): 5.28 (d, J = 3.7 Hz, 2 H, C*H*$_2$ONO$_2$), 7.02 (t, J = 3.7 Hz, 1 H, C*H*). FTIR (thin layer): 1633 (s, ONO_2). MS (EI): m/z = 192 (1) $[M]^{+\cdot}$, 177 (70) $[M–CH_3]^+$, 130 (80) $[M–NO_3]^+$, 116 (80) $[M–CH_2ONO_2]^+$, 76 (20) $[CH_2ONO_2]^+$, 73 (100) $[(CH_3)_3Si]^+$.

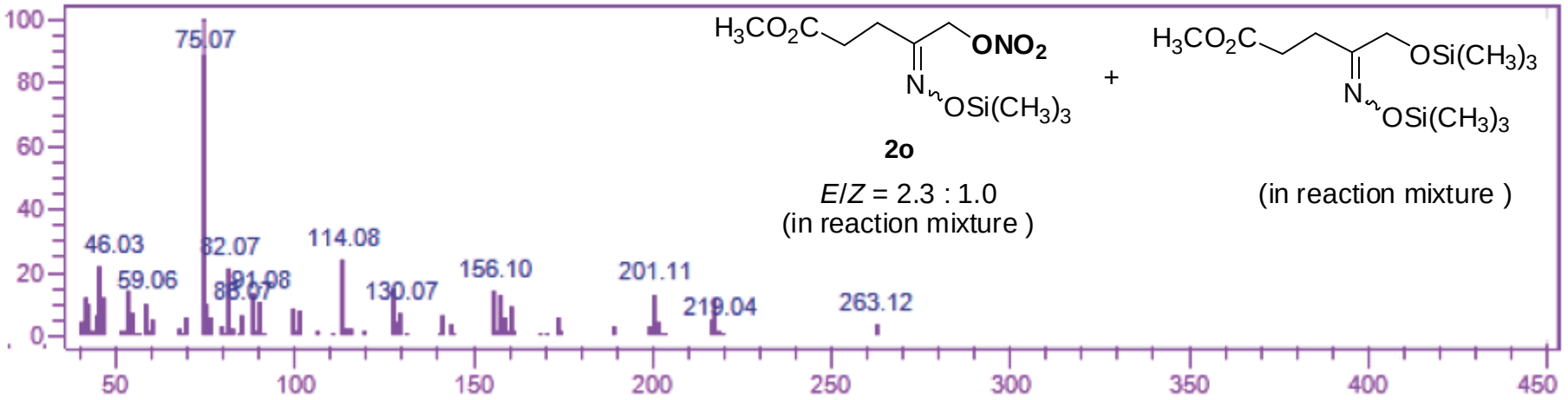
* A. A. Tabolin, A. V. Lesiv, Yu. A. Khomutova, P. A. Belyakov, Yu. A. Strelenko, S. L. Ioffe, *Synthesis*, 2005, 1656-1662.



D:\EDL\SA-1957.0 SA-1957

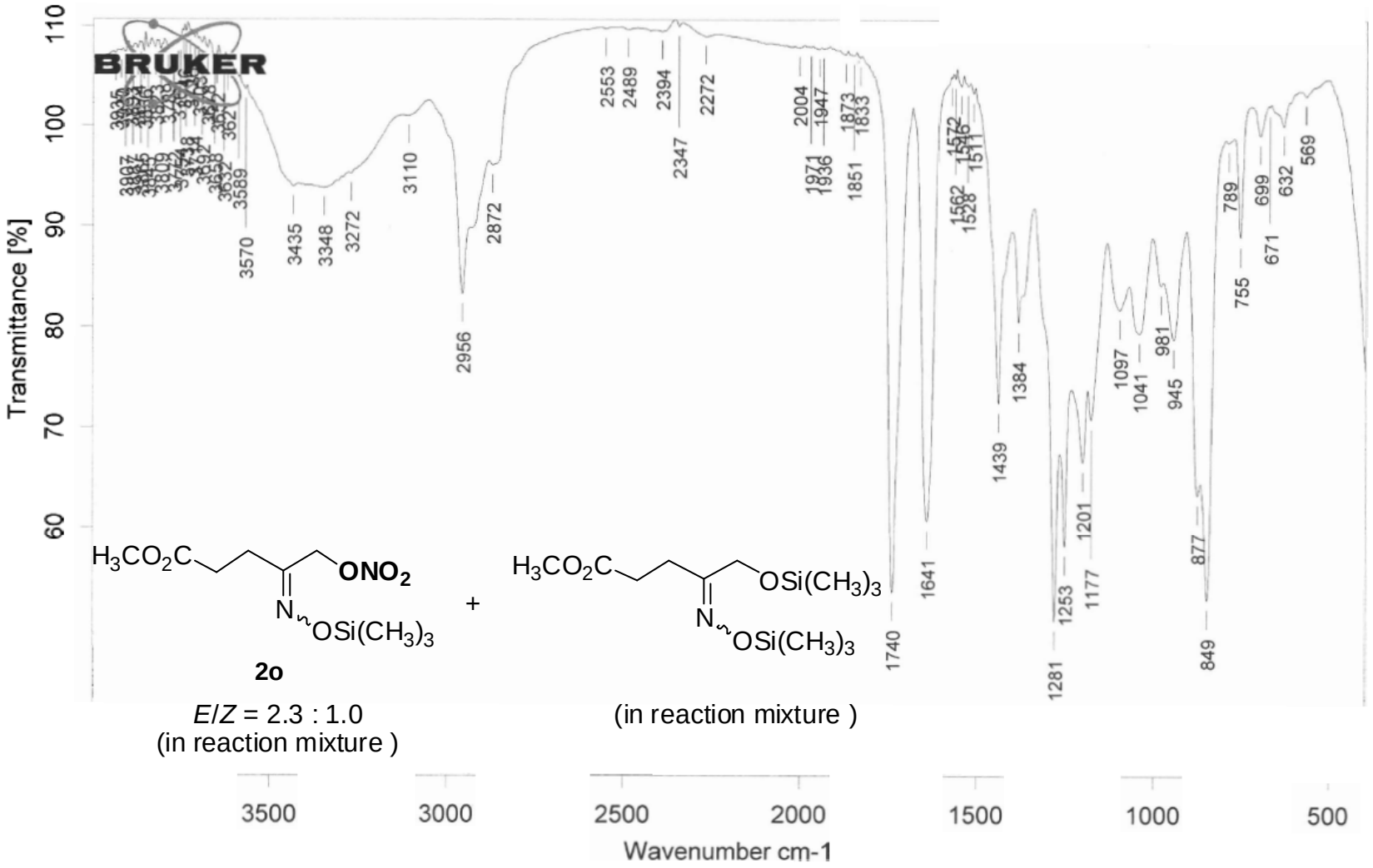

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=1.00 NS=4 SR=-1.40 TE=313K 13 April 2014 Opr: Homutova Yu.A.; Solv: CDCl3;
The Best Applied NMR On-Line: "http://nmr.ioc.ac.ru:8080"
/ILDT ny017.201
H3CO2C
ONO2
N
OSi(CH3)3
2o
E/Z = 2.3 : 1.0
(in reaction mixture)
+
H3CO2C
OSi(CH3)3
N
OSi(CH3)3
(in reaction mixture)
5.30 5.07 5.06
4.48 4.45 4.18 4.17 4.02 3.78 3.77 3.67 3.67 3.45 3.43 3.42 3.41 2.71 2.69 2.66 2.63 2.62 2.60 2.57 2.54 2.00 1.99 1.84 1.81 1.79 1.68 1.66 1.62 1.26
0.24 0.23 0.18 0.18 0.15 0.12 0.11
125
43
39
14
4.0
3.3
1.4
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.447
3.311
4.039
14.272
39.390
42.926
124.994

MS-spectra (EI)



Methyl 5-(nitrooxy)-4-(trimethylsilyloxyimino)pentanoate (2o). Yield: c.a. 10% (procedure *iii*, determined by ^{1}H NMR with internal standard). Characterized in reaction mixture containing **2o** (16%), methyl -5-[(trimethylsilyl)oxy]-4-{[(trimethylsilyl)oxy]imino}pentanoate[†] (24%) and unidentified products by ^{1}H NMR and GC-MS. Mixture of E/Z-isomers, ratio 2.3 : 1. ^{1}H NMR ($CDCl_3$, 300.13 MHz, E-isomer): 0.18 ($(CH_3)_3Si$), 2.54-2.71 (m, 4 H, CH_2-CH_2), 5.07 (s, 2 H, CH_2ONO_2). Characteristic signals of *Z*-**2o**: 5.30 (s, 2 H, CH_2ONO_2). FTIR (thin layer): 1740 (s, C=O), 1641 (s, ONO_2). MS (EI): *m*/*z* = 263 (5) [M–CH_3], 76 (15) $[CH_2ONO_2]^+$, 75 (100) $[(CH_3)_2SiOH]^+$.

[†] A. A. Tabolin, A. V. Lesiv, Yu. A. Khomutova, P. A. Belyakov, Yu. A. Strelenko, S. L. Ioffe, *Synthesis,* 2005, 1656-1662.



2o

E/*Z* = 2.3 : 1.0
(in reaction mixture)

+

(in reaction mixture)

D:\EDL\SA-1958.0 SA-1958

X-Ray of Nitrate Ester 2g

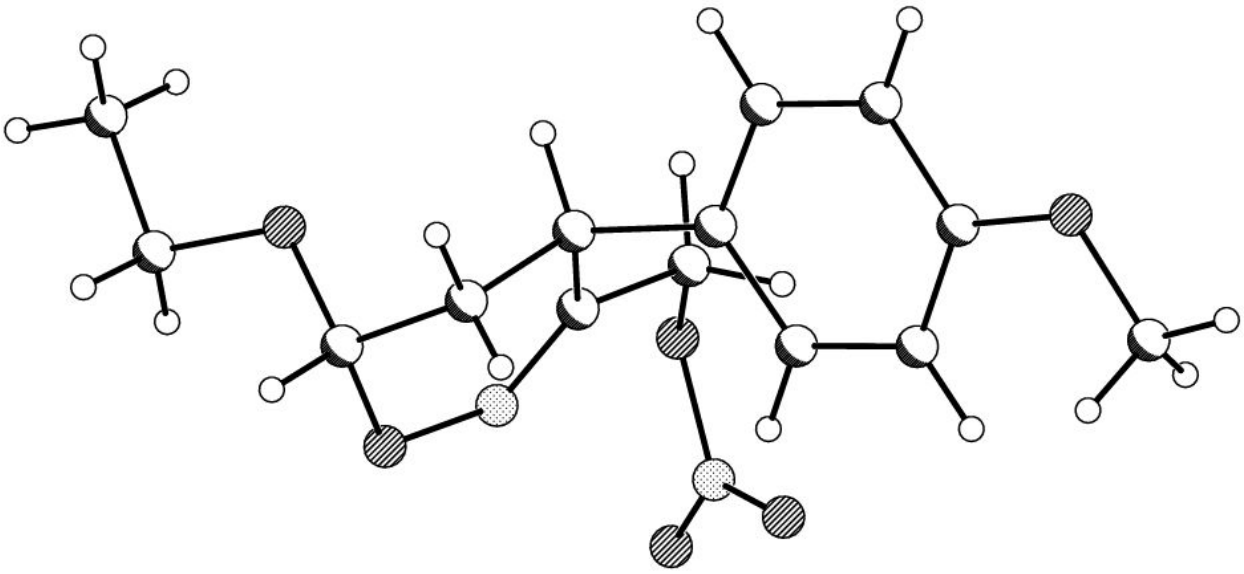


Fig. S1. General view of the compound **2g** from X-ray diffraction data.

Crystallographic data: Crystals of **2g** ($C_{14}H_{18}N_2O_6$, M = 310.30) are triclinic, space group P-1, at 120 K: a = 5.1957(4), b = 12.0390(9), c = 12.8927(10) Å, α = 112.826(2), β = 90.743(2), γ = 102.195(2)˚, V = 722.48(10) $Å^3$, Z = 2 (Z' = 1), d_{calc} = 1.426 gcm^{-3}, μ(MoKα) = 1.13 cm^{-1}, F(000) = 328. Intensities of 8717 reflections were measured with a Bruker SMART APEX2 CCD diffractometer [λ(MoKα) = 0.71072Å, ω-scans, 2θ<58°], and 3824 independent reflections [R_{int} = 0.0253] were used in further refinement. The structure was solved by direct method and refined by the full-matrix least-squares technique against F^2 in the anisotropic-isotropic approximation. The H(C) atom positions were calculated, and they were refined in the isotropic approximation within the riding model. The refinement converged to wR2 = 0.1493 and GOF = 1.009 for all independent reflections (R1 = 0.0442 was calculated against F for 3020 observed reflections with I>2σ(I))**.** All calculations were performed using SHELXTL PLUS 5.0. [Sheldrick, G. M. *SHELXTL v. 5.10, Structure Determination Software Suit*. Bruker AXS, Madison, Wisconsin, USA].

CCDC 1419082 contains the supplementary crystallographic data for **2g**. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html (or from the CCDC, 12 Union Road, Cambridge, CB21EZ, UK; or deposit@ccdc.cam.ac.uk).

UV titration of $Cr(NO_3)_3 \bullet 9H_2O$ solution in THF with bis(oxy)enamine 1a solution in CH_2Cl_2