Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

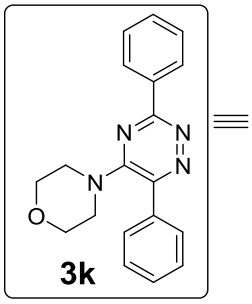
Solvent-free synthesis of 5-(aryl/alkyl)amino-1,2,4-triazines and α-arylamino-2,2′-bipyridines with a greener prospect

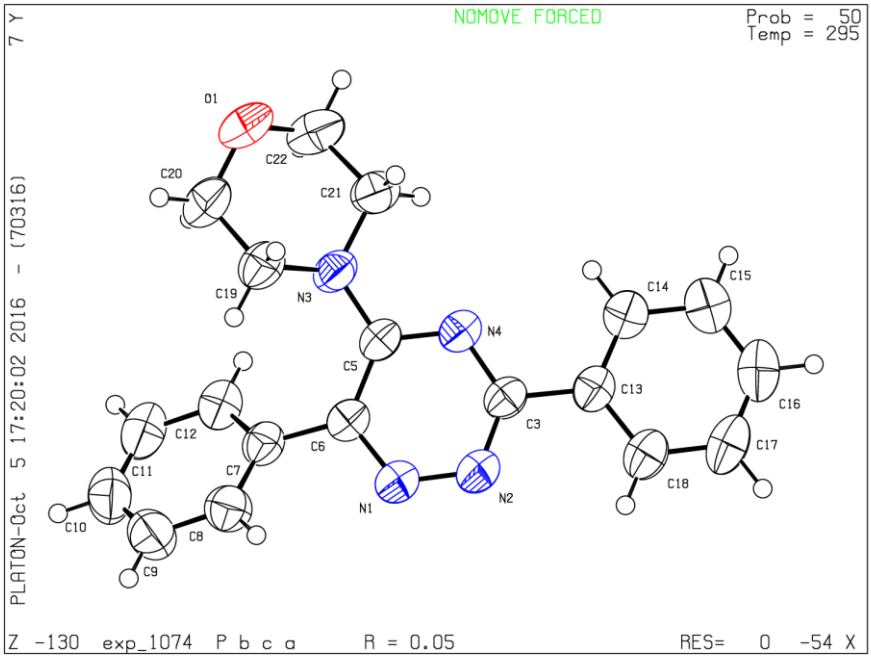
Dmitry S. Kopchuk, Nikolay V. Chepchugov, Igor S. Kovalev, Sougata Santra,* Matiur Rahman, Kousik Giri, Grigory V. Zyryanov, Adinath Majee,* Valery N. Charushin, Oleg N. Chupakhin

Table of Contents

Structure Determination (X-ray crystallographic data for 3k)

The white crystals of **3k** were obtained by crystallization from a solution in ethanol after purification by column chromatography. Chemical formula $C_{19}H_{18}N_4O$.





ORTEP (with 50% probability) diagram for the structure 5-(Morpholine-4-yl)-3,6-diphenyl-1,2,4-triazine **(3k)**.

Wavelength	0.71070 Å	
Formula	C_{19} H_{18} N_4 O	
Crystal system	Orthorhombic	
Space group	P b c a	
Unit cell dimensions	a = 11.5790(8) Å	α = 90 °
	b = 9.4791(5) Å	β = 90 °
	c = 30.0335(19) Å	γ = 90 °
Volume	3296.43 $Å^3$	
Z	8	
R-factor (%)	5.14	

The crystallographic data have been deposited with the Cambridge Crystallographic Data Centreas supplementary publication with a CCDC reference number CCDC **1505314.**

E-factor calculations for compounds 3:

For **3a:** E = [0.137 g (**1a**) + 0.047 g (**2a**) – 0.160 g (product × yield)] / 0.160 g = 0.15

For **3b**: E = [0.130 g (**1b**) + 0.047 g (**2a**) – 0.151 g (product × yield)] / 0.151 g = 0.17

For **3c**: E = [0.137 g (**1a**) + 0.082 g (**2b**) – 0.190 g (product × yield)] / 0.190 g = 0.15

For **3d**: E = [0.137 g (**1a**) + 0.069 g (**2c**) – 0.179 g (product × yield)] / 0.179 g = 0.15

For **3e**: E = [0.137 g (**1a**) + 0.062 g (**2d**) – 0.175 g (product × yield)] / 0.175 g = 0.14

For **3f**: E = [0.130 g (**1b**) + 0.062 g (**2e**) – 0.169 g (product × yield)] / 0.169 g = 0.14

For **3g**[a]**:** E = [0.131 g (**1c**) + 0.047 g (**2a**) + 8.65 g (Toluene) – 0.150 g (product × yield)] / 0.150 g = 58

For **3h**[a]**:** E = [0.139 g (**1d**) + 0.047 g (**2a**) + 8.65 g (Toluene) – 0.150 g (product × yield)] / 0.150 g = 58

For **3i:** E = [0.129 g (**1e**) + 0.047 g (**2a**) – 0.160 g (product × yield)] / 0.160 g = 0.10

For **3j**[a]: E = [0.129 g (**1e**) + 0.044 g (**2f**) + 8.65 g (Toluene) – 0.146 g (product × yield)] / 0.146 g = 59

For **3k**[a]: E = [0.129 g (**1e**) + 0.051 g (**2g**) + 8.65 g (Toluene) – 0.143 g (product × yield)] / 0.143 g = 61

For **3l**: E = [0.129 g (**1e**) + 0.036 g (**2h**) – 0.137 g (product × yield)] / 0.137 g = 0.20

For **3m**: E = [0.129 g (**1e**) + 0.050 g (**2i**) – 0.149 g (product × yield)] / 0.149 g = 0.20

For **3n**[a]**:** E = [0.105 g (**1f**) + 0.047 g (**2a**) + 8.65 g (Toluene) – 0.120 g (product × yield)] / 0.120 g = 72

For **3o**[a]**:** E = [0.137 g (**1a**) + 0.085 g (**2j**) + 8.65 g (Toluene) – 0.053 g (product × yield)] / 0.053 g = 166

[a]Flash chromatography was done using toluene as eluent.

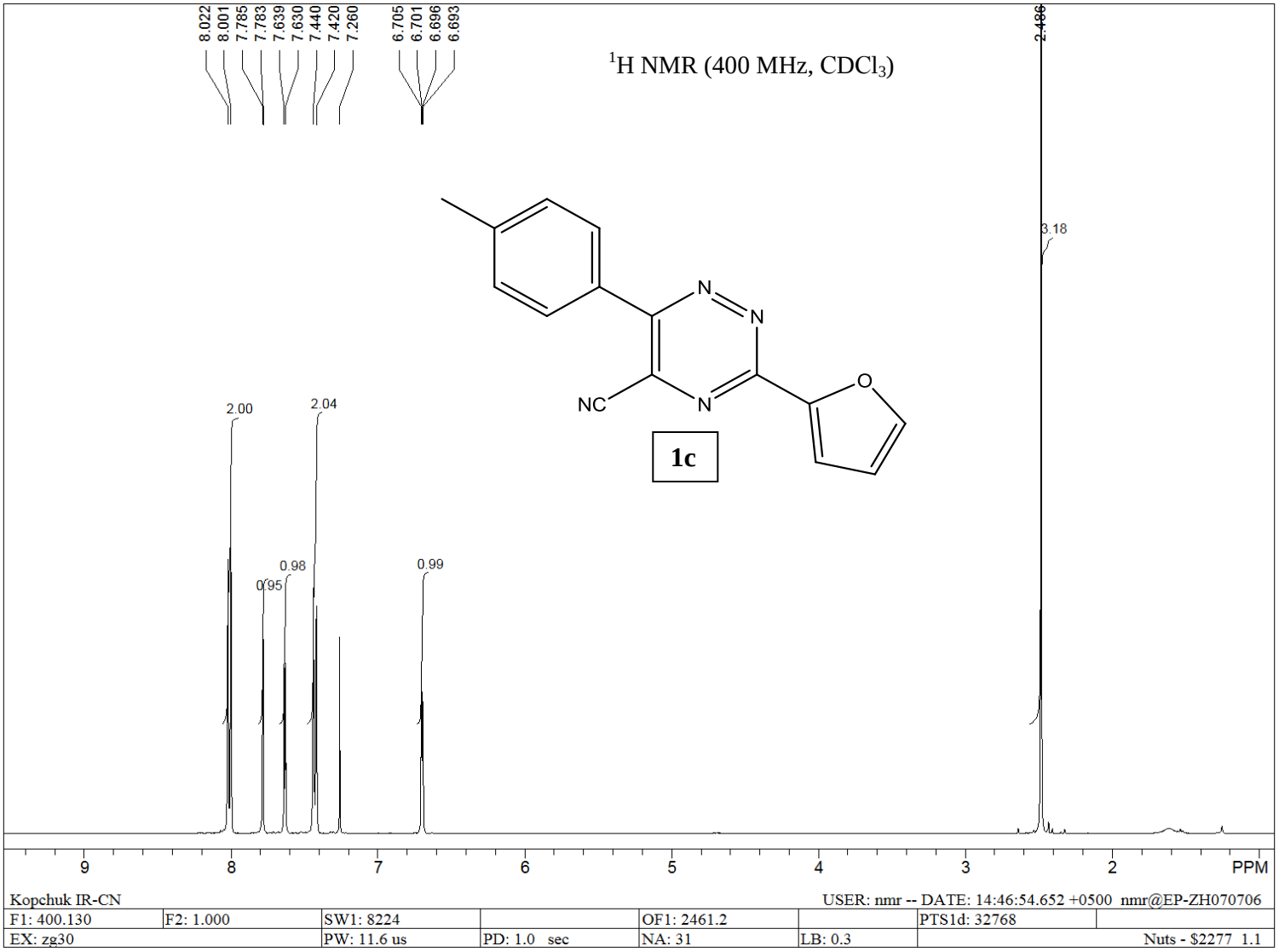
E-factor calculations for compounds 4:[a]

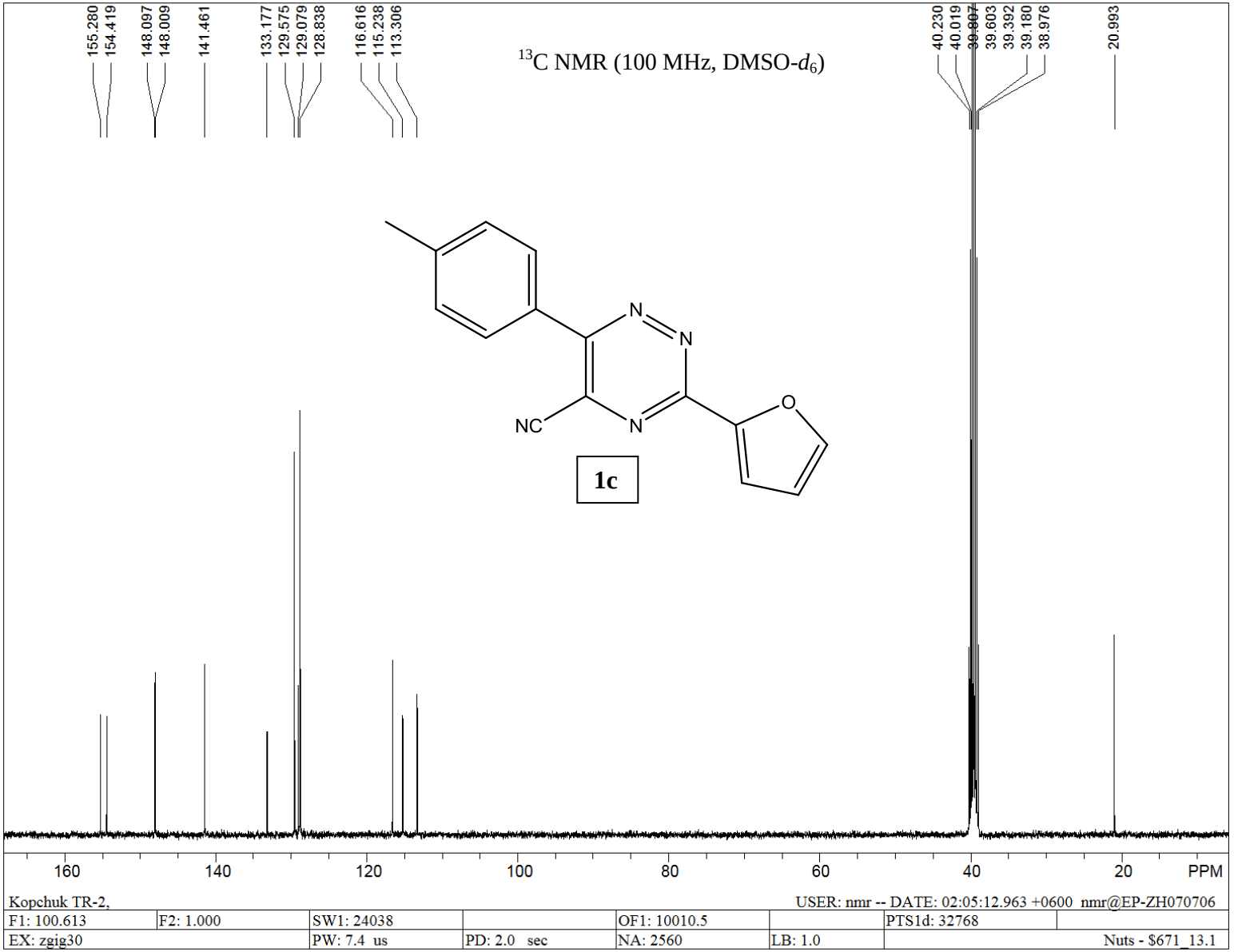
For **4a**: E = [0.095 g (**3a**) + 0.064 g (1-morpholinocyclopentene) + 3.945 g (Ethanol) – 0.100 g (product × yield)] / 0.100 g = 40. (0.28 mmol reaction was carried out)

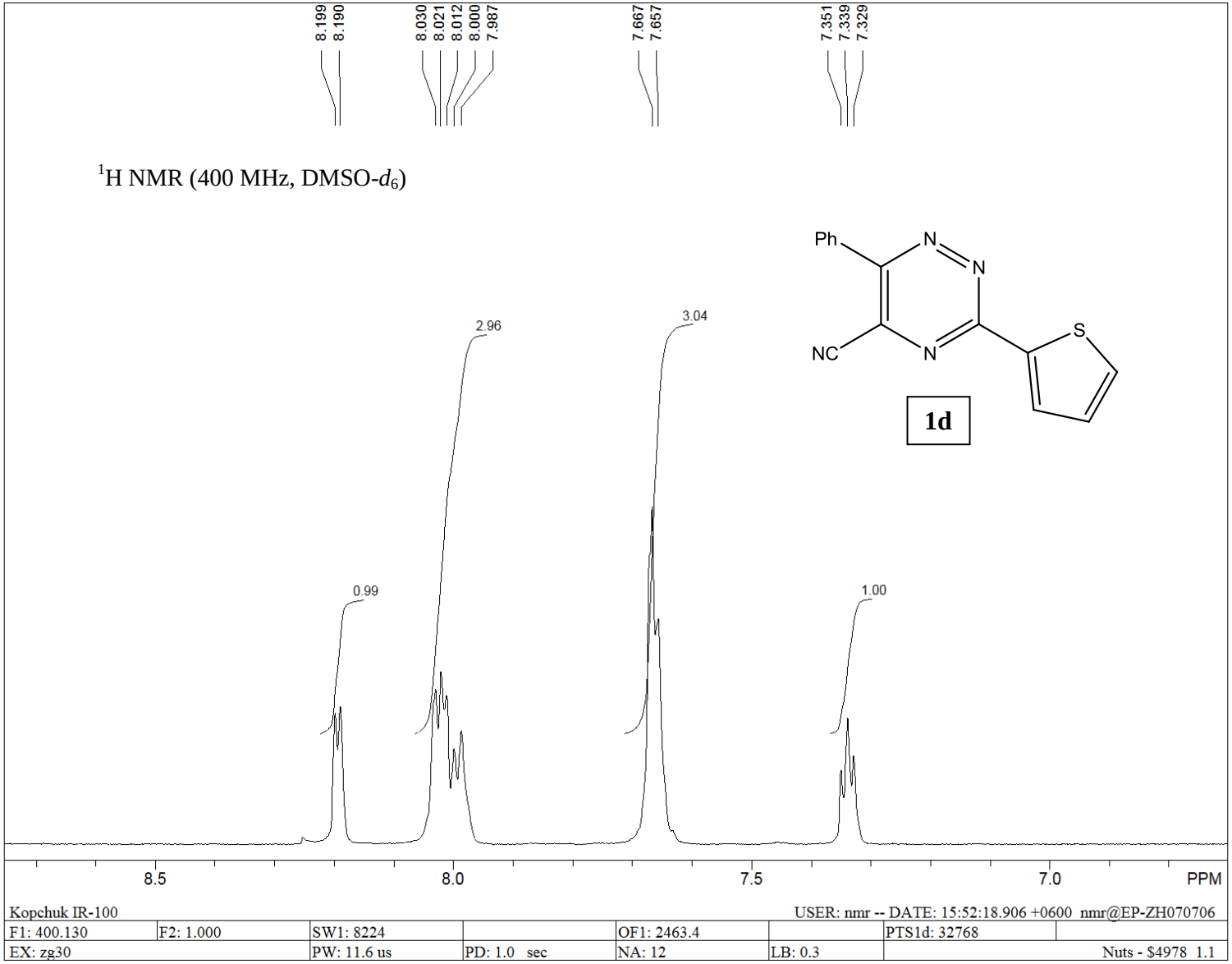
For **4b**: E = [0.084 g (**3b**) + 0.060 g (1-morpholinocyclopentene) + 3.945 g (Ethanol) – 0.096 g (product × yield)] / 0.096 g = 41. (0.26 mmol reaction was carried out.)

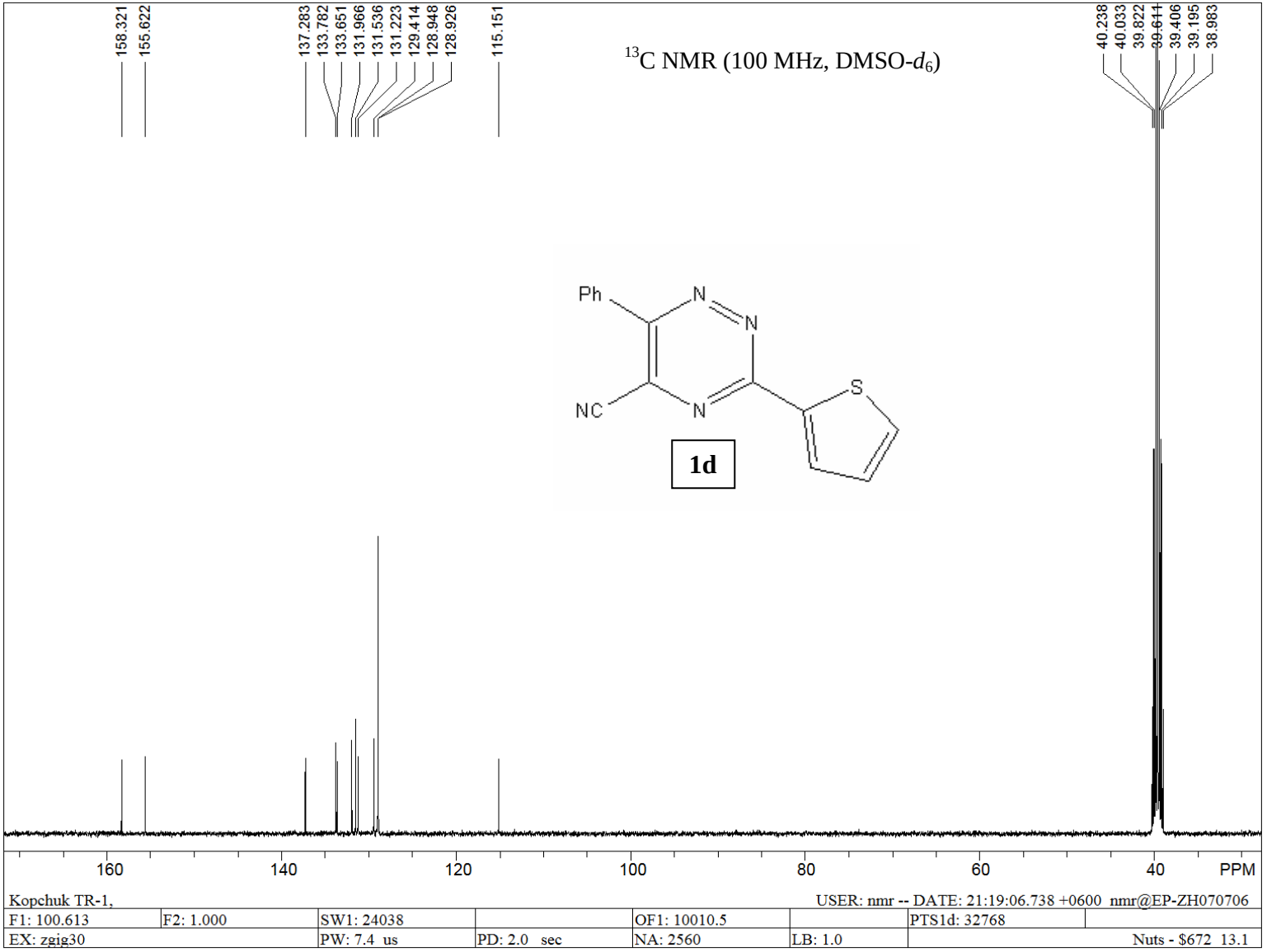
For **4c**: E = [0.092 g (**3e**) + 0.057 g (1-morpholinocyclopentene) + 3.945 g (Ethanol) – 0.100 g (product × yield)] / 0.100 g = 40. (0.25 mmol reaction was carried out)

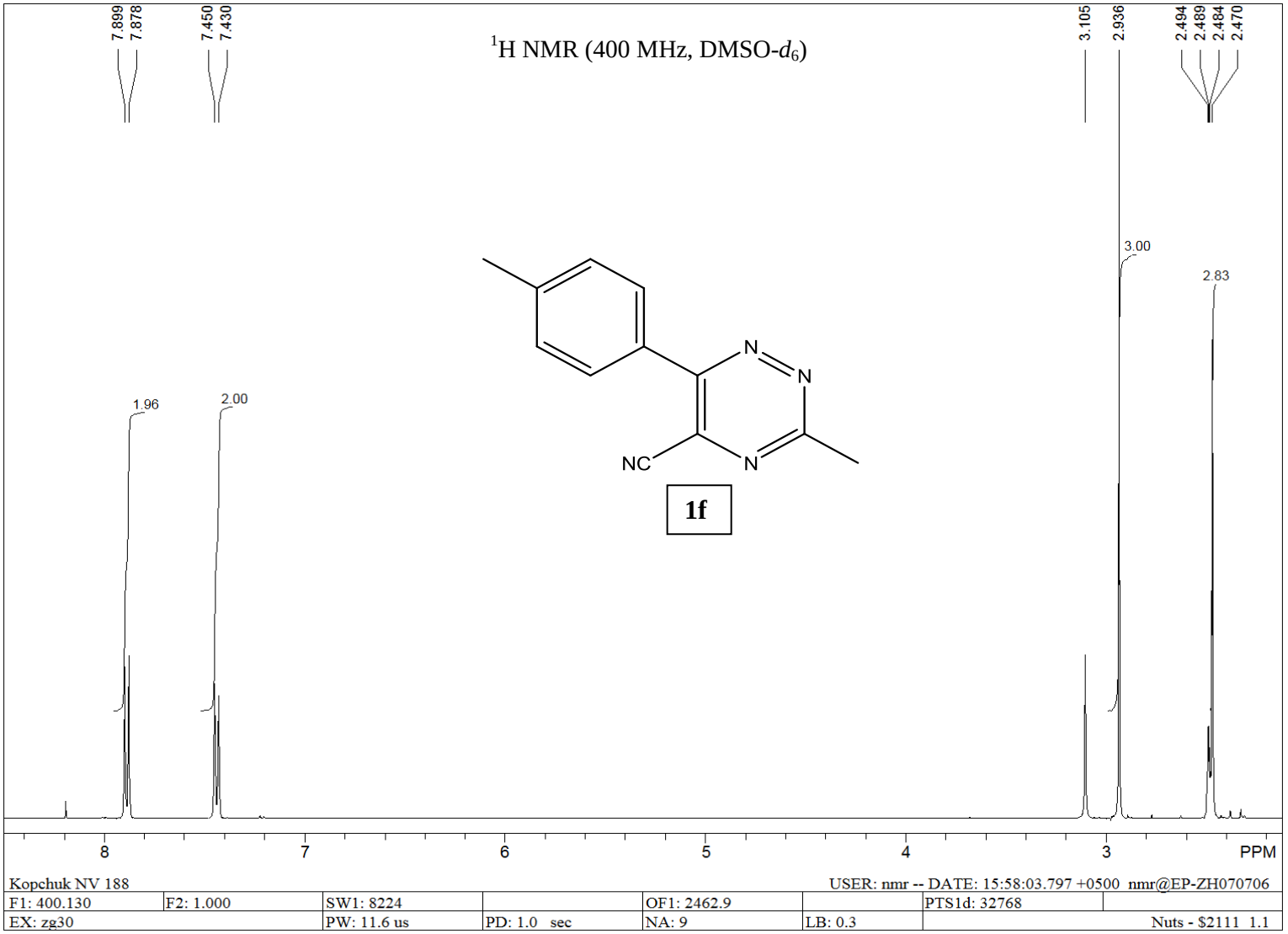
[a] 5 mL ethanol was used for recrystallization.

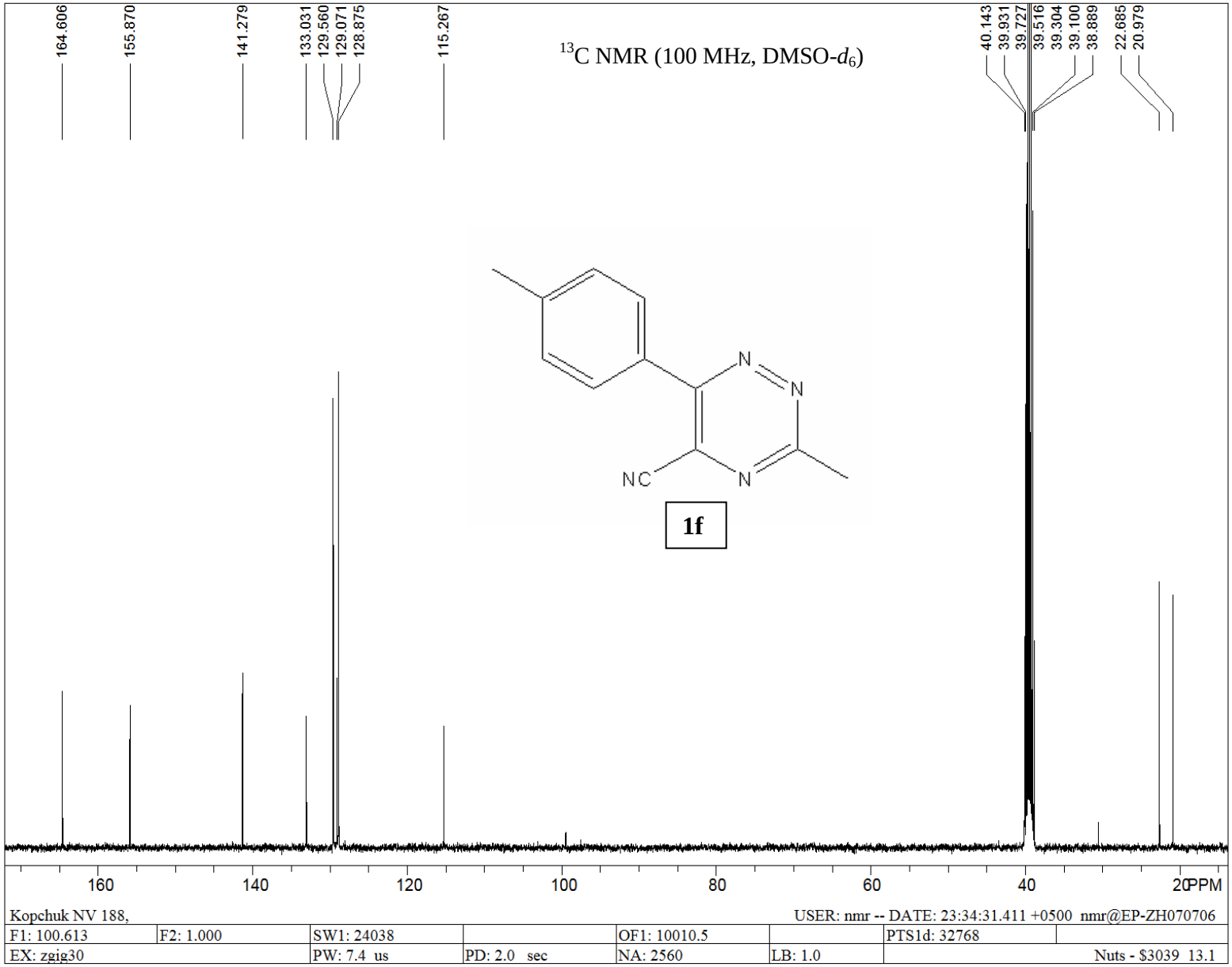

8.022
8.001
7.785
7.783
7.639
7.630
7.440
7.420
7.260
6.705
6.701
6.696
6.693
2.486
^{1}H NMR (400 MHz, $CDCl_3$)
3.18
2.00
2.04
0.98
0.95
0.99
N
N
N
NC
O
1c
9
8
7
6
5
4
3
2
PPM
Kopchuk IR-CN
USER: nmr -- DATE: 14:46:54.652 +0500 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2461.2
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 31
LB: 0.3
Nuts - $2277 1.1

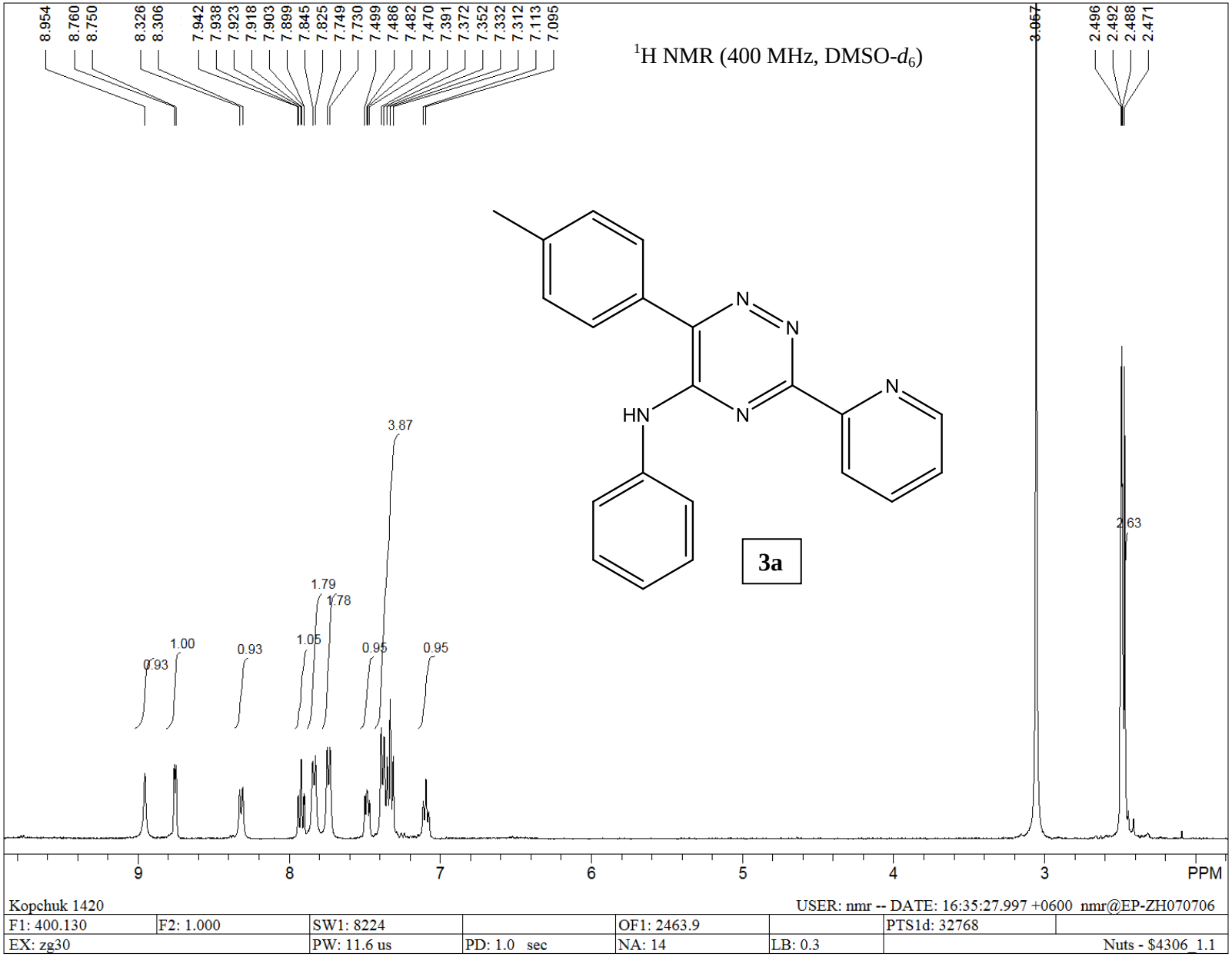

155.280
154.419
148.097
148.009
141.461
133.177
129.575
129.079
128.838
116.616
115.238
113.306
40.230
40.019
39.807
39.603
39.392
39.180
38.976
20.993
^{13}C NMR (100 MHz, DMSO-d_6)
N
N
NC
N
O
1c
160
140
120
100
80
60
40
20
PPM
Kopchuk TR-2,
USER: nmr -- DATE: 02:05:12.963 +0600 nmr@EP-ZH070706
F1: 100.613
F2: 1.000
SW1: 24038
OF1: 10010.5
PTS1d: 32768
EX: zgig30
PW: 7.4 us
PD: 2.0 sec
NA: 2560
LB: 1.0
Nuts - $671_13.1

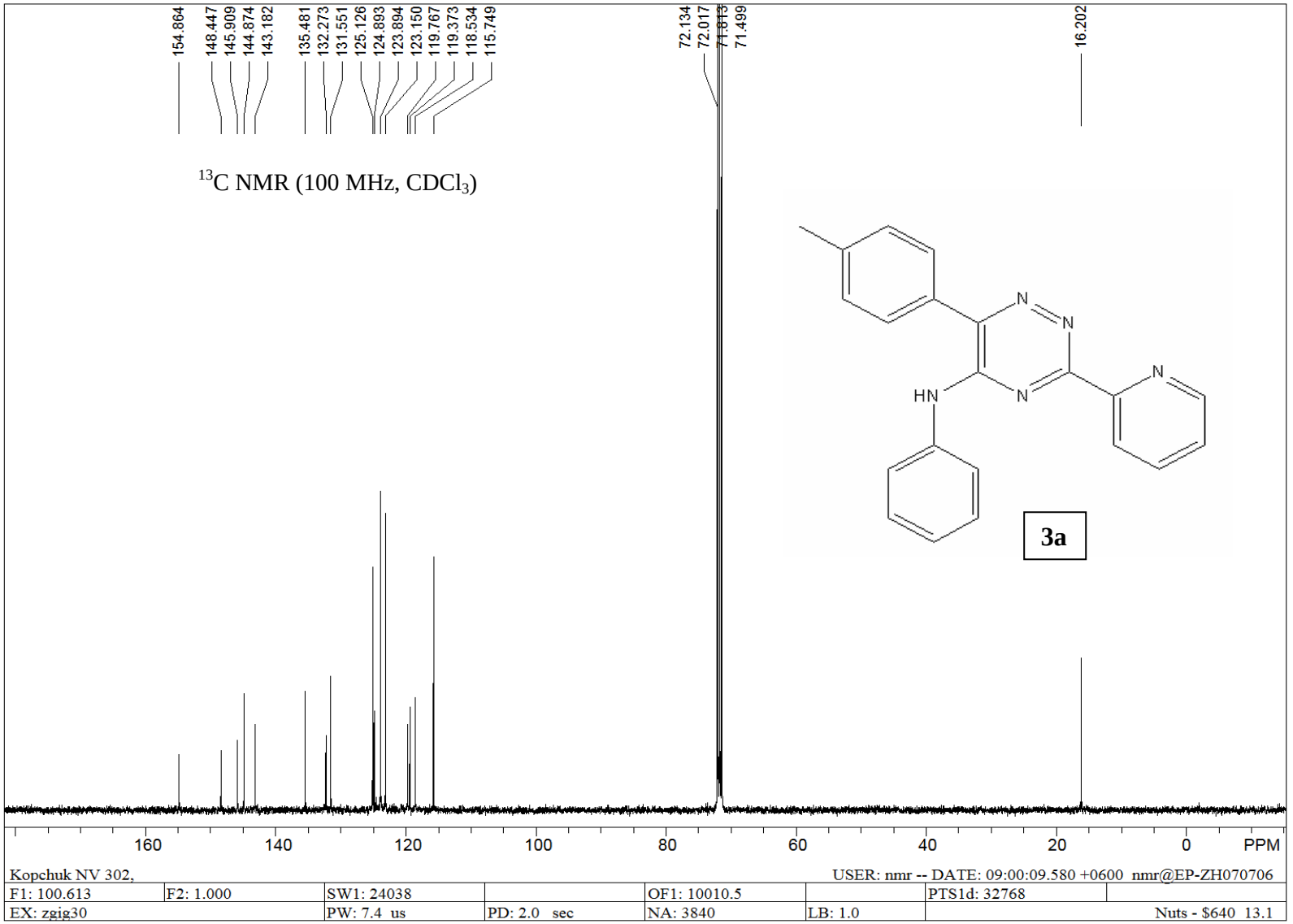

8.199
8.190
8.030
8.021
8.012
8.000
7.987
7.667
7.657
7.351
7.339
7.329
^{1}H NMR (400 MHz, DMSO-d_6)
Ph
N
N
NC
N
S
1d
2.96
3.04
0.99
1.00
8.5
8.0
7.5
7.0
PPM
Kopchuk IR-100
USER: nmr -- DATE: 15:52:18.906 +0600 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2463.4
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 12
LB: 0.3
Nuts - $4978 1.1

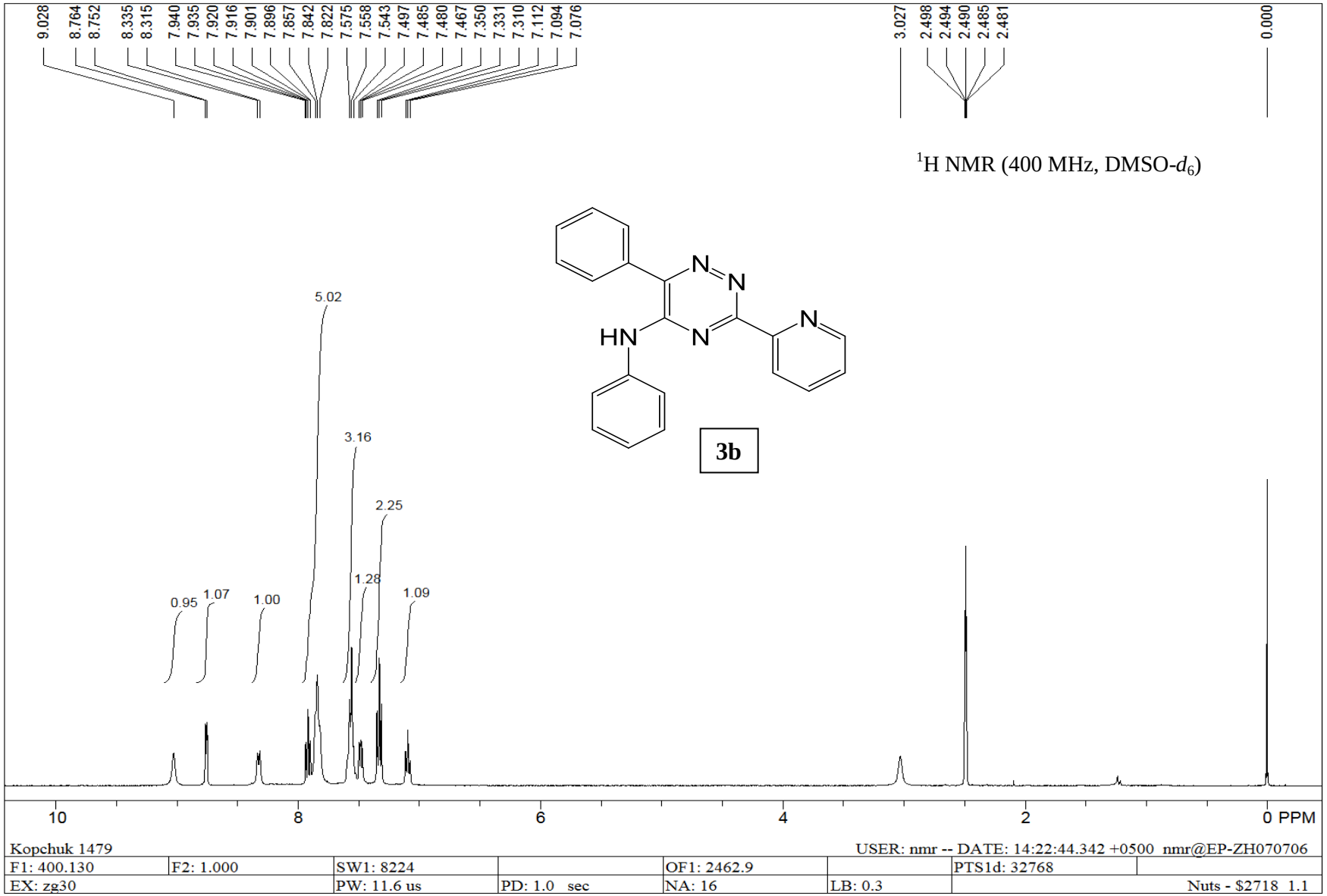

158.321
155.622
137.283
133.782
133.651
131.966
131.536
131.223
129.414
128.948
128.926
115.151
40.238
40.033
39.822
39.611
39.406
39.195
38.983
^{13}C NMR (100 MHz, DMSO-d_6)
Ph
N
N
NC
N
S
1d
160
140
120
100
80
60
40
PPM
Kopchuk TR-1,
USER: nmr -- DATE: 21:19:06.738 +0600 nmr@EP-ZH070706
F1: 100.613
F2: 1.000
SW1: 24038
OF1: 10010.5
PTS1d: 32768
EX: zgig30
PW: 7.4 us
PD: 2.0 sec
NA: 2560
LB: 1.0
Nuts - $672 13.1

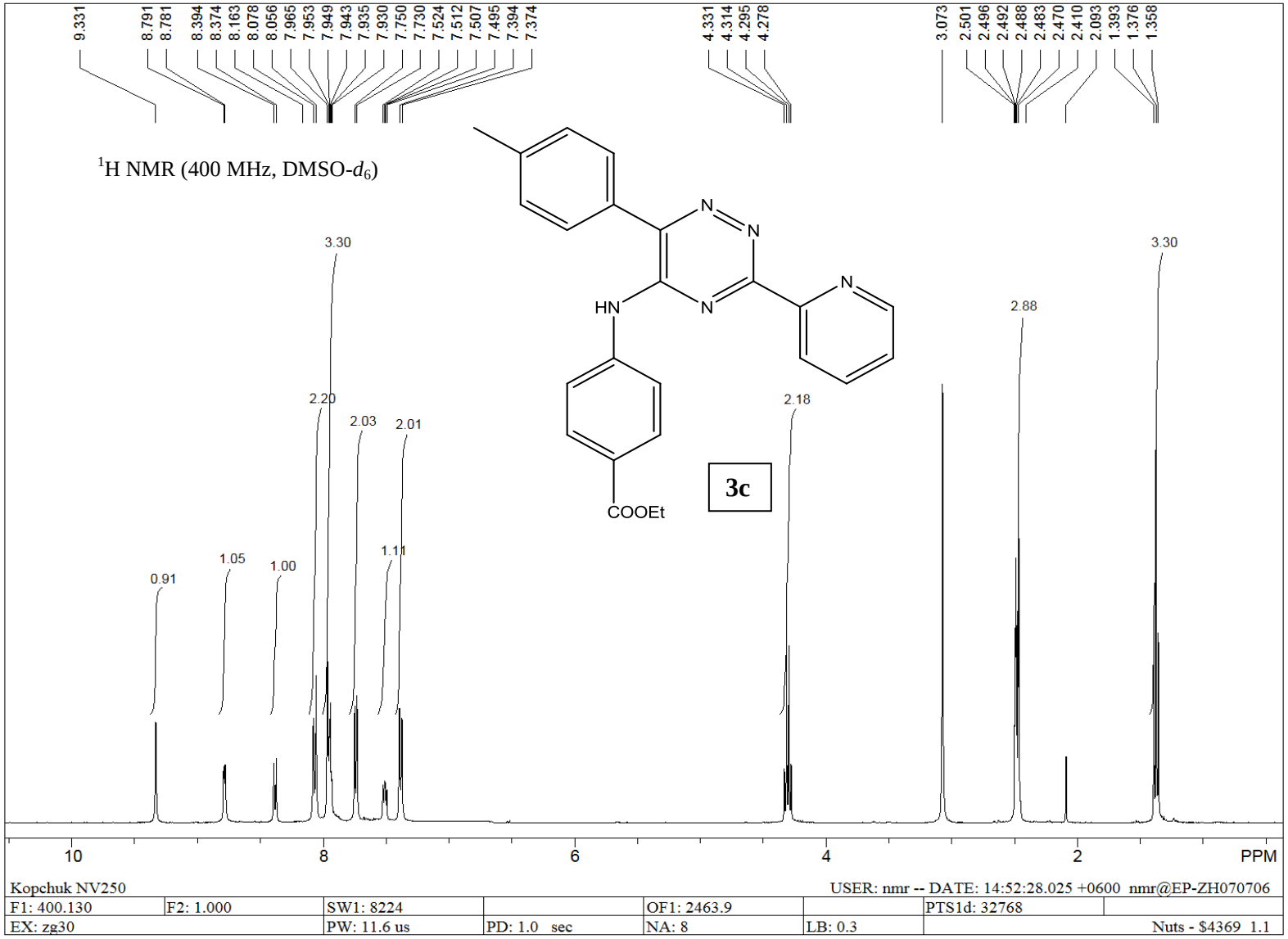

^{1}H NMR (400 MHz, DMSO-d_6)
7.899
7.878
7.450
7.430
3.105
2.936
2.494
2.489
2.484
2.470
3.00
2.83
1.96
2.00
N
N
N
NC
1f
8
7
6
5
4
3
PPM
Kopchuk NV 188
USER: nmr -- DATE: 15:58:03.797 +0500 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2462.9
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 9
LB: 0.3
Nuts - $2111 1.1

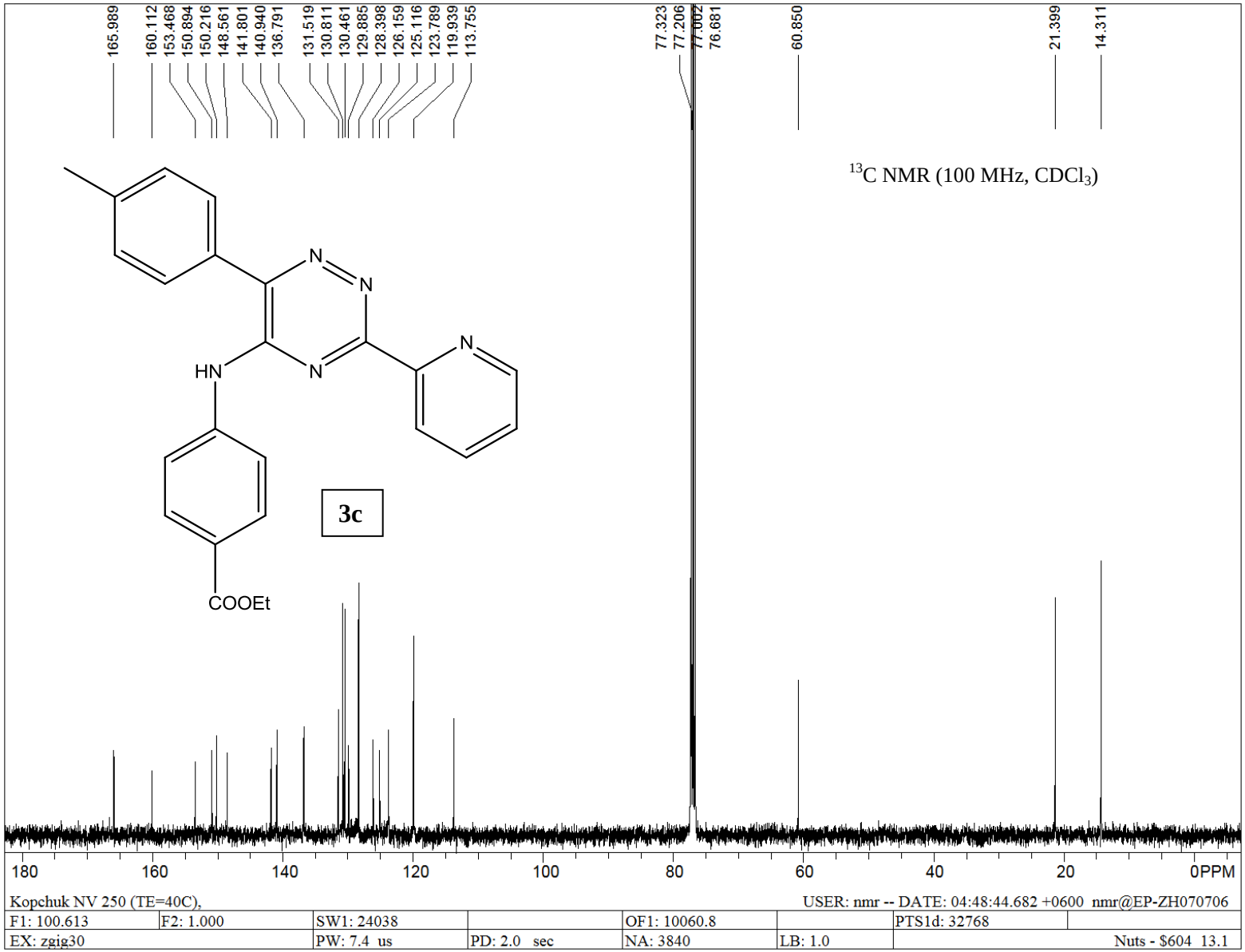

164.606
155.870
141.279
133.031
129.560
129.071
128.875
115.267
40.143
39.931
39.727
39.516
39.304
39.100
38.889
22.685
20.979
^{13}C NMR (100 MHz, DMSO-d_6)
N
N
NC
N
1f
160
140
120
100
80
60
40
20
PPM
Kopchuk NV 188,
USER: nmr -- DATE: 23:34:31.411 +0500 nmr@EP-ZH070706
F1: 100.613
F2: 1.000
SW1: 24038
OF1: 10010.5
PTS1d: 32768
EX: zgig30
PW: 7.4 us
PD: 2.0 sec
NA: 2560
LB: 1.0
Nuts - $3039 13.1

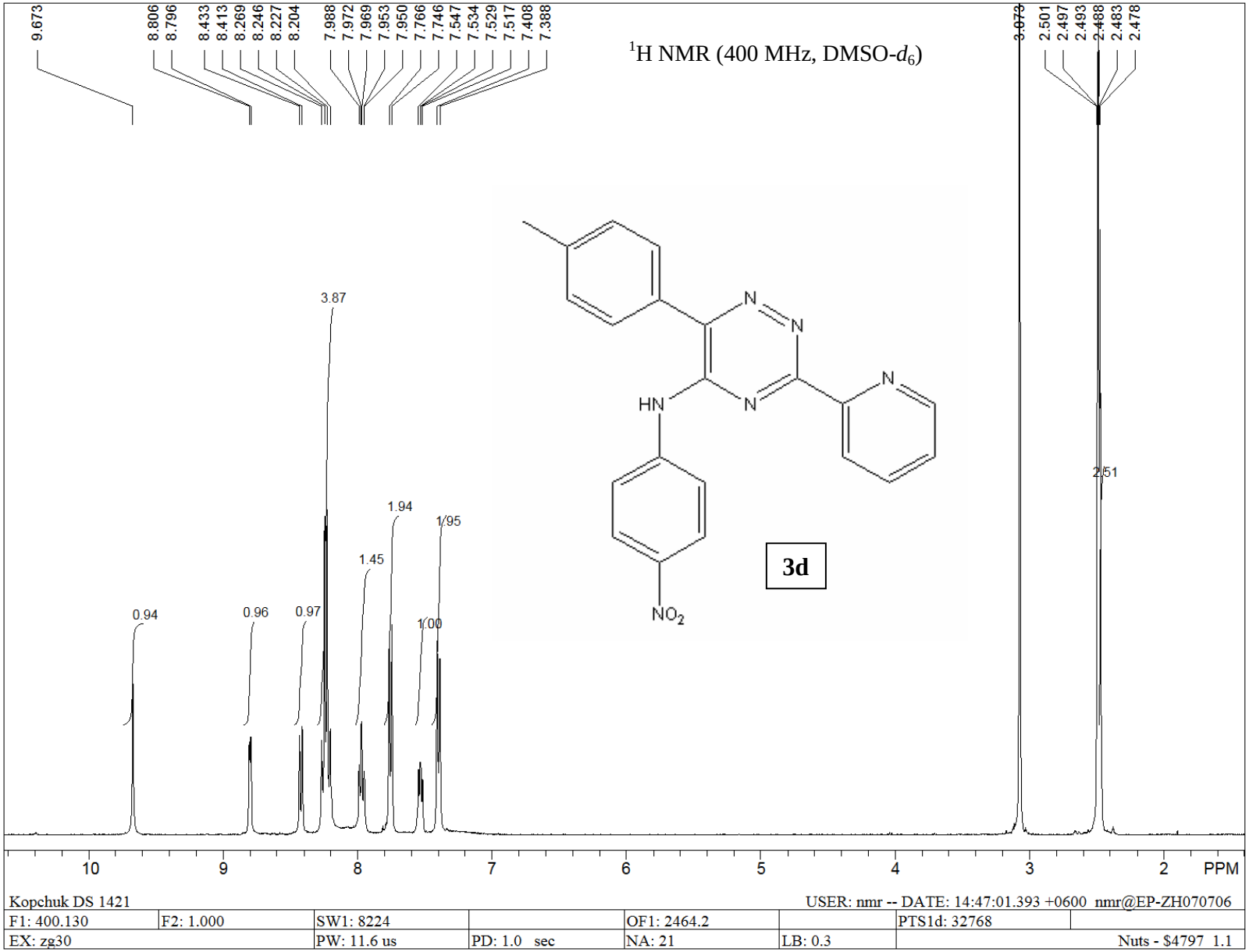

8.954
8.760
8.750
8.326
8.306
7.942
7.938
7.923
7.918
7.903
7.899
7.845
7.825
7.749
7.730
7.499
7.486
7.482
7.470
7.391
7.372
7.352
7.332
7.312
7.113
7.095
3.867
2.496
2.492
2.488
2.471
1H NMR (400 MHz, DMSO-d6)
N
N
HN
N
N
3a
3.87
2.63
1.79
1.78
1.00
0.93
0.93
1.05
0.95
0.95
9
8
7
6
5
4
3
PPM
Kopchuk 1420
USER: nmr -- DATE: 16:35:27.997 +0600 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2463.9
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 14
LB: 0.3
Nuts - $4306_1.1

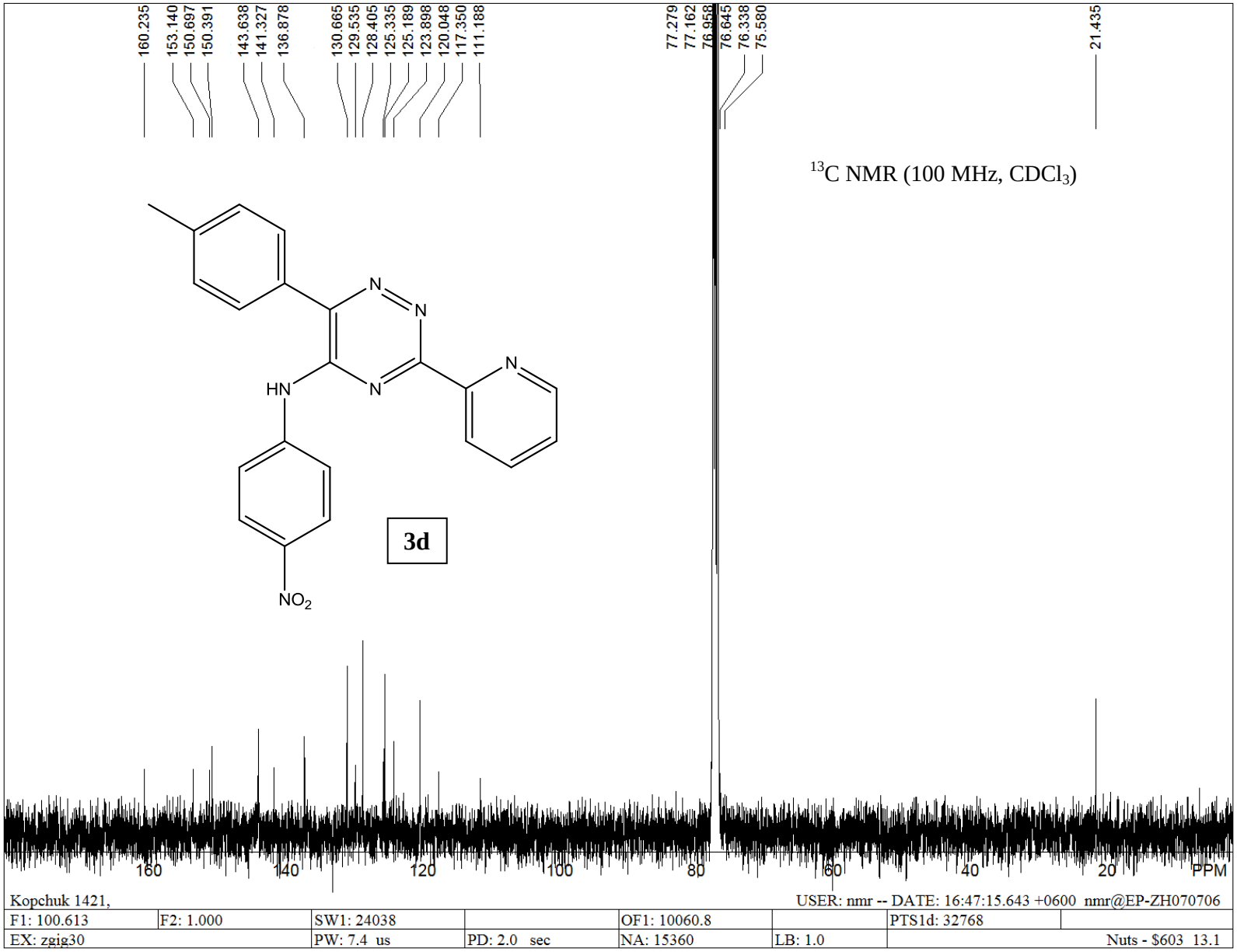

154.864
148.447
145.909
144.874
143.182
135.481
132.273
131.551
125.126
124.893
123.894
123.150
119.767
119.373
118.534
115.749
72.134
72.017
71.813
71.499
16.202
^{13}C NMR (100 MHz, $CDCl_3$)
N
N
N
HN
N
3a
160
140
120
100
80
60
40
20
0
PPM
Kopchuk NV 302,
USER: nmr -- DATE: 09:00:09.580 +0600 nmr@EP-ZH070706
F1: 100.613
F2: 1.000
SW1: 24038
OF1: 10010.5
PTS1d: 32768
EX: zgig30
PW: 7.4 us
PD: 2.0 sec
NA: 3840
LB: 1.0
Nuts - $640 13.1

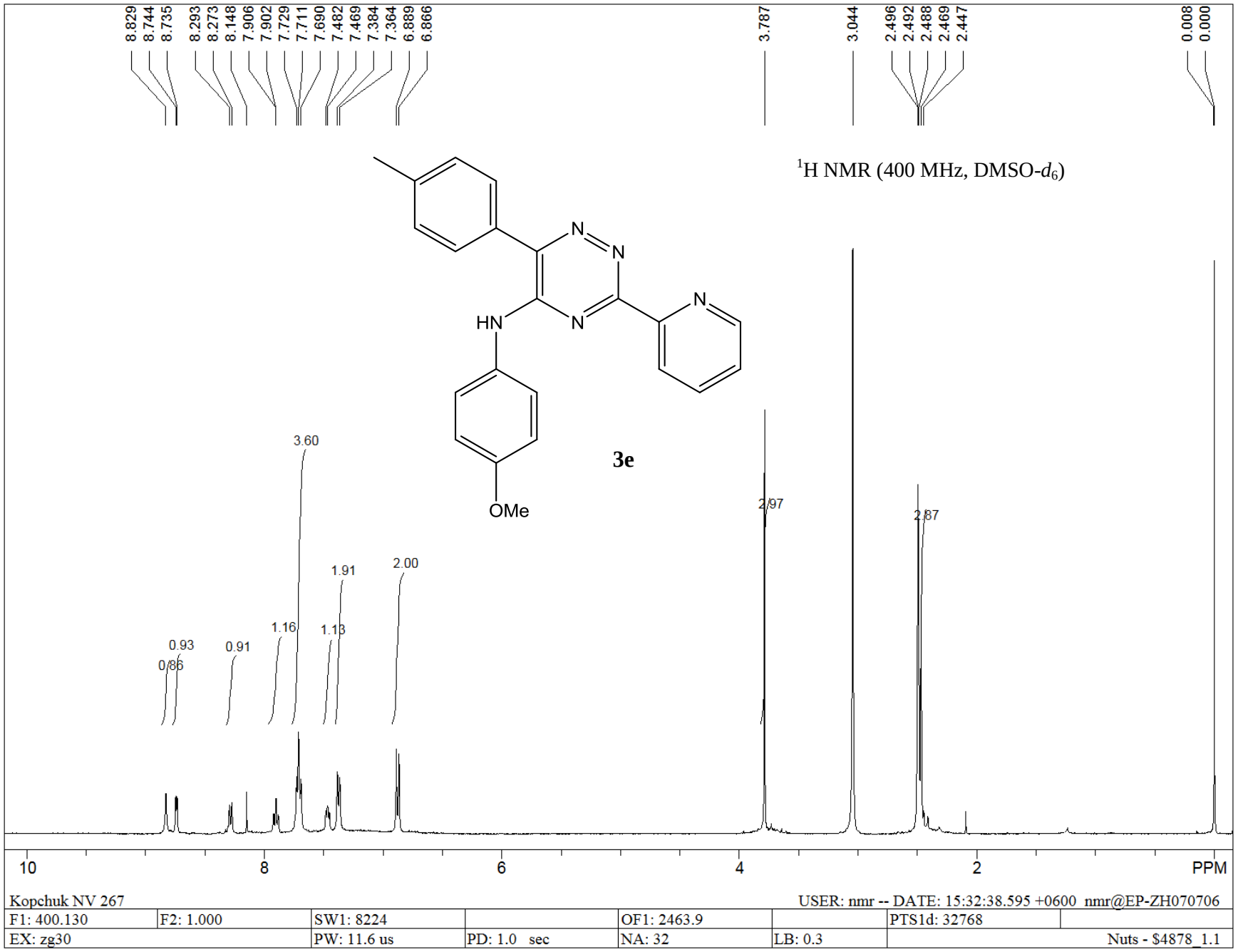

9.028
8.764
8.752
8.335
8.315
7.940
7.935
7.920
7.916
7.901
7.896
7.857
7.842
7.822
7.575
7.558
7.543
7.497
7.485
7.480
7.467
7.350
7.331
7.310
7.112
7.094
7.076
3.027
2.498
2.494
2.490
2.485
2.481
0.000
^{1}H NMR (400 MHz, DMSO-d_6)
HN
N
N
N
3b
5.02
3.16
2.25
1.28
1.09
0.95
1.07
1.00
10
8
6
4
2
0 PPM
Kopchuk 1479
USER: nmr -- DATE: 14:22:44.342 +0500 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2462.9
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 16
LB: 0.3
Nuts - $2718 1.1

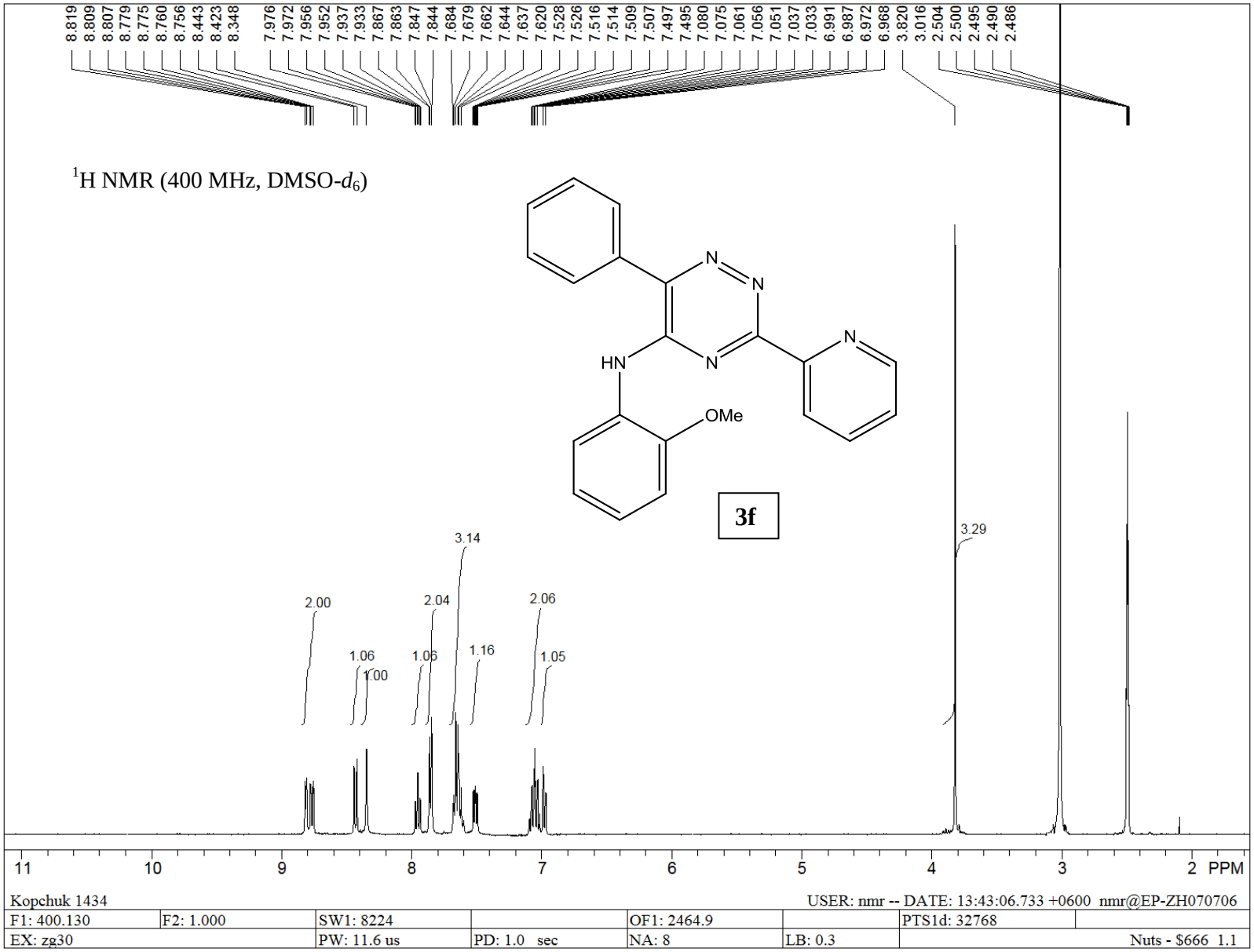

9.331
8.791
8.781
8.394
8.374
8.163
8.078
8.056
7.965
7.953
7.949
7.943
7.935
7.930
7.750
7.730
7.524
7.512
7.507
7.495
7.394
7.374
4.331
4.314
4.295
4.278
3.073
2.501
2.496
2.492
2.488
2.483
2.470
2.410
2.093
1.393
1.376
1.358
[1]H NMR (400 MHz, DMSO-d6)
N
N
N
HN
N
COOEt
3c
3.30
2.20
2.03
2.01
1.11
1.05
1.00
0.91
2.18
2.88
3.30
10
8
6
4
2
PPM
Kopchuk NV250
USER: nmr -- DATE: 14:52:28.025 +0600 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2463.9
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 8
LB: 0.3
Nuts - $4369 1.1

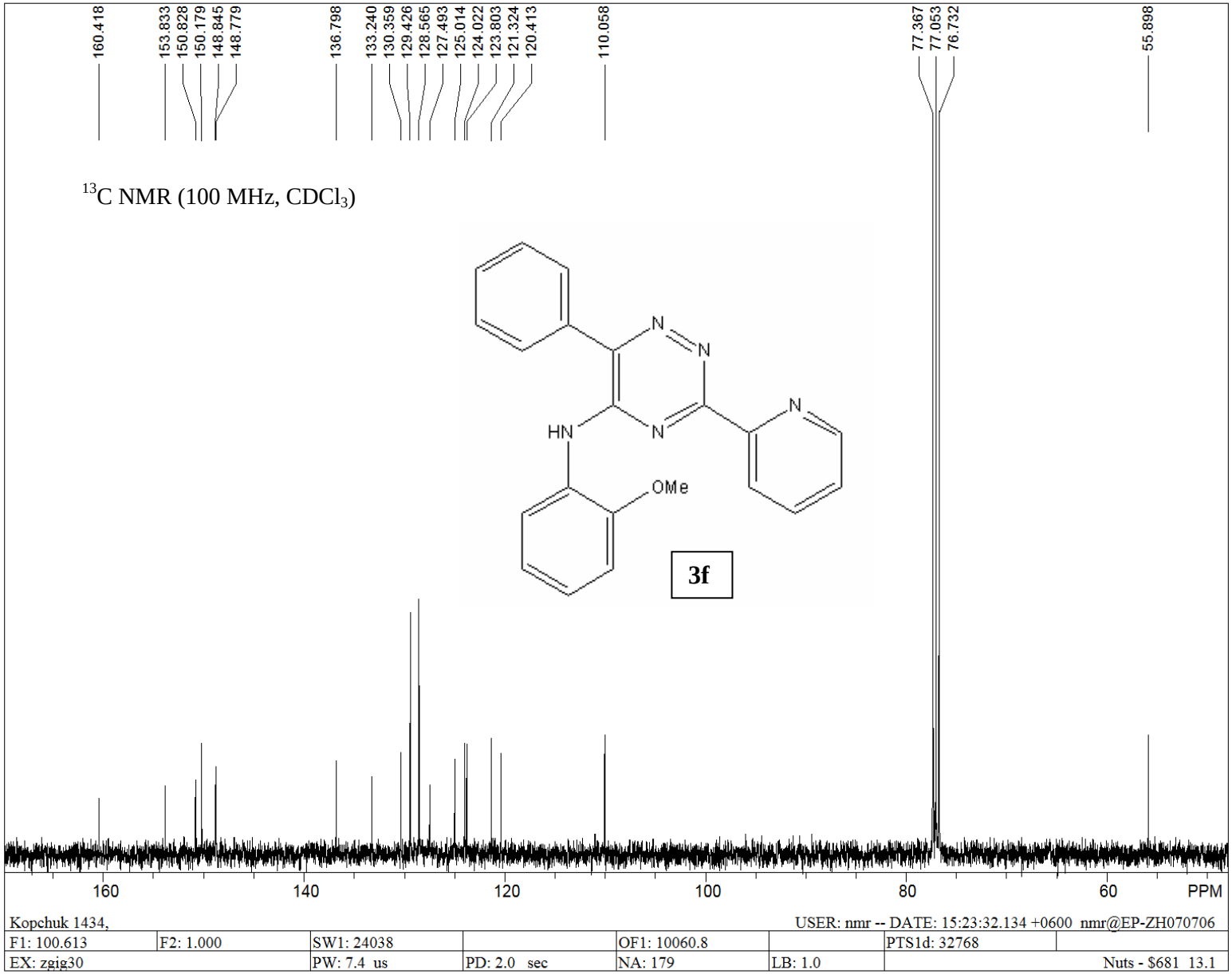

165.989
160.112
153.468
150.894
150.216
148.561
141.801
140.940
136.791
131.519
130.811
130.461
129.885
128.398
126.159
125.116
123.789
119.939
113.755
77.323
77.206
77.002
76.681
60.850
21.399
14.311
^{13}C NMR (100 MHz, CDCl$_3$)
N
N
N
HN
N
COOEt
3c
180
160
140
120
100
80
60
40
20
0PPM
Kopchuk NV 250 (TE=40C),
USER: nmr -- DATE: 04:48:44.682 +0600 nmr@EP-ZH070706
F1: 100.613
F2: 1.000
SW1: 24038
OF1: 10060.8
PTS1d: 32768
EX: zgig30
PW: 7.4 us
PD: 2.0 sec
NA: 3840
LB: 1.0
Nuts - $604 13.1

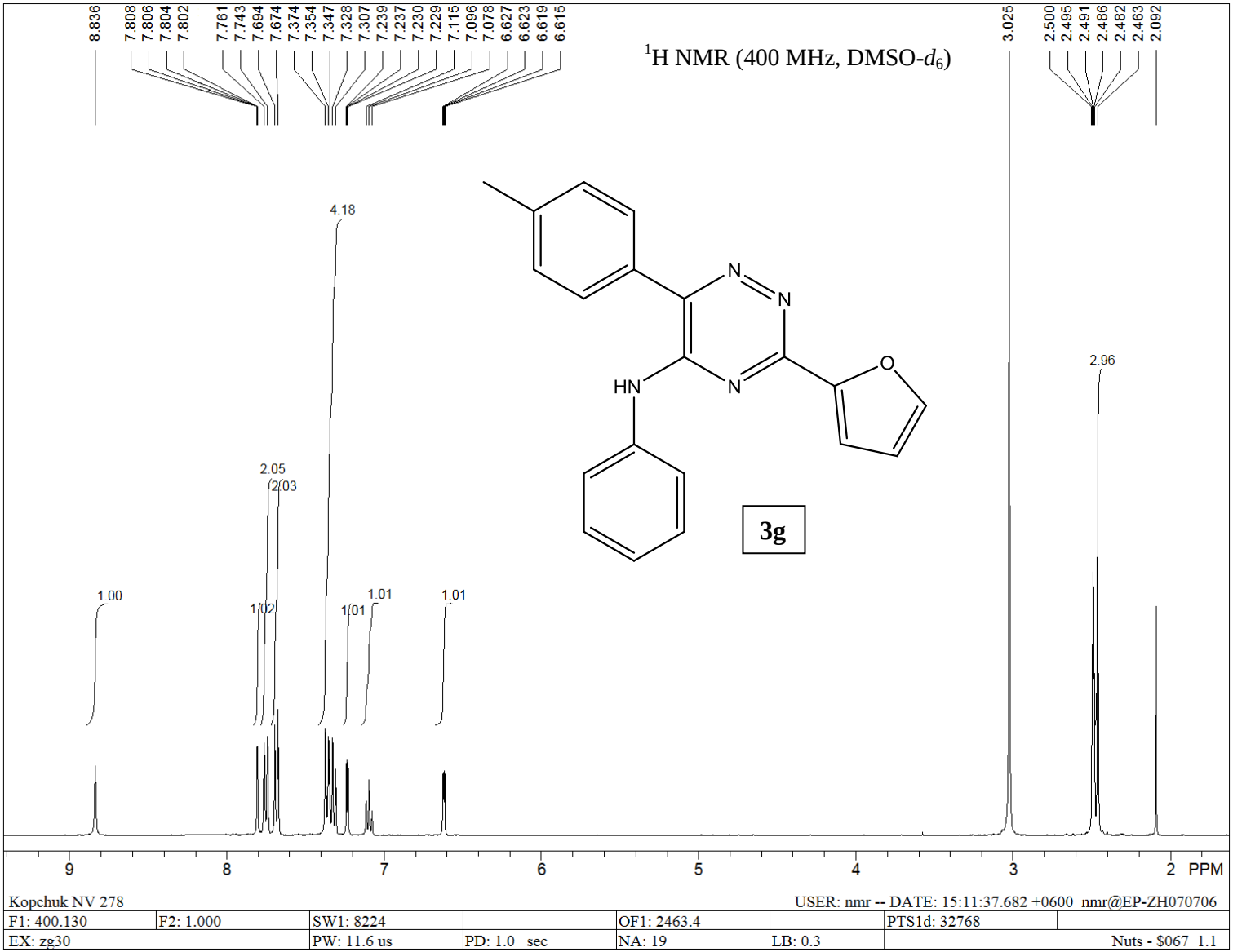

^{1}H NMR (400 MHz, DMSO-d_6)
9.673
8.806
8.796
8.433
8.413
8.269
8.246
8.227
8.204
7.988
7.972
7.969
7.953
7.950
7.766
7.746
7.547
7.534
7.529
7.517
7.408
7.388
3.873
2.501
2.497
2.493
2.488
2.483
2.478
3.87
2.51
1.94
1.95
1.45
0.94
0.96
0.97
1.00
HN
N
N
N
N
NO2
3d
10
9
8
7
6
5
4
3
2
PPM
Kopchuk DS 1421
USER: nmr -- DATE: 14:47:01.393 +0600 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2464.2
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 21
LB: 0.3
Nuts - $4797 1.1

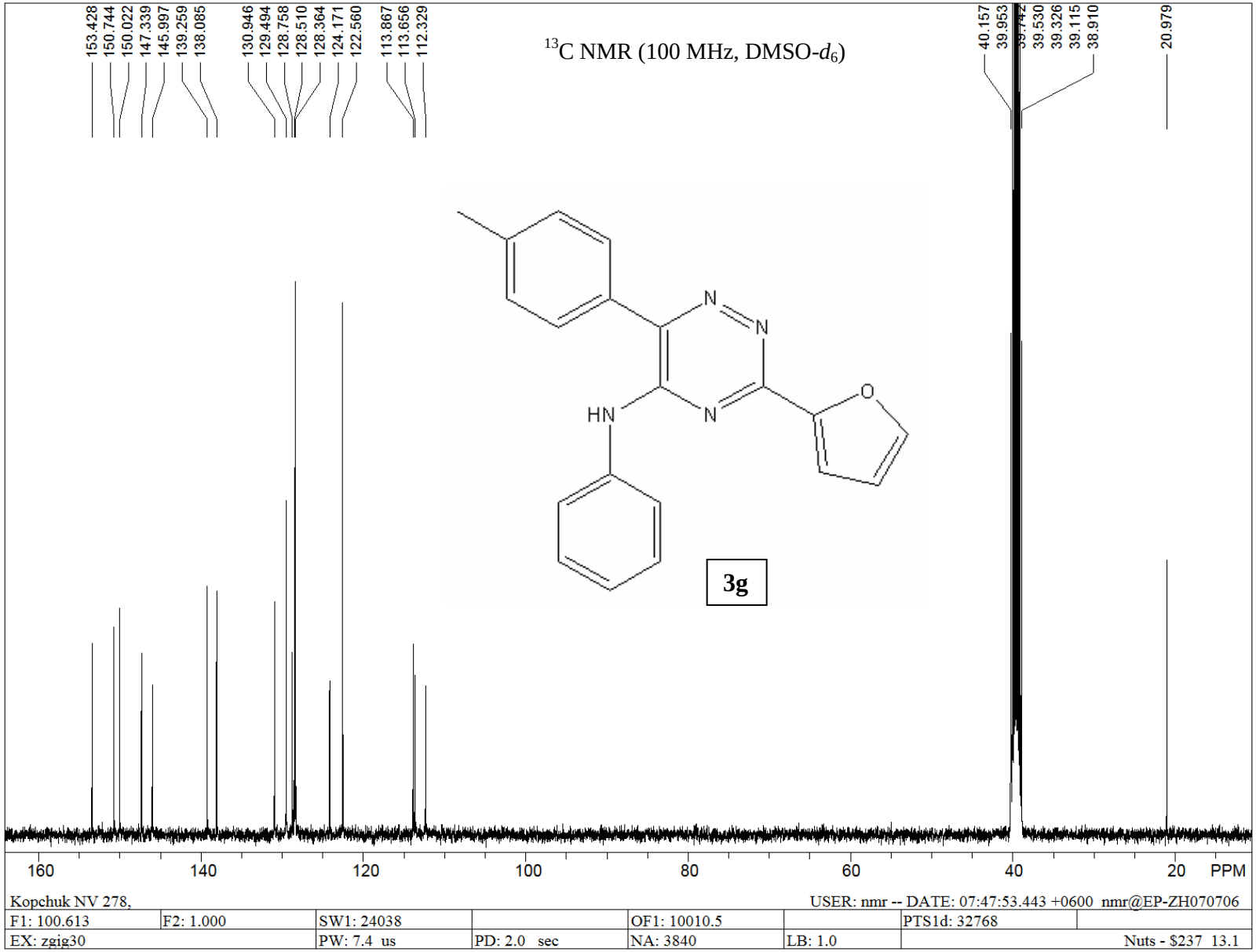

160.235
153.140
150.697
150.391
143.638
141.327
136.878
130.665
129.535
128.405
125.335
125.189
123.898
120.048
117.350
111.188
77.279
77.162
76.958
76.645
76.338
75.580
21.435
^{13}C NMR (100 MHz, CDCl$_3$)
N
N
N
HN
N
NO$_2$
3d
160
140
120
100
80
60
40
20
PPM
Kopchuk 1421,
USER: nmr -- DATE: 16:47:15.643 +0600 nmr@EP-ZH070706
F1: 100.613
F2: 1.000
SW1: 24038
OF1: 10060.8
PTS1d: 32768
EX: zgig30
PW: 7.4 us
PD: 2.0 sec
NA: 15360
LB: 1.0
Nuts - $603 13.1

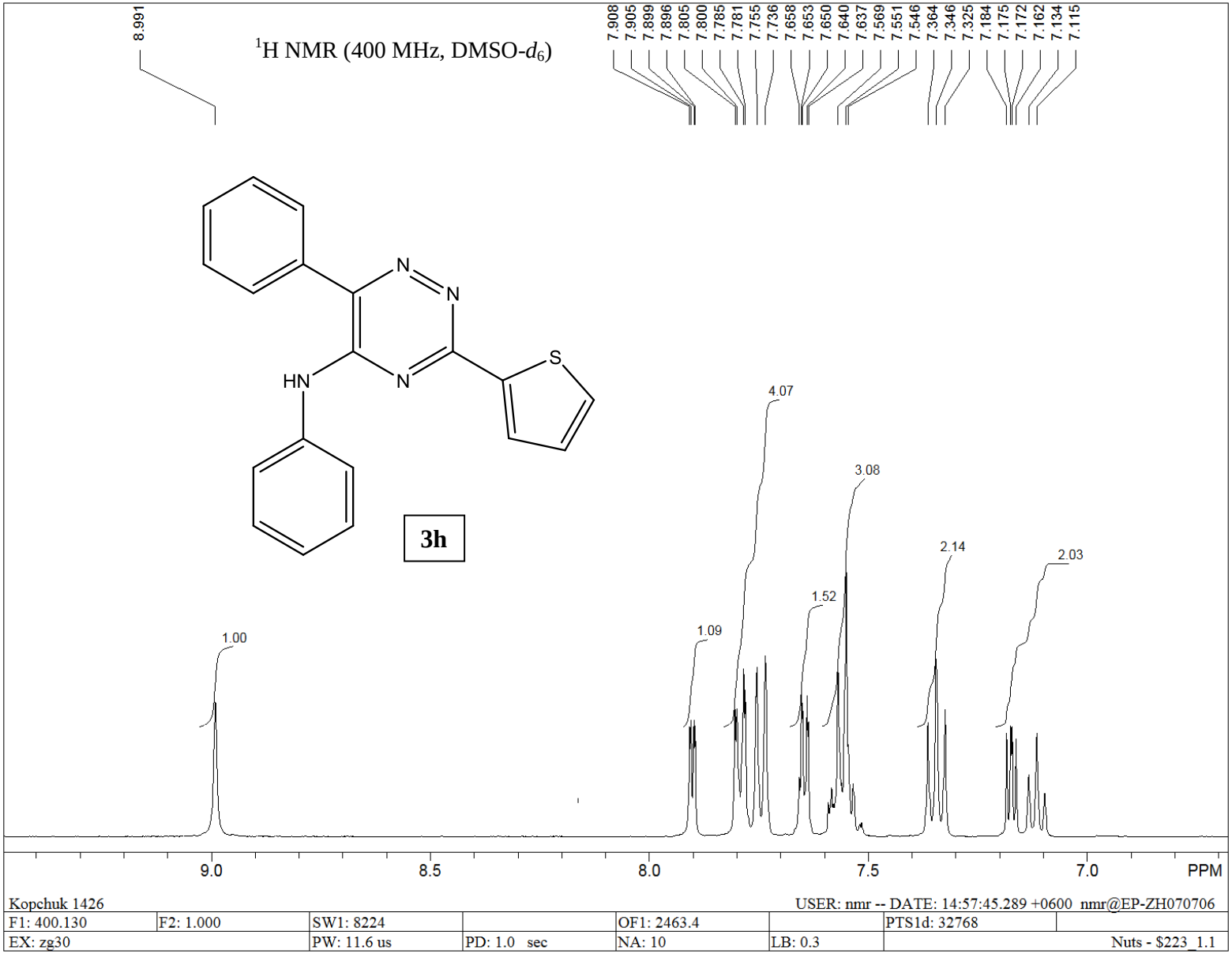

8.829
8.744
8.735
8.293
8.273
8.148
7.906
7.902
7.729
7.711
7.690
7.482
7.469
7.384
7.364
6.889
6.866
3.787
3.044
2.496
2.492
2.488
2.469
2.447
0.008
0.000
^{1}H NMR (400 MHz, DMSO-d_6)
N
N
N
HN
N
OMe
3e
3.60
2.97
2.87
2.00
1.91
1.16
1.13
0.93
0.91
0.86
10
8
6
4
2
PPM
Kopchuk NV 267
USER: nmr -- DATE: 15:32:38.595 +0600 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2463.9
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 32
LB: 0.3
Nuts - $4878_1.1

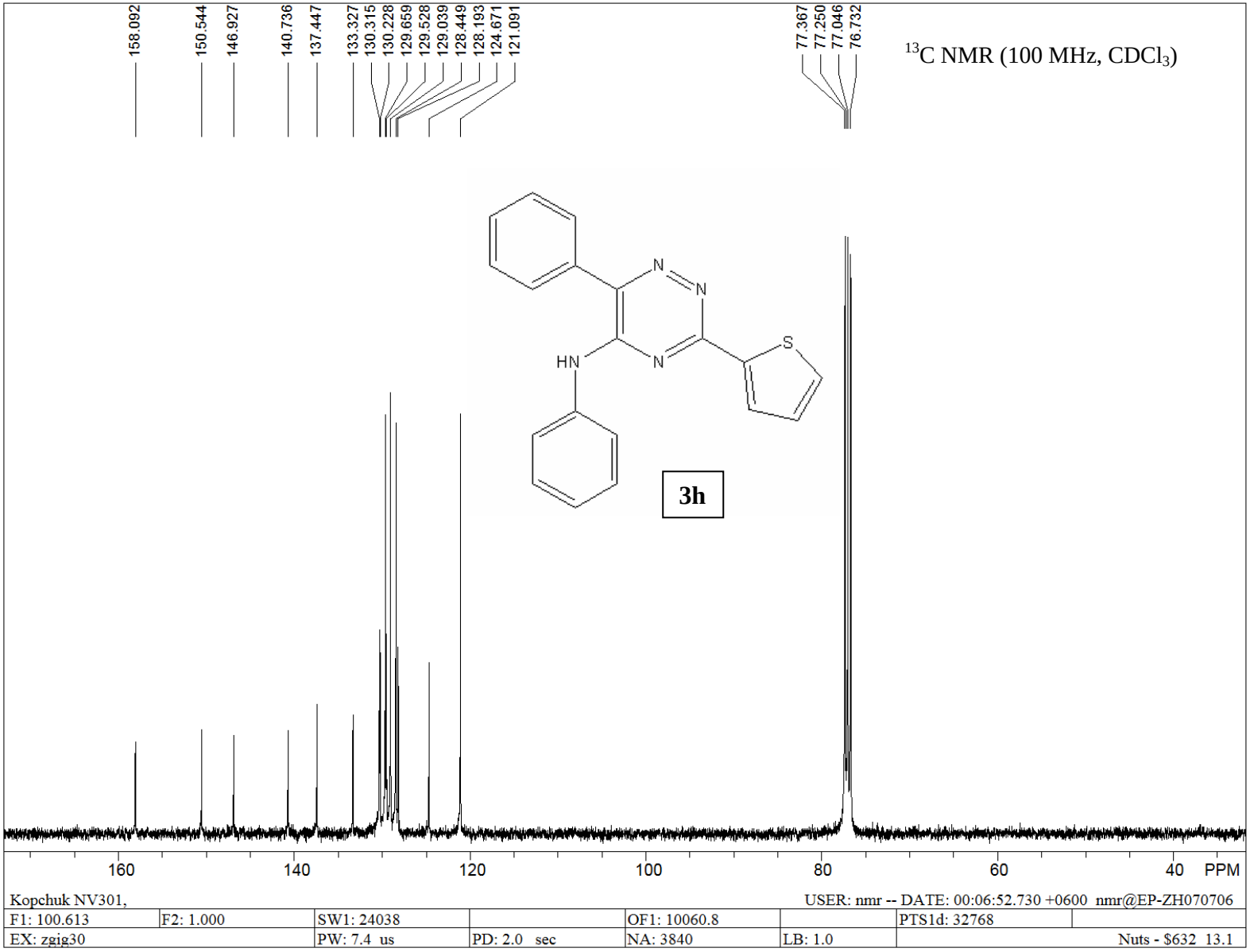

8.819
8.809
8.807
8.779
8.775
8.760
8.756
8.443
8.423
8.348
7.976
7.972
7.956
7.952
7.937
7.933
7.867
7.863
7.847
7.844
7.684
7.679
7.662
7.644
7.637
7.620
7.528
7.526
7.516
7.514
7.509
7.507
7.497
7.495
7.080
7.075
7.061
7.056
7.051
7.037
7.033
6.991
6.987
6.972
6.968
3.820
3.016
2.504
2.500
2.495
2.490
2.486
[1]H NMR (400 MHz, DMSO-d_6)
N
N
N
HN
N
OMe
3f
3.14
3.29
2.00
2.04
2.06
1.06
1.00
1.06
1.16
1.05
11
10
9
8
7
6
5
4
3
2 PPM
Kopchuk 1434
USER: nmr -- DATE: 13:43:06.733 +0600 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2464.9
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 8
LB: 0.3
Nuts - $666 1.1

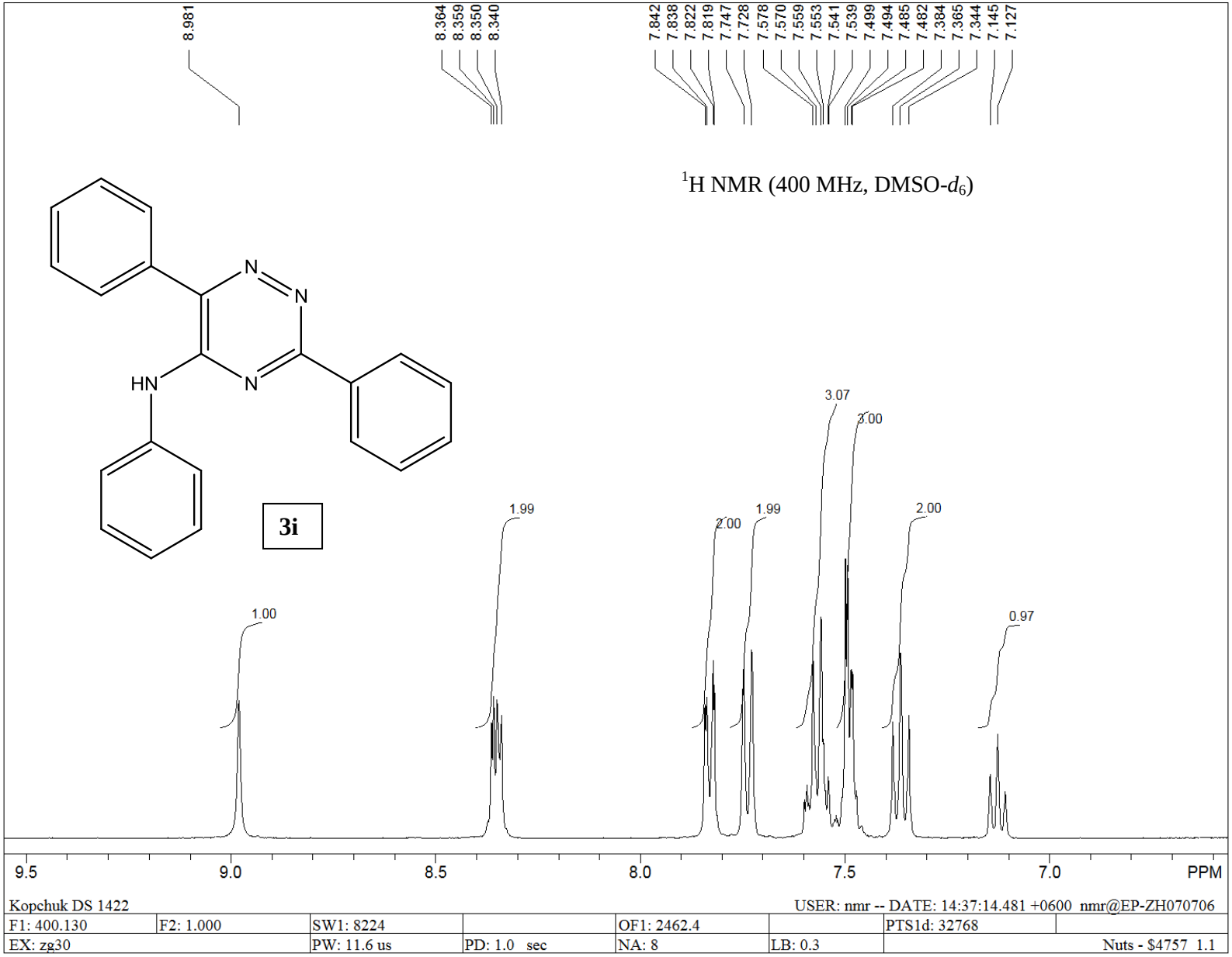

160.418
153.833
150.828
150.179
148.845
148.779
136.798
133.240
130.359
129.426
128.565
127.493
125.014
124.022
123.803
121.324
120.413
110.058
77.367
77.053
76.732
55.898
13C NMR (100 MHz, CDCl3)
N
N
N
HN
N
OMe
3f
160
140
120
100
80
60
PPM
Kopchuk 1434,
USER: nmr -- DATE: 15:23:32.134 +0600 nmr@EP-ZH070706
F1: 100.613
F2: 1.000
SW1: 24038
OF1: 10060.8
PTS1d: 32768
EX: zgig30
PW: 7.4 us
PD: 2.0 sec
NA: 179
LB: 1.0
Nuts - $681 13.1

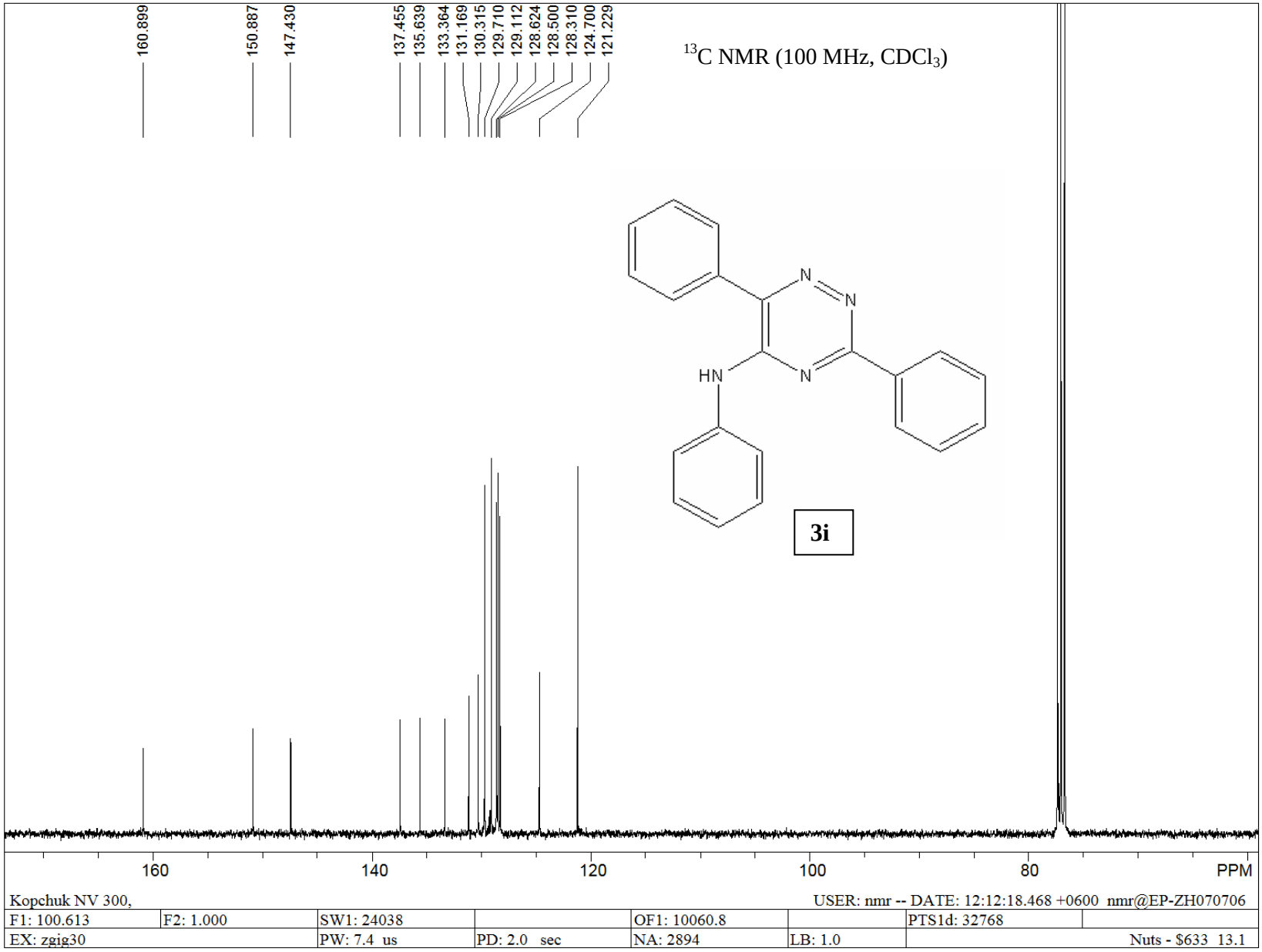

8.836
7.808
7.806
7.804
7.802
7.761
7.743
7.694
7.674
7.374
7.354
7.347
7.328
7.307
7.239
7.237
7.230
7.229
7.115
7.096
7.078
6.627
6.623
6.619
6.615
3.025
2.500
2.495
2.491
2.486
2.482
2.463
2.092
^{1}H NMR (400 MHz, DMSO-d_6)
4.18
2.96
2.05
2.03
1.00
1.02
1.01
1.01
1.01
N
N
N
HN
O
3g
9
8
7
6
5
4
3
2 PPM
Kopchuk NV 278
USER: nmr -- DATE: 15:11:37.682 +0600 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2463.4
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 19
LB: 0.3
Nuts - $067 1.1

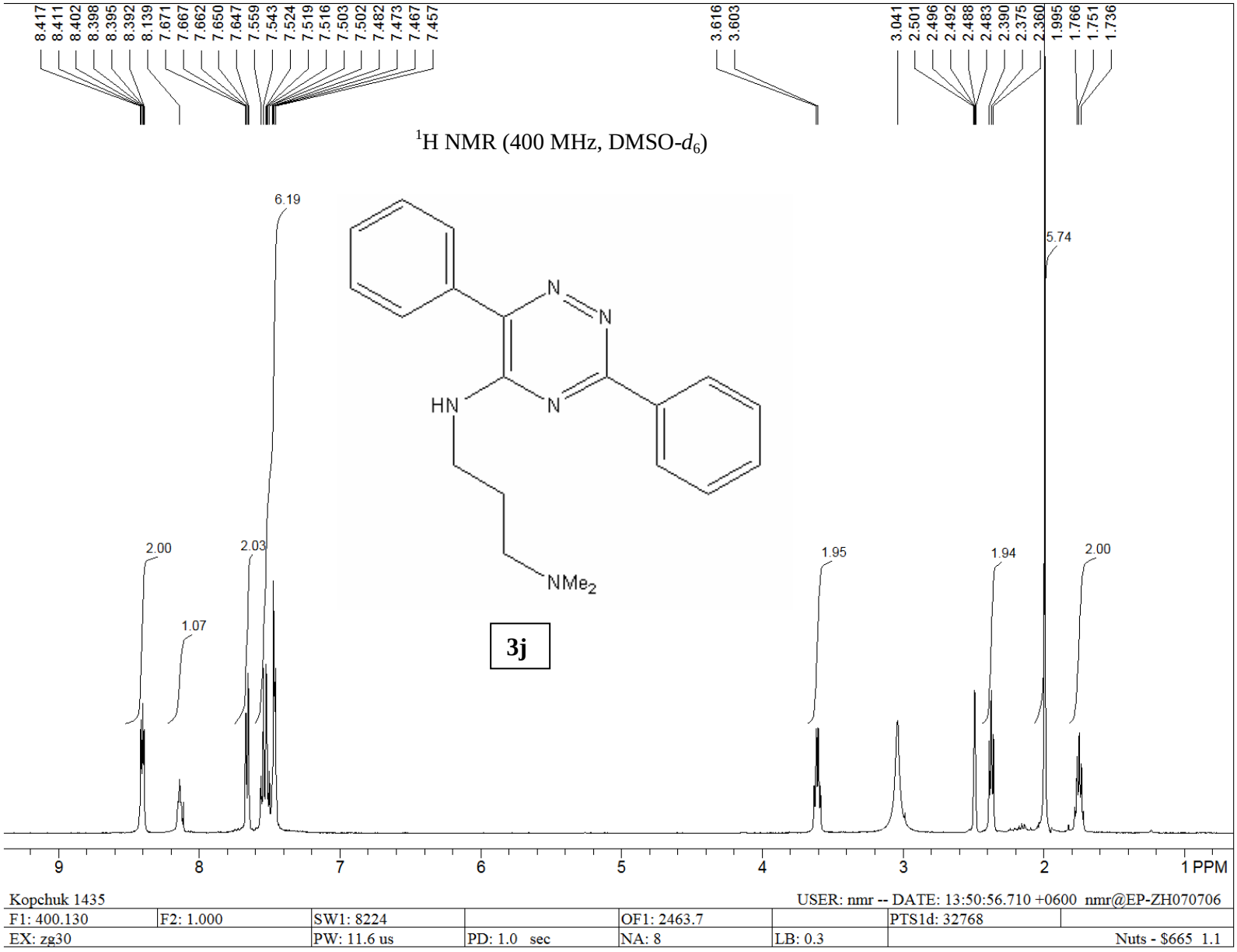

153.428
150.744
150.022
147.339
145.997
139.259
138.085
130.946
129.494
128.758
128.510
128.364
124.171
122.560
113.867
113.656
112.329
40.157
39.953
39.530
39.326
39.115
38.910
20.979
^{13}C NMR (100 MHz, DMSO-d_6)
N
N
HN
N
O
3g
160
140
120
100
80
60
40
20
PPM
Kopchuk NV 278,
USER: nmr -- DATE: 07:47:53.443 +0600 nmr@EP-ZH070706
F1: 100.613
F2: 1.000
SW1: 24038
OF1: 10010.5
PTS1d: 32768
EX: zgig30
PW: 7.4 us
PD: 2.0 sec
NA: 3840
LB: 1.0
Nuts - $237 13.1

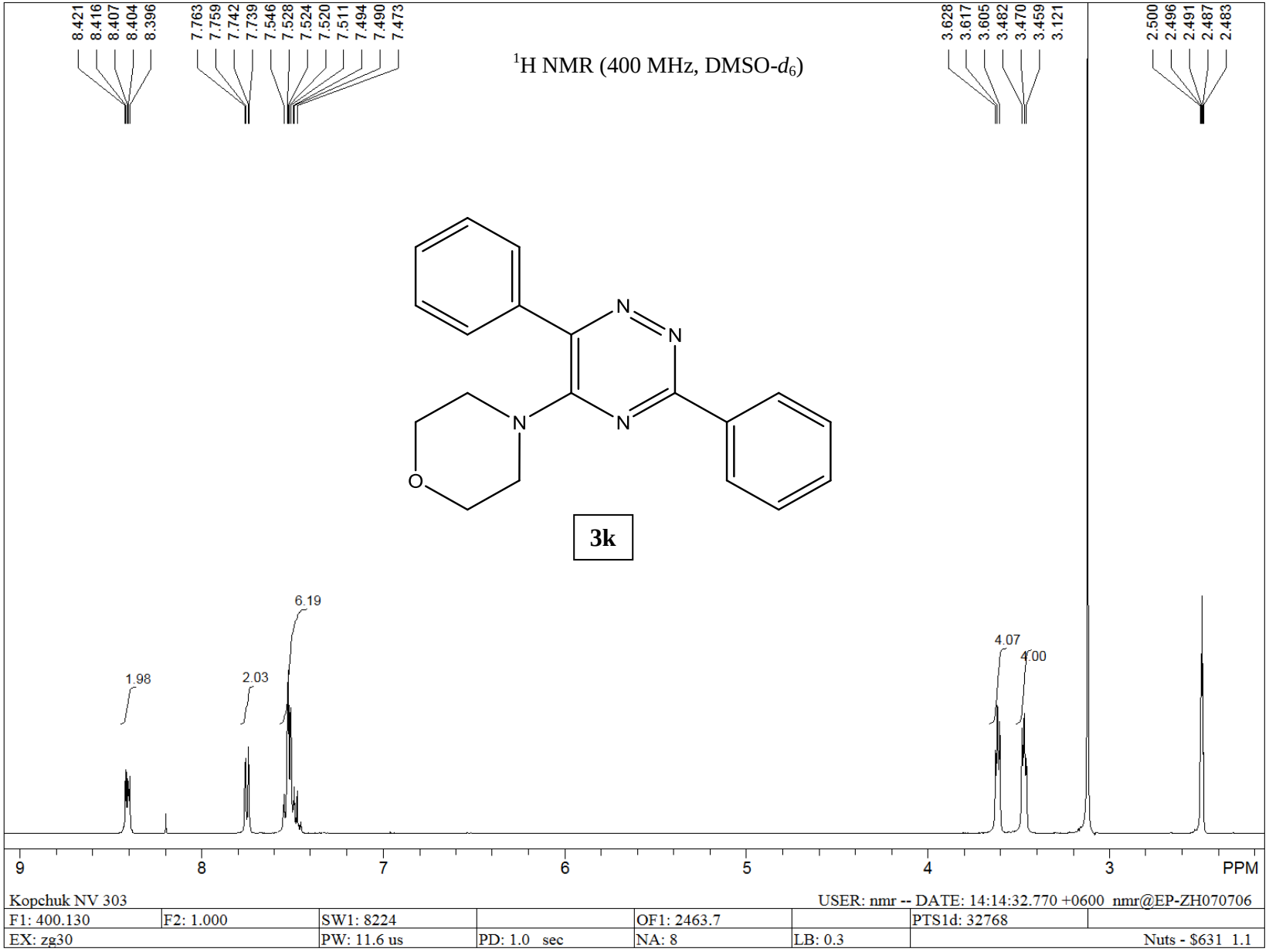

^{1}H NMR (400 MHz, DMSO-d_6)
8.991
7.908
7.905
7.899
7.896
7.805
7.800
7.785
7.781
7.755
7.736
7.658
7.653
7.650
7.640
7.637
7.569
7.551
7.546
7.364
7.346
7.325
7.184
7.175
7.172
7.162
7.134
7.115
N
N
N
HN
S
3h
1.00
1.09
4.07
1.52
3.08
2.14
2.03
9.0
8.5
8.0
7.5
7.0
PPM
Kopchuk 1426
USER: nmr -- DATE: 14:57:45.289 +0600 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2463.4
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 10
LB: 0.3
Nuts - $223_1.1

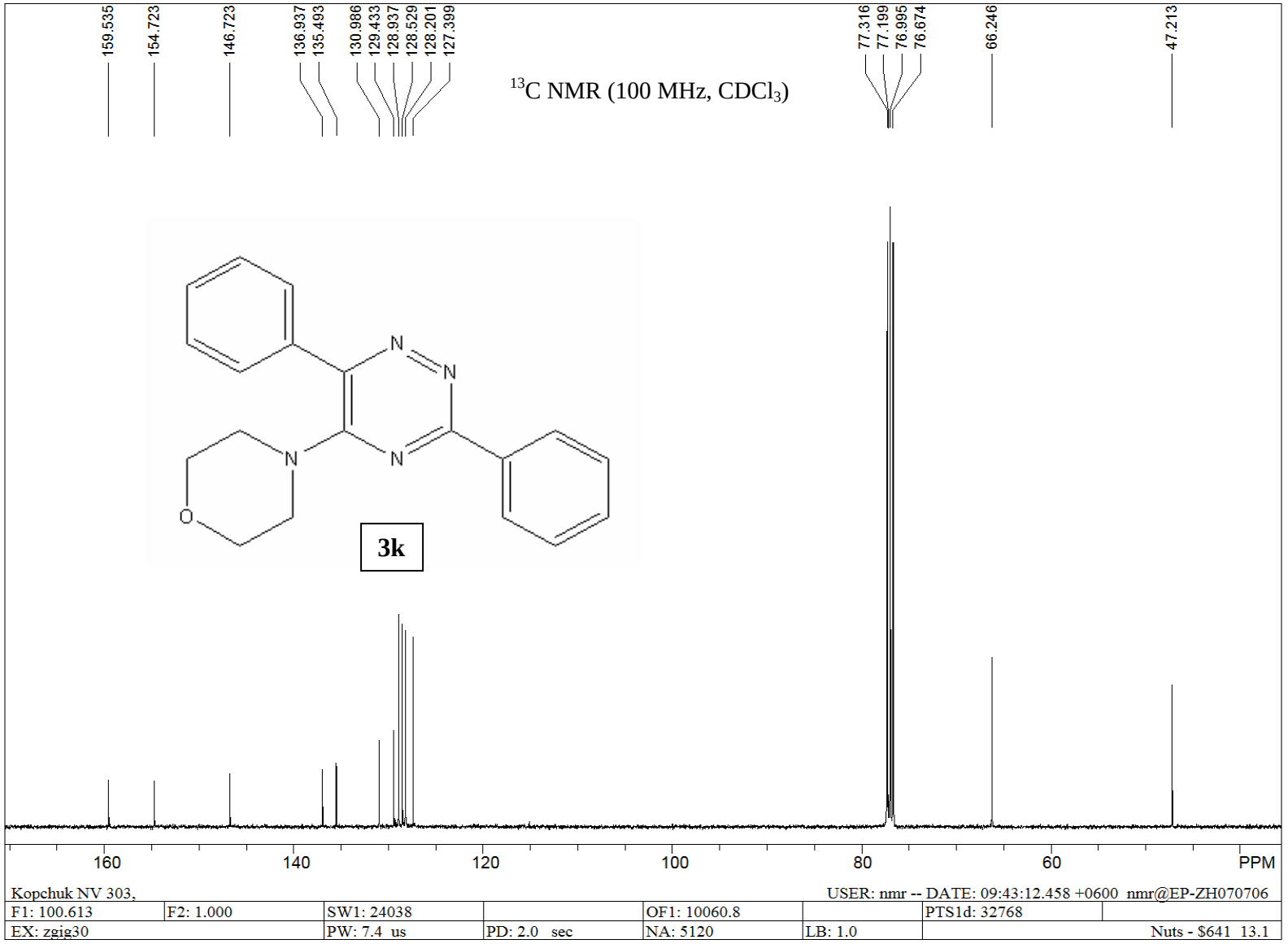

158.092
150.544
146.927
140.736
137.447
133.327
130.315
130.228
129.659
129.528
129.039
128.449
128.193
124.671
121.091
77.367
77.250
77.046
76.732
^{13}C NMR (100 MHz, $CDCl_3$)
N
N
N
HN
S
3h
160
140
120
100
80
60
40
PPM
Kopchuk NV301,
USER: nmr -- DATE: 00:06:52.730 +0600 nmr@EP-ZH070706
F1: 100.613
F2: 1.000
SW1: 24038
OF1: 10060.8
PTS1d: 32768
EX: zgig30
PW: 7.4 us
PD: 2.0 sec
NA: 3840
LB: 1.0
Nuts - $632 13.1

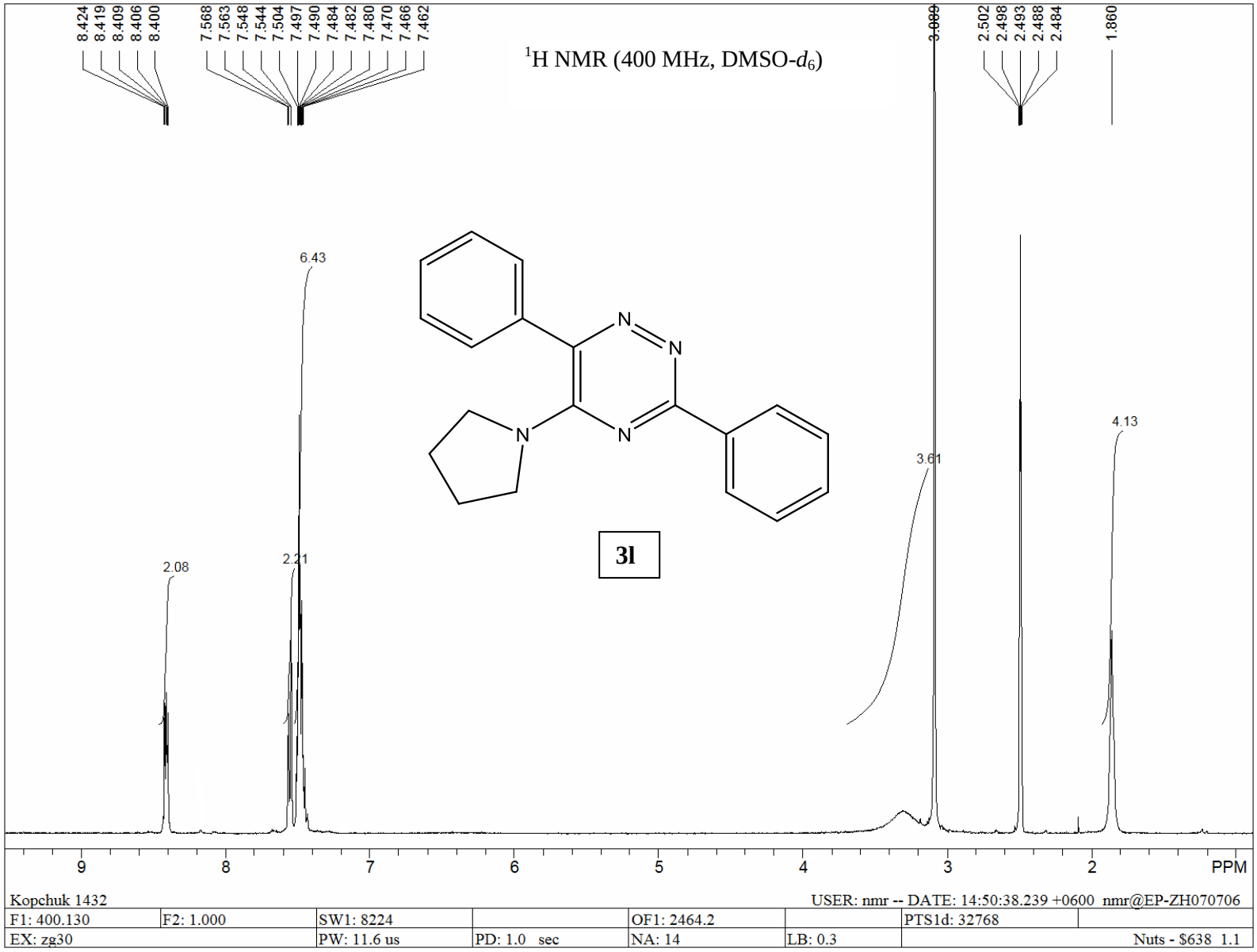

8.981
8.364
8.359
8.350
8.340
7.842
7.838
7.822
7.819
7.747
7.728
7.578
7.570
7.559
7.553
7.541
7.539
7.499
7.494
7.485
7.482
7.384
7.365
7.344
7.145
7.127
^{1}H NMR (400 MHz, DMSO-d_6)
N
N
N
HN
3i
1.00
1.99
2.00
1.99
3.07
3.00
2.00
0.97
9.5
9.0
8.5
8.0
7.5
7.0
PPM
Kopchuk DS 1422
USER: nmr -- DATE: 14:37:14.481 +0600 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2462.4
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 8
LB: 0.3
Nuts - $4757 1.1

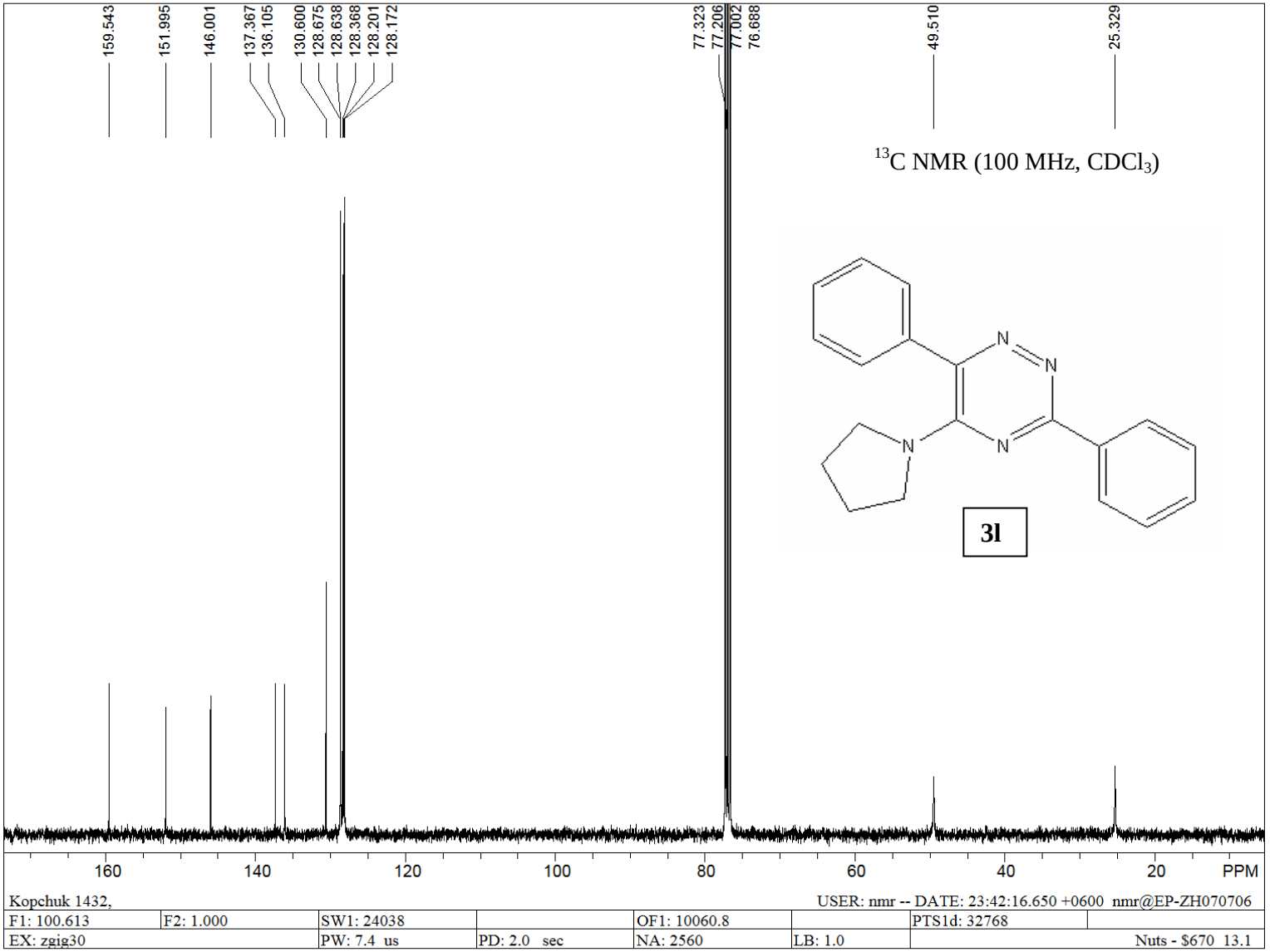

160.899
150.887
147.430
137.455
135.639
133.364
131.169
130.315
129.710
129.112
128.624
128.500
128.310
124.700
121.229
^{13}C NMR (100 MHz, CDCl$_3$)
N
N
HN
N
3i
160
140
120
100
80
PPM
Kopchuk NV 300,
USER: nmr -- DATE: 12:12:18.468 +0600 nmr@EP-ZH070706
F1: 100.613
F2: 1.000
SW1: 24038
OF1: 10060.8
PTS1d: 32768
EX: zgig30
PW: 7.4 us
PD: 2.0 sec
NA: 2894
LB: 1.0
Nuts - $633 13.1

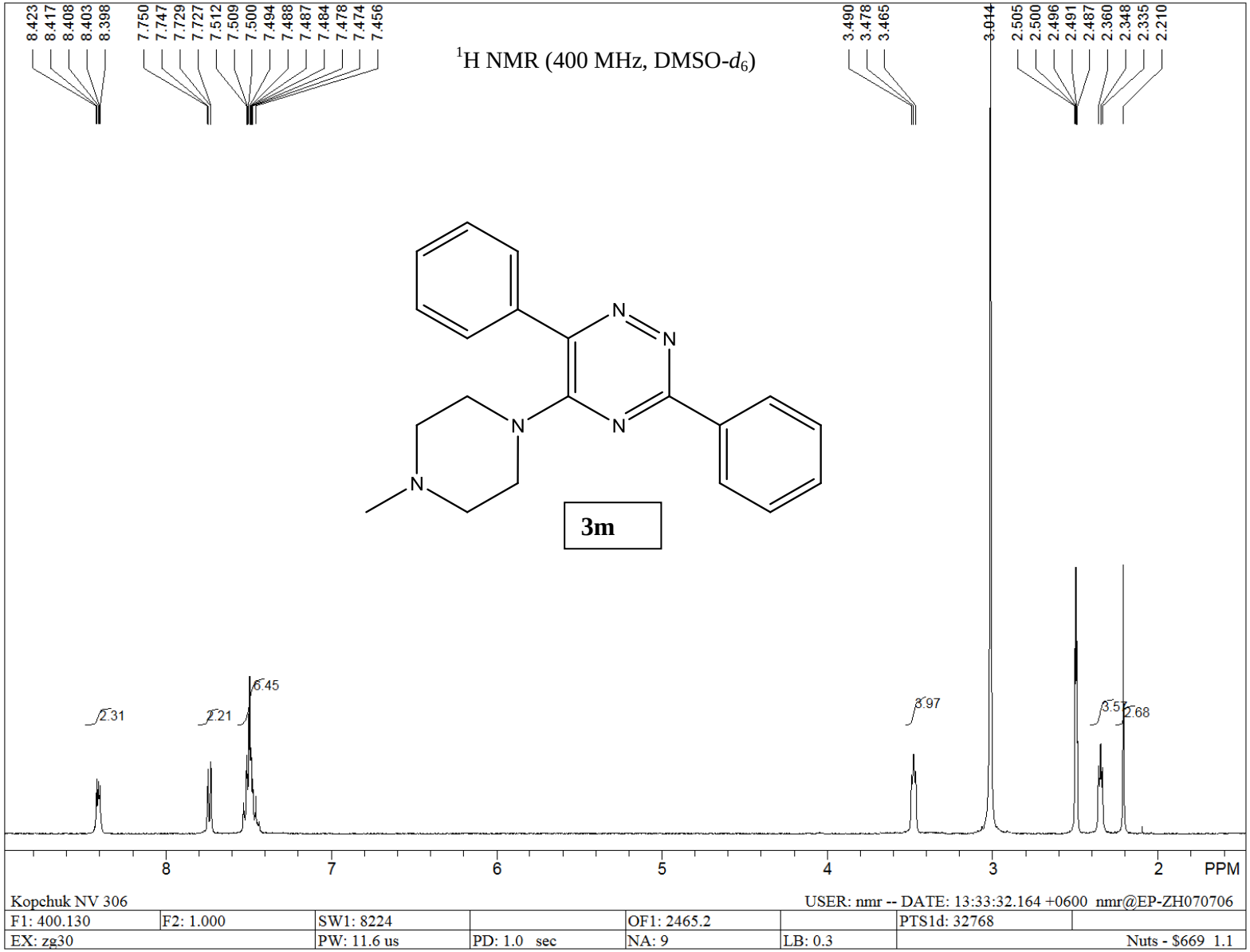

8.417
8.411
8.402
8.398
8.395
8.392
8.139
7.671
7.667
7.662
7.650
7.647
7.559
7.543
7.524
7.519
7.516
7.503
7.502
7.482
7.473
7.467
7.457
3.616
3.603
3.041
2.501
2.496
2.492
2.488
2.483
2.390
2.375
2.360
1.995
1.766
1.751
1.736
^{1}H NMR (400 MHz, DMSO-d_6)
6.19
5.74
2.00
2.03
1.07
1.95
1.94
2.00
N
N
N
HN
NMe$_2$
3j
9
8
7
6
5
4
3
2
1 PPM
Kopchuk 1435
USER: nmr -- DATE: 13:50:56.710 +0600 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2463.7
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 8
LB: 0.3
Nuts - $665 1.1

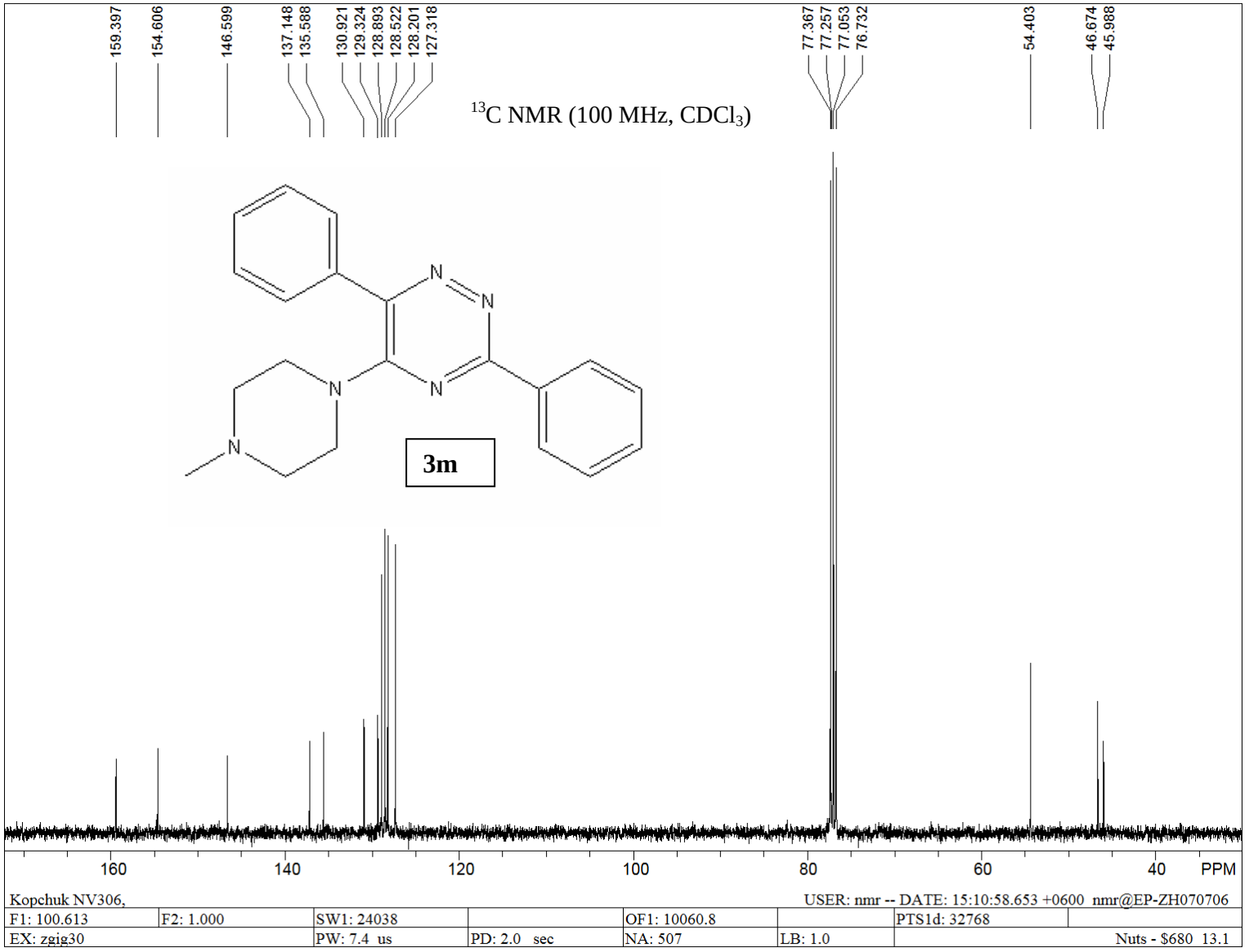

8.421
8.416
8.407
8.404
8.396
7.763
7.759
7.742
7.739
7.546
7.528
7.524
7.520
7.511
7.494
7.490
7.473
3.628
3.617
3.605
3.482
3.470
3.459
3.121
2.500
2.496
2.491
2.487
2.483
^{1}H NMR (400 MHz, DMSO-d_6)
N
N
N
N
O
3k
1.98
2.03
6.19
4.07
4.00
9
8
7
6
5
4
3
PPM
Kopchuk NV 303
USER: nmr -- DATE: 14:14:32.770 +0600 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2463.7
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 8
LB: 0.3
Nuts - $631 1.1

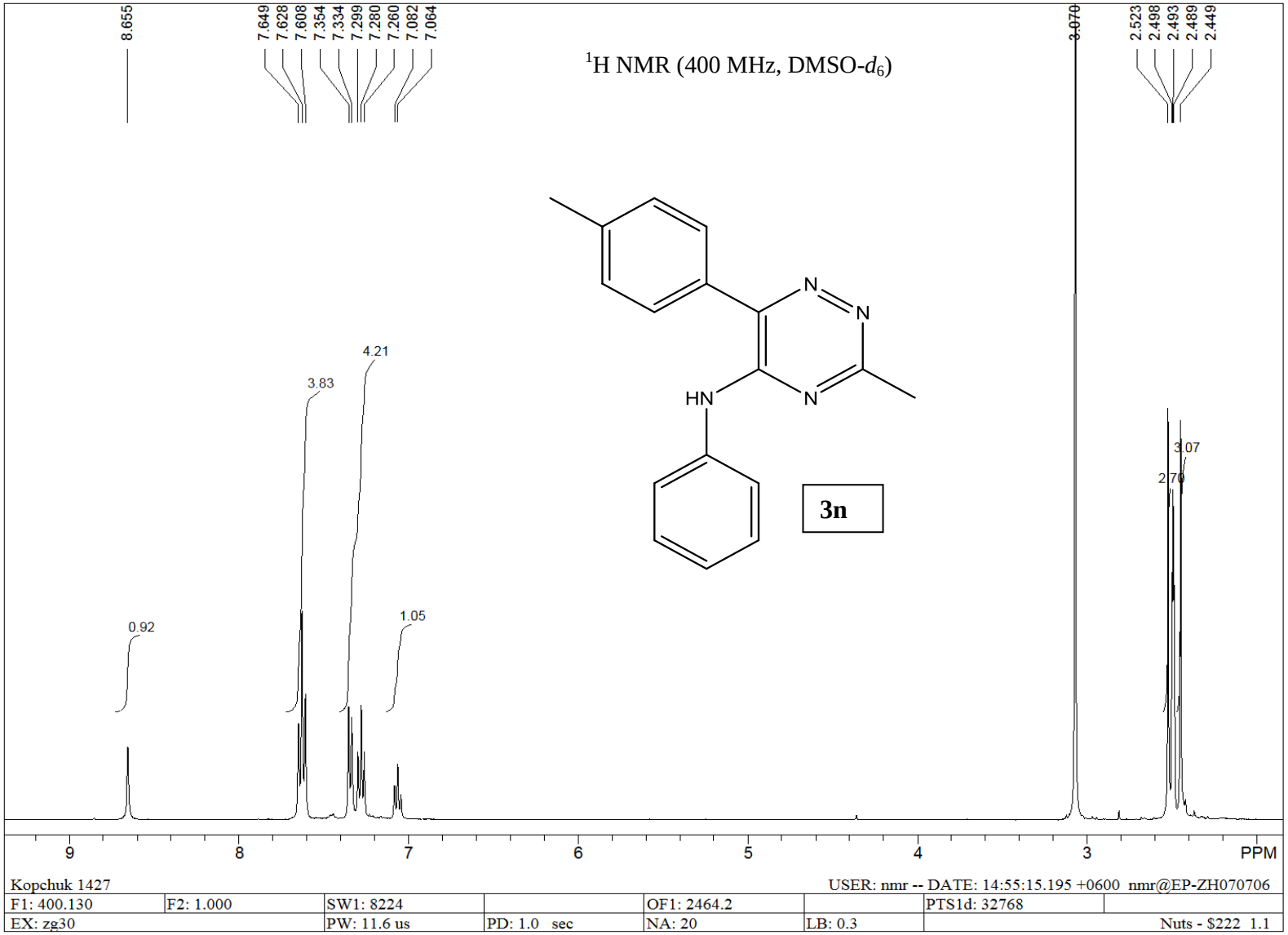

159.535
154.723
146.723
136.937
135.493
130.986
129.433
128.937
128.529
128.201
127.399
77.316
77.199
76.995
76.674
66.246
47.213
^{13}C NMR (100 MHz, $CDCl_3$)
N
N
N
O
3k
160
140
120
100
80
60
PPM
Kopchuk NV 303,
USER: nmr -- DATE: 09:43:12.458 +0600 nmr@EP-ZH070706
F1: 100.613
F2: 1.000
SW1: 24038
OF1: 10060.8
PTS1d: 32768
EX: zgig30
PW: 7.4 us
PD: 2.0 sec
NA: 5120
LB: 1.0
Nuts - $641 13.1

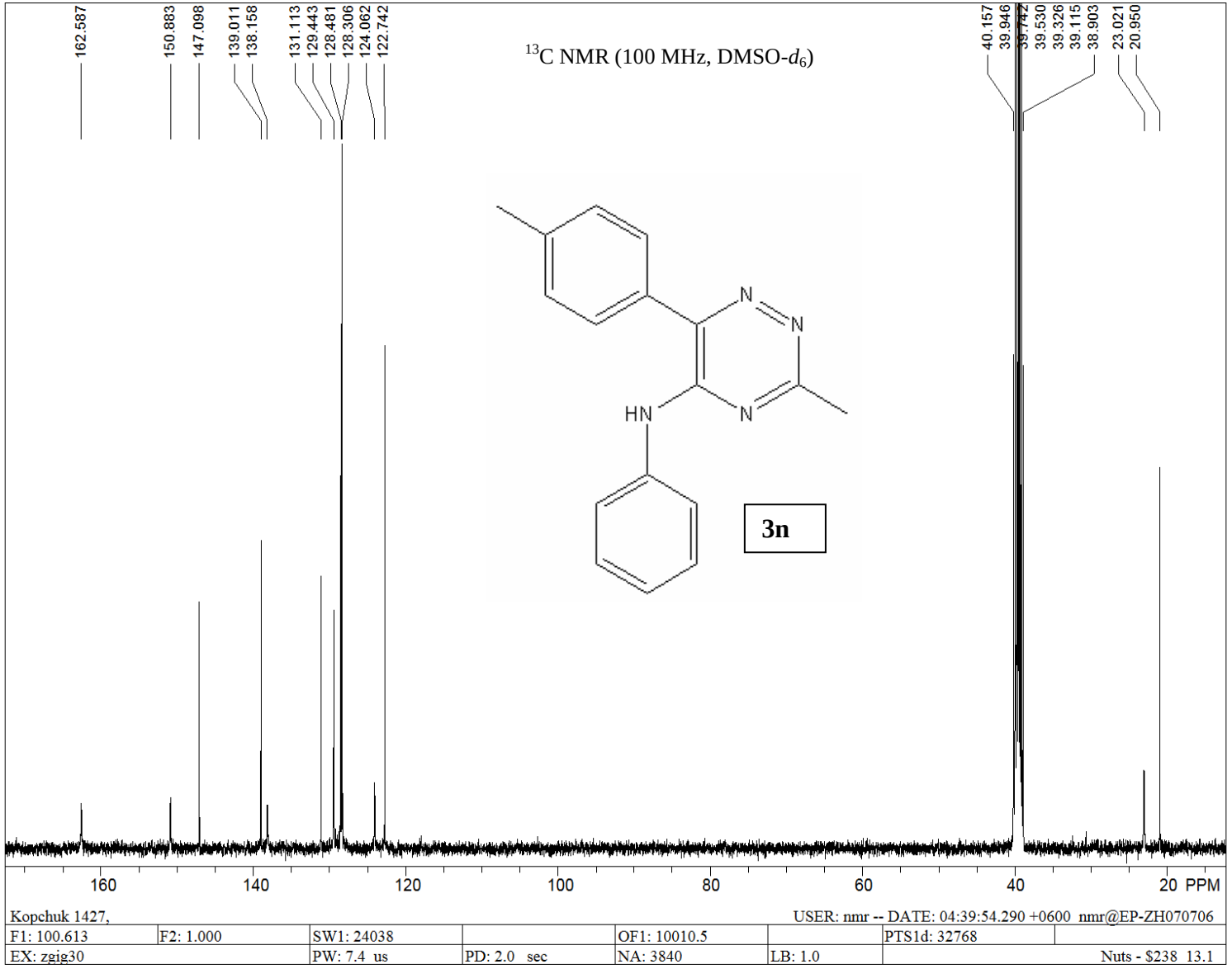

8.424
8.419
8.409
8.406
8.400
7.568
7.563
7.548
7.544
7.504
7.497
7.490
7.484
7.482
7.480
7.470
7.466
7.462
3.089
2.502
2.498
2.493
2.488
2.484
1.860
^{1}H NMR (400 MHz, DMSO-d_6)
6.43
2.08
2.21
3.61
4.13
N
N
N
N
3l
9
8
7
6
5
4
3
2
PPM
Kopchuk 1432
USER: nmr -- DATE: 14:50:38.239 +0600 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2464.2
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 14
LB: 0.3
Nuts - $638 1.1

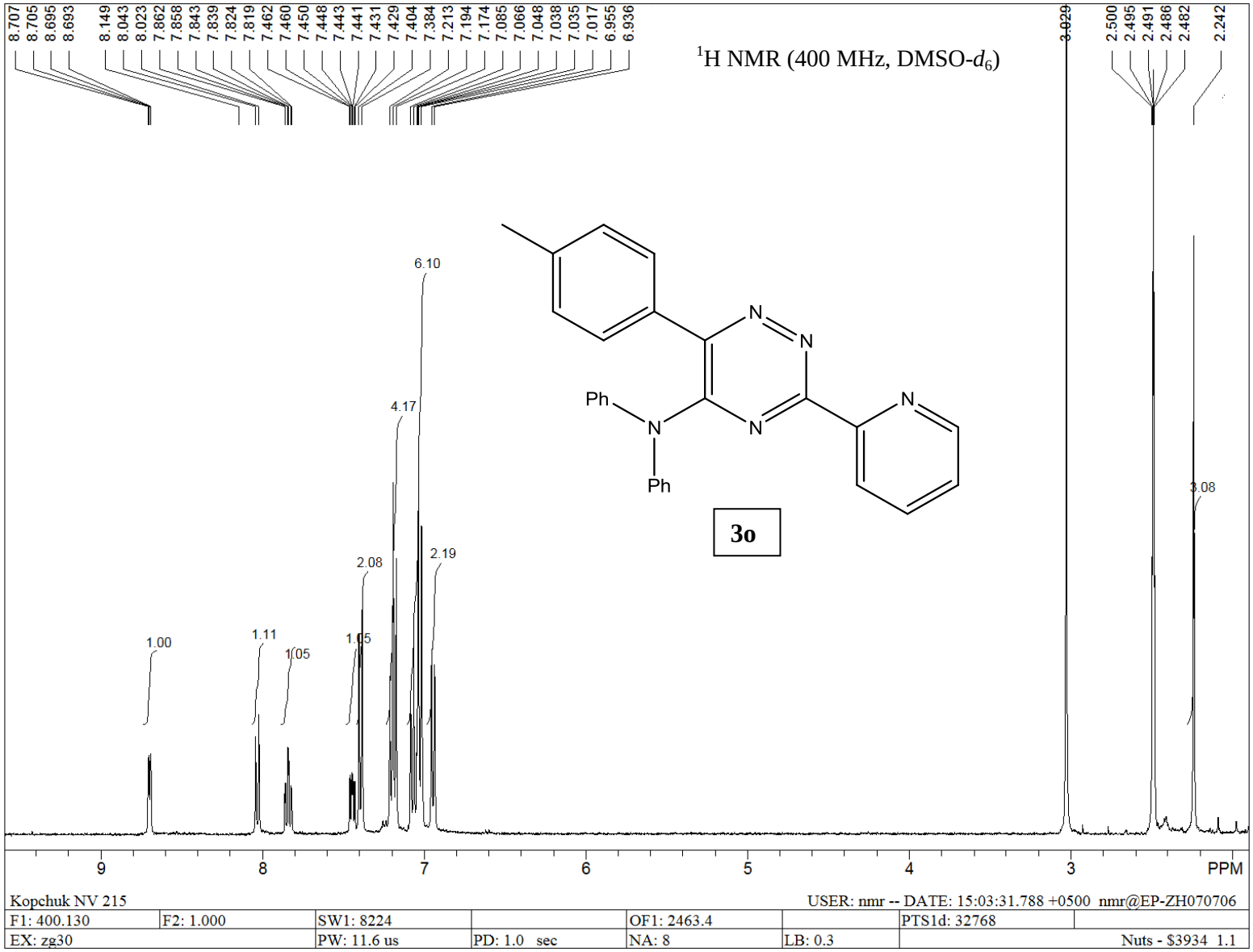

159.543
151.995
146.001
137.367
136.105
130.600
128.675
128.638
128.368
128.201
128.172
77.323
77.206
77.002
76.688
49.510
25.329
^{13}C NMR (100 MHz, $CDCl_3$)
N
N
N
N
3l
160
140
120
100
80
60
40
20
PPM
Kopchuk 1432,
USER: nmr -- DATE: 23:42:16.650 +0600 nmr@EP-ZH070706
F1: 100.613
F2: 1.000
SW1: 24038
OF1: 10060.8
PTS1d: 32768
EX: zgig30
PW: 7.4 us
PD: 2.0 sec
NA: 2560
LB: 1.0
Nuts - $670 13.1

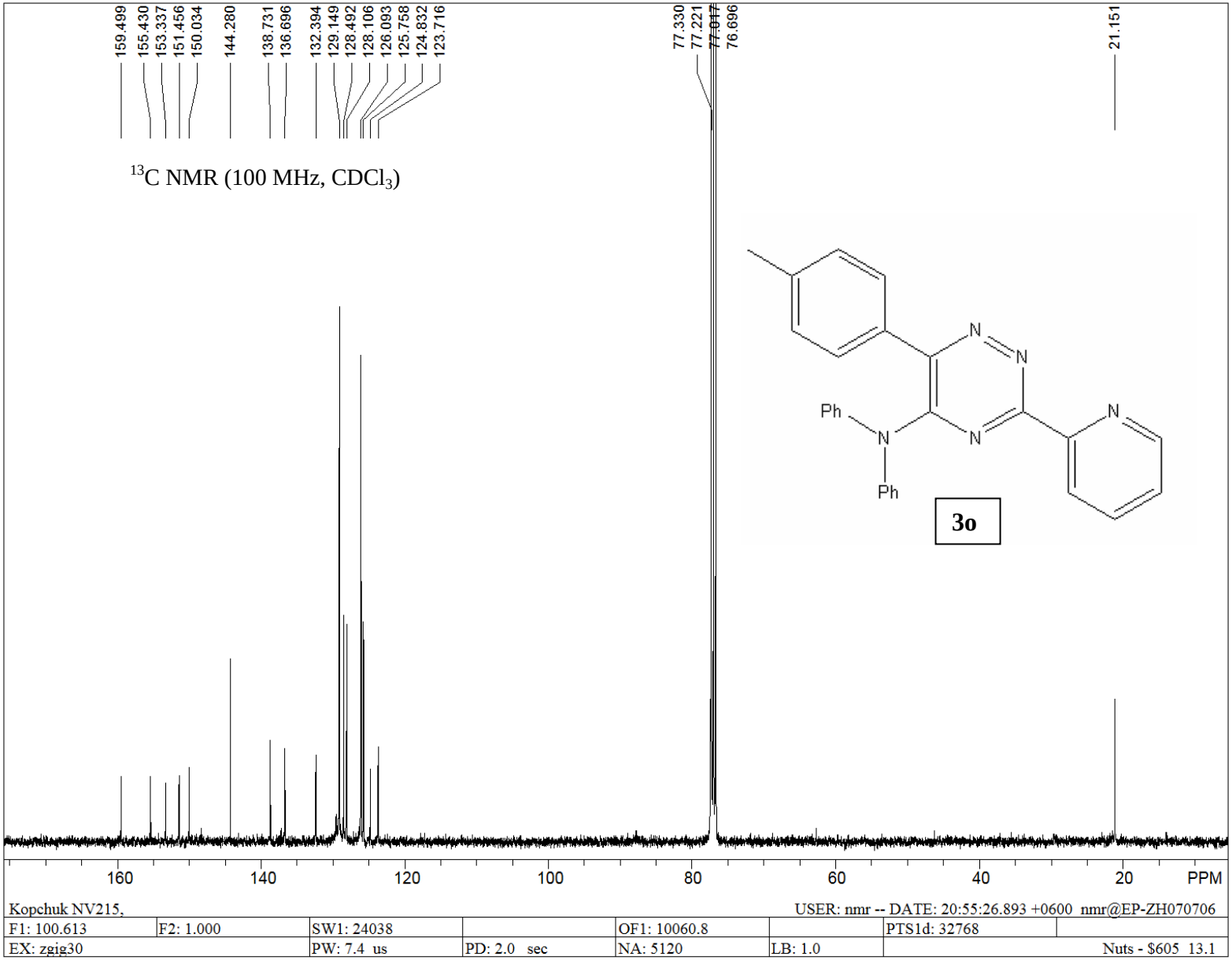

8.423
8.417
8.408
8.403
8.398
7.750
7.747
7.729
7.727
7.512
7.509
7.500
7.494
7.488
7.487
7.484
7.478
7.474
7.456
3.490
3.478
3.465
3.014
2.505
2.500
2.496
2.491
2.487
2.360
2.348
2.335
2.210
^{1}H NMR (400 MHz, DMSO-d_6)
N
N
N
N
N
3m
2.31
2.21
6.45
8.97
3.57
2.68
8
7
6
5
4
3
2
PPM
Kopchuk NV 306
USER: nmr -- DATE: 13:33:32.164 +0600 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2465.2
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 9
LB: 0.3
Nuts - $669 1.1

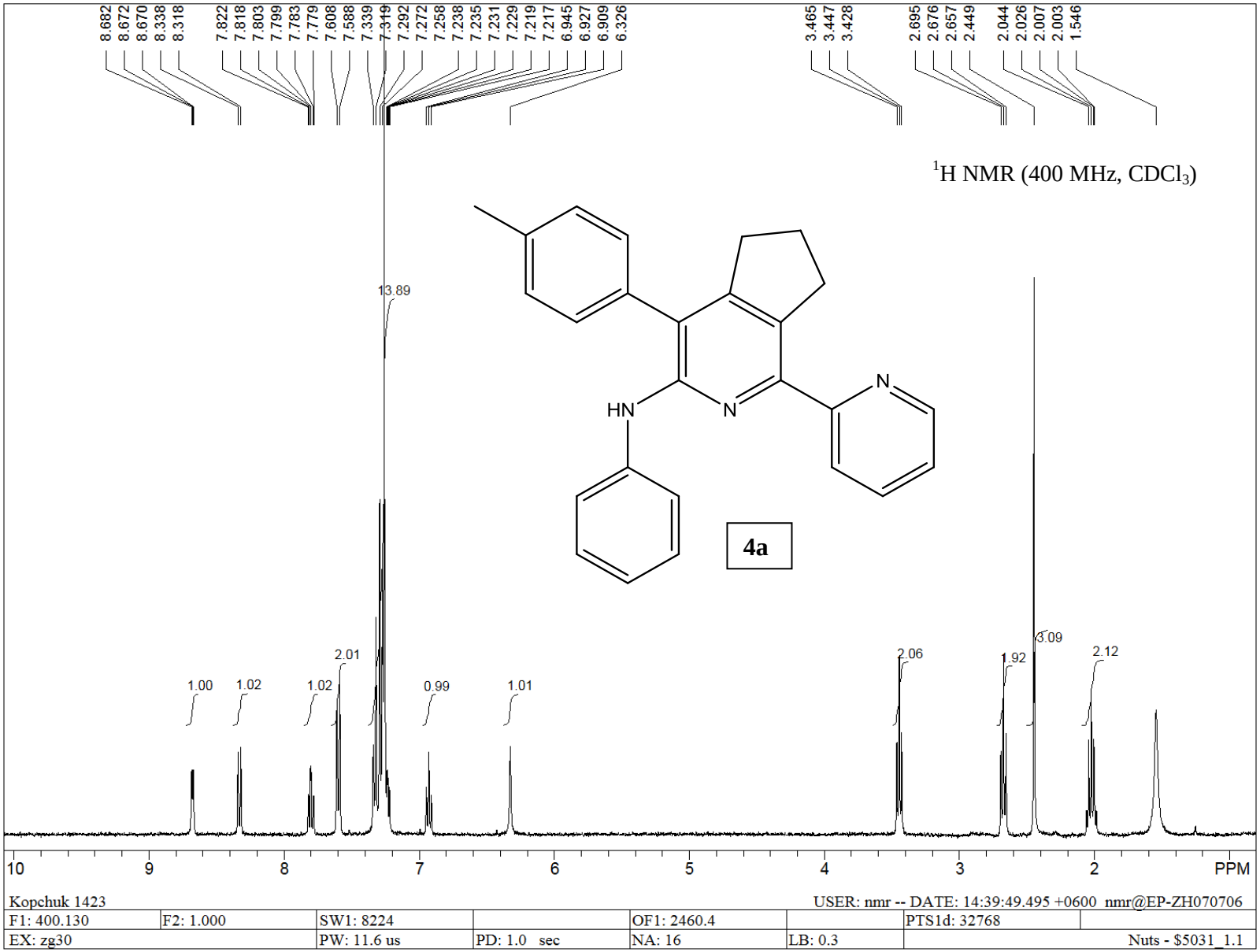

159.397
154.606
146.599
137.148
135.588
130.921
129.324
128.893
128.522
128.201
127.318
77.367
77.257
77.053
76.732
54.403
46.674
45.988
^{13}C NMR (100 MHz, CDCl$_3$)
N
N
N
N
N
3m
160
140
120
100
80
60
40
PPM
Kopchuk NV306,
USER: nmr -- DATE: 15:10:58.653 +0600 nmr@EP-ZH070706
F1: 100.613
F2: 1.000
SW1: 24038
OF1: 10060.8
PTS1d: 32768
EX: zgig30
PW: 7.4 us
PD: 2.0 sec
NA: 507
LB: 1.0
Nuts - $680 13.1

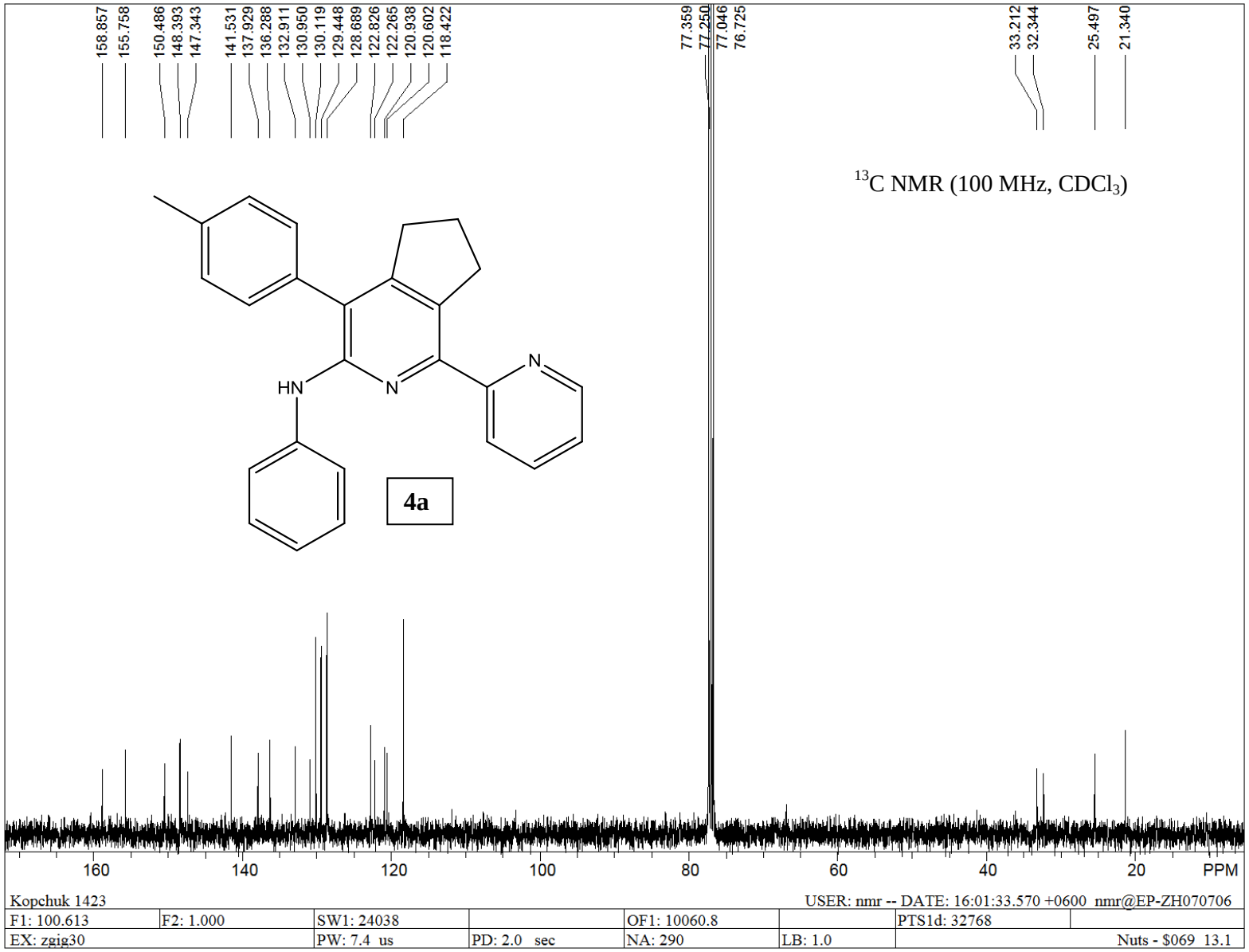

8.655
7.649
7.628
7.608
7.354
7.334
7.299
7.280
7.260
7.082
7.064
3.070
2.523
2.498
2.493
2.489
2.449
^{1}H NMR (400 MHz, DMSO-d_6)
N
N
HN
N
3n
4.21
3.83
1.05
0.92
3.07
2.70
9
8
7
6
5
4
3
PPM
Kopchuk 1427
USER: nmr -- DATE: 14:55:15.195 +0600 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2464.2
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 20
LB: 0.3
Nuts - $222 1.1

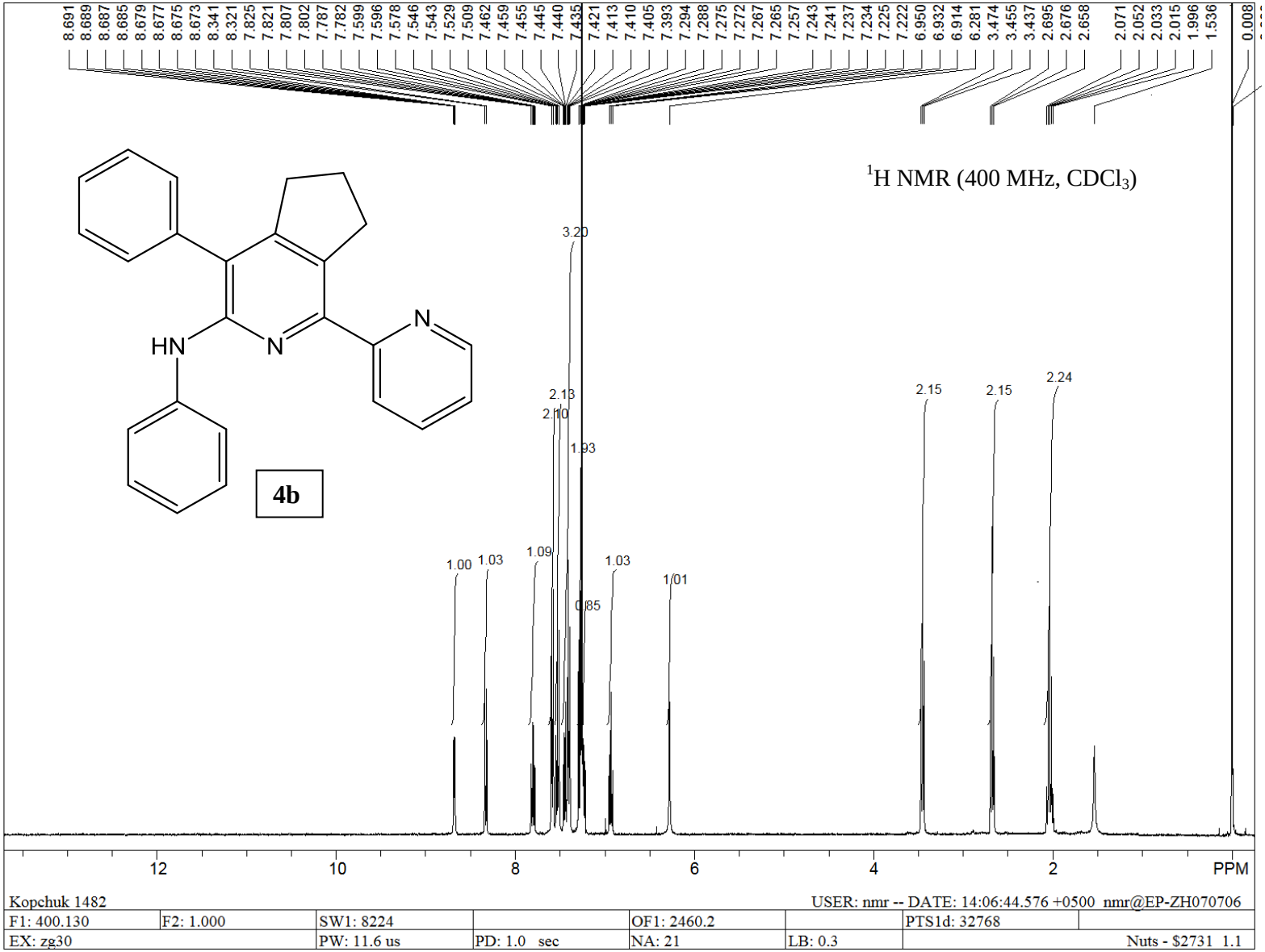

^{13}C NMR (100 MHz, DMSO-d_6)
162.587
150.883
147.098
139.011
138.158
131.113
129.443
128.481
128.306
124.062
122.742
40.157
39.946
39.530
39.326
39.115
38.903
23.021
20.950
N
N
HN
N
3n
160
140
120
100
80
60
40
20 PPM
Kopchuk 1427,
USER: nmr -- DATE: 04:39:54.290 +0600 nmr@EP-ZH070706
F1: 100.613
F2: 1.000
SW1: 24038
OF1: 10010.5
PTS1d: 32768
EX: zgig30
PW: 7.4 us
PD: 2.0 sec
NA: 3840
LB: 1.0
Nuts - $238 13.1

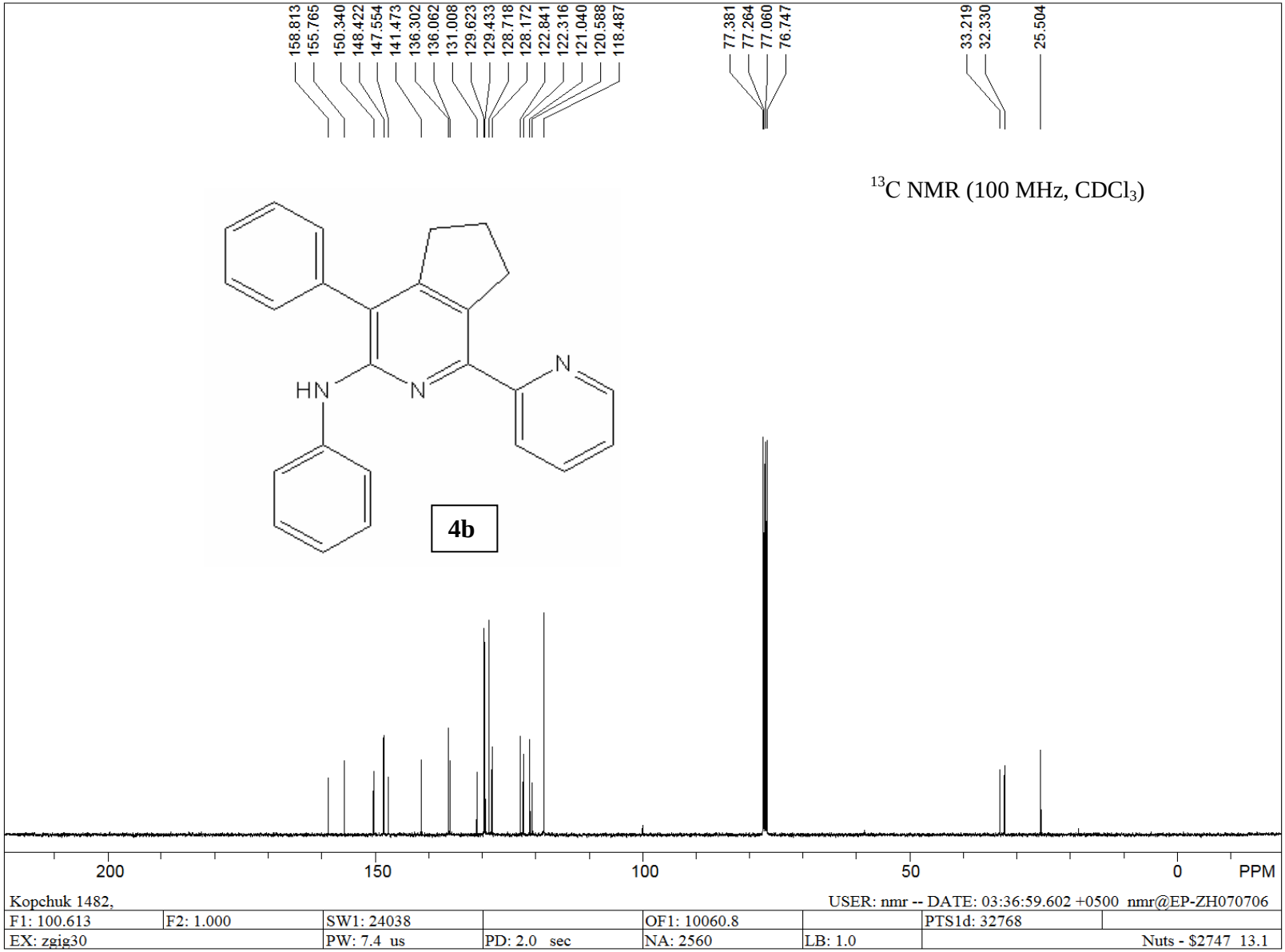

8.707
8.705
8.695
8.693
8.149
8.043
8.023
7.862
7.858
7.843
7.839
7.824
7.819
7.462
7.460
7.450
7.448
7.443
7.441
7.431
7.429
7.404
7.384
7.213
7.194
7.174
7.085
7.066
7.048
7.038
7.035
7.017
6.955
6.936
3.029
2.500
2.495
2.491
2.486
2.482
2.242
^{1}H NMR (400 MHz, DMSO-d_6)
6.10
4.17
2.08
2.19
1.00
1.11
1.05
1.05
3.08
Ph
N
N
N
N
N
Ph
3o
9
8
7
6
5
4
3
PPM
Kopchuk NV 215
USER: nmr -- DATE: 15:03:31.788 +0500 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2463.4
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 8
LB: 0.3
Nuts - $3934 1.1


159.499
155.430
153.337
151.456
150.034
144.280
138.731
136.696
132.394
129.149
128.492
128.106
126.093
125.758
124.832
123.716
77.330
77.221
77.017
76.696
21.151
^{13}C NMR (100 MHz, CDCl$_3$)
Ph
N
N
N
N
N
Ph
3o
160
140
120
100
80
60
40
20
PPM
Kopchuk NV215,
USER: nmr -- DATE: 20:55:26.893 +0600 nmr@EP-ZH070706
F1: 100.613
F2: 1.000
SW1: 24038
OF1: 10060.8
PTS1d: 32768
EX: zgig30
PW: 7.4 us
PD: 2.0 sec
NA: 5120
LB: 1.0
Nuts - $605 13.1

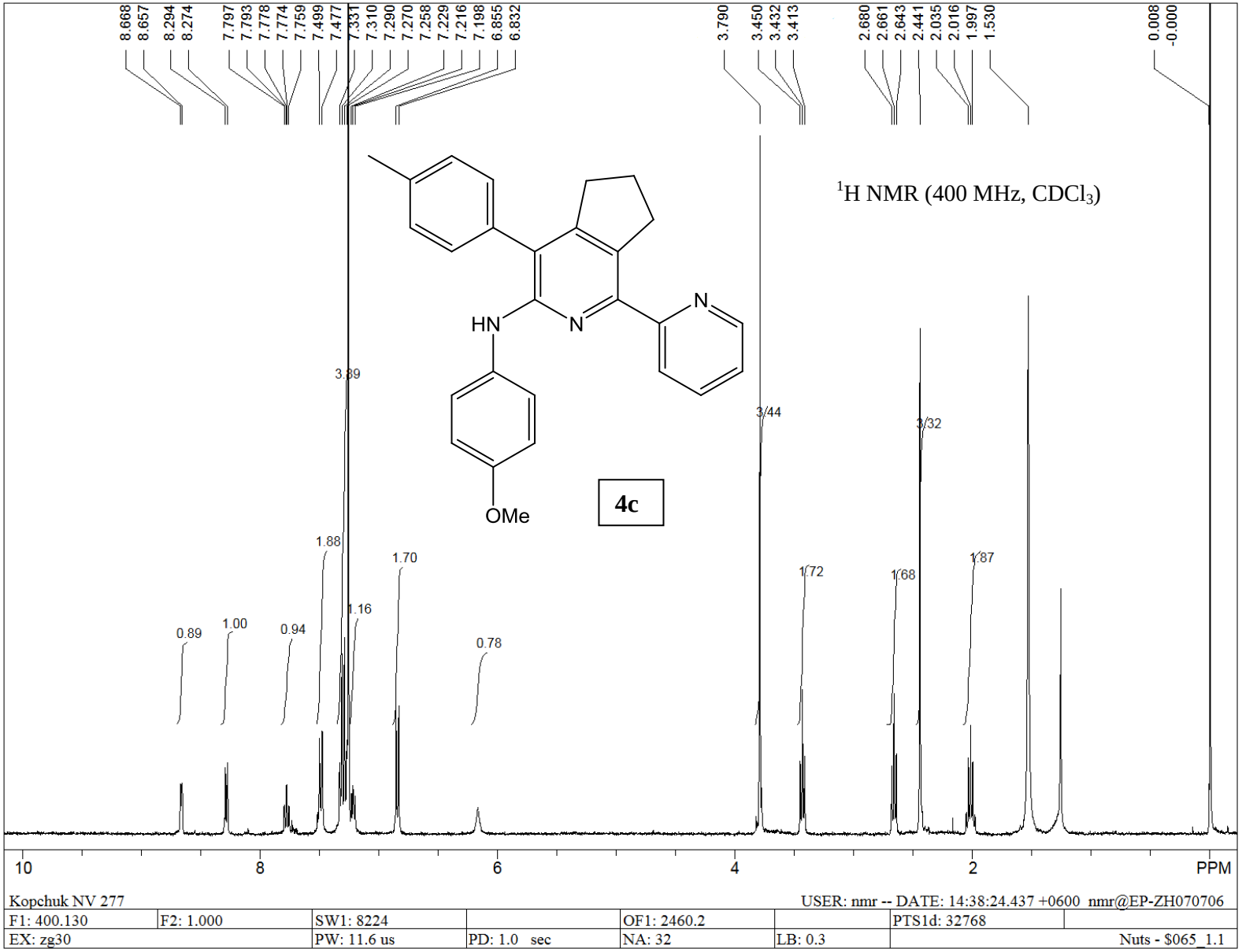

8.682
8.672
8.670
8.338
8.318
7.822
7.818
7.803
7.799
7.783
7.779
7.608
7.588
7.339
7.319
7.292
7.272
7.258
7.238
7.235
7.231
7.229
7.219
7.217
6.945
6.927
6.909
6.326
3.465
3.447
3.428
2.695
2.676
2.657
2.449
2.044
2.026
2.007
2.003
1.546
^{1}H NMR (400 MHz, $CDCl_3$)
HN
N
N
4a
13.89
1.00
1.02
1.02
2.01
0.99
1.01
2.06
1.92
3.09
2.12
10
9
8
7
6
5
4
3
2
PPM
Kopchuk 1423
USER: nmr -- DATE: 14:39:49.495 +0600 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2460.4
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 16
LB: 0.3
Nuts - $5031_1.1


158.857
155.758
150.486
148.393
147.343
141.531
137.929
136.288
132.911
130.950
130.119
129.448
128.689
122.826
122.265
120.938
120.602
118.422
77.359
77.250
77.046
76.725
33.212
32.344
25.497
21.340
13C NMR (100 MHz, CDCl3)
HN
N
N
4a
160
140
120
100
80
60
40
20
PPM
Kopchuk 1423
USER: nmr -- DATE: 16:01:33.570 +0600 nmr@EP-ZH070706
F1: 100.613
F2: 1.000
SW1: 24038
OF1: 10060.8
PTS1d: 32768
EX: zgig30
PW: 7.4 us
PD: 2.0 sec
NA: 290
LB: 1.0
Nuts - $069 13.1


8.691 8.689 8.687 8.685 8.679 8.677 8.675 8.673 8.341 8.321 7.825 7.821 7.807 7.802 7.787 7.782 7.599 7.596 7.578 7.546 7.543 7.529 7.509 7.462 7.459 7.455 7.445 7.440 7.435 7.421 7.413 7.410 7.405 7.393 7.294 7.288 7.275 7.272 7.267 7.265 7.257 7.243 7.241 7.237 7.234 7.225 7.222 6.950 6.932 6.914 6.281 3.474 3.455 3.437 2.695 2.676 2.658 2.071 2.052 2.033 2.015 1.996 1.536 0.008
^{1}H NMR (400 MHz, $CDCl_3$)
HN
N
N
4b
3.20
2.13
2.10
1.93
2.15
2.15
2.24
1.00
1.03
1.09
1.03
1.01
0.85
12
10
8
6
4
2
PPM
Kopchuk 1482
USER: nmr -- DATE: 14:06:44.576 +0500 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2460.2
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 21
LB: 0.3
Nuts - $2731 1.1



^{13}C NMR (100 MHz, $CDCl_3$)


8.668
8.657
8.294
8.274
7.797
7.793
7.778
7.774
7.759
7.499
7.477
7.331
7.310
7.290
7.270
7.258
7.229
7.216
7.198
6.855
6.832
3.790
3.450
3.432
3.413
2.680
2.661
2.643
2.441
2.035
2.016
1.997
1.530
0.008
-0.000
^{1}H NMR (400 MHz, CDCl$_3$)
HN
N
N
OMe
4c
3.89
3.44
3.32
1.88
1.70
1.72
1.68
1.87
1.16
0.89
1.00
0.94
0.78
10
8
6
4
2
PPM
Kopchuk NV 277
USER: nmr -- DATE: 14:38:24.437 +0600 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2460.2
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 32
LB: 0.3
Nuts - $065_1.1