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A PASE-based approach towards 12-(1*H*-1,2,3-triazol-1-yl)indolo-[2,1-*a*]isoquinolines *via* the reaction of 3-(isoquinolin-1-yl)-1,2,4-triazines with benzyne

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General. Unless otherwise indicated, all common reagents and solvents were used from commercial suppliers without further purification. Melting points were measured in a Boethius apparatus. ^{1}H, ^{13}C and ^{19}F NMR spectra were acquired on a Bruker Avance-400 spectrometer, 298 K, digital resolution ± 0.01 ppm, using TMS as internal reference for ^{1}H and ^{13}C NMR or $CFCl_3$ for ^{19}F NMR. Mass-spectra were recorded on MicrOTOF-Q II (Bruker Daltonics), electrospray as a method of ionization. Microanalyses (C, H, N) were performed using a Perkin–Elmer 2400 elemental analyzer. The structure of compound **8a** was determined on a X-ray diffractometer "Xcalibur E".

Isoquinoline-1-carbohydrazonamide **3**,[S1] isoquinoline-1-carbaldehyde **5**, [S2] isonitrosoacetophenone hydrazones **4**[S3] were synthesized as described in literature.

1-Cyanoisoquinoline 6. Isoquinoline *N*-oxide **7** (1.015 g, 7 mmol) was dissolved in dry DMF (50 ml). Triethylamine (1.07 ml, 7.7 mmol), potassium cyanide (1.37 g, 21 mmol) were added. Then Me_3SiCl (3.55 ml, 28 mmol) was added dropwise, and the resulting mixture was stirred at 50 °C for 8 h. Then potassium cyanide (0.91 g, 14 mmol) and Me_3SiCl (2.37 ml, 18.7 mmol, dropwise) were added, and the resulting mixture was stirred at 50 °C for additional 8 h. The resulting mixture was cooled to room temperature, water (100 ml) was added, and this was stirred at room temperature for 1 h. The obtained precipitate was filtered off, washed with water and dried in vacuum. The product was used in the next step without additional purification. Yield 755 mg (70%). M.p. 86-88 °C. ^{1}H NMR (DMSO-d_6, δ, ppm): 7.78-7.86 (m, 2H), 7.90 (m, 1H), 7.95 (m, 1H), 8.36 (m, 1H), 8.66 (d, 1H, ^{3}J 5.6 Hz). ESI-MS, *m/z*: 155.06 $(M+H)^+$.

*General method for the synthesis of triazines **1b-d***

Isoquinoline-1-carbaldehyde **5** (85 mg, 0.54 mmol) was dissolved in acetic acid (20 ml). The corresponding hydrazone **4** (0.54 mmol) was dissolved in acetic acid (20 ml). The two solutions have been mixed and this was kept at room temperature overnight. Then mixture was

heated to reflux 10-20 min until clear solution was formed. The solvent was removed under reduced pressure. Ethanol (30 ml) was added to the residue; the resulting solid was filtered off, washed with ethanol and dried. The crude triazines were used directly in the next step without additional purification.

1-(6-Phenyl-1,2,4-triazin-3-yl)isoquinoline (1b). Yield 100 mg (61%). ^{1}H NMR (DMSO-d_6, δ, ppm): 7.61-7.65 (m, 3H, Ph), 7.66-7.70 and 7.79-7.84 (both m, 2H, H-6, H-7), 8.00 (d, 1H, ^3J 5.6 Hz, H-4), 8.08 and 8.27 (both m, 2H, H-5, H-8), 8.33-8.38 (m, 2H, Ph), 8.69 (d, 1H, ^3J 5.6 Hz, H-3), 9.60 (s, 1H, triazine). ESI-MS, *m/z*: 285.11 $(M+H)^+$. Anal. Calcd. for $C_{18}H_{12}N_4$: C 76.04, H 4.25, N 19.71%; Found: C 76.13, H 4.33, N 19.63%.

1-(6-*p*-Tolyl-1,2,4-triazin-3-yl)isoquinoline (1c). Yield 89 mg (0.30 mmol, 52%). M.p. 204-206 °C. ^{1}H NMR (DMSO-d_6, δ, ppm): (s, 3H, $\underline{CH_3}$Ph),7.42-7.46 (m, 2H, $\underline{Ph}CH_3$), 7.64-7.70 and 7.79-7.84 (both m, 2H, H-6, H-7), 8.00 (d, 1H, ^3J 5.6 Hz, H-4), 8.08 and 8.26 (both m, 2H, H-5, H-8), 8.25 (m, 2H, $\underline{Ph}CH_3$), 8.69 (d, 1H, ^3J 5.6 Hz, H-3), 9.56 (s, 1H, triazine). ESI-MS, *m/z*: 299.13 $(M+H)^+$. Anal. Calcd. for $C_{19}H_{14}N_4$: C 76.49, H 4.73, N 18.78%; Found: C 76.38, H 4.61, N 18.64%.

1-[6-(4-Fluorophenyl)-1,2,4-triazin-3-yl]isoquinoline (1d). Yield 125 mg (76%). M.p. 194-196 °C. ^{1}H NMR (DMSO-d_6, δ, ppm): 7.35-7.42 (m, 2H, PhF), 7.64-7.69 and 7.77-7.83 (both m, 1H, H-6, H-7), 7.97 (d, 1H, ^3J 5.6 Hz, H-4), 8.06 and 8.28 (both m, 1H, H-5, H-8), 8.40-8.47 (m, 2H, PhF), 8.69 (d, 1H, ^3J 5.6 Hz, H-3), 9.60 (s, 1H, triazine). ^{19}F NMR (DMSO-d_6, δ, ppm): -109.1. ESI-MS, *m/z*: 303.10 $(M+H)^+$. Anal. Calcd. for $C_{18}H_{11}FN_4$: C 71.52, H 3.67, N 18.53%; Found: C 71.41, H 3.55, N 18.44%

1-(5,6-Diphenyl-1,2,4-triazin-3-yl)isoquinoline (1a). Benzil **2** (210 mg, 1.00 mmol) was dissolved in a mixture of ethanol and THF (50 ml, 1:1), then solution of isoquinoline-1-carbohydrazonamide **3** (186 mg, 1 mmol) in ethanol (50 ml) was added, and the resulting mixture was refluxed for 10 h. After cooling to room temperature, the precipitate was filtered off, washed with ethanol and dried. The crude triazine was used directly in the next step without additional purification. Yield 360 mg (50%). ^{1}H NMR ($CDCl_3$, δ, ppm): 7.32-7.39 (m, 2H, Ph), 7.40-7.48 (m, 4H, Ph), 7.63-7.73 (m, 5H, Ph), 7.74-7.79 (m, 1H), 7.84-7.88 (m, 1H), 7.94-7.99 (m, 1H), 8.61-8.65 (m, 1H), 8.79-8.82 (d, 1H, ^3J 5.6 Hz, H-3). ESI-MS, *m/z*: 461.15 $(M+H)^+$. Anal. Calcd. for $C_{24}H_{16}N_4$: C 79.98, H 4.47, N 15.54%; Found: C 79.87, H 4.34, N 15.40%.

*General method for the reaction 1,2,4-triazines **1** with arynes*

The corresponding 1,2,4-triazine **1** (0.30 mmol) was dissolved in dry toluene (25 ml), and isoamyl nitrite (0.12 ml, 0.90 mmol) was added. The mixture was stirred under reflux under argon, while a solution of 2-aminobenzoic acid (124 mg, 0.90 mmol) in dry 1,4-dioxane (10 ml) was added from dropping funnel for 30 min. The mixture was heated under reflux for 1 h and

then cooled to room temperature. Then, the reaction mass was washed with potassium hydroxide solution (3 M, 3×50 ml) and dried with anhydrous sodium sulfate. The solvents were removed under reduced pressure. The products were separated by column chromatography (silica gel, ethyl acetate as the eluent, R_f = 0.8). Analytical samples of products were obtained by recrystallization (from MeCN).

12-(4,5-Diphenyl-1*H*-1,2,3-triazol-1-yl)indolo[2,1-*a*]isoquinoline (8a). Yield 67 mg (51%). ^{1}H NMR ($CDCl_3$, δ, ppm): 6.83 (d, 1H, ^3J 7.6 Hz, H-5), 7.05-7.11 (m, 2H), 7.12-7.16 (m, 2H), 7.18-7.22 (m, 2H), 7.31-7.38 (m, 6H), 7.39-7.44 (m, 2H), 7.45-7.50 (m, 1H), 7.59-7.63 (m, 1H), 7.77-7.83 (m, 2H), 8.11 (d, 1H, ^3J 7.6 Hz, H-6). ^{13}C NMR ($CDCl_3$, δ, ppm): 109.8, 110.3, 117.2, 121.6, 122.2, 123.7, 123.9, 124.0, 126.0, 127.2, 127.2, 127.5, 128.0, 128.2, 128.6, 128.7, 129.0, 129.0, 129.2, 129.4, 129.9, 130.0, 131.1, 136.6. ESI-MS, *m/z*: 437.18 $(M+H)^+$. Anal. Calcd. for $C_{30}H_{20}N_4$: C 82.55, H 4.62, N 12.83%; Found: C 82.53, H 4.34, N 12.71%. The XRD analysis of the single crystal of $C_{30}H_{20}N_4$ was accomplished on an “Xcsalibur 3” diffractometer on standard procedure (MoK-irradiation, graphite monochromator, ω-scans with 1o step, T=295(2) K). Using Olex2,[S4] the structure was solved with the ShelXS[S5] structure solution program using Direct Methods and refined with the ShelXL[S5] refinement package using Least Squares minimization in anisotropic approximation for the nonhydrogen atoms. The H-atoms were added in the calculated positions and were refined in the “riding” model in the isotropic approximation. Crystal Data: $C_{30}H_{20}N_4$, M = 436.50, system is monoclinic, a = 13.2659(11) Å, b = 12.4205(6) Å, c = 14.6024(12) Å, β = 111.610(8)°, V = 2236.9(3) Å3, space group P21/c, Z = 4, μ(MoKα) = 0.078 mm^{-1}. On the angles 2.84 < Θ < 33.24o 32849 reflections measured, 7409 unique (Rint = 0.0442) which were used in all calculations. The final R1 = 0.1222, wR2 = 0.1205 (all data) and R1 = 0.0496, wR2 = 0.1078 (I>2σ(I)). Largest diff. peak and hole are 0.225 and -0.182 ē $Å^{-3}$. CCDC 1861705 contains the crystallographic data for this compound.

12-(4-Phenyl-1*H*-1,2,3-triazol-1-yl)indolo[2,1-*a*]isoquinoline (8b). Yield 60 mg (56%). ^{1}H NMR ($CDCl_3$, δ, ppm): 6.89 (d, 1H, ^3J 7.2 Hz, H-5), 7.16-7.20 (m, 1H), 7.29-7.35 (m, 1H), 7.38-7.54 (m, 7H), 7.61-7.65 (m, 1H), 7.89-7.93 (m, 1H), 8.01-8.06 (m, 2H), 8.16 (s, 1H, triazole), 8.20 (d, 1H, ^3J 7.2 Hz, H-6). ^{13}C NMR ($CDCl_3$, δ, ppm): 106.4, 110.0, 110.6, 117.0, 121.5, 122.6, 123.5, 123.7, 123.9, 123.9, 125.5, 126.0, 127.3, 128.2, 128.7, 128.9, 129.0, 129.1 (2C), 129.9, 130.0, 148.0. ESI-MS, *m/z*: 361.15 $(M+H)^+$. Anal. Calcd. For $C_{24}H_{16}N_4$: C 79.98, H 4.47, N 15.54%; Found: C 79.84, H 4.33, N 15.41%.

12-(4-*p*-Tolyl-1*H*-1,2,3-triazol-1-yl)indolo[2,1-*a*]isoquinoline (8c). Yield 61 mg (54%). ^{1}H NMR ($CDCl_3$, δ, ppm): 2.40 (s, 3H, CH_3), 6.71-6.75 (d, 1H, ^3J 7.2 Hz, H-5), 7.09-7.13 (m, 1H), 7.20-7.25 (m, 1H), 7.27-7.30 (m, 2H, PhCH_3), 7.31-7.34 (m, 2H), 7.34-7.40 (m, 1H), 7.41-7.46 (m, 1H), 7.49-7.52 (m, 1H), 7.76-7.81 (m, 1H), 7.88-7.92 (m, 2H, PhCH_3), 8.02-8.06 (d,

1H, ^{3}J 7.2 Hz, H-6), 8.04 (s, 1H, triazole). ^{13}C NMR ($CDCl_3$, δ, ppm): 24.5, 101.4, 104.7, 105.1, 111.7, 116.2, 117.2, 117.7, 118.5, 118.7, 120.3, 120.6, 122.0, 122.4, 122.8, 123.4, 123.6, 124.5, 124.7, 133.1, 143.0. ESI-MS, *m/z*: 375.16 $(M+H)^+$. Anal. Calcd. For $C_{25}H_{18}N_4$: C 80.19, H 4.85, N 14.96%; Found: C 80.03, H 4.72, N 14.83%.

12-[4-(4-Fluorophenyl)-1*H*-1,2,3-triazol-1-yl]indolo[2,1-*a*]isoquinoline (8d). Yield 59 mg (0.156 mmol, 52%). ^{1}H NMR ($CDCl_3$, δ, ppm): 6.82-6.86 (d, 1H, ^{3}J 7.6 Hz, H-5), 7.08-7.16 (m, 3H, C_6H_4F, isoquin.), 7.23-7.29 (m, 1H), 7.32-7.39 (m, 2H), 7.40-7.46 (m, 2H), 7.55-7.60 (m, 1H), 7.83-7.88 (m, 1H), 7.90-7.96 (m, 2H, C_6H_4F), 8.06 (s, 1H, triazole), 8.12-8.16 (d, 1H, ^{3}J 7.6 Hz, H-5). ^{19}F NMR (DMSO-d_6, δ, ppm): -113.1. ^{13}C NMR ($CDCl_3$, δ, ppm): 101.2, 104.7, 105.1, 110.8 (d, *J* 22.0 Hz), 111.6, 116.1, 117.2, 117.8, 118.3, 118.5. 118.6, 120.2, 121.5 (d, *J* 3.0 Hz), 122.0, 122.4, 122.5, 122.7, 123.4, 123.6, 124.7, 142.1, 157.6 (d, *J* 246.4 Hz). ESI-MS, *m/z*: 379.14 $(M+H)^+$. Anal. Calcd. For $C_{24}H_{15}FN_4$: C 76.18, H 4.00, N 14.81%. Found: C 76.11, H 4.10, N 14.77%.

References

[S1] S. P. J. T. Bachollet, D. Volz, B. Fiser, S. Münch, F. Rönicke, C. Franziska, J. Carrillo, H. Adams, S. Bräse and J. P. A. Harrity, *Chem.-Eur. J.*, 2016, **22**, 12430.

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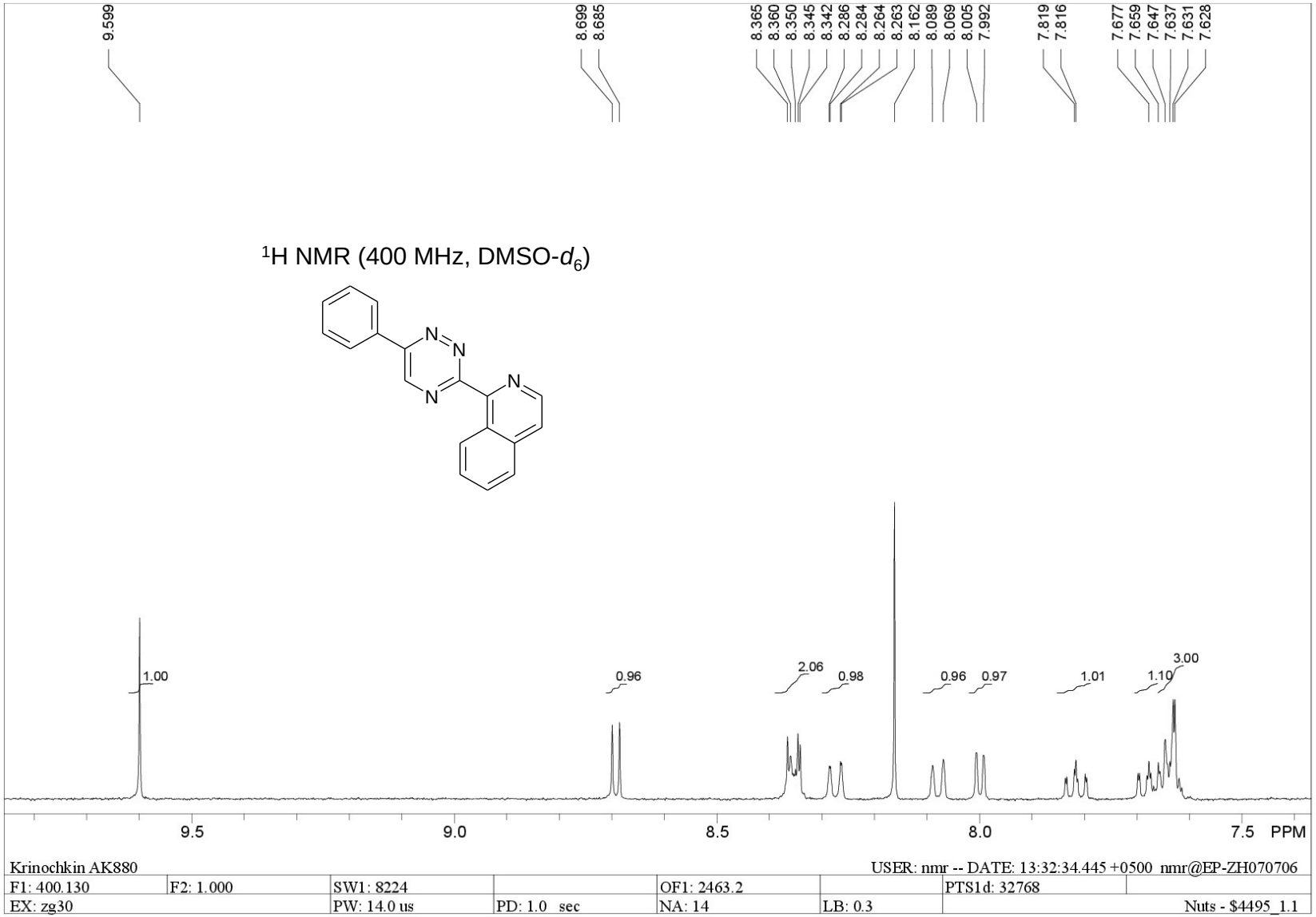
[S5] G. M. Sheldrick, *Acta Crystallogr*., 2008, **A64**, 112.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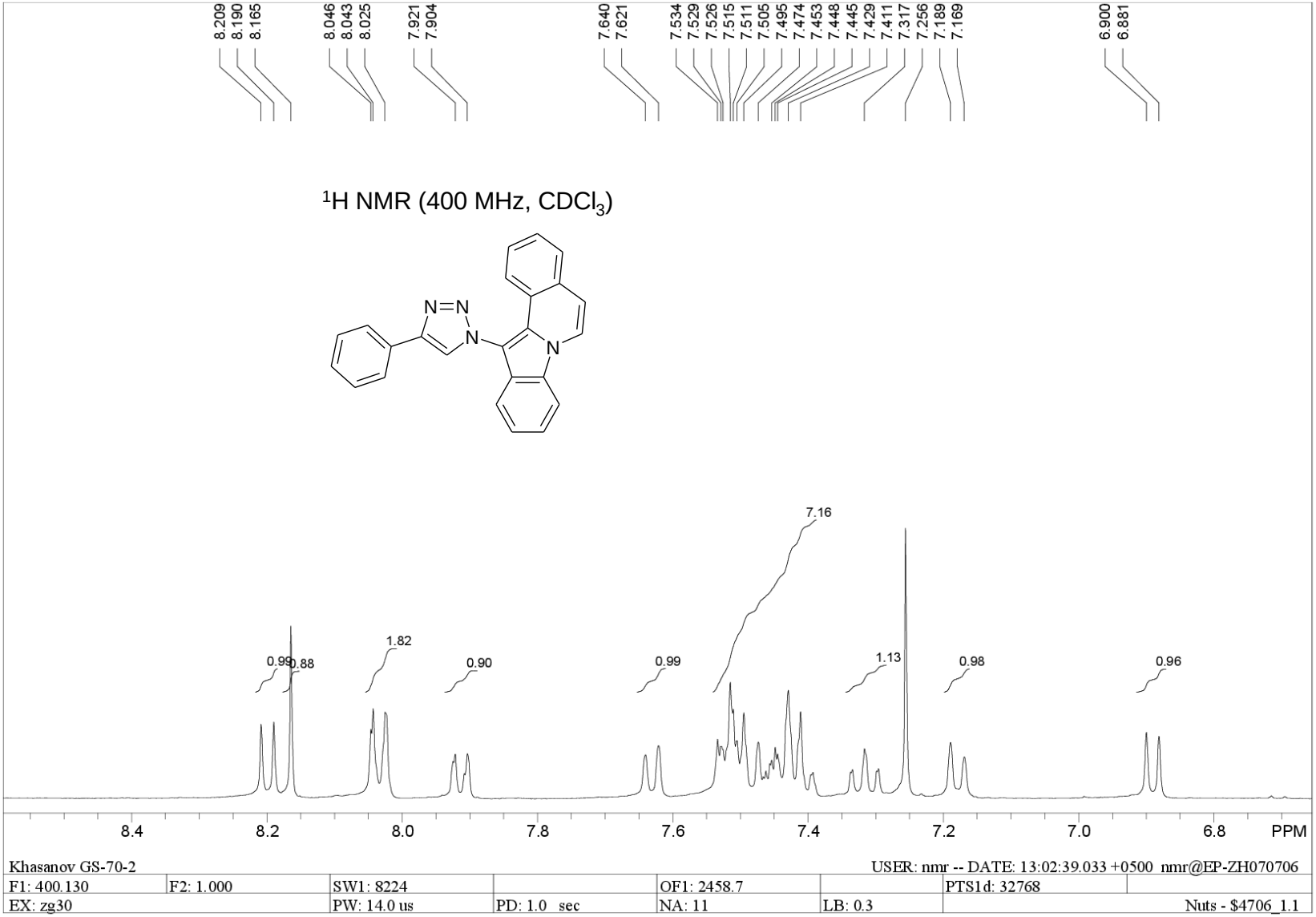
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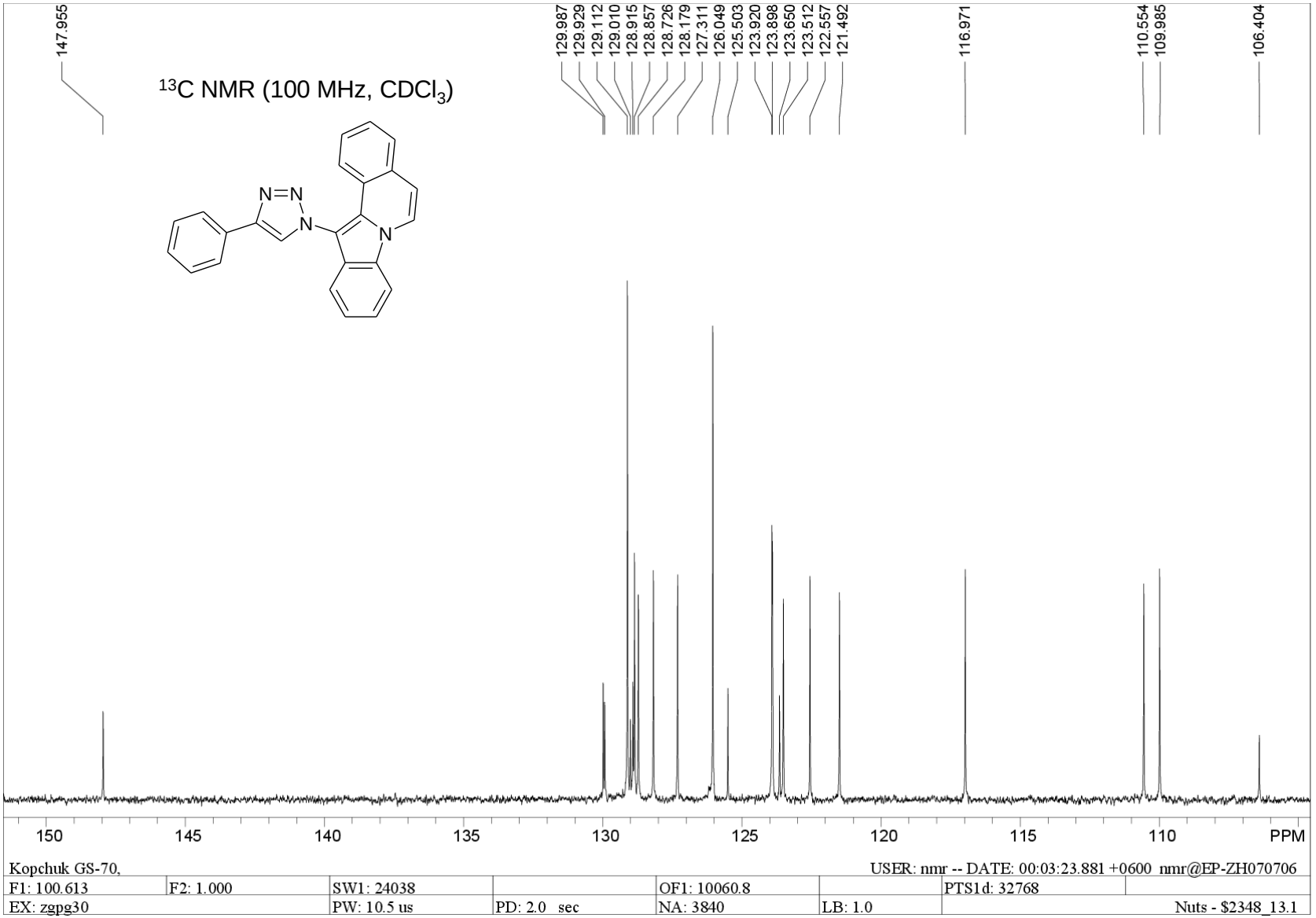
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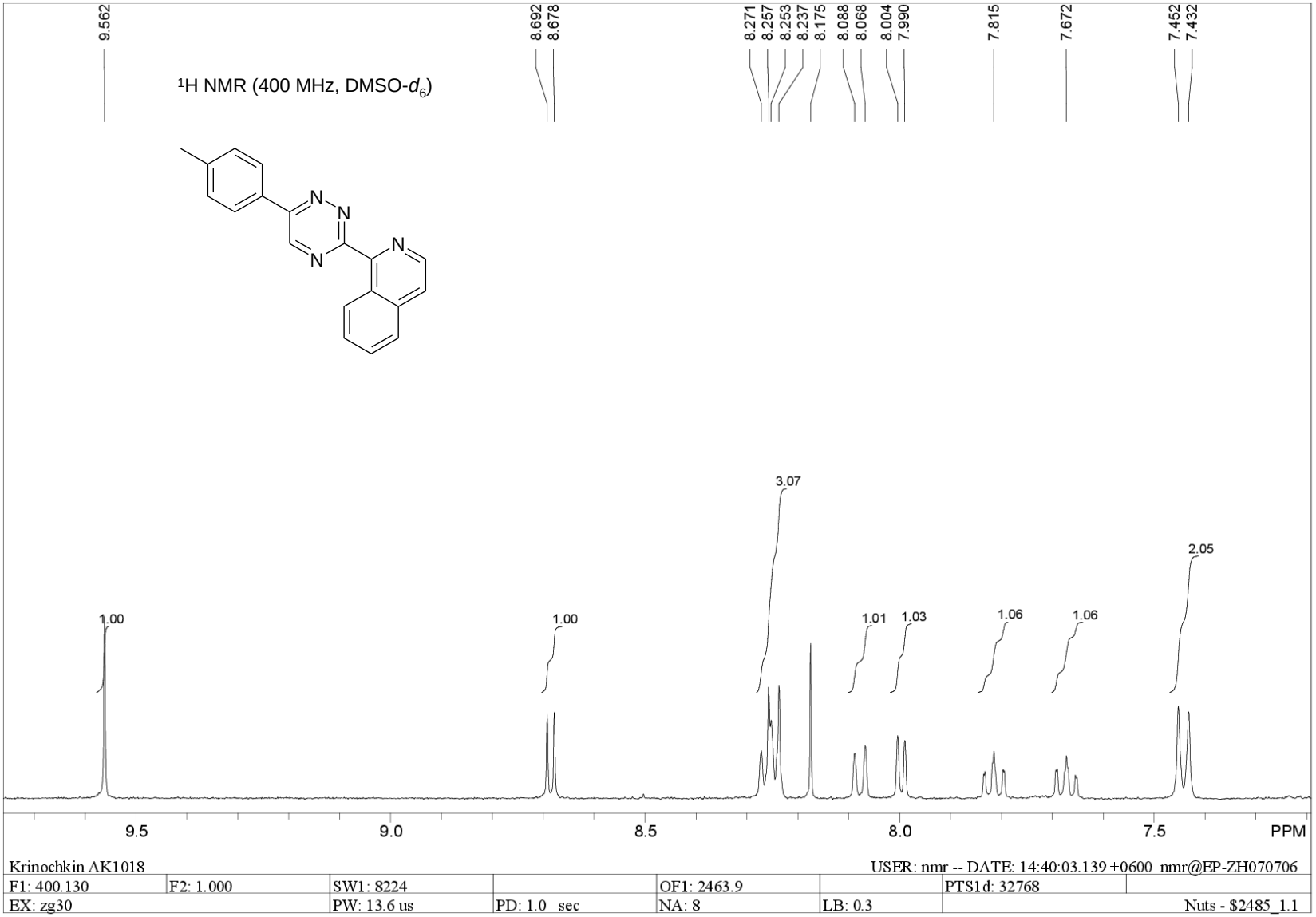
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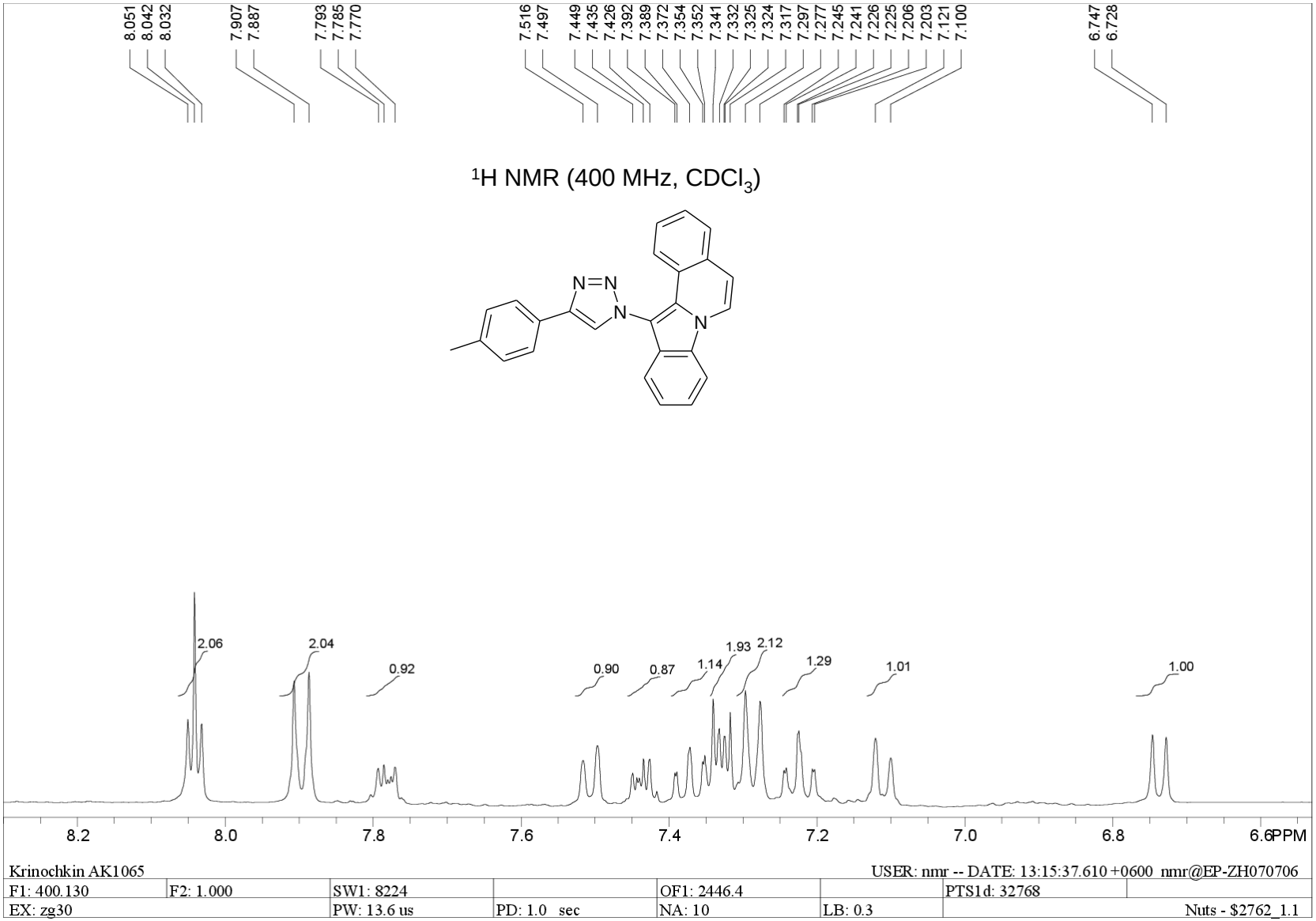
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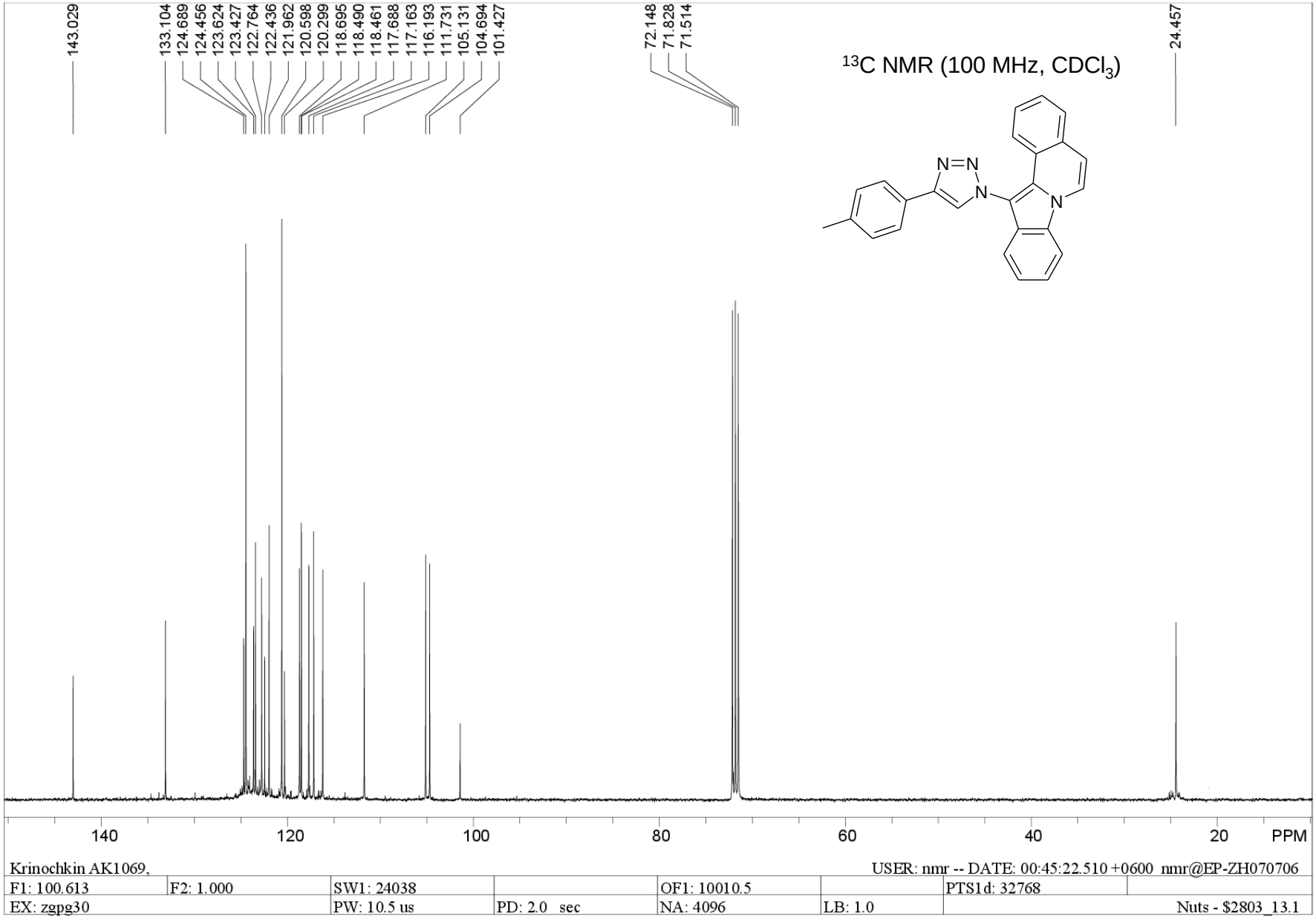
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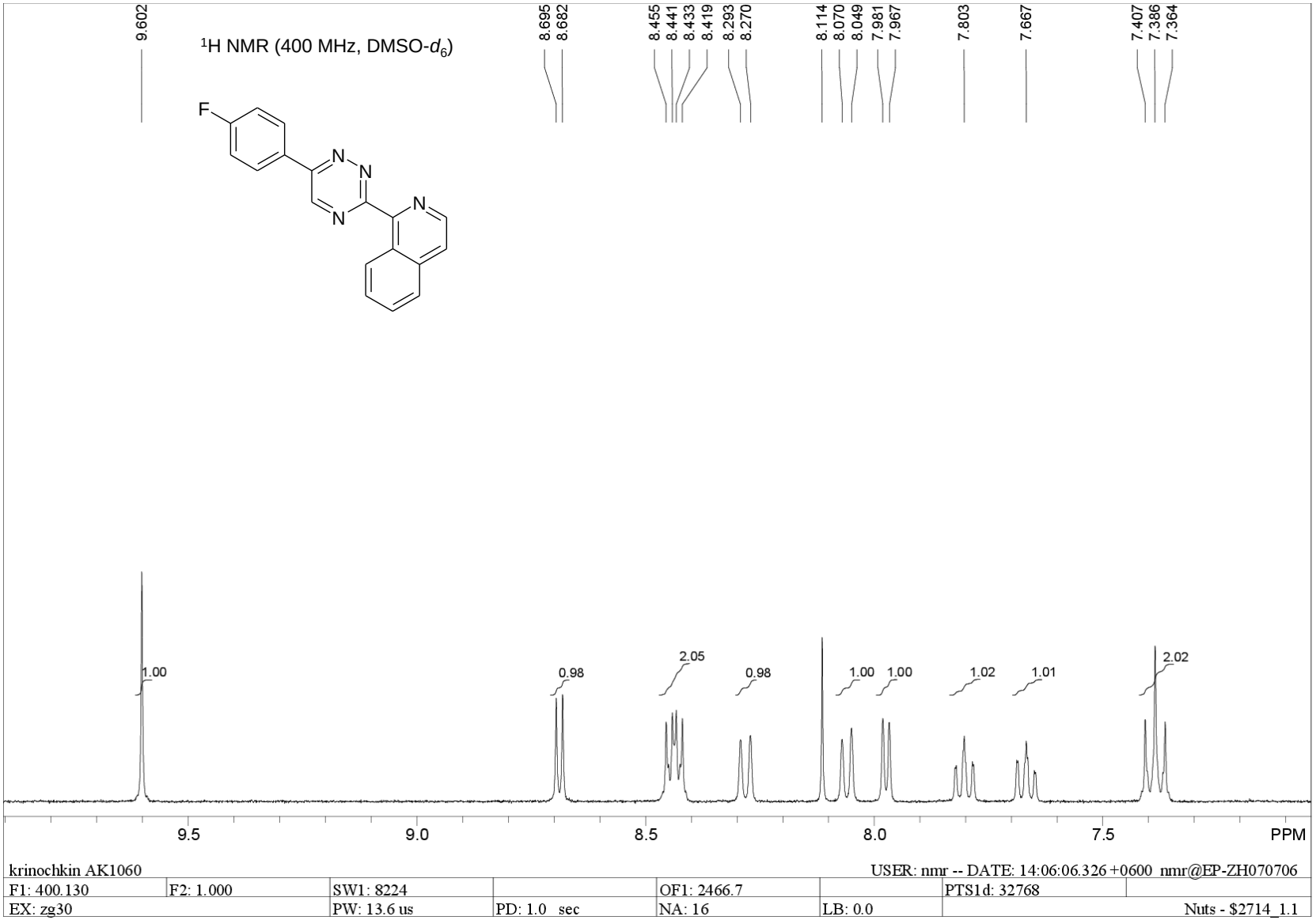
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Nuts - $2714_1.1

Figure S7

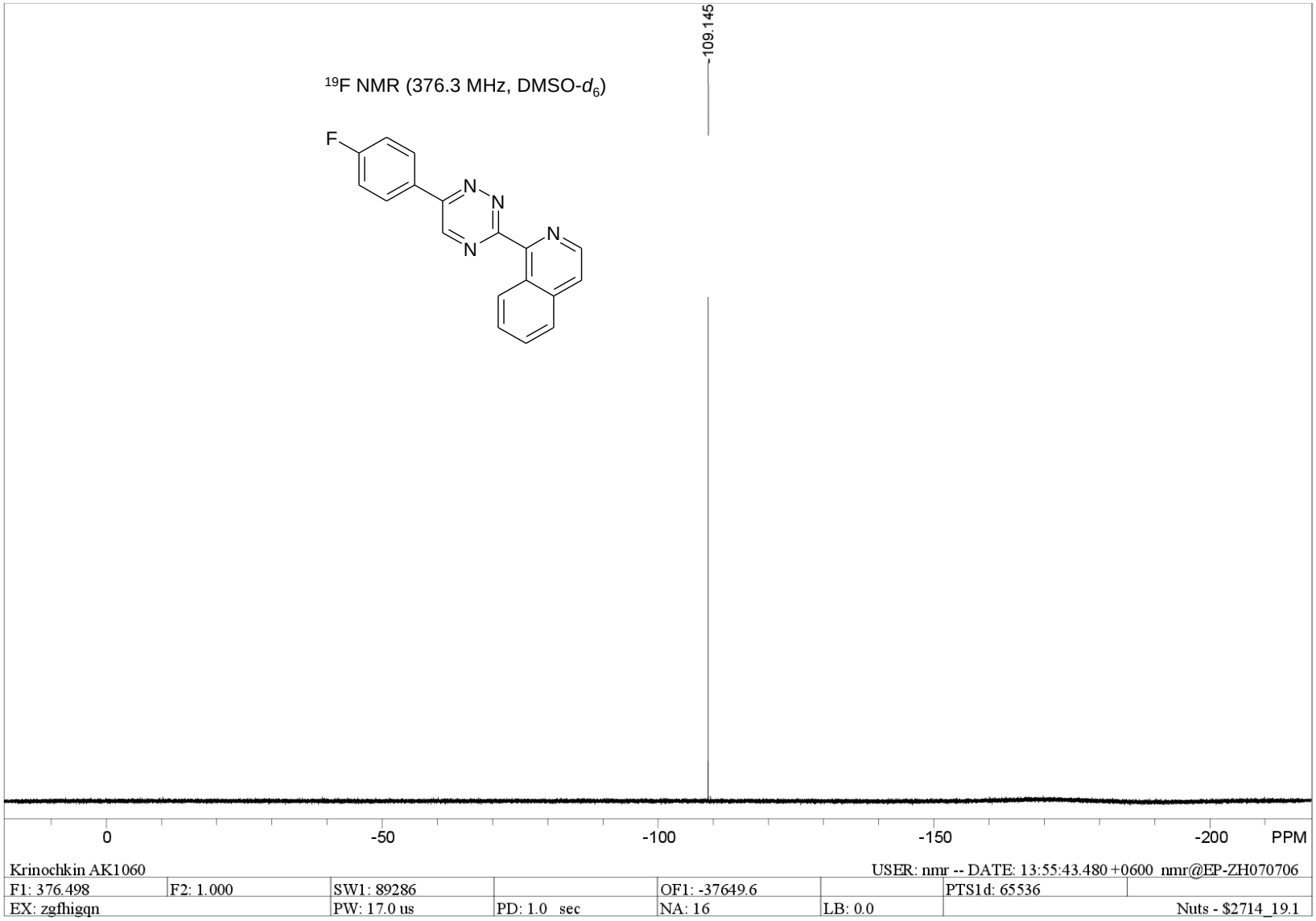
^{19}F NMR (376.3 MHz, DMSO-d_6)
F
N
N
N
N
-109.145
0
-50
-100
-150
-200
PPM
Krinochkin AK1060
USER: nmr -- DATE: 13:55:43.480 +0600 nmr@EP-ZH070706
F1: 376.498
F2: 1.000
SW1: 89286
OF1: -37649.6
PTS1d: 65536
EX: zgfhigqn
PW: 17.0 us
PD: 1.0 sec
NA: 16
LB: 0.0
Nuts - $2714_19.1

Figure S8

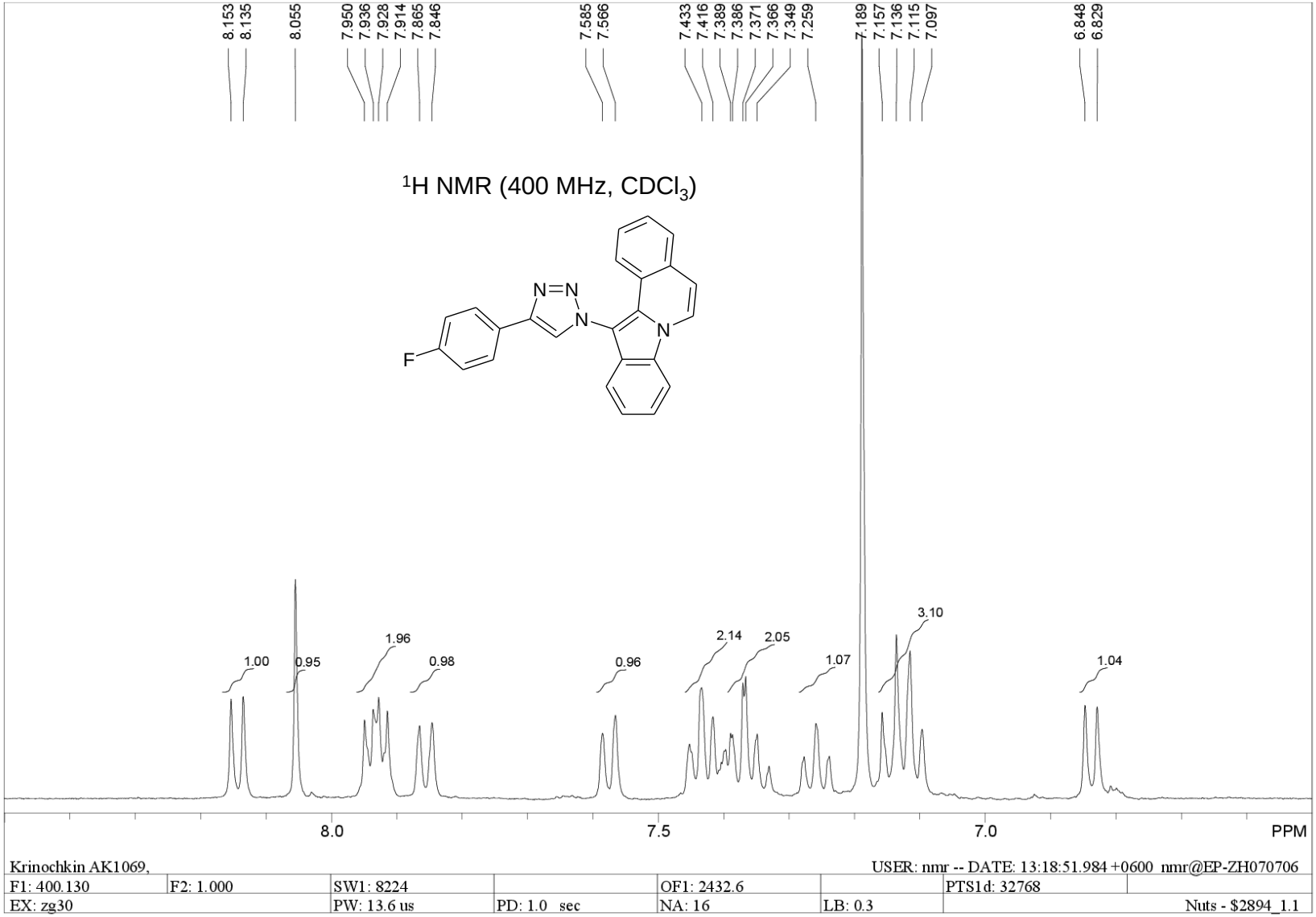
8.153
8.135
8.055
7.950
7.936
7.928
7.914
7.865
7.846
7.585
7.566
7.433
7.416
7.389
7.386
7.371
7.366
7.349
7.259
7.189
7.157
7.136
7.115
7.097
6.848
6.829
^{1}H NMR (400 MHz, $CDCl_3$)
N=N
N
N
F
1.00
0.95
1.96
0.98
0.96
2.14
2.05
1.07
3.10
1.04
8.0
7.5
7.0
PPM
Krinochkin AK1069,
USER: nmr -- DATE: 13:18:51.984 +0600 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2432.6
PTS1d: 32768
EX: zg30
PW: 13.6 us
PD: 1.0 sec
NA: 16
LB: 0.3
Nuts - $2894_1.1

Figure S9

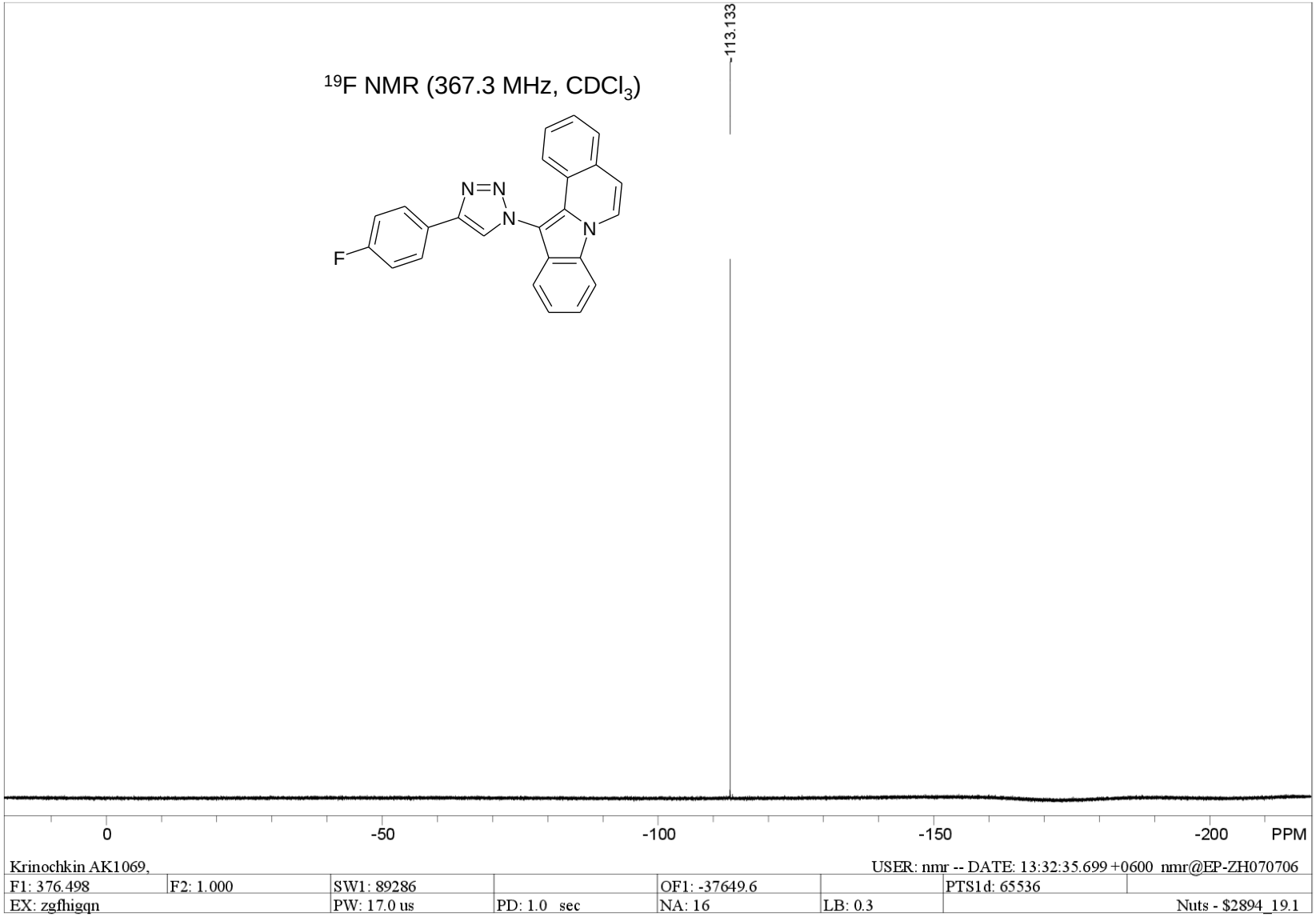
^{19}F NMR (367.3 MHz, $CDCl_3$)
-113.133
N=N
N
N
F
0
-50
-100
-150
-200
PPM
Krinochkin AK1069,
USER: nmr -- DATE: 13:32:35.699 +0600 nmr@EP-ZH070706
F1: 376.498
F2: 1.000
SW1: 89286
OF1: -37649.6
PTS1d: 65536
EX: zgfhigqn
PW: 17.0 us
PD: 1.0 sec
NA: 16
LB: 0.3
Nuts - $2894_19.1

Figure S10

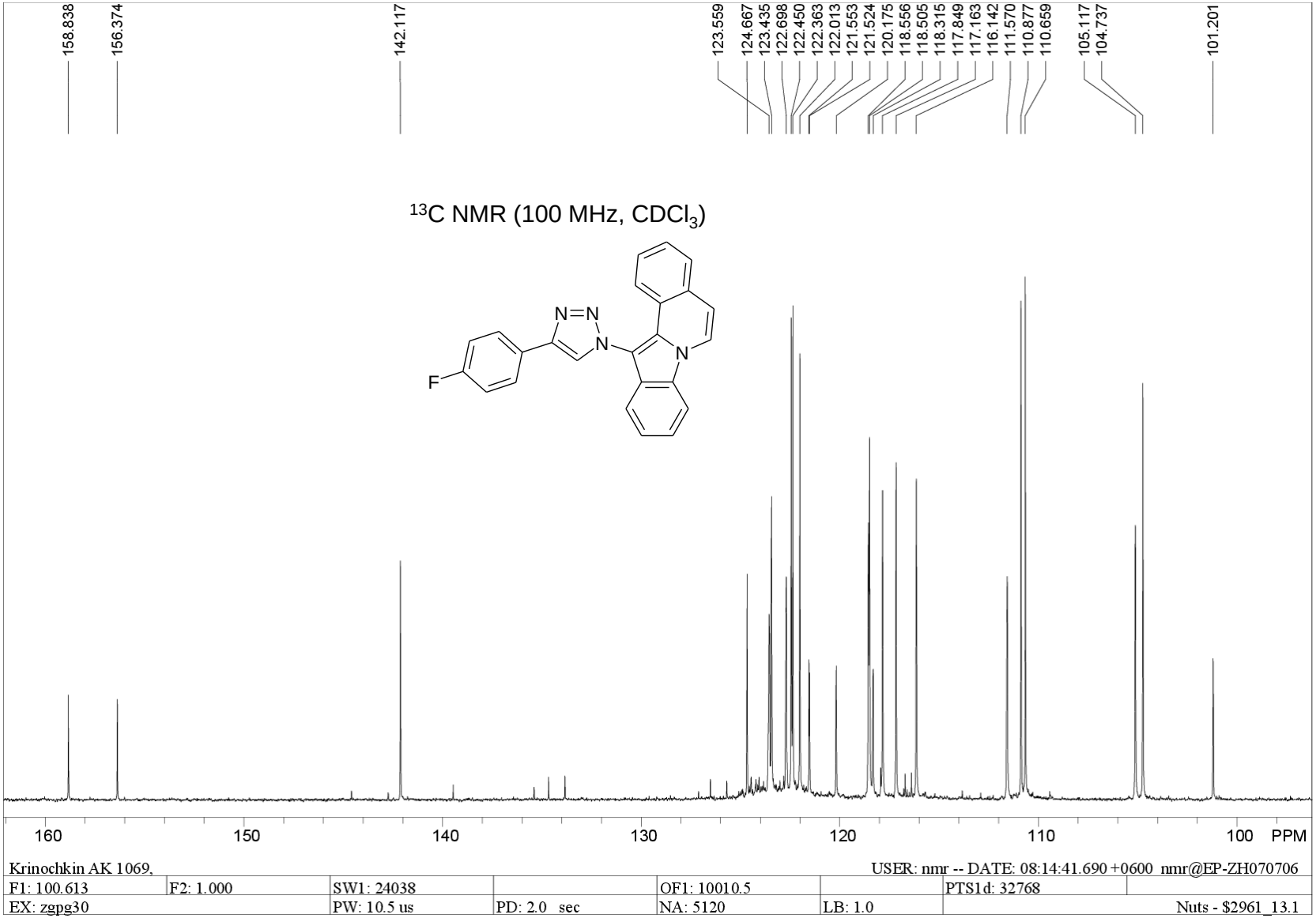
158.838
156.374
142.117
123.559
124.667
123.435
122.698
122.450
122.363
122.013
121.553
121.524
120.175
118.556
118.505
118.315
117.849
117.163
116.142
111.570
110.877
110.659
105.117
104.737
101.201
13C NMR (100 MHz, CDCl3)
N=N
N
N
F
160
150
140
130
120
110
100
PPM
Krinochkin AK 1069,
USER: nmr -- DATE: 08:14:41.690 +0600 nmr@EP-ZH070706
F1: 100.613
F2: 1.000
SW1: 24038
OF1: 10010.5
PTS1d: 32768
EX: zgpg30
PW: 10.5 us
PD: 2.0 sec
NA: 5120
LB: 1.0
Nuts - $2961_13.1

Figure S11

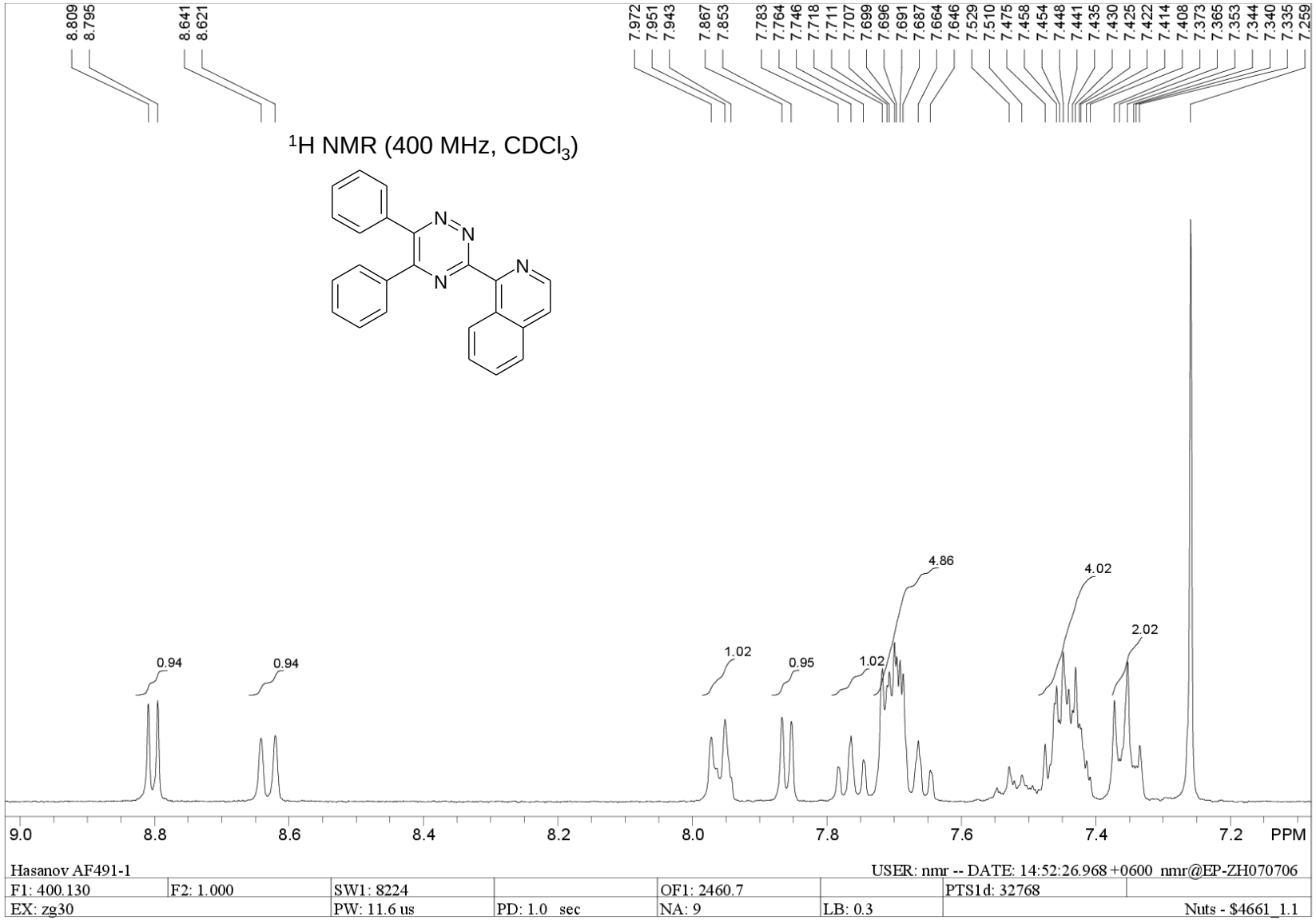
8.809
8.795
8.641
8.621
7.972
7.951
7.943
7.867
7.853
7.783
7.764
7.746
7.718
7.711
7.707
7.699
7.696
7.691
7.687
7.664
7.646
7.529
7.510
7.475
7.458
7.454
7.448
7.441
7.435
7.430
7.425
7.422
7.414
7.408
7.373
7.365
7.353
7.344
7.340
7.335
7.259
^{1}H NMR (400 MHz, $CDCl_3$)
N
N
N
N
0.94
0.94
1.02
0.95
1.02
4.86
4.02
2.02
9.0
8.8
8.6
8.4
8.2
8.0
7.8
7.6
7.4
7.2
PPM
Hasanov AF491-1
USER: nmr -- DATE: 14:52:26.968 +0600 nmr@EP-ZH070706
F1: 400.130
F2: 1.000
SW1: 8224
OF1: 2460.7
PTS1d: 32768
EX: zg30
PW: 11.6 us
PD: 1.0 sec
NA: 9
LB: 0.3
Nuts - $4661_1.1

Figure S12

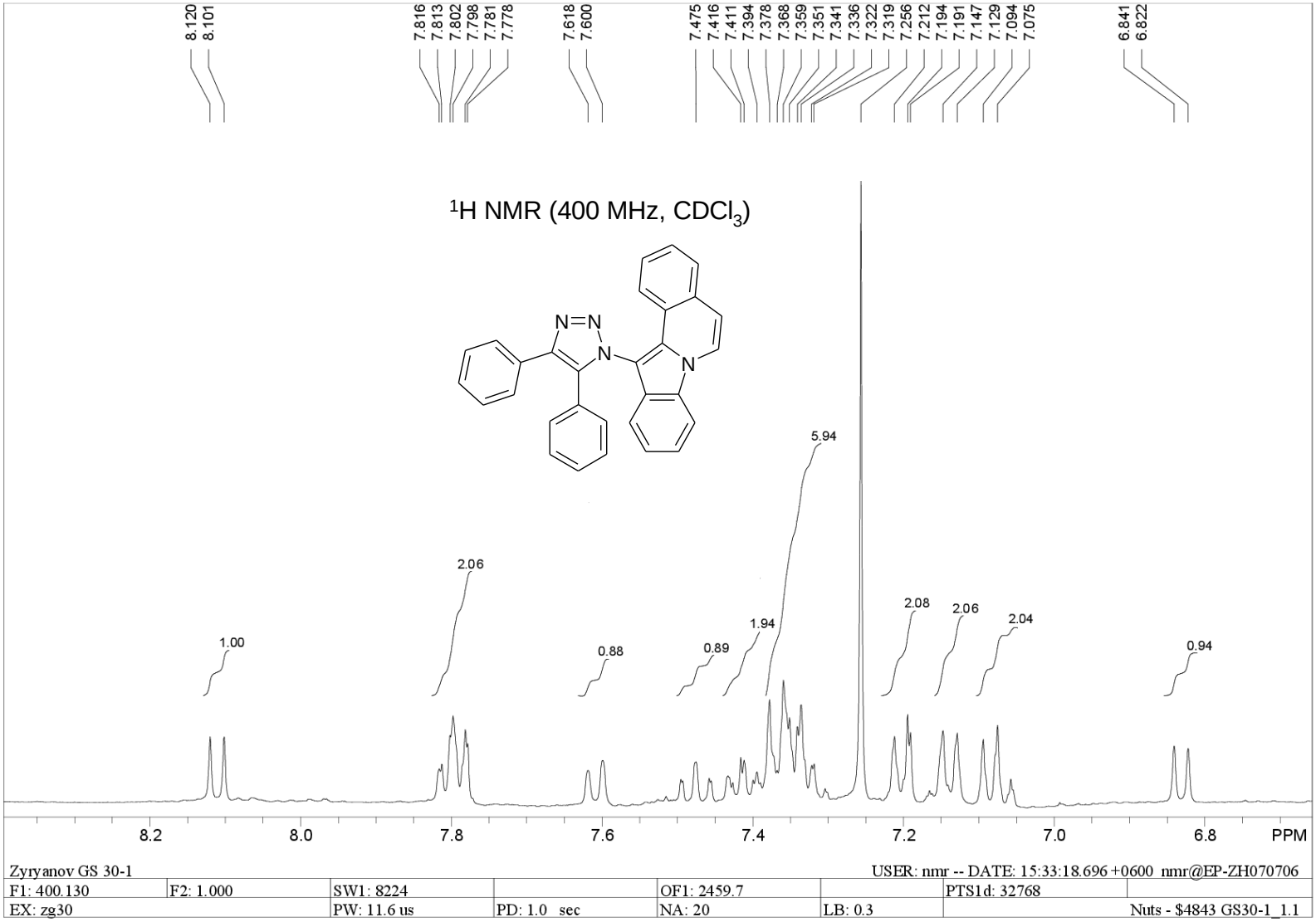

Figure S13