Electronic supplementary materials *Mendeleev Commun.*, 2022, **32**, 678–679

Synthesis of 7-oxindolylidene-3a,9a-diphenylimidazothiazolo[2,3-*c*][1,2,4]triazines by skeletal rearrangement of their [3,2-*b*]-fused isomers

Alexei N. Izmest'ev, Angelina N. Kravchenko and Galina A. Gazieva

Table S1 Rearrangement conditions and yields of products **4a-h**[a]

Entry	Product	R	NaOMe, equiv.	*t*/ min	Yield of compounds **4**, %
1	**4a**	H	0.5	30	83
2	**4b**	Me	1.0	30	90
3	**4c**	Et	1.0	15	74
4	**4d**	Pr^{i}	0.5	60	79
5	**4e**	Bu	1.0	30	77
6	**4f**	H_2C=$CHCH_2$	0.5	15	63
7	**4g**	HC≡CCH_2	0.5	15	79
8	**4h**	4$ClC_6H_4CH_2$	0.5	15	79

[a] Reaction conditions: refluxing compounds **1a-h** (0.5 mmol) and NaOMe (0.25-0.5 mmol) in MeOH (5 ml).

Experimental section

All standard reagents and methanol were purchased from Aldrich or Acros Organics and used without further purification. Melting points were determined on a Stuart SMP20 apparatus. IR spectra were recorded on a Bruker "Alpha" spectrophotometer in the range 400–4000 cm^{-1} (resolution: 2 cm^{-1}). ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AM-300 (300.13 and 75.47 MHz, respectively), Bruker DRX500 (125.76 MHz, (^{13}C)) and Bruker AV600 (150.90 MHz (^{13}C)) spectrometers and referenced to the residual solvent peak. The chemical shifts are reported in ppm (δ); multiplicities are indicated by s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet) and br (broad). Coupling constants, J, are reported in Hertz. High-resolution mass spectra (HRMS) were measured on the Bruker micrOTOF II instrument using electrospray ionization (ESI). The measurements were done in a positive ion mode (interface capillary voltage: 4500 V); mass range from m/z 50 to 3000 Da; external or internal calibration was done with Electrospray Calibrant Solution (Fluka). A syringe injection was used for solutions in MeCN or MeOH (flow rate 3 μL/min). N_2 was applied as a dry gas; interface temperature was set at 180 °C.

Synthesis of compounds 4a-h.

General Procedure

To a stirred suspension of compounds **1a–h** (0.5 mmol) in refluxing methanol (5 mL), NaOMe (30% solution in MeOH) was added (0.046 ml, 0.25 mmol for **1a,d,f-h**, 0.092 ml, 0.5 mmol for **1b,c,e**). The resulting mixture was refluxed with stirring for 15 min for **1c,f-h**, 30 min for **1a,b,e** and 1 h for **1d**. After cooling, the precipitate of compounds **4a–h** was filtered off, washed with methanol and dried at 50 °C.

(*Z*)-1,3-Dimethyl-7-(2-oxoindolin-3-ylidene)-3a,9a-diphenyl-1,3a,4,9a-tetrahydroimidazo[4,5-*e*]-thiazolo[2,3-*c*][1,2,4]triazine-2,8(3*H*,7*H*)-dione (4a). Yield 216 mg (83%) as an orange solid. Mp: >300 °C. IR (KBr), ν 3271 (NH), 3062 (Ar), 2959, 2879 (Alk), 1718, 1699, 1688 (C=N, C=O) cm^{-1}. ^{1}H NMR (300 MHz, DMSO-d_6) δ 2.63 (s, 3H, CH_3), 2.96 (s, 3H, CH_3), 6.72 (br.s, 3H, Ph), 6.91-6.96 (m, 2H, 5'-H, 7'-H), 7.15 (br.s, 3H, Ph), 7.23-7.32 (m, 5H, 6'-H, Ph), 8.39 (s, 1H, 4-NH), 8.47 (d, *J* = 8.0 Hz, 1H, 4'-H), 11.15 (br.s, 1H, 1'-NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ 25.52, 30.89 ($2CH_3$), 81.44, 86.58 (C-3a, C-9a), 110.09 (C-7'), 120.15 (C-3a'), 121.57 (C-5'), 123.07 (C-3'), 127.14, 127.65, 127.93, 128.40, 128.85 (C-4', 2Ph-2-6), 130.83, 131.68, 132.48, 132.80 (C-7, C-6', 2Ph-1), 138.75 (C=N), 142.46 (C-7a'), 158.11 (2-C=O), 163.35 (8-C=O), 168.63 (2'-C=O). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $[C_{28}H_{22}N_6O_3S + H]^+$: 523.1547; found: 523.1543.

(*Z*)-1,3-Dimethyl-7-(1-methyl-2-oxoindolin-3-ylidene)-3a,9a-diphenyl-1,3a,4,9a-tetrahydroimidazo-[4,5-*e*]thiazolo[2,3-*c*][1,2,4]triazine-2,8(3*H*,7*H*)-dione (4b). Yield 241 mg (90%) as an orange solid. Mp: 253-256 °C. IR (KBr), ν 3297 (NH), 3063 (Ar), 2946 (Alk), 1724, 1709, 1678 (C=N, C=O) cm^{-1}. ^{1}H NMR (300 MHz, DMSO-d_6) δ 2.64 (s, 3H, CH_3), 2.97 (s, 3H, CH_3), 3.28 (s, 3H, 1'-NCH_3), 6.73 (br.s, 3H, Ph), 7.00 (t, *J* = 7.6 Hz, 1H, 5'-H), 7.09-7.26 (m, 8H, 7'-H, Ph), 7.38 (t, *J* = 7.5 Hz, 1H, 6'-H), 8.43 (s, 1H, NH), 8.50 (d, *J* = 7.5 Hz, 1H, 4'-H). ^{13}C NMR (150 MHz, DMSO-d_6): δ 25.66, 26.31, 31.03 ($3CH_3$), 81.52, 86.64 (C-3a, C-9a), 108.87 (C-7'), 119.46 (C-3a'), 122.13, 122.20 (C-3', C-5'), 126.98, 127.23, 128.07, 128.54, 128.99 (C-4', 2Ph-2-6), 130.79, 131.74, 132.85, 133.29 (C-7, C-6', 2Ph-1), 138.60 (C=N), 143.43 (C-7a'), 158.24 (2-C=O), 163.25 (8-C=O), 167.09 (2'-C=O). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $[C_{29}H_{24}N_6O_3S + H]^+$: 537.1703; found: 537.1696.

(*Z*)-7-(1-Ethyl-2-oxoindolin-3-ylidene)-1,3-dimethyl-3a,9a-diphenyl-1,3a,4,9a-tetrahydroimidazo-[4,5-*e*]thiazolo[2,3-*c*][1,2,4]triazine-2,8(3*H*,7*H*)-dione (4c). Yield 203 mg (74%) as an orange solid. Mp: 237-239 °C. IR (KBr), ν 3468, 3253 (NH), 3112, 3086, 3060, 3032 (Ar), 2985, 2959, 2938, 2880 (Alk),

1716, 1686, 1647 (C=N, C=O) cm^{-1}. ^1H NMR (300 MHz, DMSO-d_6) δ 1.21 (t, J = 7.0 Hz, 3H, CH_3), 2.64 (s, 3H, CH_3), 2.96 (s, 3H, CH_3), 3.87 (q, J = 7.1 Hz, 2H, 1'-NCH_2), 6.72 (br.s, 3H, Ph), 7.00 (t, J = 7.6 Hz, 1H, 5'-H), 7.16-7.26 (m, 8H, 7'-H, Ph), 7.38 (t, J = 7.6 Hz, 1H, 6'-H), 8.42 (s, 1H, NH), 8.53 (d, J = 8.0 Hz, 1H, 4'-H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 12.59 (CH_3), 25.52, 30.88 ($2NCH_3$), 34.42 (1'-NCH_2), 81.43, 86.62 (C-3a, C-9a), 108.83 (C-7'), 119.59 (C-3a'), 122.00, 122.11 (C-3', C-5'), 127.15, 127.61, 127.93, 128.41, 128.85 (C-4', 2Ph-2-6), 130.74, 131.64, 132.77, 133.33 (C-7, C-6', 2Ph-1), 138.50 (C=N), 142.29 (C-7a'), 158.10 (2-C=O), 163.16 (8-C=O), 166.71 (2'-C=O). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $[C_{30}H_{26}N_6O_3S + H]^+$: 551.1860; found: 551.1863.

(*Z*)-7-(1-Isopropyl-2-oxoindolin-3-ylidene)-1,3-dimethyl-3a,9a-diphenyl-1,3a,4,9a-tetrahydro-imidazo[4,5-*e*]thiazolo[2,3-*c*][1,2,4]triazine-2,8(3*H*,7*H*)-dione (4d). Yield 223 mg (79%) as an orange solid. Mp: 243-245 °C (decomp). IR (KBr), ν 3435, 3254 (NH), 3058, 3037 (Ar), 2979, 2936, 2879 (Alk), 1716, 1694, 1642, 1602 (C=N, C=O) cm^{-1}. ^1H NMR (300 MHz, DMSO-d_6) δ 1.48 (d, J = 6.7 Hz, 6H, $2CH_3$), 2.64 (s, 3H, CH_3), 2.96 (s, 3H, CH_3), 4.58-4.67 (m, 1H, 1'-NCH), 6.73 (br.s, 3H, Ph), 6.99 (t, J = 7.5 Hz, 1H, 5'-H), 7.15 (br.s, 3H, Ph), 7.21-7.29 (m, 5H, 7'-H, Ph), 7.36 (t, J = 7.5 Hz, 1H, 6'-H), 8.42 (s, 1H, NH), 8.57 (d, J = 7.7 Hz, 1H, 4'-H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 19.17 ($2CH_3$), 25.52, 30.89 ($2NCH_3$), 44.11 (1'-NCH_2), 81.43, 86.60 (C-3a, C-9a), 109.76 (C-7'), 119.76 (C-3a'), 121.76, 122.36 (C-3', C-5'), 127.13, 127.26, 127.67, 127.94, 128.41, 128.86 (C-4', 2Ph-2-6), 130.67, 131.67, 12.78, 133.21 (C-7, C-6', 2Ph-1), 138.66 (C=N), 142.13 (C-7a'), 158.11 (2-C=O), 163.21 (8-C=O), 166.87 (2'-C=O). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $[C_{31}H_{28}N_6O_3S + H]^+$: 565.2016; found: 565.2009.

(*Z*)-7-(1-Butyl-2-oxoindolin-3-ylidene)-1,3-dimethyl-3a,9a-diphenyl-1,3a,4,9a-tetrahydroimidazo-[4,5-*e*]thiazolo[2,3-*c*][1,2,4]triazine-2,8(3*H*,7*H*)-dione (4e). Yield 223 mg (77%) as an bright orange solid. Mp: 236-238 °C (decomp). IR (KBr), ν 3494, 3229 (NH), 3057 (Ar), 2935, 2873 (Alk), 1718, 1688, 1648, 1608 (C=N, C=O) cm^{-1}. ^1H NMR (300 MHz, DMSO-d_6) δ 0.92 (t, J = 7.2 Hz, 3H, CH_3), 1.28-1.35 (m, 2H, CH_2), 1.57-1.67 (m, 2H, CH_2), 2.64 (s, 3H, CH_3), 2.96 (s, 3H, CH_3), 3.83 (q, J = 6.6 Hz, 2H, 1'-NCH_2), 6.723 (br.s, 3H, Ph), 7.00 (t, J = 7.7 Hz, 1H, 5'-H), 7.12-7.26 (m, 8H, 7'-H, Ph), 7.37 (t, J = 7.6 Hz, 1H, 6'-H), 8.42 (s, 1H, NH), 8.53 (d, J = 7.8 Hz, 1H, 4'-H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 13.43 (CH_3), 19.37 (CH_2), 25.48, 29.06, 30.85 ($2NCH_3$, CH_2), 39.25 (1'-NCH_2), 81.41, 86.60 (C-3a, C-9a), 108.88 (C-7'), 119.51 (C-3a'), 121.93 (C-3', C-5'), 127.23, 127.59, 127.88, 128.36, 128.81, 129.87 (C-4', 2Ph-2-6), 130.65, 131.64, 132.77, 133.38 (C-7, C-6', 2Ph-1), 138.48 (C=N), 142.62 (C-7a'), 158.07 (2-C=O), 163.14 (8-C=O), 167.03 (2'-C=O). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $[C_{32}H_{30}N_6O_3S + H]^+$: 579.2173; found: 579.2165.

(*Z*)-7-(1-Allyl-2-oxoindolin-3-ylidene)-1,3-dimethyl-3a,9a-diphenyl-1,3a,4,9a-tetrahydroimidazo[4,5-*e*]-thiazolo[2,3-*c*][1,2,4]triazine-2,8(3*H*,7*H*)-dione (4f). Yield 177 mg (63%) as a bright scarlet powder. Mp: 208-210 °C. [Lit. S1: yield 55%, scarlet powder, mp 181–183.5 °C].

(*Z*)-1,3-Dimethyl-7-(2-oxo-1-(prop-2-yn-1-yl)indolin-3-ylidene)-3a,9a-diphenyl-1,3a,4,9a-tetrahydroimidazo[4,5-*e*]thiazolo[2,3-*c*][1,2,4]triazine-2,8(3*H*,7*H*)-dione (4g). Yield 221 mg (79%) as a bright scarlet powder. Mp: 238-240 °C. IR (KBr), ν 3412, 3272 (NH), 3058, 3030 (Ar), 2947 (Alk), 1704, 1686, 1639, 1605 (C=N, C=O) cm^{-1}. ^1H NMR (300 MHz, DMSO-d_6) δ 2.64 (s, 3H, NCH_3), 2.97 (s, 3H, CH_3), 3.31 (s, 1H, ≡CH), 4.71 (s, 1H, 1'-NCH_2), 6.72 (br.s, 3H, Ph), 7.06 (t, J = 7.7 Hz, 1H, 5'-H), 7.14 (br.s, 3H, Ph), 7.19-7.28 (m, 5H, 7'-H, Ph), 7.43 (t, J = 7.6 Hz, 1H, 6'-H), 8.46 (s, 1H, NH), 8.55 (d, J = 7.9 Hz, 1H, 4'-H). ^{13}C NMR (150 MHz, DMSO-d_6): δ 25.67, 29.18,31.01 ($2NCH_3$, 1'-NCH_2), 74.67, 77.76 (C≡CH), 81.52, 86.74 (C-3a, C-9a), 109.54 (C-7'), 119.72 (C-3a'), 121.44, 122.68 (C-3', C-5'), 127.23, 128.08, 128.55, 129.00 (C-4', 2Ph-2-6), 130.74, 131.68, 132.82, 134.64 (C-7, C-6', 2Ph-1), 138.42 (C=N),

141.43 (C-7a'), 158.21 (2-C=O), 163.12 (8-C=O), 166.51 (2'-C=O). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $[C_{31}H_{24}N_6O_3S + H]^+$: 561.1703; found: 561.1685.

(*Z*)-7-(1-(4-Chlorobenzyl)-2-oxoindolin-3-ylidene)-1,3-dimethyl-3a,9a-diphenyl-1,3a,4,9a-tetrahydroimidazo[4,5-*e*]thiazolo[2,3-*c*][1,2,4]triazine-2,8(3*H*,7*H*)-dione (4h). Yield 255 mg (79%) as an orange solid. Mp: 256-258 °C. IR (KBr), ν 3443, 3275 (NH), 1696, 1647, 1607 (C=N, C=O) cm^{-1}. ^{1}H NMR (300 MHz, DMSO-d_6) δ 2.62 (s, 3H, CH_3), 2.95 (s, 3H, CH_3), 5.06 (s, 2H, 1'-NCH_2), 6.71 (br.s, 3H, Ph), 6.96-7.04 (m, 2H, 5'-H, 7'-H), 7.12 (br.s, 3H, Ph), 7.19-7.29 (m, 5H, 6'-H, Ph), 7.33 (d, *J* = 8.7 Hz, 2H, Ar), 7.39 (d, *J* = 8.4 Hz, 2H, Ar), 8.43 (s, 1H, NH), 8.53 (d, *J* = 7.9 Hz, 1H, 4'-H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 25.52, 30.89 ($2CH_3$), 42.28 (1'-NCH_2), 81.44, 86.71 (C-3a, C-9a), 109.31 (C-7'), 119.70 (C-3a'), 121.69, 122.37 (C-3', C-5'), 127.17, 127.64, 127.87, 127.94, 128.43, 128.58, 128.87, 129.04, 129.91, 130.62, 131.60, 132.09, 132.74, 134.18, 135.06 (C-4', C-6', C-7, Ar, 2Ph), 138.38 (C=N), 142.09 (C-7a'), 158.10 (2-C=O), 163.12 (8-C=O), 167.30 (2'-C=O). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $[C_{35}H_{27}ClN_6O_3S + H]^+$: 647.1627; found: 647.1612.

Procedure for the synthesis of compound 5.

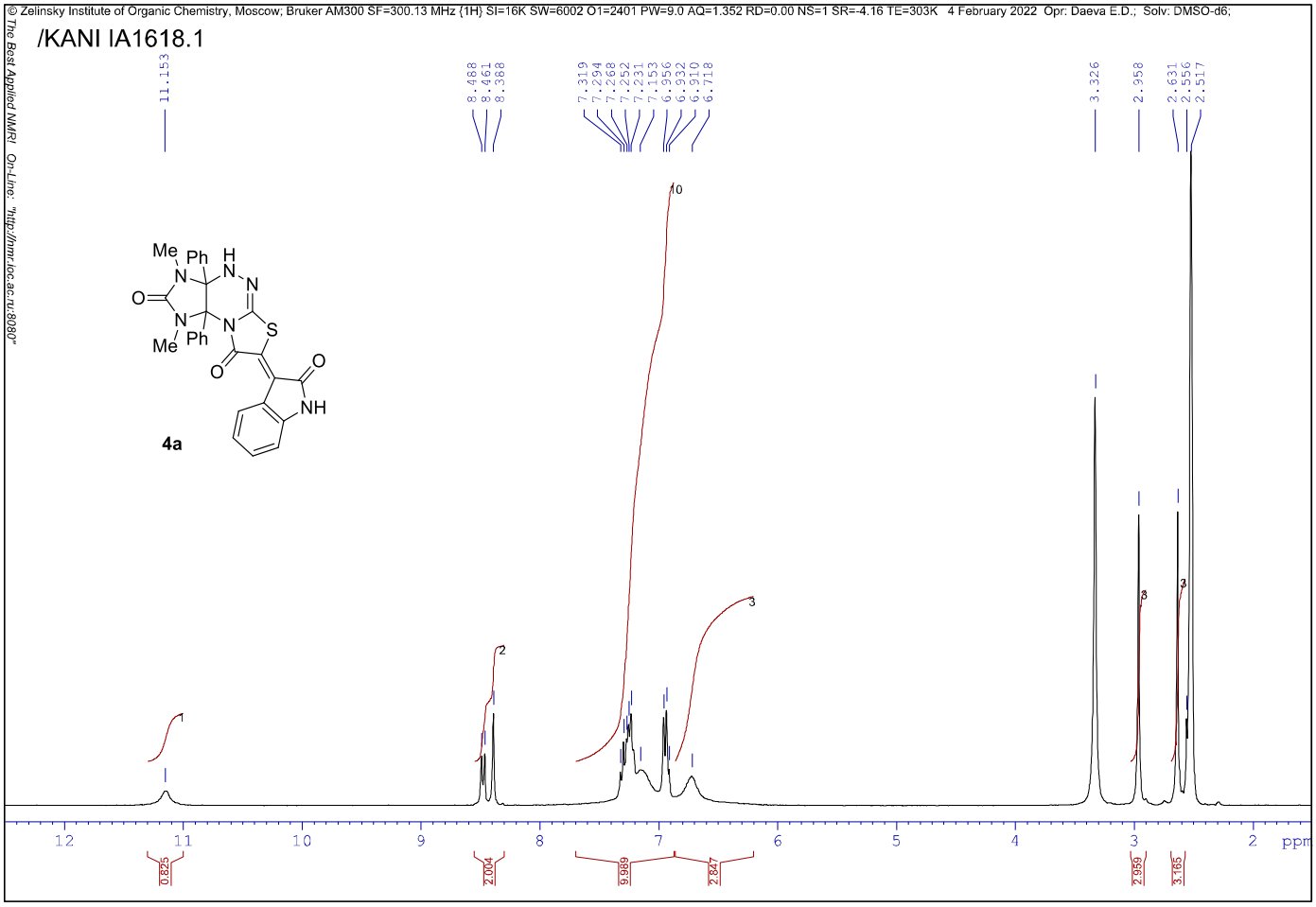
To a stirred suspension of imidazothiazolotriazine **2** (0.5 mmol) in refluxing methanol (5 mL), NaOMe (30% solution in MeOH, 0.092 ml) was added. The resulting mixture was refluxed with stirring for 1 h. After standing until the methanol had evaporated, the precipitate of compound **5** was filtered off, washed with methanol and dried at 50 °C.

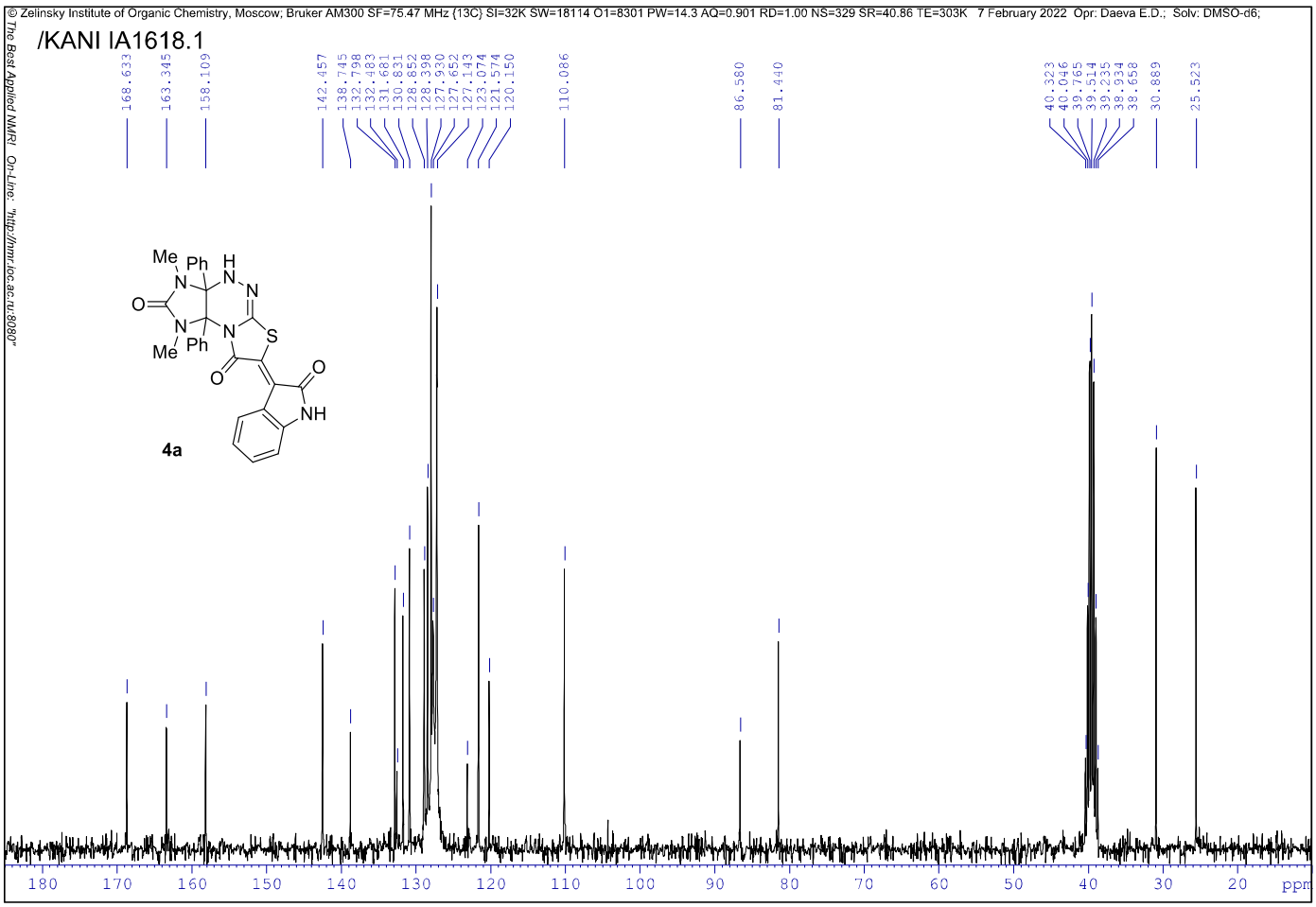
Sodium 2-((5,7-dimethyl-6-oxo-4a,7a-diphenyl-4,4a,5,6,7,7a-hexahydro-1*H*-imidazo[4,5-*e*]-[1,2,4]triazin-3-yl)thio)acetate (5). Yield 156 mg (72%) as a white solid. Mp: 256-259 (decomp) °C. IR (KBr), ν 3323 (NH), 3079, 3057, 3025 (Ph), 2862 (Alk), 1688 (C=N, C=O) cm^{-1}. ^{1}H NMR (300 MHz, DMSO-d_6) δ 2.56 (s, 6H, $2CH_3$), 3.09 (d, *J* = 14.8 Hz, 1H, CH_2), 3.20 (d, *J* = 14.8 Hz, 1H, CH_2), 6.80 (s, 1H, NH), 6.85 (d, *J* = 7.8 Hz, 2H, Ph), 7.00-7.12 (m, 8H, Ph), 10.38 (s, 1H, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ 25.25, 25.53 ($2CH_3$), 36.86 (CH_2), 81.29, 83.09 (C-3a, C-9a), 126.95, 127.40, 127.72, 128.20, 128.43, 129.91 (2Ph-2,6), 136.14, 137.32 (2Ph-1), 147.50 (C=N), 158.28 (6-C=O), 172.99 (COONa). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $[C_{20}H_{20}N_5NaO_3S + H]^+$: 434.1226; found: 434.1258.

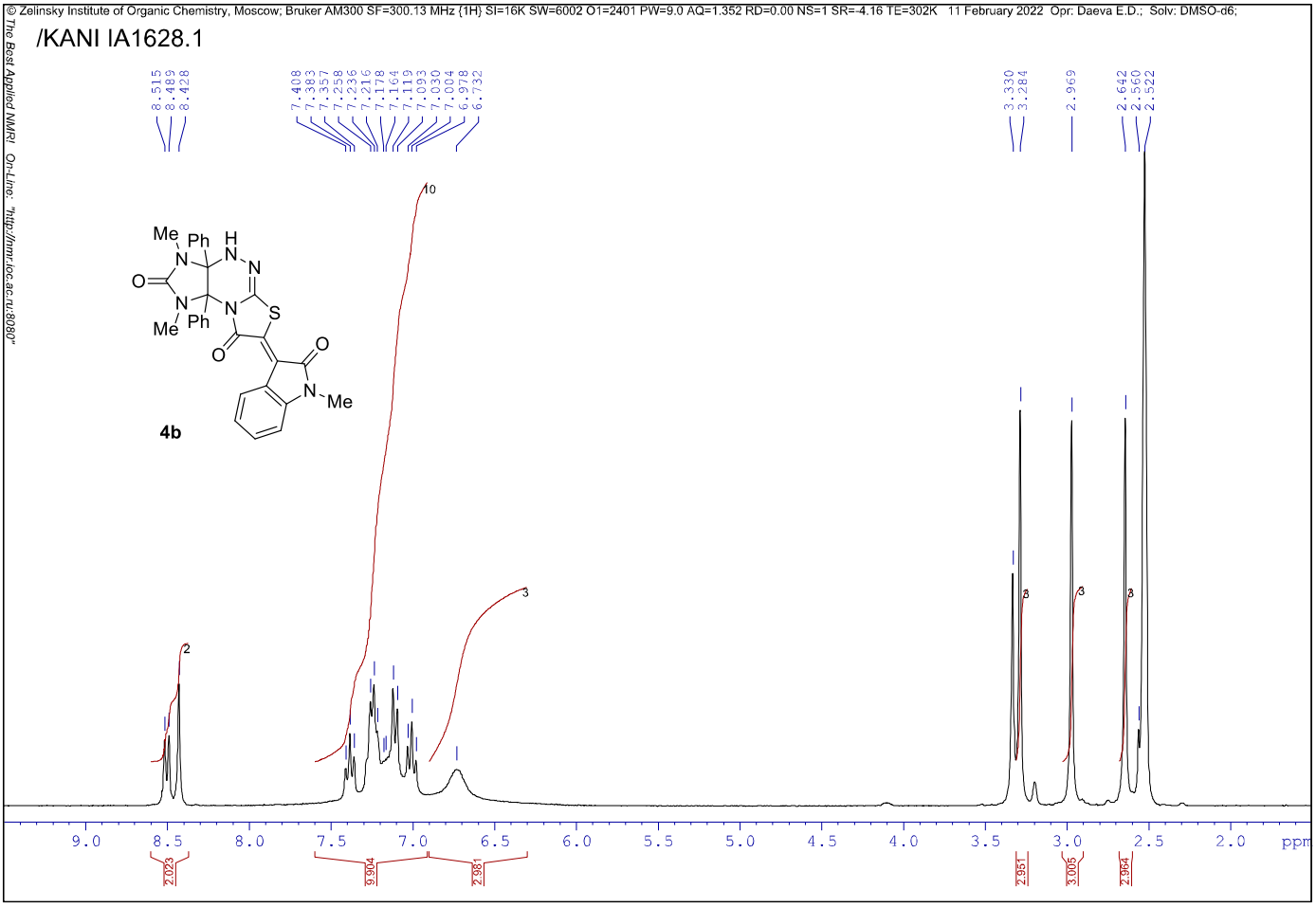
Procedure for the synthesis of acid 6.

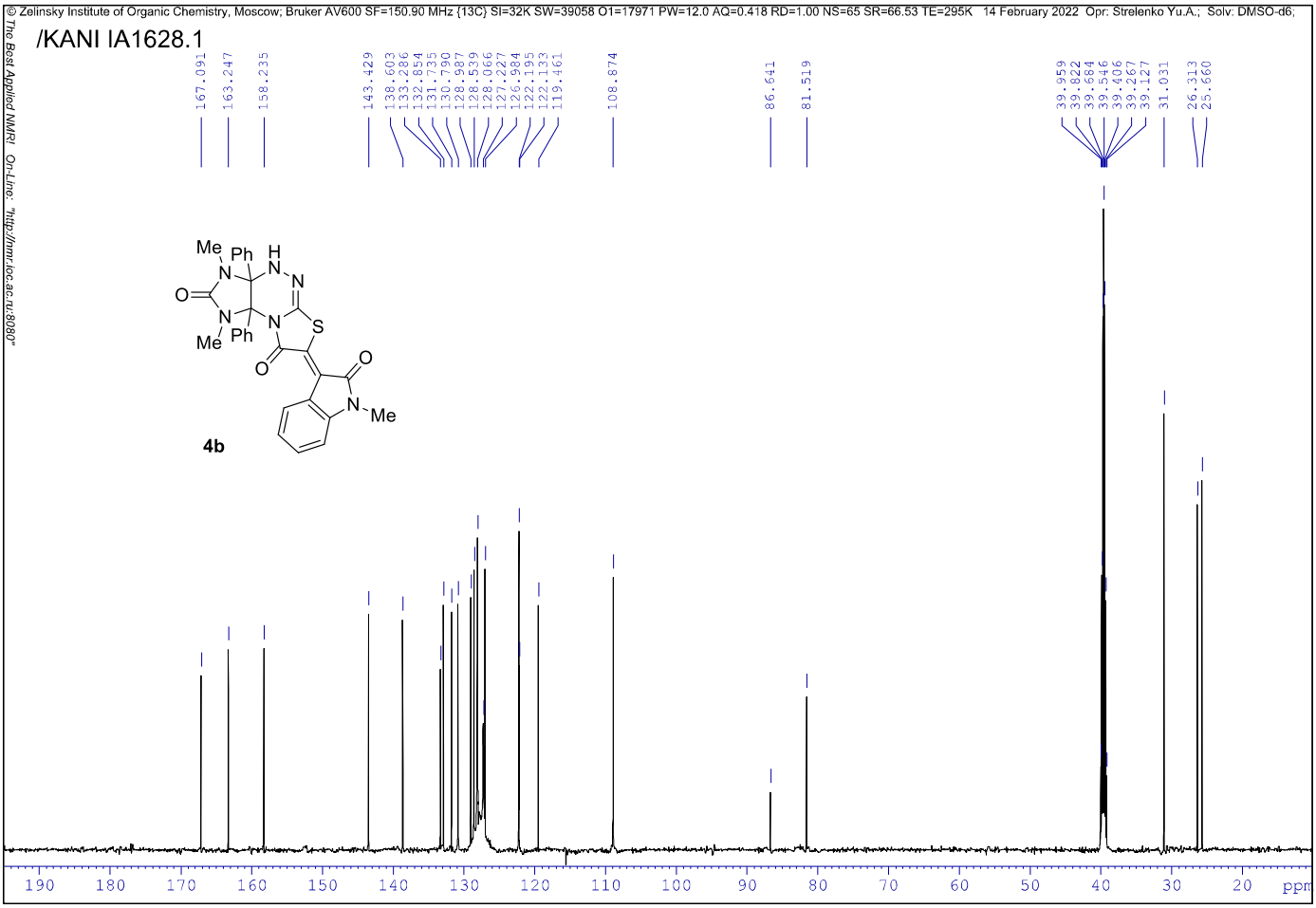
To a stirred solution of compound **5** (0.25 mmol) in water (5 mL), HCl was added to pH 3–4. The precipitate of acid **6** was filtered off, washed with water and dried at 50 °C.

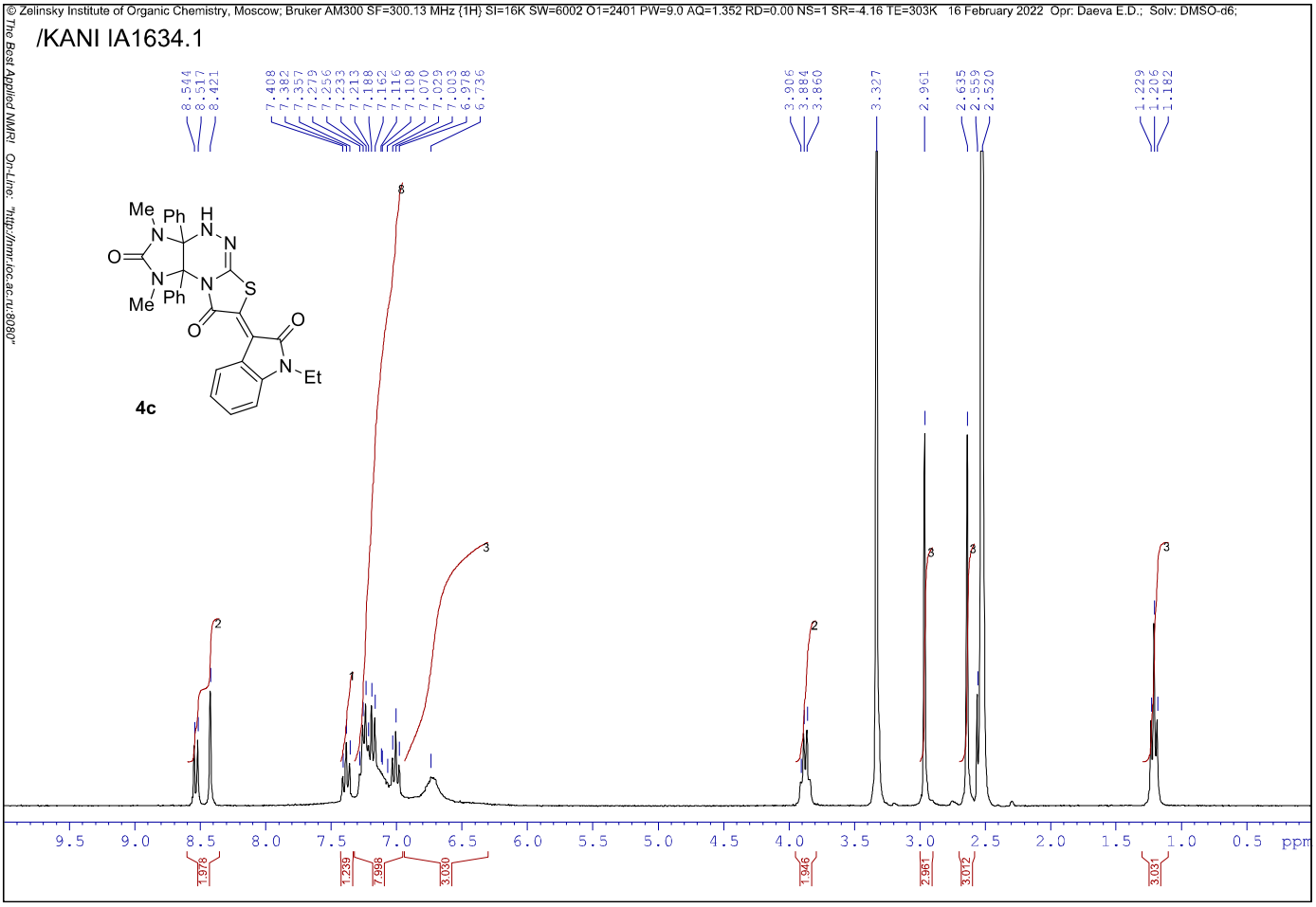
2-((5,7-Dimethyl-6-oxo-4a,7a-diphenyl-4,4a,5,6,7,7a-hexahydro-1*H*-imidazo[4,5-*e*][1,2,4]triazin-3-yl)thio)acetic acid (6) [S2]. Yield 91 mg (89%) as a white powder. Mp: 214-217 °C. [Lit.S2: yield 85-90%, white powder, mp 243–245 °C]. HRMS (ESI): *m/z* $[M + H]^+$ calcd for $[C_{20}H_{21}N_5O_3S + H]^+$: 412.1438; found: 412.1429.

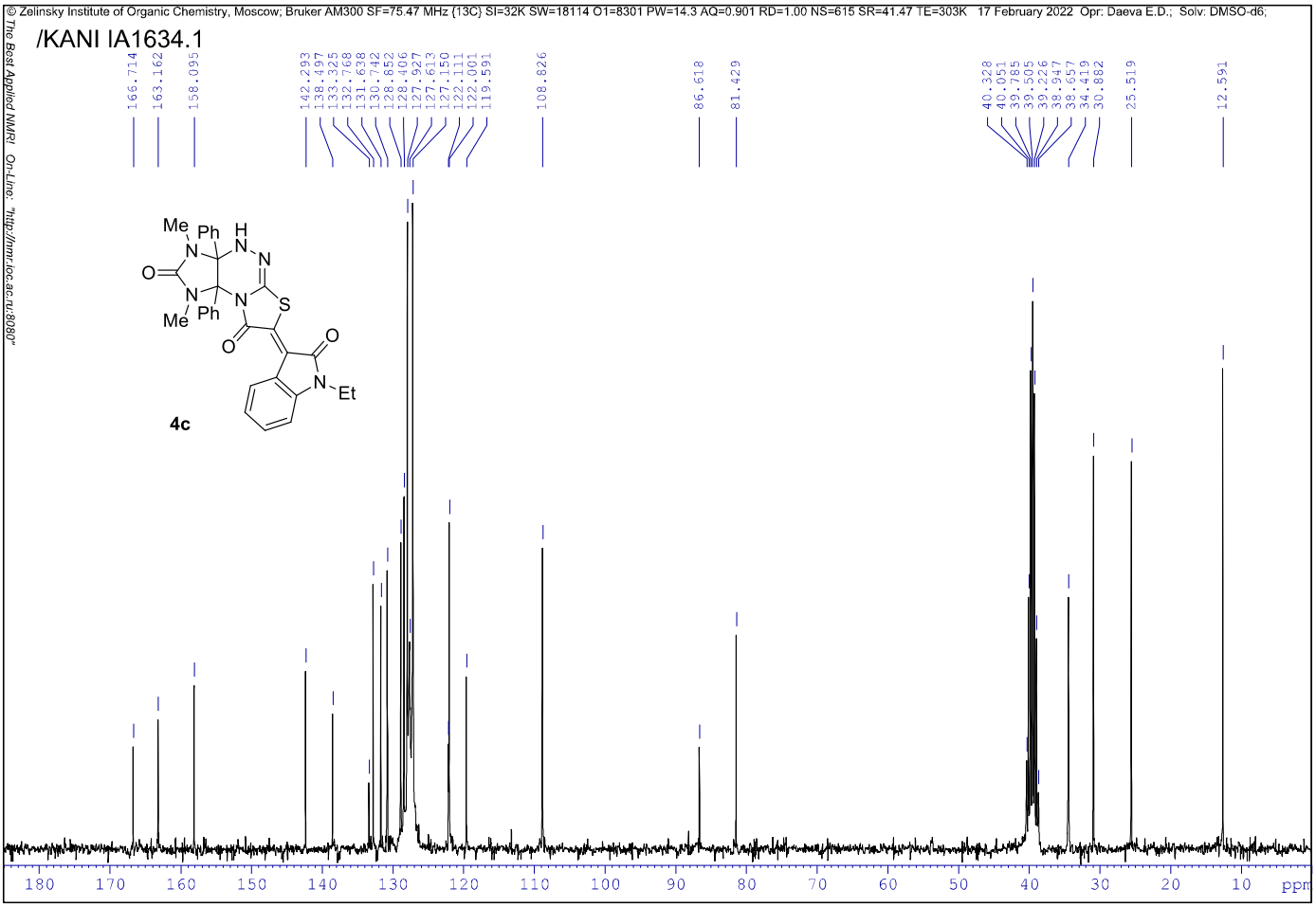

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6002 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=1 SR=-4.16 TE=303K 4 February 2022 Opr: Daeva E.D.; Solv: DMSO-d6;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1618.1
11.153
8.488
8.461
8.388
7.319
7.294
7.268
7.252
7.231
7.153
6.956
6.932
6.910
6.718
3.326
2.958
2.631
2.556
2.517
4a
ppm
0.825
2.004
9.989
2.847
2.959
3.165

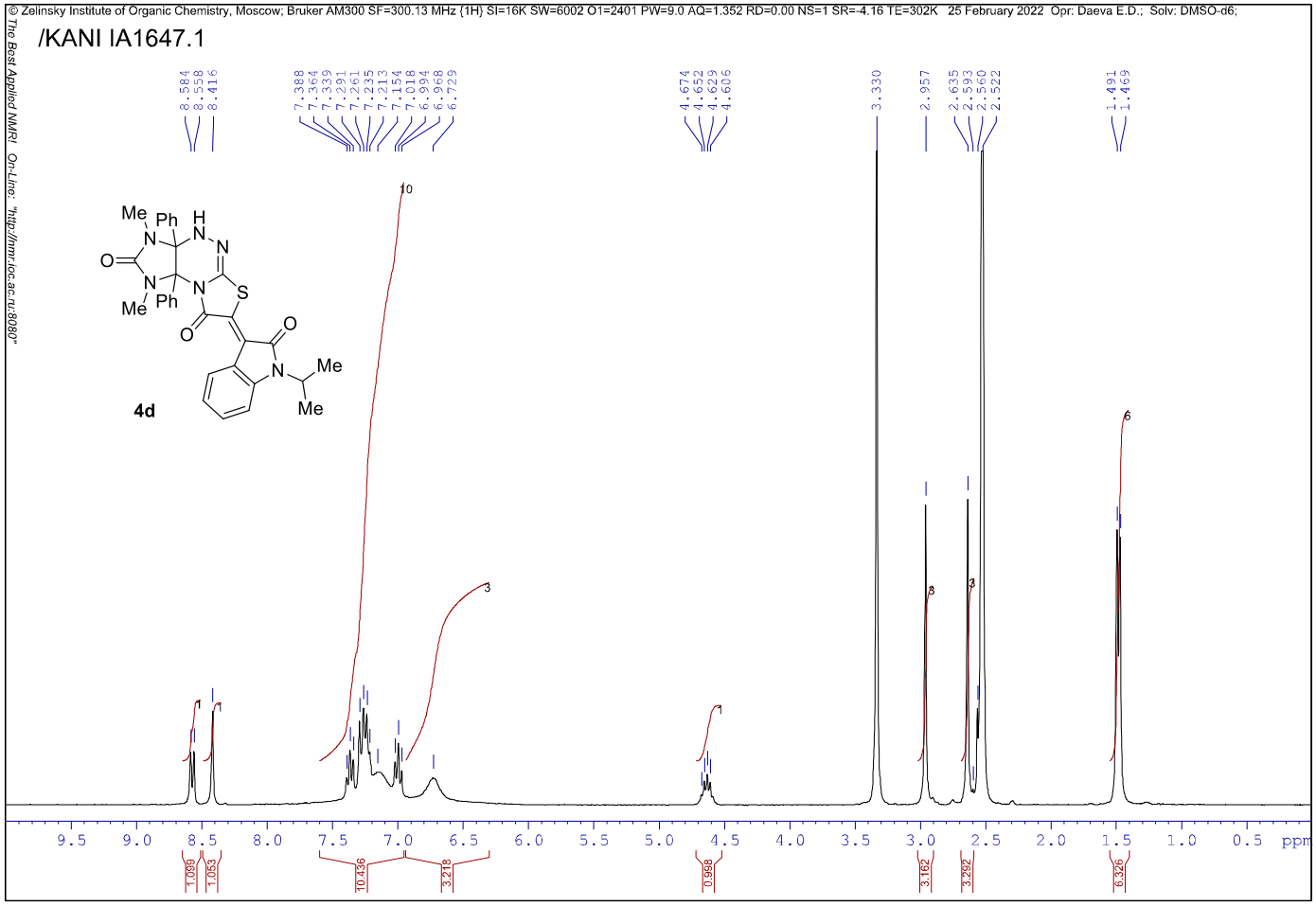

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=14.3 AQ=0.901 RD=1.00 NS=329 SR=40.86 TE=303K 7 February 2022 Opr: Daeva E.D.; Solv: DMSO-d6;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1618.1
168.633
163.345
158.109
142.457
138.745
132.798
132.483
131.681
130.831
128.852
128.398
127.930
127.652
127.143
123.074
121.574
120.150
110.086
86.580
81.440
40.323
40.046
39.765
39.514
39.235
38.934
38.658
30.889
25.523
4a
ppm

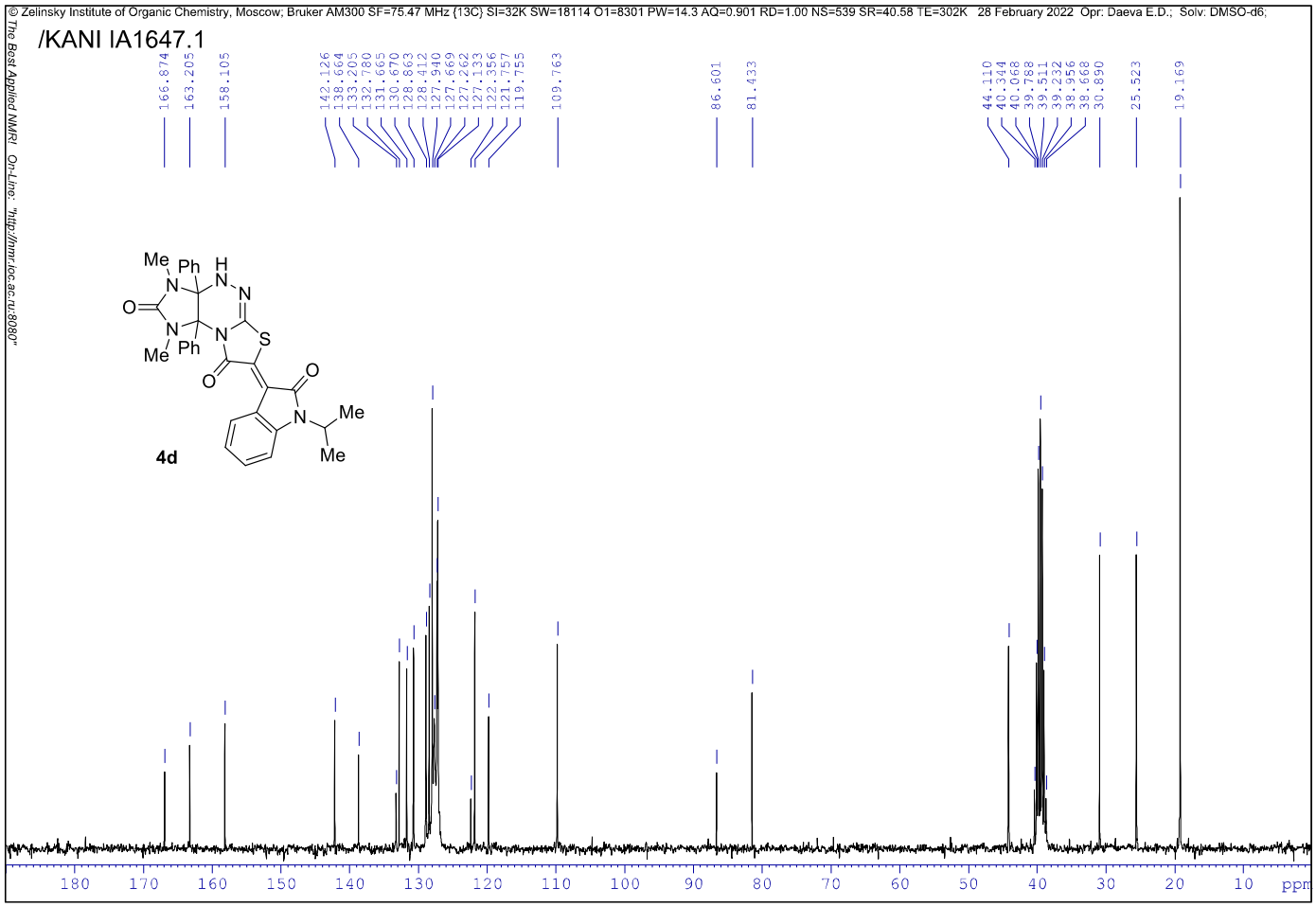

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6002 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=1 SR=-4.16 TE=302K 11 February 2022 Opr: Daeva E.D.; Solv: DMSO-d6;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1628.1
8.515
8.489
8.428
7.408
7.383
7.357
7.258
7.236
7.216
7.178
7.164
7.119
7.093
7.030
7.004
6.978
6.732
3.330
3.284
2.969
2.642
2.560
2.522
4b
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
ppm
2.023
9.304
2.381
2.951
3.005
2.964

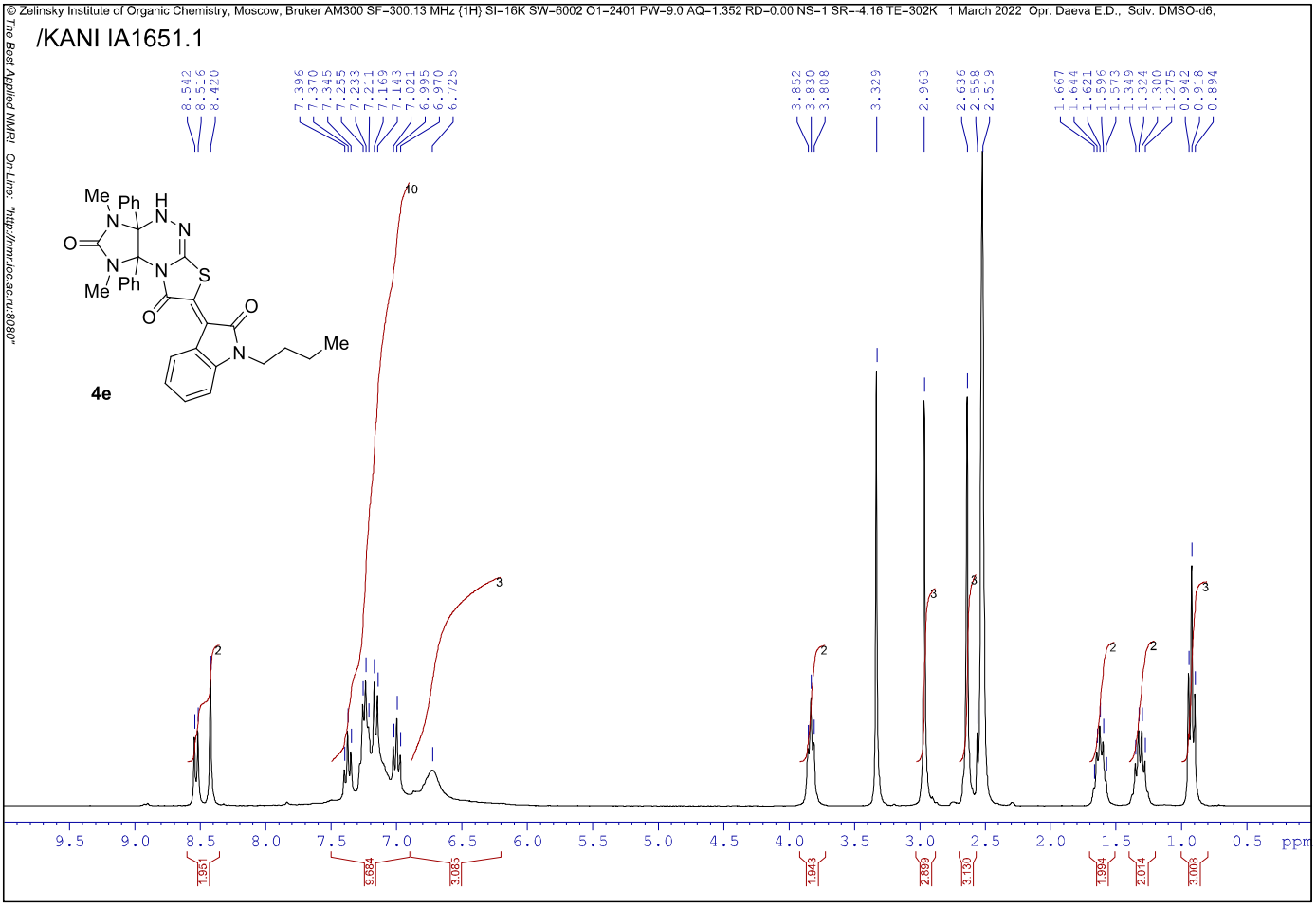

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AV600 SF=150.90 MHz {13C} SI=32K SW=39058 O1=17971 PW=12.0 AQ=0.418 RD=1.00 NS=65 SR=66.53 TE=295K 14 February 2022 Opr: Strelenko Yu.A.; Solv: DMSO-d6;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1628.1
167.091
163.247
158.235
143.429
138.603
133.286
132.654
131.735
130.790
128.987
128.539
128.066
127.227
126.984
122.195
122.133
119.461
108.874
86.641
81.519
39.959
39.822
39.684
39.546
39.406
39.267
39.127
31.031
26.313
25.660
4b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

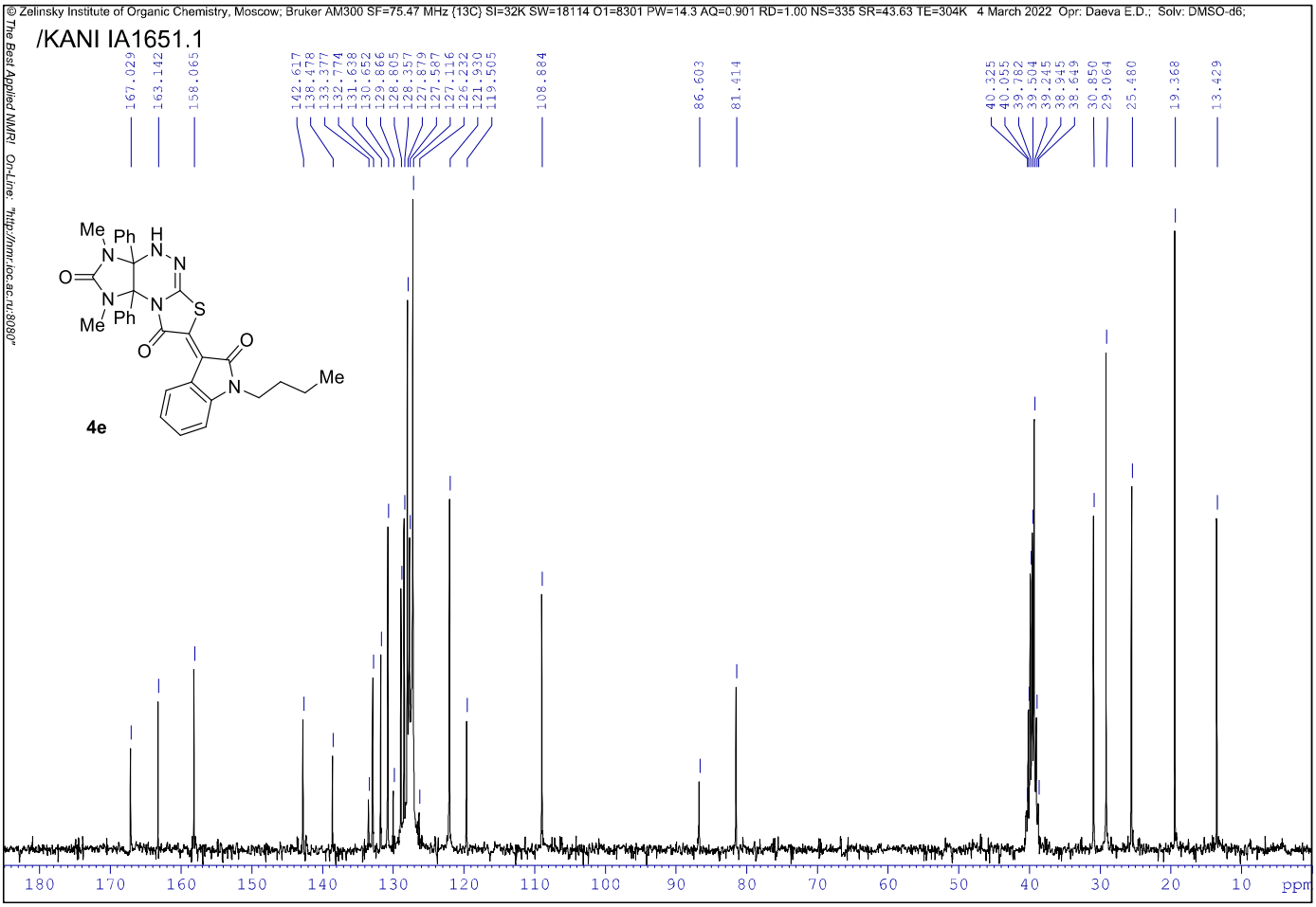

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6002 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=1 SR=-4.16 TE=303K 16 February 2022 Opr: Daeva E.D.; Solv: DMSO-d6;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1634.1
8.544
8.517
8.421
7.408
7.382
7.357
7.279
7.256
7.233
7.213
7.188
7.162
7.116
7.108
7.070
7.029
7.003
6.978
6.736
3.906
3.884
3.860
3.327
2.961
2.635
2.559
2.520
1.229
1.206
1.182
4c
ppm

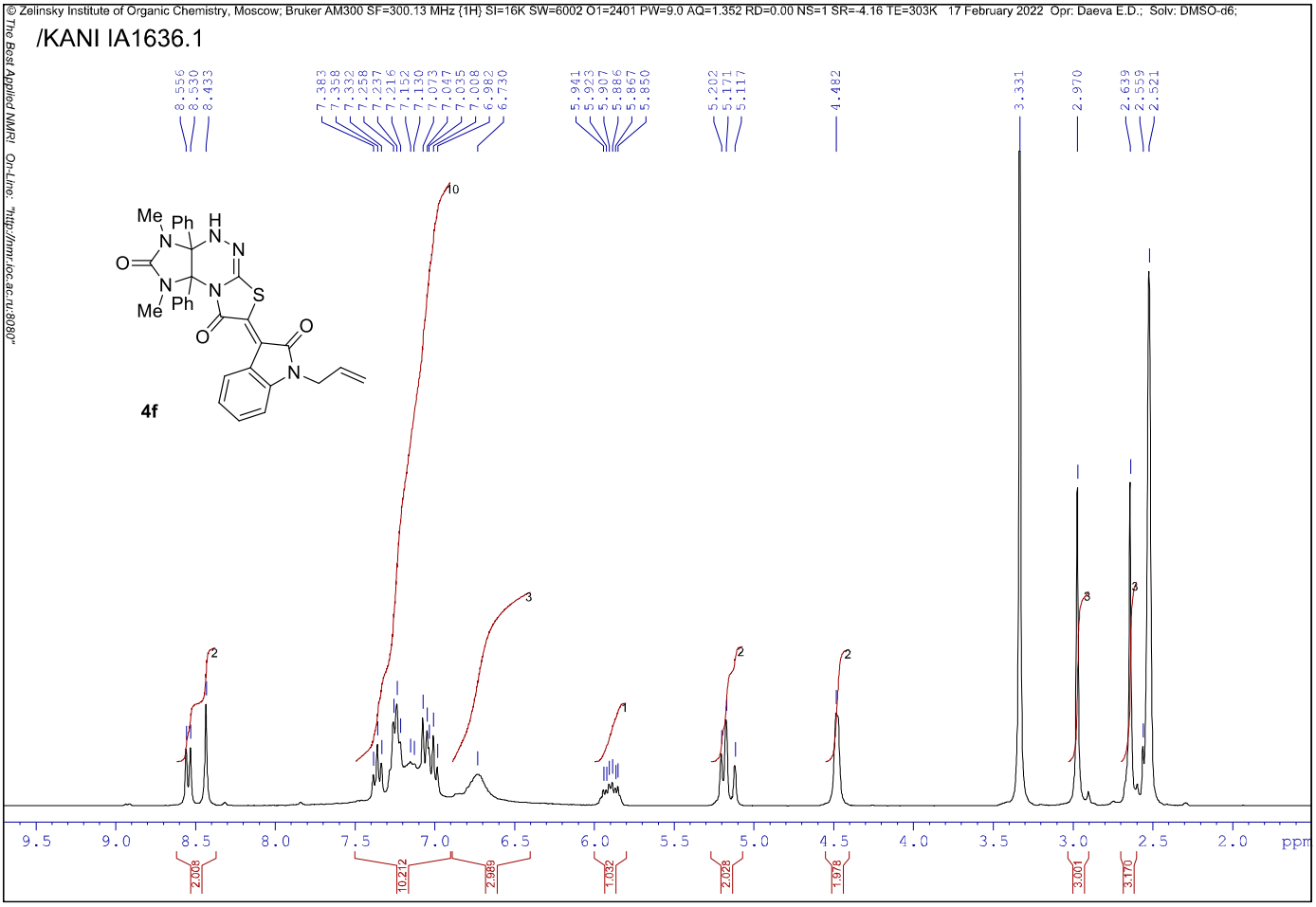

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=14.3 AQ=0.901 RD=1.00 NS=615 SR=41.47 TE=303K 17 February 2022 Opr: Daeva E.D.; Solv: DMSO-d6;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1634.1
166.714
163.162
158.095
142.293
138.497
133.325
132.768
131.638
130.742
128.852
128.406
127.927
127.613
127.150
122.111
122.001
119.591
108.826
86.618
81.429
40.328
40.051
39.785
39.505
39.226
38.947
38.657
34.419
30.882
25.519
12.591
4c
ppm

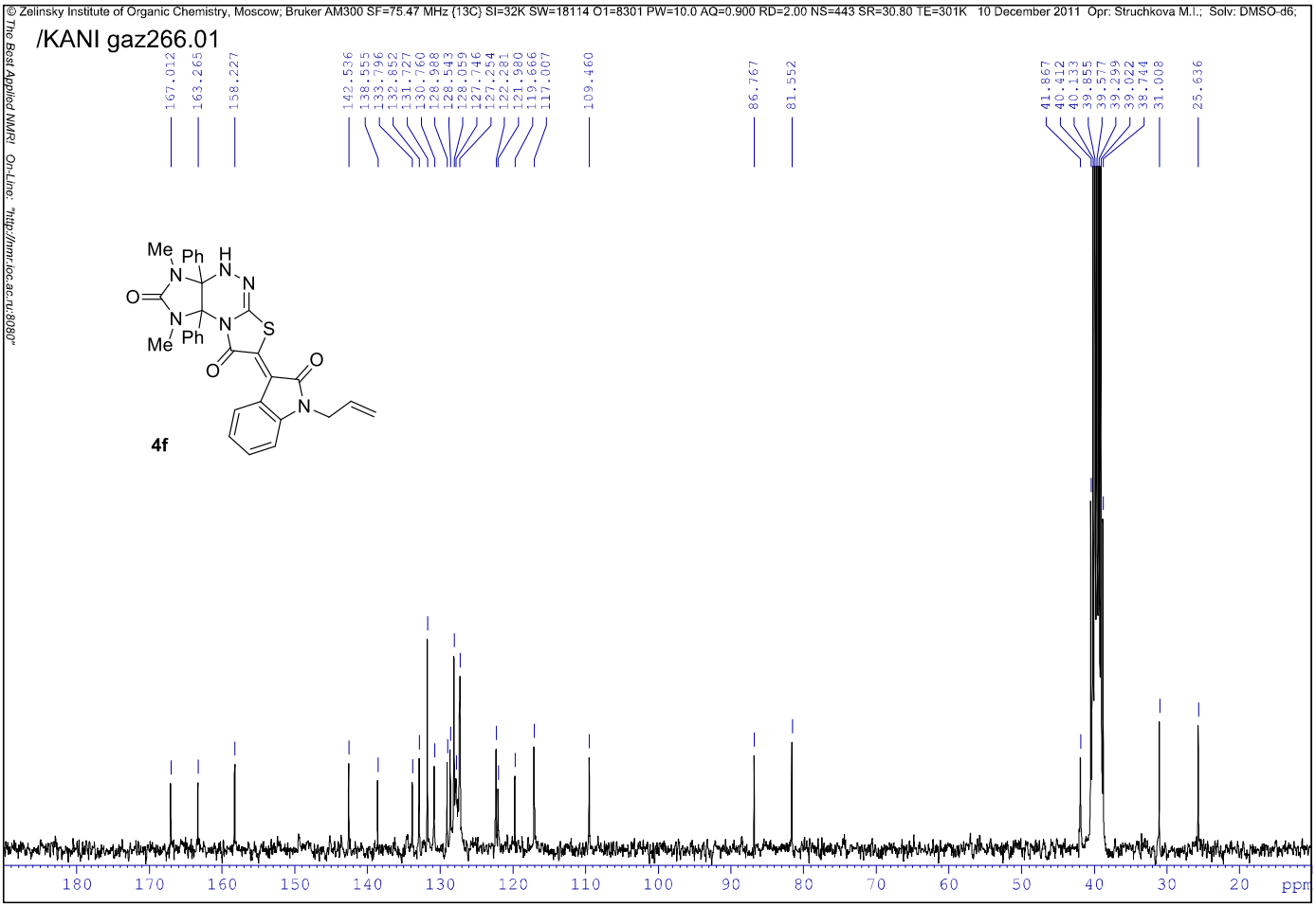

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6002 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=1 SR=-4.16 TE=302K 25 February 2022 Opr: Daeva E.D.; Solv: DMSO-d6;
/KANI IA1647.1
4d

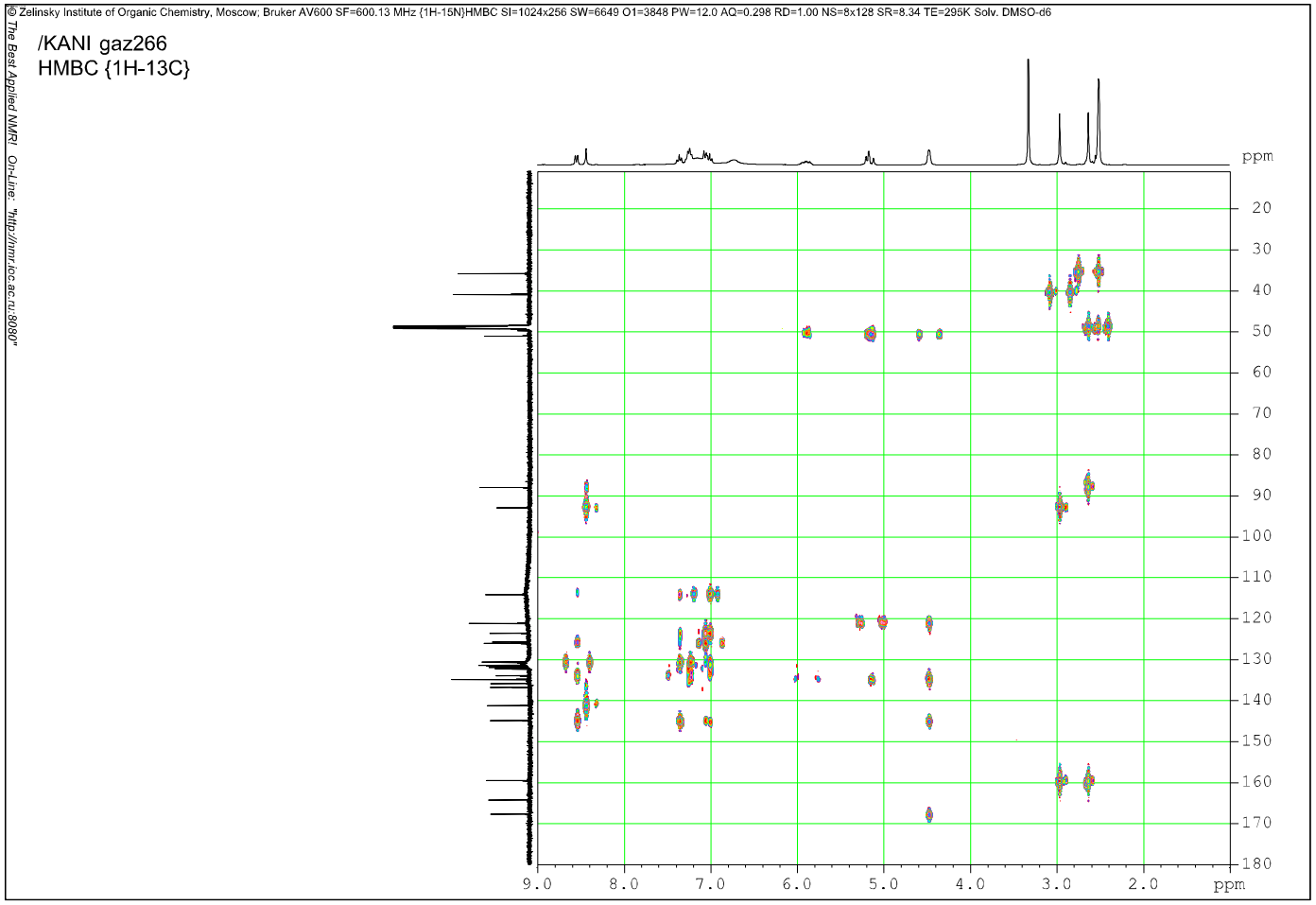

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=14.3 AQ=0.901 RD=1.00 NS=539 SR=40.58 TE=302K 28 February 2022 Opr: Daeva E.D.; Solv: DMSO-d6;
/KANI IA1647.1
4d

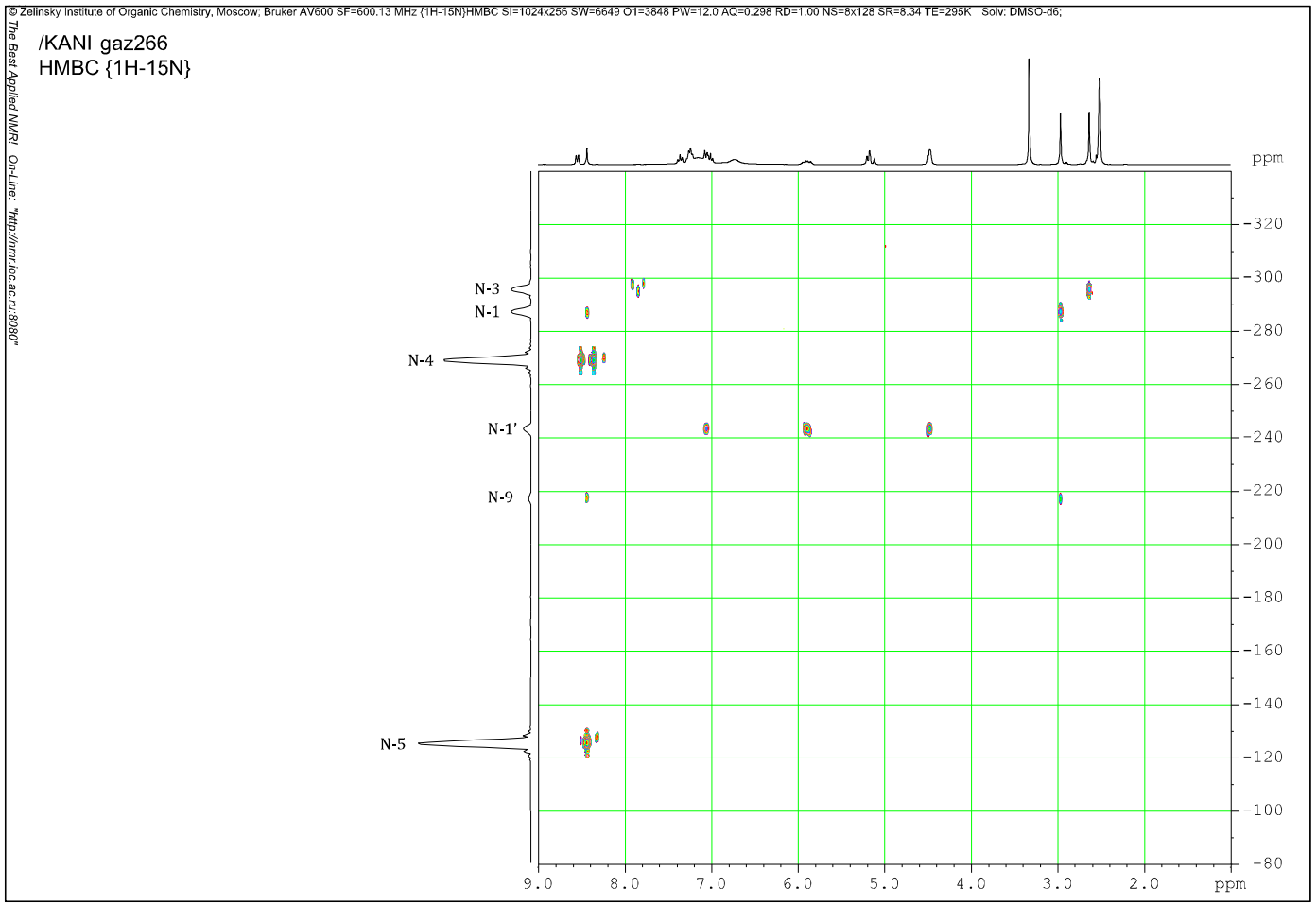

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6002 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=1 SR=-4.16 TE=302K 1 March 2022 Opr: Daeva E.D.; Solv: DMSO-d6;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1651.1
8.542 8.516 8.420
7.396 7.370 7.345 7.255 7.233 7.211 7.169 7.143 7.021 6.995 6.970 6.725
3.852 3.830 3.808
3.329
2.963
2.636 2.558 2.519
1.667 1.644 1.621 1.596 1.573 1.349 1.324 1.300 1.275 0.942 0.918 0.894
4e
ppm
1.951 9.684 3.085 1.943 2.899 3.130 1.994 2.014 3.008

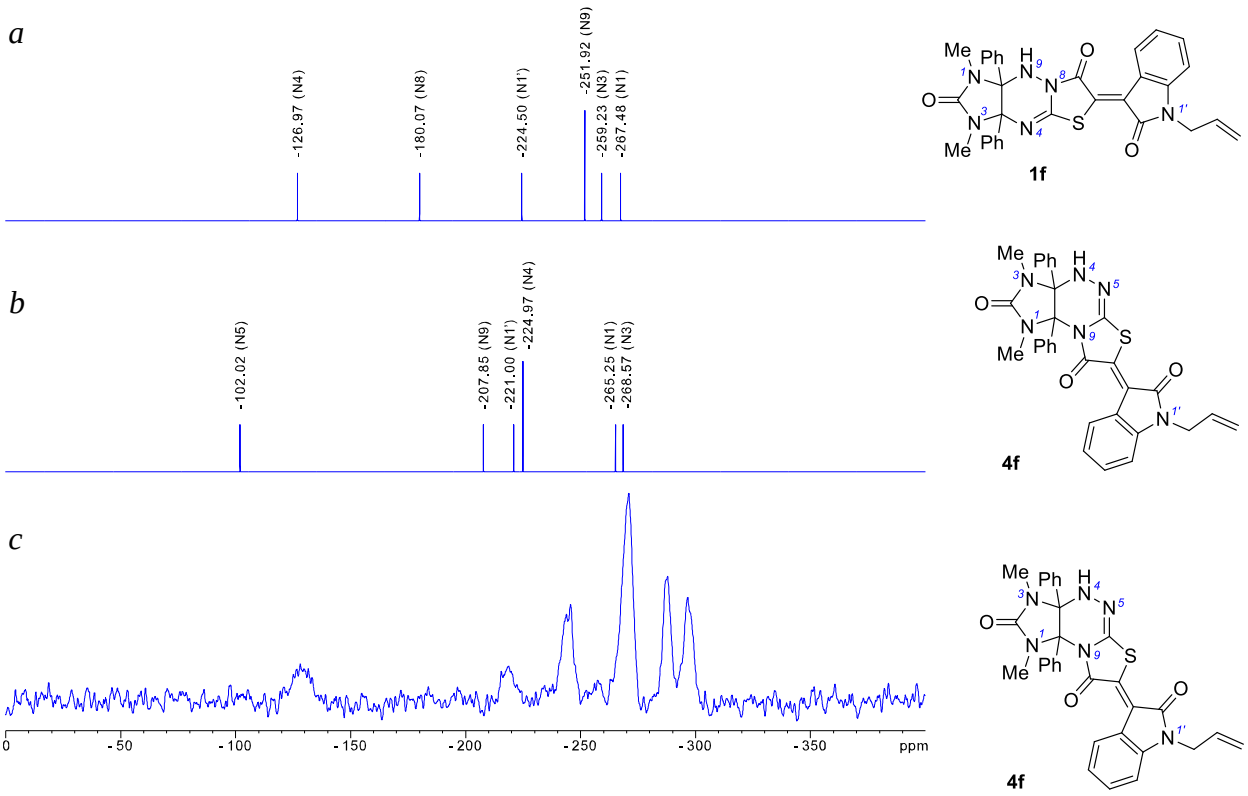

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=14.3 AQ=0.901 RD=1.00 NS=335 SR=43.63 TE=304K 4 March 2022 Opr: Daeva E.D.; Solv: DMSO-d6;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1651.1
167.029 163.142 158.065
142.617 138.478 133.377 132.774 131.638 130.652 129.866 128.805 128.357 127.879 127.587 127.116 126.232 121.930 119.505
108.884
86.603 81.414
40.325 40.055 39.782 39.504 39.245 38.945 38.649
30.850 29.064 25.480 19.368 13.429
4e
ppm


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6002 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=1 SR=-4.16 TE=303K 17 February 2022 Opr: Daeva E.D.; Solv: DMSO-d6;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1636.1
8.556
8.530
8.433
7.383
7.358
7.332
7.258
7.237
7.216
7.152
7.130
7.073
7.047
7.035
7.008
6.982
6.730
5.941
5.923
5.907
5.886
5.867
5.850
5.202
5.171
5.117
4.482
3.331
2.970
2.639
2.559
2.521
Me
Ph
H
N
N
O
N
S
Me
Ph
O
O
N
4f
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
ppm
2.008
10.212
2.986
1.032
2.028
1.978
3.001
3.170

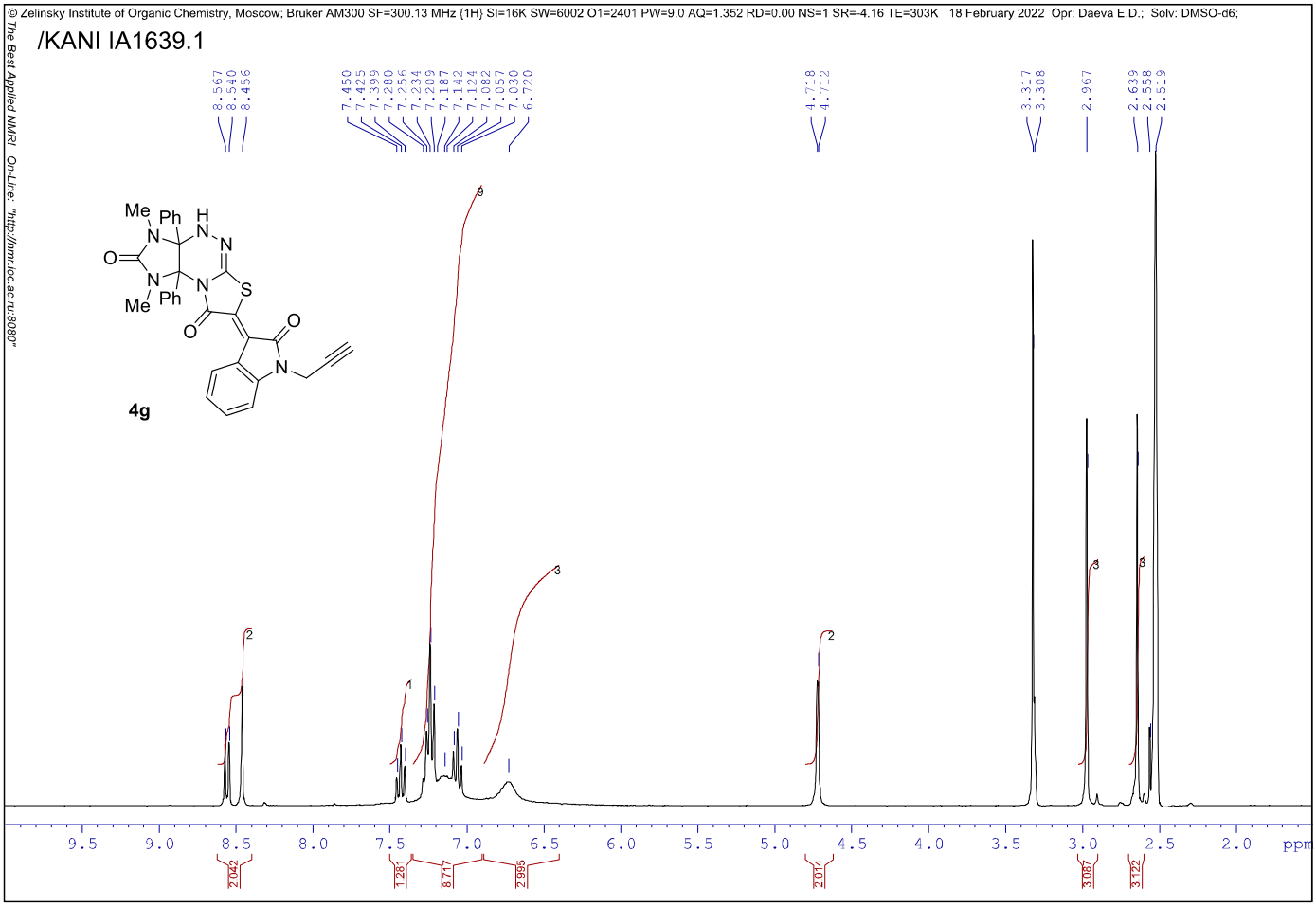

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=10.0 AQ=0.900 RD=2.00 NS=443 SR=30.80 TE=301K 10 December 2011 Opr: Struchkova M.I.; Solv: DMSO-d6;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI gaz266.01
167.012
163.265
158.227
142.536
138.555
133.796
132.852
131.727
130.760
128.988
128.543
128.059
127.746
127.254
122.281
121.980
119.666
117.007
109.460
86.767
81.552
41.867
40.412
40.133
39.855
39.577
39.299
39.022
38.744
31.008
25.636
Me
Ph
H
N
N
O
N
S
Me
Ph
O
O
N
4f
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

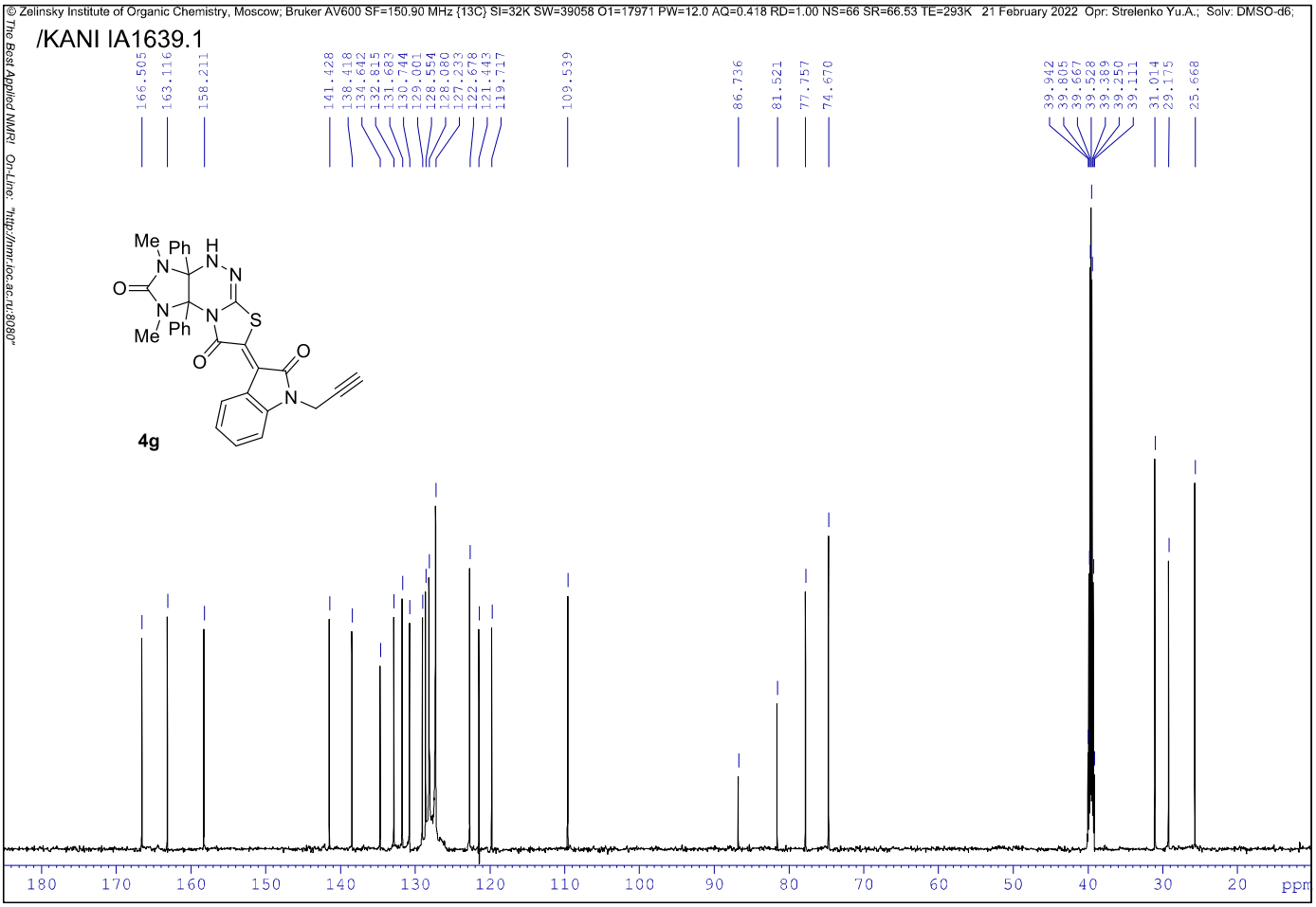

© Zelinsky Institute of Organic Chemistry, Moscow, Bruker AV600 SF=600.13 MHz {1H-15N}HMBC SI=1024x256 SW=6649 O1=3848 PW=12.0 AQ=0.298 RD=1.00 NS=8x128 SR=8.34 TE=295K Solv. DMSO-d6
© The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI gaz266
HMBC {1H-13C}
ppm
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
ppm
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180

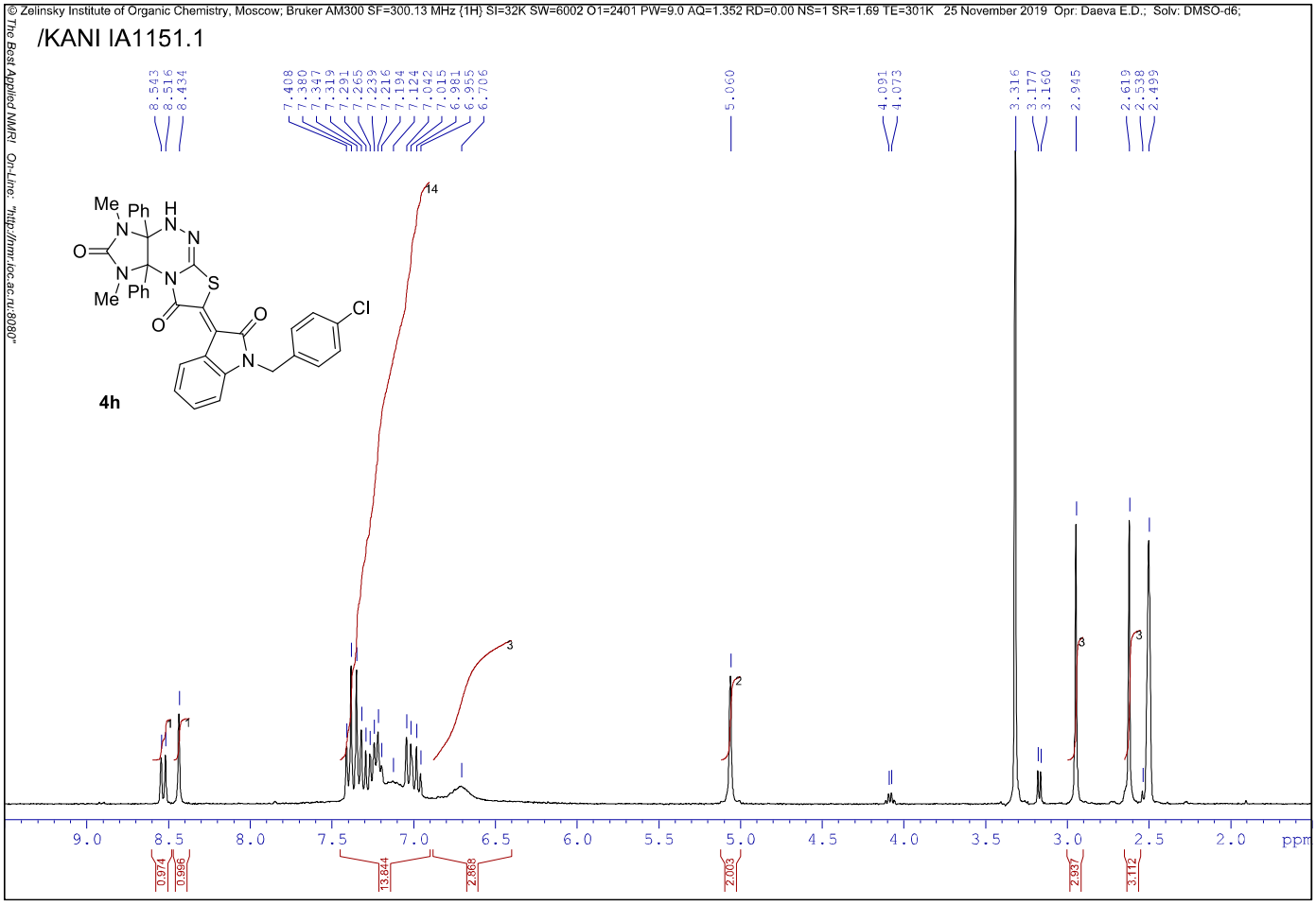

© Zelinsky Institute of Organic Chemistry, Moscow, Bruker AV600 SF=600.13 MHz {1H-15N}HMBC SI=1024x256 SW=6649 O1=3848 PW=12.0 AQ=0.298 RD=1.00 NS=8x128 SR=8.34 TE=295K Solv: DMSO-d6;
© The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI gaz266
HMBC {1H-15N}
N-3
N-1
N-4
N-1'
N-9
N-5
ppm
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
ppm
-320
-300
-280
-260
-240
-220
-200
-180
-160
-140
-120
-100
-80

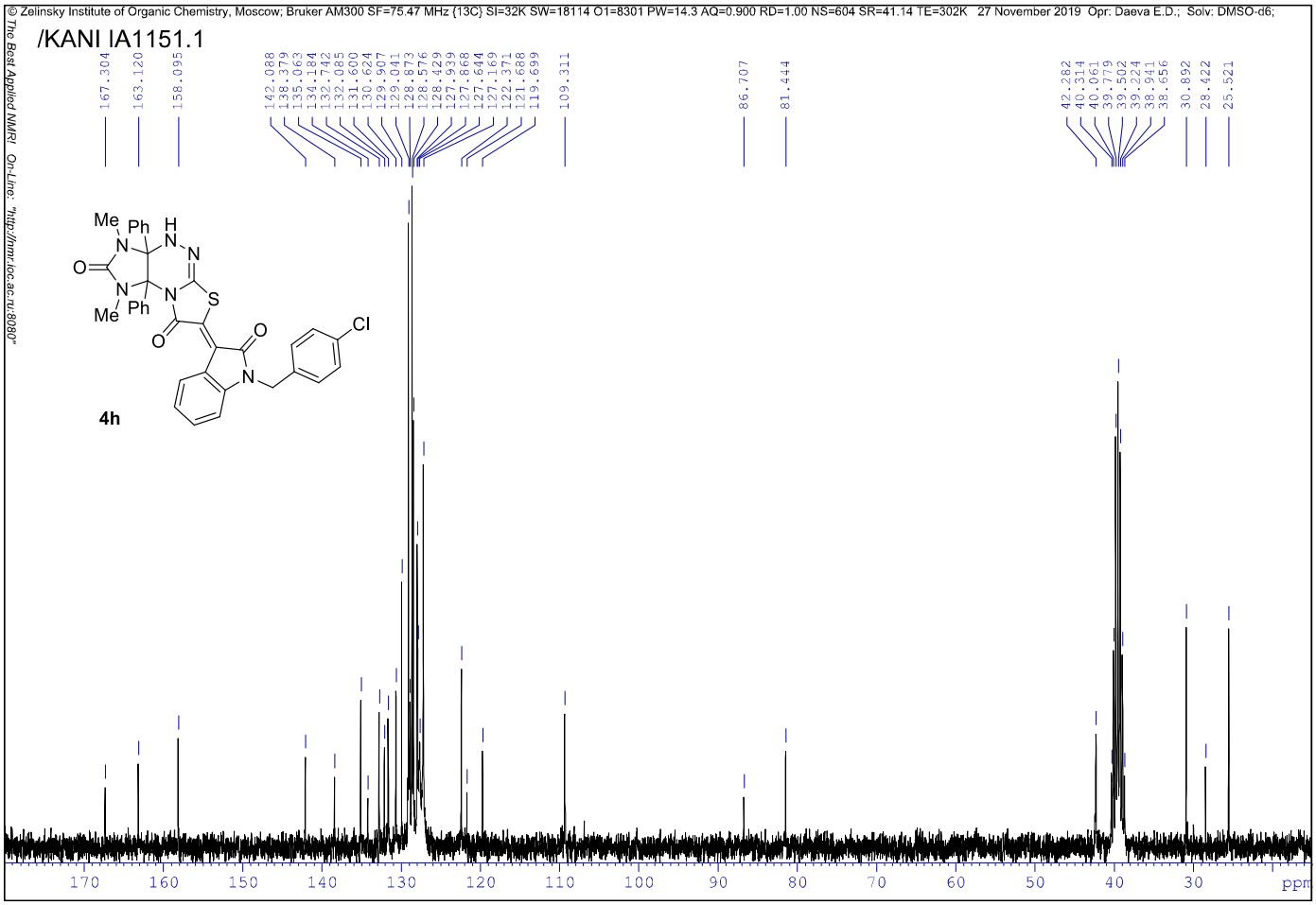


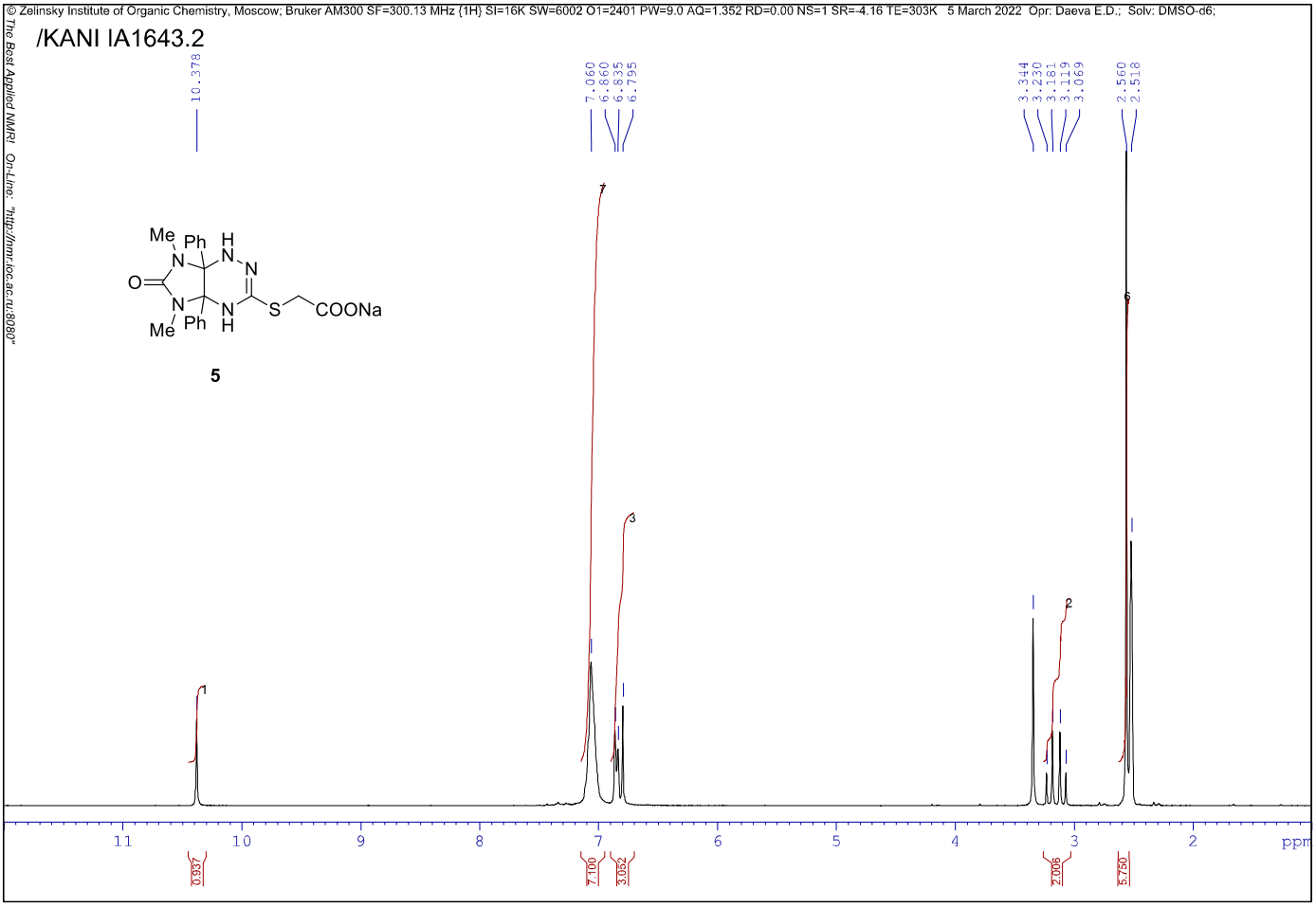
Calculated ^{15}N NMR spectra of isolated structures **1f** and **4f** (*a*,*b*) in vacuum (excluding solvent), and ^{15}N NMR spectrum of compound **4f**, registered in the solid phase by cross-polarization during rotation at a magic angle (^{15}N CP MAS) [S3].

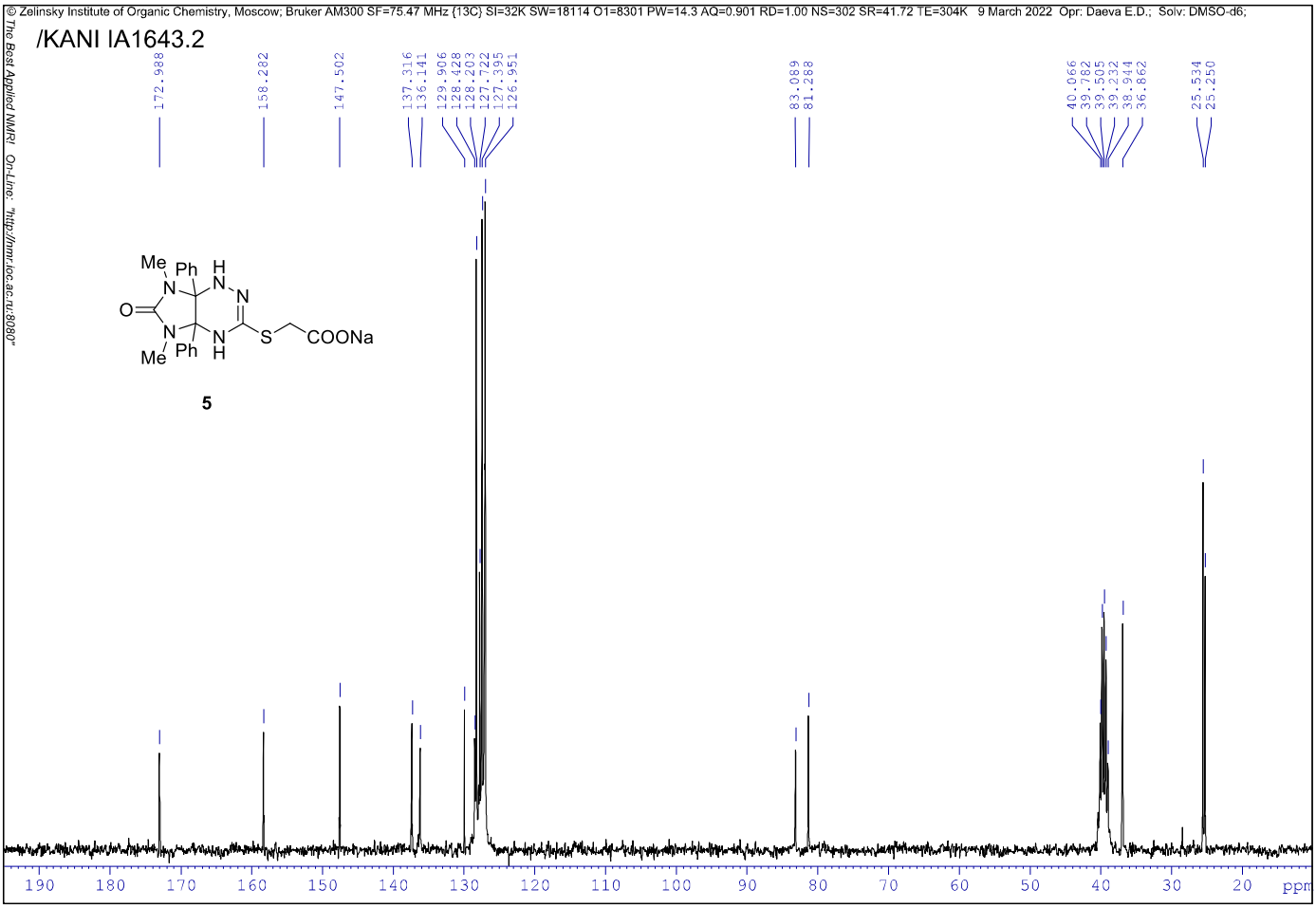

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6002 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=1 SR=-4.16 TE=303K 18 February 2022 Opr: Daeva E.D.; Solv: DMSO-d6;
/KANI IA1639.1
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
8.567
8.540
8.456
7.450
7.425
7.399
7.280
7.256
7.234
7.209
7.187
7.142
7.124
7.082
7.057
7.030
6.720
4.718
4.712
3.317
3.308
2.967
2.639
2.558
2.519
4g
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
ppm
2.042
1.281
8.717
2.995
2.014
3.087
3.122

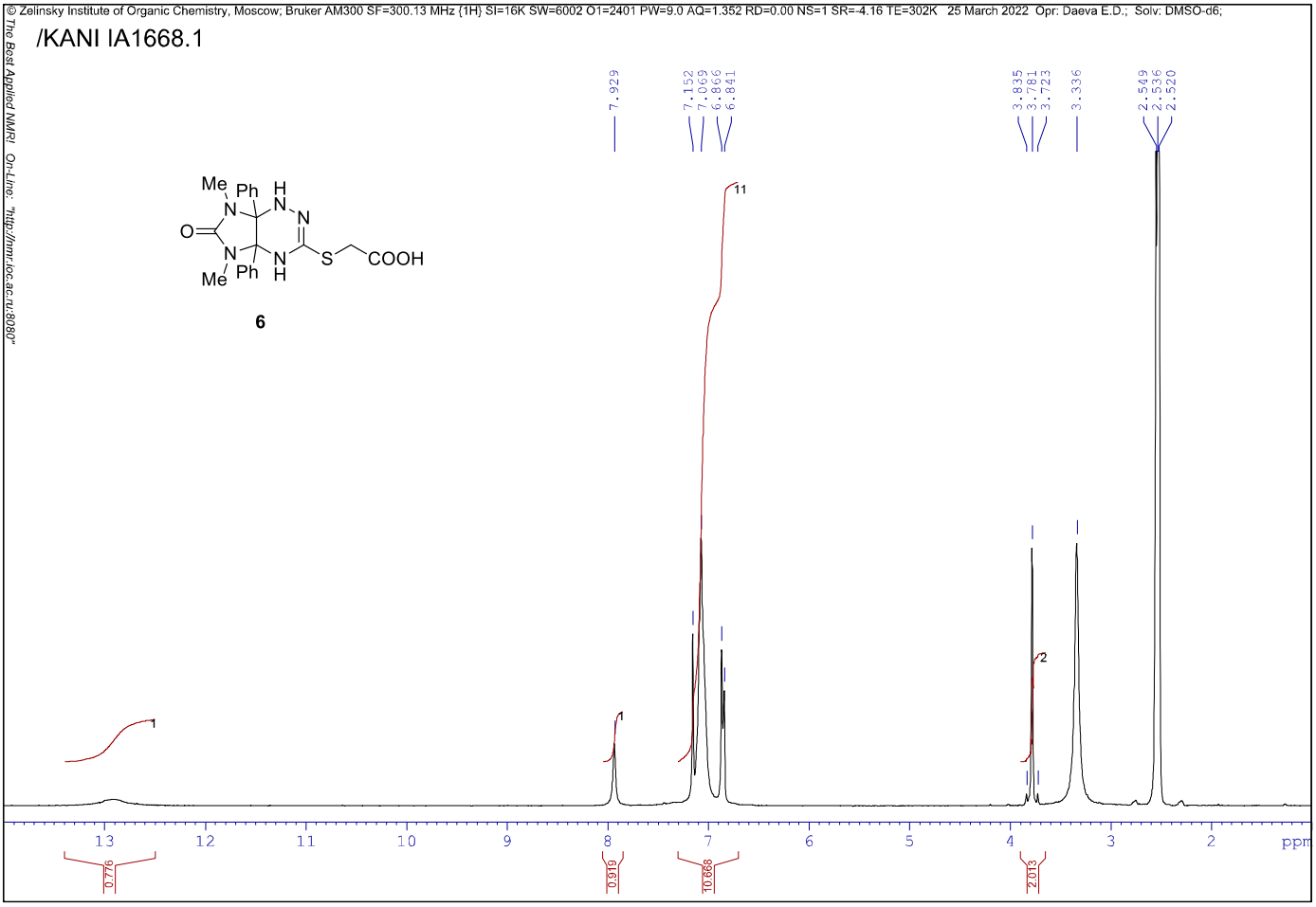

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AV600 SF=150.90 MHz {13C} SI=32K SW=39058 O1=17971 PW=12.0 AQ=0.418 RD=1.00 NS=66 SR=66.53 TE=293K 21 February 2022 Opr: Strelenko Yu.A.; Solv: DMSO-d6;
/KANI IA1639.1
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
166.505
163.116
158.211
141.428
138.418
134.642
132.815
131.683
130.744
129.001
128.554
128.080
127.233
122.678
121.443
119.717
109.539
86.736
81.521
77.757
74.670
39.942
39.805
39.667
39.528
39.389
39.250
39.111
31.014
29.175
25.668
4g
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

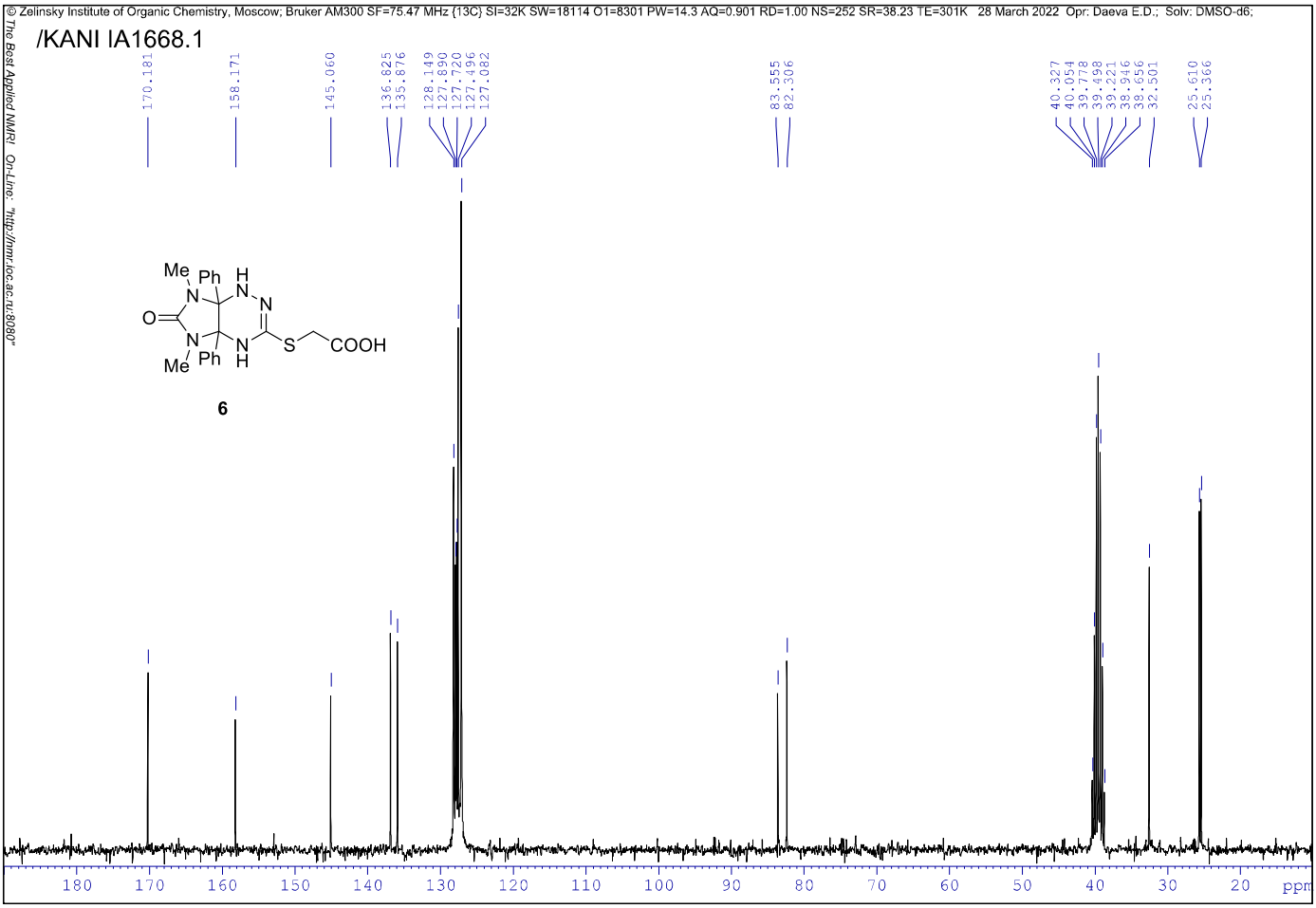

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=32K SW=6002 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=1 SR=1.69 TE=301K 25 November 2019 Opr: Daeva E.D.; Solv: DMSO-d6;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1151.1
8.543
8.516
8.434
7.408
7.380
7.347
7.319
7.291
7.265
7.239
7.216
7.194
7.124
7.042
7.015
6.981
6.955
6.706
5.060
4.091
4.073
3.316
3.177
3.160
2.945
2.619
2.538
2.499
4h
ppm
0.974
0.996
13.844
2.868
2.003
2.937
3.112


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=14.3 AQ=0.900 RD=1.00 NS=604 SR=41.14 TE=302K 27 November 2019 Opr: Daeva E.D.; Solv: DMSO-d6;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1151.1
167.304
163.120
158.095
142.088
138.379
135.063
134.184
132.742
132.085
131.600
130.624
129.907
129.041
128.873
128.576
128.429
127.939
127.868
127.644
127.169
122.371
121.688
119.699
109.311
86.707
81.444
42.282
40.314
40.061
39.779
39.502
39.224
38.941
38.656
30.892
28.422
25.521
4h
ppm


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6002 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=1 SR=-4.16 TE=303K 5 March 2022 Opr: Daeva E.D.; Solv: DMSO-d6;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1643.2
10.378
7.060
6.860
6.835
6.795
3.344
3.230
3.181
3.119
3.069
2.560
2.518
Me
Ph
H
N
N
O
N
N
S
COONa
Me
Ph
H
5
ppm
0.937
7.100
3.052
2.006
5.750


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=14.3 AQ=0.901 RD=1.00 NS=302 SR=41.72 TE=304K 9 March 2022 Opr: Daeva E.D.; Solv: DMSO-d6;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1643.2
172.988
158.282
147.502
137.316
136.141
129.906
128.428
128.203
127.722
127.395
126.951
83.089
81.288
40.066
39.782
39.505
39.232
38.944
36.862
25.534
25.250
Me
Ph
H
N
N
O
N
N
S
COONa
Me
Ph
H
5
ppm


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6002 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=1 SR=-4.16 TE=302K 25 March 2022 Opr: Daeva E.D.; Solv: DMSO-d6;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1668.1
7.929
7.152
7.069
6.866
6.841
3.835
3.781
3.723
3.336
2.549
2.536
2.520
Me
Ph
H
N
N
O
N
N
S
COOH
Me
Ph
H
6
11
1
2
0.776
0.819
10.668
2.013
13
12
11
10
9
8
7
6
5
4
3
2
ppm


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=14.3 AQ=0.901 RD=1.00 NS=252 SR=38.23 TE=301K 28 March 2022 Opr: Daeva E.D.; Solv: DMSO-d6;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1668.1
170.181
158.171
145.060
136.825
135.876
128.149
127.890
127.720
127.496
127.082
83.555
82.306
40.327
40.054
39.778
39.498
39.221
38.946
38.656
32.501
25.610
25.366
Me
Ph
H
N
N
O
N
N
S
COOH
Me
Ph
H
6
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

References

[1] G. A. Gazieva, E. A. Shishkova, L. B. Kulikova, N. G. Kolotyrkina, N. V. Sigay, A. N. Kravchenko, *J. Heterocycl. Chem*., 2014, **51**, 921.
[2] S. V. Vasilevskii, P. A. Belyakov, G. A. Gazieva, Y. V. Nelyubina, A. N. Kravchenko, *Mendeleev Commun*., 2010, **20**, 47.
[3] P. A. Belyakov, V. P. Ananikov, *Russ. Chem. Bul.*, 2011, **60**, 783.