Supplementary Information

Tandem Michael addition/elimination – novel reactivity of pyridinium ylides in reaction with electron-deficient alkenes

Alexei N. Izmest'ev,*[a] Vladimir A. Motornov,*[b] Dmitry B. Vinogradov,[a] Sema L. Ioffe,[a] Angelina N. Kravchenko[a] and Galina A. Gazieva[a]

[a] *N. D. Zelinsky Institute of Organic Chemistry, Russian Academy of Sciences, 47 Leninsky Prosp., Moscow 119991, Russian Federation;*
[b] *Institute of Organic Chemistry and Biochemistry of the Czech Academy of Sciences, Flemingovo nám. 2, 160 00 Prague 6, Czech Republic.*
Corresponding author e-mail: nebeli@mail.ru (Alexei N. Izmest'ev); vladimir.motornov@uochb.cas.cz (Vladimir A. Motornov).

Table of Contents

Experimental section

All standard reagents 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 AV-300 (300.13 MHz (^{1}H)), Bruker AM-300 (300.13 and 75.47 MHz, respectively), Bruker Avance III HD 400 MHz (400.13 and 100.61 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) or on Agilent 7890A GC coupled with Waters GCT Premier orthogonal acceleration time-of-flight detector using electron impact (EI) ionization. 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.

The starting compounds were prepared according to literature procedures for **2a-n** and **7a-l**,[1] **8** and **9**,[2] and **10**.[3]

X-ray crystallographic data and refinement details for 3o, 6h

Single crystals of **3o**, **6h** were grown from methanol.

Data collection for sample **3o** X-ray were collected at 100K on a four-circle Rigaku Synergy S diffractometer equipped with a HyPix600HE area-detector (kappa geometry, shutterless ω-scan technique), using graphite monochromatized Cu K_α-radiation. The intensity data were integrated and corrected for absorption and decay by the CrysAlisPro program[4]. The structure was solved by direct methods using SHELXT[5] and refined on F^2 using SHELXL-2018[6] in the OLEX2 program.[7] All non-hydrogen atoms were refined with individual anisotropic displacement parameters. The location of hydrogen atom N(9)H was found from the electron density-difference map; this hydrogen atom was refined with an individual isotropic displacement parameter. All other hydrogen atoms were placed in ideal calculated positions and refined as riding atoms with relative isotropic displacement parameters. The ORTEP program suite[8] was used for molecular graphics.

Data collection for sample **6h** were collected at 276K on a Bruker Quest D8 diffractometer equipped with a Photon-III area-detector (shutterless φ- and ω-scan technique), using graphite-monochromatized Mo K_α-radiation. The intensity data were integrated by the SAINT program[9] and were corrected for absorption and decay using SADABS.[10] The structure was solved by direct methods using SHELXT[5] and refined on F[7] using SHELXL-2018.[6] Positions of all atoms were found from the electron density-difference map. All non-hydrogen atoms were refined with individual anisotropic displacement parameters. The location of hydrogen atom N(4)H was found from the electron density-difference map; this hydrogen atom was refined with an individual isotropic displacement parameter. All other hydrogen atoms were placed in ideal calculated positions and refined as riding atoms with relative isotropic displacement parameters. The ORTEP program suite[8] was used for molecular graphics.

The phenyl substituent (N(1)Ph) is disordered over two positions with the contribution of main component of 0.573. One ester group (1-COOEt) is disordered over three positions with the contribution of each component 0.268:0.252:0.480.

Deposition Numbers 2172820, 2172821 contain the supplementary crystallographic data for this paper. These data are provided free of charge by the joint Cambridge Crystallographic Data Centre and Fachinformationszentrum Karlsruhe Access Structures service www.ccdc.cam.ac.uk/structures.

General procedure for the synthesis of compounds 1a-e

To a solution of pyridine (3.020 ml, 37.5 mmol) in THF (50 ml), corresponding bromide (25 mmol) was added. The reaction mixture was stirred at room temperature for 48 h. After the reaction, the precipitate was filtered off, washed by THF and dried.

1-(2-Ethoxy-2-oxoethyl)pyridin-1-ium bromide (1a). Yield 6.08 g (99%); white powder; mp: 130–131 °C (decomp.). [Lit.[11]: yield 82%; white solid; mp: 135–136 °C].

1-(2-Methoxy-2-oxoethyl)pyridin-1-ium bromide (1b). Yield 5.27 g (91%); white powder; mp: 174–175 °C (decomp.). [Lit.[12]: yield 93%; white powder; mp: 186–188 °C].

1-(2-Oxopropyl)pyridin-1-ium bromide (1c). Yield 3.29 g (61%); white powder; mp: 181–182 °C. [Lit.[11]: yield 45%; white powder; mp: 208–209 °C; lit.[13]: mp: 184 °C].

1-(2-Oxo-2-phenylethyl)pyridin-1-ium bromide (1d). Yield 6.46 g (93%); white powder; mp: 198 °C. [Lit.[12]: yield 80%; white powder; mp: 200–202 °C].

1-(Cyanomethyl)pyridin-1-ium bromide (1e). Yield 4.33 g (87%); grey powder; mp: 157–158 °C. [Lit.[11]: yield 100%; white powder; mp: 164–165 °C].

General procedure for the synthesis of compounds 3a-u

To a suspension of imidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazine **2a-n**[1] (1 mmol) and pyridinium ylide **1a-e** (1.5 mmol) in dichloromethane (5 ml), triethylamine (0.208 ml, 1.5 mmol) was added. The reaction mixture was stirred at room temperature for 2 h.

For **3a-i,k,n-u**: After standing until the solvent had evaporated, 20 ml of water was added. The resulting mixture was stirred and the precipitate formed was filtered off, washed with 5 ml of water and dried.

For **3j,l,m**: The reaction mixture was extracted by 5 ml of HCl (10% aqueous solution), then organic layer was washed by 5 ml of $NaHCO_3$ (10% aqueous solution) and 5 ml of water. The organic layer was dried by anh. Na_2SO_4, evaporated and dried.

The compounds were recrystallized from MeOH if necessary.

Diethyl (*Z*)-2-(1,3-dimethyl-2,7-dioxo-1,2,3,3a,9,9a-hexahydroimidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazin-6(7*H*)-ylidene)succinate (3a). Yield 334 mg (81%); pale yellow solid; mp: 162–163 °C. IR (KBr), ν 3214 (NH), 2985, 2940 (Alk), 1737, 1716, 1698, 1633 (C=O) cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.28 (t, *J* = 7.2 Hz, 3H, CH_3), 1.35 (t, *J* = 7.2 Hz, 3H, CH_3), 2.78 (s, 3H, NCH_3), 2.96 (s, 3H, NCH_3), 4.09-4.21 (m, 3H, CH_2, OCH_2), 4.29-4.38 (m, 3H, CH_2, OCH_2), 4.71 (dd, *J* = 5.9, 2.4 Hz, 1H, 9a'-H), 4.94 (d, *J* = 5.9 Hz, 1H, 3a'-H), 5.46 (s, 1H, NH). ^{13}C NMR (75 MHz, $CDCl_3$): δ 13.97, 14.10 ($2CH_3$), 27.35, 27.97 ($2NCH_3$), 33.13 (CH_2), 61.11, 62.61 ($2OCH_2$), 66.20, 67.11 (C-3a',

C-9a'), 126.87 (C-6'), 134.22 (C-2), 149.56 (4a'-C=N), 159.18, 160.41 (2'-C=O, 7'-C=O), 166.12 (1-COOEt), 169.98 (4-COOEt). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{16}H_{21}N_5O_6S$: 412.1285; found: 412.1290.

4-Ethyl 1-methyl (*Z*)-2-(1,3-dimethyl-2,7-dioxo-1,2,3,3a,9,9a-hexahydroimidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazin-6(7*H*)-ylidene)succinate (3b). Yield 369 mg (93%); pale yellow solid; mp: 170–172 °C. IR (KBr), ν 3464, 3217 (NH), 2979, 2958, 2935 (Alk), 1738, 1716, 1699, 1636 (C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 1.29 (t, *J* = 7.1 Hz, 3H, CH_3), 2.78 (s, 3H, NCH_3), 2.96 (s, 3H, NCH_3), 3.89 (s, 3H, OCH_3), 4.09-4.22 (m, 3H, CH_2, OCH_2), 4.30 (d, *J* = 17.1 Hz, 1H, CH_2), 4.71 (dd, *J* = 5.8, 2.0 Hz, 1H, 9a'-H), 4.95 (d, *J* = 5.9 Hz, 1H, 3a'-H), 5.43 (d, *J* = 1.9 Hz, 1H, NH). ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.92 (CH_3), 28.24, 28.84 ($2NCH_3$), 33.90 (CH_2), 54.08 (OCH_3), 61.95 (OCH_2), 67.40, 68.09 (C-3a', C-9a'), 127.63 (C-6'), 135.23 (C-2), 150.09 (4a'-C=N), 159.75, 160.71 (2'-C=O, 7'-C=O), 167.43 (1-COOMe), 170.47 (4-COOEt). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{15}H_{19}N_5O_6S$: 398.1129; found: 398.1123.

1-Ethyl 4-methyl (*Z*)-2-(1,3-dimethyl-2,7-dioxo-1,2,3,3a,9,9a-hexahydroimidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazin-6(7*H*)-ylidene)succinate (3c). Yield 318 mg (80%); pale yellow solid; mp: 181–183 °C. IR (KBr), ν 3219 (NH), 2981, 2947, 2849 (Alk), 1744, 1698, 1635 (C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 1.30 (t, *J* = 7.1 Hz, 3H, CH_3), 2.73 (s, 3H, NCH_3), 2.91 (s, 3H, NCH_3), 3.67 (s, 3H, OCH_3), 4.09 (d, *J* = 17.1 Hz, 1H, CH_2), 4.24-4.33 (m, 3H, CH_2, OCH_2), 4.66 (dd, *J* = 5.9, 2.3 Hz, 1H, 9a'-H), 4.90 (d, *J* = 5.9 Hz, 1H, 3a'-H), 5.35 (d, *J* = 2.1 Hz, 1H, NH). ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.02 (CH_3), 27.41, 28.03 ($2NCH_3$), 32.96 (CH_2), 52.20 (OCH_3), 62.69 (OCH_2), 66.34, 67.19 (C-3a', C-9a'), 126.79 (C-6'), 134.37 (C-2), 149.51 (4a'-C=N), 159.17, 160.39 (2'-C=O, 7'-C=O), 166.10 (1-COOEt), 170.44 (4-COOMe). HRMS (ESI): *m/z* $[M + Na]^+$ calcd for $C_{15}H_{19}N_5O_6S$: 420.0948; found: 420.0936.

Dimethyl (*Z*)-2-(1,3-dimethyl-2,7-dioxo-1,2,3,3a,9,9a-hexahydroimidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazin-6(7*H*)-ylidene)succinate (3d). Yield 356 mg (91%); pale yellow solid; mp: 175–176 °C. IR (KBr), ν 3212 (NH), 2982, 2951, 2849 (Alk), 1747, 1716, 1700, 1634 (C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 2.73 (s, 3H, NCH_3), 2.90 (s, 3H, NCH_3), 3.67 (s, 3H, OCH_3), 3.84 (s, 3H, OCH_3), 4.08 (d, *J* = 17.2 Hz, 1H, CH_2), 4.25 (d, *J* = 17.1 Hz, 1H, CH_2), 4.65 (dd, *J* = 5.9, 2.4 Hz, 1H, 9a'-H), 4.90 (d, *J* = 5.5 Hz, 1H, 3a'-H), 5.39 (d, *J* = 2.2 Hz, 1H, NH). ^{13}C NMR (150 MHz, $CDCl_3$): δ 28.14, 28.78 ($2NCH_3$), 33.63 (CH_2), 53.03, 54.13 ($2OCH_3$), 66.03, 67.86 (C-3a', C-9a'), 126.99 (C-6'), 135.58 (C-2), 150.23 (4a'-C=N), 159.92, 161.10 (2'-C=O, 7'-C=O), 167.37 (1-COOMe), 171.14 (4-COOMe). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{14}H_{17}N_5O_6S$: 384.0972; found: 384.0972.

Diethyl (*Z*)-2-(1,3-dimethyl-2,7-dioxo-3a,9a-diphenyl-1,2,3,3a,9,9a-hexahydroimidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazin-6(7*H*)-ylidene)succinate (3e). Yield 546 mg (97%); pale beige solid;

mp: 150–152 °C. IR (KBr), ν 3434, 3119 (NH), 2982, 2936 (Alk), 1738, 1699, 1642 (C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 1.30 (t, *J* = 7.1 Hz, 3H, CH_3), 1.39 (t, *J* = 7.1 Hz, 3H, CH_3), 2.74 (s, 3H, NCH_3), 2.83 (s, 3H, NCH_3), 4.16-4.25 (m, 3H, CH_2, OCH_2), 4.35-4.23 (m, 3H, CH_2, OCH_2), 6.23 (s, 1H, NH), 6.81-6.87 (m, 4H, 2Ph-2,6), 7.04-7.28 (m, 6H, 2Ph-3-5). ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.11, 14.25 ($2CH_3$), 25.13, 26.53 ($2NCH_3$), 33.26 (CH_2), 61.19, 62.76 ($2OCH_2$), 80.70, 85.04 (C-3a', C-9a'), 127.10, 127.50, 127.61, 127.67, 128.23, 128.40, 129.06 (C-6', 2Ph-2-6), 133.35, 134.48, 134.76 (C-2, 2Ph-1), 146.48 (4a'-C=N), 158.31, 159.85 (2'-C=O, 7'-C=O), 166.14 (1-COOEt), 169.77 (4-COOEt). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{28}H_{29}N_5O_6S$: 564.1911; found: 564.1909.

4-Ethyl 1-methyl (*Z*)-2-(1,3-dimethyl-2,7-dioxo-3a,9a-diphenyl-1,2,3,3a,9,9a-hexahydroimidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazin-6(7*H*)-ylidene)succinate (3f). Yield 532 mg (97%); pale yellow solid; mp: 208–210 °C. IR (KBr), ν 3434, 3114 (NH), 2979, 2930, 2852 (Alk), 1738, 1698, 1642 (C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 1.28 (t, *J* = 7.1 Hz, 3H, CH_3), 2.72 (s, 3H, NCH_3), 2.81 (s, 3H, NCH_3), 3.92 (s, 3H, OCH_3), 4.14-4.24 (m, 3H, CH_2, OCH_2), 4.35 (d, *J* = 17.1 Hz, 1H, CH_2), 6.24 (s, 1H, NH), 6.80-6.85 (m, 4H, 2Ph-2,6), 7.03-7.20 (m, 6H, 2Ph-3-5). ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.26 (CH_3), 25.17, 26.55 ($2NCH_3$), 33.21 (CH_2), 53.42 (OCH_3), 61.28 (OCH_2), 80.70, 85.04 (C-3a', C-9a'), 127.01, 127.39, 127.65, 128.27, 128.43, 129.10 (C-6', 2Ph-2-6), 133.28, 134.41, 135.26 (C-2, 2Ph-1), 146.46 (4a'-C=N), 158.26, 159.86 (2'-C=O, 7'-C=O), 166.72 (1-COOMe), 169.76 (4-COOEt). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{27}H_{27}N_5O_6S$: 550.1755; found: 550.1753.

1-Ethyl 4-methyl (*Z*)-2-(1,3-dimethyl-2,7-dioxo-3a,9a-diphenyl-1,2,3,3a,9,9a-hexahydroimidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazin-6(7*H*)-ylidene)succinate (3g). Yield 538 mg (98%); pale yellow solid; mp: 177–179 °C. IR (KBr), ν 3091, 3062, 3032 (Ph), 2979, 2950 (Alk), 1719, 1700, 1643 (C=N, C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 1.34 (t, *J* = 7.1 Hz, 3H, CH_3), 2.68 (s, 3H, NCH_3), 2.78 (s, 3H, NCH_3), 3.70 (s, 3H, OCH_3), 4.15 (d, *J* = 17.0 Hz, 1H, CH_2), 4.32-4.37 (m, 3H, CH_2, OCH_2), 6.18 (s, 1H, NH), 6.77-6.82 (m, 4H, 2Ph-2,6), 6.99-7.18 (m, 6H, 2Ph-3-5). ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.10 (CH_3), 25.13, 26.52 ($2NCH_3$), 33.02 (CH_2), 52.23 (OCH_3), 62.79 (OCH_2), 80.70, 85.03 (C-3a', C-9a'), 127.08, 127.29, 127.61, 127.66, 128.23, 128.39, 129.05 (C-6', 2Ph-2-6), 133.34, 134.46, 134.90 (C-2, 2Ph-1), 146.43 (4a'-C=N), 158.31, 159.81 (2'-C=O, 7'-C=O), 166.07 (1-COOEt), 170.20 (4-COOMe). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{27}H_{27}N_5O_6S$: 550.1755; found: 550.1748.

Dimethyl (*Z*)-2-(1,3-dimethyl-2,7-dioxo-3a,9a-diphenyl-1,2,3,3a,9,9a-hexahydroimidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazin-6(7*H*)-ylidene)succinate (3h). Yield 508 mg (95%); pale yellow solid; mp: 195–197 °C. IR (KBr), ν 3035, 3002 (Ph), 2952, 2845 (Alk), 1700, 1641 (C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 2.68 (s, 3H, NCH_3), 2.76 (s, 3H, NCH_3), 3.70 (s, 3H, OCH_3), 3.92 (s, 3H, OCH_3),

4.14 (d, *J* = 17.1 Hz, 1H, CH_2), 4.33 (d, *J* = 17.1 Hz, 1H, CH_2), 6.17 (s, 1H, NH), 6.77-6.81 (m, 4H, 2Ph-2,6), 6.99-7.18 (m, 6H, 2Ph-3-5). ^{13}C NMR (75 MHz, $CDCl_3$): δ 25.16, 26.57 ($2NCH_3$), 33.01 (CH_2), 52.33, 53.46 ($2OCH_3$), 80.74, 85.14 (C-3a', C-9a'), 126.85, 127.12, 127.65, 127.70, 128.28, 128.45, 129.11 (C-6', 2Ph-2-6), 133.36, 134.47, 135.44 (C-2, 2Ph-1), 146.37 (4a'-C=N), 158.24, 159.84 (2'-C=O, 7'-C=O), 166.67 (1-COOMe), 170.16 (4-COOMe). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{26}H_{25}N_5O_6S$: 536.1598; found: 536.1591.

Methyl (*Z*)-2-(1,3-dimethyl-2,7-dioxo-3a,9a-diphenyl-1,2,3,3a,9,9a-hexahydroimidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazin-6(7*H*)-ylidene)-4-oxopentanoate (3i). Yield 443 mg (85%); pale yellow solid; mp: 162–164 °C. IR (KBr), ν 3436, 3142 (NH), 3061, 3034 (Ph), 2952 (Alk), 1702, 1641 (C=O) cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.31 (s, 3H, CH_3), 2.73 (s, 3H, NCH_3), 2.82 (s, 3H, NCH_3), 3.91 (s, 3H, OCH_3), 4.24 (d, *J* = 17.5 Hz, 1H, CH_2), 4.49 (d, *J* = 17.5 Hz, 1H, CH_2), 6.18 (s, 1H, NH), 6.73-6.88 (m, 4H, 2Ph-2,6), 7.03-7.23 (m, 6H, 2Ph-3-5). ^{13}C NMR (75 MHz, $CDCl_3$): δ 25.16, 26.54 ($2NCH_3$), 29.94 ($COCH_3$), 42.50 (CH_2), 53.43 (OCH_3), 80.67, 84.97 (C-3a', C-9a'), 127.06, 127.66, 127.78, 128.26, 128.43, 129.08 (C-6', 2Ph-2-6), 133.29, 134.41, 134.90 (C-2, 2Ph-1), 146.61 (4a'-C=N), 158.36, 159.84 (2'-C=O, 7'-C=O), 166.62 (1-COOMe), 203.59 (4-COMe). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{26}H_{25}N_5O_5S$: 520.1649; found: 520.1641.

Methyl (*Z*)-2-(1,3-dimethyl-2,7-dioxo-3a,9a-diphenyl-1,2,3,3a,9,9a-hexahydroimidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazin-6(7*H*)-ylidene)-4-oxo-4-phenylbutanoate (3j). Yield 470 mg (81%); pale yellow solid; mp: 356–358 °C. IR (KBr), ν 3434, 3247 (NH), 3062, 3032 (Ph), 2949, 2849 (Alk), 1727, 1690, 1646 (C=O) cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.71 (s, 3H, NCH_3), 2.83 (s, 3H, NCH_3), 3.90 (s, 3H, OCH_3), 4.81 (d, *J* = 17.5 Hz, 1H, CH_2), 5.12 (d, *J* = 17.4 Hz, 1H, CH_2), 6.20 (s, 1H, NH), 6.82-6.91 (m, 4H, 2Ph-2,6), 7.00-7.22 (m, 6H, 2Ph-3-5), 7.52 (t, *J* = 7.4 Hz, 2H, 4-Ph-3,5), 7.62 (t, *J* = 7.4 Hz, 1H, 4-Ph-4), 8.05 (d, *J* = 7.2 Hz, 2H, 4-Ph-2,6). ^{13}C NMR (75 MHz, $CDCl_3$): δ 25.18, 26.60 ($2NCH_3$), 38.26 (CH_2), 53.44 (OCH_3), 80.69, 85.10 (C-3a', C-9a'), 127.09, 127.67, 127.71, 128.23, 128.28, 128.44, 128.53, 128.82, 129.10, 129.23 (C-6', 3Ph-2-6), 133.31, 133.54, 134.47, 136.59 (C-2, 3Ph-1), 146.66 (4a'-C=N), 158.36, 159.91 (2'-C=O, 7'-C=O), 166.80 (1-COOMe), 195.60 (4-COPh). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{31}H_{27}N_5O_5S$: 582.1806; found: 582.1795.

Methyl (*Z*)-3-cyano-2-(1,3-dimethyl-2,7-dioxo-3a,9a-diphenyl-1,2,3,3a,9,9a-hexahydroimidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazin-6(7*H*)-ylidene)propanoate (3k). Yield 448 mg (89%); pale yellow solid; mp: 178–181 °C. IR (KBr), ν 3438, 3139 (NH), 3062, 3034 (Ph), 2951 (Alk), 2253, 2216 (C≡N), 1704, 1643 (C=O) cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.76 (s, 3H, NCH_3), 2.83 (s, 3H, NCH_3), 4.03 (s, 3H, OCH_3), 4.23 (d, *J* = 16.6 Hz, 1H, CH_2), 4.47 (d, *J* = 16.6 Hz, 1H, CH_2), 6.36 (s, 1H, NH), 6.81-6.87 (m, 4H, 2Ph-2,6), 7.06-7.23 (m, 6H, 2Ph-3-5). ^{13}C NMR (75 MHz, $CDCl_3$): δ 16.04 (CH_2), 25.31, 26.60 ($2NCH_3$), 54.01 (OCH_3), 80.77, 85.28 (C-3a', C-9a'), 115.88

(C≡N), 121.60, 127.12, 127.62, 127.75, 128.45, 128.56, 129.28 (C-6', 2Ph-2-6), 133.01, 134.07, 137.93 (C-2, 2Ph-1), 145.46 (4a'-C=N), 157.55, 159.79 (2'-C=O, 7'-C=O), 165.46 (1-COOMe). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{25}H_{22}N_6O_4S$: 503.1496; found: 503.1493.

Diethyl (*Z*)-2-(1,3-diethyl-2,7-dioxo-1,2,3,3a,9,9a-hexahydroimidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazin-6(7*H*)-ylidene)succinate (3l). Yield 364 mg (83%); yellow solid; mp: 60–62 °C. IR (KBr), ν 3235 (NH), 2980, 2937, 2877 (Alk), 1722, 1697, 1642 (C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 1.10 (t, *J* = 7.0 Hz, 3H, CH_3), 1.21-1.33 (m, 6H, $2CH_3$), 1.36 (t, *J* = 7.0 Hz, 3H, CH_3), 3.19-3.41 (m, 3H, NCH_2), 3.50-3.65 (m, 1H, NCH_2), 4.08-4.22 (m, 3H, OCH_2, CH_2), 4.26-4.40 (m, 3H, OCH_2, CH_2), 4.88 (d, *J* = 5.6 Hz, 1H, 9a-H), 5.03 (d, *J* = 5.8 Hz, 1H, 3a-H), 5.26 (s, 1H, NH). ^{13}C NMR (75 MHz, $CDCl_3$): δ 12.71, 13.44, 14.10, 14.23 ($4CH_3$), 33.32 (CH_2), 35.08, 35.67 ($2NCH_2$), 61.21, 62.72 ($2OCH_2$), 64.33, 65.31 (C-3a', C-9a'), 127.18 (C-6'), 134.34 (C-2), 149.46 (4a'-C=N), 158.32, 160.37 (2'-C=O, 7'-C=O), 166.25 (1-COOEt), 169.95 (4-COOEt). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{18}H_{25}N_5O_6S$: 440.1598; found: 440.1589.

4-Ethyl 1-methyl (*Z*)-2-(1,3-diethyl-2,7-dioxo-1,2,3,3a,9,9a-hexahydroimidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazin-6(7*H*)-ylidene)succinate (3m). Yield 400 mg (94%); yellow solid; mp: 70–72 °C. IR (KBr), ν 3235 (NH), 2980, 2937, 2877 (Alk), 1702, 1642 (C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 1.09 (t, *J* = 7.1 Hz, 3H, CH_3), 1.22-1.29 (m, 6H, $2CH_3$), 3.21-3.38 (m, 3H, NCH_2), 3.51-3.61 (m, 1H, NCH_2), 3.89 (s, 3H, OCH_3), 4.07-4.20 (m, 3H, OCH_2, CH_2), 4.30 (d, *J* = 17.1 Hz, 1H, CH_2), 4.87 (dd, *J* = 6.0, 2.4 Hz, 1H, 9a-H), 5.02 (d, *J* = 6.0 Hz, 1H, 3a-H), 5.34 (s, 1H, NH). ^{13}C NMR (75 MHz, $CDCl_3$): δ 12.70, 13.44, 14.22 ($3CH_3$), 33.29 (CH_2), 35.09, 35.67 ($2NCH_2$), 53.31 (OCH_3), 61.23 (OCH_2), 64.34, 65.30 (C-3a', C-9a'), 126.73 (C-6'), 134.78 (C-2), 149.37 (4a'-C=N), 158.31, 160.30 (2'-C=O, 7'-C=O), 166.77 (1-COOMe), 169.87 (4-COOEt). HRMS (ESI): *m/z* $[M + Na]^+$ calcd for $C_{17}H_{23}N_5O_6S$: 448.1258; found: 448.1261.

Diethyl (*Z*)-2-(1-methyl-2,7-dioxo-3-phenyl-1,2,3,3a,9,9a-hexahydroimidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazin-6(7*H*)-ylidene)succinate (3n). Yield 474 mg (97%); pale yellow solid; mp: 149–151 °C. IR (KBr), ν 3435, 3283 (NH), 3063 (Ph), 2998, 2963, 2936 (Alk), 1725, 1692, 1633 (C=N, C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 1.27-1.37 (m, 6H, $2CH_3$), 2.86 (s, 3H, NCH_3), 4.10-4.22 (m, 3H, CH_2, OCH_2), 4.31-4.37 (m, 3H, CH_2, OCH_2), 4.82 (d, *J* = 5.6 Hz, 1H, 9a'-H), 5.44-5.57 (m, 2H, 3a'-H, NH), 7.15 (t, *J* = 7.5 Hz, 1H, Ph-4), 7.38 (t, *J* = 7.9 Hz, 2H, Ph-3,5), 7.71 (d, *J* = 8.1 Hz, 2H, Ph-2,6). ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.11, 14.26 ($2CH_3$), 27.98 (NCH_3), 33.35 (CH_2), 61.27, 62.77 ($2OCH_2$), 65.76, 66.65 (C-3a', C-9a'), 120.79 (Ph-2,6), 124.31, 127.45 (C-6', Ph-4), 129.01 (Ph-3,5), 134.17, 137.99 (C-2, Ph-1), 149.98 (4a'-C=N), 156.27 (2'-C=O), 160.36 (7'-C=O), 166.17 (1-COOEt), 170.04 (4-COOEt). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{21}H_{23}N_5O_6S$: 474.1441; found: 474.1425.

4-Ethyl 1-methyl (*Z*)-2-(1-methyl-2,7-dioxo-3-phenyl-1,2,3,3a,9,9a-hexahydroimidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazin-6(7*H*)-ylidene)succinate (3o). Yield 454 mg (99%); pale yellow solid; mp: 158–160 °C. IR (KBr), ν 3433, 3292 (NH), 3065, 3045 (Ph), 2982, 2951 (Alk), 1724, 1695, 1637 (C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 1.29 (t, *J* = 7.1 Hz, 3H, CH_3), 2.87 (s, 3H, NCH_3), 3.89 (s, 3H, OCH_3), 4.10-4.23 (m, 3H, CH_2, OCH_2), 4.32 (d, *J* = 17.1 Hz, 1H, CH_2), 4.83 (d, *J* = 6.1 Hz, 1H, 9a'-H), 5.46-5.51 (m, 3a'-H, NH), 7.15 (t, *J* = 7.4 Hz, 1H, Ph-4), 7.38 (t, *J* = 7.9 Hz, 2H, Ph-3,5), 7.72 (d, *J* = 8.0 Hz, 2H, Ph-2,6). ^{13}C NMR (150 MHz, $CDCl_3$): δ 14.93 (CH_3), 28.66 (NCH_3), 33.95 (CH_2), 54.10 (OCH_3), 62.02 (OCH_2), 66.28, 67.21 (C-3a', C-9a'), 121.41 (Ph-2,6), 124.99, 127.55 (C-6', Ph-4), 129.70 (Ph-3,5), 135.33, 138.59 (C-2, Ph-1), 150.64 (4a'-C=N), 156.95 (2'-C=O), 161.03 (7'-C=O), 167.38 (1-COOMe), 170.71 (4-COOEt). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{20}H_{21}N_5O_6S$: 460.1285; found: 460.1282.

Diethyl (*Z*)-2-(1-ethyl-2,7-dioxo-3-phenyl-1,2,3,3a,9,9a-hexahydroimidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazin-6(7*H*)-ylidene)succinate (3p). Yield 474 mg (97%); yellow solid; mp: 83–85 °C. IR (KBr), ν 3435, 3248 (NH), 3065 (Ph), 2980, 2936, 2906, 2874 (Alk), 1728, 1640 (C=N, C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 1.15 (t, *J* = 7.1 Hz, 3H, CH_3), 1.29 (t, *J* = 7.2 Hz, 3H, CH_3), 1.35 (t, *J* = 7.1 Hz, 3H, CH_3), 3.27-3.50 (m, 2H, NCH_2), 4.09-4.22 (m, 3H, CH_2, OCH_2), 4.31-4.37 (m, 3H, CH_2, OCH_2), 5.01 (dd, *J* = 6.0, 2.2 Hz, 1H, 9a'-H), 5.41 (d, *J* = 2.3 Hz, 1H, NH), 5.49 (d, *J* = 6.0 Hz, 1H, 3a'-H), 7.14 (t, *J* = 7.4 Hz, 1H, Ph-4), 7.38 (t, *J* = 8.0 Hz, 2H, Ph-3,5), 7.72 (d, *J* = 7.7 Hz, 2H, Ph-2,6). ^{13}C NMR (125 MHz, $CDCl_3$): δ 12.49, 14.01, 14.14 ($3CH_3$), 33.25 (CH_2), 35.57 (NCH_2), 61.17, 62.66 ($2OCH_2$), 64.33, 65.43 (C-3a', C-9a'), 120.62 (Ph-2,6), 124.09, 127.26 (C-6', Ph-4), 128.87 (Ph-3,5), 134.11, 137.91 (C-2, Ph-1), 149.97 (4a'-C=N), 155.61 (2'-C=O), 160.35 (7'-C=O), 166.07 (1-COOEt), 169.95 (4-COOEt). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{22}H_{25}N_5O_6S$: 488.1598; found: 488.1602.

4-Ethyl 1-methyl (*Z*)-2-(1-ethyl-2,7-dioxo-3-phenyl-1,2,3,3a,9,9a-hexahydroimidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazin-6(7*H*)-ylidene)succinate (3q). Yield 450 mg (95%); pale yellow solid; mp: 102–104 °C. IR (KBr), ν 3434, 3250 (NH), 3064 (Ph), 2979 (Alk), 1728, 1641 (C=N, C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 1.15 (t, *J* = 7.2 Hz, 3H CH_3), 1.28 (t, *J* = 7.1 Hz, 3H, CH_3), 3.28-3.49 (m, 2H, NCH_2), 3.89 (s, 3H, OCH_3), 4.08-4.23 (m, 3H, CH_2, OCH_2), 4.32 (d, *J* = 17.1 Hz, 1H, CH_2), 5.01 (dd, *J* = 6.0, 2.5 Hz, 1H, 9a'-H), 5.35 (d, , *J* = 2.6 Hz, 1H, NH), 5.49 (d, *J* = 6.1 Hz, 1H, 3a'-H), 7.15 (t, *J* = 7.4 Hz, 1H, Ph-4), 7.32-7.44 (m, 2H, Ph-3,5), 7.65-7.80 (m, 2H, Ph-2,6). ^{13}C NMR (75 MHz, $CDCl_3$): δ 12.61, 14.25 ($2CH_3$), 33.34, 35.72 (CH_2, NCH_2), 53.36, 61.30 ($2OCH_2$), 64.51, 65.64 (C-3a', C-9a'), 120.77 (Ph-2,6), 124.22, 126.98 (C-6', Ph-4), 128.98 (Ph-3,5), 134.66, 138.06 (C-2, Ph-1), 150.00 (4a'-C=N), 155.73 (2'-C=O), 160.36 (7'-C=O), 166.71 (1-COOMe), 169.94 (4-COOEt). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{21}H_{23}N_5O_6S$: 474.1441; found: 474.1437.

Diethyl (*Z*)-2-(1-methyl-7-oxo-3-phenyl-2-thioxo-1,2,3,3a,9,9a-hexahydroimidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazin-6(7*H*)-ylidene)succinate (3r). Yield 484 mg (99%); pale yellow solid; mp: 188–190 °C. IR (KBr), ν 3445, 3292 (NH), 3063 (Ph), 2983, 2939 (Alk), 1734, 1713, 1646 (C=N, C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 1.30 (t, *J* = 7.2 Hz, 3H, CH_3), 1.36 (t, *J* = 7.1 Hz, 3H, CH_3), 3.18 (s, 3H, NCH_3), 4.09-4.23 (m, 3H, CH_2, OCH_2), 4.33-4.38 (m, 3H, CH_2, OCH_2), 5.09 (dd, *J* = 6.4, 2.4 Hz, 1H, 9a'-H), 5.40 (d, *J* = 6.5 Hz, 1H, 3a'-H), 5.50 (d, *J* = 2.2 Hz, 1H, NH), 7.36 (t, *J* = 7.3 Hz, 1H, Ph-4), 7.45 (t, *J* = 7.5 Hz, 2H, Ph-3,5), 7.54 (d, *J* = 7.7 Hz, 2H, Ph-2,6). ^{13}C NMR (125 MHz, $CDCl_3$): δ 14.01, 14.16 ($2CH_3$), 31.23 (CH_2), 33.27 (NCH_3), 61.28, 62.81 ($2OCH_2$), 69.01, 62.81 (C-3a', C-9a'), 127.69, 127.83 (C-6', Ph-4), 128.15, 128.87 (Ph-2,3,5,6), 133.65, 138.09 (C-2, Ph-1), 150.80 (4a'-C=N), 160.18 (7'-C=O), 166.06 (1-COOEt), 169.92 (4-COOEt), 182.79 (2'-C=S). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{21}H_{23}N_5O_5S_2$: 490.1213; found: 490.1215.

4-Ethyl 1-methyl (*Z*)-2-(1-methyl-7-oxo-3-phenyl-2-thioxo-1,2,3,3a,9,9a-hexahydroimidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazin-6(7*H*)-ylidene)succinate (3s). Yield 465 mg (98%); yellow solid; mp: 183–185 °C. IR (KBr), ν 3436, 3252 (NH), 3064 (Ph), 2979, 953 (Alk), 1731, 1641 (C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 1.28 (t, *J* = 7.2 Hz, 3H, CH_3), 3.15 (s, 3H, NCH_3), 3.90 (s, 3H, OCH_3), 4.03-4.22 (m, 3H, CH_2, OCH_2), 4.33 (d, *J* = 17.1 Hz, 1H, CH_2), 5.06 (dd, *J* = 6.6, 2.7 Hz, 1H, 9a'-H), 5.37 (d, *J* = 6.6 Hz, 1H, 3a'-H), 5.50 (d, *J* = 2.8 Hz, 1H, NH), 7.34 (t, *J* = 7.2 Hz, 1H, Ph-4), 7.44 (t, *J* = 7.6 Hz, 2H, Ph-3,5), 7.54 (d, *J* = 7.6 Hz, 2H, Ph-2,6). ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.26 (CH_3), 31.34, 33.34 (CH_2, NCH_3), 53.45 (OCH_3), 61.38 (OCH_2), 69.09, 69.65 (C-3a', C-9a'), 127.28, 127.84 (C-6', Ph-4), 128.22, 128.93 (Ph-2,3,5,6), 134.27, 138.30 (C-2, Ph-1), 150.79 (4a'-C=N), 160.27 (7'-C=O), 166.72 (1-COOMe), 169.94 (4-COOEt), 182.88 (2'-C=S). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{20}H_{21}N_5O_5S_2$: 476.1056; found: 476.1044.

Diethyl (*Z*)-2-(1-ethyl-7-oxo-3-phenyl-2-thioxo-1,2,3,3a,9,9a-hexahydroimidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazin-6(7*H*)-ylidene)succinate (3t). Yield 497 mg (99%); yellow solid; mp: 119–121 °C. IR (KBr), ν 3434, 3185 (NH), 3067 (Ph), 2972, 2935, 2897, 2875 (Alk), 1737, 1718, 1691, 1643 (C=N, C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 1.20 (t, *J* = 7.1 Hz, 3H, CH_3), 1.29 (t, *J* = 7.1 Hz, 3H, CH_3), 1.37 (t, *J* = 7.1 Hz, 3H, CH_3), 3.60 (m, 1H, NCH_2), 3.85-3.97 (m, 1H, CH_2), 4.09-4.22 (m, 3H, CH_2, OCH_2), 4.32-4.39 (m, 3H, CH_2, OCH_2), 5.25 (dd, *J* = 6.4, 2.3 Hz, 1H, 9a'-H), 5.37-5.42 (m, 2H, 3a'-H, NH), 7.36 (t, *J* = 7.3 Hz, 1H, Ph-4), 7.45 (t, *J* = 7.7 Hz, 2H, Ph-3,5), 7.54 (d, *J* = 7.7 Hz, 2H, Ph-2,6). ^{13}C NMR (75 MHz, $CDCl_3$): δ 11.81, 14.11, 14.25 ($3CH_3$), 33.41 (CH_2), 38.92 (NCH_2), 61.35, 62.88 ($2OCH_2$), 67.71, 62.88 (C-3a', C-9a'), 127.78, 127.84 (C-6', Ph-4), 128.37, 128.90 (Ph-2,3,5,6), 133.77, 138.27 (C-2, Ph-1) 150.90 (4a'-C=N), 160.34 (7'-C=O), 166.18 (1-COOEt), 169.97 (4-COOEt), 182.01 (2'-C=S). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{22}H_{25}N_5O_5S_2$: 504.1369; found: 504.1359.

4-Ethyl 1-methyl (*Z*)-2-(1-ethyl-7-oxo-3-phenyl-2-thioxo-1,2,3,3a,9,9a-hexahydroimidazo[4,5-*e*]thiazolo[3,2-*b*][1,2,4]triazin-6(7*H*)-ylidene)succinate (3u). Yield 474 mg (97%); yellow solid; mp: 89–91 °C. IR (KBr), ν 3434, 3253 (NH), 3064 (Ph), 2978, 2953 (Alk), 1731, 1641 (C=N, C=O) cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.21 (t, *J* = 7.1 Hz, 3H, CH_3), 1.29 (t, *J* = 7.2 Hz, 3H, CH_3), 3.60-3.71 (m, 1H, NCH_2), 3.88-3.95 (m, 4H, NCH_2, OCH_3), 4.08-4.23 (m, 3H, CH_2, OCH_2), 4.33 (d, *J* = 17.1 Hz, 1H, CH_2), 5.27 (d, *J* = 6.2 Hz, 1H, 9a'-H), 5.39 (d, *J* = 6.6 Hz, 1H, 3a'-H), 5.46 (s, 1H, NH), 7.36 (t, *J* = 7.2 Hz, 1H, Ph-4), 7.45 (t, *J* = 7.5 Hz, 2H, Ph-3,5), 7.54 (d, *J* = 7.9 Hz, 2H, Ph-2,6). ^{13}C NMR (150 MHz, $CDCl_3$): 12.45, 14.93 ($2CH_3$), 34.01 (CH_2), 39.55 (NCH_2), 54.18 (OCH_3), 62.07 (OCH_2), 68.26, 69.56 (C-3a', C-9a'), 127.89, 128.55 (C-6', Ph-4), 129.08, 129.59 (Ph-2,3,5,6), 134.92, 138.85 (C-2, Ph-1), 151.55 (4a'-C=N), 160.99 (7'-C=O), 167.40 (1-COOMe), 170.61 (4-COOEt), 182.59 (2'-C=S). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{21}H_{23}N_5O_5S_2$: 490.1213; found: 490.1200.

General procedure for the synthesis of compounds 6a-n.

Method A. To a suspension of imidazo[4,5-*e*]thiazolo[2,3-*c*][1,2,4]triazine **7a-l**[1] (1 mmol) and pyridinium ylide **1a,b** (1.5 mmol) in dichloromethane (5 ml), triethylamine (0.208 ml, 1.5 mmol) was added. The reaction mixture was stirred at room temperature for 2 h.

For **6a-d,g-n**: After standing until the solvent had evaporated, 20 ml of water was added. The resulting mixture was stirred and the precipitate formed was filtered off, washed with 5 ml of water and dried.

For **6e,f**: The reaction mixture was extracted by 5 ml of HCl (10% aqueous solution), then organic layer was washed by 5 ml of $NaHCO_3$ (10% aqueous solution) and 5 ml of water. The organic layer was dried by anh. Na_2SO_4, evaporated and dried.

Method B. To the boiling suspension of **3a** (411 mg, 1 mmol) in 10 ml of EtOH (for **6a**) or 10 ml of MeOH (for **6b**) triethylamine (0.139 ml, 1 mmol) was added. The reaction mixture was stirred under reflux for 1 h. After standing to room temperature, the precipitate formed was filtered off, washed with cold MeOH, and dried.

The compounds were recrystallized from MeOH if necessary.

Diethyl (*Z*)-2-(1,3-dimethyl-2,8-dioxo-1,2,3,3a,4,9a-hexahydroimidazo[4,5-*e*]thiazolo[2,3-*c*][1,2,4]triazin-7(8*H*)-ylidene)succinate (6a). Method A: yield 402 mg (98%); Method B: yield 177 mg (44%); bright yellow solid; mp: 152–154 °C. IR (KBr), ν 3465, 3434, 3291, 3259 (NH), 2983, 2963 (Alk), 1728, 1710, 1689, 1642 (C=O) cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.29 (t, *J* = 7.1 Hz, 3H, CH_3), 1.35 (t, *J* = 7.1 Hz, 3H, CH_3), 2.79 (s, 3H, NCH_3), 3.04 (s, 3H, NCH_3), 4.04 (d, *J* = 17.1 Hz, 1H, CH_2), 4.18 (q, *J* = 7.1 Hz, 2H, OCH_2), 4.31-4.40 (m, 3H, CH_2, OCH_2), 4.72 (dd, *J* = 6.3, 1.9 Hz, 1H, 3a'-H), 5.53 (d, *J* = 6.2 Hz, 1H, 9a'-H), 6.09 (d, *J* = 1.5 Hz, 1H, NH). ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.13, 14.13 ($2CH_3$), 27.60, 31.73, 32.76 ($2NCH_3$, CH_2), 61.27, 62.54 ($2OCH_2$), 65.96, 66.20 (C-3a',

C-9a'), 124.10 (C-7'), 137.61, 139.09 (C-2, 5a'-C=N), 158.85 (2'-C=O), 164.22 (8'-C=O), 166.66 (1-COOEt), 170.66 (4-COOEt). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{16}H_{21}N_5O_6S$: 412.1285; found: 412.1283.

4-Ethyl 1-methyl (*Z*)-2-(1,3-dimethyl-2,8-dioxo-1,2,3,3a,4,9a-hexahydroimidazo[4,5-*e*]thiazolo[2,3-*c*][1,2,4]triazin-7(8*H*)-ylidene)succinate (6b). Method A: yield 373 mg (94%); Method B: yield 250 mg (63%); bright yellow solid; mp: 185–187 °C. IR (KBr), ν 3300, 3279 (NH), 2984, 2959 (Alk), 1728, 1705, 1643 (C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 1.29 (t, *J* = 7.1 Hz, 3H, CH_3), 2.79 (s, 3H, NCH_3), 3.04 (s, 3H, NCH_3), 3.89 (s, 3H, OCH_3), 4.03 (d, *J* = 17.1 Hz, 1H, CH_2), 4.19 (q, *J* = 7.1 Hz, 2H, OCH_2), 4.37 (d, *J* = 17.1 Hz, 1H, CH_2), 4.72 (dd, *J* = 6.3, 1.6 Hz, 1H, 3a'-H), 5.53 (d, *J* = 6.3 Hz, 1H, 9a'-H), 6.12 (s, 1H, NH). ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.19 (CH_3), 27.55, 31.69, 32.69 ($2NCH_3$, CH_2), 53.22 (OCH_3), 61.22 (OCH_2), 65.93, 66.19 (C-3a', C-9a'), 123.48 (C-7'), 138.16, 138.84 (C-2, 5a'-C=N), 158.82 (2'-C=O), 164.08 (8'-C=O), 167.14 (1-COOMe), 170.67 (4-COOEt). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{15}H_{19}N_5O_6S$: 398.1129; found: 398.1119.

1-Ethyl 4-methyl (*Z*)-2-(1,3-dimethyl-2,8-dioxo-1,2,3,3a,4,9a-hexahydroimidazo[4,5-*e*]thiazolo[2,3-*c*][1,2,4]triazin-7(8*H*)-ylidene)succinate (6c). Yield 373 mg (94%); bright yellow solid; mp: 164–166 °C. IR (KBr), ν 3434, 3292 (NH), 2981, 2954 (Alk), 1711, 1644 (C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 1.30 (t, *J* = 7.1 Hz, 3H, CH_3), 2.74 (s, 3H, NCH_3), 3.00 (s, 3H, NCH_3), 3.67 (s, 3H, OCH_3), 4.03 (d, *J* = 17.1 Hz, 1H, CH_2), 4.26-4.33 (m, 3H, OCH_2, CH_2), 4.68 (d, *J* = 6.0 Hz, 1H, 3a'-H), 5.48 (d, *J* = 6.2 Hz, 1H, 9a'-H), 5.98 (s, 1H, NH). ^{13}C NMR (125 MHz, $CDCl_3$): δ 14.04 (CH_3), 27.51, 31.65, 32.43 ($2NCH_3$, CH_2), 53.23 (OCH_3), 62.50 (OCH_2), 65.86, 66.11 (C-3a', C-9a'), 123.73 (C-7'), 137.66, 138.95 (C-2, 5a'-C=N), 158.75 (2'-C=O), 164.10 (8'-C=O), 166.50 (1-COOEt), 171.04 (4-COOMe). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{15}H_{19}N_5O_6S$: 398.1129; found: 398.1127.

Dimethyl (*Z*)-2-(1,3-dimethyl-2,8-dioxo-1,2,3,3a,4,9a-hexahydroimidazo[4,5-*e*]thiazolo[2,3-*c*][1,2,4]triazin-7(8*H*)-ylidene)succinate (6d). Yield 352 mg (92%); bright yellow solid; mp: 185–187 °C. IR (KBr), ν 3315, 3261 (NH), 2955 (Alk), 1732, 1709, 1690, 1641 (C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 2.71 (s, 3H, NCH_3), 2.97 (s, 3H, NCH_3), 3.67 (s, 3H, OCH_3), 3.83 (s, 3H, OCH_3), 3.94 (d, *J* = 17.2 Hz, 1H, CH_2), 4.35 (d, *J* = 17.1 Hz, 1H, CH_2), 4.64 (d, *J* = 6.0, 1.6 Hz, 1H, 3a'-H), 5.45 (d, *J* = 6.2 Hz, 1H, 9a'-H), 6.26 (s, 1H, NH). ^{13}C NMR (75 MHz, $CDCl_3$): δ 27.63, 31.74, 32.54 ($2NCH_3$, CH_2), 52.32, 53.30 (OCH_3), 65.94, 66.26 (C-3a', C-9a'), 123.53 (C-7'), 138.09, 138.95 (C-2, 5a'-C=N), 158.83 (2'-C=O), 164.15 (8'-C=O), 167.12 (1-COOMe), 170.93 (4-COOMe). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{14}H_{17}N_5O_6S$: 384.0972; found: 384.0964.

Diethyl (*Z*)-2-(1,3-diethyl-2,8-dioxo-1,2,3,3a,4,9a-hexahydroimidazo[4,5-*e*]thiazolo[2,3-*c*][1,2,4]triazin-7(8*H*)-ylidene)succinate (6e). Yield 356 mg (81%); yellow solid; mp: 128–130 °C.

IR (KBr), ν 3261 (NH), 2982, 2934, 2877 (Alk), 1725, 1699, 1685, 1647 (C=O) cm–1. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.09 (t, *J* = 7.2 Hz, 3H, CH_3), 1.15 (t, *J* = 7.1 Hz, 3H, CH_3), 1.23 (t, *J* = 7.2 Hz, 3H, CH_3), 1.29 (t, *J* = 7.2 Hz, 3H, CH_3), 3.06-3.25 (m, 1H, NCH_2), 3.27-3.42 (m, 2H, NCH_2), 3.58-3.69 (m, 1H, NCH_2), 3.98 (d, *J* = 17.1 Hz, 1H, CH_2), 4.13 (q, *J* = 7.1 Hz, 2H, OCH_2), 4.20-4.38 (m, 3H, OCH_2, CH_2), 4.75 (d, *J* = 6.1 Hz, 1H, 3a-H), 5.54 (d, *J* = 6.1 Hz, 1H, 9a-H), 6.01 (s, 1H, NH). ^{13}C NMR (75 MHz, $CDCl_3$): δ 12.80, 13.54, 14.15, 14.26 ($4CH_3$), 32.82 (CH_2), 35.43, 38.52 ($2NCH_2$), 61.21, 62.52 ($2OCH_2$), 63.71, 63.91 (C-3a', C-9a'), 124.23 (C-7'), 137.50, 138.93 (C-2, 5a'-C=N), 158.12 (2'-C=O), 164.22 (8'-C=O), 166.68 (1-COOEt), 170.55 (4-COOEt). HRMS (ESI): *m/z* [M + H]$^+$ calcd for $C_{18}H_{25}N_5O_6S$: 440.1598; found: 440.1602.

4-Ethyl 1-methyl (*Z*)-2-(1,3-diethyl-2,8-dioxo-1,2,3,3a,4,9a-hexahydroimidazo[4,5-*e*]thiazolo[2,3-*c*][1,2,4]triazin-7(8*H*)-ylidene)succinate (6f). Yield 315 mg (74%); yellow solid; mp: 157–159 °C. IR (KBr), ν 3296, 3269 (NH), 2974, 2934, 2876 (Alk), 1721, 1684, 1644 (C=O) cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.11-1.24 (m, 6H, $2CH_3$), 1.29 (t, *J* = 7.1 Hz, 3H, CH_3), 3.12-3.24 (m, 1H, NCH_2), 3.26-3.47 (m, 2H, NCH_2), 3.63-3.75 (m, 1H, NCH_2), 3.89 (s, 3H, OCH_3), 4.04 (d, *J* = 17.1 Hz, 1H, CH_2), 4.19 (q, *J* = 7.1 Hz, 2H, OCH_2), 4.36 (d, *J* = 17.1 Hz, 1H, CH_2), 4.81 (d, *J* = 6.0 Hz, 1H, 3a-H), 5.59 (d, *J* = 6.0 Hz, 1H, 9a-H), 6.06 (s, 1H, NH). ^{13}C NMR (75 MHz, $CDCl_3$): δ 12.76, 13.55, 14.22 ($3CH_3$), 32.75 (CH_2), 35.40, 38.51 ($2NCH_2$), 53.20 (OCH_3), 61.26 (OCH_2), 63.75, 63.91 (C-3a', C-9a'), 123.59 (C-7'), 138.10, 138.71 (C-2, 5a'-C=N), 158.10 (2'-C=O), 164.08 (8'-C=O), 167.18 (1-COOMe), 170.58 (4-COOEt). HRMS (ESI): *m/z* [M + H]$^+$ calcd for $C_{17}H_{23}N_5O_6S$: 426.1442; found: 426.1428.

Diethyl (*Z*)-2-(3-methyl-2,8-dioxo-1-phenyl-1,2,3,3a,4,9a-hexahydroimidazo[4,5-*e*]thiazolo[2,3-*c*][1,2,4]triazin-7(8*H*)-ylidene)succinate (6g). Yield 463 mg (98%); yellow solid; mp: 153–155 °C. IR (KBr), ν 3308 (NH), 3065 (Ph), 2983, 2939, 2905 (Alk), 1718, 1645 (C=N, C=O) cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.22 (t, *J* = 7.1 Hz, 3H, CH_3), 1.33 (t, *J* = 7.2 Hz, 3H, CH_3), 2.85 (s, 3H, NCH_3), 3.78 (d, *J* = 17.1 Hz, 1H, CH_2), 4.05-4.12 (m, 3H, CH_2, OCH_2), 4.31 (q, *J* = 7.2 Hz, 2H, OCH_2), 4.92 (d, *J* = 6.2 Hz, 1H, 3a'-H), 6.13 (d, *J* = 6.3 Hz, 1H, 9a'-H), 6.18 (s, 1H, NH), 7.23-7.39 (m, 5H, Ph). ^{13}C NMR (150 MHz, $CDCl_3$): δ 14.79, 14.86 ($2CH_3$), 28.10 (NCH_3), 33.11 (CH_2), 61.82, 63.21 ($2OCH_2$), 65.97, 66.49 (C-3a', C-9a'), 124.95, 126.30, 127.24, 129.59 (C-7', Ph-2-6), 137.89, 138.211, 140.01 (C-2, 5a'-C=N, Ph-1) 157.59 (2'-C=O), 163.57 (8'-C=O), 167.33 (1-COOEt), 171.20 (4-COOEt). HRMS (ESI): *m/z* [M + H]$^+$ calcd for $C_{21}H_{23}N_5O_6S$: 474.1442; found: 474.1427.

4-Ethyl 1-methyl (*Z*)-2-(3-methyl-2,8-dioxo-1-phenyl-1,2,3,3a,4,9a-hexahydroimidazo[4,5-*e*]thiazolo[2,3-*c*][1,2,4]triazin-7(8*H*)-ylidene)succinate (6h). Yield 454 mg (99%); yellow solid; mp: 200–202 °C. IR (KBr), ν 3308 (NH), 3074 (Ph), 2985, 2953, 2901, 2848 (Alk), 1717, 1693, 1646 (C=N, C=O) cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.22 (t, *J* = 7.1 Hz, 3H, CH_3), 2.88 (s, 3H, NCH_3), 3.75-3.89 (m, 4H, CH_2, OCH_3), 4.00-4.12 (m, 3H, CH_2, OCH_2), 4.96 (d, *J* = 6.4 Hz, 1H, 3a'-H), 6.12-

6.16 (m, 2H, 9a'-H, NH), 7.23-7.40 (m, 5H, Ph). ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.17 (CH_3), 27.46 (NCH_3), 32.48 (CH_2), 53.22 (OCH_3), 61.14 (OCH_2), 65.38, 65.80 (C-3a', C-9a'), 124.07 (C-7'), 125.76 (Ph-2,6), 126.63 (Ph-4), 128.93 (Ph-3,5), 137.46, 137.56, 139.24 (C-2, 5a'-C=N, Ph-1), 156.97 (2'-C=O), 162.86 (8'-C=O), 167.16 (1-COOMe), 170.31 (4-COOEt). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{20}H_{21}N_5O_6S$: 460.1285; found: 460.1273.

Diethyl (*Z*)-2-(3-ethyl-2,8-dioxo-1-phenyl-1,2,3,3a,4,9a-hexahydroimidazo[4,5-*e*]thiazolo[2,3-*c*][1,2,4]triazin-7(8*H*)-ylidene)succinate (6i). Yield 463 mg (95%); yellow solid; mp: 148–150 °C. IR (KBr), ν 3291 (NH), 2982, 2937, 2905 (Alk), 1733, 1709, 1646 (C=N, C=O) cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.12-1.27 (m, 6H, $2CH_3$), 1.33 (t, *J* = 7.1 Hz, 3H, CH_3), 3.18-3.30 (m, 1H, NCH_2), 3.45-3.57 (m, 1H, NCH_2), 3.79 (d, *J* = 17.1 Hz, 1H, CH_2), 4.00-4.18 (m, 3H, CH_2, OCH_2), 4.31 (q, *J* = 7.1 Hz, 2H, OCH_2), 5.06 (dd, *J* = 6.2, 2.2 Hz, 1H, 3a'-H), 6.06-6.17 (m, 2H, 9a'-H, NH), 7.26-7.41 (m, 5H, Ph). ^{13}C NMR (75 MHz, $CDCl_3$): δ 12.62 (CH_3), 14.12 (CH_3), 14.18 (CH_3), 32.49 (CH_2), 35.41 (NCH_2), 61.10, 62.48, 63.61, 65.31 ($2OCH_2$, C-3a', C-9a'), 124.29 (C-7'), 125.62 (Ph-2,6), 126.45 (Ph-4), 128.86 (Ph-3,5), 137.25, 137.57, 139.09 (Ph-1, 5a'-C=N, C-2), 156.45 (2'-C=O), 162.91 (8'-C=O), 166.66 (1-COOEt), 170.51 (4-COOEt). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{22}H_{25}N_5O_6S$: 488.1598; found: 488.1590.

4-Ethyl 1-methyl (*Z*)-2-(3-ethyl-2,8-dioxo-1-phenyl-1,2,3,3a,4,9a-hexahydroimidazo[4,5-*e*]thiazolo[2,3-*c*][1,2,4]triazin-7(8*H*)-ylidene)succinate (6j). Yield 468 mg (99%); yellow solid; mp: 196–198 °C. IR (KBr), ν 3268, 3155 (NH), 2977, 2957, 2935 (Alk), 1729, 1711 (C=N, C=O) cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.18-1.24 (m, 6H, $2CH_3$), 3.18-3.30 (m, 1H, NCH_2), 3.45-3.57 (m, 1H, NCH_2), 3.79 (d, *J* = 17.1 Hz, 1H, CH_2), 3.86 (s, 3H, OCH_3), 4.04-4.12 (m, 3H, CH_2, OCH_2), 5.07 (d, *J* = 6.2 Hz, 1H, 3a'-H), 6.09-6.14 (m, 2H, 9a'-H, NH), 7.22-7.39 (m, 5H, Ph). ^{13}C NMR (75 MHz, $CDCl_3$): δ 12.69, 14.19 ($2CH_3$), 32.48 (CH_2), 35.47 (NCH_2), 53.21 (OCH_3), 61.13 (OCH_2), 63.63, 65.36 (C-3a', C-9a'), 124.00, 125.66, 126.51, 128.90 (C-7', Ph-2-6), 137.55, 139.10 (C-2, 5a'-C=N, Ph-1), 156.45 (2'-C=O), 162.85 (8'-C=O), 167.18 (1-COOMe), 170.32 (4-COOEt). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{21}H_{23}N_5O_6S$: 474.1442; found: 474.1445.

Diethyl (*Z*)-2-(3-methyl-8-oxo-1-phenyl-2-thioxo-1,2,3,3a,4,9a-hexahydroimidazo[4,5-*e*]thiazolo[2,3-*c*][1,2,4]triazin-7(8*H*)-ylidene)succinate (6k). Yield 484 mg (99%); yellow solid; mp: 110–112 °C. IR (KBr), ν 3329, 3289 (NH), 3063 (Ph), 2981, 2934, 2873 (Alk), 1735, 1718, 1643 (C=N, C=O) cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.24 (t, *J* = 7.1 Hz, 3H, CH_3), 1.34 (t, *J* = 7.1 Hz, 3H, CH_3), 3.21 (s, 3H, NCH_3), 3.78 (d, *J* = 17.1 Hz, 1H, CH_2), 3.92-4.18 (m, 3H, CH_2, OCH_2), 4.33 (q, *J* = 7.1 Hz, 2H, OCH_2), 5.18 (d, *J* = 7.1 Hz, 1H, 3a'-H), 6.15 (d, *J* = 7.0 Hz, 1H, 9a'-H), 6.28 (s, 1H, NH), 7.23-7.42 (m, 5H, Ph). ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.13 (CH_3), 14.21 (CH_3), 31.47, 32.49 (NCH_3, CH_2), 61.20 (OCH_2), 62.64 (OCH_2), 67.51, 68.82 (C-3a', C-9a'), 124.95 (C-7'), 128.35 (Ph-4), 128.92, 129.14 (Ph-2,6, Ph-3,5), 136.74, 138.78, 139.47 (Ph-1, 5a'-C=N, C-2), 162.35 (8'-C=O),

166.55 (1-COOEt), 170.32 (4-COOEt), 183.64 (2'-C=S). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{21}H_{23}N_5O_5S_2$: 490.1213; found: 490.1203.

4-Ethyl 1-methyl (*Z*)-2-(3-methyl-8-oxo-1-phenyl-2-thioxo-1,2,3,3a,4,9a-hexahydroimidazo[4,5-*e*]thiazolo[2,3-*c*][1,2,4]triazin-7(8*H*)-ylidene)succinate (6l). Yield 470 mg (99%); yellow solid; mp: 188–190 °C. IR (KBr), ν 3359 (NH), 3056 (Ph), 2987, 2945, 2908 (Alk), 1728, 1711, 1688, 1644 (C=N, C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 1.24 (t, *J* = 7.1 Hz, 3H, CH_3), 3.19 (s, 3H, NCH_3), 3.75 (d, *J* = 17.1 Hz, 1H, CH_2), 3.88 (s, 3H, OCH_3), 4.03 (d, *J* = 17.1 Hz, 1H, CH_2), 4.11 (q, *J* = 7.2 Hz, 2H, OCH_2), 5.16 (d, *J* = 9.1 Hz, 1H, 3a'-H), 6.14 (d, *J* = 7.0 Hz, 1H, 9a'-H), 6.30 (s, 1H, NH), 7.23-7.46 (m, 5H, Ph). ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.15 (CH_3), 31.34, 32.42 (NCH_3, CH_2), 53.32 (OCH_3), 61.30 (OCH_2), 67.44, 68.86 (C-3a', C-9a'), 124.25 (C-7'), 128.30 (Ph-4), 128.81, 129.09 (Ph-2,6, Ph-3,5), 137.43, 138.75, 139.33 (Ph-1, 5a'-C=N, C-2), 162.19 (8'-C=O), 167.03 (1-COOMe), 170.47 (4-COOEt), 183.50 (2'-C=S). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{20}H_{21}N_5O_5S_2$: 476.1056; found: 476.1048.

Diethyl (*Z*)-2-(3-ethyl-8-oxo-1-phenyl-2-thioxo-1,2,3,3a,4,9a-hexahydroimidazo[4,5-*e*]thiazolo[2,3-*c*][1,2,4]triazin-7(8*H*)-ylidene)succinate (6m). Yield 483 mg (96%); yellow solid; mp: 170–172 °C. IR (KBr), ν 3324 (NH), 3038 (Ph), 2986, 2958, 2939, 2904 (Alk), 1722, 1691, 1646 (C=N, C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 1.20-1.40 (m, 9H, $3CH_3$), 3.52-3.63 (m, 1H, NCH_2), 3.75 (d, *J* = 17.1 Hz, 1H, CH_2), 3.89-4.02 (m, 1H, NCH_2), 4.00-4.18 (m, 3H, CH_2, OCH_2), 4.33 (q, *J* = 7.1 Hz, 2H, OCH_2), 5.25 (d, *J* = 7.1 Hz, 1H, 3a'-H), 6.12 (d, *J* = 7.1 Hz, 1H, 9a'-H), 6.27 (s, 1H, NH), 7.25-7.23 (m, 2H, Ph), 7.31-7.44 (m, 3H, Ph). ^{13}C NMR (150 MHz, $CDCl_3$): δ 12.53 (CH_3), 14.82 (CH_3), 14.86 (CH_3), 33.11 (CH_2), 39.78 (NCH_2), 63.02 (OCH_2), 63.32 (OCH_2), 67.72, 68.06 (C-3a', C-9a'), 125.05 (C-7'), 128.95 (Ph-4), 129.62, 129.75 (Ph-2,6, Ph-3,5), 138.03, 139.33, 140.10 (Ph-1, 5a'-C=N, C-2), 162.86 (8'-C=O), 167.23 (1-COOEt), 171.50 (4-COOEt), 183.31 (2'-C=S). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{22}H_{25}N_5O_5S_2$: 504.1370; found: 504.1356.

4-Ethyl 1-methyl (*Z*)-2-(3-ethyl-8-oxo-1-phenyl-2-thioxo-1,2,3,3a,4,9a-hexahydroimidazo[4,5-*e*]thiazolo[2,3-*c*][1,2,4]triazin-7(8*H*)-ylidene)succinate (6n). Yield 460 mg (94%); yellow solid; mp: 176–178 °C. IR (KBr), ν 3337, 3318 (NH), 3037 (Ph), 2984, 2949, 2904 (Alk), 1722, 1699, 1645 (C=N, C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 1.25 (m, 6H, $2CH_3$), 3.50-3.62 (m, 1H, NCH_2), 3.76 (d, *J* = 17.1 Hz, 1H, CH_2), 3.83-3.99 (m, 4H, NCH_2, OCH_3), 4.00-4.15 (m, 3H, CH_2, OCH_2), 5.24 (dd, *J* = 7.2, 2.0 Hz, 1H, 3a'-H), 6.11 (d, *J* = 7.2 Hz, 1H, 9a'-H), 6.39 (d, *J* = 2.0 Hz, 1H, NH), 7.22-7.30 (m, 2H, Ph), 7.34-7.43 (m, 3H, Ph). ^{13}C NMR (150 MHz, $CDCl_3$): δ 12.60 (CH_3), 14.84 (CH_3), 33.09 (CH_2), 39.84 (NCH_2), 54.05 (OCH_3), 62.03 (OCH_2), 67.68, 68.11 (C-3a', C-9a'), 124.74 (C-7'), 128.98 (Ph-4), 129.63, 19.77 (Ph-2,6, Ph-3,5), 138.32, 139.23, 140.04 (Ph-1, 5a'-C=N, C-2), 162.80 (8'-C=O), 167.75 (1-COOMe), 171.28 (4-COOEt), 183.36 (2'-C=S). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{21}H_{23}N_5O_5S_2$: 490.1213; found: 490.1205.

Procedure for the synthesis of compound 11

To a suspension of benzo[4,5]imidazo[2,1-*b*]thiazol **8**[2] (260 mg, 1 mmol) and pyridinium ylide **1a** (369 mg, 1.5 mmol) in dichloromethane (5 ml), triethylamine (0.208 ml, 1.5 mmol) was added. The reaction mixture was stirred at room temperature for 2 h. The reaction mixture was extracted by 5 ml of HCl (10% aqueous solution), then organic layer was washed by 5 ml of $NaHCO_3$ (10% aqueous solution) and 5 ml of water. The organic layer was dried by anh. Na_2SO_4 and evaporated. The compound was recrystallized from MeCN.

(*Z*)-4-Ethyl 1-methyl 2-(3-oxobenzo[4,5]imidazo[2,1-*b*]thiazol-2(3*H*)-ylidene)succinate (11). Yield 225 mg (65%); yellow solid; mp: 177–180 °C. IR (KBr), ν 2983, 2958, 2878, 2848 (Alk), 1730, 1704 (C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 1.26 (t, *J* = 7.1 Hz, 3H, CH_3), 3.91 (s, 3H, OCH_3), 4.17 (q, *J* = 7.2 Hz, 2H, OCH_2), 4.29 (s, 2H, CH_2), 7.26-7.37 (m, 2H, Ar), 7.61 (d, *J* = 7.7 Hz, 1H, Ar), 7.89 (d, *J* = 7.9 Hz, 1H, Ar). ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.29 (CH_3), 33.24 (CH_2), 53.71 (OCH_3), 61.52 (OCH_2), 112.92, 120.02, 124.75, 126.46 (C-5'-8'), 130.35, 131.37 (C-2', C-8a'), 140.47 (C-2), 148.98 (C-4a'), 154.82 (9a'-C=N), 158.69 (3'-C=O), 166.52 (1-COOMe), 169.29 (4-COOEt). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{16}H_{14}N_2O_5S$: 347.0696; found: 347.0703.

Procedure for the synthesis of compound 12

To a suspension of thiazolidin-4-one **9**[2] (338 mg, 1 mmol) and pyridinium ylide **1a** (369 mg, 1.5 mmol) in dichloromethane (5 ml), triethylamine (0.208 ml, 1.5 mmol) was added. The reaction mixture was stirred at room temperature for 2 h. After standing until the solvent had evaporated, 20 ml of water was added. The resulting mixture was stirred and the precipitate formed was filtered off, washed with 5 ml of water and dried.

4-Ethyl 1-methyl (*Z*)-2-((*Z*)-4-oxo-3-phenyl-2-(phenylimino)thiazolidin-5-ylidene)succinate (12). Yield 380 mg (90%); yellow solid; mp: 113–114 °C. IR (KBr), ν 3066 (Ph), 2990, 2950, 2906, 2842 (Alk), 1737, 1714, 1634 (C=N, C=O) cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 1.27 (t, *J* = 7.1 Hz, 3H, CH_3), 3.86 (s, 3H, OCH_3), 4.18 (q, *J* = 7.1 Hz, 2H, OCH_2), 4.28 (s, 2H, CH_2), 6.97 (d, *J* = 7.2 Hz, 2H, Ph-2,6), 7.16 (t, *J* = 7.4 Hz, 1H, Ph-4), 7.35 (t, *J* = 7.8 Hz, 2H, Ph-3,5), 7.40-7.50 (m, 3H, Ph-3,4,5), 7.51-7.58 (m, 2H, Ph-2,6). ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.24 (CH_3), 33.21 (CH_2), 53.23 (OCH_3), 61.16 (OCH_2), 121.03 (Ph-2,6), 125.06 (Ph-4), 125.80 (C-5'), 128.09 (Ph-3,5), 129.17 (Ph-4), 129.30 (Ph-2,6), 129.36 (Ph-3,5), 134.18 (2'-C=N), 137.05 (C-2), 147.43 (Ph-1), 151.81 (Ph-1), 165.33 (4'-C=O), 167.08 (1-COOMe), 170.01 (4-COOEt). HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{22}H_{20}N_2O_5S_2$: 425.1165; found: 425.1173.

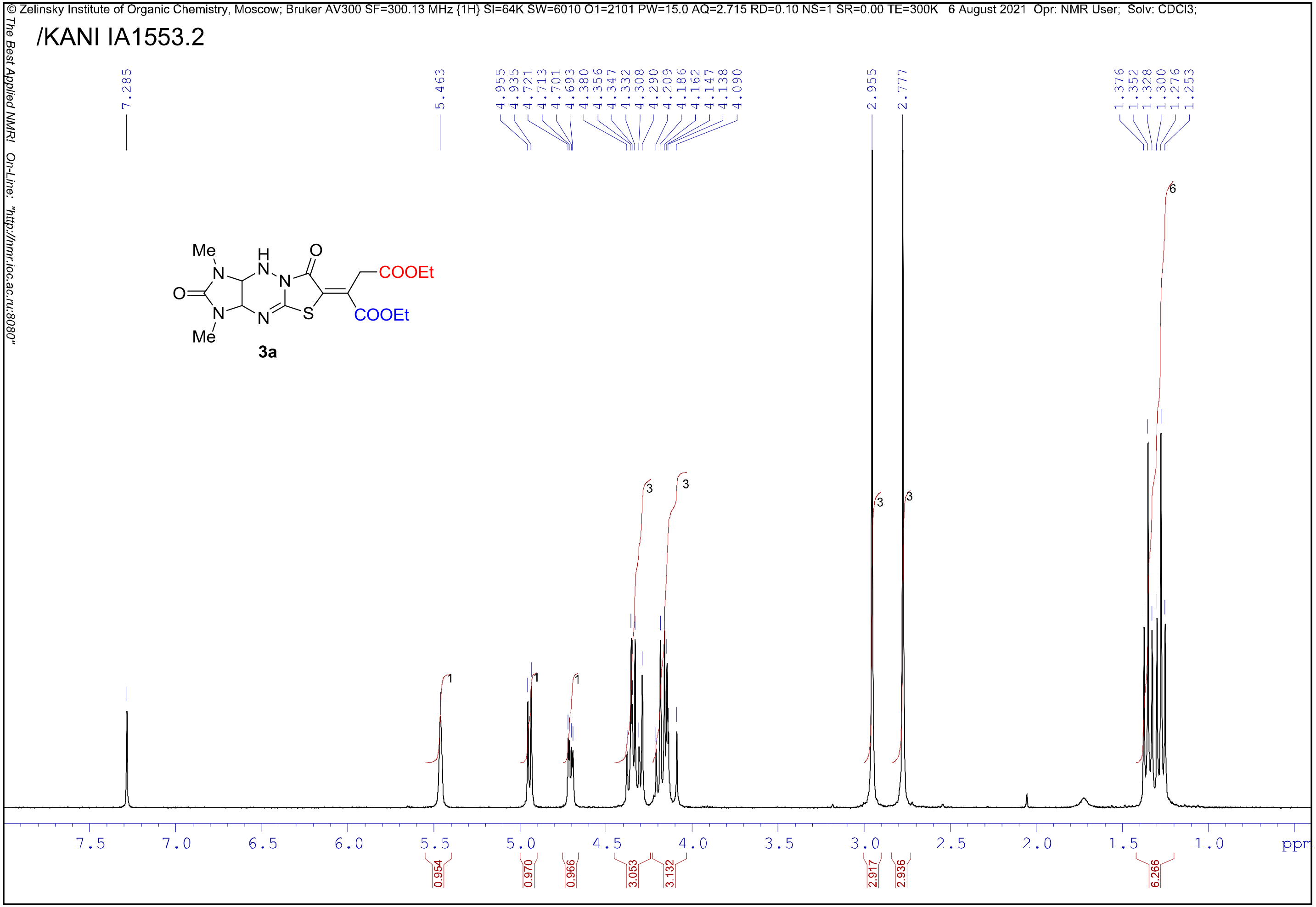
Procedure for the synthesis of compound 13

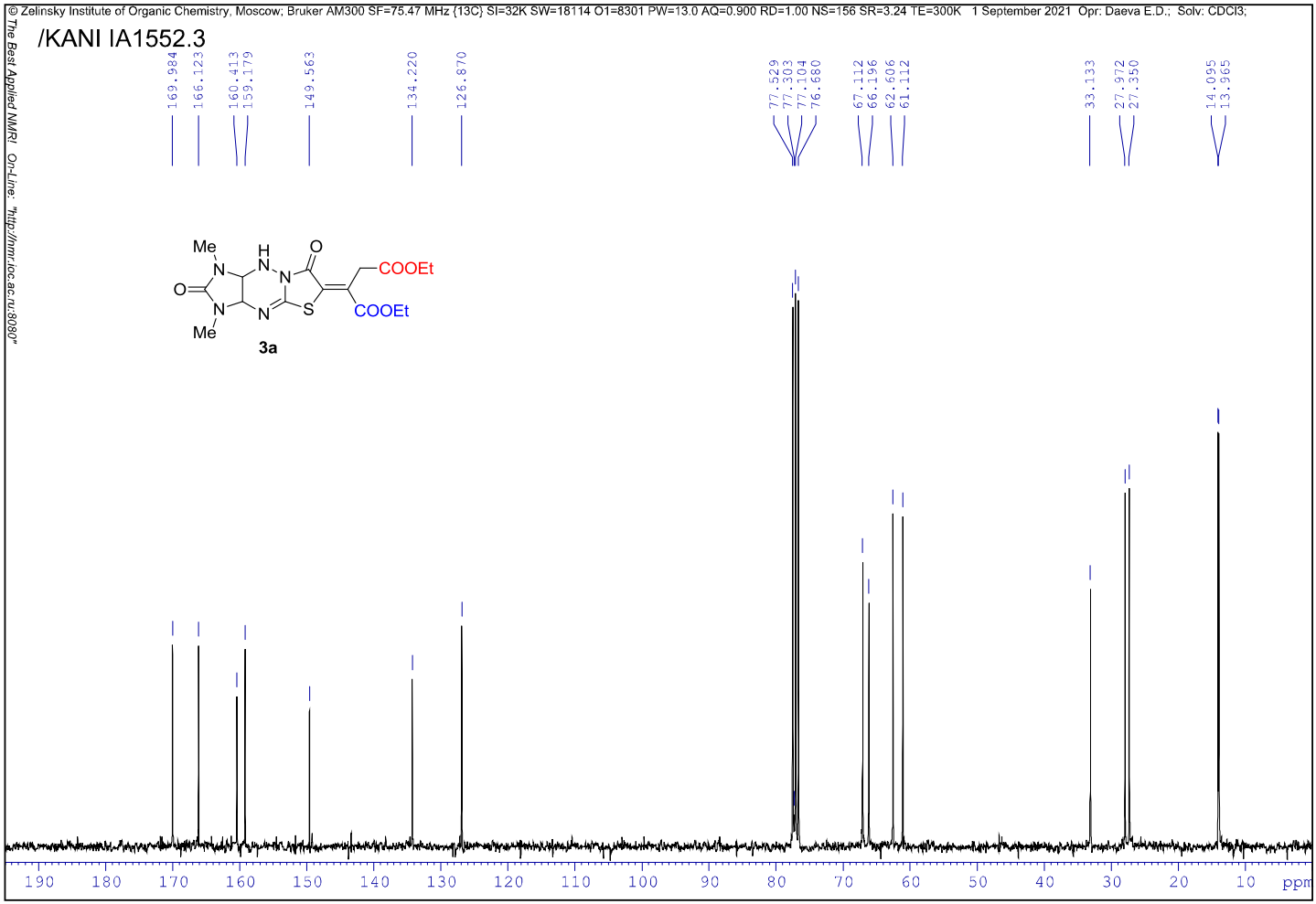
To the solution of diethyl 2-((4-chlorophenyl)thio)ethylene-1,2-dicarboxylate (31.5 mg, 0.1 mmol, 9:1 *Z*/*E* ratio) in dichloromethane (1 ml) pyridinium salt (37 mg, 0.15 mmol, 1.5 equiv.) and triethylamine (21 μl, 15 mg, 1.5 equiv.) were added, and the resulting mixture was stirred at r.t. for 1 hour. Then it was evaporated after addition of silica gel and the crude product was purified by column chromatography (hexane/EtOAc, 14:1 to 4:1).

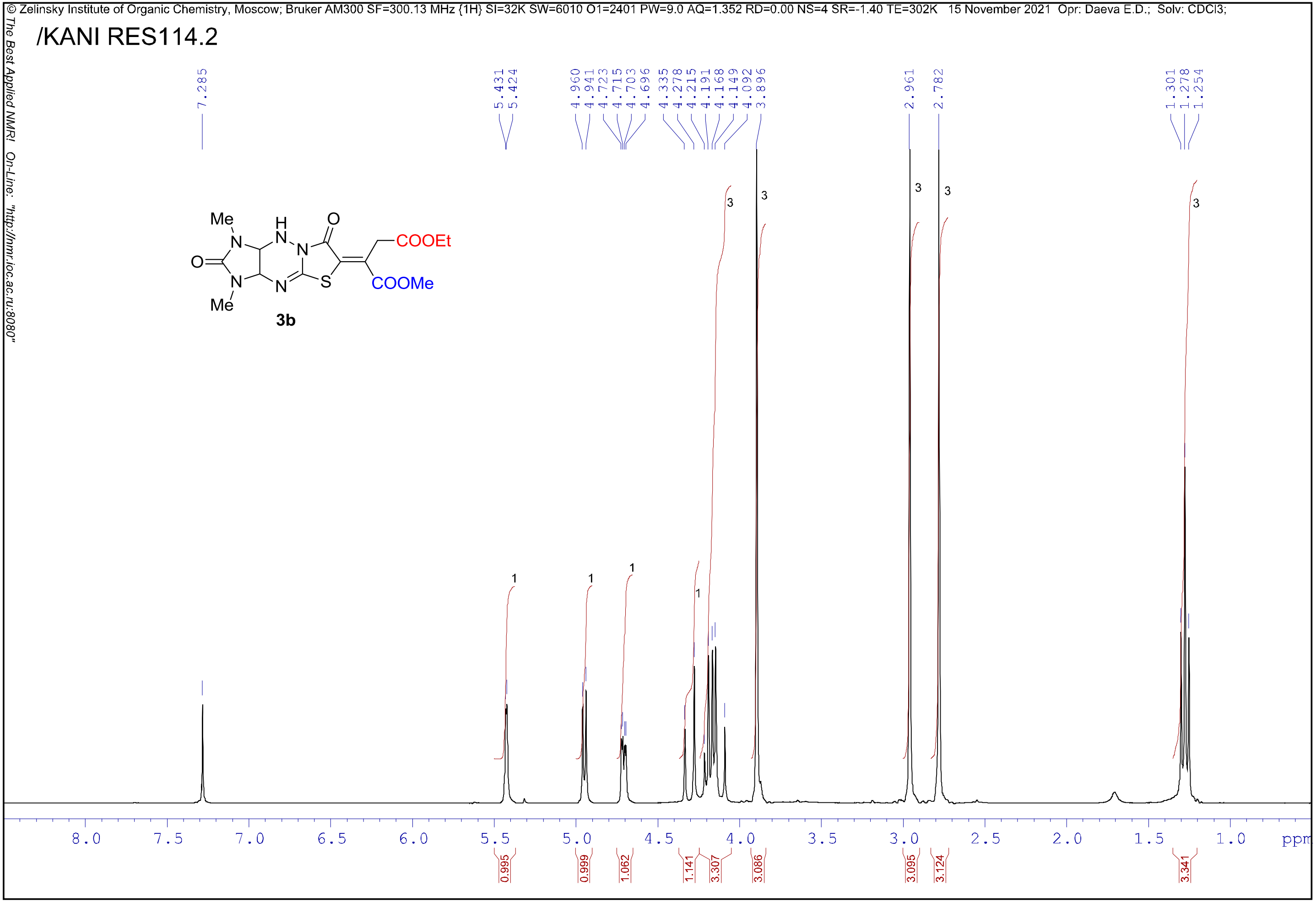
Triethyl 1-((4-chlorophenyl)thio)prop-1-ene-1,2,3-tricarboxylate (13). It has been separated as a mixture of *Z*/*E* diastereoisomers in 9:1 ratio according to ^{1}H NMR spectrum. Yield 30.5 mg (76%); yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.99-1.05 (m, 6H, CH_3 of *E*- and *Z*-isomers), 1.25-1.36 (m, 12H, $2CH_3$ of *E*- and *Z*-isomers), 3.40 (s, 2H, CH_2 of *E*-isomer), 3.67 (s, 2H, CH_2 of *Z*-isomer), 3.75 (q, *J* = 7.2 Hz, 2H, OCH_2 of *E*-isomer), 3.87 (q, *J* = 7.2 Hz, 2H, OCH_2 of *Z*-isomer), 4.16 (q, *J* = 7.1 Hz, 2H, OCH_2 of *E*-isomer), 4.21 (q, *J* = 7.1 Hz, 4H, OCH_2 of *Z*-isomer), 4.31 (q, *J* = 7.1 Hz, 2H, OCH_2 of *E*-isomer), 7.31-7.35 (m, 4H, Ar-2,6 of *E*- and *Z*-isomers), 7.45-7.50 (m, 4H, Ar-3,5 of *E*- and *Z*-isomers). ^{13}C NMR (100 MHz, $CDCl_3$): δ 13.64, 13.94, 14.11, 14.17 ($3CH_3$), 35.69, 36.65 ($3-CH_2$), 61.00, 61.21, 61.48, 61.61, 61.71, 62.00 ($3OCH_2$), 120.10, 123.86 (C-2), 127.09, 129.00, 129.22, 129.84, 136.08, 136.25, 136.36, 136.62 (Ar-1-6), 145.22, 147.05 (C-1), 163.70, 164.13, 164.21, 165.69, 169.24, 169.70 (3COOEt). HRMS (EI^+): *m/z* [M^+] calcd for $C_{18}H_{21}ClO_6S$: 400.0742; found: 400.0748.

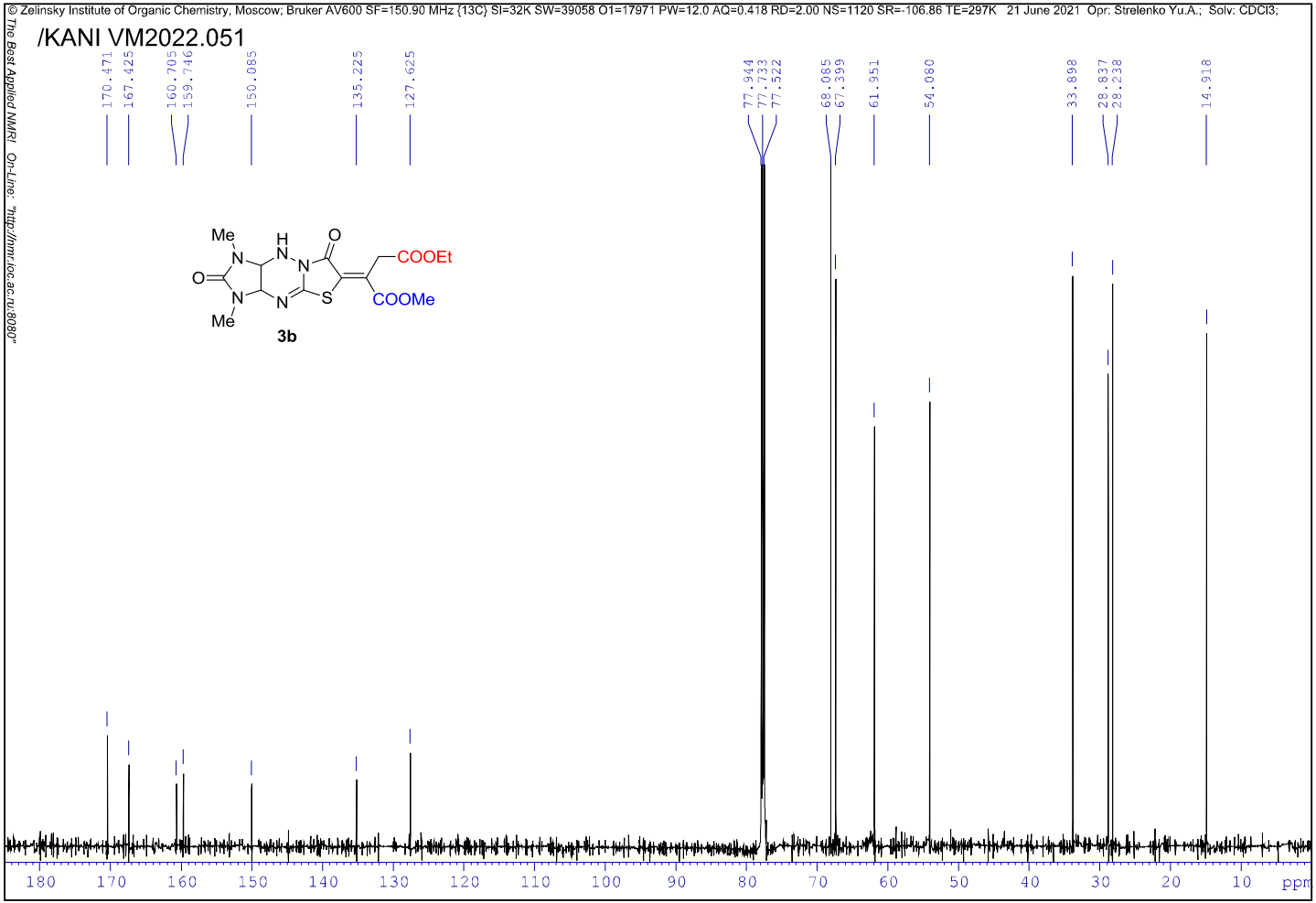
References

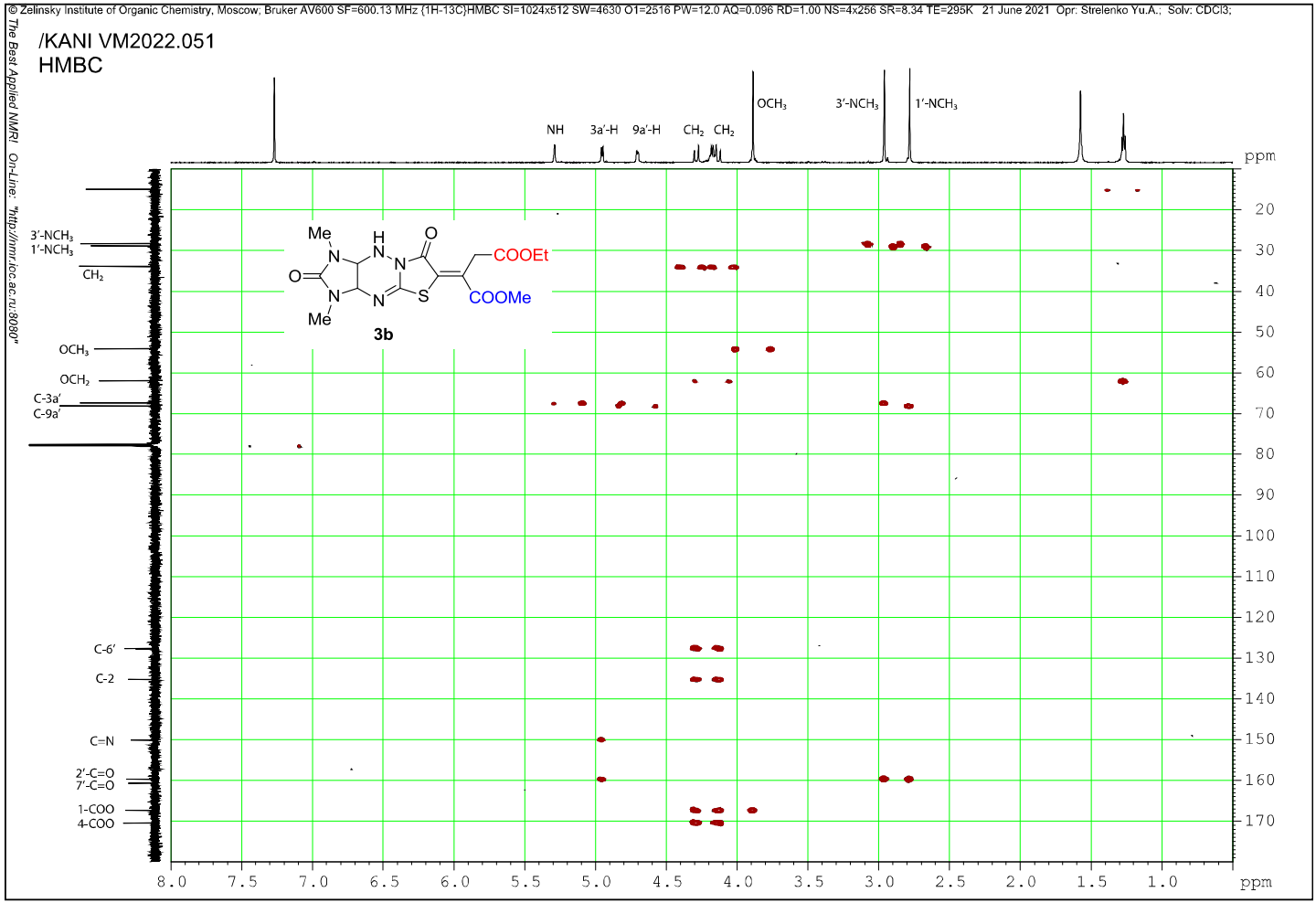
1. A. N. Izmest'ev, D. B. Vinogradov, N. G. Kolotyrkina, A. N. Kravchenko, G. A. Gazieva, Synthesis of functionalized imidazo[4,5-*e*]thiazolo[3,2-*b*]triazines by condensation of imidazo[4,5-*e*]triazinethiones with DMAD or DEAD and rearrangement to imidazo[4,5-*e*]thiazolo[2,3-*c*]triazines, *Beilstein J. Org. Chem*., 2021, **17**, 1141.
2. R. M. Acheson, J. D. Wallis, Addition reactions of heterocyclic compounds. Part 74. Products from dimethyl acetylenedicarboxylate with thiourea, thioamide, and guanidine derivatives, *J. Chem. Soc., Perkin Trans*. 1, 1981, 415.
3. S. A. Hashemi, M. R. Mohammadizadeh, Triethylamine-Promoted Stereoselective Synthesis of Dialkyl 2-Thiofumarates by the Reaction of Thiols and Dialkyl Acetylenedicarboxylates, *ChemistrySelect*, 2021, **6**, 733.
4. CrysAlisPro. Version 1.171.41.106a. *Rigaku Oxford Diffraction*, 2021.
5. G. M. Sheldrick, SHELXT — Integrated space-group and crystal-structure determination, *Acta Crystallogr., Sect. A: Found. Adv.,* 2015, **A71(1)**, 3.
6. G. M. Sheldrick, Crystal structure refinement with SHELXL, *Acta Crystallogr., Sect. C: Struct. Chem.,* 2015, **C71(1)**, 3.
7. O. V. Dolomanov, L. J. Bourhis, R. J. Gildea, J. A. K. Howard, H. Puschmann, OLEX2: a complete structure solution, refinement and analysis program, *J. Appl. Cryst.*, 2009, **42(2)**, 229.
8. L. J. Farrugia, *J. Appl. Cryst.*, 2012, **45**, 849.
9. Bruker. APEX-III. *Bruker AXS Inc.*, Madison, Wisconsin, USA, 2019.
10. L. Krause, R. Herbst-Irmer, G. M. Sheldrick, D. Stalke, Comparison of silver and molybdenum microfocus X-ray sources for single-crystal structure determination, *J. Appl. Cryst.*, 2015, **48**, 3.
11. D. S. Allgäuer, H. Mayr, *One-pot* two-step synthesis of 1-(ethoxycarbonyl)indolizines via pyridinium ylides, *Eur. J. Org. Chem.*, 2013, **28**, 6379.
12. J. Brioche, C. Meyer, J. Cossy, Synthesis of 2-aminoindolizines by 1,3-dipolar cycloaddition of pyridinium ylides with electron-deficient ynamides, *Org. Lett.*, 2015, **17**, 2800.
13. G. P. Kondratenko, N. I. Geonya, L. A. Perel'man, L. M. Litvinenko, Antimicrobial activity of the pyridinium salts of someα-halo carbonyl compounds, *Pharm. Chem. J.*, 1976, **10**, 201.

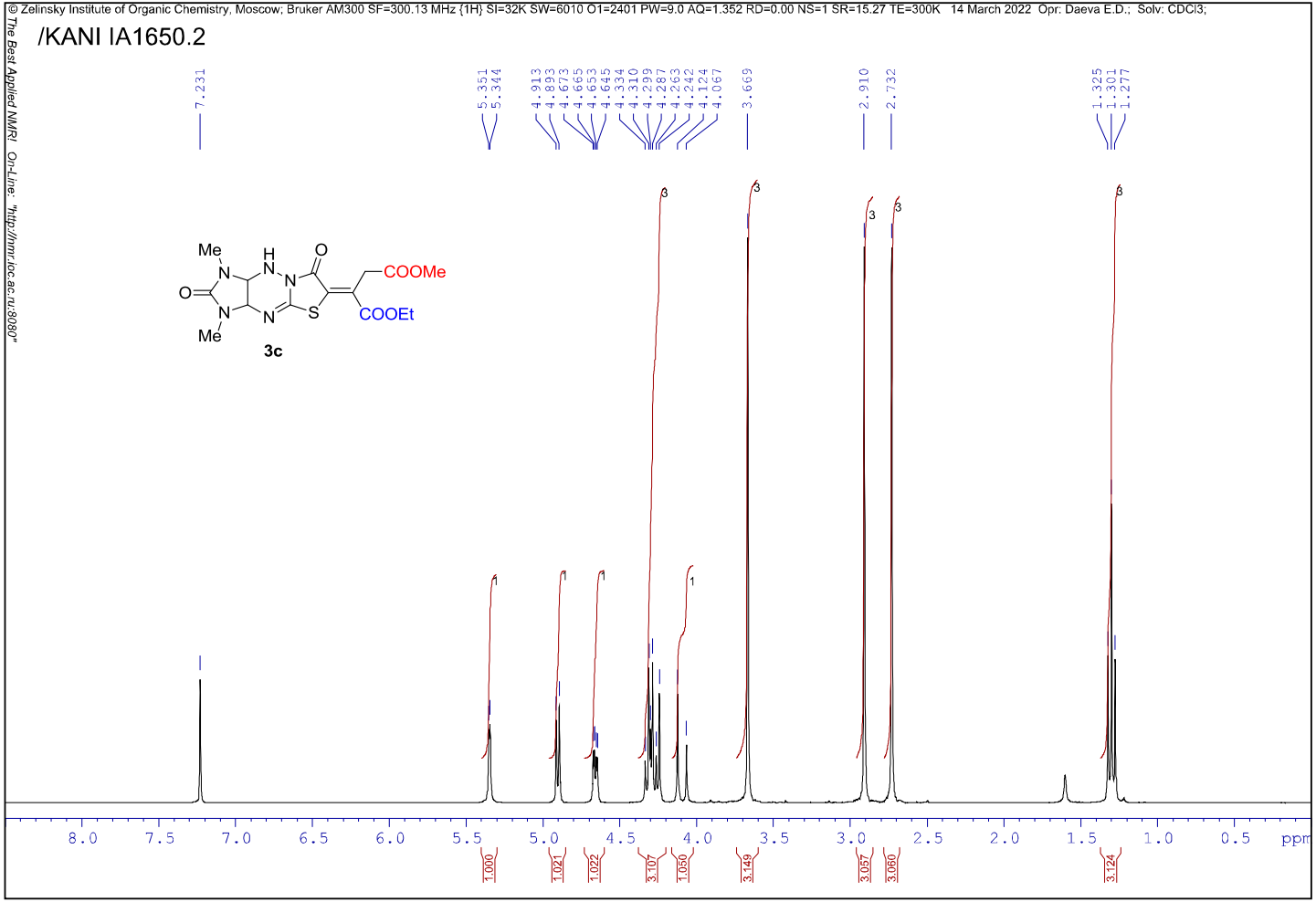

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AV300 SF=300.13 MHz {1H} SI=64K SW=6010 O1=2101 PW=15.0 AQ=2.715 RD=0.10 NS=1 SR=0.00 TE=300K 6 August 2021 Opr: NMR User; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1553.2
7.285
5.463
4.955 4.935 4.721 4.713 4.701 4.693 4.380 4.356 4.347 4.332 4.308 4.290 4.209 4.186 4.162 4.147 4.138 4.090
2.955
2.777
1.376 1.352 1.328 1.300 1.276 1.253
Me
H
O
N
COOEt
COOEt
S
Me
3a
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
0.954 0.970 0.966 3.053 3.132 2.917 2.936 6.266

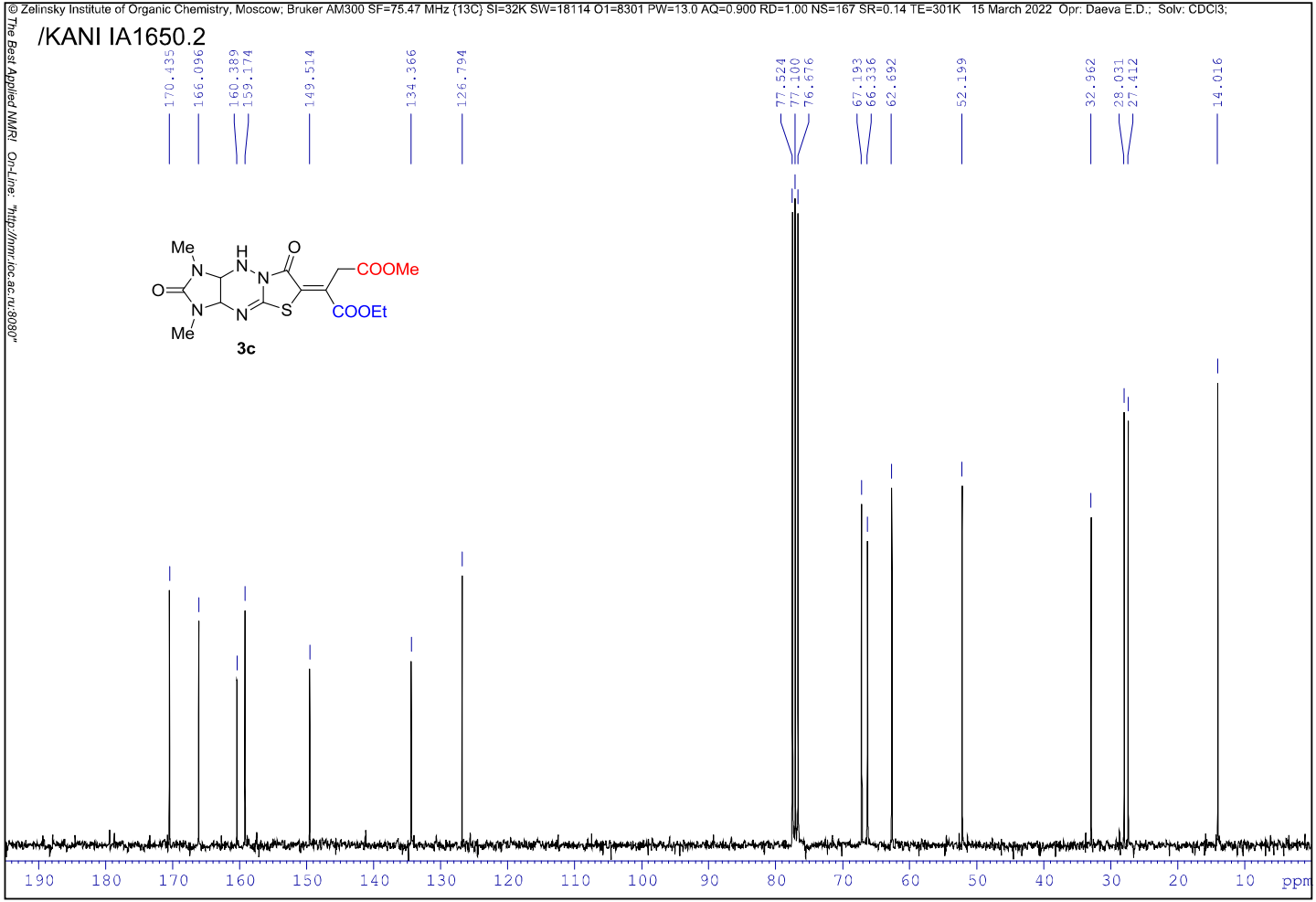

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=156 SR=3.24 TE=300K 1 September 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1552.3
169.984
166.123
160.413
159.179
149.563
134.220
126.870
77.529
77.303
77.104
76.680
67.112
66.196
62.606
61.112
33.133
27.972
27.350
14.095
13.965
Me
N
H
N
O
O
N
N
S
COOEt
COOEt
Me
3a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

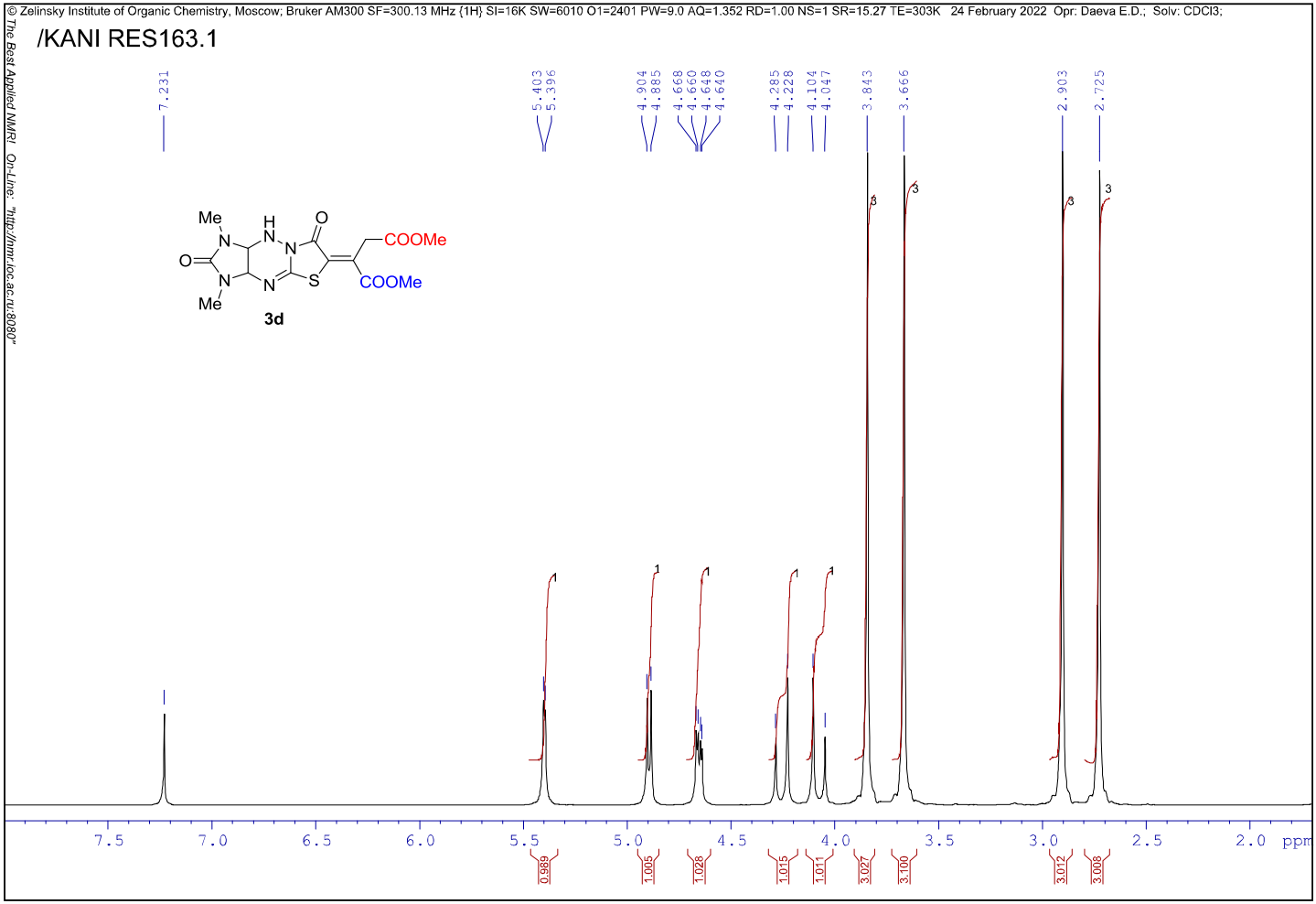

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=32K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=4 SR=-1.40 TE=302K 15 November 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI RES114.2
7.285
5.431
5.424
4.960
4.941
4.723
4.715
4.703
4.696
4.335
4.278
4.215
4.191
4.168
4.149
4.092
3.896
2.961
2.782
1.301
1.278
1.254
Me
N
O
N
Me
H
N
N
N
O
S
COOEt
COOMe
3b
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
0.995
0.999
1.062
1.141
3.307
3.086
3.095
3.124
3.341

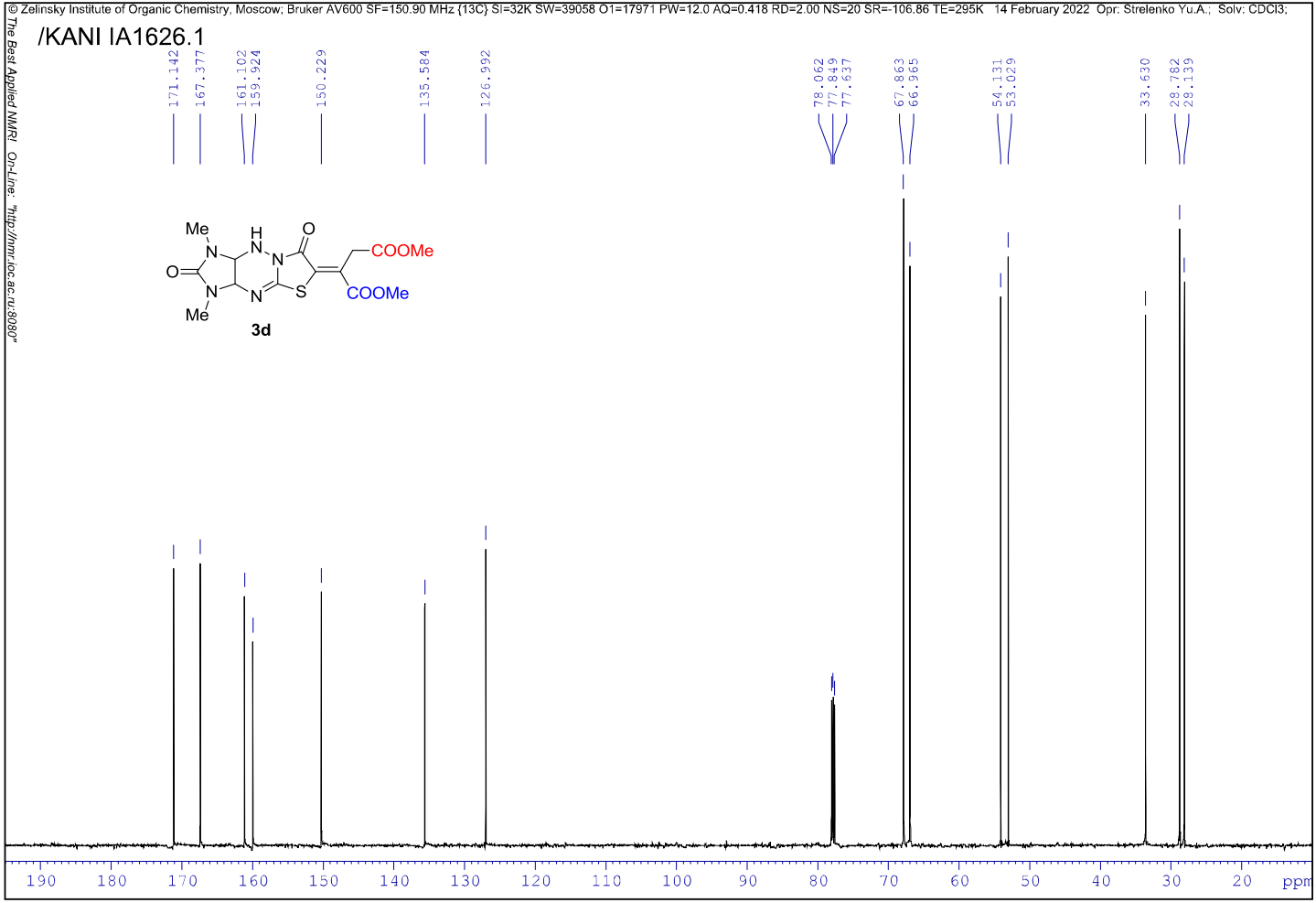

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AV600 SF=150.90 MHz {13C} SI=32K SW=39058 O1=17971 PW=12.0 AQ=0.418 RD=2.00 NS=1120 SR=-106.86 TE=297K 21 June 2021 Opr: Strelenko Yu.A.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI VM2022.051
170.471
167.425
160.705
159.746
150.085
135.225
127.625
77.944
77.733
77.522
69.085
67.399
61.951
54.080
33.898
28.837
28.238
14.918
Me
N
H
N
O
N
N
O
S
N
Me
COOEt
COOMe
3b
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AV600 SF=600.13 MHz {1H-13C}HMBC SI=1024x512 SW=4630 O1=2516 PW=12.0 AQ=0.096 RD=1.00 NS=4x256 SR=8.34 TE=295K 21 June 2021 Opr: Strelenko Yu.A.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI VM2022.051
HMBC
NH
3a'-H
9a'-H
CH2
CH2
OCH3
3'-NCH3
1'-NCH3
ppm
3'-NCH3
1'-NCH3
CH2
OCH3
OCH2
C-3a'
C-9a'
C-6'
C-2
C=N
2'-C=O
7'-C=O
1-COO
4-COO
Me
N
H
N
O
O
N
N
S
Me
COOEt
COOMe
3b
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

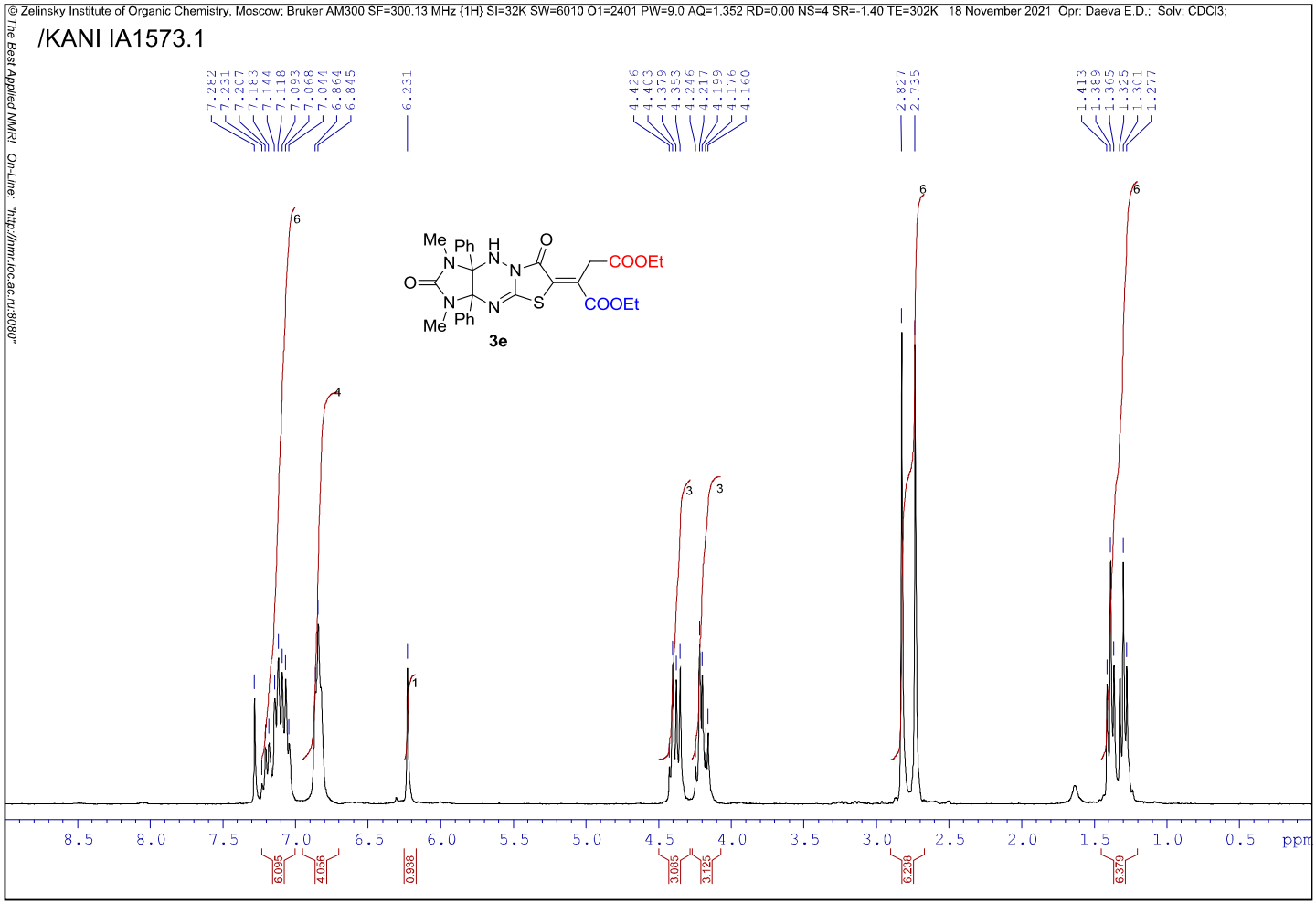

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=32K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=1 SR=15.27 TE=300K 14 March 2022 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1650.2
7.231
5.351
5.344
4.913
4.893
4.673
4.665
4.653
4.645
4.334
4.310
4.299
4.287
4.263
4.242
4.124
4.067
3.669
2.910
2.732
1.325
1.301
1.277
Me
N
H
N
O
O
N
N
S
COOMe
COOEt
Me
3c
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
1.000
1.021
1.022
3.107
1.050
3.149
3.057
3.060
3.124

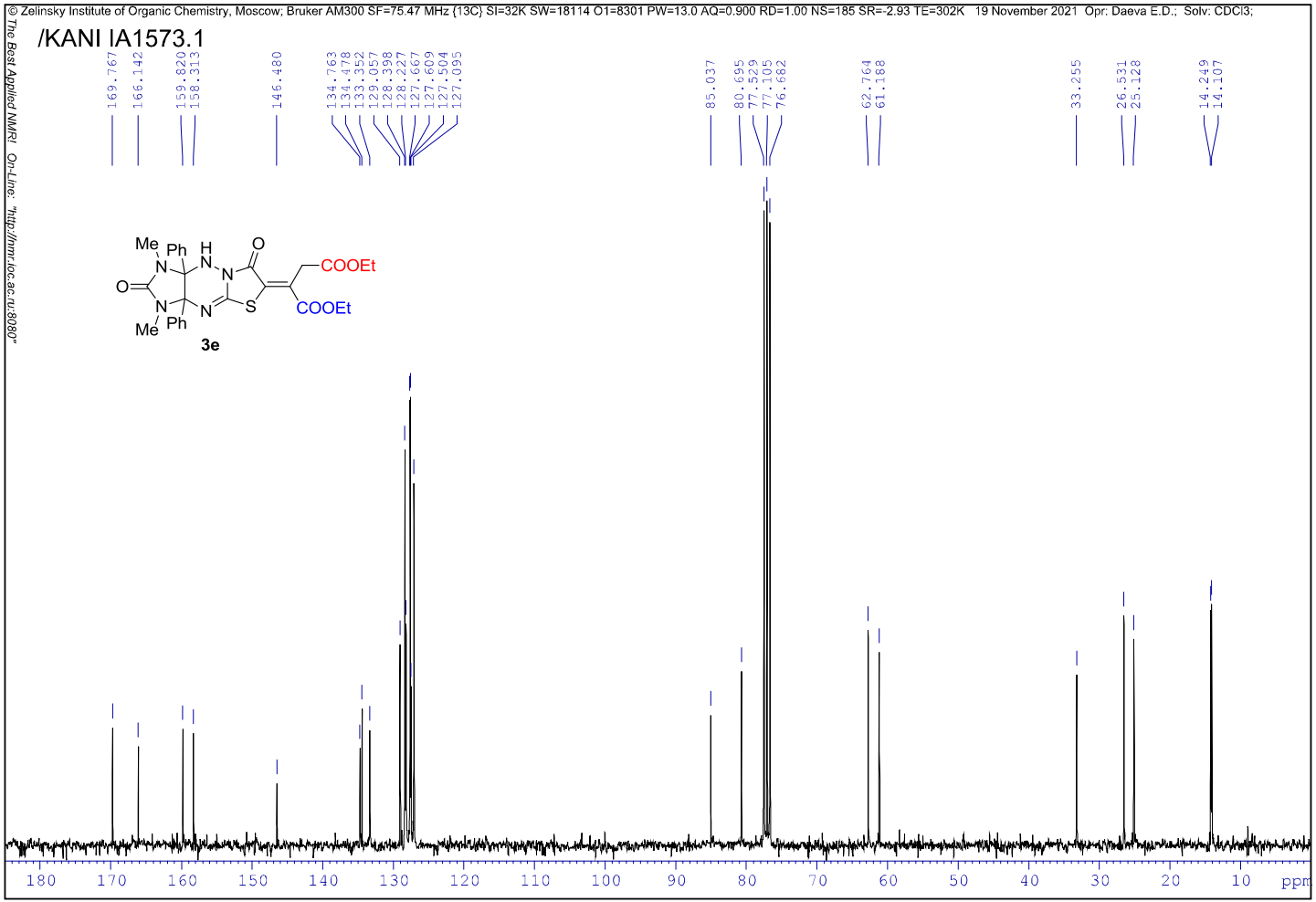

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=167 SR=0.14 TE=301K 15 March 2022 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1650.2
170.435
166.096
160.389
159.174
149.514
134.366
126.794
77.524
77.100
76.676
67.193
66.336
62.692
52.199
32.962
28.031
27.412
14.016
Me
N
H
N
O
O
N
N
S
N
Me
COOMe
COOEt
3c
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

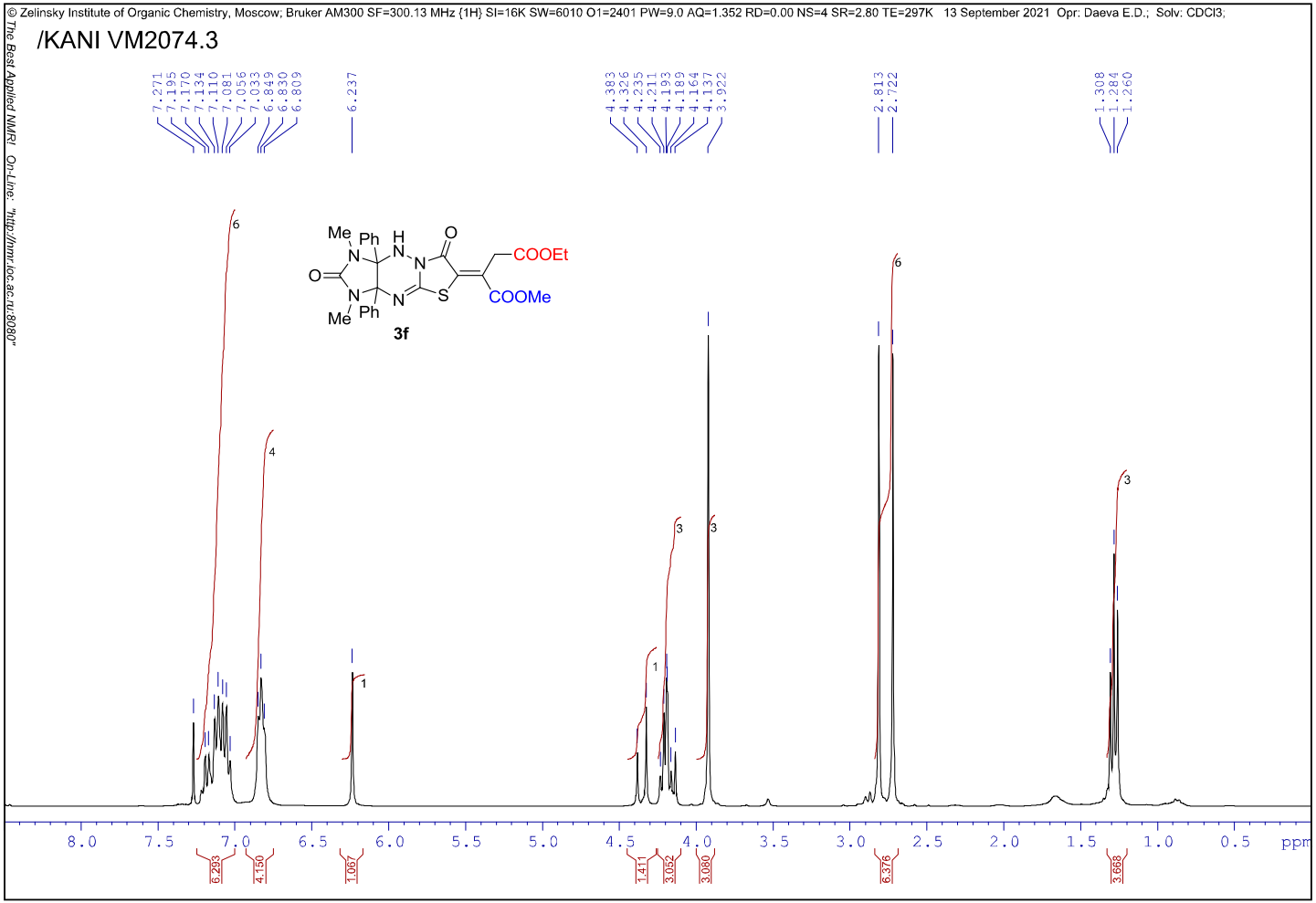

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=1.00 NS=1 SR=15.27 TE=303K 24 February 2022 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI RES163.1
7.231
5.403
5.396
4.904
4.885
4.668
4.660
4.648
4.640
4.285
4.228
4.104
4.047
3.843
3.666
2.903
2.725
Me
N
H
N
N
O
O
N
N
S
COOMe
COOMe
Me
3d
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
0.989
1.005
1.028
1.015
1.011
3.027
3.100
3.012
3.008

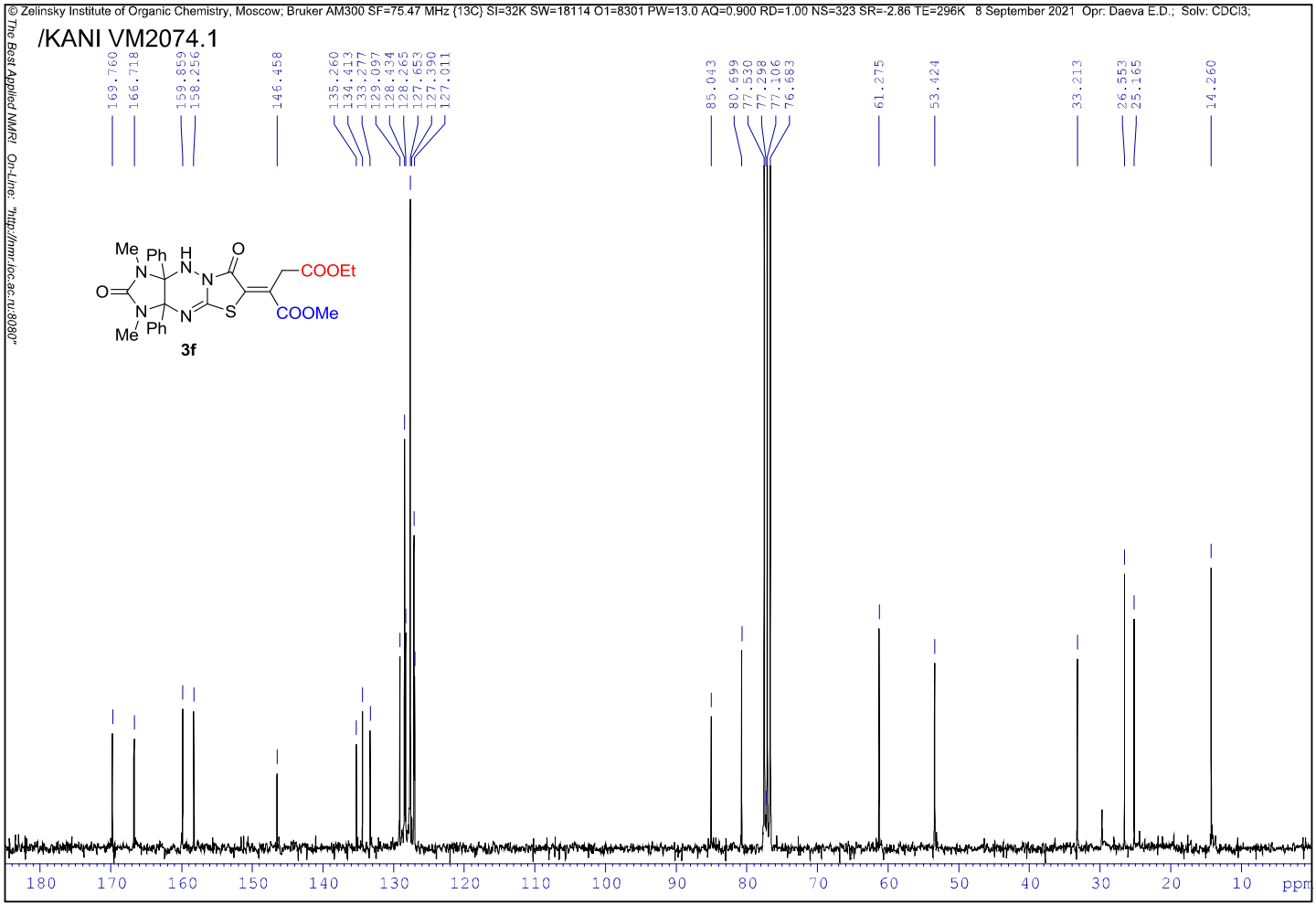

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AV600 SF=150.90 MHz {13C} SI=32K SW=39058 O1=17971 PW=12.0 AQ=0.418 RD=2.00 NS=20 SR=-106.86 TE=295K 14 February 2022 Opr: Strelenko Yu.A.; Solv: CDCl3;
/KANI IA1626.1
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
171.142
167.377
161.102
159.924
150.229
135.584
126.992
78.062
77.849
77.637
67.863
66.965
54.131
53.029
33.630
28.782
28.139
Me
N
H
N
O
O
N
N
COOMe
N
S
COOMe
Me
3d
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

/KANI IA1573.1

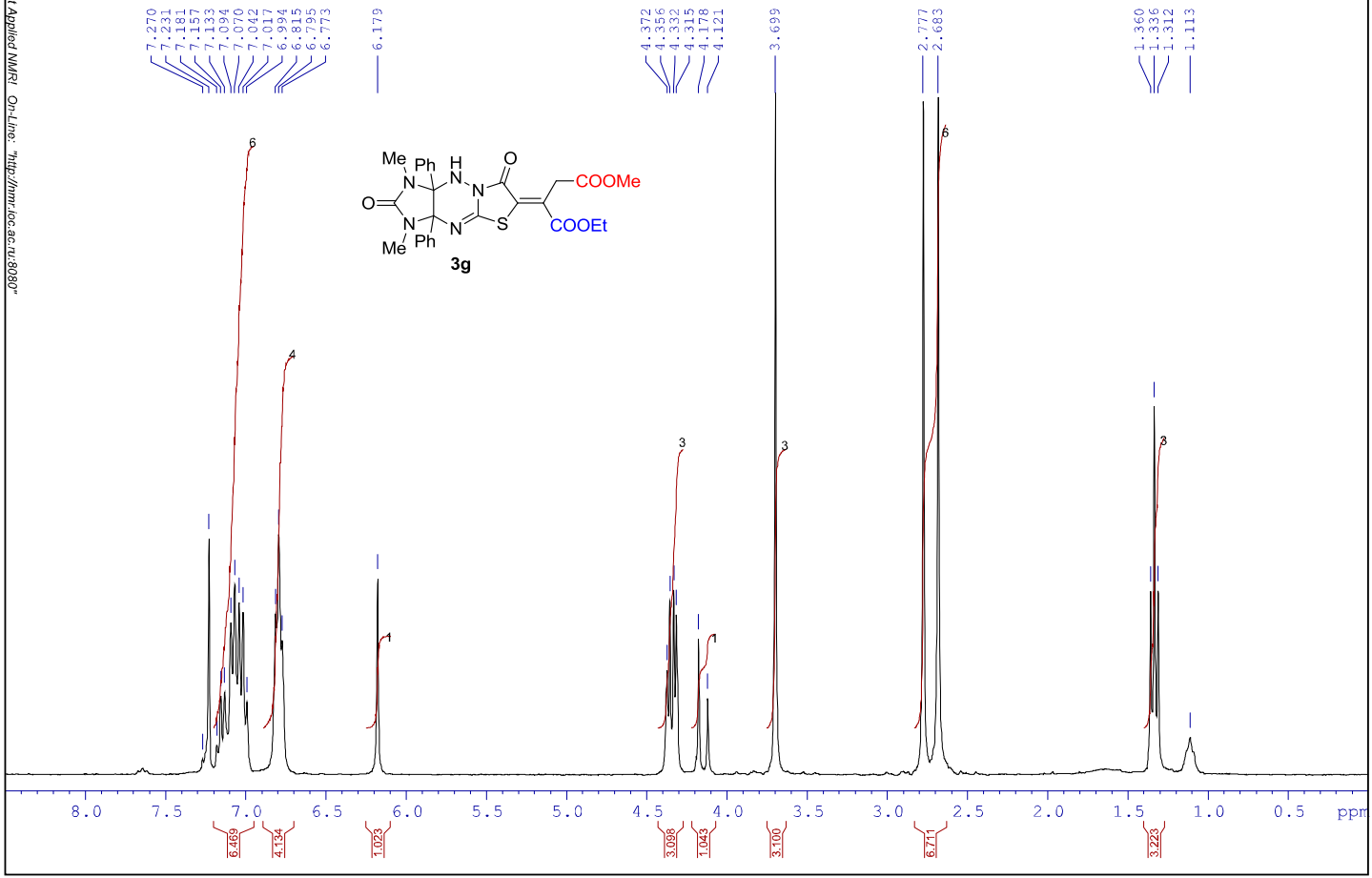


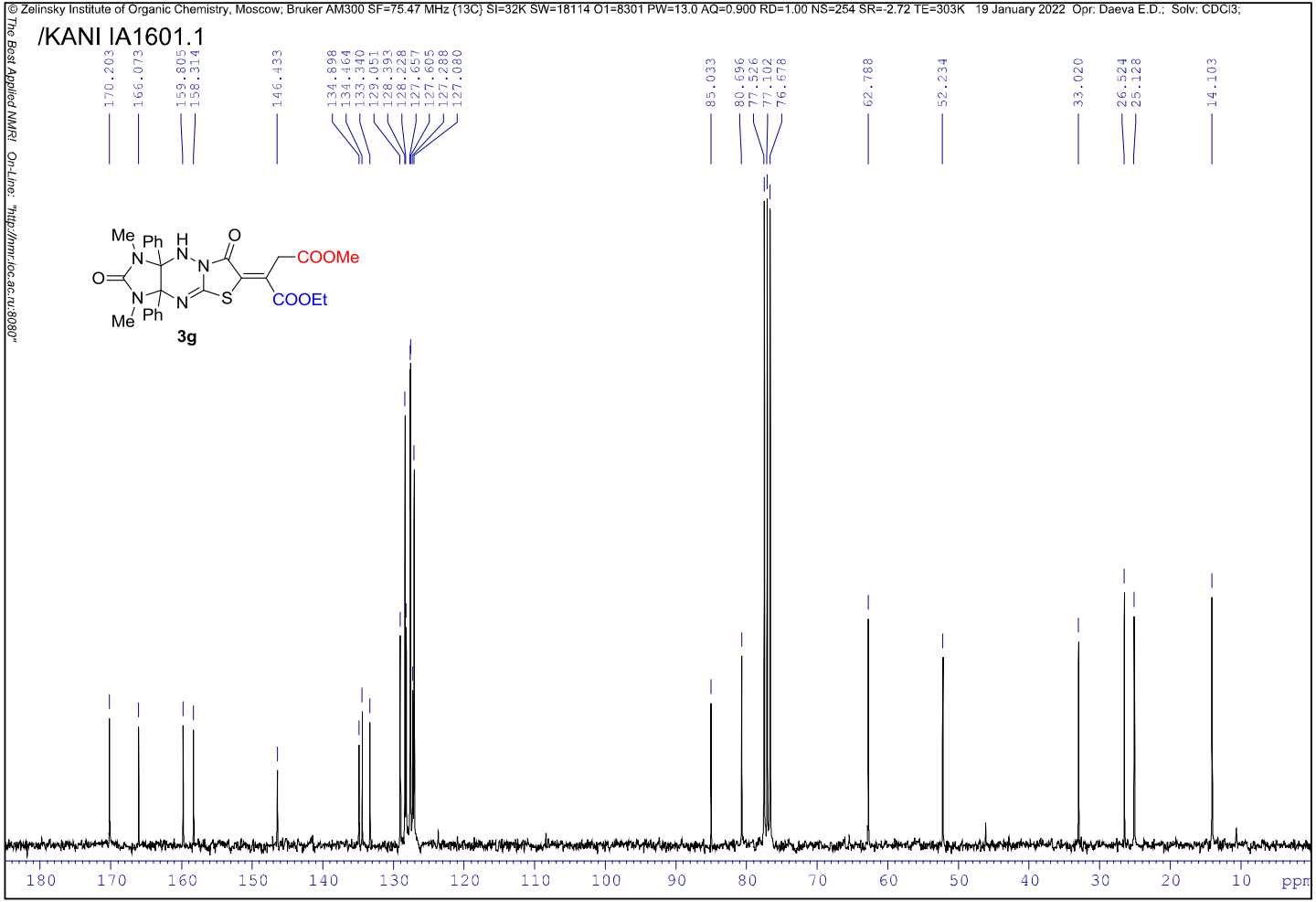

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=185 SR=-2.93 TE=302K 19 November 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1573.1
169.767
166.142
159.820
158.313
146.480
134.763
134.478
133.352
129.057
128.398
128.227
127.667
127.609
127.504
127.095
85.037
80.695
77.529
77.105
76.682
62.764
61.188
33.255
26.531
25.128
14.249
14.107
Me
Ph
H
N
O
COOEt
COOEt
S
3e
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

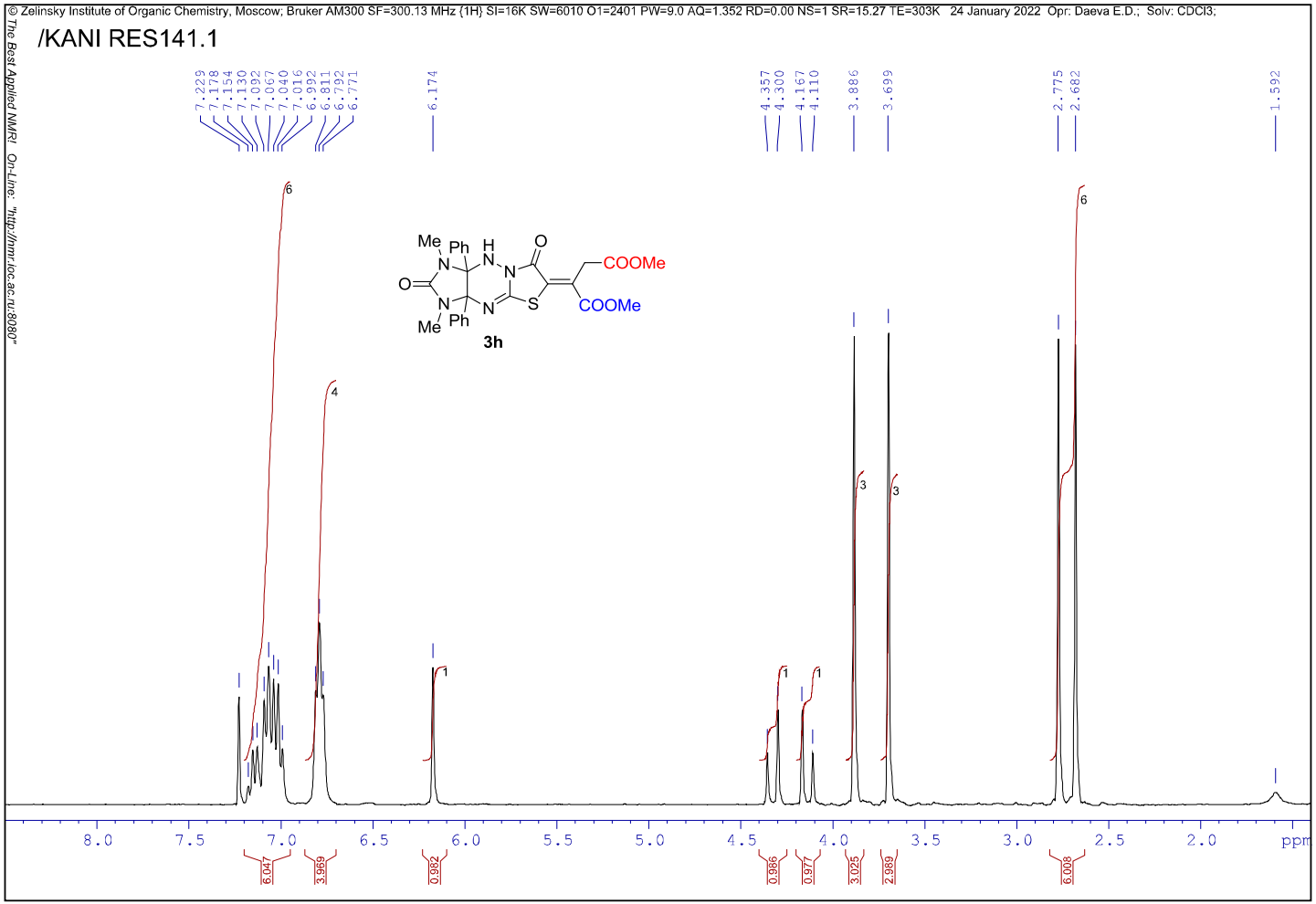

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=4 SR=2.80 TE=297K 13 September 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI VM2074.3
7.271
7.195
7.170
7.134
7.110
7.081
7.056
7.033
6.849
6.830
6.809
6.237
4.383
4.326
4.235
4.211
4.193
4.189
4.164
4.137
3.922
2.813
2.722
1.308
1.284
1.260
Me
Ph
N
H
N
O
O
N
N
S
COOEt
COOMe
Me
Ph
3f
6
4
1
3
3
1
6
3
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
6.293
4.150
1.067
1.411
3.052
3.080
6.376
3.668

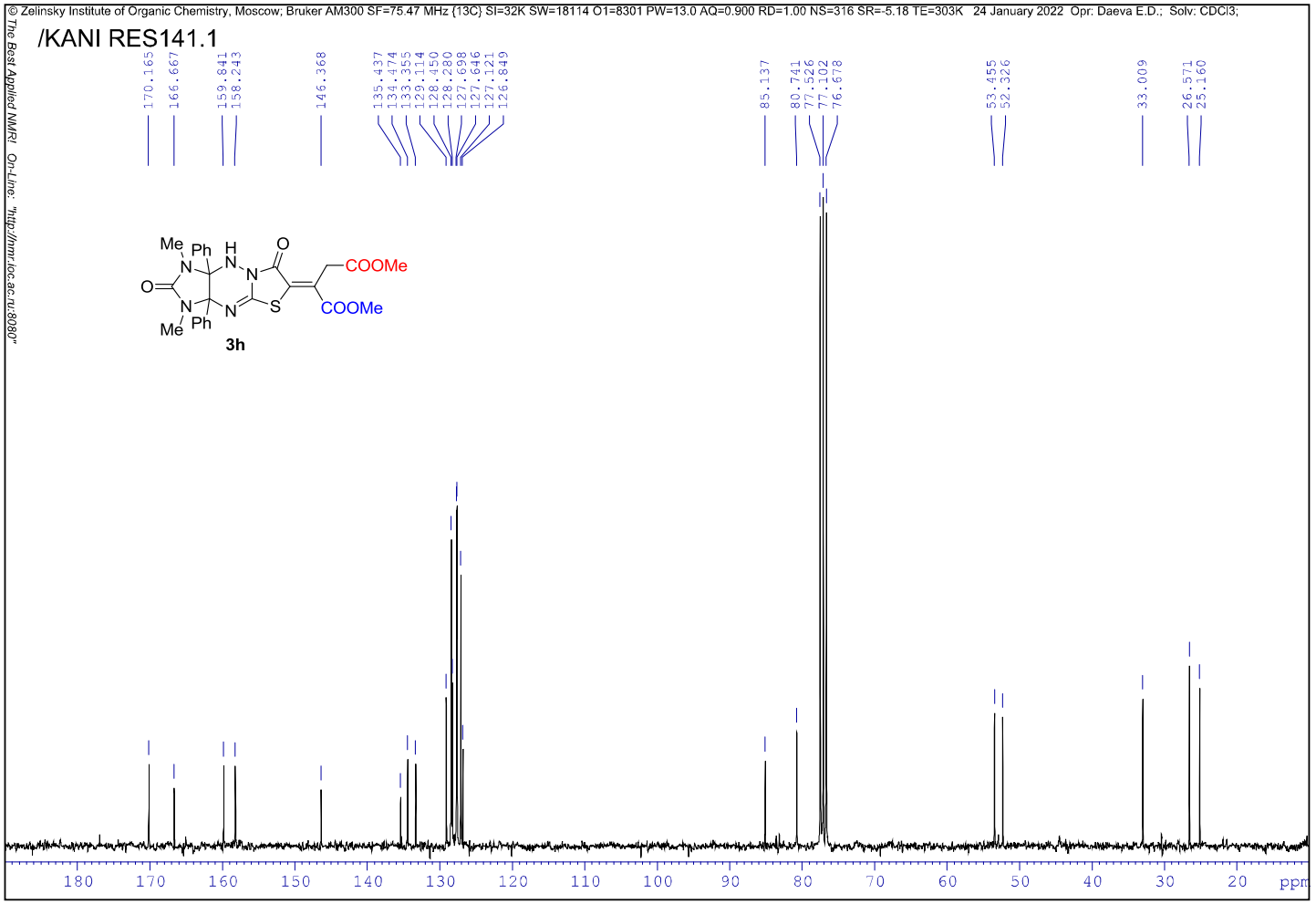

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=323 SR=-2.86 TE=296K 8 September 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI VM2074.1
169.760
166.718
159.859
158.256
146.458
135.260
134.413
133.277
129.097
128.434
128.265
127.653
127.390
127.011
85.043
80.699
77.530
77.298
77.106
76.683
61.275
53.424
33.213
26.553
25.165
14.260
Me
Ph
H
N
N
O
O
N
N
S
COOEt
COOMe
Me
Ph
3f
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

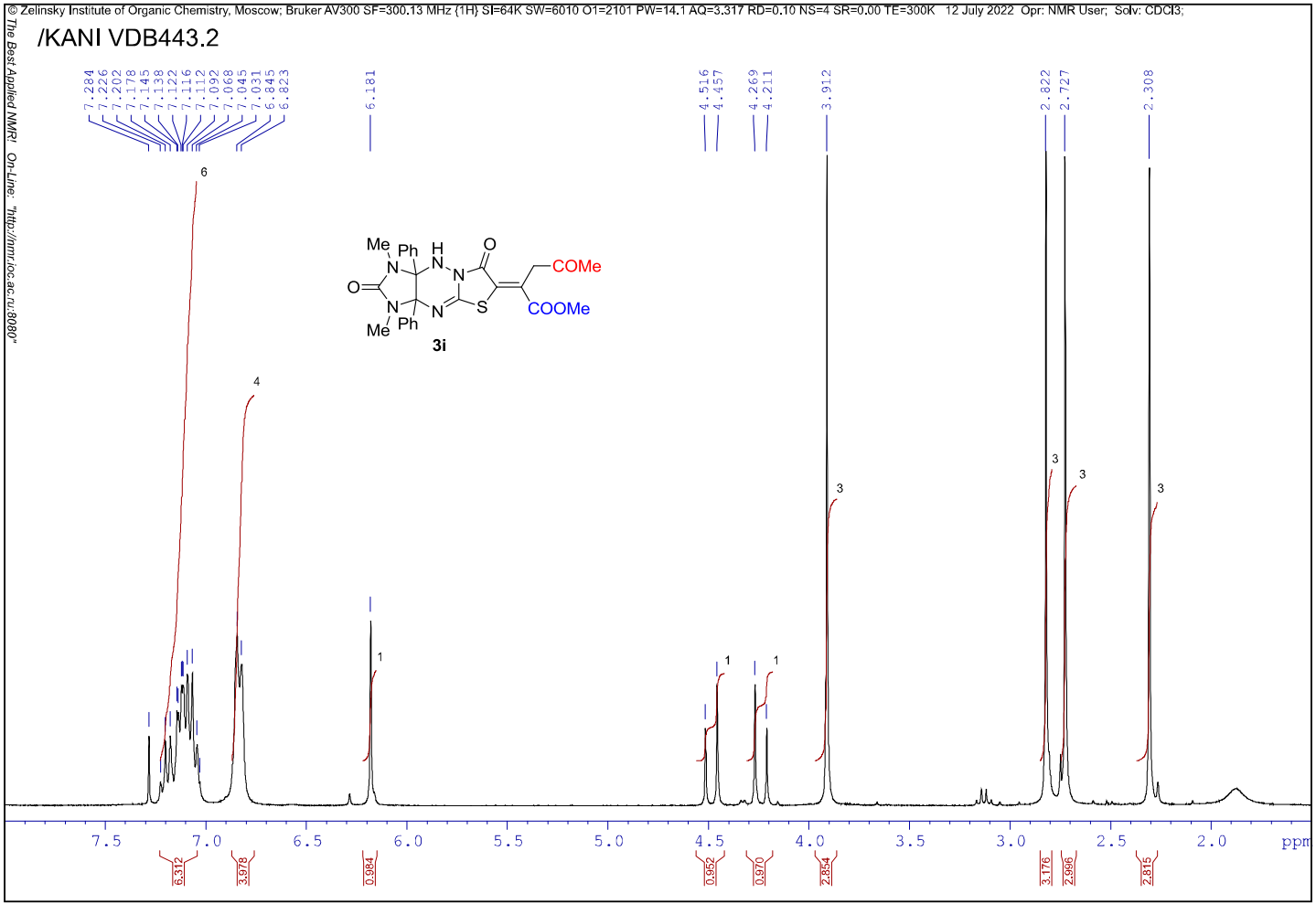
/KANI IA1601.1

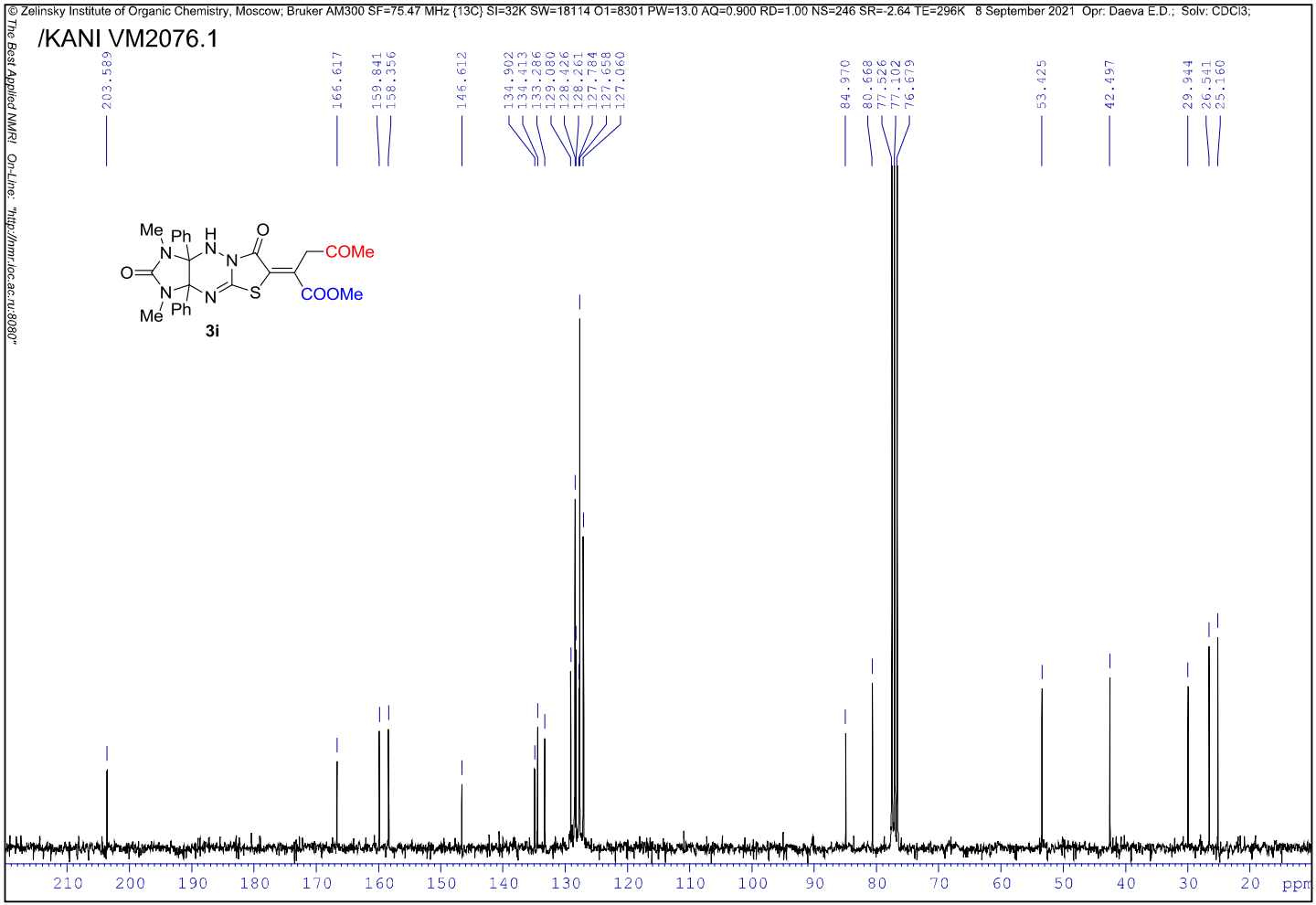



© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=254 SR=-2.72 TE=303K 19 January 2022 Opr: Daeva E.D.; Solv: CDCl3;
/KANI IA1601.1
The Best Applied NMR! On-Line. "http://nmr.ioc.ac.ru:8080"
170.203
166.073
159.805
158.314
146.433
134.898
134.464
133.340
129.051
128.393
128.228
127.657
127.605
127.288
127.080
85.033
80.696
77.526
77.102
76.678
62.788
52.234
33.020
26.524
25.128
14.103
Me
Ph
H
N
O
COOMe
COOEt
S
3g
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

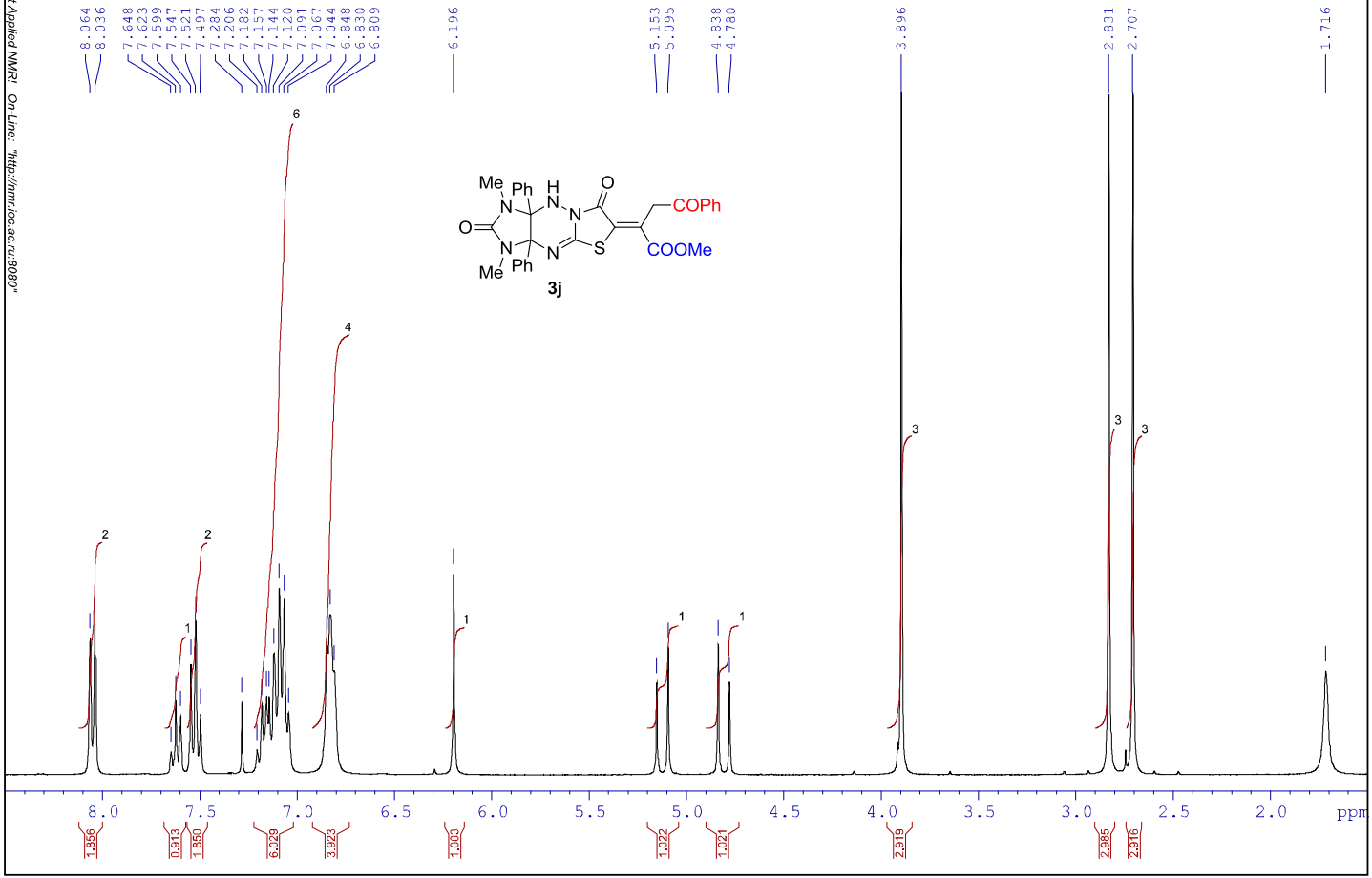

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=1 SR=15.27 TE=303K 24 January 2022 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI RES141.1
7.229
7.178
7.154
7.130
7.092
7.067
7.040
7.016
6.992
6.811
6.792
6.771
6.174
4.357
4.300
4.167
4.110
3.886
3.699
2.775
2.682
1.592
Me
Ph
H
N
O
COOMe
COOMe
S
3h
6
4
1
3
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
6.047
3.969
0.982
0.966
0.977
3.025
2.989
6.008

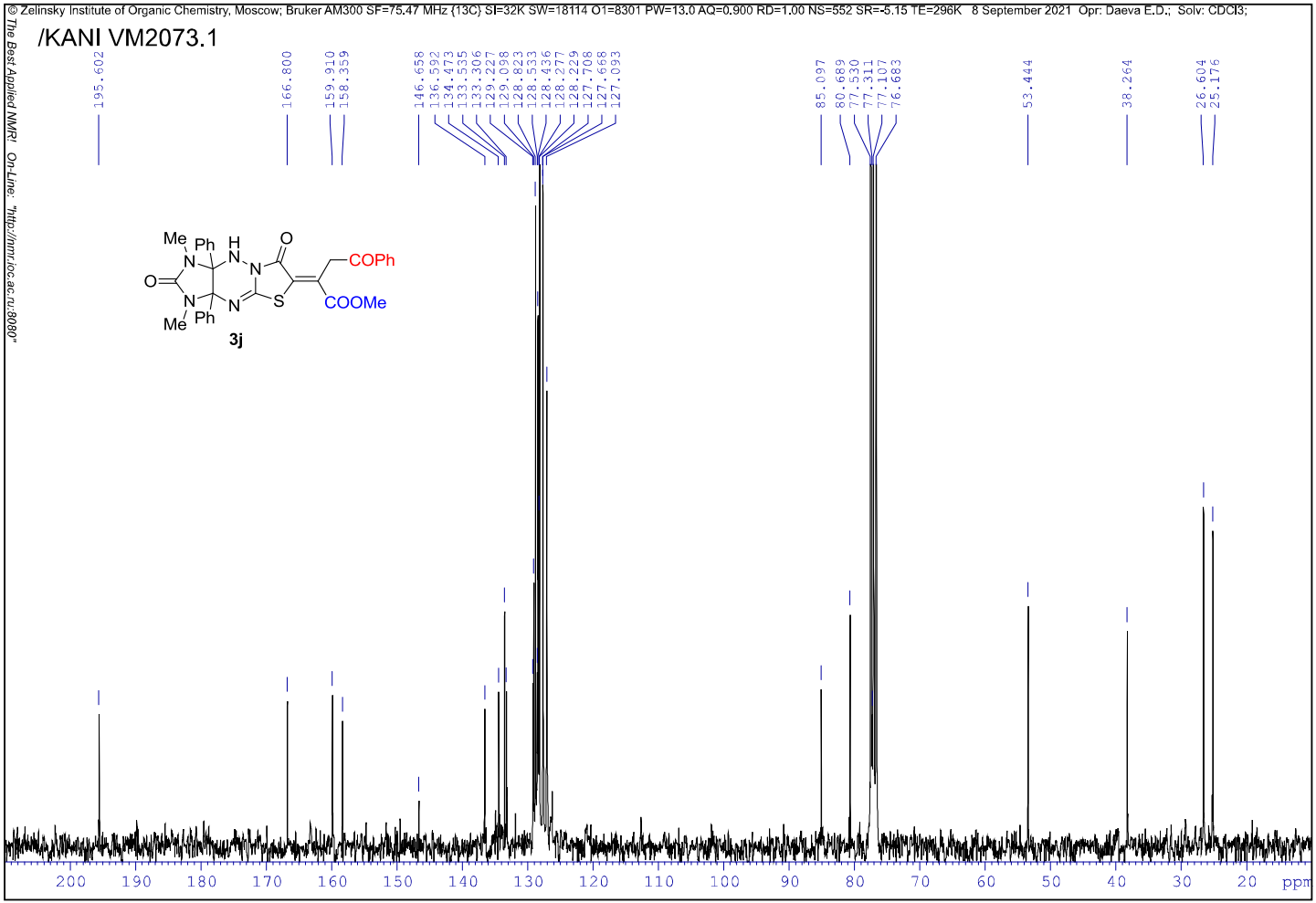

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=316 SR=-5.18 TE=303K 24 January 2022 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI RES141.1
170.165
166.667
159.841
158.243
146.368
135.437
134.474
133.355
129.114
128.450
128.280
127.698
127.646
127.121
126.849
85.137
80.741
77.326
77.102
76.678
53.455
52.326
33.009
26.571
25.160
Me
Ph
H
N
N
N
O
O
N
N
S
Me
Ph
COOMe
COOMe
3h
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

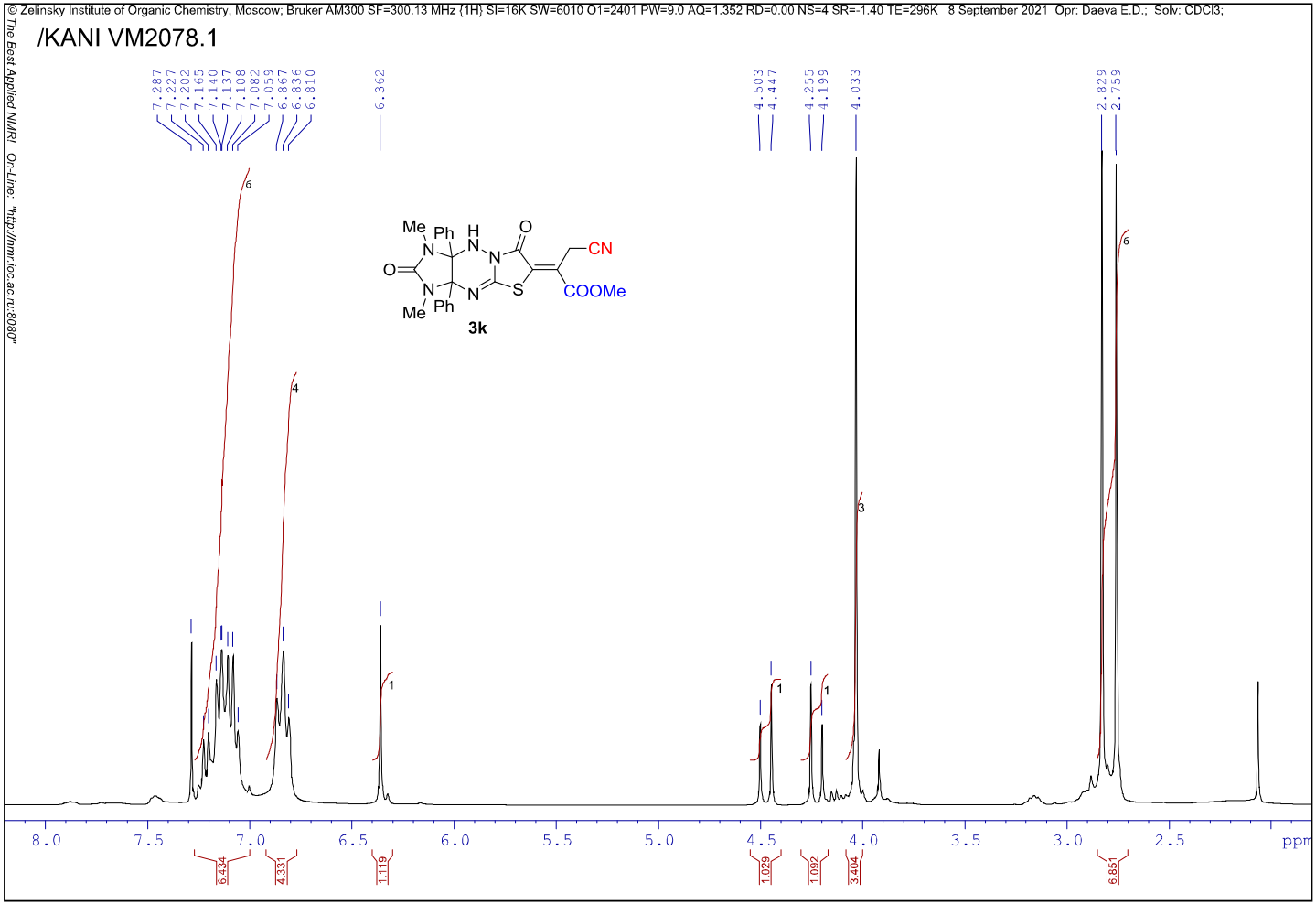

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AV300 SF=300.13 MHz {1H} SI=64K SW=6010 O1=2101 PW=14.1 AQ=3.317 RD=0.10 NS=4 SR=0.00 TE=300K 12 July 2022 Opr: NMR User; Solv: CDCl3;
The Best Applied NMR!. On-Line. "http://nmr.ioc.ac.ru:8080"
/KANI VDB443.2
7.284
7.226
7.202
7.178
7.145
7.138
7.122
7.116
7.112
7.092
7.068
7.045
7.031
6.845
6.823
6.181
4.516
4.457
4.269
4.211
3.912
2.822
2.727
2.308
Me
Ph
H
N
O
COMe
COOMe
S
3i
6
4
3
1
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
6.312
3.978
0.984
0.952
0.970
2.854
3.176
2.996
2.815

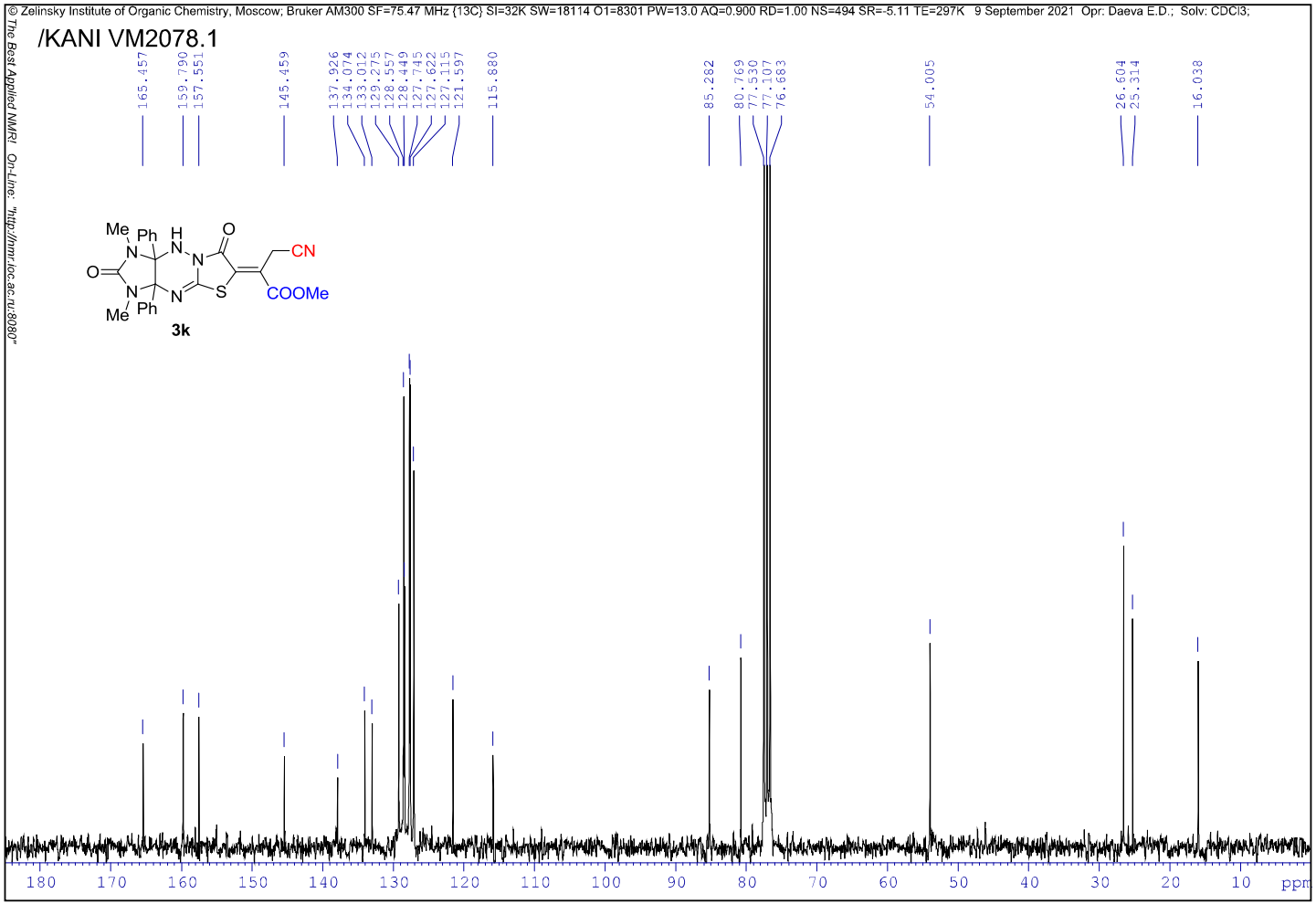

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=246 SR=-2.64 TE=296K 8 September 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI VM2076.1
203.589
166.617
159.841
158.356
146.612
134.902
134.413
133.286
129.080
128.426
128.261
127.784
127.658
127.060
84.970
80.668
77.526
77.102
76.679
53.425
42.497
29.944
26.541
25.160
Me
Ph
H
N
O
COMe
COOMe
S
3i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

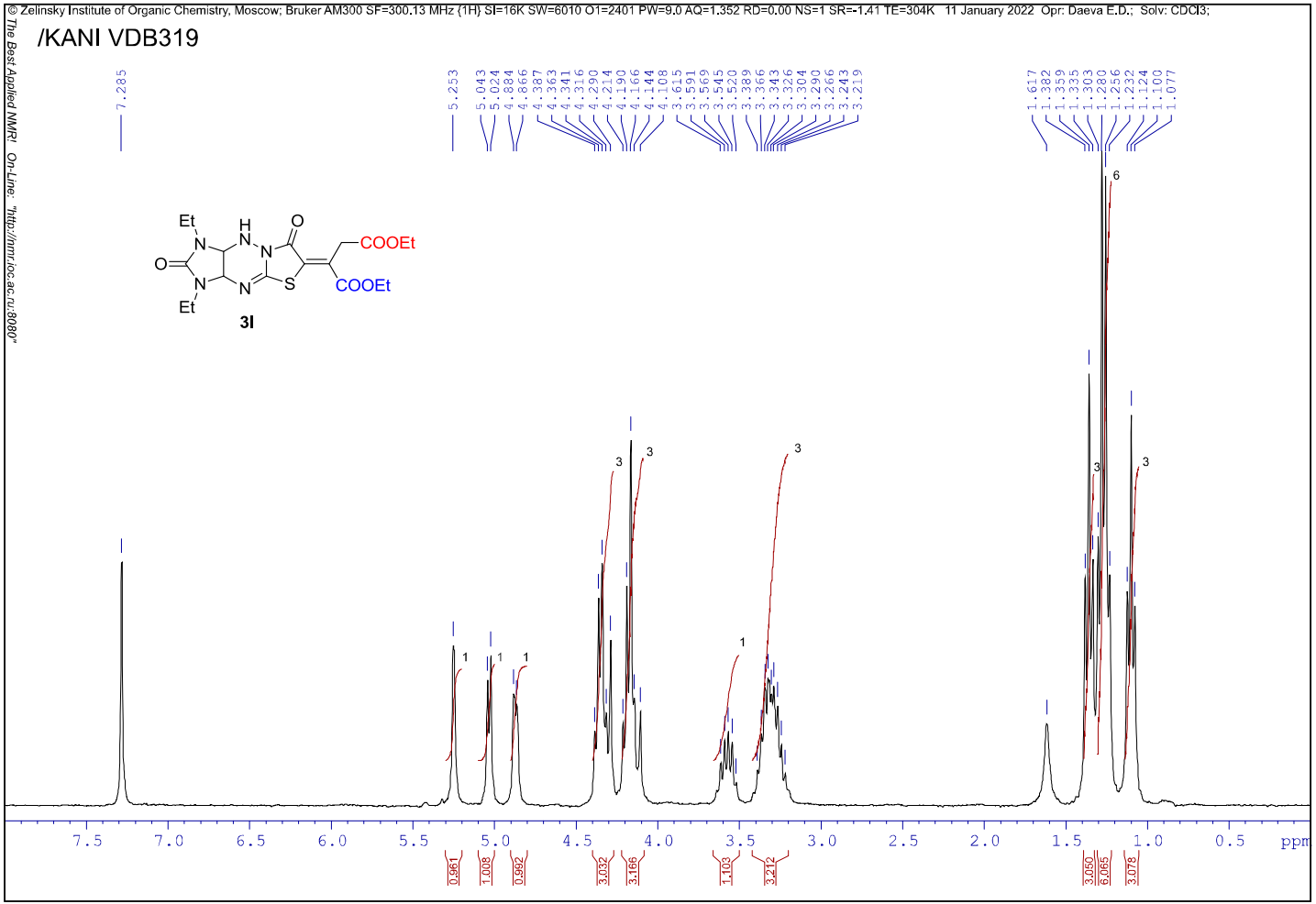
/KANI VDB456.2.2

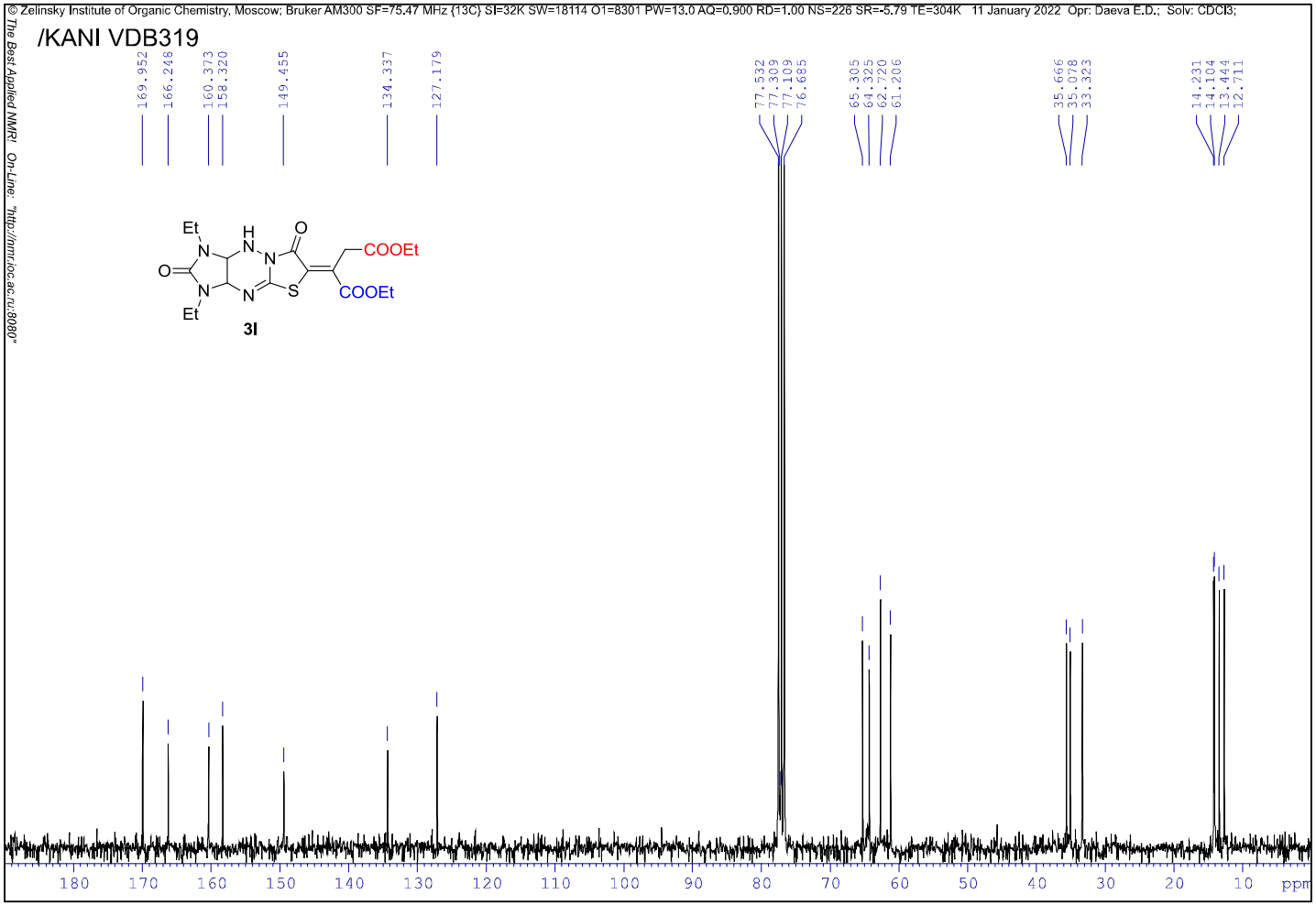


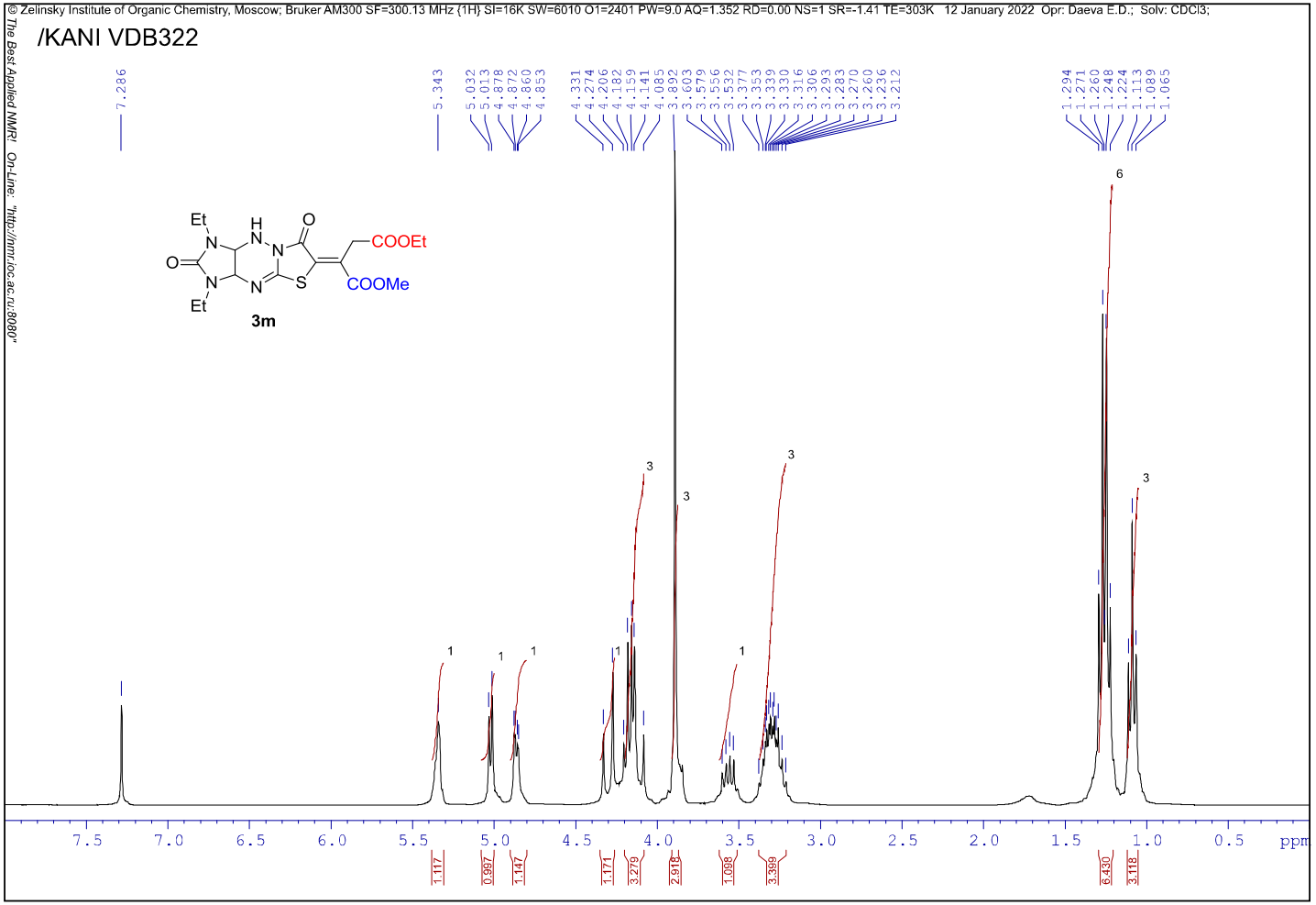

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=552 SR=-5.15 TE=296K 8 September 2021 Opr: Daeva E.D.; Solv: CDCl3;
/KANI VM2073.1
The Best Applied NMR! On-Line. "http://nmr.joc.ac.ru:8080"
195.602
166.800
159.910
158.359
146.658
136.592
134.473
133.535
133.306
129.227
129.098
128.823
128.533
128.436
128.277
128.229
127.708
127.668
127.093
85.097
80.689
77.530
77.311
77.107
76.683
53.444
38.264
26.604
25.176
Me
Ph
H
N
N
N
O
O
N
N
S
Me
Ph
COPh
COOMe
3j
200
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=6010 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=4 SR=-1.40 TE=296K 8 September 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI VM2078.1
7.287
7.227
7.202
7.165
7.140
7.137
7.108
7.082
7.059
6.857
6.836
6.810
6.362
4.503
4.447
4.255
4.199
4.033
2.829
2.759
Me
Ph
H
N
O
CN
COOMe
3k
6
4
1
3
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
6.434
4.331
1.119
1.029
1.092
3.404
6.851


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=494 SR=-5.11 TE=297K 9 September 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI VM2078.1
165.457
159.790
157.551
145.459
137.926
134.074
133.012
129.275
128.557
128.449
127.745
127.622
127.115
121.597
115.880
85.282
80.769
77.530
77.107
76.683
54.005
26.604
25.314
16.038
Me
Ph
H
N
O
CN
COOMe
S
3k
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

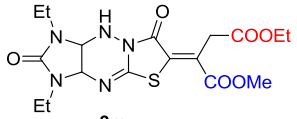

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=1 SR=-1.41 TE=304K 11 January 2022 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line. "http://nmr.ioc.ac.ru:8080"
/KANI VDB319
7.285
5.253
5.043
5.024
4.884
4.866
4.387
4.363
4.341
4.316
4.290
4.214
4.190
4.166
4.144
4.108
3.615
3.591
3.569
3.545
3.520
3.389
3.366
3.343
3.326
3.304
3.290
3.266
3.243
3.219
1.617
1.382
1.359
1.335
1.303
1.280
1.256
1.232
1.124
1.100
1.077
Et
N
H
N
O
O
N
N
N
S
COOEt
COOEt
Et
3l
1
1
1
3
3
1
3
3
3
6
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
0.961
1.008
0.992
3.032
3.166
1.103
3.212
3.050
6.065
3.078


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=226 SR=-5.79 TE=304K 11 January 2022 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line. "http://nmr.ioc.ac.ru:8080"
/KANI VDB319
169.952
166.248
160.373
158.320
149.455
134.337
127.179
77.532
77.309
77.109
76.685
65.305
64.325
62.720
61.206
35.666
35.078
33.323
14.231
14.104
13.444
12.711
Et
N
H
N
O
O
N
N
S
COOEt
COOEt
Et
3l
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

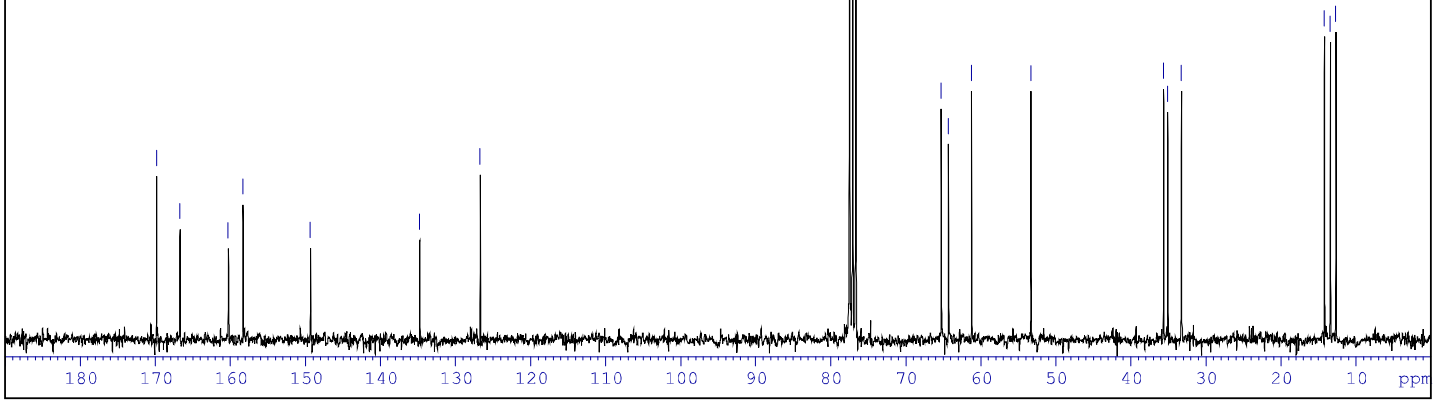

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=1 SR=-1.41 TE=303K 12 January 2022 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI VDB322
7.286
5.343
5.032
5.013
4.878
4.872
4.860
4.853
4.331
4.274
4.206
4.182
4.159
4.141
4.085
3.892
3.603
3.579
3.556
3.532
3.377
3.353
3.339
3.330
3.316
3.306
3.293
3.283
3.270
3.260
3.236
3.212
1.294
1.271
1.260
1.248
1.224
1.113
1.089
1.065
Et
N
H
N
O
N
N
O
N
S
Et
COOEt
COOMe
3m
6
3
3
3
1
1
1
1
1
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
1.117
0.997
1.147
1.171
3.279
2.918
1.098
3.399
6.430
3.118

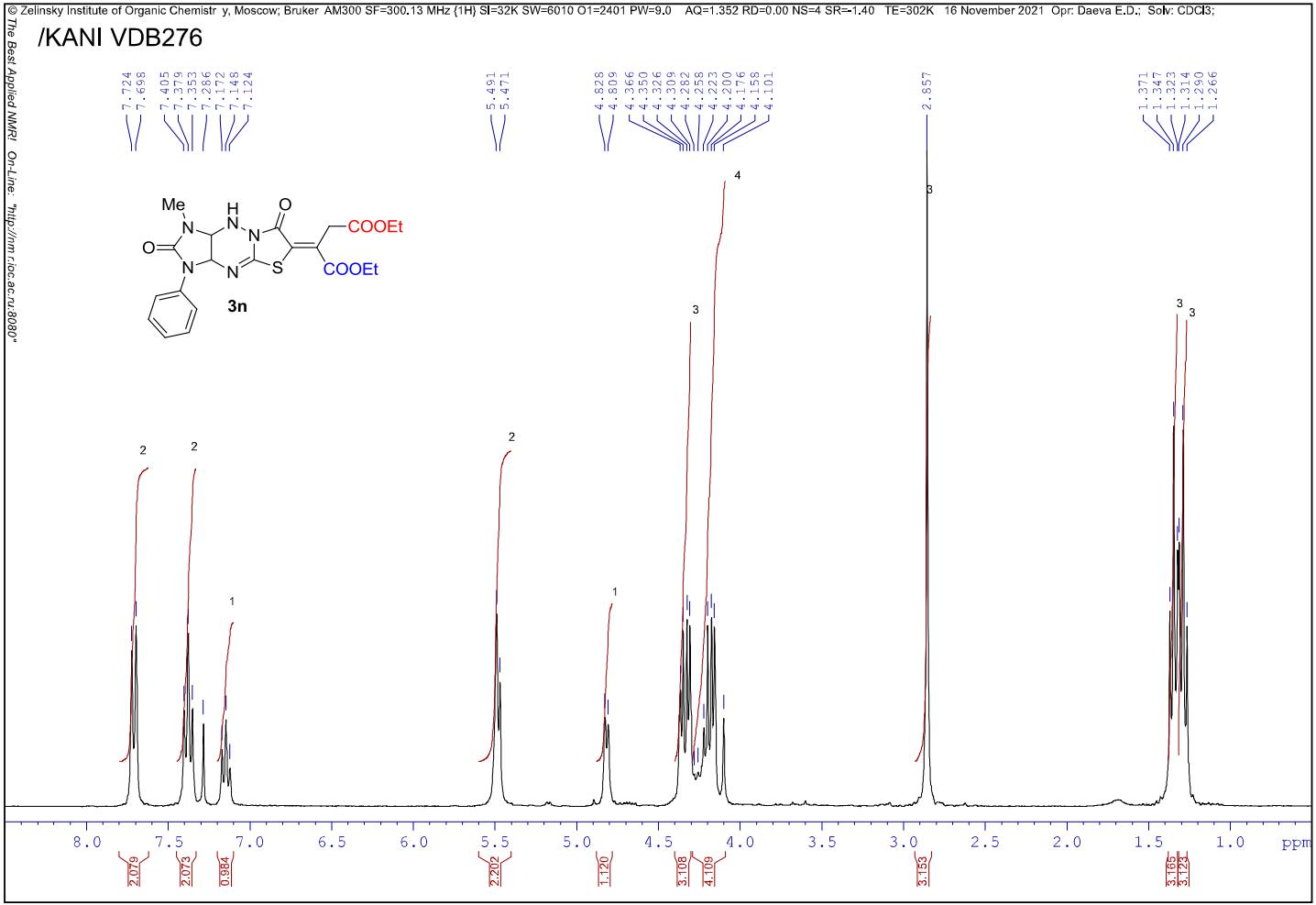
/KANI VDB322

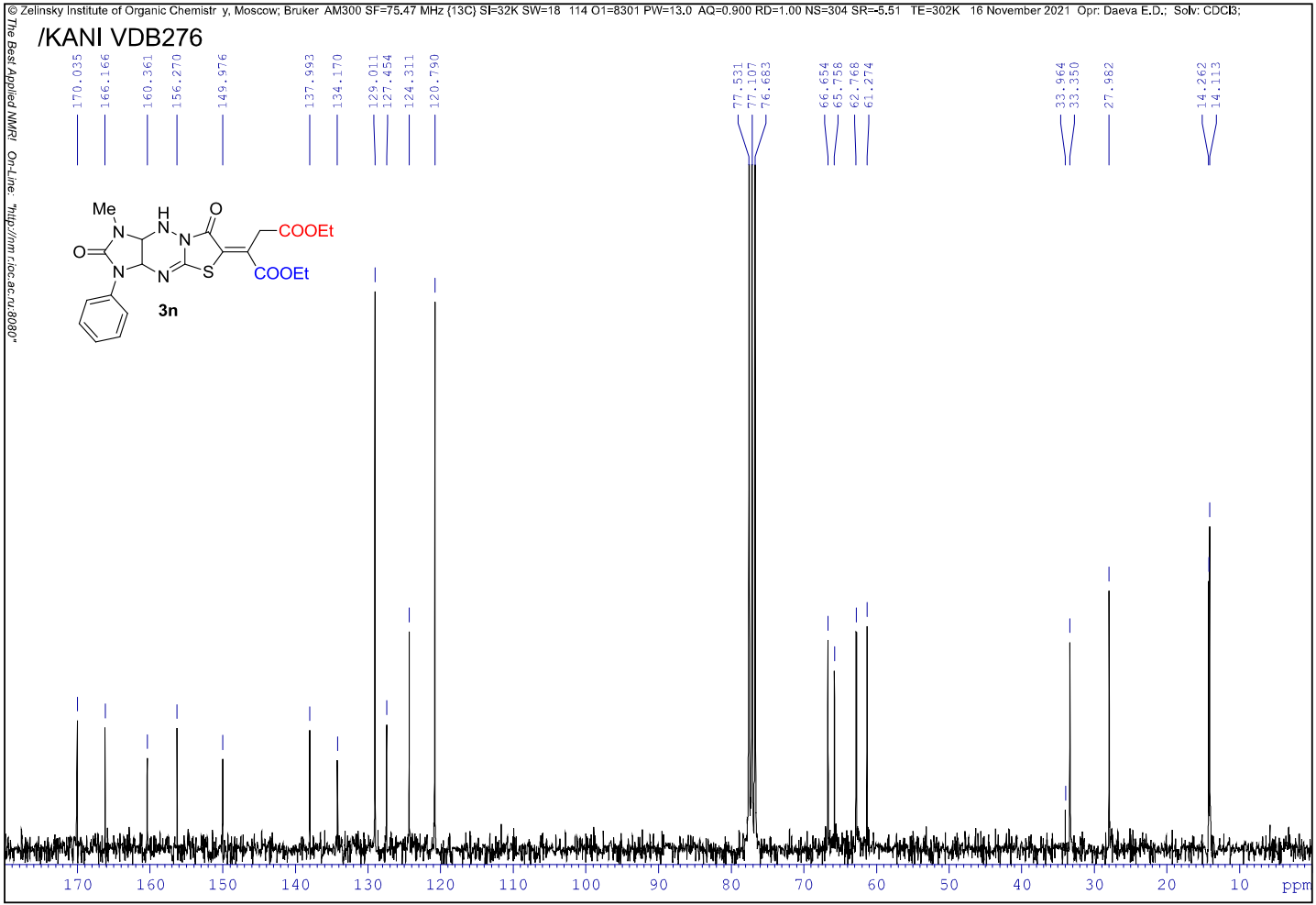
169.866
166.773
160.296
158.310
149.373
134.783
126.729
77.529
77.106
76.682
65.299
64.341
61.233
53.314
35.669
35.093
33.288
14.222
13.440
12.701

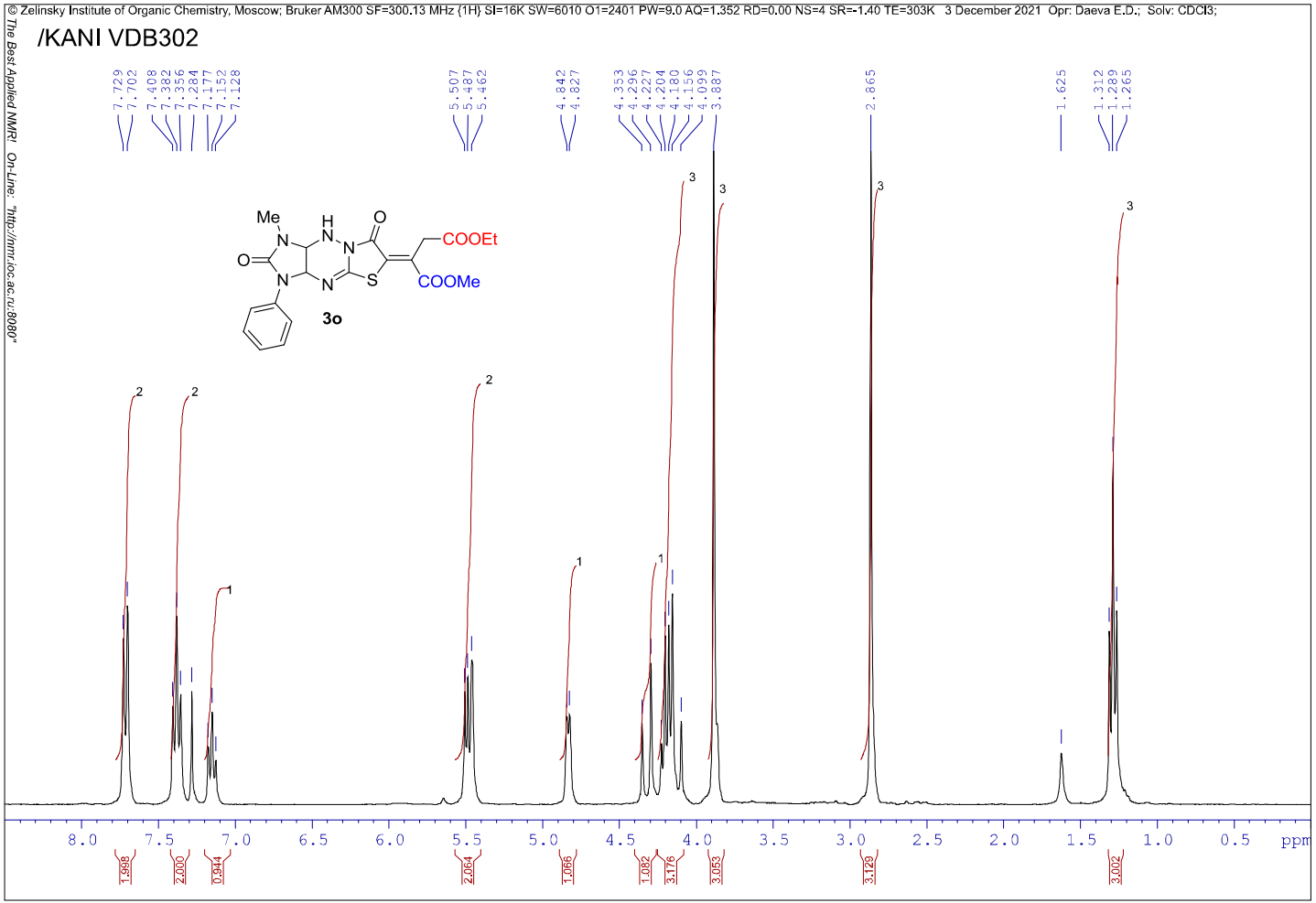


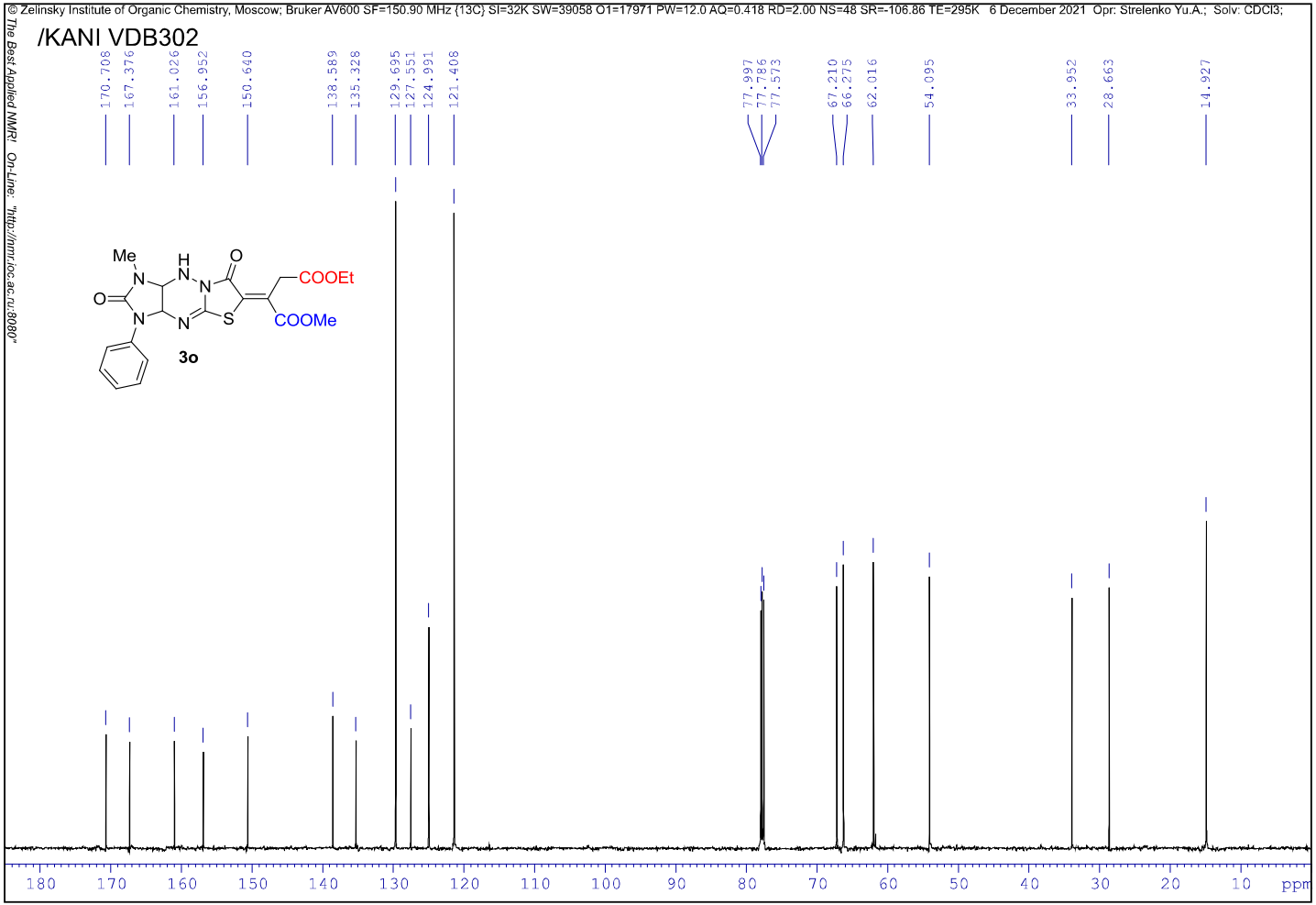
3m

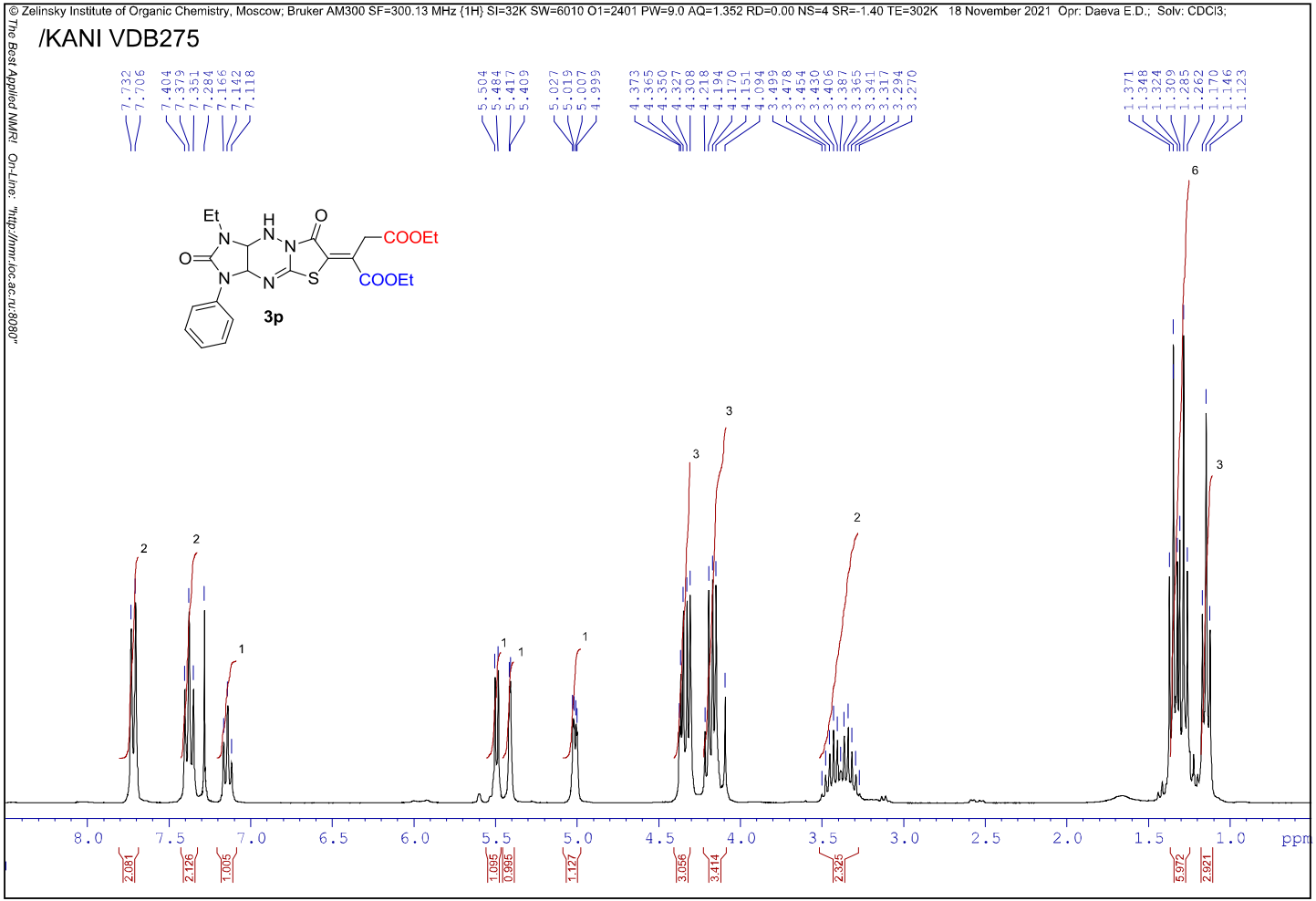


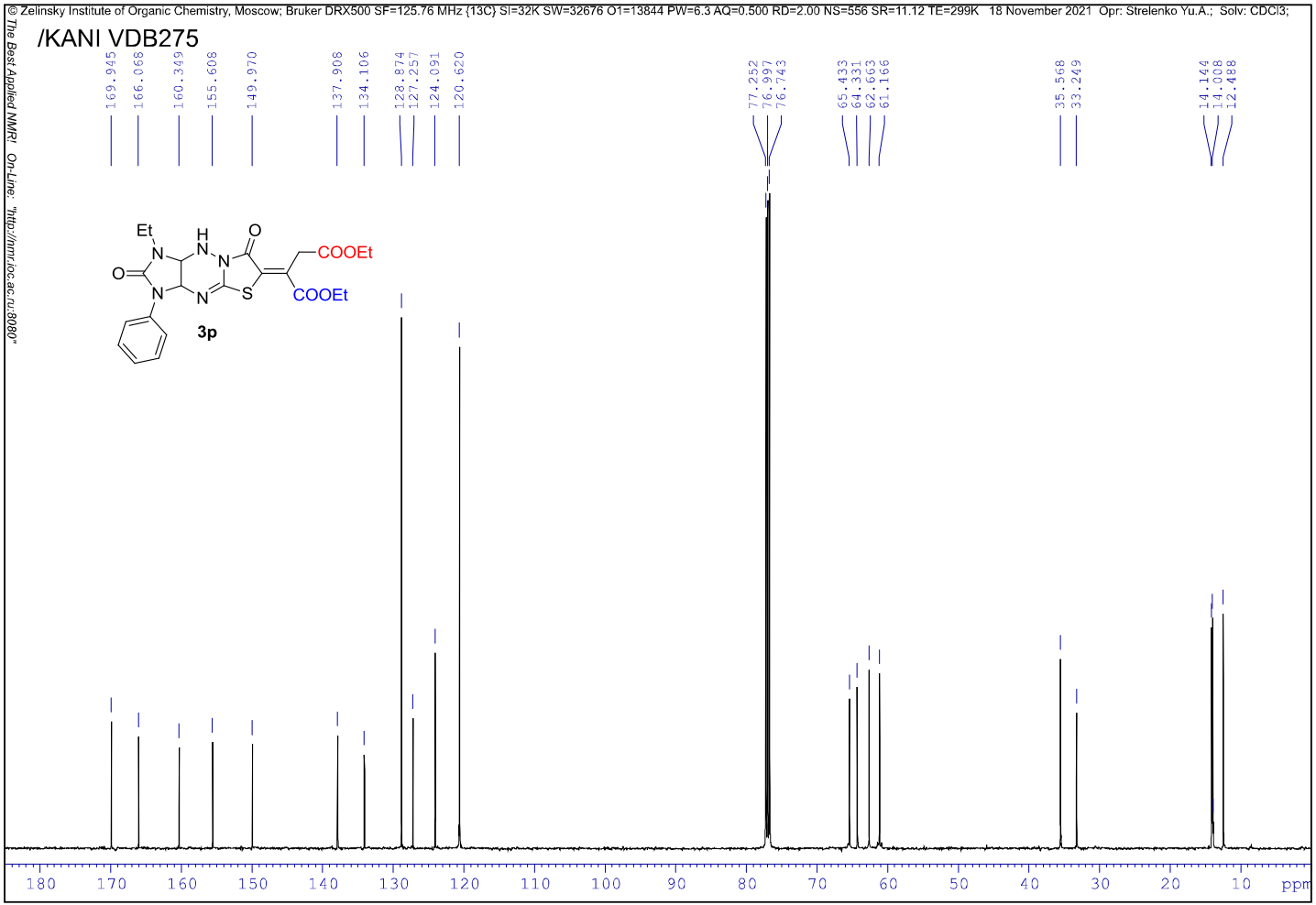

© Zelinsky Institute of Organic Chemistr y, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=32K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=4 SR=-1.40 TE=302K 16 November 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI VDB276
7.724
7.698
7.405
7.379
7.353
7.286
7.172
7.148
7.124
5.491
5.471
4.828
4.809
4.366
4.350
4.326
4.309
4.282
4.258
4.223
4.200
4.176
4.158
4.101
2.657
1.371
1.347
1.323
1.314
1.290
1.266
Me
N
H
N
N
O
O
N
N
S
COOEt
COOEt
3n
2
2
1
2
1
3
4
3
3
3
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
2.079
2.073
0.984
2.202
1.120
3.108
4.109
3.153
3.165
3.123


© Zelinsky Institute of Organic Chemistr y, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18 114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=304 SR=-5.51 TE=302K 16 November 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI VDB276
170.035
166.166
160.361
156.270
149.976
137.993
134.170
129.011
127.454
124.311
120.790
77.531
77.107
76.683
66.654
65.758
62.768
61.274
33.964
33.350
27.982
14.262
14.113
Me
N
H
N
N
O
O
N
N
S
COOEt
COOEt
3n
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

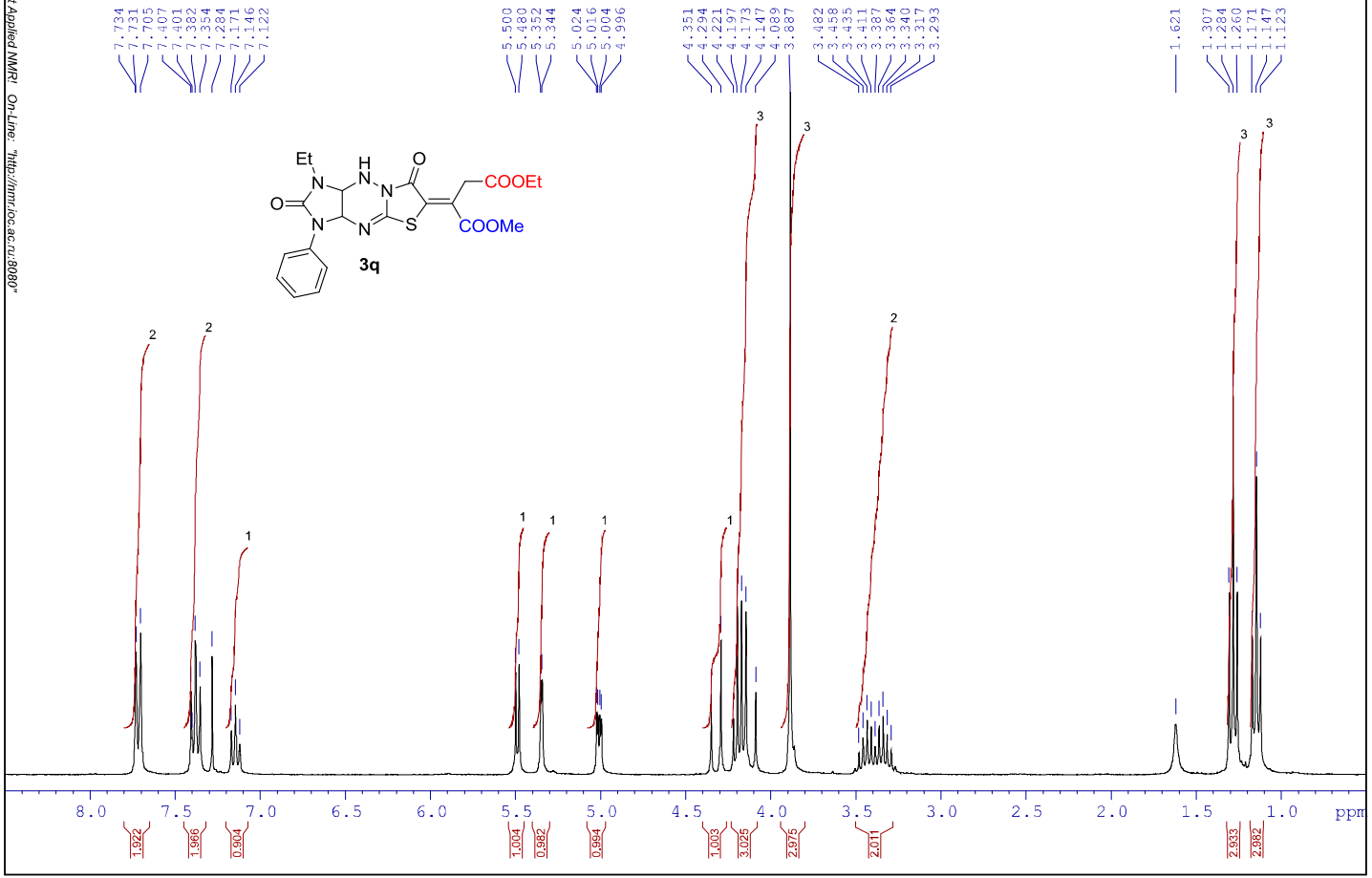

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=4 SR=-1.40 TE=303K 3 December 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI VDB302
7.729
7.702
7.408
7.382
7.356
7.284
7.177
7.152
7.128
5.507
5.487
5.462
4.842
4.827
4.353
4.296
4.227
4.204
4.180
4.156
4.099
3.887
2.865
1.625
1.312
1.289
1.265
Me
N
H
N
N
O
O
N
N
S
COOEt
COOMe
3o
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
1.998
2.000
0.944
2.064
1.066
1.082
3.176
3.053
3.129
3.002

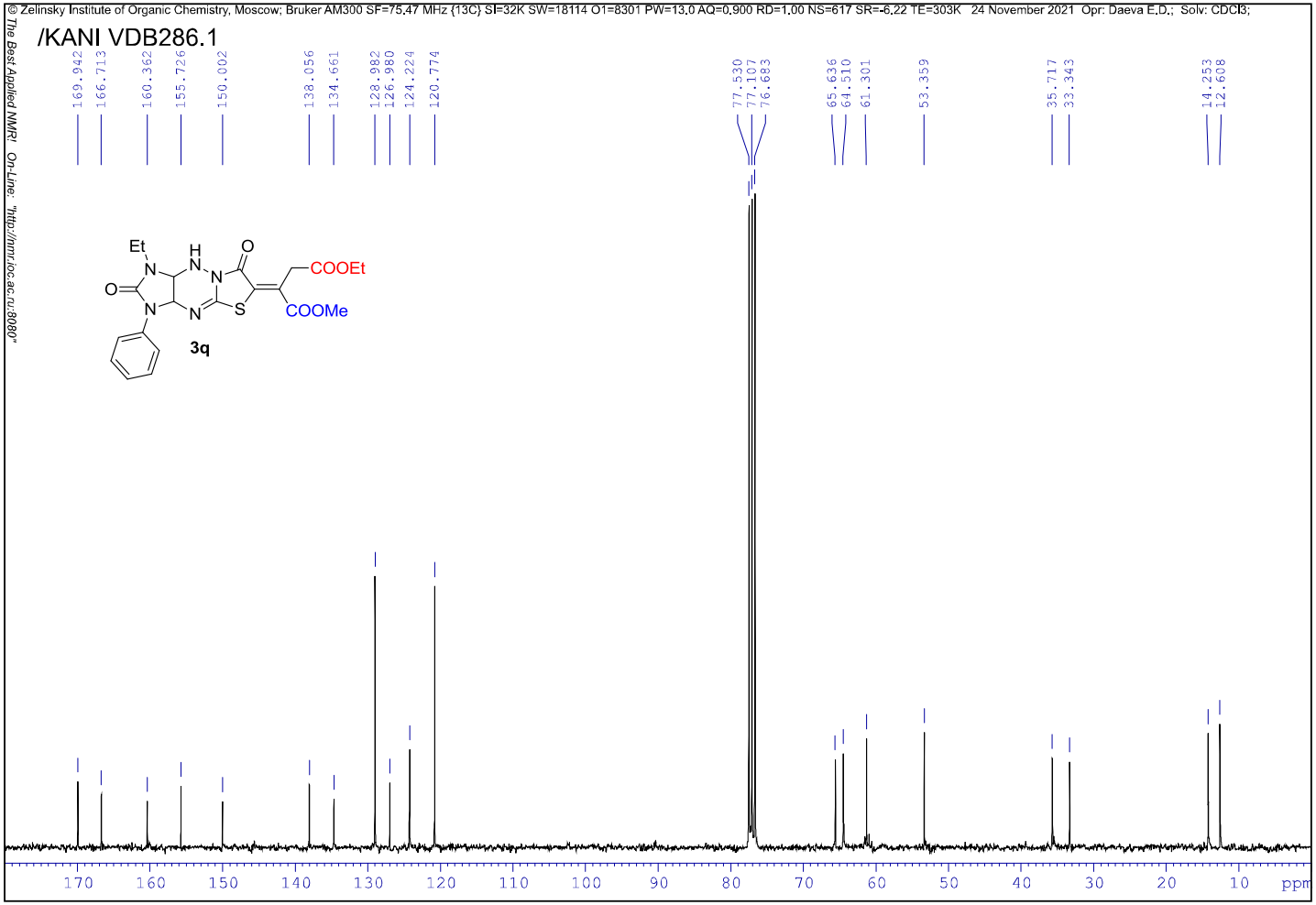

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AV600 SF=150.90 MHz {13C} SI=32K SW=39058 O1=17971 PW=12.0 AQ=0.418 RD=2.00 NS=48 SR=-106.86 TE=295K 6 December 2021 Opr: Strelenko Yu.A.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI VDB302
170.708
167.376
161.026
156.952
150.640
138.589
135.328
129.695
127.551
124.991
121.408
77.997
77.786
77.573
67.210
66.275
62.016
54.095
33.952
28.663
14.927
Me
N
H
N
N
O
O
N
N
S
COOEt
COOMe
3o
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

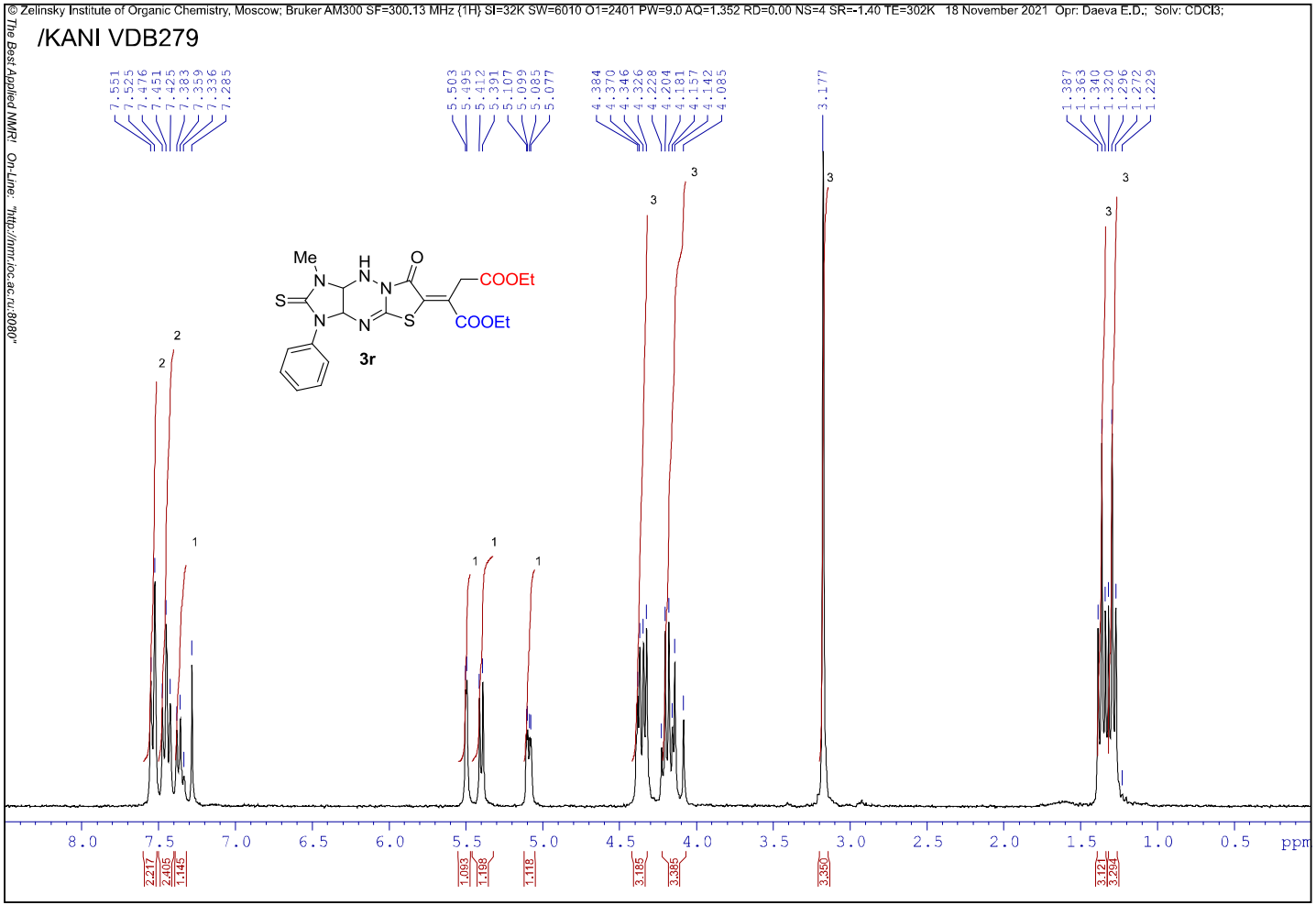

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=32K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=4 SR=-1.40 TE=302K 18 November 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI VDB275
7.732 7.706 7.404 7.379 7.351 7.284 7.166 7.142 7.118
5.504 5.484 5.417 5.409 5.027 5.019 5.007 4.999
4.373 4.365 4.350 4.327 4.308 4.218 4.194 4.170 4.151 4.094 3.499 3.478 3.454 3.430 3.406 3.387 3.365 3.341 3.317 3.294 3.270
1.371 1.348 1.324 1.309 1.285 1.262 1.170 1.146 1.123
Et
N
H
N
N
O
O
N
N
S
COOEt
COOEt
3p
2 2 1 1 1 1 3 3 2 6 3
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
2.081 2.126 1.005 1.095 0.995 1.127 3.056 3.414 2.325 5.972 2.921

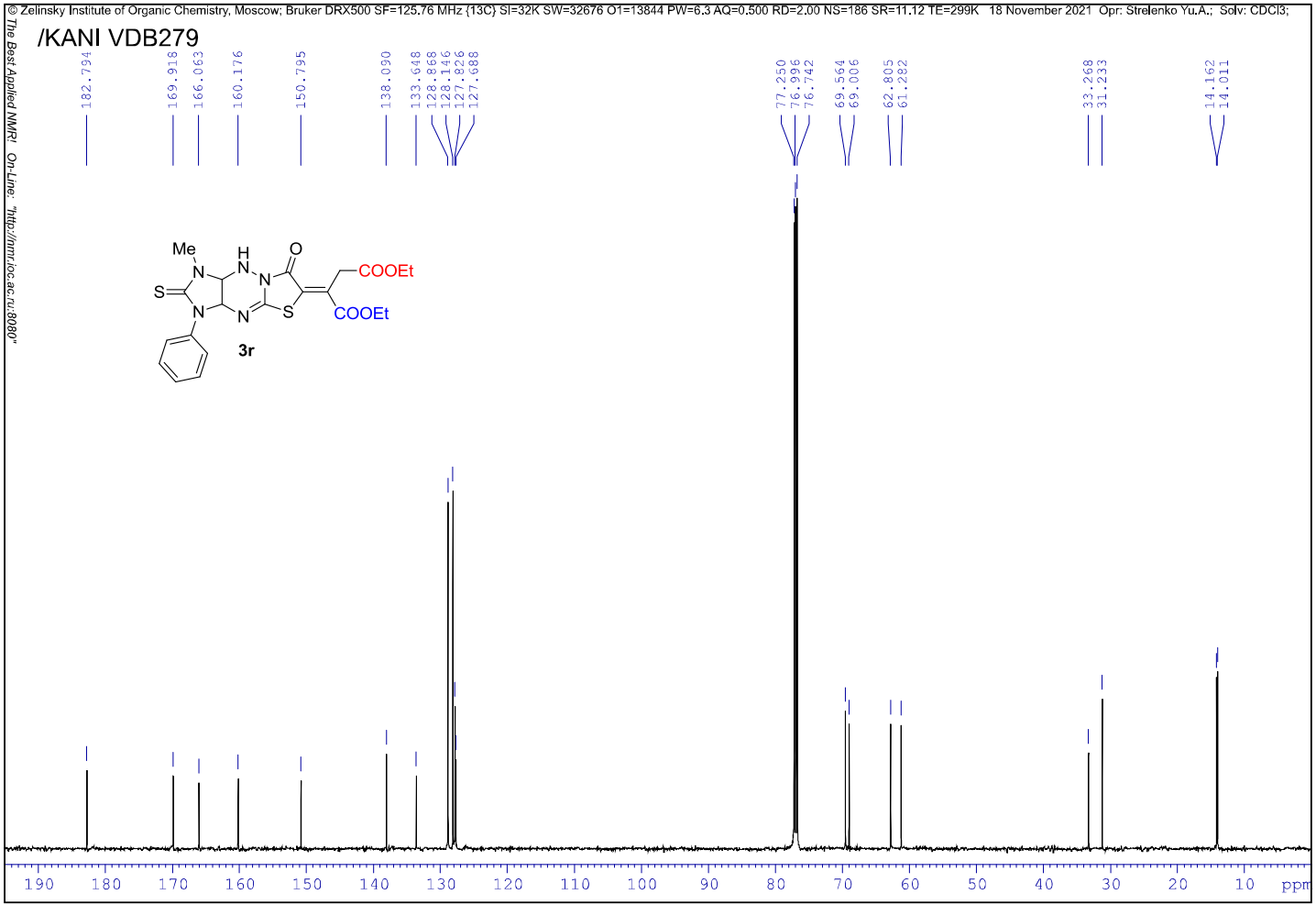

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker DRX500 SF=125.76 MHz {13C} SI=32K SW=32676 O1=13844 PW=6.3 AQ=0.500 RD=2.00 NS=556 SR=11.12 TE=299K 18 November 2021 Opr: Strelenko Yu.A.; Solv: CDCl3;
The Best Applied NMR! On-Line. "http://nmr.ioc.ac.ru:8080"
/KANI VDB275
169.945
166.068
160.349
155.608
149.970
137.908
134.106
128.874
127.257
124.091
120.620
77.252
76.997
76.743
65.433
64.331
62.663
61.166
35.568
33.249
14.144
14.008
12.488
Et
N
H
N
O
O
N
N
N
S
COOEt
COOEt
3p
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

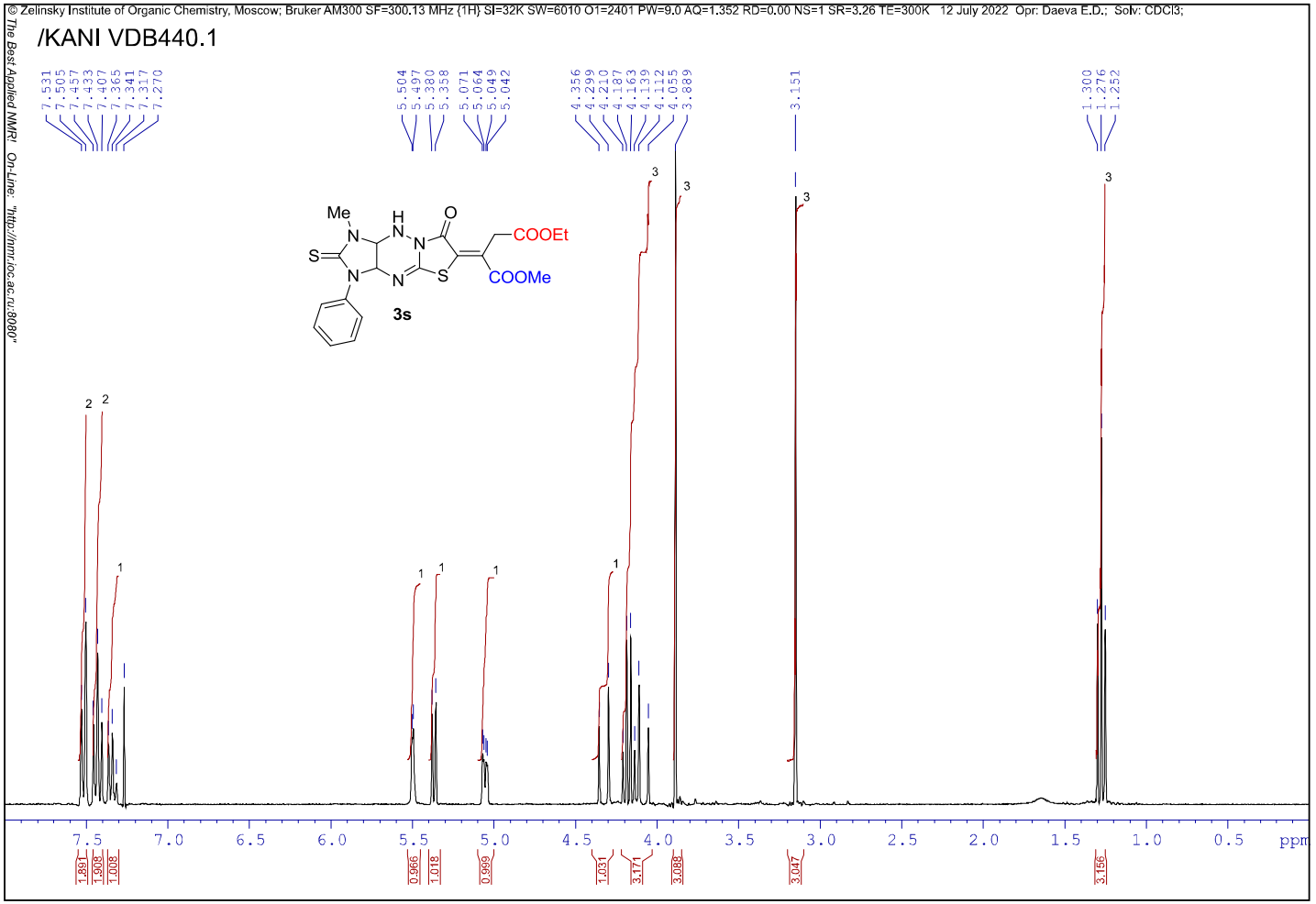
/KANI VDB448.2

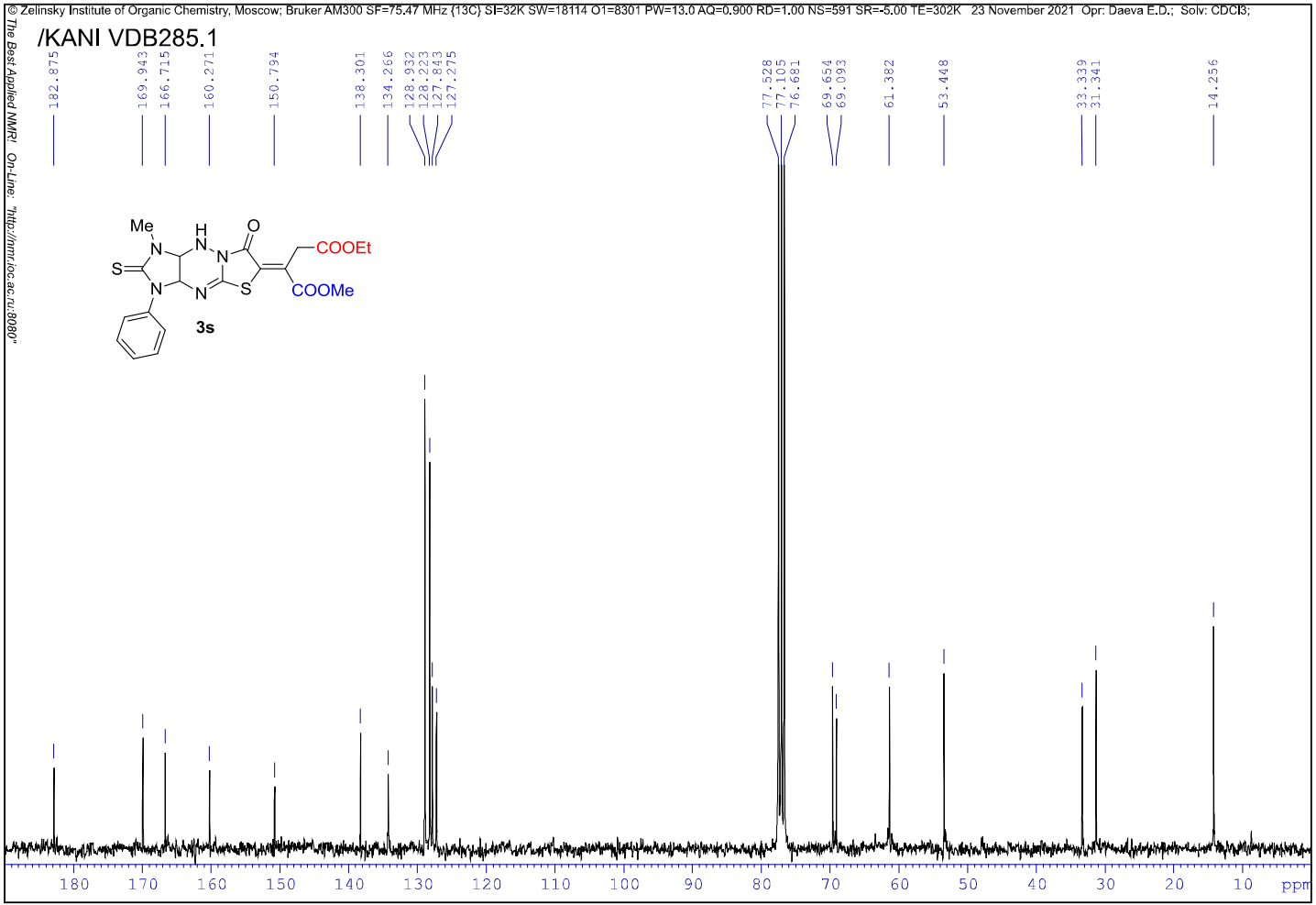


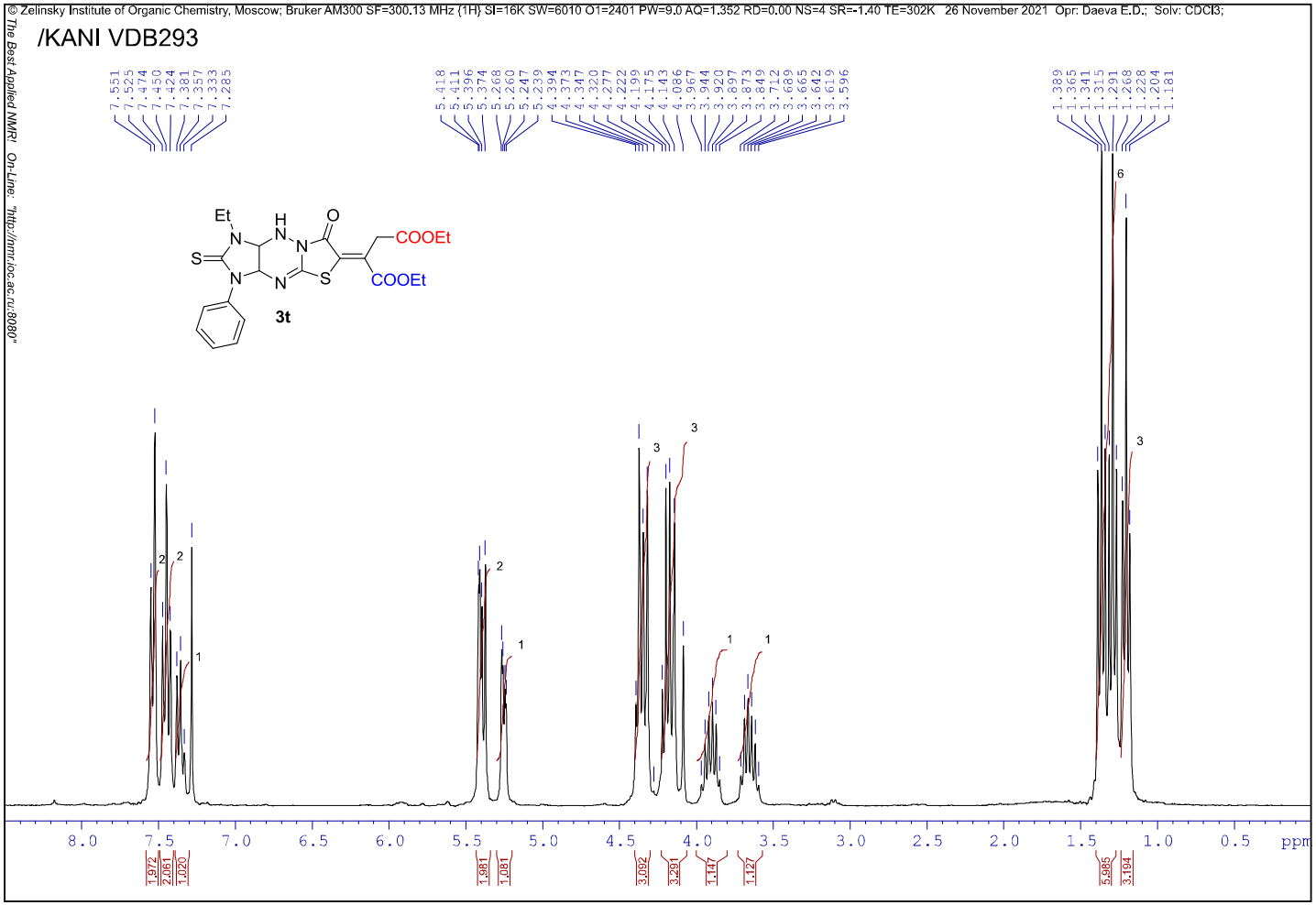

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=617 SR=-6.22 TE=303K 24 November 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line. "http://nmr.ioc.ac.ru:8080"
/KANI VDB286.1
169.942
166.713
160.362
155.726
150.002
138.056
134.661
128.982
126.980
124.224
120.774
77.530
77.107
76.683
65.636
64.510
61.301
53.359
35.717
33.343
14.253
12.608
Et
N
H
N
O
O
N
N
N
S
COOEt
COOMe
3q
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

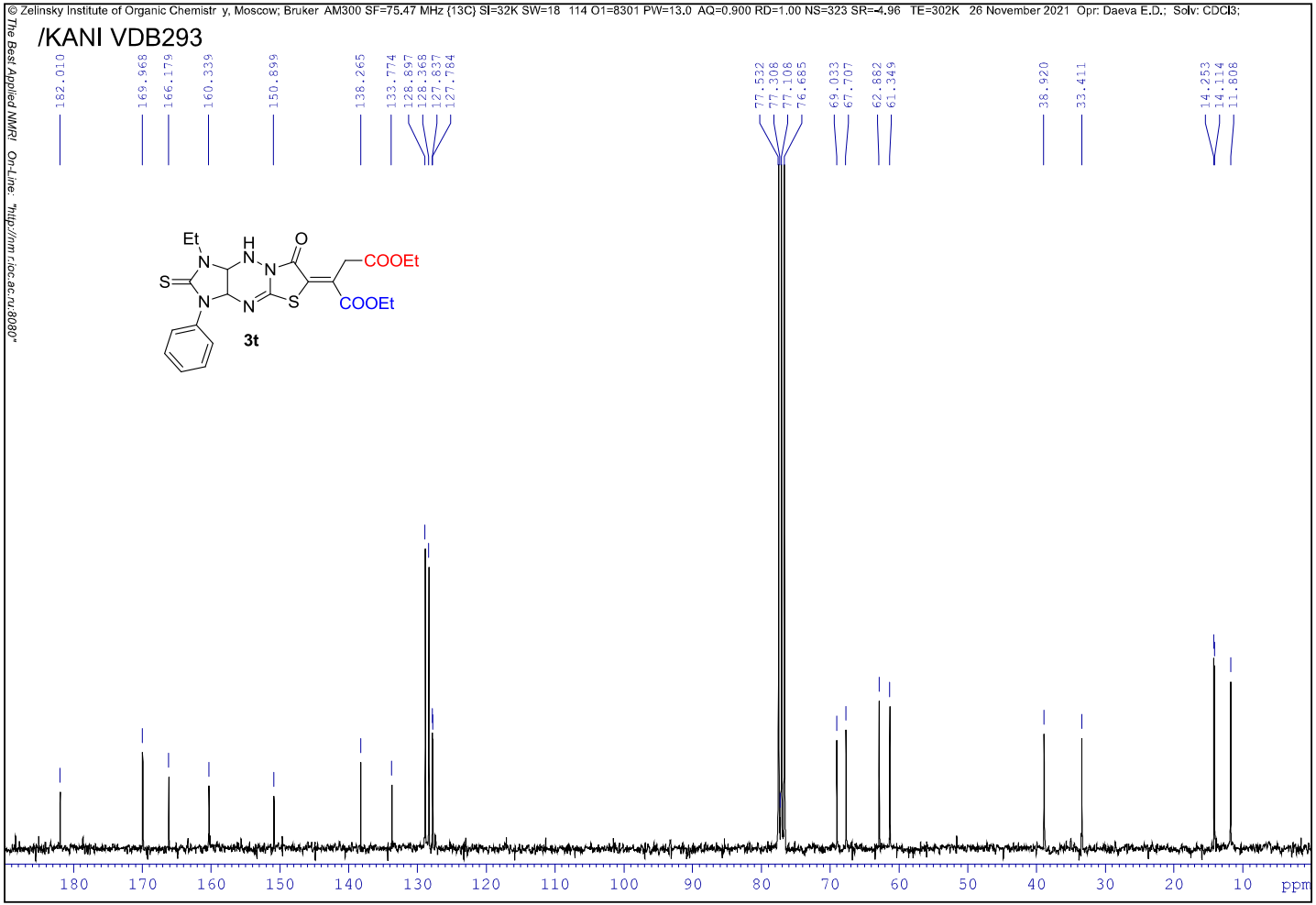

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=32K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=4 SR=-1.40 TE=302K 18 November 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line. "http://nmr.ioc.ac.ru:8080"
/KANI VDB279
7.551
7.525
7.476
7.451
7.425
7.383
7.359
7.336
7.285
5.503
5.495
5.412
5.391
5.107
5.099
5.085
5.077
4.384
4.370
4.346
4.326
4.228
4.204
4.181
4.157
4.142
4.085
3.177
1.387
1.363
1.340
1.320
1.296
1.272
1.229
Me
N
H
N
O
S
N
N
N
S
COOEt
COOEt
3r
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
2.217
2.405
1.145
1.093
1.198
1.118
3.185
3.385
3.350
3.121
3.294


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker DRX500 SF=125.76 MHz {13C} SI=32K SW=32676 O1=13844 PW=6.3 AQ=0.500 RD=2.00 NS=186 SR=11.12 TE=299K 18 November 2021 Opr: Strelenko Yu.A.; Solv: CDCl3;
/KANI VDB279
The Best Applied NMR! On-Line. "http://nmr.ioc.ac.ru:8080"
182.794
169.918
166.063
160.176
150.795
138.090
133.648
128.868
128.146
127.826
127.688
77.250
76.996
76.742
69.564
69.006
62.805
61.282
33.268
31.233
14.162
14.011
Me
H
O
N
N
N
N
S
S
N
COOEt
COOEt
3r
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

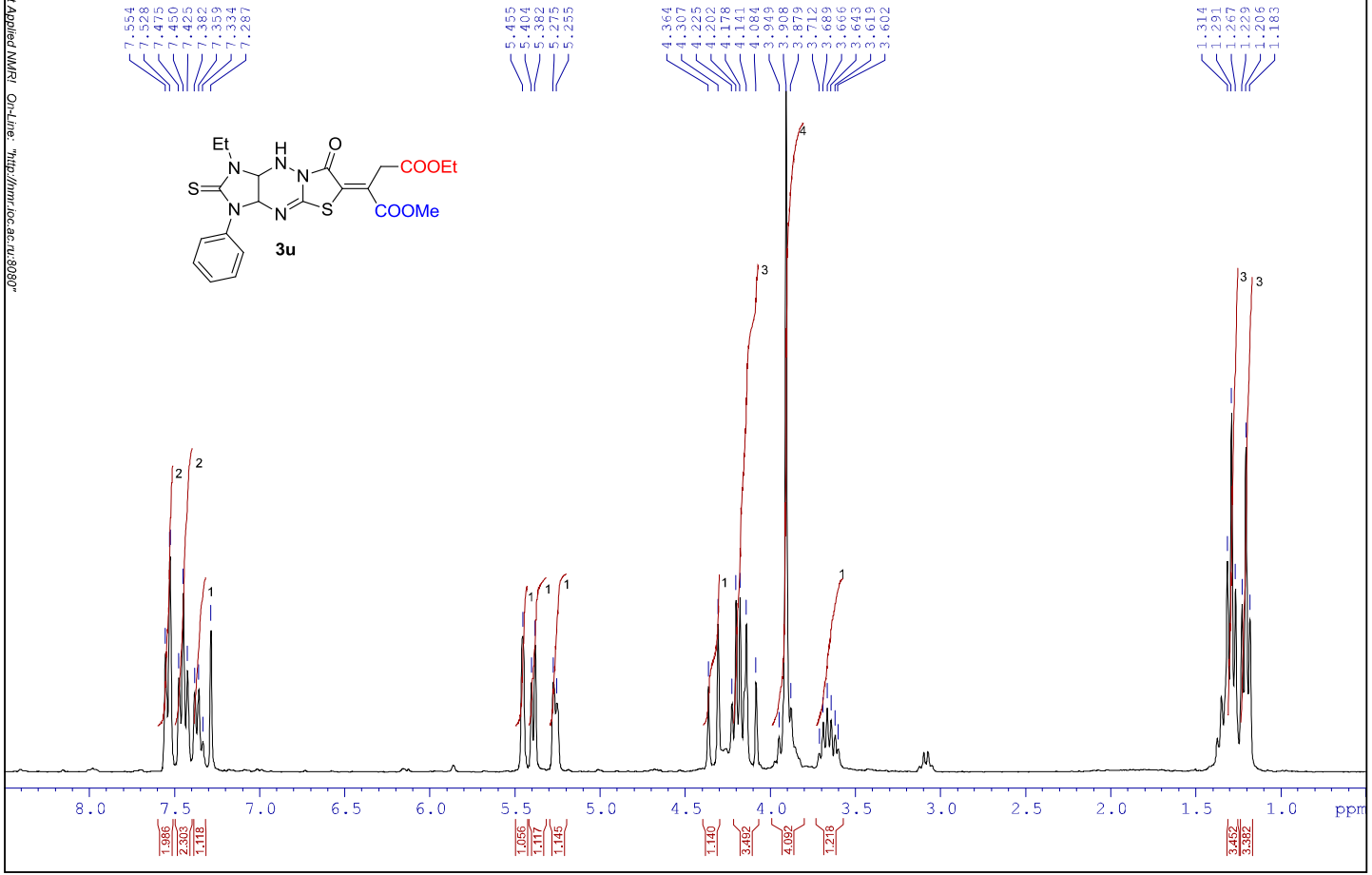

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=32K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=1 SR=3.26 TE=300K 12 July 2022 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI VDB440.1
7.531
7.505
7.457
7.433
7.407
7.365
7.341
7.317
7.270
5.504
5.497
5.380
5.358
5.071
5.064
5.049
5.042
4.356
4.299
4.210
4.187
4.163
4.139
4.122
4.055
3.889
3.151
1.300
1.276
1.252
Me
H
N
O
S
COOEt
COOMe
3s
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
1.891
1.908
1.008
0.966
1.018
0.999
1.031
3.171
3.088
3.047
3.156

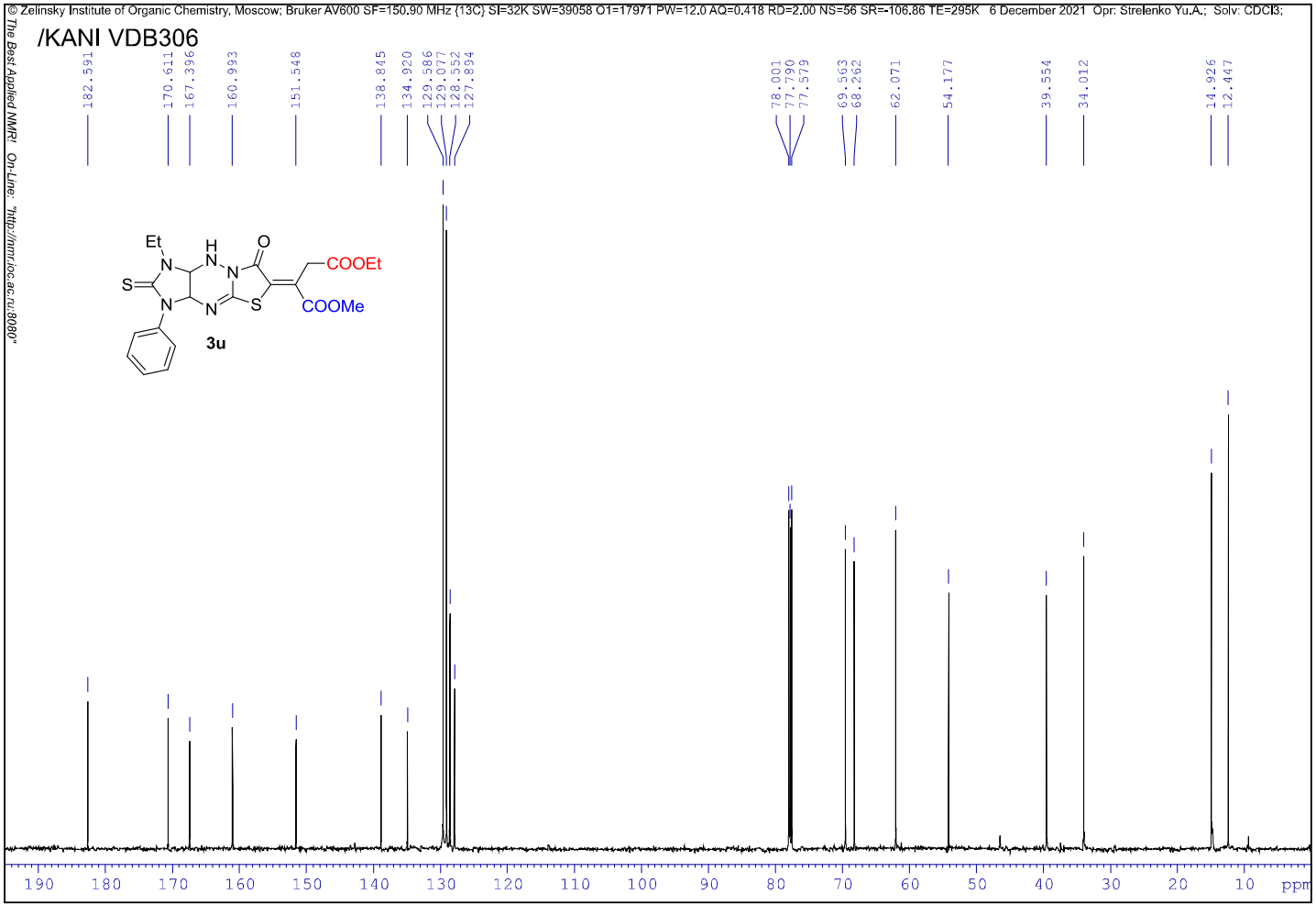

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=591 SR=-5.00 TE=302K 23 November 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line. "http://nmr.ioc.ac.ru:8080"
/KANI VDB285.1
182.875
169.943
166.715
160.271
150.794
138.301
134.266
128.932
128.223
127.843
127.275
77.528
77.105
76.681
69.654
69.093
61.382
53.448
33.339
31.341
14.256
Me
H
O
S
COOEt
COOMe
3s
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

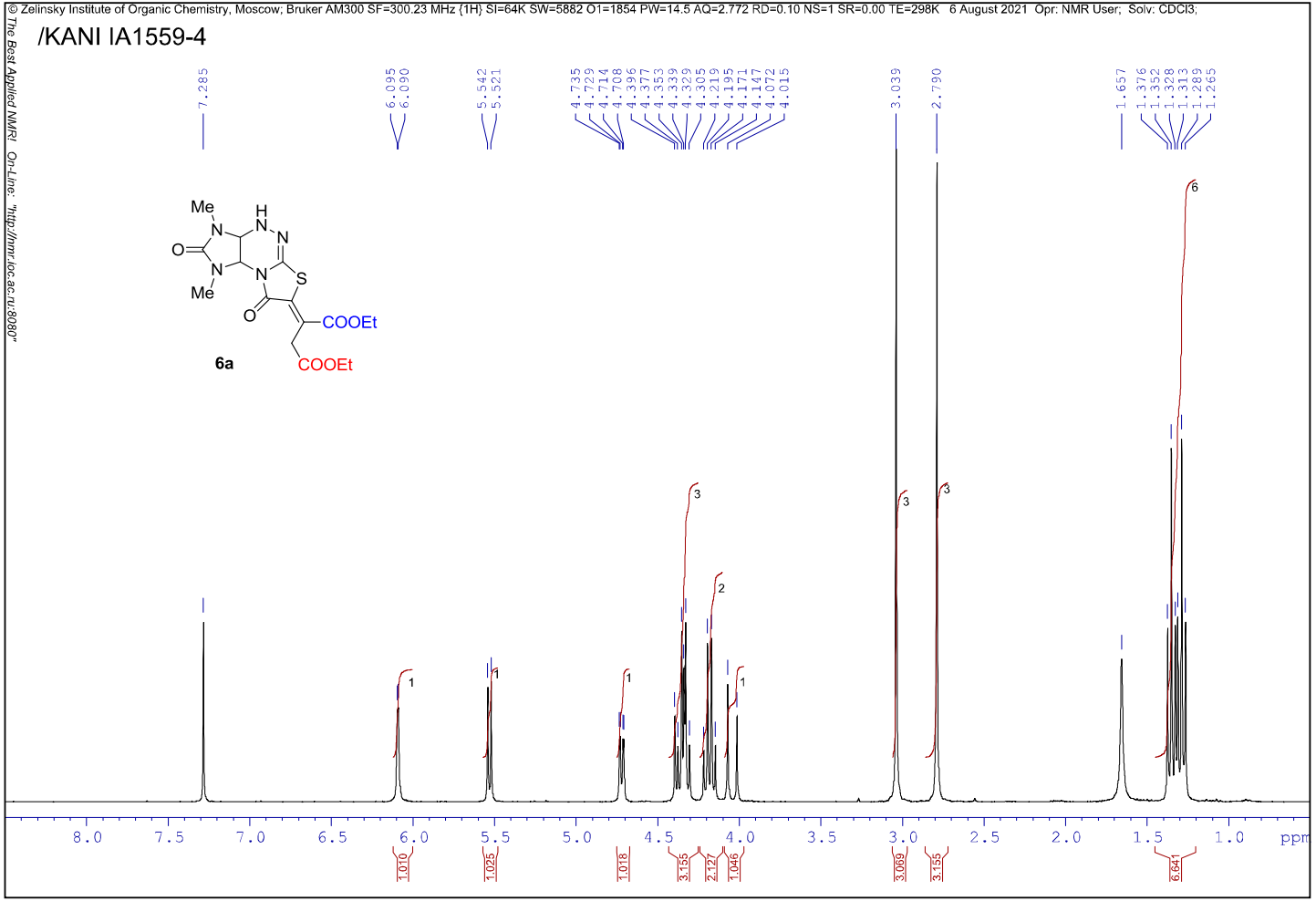

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=4 SR=-1.40 TE=302K 26 November 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line. "http://nmr.ioc.ac.ru:8080"
/KANI VDB293
7.551 7.525 7.474 7.450 7.424 7.381 7.357 7.333 7.285
5.418 5.411 5.396 5.374 5.268 5.260 5.247 5.239 4.394 4.373 4.347 4.320 4.277 4.222 4.199 4.175 4.143 4.086 3.967 3.944 3.920 3.897 3.873 3.849 3.712 3.689 3.665 3.642 3.619 3.596
1.389 1.365 1.341 1.315 1.291 1.268 1.228 1.204 1.181
Et
N
H
N
O
N
S
COOEt
N
N
S
COOEt
3t
2 2 1
2 1
3 3 1 1
6 3
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
1.972 2.061 1.020
1.981 1.081
3.092 3.291 1.147 1.127
5.985 3.194

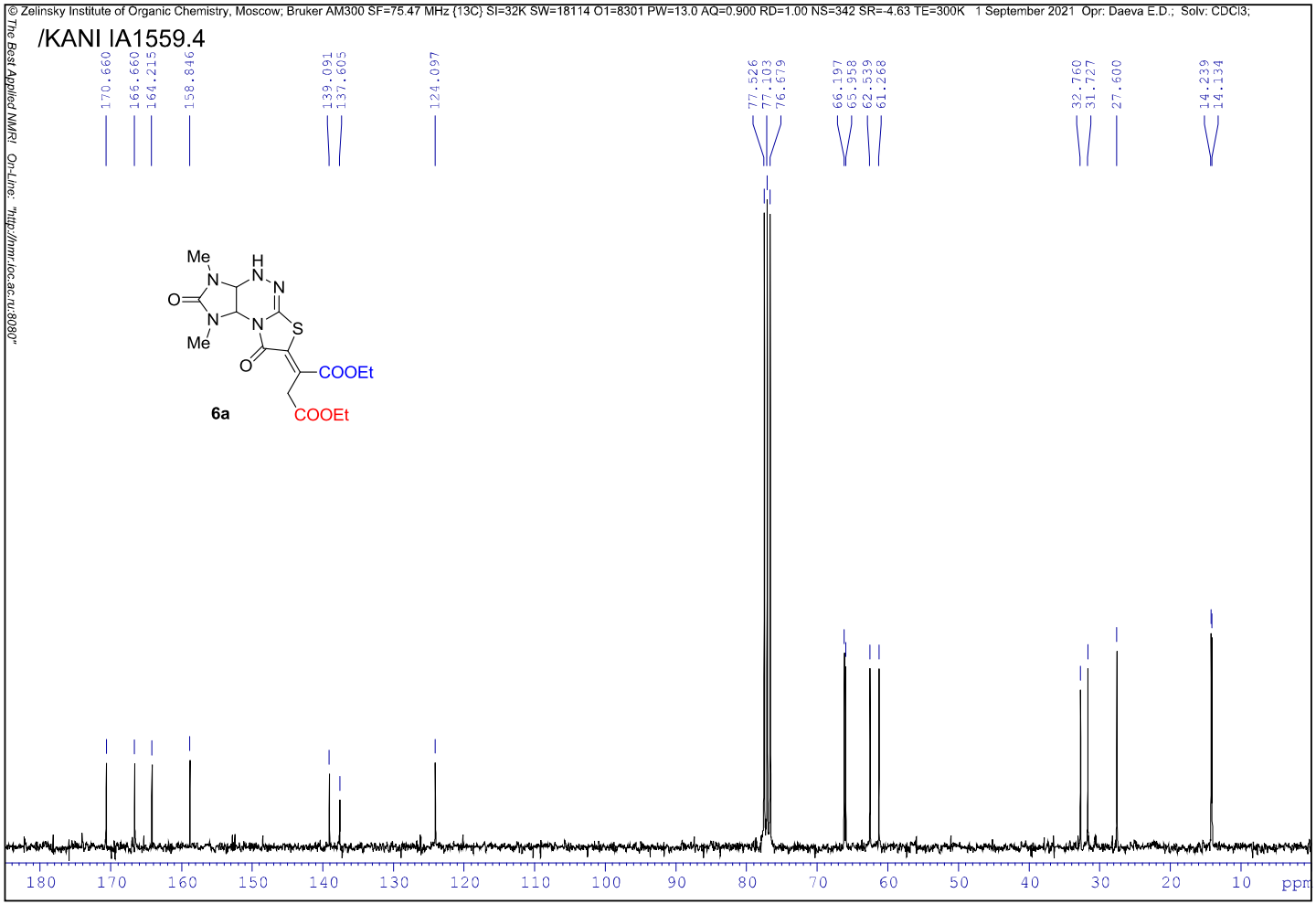

© Zelinsky Institute of Organic Chemistr y, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18 114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=323 SR=-4.96 TE=302K 26 November 2021 Opr: Daeva E.D.; Solv: CDCl3;
/KANI VDB293
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
182.010
169.968
166.179
160.339
150.899
138.265
133.774
128.897
128.368
127.837
127.784
77.532
77.308
77.108
76.685
69.033
67.707
62.882
61.349
38.920
33.411
14.253
14.114
11.808
Et
N
H
N
O
S
N
N
S
COOEt
COOEt
3t
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

/KANI VDB306

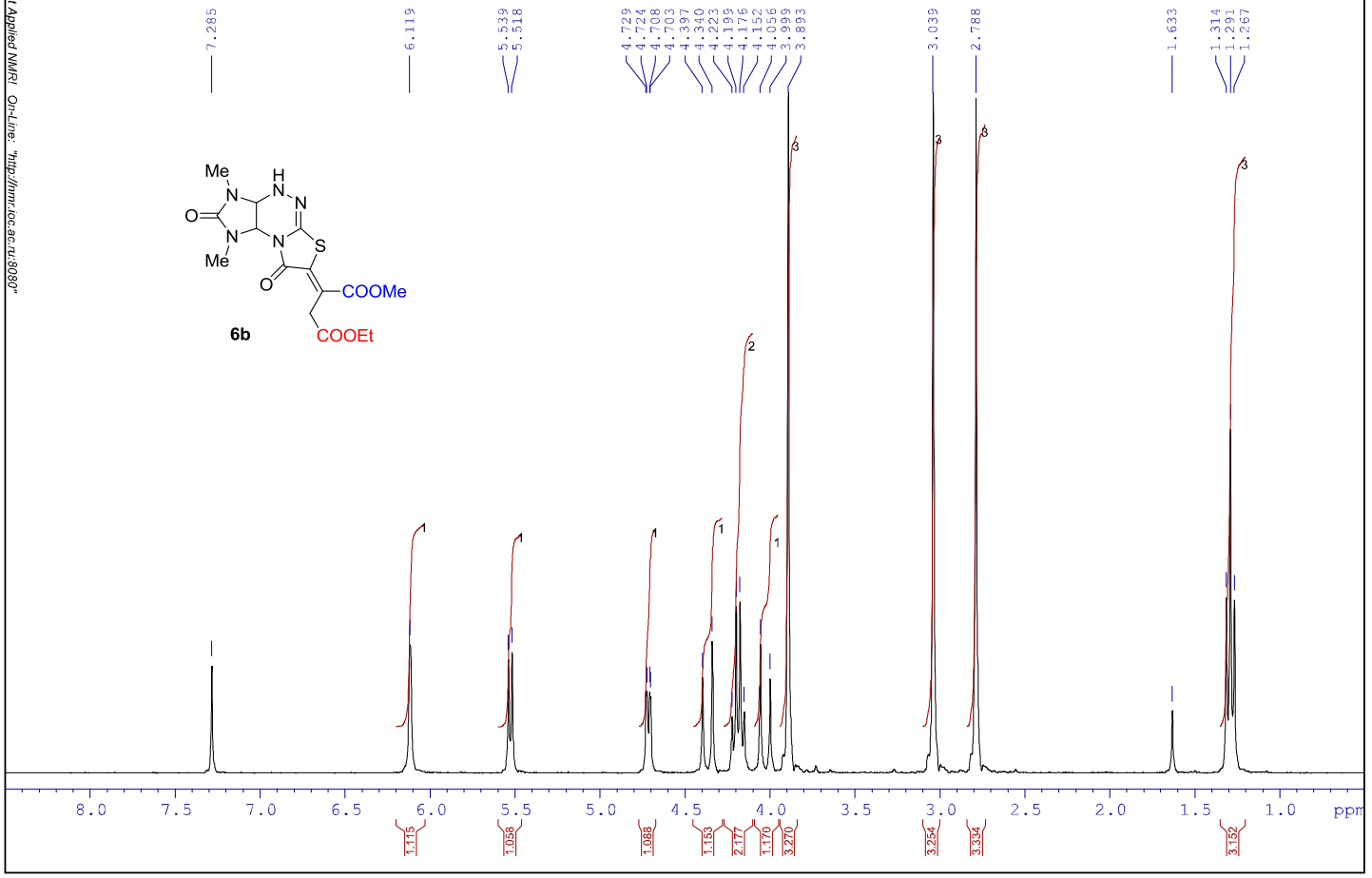


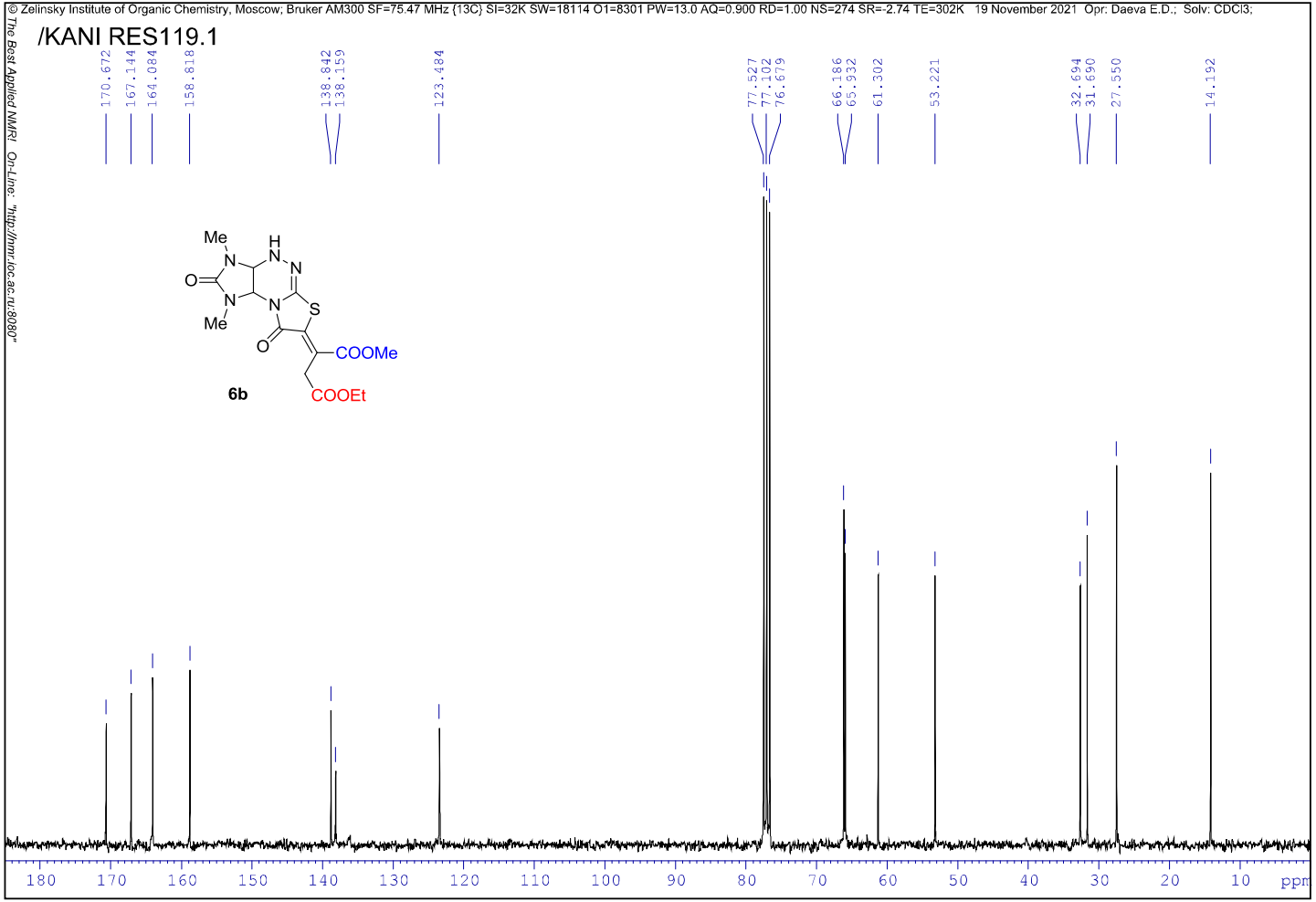

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AV600 SF=150.90 MHz {13C} SI=32K SW=39058 O1=17971 PW=12.0 AQ=0.418 RD=2.00 NS=56 SR=-106.86 TE=295K 6 December 2021 Opr: Strelenko Yu.A.; Solv: CDCl3;
The Best Applied NMR! On-Line. "http://nmr.ioc.ac.ru:8080"
/KANI VDB306
182.591
170.611
167.396
160.993
151.548
138.845
134.920
129.586
129.077
128.552
127.894
78.001
77.790
77.579
69.563
68.262
62.071
54.177
39.554
34.012
14.926
12.447
Et
N
H
N
O
S
N
N
S
COOEt
COOMe
3u
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.23 MHz {1H} SI=64K SW=5882 O1=1854 PW=14.5 AQ=2.772 RD=0.10 NS=1 SR=0.00 TE=298K 6 August 2021 Opr: NMR User; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1559-4
7.285
6.095
6.090
5.542
5.521
4.735
4.729
4.714
4.708
4.396
4.377
4.353
4.339
4.329
4.305
4.219
4.195
4.171
4.147
4.072
4.015
3.039
2.790
1.657
1.376
1.352
1.328
1.313
1.289
1.265
Me
N
H
N
O
N
N
S
Me
O
COOEt
COOEt
6a
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
1.010
1.025
1.018
3.155
2.127
1.046
3.069
3.155
6.641

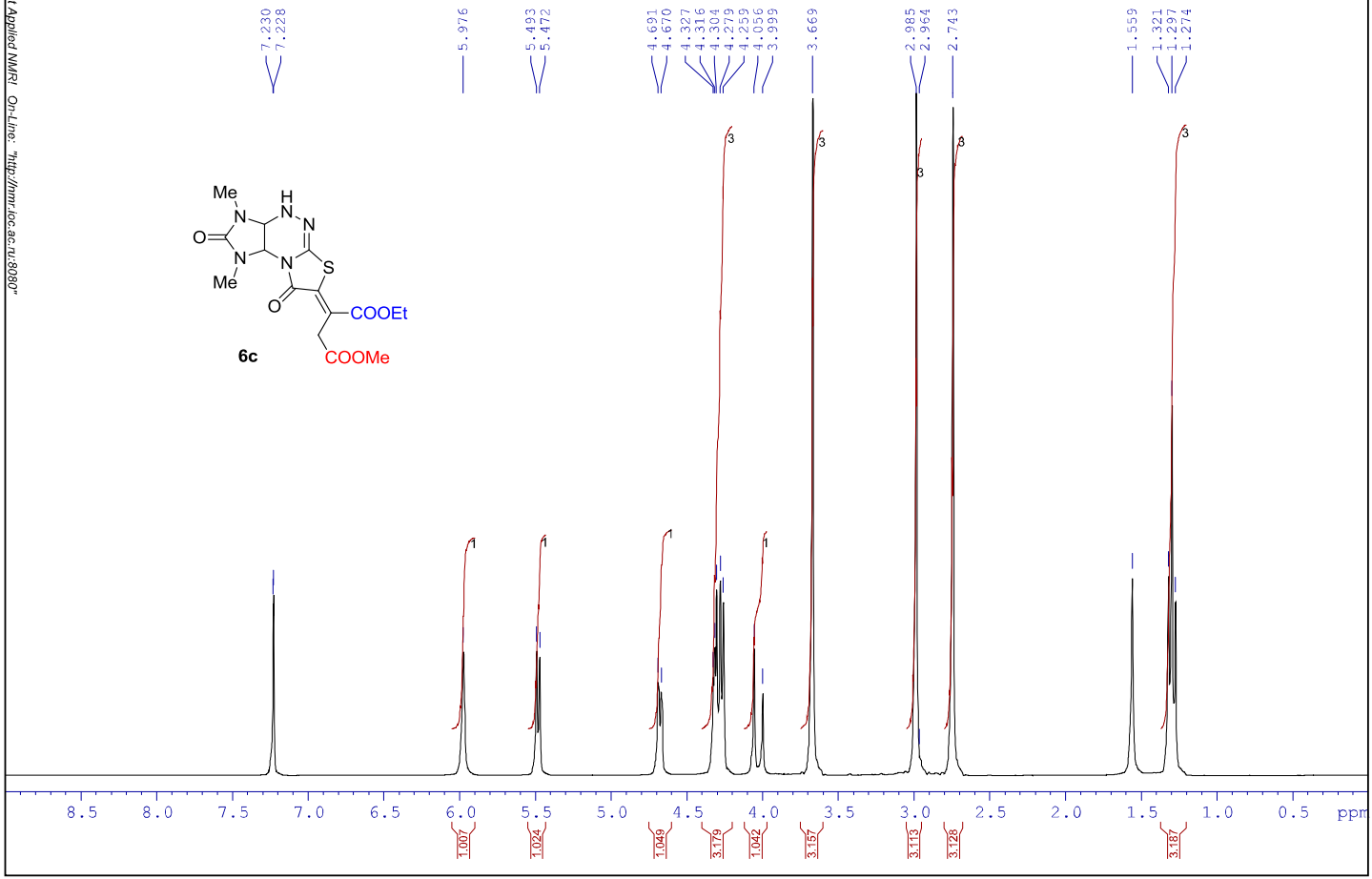

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=342 SR=-4.63 TE=300K 1 September 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line. "http://nmr.ioc.ac.ru:8080"
/KANI IA1559.4
170.660
166.660
164.215
158.846
139.091
137.605
124.097
77.526
77.103
76.679
66.197
65.958
62.539
61.268
32.760
31.727
27.600
14.239
14.134
Me
N
H
N
N
O
N
N
S
Me
O
COOEt
COOEt
6a
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

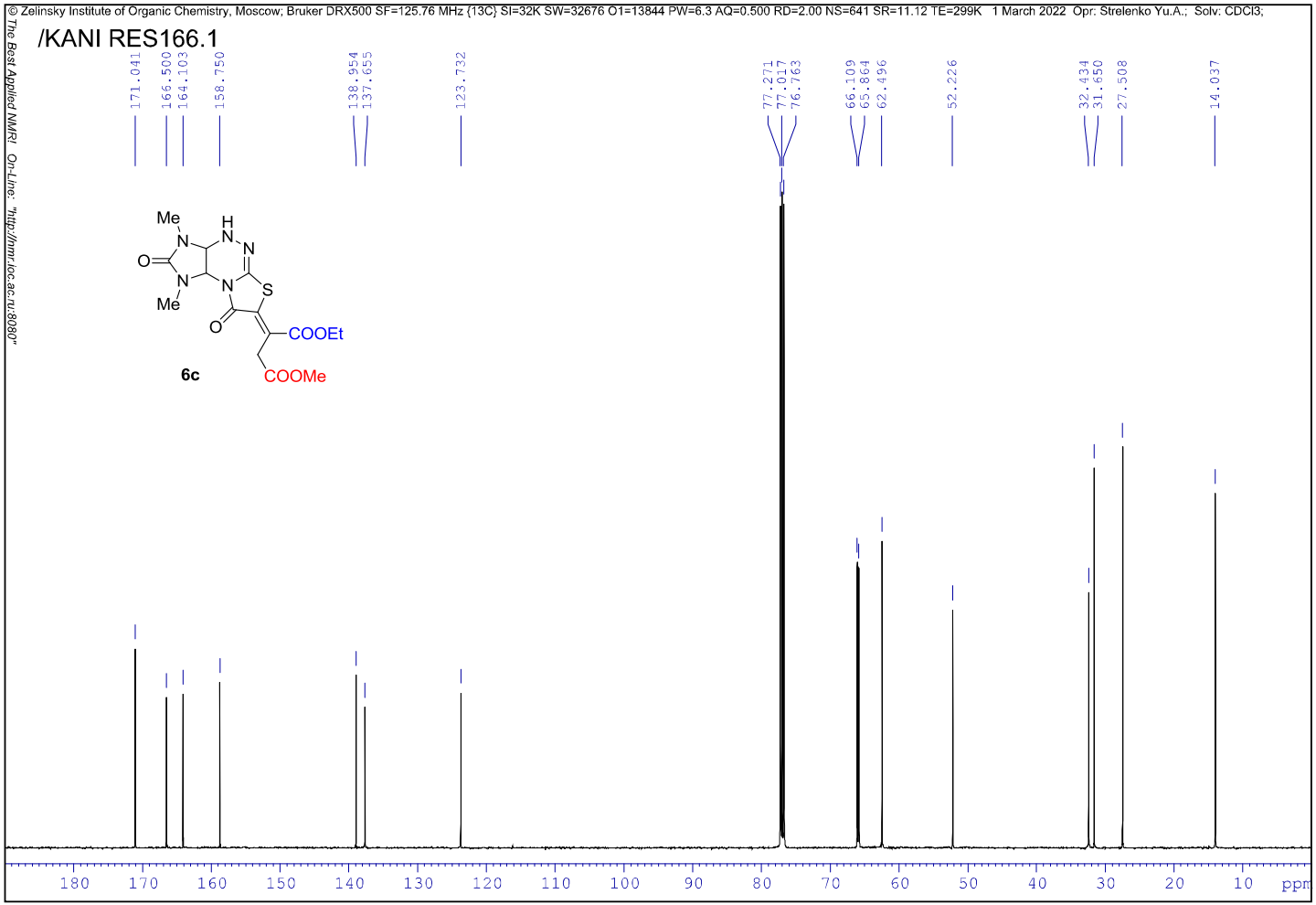
/KANI RES119.1




© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=274 SR=-2.74 TE=302K 19 November 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI RES119.1
170.672
167.144
164.084
158.818
138.842
138.159
123.484
77.527
77.102
76.679
66.186
65.932
61.302
53.221
32.694
31.690
27.550
14.192
Me
N
H
N
N
O
N
N
S
Me
O
COOMe
COOEt
6b
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

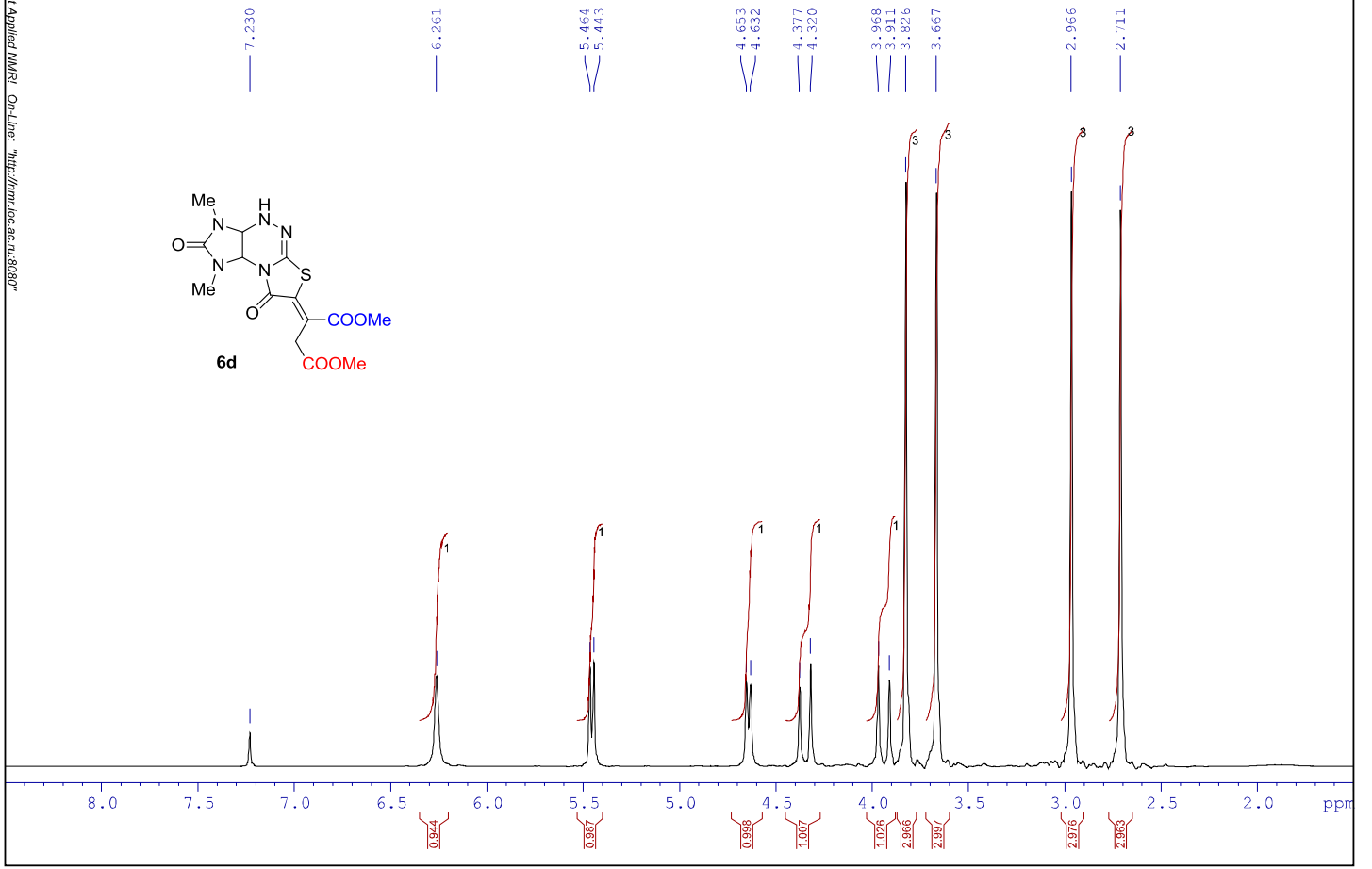
/KANI RES166.1

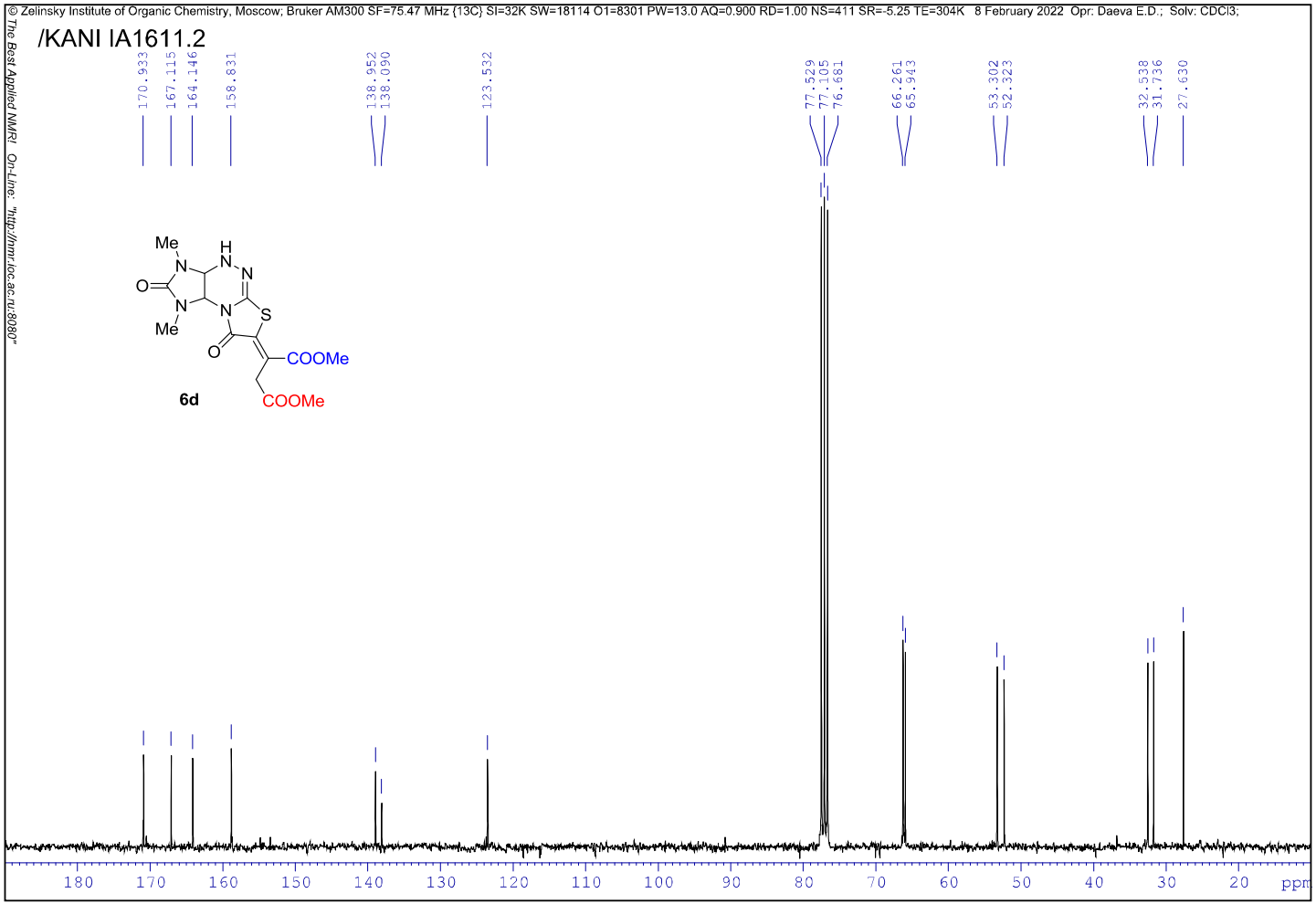


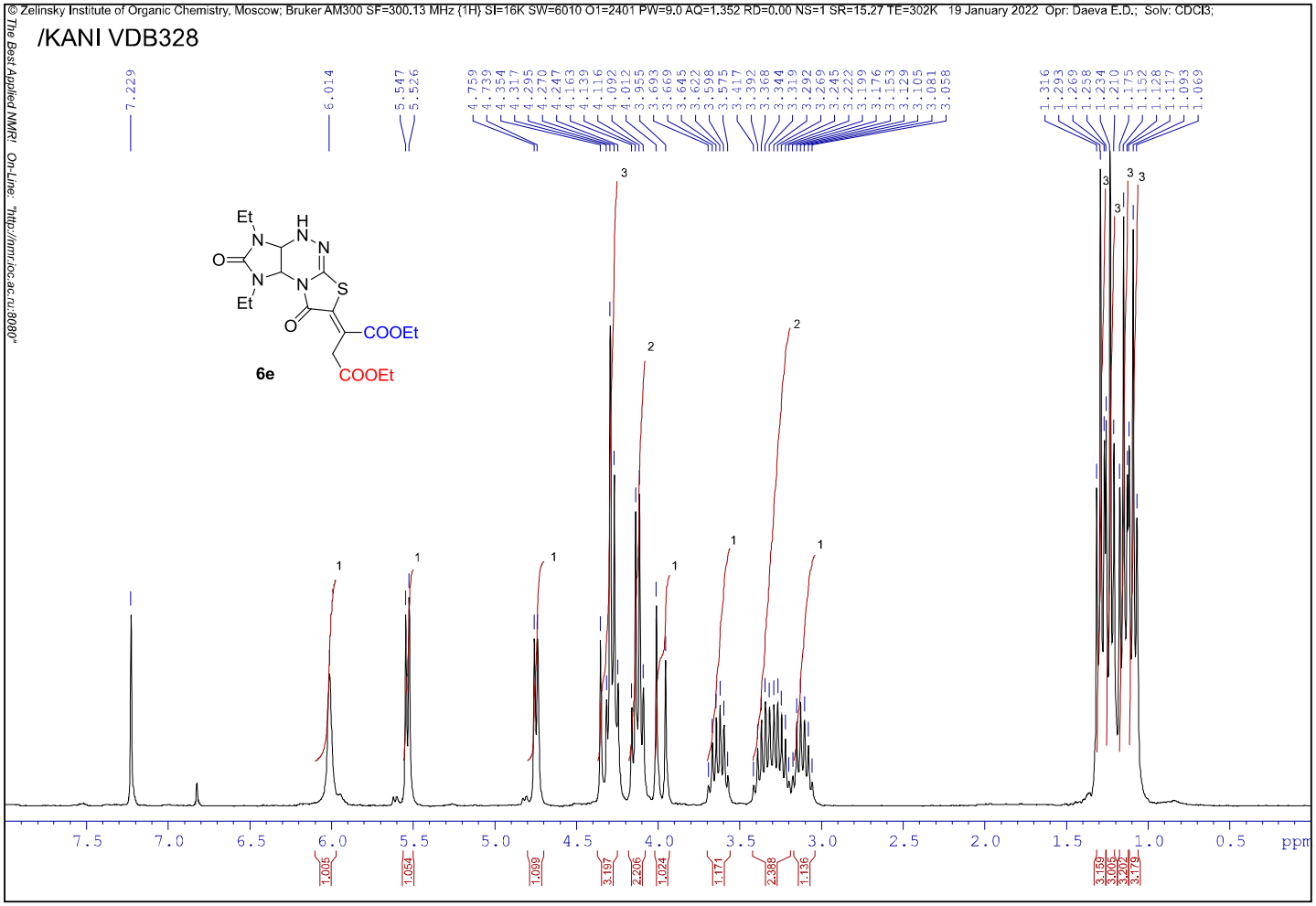

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker DRX500 SF=125.76 MHz {13C} SI=32K SW=32676 O1=13844 PW=6.3 AQ=0.500 RD=2.00 NS=641 SR=11.12 TE=299K 1 March 2022 Opr: Strelenko Yu.A.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI RES166.1
171.041
166.500
164.103
158.750
138.954
137.655
123.732
77.271
77.017
76.763
66.109
65.864
62.496
52.226
32.434
31.650
27.508
14.037
Me
N
H
N
N
O
N
S
Me
O
COOEt
COOMe
6c
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

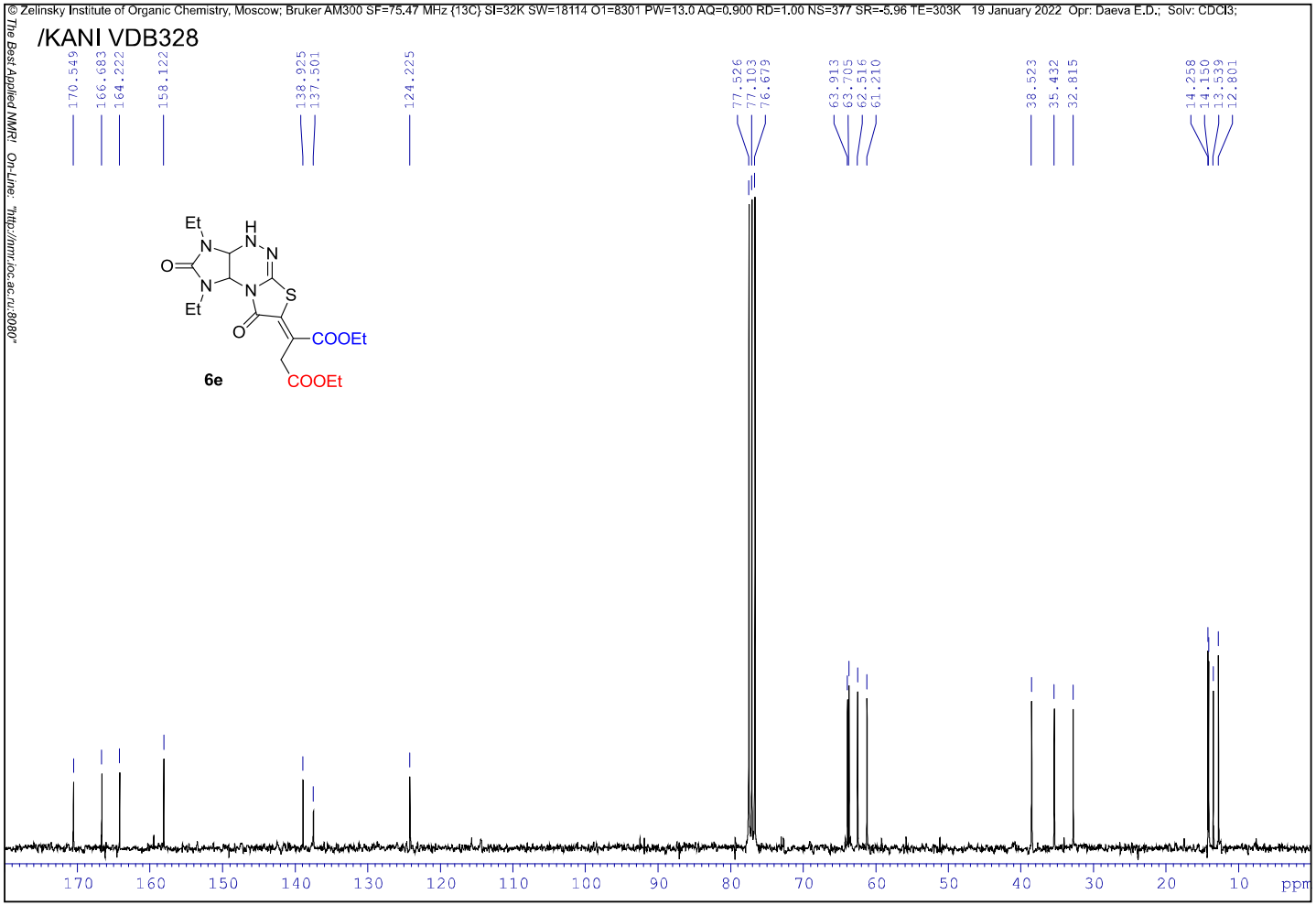
/KANI IA1611.2

6d

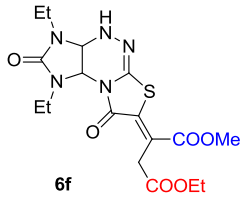



© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=411 SR=-5.25 TE=304K 8 February 2022 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1611.2
170.933
167.115
164.146
158.831
138.952
138.090
123.532
77.529
77.105
76.681
66.261
65.943
53.302
52.323
32.538
31.736
27.630
Me
N
H
N
O
N
N
S
Me
O
COOMe
COOMe
6d
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=6010 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=1 SR=15.27 TE=302K 19 January 2022 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR!. On-Line. "http://nmr.ioc.ac.ru:8080"
/KANI VDB328
6e
COOEt
COOEt
ppm

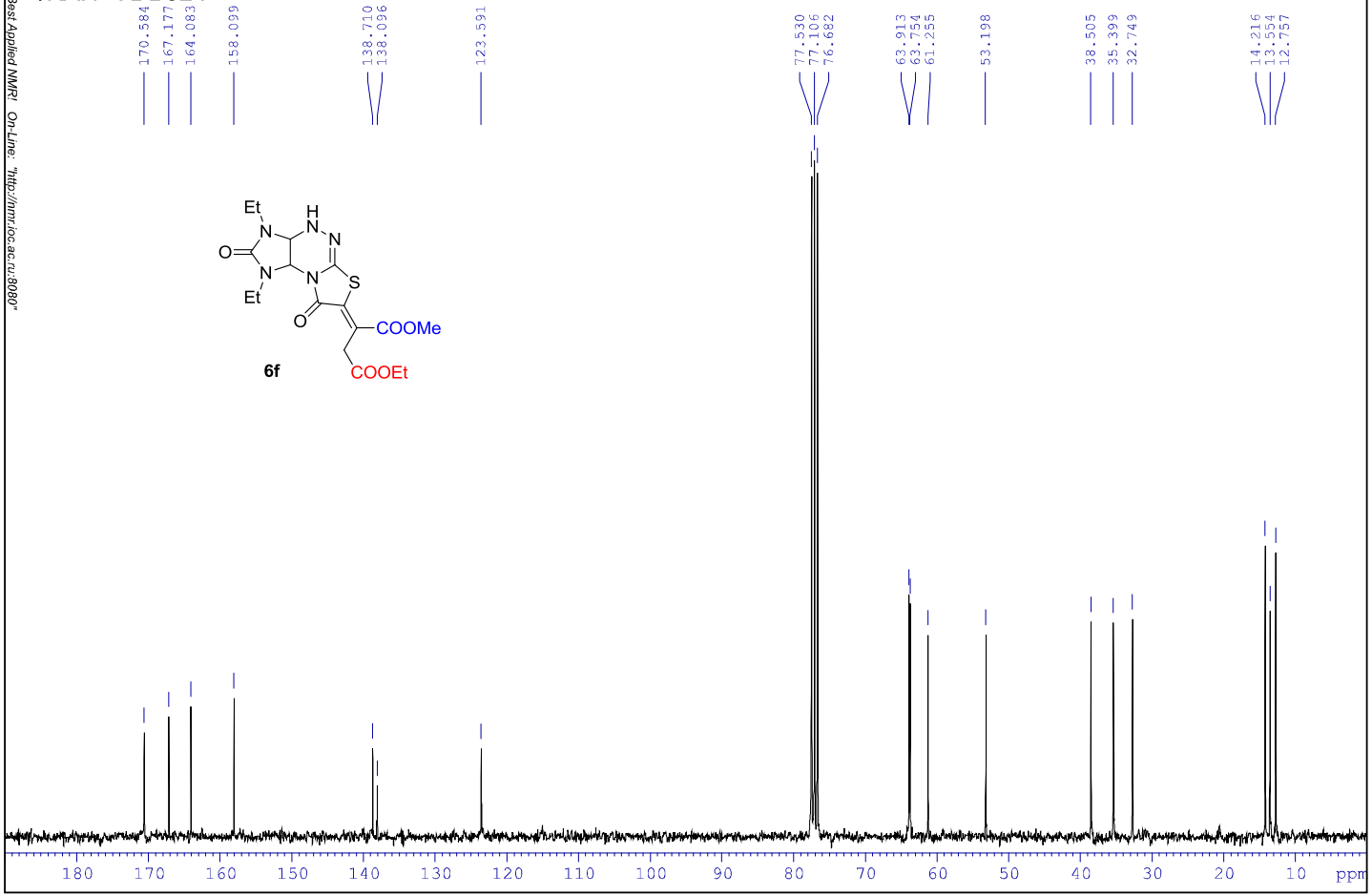

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=377 SR=-5.96 TE=303K 19 January 2022 Opr: Daeva E.D.; Solv: CDCl3;
/KANI VDB328
The Best Applied NMR! On-Line. "http://nmr.joc.ac.ru:8080"
170.549
166.683
164.222
158.122
138.925
137.501
124.225
77.526
77.103
76.679
63.913
63.705
62.516
61.210
38.523
35.432
32.815
14.258
14.150
13.539
12.801
Et
N
H
N
N
O
N
N
S
Et
O
COOEt
COOEt
6e
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

/KANI VDB324

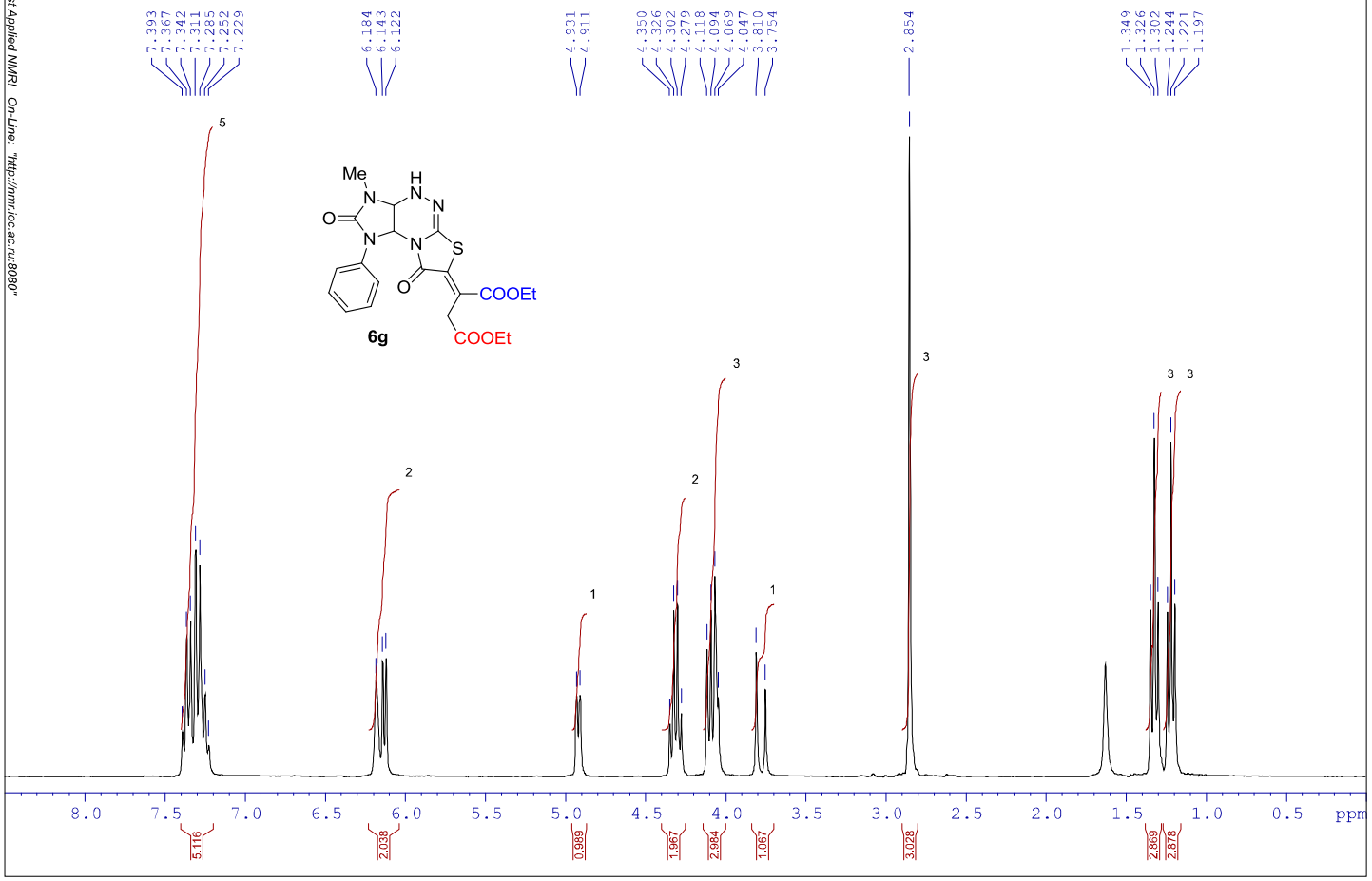


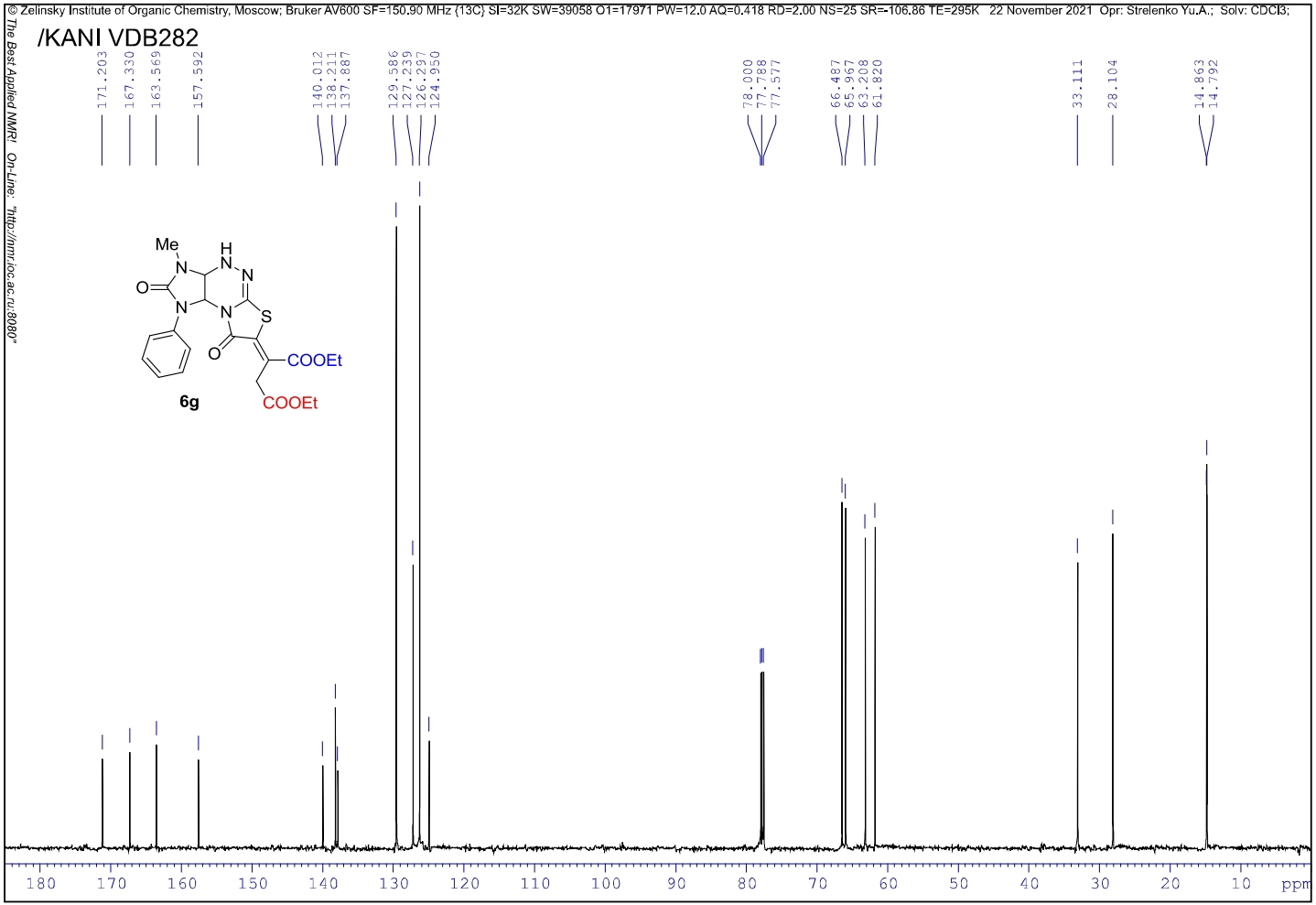
6f

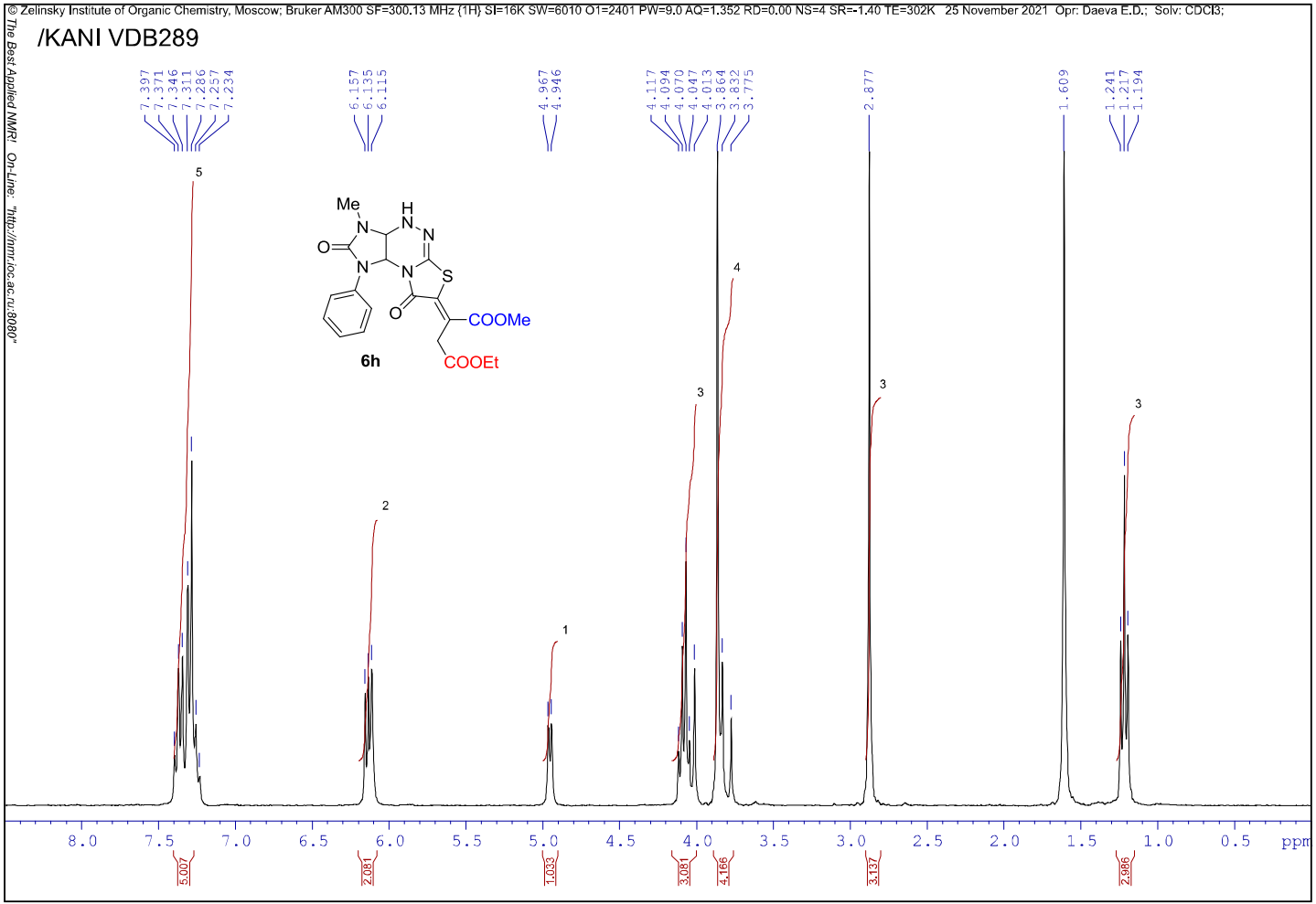
/KANI VDB324

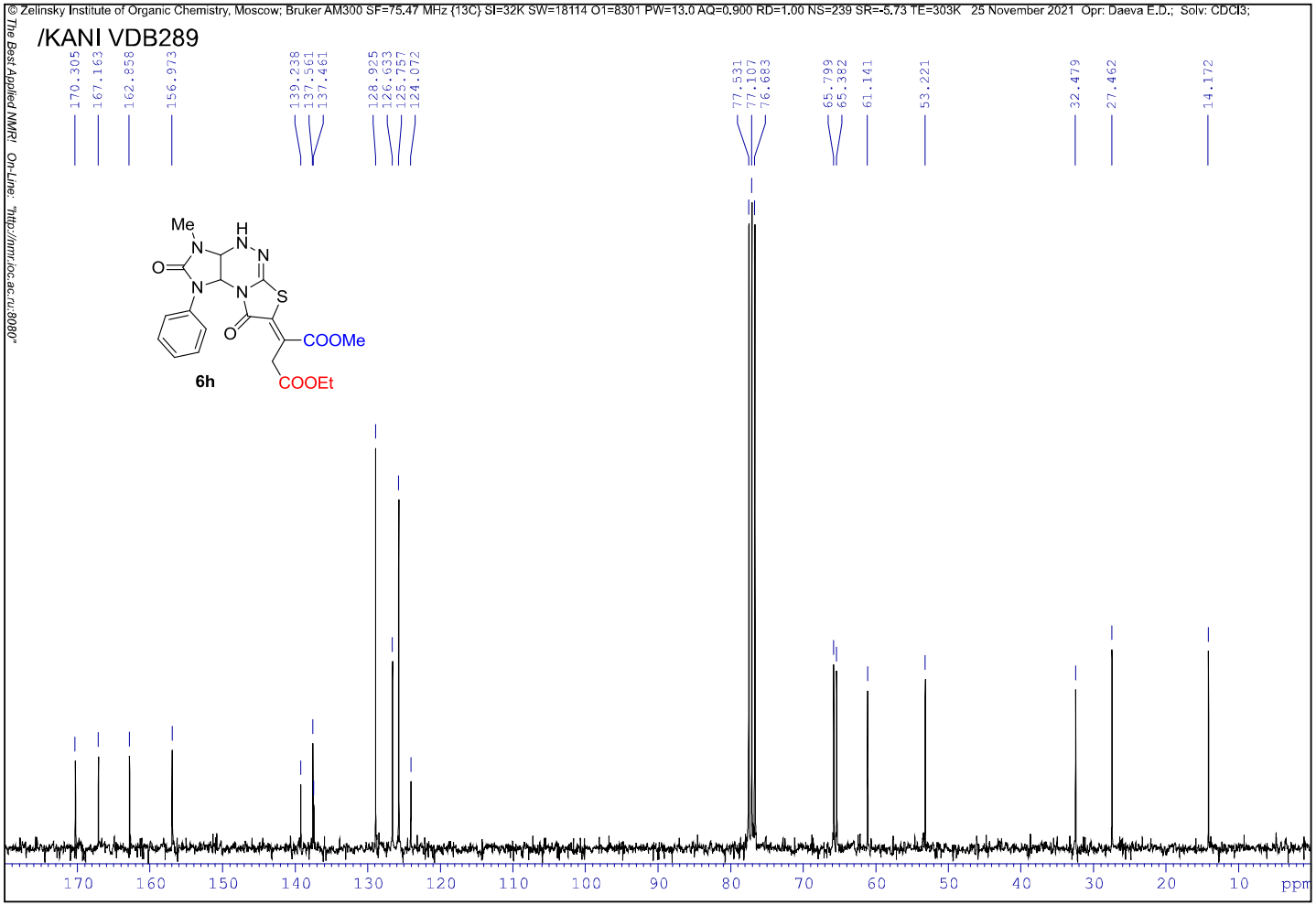


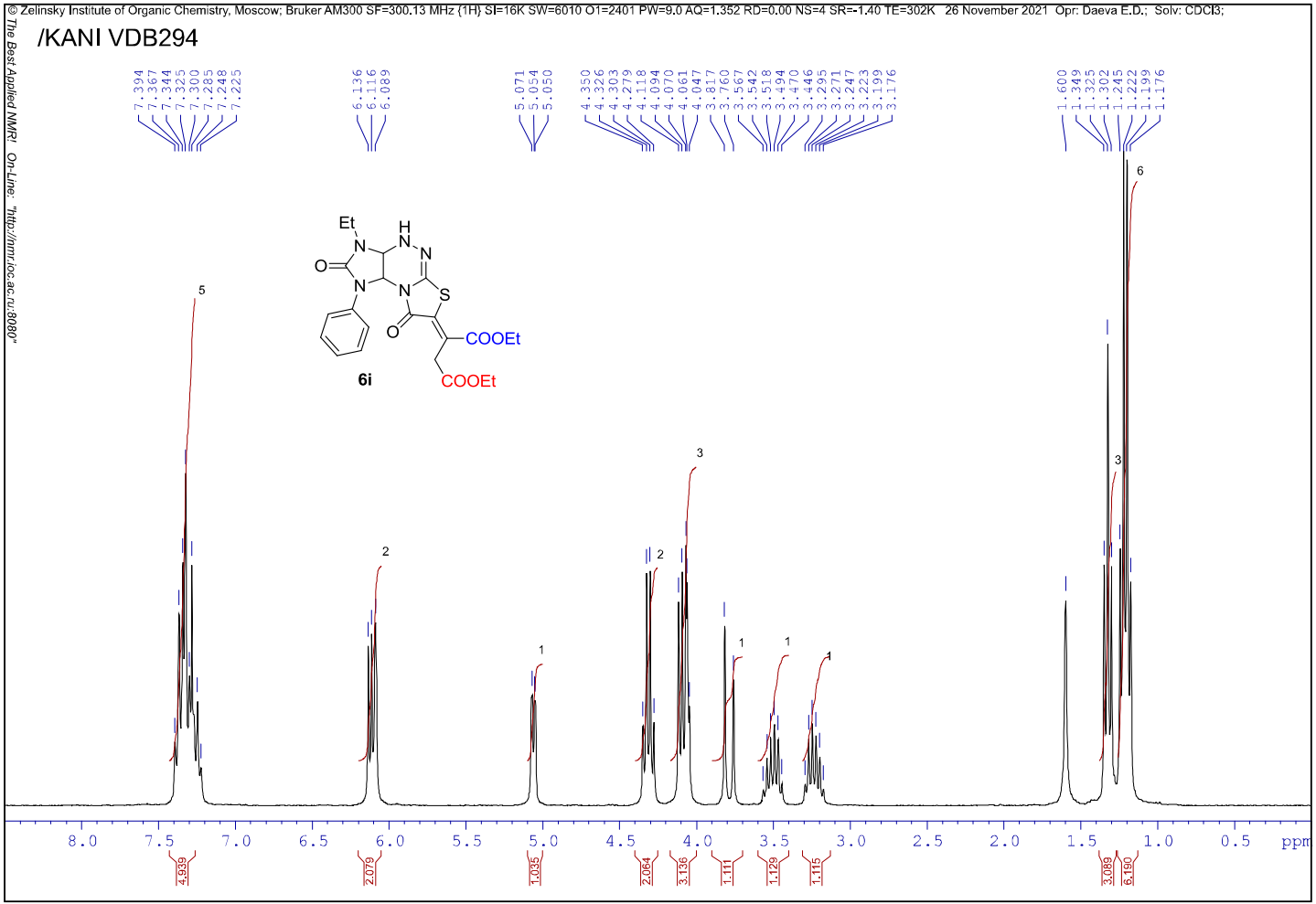
/KANI VDB282

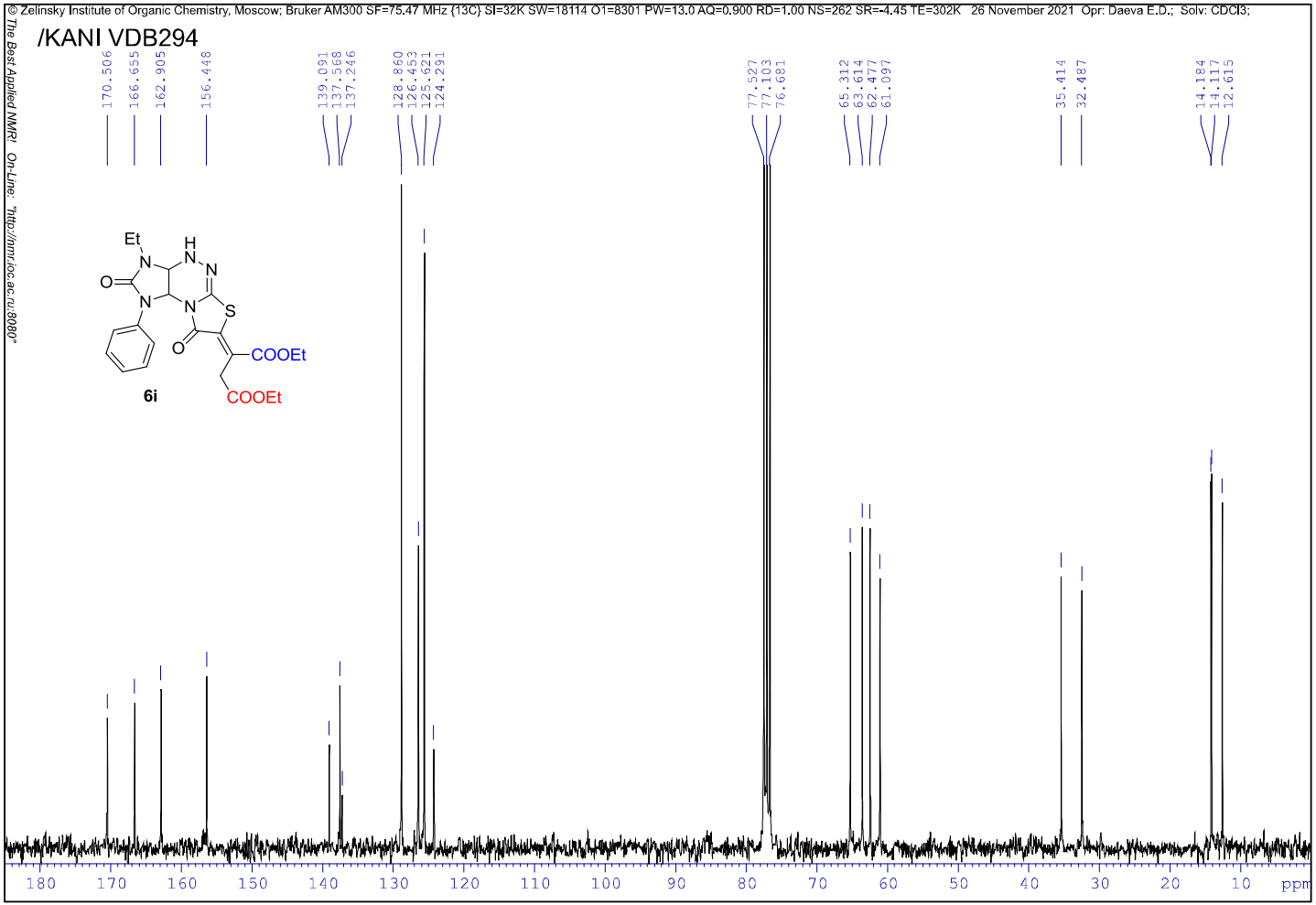


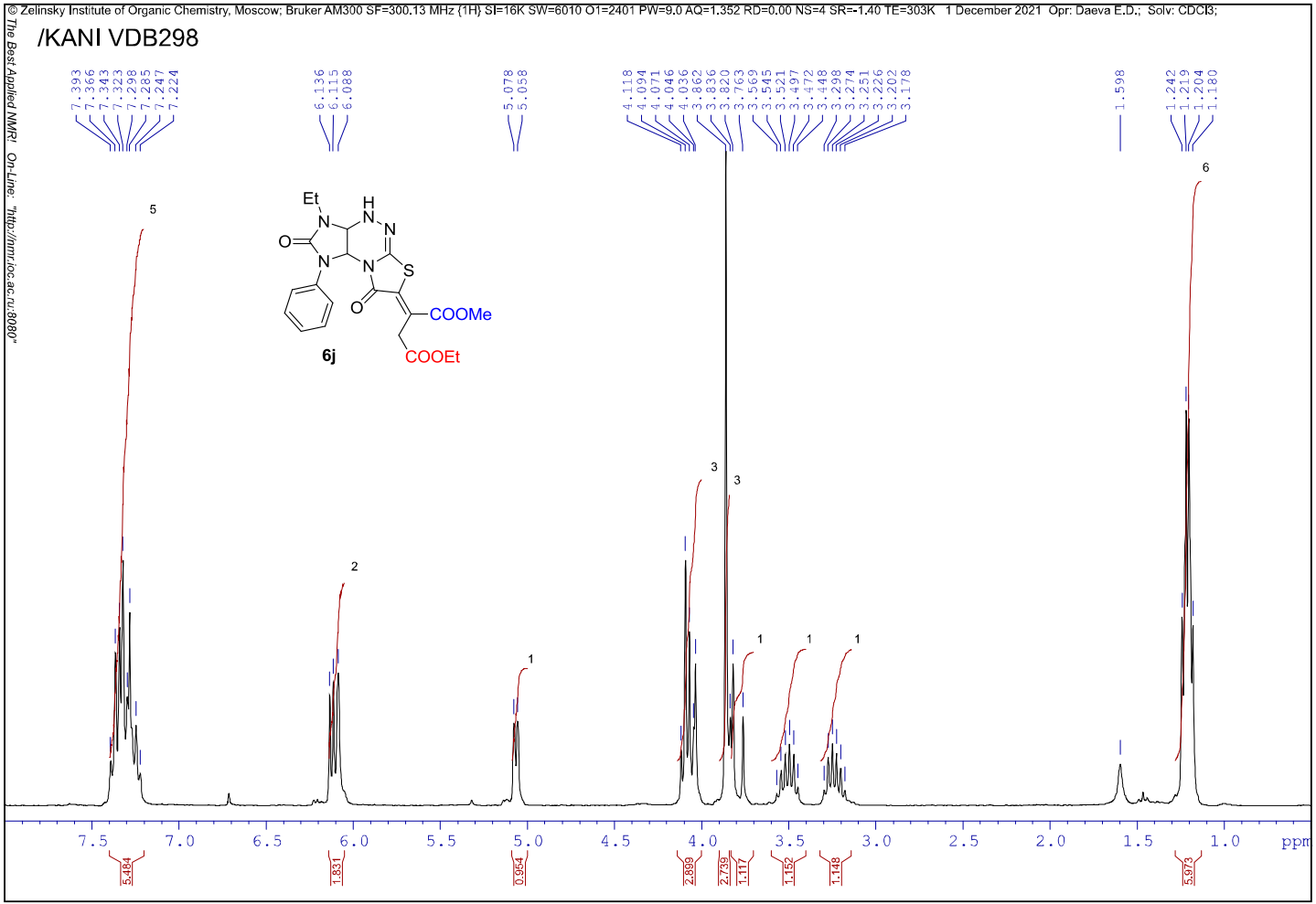

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AV600 SF=150.90 MHz {13C} SI=32K SW=39058 O1=17971 PW=12.0 AQ=0.418 RD=2.00 NS=25 SR=-106.86 TE=295K 22 November 2021 Opr: Strelenko Yu.A.; Solv: CDCl3;
The Best Applied NMR! On-Line, "http://nmr.ioc.ac.ru:8080"
/KANI VDB282
171.203
167.330
163.569
157.592
140.012
138.211
137.887
129.586
127.239
126.297
124.950
78.000
77.788
77.577
66.487
65.967
63.208
61.820
33.111
28.104
14.863
14.792
Me
N
H
N
N
O
N
N
S
O
COOEt
COOEt
6g
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

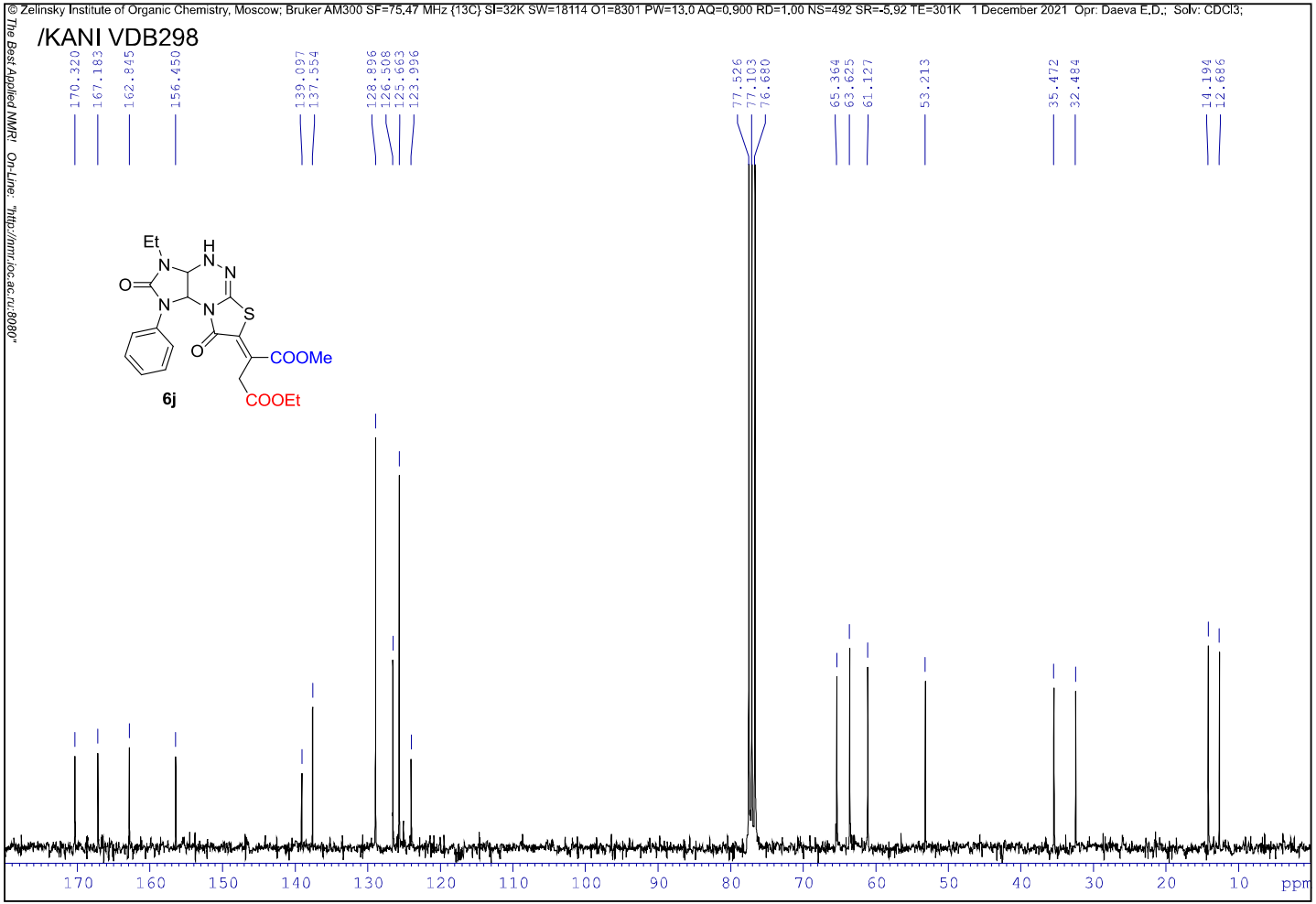

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=4 SR=-1.40 TE=302K 25 November 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line. "http://nmr.ioc.ac.ru:8080"
/KANI VDB289
7.397
7.371
7.346
7.311
7.286
7.257
7.234
6.157
6.135
6.115
4.967
4.946
4.117
4.094
4.070
4.047
4.013
3.864
3.832
3.775
2.877
1.609
1.241
1.217
1.194
Me
H
N
O
S
COOMe
COOEt
6h
5
2
1
3
4
3
3
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
5.007
2.081
1.033
3.081
4.166
3.137
2.986

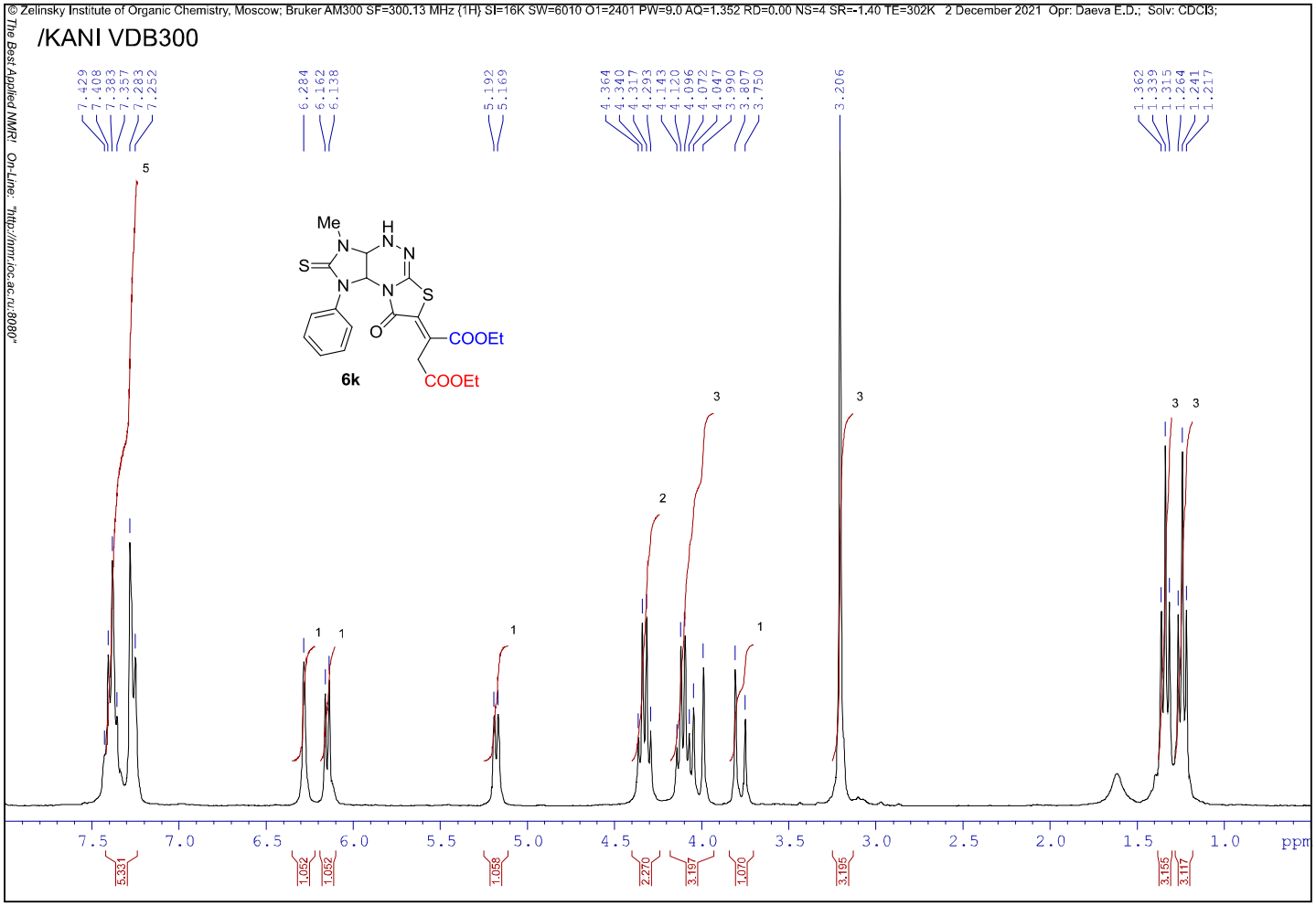

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=239 SR=-5.73 TE=303K 25 November 2021 Opr: Daeva E.D.; Solv: CDCl3;
/KANI VDB289
The Best Applied NMR! On-Line. "http://nmr.ioc.ac.ru:8080"
170.305
167.163
162.858
156.973
139.238
137.561
137.461
128.925
126.633
125.757
124.072
77.531
77.107
76.683
65.799
65.382
61.141
53.221
32.479
27.462
14.172
Me
H
N
O
S
COOMe
COOEt
6h
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

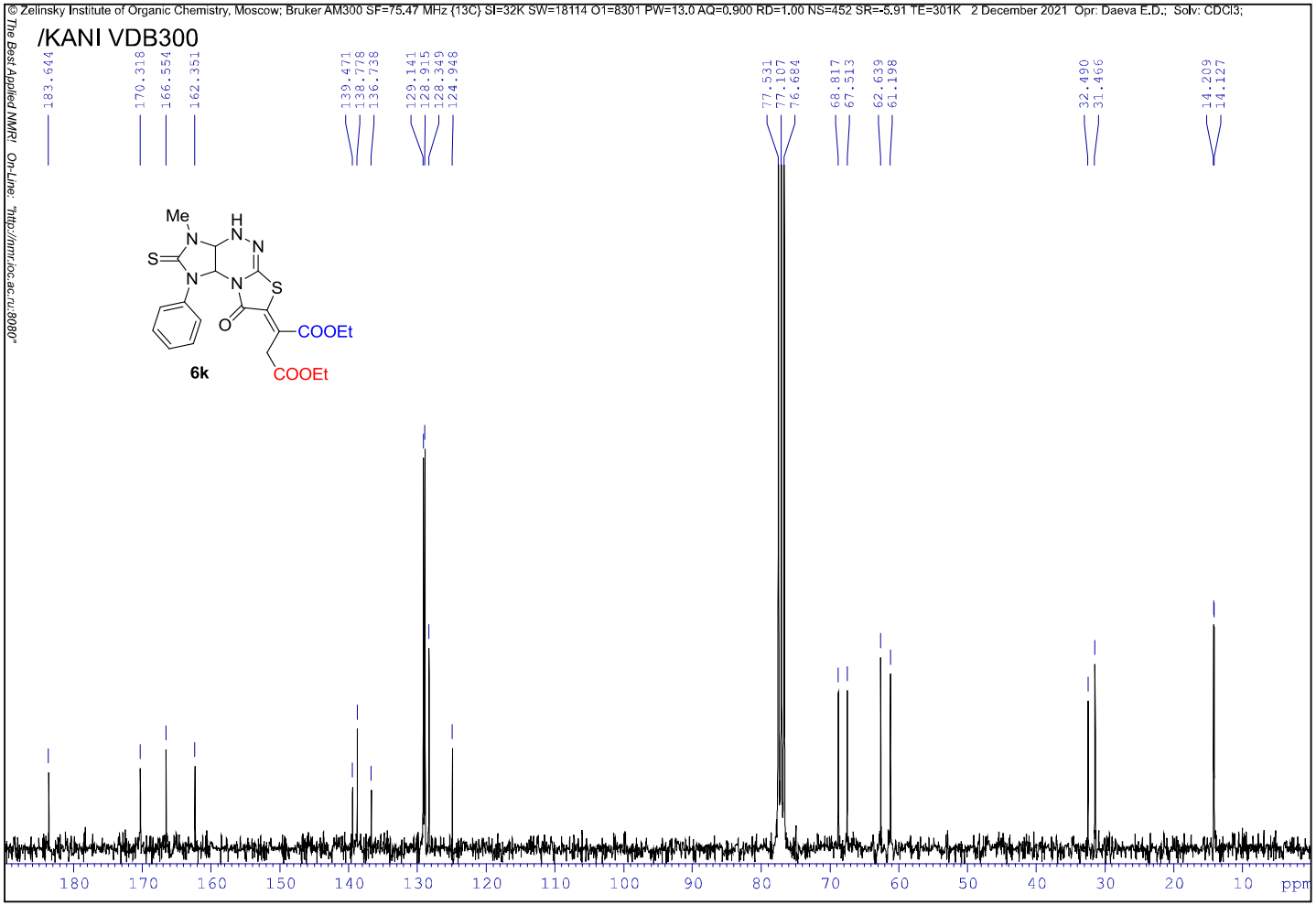

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=4 SR=-1.40 TE=302K 26 November 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR!. On-Line. "http://nmr.ioc.ac.ru:8080"
/KANI VDB294
7.394
7.367
7.344
7.325
7.300
7.285
7.248
7.225
6.136
6.116
6.089
5.071
5.054
5.050
4.350
4.326
4.303
4.279
4.118
4.094
4.070
4.061
4.047
3.817
3.760
3.567
3.542
3.518
3.494
3.470
3.446
3.295
3.271
3.247
3.223
3.199
3.176
1.600
1.349
1.325
1.302
1.245
1.222
1.199
1.176
Et
H
N
O
S
COOEt
COOEt
6i
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
4.939
2.079
1.035
2.064
3.136
1.111
1.129
1.115
3.089
6.190

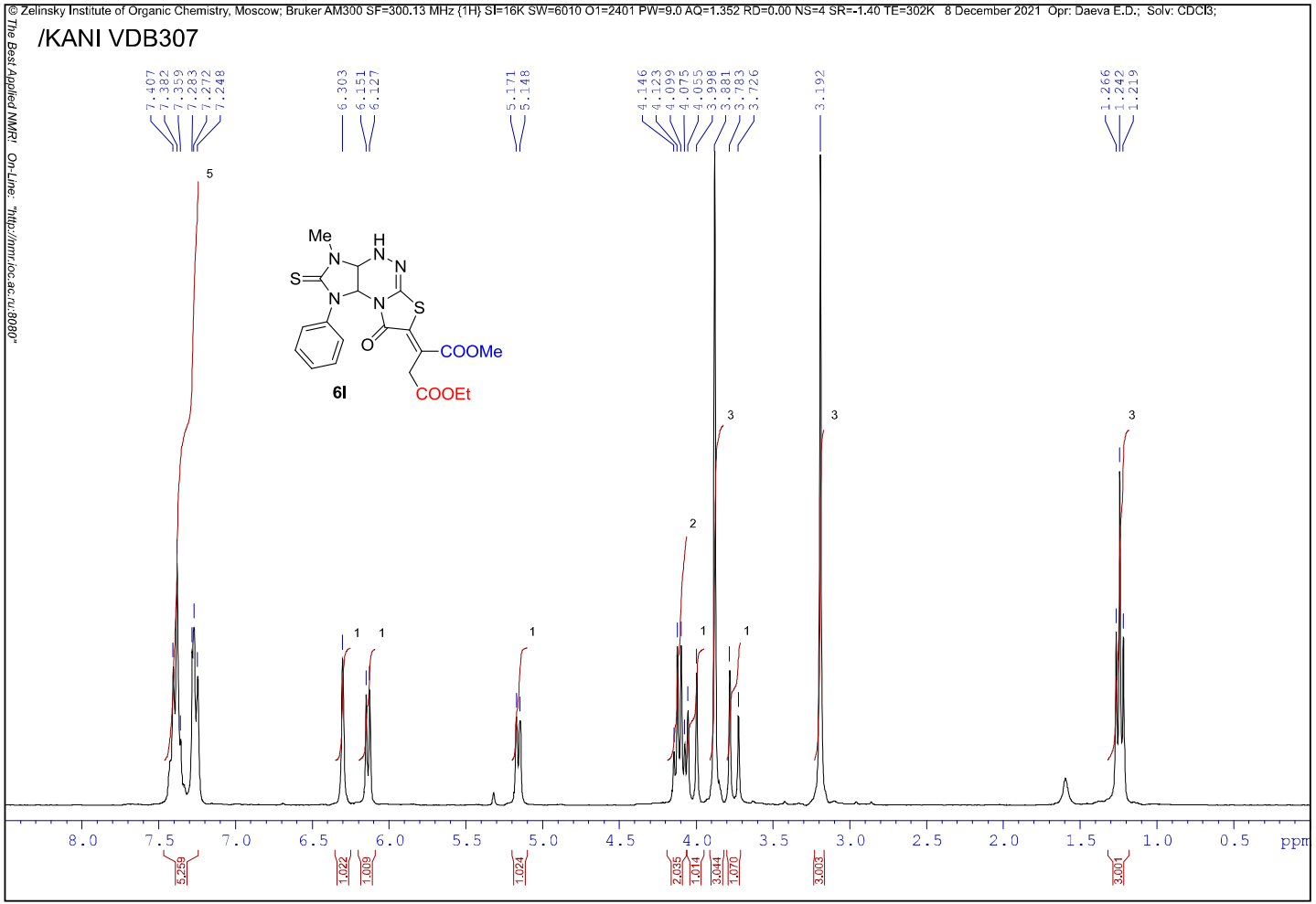

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=262 SR=-4.45 TE=302K 26 November 2021 Opr: Daeva E.D.; Solv: CDCl3;
/KANI VDB294
The Best Applied NMR!. On-Line. "http://nmr.joc.ac.ru:8080"
170.506
166.655
162.905
156.448
139.091
137.568
137.246
128.860
126.453
125.621
124.291
77.527
77.103
76.681
65.312
63.614
62.477
61.097
35.414
32.487
14.184
14.117
12.615
Et
H
N
N
O
N
N
S
O
COOEt
COOEt
6i
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

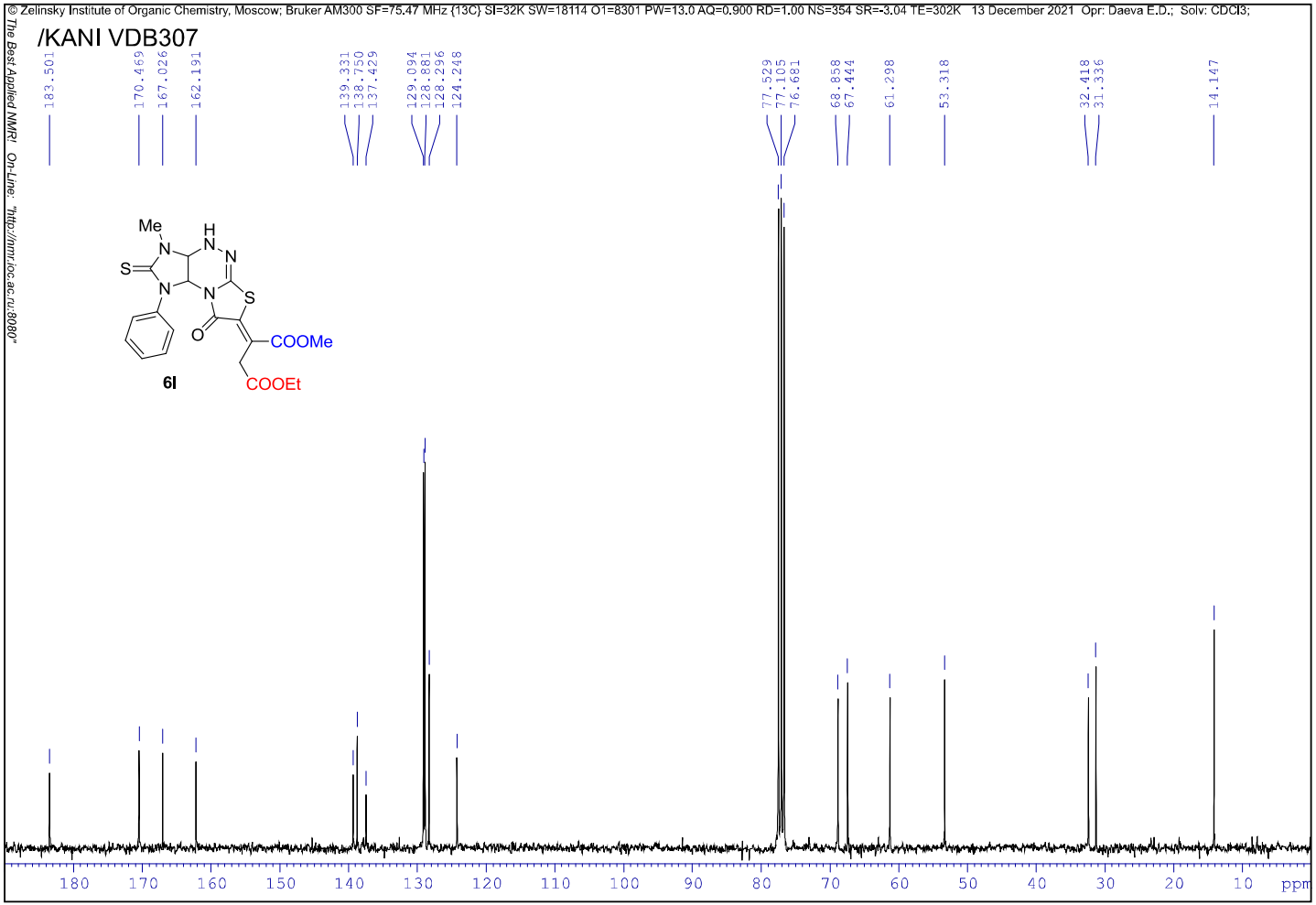

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=4 SR=-1.40 TE=303K 1 December 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line. "http://nmr.ioc.ac.ru:8080"
/KANI VDB298
7.393 7.366 7.343 7.323 7.298 7.285 7.247 7.224
6.136 6.115 6.088
5.078 5.058
4.118 4.094 4.071 4.046 4.036 3.862 3.836 3.820 3.763 3.569 3.545 3.521 3.497 3.472 3.448 3.298 3.274 3.251 3.226 3.202 3.178
1.598
1.242 1.219 1.204 1.180
Et
H
N
N
O
N
N
S
O
COOMe
COOEt
6j
5
2
1
3
3
1
1
1
6
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
5.484
1.831
0.964
2.869
2.739
1.117
1.152
1.148
5.973


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=492 SR=-5.92 TE=301K 1 December 2021 Opr: Daeva E.D.; Solv: CDCl3;
/KANI VDB298
The Best Applied NMR! On-Line. "http://nmr.ioc.ac.ru:8080"
170.320
167.183
162.845
156.450
139.097
137.554
128.896
126.508
125.663
123.996
77.526
77.103
76.680
65.364
63.625
61.127
53.213
35.472
32.484
14.194
12.686
Et
H
N
N
O
N
N
S
O
COOMe
COOEt
6j
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

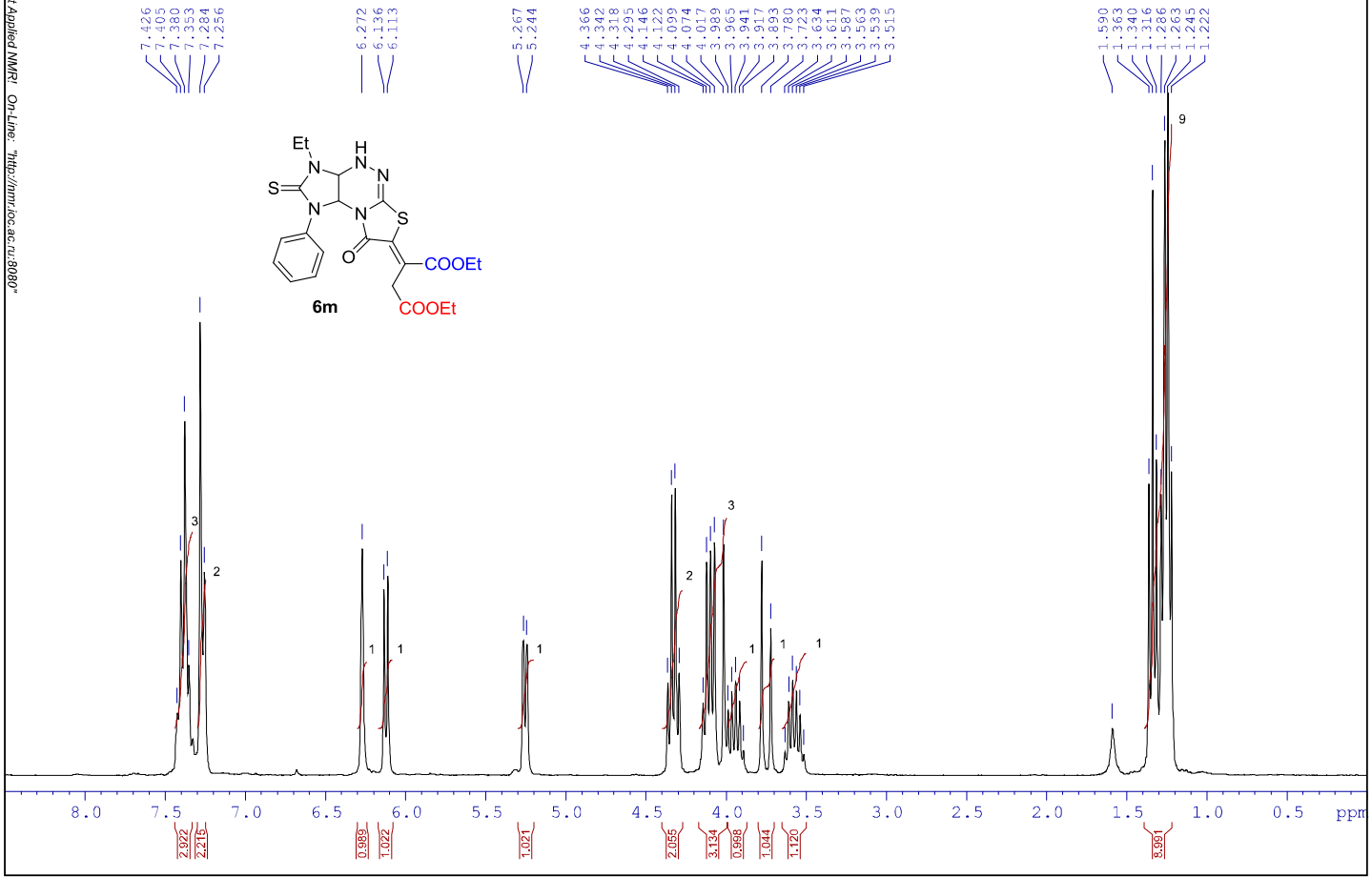

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=4 SR=-1.40 TE=302K 2 December 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line. "http://nmr.joc.ac.ru:8080"
/KANI VDB300
7.429
7.408
7.383
7.357
7.283
7.252
6.284
6.162
6.138
5.192
5.169
4.364
4.340
4.317
4.293
4.143
4.120
4.096
4.072
4.047
3.990
3.807
3.750
3.206
1.362
1.339
1.315
1.264
1.241
1.217
Me
H
N
S
O
COOEt
COOEt
6k
ppm
5.331
1.052
1.052
1.058
2.270
3.197
1.070
3.195
3.155
3.117

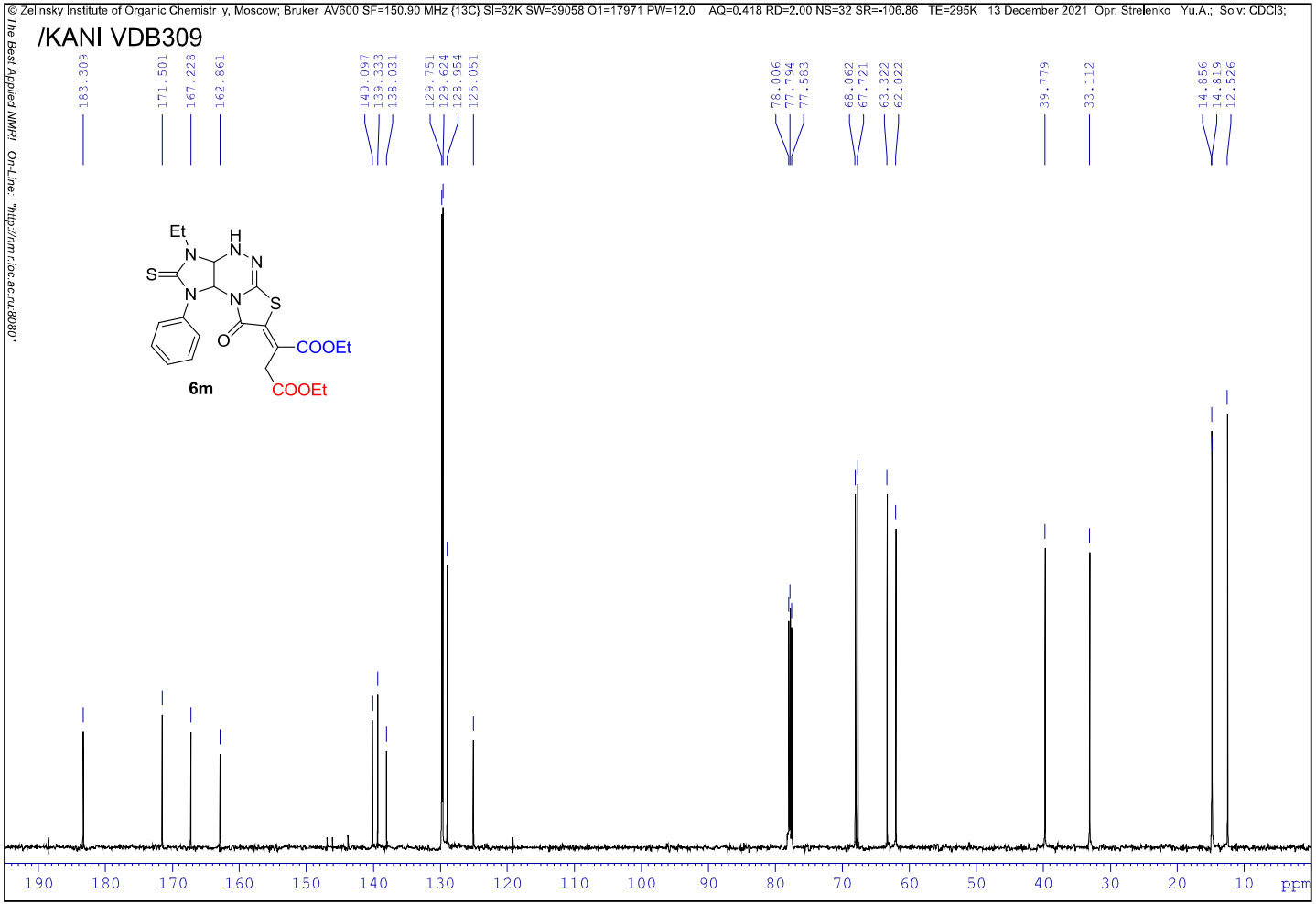

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=452 SR=-5.91 TE=301K 2 December 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line, "http://nmr.ioc.ac.ru:8080"
/KANI VDB300
183.644
170.318
166.554
162.351
139.471
138.778
136.738
129.141
128.915
128.349
124.948
77.531
77.107
76.684
68.817
67.513
62.639
61.198
32.490
31.466
14.209
14.127
Me
H
N
N
S
N
N
S
O
COOEt
COOEt
6k
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

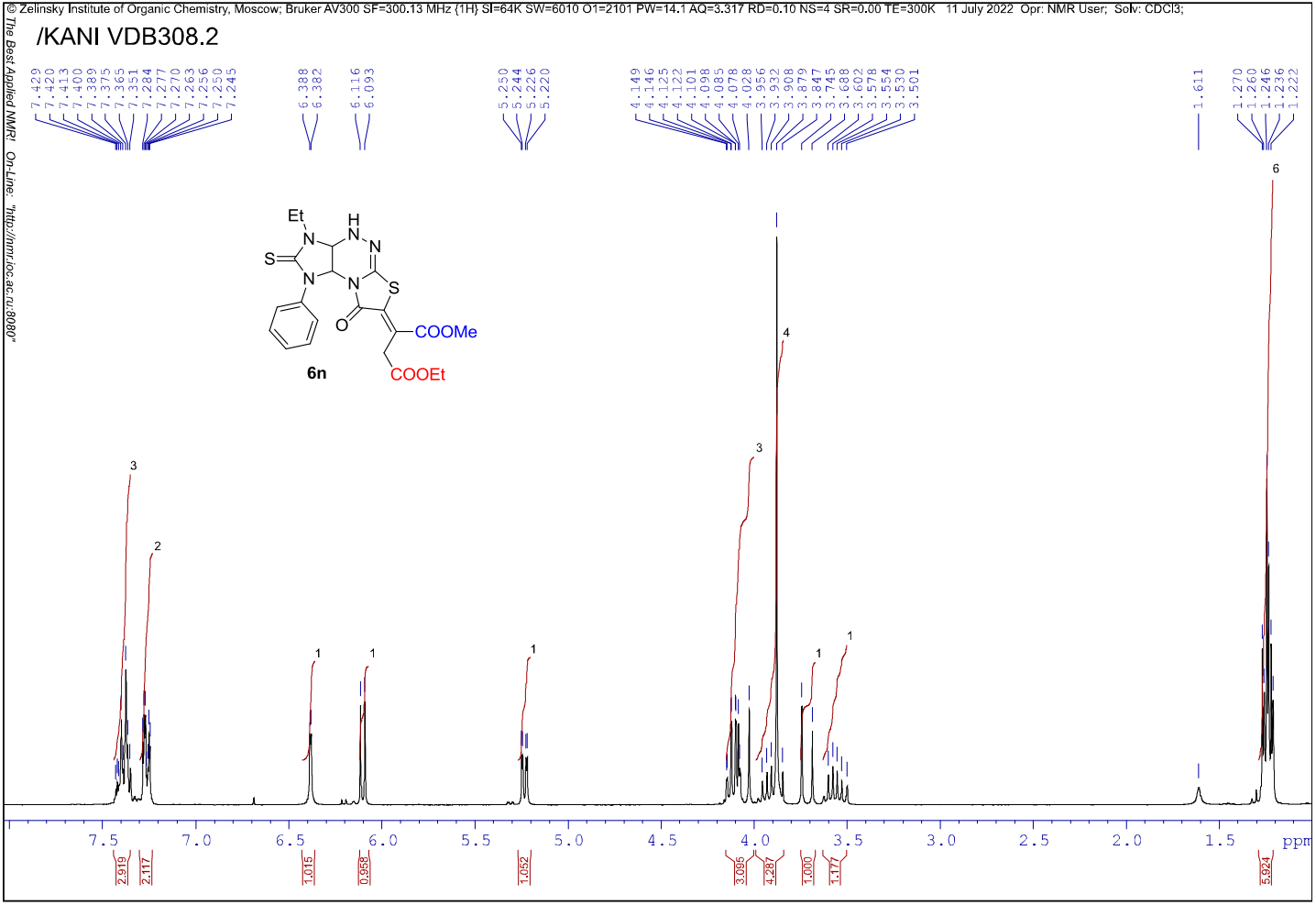

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=4 SR=-1.40 TE=302K 8 December 2021 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI VDB307
7.407
7.382
7.359
7.283
7.272
7.248
6.303
6.151
6.127
5.171
5.148
4.146
4.123
4.099
4.075
4.055
3.998
3.881
3.783
3.726
3.192
1.266
1.242
1.219
Me
H
N
S
O
COOMe
COOEt
6l
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
5.259
1.022
1.009
1.024
2.035
1.014
3.044
1.070
3.003
3.001

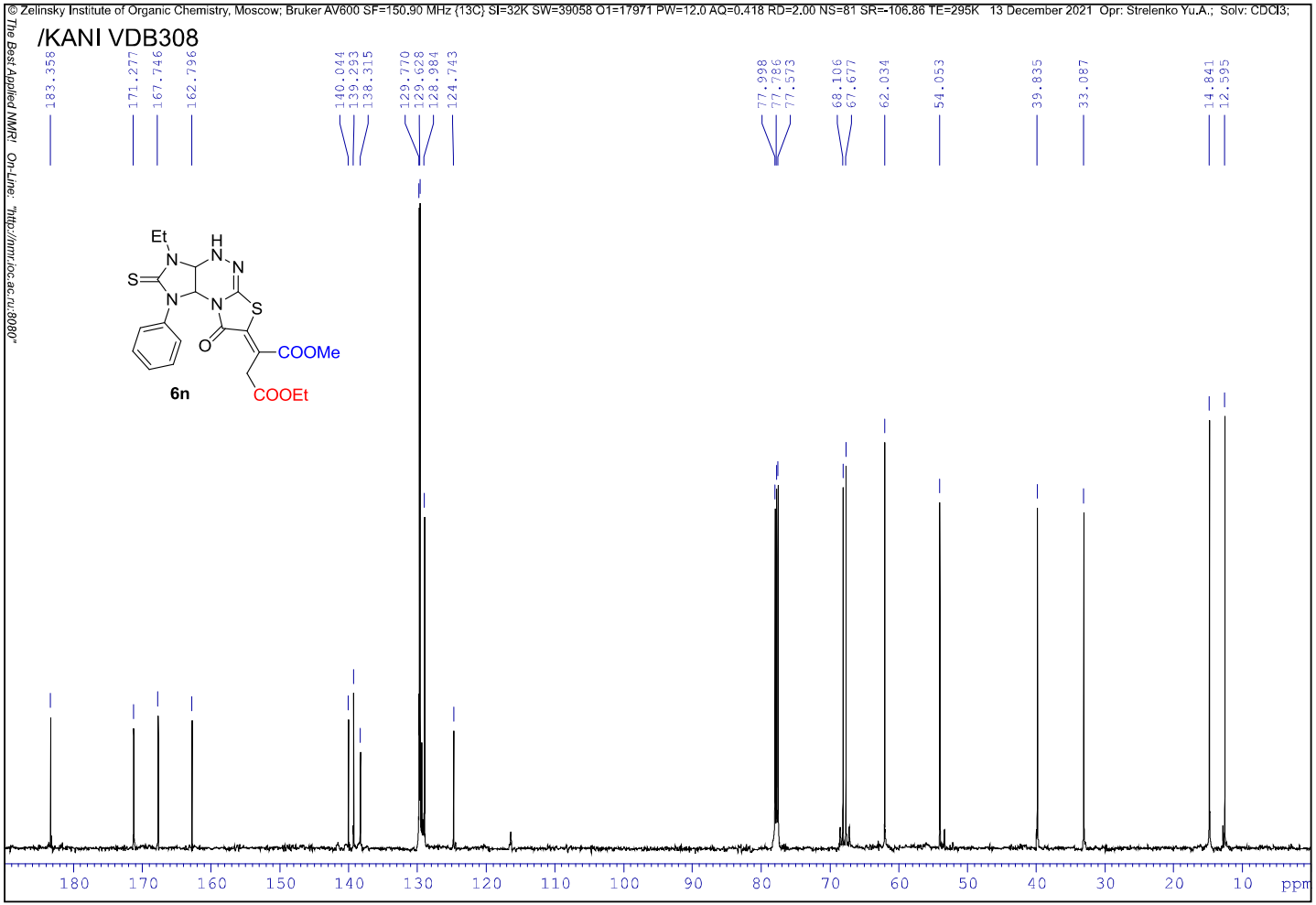

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=354 SR=-3.04 TE=302K 13 December 2021 Opr: Daeva E.D.; Solv: CDCl3;
/KANI VDB307
The Best Applied NMR! On-Line, "http://nmr.ioc.ac.ru:8080"
183.501
170.469
167.026
162.191
139.331
138.750
137.429
129.094
128.881
128.296
124.248
77.529
77.105
76.681
68.858
67.444
61.298
53.318
32.418
31.336
14.147
Me
H
N
S
O
COOMe
COOEt
6l
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

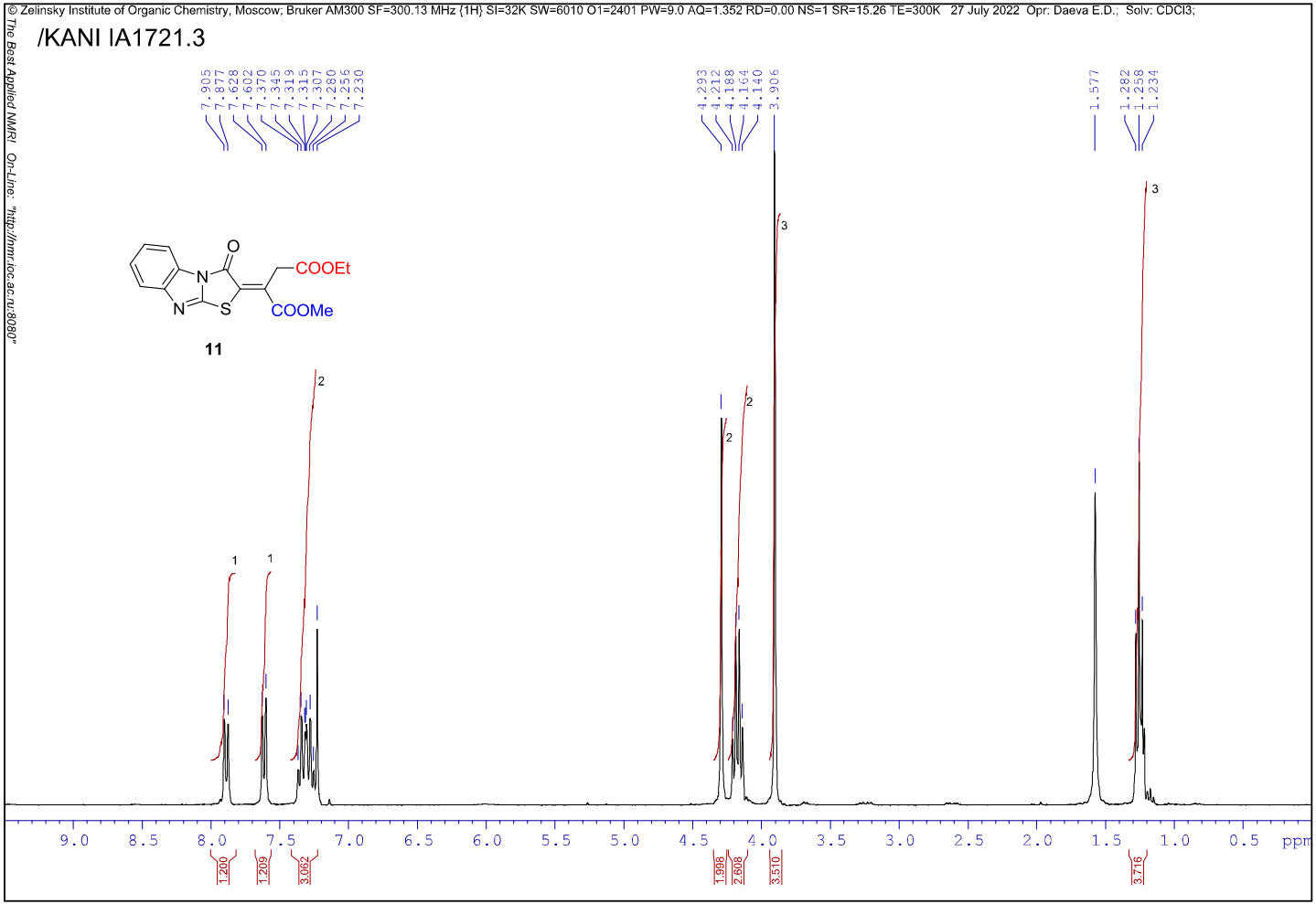
/KANI VDB309

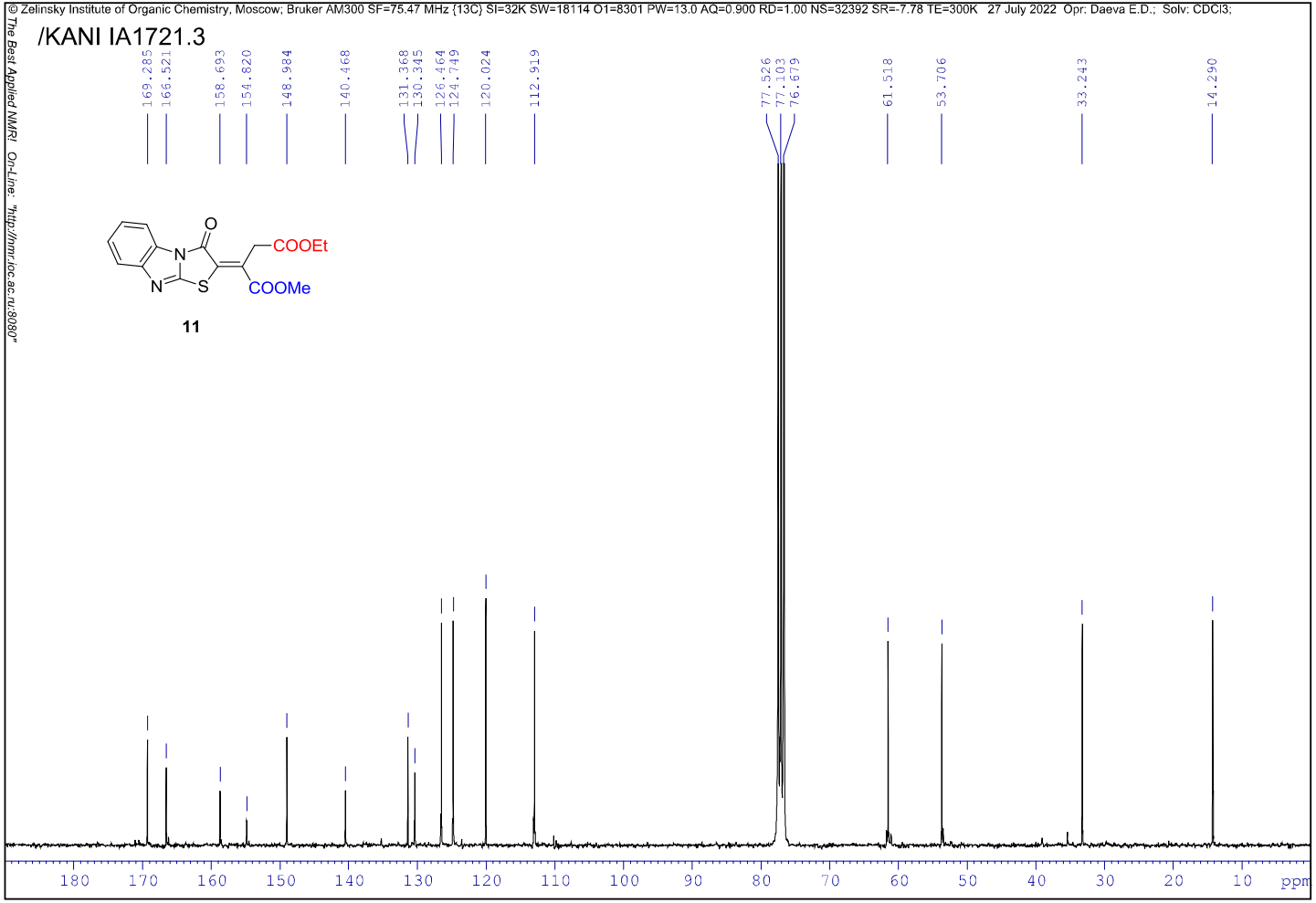


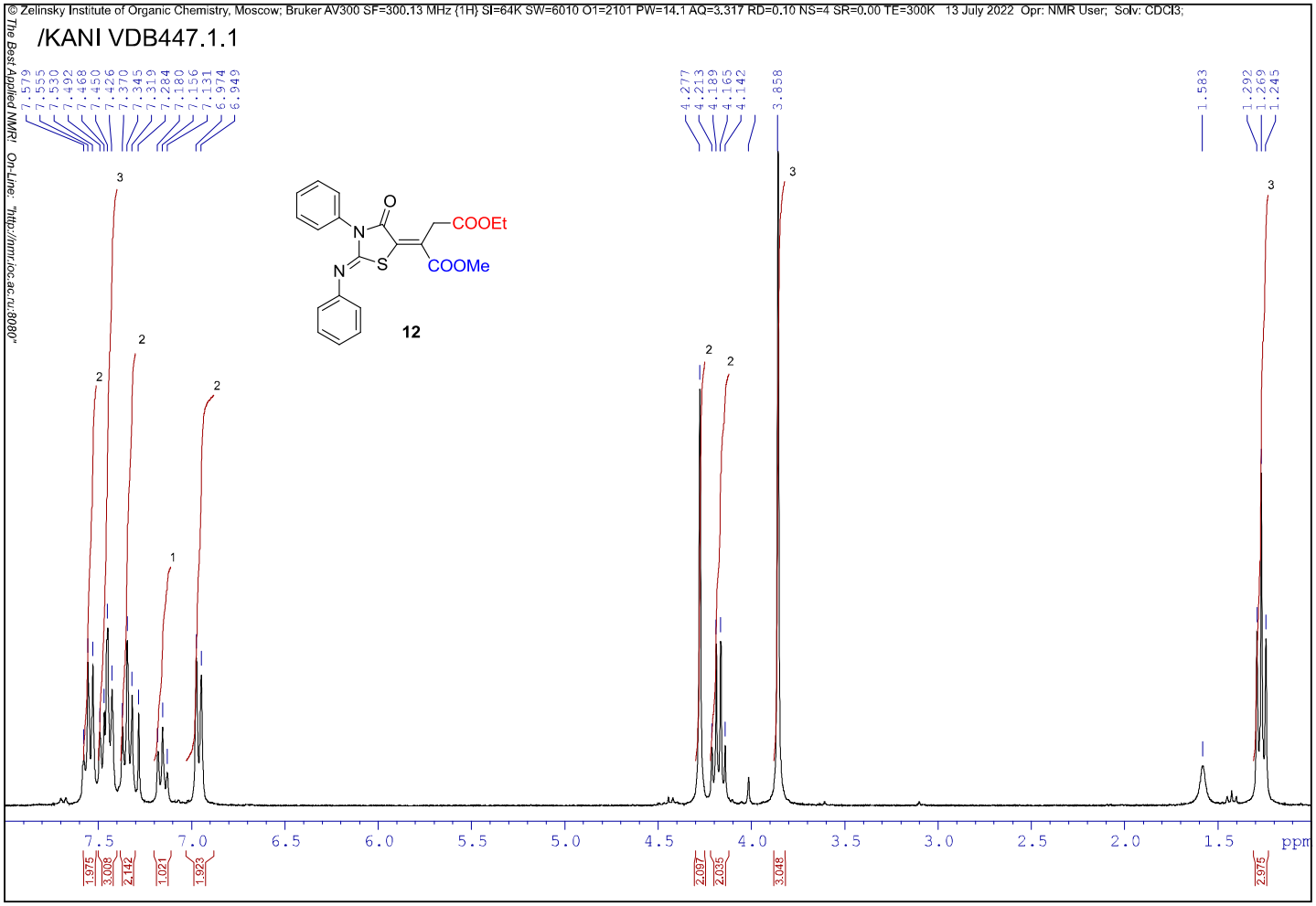

© Zelinsky Institute of Organic Chemistr y, Moscow; Bruker AV600 SF=150.90 MHz (13C) SI=32K SW=39058 O1=17971 PW=12.0 AQ=0.418 RD=2.00 NS=32 SR=-106.86 TE=295K 13 December 2021 Opr: Strelenko Yu.A.; Solv: CDCl3;
/KANI VDB309
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
183.309
171.501
167.228
162.861
140.097
139.333
138.031
129.751
129.624
128.954
125.051
78.006
77.794
77.583
68.062
67.721
63.322
62.022
39.779
33.112
14.856
14.619
12.526
Et
N
H
N
S
N
N
S
O
COOEt
COOEt
6m
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

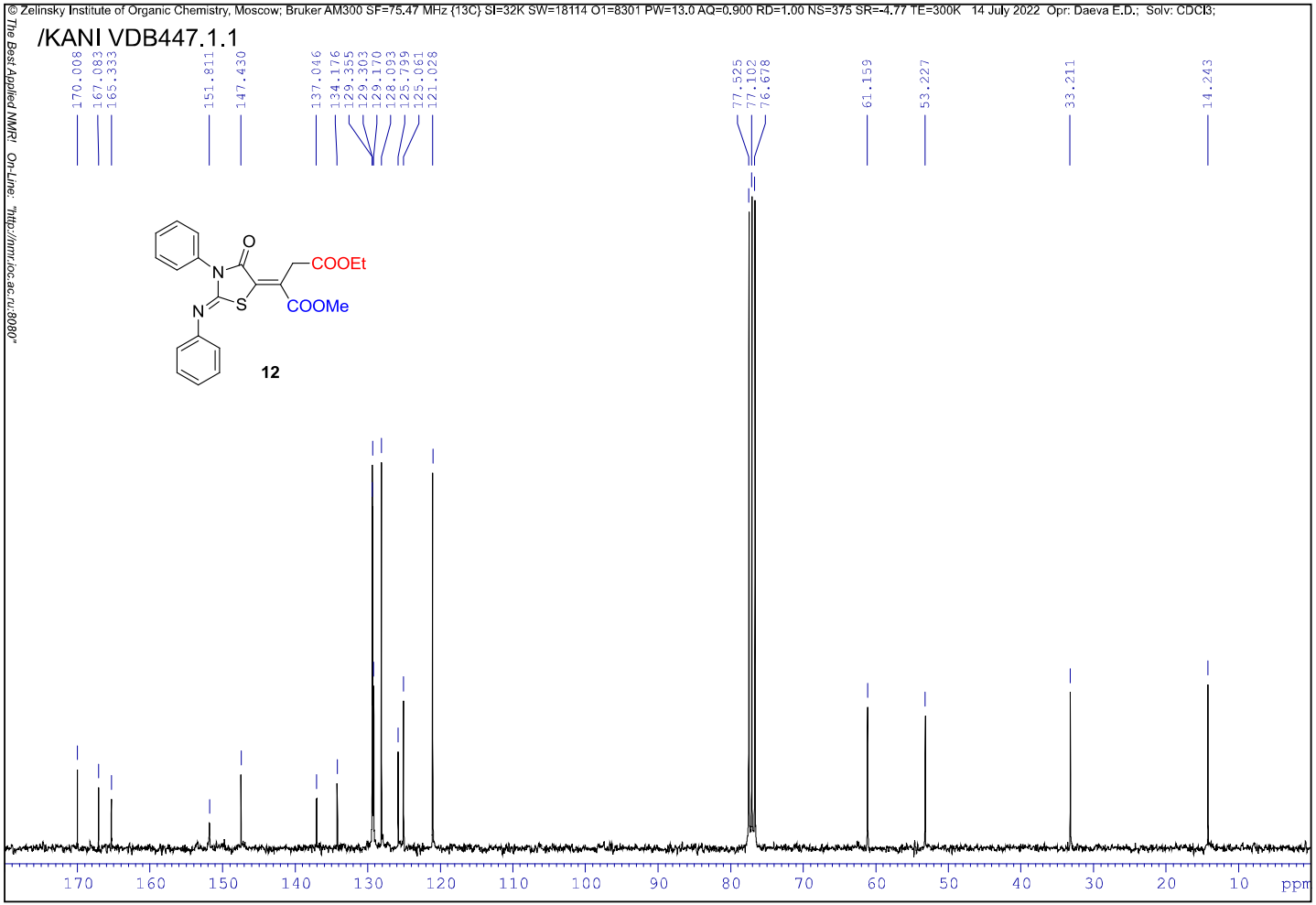

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AV300 SF=300.13 MHz {1H} SI=64K SW=6010 O1=2101 PW=14.1 AQ=3.317 RD=0.10 NS=4 SR=0.00 TE=300K 11 July 2022 Opr: NMR User; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI VDB308.2
6n
COOMe
COOEt
ppm


© 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=2.00 NS=81 SR=-106.86 TE=295K 13 December 2021 Opr: Strelenko Yu.A.; Solv: CDCl3;
The Best Applied NMR! On-Line. "http://nmr.ioc.ac.ru:8080"
/KANI VDB308
183.358
171.277
167.746
162.796
140.044
139.293
138.315
129.770
129.628
128.984
124.743
77.998
77.786
77.573
68.106
67.677
62.034
54.053
39.835
33.087
14.841
12.595
Et
H
N
S
COOMe
COOEt
6n
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

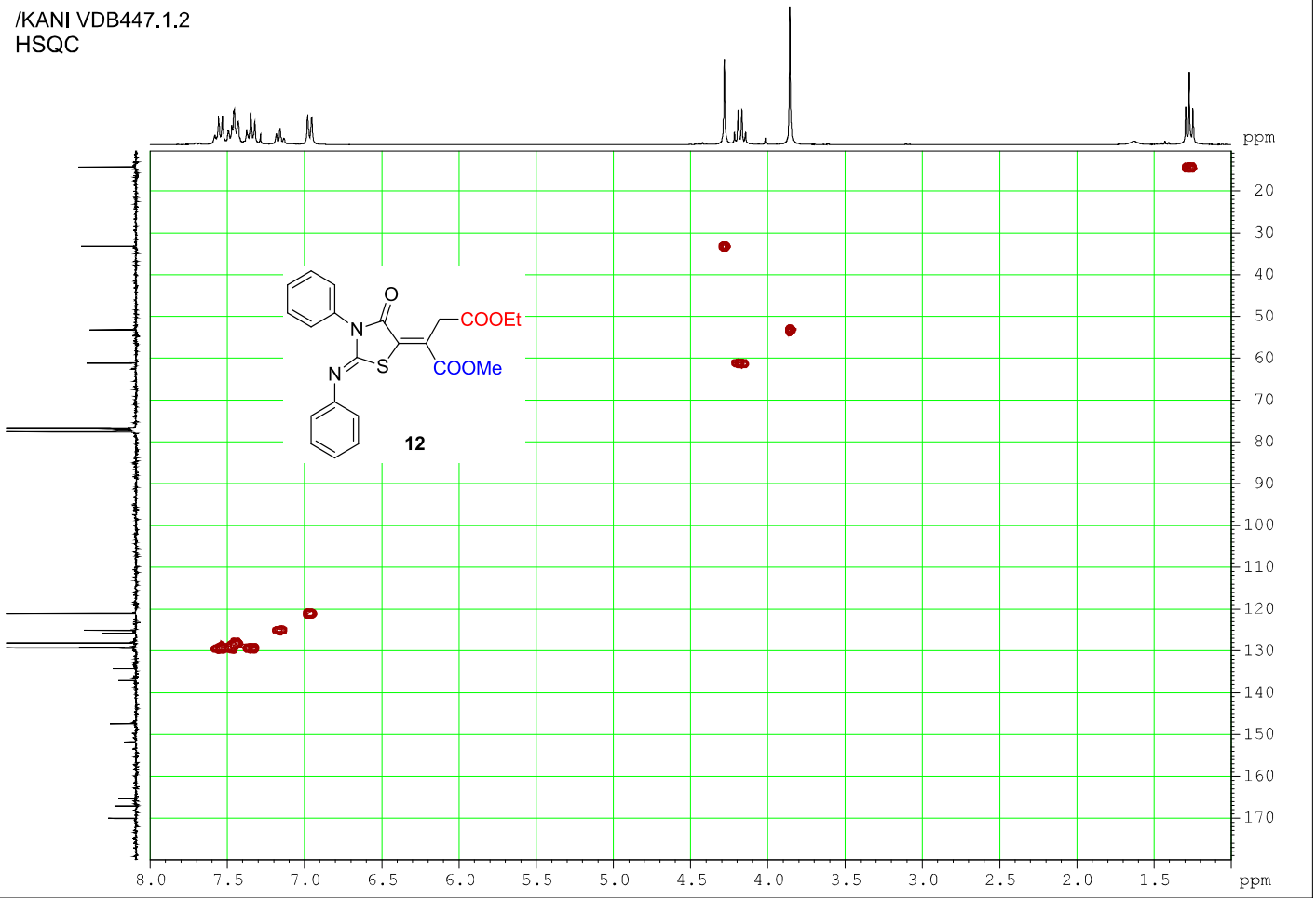

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=32K SW=6010 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=1 SR=15.26 TE=300K 27 July 2022 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1721.3
7.905
7.877
7.628
7.622
7.370
7.345
7.319
7.315
7.307
7.280
7.256
7.230
4.293
4.212
4.188
4.164
4.140
3.906
1.577
1.282
1.258
1.234
O
N
S
N
COOEt
COOMe
11
1
1
2
2
2
3
3
3
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
1.200
1.209
3.062
1.998
2.608
3.510
3.716


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=32392 SR=-7.78 TE=300K 27 July 2022 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
/KANI IA1721.3
169.285
166.521
158.693
154.820
148.984
140.468
131.368
130.345
126.464
124.749
120.024
112.919
77.526
77.103
76.679
61.518
53.706
33.243
14.290
O
N
N
S
COOEt
COOMe
11
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

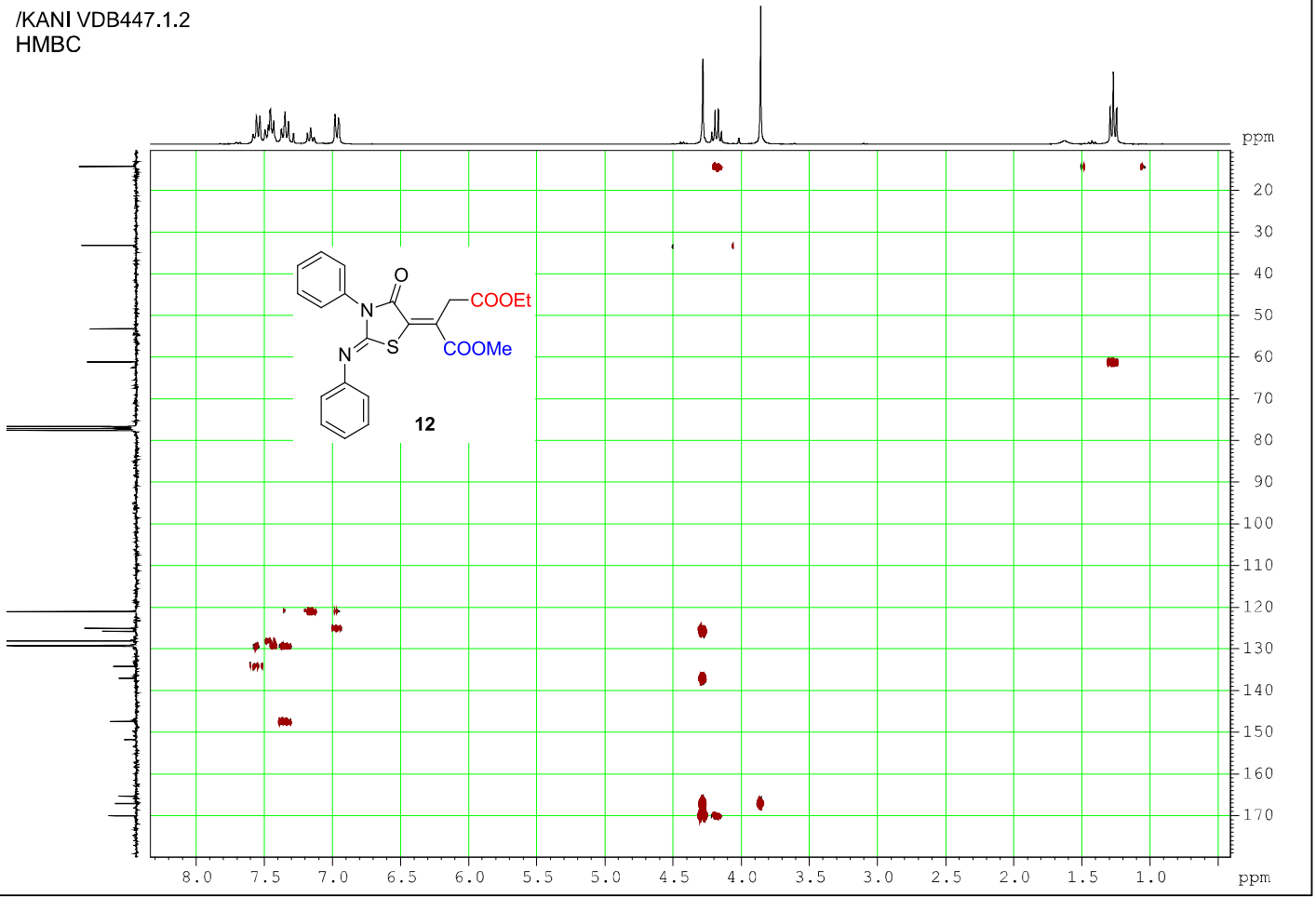

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AV300 SF=300.13 MHz {1H} SI=64K SW=6010 O1=2101 PW=14.1 AQ=3.317 RD=0.10 NS=4 SR=0.00 TE=300K 13 July 2022 Opr: NMR User; Solv: CDCl3;
The Best Applied NMR!. On-Line. "http://nmr.ioc.ac.ru:8080"
/KANI VDB447.1.1
7.579
7.555
7.530
7.492
7.468
7.450
7.426
7.370
7.345
7.319
7.284
7.180
7.156
7.131
6.974
6.949
4.277
4.213
4.189
4.165
4.142
3.858
1.583
1.292
1.269
1.245
COOEt
COOMe
12
ppm
1.975
3.008
2.142
1.021
1.923
2.097
2.036
3.048
2.975

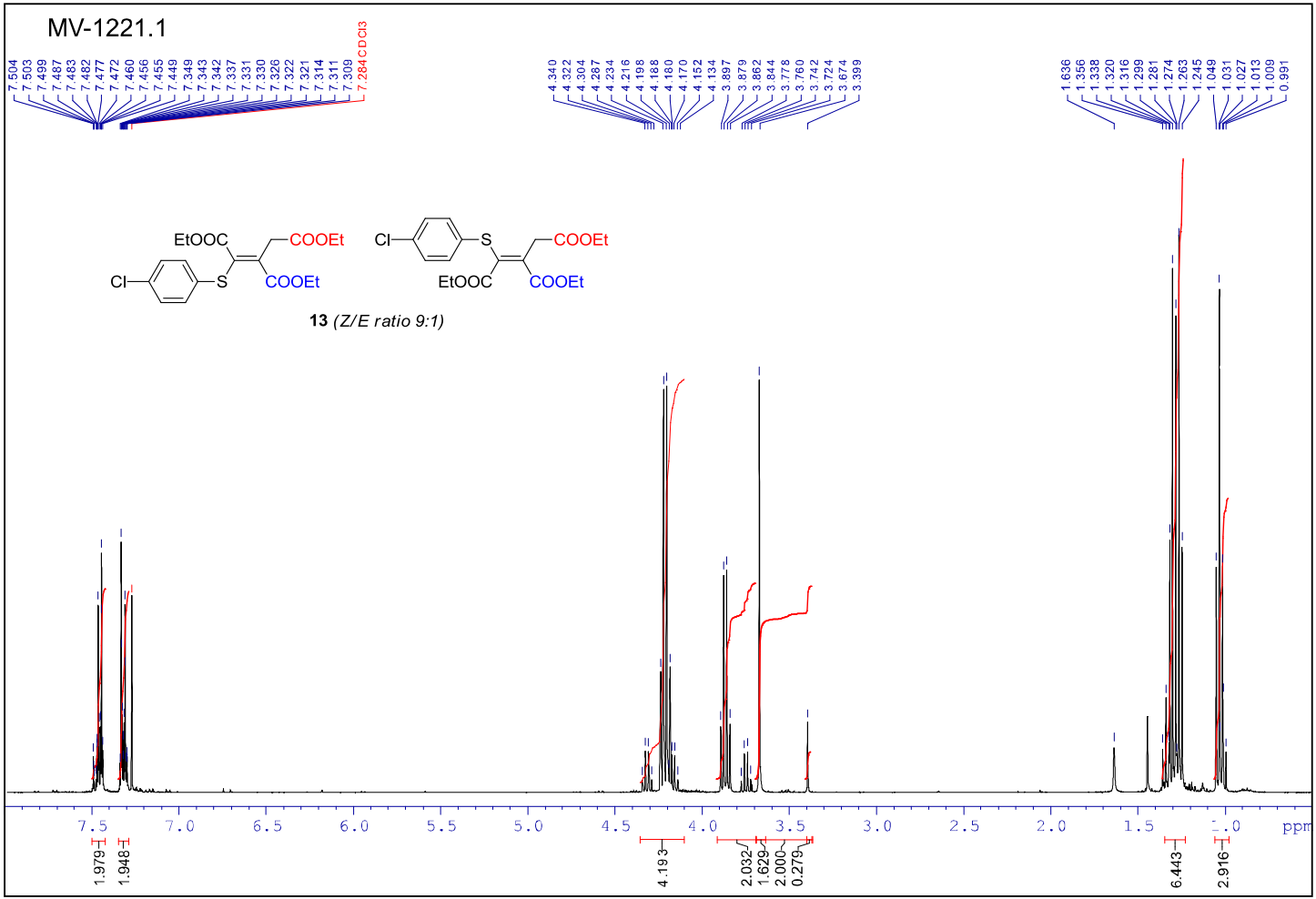

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=13.0 AQ=0.900 RD=1.00 NS=375 SR=-4.77 TE=300K 14 July 2022 Opr: Daeva E.D.; Solv: CDCl3;
The Best Applied NMR! On-Line, "http://nmr.ioc.ac.ru:8080"
/KANI VDB447.1.1
170.008
167.083
165.333
151.811
147.430
137.046
134.176
129.355
129.303
129.170
128.093
125.799
125.061
121.028
77.525
77.102
76.678
61.159
53.227
33.211
14.243
O
N
S
N
COOEt
COOMe
12
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

/KANI VDB447.1.2
HSQC



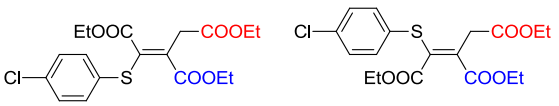
/KANI VDB447.1.2
HMBC





13 *(Z/E ratio 9:1)*

MV-1221.1



13 *(Z/E ratio 9:1)*

MV-1221.1 HMBC