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Synthesis of trialkyl semithioglycolurils from alkylthiourea-glyoxal cyclic adducts and dialkylureas

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General

All reagents (Acros) were used as purchased, unless otherwise indicated. ^{1}H and ^{13}C NMR were recorded at 25 to 30 °C using Bruker AM300 (^{1}H, 300.13 MHz; ^{13}C, 75.5 MHz). Chemical shifts are reported in the δ scale relative to Me_4Si as internal standard. High resolution mass spectra (HRMS) were recorded on a Bruker micrOTOF II instrument using the electrospray ionization method (ESI). Infrared spectra were obtained on a BrukerALPHA spectrometer using KBr pellets and reported in wavenumbers (cm^{-1}). Melting points were determined in a SMP10 instrument (Stuart).

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Spectral data of compounds 1a-h

1,3-Diethyl-4-methyl-5-thioxohexahydroimidazo[4,5-d]imidazol-2(1H)-one **1a.** Beige powder. Yield 52% (0.30 g), mp 180 – 182 °C (H_2O). IR (ν/cm^{-1}): 3206, 1693, 1672, 1508, 1450, 1405, 1359, 1322, 1288, 1260, 1222, 1206, 1119, 1102, 1067, 1022, 976, 929, 917, 840, 798, 777, 755, 700, 657, 632, 586. ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 1.06 (t, 3H, *J* = 7.2 Hz, $CH_2\underline{CH_3}$); 1.10 (t, 3H, *J* = 7.0 Hz, $CH_2\underline{CH_3}$); 3.11 (s, 3H, NMe); 3.09 (dq, 1H, *J* = 7.1 Hz, *J* = 14.2 Hz, $\underline{CH_2}CH_3$); 3.17 – 3.33 (m, 2H, $\underline{CH_2}CH_3$); 3.42 (dq, 1H, *J* = 7.2 Hz, *J* = 14.3 Hz, $\underline{CH_2}CH_3$); 5.40 (d, 1H, *J* = 8.4 Hz, CH); 5.52 (d, 1H, *J* = 8.5 Hz, CH); 9.31 (s, 1H, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ (ppm) 12.81, 13.16 (CMe), 32.22 (NMe), 35.68, 37.56 (NCH_2), 66.59, 74.41 (HC-CH), 157.05 (C=O), 182.23 (C=S). HRMS (ESI), *m/z*: 229.1114 $[M+H]^+$ (calc. for: $C_9H_{16}N_4OS$+H, *m/z*: 229.1118); *m/z*: 251.0936 $[M+Na]^+$ (calc. for: $C_9H_{16}N_4OS$+Na, *m/z*: 251.0937).

1,3,4-Trimethyl-5-thioxohexahydroimidazo[4,5-d]imidazol-2(1H)-one **1b.** Beige powder. Yield 58% (0.29 g), mp 191 – 193 °C (H_2O). IR (ν/cm^{-1}): 3180, 1724, 1505, 1449, 1410, 1392, 1336, 1269, 1239, 1209, 1116, 1062, 1035, 1002, 918, 862, 788, 748, 728, 670, 652, 609, 575. ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 2.70 (s, 3H, NMe); 2.88 (s, 3H, NMe); 3.12 (s, 3H, NMe); 5.27 (d, 1H, *J* = 8.4 Hz, CH); 5.39 (d, 1H, *J* = 8.4 Hz, CH); 9.32 (s, 1H, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ (ppm) 28.15, 30.26, 32.28 (Me), 68.42, 76.51 (HC-CH), 157.72 (C=O), 182.25 (C=S). HRMS (ESI), *m/z*: 201.0811 $[M+H]^+$ (calc. for: $C_7H_{12}N_4OS$+H, *m/z*: 201.0805); *m/z*: 223.0622 $[M+Na]^+$ (calc. for: $C_7H_{12}N_4OS$+Na, *m/z*: 223.0624).

4-Methyl-1,3-dipropyl-5-thioxohexahydroimidazo[4,5-d]imidazol-2(1H)-one **1c.** Grey powder. Yield 46% (0.30 g), mp 166 – 168 °C (H_2O). IR (ν/cm^{-1}): 3234, 1672, 1503, 1459, 1429, 1404, 1382, 1330, 1270, 1250, 1218, 1197, 1117, 1076, 1010, 969, 942, 896, 858, 795, 760, 743, 727, 682, 662, 648, 627, 584. ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 0.81 (t, 3H, *J* = 7.3 Hz, $CH_2\underline{CH_3}$); 0.82 (t, 3H, *J* = 7.3 Hz, $CH_2\underline{CH_3}$); 1.37 – 1.67 (m, 4H, $\underline{CH_2}CH_3$); 3.10 (s, 3H, NMe); 2.96 – 3.22 (m, 3H, $N\underline{CH_2}CH_2$); 3.28 – 3.39 (m, 1H, $N\underline{CH_2}CH_2$); 5.38 (d, 1H, *J* = 8.5 Hz, CH); 5.51 (d, 1H, *J* = 8.5 Hz, CH); 9.31 (s, 1H, NH). ^{13}C NMR (75 MHz, DMSO-d_6/D_2O (98/2)): δ (ppm) 11.04, 11.16 (C<u>Me</u>), 20.36, 20.66 ($C\underline{CH_2}C$), 32.56 (NMe), 42.59, 44.38 (NCH_2), 66.85, 66.96 (HC(6a)), 74.66 (HC(3a)), 157.73 (C=O), 182.35 (C=S). HRMS (ESI), *m/z*: 257.1438 $[M+H]^+$ (calc. for: $C_{11}H_{20}N_4OS$+H, *m/z*: 257.1431); *m/z*: 279.1256 $[M+Na]^+$ (calc. for: $C_{11}H_{20}N_4OS$+Na, *m/z*: 279.1250).

4-Ethyl-1,3-dimethyl-5-thioxohexahydroimidazo[4,5-d]imidazol-2(1H)-one **1d.** Beige powder. Yield 63% (0.34 g), mp 183 – 185 °C (H_2O). IR (ν/cm^{-1}): 3275, 1692, 1508, 1484, 1422, 1403, 1379, 1324, 1250, 1228, 1123, 1082, 1068, 1038, 958, 861, 792, 750, 666, 645, 578,

556. ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 1.15 (t, 3H, *J* = 7.1 Hz, $CH_2\underline{CH_3}$); 2.69 (s, 3H, NMe); 2.88 (s, 3H, NMe); 3.43 (dq, 1H, *J* = 7.1 Hz, *J* = 14.2 Hz, $\underline{CH_2}CH_3$); 3.79 (dq, 1H, *J* = 7.2 Hz, *J* = 14.4 Hz, $\underline{CH_2}CH_3$); 5.25 (d, 1H, *J* = 8.5 Hz, CH); 5.48 (d, 1H, *J* = 8.5 Hz, CH); 9.31 (s, 1H, NH). ^{13}C NMR (75 MHz, DMSO-d_6/D_2O (98/2)): δ (ppm) 12.45 (CMe), 28.15(NMe), 30.59 (NCH_2), 68.59, 68.70 (HC(6a)), 74.76 (HC(3a)), 158.07 (C=O), 181.72 (C=S). HRMS (ESI), *m/z:* 215.0971 $[M+H]^+$ (calc. for: $C_8H_{14}N_4OS$+H, *m*/*z*: 215.0961); *m/z*: 237.0787 $[M+Na]^+$ (calc. for: $C_8H_{14}N_4OS$+Na, *m*/*z*: 237.0781).

1,3,4-Triethyl-5-thioxohexahydroimidazo[4,5-d]imidazol-2(1H)-one **1e.** Beige powder. Yield 58% (0.35 g), mp 154 – 156 °C (H_2O). IR (ν/cm^{-1}): 3232, 1692, 1673, 1492, 1437, 1379, 1355, 1320, 1260, 1238, 1209, 1123, 1064, 1027, 952, 918, 838, 800, 775, 756, 703, 660, 625, 584. ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 1.05 (t, 3H, *J* = 7.2 Hz, $CH_2\underline{CH_3}$); 1.08 (t, 3H, *J* = 7.1 Hz, $CH_2\underline{CH_3}$); 1.14 (t, 3H, *J* = 7.0 Hz, $CH_2\underline{CH_3}$); 3.01 – 3.54 (m, 5H, $\underline{CH_2}CH_3$); 3.87 (dq, 1H, *J* = 7.1 Hz, *J* = 14.4 Hz, $\underline{CH_2}CH_3$); 5.37 (d, 1H, *J* = 8.6 Hz, CH); 5.61 (d, 1H, *J* = 8.6 Hz, CH); 9.29 (s, 1H, NH). ^{13}C NMR (75 MHz, DMSO-d_6/D_2O (98/2)): δ (ppm) 12.37, 12.93, 13.19 (CMe), 35.85, 37.88, 39.43 (NCH_2), 66.75, 66.85 (HC(6a)), 72.23 (HC(3a)), 157.38 (C=O), 181.72 (C=S). HRMS (ESI), *m/z*: 243.1285 $[M+H]^+$ (calc. for: $C_{10}H_{18}N_4OS$+H, *m*/*z*: 243.1274); *m/z*: 265.1099 $[M+Na]^+$ (calc. for: $C_{10}H_{18}N_4OS$+Na, *m*/*z*: 265.1094).

4-Ethyl-1,3-dipropyl-5-thioxohexahydroimidazo[4,5-d]imidazol-2(1H)-one **1f.** Grey powder. Yield 60% (0.40 g), mp 132 – 133 °C (H_2O). IR (ν/cm^{-1}): 3231, 1673, 1487, 1462, 1431, 1374, 1321, 1239, 1199, 1124, 1076 1017, 959, 935, 893, 856, 798, 780, 755, 699, 653, 627, 594, 574. ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 0.81 (t, 3H, *J* = 7.2 Hz, $CH_2CH_2\underline{CH_3}$); 0.82 (t, 3H, *J* = 7.2 Hz, $CH_2CH_2\underline{CH_3}$); 1.14 (t, 3H, *J* = 7.0 Hz, $CH_2\underline{CH_3}$); 1.37 – 1.67 (m, 4H, $\underline{CH_2}CH_3$); 2.96 – 3.22 (m, 3H, $N\underline{CH_2}$); 3.28 – 3.43 (m, 2H, $N\underline{CH_2}$); 3.86 (dq, 1H, *J* = 7.2 Hz, *J* = 14.4 Hz, $N\underline{CH_2}CH_3$); 5.36 (d, 1H, *J* = 8.5 Hz, CH); 5.61 (d, 1H, *J* = 8.5 Hz, CH); 9.28 (s, 1H, NH). ^{13}C NMR (75 MHz, DMSO-d_6/D_2O (98/2)): δ (ppm) 11.08, 11.17 ($CH_2CH_2\underline{Me}$), 12.42 ($NCH_2\underline{Me}$), 20.31, 20.68 (CCH_2C), 39.59, 42.54, 44.48 (NCH_2), 66.92, 67.03 (HC(6a)), 72.35 (HC(3a)), 157.84 (C=O), 181.75 (C=S). HRMS (ESI), *m/z*: 271.1587 $[M+H]^+$ (calc. for: $C_{12}H_{22}N_4OS$+H, *m*/*z*: 271.1587); *m/z*: 293.1402 $[M+Na]^+$ (calc. for: $C_{12}H_{22}N_4OS$+Na, *m*/*z*: 293.1407).

4-Isobutyl-1,3-dimethyl-5-thioxohexahydroimidazo[4,5-d]imidazol-2(1H)-one **1g.** Beige powder. Yield 66% (0.40 g), mp 153 – 155 °C (H_2O). IR (ν/cm^{-1}): 3269, 1699, 1680, 1505, 1416, 1400, 1372, 1317, 1282, 1265, 1230, 1172, 1038, 1017, 888, 862, 793, 761, 748, 666, 642, 588, 573. ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 0.84 (d, 3H, *J* = 6.5 Hz, $CH\underline{CH_3}$); 0.94 (d, 3H, *J* = 6.5 Hz, $CH\underline{CH_3}$); 2.08 – 2.23 (m, 1H, *J* = 7.2 Hz, $CH_2\underline{CH}(CH_3)_2$); 2.70 (s, 3H, *J* = 7.0 Hz, NCH_3); 2.88 (s, 3H, *J* = 7.0 Hz, NCH_3); 3.10 (dd, 1H, *J* = 5.6 Hz, *J* = 14.0 Hz, $N\underline{CH_2}CH$);

3.78 (dd, 1H, *J* = 9.6 Hz, *J* = 13.8 Hz, NCH$_2$CH); 5.28 (d, 1H, *J* = 8.5 Hz, CH); 5.45 (d, 1H, *J* = 8.4 Hz, CH); 9.33 (s, 1H, NH). ^{13}C NMR (75 MHz, DMSO-*d*$_6$/D$_2$O (98/2)): δ (ppm) 19.60, 20.28 (CH$_2$CHMe$_2$), 25.80 (NCH$_2$CH), 28.16, 31.29 (NMe), 51.18 (NCH$_2$), 68.52, 68.63 (HC(6a)), 74.82 (HC(3a)), 158.26 (C=O), 182.36, 182.43 (C=S). HRMS (ESI), *m/z*: 243.1282 [M+H]$^+$ (calc. for: C$_{10}$H$_{18}$N$_4$OS+H, *m/z*: 243.1274); *m/z*: 265.1101 [M+Na]$^+$ (calc. for: C$_{10}$H$_{18}$N$_4$OS+Na, *m/z*: 265.1094).

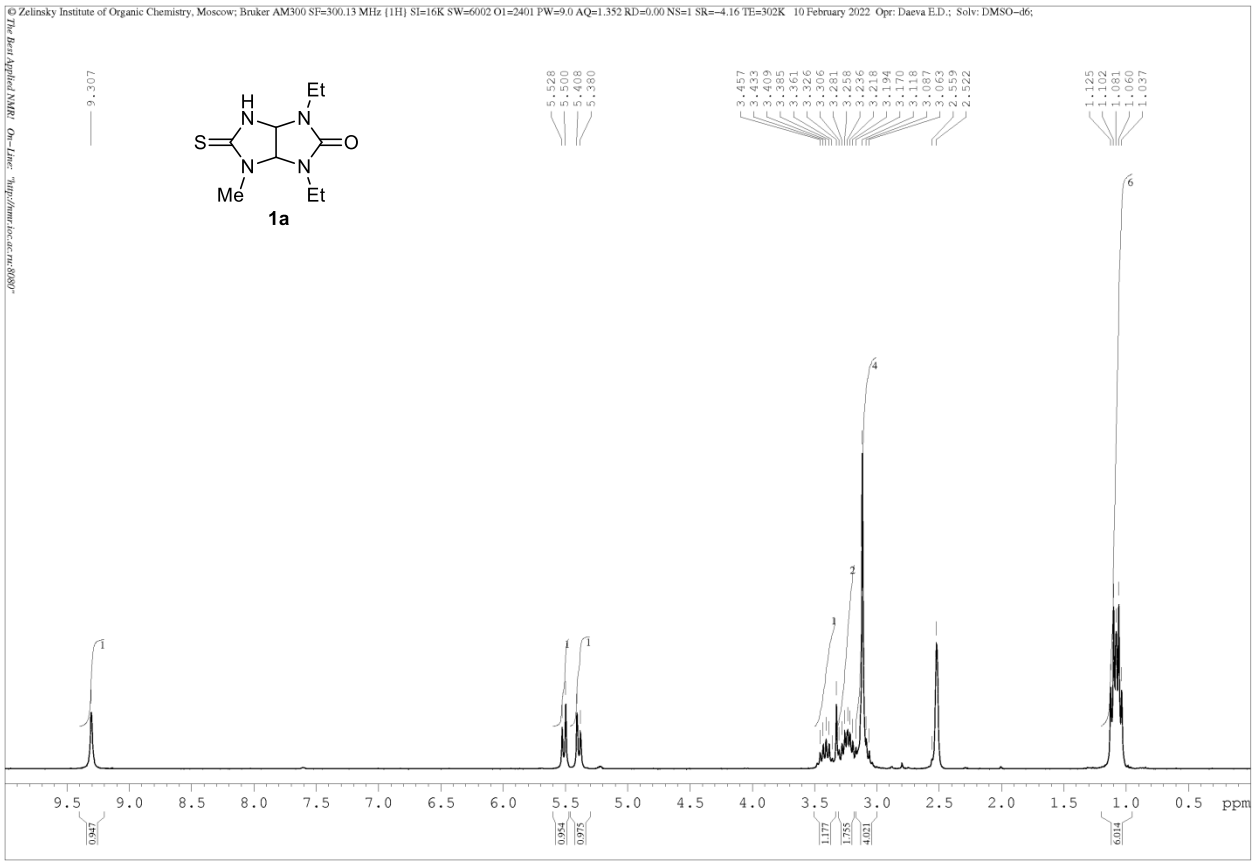
1,3-Diethyl-4-isobutyl-5-thioxohexahydroimidazo[4,5-d]imidazol-2(1H)-one **1h.** Beige crystalls. Yield 58% (0.39 g), mp 132 – 134 °C (H$_2$O), 132 – 134 °C (EtOAc). IR (ν/cm^{-1}): 3225, 1696, 1500, 1465, 1437, 1380, 1358, 1330, 1297, 1275, 1256, 1239, 1200, 1132, 1069, 1019, 913, 888, 851, 820, 789, 771, 757, 742, 655, 596, 567. ^{1}H NMR (300 MHz, DMSO-*d*$_6$): δ (ppm) 0.84 (d, 3H, *J* = 6.5 Hz, CHCH$_3$); 0.94 (d, 3H, *J* = 6.7 Hz, CHCH$_3$); 1.06 (t, 3H, *J* = 7.0 Hz, CH$_2$CH$_3$); 1.07 (t, 3H, *J* = 6.9 Hz, CH$_2$CH$_3$); 2.03 – 2.21 (m, 1H, *J* = 7.2 Hz, CH$_2$CH(CH$_3$)$_2$); 2.96 – 3.14 (m, 2H+1H, NCH$_2$CH$_3$+NCH$_2$CH); 3.28 (dq, 1H, *J* = 7.2 Hz, *J* = 14.4 Hz, NCH$_2$CH$_3$); 3.38 – 3.49 (m, 1H, NCH$_2$CH$_3$); 3.82 (dd, 1H, *J* = 9.5 Hz, *J* = 14.0 Hz, NCH$_2$CH); 5.41 (d, 1H, *J* = 8.5 Hz, CH); 5.57 (d, 1H, *J* = 8.5 Hz, CH); 9.31 (s, 1H, NH). ^{13}C NMR (75 MHz, DMSO-*d*$_6$/D$_2$O (98/2)): δ (ppm) 12.87, 13.21 (NCH$_2$Me), 19.66, 20.26 (CH$_2$CHMe$_2$), 26.00 (NCH$_2$CH), 35.78, 38.48 (NCH$_2$Me), 50.93 (NCH$_2$CH), 66.62, 66.73 (HC(6a)), 72.36 (HC(3a)), 157.48 (C=O), 182.33 (C=S). HRMS (ESI), *m/z*: 271.1596 [M+H]$^+$ (calc. for: C$_{12}$H$_{22}$N$_4$OS+H, *m/z*: 271.1587); *m/z*: 293.1416 [M+Na]$^+$ (calc. for: C$_{12}$H$_{22}$N$_4$OS+Na, *m/z*: 293.1407).

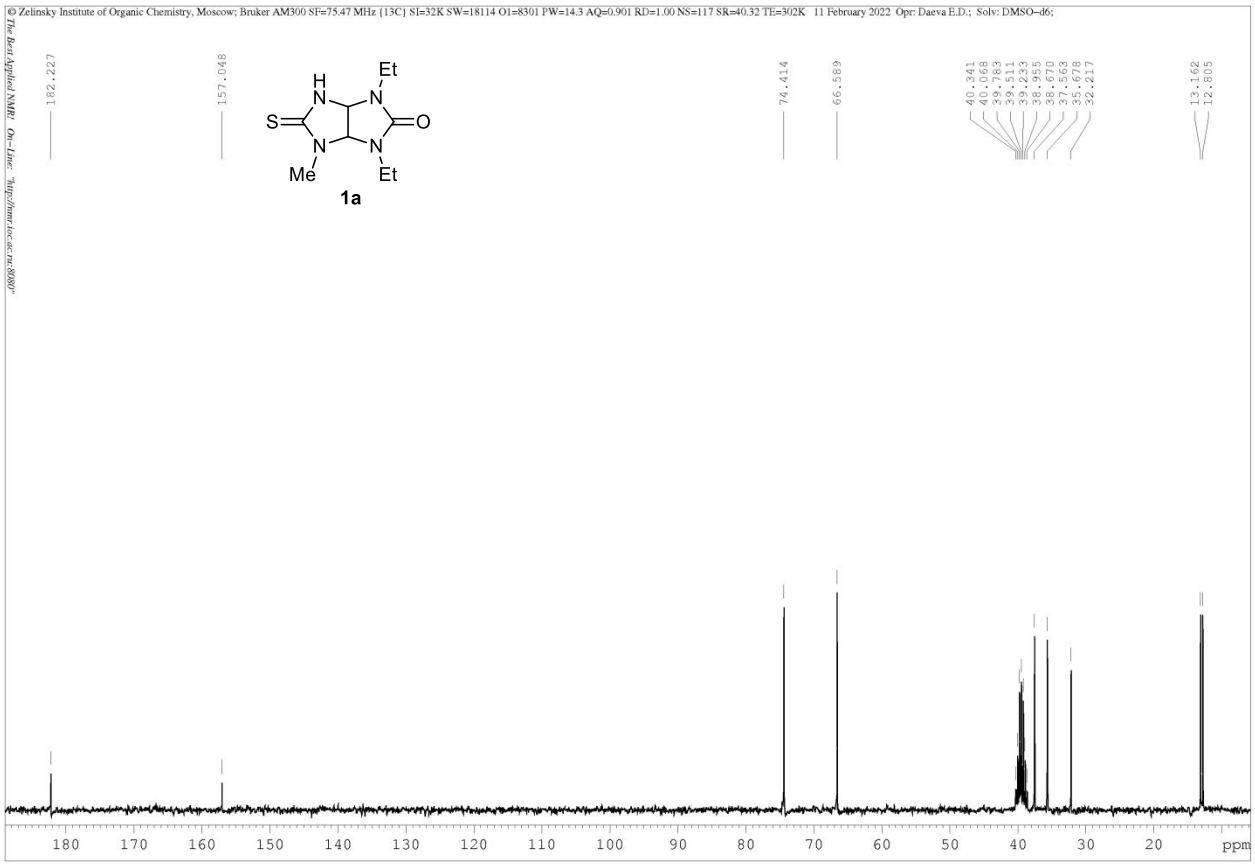
Synthesis and spectral data of compound 4c

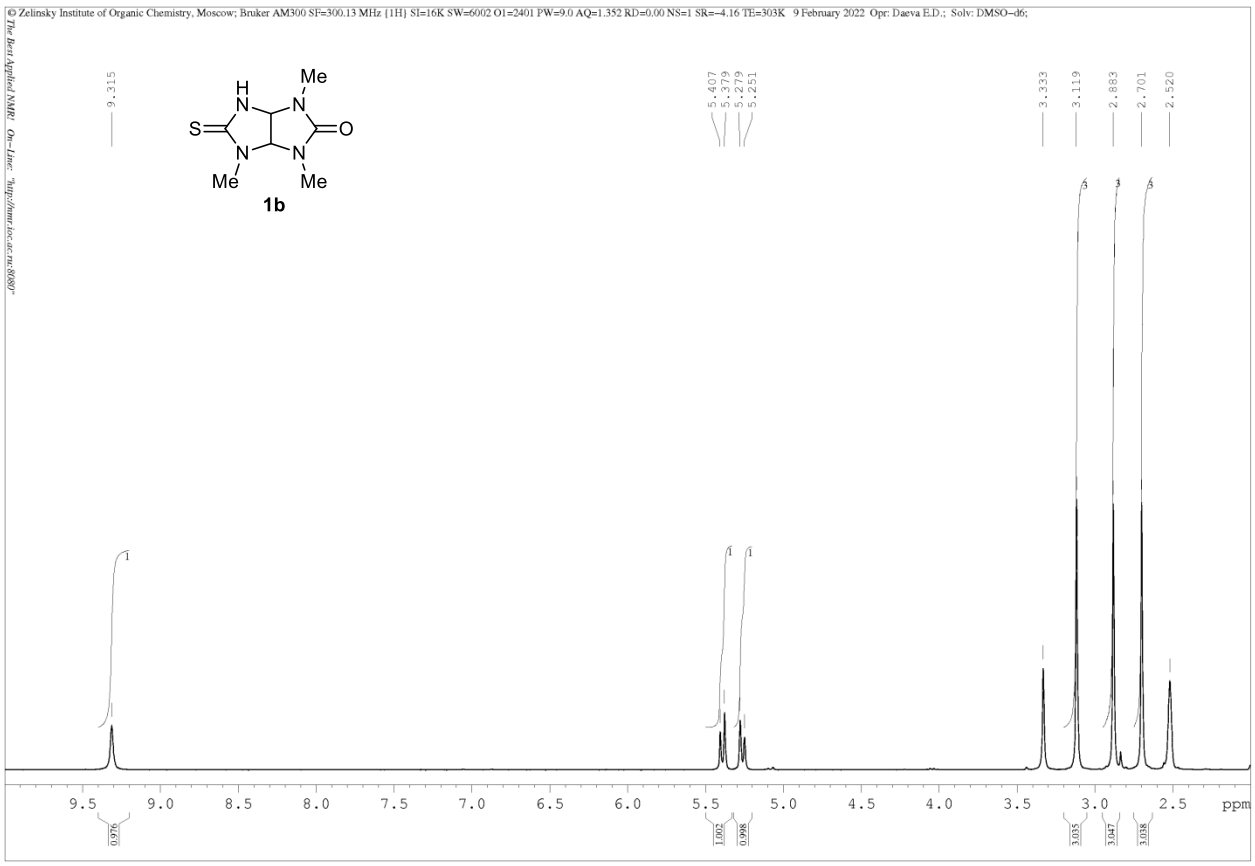
A suspension of 1-isobutylthiourea **2c** (3.30 g, 25 mmol), glyoxal trimer dihydrate (1.75 g, 8.33 mmol) in water (20 ml) was stirred at 45 °C for 1 h. The formed precipitate of compound **4c** was filtered off and dried in air. The second crop of the product was obtained by evaporation of filtrate to dryness. The residue was triturated with H$_2$O, and the formed precipitate of compound **4c** was filtered off.

4,5-Dihydroxy-1-isobutylimidazolidine-2-thione **4c**. Grey powder. Yield 94% (4.465 g), mp 155 – 156 °C (H$_2$O). IR (ν/cm^{-1}): 3306, 3210, 3064, 2957, 1499, 1424, 1390, 1368, 1303, 1249, 1202, 1156, 1092, 1078, 1052, 906, 832, 812, 741, 654, 616, 526, 441. ^{1}H NMR (300 MHz, DMSO-*d*$_6$): δ (ppm) 0.82 (d, 3H, *J* = 6.7 Hz, CHCH$_3$); 0.89 (d, 3H, *J* = 6.7 Hz, CHCH$_3$); 1.96 – 2.11 (m, 1H, CHMe$_2$); 3.04 (dd, 1H, *J* = 6.3 Hz, *J* = 13.5 Hz, CH$_2$); 3.56 (dd, 1H, *J* = 8.7 Hz, *J* = 13.5 Hz, CH$_2$); 5.04 (s, 2H, CH-CH); 5.89 (d 1H, *J* = 4.6 Hz, OH), 6.09 (d 1H, *J* = 7.6 Hz, OH), 8.67 (s, 1H, NH). ^{13}C NMR (75 MHz, DMSO-*d*$_6$): δ (ppm) 20.02, 20.38 (CHMe$_2$), 26.32 (CHMe$_2$), 49.22 (NCH$_2$), 76.75, 81.84 (HC-CH), 180.90 (C=S). HRMS (ESI), m/z: 191.0856 [M+H]$^+$ (calc. for: C$_7$H$_{14}$N$_2$O$_2$S+H, m/z: 191.0849).

NMR spectra of compounds 1a-h, 4c



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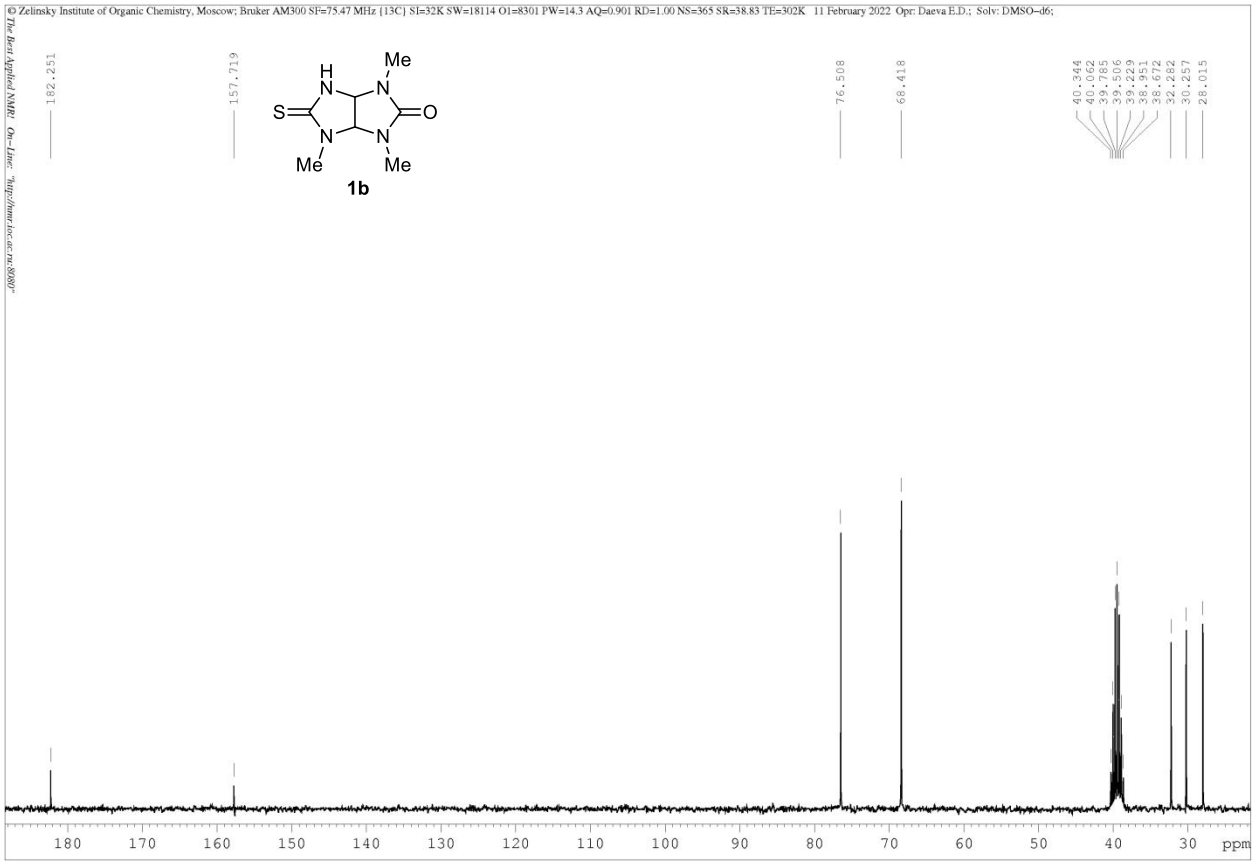
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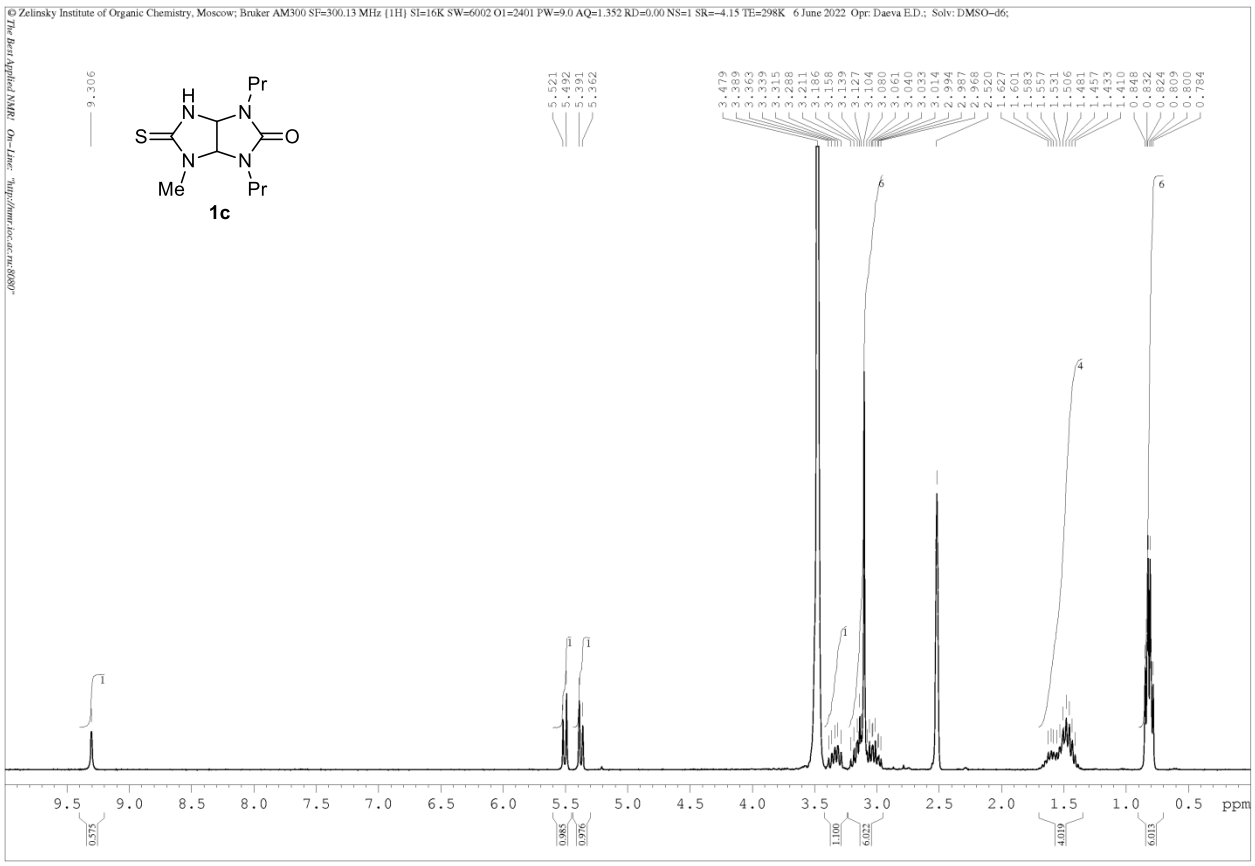
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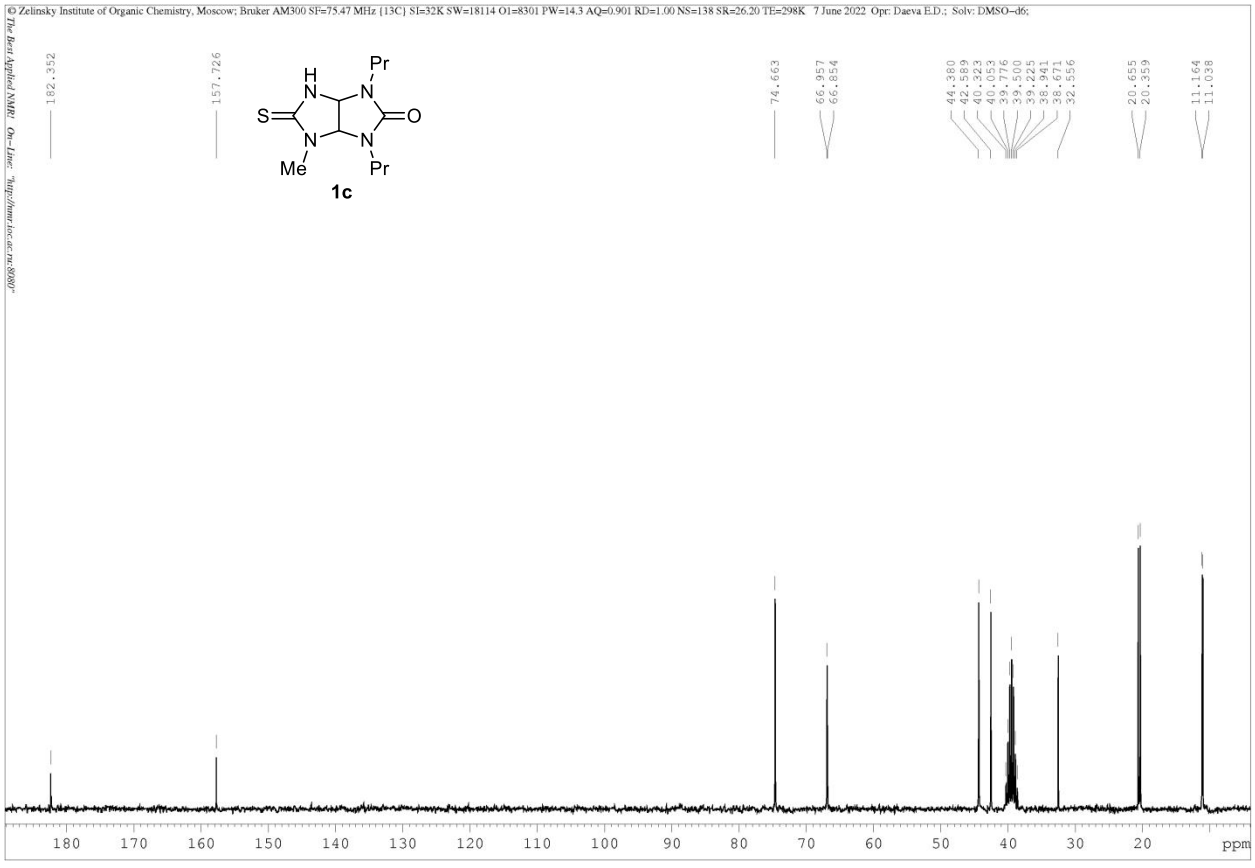
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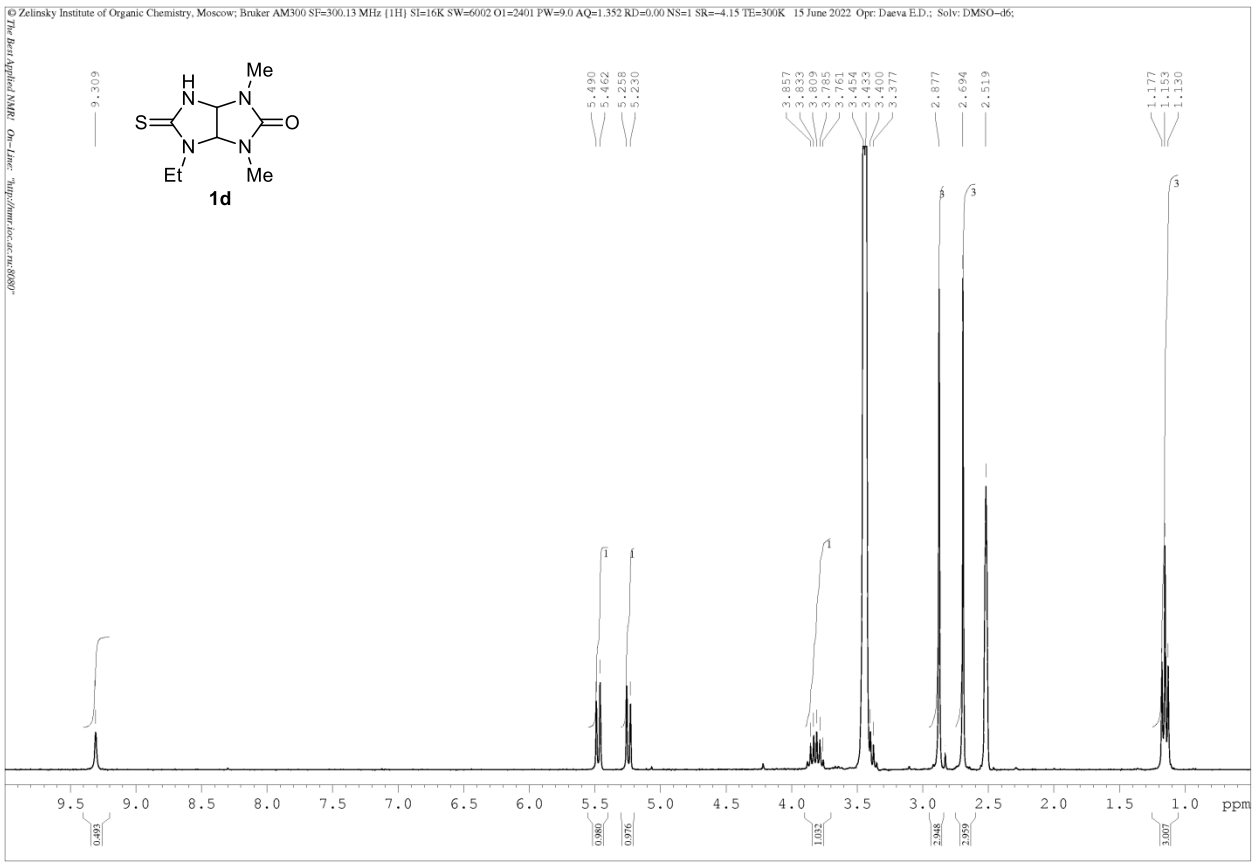
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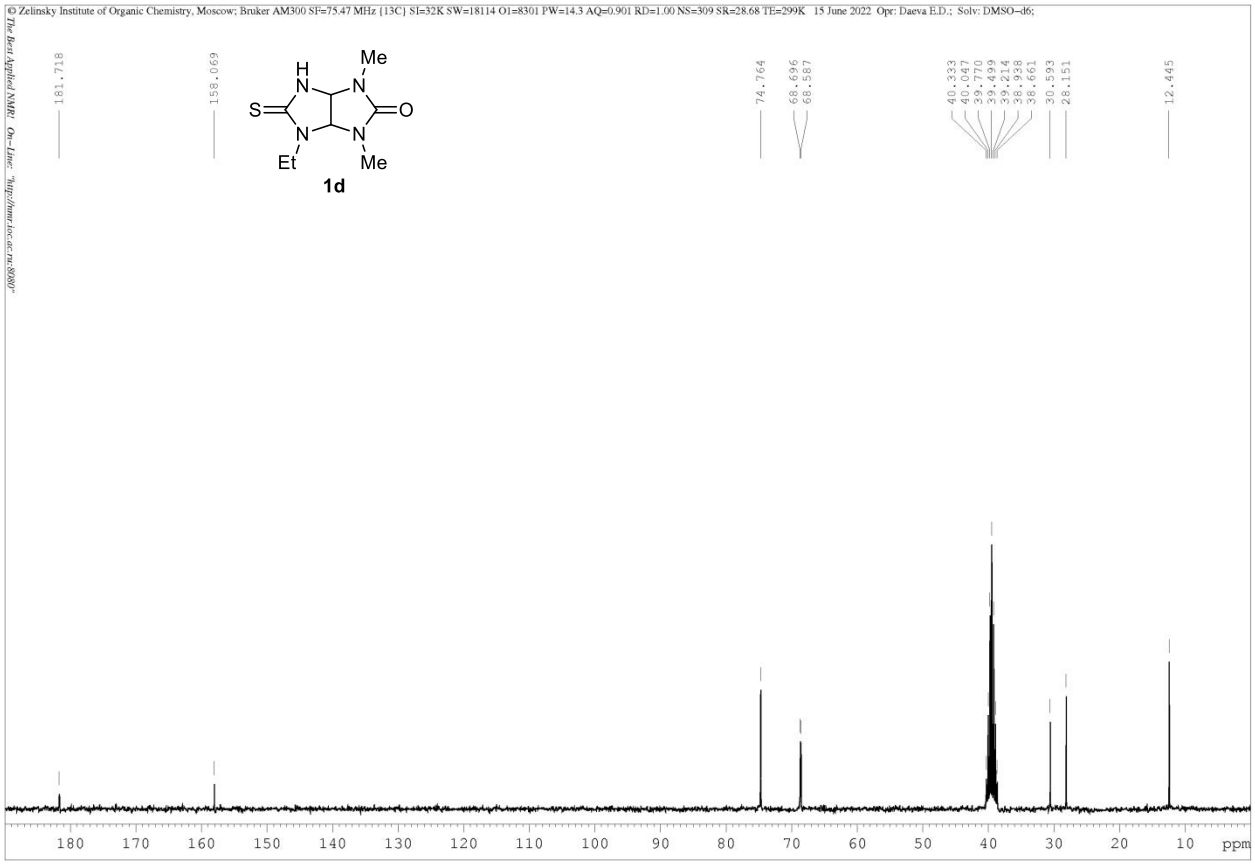
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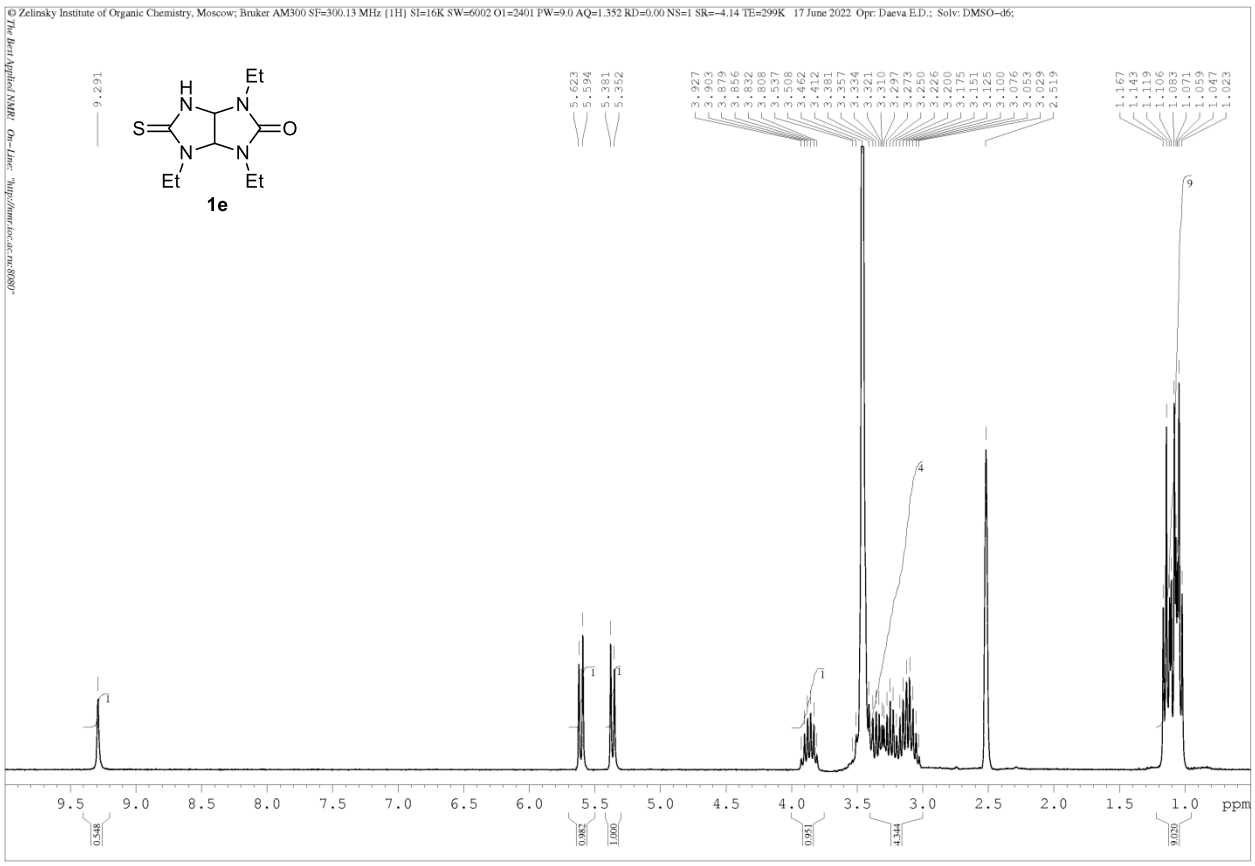
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3.273
3.250
3.226
3.200
3.175
3.151
3.125
3.100
3.076
3.053
3.029
2.519
1.167
1.143
1.119
1.106
1.083
1.071
1.059
1.047
1.023
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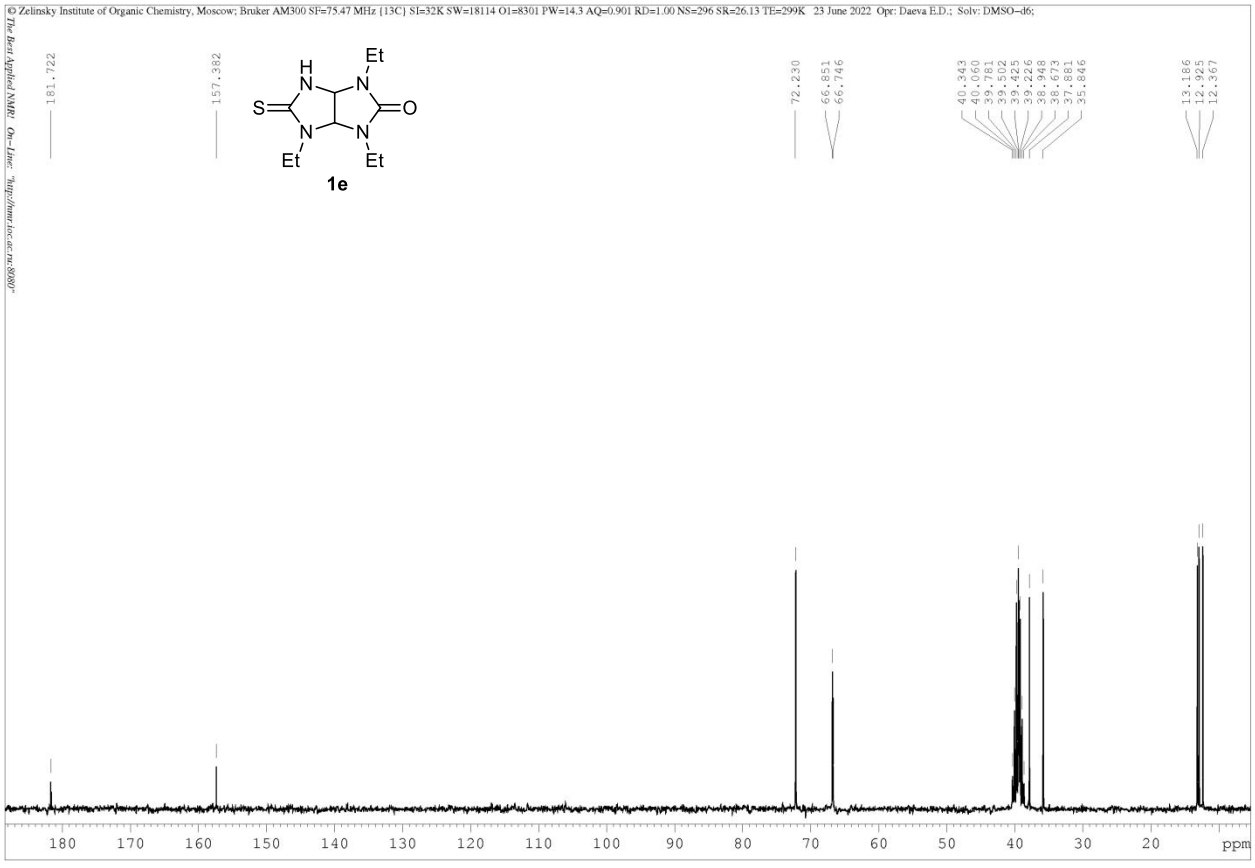
1.000
0.951
4.344
9.020


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=14.3 AQ=0.901 RD=1.00 NS=296 SR=26.13 TE=299K 23 June 2022 Opr: Daeva E.D.; Solv: DMSO-d6;
The Best Applied NMR On-Line: "http://nmr.isoc.ac.ru:8080/"
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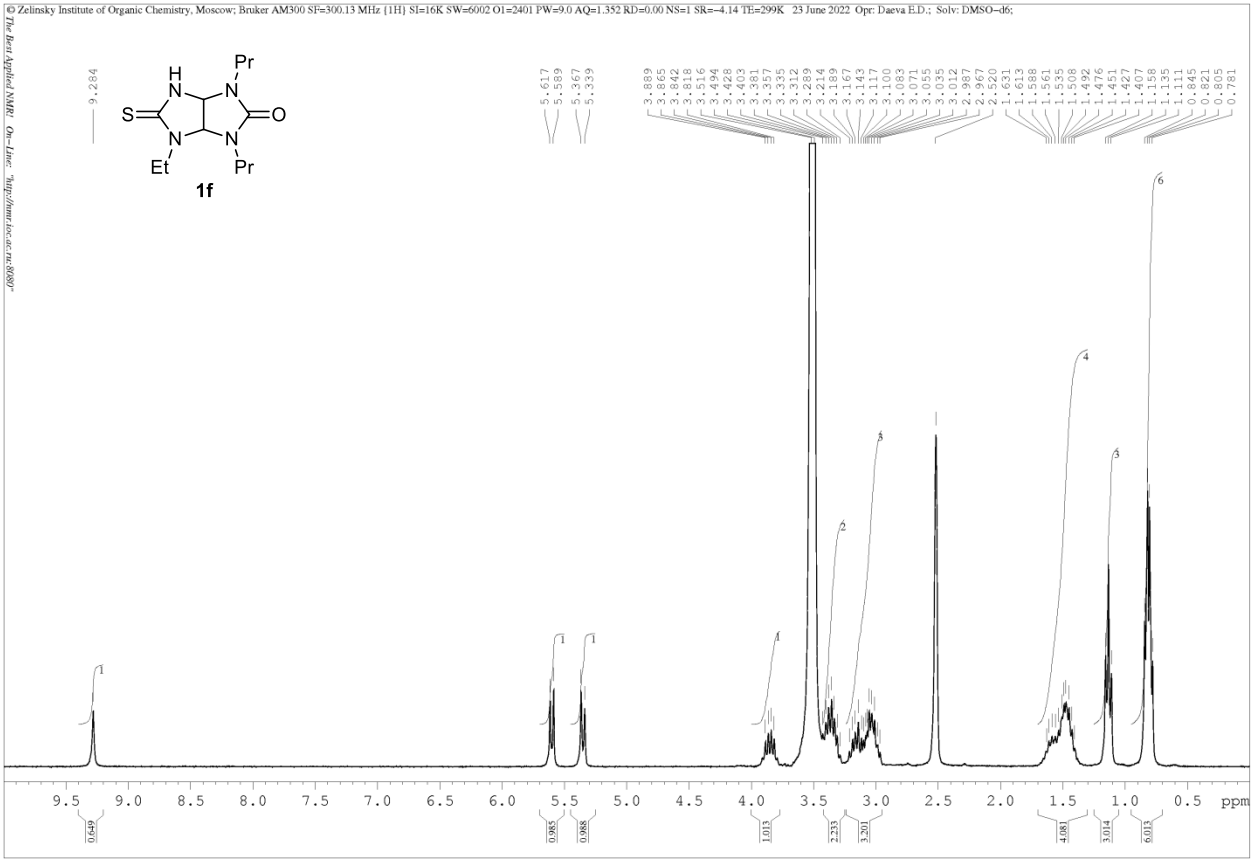
157.382
72.230
66.851
66.746
40.343
40.060
39.781
39.502
39.425
39.226
38.948
38.673
37.881
35.846
13.186
12.925
12.367
1e
ppm


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6002 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=1 SR=-4.14 TE=299K 23 June 2022 Opr: Daeva E.D.; Solv: DMSO-d6;
The Best Applied NMR On-Line: "http://nmr.isoc.ac.ru:8080/"
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5.617
5.589
5.367
5.339
3.889
3.865
3.842
3.818
3.516
3.494
3.428
3.403
3.381
3.357
3.335
3.312
3.289
3.214
3.189
3.167
3.143
3.117
3.100
3.083
3.071
3.055
3.035
3.012
2.987
2.967
2.520
2.631
1.613
1.588
1.561
1.535
1.508
1.492
1.476
1.451
1.427
1.407
1.158
1.135
1.111
0.845
0.821
0.805
0.781
H
N
Pr
N
S
O
N
N
Et
Pr
1f
ppm
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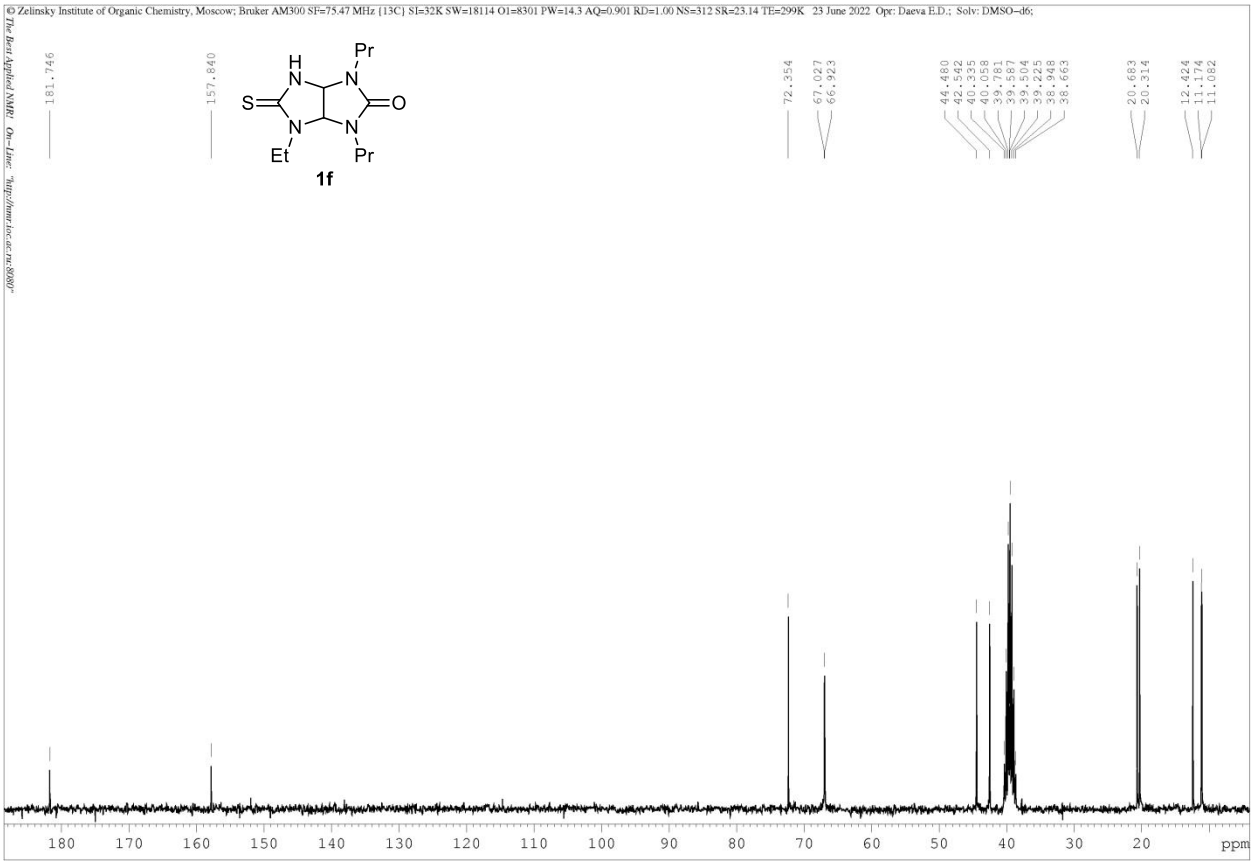
0.888
1.013
2.233
3.201
4.081
3.014
6.013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The Best Applied NMR On-Line: "http://nmr.isoc.ac.ru:8080/"
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157.840
72.354
67.027
66.923
44.480
42.542
40.335
40.058
39.781
39.587
39.504
39.225
38.948
38.663
20.683
20.314
12.424
11.174
11.082
H
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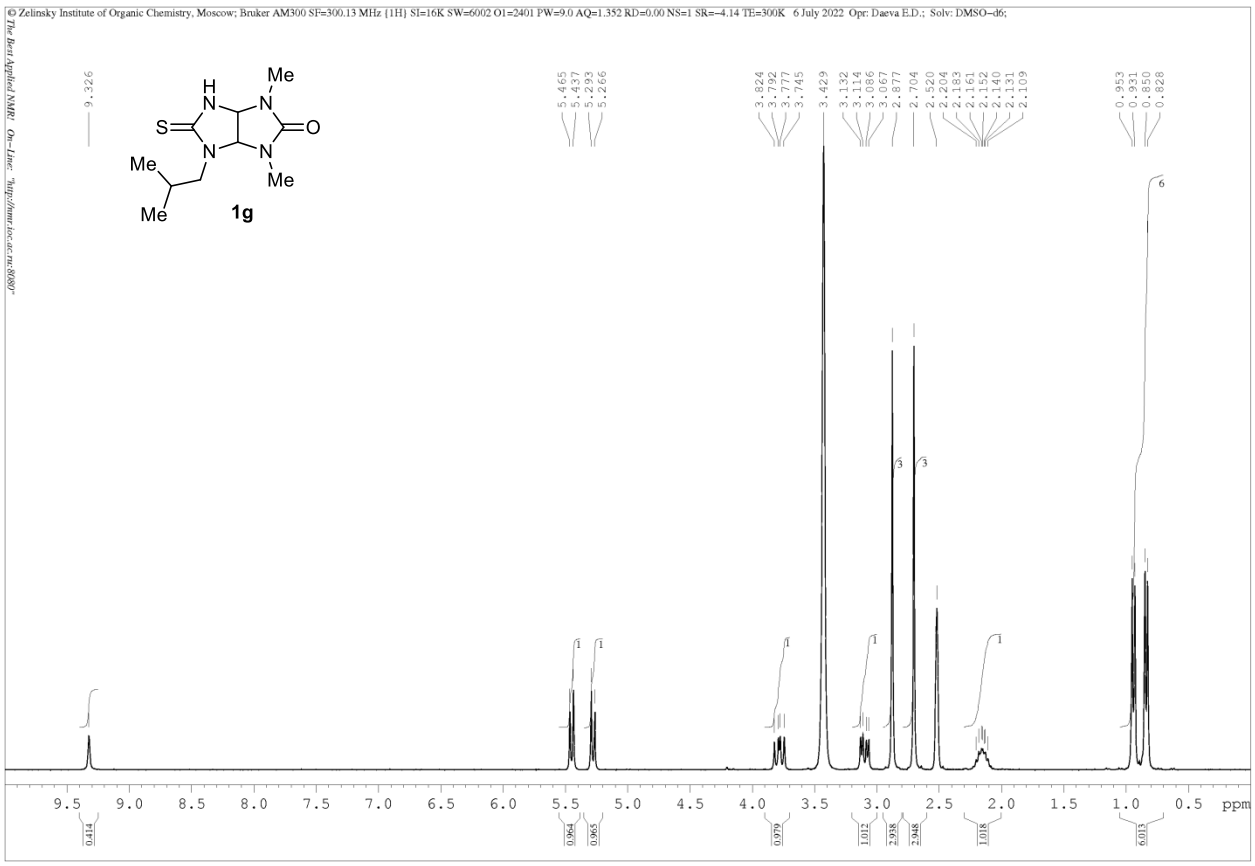
Pr
N
S
O
N
N
Et
Pr
1f
ppm


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6002 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=1 SR=-4.14 TE=300K 6 July 2022 Opr: Daeva E.D.; Solv: DMSO-d6;
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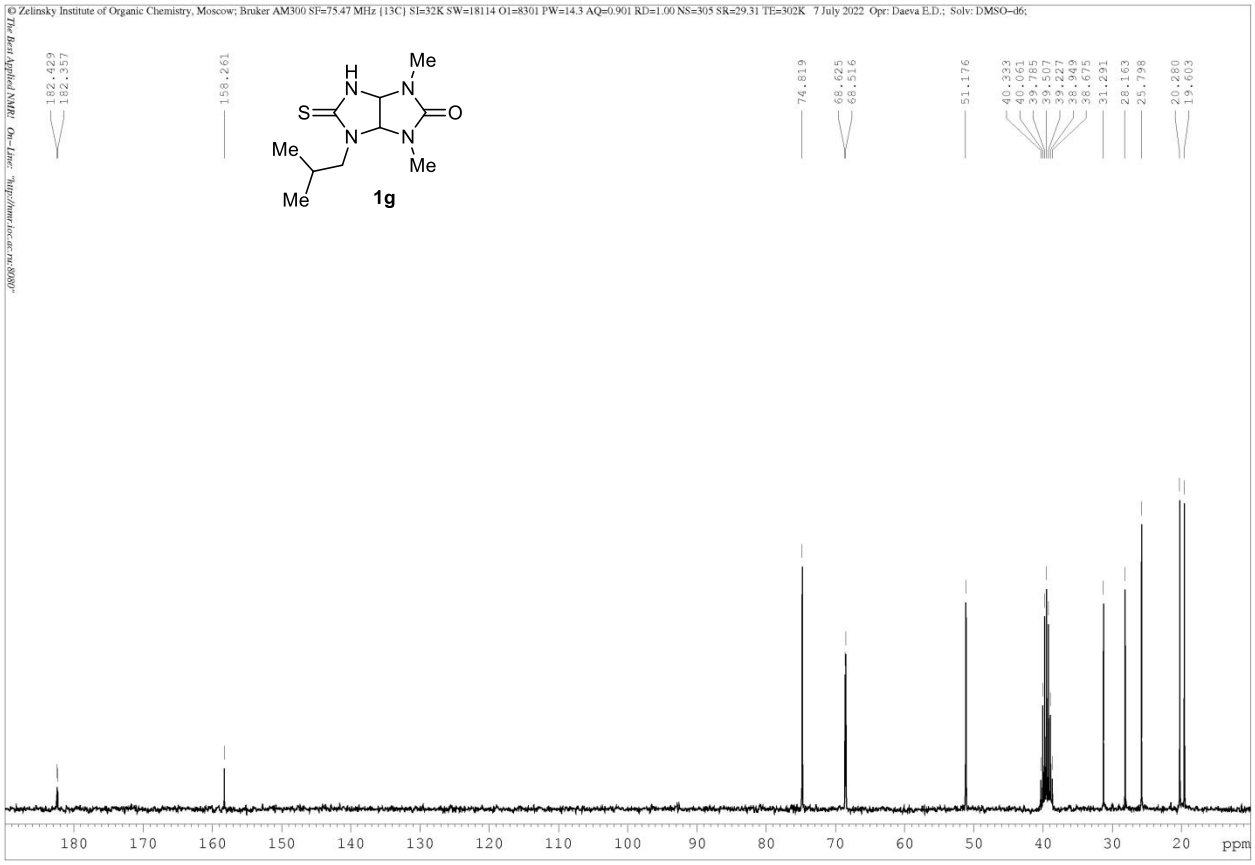
1g
ppm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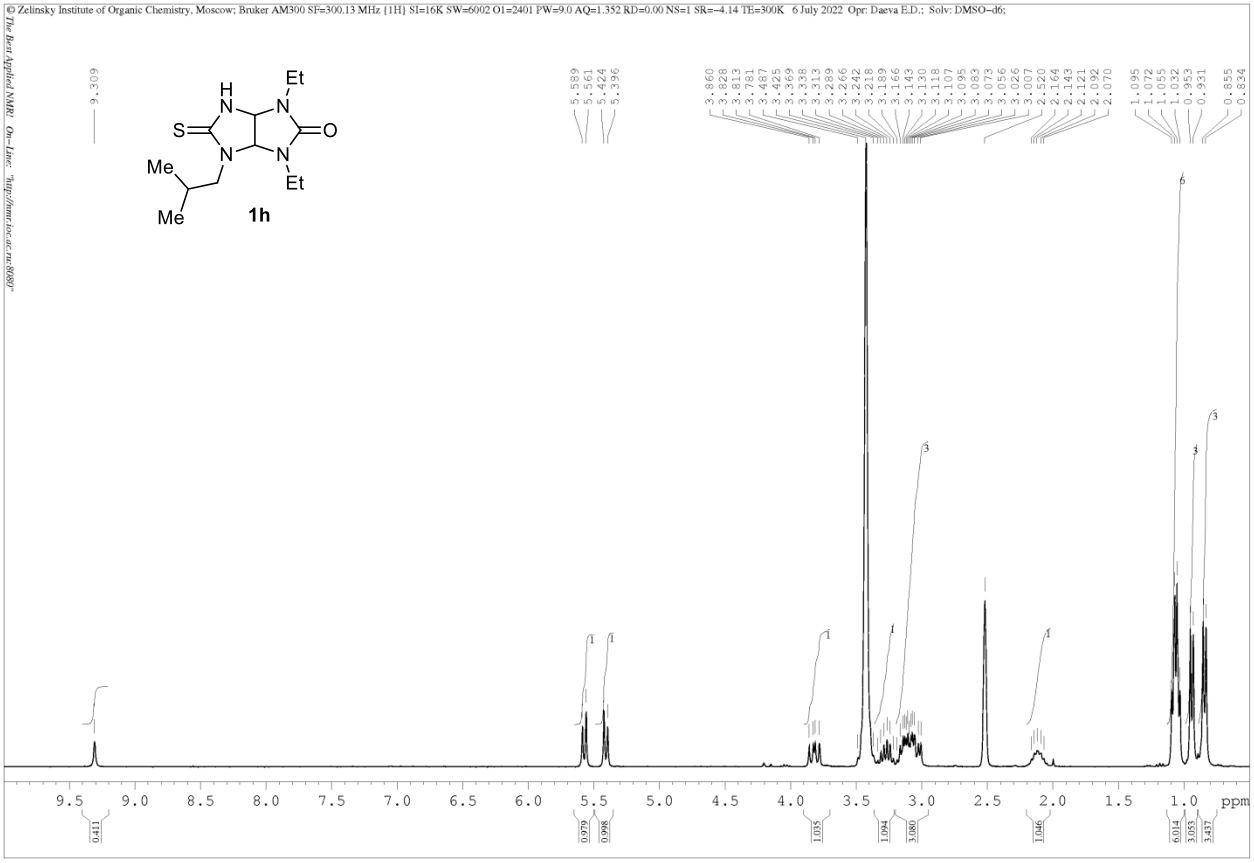
© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6002 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=1 SR=-4.14 TE=300K 6 July 2022 Opr: Daeva E.D.; Solv: DMSO-d6;
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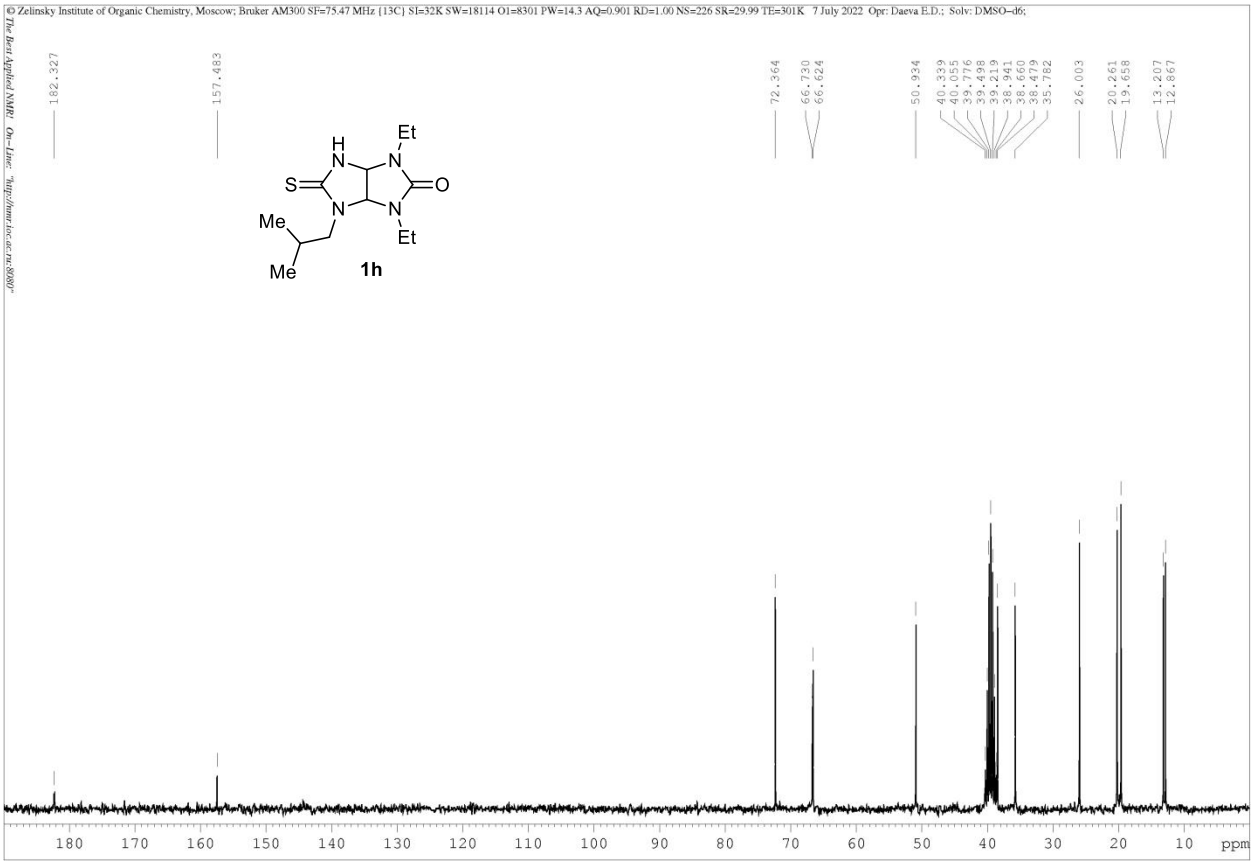
1h
ppm


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=75.47 MHz {13C} SI=32K SW=18114 O1=8301 PW=14.3 AQ=0.901 RD=1.00 NS=226 SR=29.99 TE=301K 7 July 2022 Opr: Daeva E.D.; Solv: DMSO-d6;
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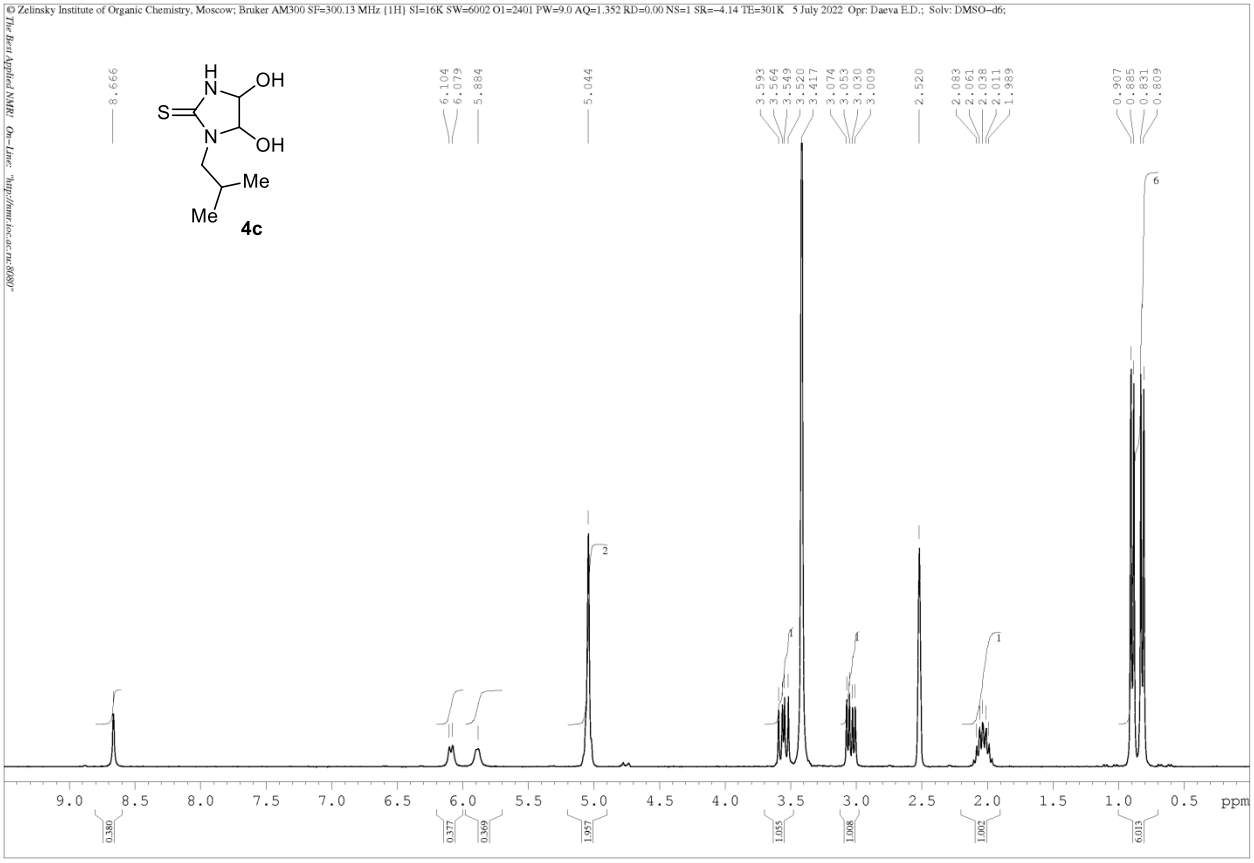
1h
ppm


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz {1H} SI=16K SW=6002 O1=2401 PW=9.0 AQ=1.352 RD=0.00 NS=1 SR=-4.14 TE=301K 5 July 2022 Opr: Daeva E.D.; Solv: DMSO-d6;
The Best Applied NMR On-Line: http://nmr.isoc.ac.ru:8080/
8.666
6.104
6.079
5.884
5.044
3.593
3.564
3.549
3.520
3.417
3.074
3.053
3.030
3.009
2.520
2.083
2.061
2.038
2.011
1.989
0.907
0.885
0.831
0.809
OH
OH
Me
Me
4c
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
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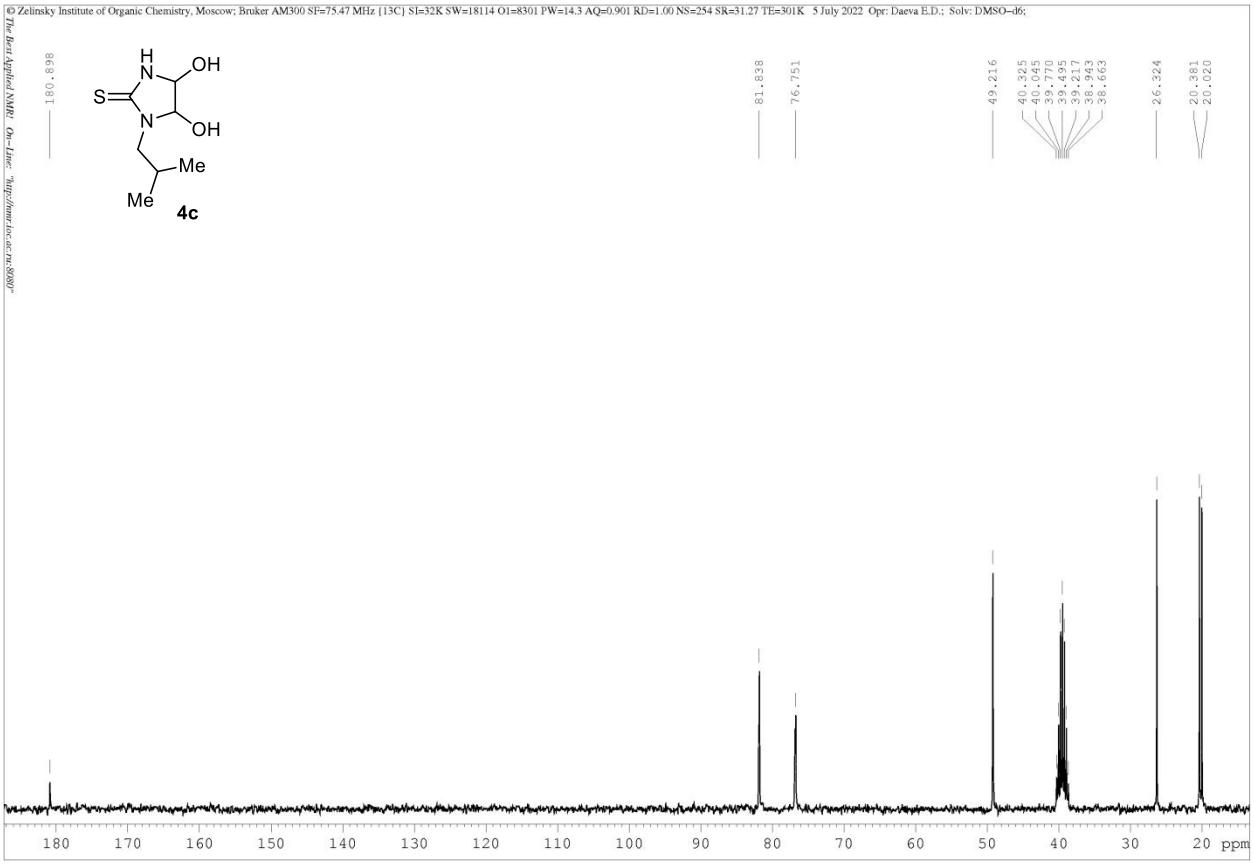
1.002
6.013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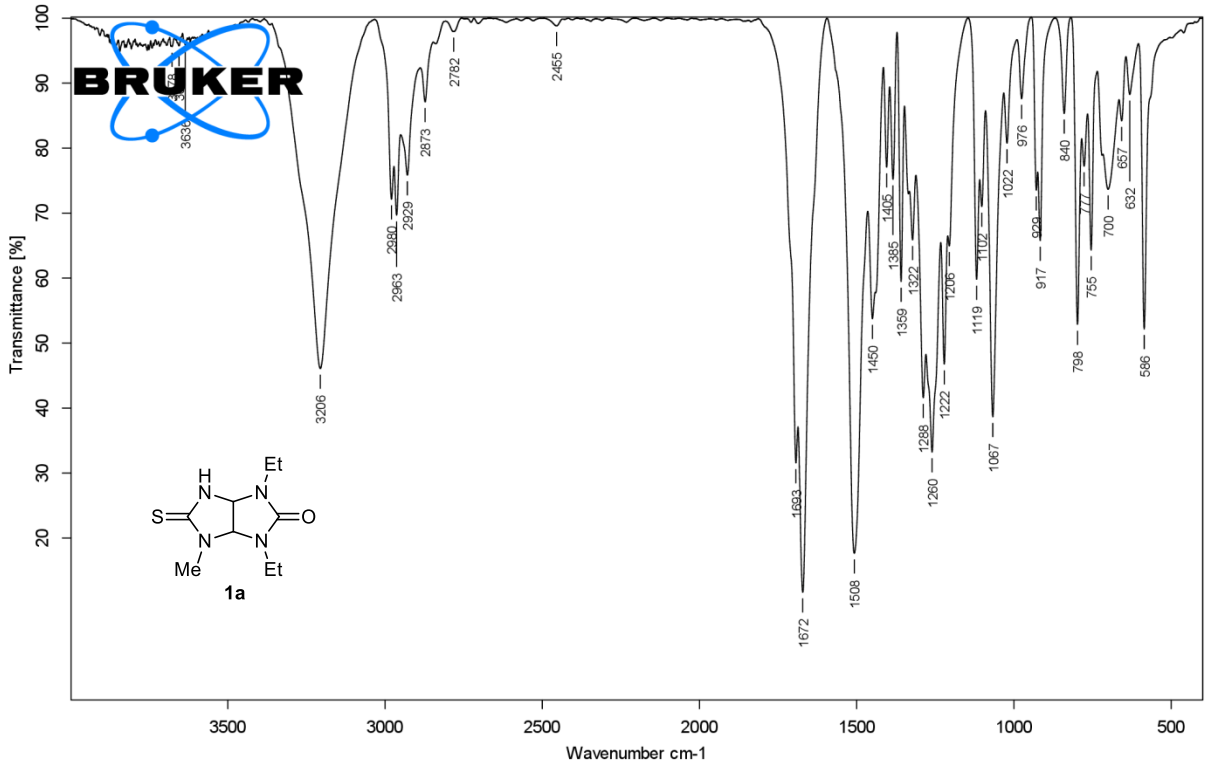
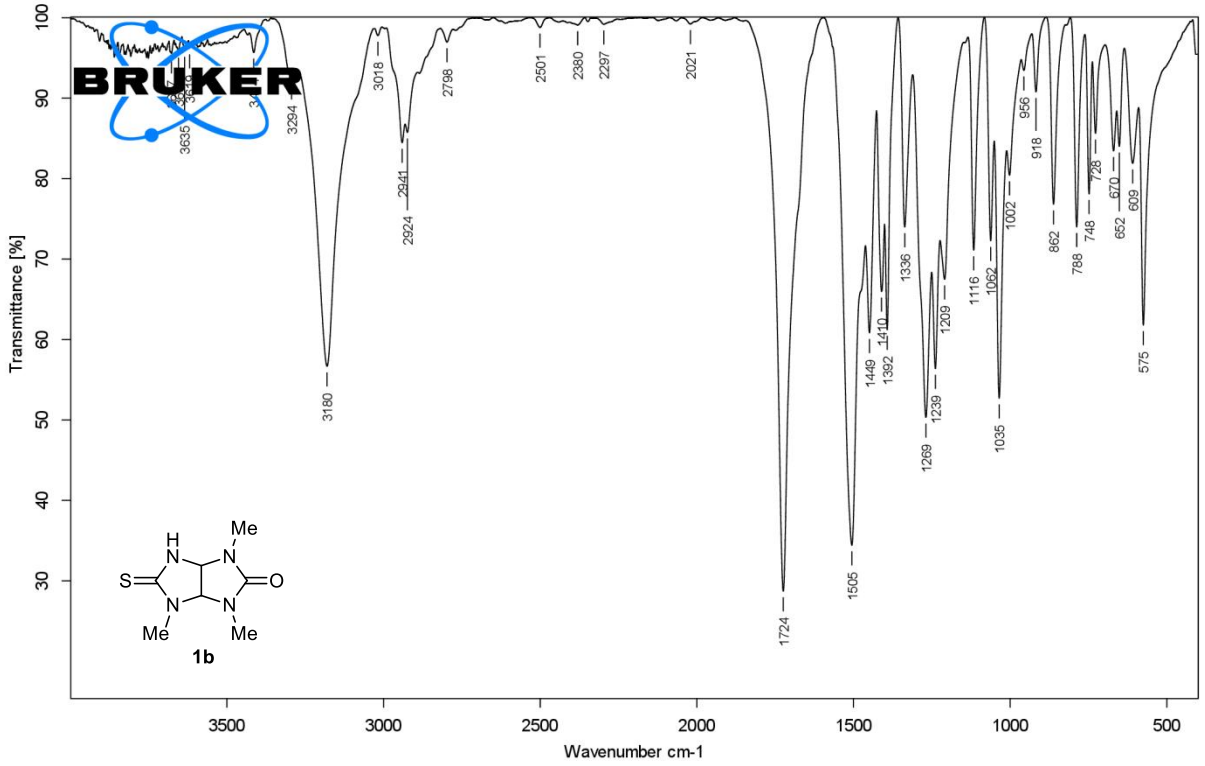
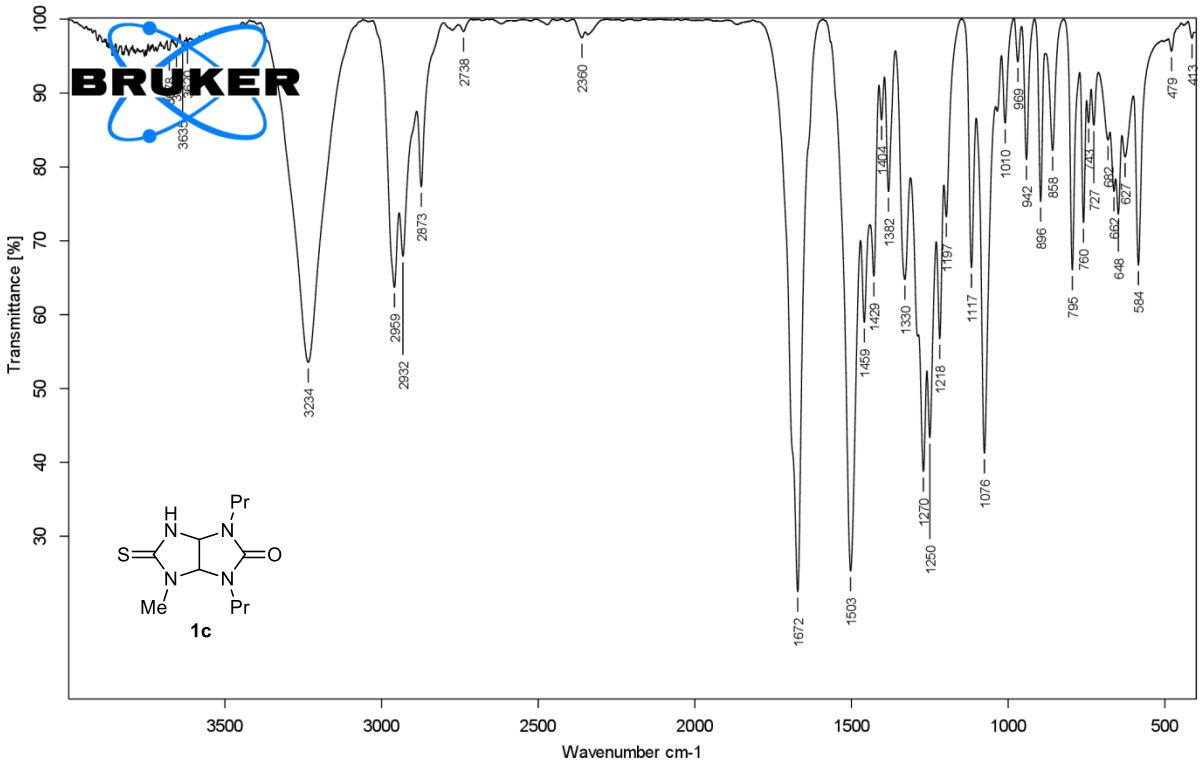
180.898
81.838
76.751
49.216
40.325
40.075
39.770
39.496
39.217
38.943
38.663
26.324
20.381
20.020
OH
OH
Me
Me
4c
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

IR spectra of compounds 1a-h, 4c



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1429
1404
1382
1330
1270
1250
1218
1197
1117
1076
1010
969
942
896
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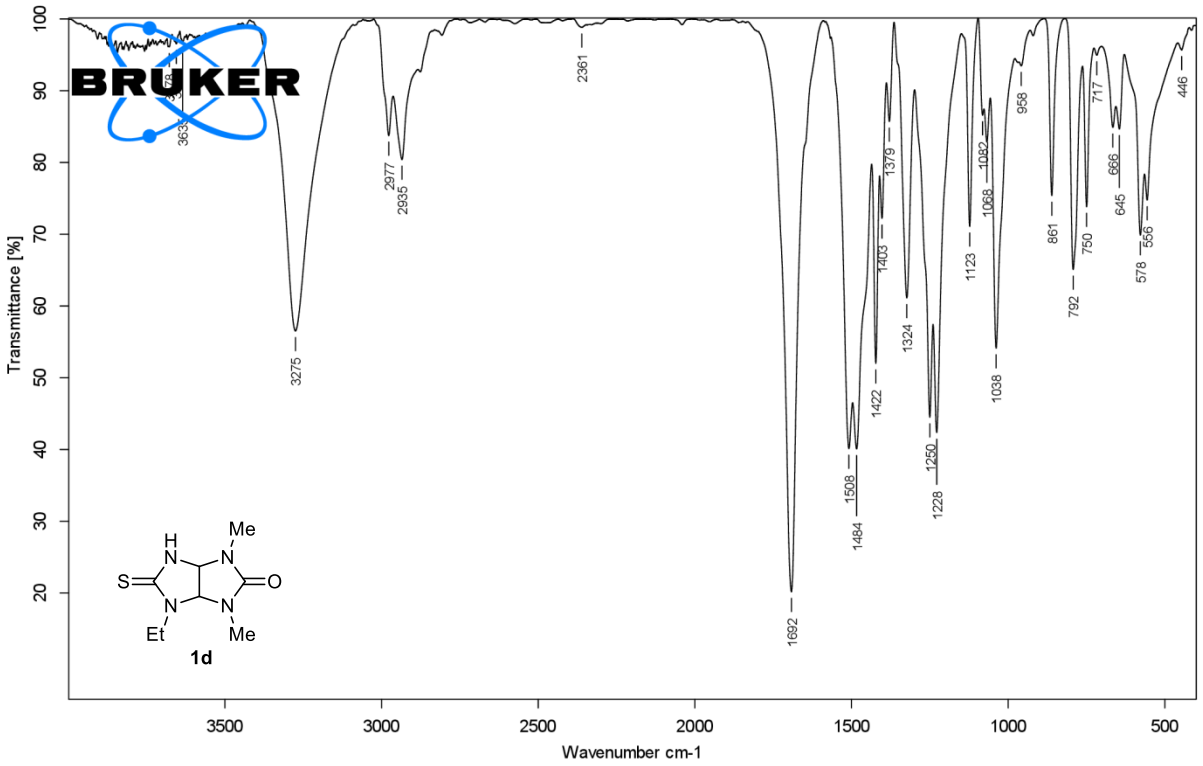
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1508
1484
1422
1403
1379
1324
1250
1228
1123
1082
1068
1038
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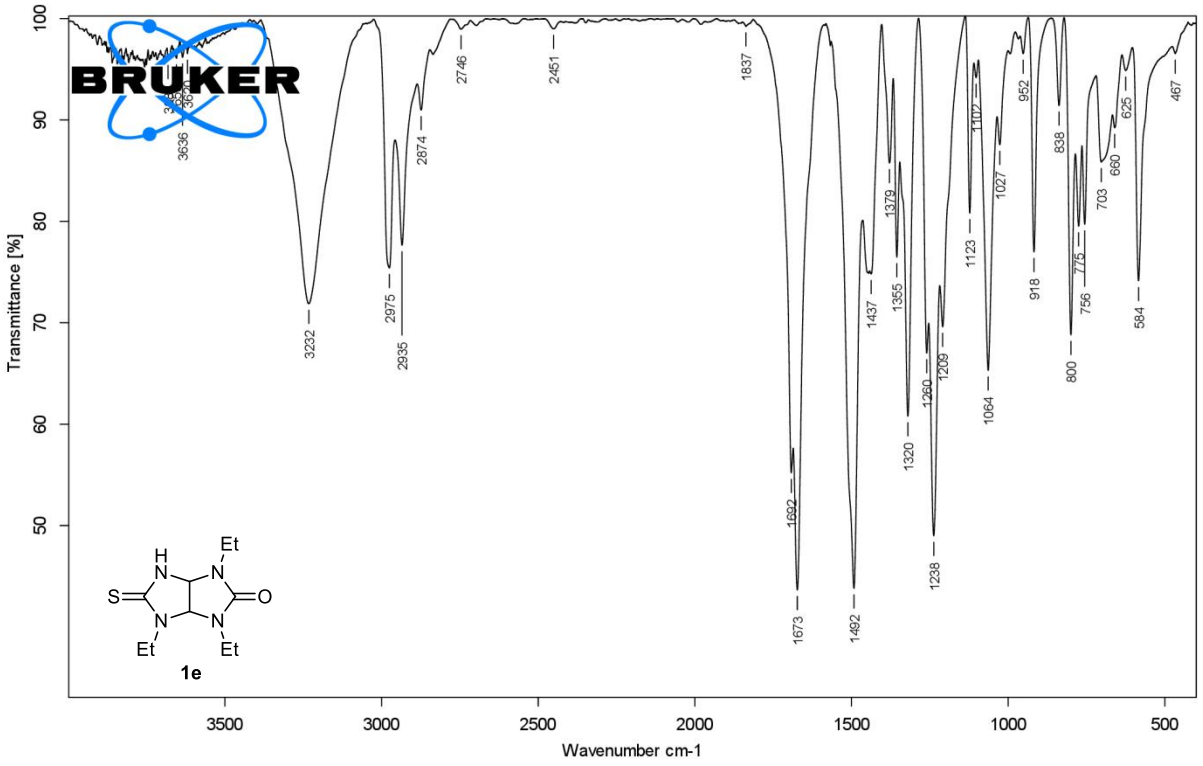
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1355
1320
1260
1238
1209
1123
1102
1064
1027
952
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S
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Et
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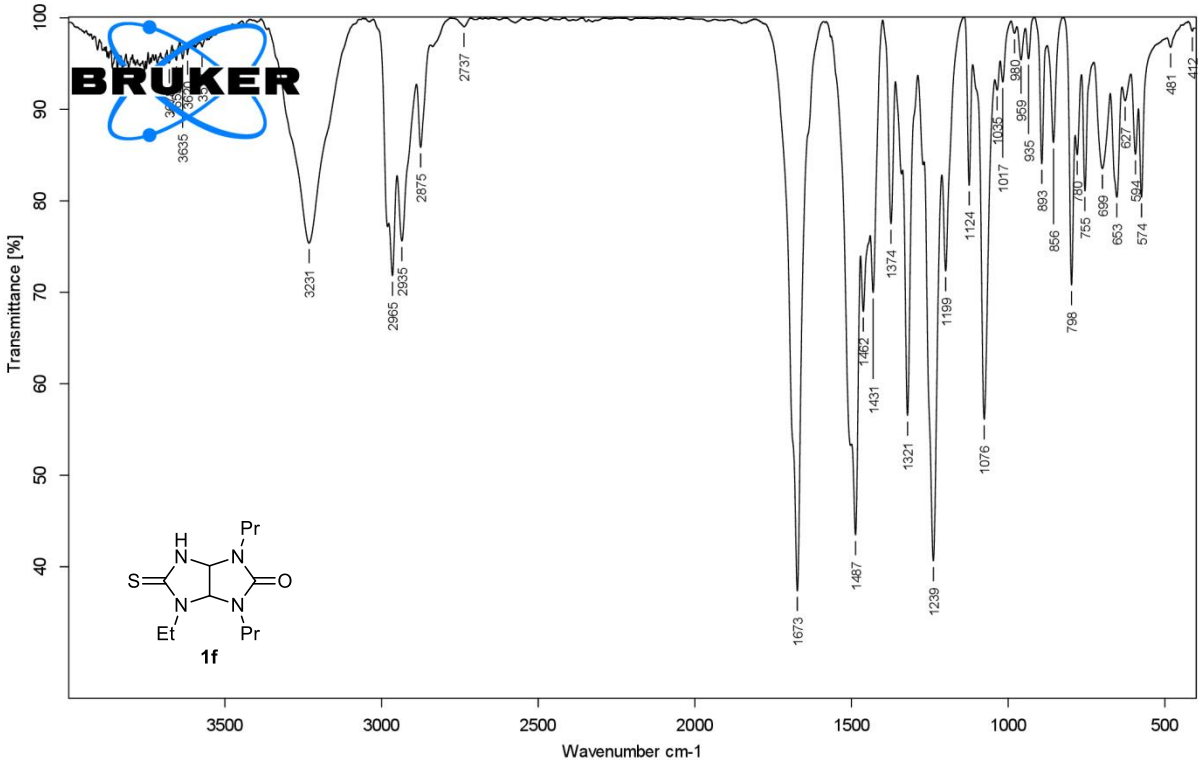
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1035
1017
980
959
935
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856
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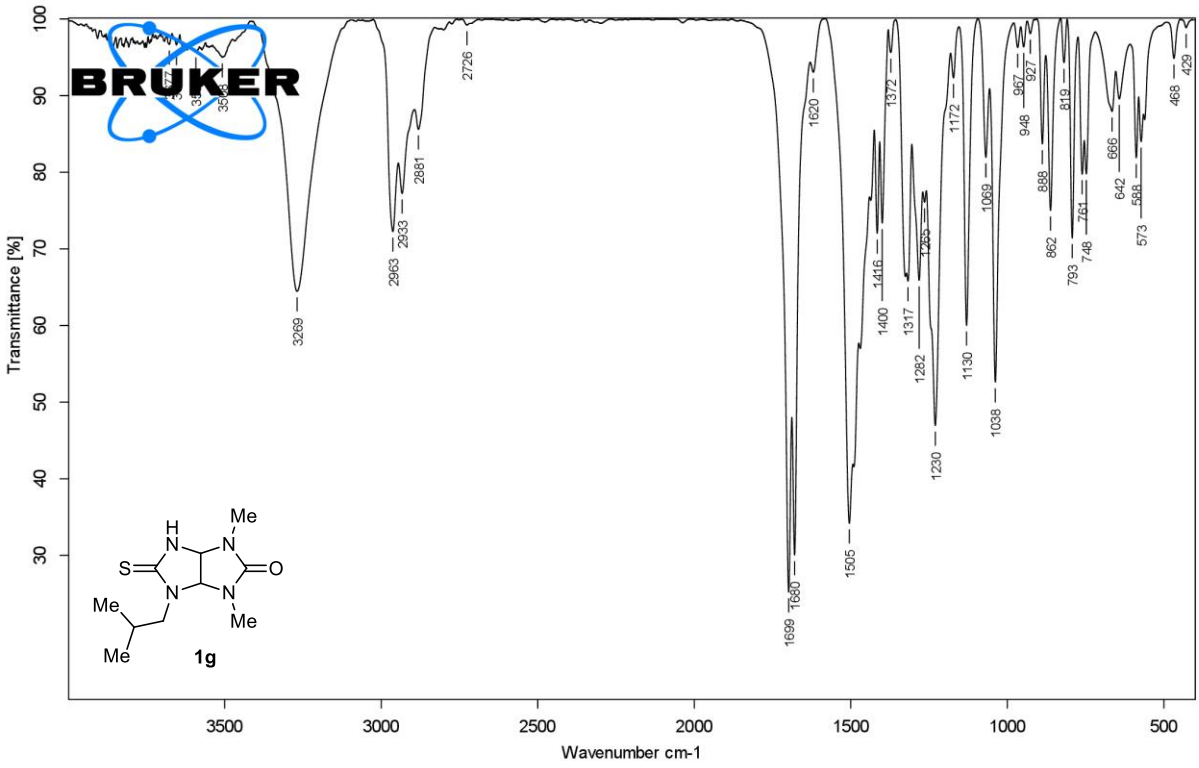
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1038
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Me
Me
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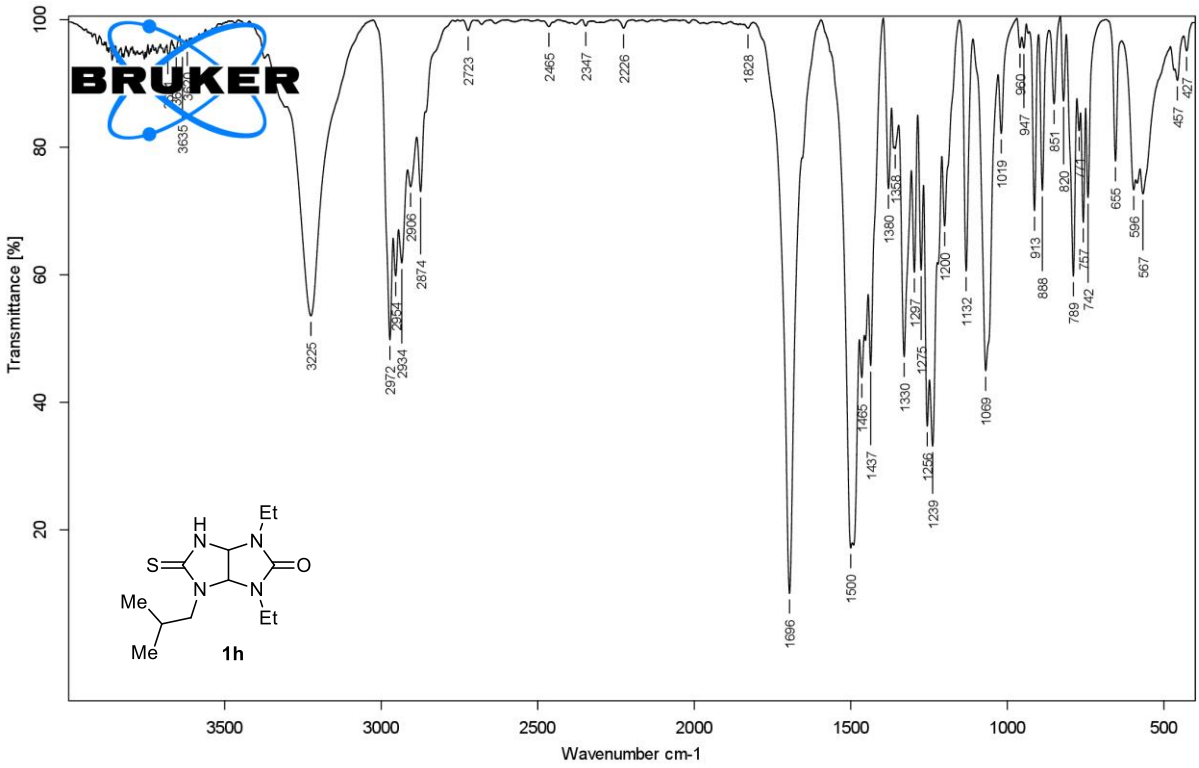
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1437
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1358
1330
1297
1275
1256
1239
1200
1132
1069
1019
960
947
913
888
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820
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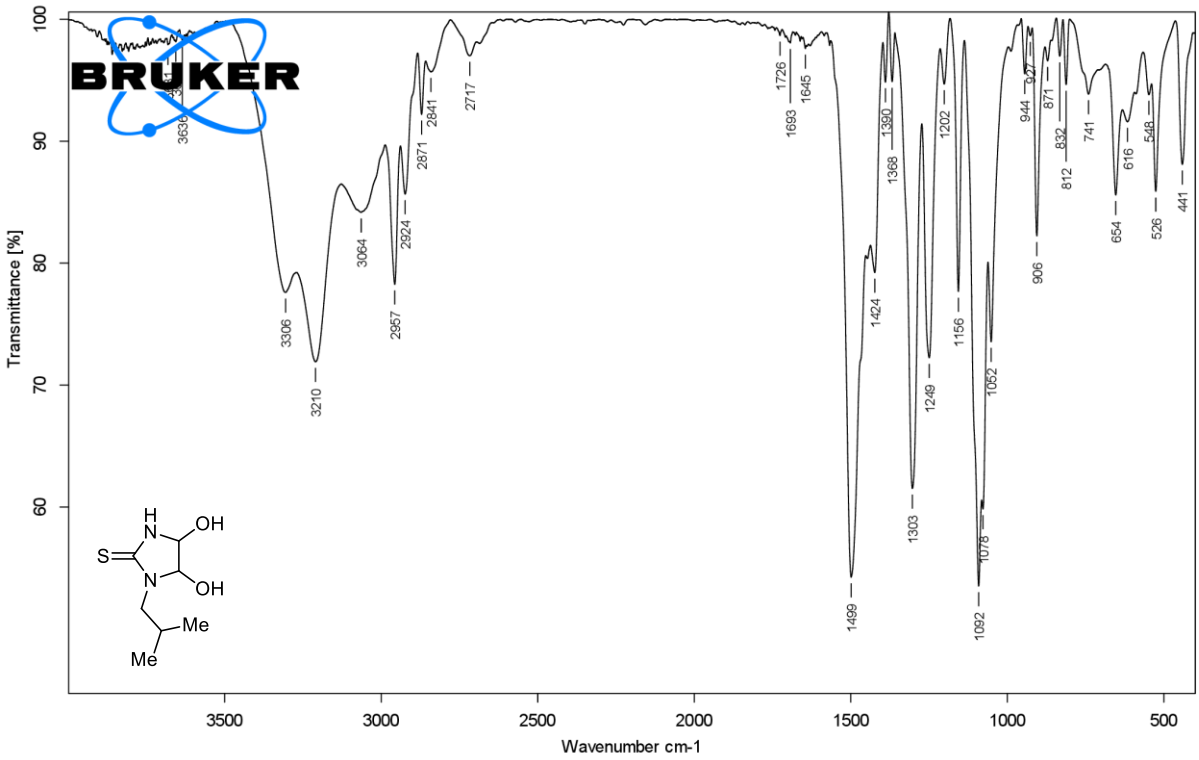
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1645
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1424
1390
1368
1303
1249
1202
1156
1092
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616
548
526
441
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S
N
OH
Me
Me