

## Supporting information

### Regiospecific way to *N*9-alkylated thioxanthines

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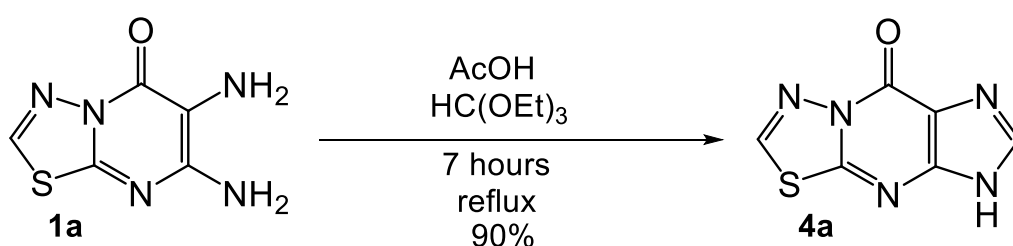
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**Materials and Methods.** Commercial reagents were obtained from Sigma-Aldrich, Acros Organics, or Alfa Aesar and used without any preprocessing. All workup and purification procedures were carried out using analytical-grade solvents. One-dimensional  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were acquired on a Bruker DRX-400 instrument (400 and 101 MHz, respectively) or a Bruker Avance NEO 600 instrument (600 and 151 MHz, respectively), equipped with a Prodigy broadband gradient cryoprobe, utilizing DMSO- $d_6$  and  $\text{CDCl}_3$  as solvents. Chemical shifts are expressed in  $\delta$  (parts per million, ppm) values, and coupling constants are expressed in hertz (Hz). The following abbreviations are used for the multiplicity of NMR signals: br., broadened; s, singlet; d, doublet; t, triplet; and m, multiplet. IR spectra were recorded on a Bruker  $\alpha$  spectrometer equipped with a ZnSe ATR accessory. Elemental analysis was performed on a PerkinElmer PE 2400 elemental analyzer. Mass spectra were recorded with a Shimadzu GCMS-QP 2010 "Ultra" (Kyoto, Japan) mass spectrometer using the electron impact (EI) ionization technique (40–200°C, 70 eV). Melting points were determined on a Stuart SMP3 (Staffordshire, UK) and are uncorrected. The monitoring of the reaction progress was performed using TLC on Silufol UV254 plates.

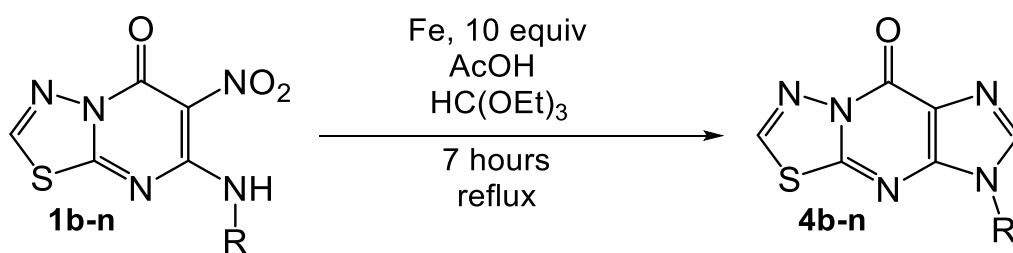
#### 1,3,4-thiadiazolo[3,2-*a*]purin-8-one (4a).



A suspension of 5,6-diamino-1,3,4-thiadiazolo[3,2-*a*]pyrimidin-7-one **2** (0.002 mol, 0.361 g) in AcOH (4 mL) and  $\text{HC(OEt)}_3$  (4 mL) in a round-bottom flask was refluxed for 7 hours, cooled to room temperature and filtered to give 0.347 g (90%) of **4a**.

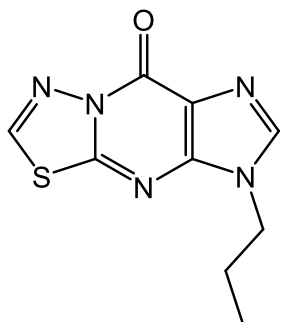
Recrystallize from AcOH; Pale yellow powder (347 mg, 90% yield);  $R_f$  (AcOEt) = 0.2; mp > 300 °C; IR (neat) 3067, 3015, 2975, 1667, 1633, 1593, 1548, 1497, 1464, 1418, 1356, 1204, 1116, 1042, 969, 916, 861, 772, 688  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz, DMSO- $d_6$ )  $\delta$  = 13.84 (s, 1H), 9.32 (s, 1H), 8.14 (s, 1H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz, DMSO- $d_6$ )  $\delta$  = 159.2, 158.6, 147.7, 143.0, 140.4, 113.7. MS (EI, 70 eV)  $m/z$ :  $[\text{M}^+]$  Calcd for  $\text{C}_6\text{H}_3\text{N}_5\text{OS}$  193, Found 193

#### General procedure for the synthesis of 4b-n



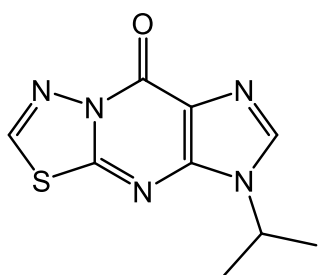
To a mixture of thiadiazolopyrimidone **1b-n** (0.01 mol, 1 equiv), AcOH (50 mL), and  $\text{HC(OEt)}_3$  (50 mL) in a round-bottom flask at 100°C was added Fe dust (0.1 mol, 5.58 g, 10 equiv) in small portions. The resulting suspension was refluxed for 7 hours, filtered while hot and mother liquor was concentrated under vacuum. The residue was quenched with water (100 mL) and extracted with  $\text{CHCl}_3$  (4 × 30 mL). The combined organic layers were dried with anhydrous  $\text{Na}_2\text{SO}_4$  and then concentrated under vacuum to give thiadiazolopurines **4a**, **4b**, **4d-4j**, **4m**. In case of heterocycles **4c**, **4k**, **4l** after residue was quenched with water the resulting precipitate was filtered to give desired product.

**5-*n*-propyl-1,3,4-thiadiazolo[3,2-*a*]purin-8-one (4b).**



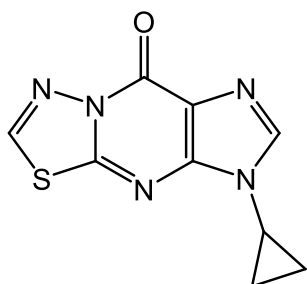
Recrystallized from H<sub>2</sub>O; Pale yellow solid (2.09 g, 89% yield); R<sub>f</sub> (CHCl<sub>3</sub>-MeOH, 4-1) = 0.9; mp 223-225 °C; IR (neat) 3069, 2968, 1714, 1538, 1488, 1369, 1214, 1175, 1062, 852, 765, 749, 654 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>) δ = 9.23 (s, 1H), 8.06 (s, 1H), 4.13 (t, J = 7.2 Hz, 2H), 1.84-1.90 (m, 2H), 0.93 (t, J = 7.2 Hz, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>) δ = 156.9, 152.9, 148.4, 144.2, 140.3, 121.0, 46.0, 23.6, 11.2. MS (EI, 70 eV) m/z: [M<sup>+</sup>] Calcd for C<sub>9</sub>H<sub>9</sub>N<sub>5</sub>OS 235; Found 235. Anal. Calcd. For C<sub>9</sub>H<sub>9</sub>N<sub>5</sub>OS: C, 45.95; H, 3.86; N, 29.77; found: C, 45.97; H, 3.90; N, 29.69

**5-*iso*-propyl-1,3,4-thiadiazolo[3,2-*a*]purin-8-one (4c).**



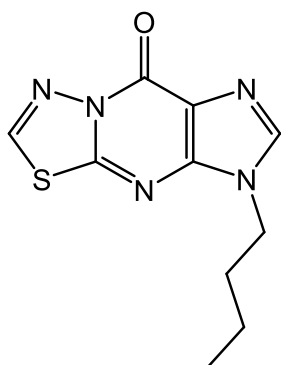
Recrystallized from H<sub>2</sub>O; Pale yellow solid (1.99 g, 85% yield); R<sub>f</sub> (CHCl<sub>3</sub>-MeOH, 4-1) = 0.9; mp 209-211 °C; IR (neat) 3068, 2942, 1725, 1537, 1492, 1362, 1317, 1226, 1179, 859, 764, 654 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>) δ = 9.23 (s, 1H), 8.17 (s, 1H), 4.76 (m, 1H), 1.56-1.58 (d, J = 7.2 Hz, 6H). <sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>) δ = 156.7, 153.0, 147.9, 144.6, 137.9, 121.2, 47.7, 22.8 (2C). MS (EI, 70 eV) m/z: [M<sup>+</sup>] Calcd for C<sub>9</sub>H<sub>9</sub>N<sub>5</sub>OS 235; Found 235. Anal. Calcd. For C<sub>9</sub>H<sub>9</sub>N<sub>5</sub>OS: C, 45.95; H, 3.86; N, 29.77; found: C, 46.00; H, 3.86; N, 29.65

**5-*cyclo*-propyl-1,3,4-thiadiazolo[3,2-*a*]purin-8-one (4d).**



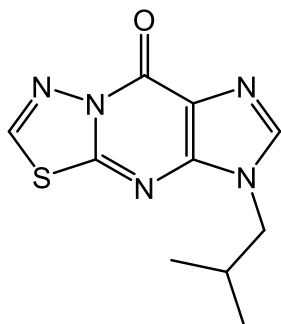
Recrystallized from H<sub>2</sub>O; White powder (1.75 g, 75% yield); R<sub>f</sub> (EtOAc) = 0.5; mp 272-274 °C; IR (neat) 3077, 1709, 1531, 1488, 1429, 1383, 1315, 1233, 1179, 1035, 896, 877, 853, 837, 767, 746, 681, 655 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>) δ = 9.24 (s, 1H), 8.00 (s, 1H), 3.45-3.51 (m, 1H), 1.13 (m, 4H). <sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, DMSO-d<sub>6</sub>) δ = 157.4, 151.9, 149.0, 146.7, 140.5, 120.0, 25.3, 5.4 (2C). MS (EI, 70 eV) m/z: [M<sup>+</sup>] Calcd for C<sub>9</sub>H<sub>7</sub>N<sub>5</sub>OS 233; Found 233. Anal. Calcd. For C<sub>9</sub>H<sub>7</sub>N<sub>5</sub>OS: C, 46.34; H, 3.03; N, 30.03; found: C, 46.28; H, 2.98; N, 29.93

**5-*n*-butyl-1,3,4-thiadiazolo[3,2-*a*]purin-8-one (4e).**



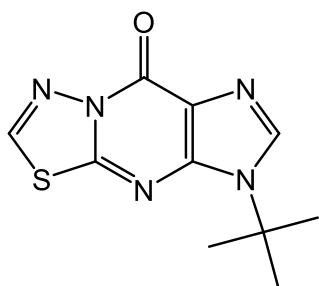
Recrystallized from H<sub>2</sub>O; White crystals (2.24 g, 90% yield); R<sub>f</sub> (CHCl<sub>3</sub>-MeOH, 4-1) = 0.95; mp 171-173 °C; IR (neat) 3055, 2955, 2869, 1706, 1534, 1494, 1371, 1295, 1260, 1231, 1203, 1176, 1041, 985, 848, 768, 743, 656 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>) δ = 9.22 (s, 1H), 8.04 (s, 1H), 4.16 (t, J = 7.2 Hz, 2H), 1.80-1.86 (m, 2H), 1.31-1.37 (m, 2H), 0.96 (t, J = 7.2 Hz, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>) δ = 156.9, 152.9, 148.4, 144.1, 140.3, 121.1, 44.1, 32.2, 19.9, 13.6. MS (EI, 70 eV) m/z: [M<sup>+</sup>] Calcd for C<sub>10</sub>H<sub>11</sub>N<sub>5</sub>OS 249; Found 249. Anal. Calcd. For C<sub>10</sub>H<sub>11</sub>N<sub>5</sub>OS: C, 48.18; H, 4.45; N, 28.09; found: C, 48.11; H, 4.45; N, 28.01

**5-*iso*-butyl-1,3,4-thiadiazolo[3,2-*a*]purin-8-one (4f).**



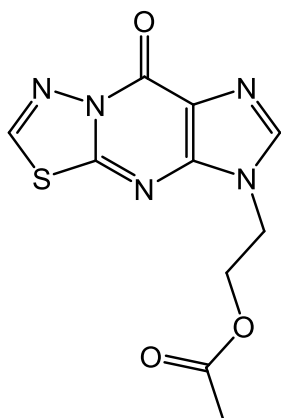
Recrystallized from H<sub>2</sub>O; Pale yellow powder (2.23 g, 90% yield); R<sub>f</sub> (CHCl<sub>3</sub>-MeOH, 4-1) = 0.95; mp 197-198 °C; IR (neat) 3068, 2956, 2871, 1716, 1540, 1486, 1369, 1352, 1289, 1219, 1175, 1107, 1060, 881, 852, 765, 745, 728, 692, 652 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>) δ = 9.23 (s, 1H), 8.03 (s, 1H), 3.98 (d, J = 7.2 Hz, 2H), 2.19-2.25 (m, 1H), 0.92 (d, J = 7.2 Hz, 6H). <sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, DMSO-d<sub>6</sub>) δ = 157.6, 152.2, 148.1, 147.1, 141.3, 119.8, 50.3, 28.5, 19.5 (2C). MS (EI, 70 eV) m/z: [M<sup>+</sup>] Calcd for C<sub>10</sub>H<sub>11</sub>N<sub>5</sub>OS 249; Found 249. Anal. Calcd. For C<sub>10</sub>H<sub>11</sub>N<sub>5</sub>OS: C, 48.18; H, 4.45; N, 28.09; found: C, 48.25; H, 4.39; N, 28.20

**5-*tert*-butyl-1,3,4-thiadiazolo[3,2-*a*]purin-8-one (4j).**



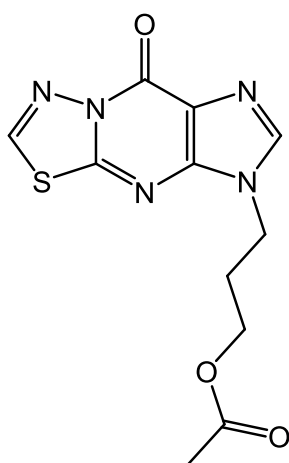
Recrystallized from H<sub>2</sub>O; Pale yellow powder (1.86 g, 75% yield); R<sub>f</sub> (CHCl<sub>3</sub>-MeOH, 4-1) = 0.95; mp 205-207 °C; IR (neat) 3061, 2932, 1715, 1532, 1488, 1354, 1328, 1221, 1026, 893, 857, 767, 749, 653 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>) δ = 9.23 (s, 1H), 8.06 (s, 1H), 1.76 (s, 9H). <sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>) δ = 155.3, 153.1, 148.3, 144.6, 138.1, 122.6, 58.1, 29.3 (3C). MS (EI, 70 eV) m/z: [M<sup>+</sup>] Calcd for C<sub>10</sub>H<sub>11</sub>N<sub>5</sub>OS 249; Found 249. Anal. Calcd. For C<sub>10</sub>H<sub>11</sub>N<sub>5</sub>OS: C, 48.18; H, 4.45; N, 28.09; found: C, 48.17; H, 4.44; N, 28.18

**5-(2-Acetoxyethyl)-1,3,4-thiadiazolo[3,2-*a*]purin-8-one (4h).**



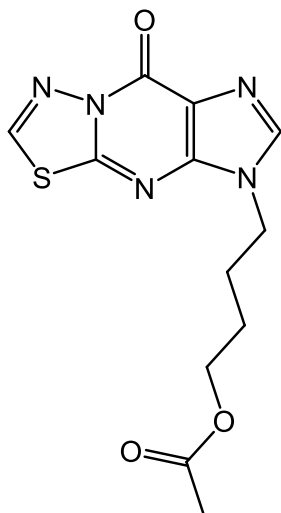
Recrystallized from *iso*-propanol; Pale yellow crystals (2.23 g, 80% yield); R<sub>f</sub> (CHCl<sub>3</sub>-MeOH, 4-1) = 0.9; mp 183-185 °C; IR (neat) 3079, 1703, 1593, 1504, 1372, 1275, 1236, 1188, 1046, 888, 857, 843, 770, 749, 695, 657 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>) δ = 9.24 (s, 1H), 8.08 (s, 1H), 4.38-4.44 (m, 4H), 1.98 (s, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, DMSO-d<sub>6</sub>) δ = 170.1, 157.9, 152.1, 148.2, 147.2, 141.2, 119.8, 62.0, 42.6, 20.5. MS (EI, 70 eV) m/z: [M<sup>+</sup>] Calcd for C<sub>10</sub>H<sub>9</sub>N<sub>5</sub>O<sub>3</sub>S 279; Found 279. Anal. Calcd. For C<sub>10</sub>H<sub>9</sub>N<sub>5</sub>O<sub>3</sub>S: C, 43.01; H, 3.25; N, 25.08; found: C, 42.97; H, 3.28; N, 24.90

**5-(3-Acetoxypropyl)-1,3,4-thiadiazolo[3,2-*a*]purin-8-one (4i).**



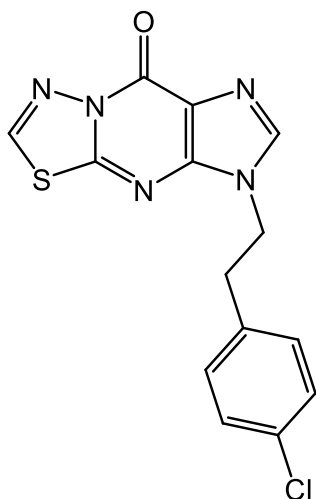
Recrystallized from *iso*-propanol; Pale yellow powder (2.40 g, 82% yield); R<sub>f</sub> (CHCl<sub>3</sub>-MeOH, 4-1) = 0.9; mp 174-176 °C; IR (neat) 3067, 1729, 1701, 1535, 1488, 1439, 1363, 1342, 1249, 1226, 1179, 1030, 960, 940, 891, 877, 845, 768, 746, 692, 656 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ = 8.83 (s, 1H), 7.82 (s, 1H), 4.28 (t, J = 6.8 Hz, 2H), 4.10 (t, J = 6.8 Hz, 2H), 2.23 (m, 2H), 2.03 (s, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>) δ = 170.9, 157.2, 152.8, 148.4, 144.5, 140.4, 121.1, 61.1, 41.4, 29.2, 20.9. MS (EI, 70 eV) m/z: [M<sup>+</sup>] Calcd for C<sub>11</sub>H<sub>11</sub>N<sub>5</sub>O<sub>3</sub>S 293; Found 293. Anal. Calcd. For C<sub>11</sub>H<sub>11</sub>N<sub>5</sub>O<sub>3</sub>S: C, 45.05; H, 3.78; N, 23.88; found: C, 44.99; H, 3.77; N, 24.02

**5-(3-Acetoxybutyl)-1,3,4-thiadiazolo[3,2-*a*]purin-8-one (4j).**



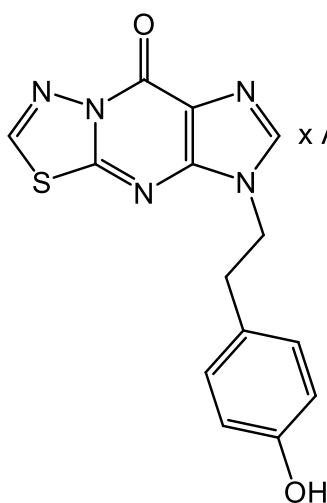
Recrystallized from *iso*-propanol; Gray powder (2.36 g, 77% yield);  $R_f$  (CHCl<sub>3</sub>-MeOH, 4-1) = 0.9; mp 158-159 °C; IR (neat) 3053, 2938, 1703, 1535, 1497, 1371, 1343, 1292, 1235, 1180, 1036, 882, 851, 768, 745, 680, 655 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  = 8.86 (s, 1H), 7.77 (s, 1H), 4.17 (t, *J* = 6.4 Hz, 2H), 4.04 (t, *J* = 6.4 Hz, 2H), 1.97 (s, 3H), 1.91 (m, 2H), 1.62 (m, 2H). <sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  = 171.1, 157.1, 152.8, 148.4, 144.8, 140.2, 120.9, 63.4, 43.8, 26.9, 25.8, 21.0. MS (EI, 70 eV) *m/z*: [M<sup>+</sup>] Calcd for C<sub>12</sub>H<sub>13</sub>N<sub>5</sub>O<sub>3</sub>S 307; Found 307. Anal. Calcd. For C<sub>12</sub>H<sub>13</sub>N<sub>5</sub>O<sub>3</sub>S: C, 46.90; H, 4.26; N, 22.79; found: C, 46.90; H, 4.22; N, 22.80

**5-(2-(*para*-Chlorophenyl)ethyl)-1,3,4-thiadiazolo[3,2-*a*]purin-8-one (4k).**



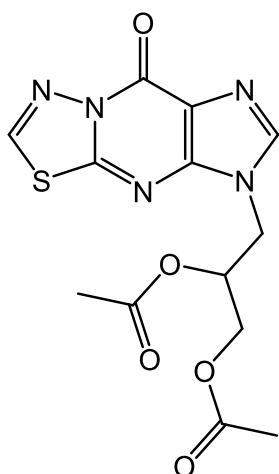
Recrystallized from *iso*-propanol; Grey powder (2.48 g, 75% yield);  $R_f$  (AcOEt) = 0.4; mp 250-251 °C; IR (neat) 3048, 1706, 1531, 1487, 1365, 1286, 1263, 1237, 1218, 1179, 1136, 1088, 1012, 941, 916, 891, 851, 810, 768, 744, 709, 682, 656 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  = 9.26 (s, 1H), 7.92 (s, 1H), 7.28 (d, *J* = 8.4 Hz, 2H), 7.17 (d, *J* = 8.4 Hz, 2H), 4.40 (t, *J* = 6.8 Hz, 2H), 3.14 (t, *J* = 6.8 Hz, 2H). <sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  = 157.6, 152.1, 147.9, 147.1, 140.8, 136.8, 131.2, 130.6(2C), 128.3 (2C), 119.8, 44.4, 34.4. MS (EI, 70 eV) *m/z*: [M<sup>+</sup>] Calcd for C<sub>14</sub>H<sub>10</sub>N<sub>5</sub>ClOS 331, 333; Found 331, 333. Anal. Calcd. For C<sub>14</sub>H<sub>10</sub>N<sub>5</sub>ClOS: C, 50.68; H, 3.04; N, 21.11; found: C, 50.62; H, 2.97; N, 20.95

**5-(2-(*para*-Hydroxyphenyl)ethyl)-1,3,4-thiadiazolo[3,2-*a*]purin-8-one (4l).**



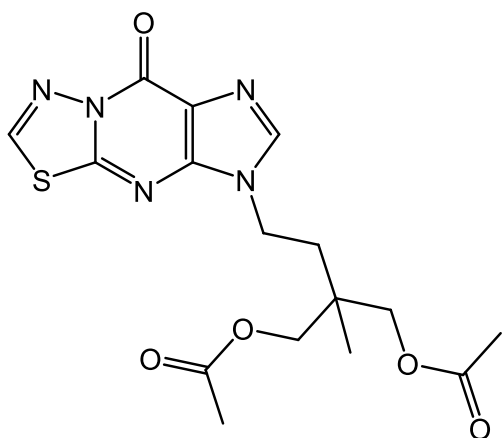
Recrystallized from AcOH; Pale yellow powder (2.79 g, 75% yield);  $R_f$  (AcOEt) = 0.2; mp 234-235 °C; IR (neat) 3100, 2743, 2713, 2600, 1689, 1541, 1504, 1455, 1376, 1266, 1218, 1186, 1146, 1068, 999, 896, 861, 767, 746, 706, 654 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  = 11.95 (s, 1H), 9.27 (s, 1H), 9.21 (s, 1H), 7.90 (s, 1H), 6.91 (d, *J* = 8.4 Hz, 2H), 6.65 (d, *J* = 8.4 Hz, 2H), 4.33 (t, *J* = 6.8 Hz, 2H), 3.00 (t, *J* = 6.8 Hz, 2H), 1.91 (s, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  = 172.0, 157.6, 156.0, 152.2, 147.9, 147.1, 140.9, 129.7 (2C), 127.7, 119.9, 115.2 (2C), 45.0, 34.4, 21.0. MS (EI, 70 eV) *m/z*: [M<sup>+</sup>] Calcd for C<sub>14</sub>H<sub>11</sub>N<sub>5</sub>O<sub>2</sub>S 313; Found 313. Anal. Calcd. For C<sub>16</sub>H<sub>15</sub>N<sub>5</sub>O<sub>4</sub>S: C, 51.47; H, 4.05; N, 18.76; found: C, 51.60; H, 4.13; N, 18.77

**5-(2,3-Diacetoxypropyl)-1,3,4-thiadiazolo[3,2-*a*]purin-8-one (4m).**



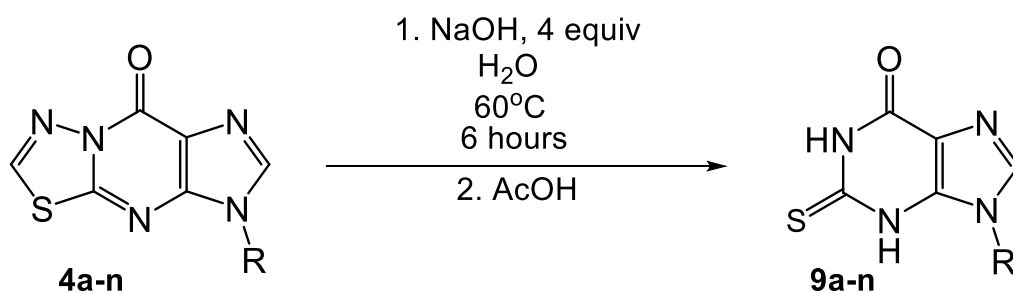
Recrystallized from EtOH; Gray powder (2.73 g, 78% yield);  $R_f$  (EtOAc) = 0.5; mp 165-166 °C; IR (neat) 3068, 1742, 1702, 1535, 1488, 1373, 1292, 1211, 1043, 953, 898, 853, 771, 748, 725, 664  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz, DMSO- $d_6$ )  $\delta$  = 9.25 (s, 1H), 8.06 (s, 1H), 5.33 (m, 1H), 4.46 (m, 2H), 4.26 (m, 1H), 4.08 (m, 1H), 2.04 (s, 3H), 1.97 (s, 3H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz, DMSO- $d_6$ )  $\delta$  = 170.0, 169.6, 157.9, 152.1, 149.3, 147.3, 141.4, 119.7, 69.2, 62.4, 43.4, 20.5 (2C). MS (EI, 70 eV)  $m/z$ :  $[\text{M}^+]$  Calcd for  $\text{C}_{13}\text{H}_{13}\text{N}_5\text{O}_5\text{S}$  351; Found 351. Anal. Calcd. For  $\text{C}_{13}\text{H}_{13}\text{N}_5\text{O}_5\text{S}$ : C, 44.44; H, 3.73; N, 19.93; found: C, 44.51; H, 3.65; N, 20.00

**5-(4-acetoxy-3-acetoxymethyl-3-methyl-butyl)-1,3,4-thiadiazolo[3,2-*a*]purin-8-one (4n).**



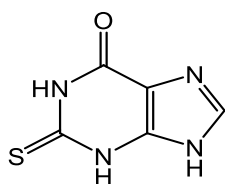
Recrystallized from EtOH; White powder (3.14 g, 80% yield);  $R_f$  (AcOEt) = 0.5; mp 109-110 °C; IR (neat) 3060, 2966, 1736, 1704, 1535, 1489, 1374, 1227, 1031, 896, 850, 769, 747, 686, 658  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz, DMSO- $d_6$ )  $\delta$  = 9.24 (s, 1H), 8.10 (s, 1H), 4.24 (t,  $J$  = 8.0 Hz, 2H), 3.93 (s, 4H), 2.04 (s, 6H), 1.88 (t,  $J$  = 8.0 Hz, 2H), 1.04 (s, 3H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz, DMSO- $d_6$ )  $\delta$  = 170.2 (2C), 157.7, 152.1, 147.8, 147.1, 140.8, 119.9, 67.0 (2C), 36.5, 34.3, 20.6 (3C), 18.6. MS (EI, 70 eV)  $m/z$ :  $[\text{M}^+]$  Calcd for  $\text{C}_{16}\text{H}_{19}\text{N}_5\text{O}_5\text{S}$  393; Found 393. Anal. Calcd. For  $\text{C}_{16}\text{H}_{19}\text{N}_5\text{O}_5\text{S}$ : C, 48.85; H, 4.87; N, 17.80; found: C, 48.95; H, 4.74; N, 17.80

**General procedure for the synthesis of 9a-m**



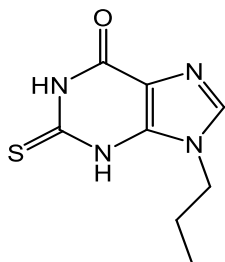
To a solution of NaOH (0.02 mol, 0.8 g, 4 equiv) in  $\text{H}_2\text{O}$  (15 mL) in round-bottom flask was added thiadiazolopurine **4a-n** (0.005 mol, 1 equiv) and the resulting mixture was heated at 60°C for 6 hours. The resulting solution was cooled to room temperature, adjusted with glacial AcOH (0.02 mol, 1.2 mL) to pH-5, precipitate was filtered and washed with  $\text{H}_2\text{O}$  (10 mL) to give thioxanthine **9a-n**.

### Thioxanthine (9a).



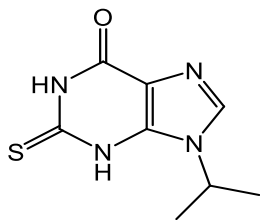
Recrystallized from DMF; White powder (0.63 g, 75% yield);  $R_f$  (CHCl<sub>3</sub>-MeOH, 4-1) = 0.6; mp > 300 °C; IR (neat) 2881, 2770, 2597, 1681, 1631, 1572, 1483, 1420, 1373, 1219, 1162, 1098, 942, 852, 762, 657 cm<sup>-1</sup>; <sup>1</sup>H NMR (600 MHz, DMSO-d<sub>6</sub>)  $\delta$  = 13.63 (s, 1H), 13.26 (s, 1H), 12.21 (s, 1H), 8.07 (s, 1H). <sup>13</sup>C{<sup>1</sup>H} NMR (150 MHz, DMSO-d<sub>6</sub>)  $\delta$  = 173.5, 153.5, 149.1, 141.7, 110.3. MS (EI, 70 eV) m/z: [M<sup>+</sup>] Calcd for C<sub>5</sub>H<sub>4</sub>N<sub>4</sub>OS 168, Found 168. Anal. Calcd. For C<sub>5</sub>H<sub>4</sub>N<sub>4</sub>OS: C, 35.71; H, 2.40; N, 33.32; found: C, 35.77; H, 2.38; N, 33.32

### 9-*n*-propyl-thioxanthine (9b).



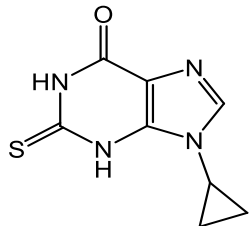
Recrystallized from EtOH; White powder (0.99 g, 95% yield);  $R_f$  (CHCl<sub>3</sub>-MeOH, 4-1) = 0.2; mp 230-231 °C; IR (neat) 3088, 2962, 2873, 1708, 1578, 1499, 1463, 1334, 1290, 1209, 1176, 1111, 871, 831, 773, 726, 693, 659 cm<sup>-1</sup>; <sup>1</sup>H NMR (600 MHz, DMSO-d<sub>6</sub>)  $\delta$  = 13.52 (s, 1H), 12.19 (s, 1H), 7.85 (s, 1H), 4.08 (t, J = 7.2 Hz, 2H), 1.68 (m, 2H), 0.83 (t, J = 7.2 Hz, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (150 MHz, DMSO-d<sub>6</sub>)  $\delta$  = 173.4, 155.9, 140.1, 138.4, 119.0, 45.6, 23.3, 10.5. MS (EI, 70 eV) m/z: [M<sup>+</sup>] Calcd for C<sub>8</sub>H<sub>10</sub>N<sub>4</sub>OS 210, Found 210. Anal. Calcd. For C<sub>8</sub>H<sub>10</sub>N<sub>4</sub>OS: C, 45.70; H, 4.79; N, 26.65; found: C, 45.75; H, 4.91; N, 26.56

### 9-*iso*-propyl-thioxanthine (9c).



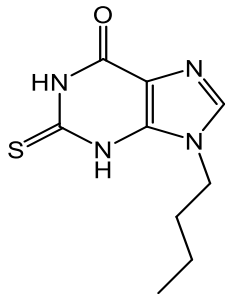
Recrystallized from EtOH; White powder (0.96 g, 92% yield);  $R_f$  (CHCl<sub>3</sub>-MeOH, 4-1) = 0.2; mp 295-296 °C; IR (neat) 3067, 2491, 1701, 1586, 1517, 1429, 1316, 1212, 1184, 1148, 1114, 967, 832, 814, 767, 673 cm<sup>-1</sup>; <sup>1</sup>H NMR (600 MHz, DMSO-d<sub>6</sub>)  $\delta$  = 13.39 (s, 1H), 12.17 (s, 1H), 8.02 (s, 1H), 4.75 (m, 1H), 1.43 (d, J = 6.6 Hz, 6H). <sup>13</sup>C{<sup>1</sup>H} NMR (150 MHz, DMSO-d<sub>6</sub>)  $\delta$  = 173.4, 155.9, 139.6, 135.5, 118.8, 47.2, 22.5 (2C). MS (EI, 70 eV) m/z: [M<sup>+</sup>] Calcd for C<sub>8</sub>H<sub>10</sub>N<sub>4</sub>OS 210, Found 210. Anal. Calcd. For C<sub>8</sub>H<sub>10</sub>N<sub>4</sub>OS: C, 45.70; H, 4.79; N, 26.65; found: C, 45.71; H, 4.83; N, 26.68

### 9-*cyclo*-propyl-thioxanthine (9d).



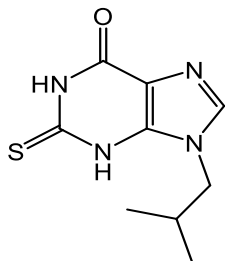
Recrystallized from EtOH; White powder (0.88 g, 85% yield);  $R_f$  (CHCl<sub>3</sub>-MeOH, 4-1) = 0.1; mp 296-297 °C; IR (neat) 3503, 3081, 2776, 1709, 1511, 1476, 1441, 1359, 1329, 1289, 1242, 1219, 1164, 1127, 1106, 1027, 956, 918, 870, 834, 813, 763, 694, 666 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>)  $\delta$  = 13.47 (s, 1H), 11.97 (s, 1H), 7.66 (s, 1H), 3.40 (m, 1H), 1.05 (m, 4H). <sup>13</sup>C{<sup>1</sup>H} NMR (150 MHz, DMSO-d<sub>6</sub>)  $\delta$  = 173.5, 155.9, 141.9, 137.8, 119.2, 26.3, 6.5 (2C). MS (EI, 70 eV) m/z: [M<sup>+</sup>] Calcd for C<sub>8</sub>H<sub>8</sub>N<sub>4</sub>OS 208, Found 208. Anal. Calcd. For C<sub>8</sub>H<sub>8</sub>N<sub>4</sub>OS: C, 46.14; H, 3.87; N, 26.91; found: C, 46.10; H, 3.84; N, 26.98

### 9-*n*-butyl-thioxanthine (9e).



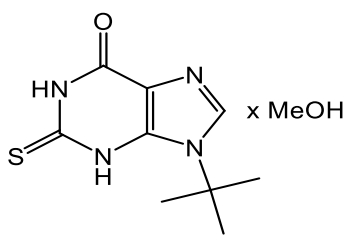
Recrystallized from EtOH; White powder (1.05 g, 94% yield);  $R_f$  (CHCl<sub>3</sub>-MeOH, 4-1) = 0.2; mp 229-230 °C; IR (neat) 3085, 2957, 1695, 1582, 1540, 1505, 1464, 1338, 1299, 1204, 1168, 1115, 872, 834, 774, 763, 723, 660 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>)  $\delta$  = 13.35 (s, 1H), 11.98 (s, 1H), 7.71 (s, 1H), 4.14 (t, J = 7.2 Hz, 2H), 1.68 (m, 2H), 1.24 (m, 2H), 0.95 (t, J = 7.2 Hz, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (150 MHz, DMSO-d<sub>6</sub>)  $\delta$  = 173.4, 155.8, 140.1, 138.4, 119.0, 44.0, 31.9, 19.1, 13.5. MS (EI, 70 eV) m/z: [M<sup>+</sup>] Calcd for C<sub>9</sub>H<sub>12</sub>N<sub>4</sub>OS 224, Found 224. Anal. Calcd. For C<sub>9</sub>H<sub>12</sub>N<sub>4</sub>OS: C, 48.20; H, 5.39; N, 24.98; found: C, 48.24; H, 5.33; N, 25.00

### 9-*iso*-butyl-thioxanthine (9f).



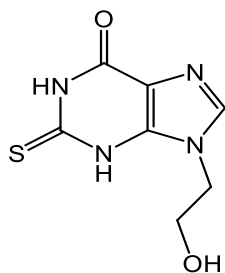
Recrystallized from EtOH; White powder (1.06 g, 95% yield);  $R_f$  (CHCl<sub>3</sub>-MeOH, 4-1) = 0.2; mp 226-227 °C; IR (neat) 3093, 2963, 1711, 1679, 1579, 1543, 1503, 1466, 1371, 1329, 1293, 1211, 1183, 1111, 883, 839, 774, 763, 733, 658 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>)  $\delta$  = 13.37 (s, 1H), 12.00 (s, 1H), 7.68 (s, 1H), 3.98 (d, J = 7.2 Hz, 2H), 2.01 (m, 1H), 0.88 (d, J = 6.6 Hz, 6H). <sup>13</sup>C{<sup>1</sup>H} NMR (150 MHz, DMSO-d<sub>6</sub>)  $\delta$  = 173.4, 155.9, 140.3, 138.8, 119.0, 50.8, 28.7, 19.1 (2C). MS (EI, 70 eV) m/z: [M<sup>+</sup>] Calcd for C<sub>9</sub>H<sub>12</sub>N<sub>4</sub>OS 224, Found 224. Anal. Calcd. For C<sub>9</sub>H<sub>12</sub>N<sub>4</sub>OS: C, 48.20; H, 5.39; N, 24.98; found: C, 48.22; H, 5.39; N, 24.99

### 9-*tert*-butyl-thioxanthine (9g).



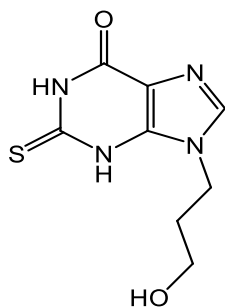
Recrystallized from MeOH; White powder (0.67 g, 60% yield);  $R_f$  (CHCl<sub>3</sub>-MeOH, 4-1) = 0.2; mp > 300 °C; IR (neat) 3055, 2879, 1708, 1580, 1545, 1473, 1372, 1338, 1289, 1202, 1174, 1127, 997, 942, 845, 774, 659 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>)  $\delta$  = 11.98 (s, 1H), 7.95 (s, 1H), 3.19 (s, 3H), 1.68 (s, 9H). <sup>13</sup>C{<sup>1</sup>H} NMR (150 MHz, DMSO-d<sub>6</sub>)  $\delta$  = 174.2, 155.6, 140.6, 135.9, 119.1, 58.0, 48.6, 28.5 (3C). MS (EI, 70 eV) m/z: [M<sup>+</sup>] Calcd for C<sub>9</sub>H<sub>12</sub>N<sub>4</sub>OS 224, Found 224. Anal. Calcd. For C<sub>10</sub>H<sub>16</sub>N<sub>4</sub>O<sub>2</sub>S: C, 46.86; H, 6.29; N, 21.86; found: C, 46.99; H, 6.25; N, 22.00

### 9-(2-hydroxyethyl)-thioxanthine (9h).



Recrystallized from H<sub>2</sub>O; White powder (0.94 g, 89% yield);  $R_f$  (CHCl<sub>3</sub>-MeOH, 4-1) = 0.1; mp 266-267 °C; IR (neat) 3081, 2831, 2326, 1697, 1587, 1541, 1505, 1443, 1327, 1289, 1198, 1153, 1121, 1039, 860, 819, 763, 659 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>)  $\delta$  = 13.19 (s, 1H), 11.97 (s, 1H), 7.65 (s, 1H), 4.89 (br. s, 1H), 4.23 (t, J = 7.2 Hz, 2H), 3.65 (t, J = 7.2 Hz, 2H). <sup>13</sup>C{<sup>1</sup>H} NMR (150 MHz, DMSO-d<sub>6</sub>)  $\delta$  = 173.2, 155.9, 140.6, 138.9, 119.0, 59.7, 47.0. Anal. Calcd. For C<sub>7</sub>H<sub>8</sub>N<sub>4</sub>O<sub>2</sub>S: C, 39.62; H, 3.80; N, 26.40; found: C, 39.63; H, 3.82; N, 26.37

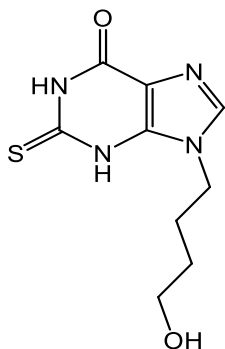
### 9-(3-hydroxypropyl)-thioxanthine (9i).



Recrystallized from H<sub>2</sub>O; Pale yellow powder (0.98 g, 87% yield);  $R_f$  (CHCl<sub>3</sub>-MeOH, 4-1) = 0.1; mp 266-267 °C; IR (neat) 3081, 2817, 1694, 1587, 1507, 1446, 1284, 1199, 1147, 1120, 1045, 904, 829, 762, 722, 659 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>)  $\delta$  = 13.36 (s, 1H), 12.01 (s, 1H), 7.70 (s, 1H), 4.54 (br. s, 1H), 4.21 (t, J = 7.2 Hz, 2H), 3.40 (t, J = 7.2 Hz, 2H), 1.84 (m, 2H). <sup>13</sup>C{<sup>1</sup>H} NMR (150 MHz, DMSO-d<sub>6</sub>)  $\delta$  = 173.3, 155.9, 140.3, 138.5, 119.1, 57.4, 41.6, 32.6. Anal. Calcd. For C<sub>8</sub>H<sub>10</sub>N<sub>4</sub>O<sub>2</sub>S: C, 42.47; H, 4.46; N, 24.76; found: C, 42.45; H, 4.32; N, 24.90

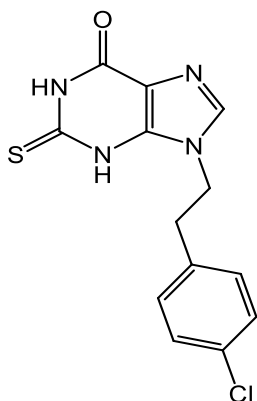


### 9-(4-hydroxybutyl)-thioxanthine (9j).



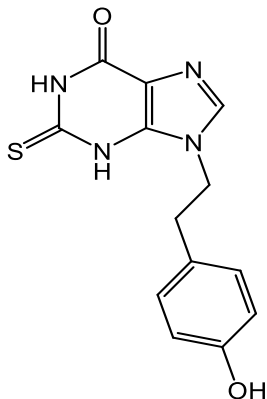
Recrystallized from H<sub>2</sub>O; Gray powder (0.90 g, 75% yield); R<sub>f</sub> (CHCl<sub>3</sub>-MeOH, 4-1) = 0.1; mp 249-250 °C; IR (neat) 3082, 2865, 1698, 1582, 1543, 1505, 1443, 1291, 1195, 1117, 1026, 873, 835, 760, 717, 657 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>) δ = 13.38 (s, 1H), 11.98 (s, 1H), 7.72 (s, 1H), 4.32 (br. s, 1H), 4.15 (t, J = 7.2 Hz, 2H), 3.44 (t, J = 7.2 Hz, 2H), 1.74 (m, 2H), 1.43 (m, 2H). <sup>13</sup>C{<sup>1</sup>H} NMR (150 MHz, DMSO-d<sub>6</sub>) δ = 173.4, 155.9, 140.1, 138.4, 119.0, 60.2, 44.2, 29.2, 26.9. Anal. Calcd. For C<sub>9</sub>H<sub>12</sub>N<sub>4</sub>O<sub>2</sub>S: C, 44.99; H, 5.03; N, 23.32; found: C, 44.96; H, 4.96; N, 23.44

### 9-(2-(para-Chlorophenyl)ethyl)-thioxanthine (9k).



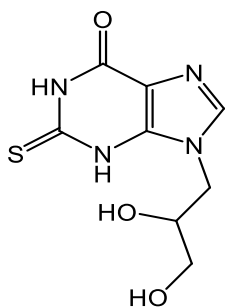
Recrystallized from AcOH; White powder (1.46 g, 95% yield); R<sub>f</sub> (CHCl<sub>3</sub>-MeOH, 4-1) = 0.3; mp 245-246 °C; IR (neat) 3099, 2839, 1698, 1591, 1515, 1490, 1427, 1335, 1184, 1139, 1092, 1056, 1014, 859, 812, 736, 705, 674, 652 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>) δ = 13.42 (s, 1H), 11.99 (s, 1H), 7.59 (s, 1H), 7.29 (d, J = 8.4 Hz, 2H), 7.27 (d, J = 8.4 Hz, 2H), 4.37 (t, J = 7.2 Hz, 2H), 3.00 (t, J = 7.2 Hz, 2H). <sup>13</sup>C{<sup>1</sup>H} NMR (150 MHz, DMSO-d<sub>6</sub>) δ = 173.3, 155.8, 140.0, 138.3, 136.5, 131.4, 130.9 (2C), 128.3 (2C), 118.9, 45.1, 35.1. MS (EI, 70 eV) m/z: [M<sup>+</sup>] Calcd for C<sub>13</sub>H<sub>11</sub>ClN<sub>4</sub>OS 306, 308, Found 306, 308. Anal. Calcd. For C<sub>13</sub>H<sub>11</sub>ClN<sub>4</sub>OS: C, 50.90; H, 3.61; N, 18.26; found: C, 51.01; H, 3.70; N, 18.16

### 9-(2-(para-Hydroxyphenyl)ethyl)-thioxanthine (9l).



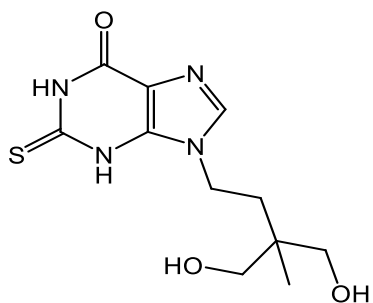
Recrystallized from AcOH; White powder (1.35 g, 94% yield); R<sub>f</sub> (CHCl<sub>3</sub>-MeOH, 4-1) = 0.1; mp 280-281 °C; IR (neat) 3103, 3012, 2874, 1681, 1592, 1513, 1434, 1340, 1250, 1204, 1162, 1140, 836, 768, 721, 662, 647 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>) δ = 13.40 (s, 1H), 11.98 (s, 1H), 9.03 (br. s, 1H), 7.52 (s, 1H), 6.96 (d, J = 8.4 Hz, 2H), 6.65 (d, J = 8.4 Hz, 2H), 4.32 (t, J = 7.2 Hz, 2H), 2.88 (t, J = 7.2 Hz, 2H). <sup>13</sup>C{<sup>1</sup>H} NMR (150 MHz, DMSO-d<sub>6</sub>) δ = 173.3, 156.1, 155.8, 140.0, 138.4, 129.9 (2C), 127.3, 118.8, 115.2 (2C), 45.6, 35.0. MS (EI, 70 eV) m/z: [M<sup>+</sup>] Calcd for C<sub>13</sub>H<sub>12</sub>N<sub>4</sub>O<sub>2</sub>S 288, Found 288. Anal. Calcd. For C<sub>13</sub>H<sub>12</sub>N<sub>4</sub>O<sub>2</sub>S: C, 54.16; H, 4.20; N, 19.43; found: C, 54.11; H, 4.28; N, 19.43

### 9-(2,3-dihydroxypropyl)-thioxanthine (9m).



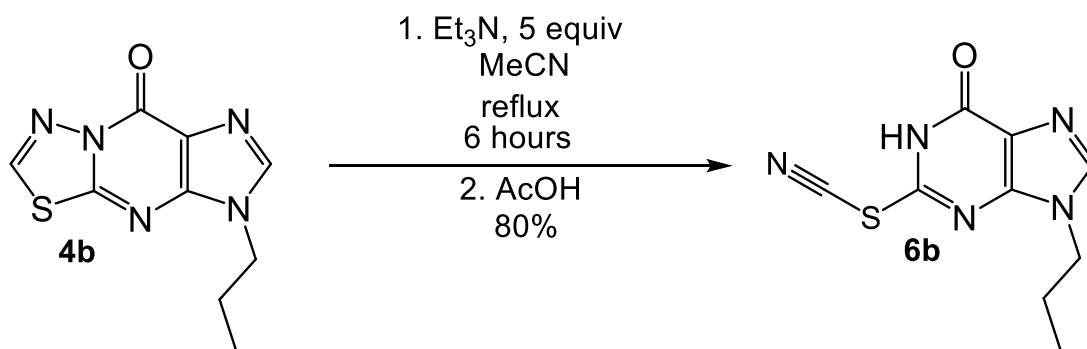
Recrystallized from EtOH; White powder (1.02 g, 84% yield); R<sub>f</sub> (CHCl<sub>3</sub>-MeOH, 4-1) = 0.1; mp 260-261 °C; IR (neat) 3053, 1699, 1577, 1508, 1425, 1288, 1200, 1156, 1115, 1033, 842, 760, 659 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>) δ = 13.09 (s, 1H), 11.95 (s, 1H), 7.63 (s, 1H), 4.98 (br. s, 1H), 4.64 (br. s, 1H), 4.28 (m, 1H), 4.11 (m, 1H), 3.72 (m, 1H), 3.40 (m, 2H). <sup>13</sup>C{<sup>1</sup>H} NMR (150 MHz, DMSO-d<sub>6</sub>) δ = 173.1, 156.0, 140.9, 139.1, 119.0, 70.1, 63.1, 47.5. Anal. Calcd. For C<sub>8</sub>H<sub>10</sub>N<sub>4</sub>O<sub>3</sub>S: C, 39.66; H, 4.16; N, 23.13; found: C, 39.70; H, 4.28; N, 23.02

### 9-(4-hydroxy-3-hydroxymethyl-3-methyl)-thioxanthine (9n).



Recrystallized from H<sub>2</sub>O; Grey powder (1.23 g, 87% yield); R<sub>f</sub> (CHCl<sub>3</sub>-MeOH, 4-1) = 0.1; mp 265-266 °C; IR (neat) 3089, 2867, 1696, 1585, 1543, 1504, 1434, 1289, 1189, 1113, 1009, 842, 759, 693, 657 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>) δ = 13.33 (s, 1H), 12.00 (s, 1H), 7.75 (s, 1H), 4.39 (br. s, 2H), 4.18 (m, 2H), 3.28 (m, 2H), 1.69 (m, 2H), 0.85 (s, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (150 MHz, DMSO-d<sub>6</sub>) δ = 173.4, 155.8, 140.1, 138.1, 118.9, 65.5 (2C), 40.8, 34.3, 18.7 (2C). Anal. Calcd. For C<sub>11</sub>H<sub>16</sub>N<sub>4</sub>O<sub>3</sub>S: C, 46.47; H, 5.67; N, 19.70; found: C, 46.50; H, 5.66; N, 19.65

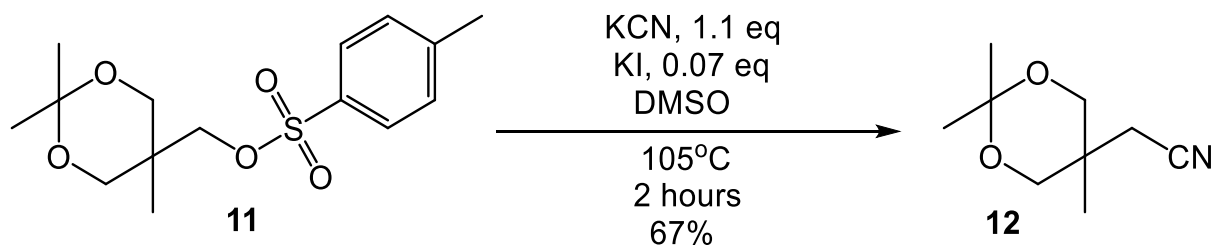
### 2-Thiocyanato-9-*n*-propyl-purin-6-one (6b).



To a suspension of 5-*n*-propyl-1,3,4-thiadiazolo[3,2-*a*]purin-8-one **4b** (0.002 mol, 0.47 g, 1 equiv) in MeCN (25 mL) in a round-bottom flask was added Et<sub>3</sub>N (0.01 mol, 1.39 mL, 5 equiv) at room temperature. Resulting mixture was refluxed for 6 hours, cooled to room temperature, concentrated under vacuum and dissolved in H<sub>2</sub>O (10 mL). This solution was treated with AcOH to pH~4, resulting precipitate was filtered and washed with H<sub>2</sub>O (10 mL) to give 0.376 g (80%) of **6b**.

White powder (0.37 g, 80% yield); R<sub>f</sub> (CHCl<sub>3</sub>-MeOH, 4-1) = 0.5; mp 185-186 °C; IR (neat) 3140, 3110, 2966, 2798, 2613, 2162, 1715, 1611, 1567, 1455, 1316, 1254, 1219, 1203, 1182, 1131, 1099, 1055, 899, 873, 808, 794, 777, 757, 738, 706, 669, 650 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>) δ = 8.12 (s, 1H), 4.15 (t, J = 6.8 Hz, 2H), 1.89 (m, 2H), 0.91 (t, J = 6.8 Hz, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, DMSO-d<sub>6</sub>) δ = 158.0, 149.4, 148.7, 141.6, 121.5, 107.5, 45.2, 22.5, 10.9. MS (EI, 70 eV) m/z: [M<sup>+</sup>] Calcd for C<sub>9</sub>H<sub>9</sub>N<sub>5</sub>OS 235, Found 235. Anal. Calcd. For C<sub>9</sub>H<sub>9</sub>N<sub>5</sub>OS: C, 45.95; H, 3.86; N, 29.77; found: C, 45.97; H, 3.90; N, 29.68

### (2,2,5-trimethyl-[1,3]dioxan-5-yl)-acetonitrile (12).

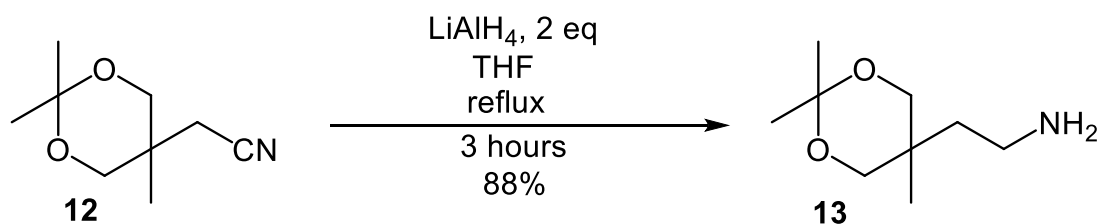


A suspension of 1,3-(dimethylmethylenedioxy)-2-methyl-2-(methylene-p-toluenesulfonyl)propane **11** (0.086 mol, 27 g, 1 equiv), KCN (0.095 mol, 6.15 g, 1.1 equiv), and KI (0.006 mol, 1 g, 0.07 equiv) in DMSO (200 mL) in round-bottom flask was heated at 105°C for 2 hours (solution was formed after 1 hour).

A resulting mixture was poured into H<sub>2</sub>O (1000 mL) and extracted with AcOEt (4 × 100 mL). The combined organic layers were dried with MgSO<sub>4</sub> and then concentrated under vacuum. Oily residue was washed with H<sub>2</sub>O (3 × 5 mL), dried with anhydrous Na<sub>2</sub>SO<sub>4</sub> to give 9.81 g (67%) of nitrile **12**.

Yellow oil (9.81 g, 67% yield); bp = 135°C/2mmHg; IR (neat) 2994, 2874, 2470, 1458, 1373, 1248, 1201, 1151, 1081, 1040, 995, 934, 883, 825, 752, 675 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ = 3.67 (m, 2H), 3.51 (m, 2H), 2.65 (s, 2H), 1.41 (s, 3H), 1.38 (s, 3H), 0.95 (s, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>) δ = 118.1, 98.5, 67.9 (2C), 32.7, 28.4, 24.0, 19.02, 18.95. MS (EI, 70 eV) m/z: [M<sup>+</sup>] Calcd for C<sub>9</sub>H<sub>15</sub>NO<sub>2</sub> 169, Found 154. Anal. Calcd. For C<sub>9</sub>H<sub>15</sub>NO<sub>2</sub>: C, 63.88; H, 8.93; N, 8.28; found: C, 64.15; H, 9.11; N, 8.08

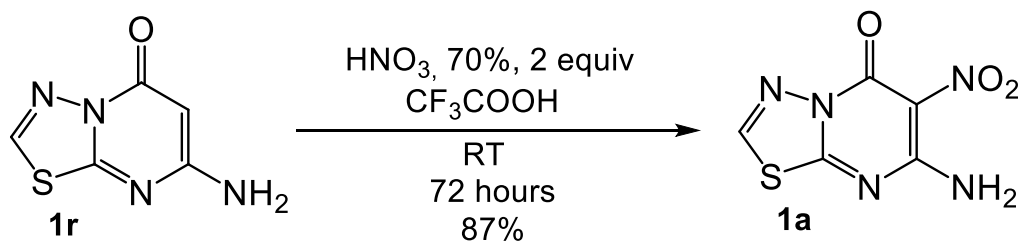
**(2,2,5-trimethyl-[1,3]dioxan-5-yl)-ethyl-2-amine (13).**



A solution of nitrile **12** (0.02 mol, 3.38 g, 1 equiv) in THF (30 mL) was added dropwise to a suspension of LiAlH<sub>4</sub> (0.04 mol, 1.52 g, 2 equiv) in THF (40 mL) during 1 hour at 25-30°C. Resulting suspension was refluxed for 3 hours, cooled to 5°C, quenched with ice and filtered. A mother liquor was extracted with CHCl<sub>3</sub> (3 × 20 mL), dried with anhydrous Na<sub>2</sub>SO<sub>4</sub> and then concentrated under vacuum to give 3.04 g (88%) of the amine **13**.

Yellow oil (3.04 g, 88% yield); bp = 95°C/5mmHg; IR (neat) 2990, 2937, 2860, 1664, 1597, 1454, 1370, 1284, 1203, 1153, 1088, 1028, 932, 829, 750, 664 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ = 3.52 (m, 2H), 3.47 (m, 2H), 2.68 (m, 2H), 1.49 (m, 2H), 1.36 (s, 6H), 0.88 (s, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>) δ = 97.9, 69.5 (2C), 39.8, 37.2, 32.3, 24.4, 23.3, 19.9. MS (EI, 70 eV) m/z: [M<sup>+</sup>] Calcd for C<sub>9</sub>H<sub>19</sub>NO<sub>2</sub> 173, Found 158. Anal. Calcd. For C<sub>9</sub>H<sub>19</sub>NO<sub>2</sub>: C, 62.39; H, 11.05; N, 8.08; found: C, 62.12; H, 10.88; N, 7.98

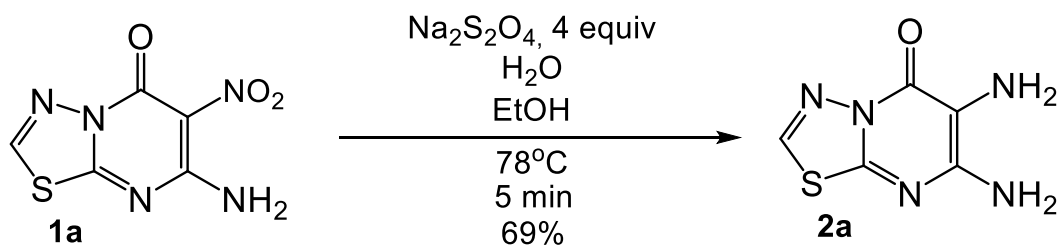
**5-amino-6-nitro-1,3,4-thiadiazolo[3,2-*a*]pyrimidin-7-one (1a).**



To a solution of 5-amino-1,3,4-thiadiazolo[3,2-*a*]pyrimidin-7-one **1r** [3] (0.03 mol, 5.04 g, 1 equiv) in CF<sub>3</sub>COOH (50 mL) in an Erlenmeyer flask was added HNO<sub>3</sub> (70%, 0.06 mol, 3.83 mL, 2 equiv) at room temperature. Resulting solution was stirred for 72 hours, concentrated, treated with EtOH (30 mL) and filtered to give 5.56 g (87%) of **1s**.

Recrystallize from EtOH; Yellow powder (5.56 g, 87% yield); R<sub>f</sub> (CHCl<sub>3</sub>-MeOH, 4-1) = 0.2; mp > 300 °C; IR (neat) 3477, 3228, 3041, 2429, 1833, 1694, 1612, 1502, 1429, 1269, 1083, 916, 853, 781, 735 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) δ = 9.29 (s, 1H), 8.86 (s, 2H). <sup>13</sup>C{<sup>1</sup>H} NMR (150 MHz, DMSO-*d*<sub>6</sub>) δ = 160.4, 158.9, 149.0, 148.8, 113.1. MS (EI, 70 eV) m/z: [M<sup>+</sup>] Calcd for C<sub>5</sub>H<sub>3</sub>N<sub>5</sub>O<sub>3</sub>S 213; Found 213. Anal. Calcd. For C<sub>5</sub>H<sub>3</sub>N<sub>5</sub>O<sub>3</sub>S: C, 28.17; H, 1.42; N, 32.85; found: C, 28.11; H, 1.55; N, 32.93

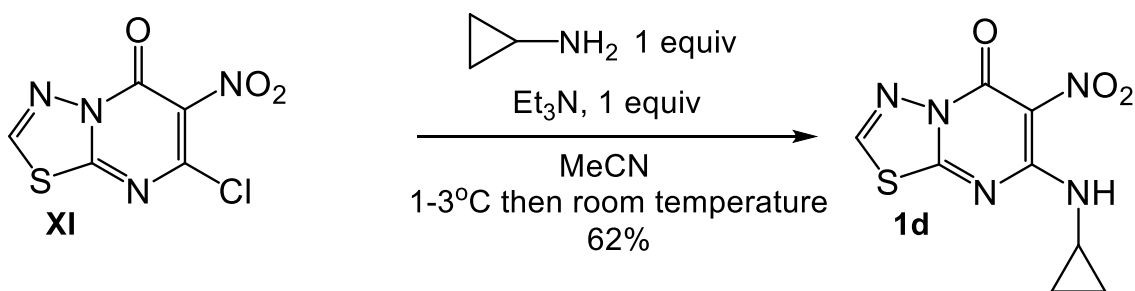
**5,6-diamino-1,3,4-thiadiazolo[3,2-*a*]pyrimidin-7-one (2a).**



To a suspension of 5-amino-6-nitro-1,3,4-thiadiazolo[3,2-*a*]pyrimidin-7-one **1a** (0.01 mol, 2.13 g, 1 equiv) in  $\text{H}_2\text{O}$  (90 mL) and  $\text{EtOH}$  (30 mL) in a round-bottom flask was added  $\text{Na}_2\text{S}_2\text{O}_4$  (85%, 0.04 mol, 8.19 g, 4 equiv) at room temperature and resulting mixture was refluxed for 5 min. Clear yellow solution was cooled to room temperature, concentrated under vacuum, treated with  $\text{H}_2\text{O}$  (10 mL) and filtered to give 1.26 g (69%) of **2a**.

Recrystallize from  $\text{H}_2\text{O}$ ; Pale yellow powder (1.26 g, 69% yield);  $R_f$  ( $\text{CHCl}_3$ - $\text{MeOH}$ , 4-1) = 0.2; mp 200-201 °C; IR (neat) 3520, 3385, 3307, 3030, 2922, 2852, 1973, 1667, 1598, 1519, 1470, 1348, 1337, 1253, 1182, 1143, 992, 859, 761, 714  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{DMSO-d}_6$ )  $\delta$  = 9.20 (s, 1H), 6.10 (s, 2H), 4.08 (br.s, 2H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (150 MHz,  $\text{DMSO-d}_6$ )  $\delta$  = 162.8, 150.8, 146.1, 133.9, 108.2. MS (EI, 70 eV)  $m/z$ : [ $\text{M}^+$ ] Calcd for  $\text{C}_5\text{H}_5\text{N}_5\text{OS}$  183; Found 183. Anal. Calcd. For  $\text{C}_5\text{H}_5\text{N}_5\text{OS}$ : C, 32.78; H, 2.75; N, 38.23; found: C, 32.88; H, 2.81; N, 38.06

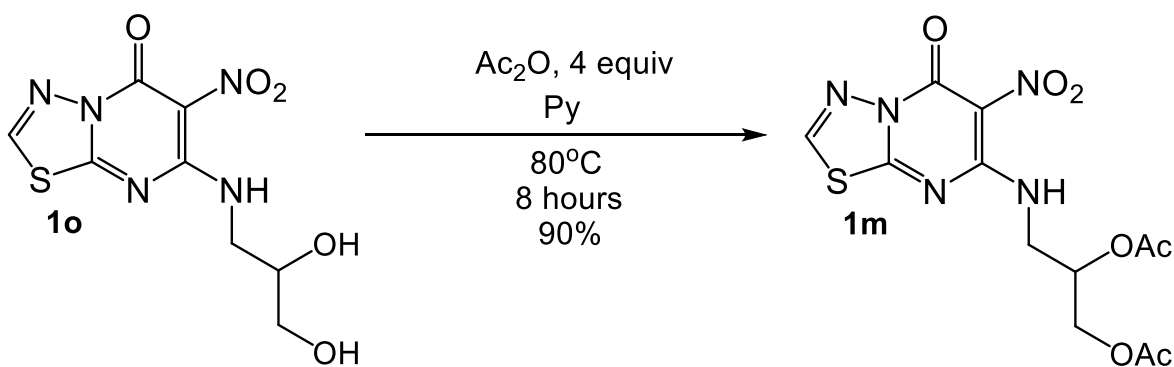
**5-cyclo-propylamino-1,3,4-thiadiazolo[3,2-*a*]pyrimidin-7-one (1d).**



To a solution of 5-chloro-6-nitro-1,3,4-thiadiazolo[3,2-*a*]pyrimidin-7-one **XI** (0.01 mol, 2.32 g, 1 equiv) in  $\text{MeCN}$  (30 mL) in an Erlenmeyer flask was added  $\text{Et}_3\text{N}$  (0.01 mol, 1.39 mL, 1 equiv) at 1-3°C followed by of aminocyclopropane (0.01 mol, 0.69 ml, 1 equiv) at 1-3°C. The resulting mixture was stirred overnight at room temperature, filtered and precipitate was recrystallized from  $\text{EtOH-H}_2\text{O}$  (1-1) to give 1.57 g (62%) of **1d**.

Recrystallize from  $\text{EtOH-H}_2\text{O}$  (1-1); Yellow powder (1.57 g, 62% yield); mp 245-246 °C; IR (neat) 3337, 3100, 3050, 1696, 1582, 1539, 1501, 1432, 1343, 1296, 1261, 1194, 1100, 1024, 999, 904, 862, 775, 721, 701  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{DMSO-d}_6$ )  $\delta$  = 9.21 (m, 1H), 9.17 (s, 1H), 3.14 (m, 1H), 0.88 (m, 2H), 0.78 (m, 2H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{DMSO-d}_6$ )  $\delta$  = 164.2, 156.9, 149.9, 147.6, 113.5, 25.0, 6.5 (2C). MS (EI, 70 eV)  $m/z$ : [ $\text{M}^+ + \text{H}^+$ ] Calcd for  $\text{C}_8\text{H}_8\text{N}_5\text{O}_3\text{S}$  254; Found 254. Anal. Calcd. For  $\text{C}_8\text{H}_7\text{N}_5\text{O}_3\text{S}$ : C, 37.94; H, 2.79; N, 27.66; found: C, 37.95; H, 2.82; N, 27.67

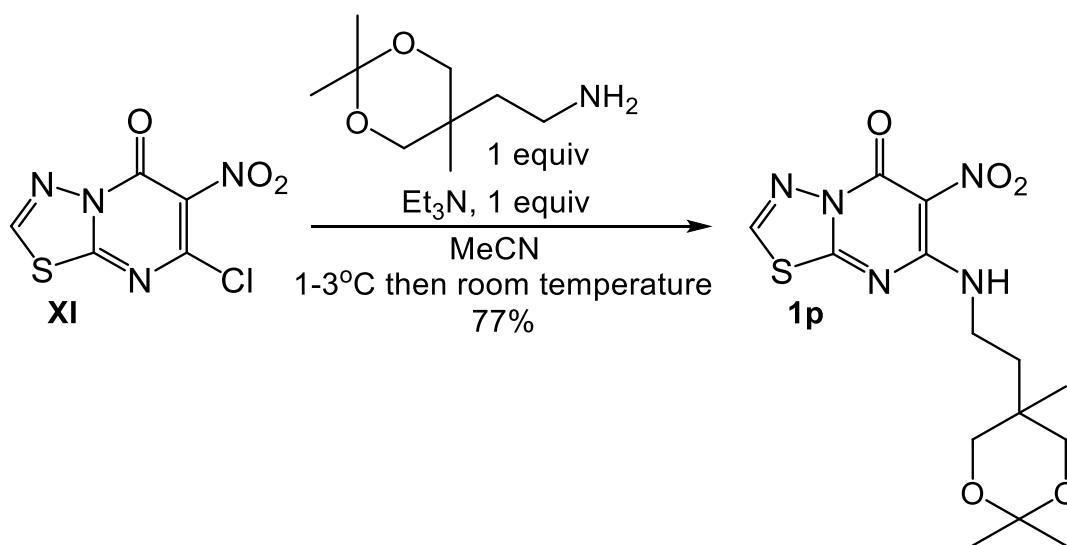
**5-(2,3-Diacetoxypropyl-1-amino)-1,3,4-thiadiazolo[3,2-*a*]pyrimidin-7-one (1m).**



To a suspension of 5-(2,3-hydroxypropyl-1-amino)-1,3,4-thiadiazolo[3,2-*a*]pyrimidin-7-one **1o** (0.005 mol, 1.44 g, 1 equiv) in Py (30 mL) in a round-bottom flask was added  $\text{Ac}_2\text{O}$  (0.02 mol, 1.89 mL, 4 equiv) at room temperature. The resulting mixture was heated at  $80^\circ\text{C}$  for 8 hours, cooled to room temperature, concentrated under vacuum, treated with  $\text{H}_2\text{O}$  and filtered to give 1.67 g (90%) of **1m**.

Recrystallize from  $\text{H}_2\text{O}$ ; Gray powder (1.67 g, 90% yield);  $R_f$  (AcOEt) = 0.4; mp  $180\text{--}181^\circ\text{C}$ ; IR (neat) 3053, 1747, 1695, 1560, 1506, 1428, 1367, 1203, 1124, 1080, 1011, 902, 862, 777, 726  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{DMSO-d}_6$ )  $\delta$  = 9.55 (t,  $J$  = 5.6 Hz, 1H), 9.20 (s, 1H), 5.23 (m, 1H), 4.25 (dd,  $J_1$  = 6.0 Hz,  $J_2$  = 6.0 Hz), 4.17 (dd,  $J_1$  = 6.0 Hz,  $J_2$  = 6.0 Hz), 3.90 (m, 1H), 3.80 (m, 1H), 2.03 (s, 3H), 2.01 (s, 3H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{DMSO-d}_6$ )  $\delta$  = 170.2, 169.9, 164.5, 156.2, 150.3, 148.0, 113.6, 69.6, 63.0, 41.5, 20.7, 20.5. Anal. Calcd. For  $\text{C}_{12}\text{H}_{13}\text{N}_5\text{O}_7\text{S}$ : C, 38.82; H, 3.53; N, 18.86; found: C, 38.85; H, 3.43; N, 19.00

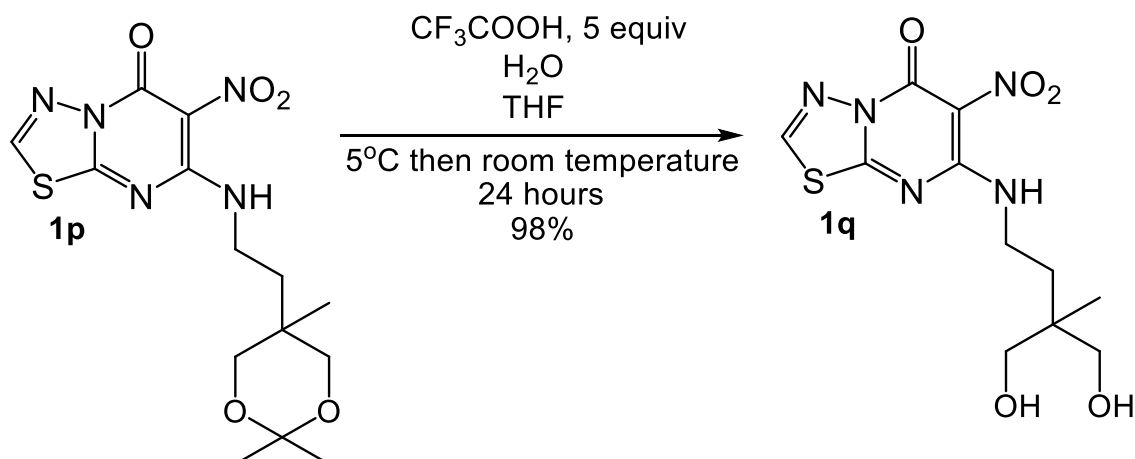
**5-(2-(2,2,5-trimethyl-[1,3]dioxan-5-yl)-ethyl)amino)-1,3,4-thiadiazolo[3,2-*a*]pyrimidin-7-one (1p).**



To a solution of 5-chloro-6-nitro-1,3,4-thiadiazolo[3,2-*a*]pyrimidin-7-one **XI** (0.01 mol, 2.32 g, 1 equiv) in MeCN (30 mL) in an Erlenmeyer flask was added  $\text{Et}_3\text{N}$  (0.01 mol, 1.39 mL, 1 equiv) at  $1\text{--}3^\circ\text{C}$  followed by a solution of (2,2,5-trimethyl-[1,3]dioxan-5-yl)-ethyl-2-amine **13** (0.01 mol, 1.73 g, 1 equiv) in MeCN (10 mL) at  $1\text{--}3^\circ\text{C}$ . The resulting mixture was stirred for 3 hours at room temperature, filtered and precipitate was recrystallized from EtOH (150 mL) to give 2.84 g (77%) of **1p**.

Recrystallize from EtOH; Pale yellow crystals (2.84 g, 77% yield);  $R_f$  (AcOEt) = 0.5; mp  $194\text{--}195^\circ\text{C}$ ; IR (neat) 3283, 3074, 2987, 2949, 2858, 1711, 1548, 1493, 1430, 1360, 1316, 1292, 1245, 1203, 1179, 1146, 1113, 1076, 1019, 988, 929, 860, 829, 776, 731, 709, 683  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{DMSO-d}_6$ )  $\delta$  = 9.54 (t,  $J$  = 6.0 Hz, 1H), 9.16 (s, 1H), 3.60 (dt,  $J_1$  = 8.0,  $J_2$  = 6.0, 2H), 3.49 (m, 4H), 1.68 (t,  $J$  = 8.0, 2H), 1.32 (s, 3H), 1.30 (s, 3H), 0.90 (s, 3H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{DMSO-d}_6$ )  $\delta$  = 164.4, 155.6, 150.4, 147.7, 133.3, 97.2, 68.1 (2C), 37.4, 34.4, 31.8, 24.5, 23.0, 19.2. MS (EI, 70 eV)  $m/z$ : [ $\text{M}^+\text{H}^+$ ] Calcd for  $\text{C}_{14}\text{H}_{19}\text{N}_5\text{O}_5\text{S}$  370; Found 370. Anal. Calcd. For  $\text{C}_{14}\text{H}_{19}\text{N}_5\text{O}_5\text{S}$ : C, 45.52; H, 5.18; N, 18.96; found: C, 45.52; H, 5.20; N, 19.01

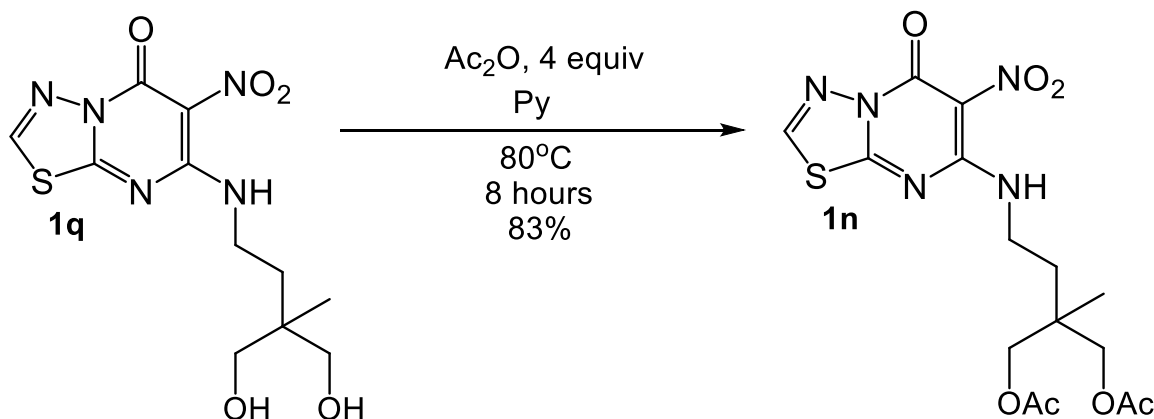
**5-(4-hydroxy-3-(hydroxymethyl)-3-methylbutyl)amino)-1,3,4-thiadiazolo[3,2-*a*]pyrimidin-7-one (1q).**



To a solution of 5-(2-(2,2,5-trimethyl-[1,3]dioxan-5-yl)ethyl)amino)-1,3,4-thiadiazolo[3,2-*a*]pyrimidin-7-one **1p** (0.005 mol, 1.85 g, 1 equiv) in THF (85 mL) and  $\text{H}_2\text{O}$  (21 mL) in an Erlenmeyer flask was added  $\text{CF}_3\text{COOH}$  (0.025 mol, 1.91 mL, 5 equiv) at  $5^\circ\text{C}$ . The resulting solution was stirred for 24 hours at room temperature, concentrated under vacuum, treated with  $\text{CH}_2\text{Cl}_2$  and filtered to give 1.61 g (98%) of **1q**.

Pale yellow crystals (1.61 g, 98% yield);  $R_f$  (AcOEt) = 0.1; mp  $195\text{--}196^\circ\text{C}$ ; IR (neat) 3499, 3275, 3045, 2881, 1774, 1692, 1590, 1544, 1501, 1436, 1381, 1262, 1198, 1124, 1071, 1026, 925, 888, 863, 777, 726, 700,  $671\text{ cm}^{-1}$ ;  $^1\text{H NMR}$  (400 MHz,  $\text{DMSO-d}_6$ )  $\delta$  = 9.56 (t,  $J$  = 6.0 Hz, 1H), 9.15 (s, 1H), 4.25 (br.s, 2H), 3.60 (dt,  $J_1$  = 8.0,  $J_2$  = 6.0, 2H), 3.23 (m, 4H), 1.54 (t,  $J$  = 8.0, 2H), 0.80 (s, 3H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{DMSO-d}_6$ )  $\delta$  = 164.5, 155.5, 150.4, 147.6, 113.2, 65.8 (2C), 39.2, 37.8, 33.6, 19.0. Anal. Calcd. For  $\text{C}_{11}\text{H}_{15}\text{N}_5\text{O}_5\text{S}$ : C, 40.12; H, 4.59; N, 21.27; found: C, 40.03; H, 4.66; N, 21.14

**5-(4-acetoxy-3-(acetoxymethyl)-3-methylbutyl)amino)-1,3,4-thiadiazolo[3,2-*a*]pyrimidin-7-one (1n).**



To a suspension of 5-(4-hydroxy-3-(hydroxymethyl)-3-methylbutyl)amino)-1,3,4-thiadiazolo[3,2-*a*]pyrimidin-7-one **1q** (0.005 mol, 1.65 g, 1 equiv) in Py (30 mL) in a round-bottom flask was added  $\text{Ac}_2\text{O}$  (0.02 mol, 1.89 mL, 4 equiv) at room temperature. The resulting mixture was heated at  $80^\circ\text{C}$  for 8 hours, cooled to room temperature, concentrated under vacuum, treated with EtOH, filtered and recrystallized from EtOH to give 1.71 g (83%) of **1n**.

Recrystallize from EtOH; Pale yellow powder (1.71 g, 83% yield);  $R_f$  (AcOEt) = 0.4; mp  $160\text{--}161^\circ\text{C}$ ; IR (neat) 3288, 3151, 2941, 2349, 1713, 1557, 1450, 1414, 1376, 1257, 1225, 1202, 1109, 1032, 878, 857, 776,  $726\text{ cm}^{-1}$ ;  $^1\text{H NMR}$  (400 MHz,  $\text{DMSO-d}_6$ )  $\delta$  = 9.54 (br.s, 1H), 9.14 (s, 1H), 3.90 (br.s, 2H), 3.64 (dt,  $J_1$  = 8.0,  $J_2$  = 6.0, 2H), 2.03 (s, 6H), 1.567 (t,  $J$  = 8.0, 2H), 0.99 (s, 3H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (100 MHz,  $\text{DMSO-d}_6$ )  $\delta$  = 170.2, 164.5, 155.6, 150.4, 147.7, 113.3, 67.1, 37.2, 36.5, 33.5, 20.6 (2C), 18.7. Anal. Calcd. For  $\text{C}_{15}\text{H}_{19}\text{N}_5\text{O}_7\text{S}$ : C, 43.58; H, 4.63; N, 16.94; found: C, 43.66; H, 4.63; N, 17.09

The structure of the compound **4k** was confirmed by XRD data. According XRD data, the compound is crystallized in the centrosymmetric space group (fig.1). The bond distances and angles are near to expectation. In particular, the distinguishes between double and single bonds in the heterocyclic part of the molecule are well expressed. The molecule has a pincers-like conformation with synclinal placed substitutes of the ethylene moiety. In the crystal the shortened planar contact between N(3)C(2)HS(1) and N(7)C(6)C(5)O(1)  $[x, 0,5-y, z-0,5]$  moieties is observed and  $\pi$ -stacked interaction between heterocyclic parts of the nearest molecules is realized.

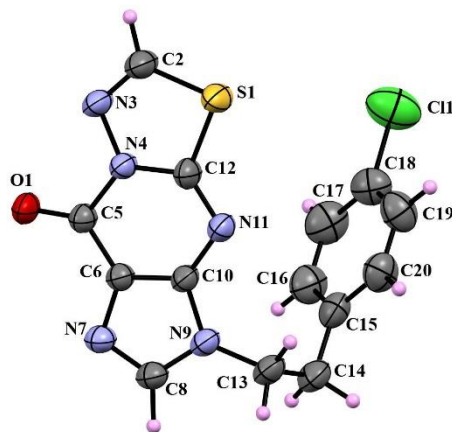


Fig.1. The molecule of the compound **4k** in the thermal ellipsoids of the 50% probability level.

The orthorhombic phase of the compound **10g** is crystallized as solvate with DMSO (1:2). The molecule of the disulfide is placed in the special position on the 2-fold axis (fig. 2). The measured S-S bond distance 2.018(2) Å and C-S distance 1.789(5) Å. The torsion angle C-S-S-C 75.5(2)°. The H-atom of the heterocyclic NH-group is localized at NC=O moiety. All other bond distances and angles are near to the standard values. The molecule of the DMSO is disordered into two position with occupancy coefficients 0.85/0.15, however, the O-atom of the DMSO is well localized and forms the intermolecular H-bond with the NH-group of the heterocycle. The other significantly shortened intermolecular contacts in the crystal do not observed.

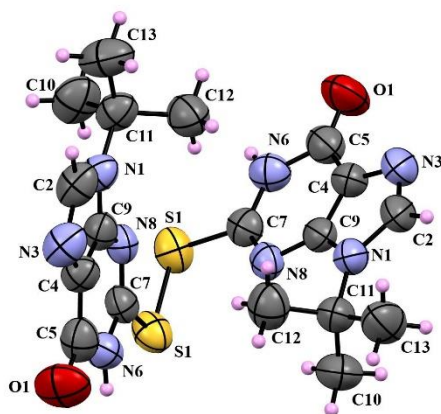


Fig.2. The molecule of the compound **10g** in the thermal ellipsoids of the 50% probability level. The molecule of the DMSO is omitted for clarity.

The monoclinic crystals of the compound **10e** also are crystallized as solvate with DMSO (1:2). The molecule of the disulfide is placed in the general position (fig. 3). The measured S-S bond distance 2.013(2) Å and C-S distances 1.781(4) Å. The torsion angle C-S-S-C 71.5(2)°. The H-atom of the heterocyclic NH-group is localized at NC=O moiety. The n-butyl moiety is disordered and taken into refinement in the particular isotropic approximation and fixed C-C bonds. All other measured bond distances and angles are near to the standard values. One molecule of the DMSO is disordered into two position with occupancy coefficients 0.8/0.2, second molecule is well localized and both form the intermolecular O...H-bond with the NH-group of the heterocycle. The other significantly shortened intermolecular contacts in the crystal do not observed.

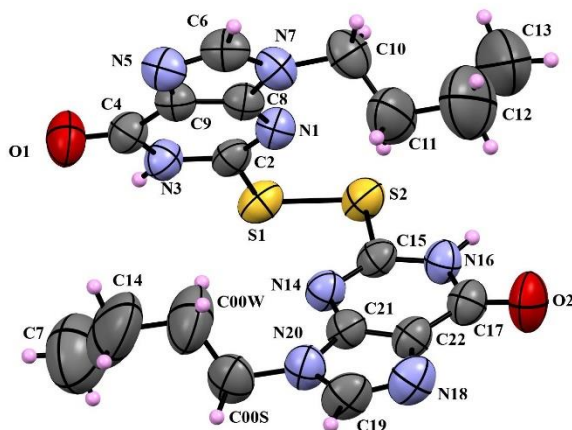


Fig.3. The molecule of the compound **10e** in the thermal ellipsoids of the 50% probability level. The molecule of the DMSO is omitted for clarity.

The XRD analyses were carried out using equipment of the Center for Joint Use “Spectroscopy and Analysis of Organic Compounds” at the Postovsky Institute of Organic Synthesis of the Ural Branch of the Russian Academy of Sciences. The experiments were accomplished on the automated X-ray diffractometer «Xcalibur 3» with CCD detector on standard procedure (MoK $\alpha$ -irradiation, graphite monochromator,  $\omega$ -scans with 1° step at T= 295(2) K). Empirical absorption correction was applied. The solution and refinement of the structures were accomplished with using Olex program package [1]. The structures were solved by method of the intrinsic phases in ShelXT program and refined by ShelXL by full-matrix least-squared method for non-hydrogen atoms [2]. The H-atoms were placed in the calculated positions and were refined in isotropic approximation in the “rider” model.

**4k** Crystal Data for C<sub>14</sub>H<sub>10</sub>ClN<sub>5</sub>OS (M = 331.78 g/mol): monoclinic, space group P2<sub>1</sub>/c, a = 12.0095(12) Å, b = 9.0931(5) Å, c = 14.1600(11) Å,  $\beta$  = 109.568(11)°, V = 1457.0(2) Å<sup>3</sup>, Z = 4,  $\mu$ (Mo K $\alpha$ ) = 0.414 mm<sup>-1</sup>, D<sub>calc</sub> = 1.512 g/cm<sup>3</sup>, 10653 reflections measured (7.202° ≤ 2 $\theta$  ≤ 61.808°), 3948 unique (R<sub>int</sub> = 0.0368, R<sub>sigma</sub> = 0.0433) which were used in all calculations. The final R<sub>1</sub> = 0.0611, wR<sub>2</sub> = 0.1761 (I > 2 $\sigma$ (I)) and R<sub>1</sub> = 0.1013, wR<sub>2</sub> = 0.2286 (all data). GooF on F<sup>2</sup> 1.089. Largest diff. peak/hole 0.45/-0.40 e<sup>-</sup>Å<sup>-3</sup>. CCDC number is 2163811.

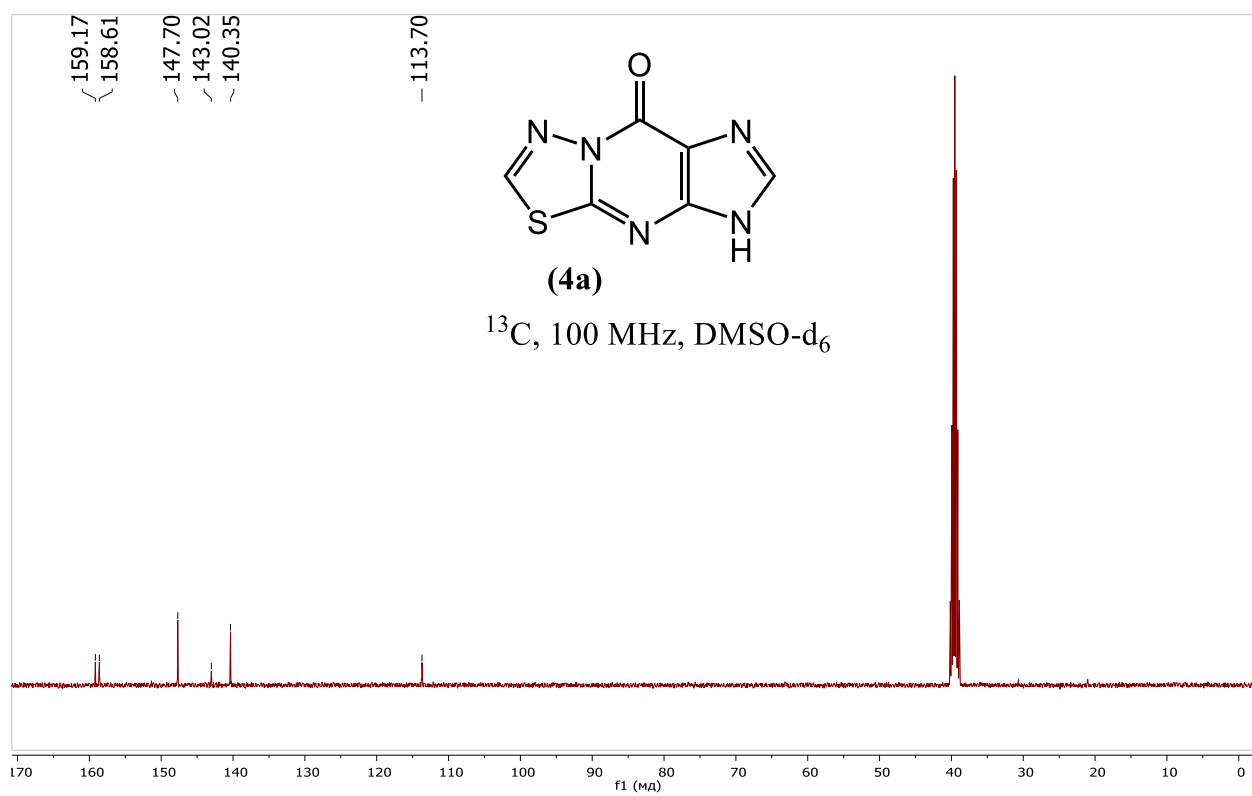
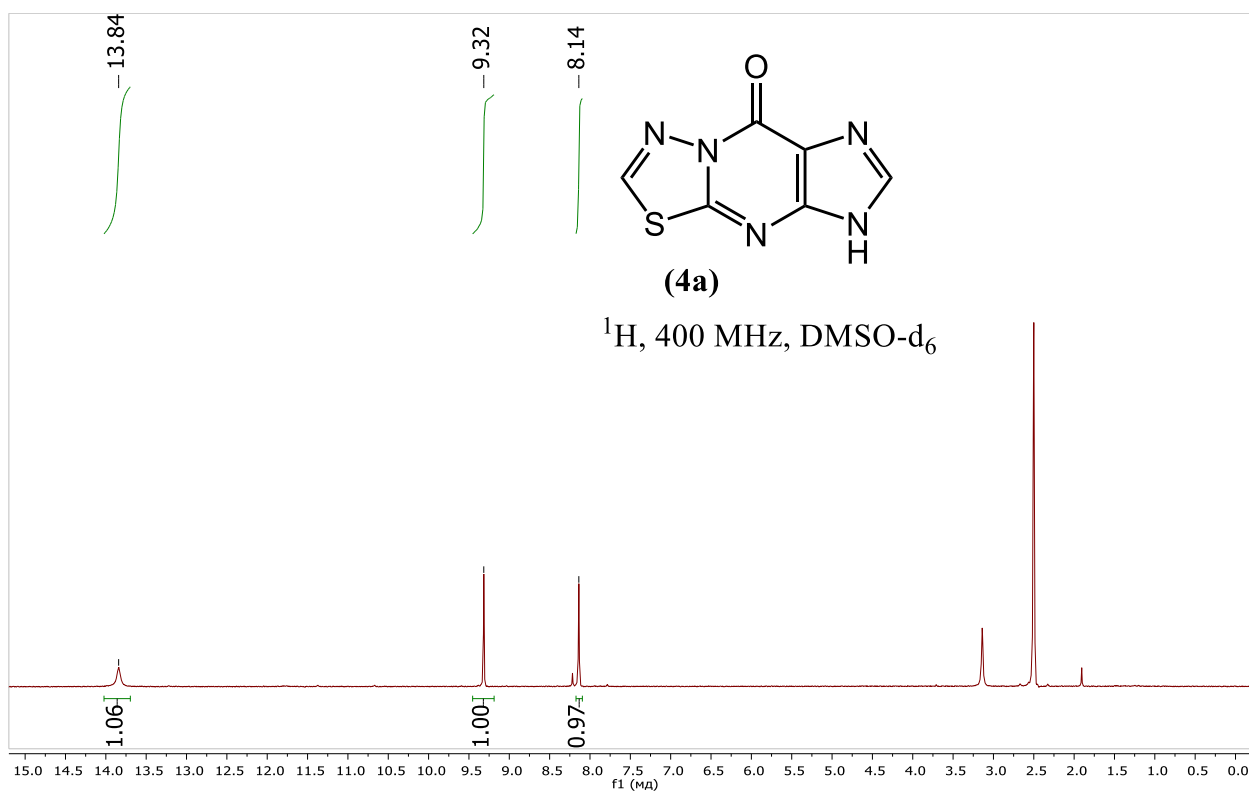
**10g** Crystal Data for C<sub>22</sub>H<sub>34</sub>N<sub>8</sub>O<sub>4</sub>S<sub>4</sub> (M = 602.81 g/mol): monoclinic, space group C2/c, a = 13.4433(9) Å, b = 27.1914(16) Å, c = 16.9657(14) Å,  $\beta$  = 103.129(7)°, V = 6039.6(8) Å<sup>3</sup>, Z = 8,  $\mu$ (Mo K $\alpha$ ) = 0.356 mm<sup>-1</sup>, D<sub>calc</sub> = 1.326 g/cm<sup>3</sup>, 23879 reflections measured (7.498° ≤ 2 $\theta$  ≤ 60.992°), 8489 unique (R<sub>int</sub> = 0.0559, R<sub>sigma</sub> = 0.0819) which were used in all calculations. The final R<sub>1</sub> = 0.0683, wR<sub>2</sub> = 0.1758 (I > 2 $\sigma$ (I)) and R<sub>1</sub> = 0.1710, wR<sub>2</sub> = 0.2486 (all data). GooF on F<sup>2</sup> 1.005. Largest diff. peak/hole 0.36/-0.31 e<sup>-</sup>Å<sup>-3</sup>. CCDC number is 2163812.

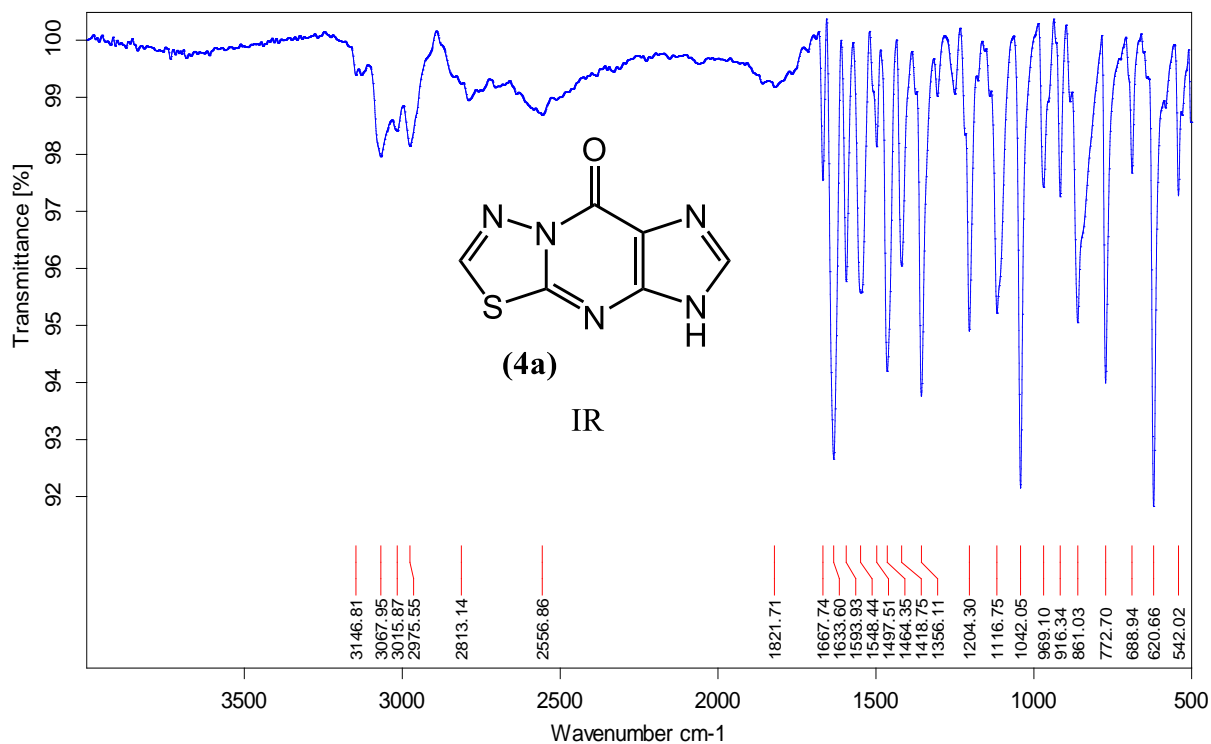
**10f** Crystal Data for C<sub>22</sub>H<sub>34</sub>N<sub>8</sub>O<sub>4</sub>S<sub>4</sub> (M = 602.81 g/mol): orthorhombic, space group Pccn, a = 11.8823(13) Å, b = 16.0031(17) Å, c = 16.3570(15) Å, V = 3110.3(6) Å<sup>3</sup>, Z = 4,  $\mu$ (Mo K $\alpha$ ) = 0.346 mm<sup>-1</sup>, D<sub>calc</sub> = 1.287



g/cm<sup>3</sup>, 11768 reflections measured ( $3.56^\circ \leq \Theta \leq 30.87^\circ$ ), 4251 unique ( $R_{\text{int}} = 0.078$ ) which were used in all calculations. The final  $R_1 = 0.0889$ ,  $wR_2 = 0.2253$  ( $I > 2\sigma(I)$ ) and  $R_1 = 0.2165$ ,  $wR_2 = 0.3188$  (all data). GooF on  $F^2$  1.088. Largest diff. peak/hole 0.47/-0.30 e<sup>-</sup>Å<sup>-3</sup>. CCDC number is 2163813.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J., Howard, J.A.K. & Puschmann, H. *J. Appl. Cryst.*, **2009**, *42*, 339-341
2. Sheldrick, G.M. *Acta Cryst.*, **2015**, *A71*, 3-8
3. Takenaka, K.; Tsuji, T. Synthesis of [1,3,4]thiadiazolo[3,2-a]pyrimidines in the presence of formic acid. *Journal of Heterocyclic Chemistry*, **1996**, *33(4)*, 1367–1370. DOI:10.1002/jhet.5570330459





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KC-1291

29/04/2021

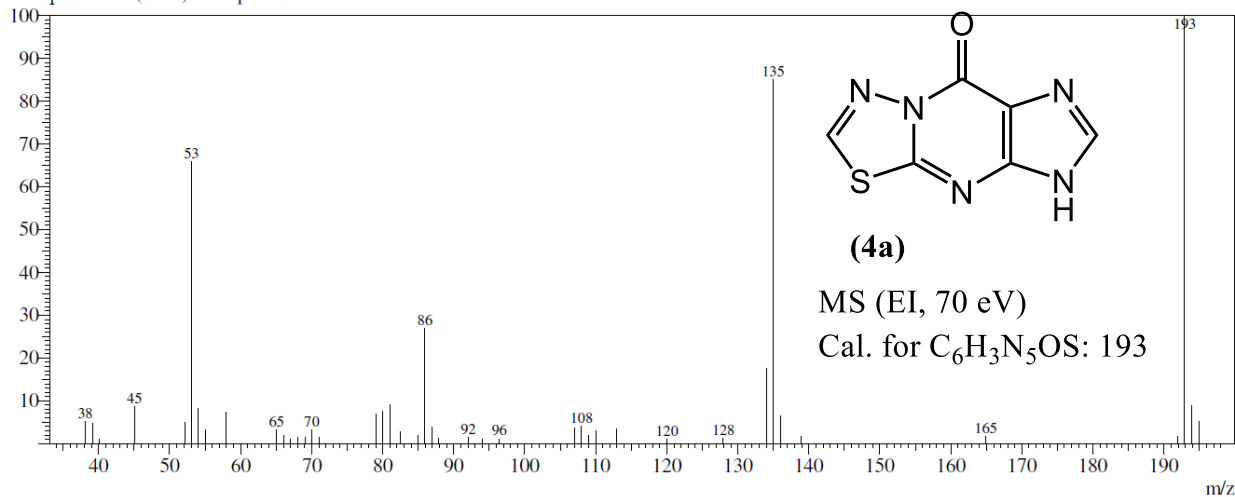
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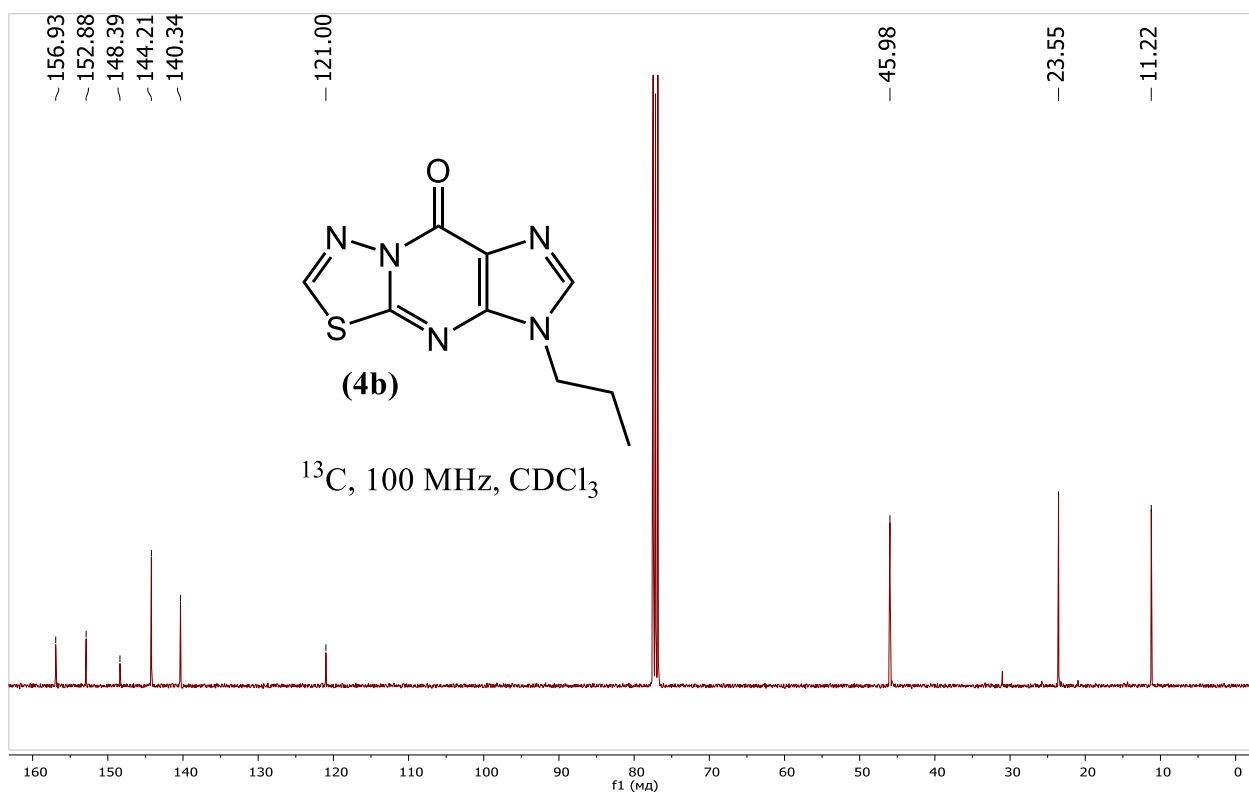
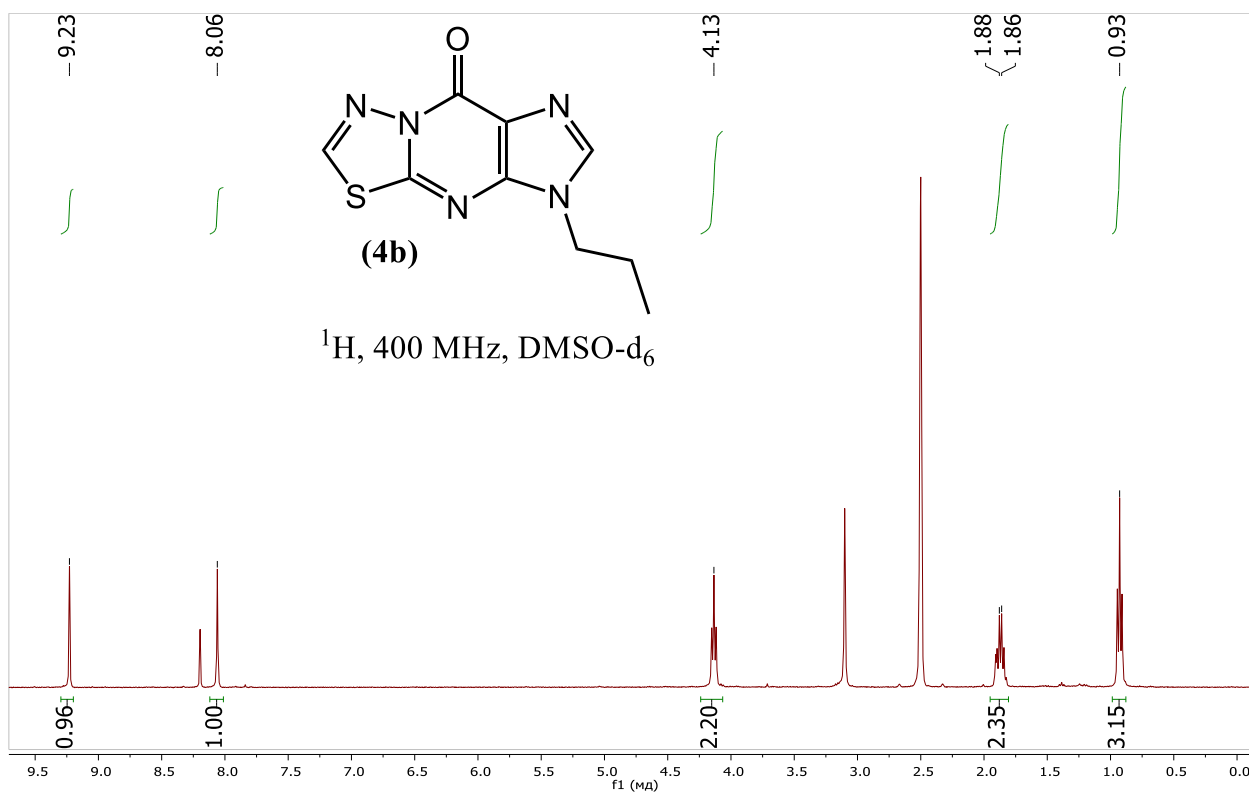
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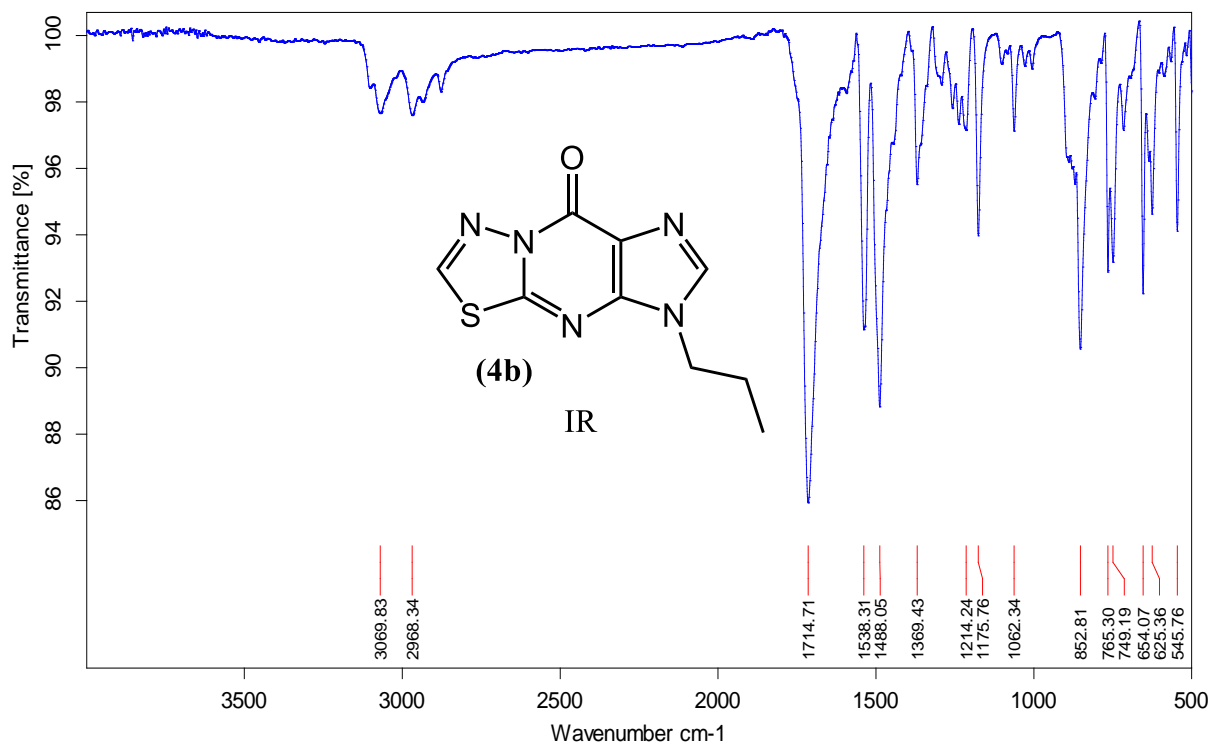
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Фон.реш.:5.135(2035) Group 1 - Event 1







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KC-1118

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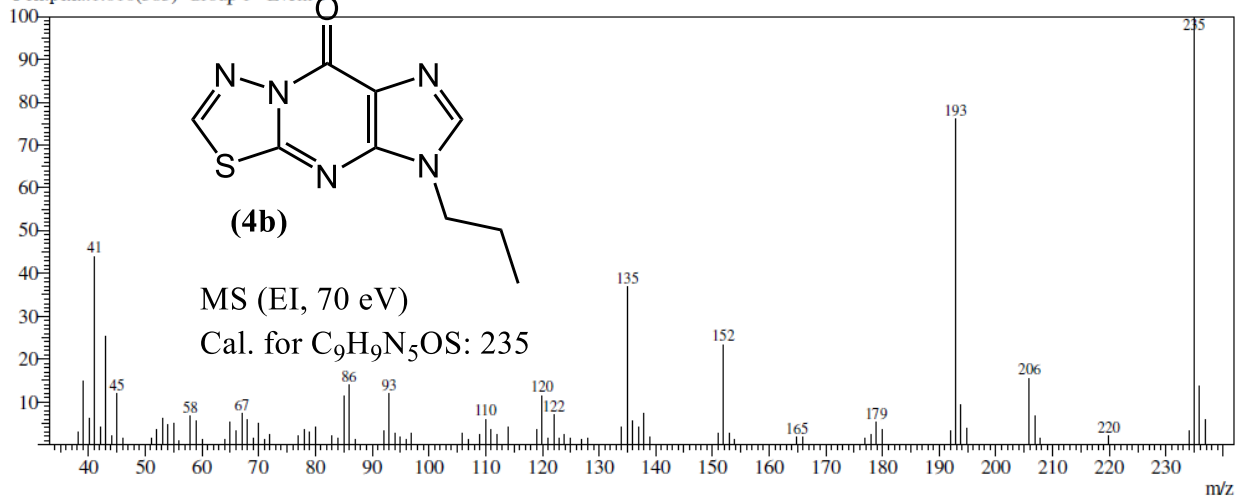
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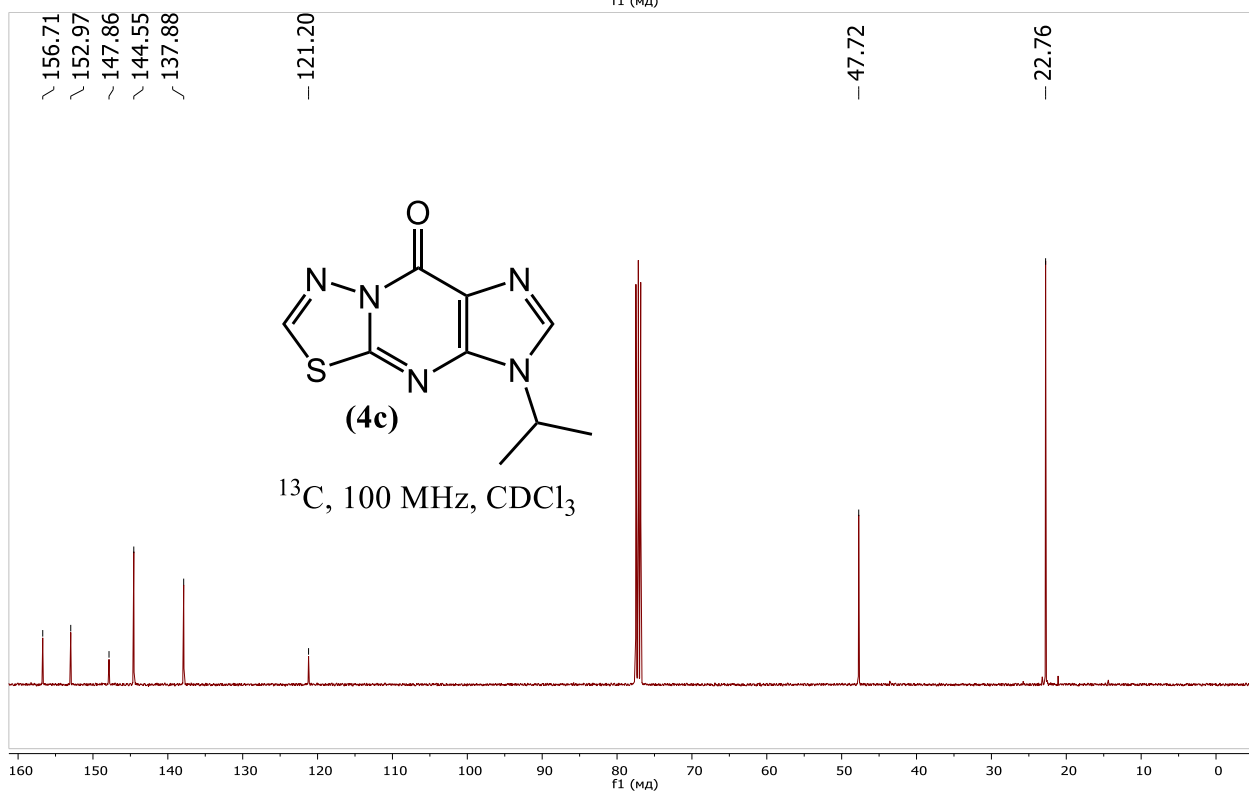
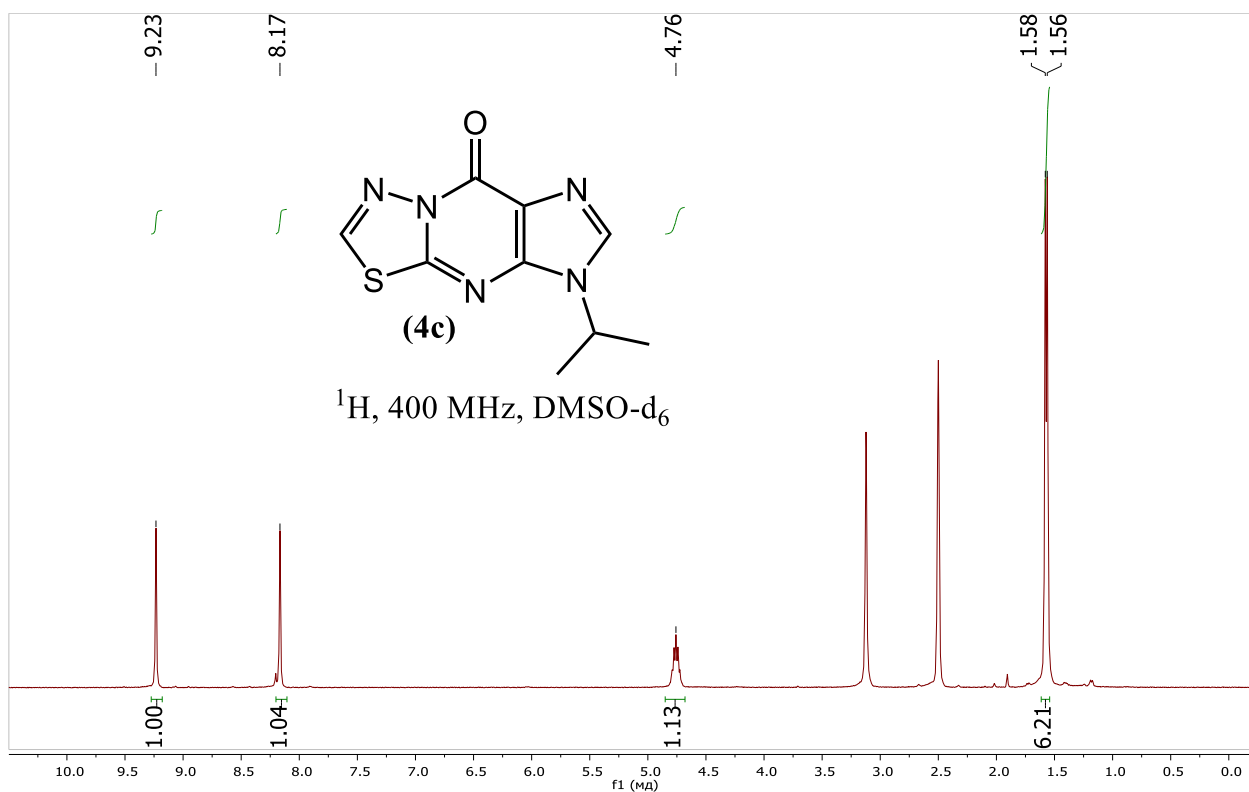
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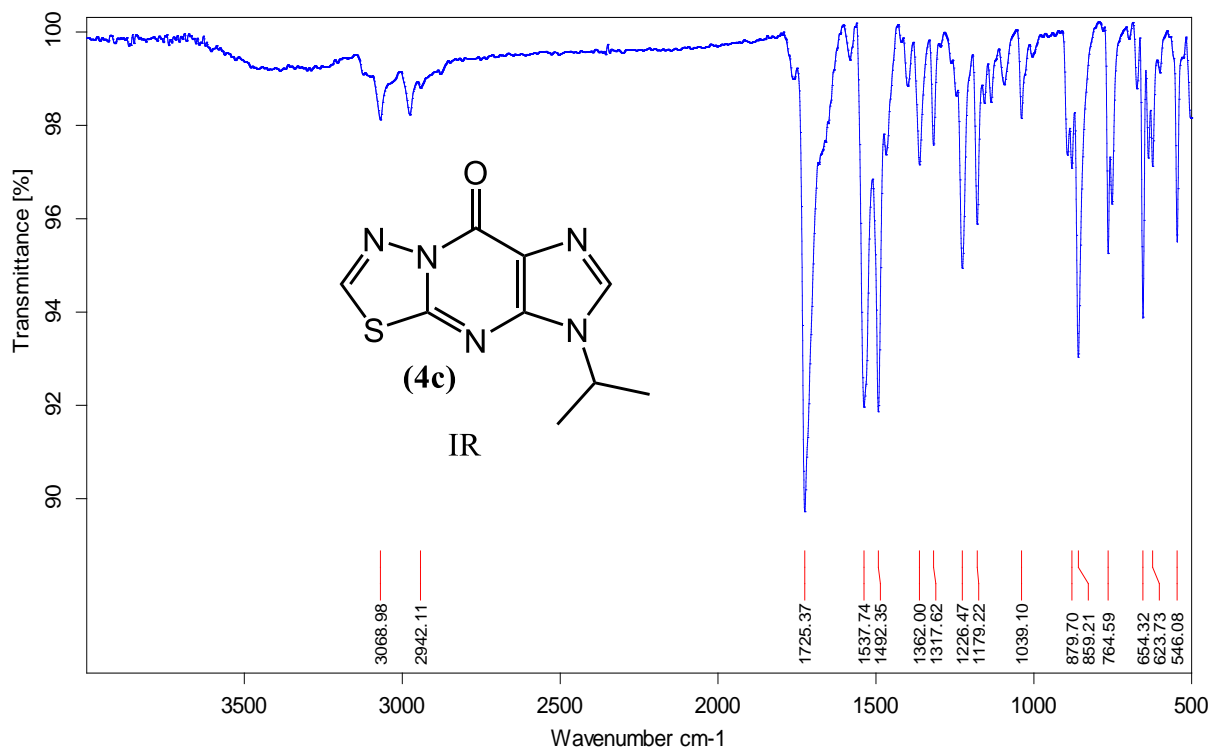
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RawMode:Single 2.255(883) BasePeak:235(6589421)

Фон.реж.:1.010(385) Group 1 - Event







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KC-1121

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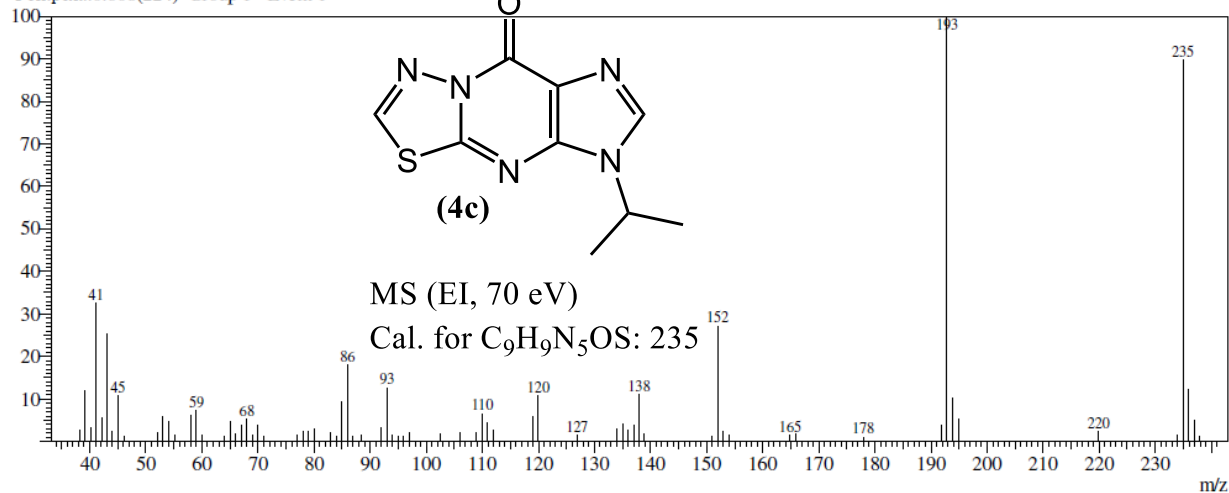
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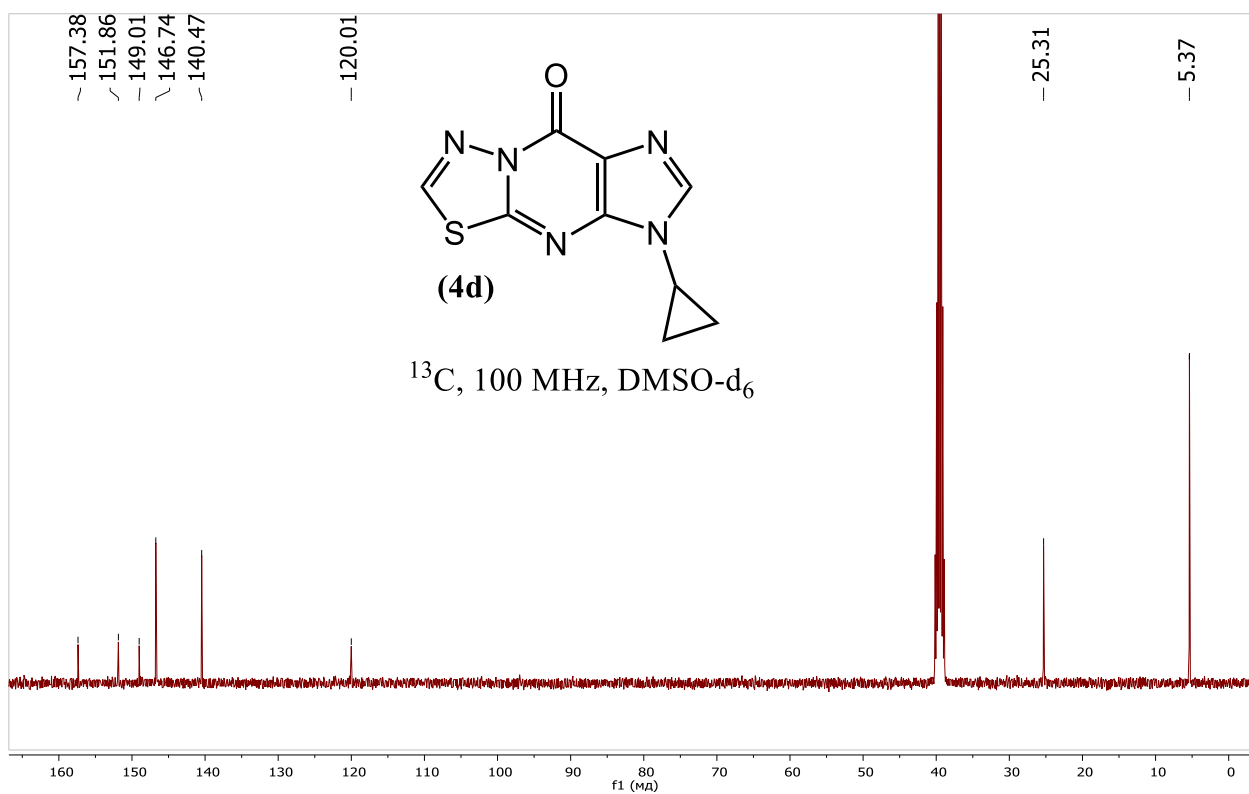
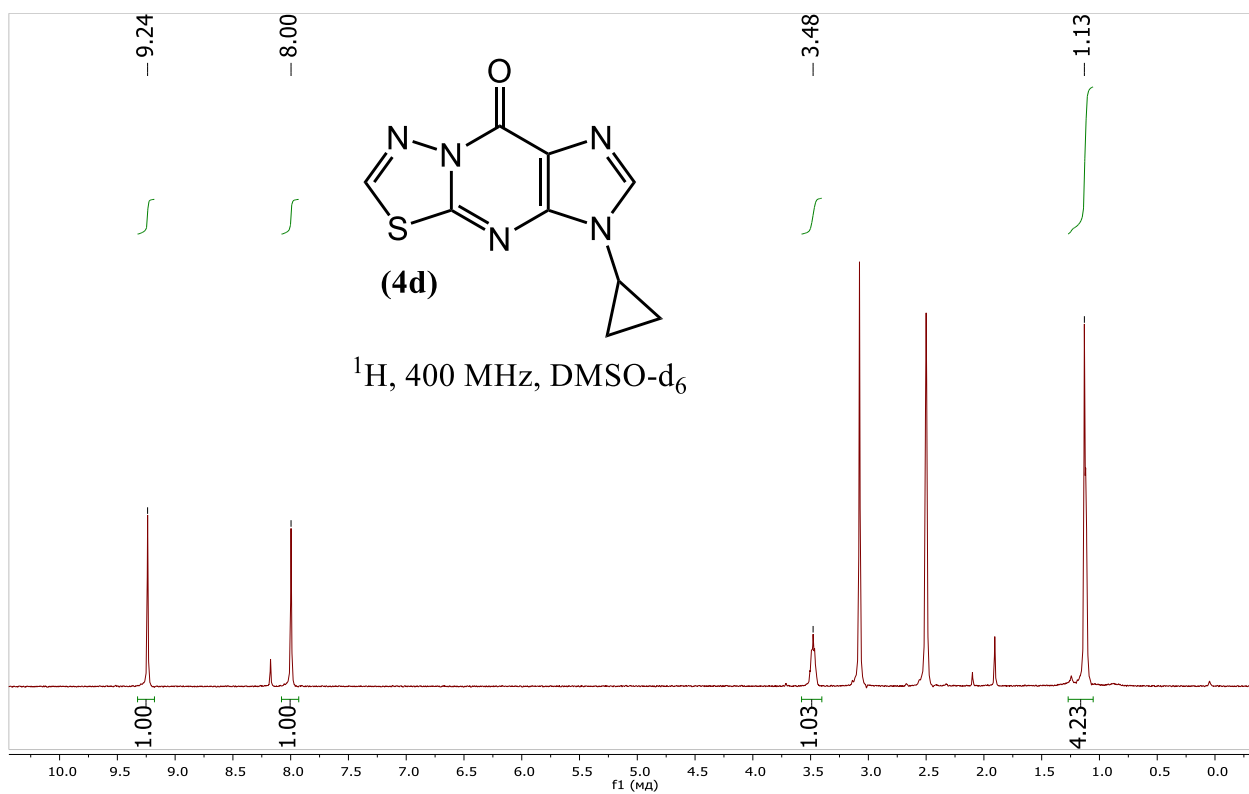
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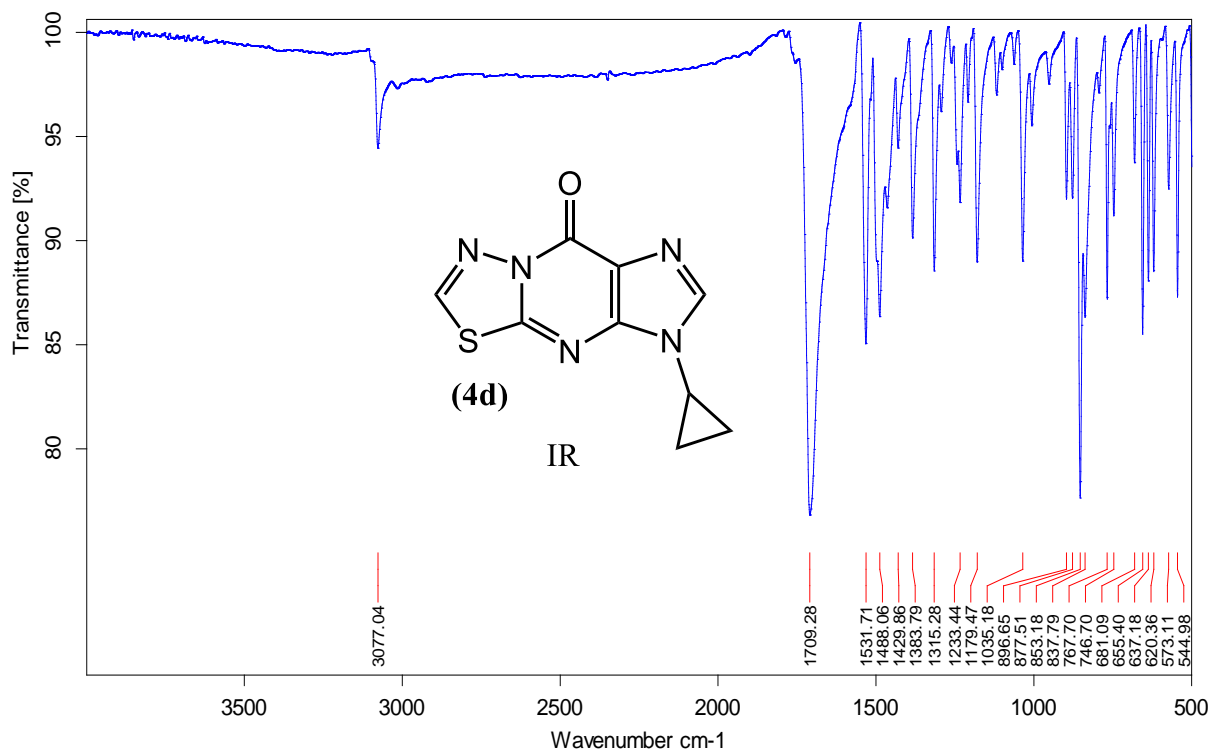
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Фон.реж.:0.608(224) Group 1 - Event 1









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KC-1200

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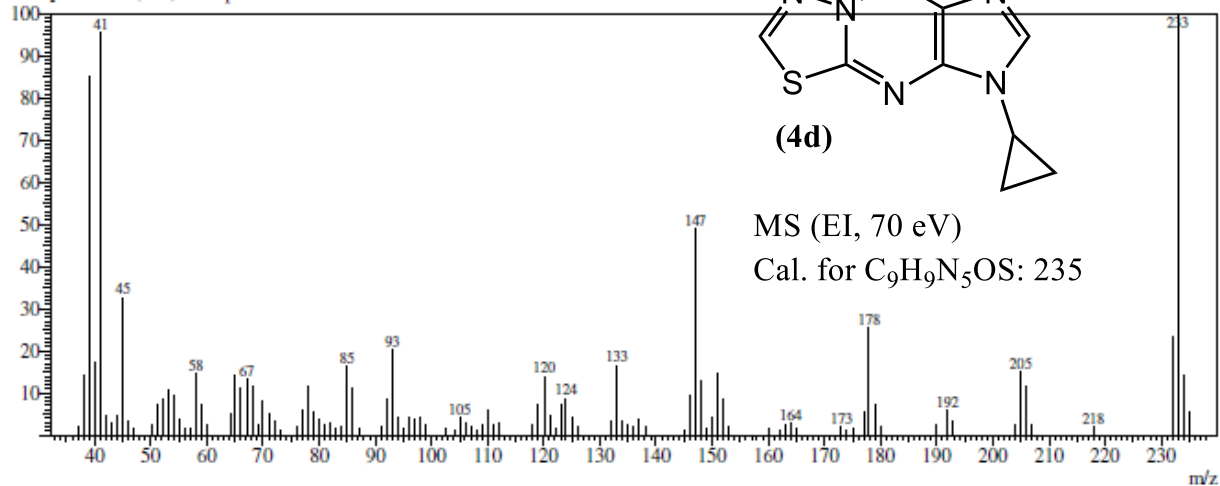
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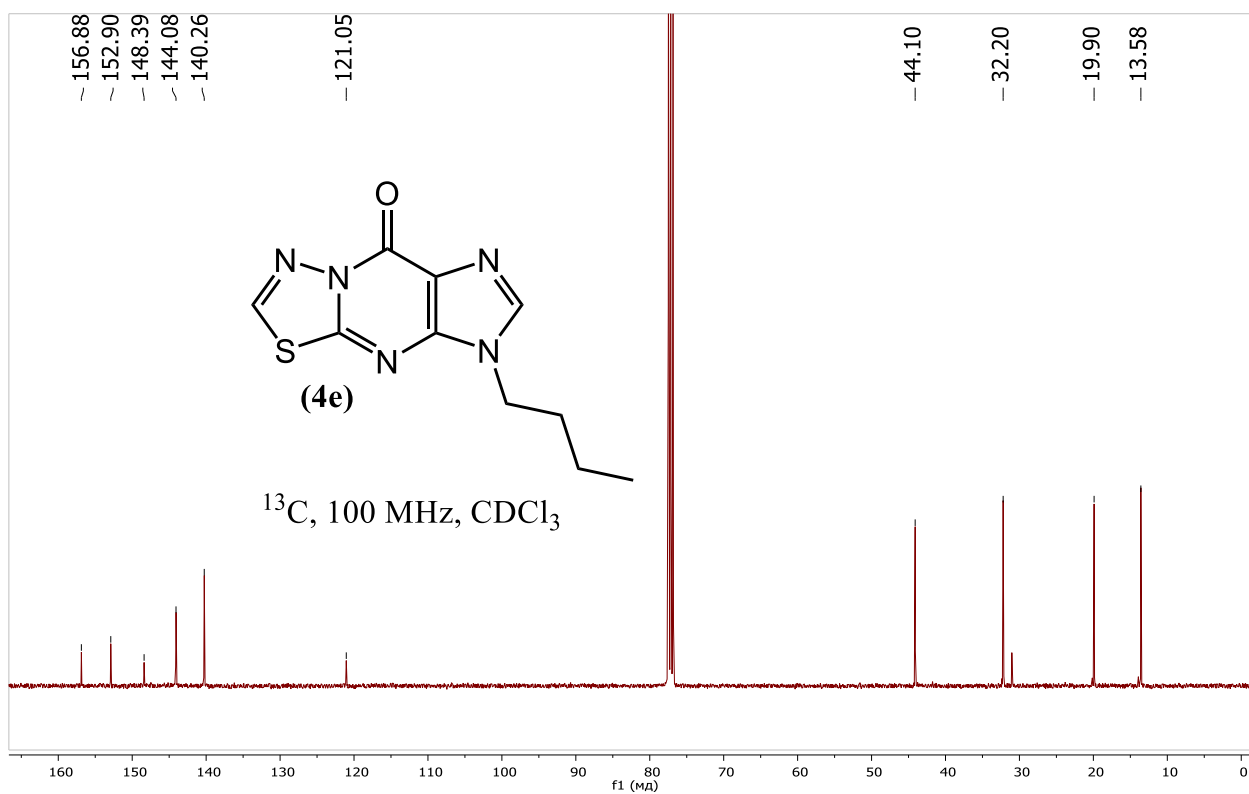
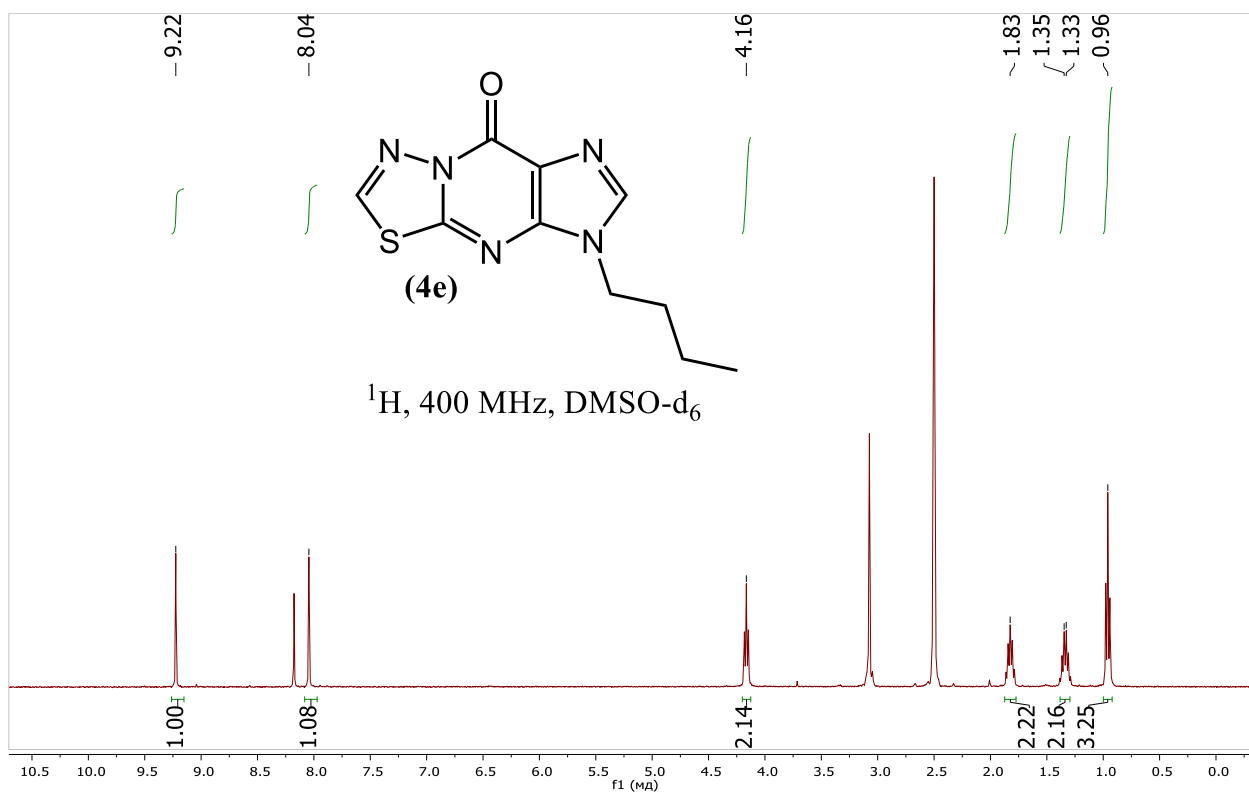
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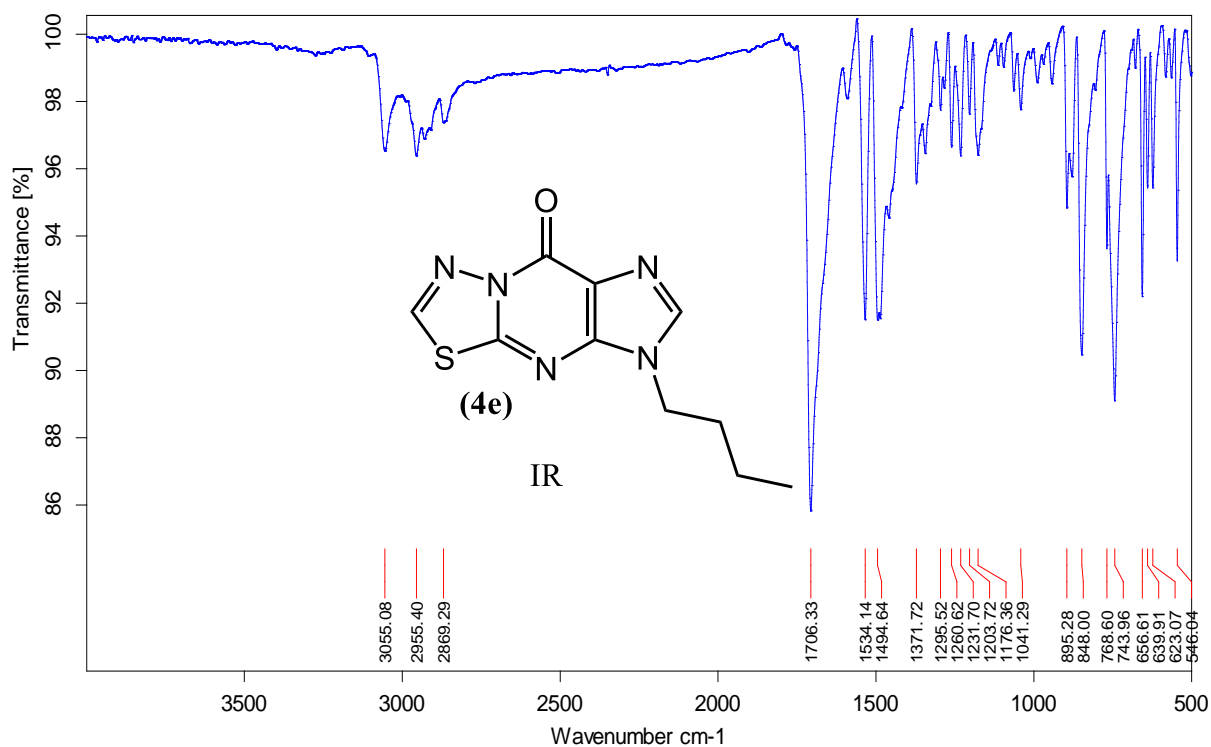
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RawMode:Single 3.163(1246) BasePeak:233(2870840)

Phi.pea.:1.165(447) Group 1 - Event 1







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KC-1144

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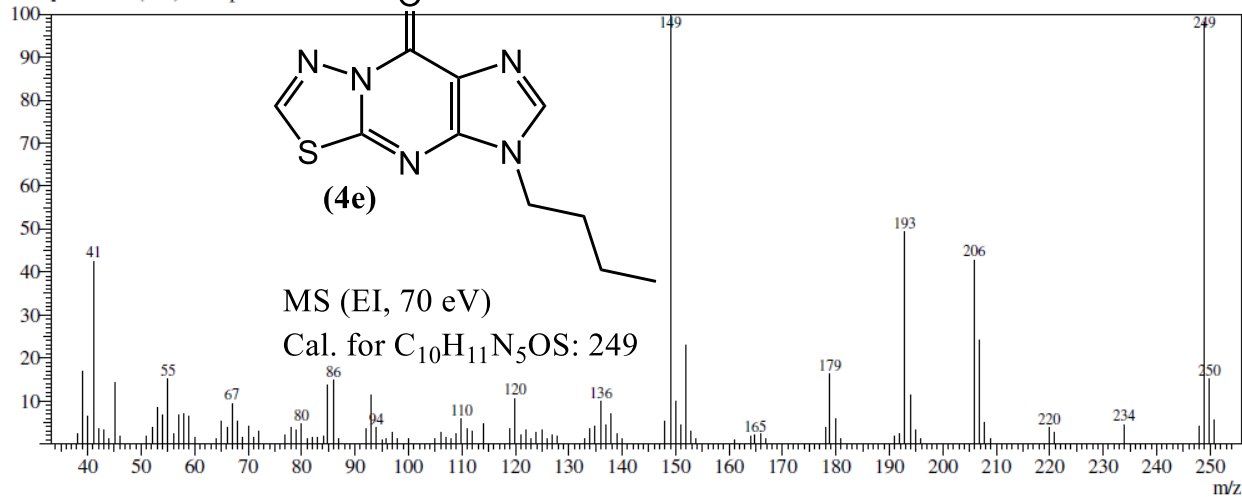
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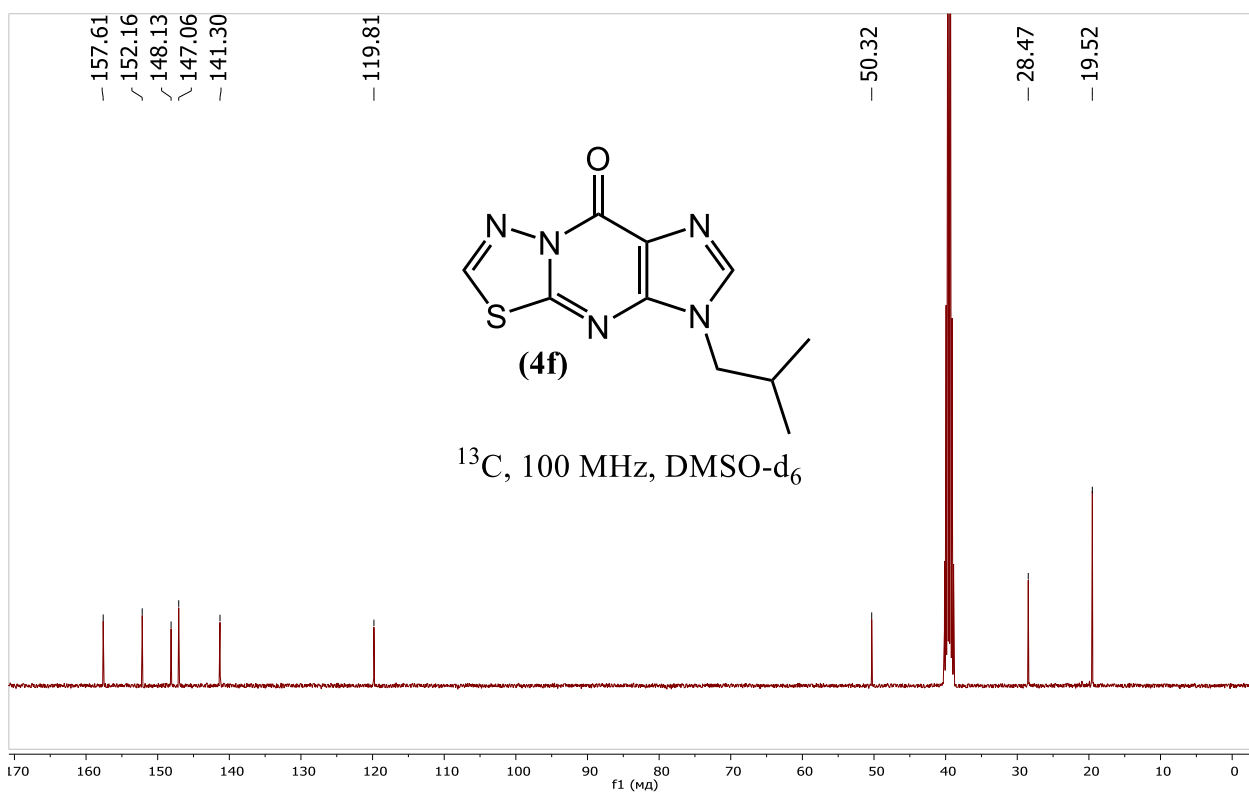
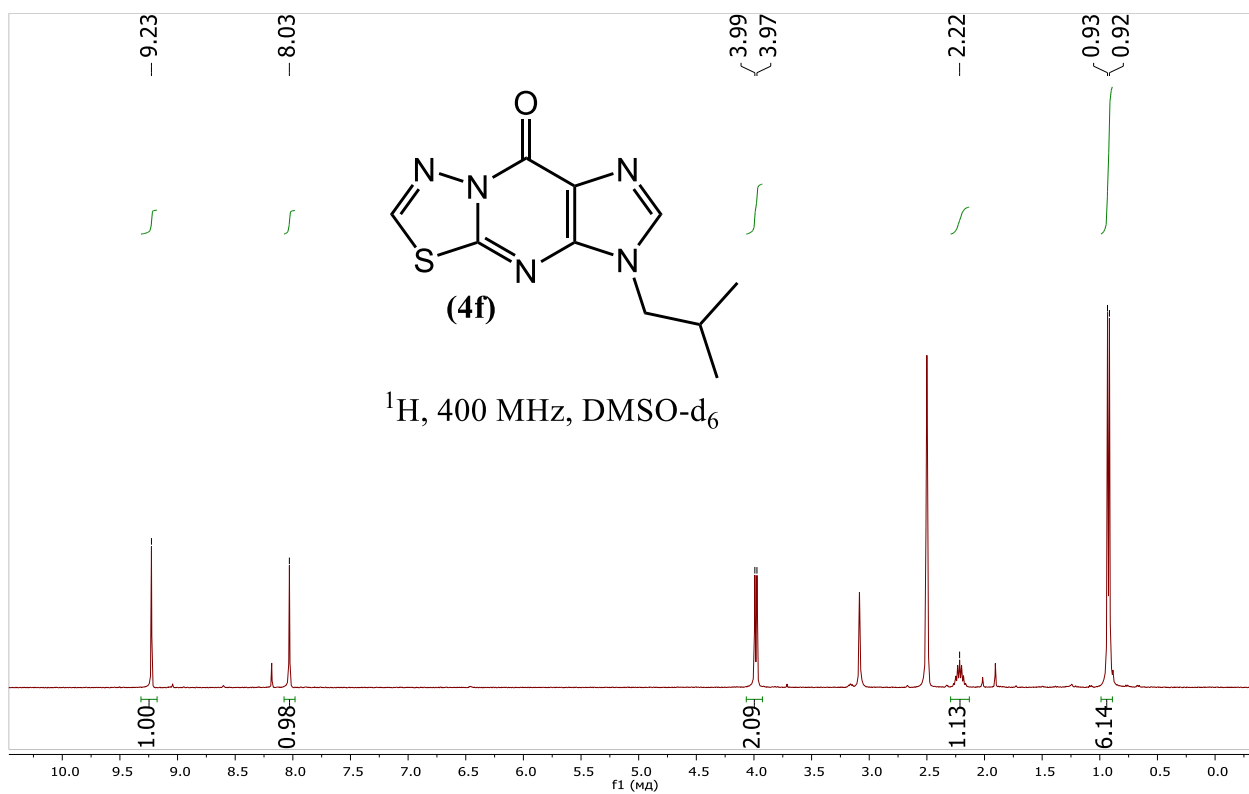
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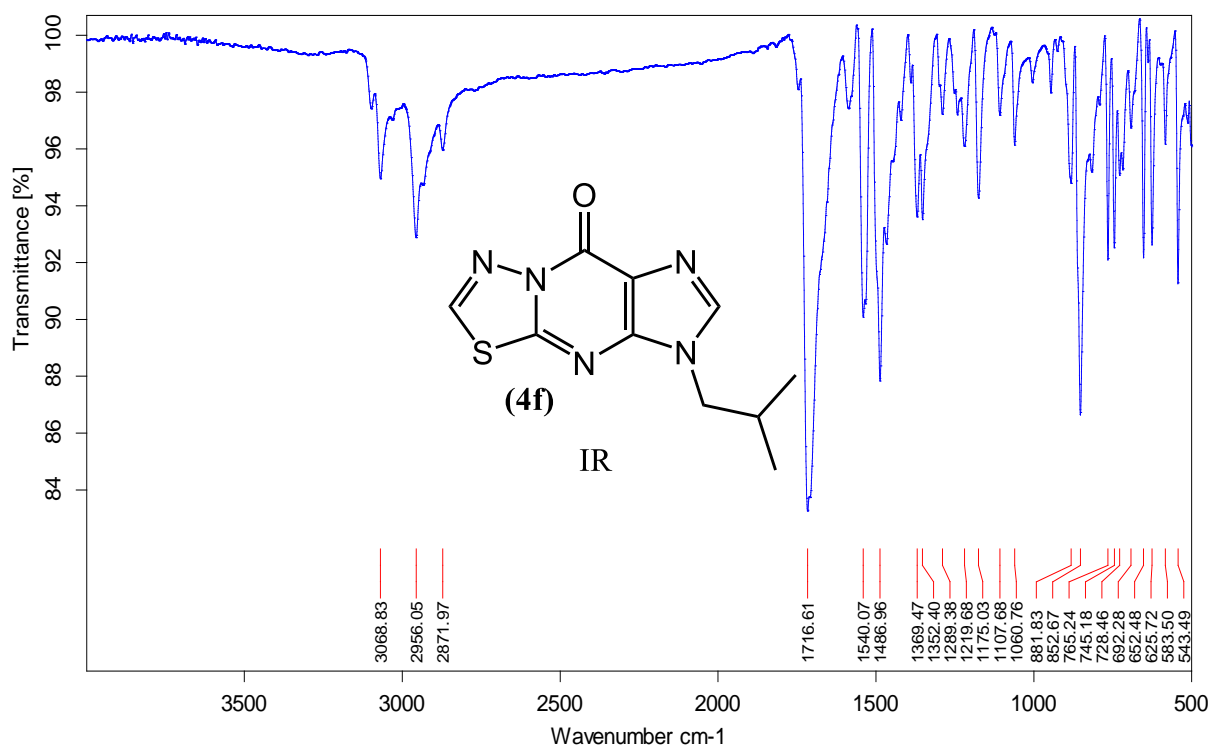
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RawMode:Single 2.255(883) BasePeak:149(3425941)

Фон.реж.:1.460(565) Group 1 - Event 1







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KC-1122

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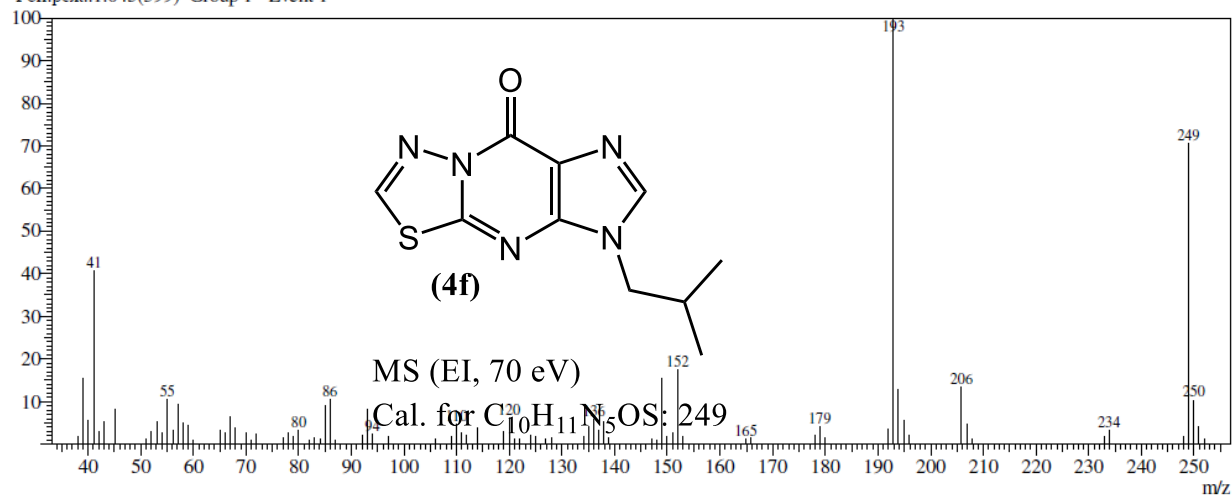
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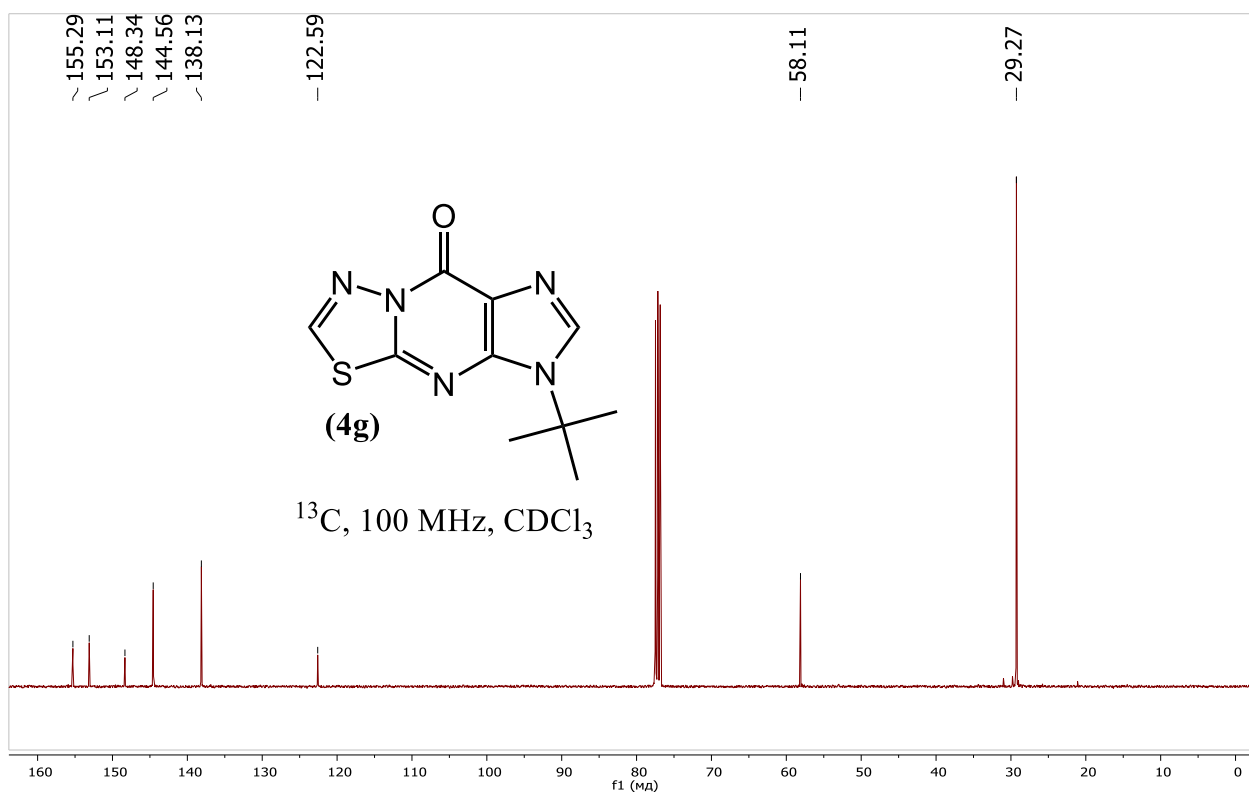
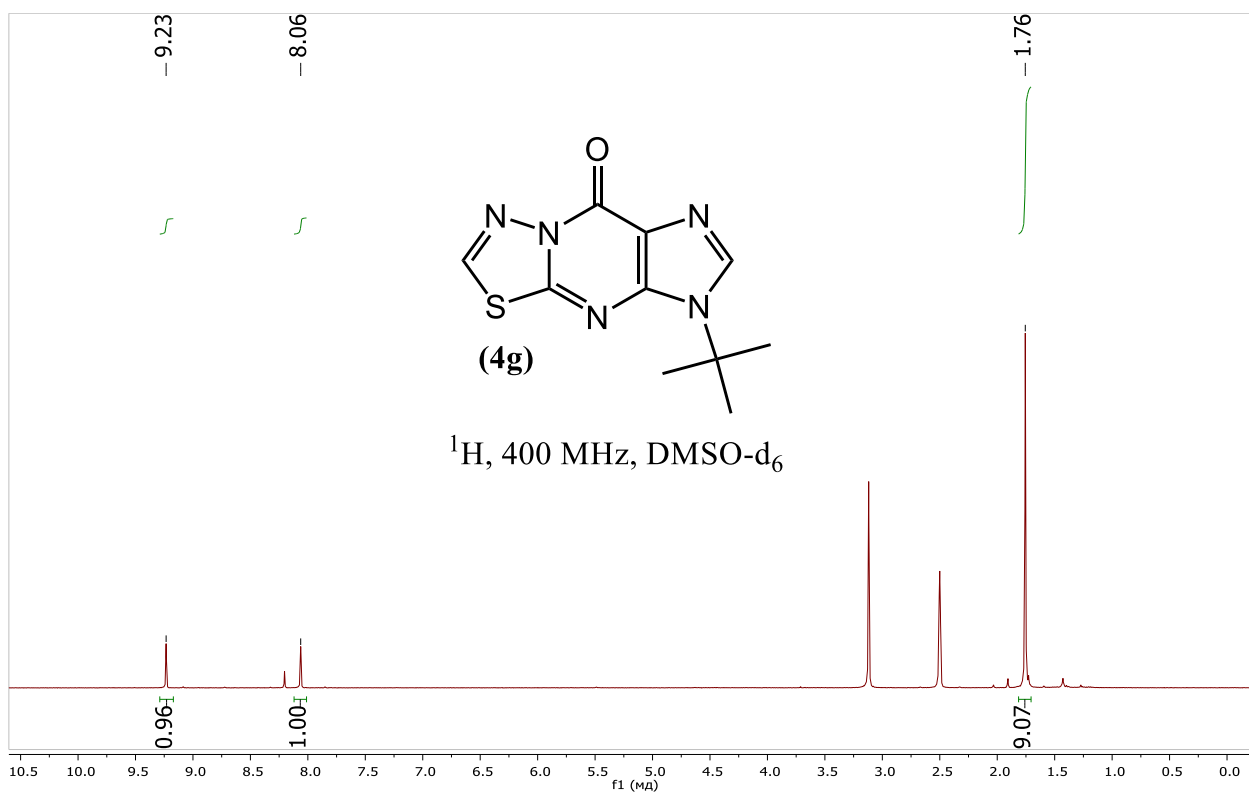
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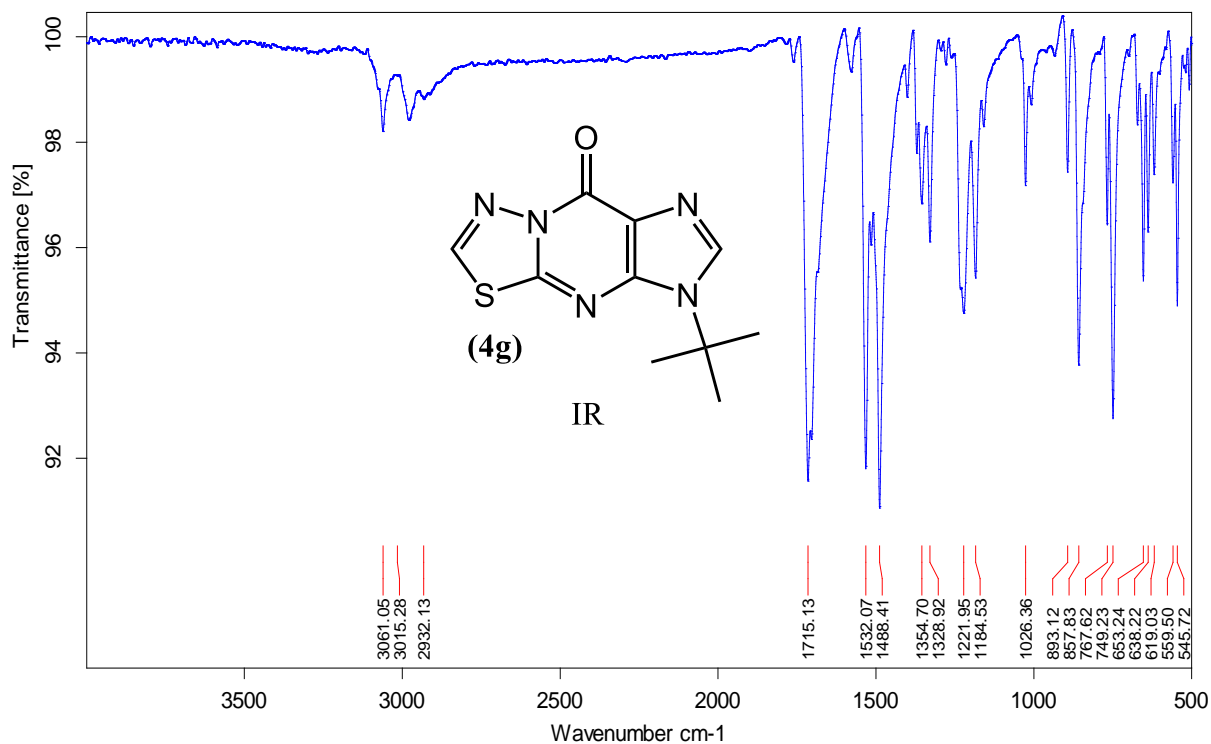
MassPeaks:85

RawMode:Single 2.855(1123) BasePeak:193(7007037)

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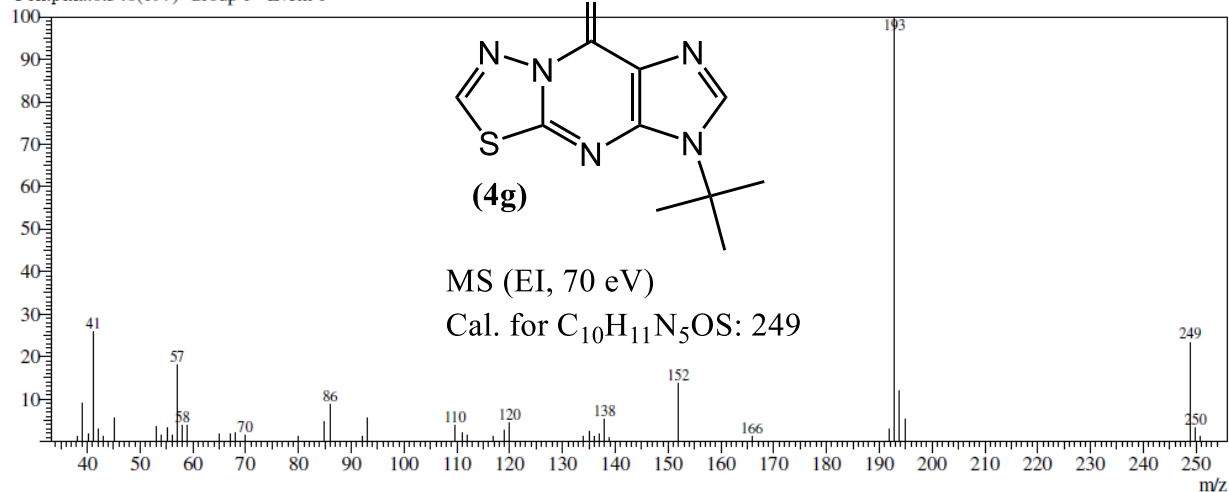
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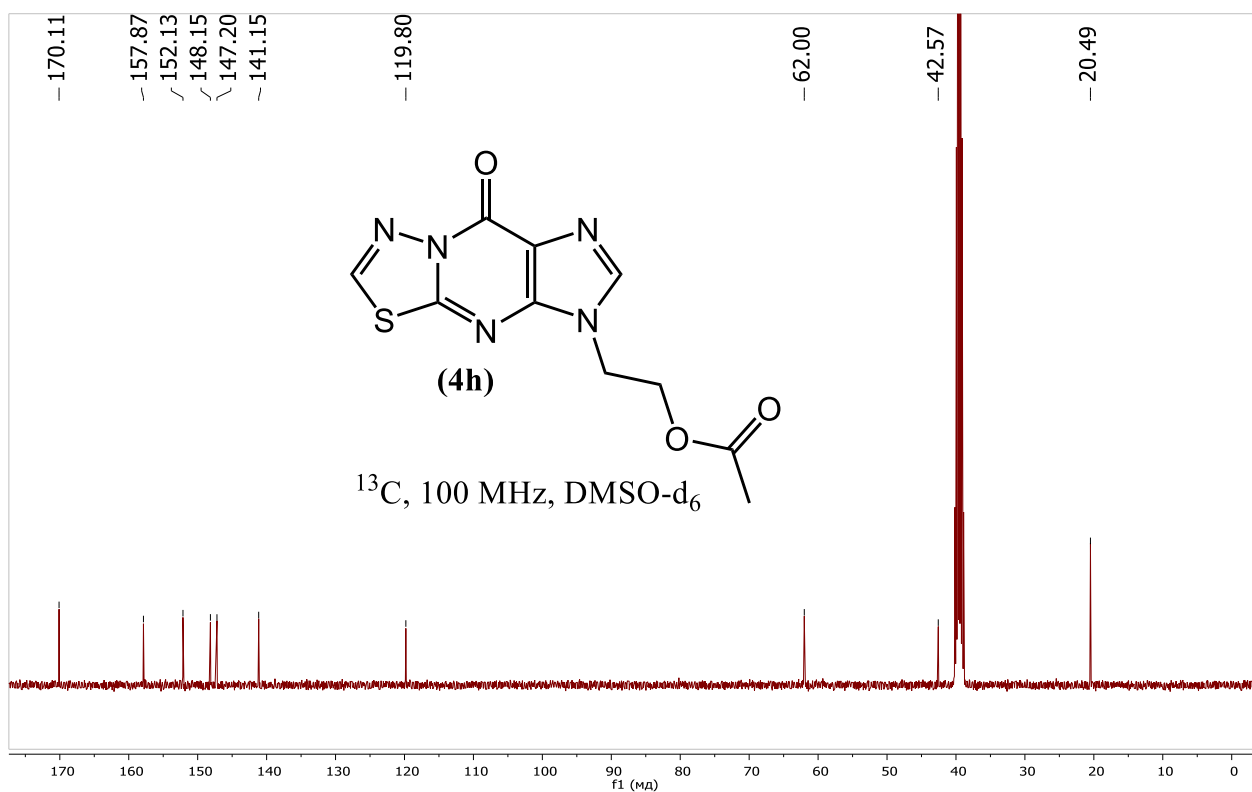
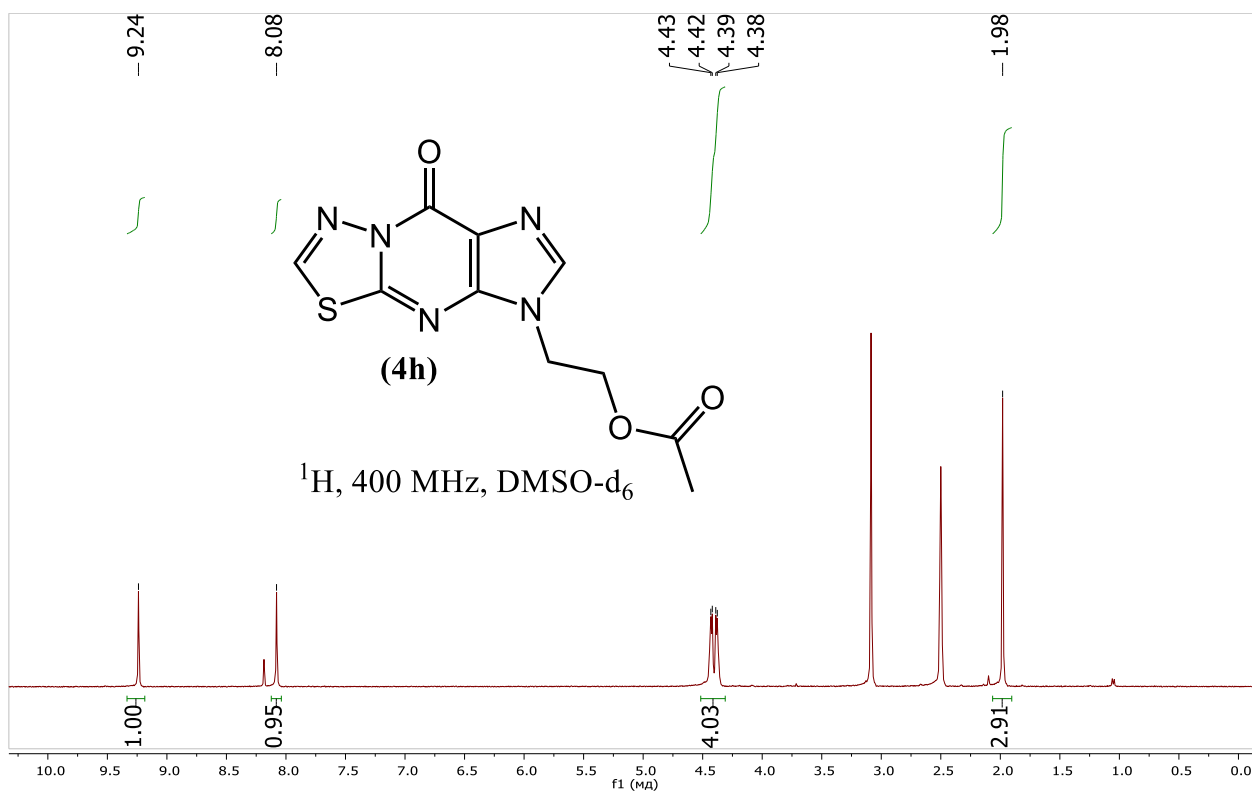
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MassPeaks:44

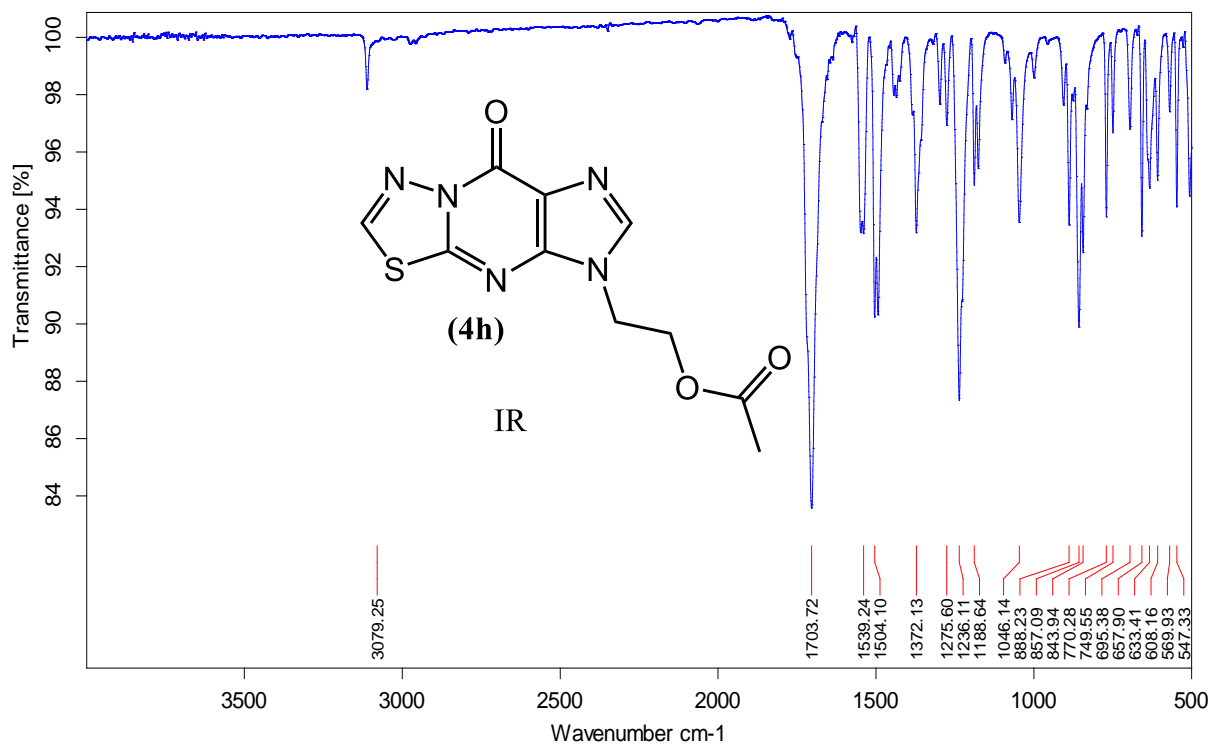
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Фон.реж.:0.540(197) Group 1 - Event 1









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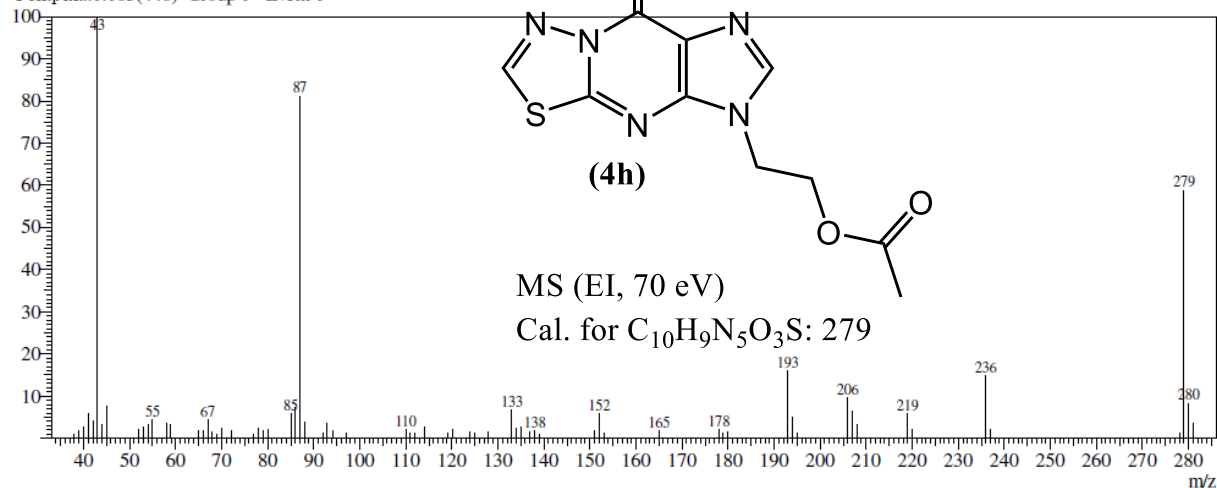
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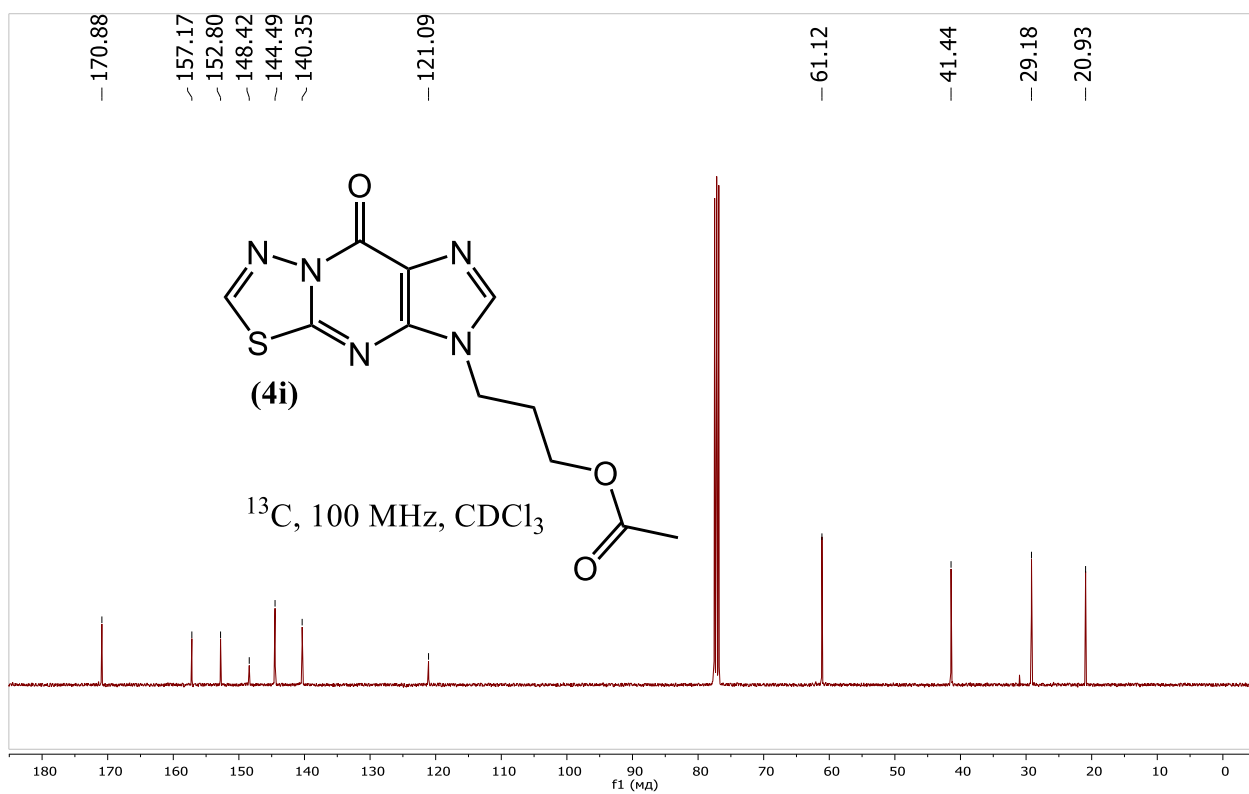
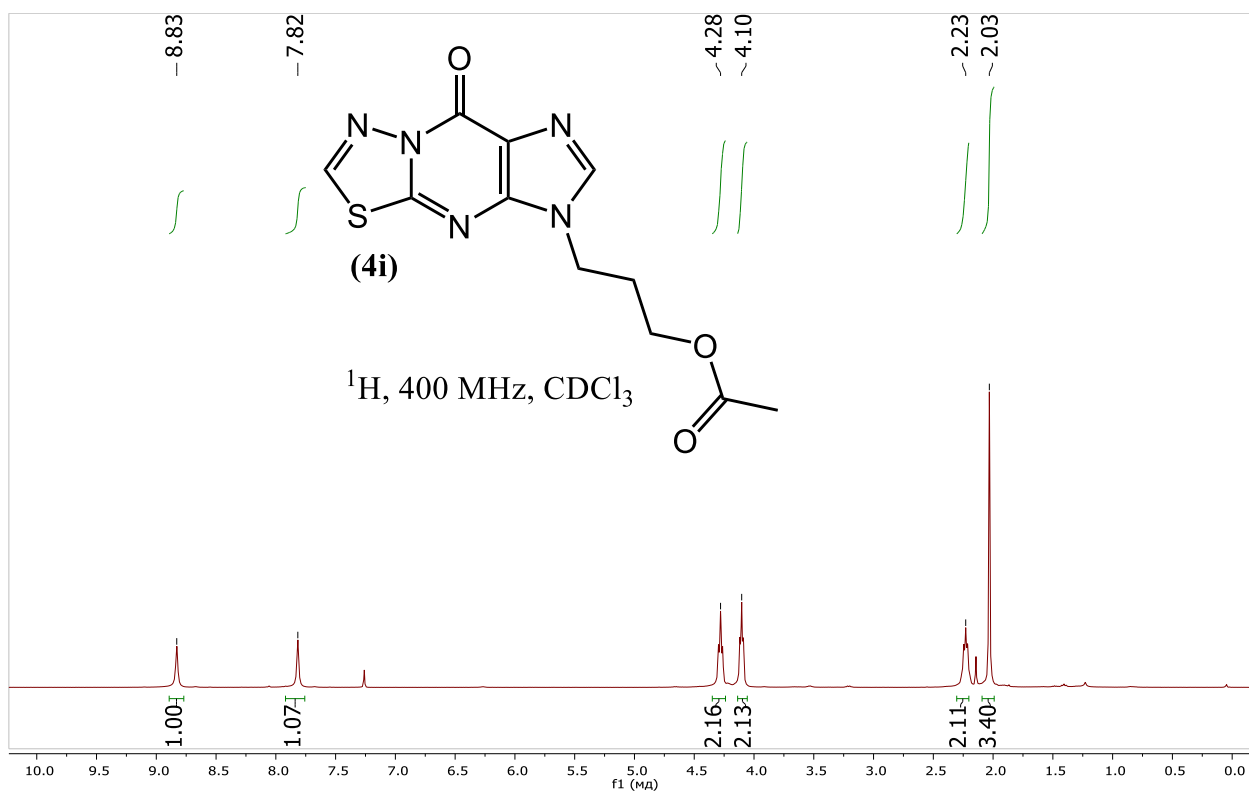
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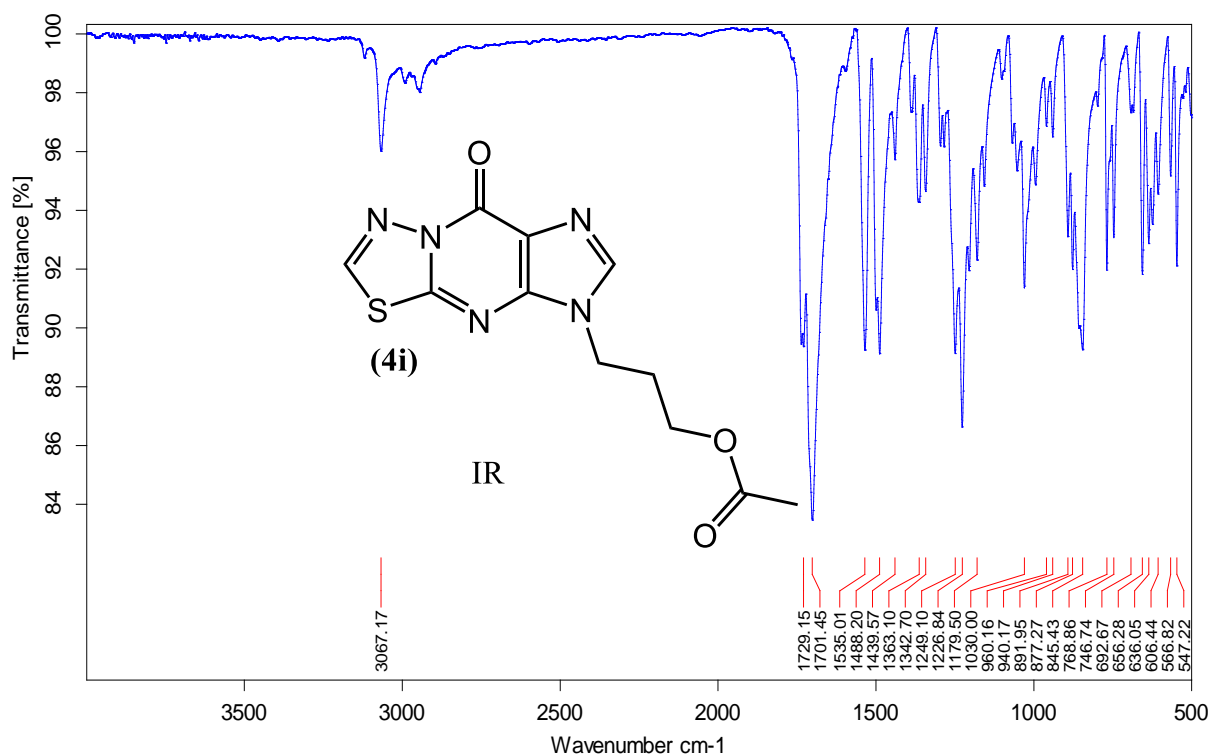
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Фон.реш.:1.163(446) Group 1 - Event 1







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KC-1136

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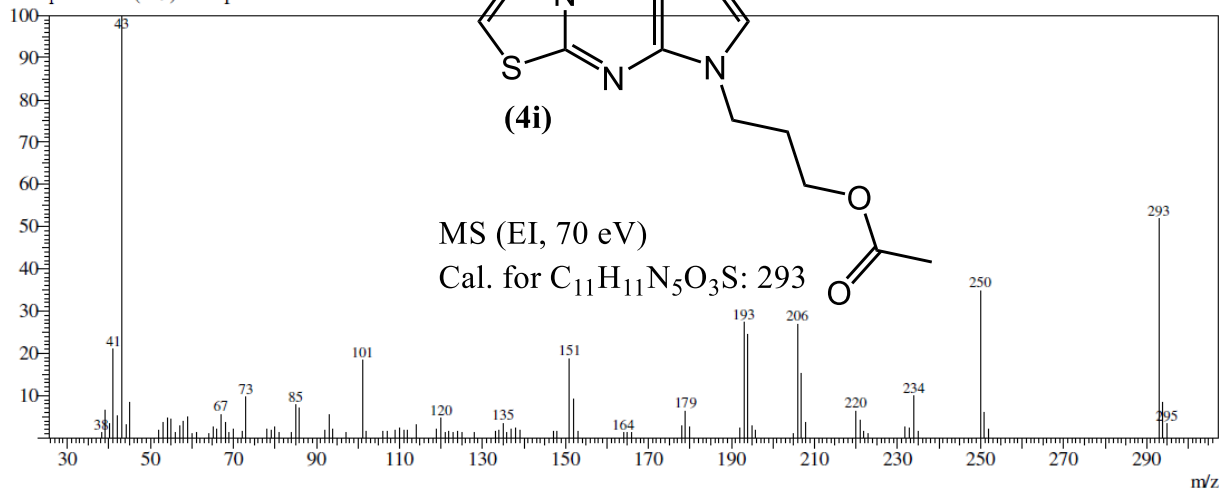
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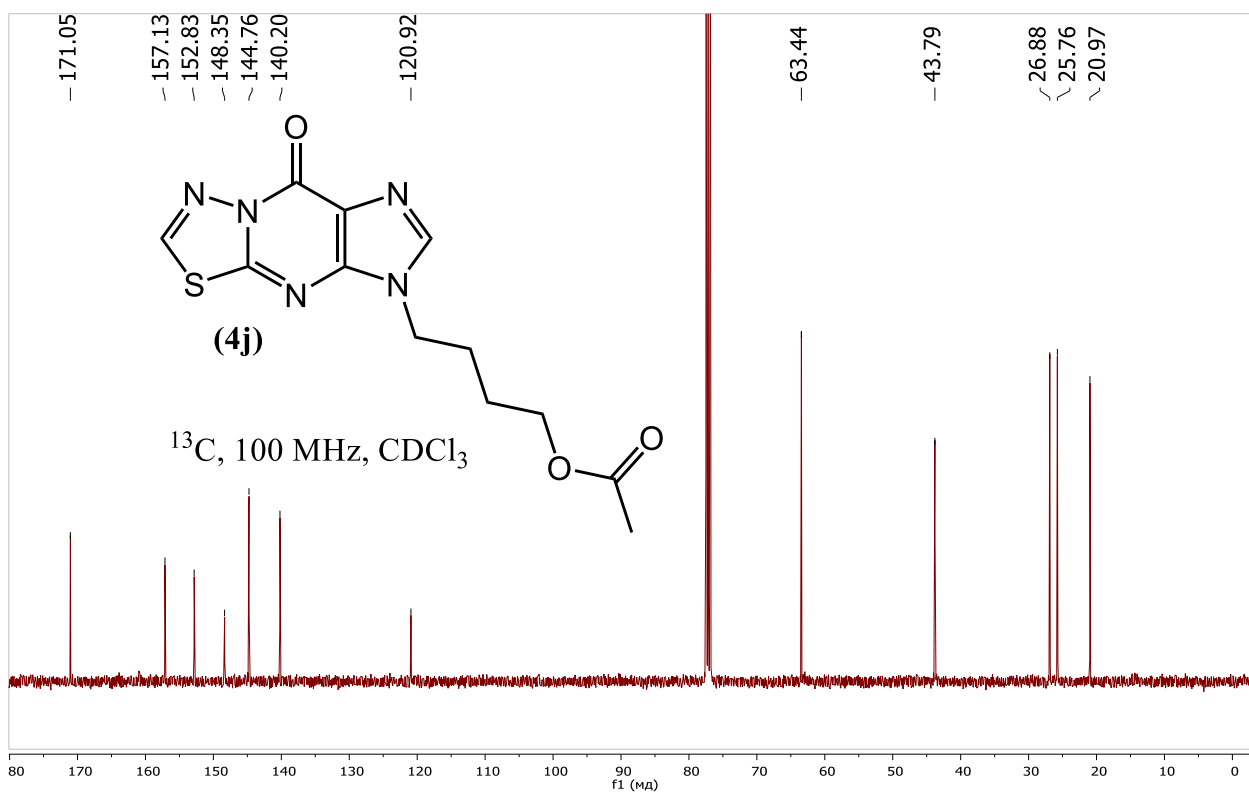
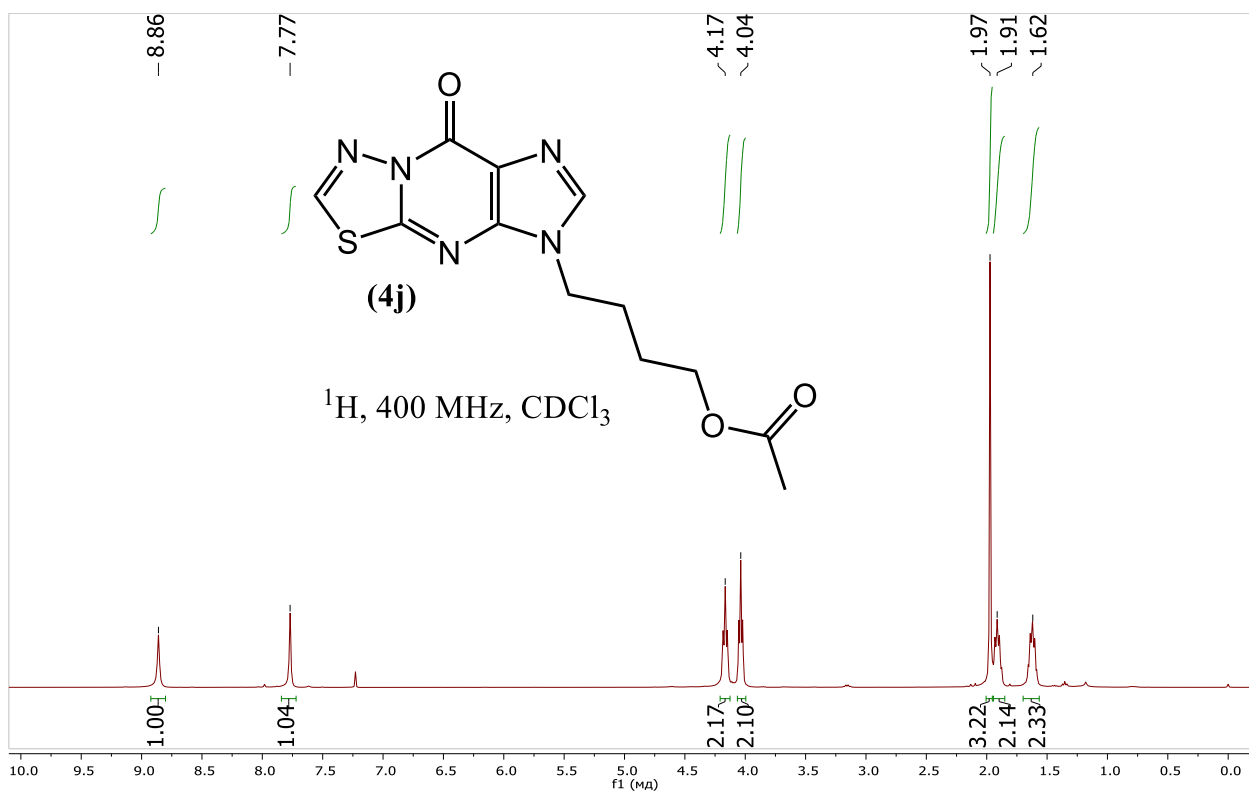
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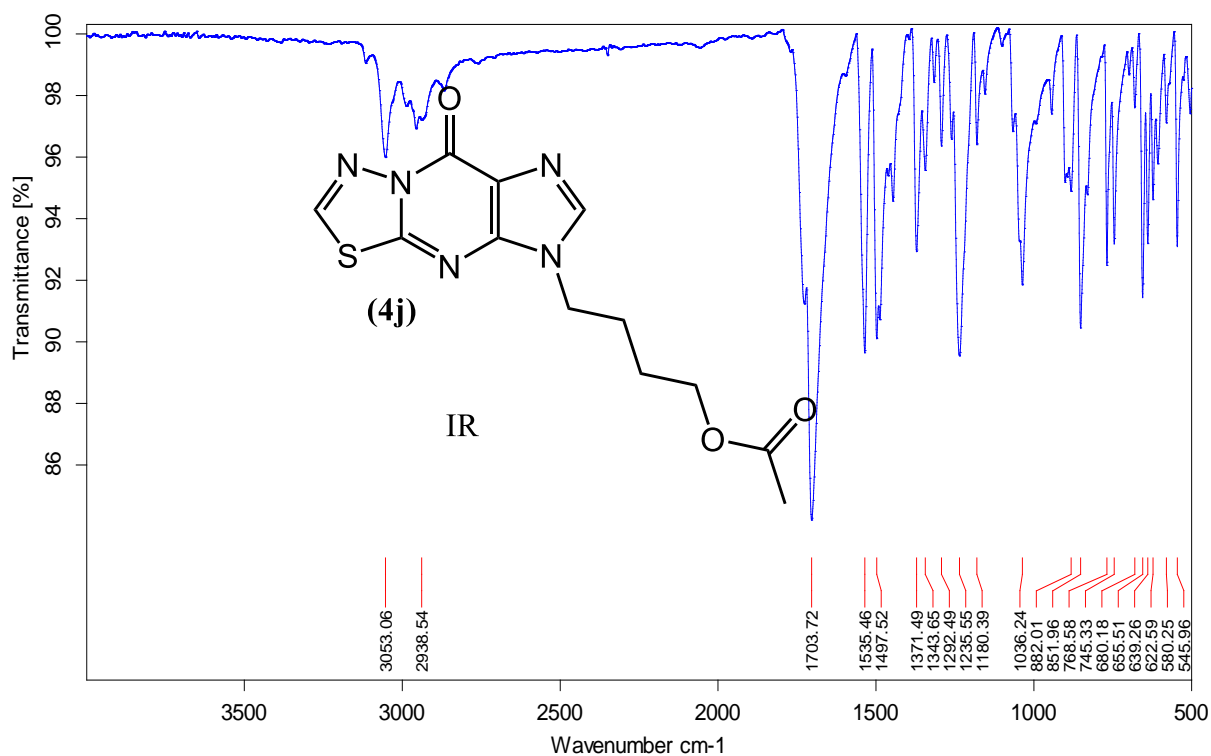
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Фон.реж.:1.470(569) Group 1 - Event 1





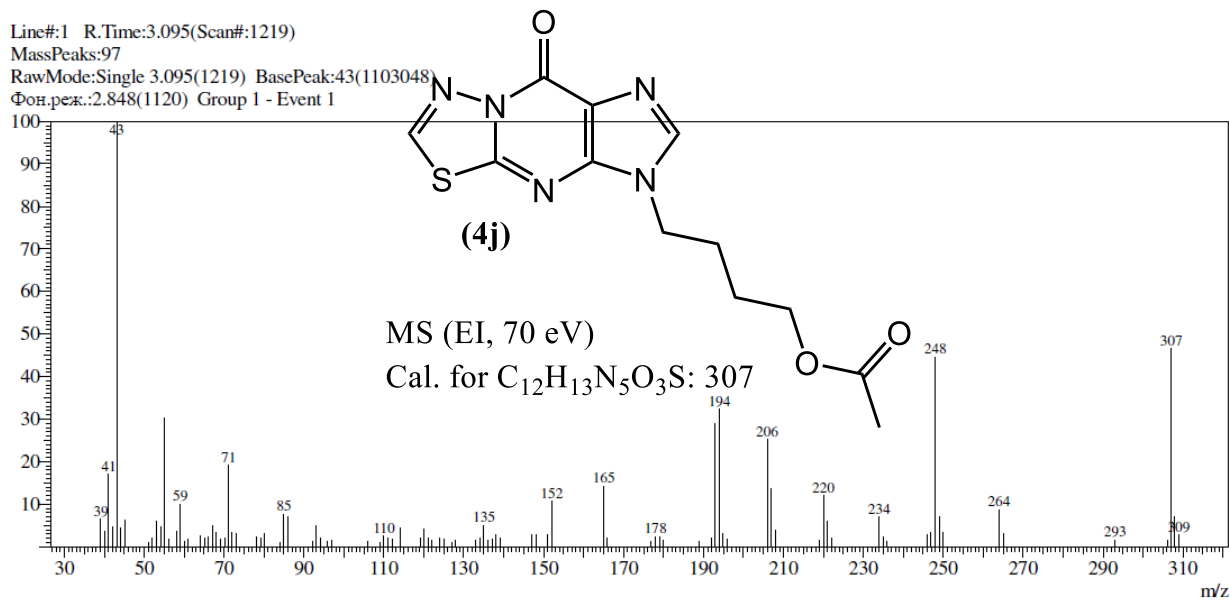


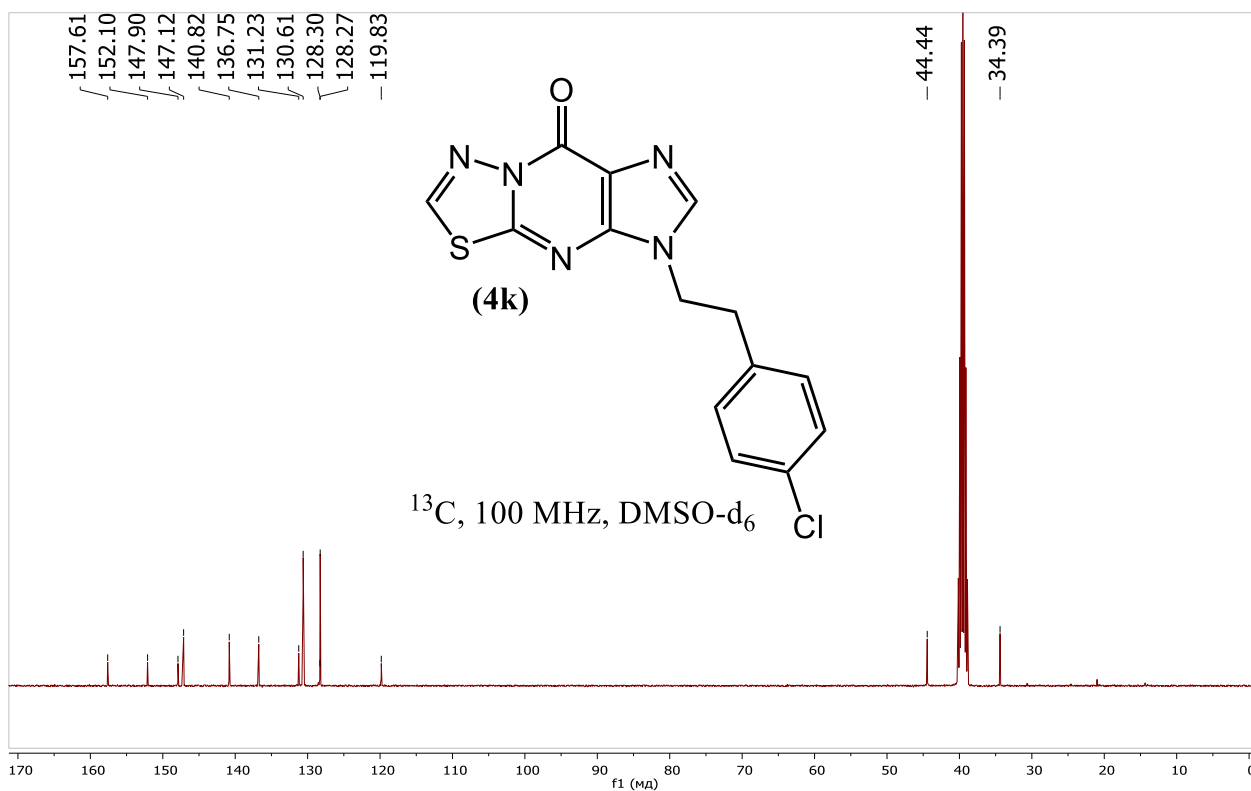
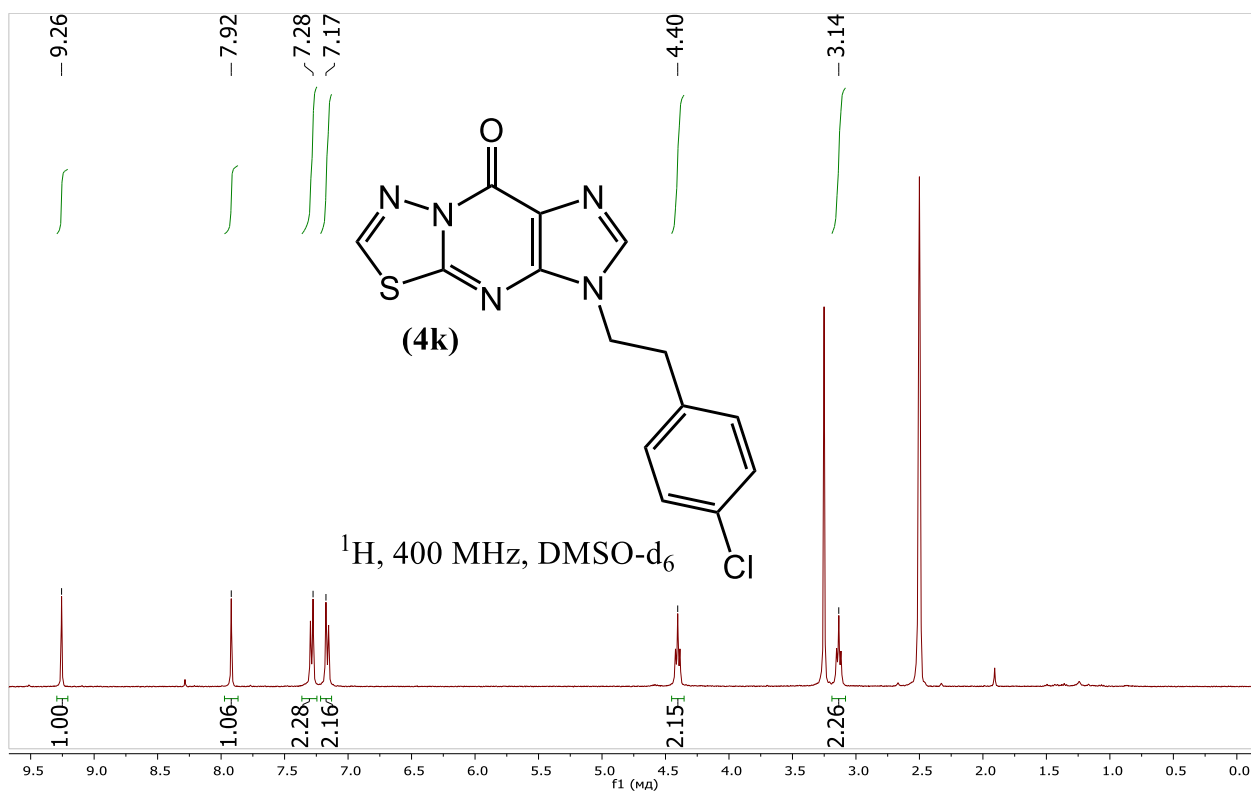
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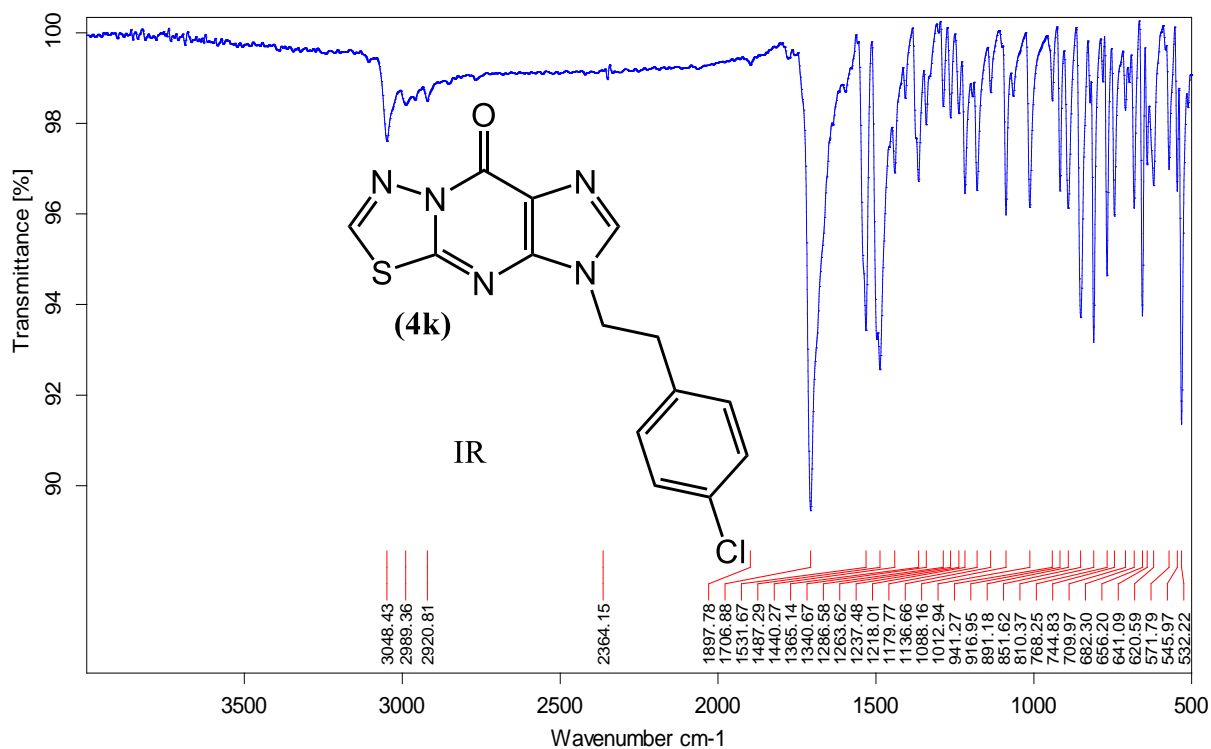
KC-1158

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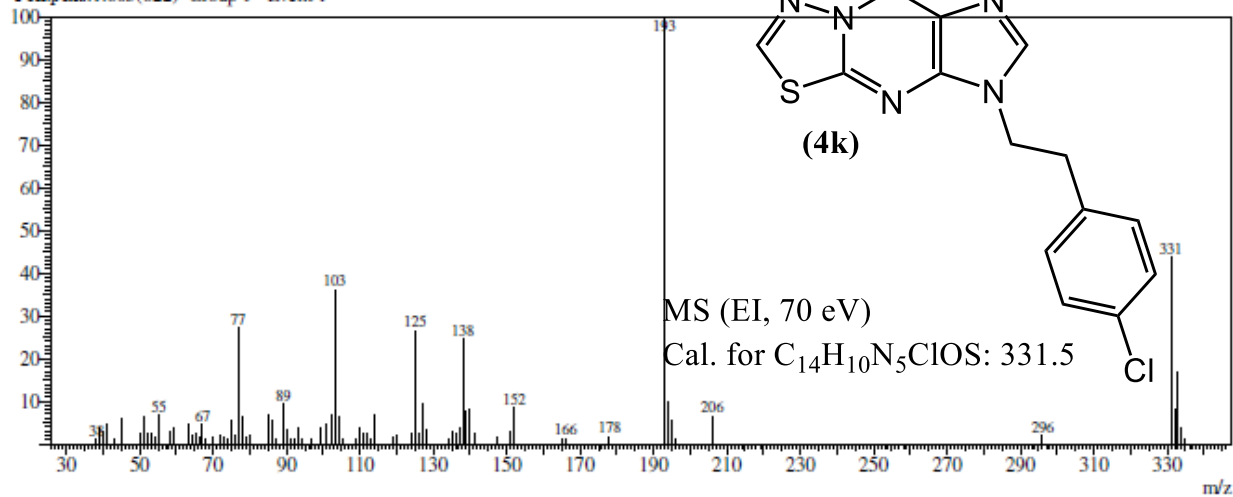
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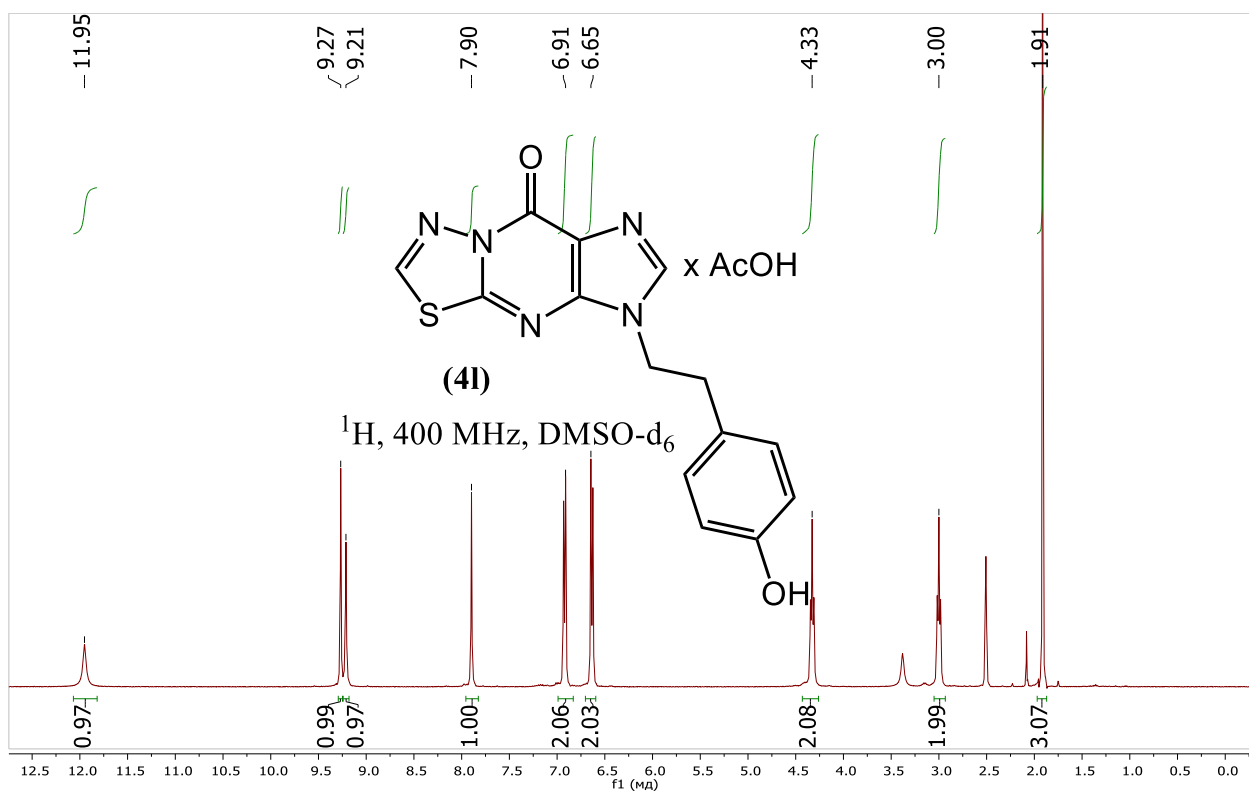
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MassPeaks:84

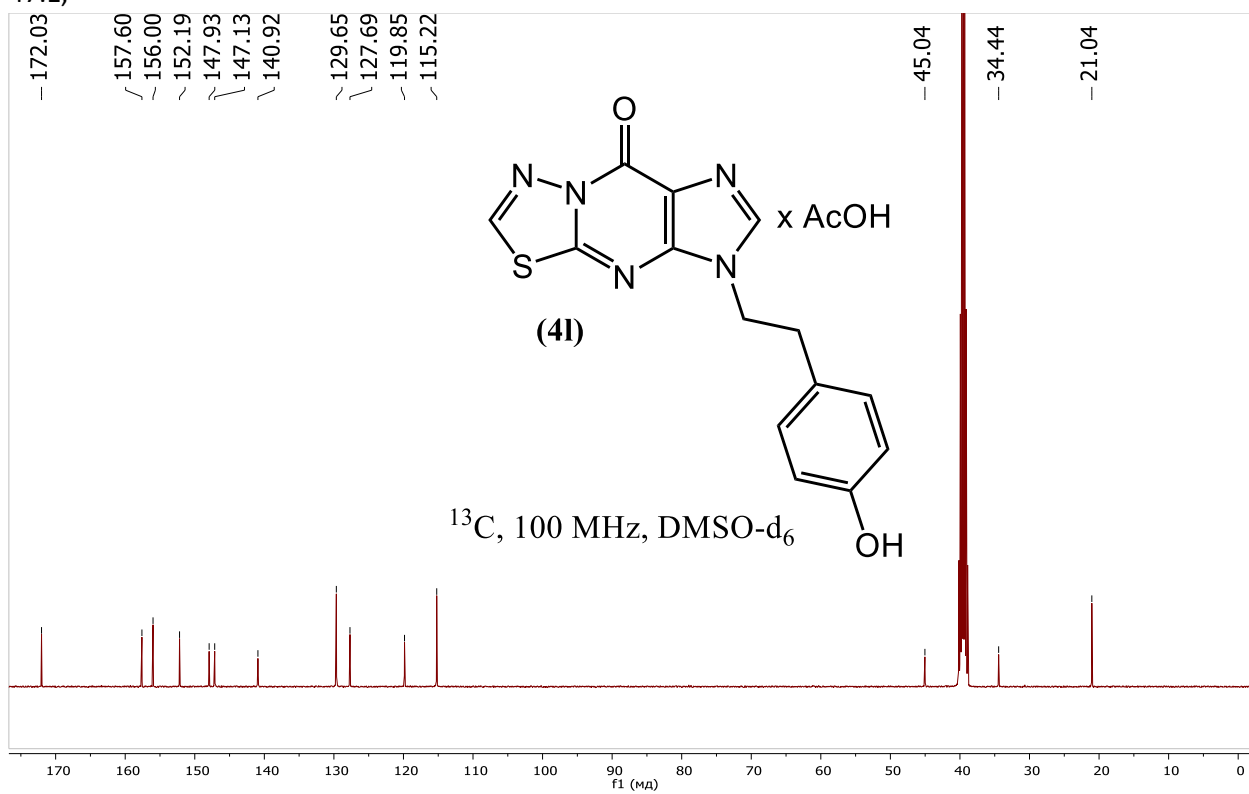
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Фон.реш.:1.603(622) Group 1 - Event 1

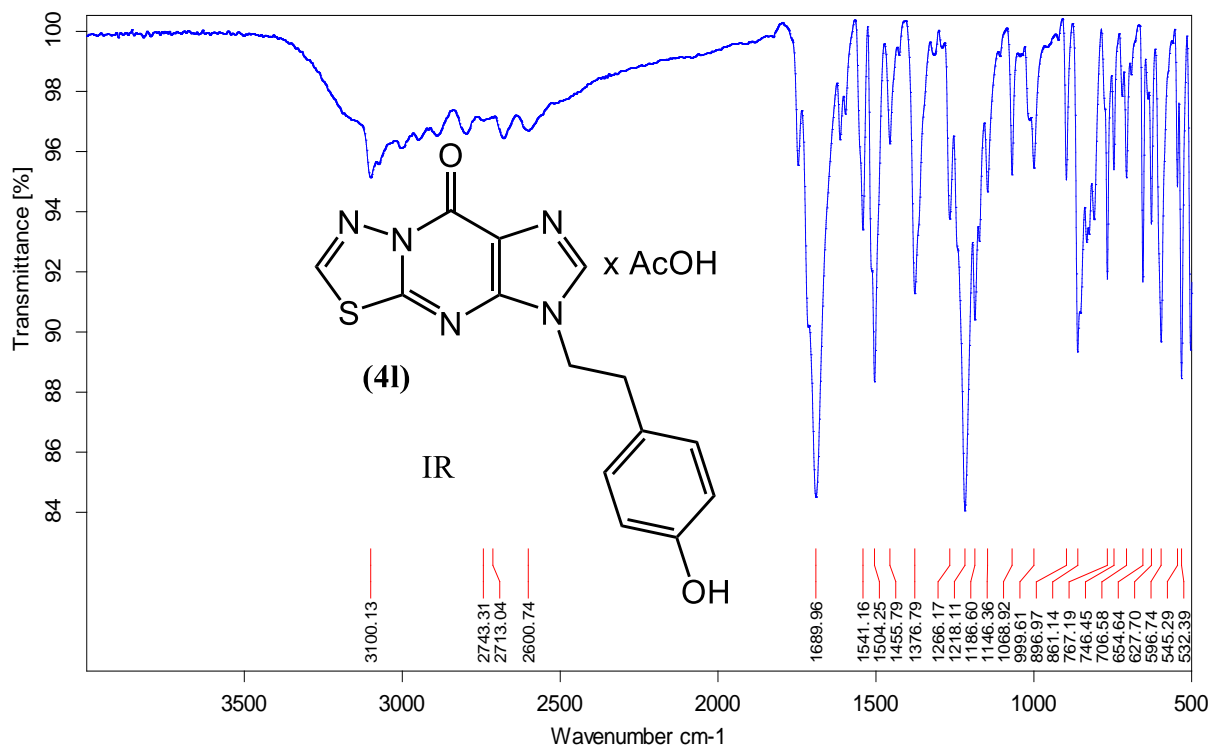




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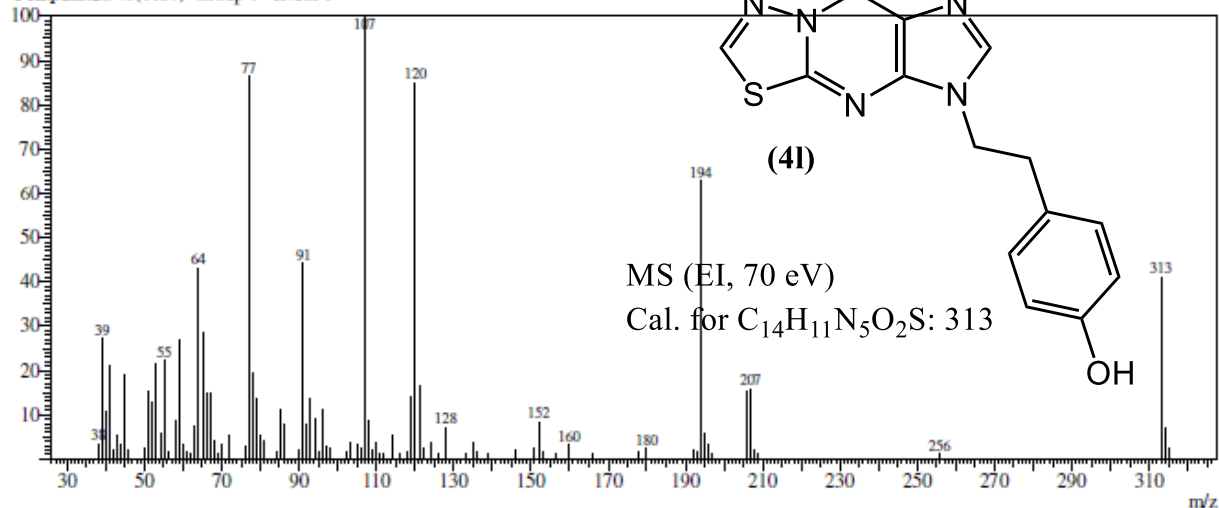
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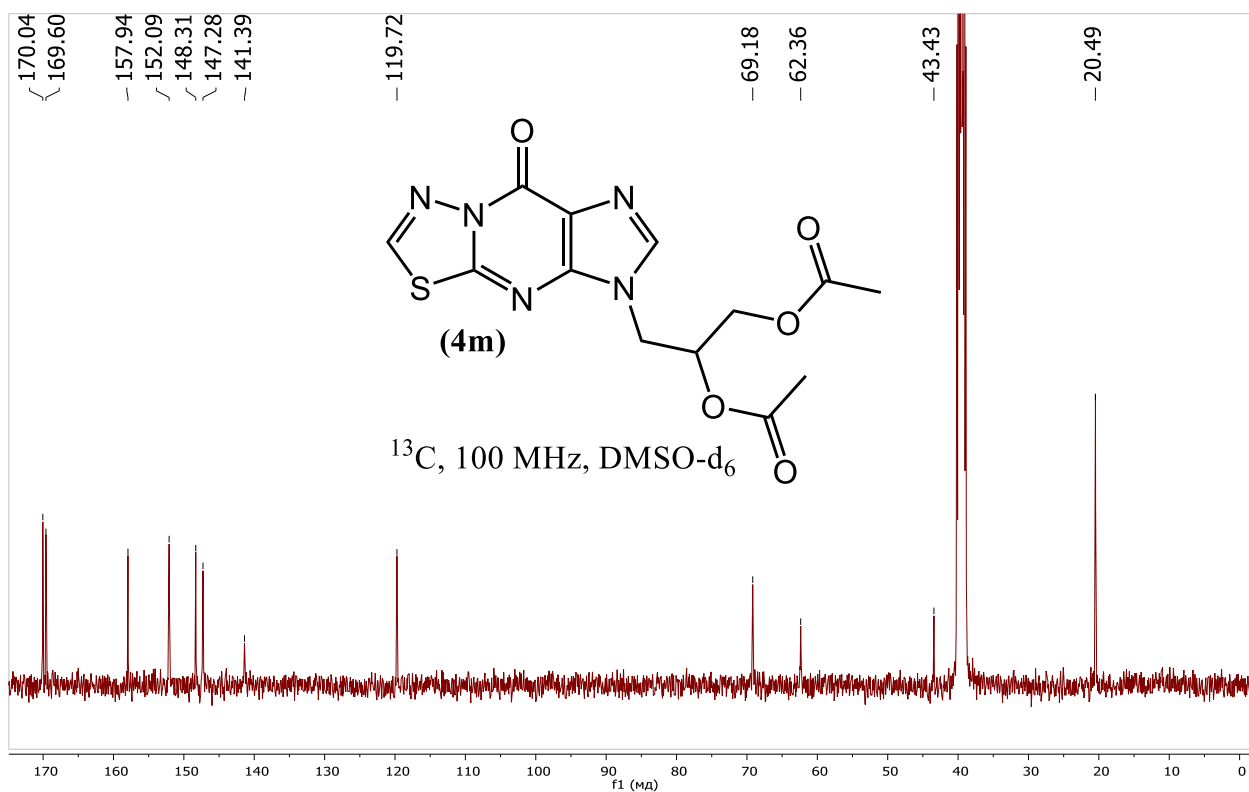
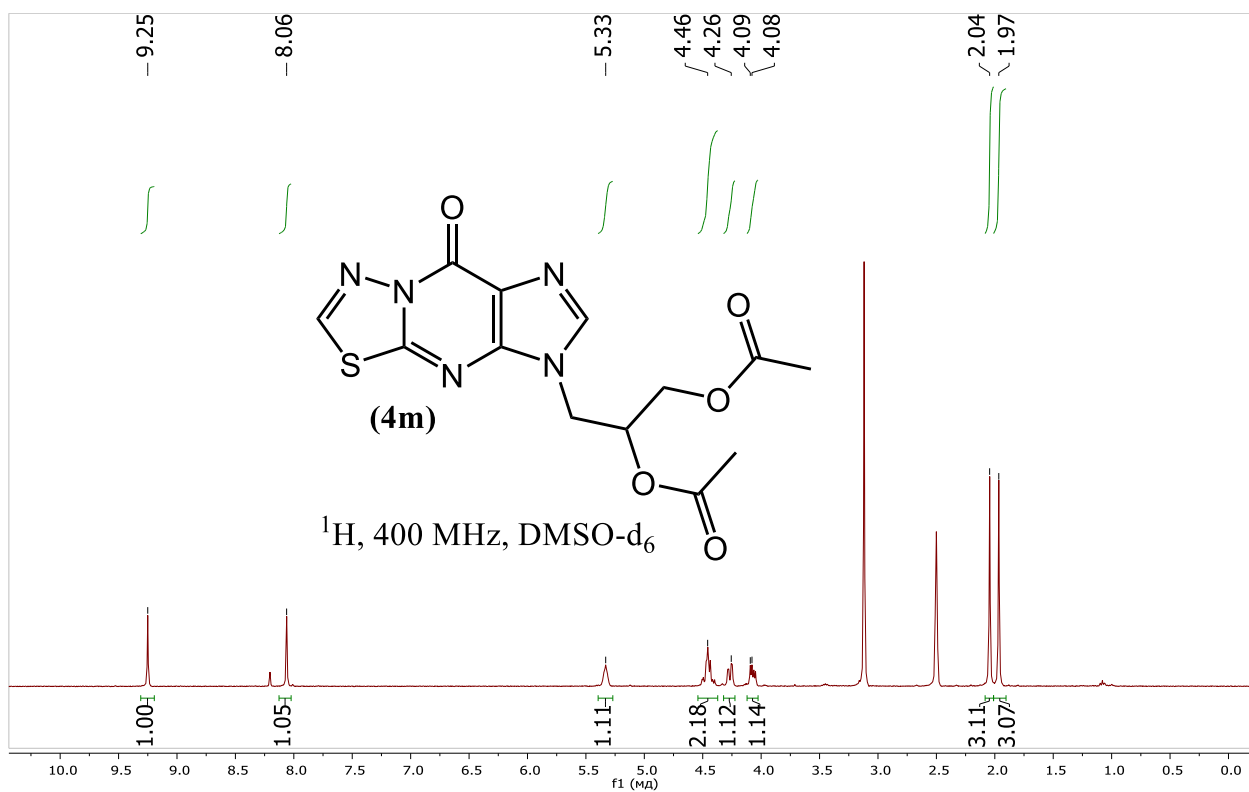
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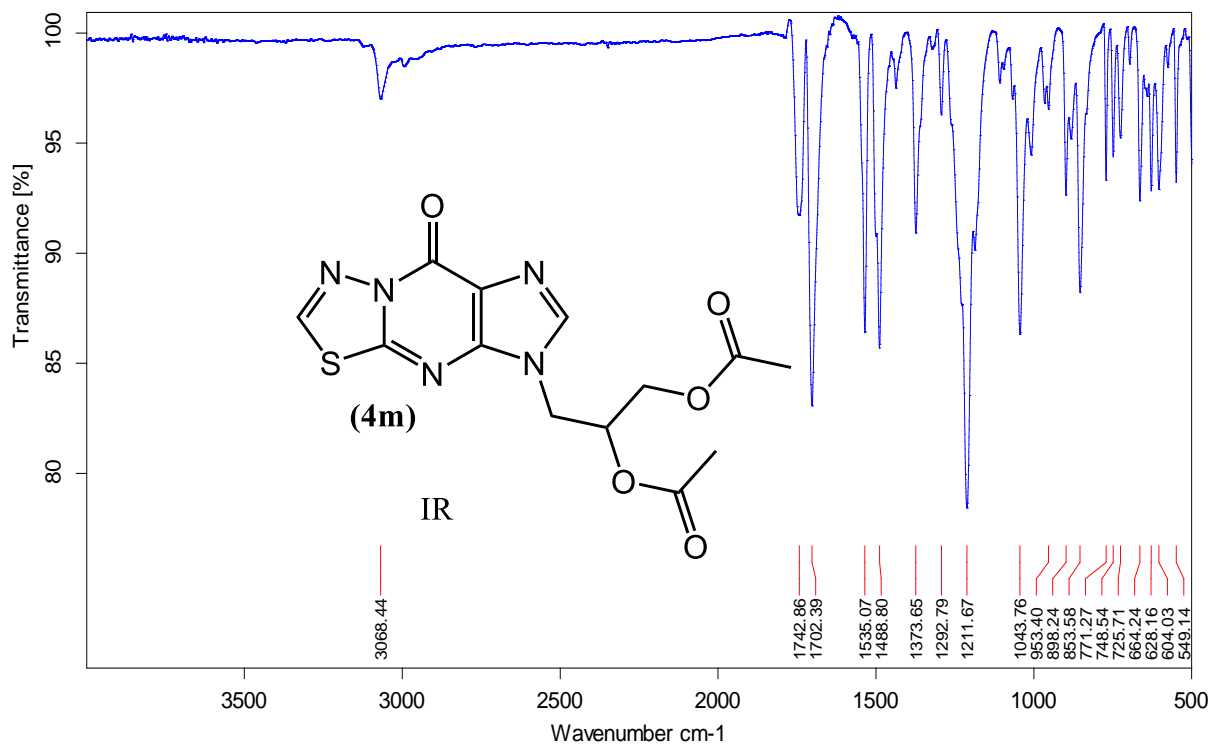
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Фон. пик.:2.940(1157) Group 1 - Event 1







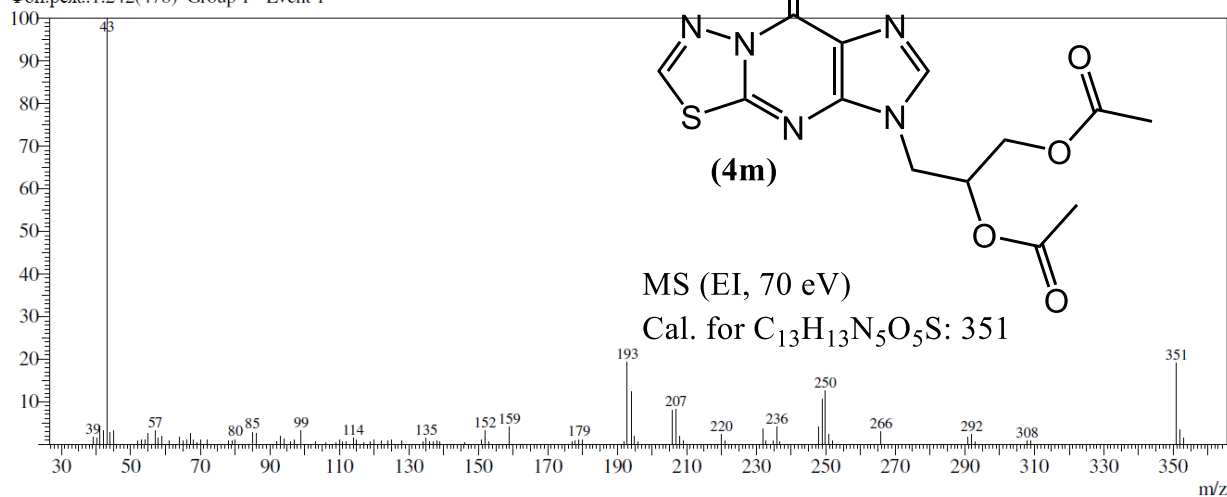
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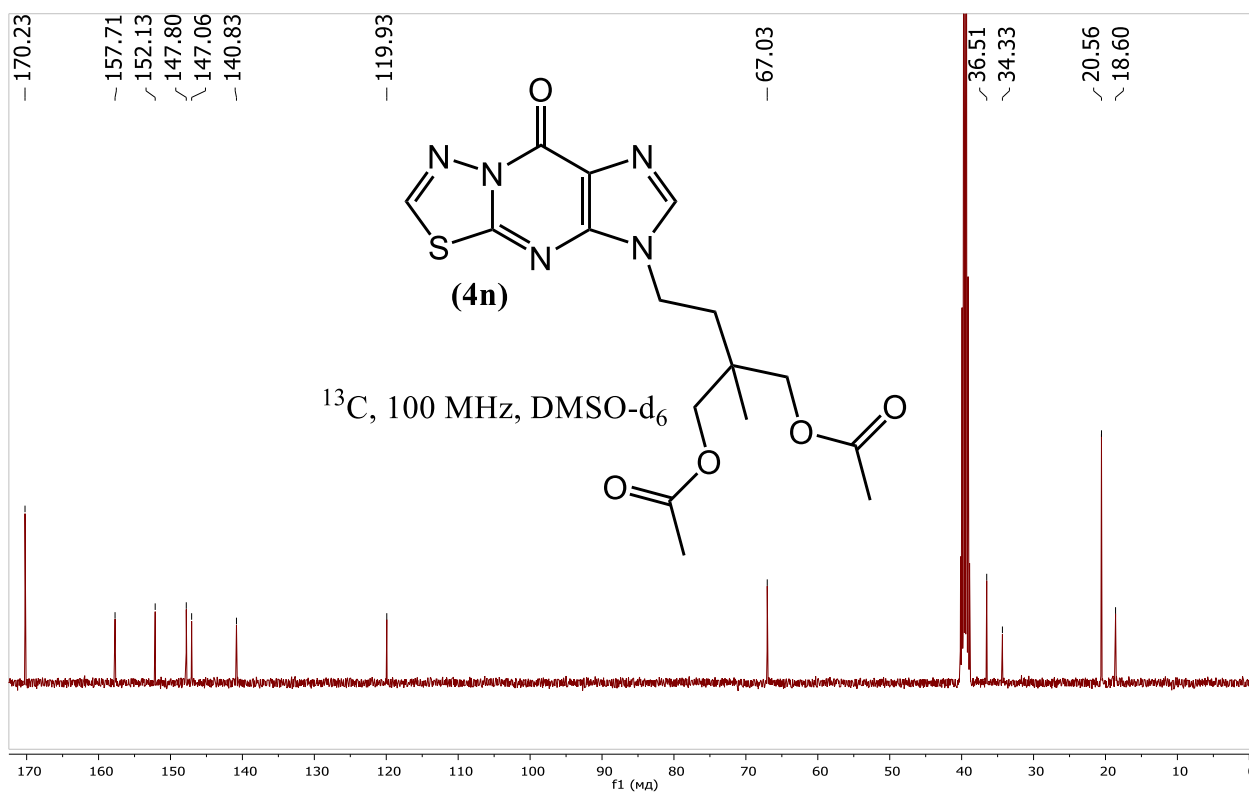
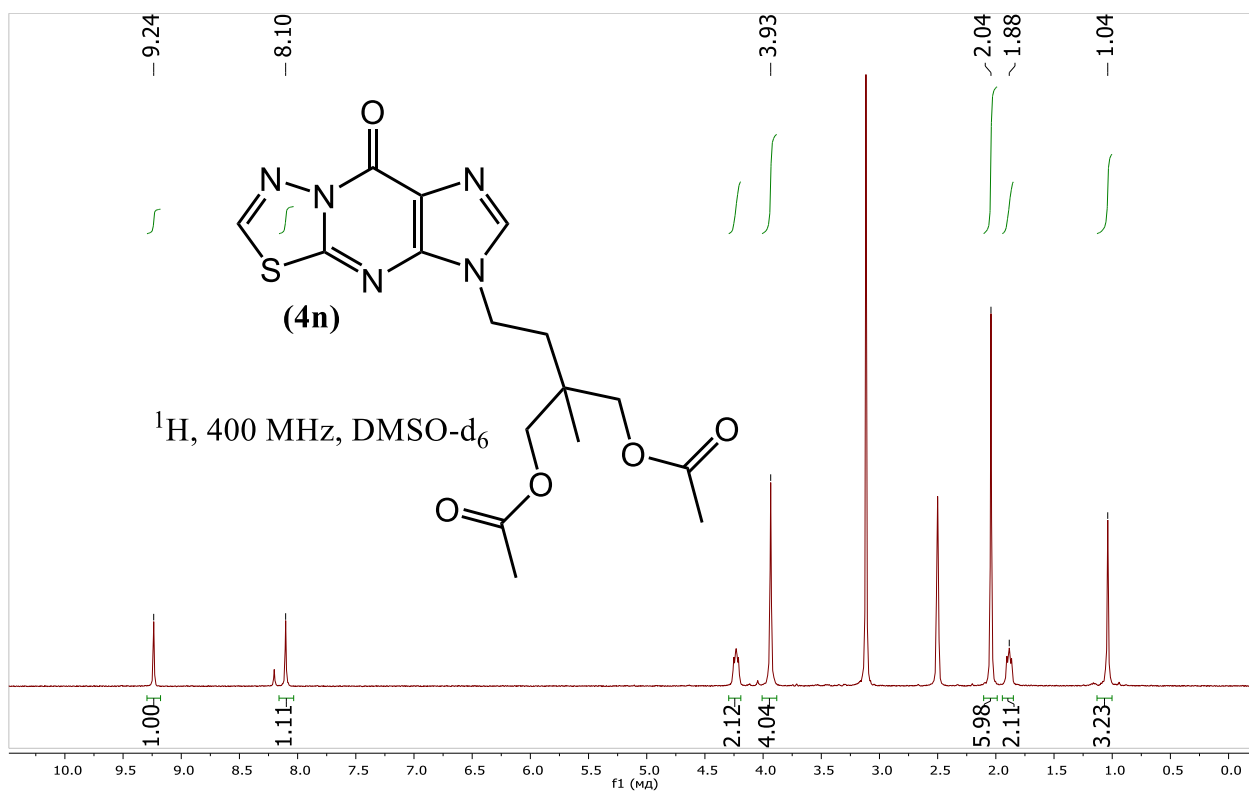
KC-1197

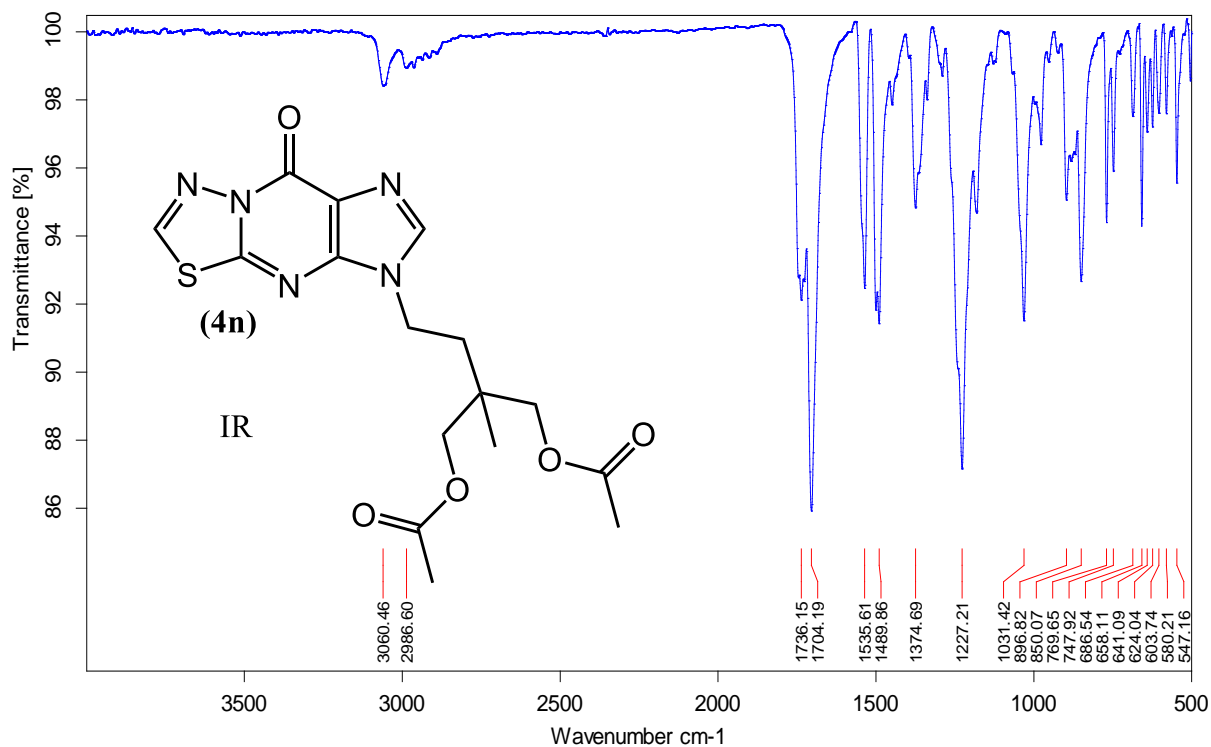
04/03/2021

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Line#: 1 R.Time: 3.820 (Scan#: 1509)  
 MassPeaks: 94  
 RawMode: Single 3.820 (1509) BasePeak: 43 (6026440)  
 Фон. рож.: 1.242 (478) Group 1 - Event 1







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KC-1222

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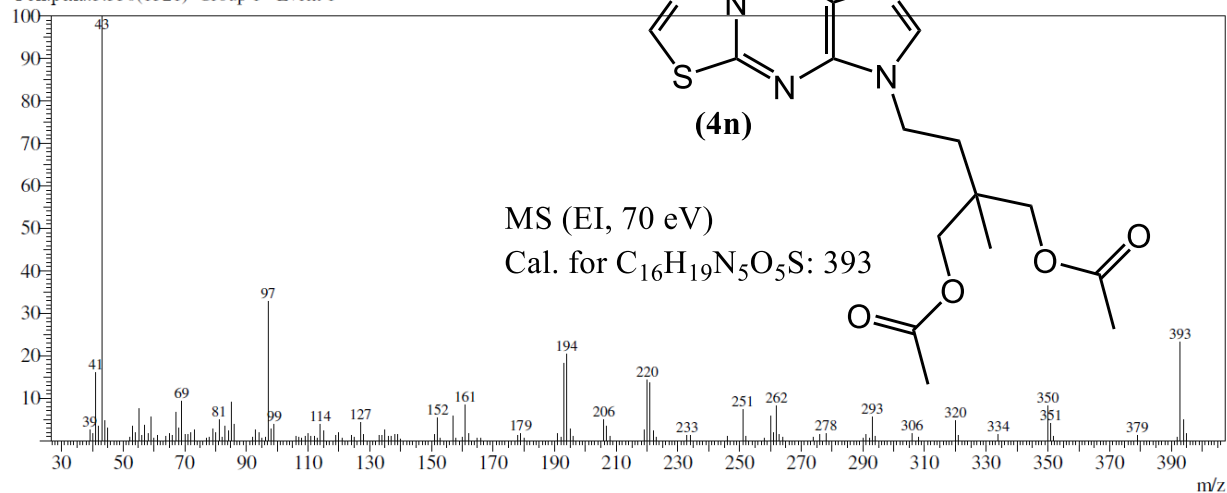
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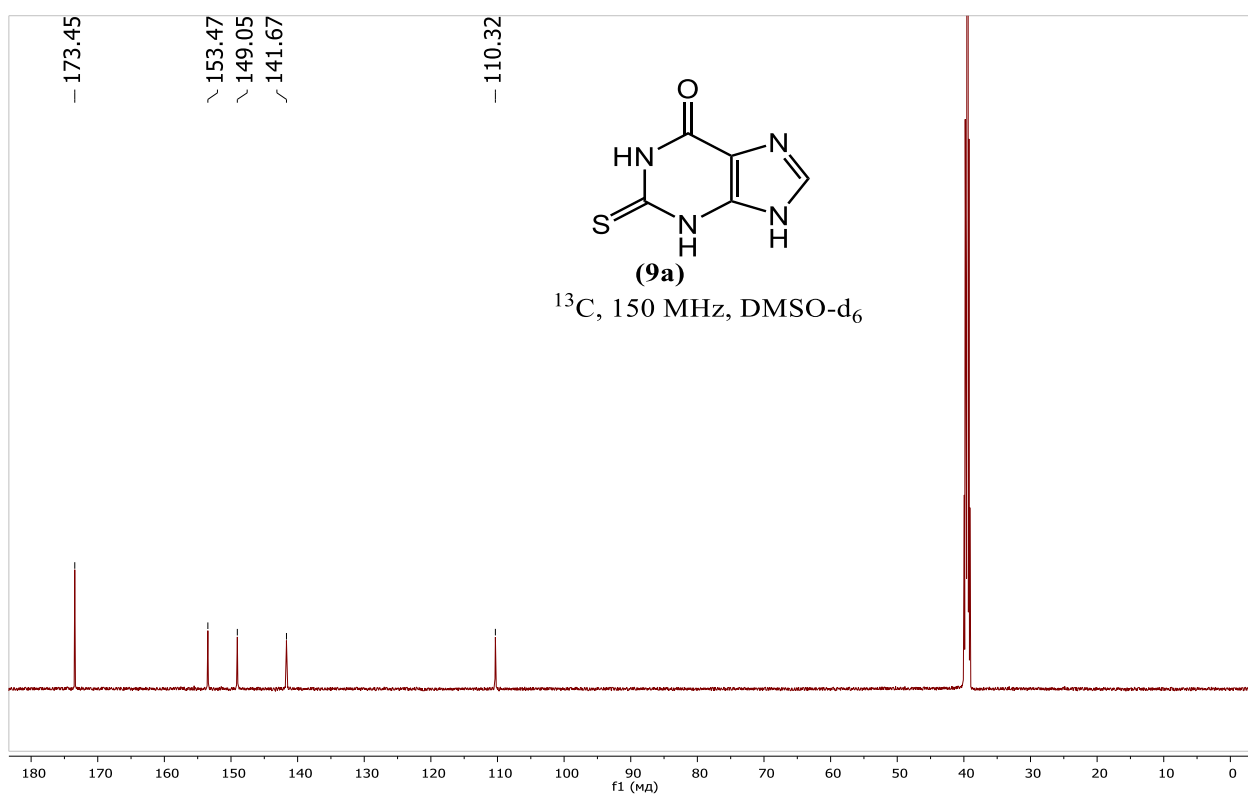
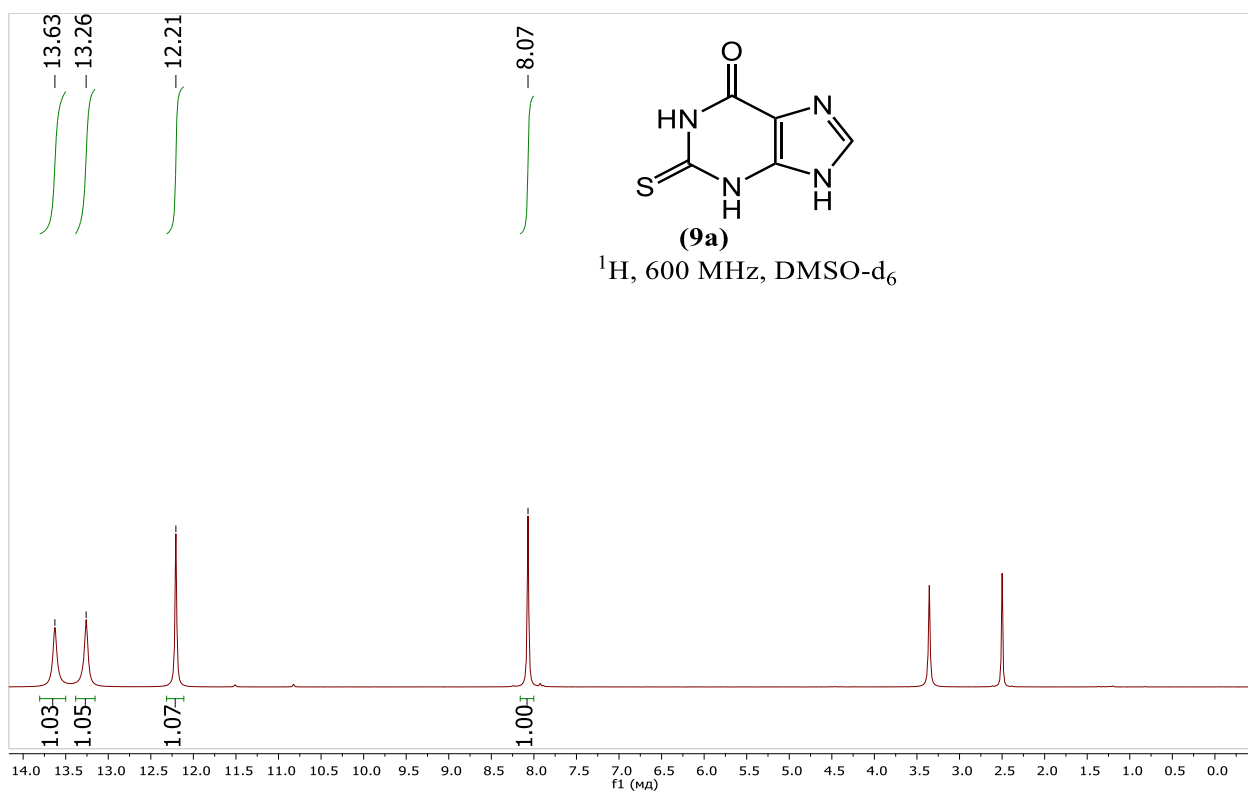
Line#:1 R.Time:5.250(Scan#:2081)

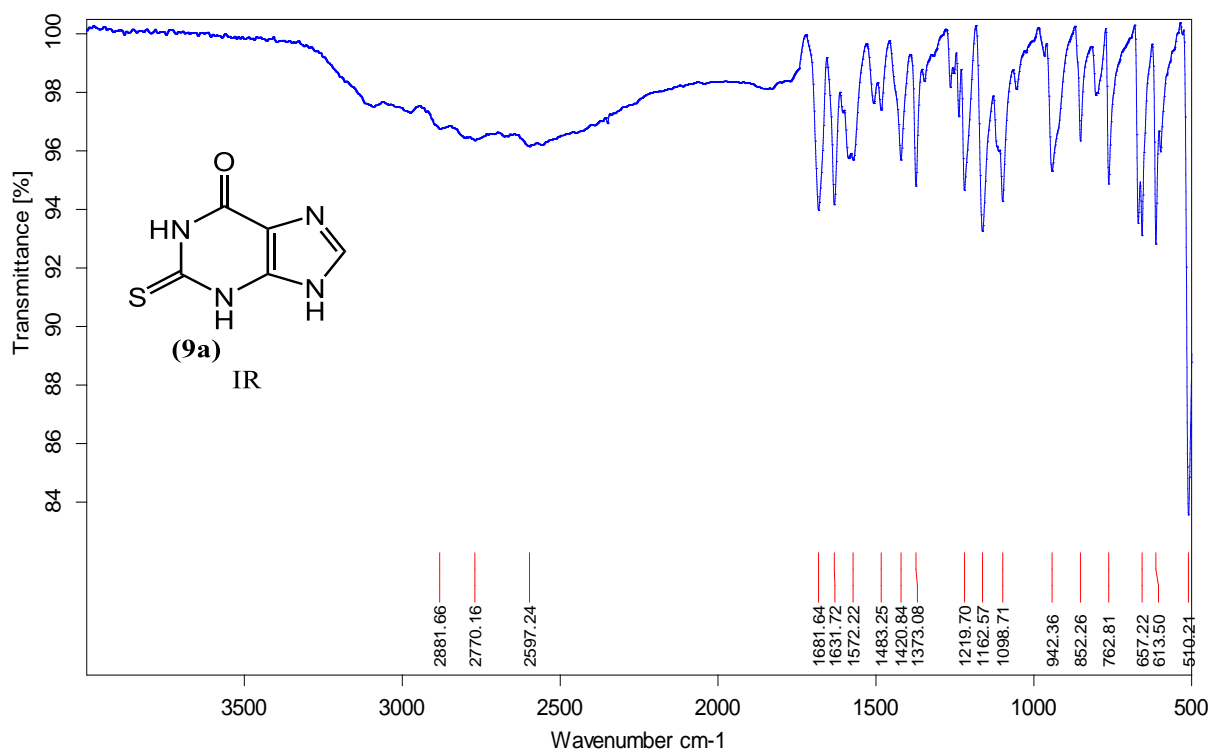
MassPeaks:129

RawMode:Single 5.250(2081) BasePeak:43(6919537)

Фон.реж.:3.350(1321) Group 1 - Event 1







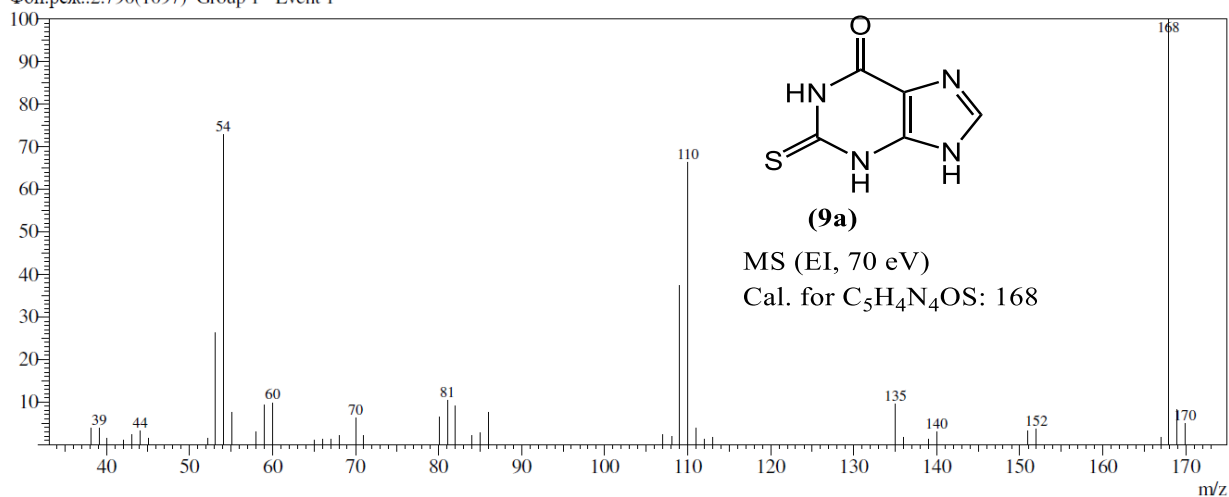
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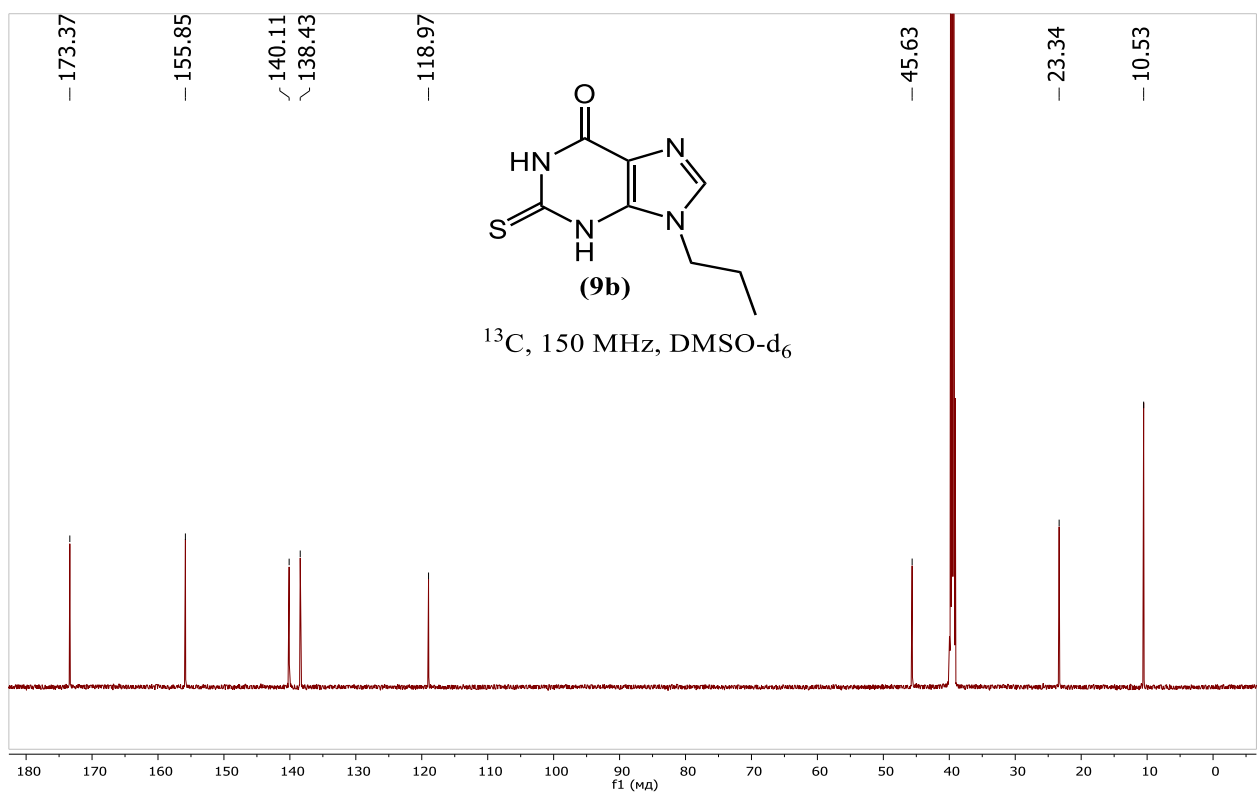
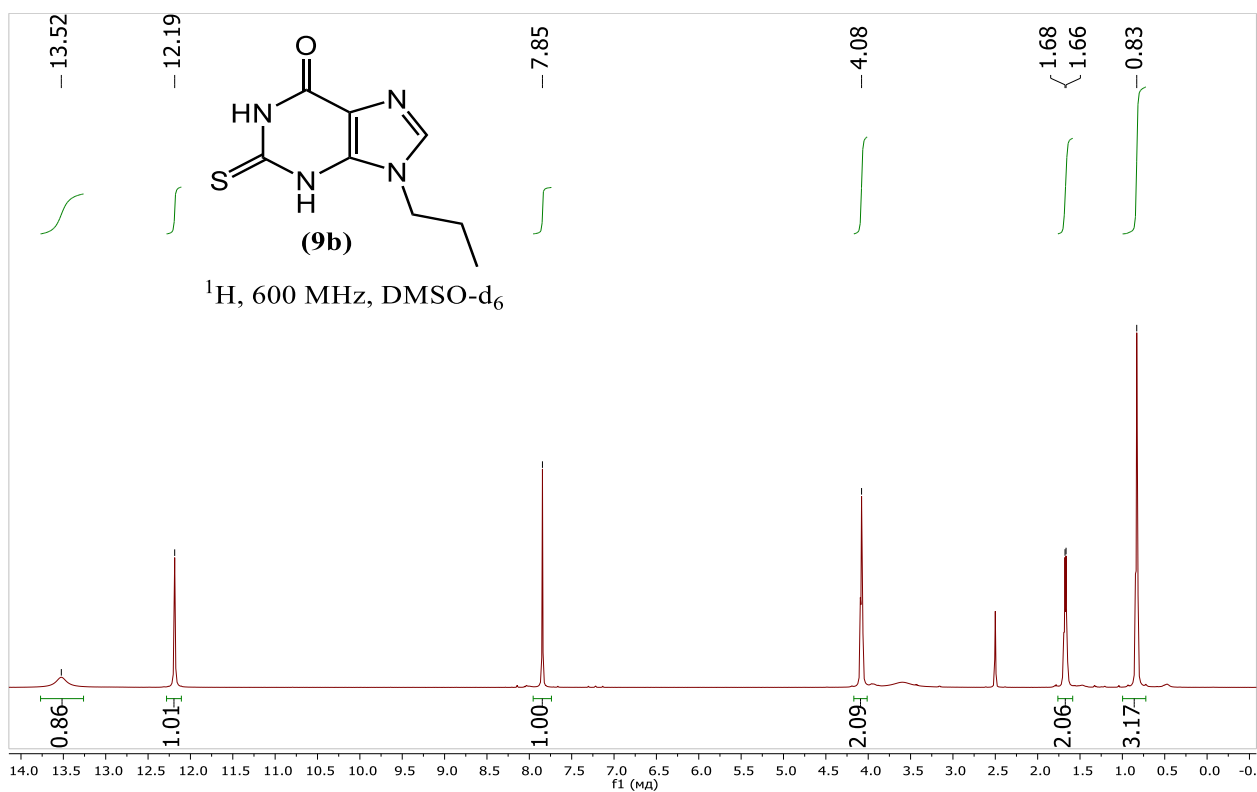
kc-1299

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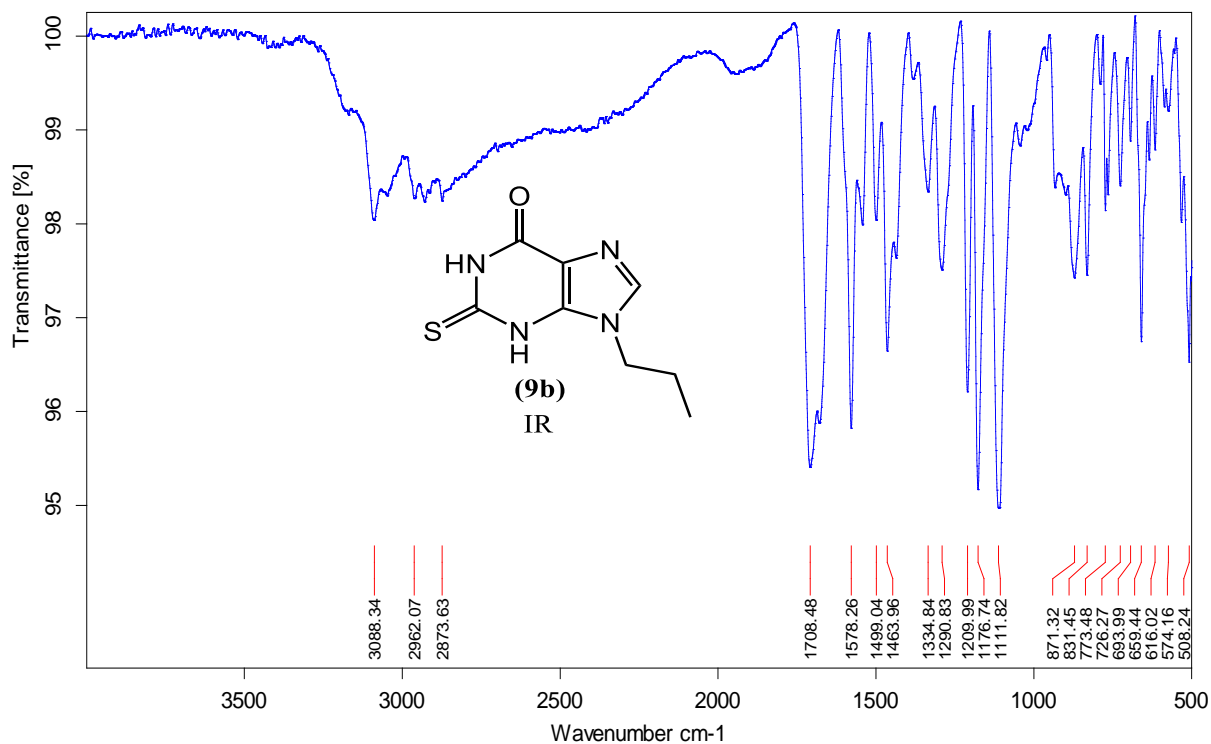
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Line#:1 R.Time:3.898(Scan#:1540)  
 MassPeaks:43  
 RawMode:Single 3.898(1540) BasePeak:168(5271658)  
 Фон.реж.:2.790(1097) Group 1 - Event 1









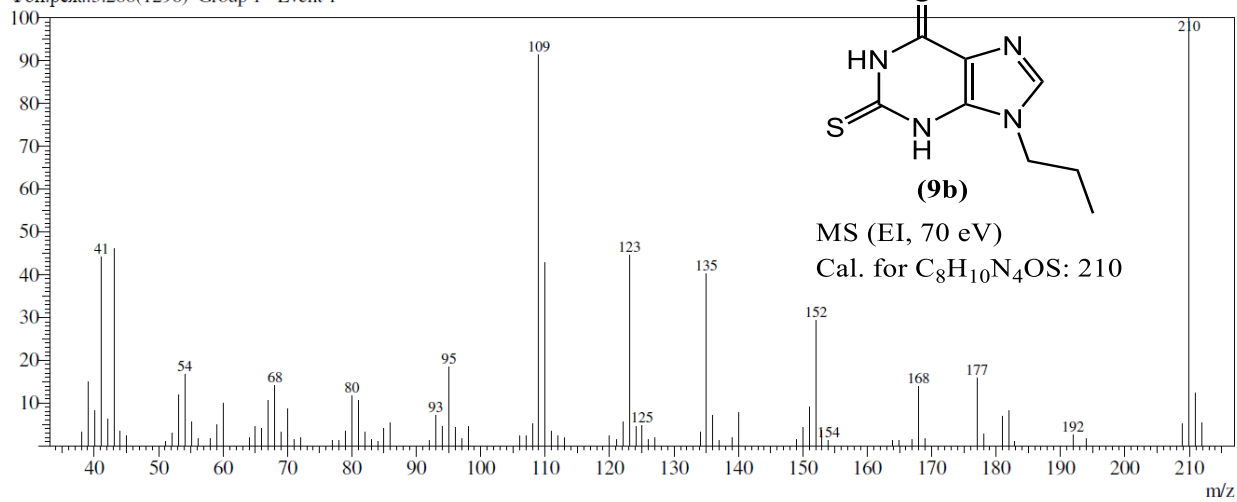
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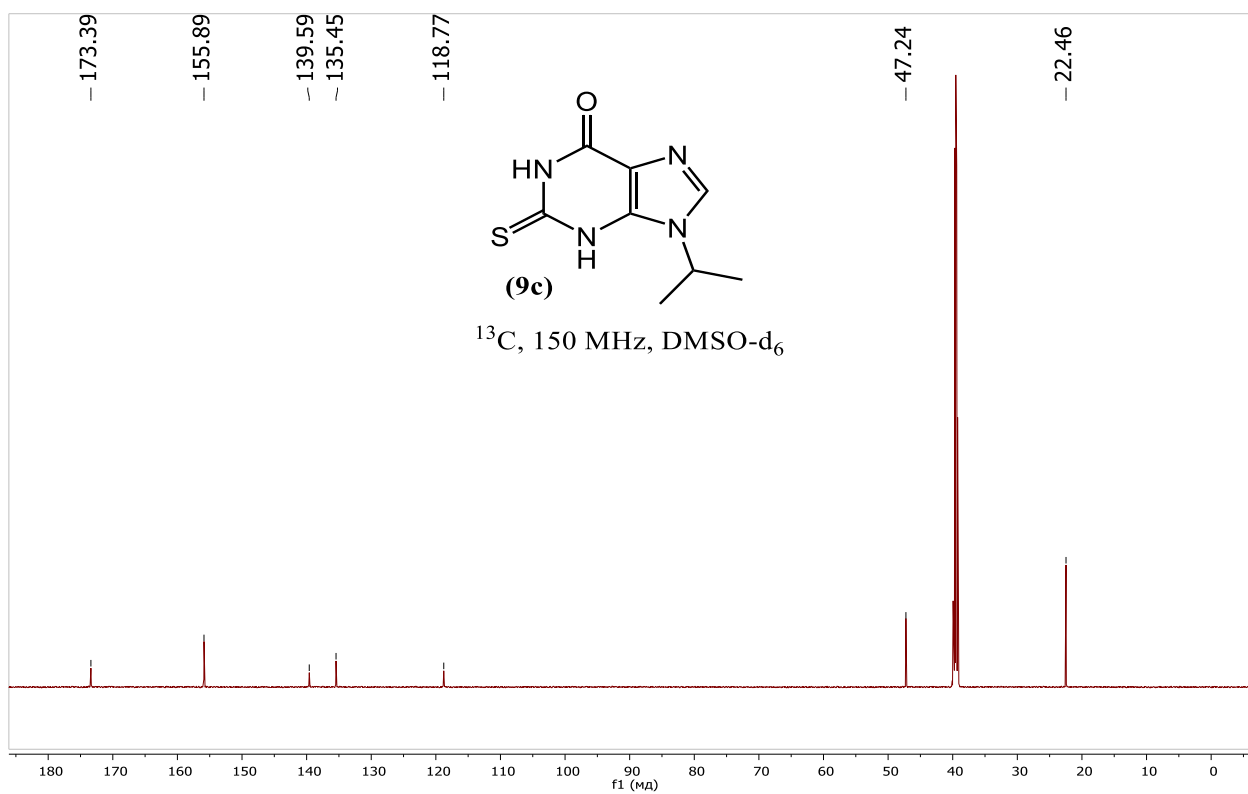
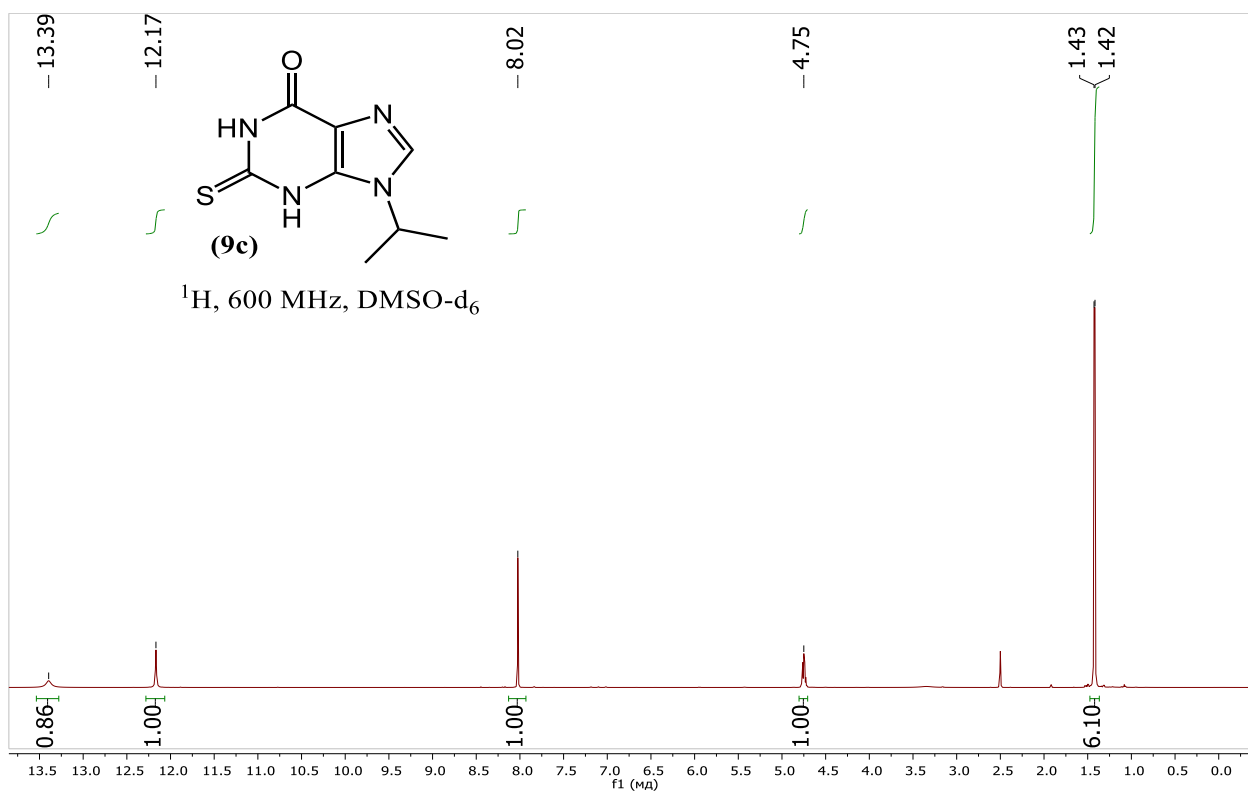
kc-1220

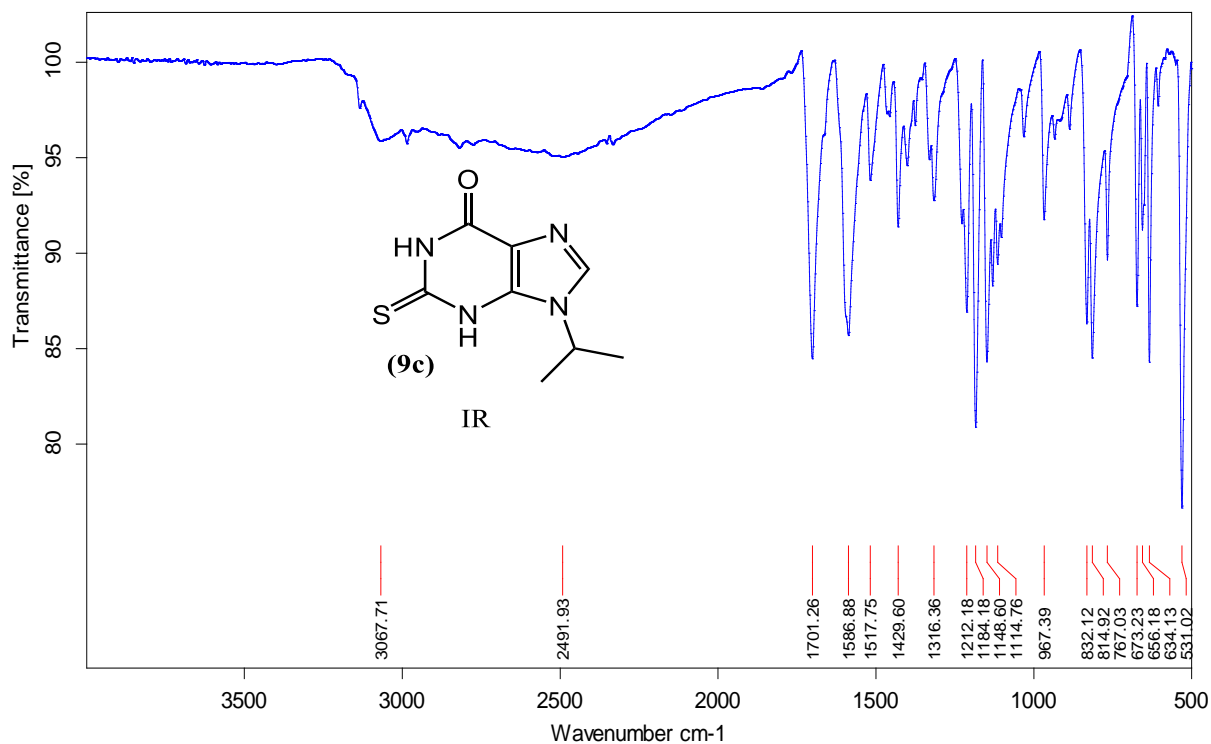
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Line#:1 R.Time:2.818(Scan#:1108)  
 MassPeaks:87  
 RawMode:Single 2.817(1108) BasePeak:210(4047453)  
 Фон.реш.:3.288(1296) Group 1 - Event 1







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kc-1211

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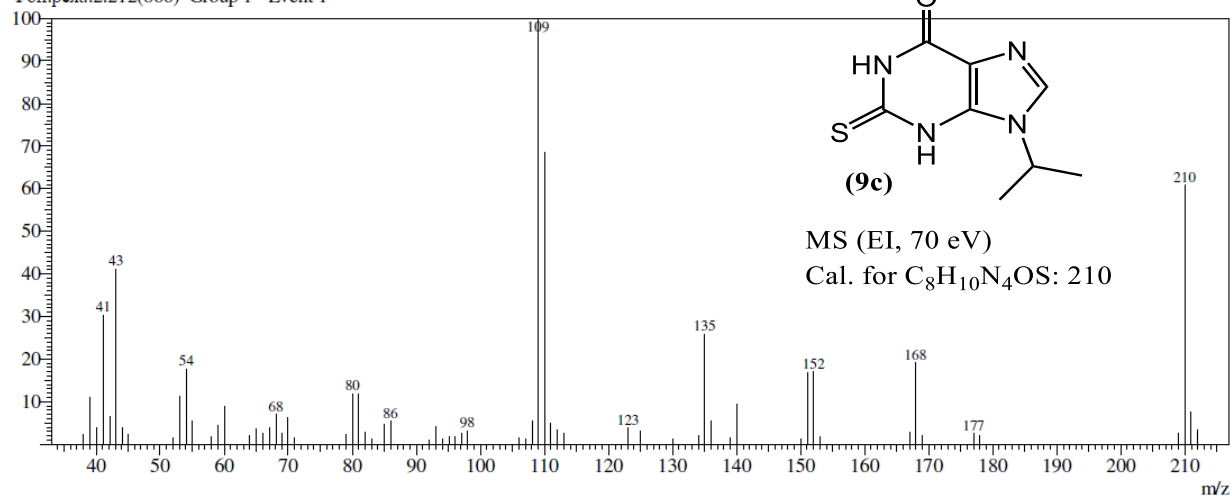
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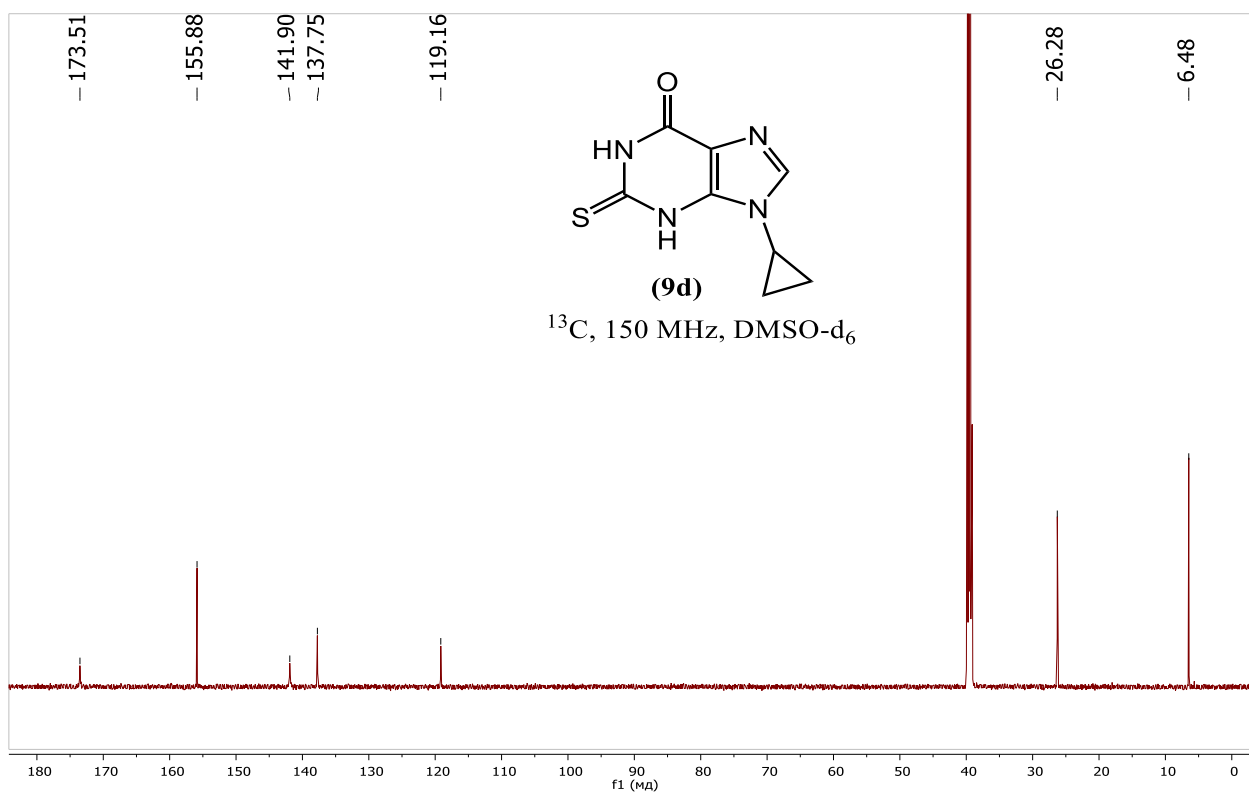
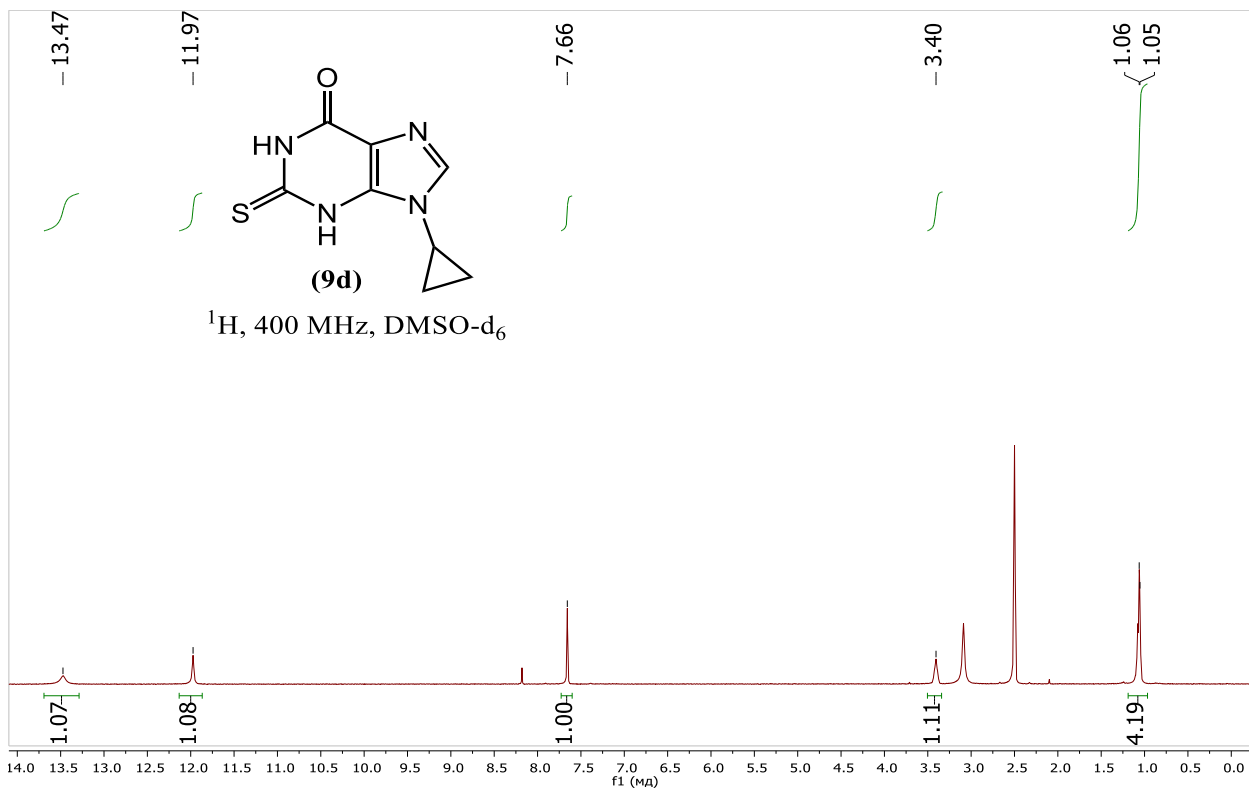
Line#:1 R.Time:4.442(Scan#:1758)

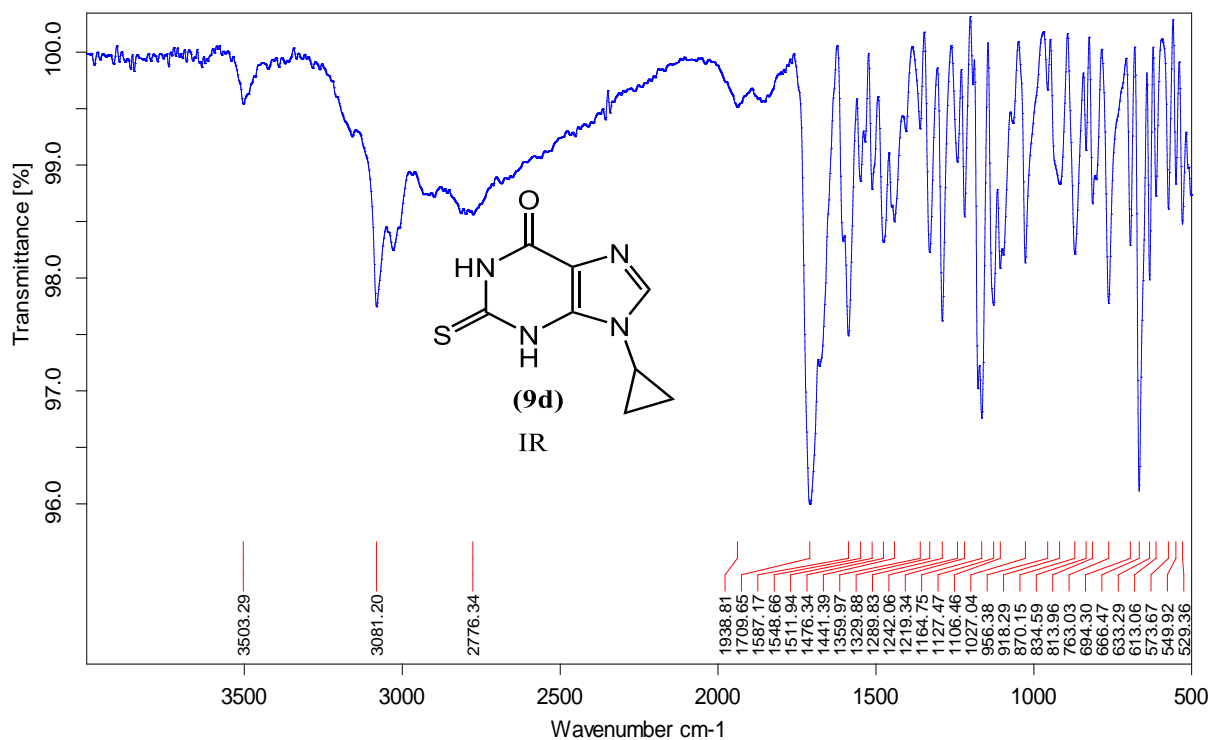
MassPeaks:66

RawMode:Single 4.442(1758) BasePeak:109(5350653)

Фон.реж.:2.212(866) Group 1 - Event 1







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kc-1202

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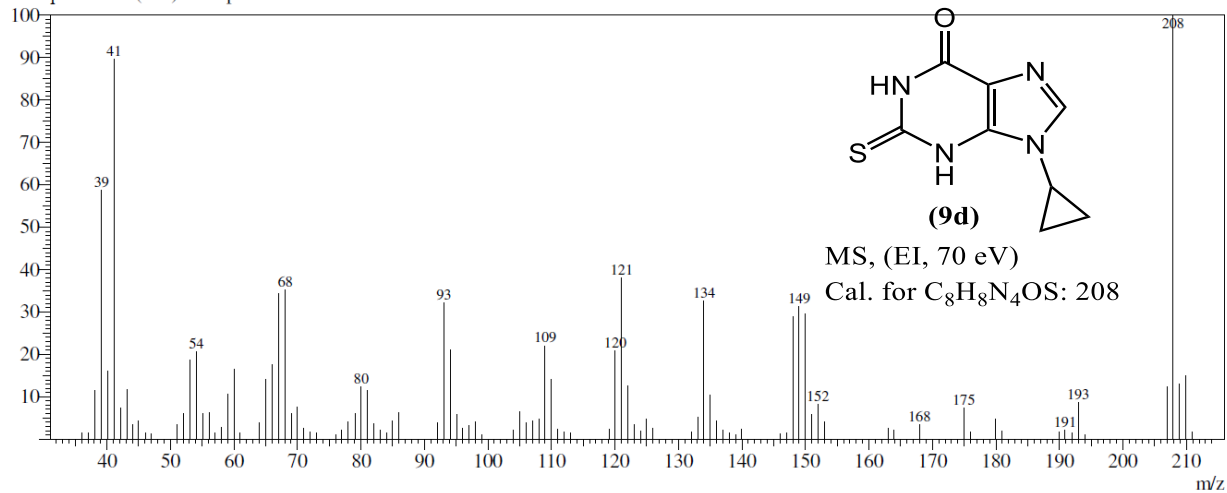
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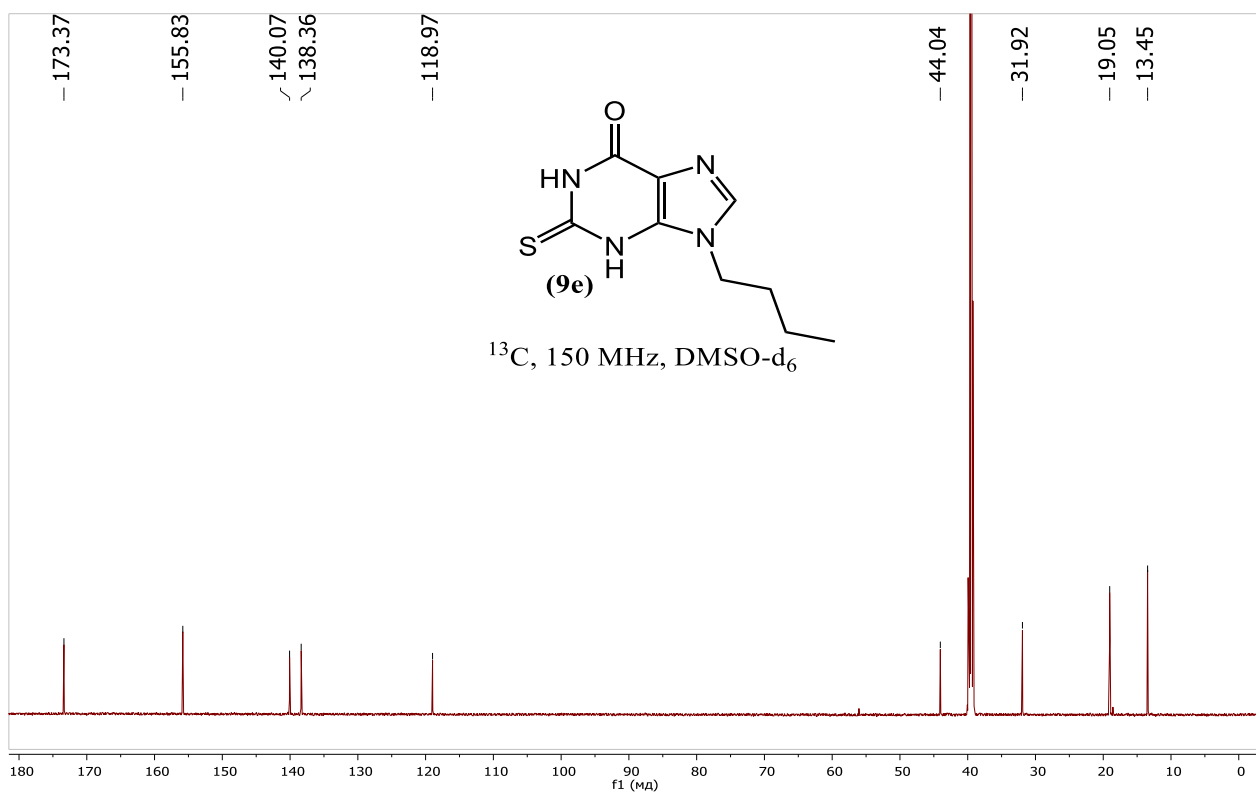
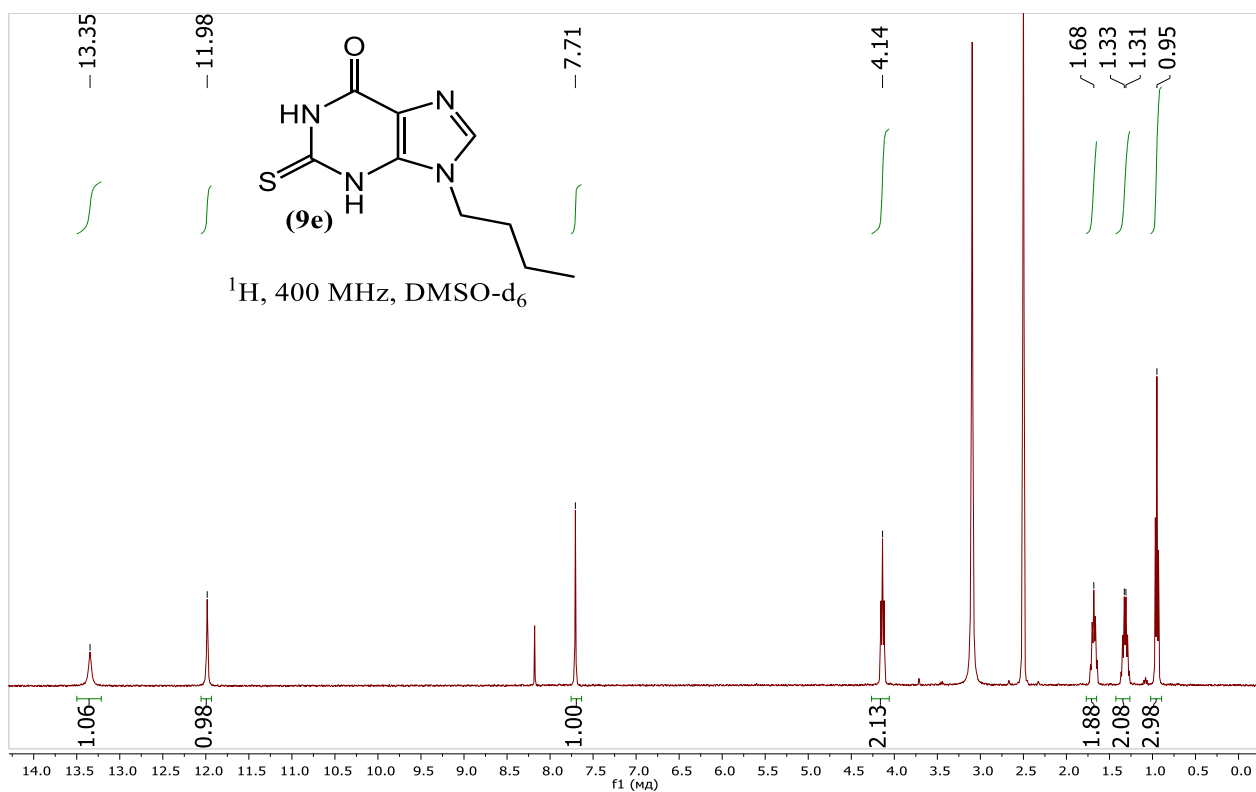
Line#:1 R.Time:3.587(Scan#:1416)

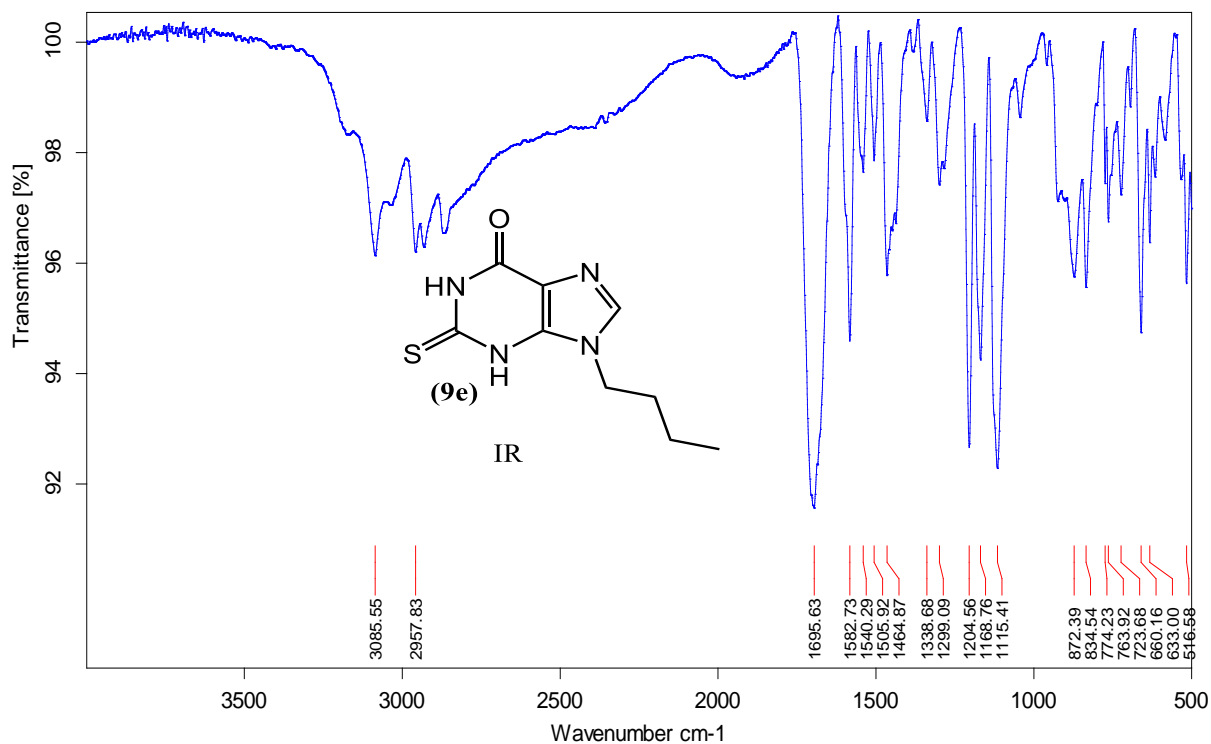
MassPeaks:104

RawMode:Single 3.587(1416) BasePeak:208(2466164)

Фон.рж.:1.855(723) Group 1 - Event 1







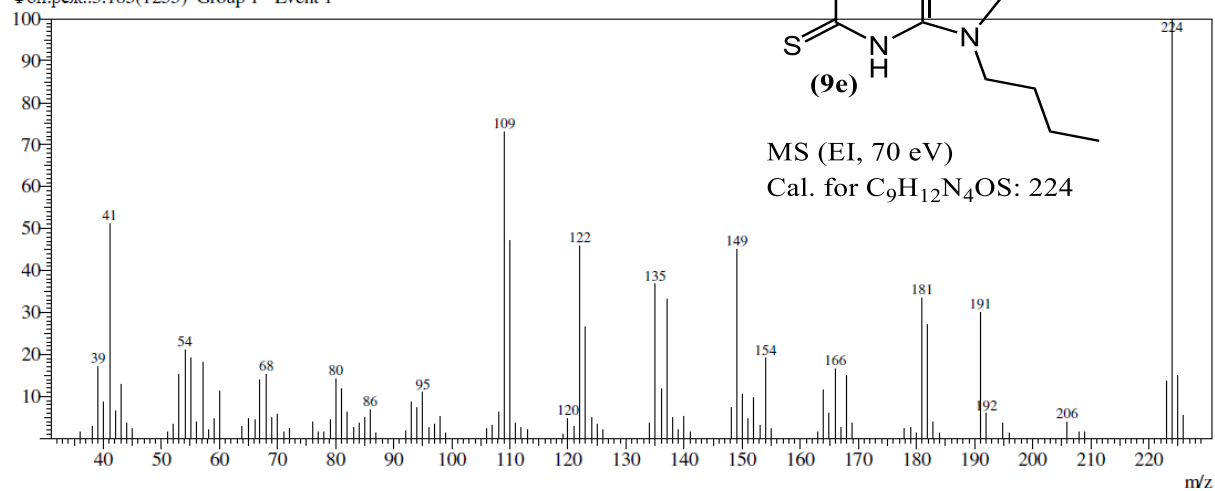
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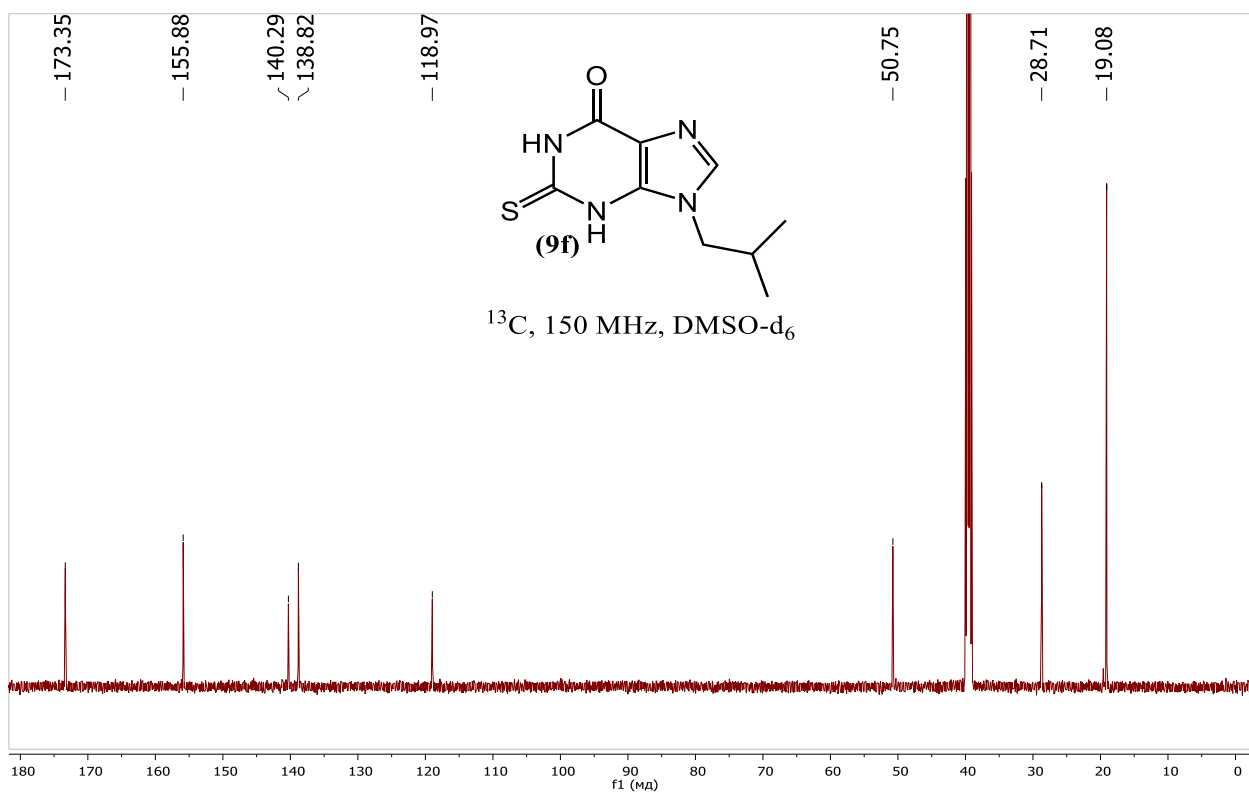
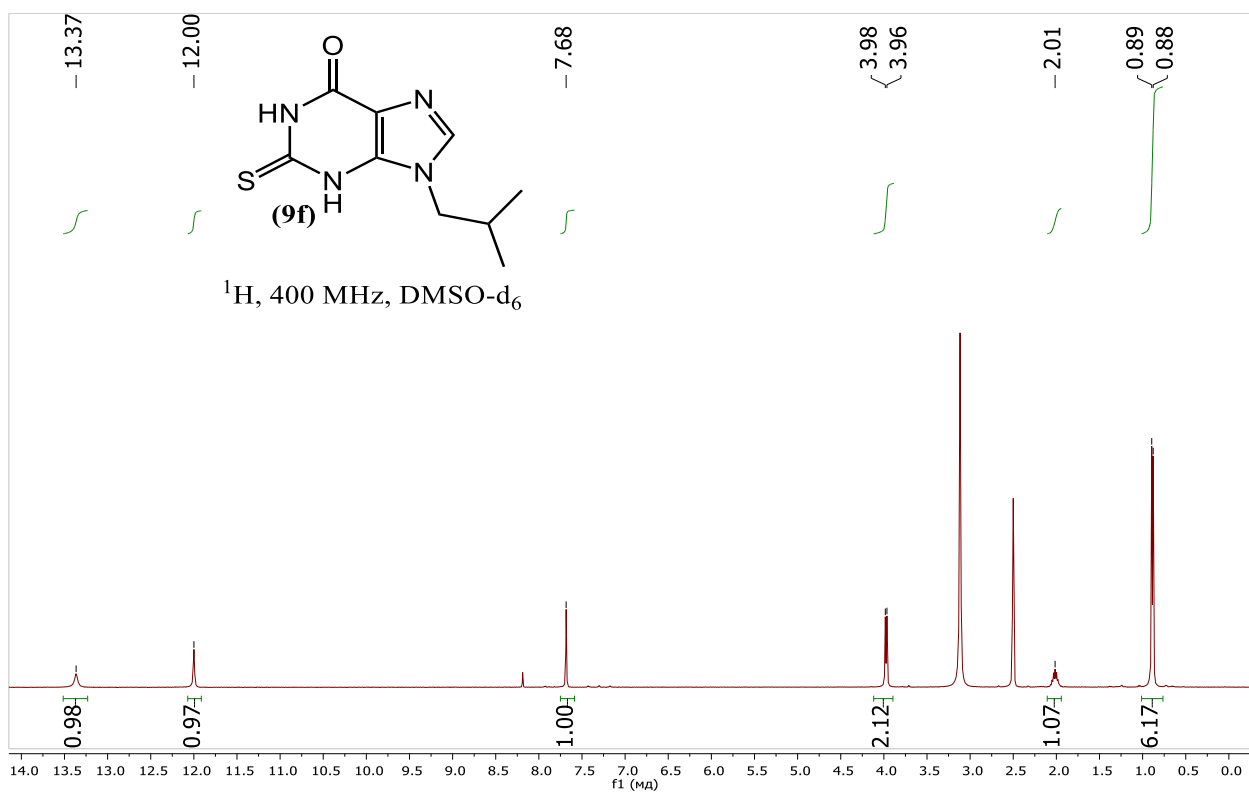
kc-1210

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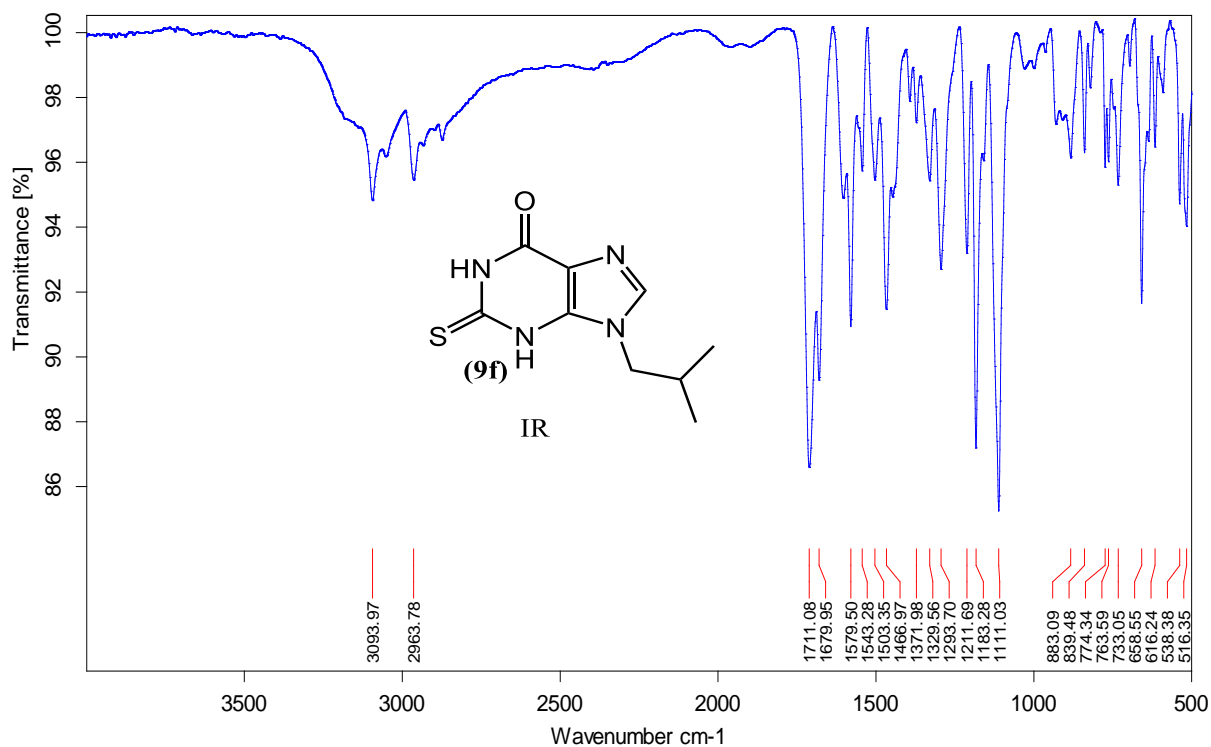
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Line#:1 R.Time:2.810(Scan#:1105)  
 MassPeaks:105  
 RawMode:Single 2.810(1105) BasePeak:224(2920821)  
 Фон.реж.:3.185(1255) Group 1 - Event 1









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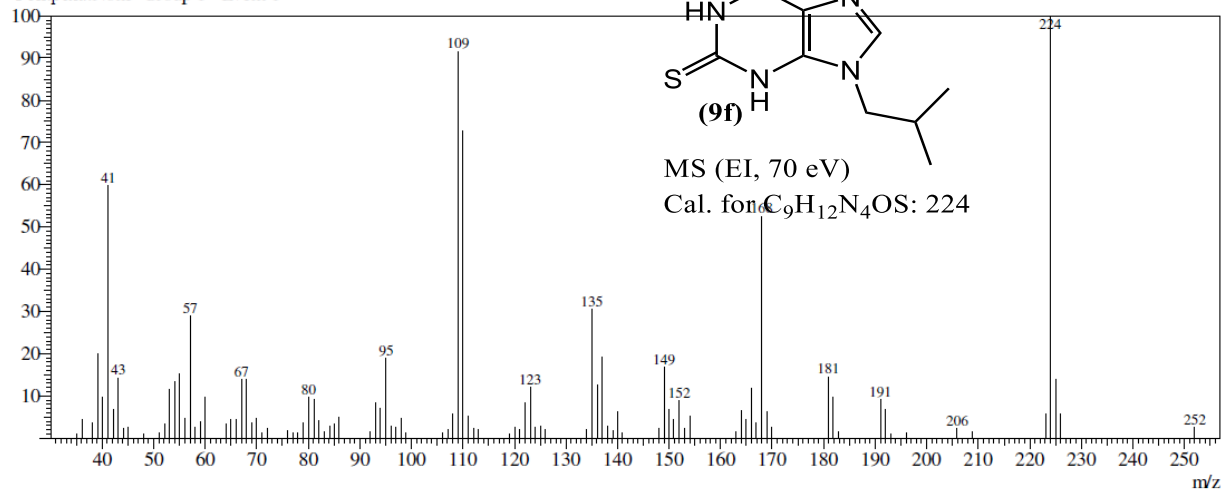
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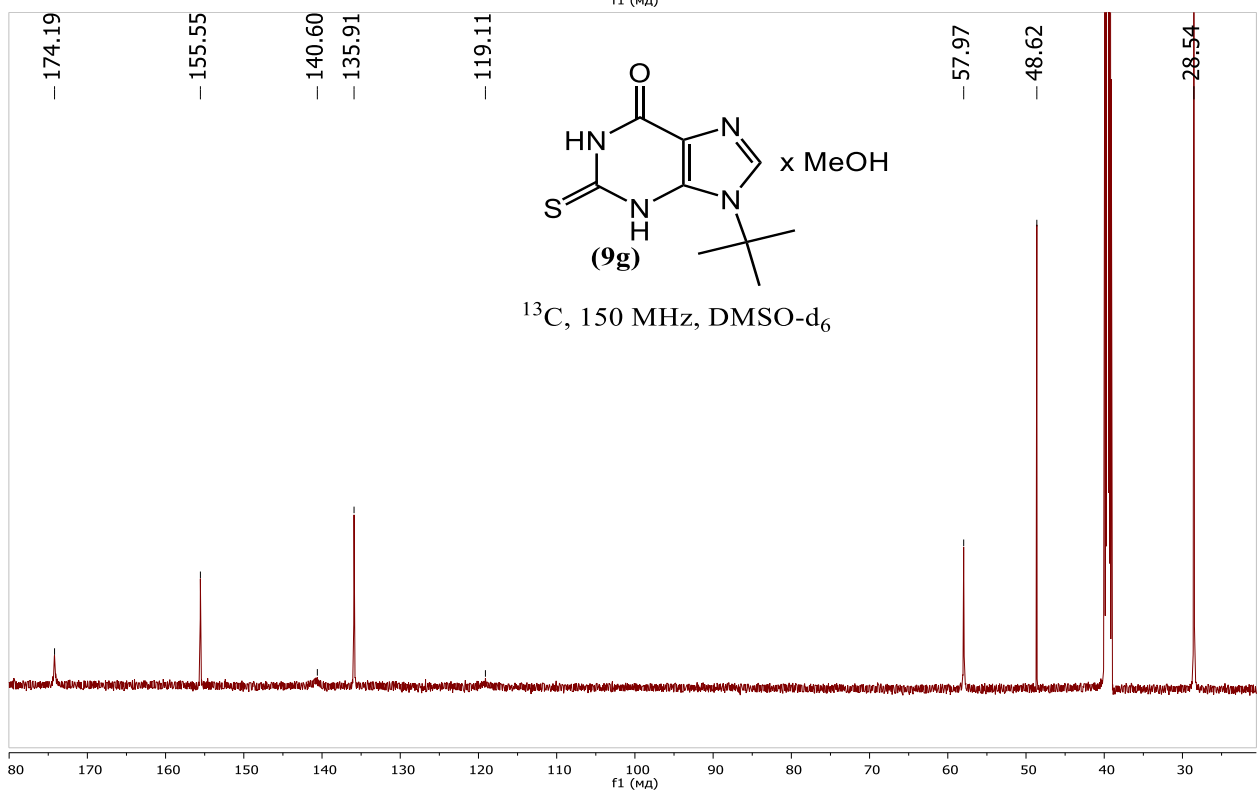
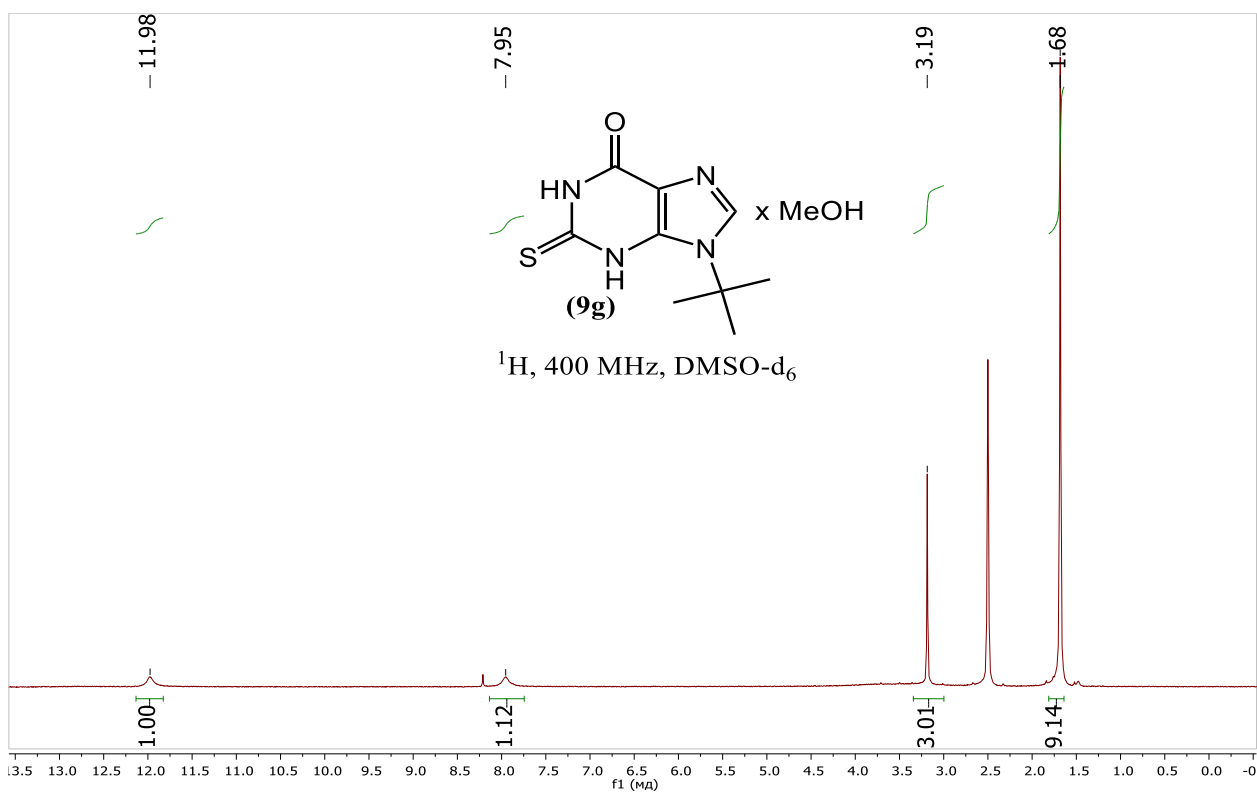
Line#:1 R.Time:3.097(Scan#:1220)

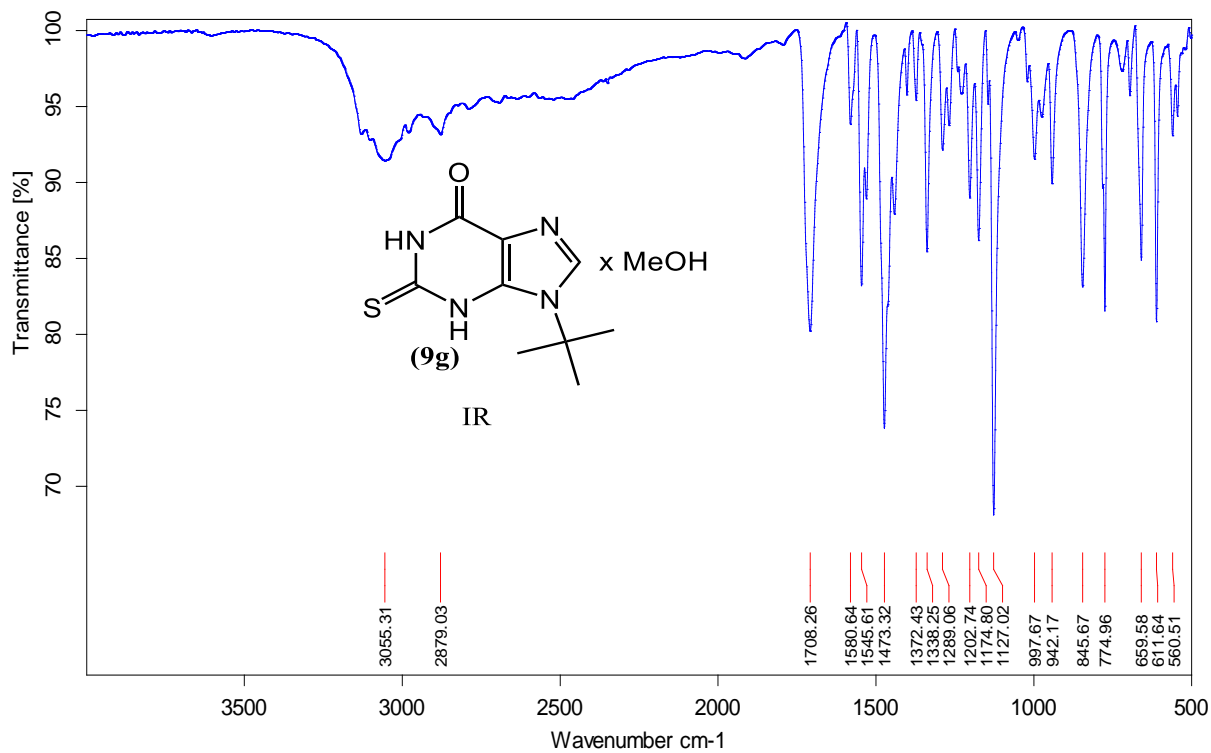
MassPeaks:102

RawMode:Single 3.098(1220) BasePeak:224(4532536)

Фон.реж.:None Group 1 - Event 1







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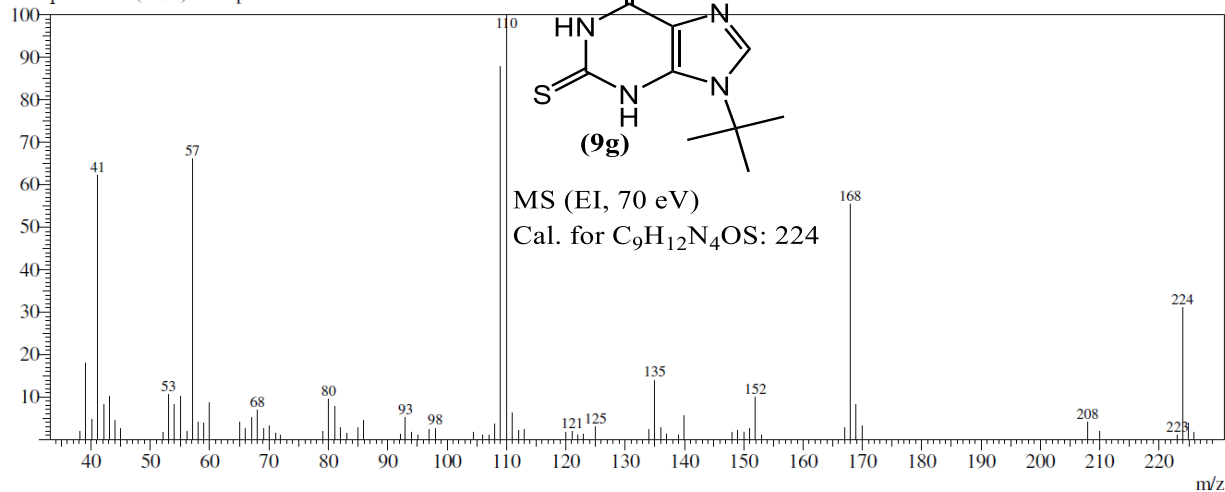
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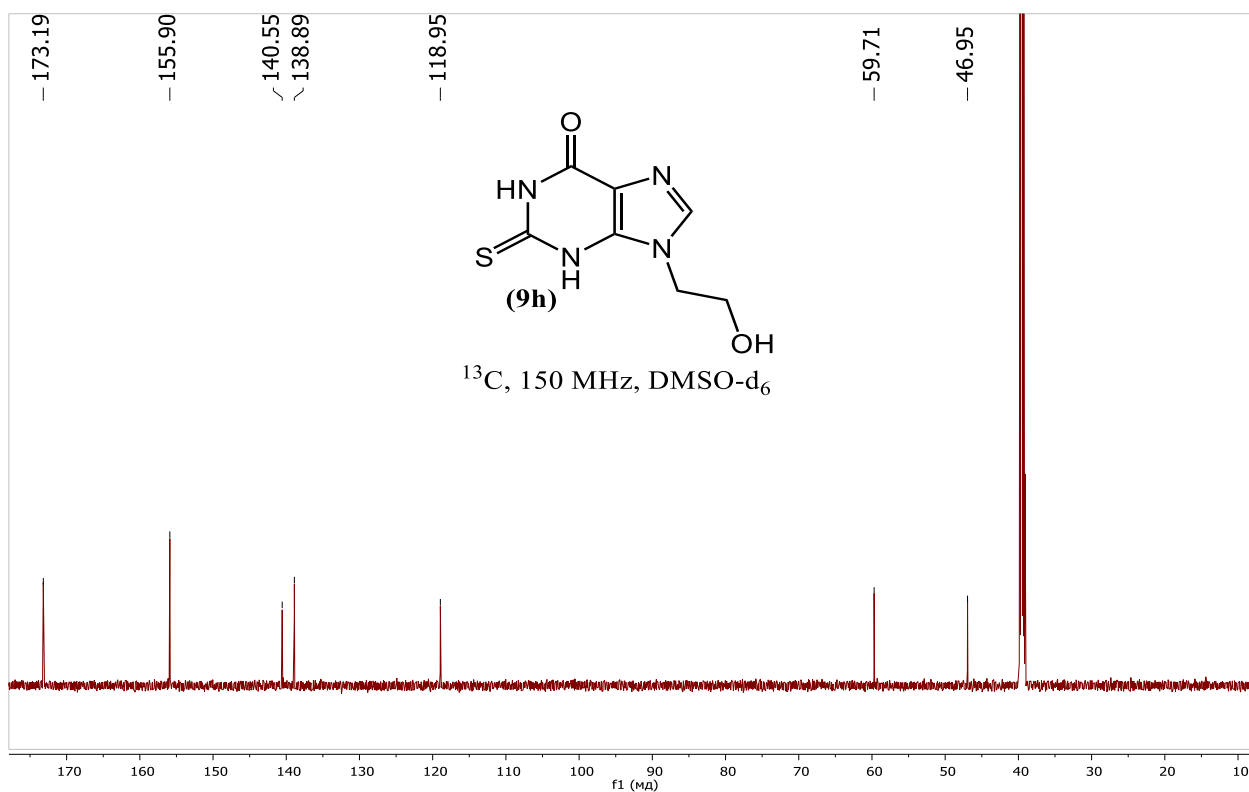
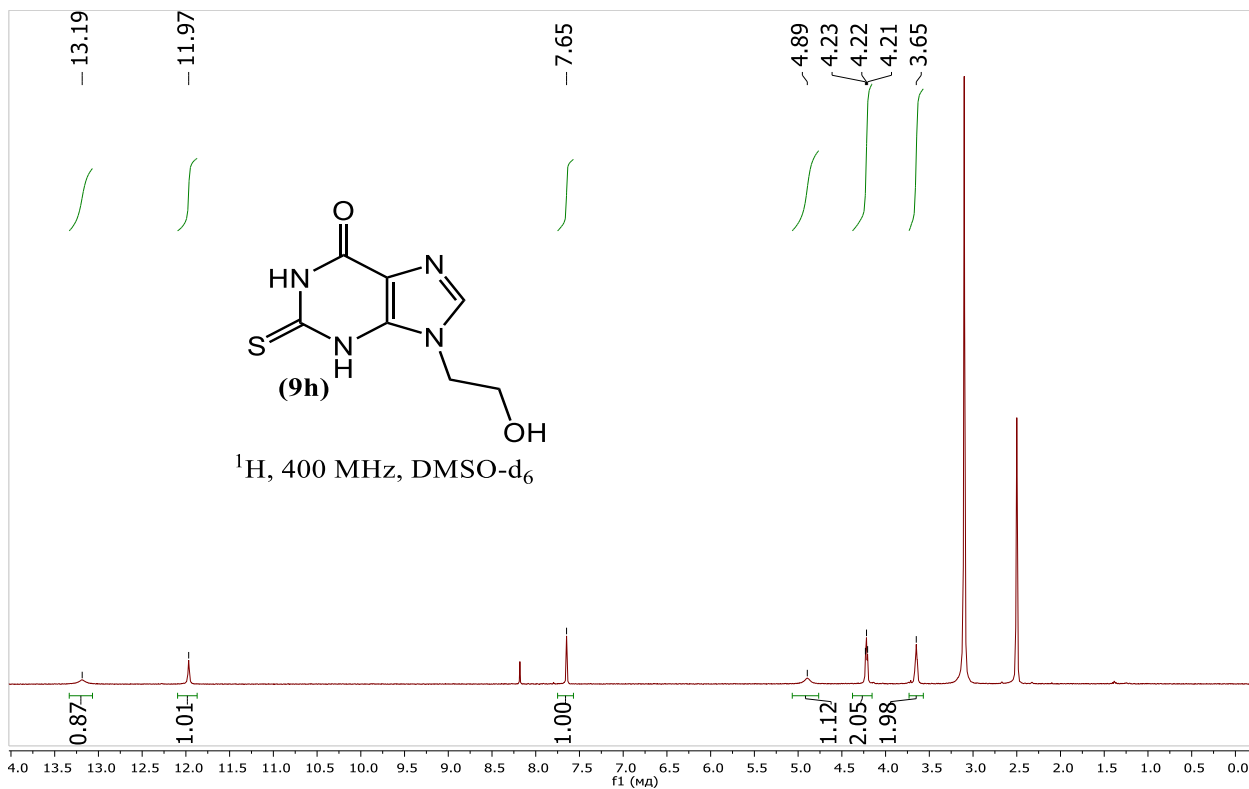
Line#:1 R.Time:2.643(Scan#:1038)

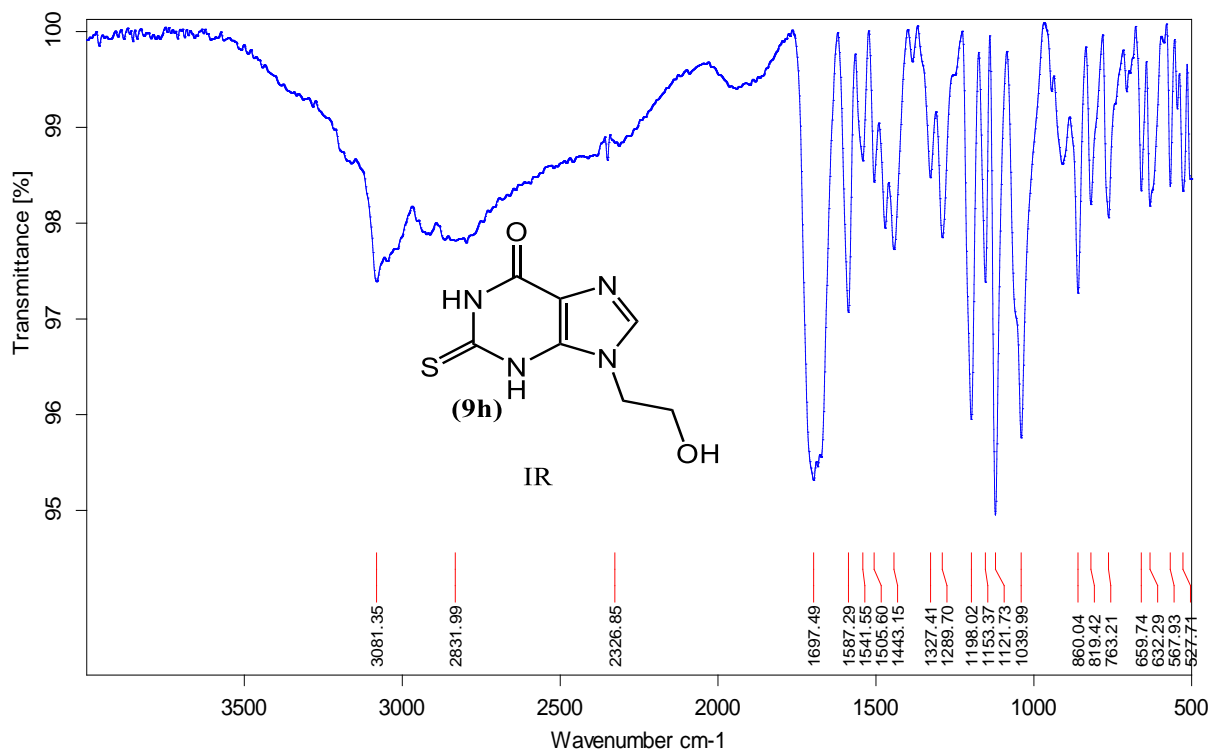
MassPeaks:74

RawMode:Single 2.643(1038) BasePeak:110(646191)

Фон.реж.:4.282(1694) Group 1 - Event 1



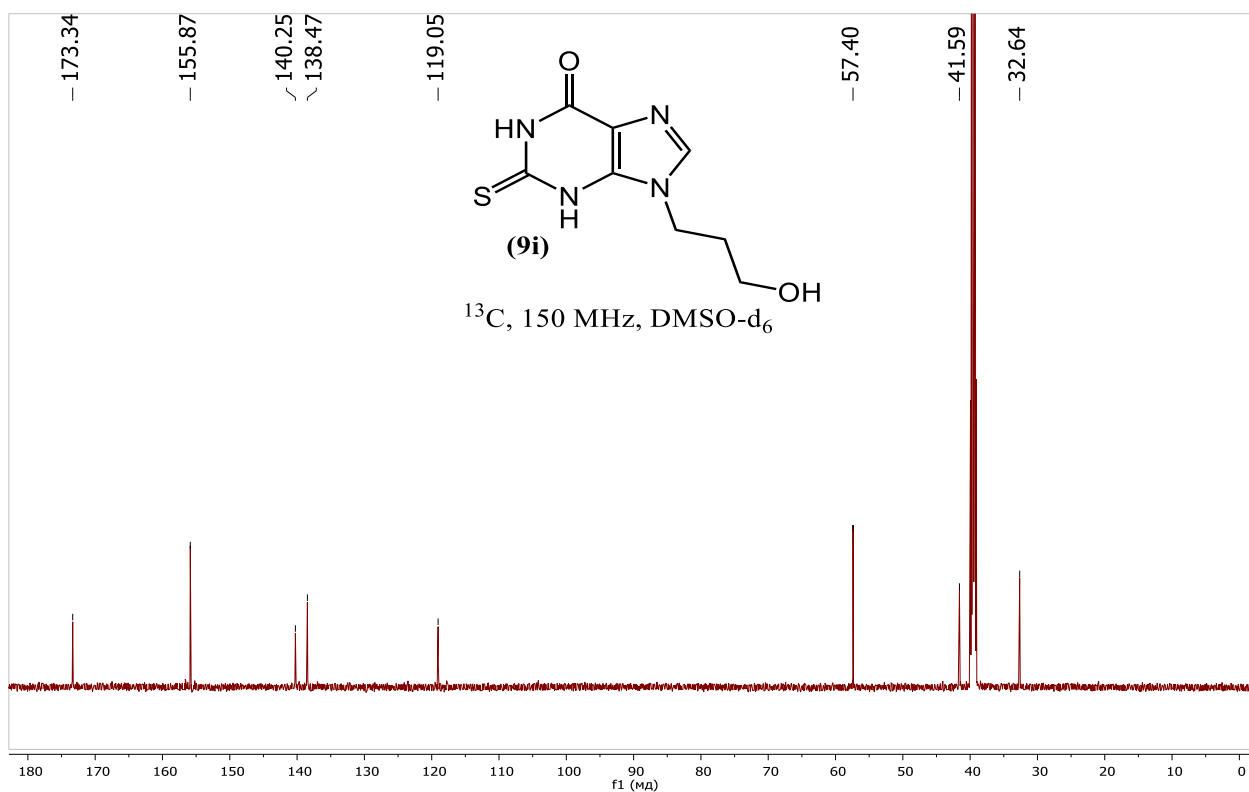
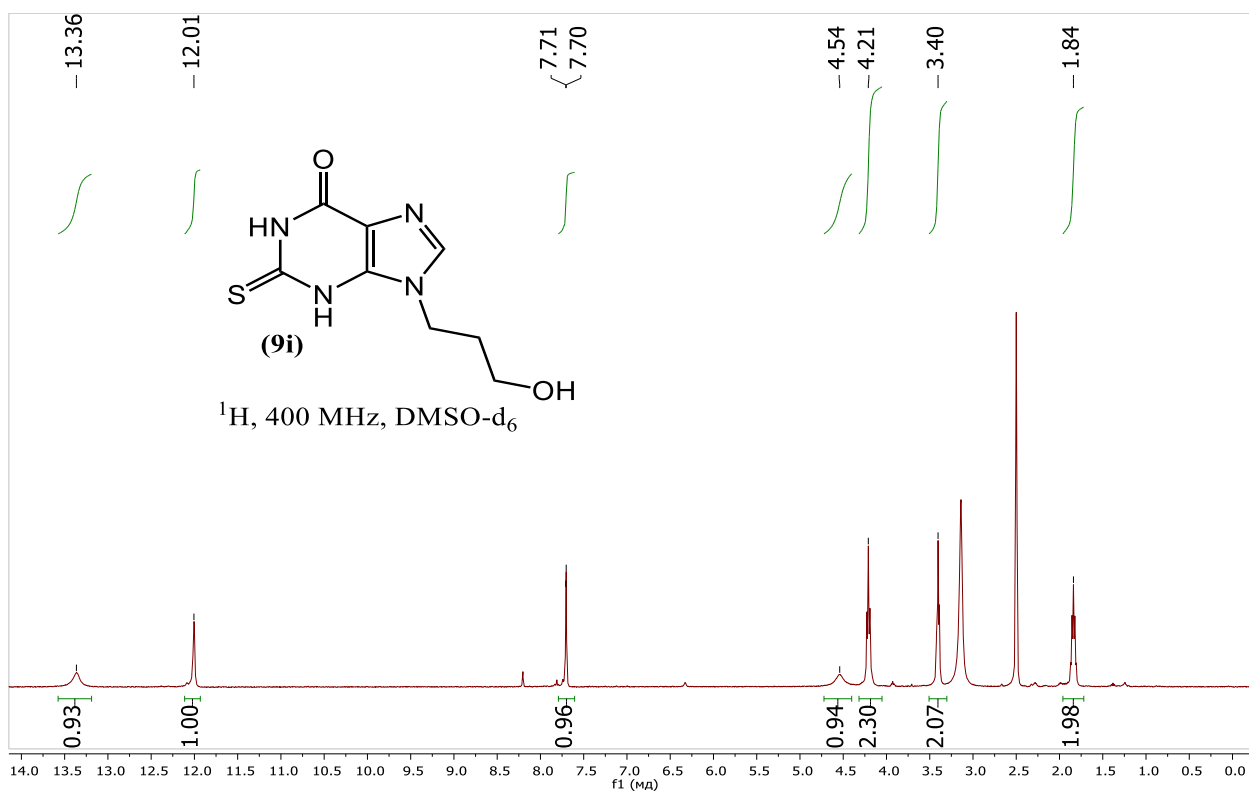


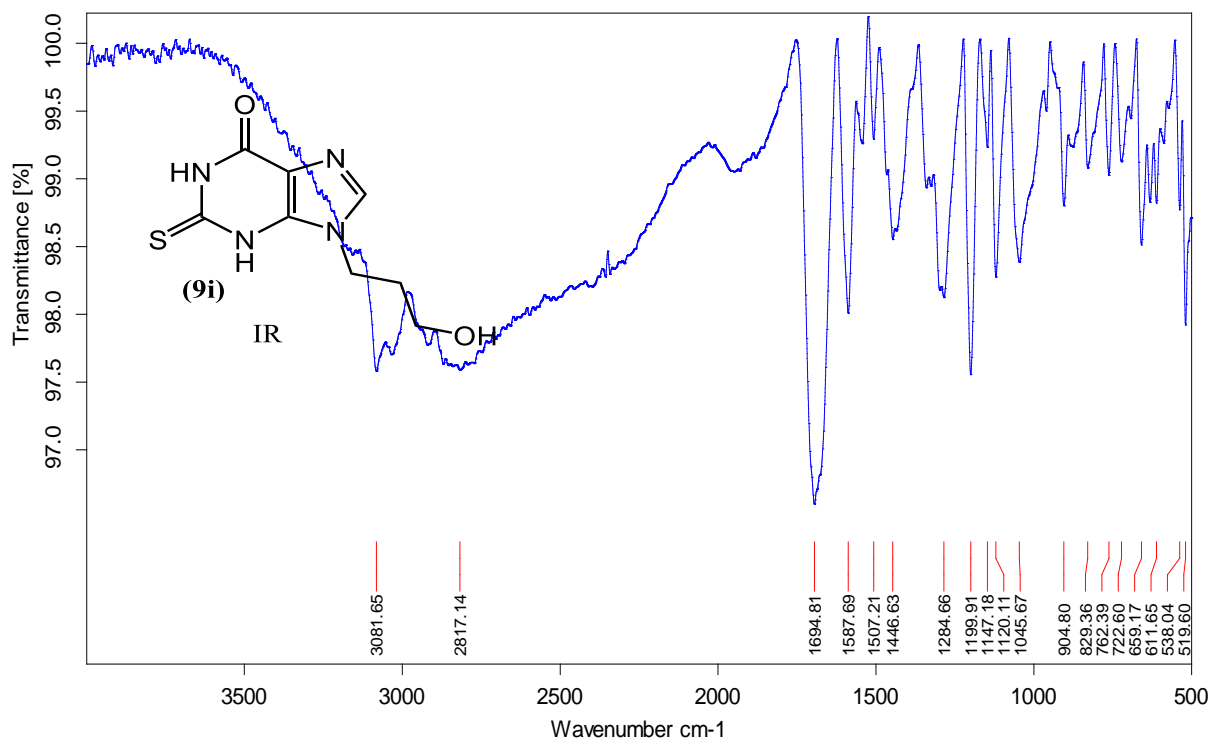


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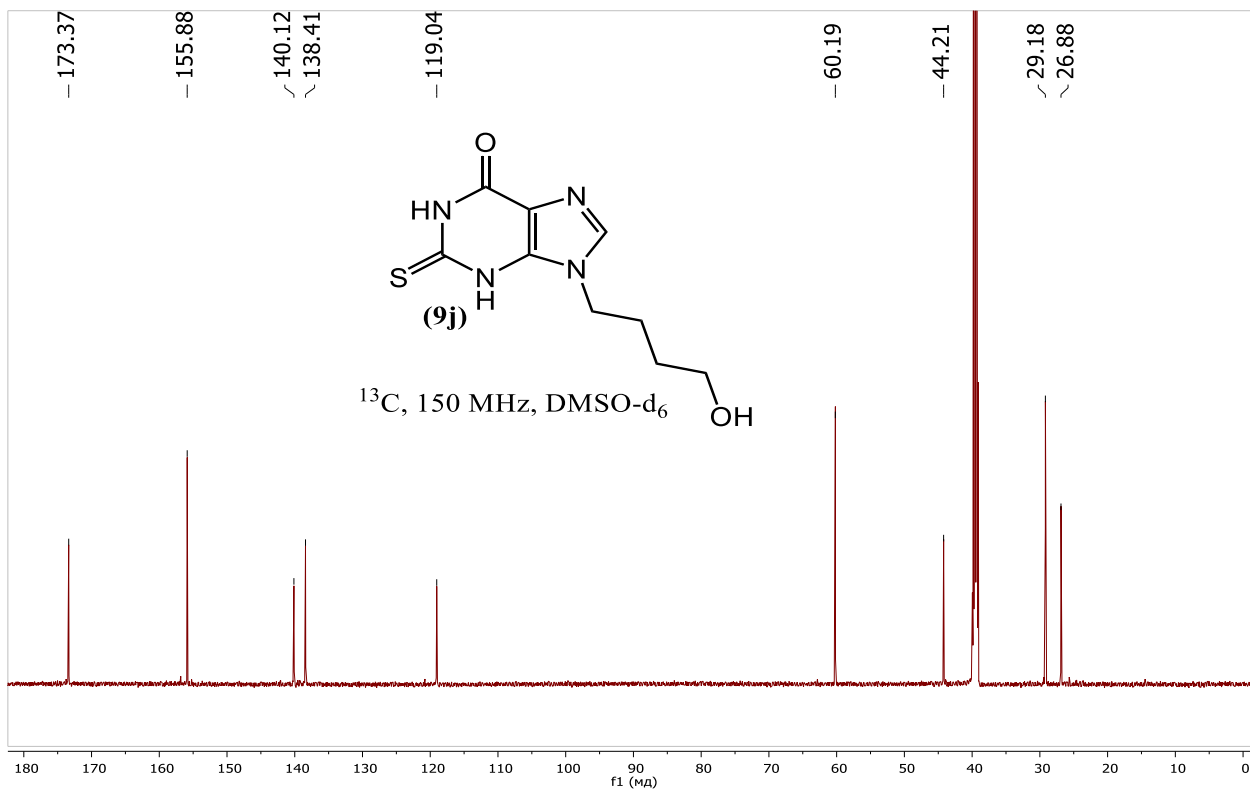
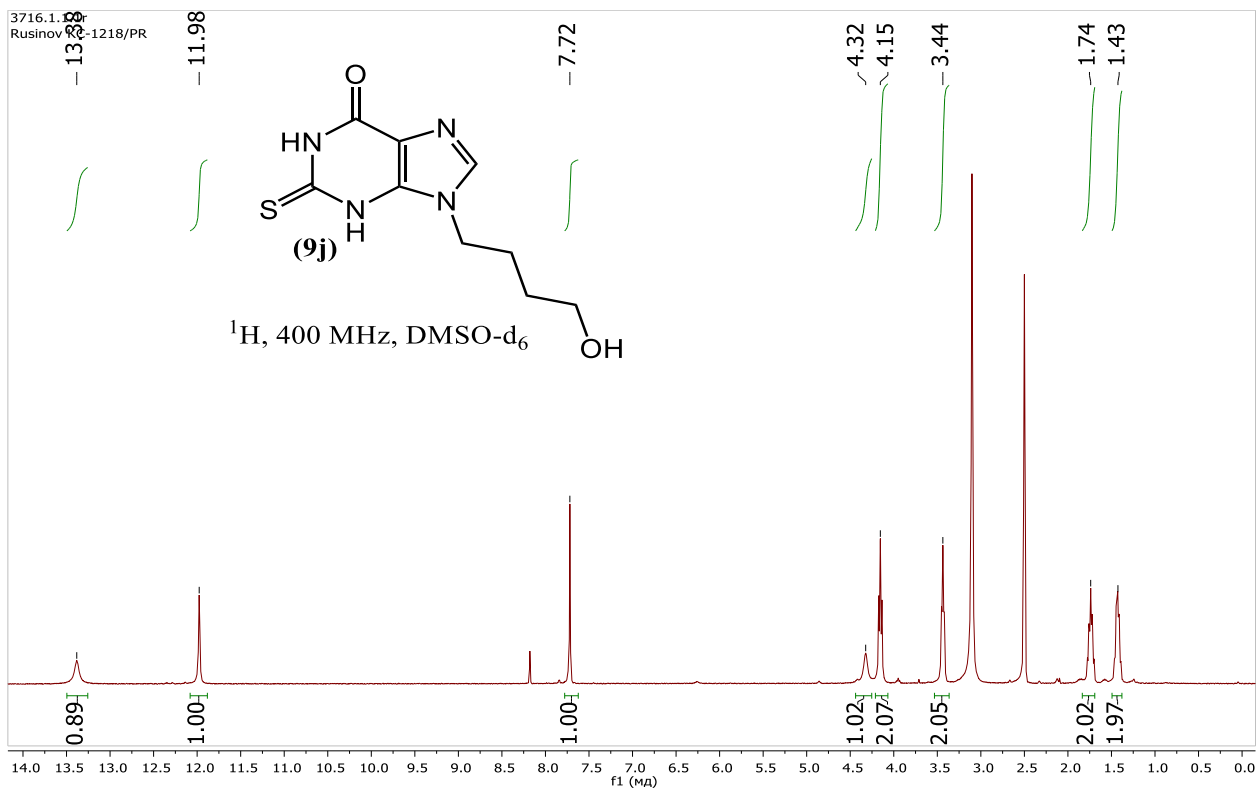


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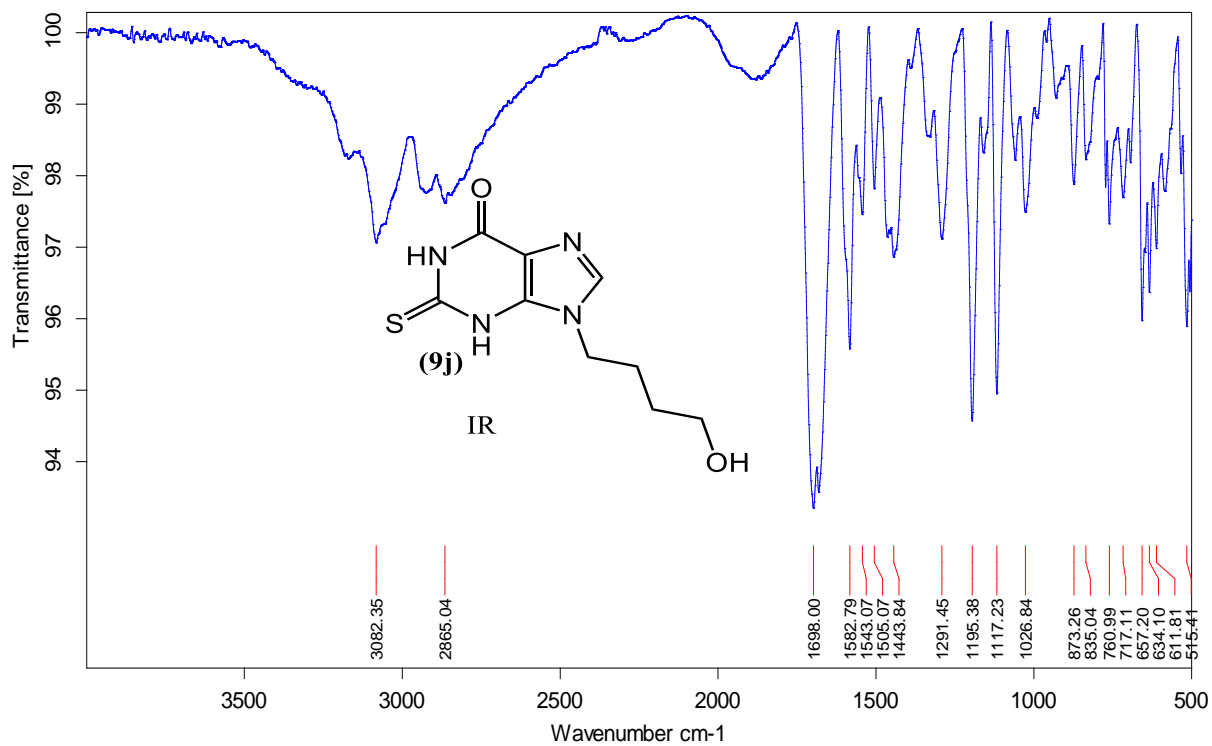
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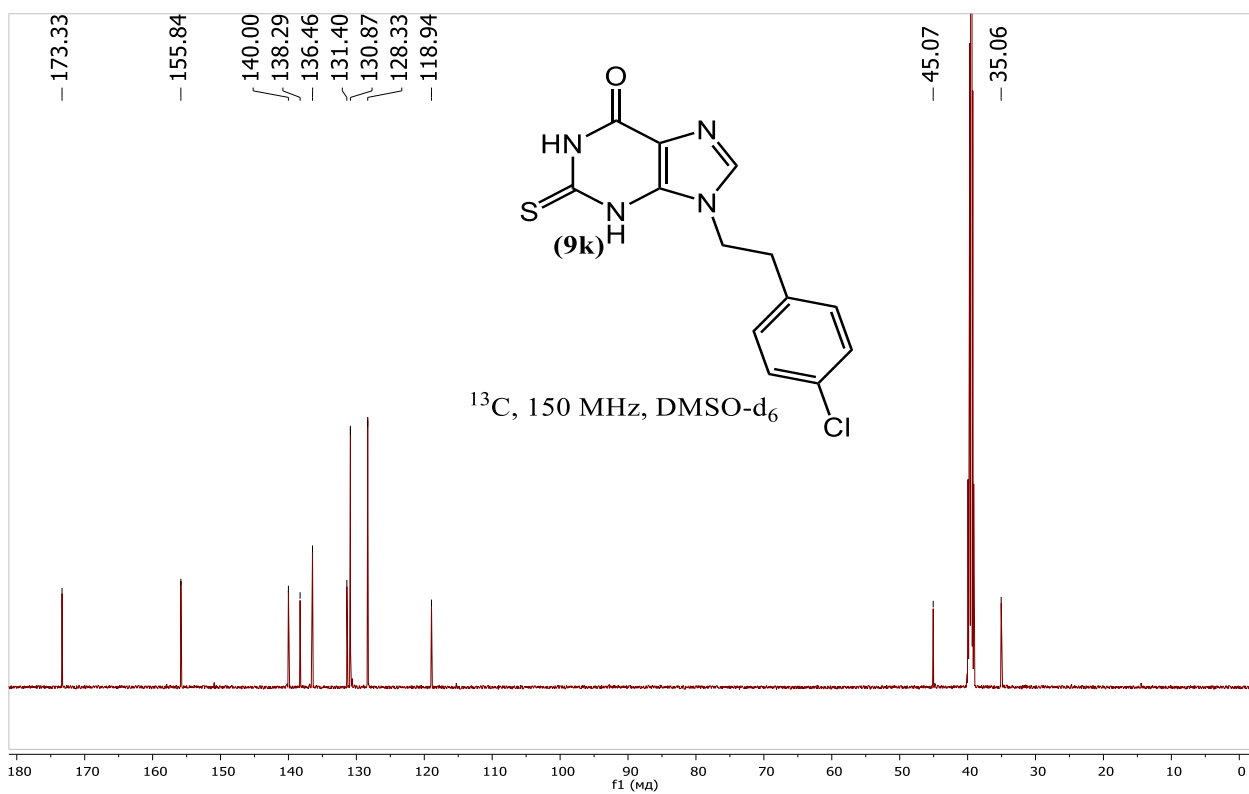
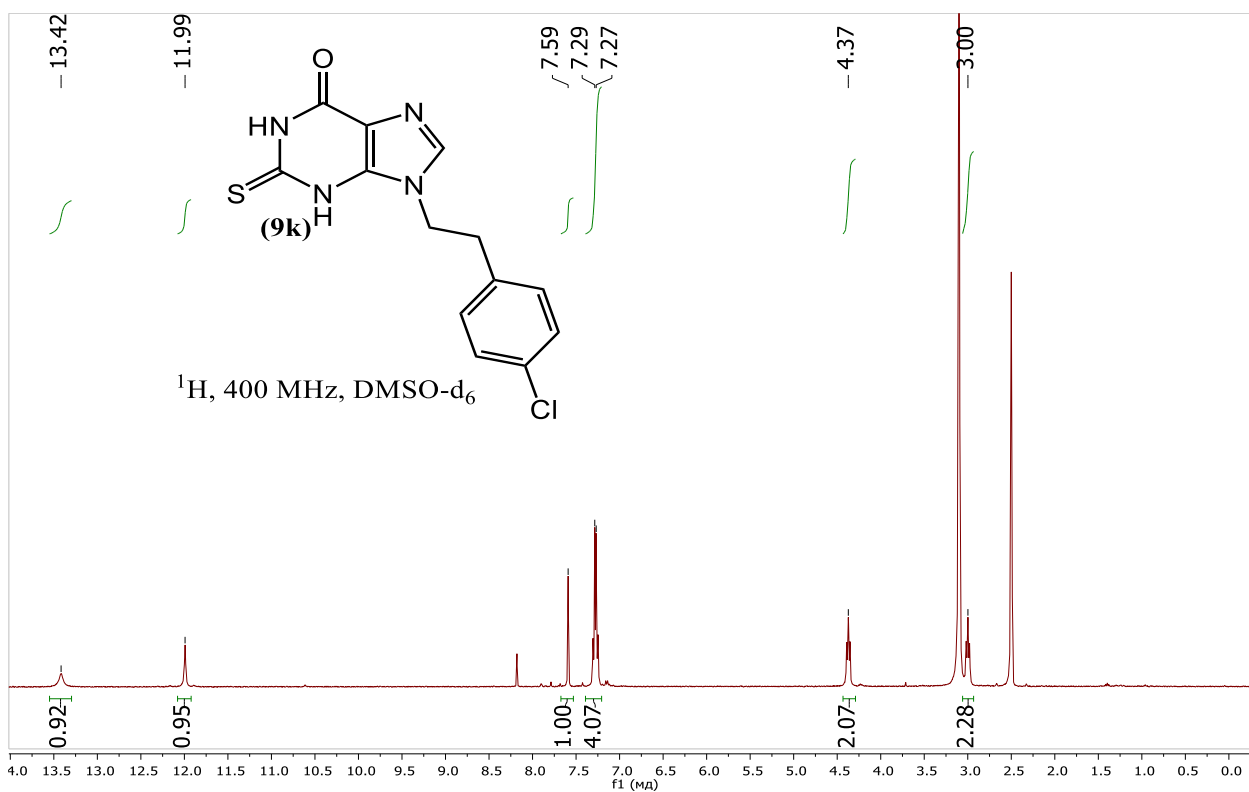


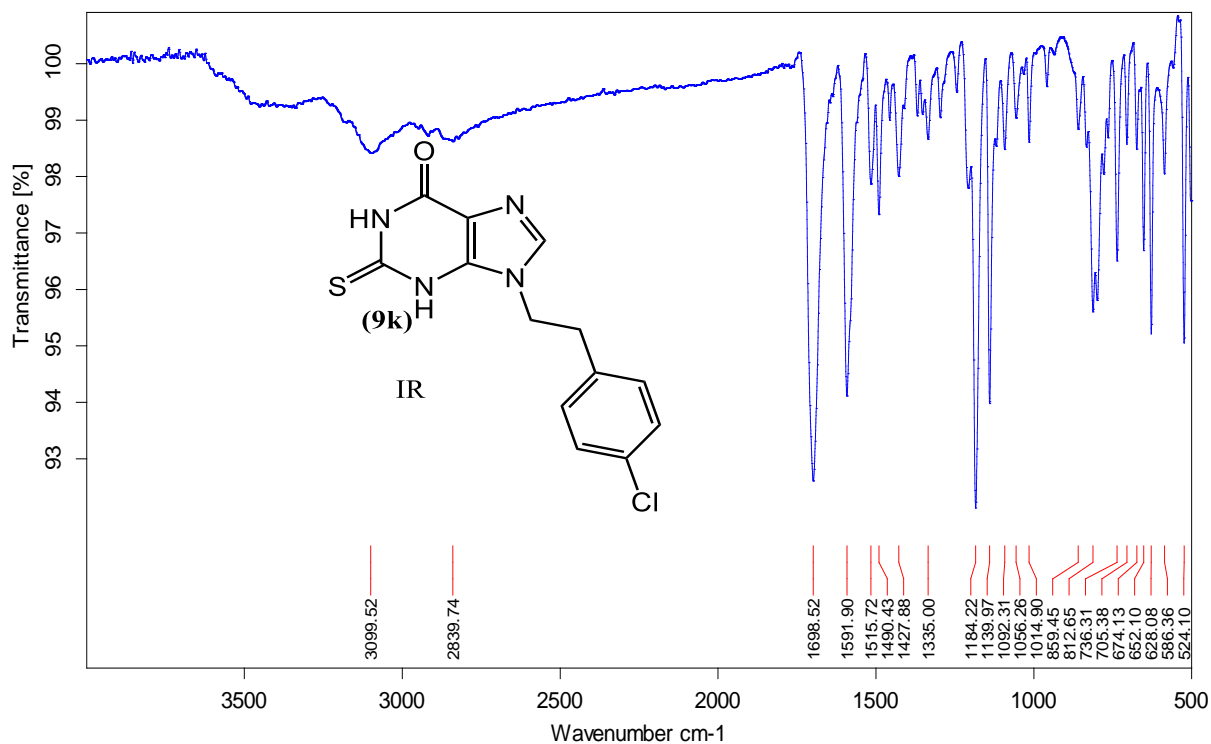


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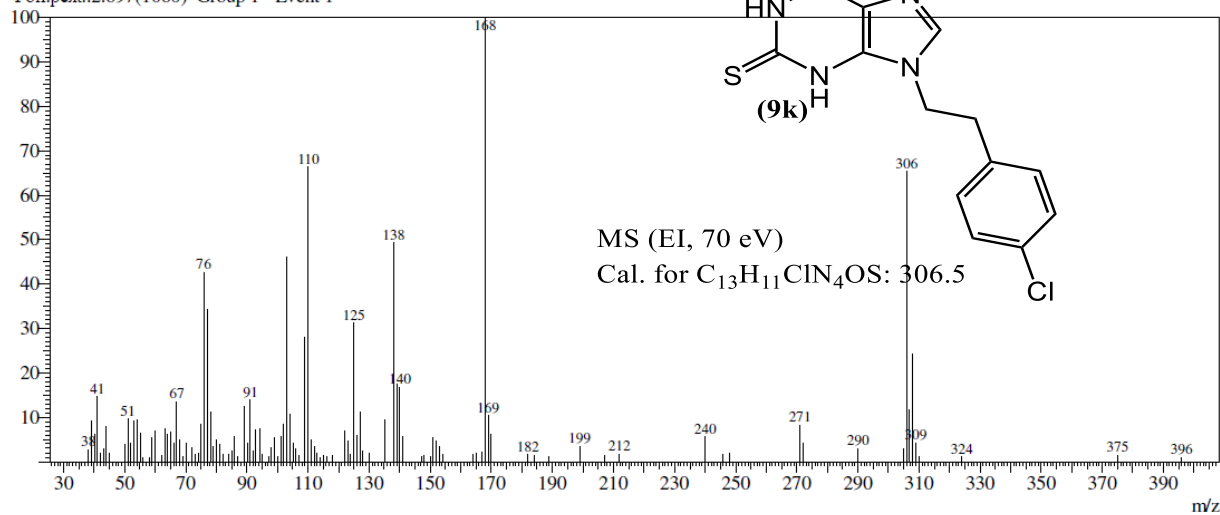
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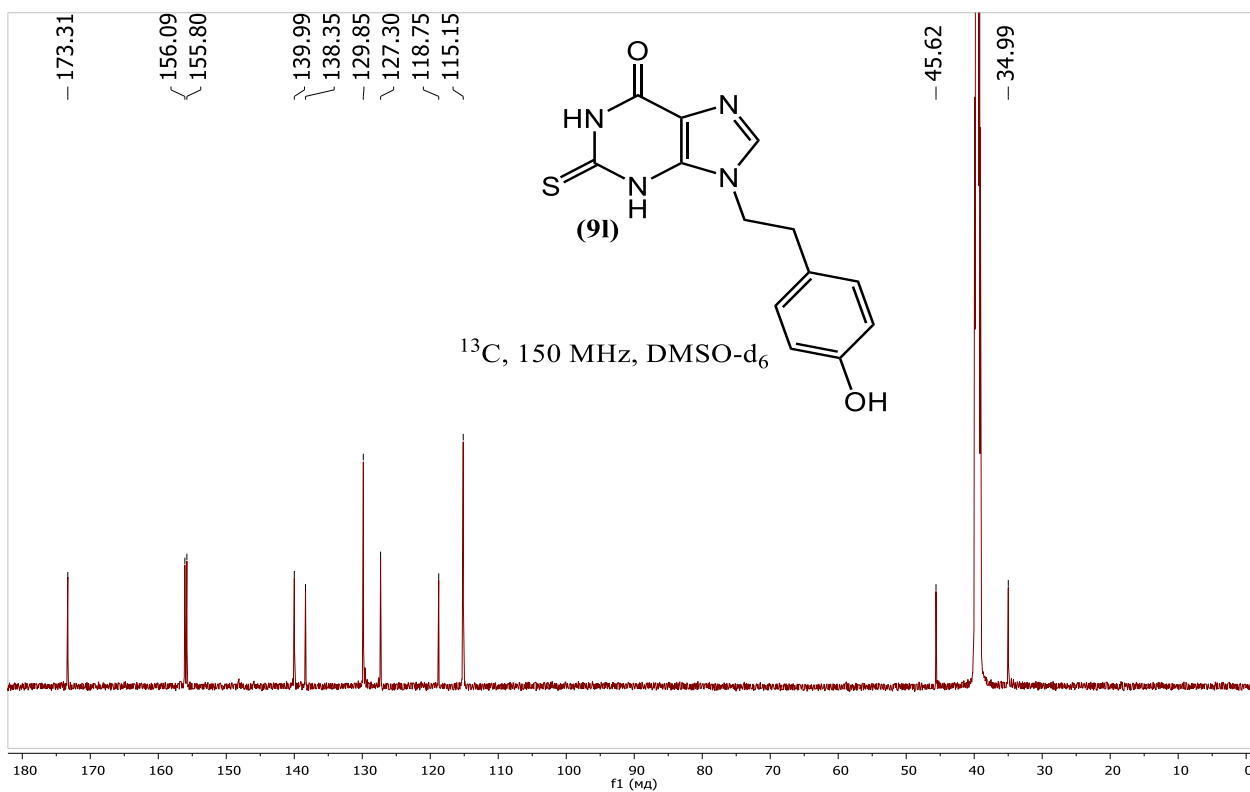
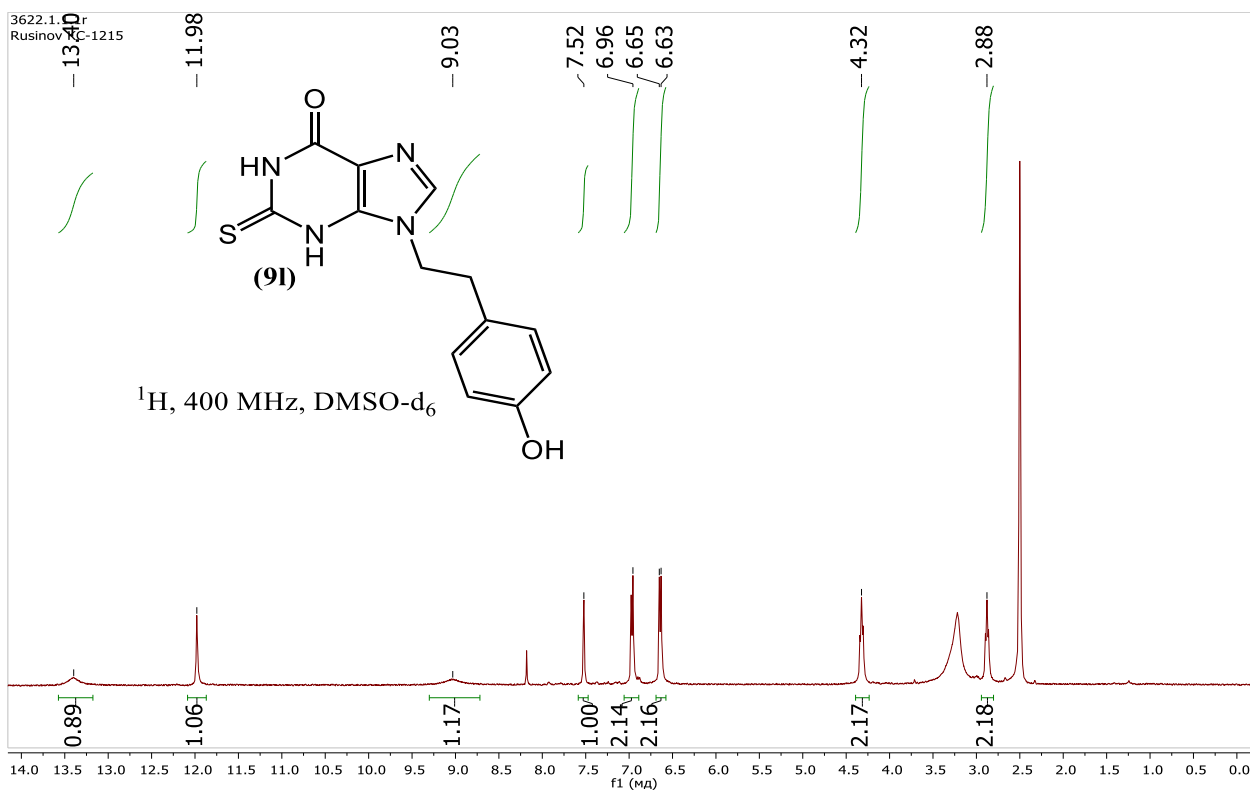
Line#:2 R.Time:3.200(Scan#:1261)

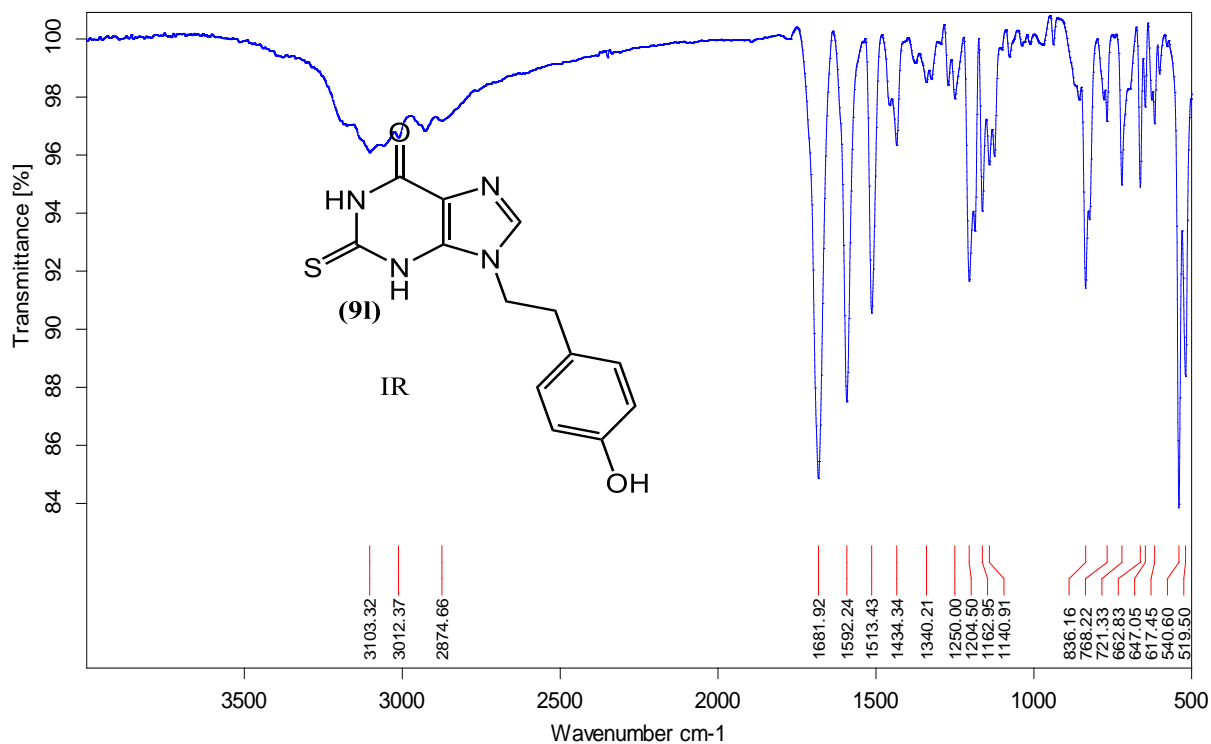
MassPeaks:116

RawMode:Single 3.200(1261) BasePeak:168(331923)

Фон.реж.:2.697(1060) Group 1 - Event 1







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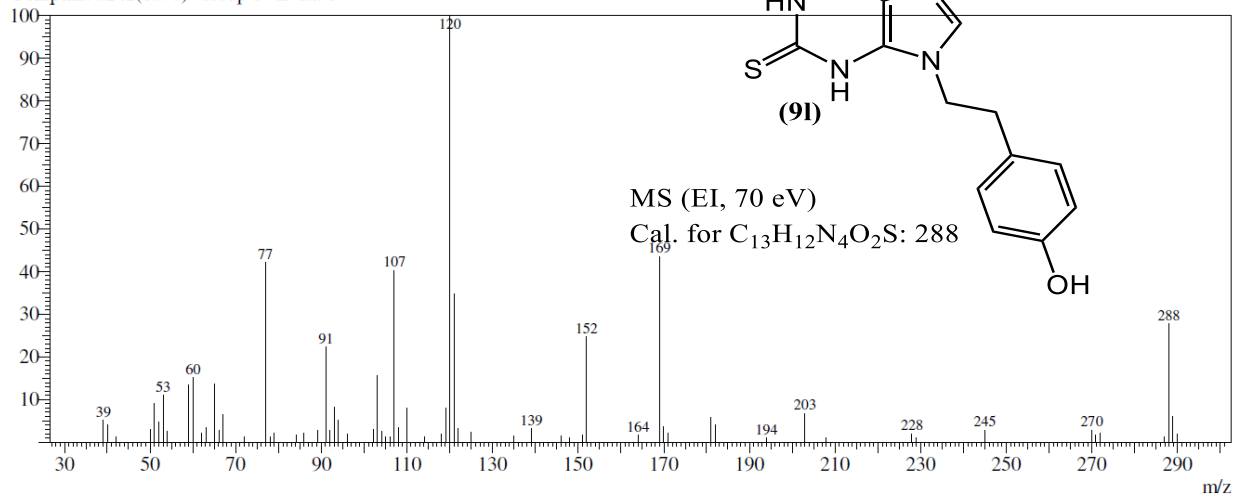
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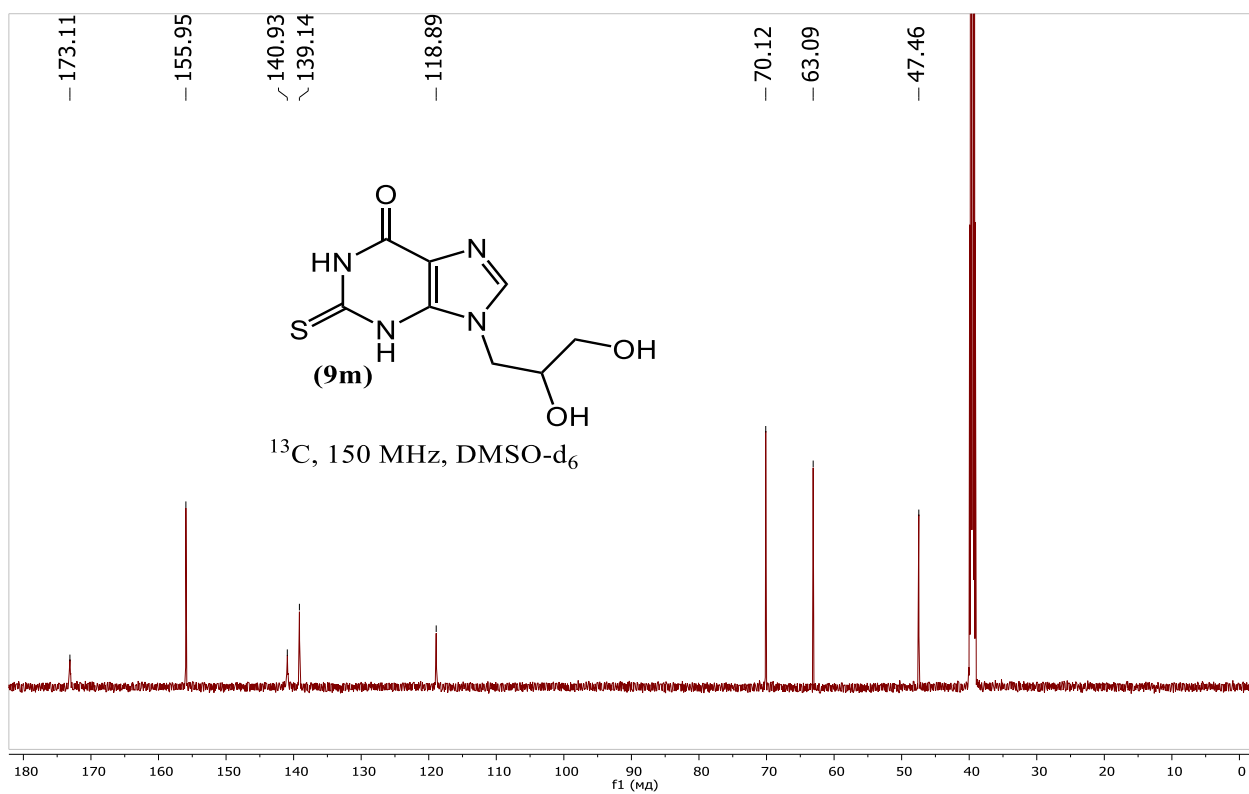
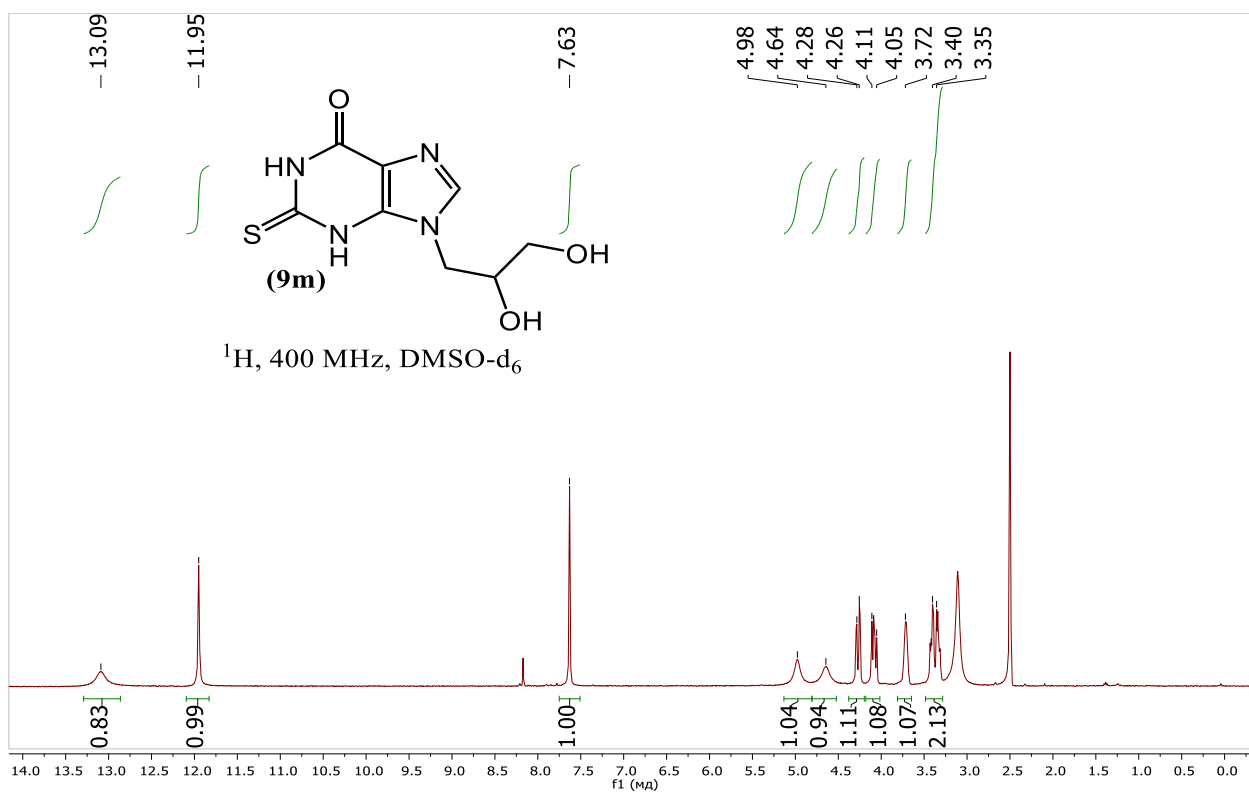
Line#:1 R.Time:4.877(Scan#:1932)

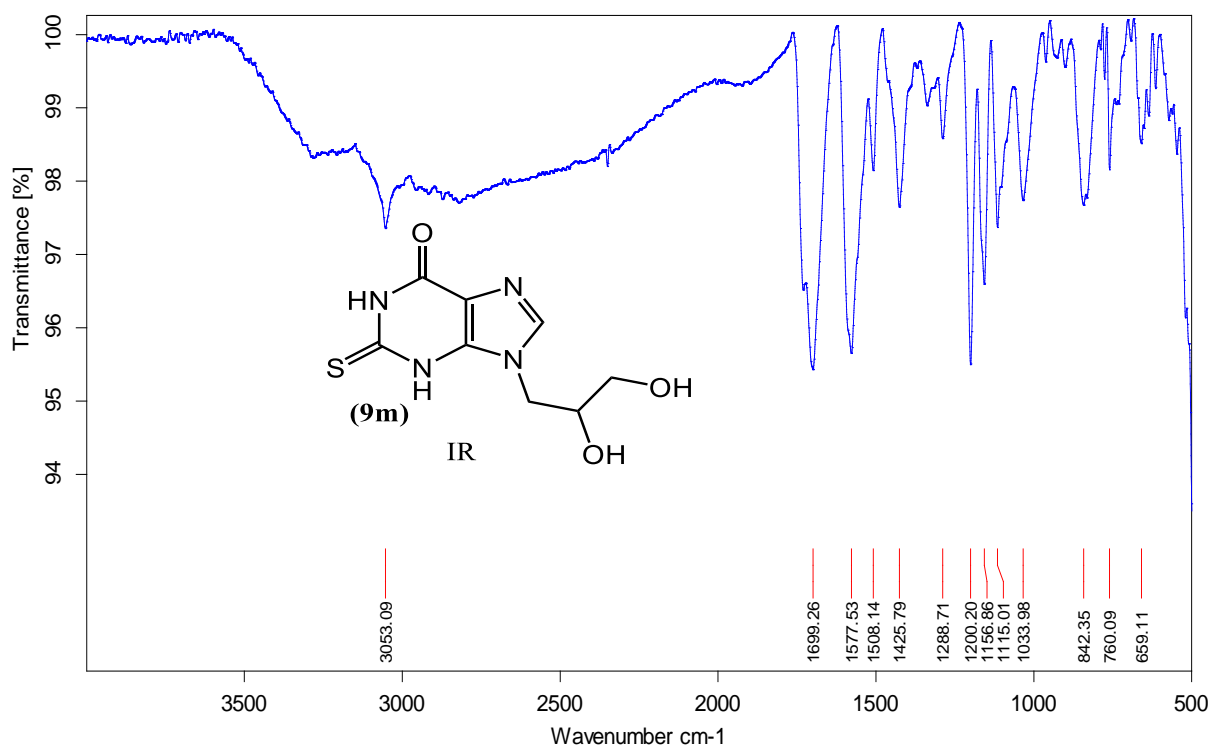
MassPeaks:67

RawMode:Single 4.878(1932) BasePeak:120(88211)

Фон.реж.:4.242(1678) Group 1 - Event 1



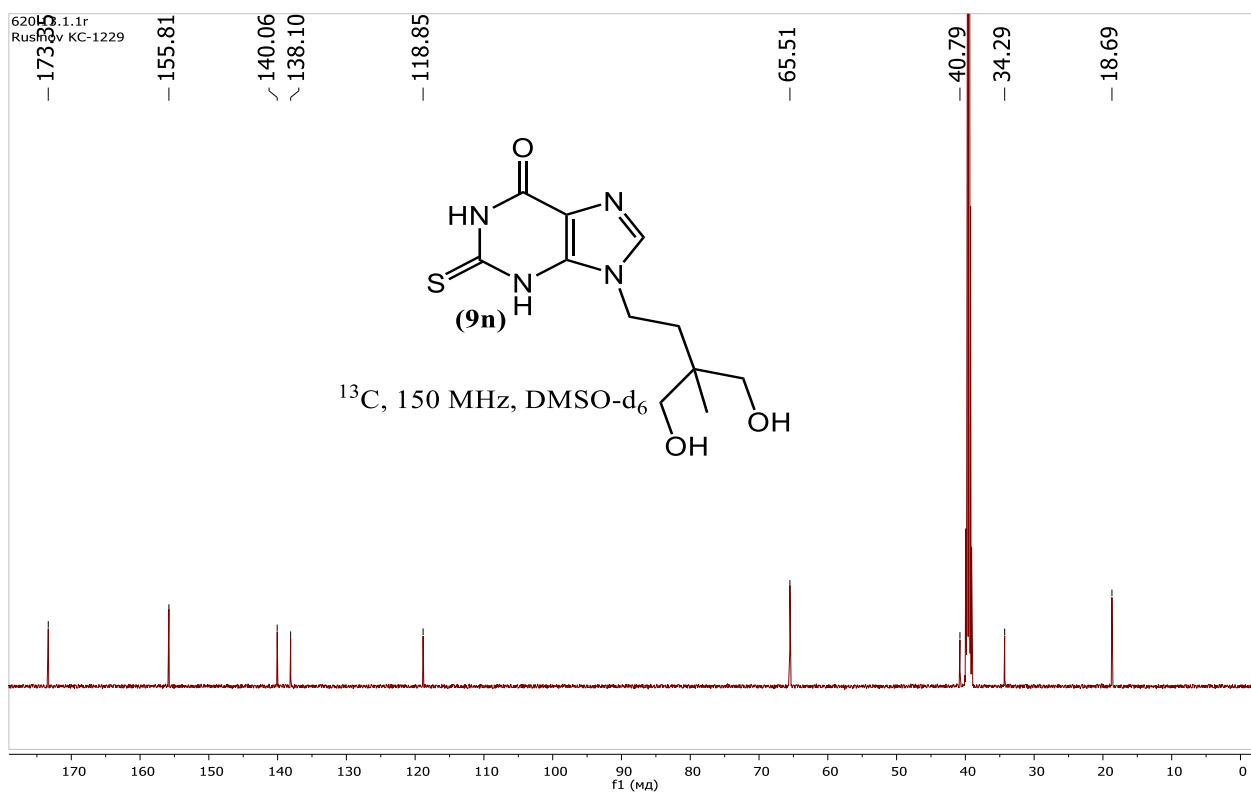
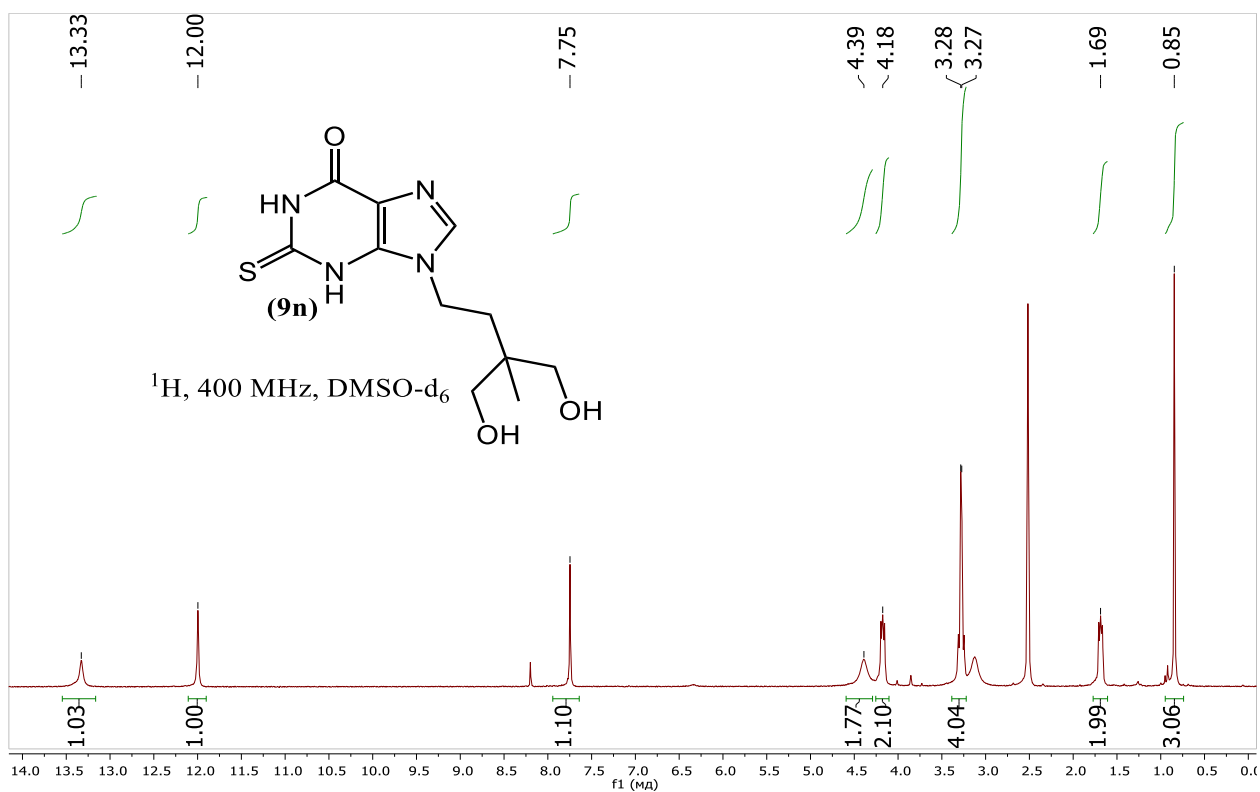




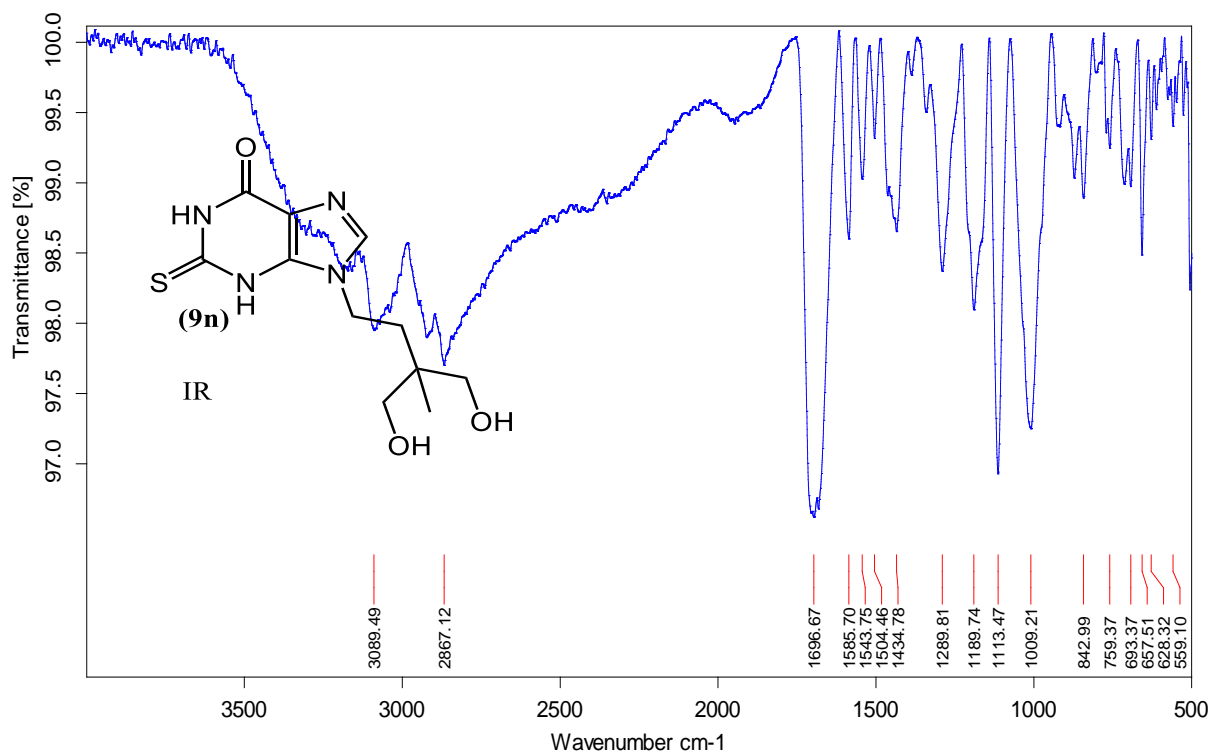
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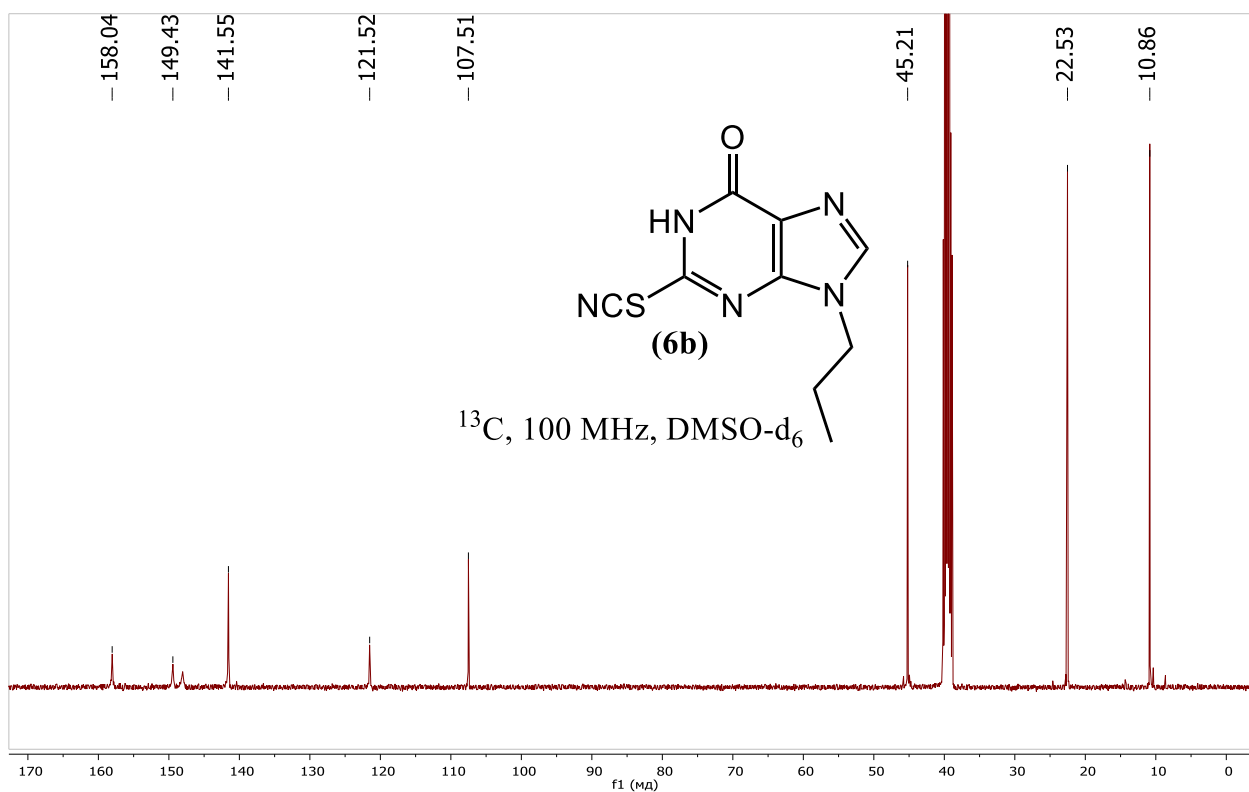
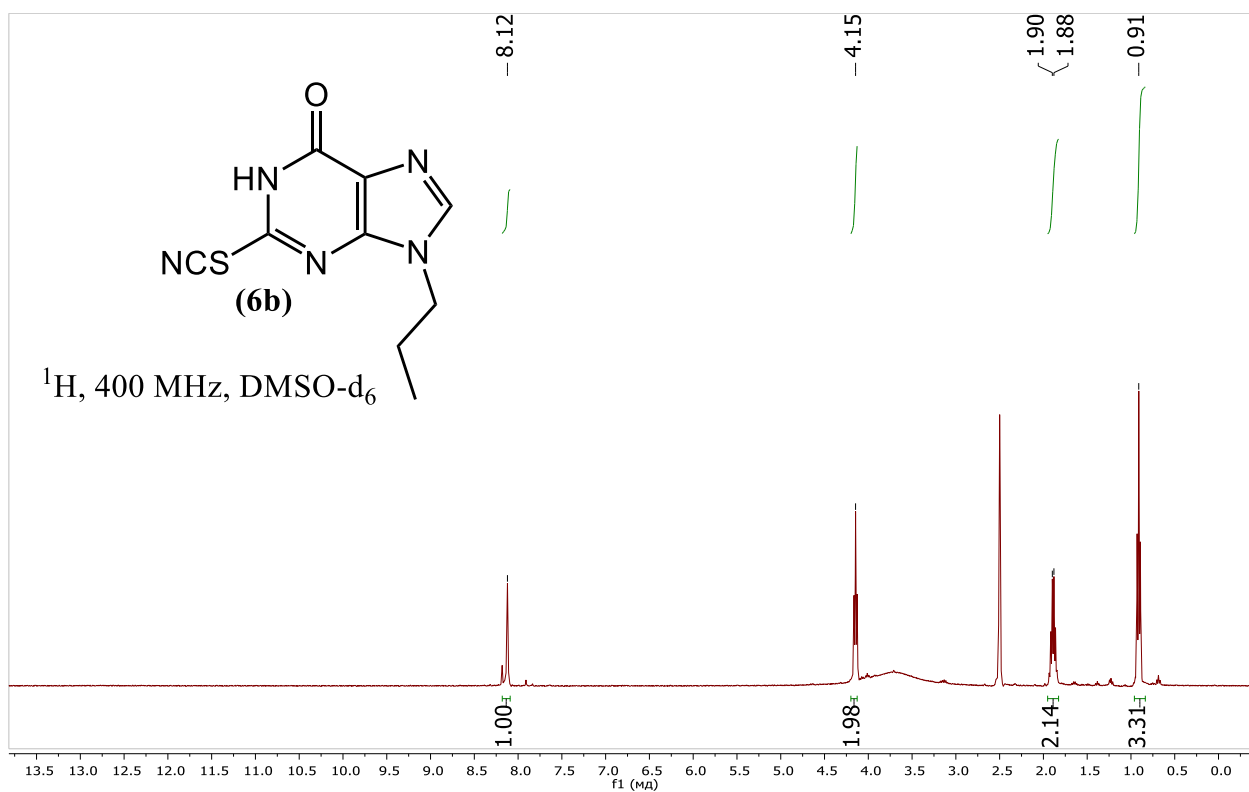


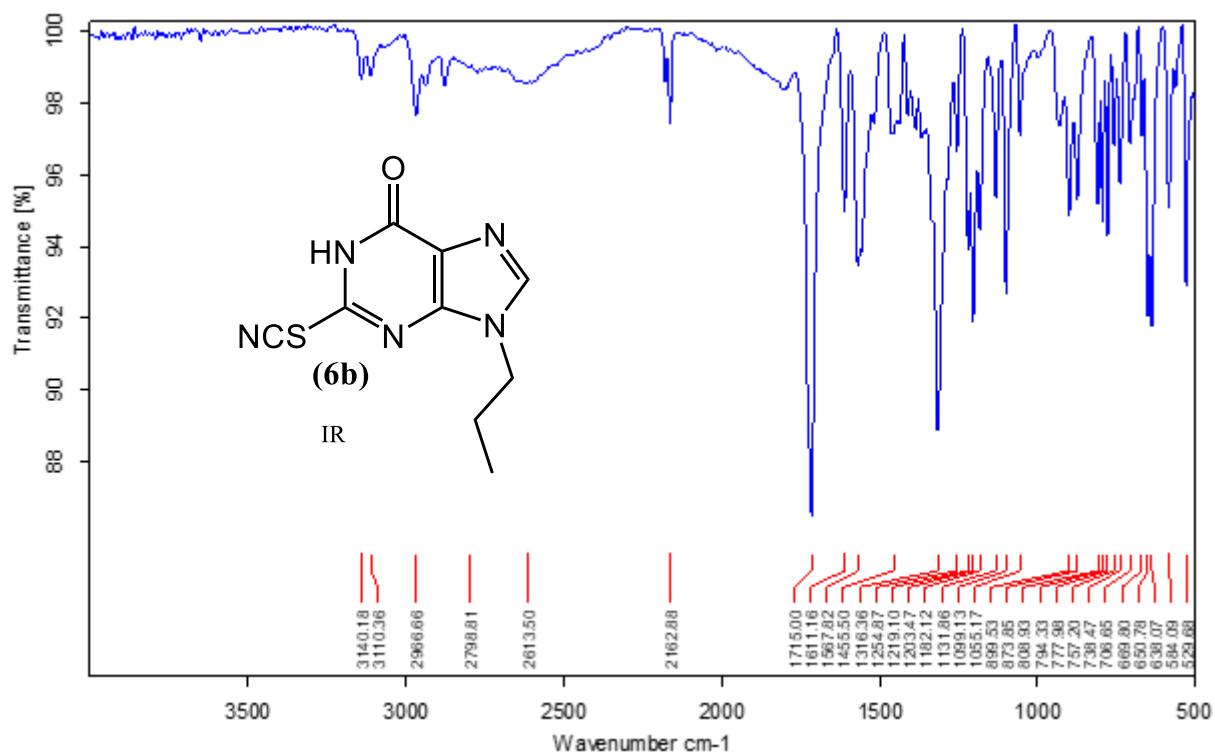


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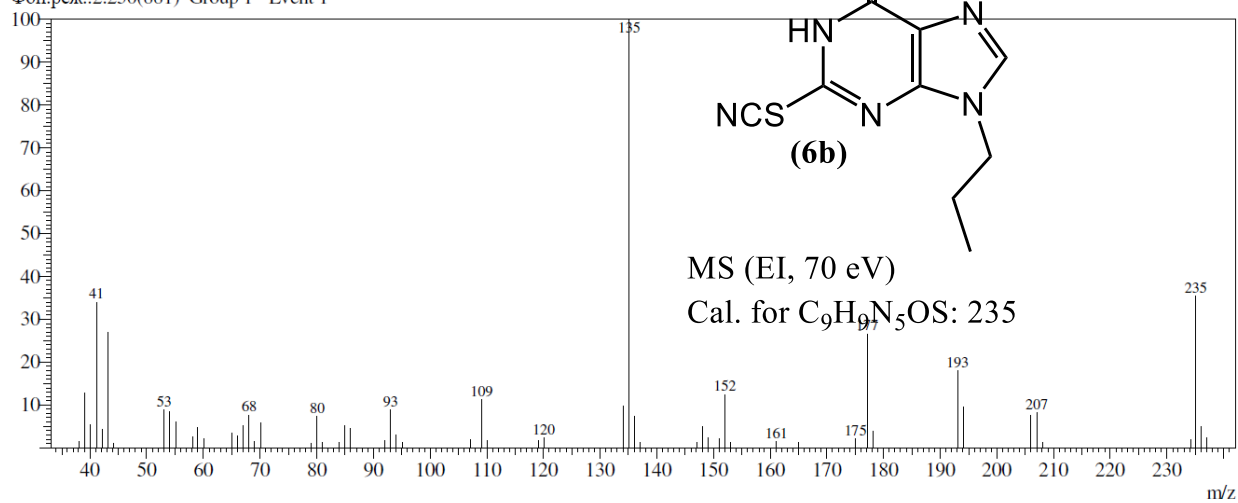
kc-1229

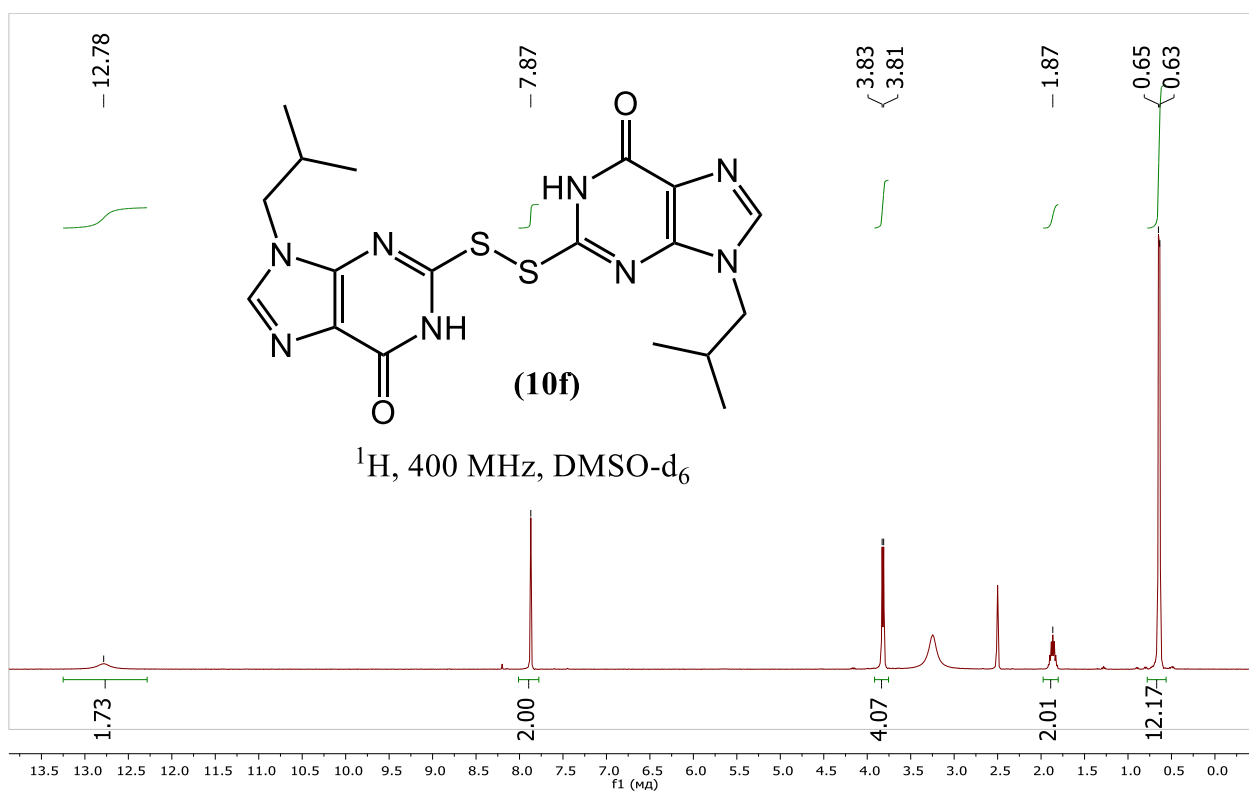
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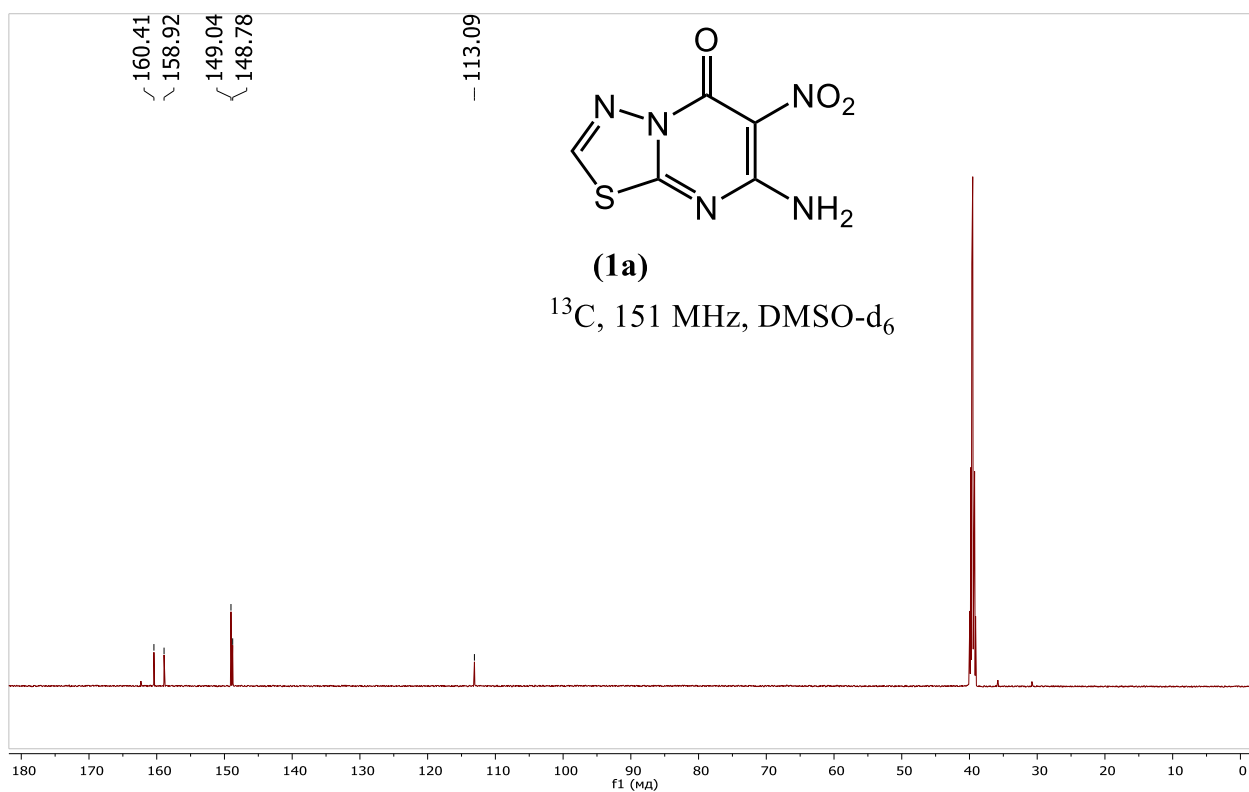
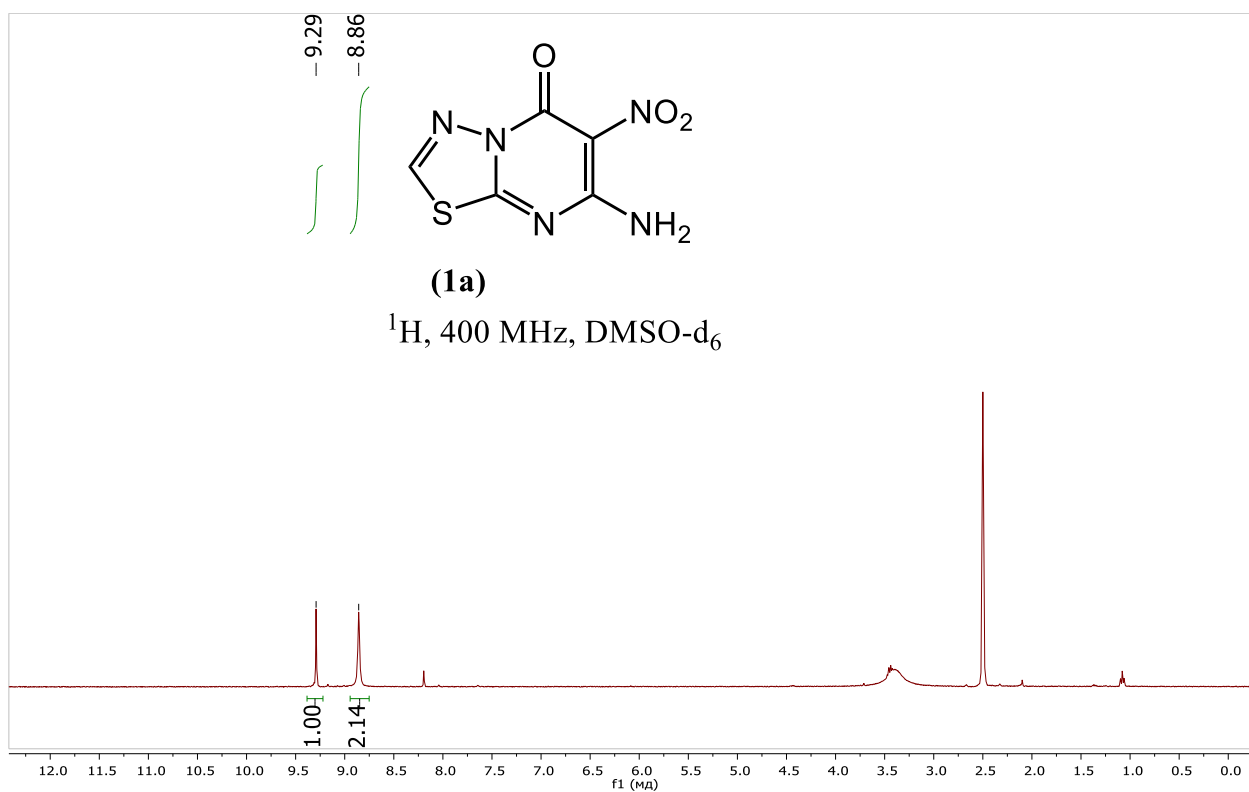


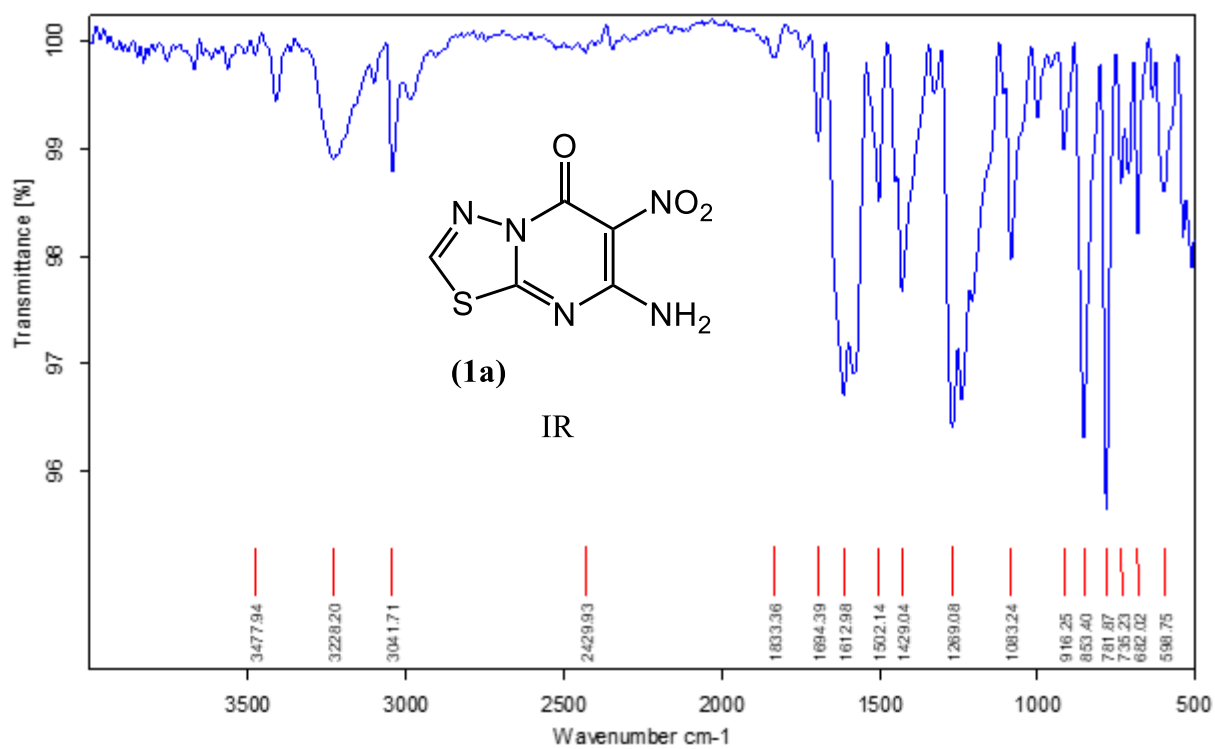


Line#:1 R.Time:2.462(Scan#:966)  
 MassPeaks:58  
 RawMode:Single 2.462(966) BasePeak:135(369165)  
 Фон.реж.:2.250(881) Group 1 - Event 1

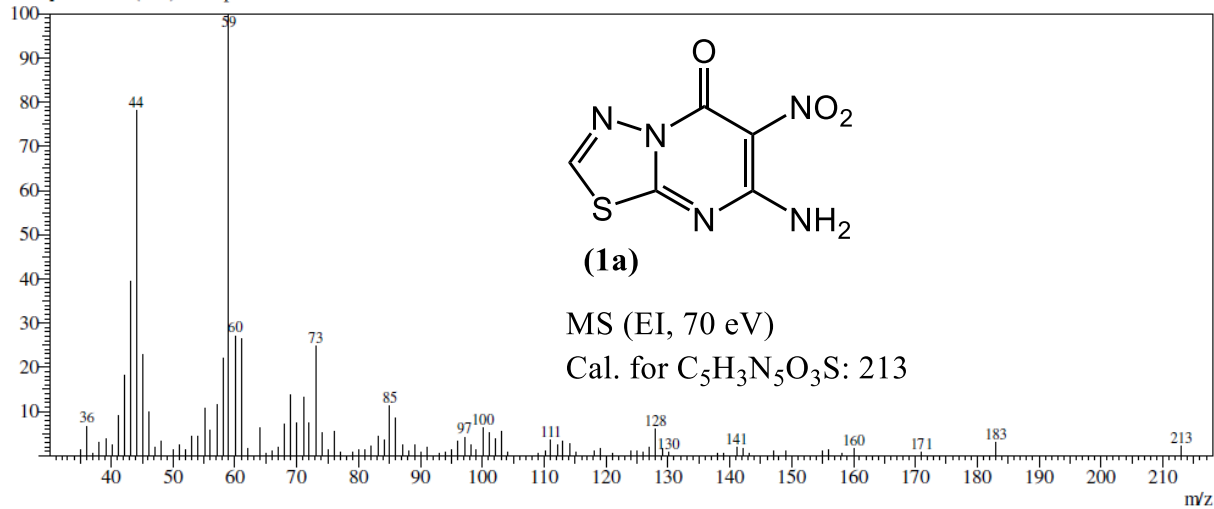


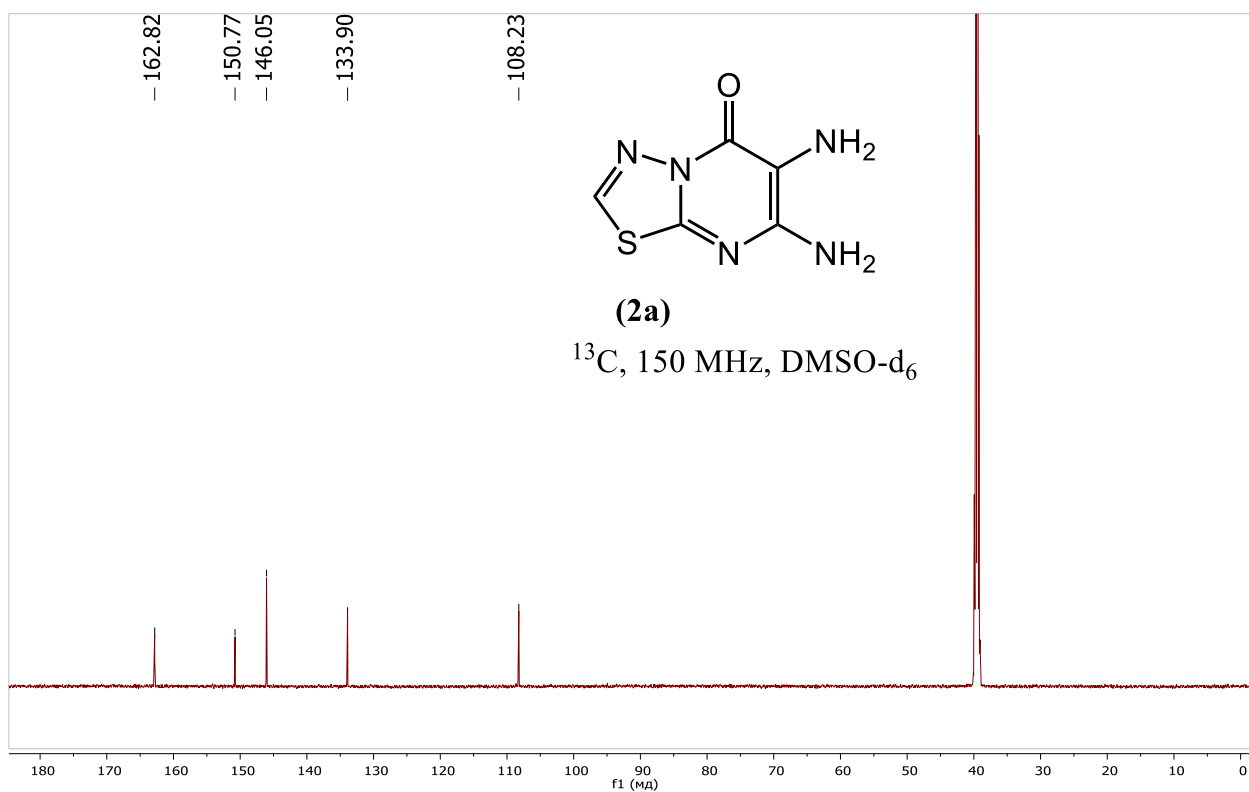
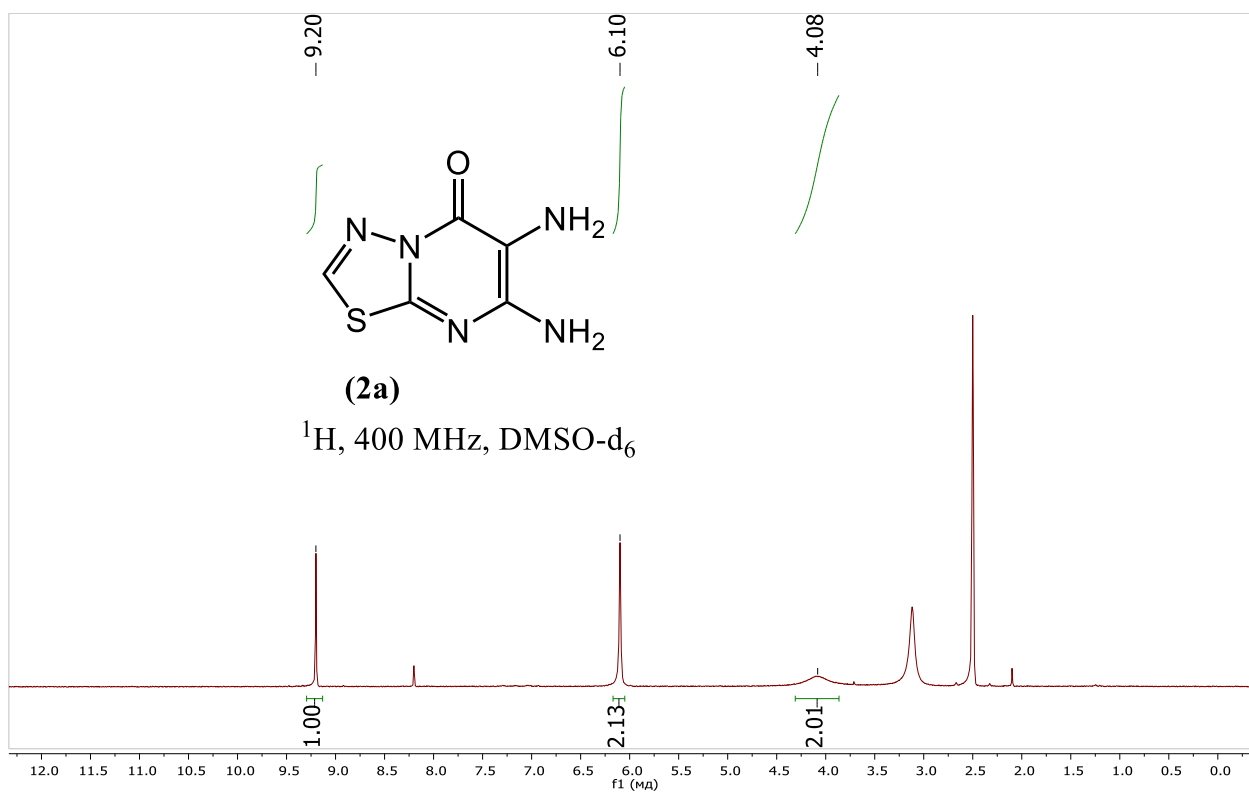


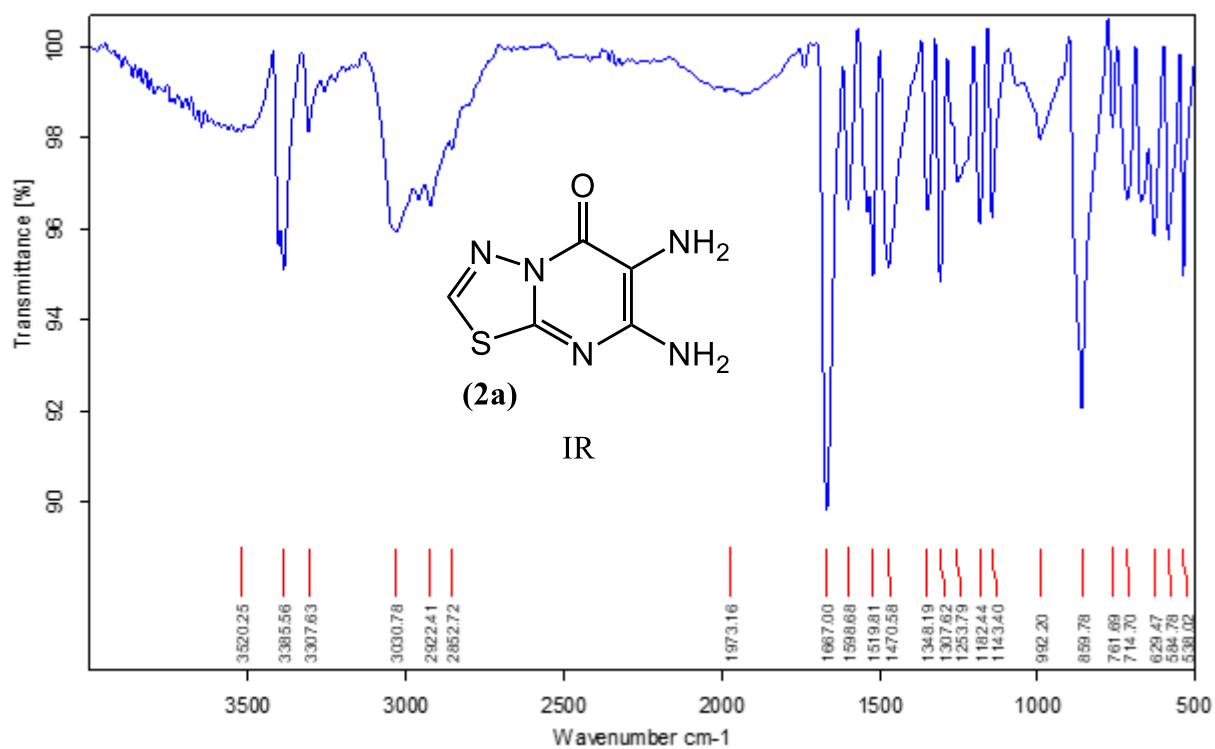




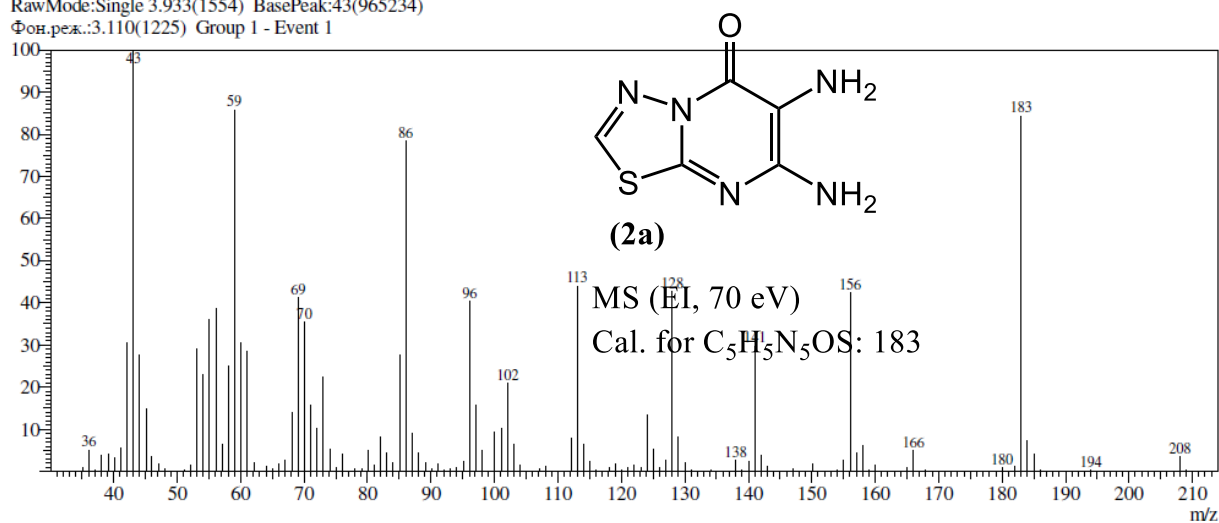
Line#:1 R.Time:2.663(Scan#:1046)  
 MassPeaks:97  
 RawMode:Single 2.663(1046) BasePeak:59(902376)  
 Фон.реж.:1.050(401) Group 1 - Event 1



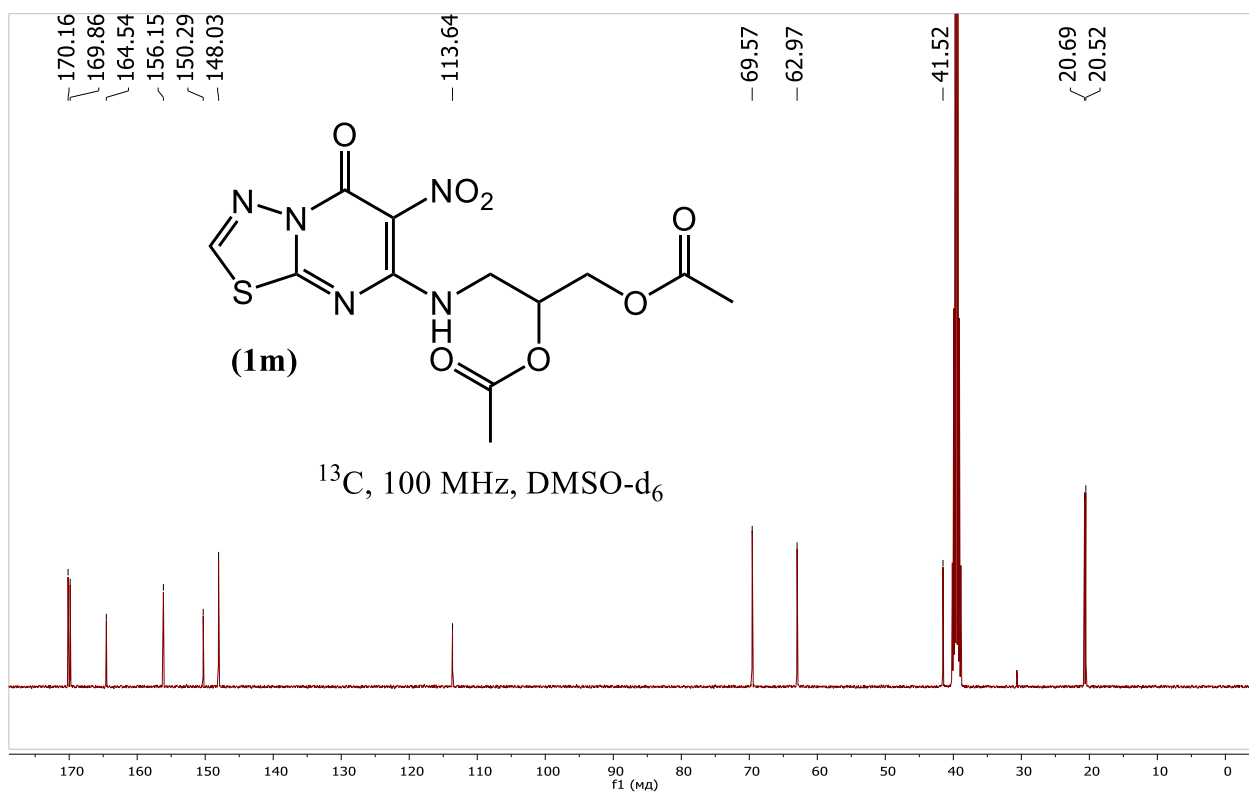
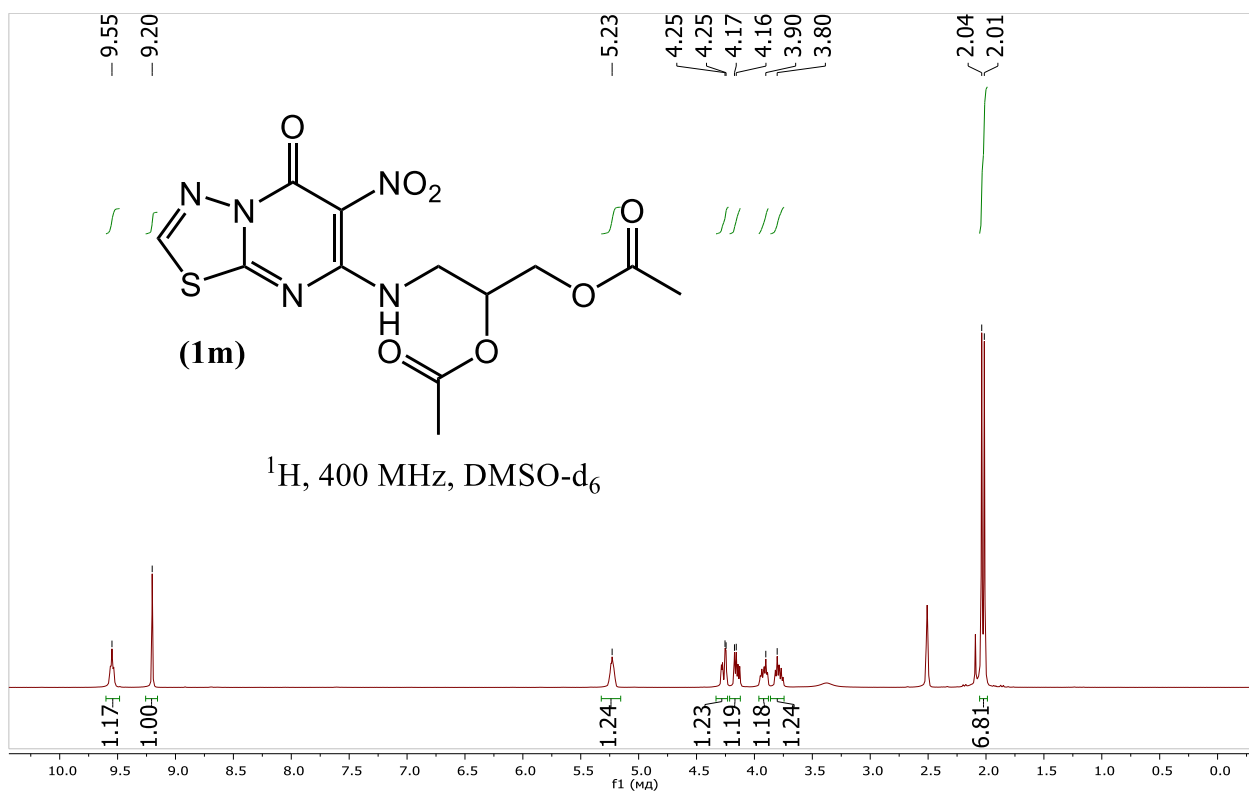


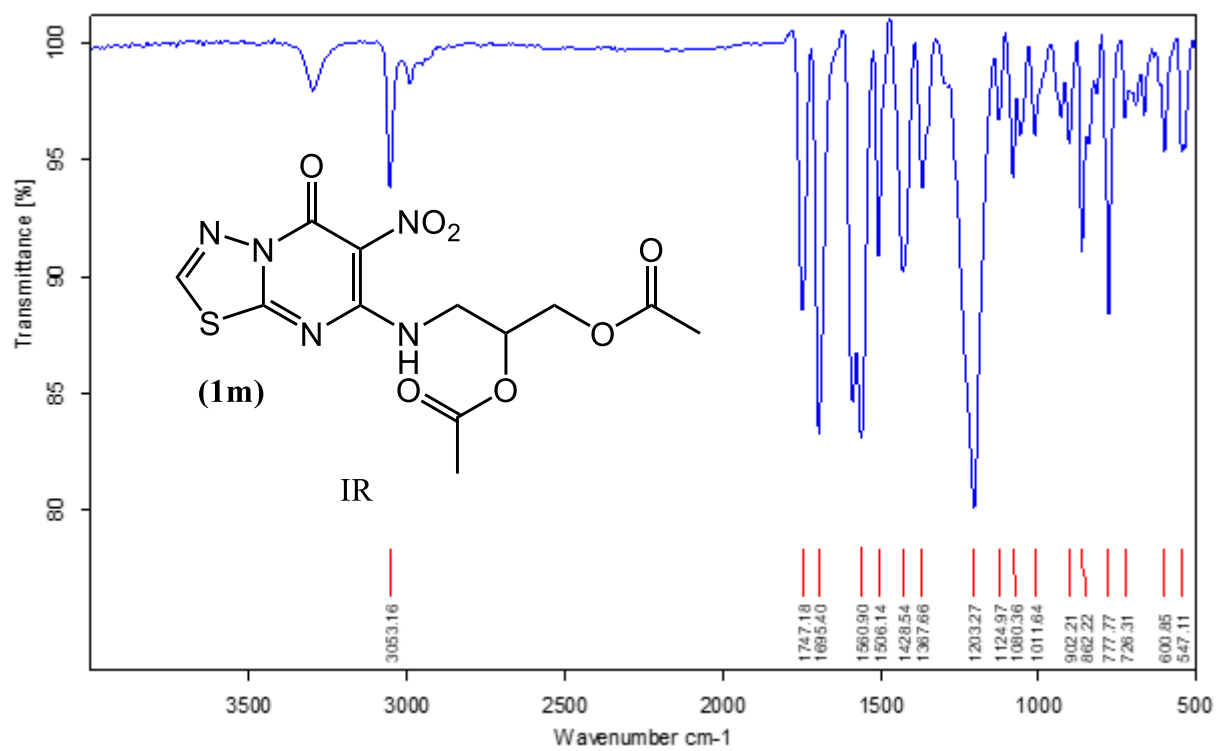


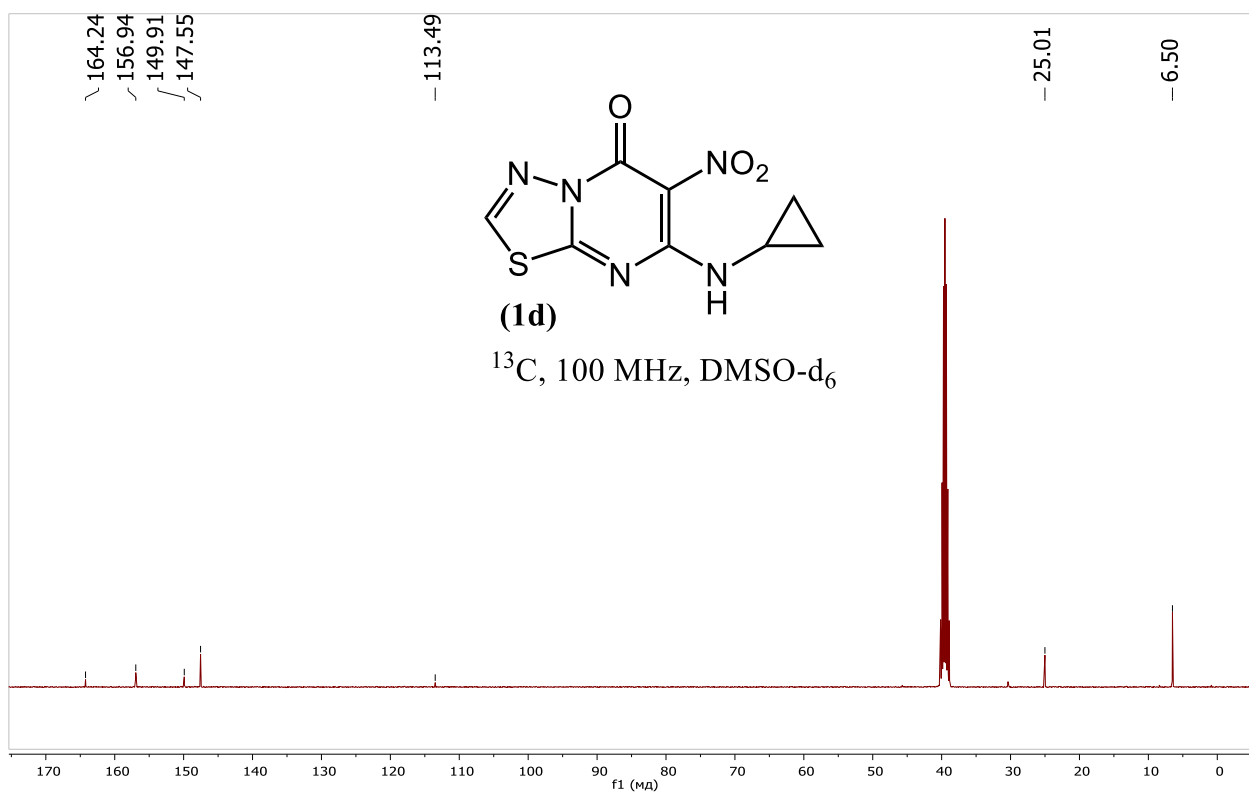
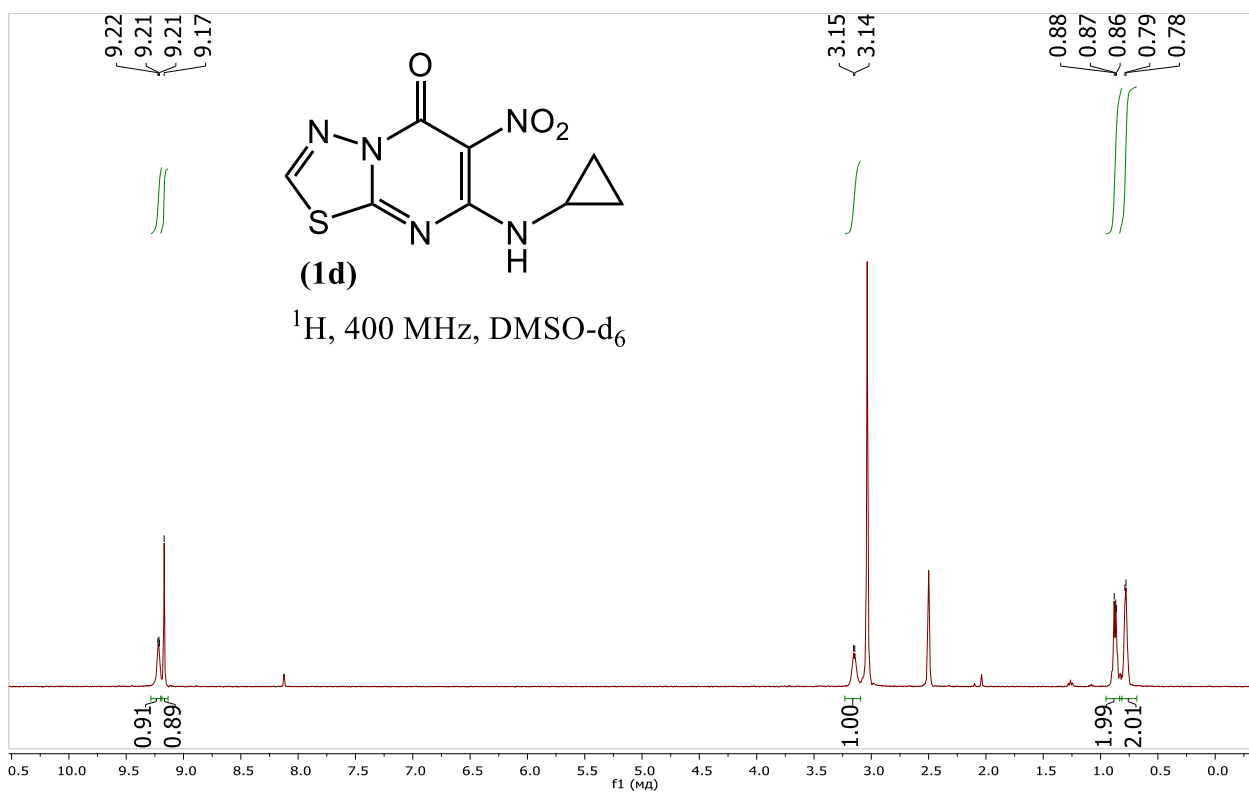
Line#:1 R.Time:3.932(Scan#:1554)  
 MassPeaks:114  
 RawMode:Single 3.933(1554) BasePeak:43(965234)  
 Фон.реж.:3.110(1225) Group 1 - Event 1

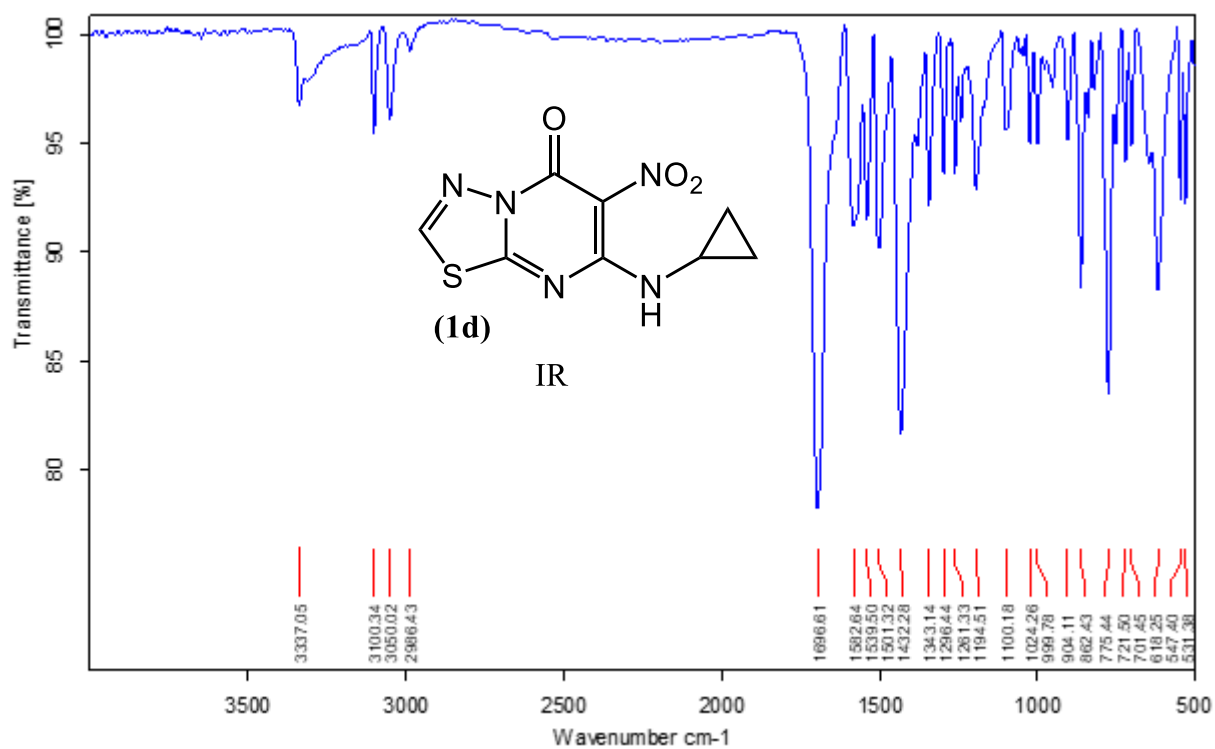










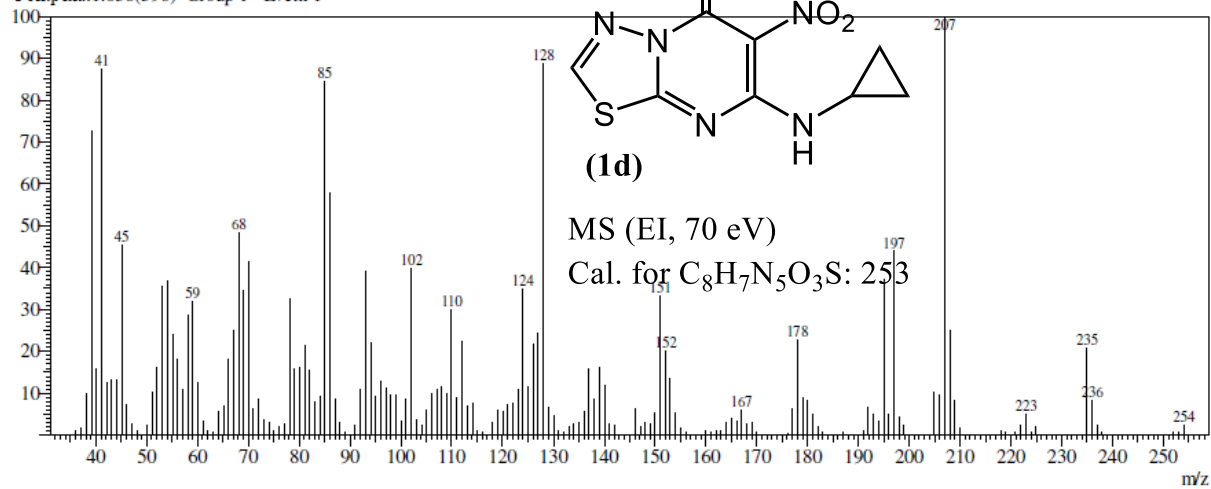


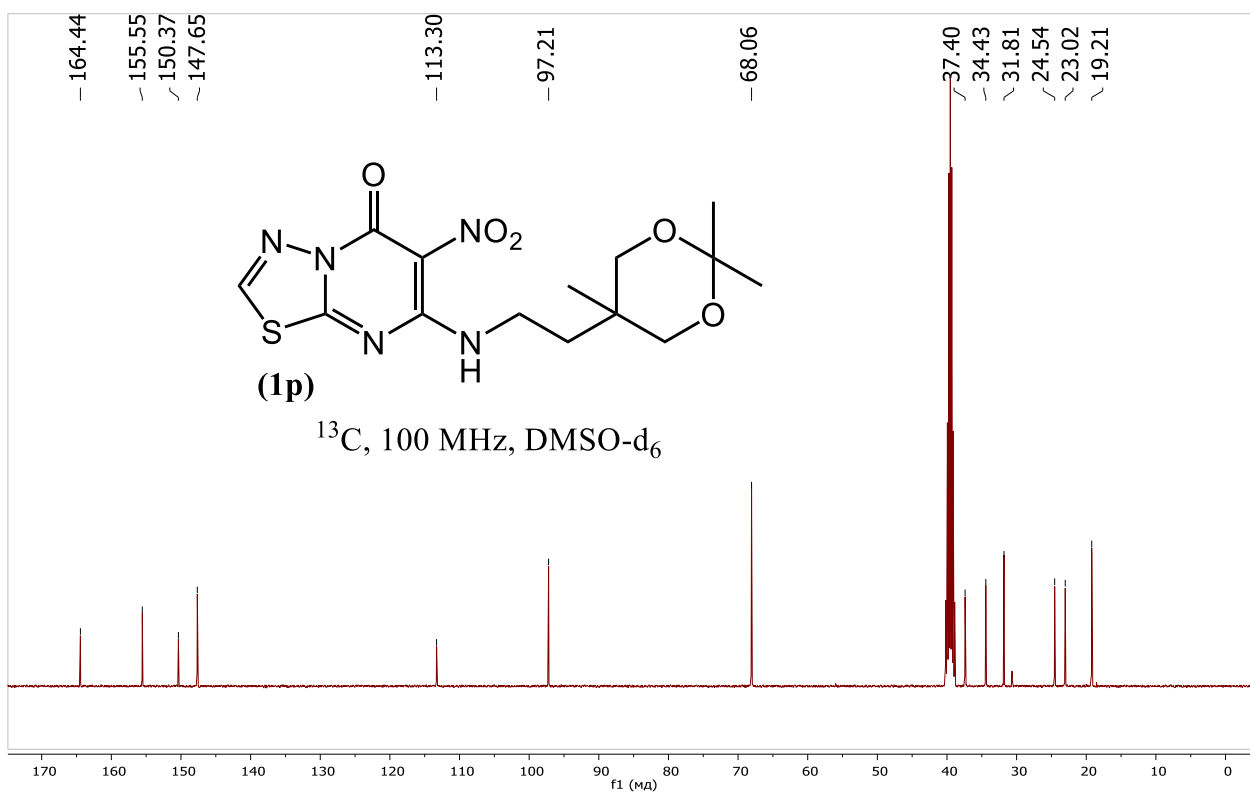
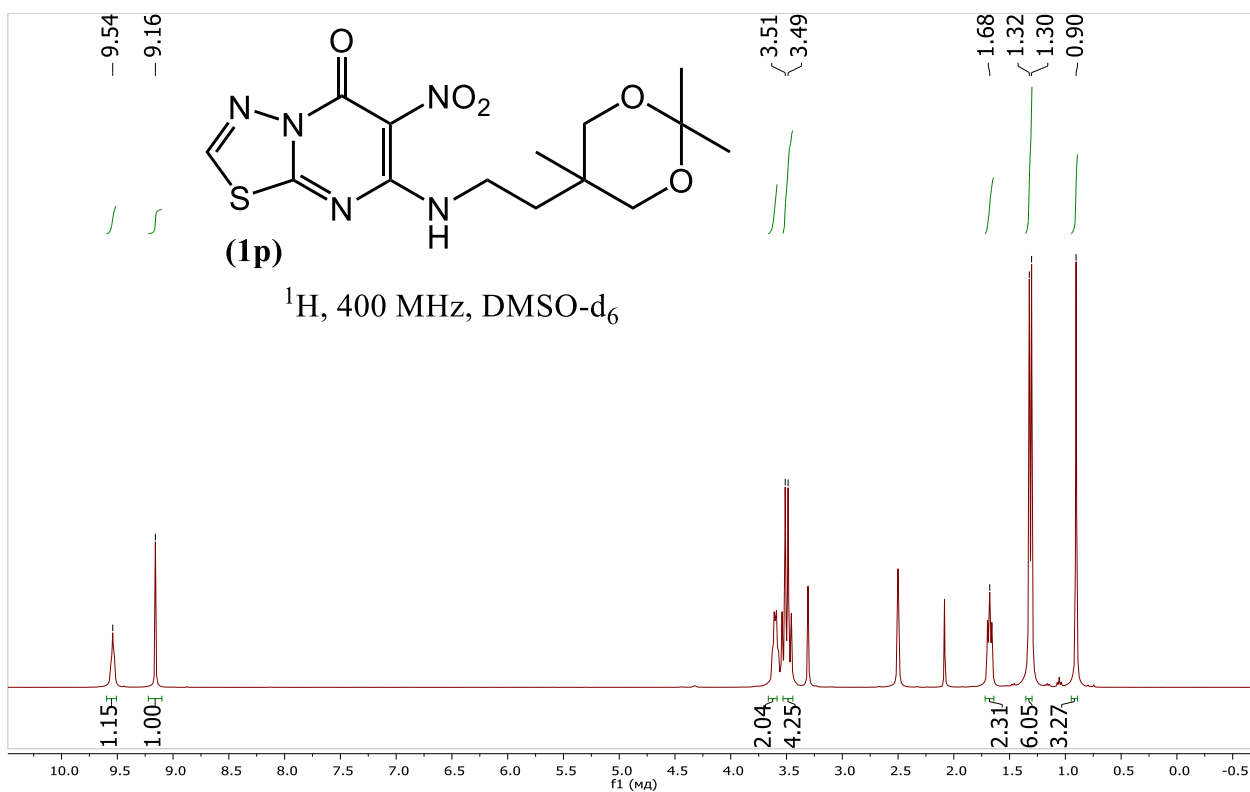
Line#:1 R.Time:2.422(Scan#:950)

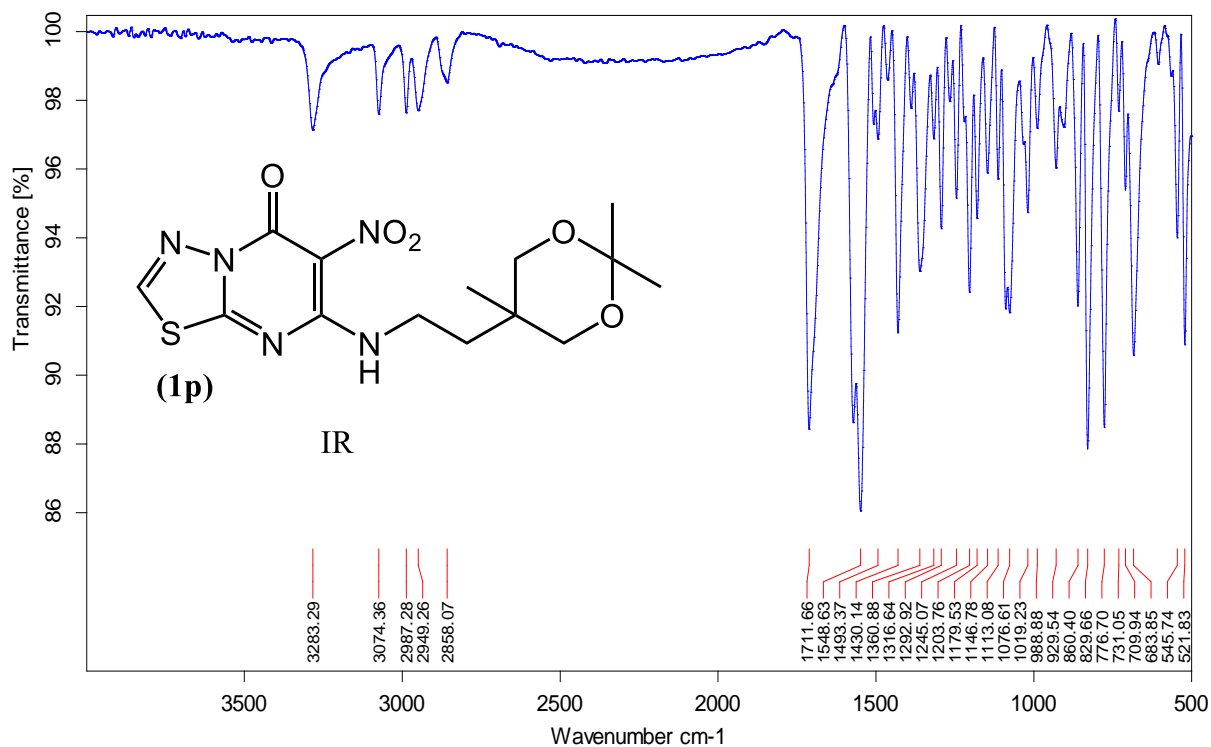
MassPeaks:164

RawMode:Single 2.422(950) BasePeak:207(1648651)

Фон.р.ж.:1.038(396) Group 1 - Event 1







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KC-1146

27/09/2021

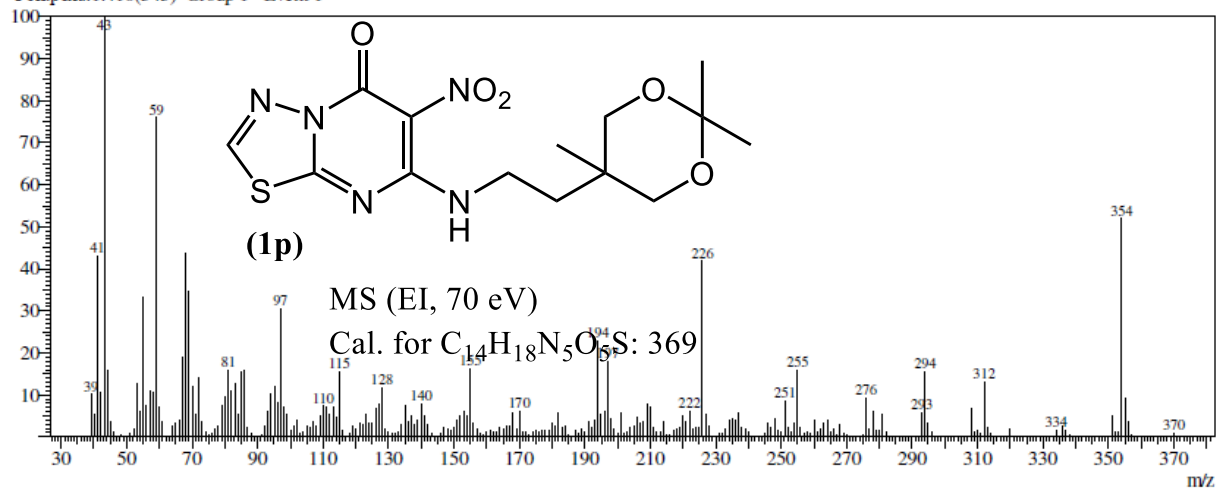
Page 1/1

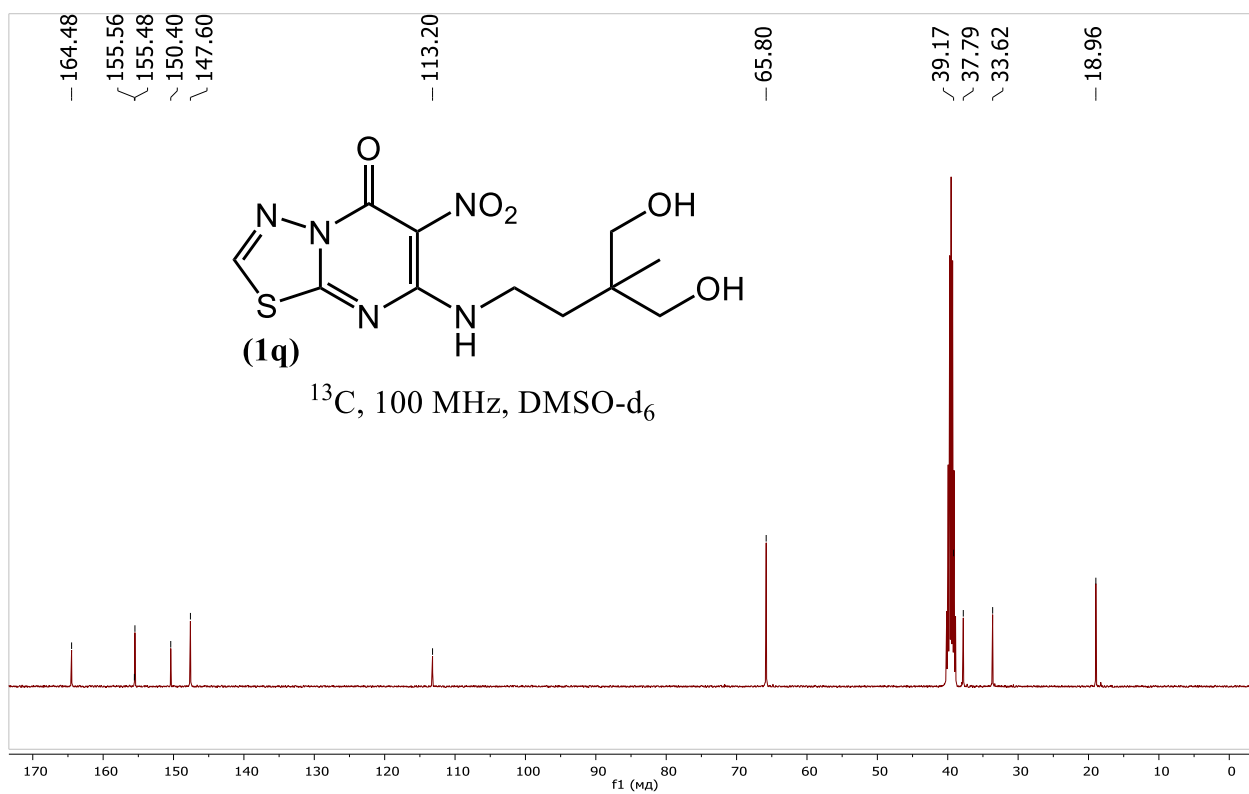
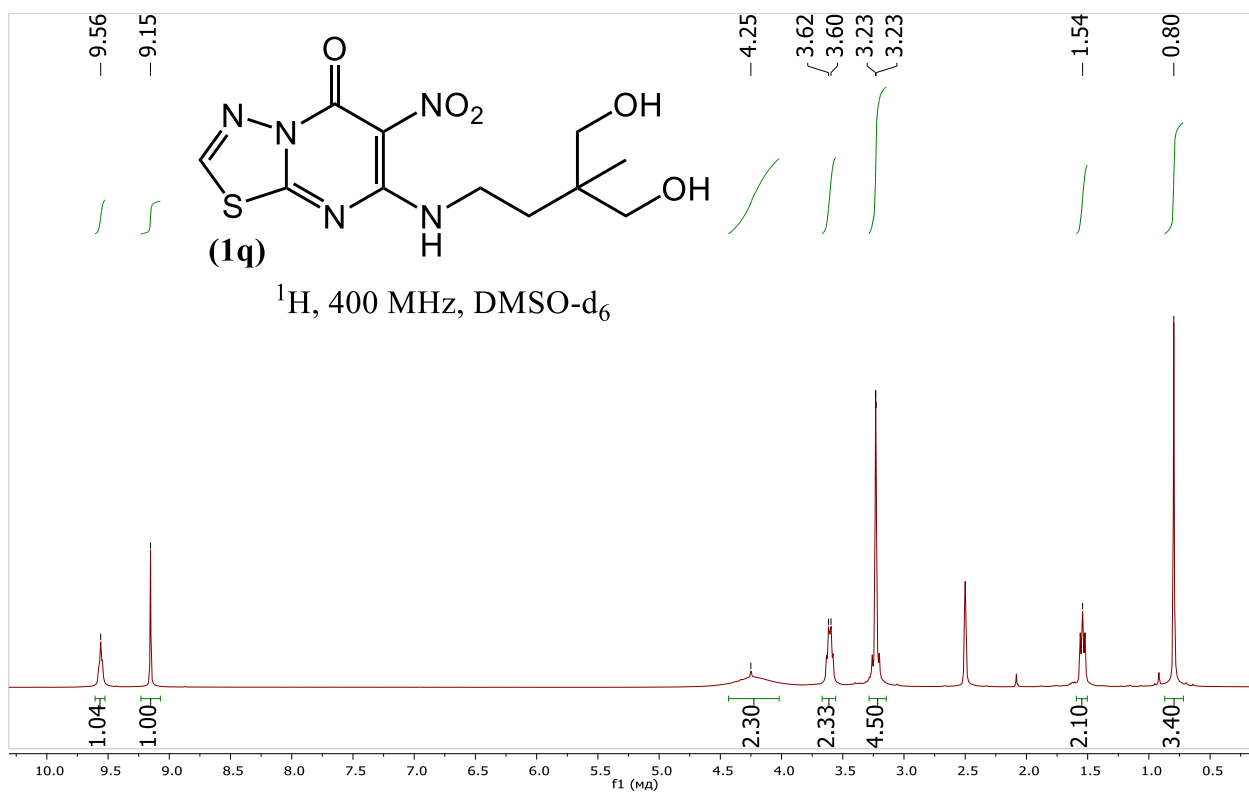
Line#:1 R.Time:2.822(Scan#:1110)

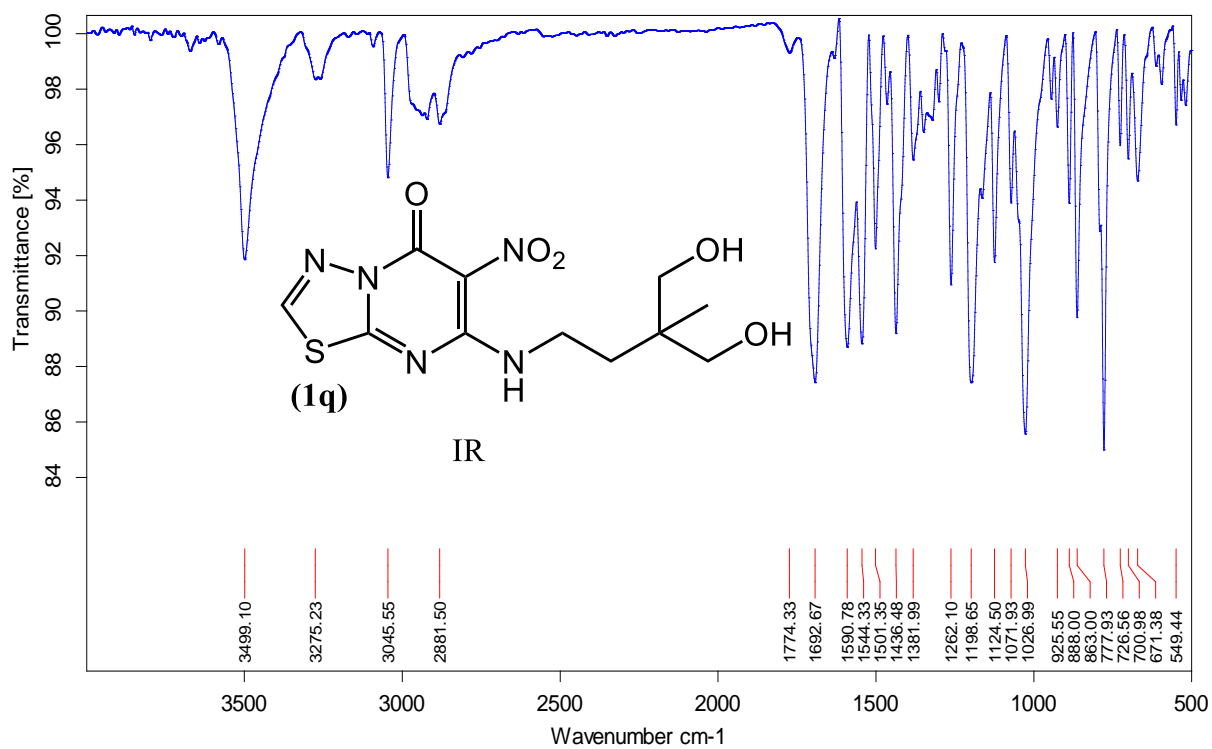
MassPeaks:247

RawMode:Single 2.822(1110) BasePeak:43(3217246)

Фон.р.к.:1.410(545) Group 1 - Event 1





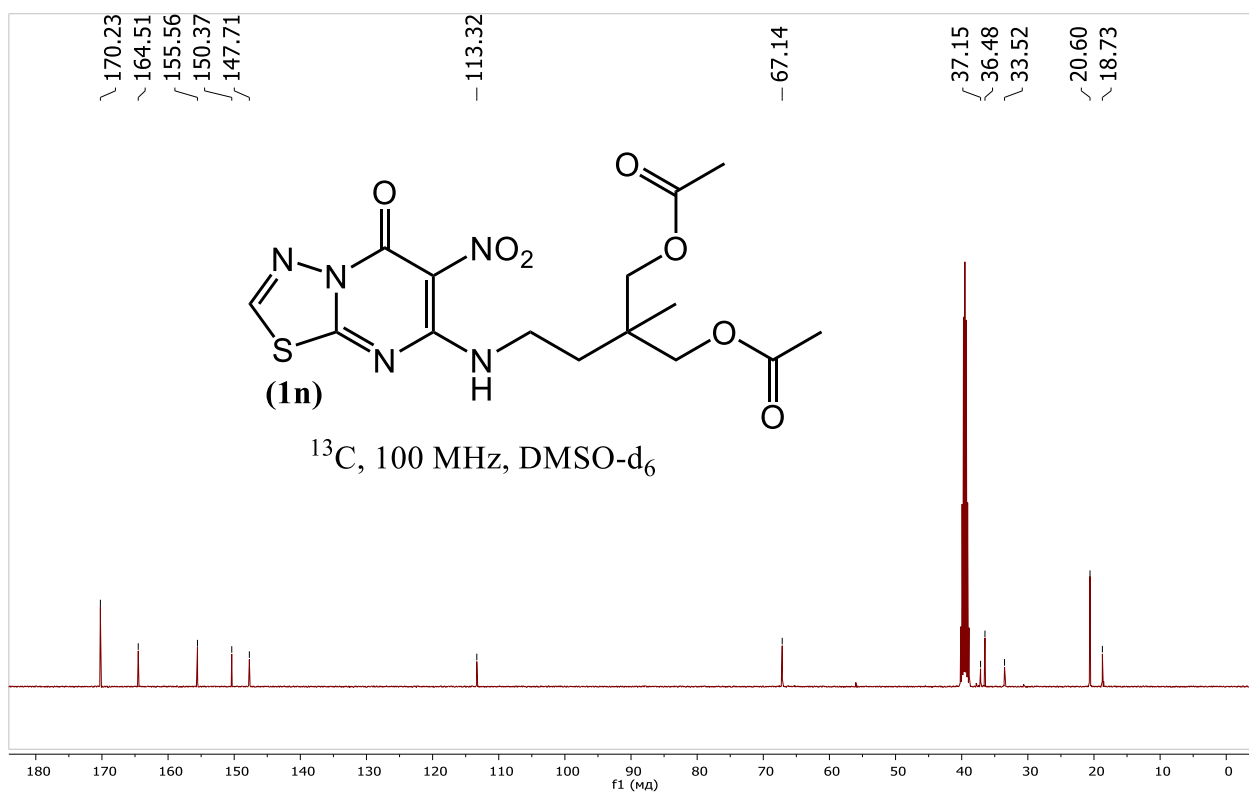
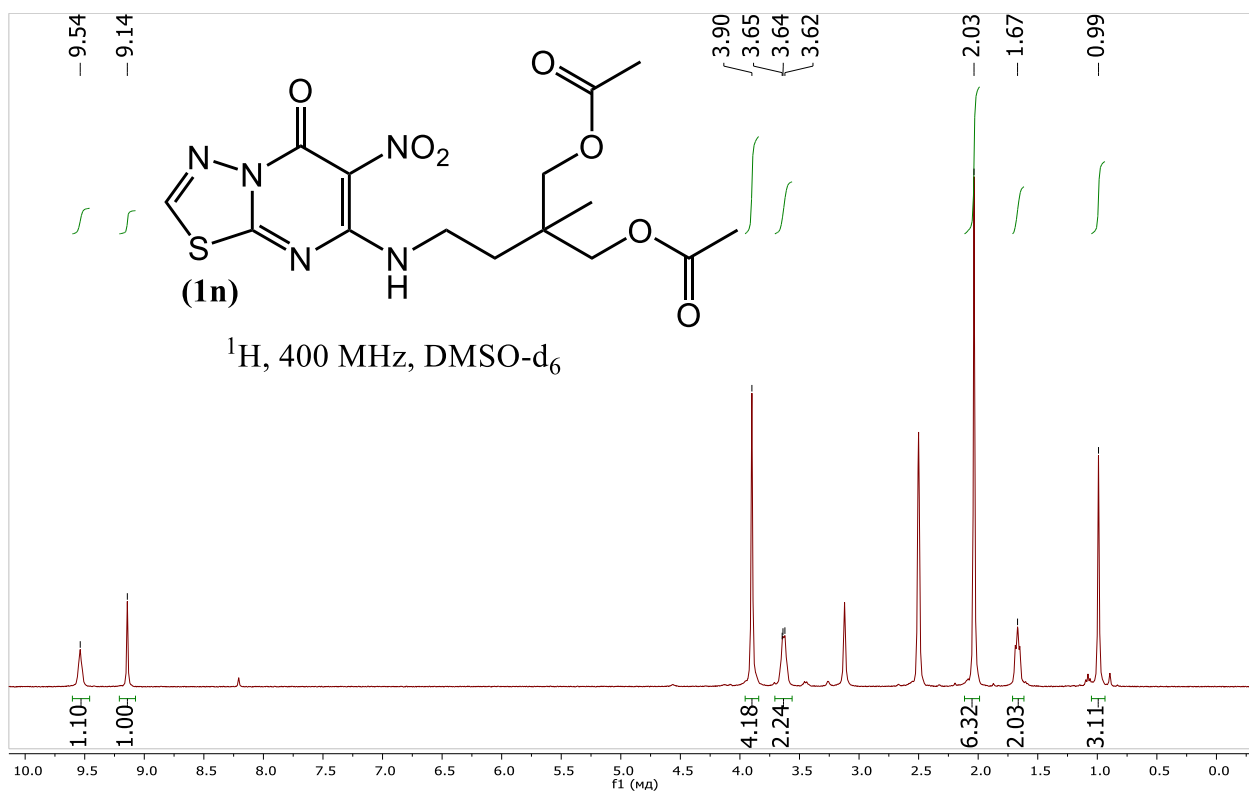


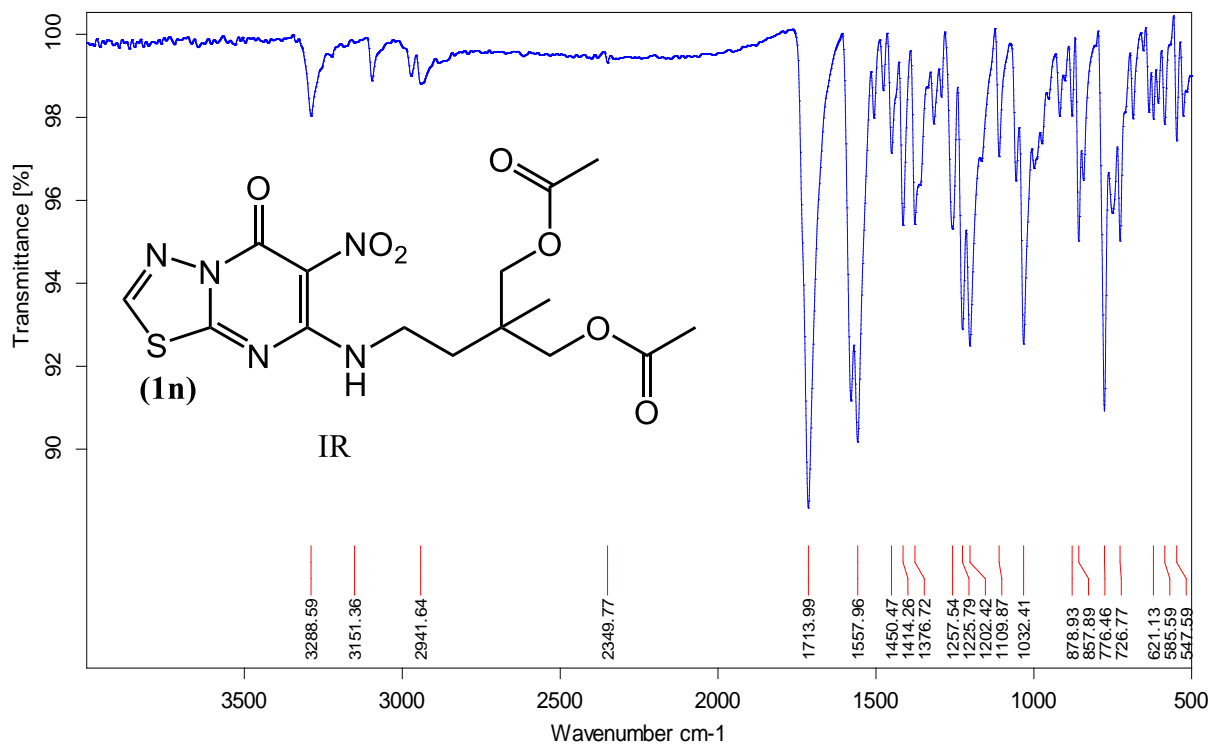
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KC-1199

27/09/2021



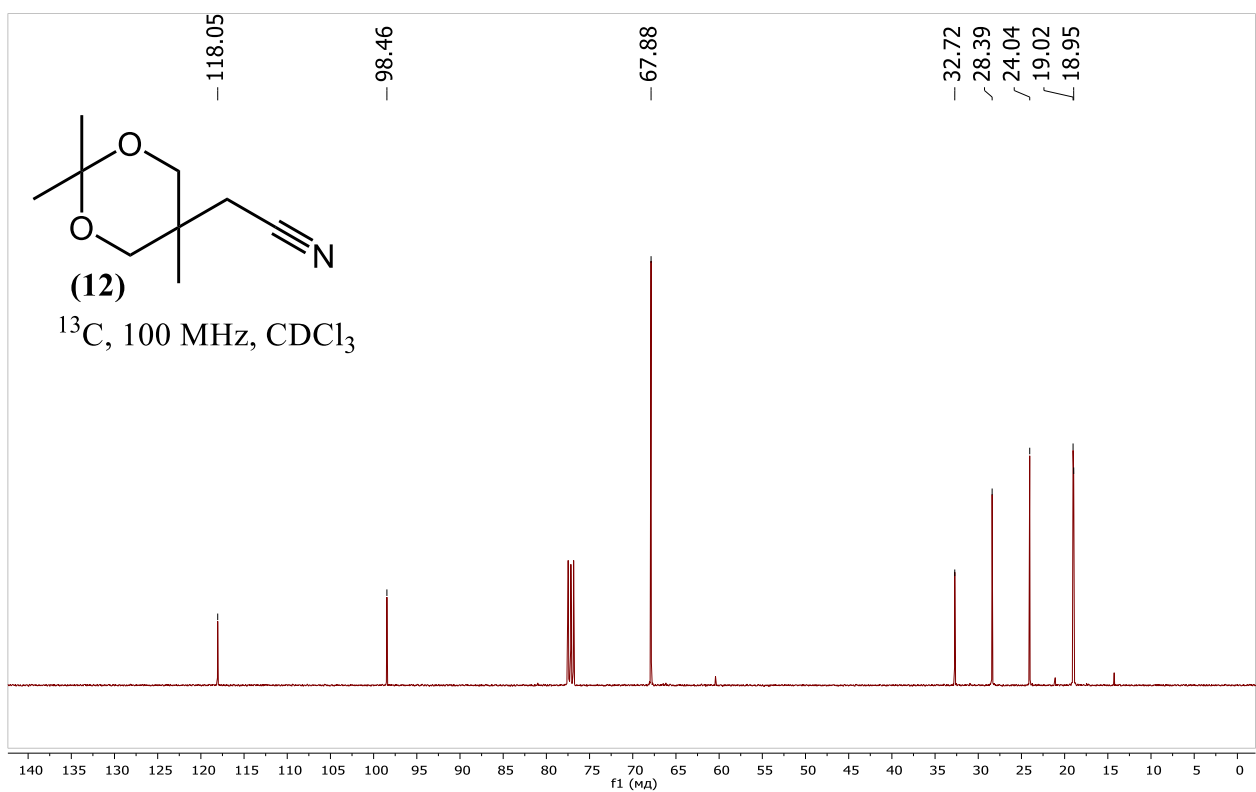
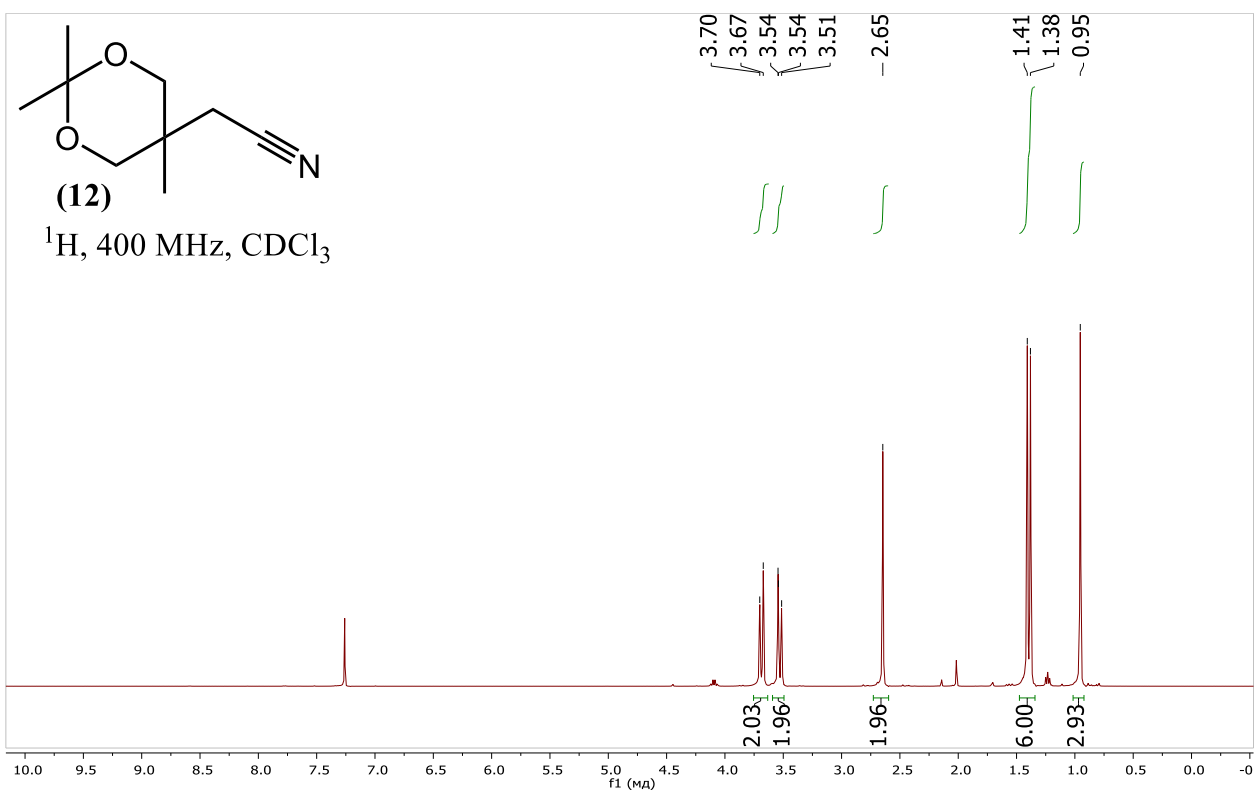


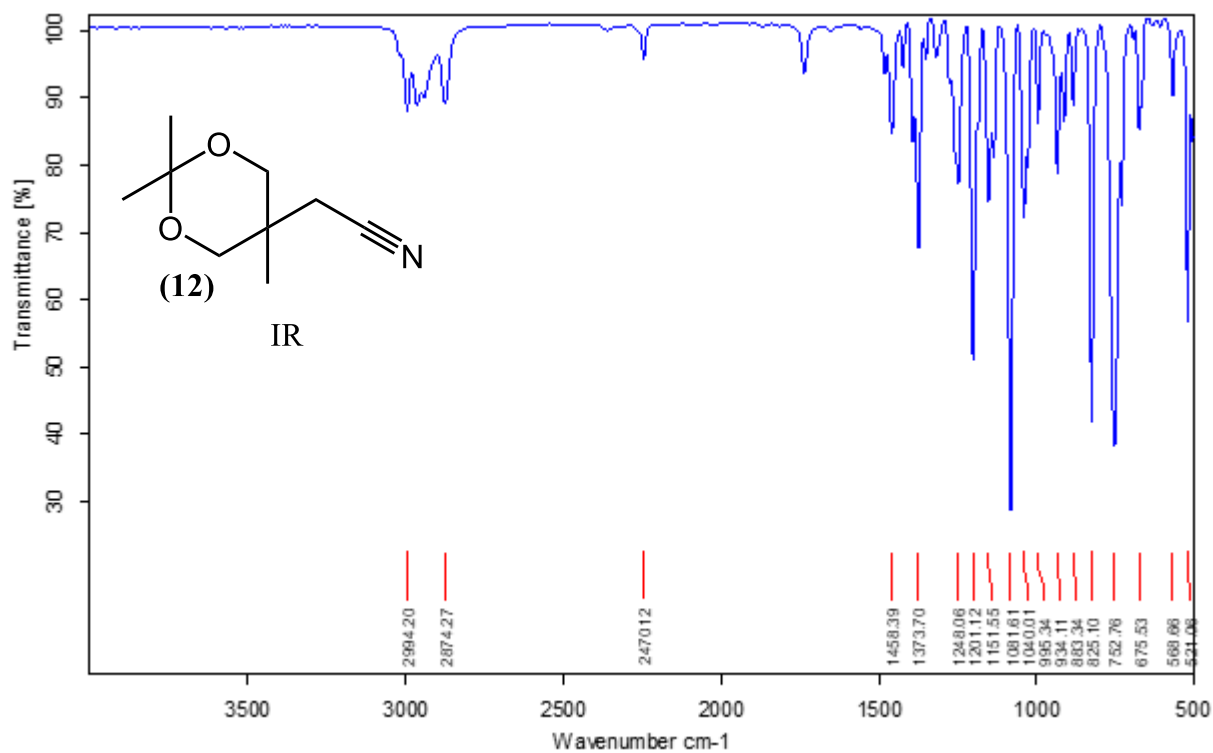


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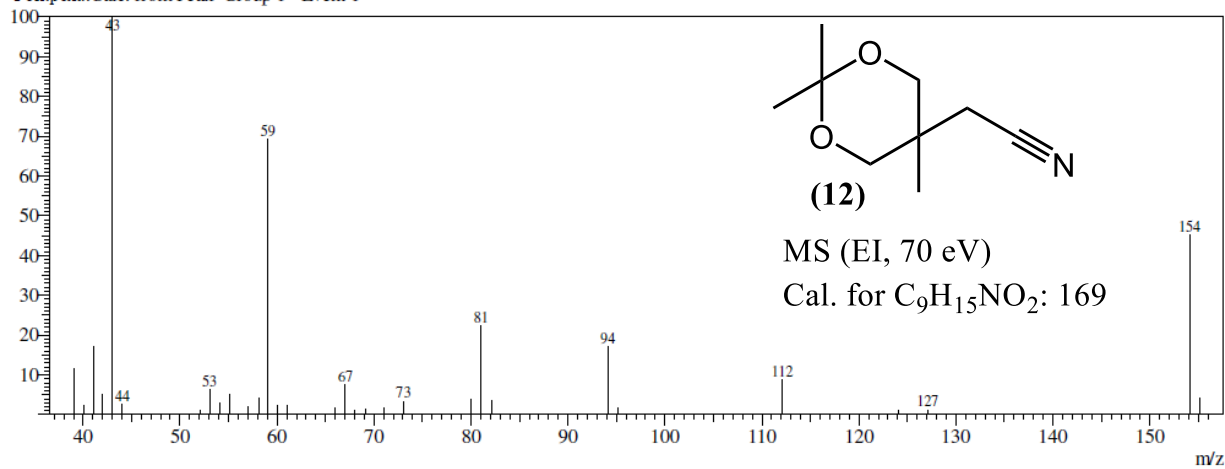
KC-1221

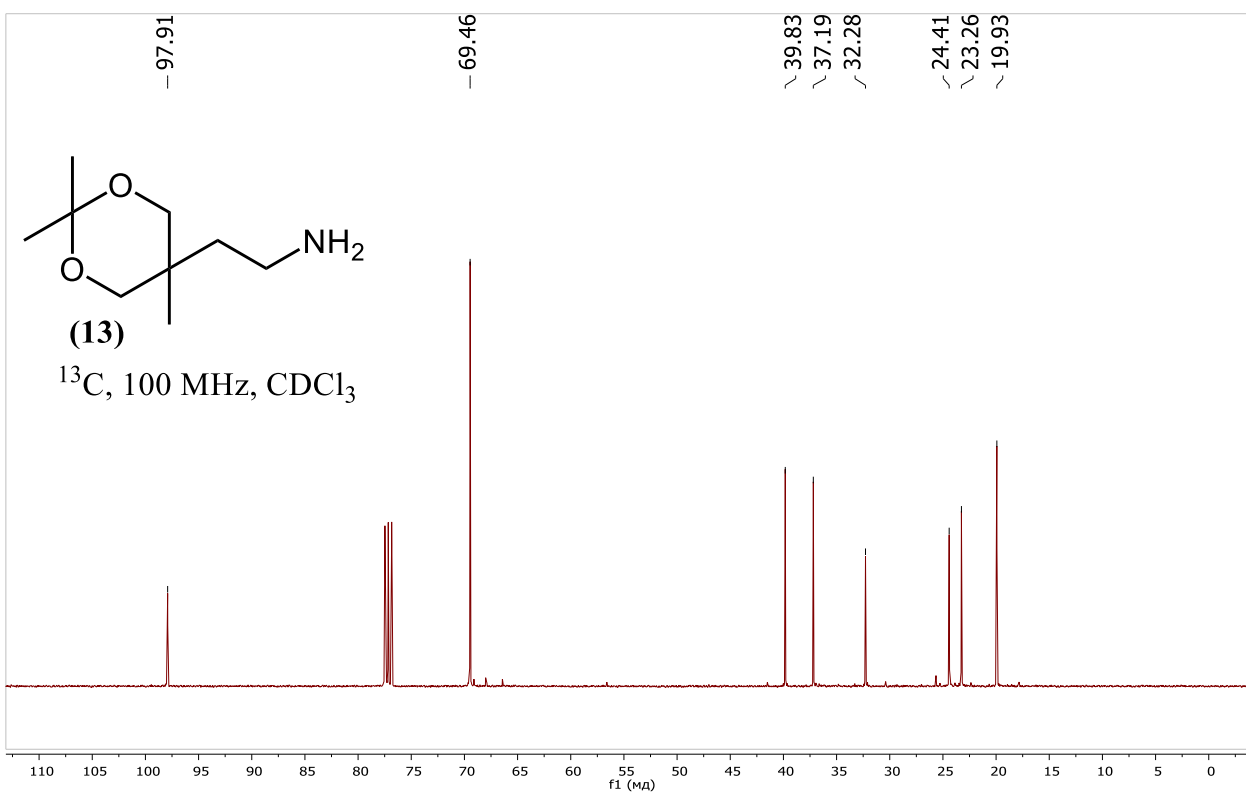
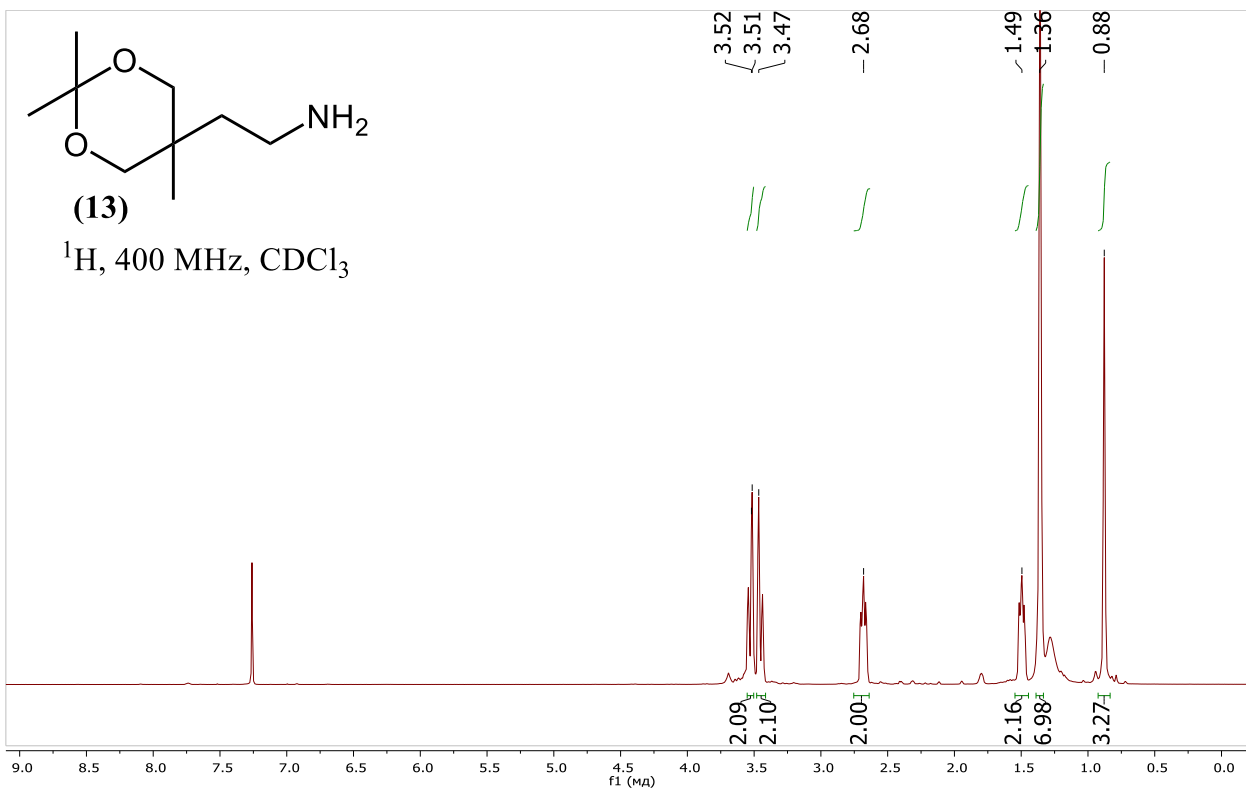
27/09/2021

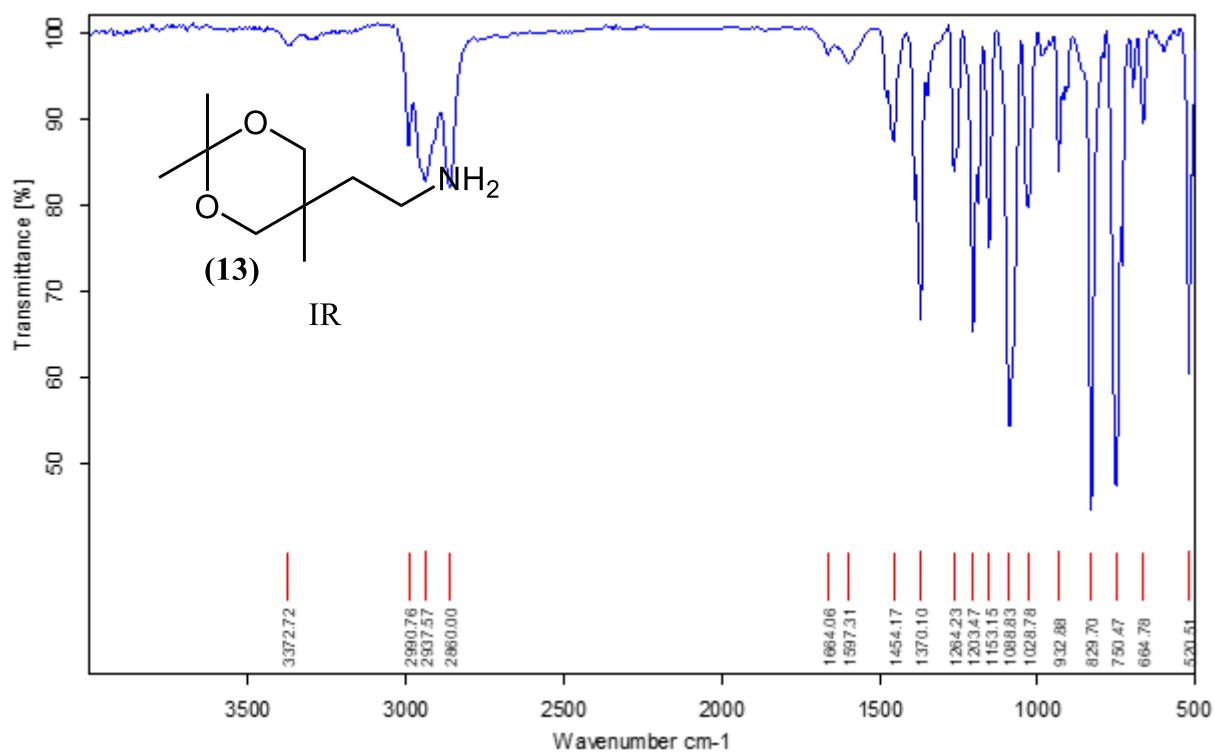




Line#:3 R.Time:5.742(Scan#:779)  
 MassPeaks:31  
 RawMode:Averaged 5.737-5.746(778-780) BasePeak:43(5737072)  
 Фон.реж.:Calc. from Peak Group 1 - Event 1







Line#:3 R.Time:5.892(Scan#:815)  
 MassPeaks:43  
 RawMode:Averaged 5.888-5.896(814-816) BasePeak:43(3140333)  
 Фон.реж.:Calc. from Peak Group 1 - Event 1

