

**A facile synthesis of ( $\pm$ )-15-deoxy- $\Delta^{12,14}$ -prostaglandin J<sub>2</sub> methyl ester**

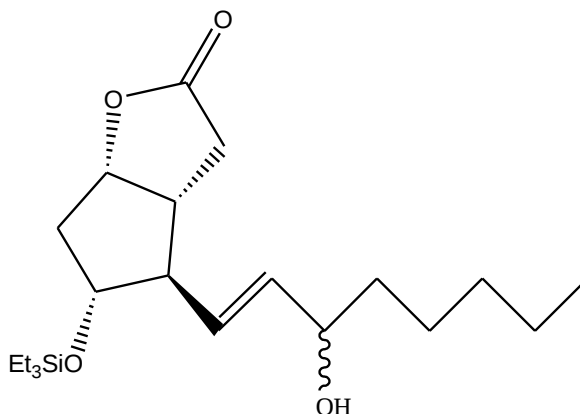
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and Mansur S. Miftakhov**

## **Experimental**

### **General**

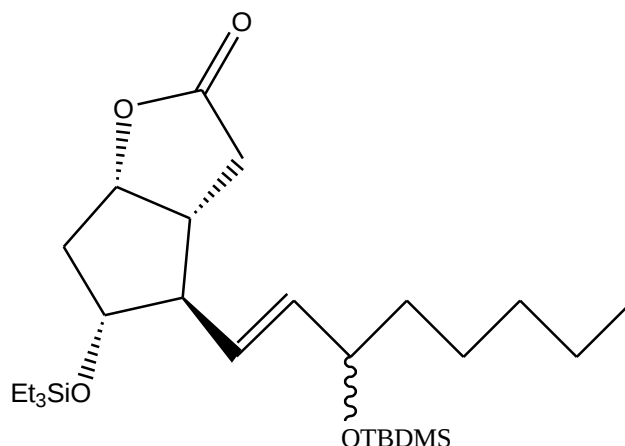
IR spectra were obtained on an IR Prestige-21 Shimadzu spectrophotometer in a film or in Nujol mulls. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on a Bruker AVANCE-500 spectrometer with operating frequencies of 500.13 and 125.77 MHz, respectively, using TMS as the internal standard. Mass spectra were obtained on an LCMS-2010EV mass spectrometer (Shimadzu) (syringe injection, a solution of the sample in chloroform/acetonitrile, flow rate 0.1 ml min<sup>-1</sup>, acetonitrile/water (95:5) as the eluent, positive and negative ion detection mode, at 4.5 and -3.5 kV potentials of a needle ionizing electrode, respectively; interface capillary temperature 250 °C, interface capillary voltage 5 V. Elemental analysis data were obtained on a Evro EA-3000 CHN analyzer. The reaction was monitored by TLC on “Sorbfil” plates (Krasnodar, Russia) with detection of compounds by wetting the plates with solutions of anisaldehyde and sulfuric acid in ethanol. The synthesized products were isolated by column chromatography on silica gel (30-60 g of the adsorbent per 1 g of a compound).

**(*E*,3a*R*\*,4*R*\*,5*R*\*,6a*S*\*)-4-(3-Hydroxyoct-1-en-1-yl)-5-(triethylsilyloxy)-hexahydro-2*H*-cyclopenta[*b*]furan-2-one (8)**



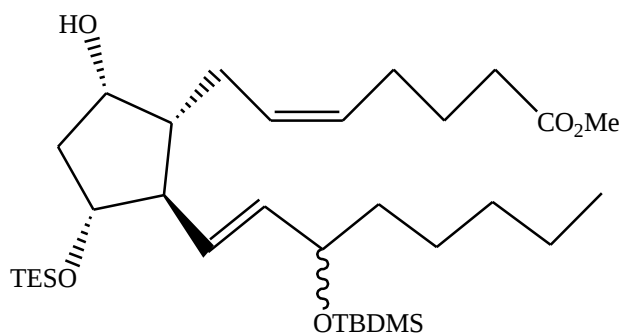
To a solution of compound **7** (1714 mg, 4.5 mmol) in MeOH at 0 °C was added CeCl<sub>3</sub>·7H<sub>2</sub>O (1676 mg, 4.5 mmol), then NaBH<sub>4</sub> (170 mg, 4.5 mmol) was gradually added, the progress of the reaction was monitored by TLC. At the end of the reaction, after standard treatment and purification on SiO<sub>2</sub>, 1533 mg of yellow oily compound **8** was obtained, yield 89%. *R*<sub>f</sub>=0.35 (hexane-ethyl acetate=7:3). IR,  $\nu$ , cm<sup>-1</sup>: 3440.2956, 2934, 1765, 1428, 1173, 1089, 1009.745. <sup>1</sup>H NMR (500 MHz) acetone-d<sub>6</sub> : (2:1 mixture of two C-3' epimers, according to the intensity of doublet signals of OH groups at 3.58 and 3.62 ppm) 5.55-5.65 (m, 2H, CH=CH ), 4.94 (m, 1H, *J*=6.9, 2.3 Hz, H-6a), 4.07 (m, 1H, H-5), 4.03 (m, 1H, H-3'), 3.63 (d, 0.5 H, *J*<sub>CH-OH</sub> =4.5 Hz, -OH), 3.58 (d, 1H, *J*<sub>CH-OH</sub> =4.7 Hz, -OH), 2.78 (dd, 2H, *J*=15.5, 7.5Hz, H-3a, epimer mixture) , 2.36 (dd, 2H, *J*=15.5, 7.3Hz, H-3<sub>b</sub>, epimer mixture), 1.80 m (1H, H-4), 1.53-1.23 (m, 11H), 0.95 (t, 9H, *J*=6.3 Hz, (CH<sub>3</sub>CH<sub>2</sub>)<sub>3</sub>Si-,major isomer), 0.87 (t, 3H, *J*=6.9Hz, (CH<sub>3</sub>CH<sub>2</sub>)<sub>3</sub>Si-,minor isomer), 0.60 and 0.68 (overlapping quartets, 6H, *J*=6.4Hz,((CH<sub>3</sub>CH<sub>2</sub>)<sub>3</sub>Si-)). <sup>13</sup>C NMR (125 MHz) acetone d<sub>6</sub>, (\*-minor isomer designation): 176.07 (C<sub>q</sub>, -CO<sub>2</sub>-, lactone), 136.42\* (C-2', CH=CH), 136.03 (C -2', CH=CH), 129.23\* (C-1', CH=CH), 128.83 (C-1', CH=CH), 82.61 (C-6<sub>a</sub>), 82.54\* (C-6<sub>a</sub>), 77.74 (C-5), 77.64\* (C-5), 71.56\* (C-3'), 71.38 (C-3'), 56.88\* (C-4), 56.75 (C-4), 42.12\* (C -3<sub>a</sub>), 42.04 (C-3<sub>a</sub>), 40.82 (C-6), 37.65 (C-4'), 25.09 (C-5'), 24.97\* (C-5'), 22.44 (C-7') , 13.43 (C-8'), 6.20 ((CH<sub>3</sub>CH<sub>2</sub>)<sub>3</sub>Si-), 4.46 ((CH<sub>3</sub>CH<sub>2</sub>)<sub>3</sub>Si. MS (APCI,%): *m/z* 497 [M+H+4H<sub>2</sub>O+MeCN]<sup>+</sup> (100%), 383 [M+H]<sup>+</sup> (85%), 365 [M+H-H<sub>2</sub>O]<sup>+</sup> (50%) .

**(*E*,3a*R*\*,4*R*\*,5*R*\*,6a*S*\*)-4-[3-(*tert*-Butyldimethylsilyloxy)oct-1-en-1-yl]-5-(triethylsilyloxy)hexahydro-2*H*-cyclopenta[*b*]furan-2-one (9).**



To a stirred solution of compound **8** (1560 mg, 4.08 mmol) in dry CH<sub>2</sub>Cl<sub>2</sub> was added imidazole (833 mg, 12.24 mmol) followed by TBSCl (738 mg, 4.9 mmol) in one portion. After completion of the reaction (TLC), the reaction mixture was washed with water, the organic layer was dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and evaporated. After purification on SiO<sub>2</sub>, 1627 mg of oily compound **9** was obtained, 86% yield. *R*<sub>f</sub>=0.6 (hexane-ethyl acetate=5:1). IR, ν, cm<sup>-1</sup>: 2968, 2930, 1779, 1462, 1249, 1090, 835. <sup>1</sup>H NMR (500 MHz) acetone-d<sub>6</sub>, δ: 5.65-5.53 (m, 2H, CH=CH), 4.94 (m, 1H, H-6a), 4.16 (m, 1H, H-5), 4.08 (m, 1H, H-3'), 2.45-2.32 (m, 3H, H-3, H-4), 1.82 (m, 1H, H-3a), 1.53-1.43 (m, 2H, H-6), 1.40-1.23 (m, 8H, H-4', H-5', H-6', H-7'), 0.96 (t, 9H, J=7.9 Hz, (CH<sub>3</sub>CH<sub>2</sub>)<sub>3</sub>Si-) 0.88 (s, 9H, -C(CH<sub>3</sub>)<sub>3</sub>), 0.60 (m, 6H, ((CH<sub>3</sub>CH<sub>2</sub>)<sub>3</sub>Si-) 0.08 (s, 3H, (CH<sub>3</sub>)<sub>2</sub>Si-), 0.05 (s, 3H, (CH<sub>3</sub>)<sub>2</sub>Si-). <sup>13</sup>C NMR (125 MHz) acetone-d<sub>6</sub>, (\*-minor isomer designation) δ: 176.03 (C<sub>q</sub>, -CO<sub>2</sub>-, lactone), 135.52 (C-2', CH=CH), 129.26 (C-1', CH=CH), 82.65 (C-6a), 82.38\* (C-6a), 77.77 (C-5), 77.46\* (C-5), 73.27\* (C-3'), 72.95 (C-3'), 56.77 (C-4), 56.55\* (C-4), 42.15 (C-3a), 41.95\* (C-3a), 40.97\* (C-6), 40.84 (C-6), 38.30 (C-4'), 38.19\* (C-4'), 34.22 (C-3), 34.10\* (C-3), 31.65 (C-6') 25.38 (CH<sub>3</sub>)<sub>3</sub>CSi-, 24.79 (C-5'), 24.62\* (C-5'), 22.40 (C-7'), 17.84 (C<sub>q</sub>, (CH<sub>3</sub>)<sub>3</sub>C-), 13.39 (C-8'), 6.21 ((CH<sub>3</sub>CH<sub>2</sub>)<sub>3</sub>Si-), 4.50 ((CH<sub>3</sub>CH<sub>2</sub>)<sub>3</sub>Si-), -4.83 ((CH<sub>3</sub>)<sub>2</sub>Si-), -5.44 ((CH<sub>3</sub>)<sub>2</sub>Si-). MS (APCI,%): *m/z* 497 [M+H]<sup>+</sup> (100%), 479 [M+H-H<sub>2</sub>O]<sup>+</sup> (40%), 365 [M+H-Et<sub>3</sub>SiOH]<sup>+</sup> (40%).

**Methyl (Z)-7-{{(1*R*\*,2*R*\*,3*R*\*,5*S*\*)-2-[(*E*)-3-(*tert*-butyldimethylsilyloxy)oct-1-en-1-yl]-5-hydroxy-3-(triethylsilyloxy)cyclopentyl}hept-5-enoate (11)**

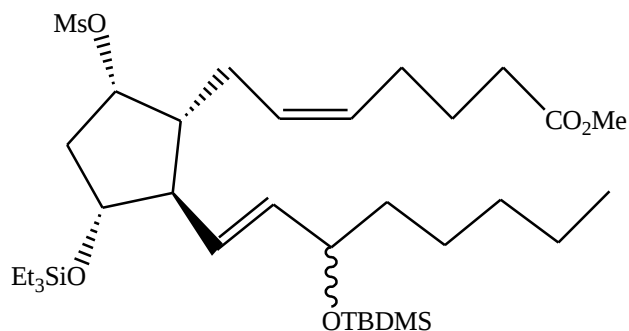


To a stirred solution of compound **9** (230 mg, 0.46 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (30 ml), DIBAL-H (0.23 ml, 1.15 mmol) was added dropwise in an argon flow at -78 °C, after 1 hour, an excess of DIBAL-H was neutralized with MeOH (1 ml), and the temperature was brought to ambient. Part of the solution was evaporated, the precipitate was filtered off on a Schott filter, the filtrate was dried over Na<sub>2</sub>SO<sub>4</sub> and evaporated to give 218 mg of crude lactol, which was used without further purification.

To a solution of phosphonium salt Br<sup>-</sup>[Ph<sub>3</sub>P<sup>+</sup>(CH<sub>2</sub>)<sub>4</sub>COOH] (212.5 mg, 0.48 mmol) in THF at -30 °C, was added NaHMDS (1 M THF solution, 1.44 ml, 1.44 mmol), after 30 min of stirring, a solution of the above lactol (218 mg) was added in one portion. After 6 hours of stirring, H<sub>2</sub>O (3 ml) was added. The solution was evaporated and extracted with Et<sub>2</sub>O. The aqueous layer was acidified to pH=4-5, extracted with CH<sub>2</sub>Cl<sub>2</sub>. The combined organic layers were dried over Na<sub>2</sub>SO<sub>4</sub>, filtered and evaporated, the resulting crude acid **10** was dissolved in MeOH-H<sub>2</sub>O (10:1) and treated with an excess of ethereal diazomethane to obtain 251 mg of yellow oily ester **11**, yield 70% (in 3 steps). R<sub>f</sub>=0.55 (hexane-ethyl acetate=5:1). IR, ν, cm<sup>-1</sup>: 3447, 3000, 2956, 2934, 1743, 1727, 1459, 1248, 1080, 836, 741. <sup>1</sup>H NMR (500 MHz) acetone d<sub>6</sub>, δ: 5.53-5.47 (m, 3H, H-5, H-6, H-2', CH=CH), 5.33 (m, 1H, H-1'', CH=CH), 4.18 (q, 1H, J=5.6 Hz, H-5'), 4.08 (m, 1H, H-3''), 4.00 (m, 1H, H-3'), 3.60 (s, 3H, -OMe), 3.32 (d, 1H, -OH, J<sub>CH-OH</sub>=6.2 Hz), 2.36-2.32 (m, 1H, H-1'), 2.28 (t, 2H, J=7.6 Hz, H-2), 2.27-2.22 (m, 3H, H-2', H<sub>a</sub>-4'), 2.17-2.07 (m, 4H, H-4, H-7), 1.64 (quintet, 2H, J=6.7 Hz, H-3), 1.62 (m, 2H, H-4''),

1.56-1.30 (m, 6H, H-5'', H-6'', H-7''), 0.95 (t, 9H, J=6.6 Hz, (CH<sub>3</sub>CH<sub>2</sub>)<sub>3</sub>Si-) 0.55 (s, 9H, -C(CH<sub>3</sub>)<sub>3</sub>), 0.88 (t, 3H, J=6.8 Hz, H-8''), 0.60 (q, 6H, J=6.8 Hz, (CH<sub>3</sub>CH<sub>2</sub>)<sub>3</sub>Si-) 0.08 (s, 3H, (CH<sub>3</sub>)<sub>2</sub>Si-), 0.06 (s, 3H, (CH<sub>3</sub>)<sub>2</sub>Si-). <sup>13</sup>C NMR (125 MHz) acetone d<sub>6</sub>, (\*-minor isomer designation) δ: 173.12 (C<sub>q</sub>, -CO<sub>2</sub>-), 135.43\* (C-2'', CH=CH), 135.01 (C-2'', CH=CH), 131.67\* (C-1'', CH=CH), 131.24 (C-1'', CH=CH), 129.67 (C-5, CH=CH), 129.62\* (C-5, CH=CH), 128.86\* (C-6, CH=CH), 128.79 (C-6, CH=CH), 78.24 (C-3'), 78.07\* (C-3'), 73.45\* (C-3''), 72.96 (C-3''), 71.32 (C-5'), 71.17\* (C-5'), 55.31\* (C-2'), 55.21 (C-2'), 50.61 (-OCH<sub>3</sub>), 49.93 (C-1'), 49.90\* (C-1'), 44.30\* (C-4'), 44.21 (C-4'), 38.61 (C-4''), 38.52\* (C-4''), 32.98 (C-2), 32.95\* (C-2), 31.77 (C-6''), 26.44\* (C-5''), 26.39 (C-5''), 25.42 (CH<sub>3</sub>)<sub>3</sub>CSi-, 25.33\* (C-4), 25.28 (C-4), 24.86\* (C-3), 24.81 (C-3), 22.48 (C-7''), 17.86 (C<sub>q</sub>, (CH<sub>3</sub>)<sub>3</sub>C-), 13.45 (C-8''), 6.21 ((CH<sub>3</sub>CH<sub>2</sub>)<sub>3</sub>Si), 4.59 ((CH<sub>3</sub>CH<sub>2</sub>)<sub>3</sub>Si-), -4.69 ((CH<sub>3</sub>)<sub>2</sub>Si-), -5.33 ((CH<sub>3</sub>)<sub>2</sub>Si-). MS (APCI,%): m/z 597 [M]<sup>+</sup> (100%), 465 [M-Et<sub>3</sub>SiOH]<sup>+</sup> (60%), 447 [M-Et<sub>3</sub>SiOH-TBSOH]<sup>+</sup> (50%).

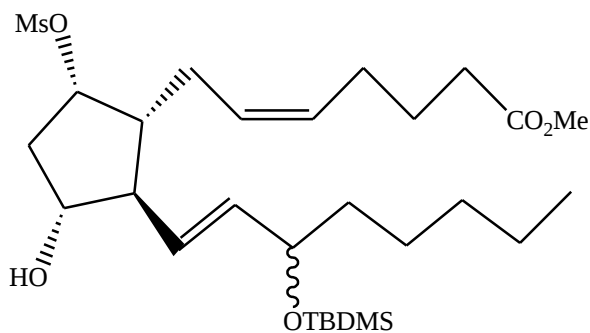
**Methyl (Z)-7-[(1*R*\*,2*R*\*,3*R*\*,5*S*\*)-2-[(*E*)-3-(*tert*-butyldimethylsilyloxy)oct-1-en-1-yl]-5-methylsulfonyloxy-3-(triethylsilyloxy)cyclopentyl]hept-5-enoate (12)**



To a solution of compound **11** (500 mg, 0.83 mmol) in absolute CH<sub>2</sub>Cl<sub>2</sub> (50 ml) at 0 °C was added Et<sub>3</sub>N (1.15 ml, 8.3 mmol), then MsCl (0.1 ml, 1.245 mmol) was gradually added, the progress of the reaction was monitored by TLC. Upon completion of the reaction, the reaction mass was washed with saturated aqueous NaHCO<sub>3</sub>, then with water (3x5 ml), the organic layer was dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and evaporated. Purification of the residue on SiO<sub>2</sub> gave 508 mg of oily compound **12**, 85% yield. *R*<sub>f</sub>=0.45 (hexane-ethyl acetate=5:1). IR,  $\nu$ , cm<sup>-1</sup>: 2954, 2930, 1739, 1360, 1249, 1174, 838, 774. <sup>1</sup>H NMR (500 MHz) acetone d<sub>6</sub>,  $\delta$ : 5.60 (m, 1H, CH=CH), 5.48-5.54 (m, 2H, CH=CH), 5.38 (m, 1H, CH=CH), 5.03 (m, 1H, H-5'), 4.22 (q, 1H, *J*=5.6 Hz, H-3'), 4.12-4.01 (m, 1H, H-3''), 3.61 (s, 3H, -OMe), 3.08 (s, 3H, -SO<sub>2</sub>Me), 2.54 (m, 1H, H-1'), 2.33 (m, 2H, H-2'), 2.30 (t, 2H, *J*=7.3 Hz, H-2), 2.25-2.12 (m, 2H, H-4), 2.10 (m, 2H, H-7), 2.01 (m, 1H, H<sub>a</sub>-4'), 1.78 (m, 1H, H<sub>b</sub>-4'), 1.64 (quintet, 2H, *J*=7.6 Hz, H-3), 1.51 (quintet, 2H, *J*=6.2 Hz, H-5''), 1.45-1.20 (m, 6H, H-4'', H-6'', H-7''), 0.96 (t, 9H, *J*=6.8 Hz, (CH<sub>3</sub>CH<sub>2</sub>)<sub>3</sub>Si-), 0.92 (s, 9H, -C(CH<sub>3</sub>)<sub>3</sub>), 0.88 (t, 3H, *J*=7.8 Hz, H-8''), 0.60 (q, 6H, *J*=6.8 Hz, (CH<sub>3</sub>CH<sub>2</sub>)<sub>3</sub>Si-), 0.07 (s, 3H, (CH<sub>3</sub>)<sub>2</sub>Si-), 0.06 (s, 3H, (CH<sub>3</sub>)<sub>2</sub>Si-). <sup>13</sup>C NMR (125 MHz) acetone d<sub>6</sub>, (\*-minor isomer designation)  $\delta$ : 173.05 (C<sub>q</sub>, -CO<sub>2</sub>-), 136.55\* (C-2''), 136.46 (C-2''), 130.18\* (C-1''), 129.97 (C-1''), 129.87\* (C-5), 129.74\* (C-5), 128.16 (C-6), 128.12\* (C-6), 82.54 (C-5'), 76.82 (C-3'), 76.96\* (C-3'), 73.14\* (C-3''), 72.61 (C-3''), 55.65\* (C-2'), 55.49 (C-2'), 50.62 (-OCH<sub>3</sub>), 48.29 (C-1'), 48.19\* (C-1'), 42.18\* (C-4'), 42.12 (C-4'), 38.53\* (C-4''), 38.42 (C-4''), 37.88 (CH<sub>3</sub>SO<sub>2</sub>-), 32.95 (C-2), 32.91\* (C-2), 26.58\* (C-4), 26.53 (C-4), 25.40

( $\text{CH}_3$ )<sub>3</sub>CSi-), 24.81\* (C-7), 24.75 (C-7), 22.45 (C-17''), 17.85 (C<sub>q</sub>, ( $\text{CH}_3$ )<sub>3</sub>C-), 17.67\* (C<sub>q</sub>, ( $\text{CH}_3$ )<sub>3</sub>C-), 13.44(C-8''), 6.40 (( $\text{CH}_3\text{CH}_2$ )<sub>3</sub>Si-), 6.31\* (( $\text{CH}_3\text{CH}_2$ )<sub>3</sub>Si-), 4.60 (( $\text{CH}_3\text{CH}_2$ )<sub>3</sub>Si-), -5.19 (( $\text{CH}_3$ )<sub>2</sub>Si-), -5.32\* (( $\text{CH}_3$ )<sub>2</sub>Si-), -5.38\* (( $\text{CH}_3$ )<sub>2</sub>Si-), -5.41 (( $\text{CH}_3$ )<sub>2</sub>Si-). MS (APCI,%):m/z 447 [M-Et<sub>3</sub>SiOH-MsOH]<sup>+</sup> (35%), 333 [M-Et<sub>3</sub>SiOH-MsOH-TBSOH+H<sub>2</sub>O]<sup>+</sup> (100%), 315 [M-Et<sub>3</sub>SiOH-MsOH-TBSOH]<sup>+</sup> (100%).

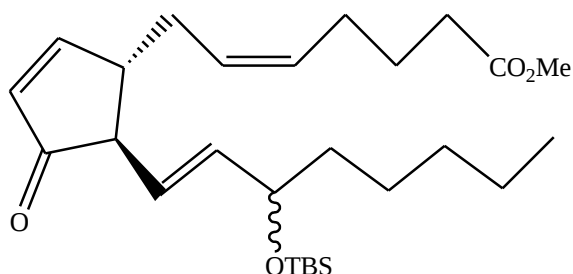
**Methyl (Z)-7-((1*R*\*,2*R*\*,3*R*\*,5*S*\*)-2-((*E*)-3-(*tert*-butyldimethylsilyloxy)oct-1-en-1-yl)-3-hydroxy-5-(methylsulfonyloxy)cyclopentyl)hept-5-enoate (5)**



To a stirred solution of compound **12** (150 mg, 0.22 mmol) in THF-H<sub>2</sub>O (5:1) system was added a saturated citric acid solution (4 ml), the progress of the reaction was monitored by TLC. At the end of the reaction, THF was evaporated, the aqueous layer was extracted with CH<sub>2</sub>Cl<sub>2</sub>, the combined organic layers were dried over Na<sub>2</sub>SO<sub>4</sub>, filtered and evaporated. Purification of the residue on a SiO<sub>2</sub> column gave 106 mg of compound **5**, 85% yield. R<sub>f</sub>=0.30 (hexane-ethyl acetate=7:3). IR, ν, cm<sup>-1</sup>: 3471, 3007, 2953, 2929, 1732, 1462, 1359, 1249, 1170, 910. <sup>1</sup>H NMR (500 MHz) acetone d<sub>6</sub>, δ: 5.62-5.48 (m, 3H, CH=CH), 5.43-5.34 (m, 1H, H-6, CH=CH), 5.03 (td, 1H, J=5.8, 1.5 Hz, H-5'), 4.18 (q, 1H, J=6.0 Hz, H-3'), 4.0-3.9 (m, 1H, H-3''), 3.60 (s, 3H, -OMe), 3.08 (s, 3H, -SO<sub>2</sub>Me), 2.52 (ddd, 1H, J=15.5, 9.2, 2.7 Hz, H<sub>a</sub>-4'), 2.34 (m, 2H, H-7), 2.28 (t, 2H, J=7.3 Hz, H-2), 2.26-2.06 (m, 2H, H-1', H-2''), 2.03-1.97 (m, 2H, H-4), 1.80 (m, 1H, H<sub>b</sub>-4'), 1.65 (quintet, 2H, J=7.6 Hz, H-3), 1.50 (m, 2H, H-4''), 1.40-1.25 (m, 6H, H-5'', H-6'',

H-7''), 0.88 s (9H, -C(CH<sub>3</sub>)<sub>3</sub>), 0.86 (t, 3H, J=7.0 Hz, H-20), 0.08 (s, 3H, (CH<sub>3</sub>)<sub>2</sub>Si-), 0.06 (s, 3H, (CH<sub>3</sub>)<sub>2</sub>Si-). <sup>13</sup>C NMR (125 MHz) acetone-d<sub>6</sub>, (\*-minor isomer designation) δ: 172.35 (C<sub>q</sub>, -CO<sub>2</sub>-), 135.68\* (C-2''), 135.48 (C-2''), 129.84 (C-1''), 129.67\* (C-1''), 129.14\* (C-5), 129.06 (C-5), 128.25 (C-6), 128.21\* (C-6), 82.13 (C-5'), 81.99\* (C-5'), 75.13 (C-3'), 75.09\* (C-3'), 72.58 (C-3''), 54.43\* (C-2'), 54.39 (C-2'), 49.91 (-OCH<sub>3</sub>), 47.80\* (C-1'), 47.71 (C-1'), 40.95 (C-4'), 40.87\* (C-4'), 37.71 (C-4''), 37.65 (C-4''), 37.17 (CH<sub>3</sub>SO<sub>2</sub>-), 32.24\* (C-2), 32.20 (C-2), 30.96 (C-6''), 25.93\* (C-4), 25.78 (C-4), 24.86 (C-5''), 24.73 (CH<sub>3</sub>)<sub>3</sub>CSi-, 24.21 (C-7), 24.18\* (C-7), 24.64 (C-3), 24.66\* (C-3), 21.73 (C-7''), 17.16 (C<sub>q</sub>, (CH<sub>3</sub>)<sub>3</sub>C-), 12.71 (C-8''), -5.37 ((CH<sub>3</sub>)<sub>2</sub>Si-), -5.41\* ((CH<sub>3</sub>)<sub>2</sub>Si-), -6.10\* ((CH<sub>3</sub>)<sub>2</sub>Si-), -6.13 ((CH<sub>3</sub>)<sub>2</sub>Si-). MS (APCI,%):m/z 333 [M+H-TBSOH-MsOH]<sup>+</sup> (85%), 315 [M+H-TBSOH-MsOH-H<sub>2</sub>O]<sup>+</sup> (100%).

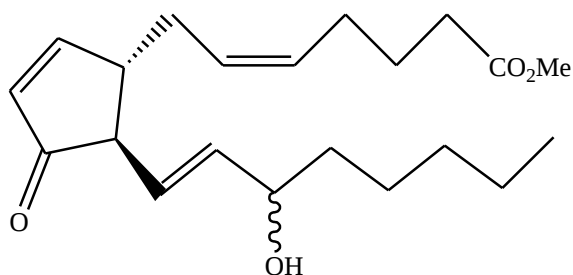
**Methyl (Z)-7-((1*S*\*,5*R*\*)-5-[(*E*)-3-(*tert*-butyldimethylsilyloxy)oct-1-en-1-yl]-4-oxocyclopent-2-en-1-yl)hept-5-enoate (13).**



To a solution of compound **5** (420 mg, 0.75 mmol) in absolute CH<sub>2</sub>Cl<sub>2</sub> at 0 °C was added diacetoxy(phenyl)iodinane (375.5 mg, 1.16 mmol). After 5 min of stirring, TEMPO (11.76 mg, 0.075 mmol) was added. After 1 hour, the temperature was raised to ambient, the reaction mass was evaporated and chromatographed on SiO<sub>2</sub> to afford 319 mg of compound **13**, 92% yield. R<sub>f</sub>=0.60 (hexane-ethyl acetate=5:1). IR, ν, cm<sup>-1</sup>: 3007, 2953, 2927, 1735, 1718, 1654, 1589, 1458, 1251, 837. <sup>1</sup>H NMR (500 MHz) acetone-d<sub>6</sub>, δ: 7.72 (dd, 1H, J=5.7, 2.6, Hz, H-2'), 6.05 (dd, 1H, J=5.6,

2.6 Hz, H-3`), 5.60 (overlapping dd, 1H, J=14.0, 7.0 Hz, H-2``), 5.58 (overlapping dd, 1H, J=14.7, 7.0 Hz, H-1``), 5.53-5.46 (m, 2H, H-5, H-6, CH=CH), 4.16 (m, 1H, H-3``), 3.60 (s, 3H, -OMe), 2.84 (m, 1H, H-2`), 2.63 (td, 1H, J=6.7, 3.1 Hz, H-1`), 2.42 (m, 1H, H<sub>a</sub>-7), 2.34 (m, 1H, H<sub>b</sub>-7), 2.32 (t, 2H, J=7.4 Hz, H-2), 2.11 (q, 2H, J=7.0 Hz, H-4), 1.66 (quintet, 2H, J=7.5 Hz, H-3), 1.60-1.20 (m, 8H, H-4``, H-5``, H-6``, H-7``), 0.90 s (9H, -C(CH<sub>3</sub>)<sub>3</sub>), 0.88 (t, 3H, J=7.5 Hz, H-8``), 0.08 (s, 6H, (CH<sub>3</sub>)<sub>2</sub>Si-). <sup>13</sup>C NMR (125 MHz) acetone d<sub>6</sub>, (\*-minor isomer designation) δ: 207.06 (C<sub>q</sub>, CO), 173.03 (C<sub>q</sub>, -CO<sub>2</sub>-), 165.85\* (C-5`), 165.78 (C-5`), 136.97\* (C-2``), 136.61 (C-2``), 132.36\* (C-4`), 132.32 (C-4`), 131.14\* (C-1``), 131.10 (C-1``), 126.94 (C-5), 126.90\* (C-5), 126.30\* (C-6), 126.21 (C-6), 73.38\* (C-3``), 73.19 (C-3``), 54.24\* (C-2`), 54.13 (C-2`), 50.63 (-OCH<sub>3</sub>), 47.99\* (C-1`), 47.89 (C-1`), 38.26\* (C-4``), 38.21 (C-4``), 32.81 (C-2), 31.62 (C-7), 30.69 (C-6``), 26.33 (C-4), 25.41 ((CH<sub>3</sub>)<sub>3</sub>C-), 24.72 (C-3), 24.66 (C-17), 22.44 (C-7``), 17.86 (C<sub>q</sub>, (CH<sub>3</sub>)<sub>3</sub>C-), 13.40 (C-8``), -4.74 ((CH<sub>3</sub>)<sub>2</sub>Si-), -5.51 ((CH<sub>3</sub>)<sub>2</sub>Si-). MS(APCI,%):m/z 461 [M-H]<sup>+</sup> (50%), 331 [M+H-TBSOH]<sup>+</sup> (100%).

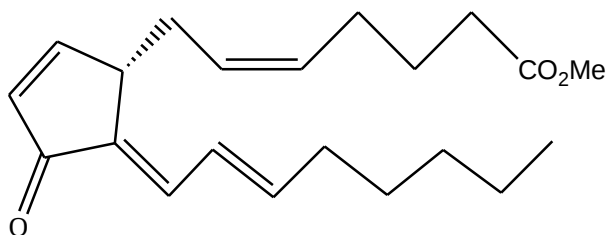
**Methyl (Z)-7-[(1S\*,5R\*)-5-((E)-3-hydroxyoct-1-en-1-yl)-4-oxocyclopent-2-en-1-yl]hept-5-enoate (14).**



To a solution of compound **13** (230 mg, 0.5 mmol) in MeCN at 0 °C was added 40% aqueous HF (0.01 ml, 0.25 mmol). The progress of the reaction was monitored by TLC. After 3 hours, the temperature of the reaction mass was raised to ambient, the mixture was treated with NaHCO<sub>3</sub> (1 ml), extracted with CH<sub>2</sub>Cl<sub>2</sub>, the combined organic layers were dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and evaporated. Purification on SiO<sub>2</sub> gave 138.8 mg of oily compound **14**, 80% yield. R<sub>f</sub>=0.40

(hexane-ethyl acetate=7:3). IR,  $\nu$ ,  $\text{cm}^{-1}$ : 3442, 3007, 2953, 2927, 1737, 1712, 1587, 1458, 1168, 968.  $^1\text{H}$  NMR (500 MHz) acetone  $d_6$ ,  $\delta$ : 7.70 (dd, 1H,  $J=1.8, 5.7$ , Hz, H-2'), 6.10 (dd, 1H,  $J=1.9, 5.8$  Hz, H-3'), 5.64 (dd, 1H,  $J=15.4, 6.1$  Hz, H-2''), 5.54 (dd, 1H,  $J=15.9, 4.0$  Hz, H-1''), 5.47-5.52 (m, 2H, H-5, H-6, CH=CH), 4.03 (m, 1H, H-3''), 3.70 и 3.65 (m, 1H, H-5' epimers), 3.60 (s, 3H, -OMe), 2.88 (m, 1H, H-1'), 2.61 (m, 1H, H-1'), 2.44-2.36 (m, 2H, H-7), 2.32 (t, 2H,  $J=7.4$  Hz, H-2), 2.12 (q, 2H,  $J=6.9$  Hz, H-4), 1.65 (quintet, 2H,  $J=7.5$  Hz, H-3), 1.33-1.60 (m, 8H, H-4'', H-5'', H-6'', H-7''), 0.88 (t, 3H,  $J=6.5$  Hz, H-8'').  $^{13}\text{C}$  NMR (125 MHz) acetone  $d_6$ ,  $\delta$ : 206.46 ( $\text{C}_q$ , CO), 173.02 ( $\text{C}_q$ ,  $-\text{CO}_2-$ ), 165.89 (C-5'), 137.40 (C-2''), 132.33 (C-3''), 131.12 (C-1''), 126.87 (C-6), 125.75 (C-5), 71.56 (C-3''), 54.70 ( $-\text{OCH}_3$ ), 54.23 (C-2'), 48.05 (C-1'), 37.53 (C-4''), 32.81 (C-2), 31.73 (C-7), 30.59 (C-6''), 26.32 (C-4), 24.99 (C-3), 24.65 (C-5''), 22.46 (C-7''), 13.43 (C-8''). MS (APCI,  $m/z$ , %):  $m/z$  331  $[\text{M}+\text{H}-\text{H}_2\text{O}]^+$  (100%), 347  $[\text{M}-\text{H}]^+$  (40%), 366  $[\text{M}+\text{H}_2\text{O}]^+$  (40%)

**Methyl ( $\pm$ )-(5*Z*,12*E*,14*E*)-11-oxoprostano-5,12,14-trien-1-oate (2).**  
**(15-deoxy- $\Delta^{12,14}$ -prostaglandin  $\text{J}_2$ )**

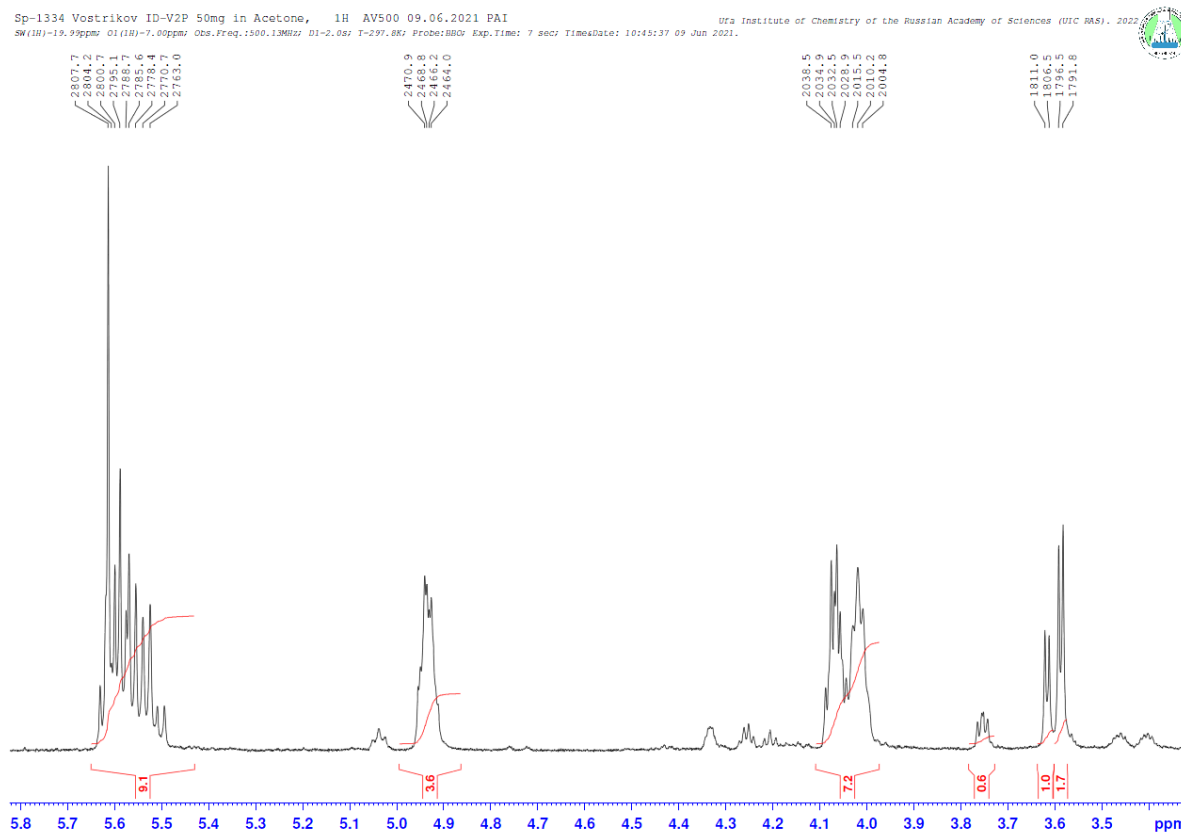
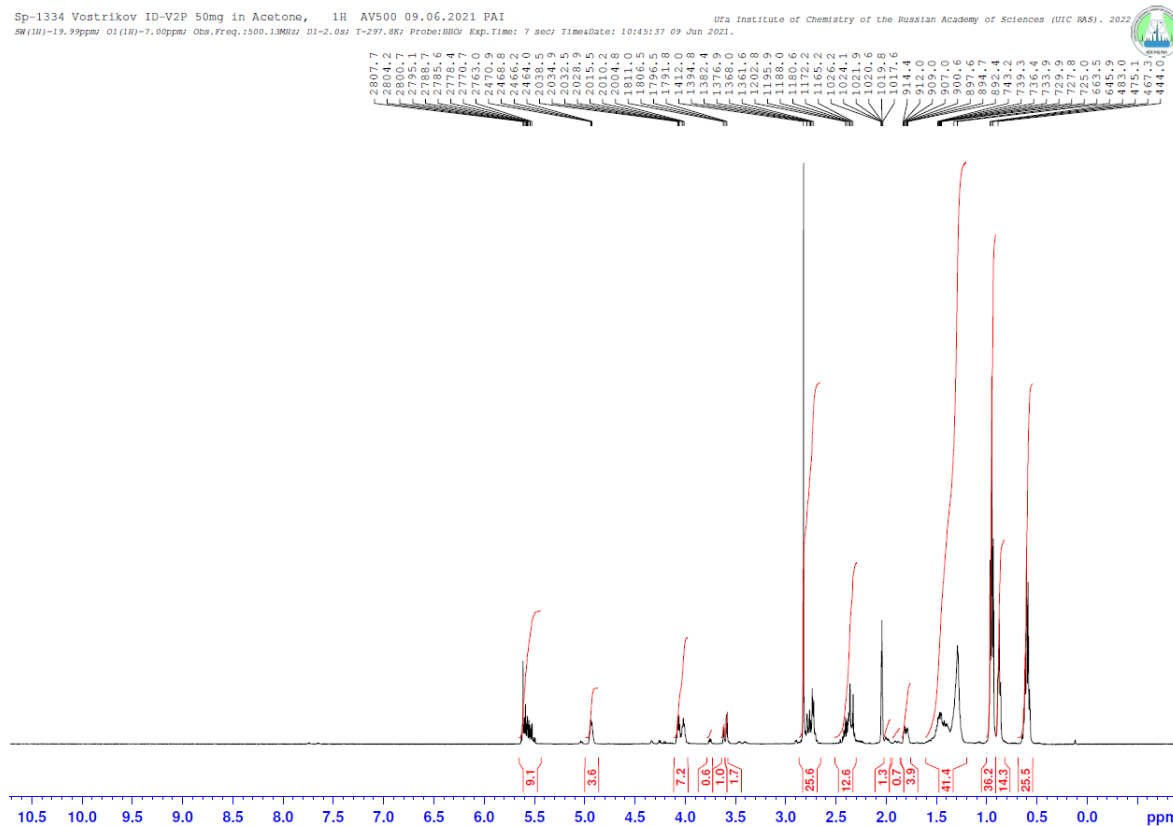


To a stirred solution of compound **14** (115 mg, 0.33 mmol) in  $\text{CH}_2\text{Cl}_2$  (10 ml) was added  $\text{TsOH} \cdot \text{H}_2\text{O}$  (63.5 mg, 0.33 mmol). After completion of the reaction (TLC), the organic layer was washed with water and dried over  $\text{Na}_2\text{SO}_4$ , filtered, and evaporated. Purification of the residue on  $\text{SiO}_2$  gave 91.6 mg of compound **2**, 84% yield.  $R_f=0.30$  (hexane-ethyl acetate=5:1). IR,  $\nu$ ,  $\text{cm}^{-1}$ : 2955, 2926, 2858,

1739, 1692, 1631, 1437, 1362, 1349, 1151, 1035, 978, 809.  $^1\text{H}$  NMR (500 MHz) acetone  $d_6$ ,  $\delta$ : 7.61 (ddd, 1H,  $J=0.8, 2.7, 6.8$  Hz, H-2'), 6.84 (d, 1H,  $J=11.7$  Hz, H-1''), 6.51 (ddt, 1H,  $J=15.0, 11.7, 1.3$  Hz, H-2''), 6.29 (dt, 1H,  $J=7.1, 14.5$  Hz, H-3''), 6.28 (dd, 1H,  $J=1.8, 5.9$  Hz, H-3'), 5.45 (dt, 1H,  $J=10.7, 7.2$  Hz, H-5), 5.37 (dt, 1H,  $J=10.9, 6.8$  Hz, H-6), 3.70 (m, 1H, H-1'), 3.62 (s, 3H, -OMe), 2.64 (dt, 1H,  $J=14.4, 4.7$  Hz, H<sub>a</sub>-7), 2.39 (dt, 1H,  $J=8.1, 15.5$  Hz, H<sub>b</sub>-7), 2.27 (t, 2H,  $J=7.4$  Hz, H-2), 2.25 (m, 2H, H-4''), 2.05 (m, 2H, H-4), 1.61 (quintet, 2H,  $J=7.5$  Hz, H-3), 1.48 (quintet, 2H,  $J=7.4$  Hz, H-5''), 1.35-1.30 (m, 4H, H-6'', H-7''), 0.89 (t, 3H,  $J=7.5$  Hz, H-8'').  $^{13}\text{C}$  NMR (125 MHz) acetone  $d_6$ ,  $\delta$ : 195.87 (C<sub>q</sub>, CO), 173.07 (C<sub>q</sub>, -CO<sub>2</sub>), 160.86 (C-5'), 145.75 (C<sub>q</sub>, C-2'), 135.60 (C-3''), 134.71 (C-1''), 131.26 (C-4'), 130.31 (C-5), 126.06 (C-6), 125.85 (C-2''), 50.60 (-OCH<sub>3</sub>), 43.21 (C-1'), 32.99 (C-7), 32.84 (C-2), 31.23 (C-4''), 30.33 (C-6''), 27.76 (C-5''), 26.39 (C-4), 24.66 (C-3), 22.22 (C-7''), 13.36 (C-8''). MS (APCI,  $m/z$ , %):  $m/z$  331 [M+H]<sup>+</sup> (100%)

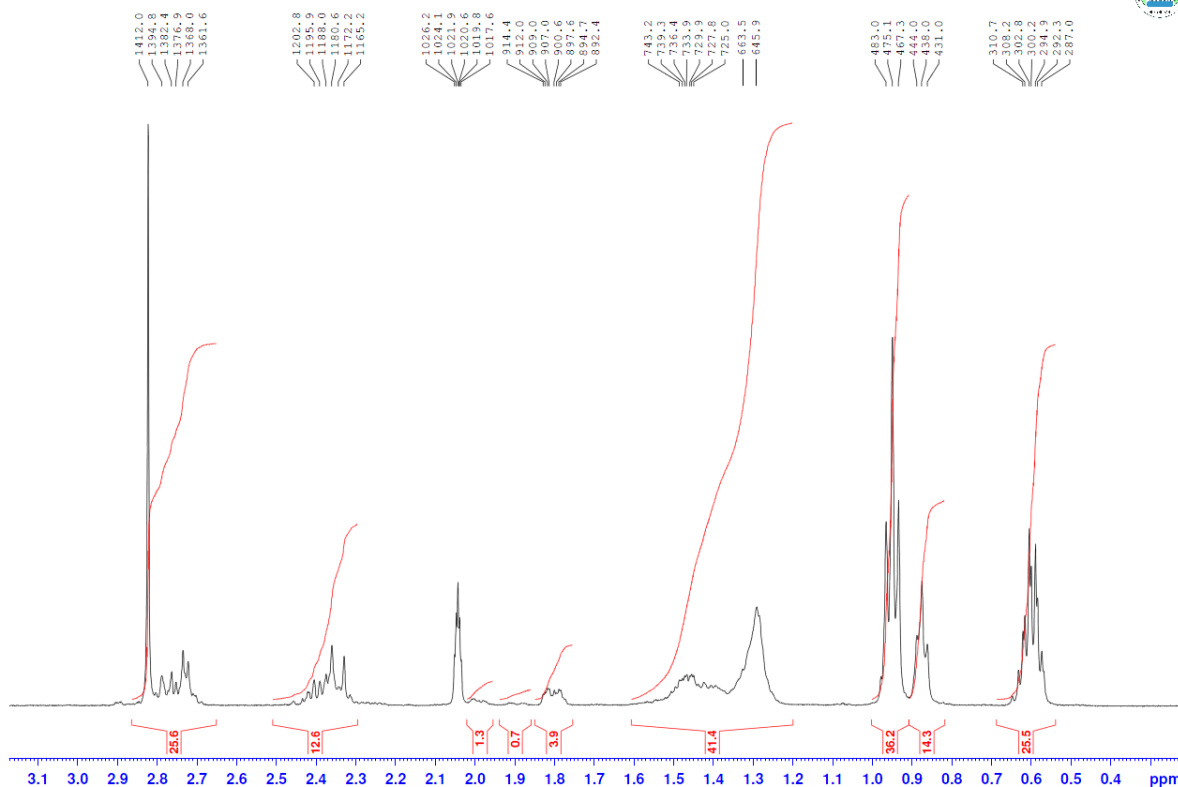
**(3a*R*\*,4*R*\*,5*R*\*,6a*S*\*)-4-((*E*)-3-hydroxyoct-1-en-1-yl)-  
5-(triethylsilyloxy)hexahydro-2*H*-cyclopenta[*b*]furan-2-one (8).**

<sup>1</sup>H NMR



Sp-1334 Vostrikov ID-V2P 50mg in Acetone, 1H AV500 09.06.2021 PAI

Ufa Institute of Chemistry of the Russian Academy of Sciences (IIC RAS), 2022



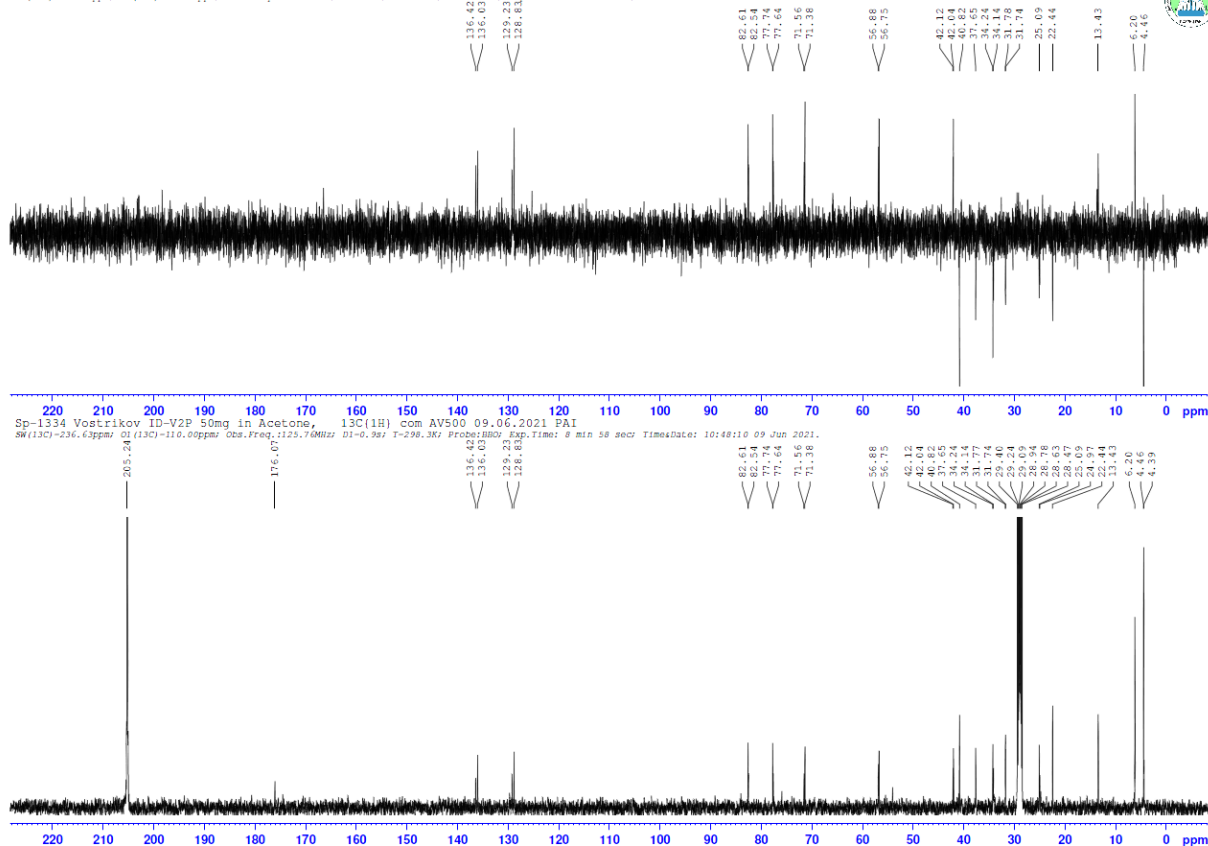
## $^{13}\text{C}$ NMR

Sp-1334 Vostrikov ID-V2P 50mg in Acetone,  $^{13}\text{C}(1\text{H})$  dept135 AV500 09.06.2021 PAI

Ufa Institute of Chemistry of the Russian Academy of Sciences (IIC RAS), 2022



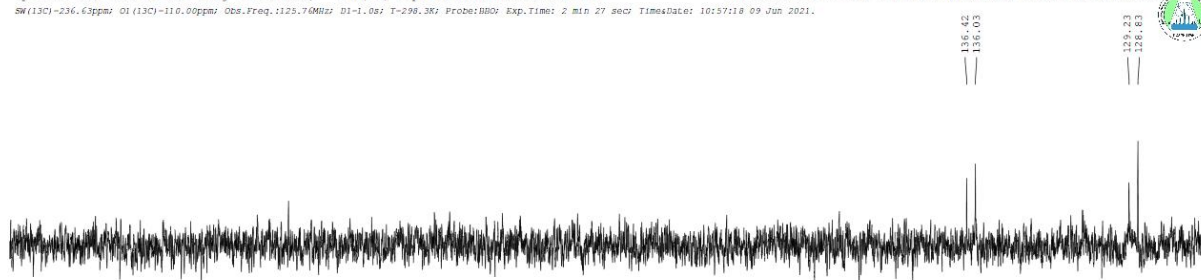
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Sp-1334 Vostrikov ID-V2P 50mg in Acetone, 13C(1H) dept135 AV500 09.06.2021 PAI

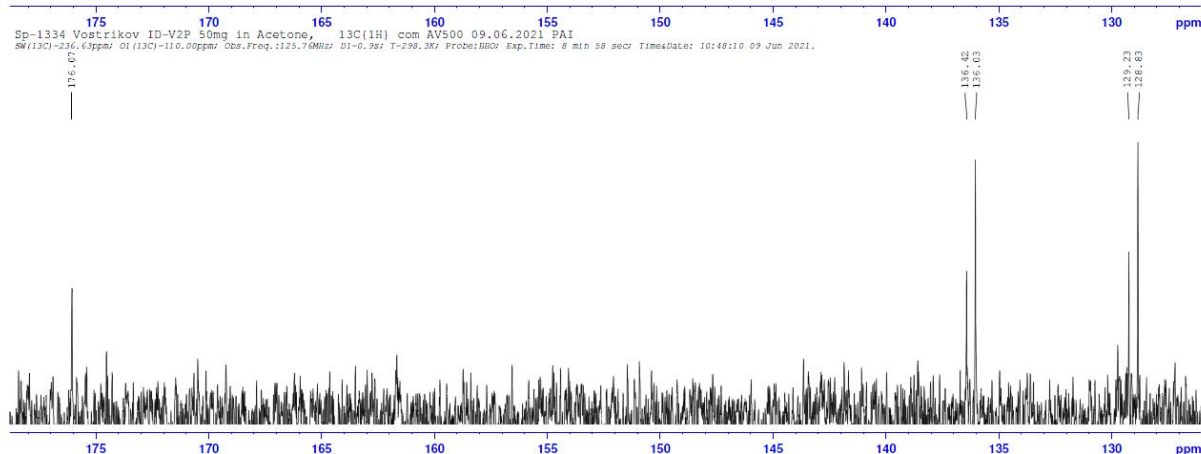
Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS), 2022

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Sp-1334 Vostrikov ID-V2P 50mg in Acetone, 13C(1H) com AV500 09.06.2021 PAI

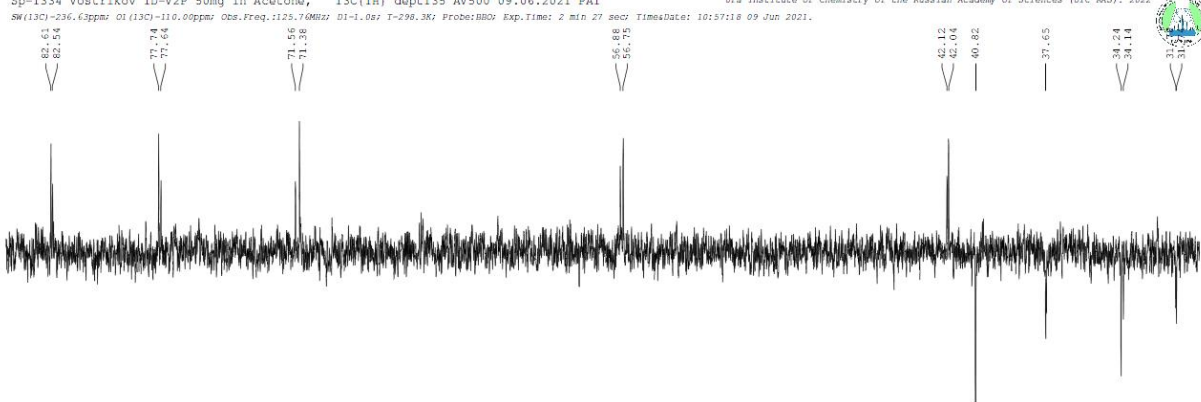
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Sp-1334 Vostrikov ID-V2P 50mg in Acetone, 13C(1H) dept135 AV500 09.06.2021 PAI

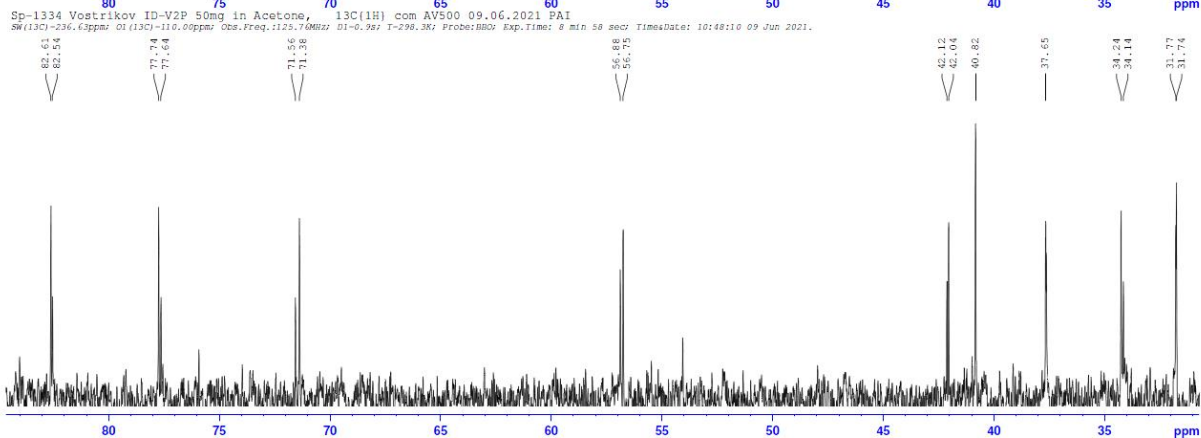
Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS), 2022

SW(13C)-236.63ppm; O1(13C)-110.00ppm; Obs.Freq.:1125.70MHz; D1-1.0s; T-298.3K; Probe:BBQ; Exp.Time: 2 min 27 sec; TimesDate: 10:57:18 09 Jun 2021.



Sp-1334 Vostrikov ID-V2P 50mg in Acetone, 13C(1H) com AV500 09.06.2021 PAI

SW(13C)-236.63ppm; O1(13C)-110.00ppm; Obs.Freq.:1125.70MHz; D1-0.9s; T-298.3K; Probe:BBQ; Exp.Time: 8 min 58 sec; TimesDate: 10:48:10 09 Jun 2021.

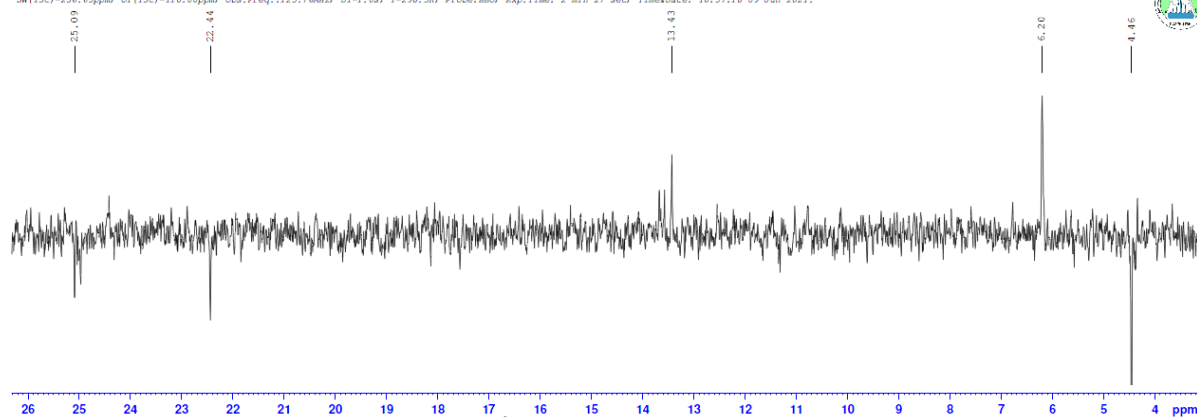


Sp-1334 Vostrikov ID-V2P 50mg in Acetone, 13C(1H) dept135 AV500 09.06.2021 PAI

Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS), 2022

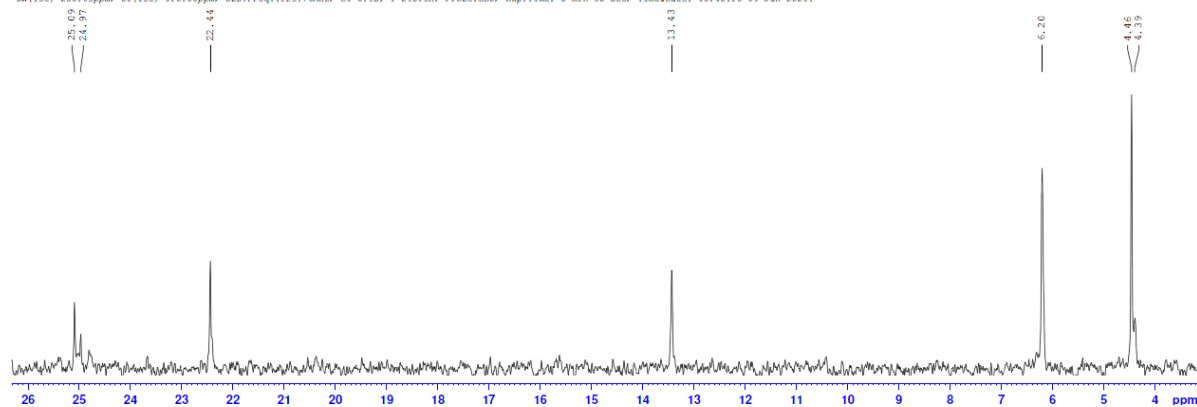


SW(13C)-236.63ppm Ol(13C)-110.00ppm Obs.Freq.:125.76MHz DI-1.09; T-230.3K; Probe:BB0V Exp.Time: 2 min 27 secr TimesDate: 10:57:18 09 Jun 2021.



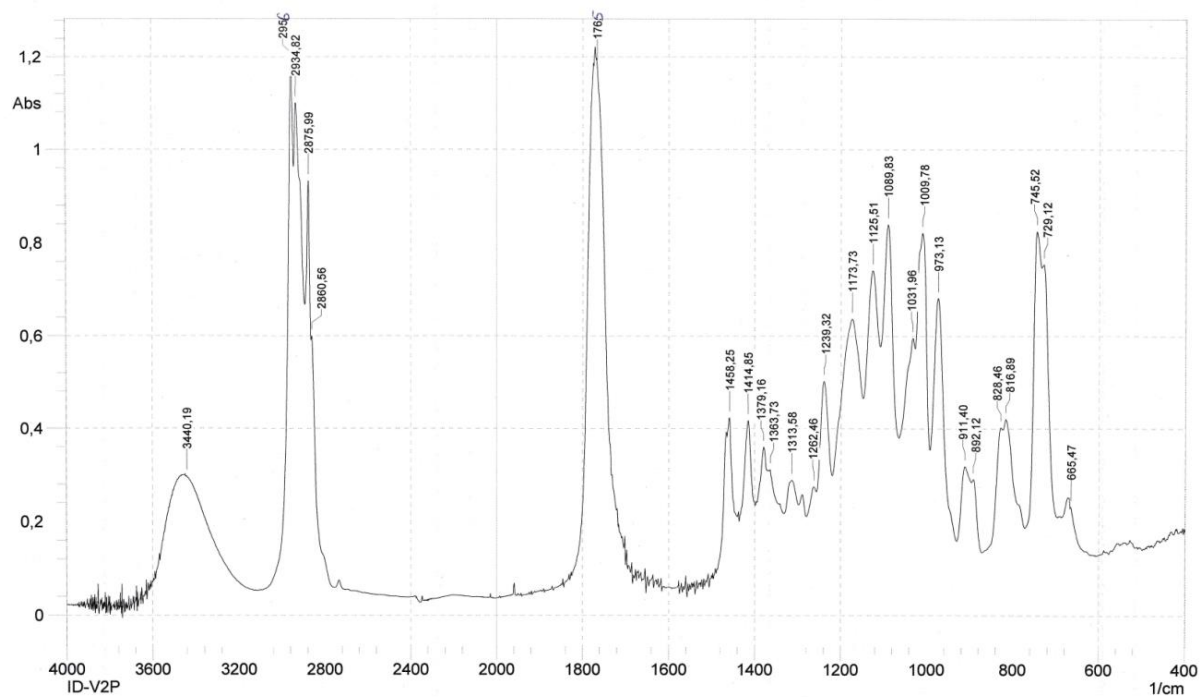
Sp-1334 Vostrikov ID-V2P 50mg in Acetone, 13C(1H) com AV500 09.06.2021 PAI

SW(13C)-236.63ppm Ol(13C)-110.00ppm Obs.Freq.:125.76MHz DI-0.99; T-230.3K; Probe:BB0V Exp.Time: 8 min 58 secr TimesDate: 10:48:10 09 Jun 2021.



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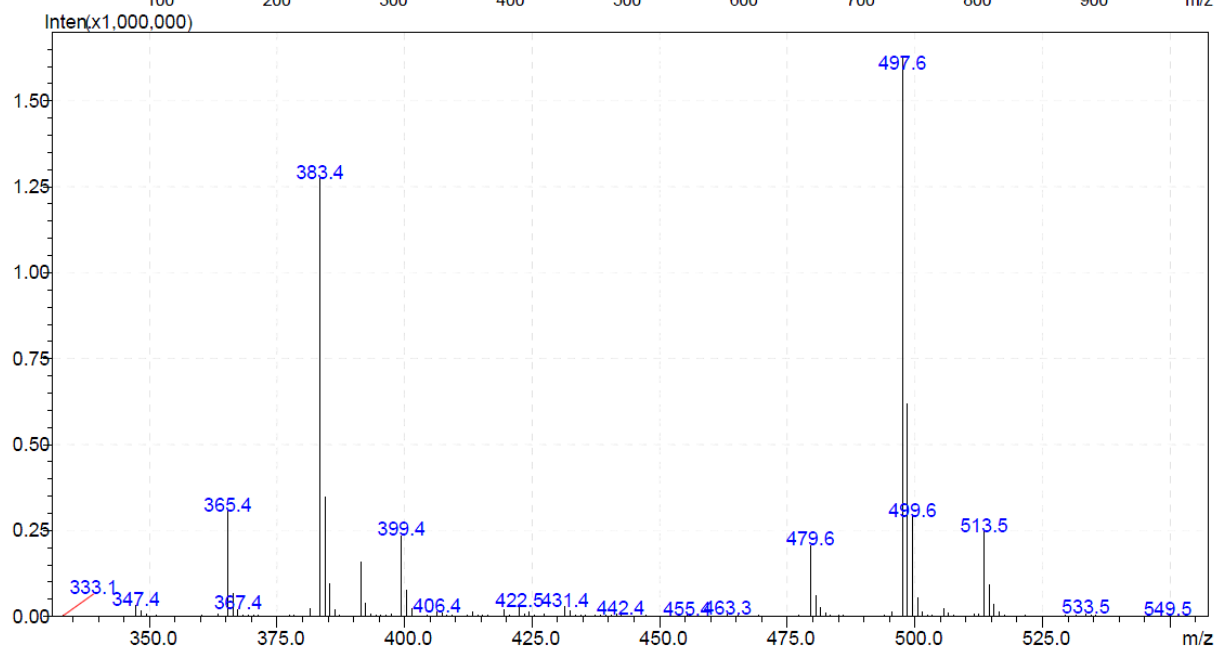
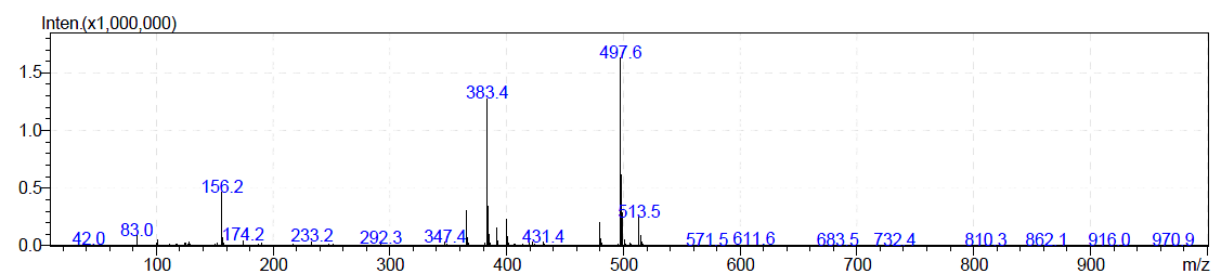


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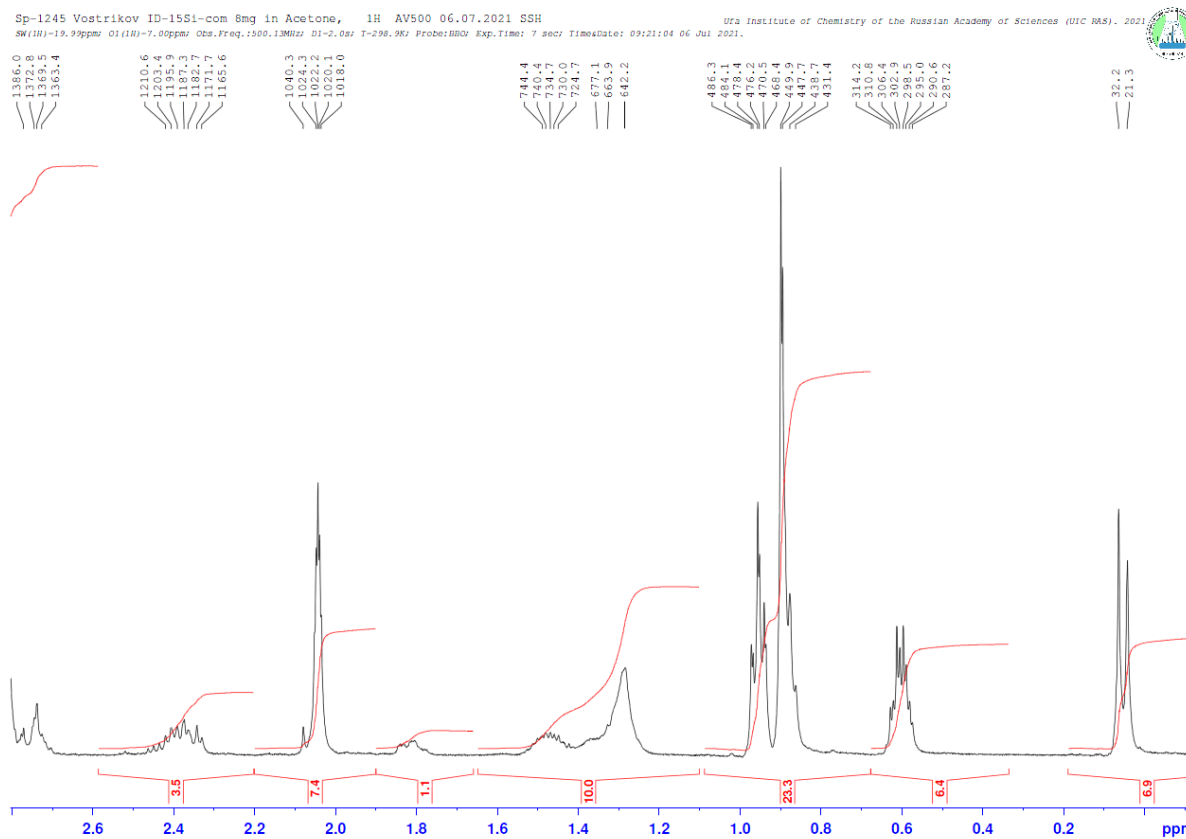
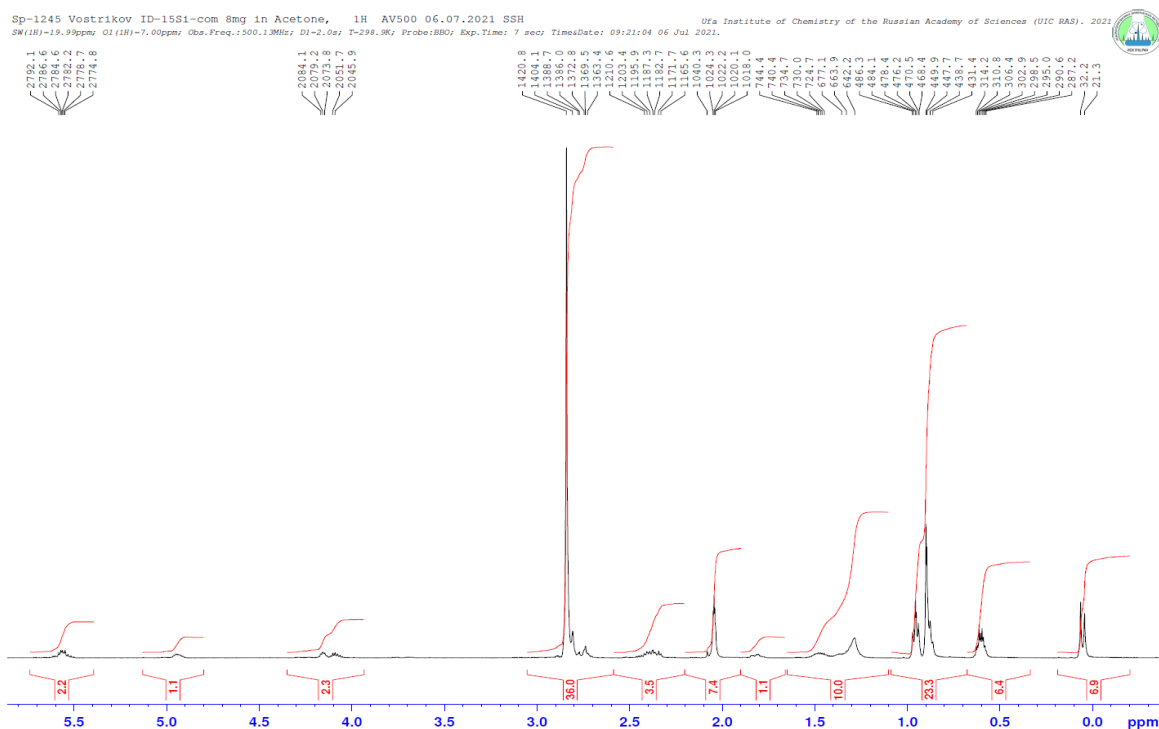
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# Mass



**(3aR\*,4R\*,5R\*,6aS\*)-4-[(E)-3-(tert-butyl dimethylsilyloxy)oct-1-en-1-yl]-5-(triethylsilyloxy)hexahydro-2H-cyclopenta[b]furan-2-one (9).**

**<sup>1</sup>H NMR**

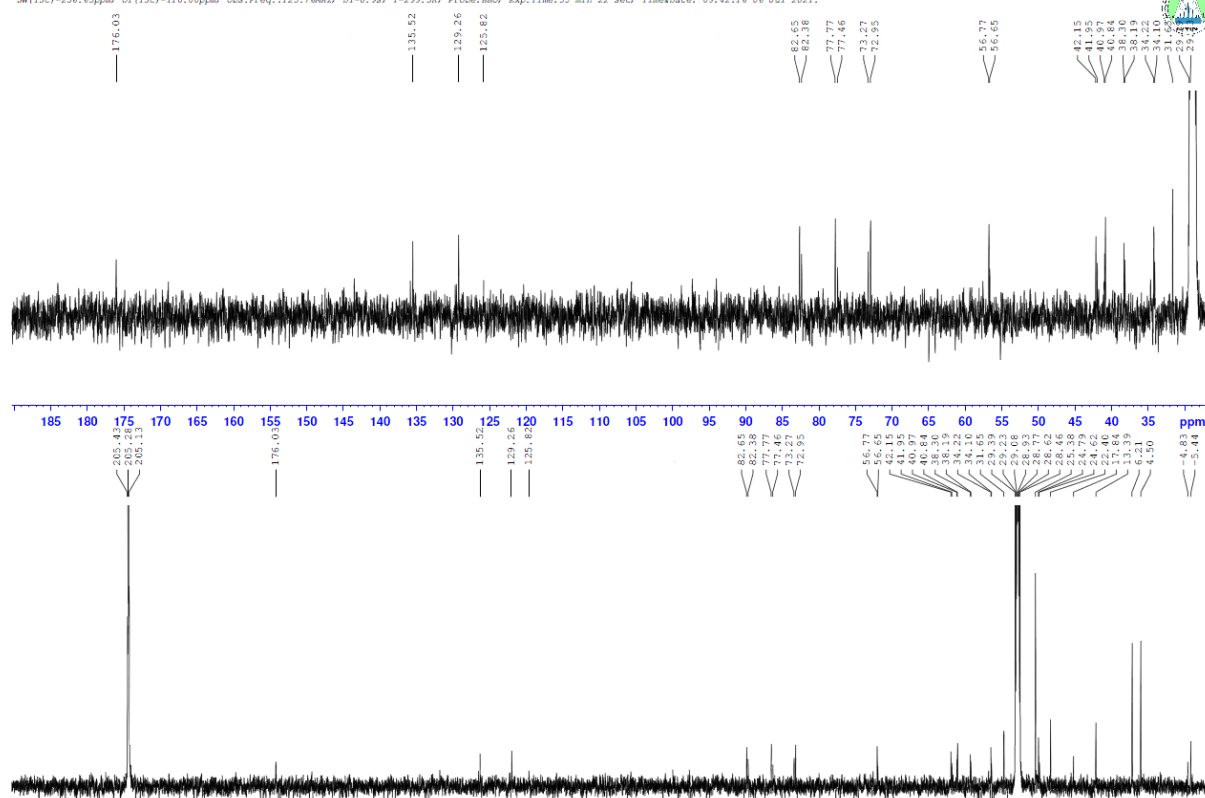


# <sup>13</sup>C NMR

Sp-1245 Vostrikov ID-15Si-com 8mg in CDCl<sub>3</sub>, <sup>13</sup>C{<sup>1</sup>H} com AV500 06.07.2021 SSH

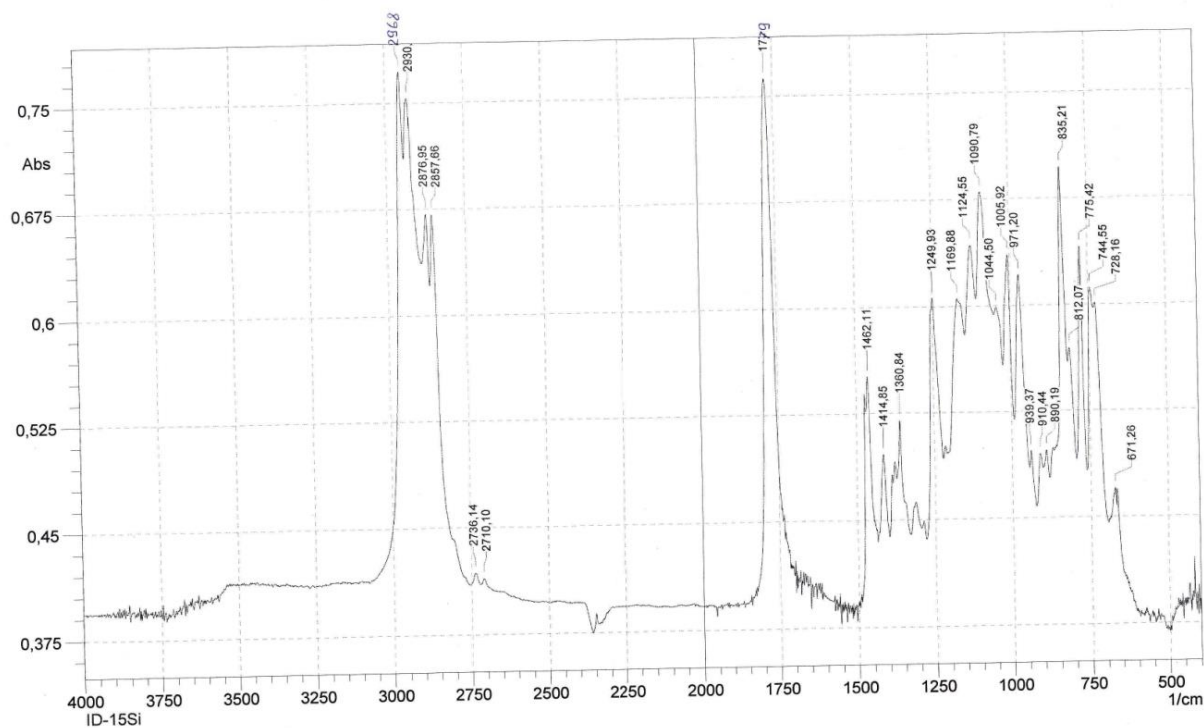
Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS), 2021

SW(13C)-236.63ppm; Q1(13C)-110.00ppm; Cda.Freq.:125.70MHz; D1-0.39; T-299.3K; Probe:BBO; Exp.Time:55 min 22 sec; TimeDate: 09:42:16 06 Jul 2021.



## IR

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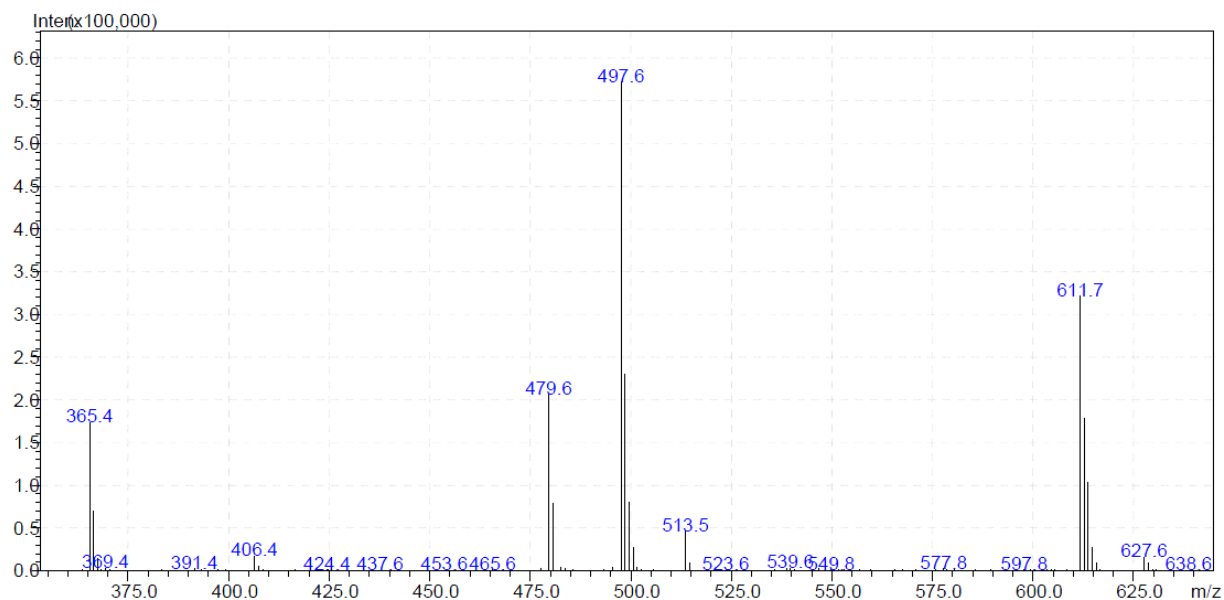
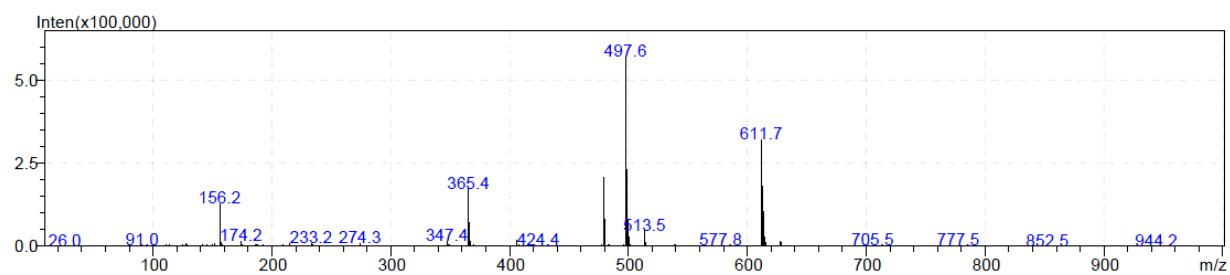


Comment;  
ID-15Si *u u p-pa*

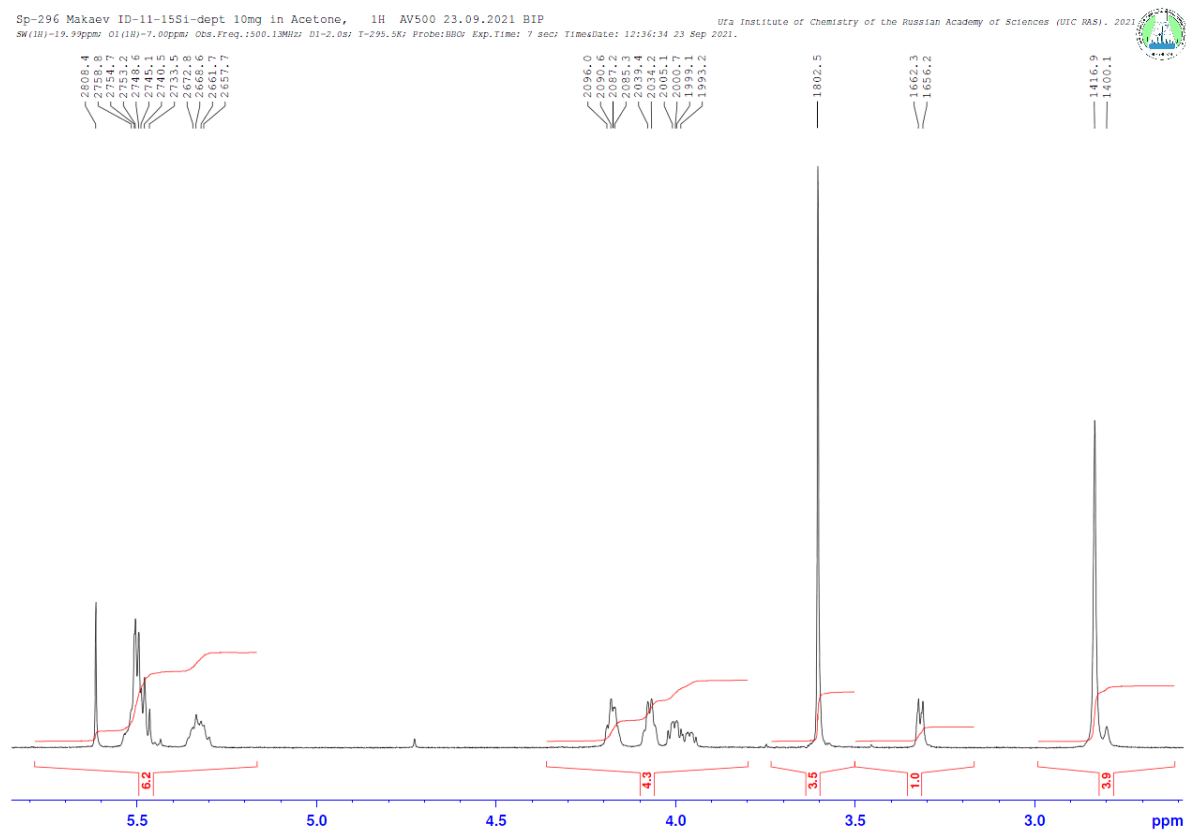
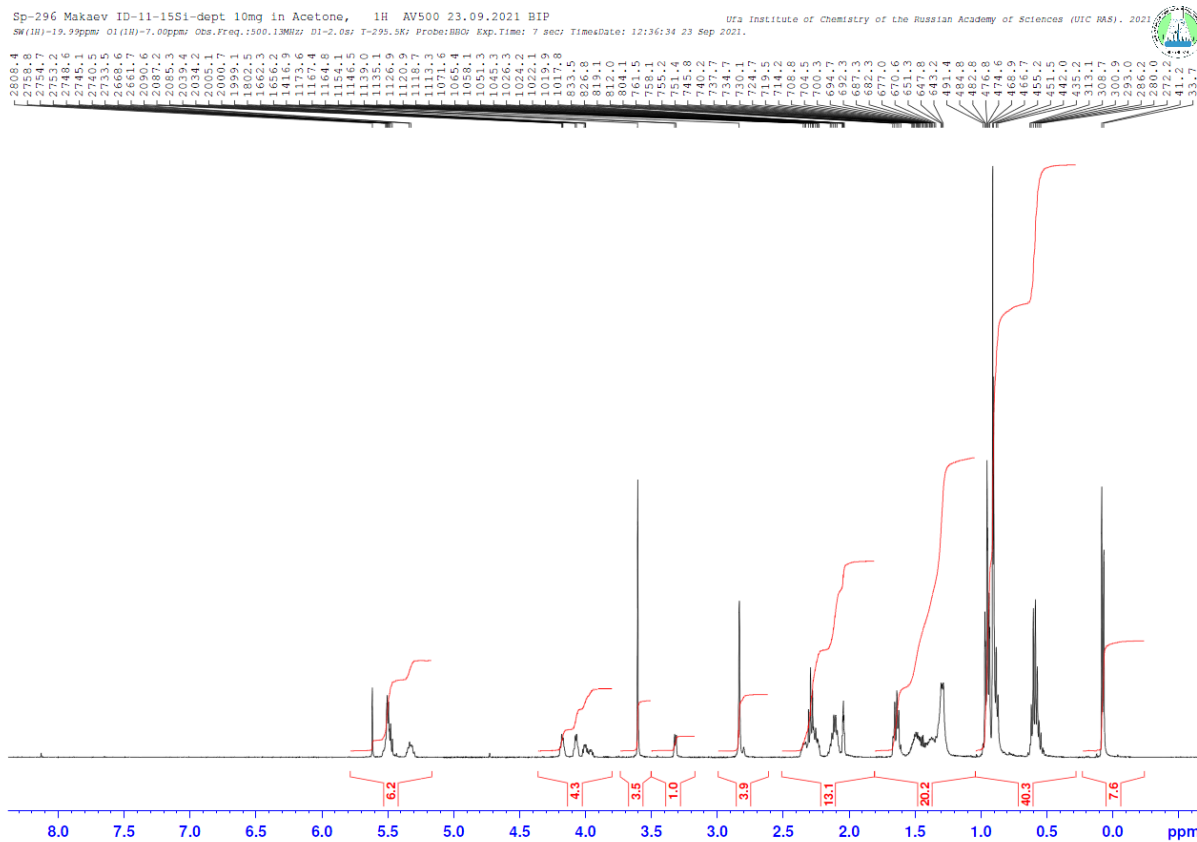
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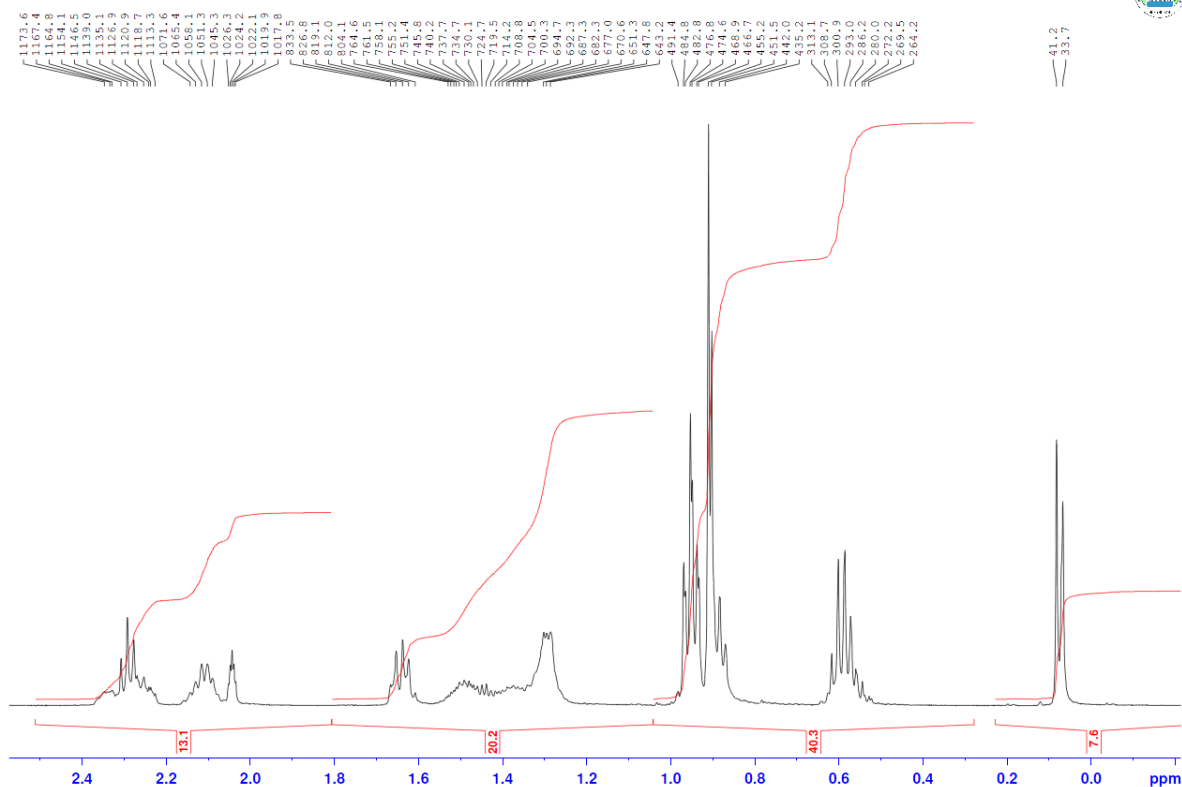
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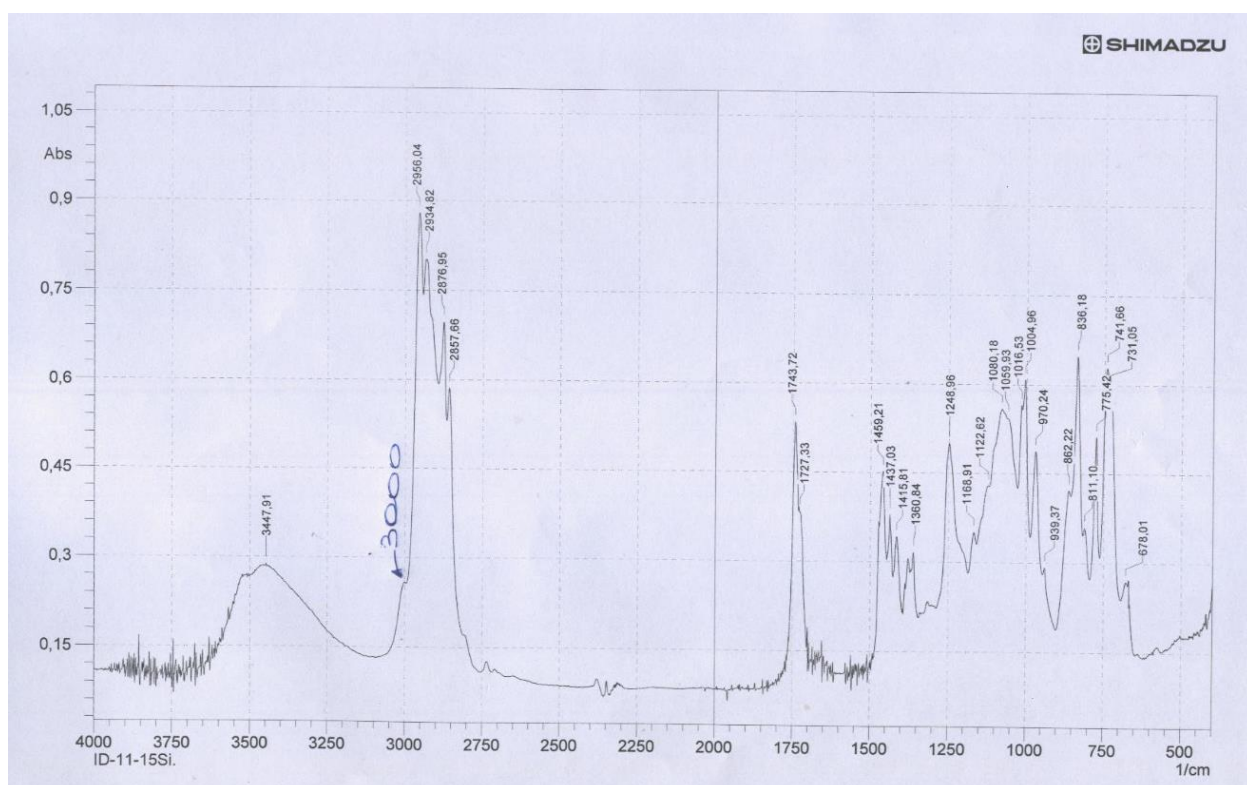


**Methyl (Z)-7-((1R\*,2R\*,3R\*,5S\*)-2-[(E)-3-(tert-butyldimethylsilyloxy)oct-1-en-1-yl]-5-hydroxy-3-(triethylsilyloxy)cyclopentyl]hept-5-enoate (11).**  
<sup>1</sup>H NMR



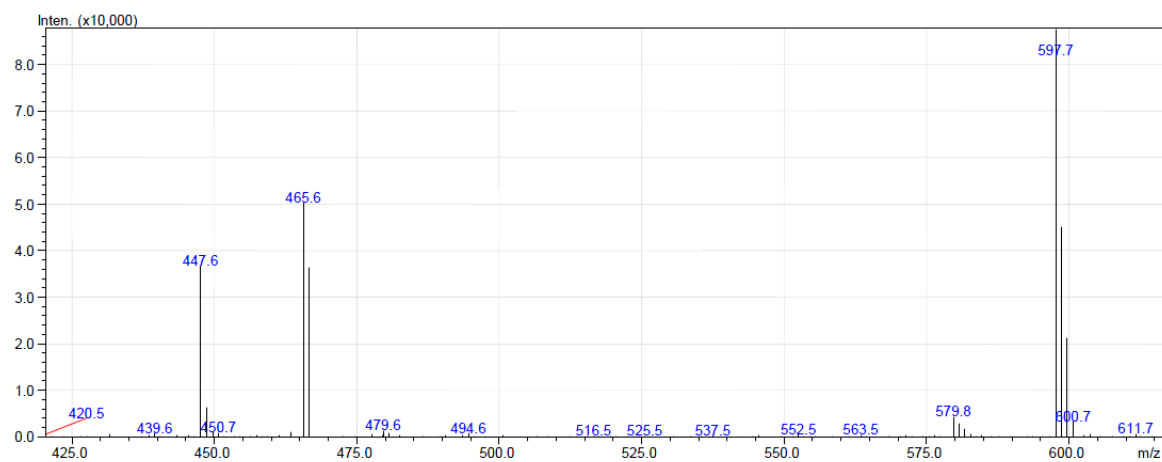
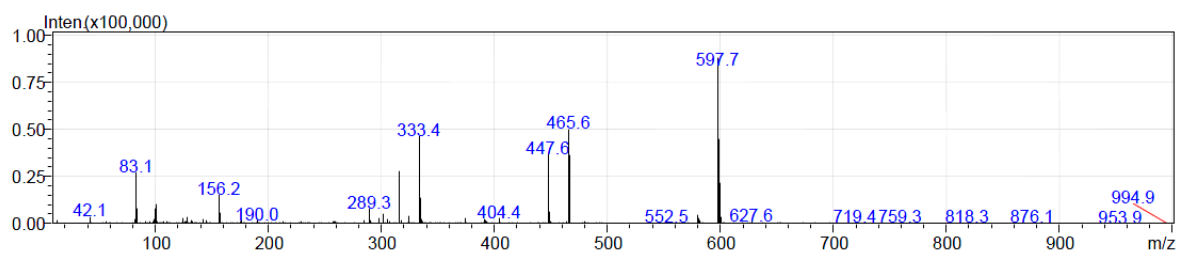


IR

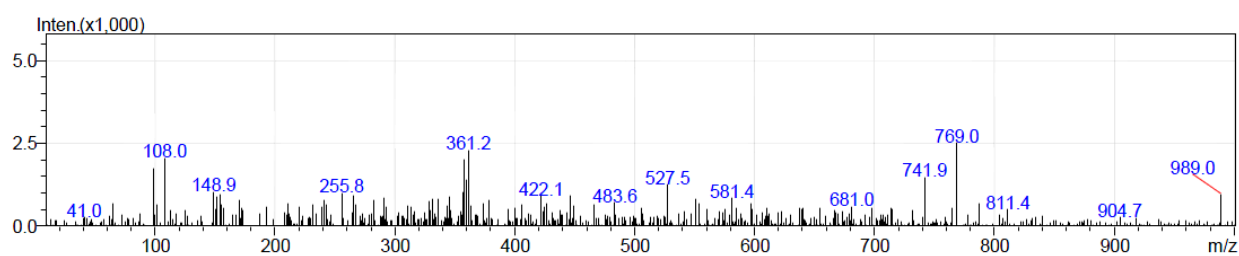


S21

# Mass

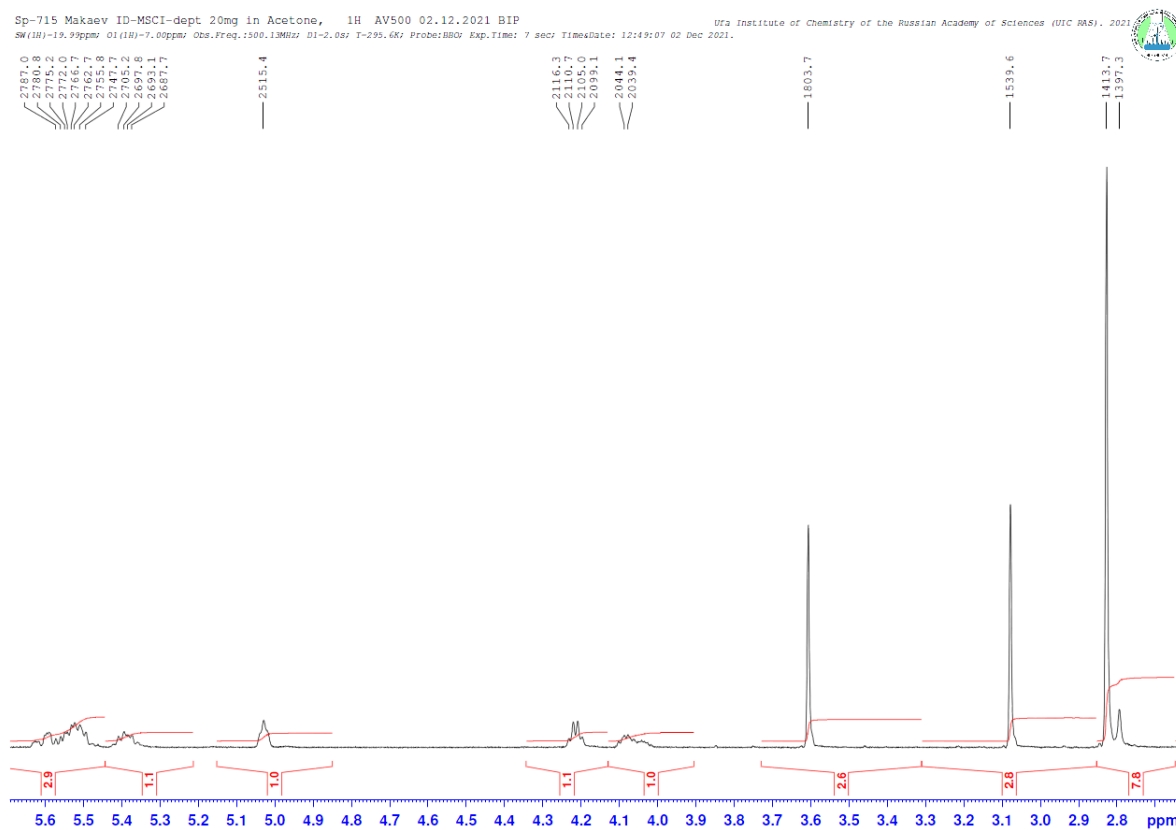
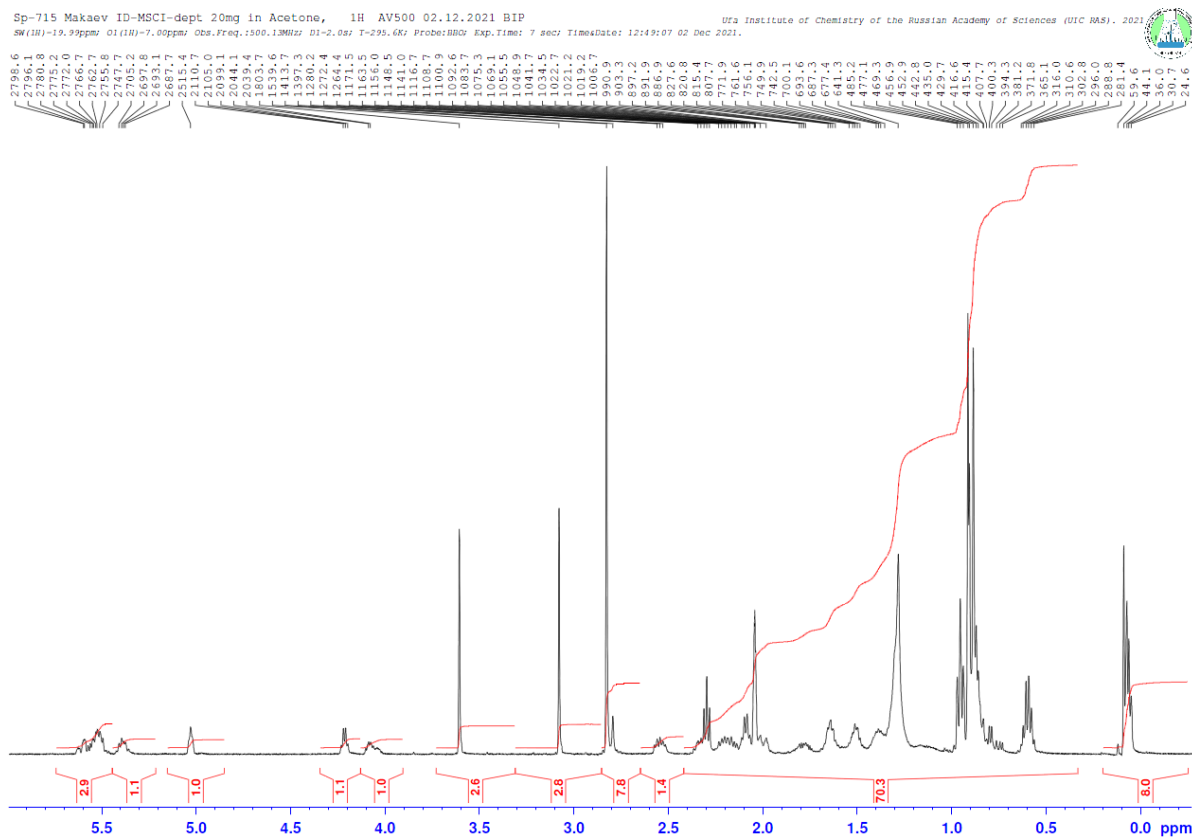


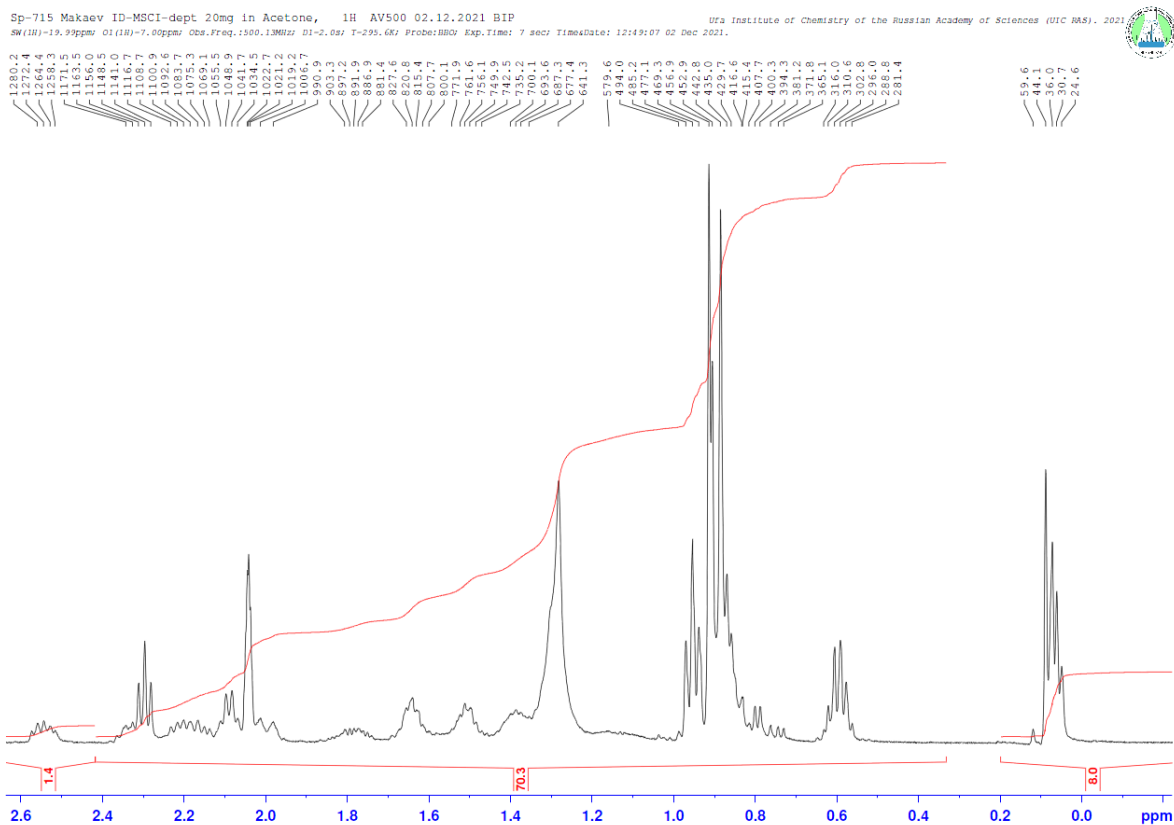
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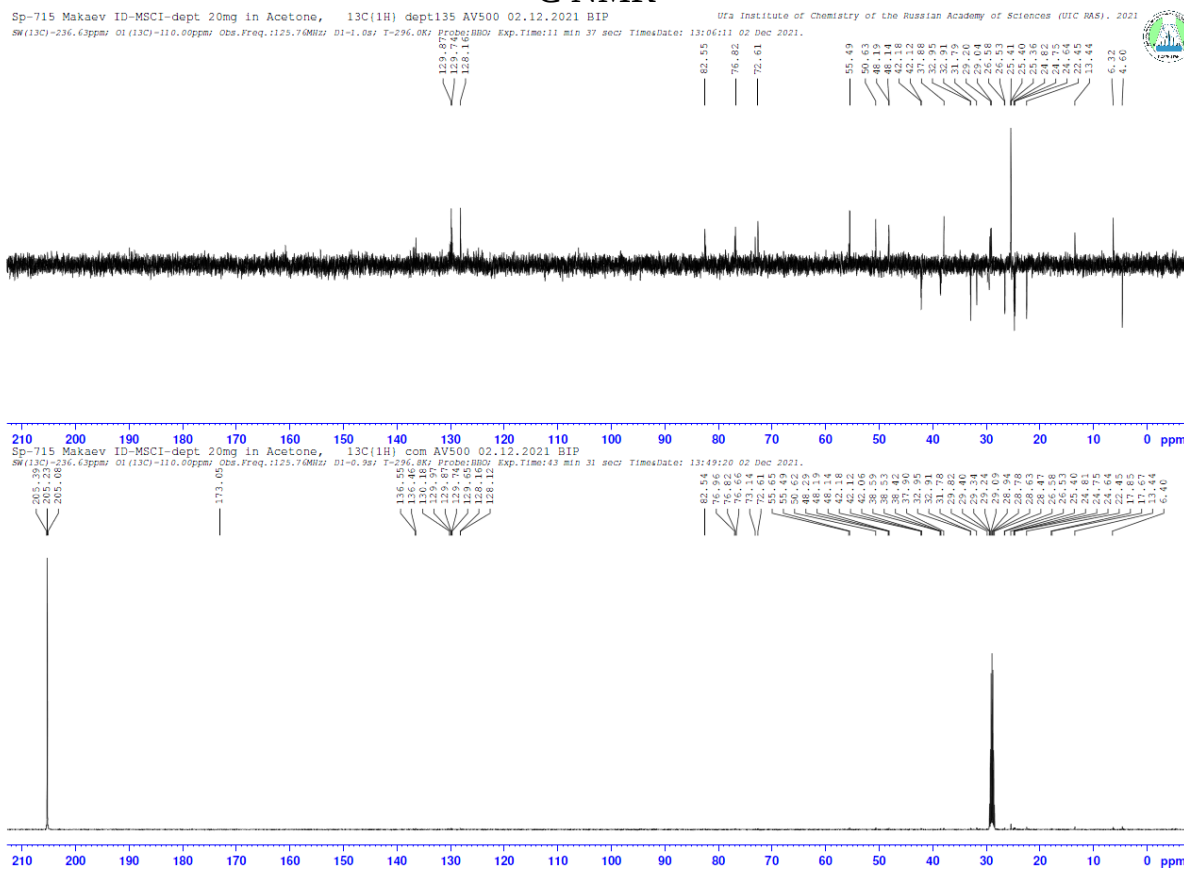
**Methyl (Z)-7-((1*R*\*,2*R*\*,3*R*\*,5*S*\*)-2-[(*E*)-3-(*tert*-butyldimethylsilyloxy)oct-1-en-1-yl]-5-(methylsulfonyloxy)-3-(triethylsilyloxy)cyclopentyl}hept-5-enoate (12).**

<sup>1</sup>H NMR





# <sup>13</sup>C NMR

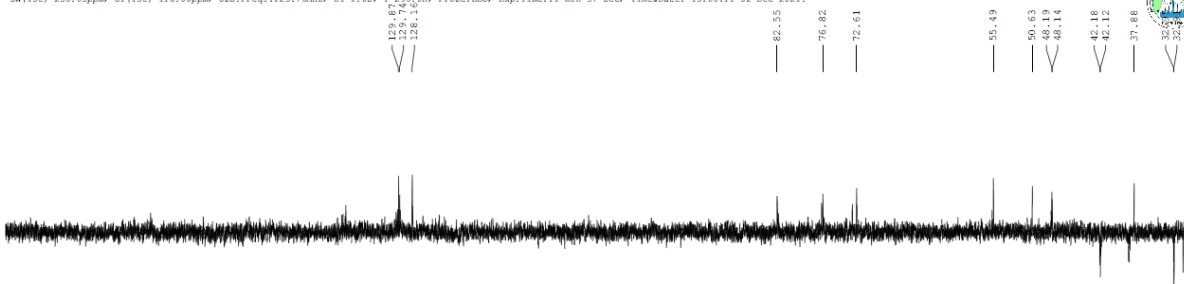


Sp-715 Makaev ID-MSCI-dept 20mg in Acetone, <sup>13</sup>C{<sup>1</sup>H} dept135 AV500 02.12.2021 BIP

Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS). 2021

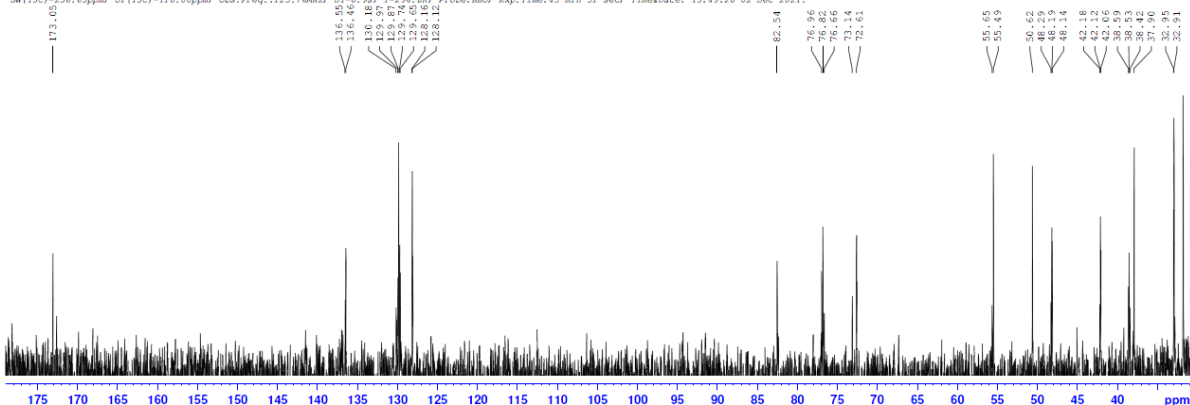


SW(13C)-236.63ppm; Q(13C)-110.00ppm; Obs.Freq.:125.76MHz; D1-1.0s; T-296.0K; Probe:BBQ; Exp.Time:11 min 37 sec; TimeDate: 13:06:11 02 Dec 2021.

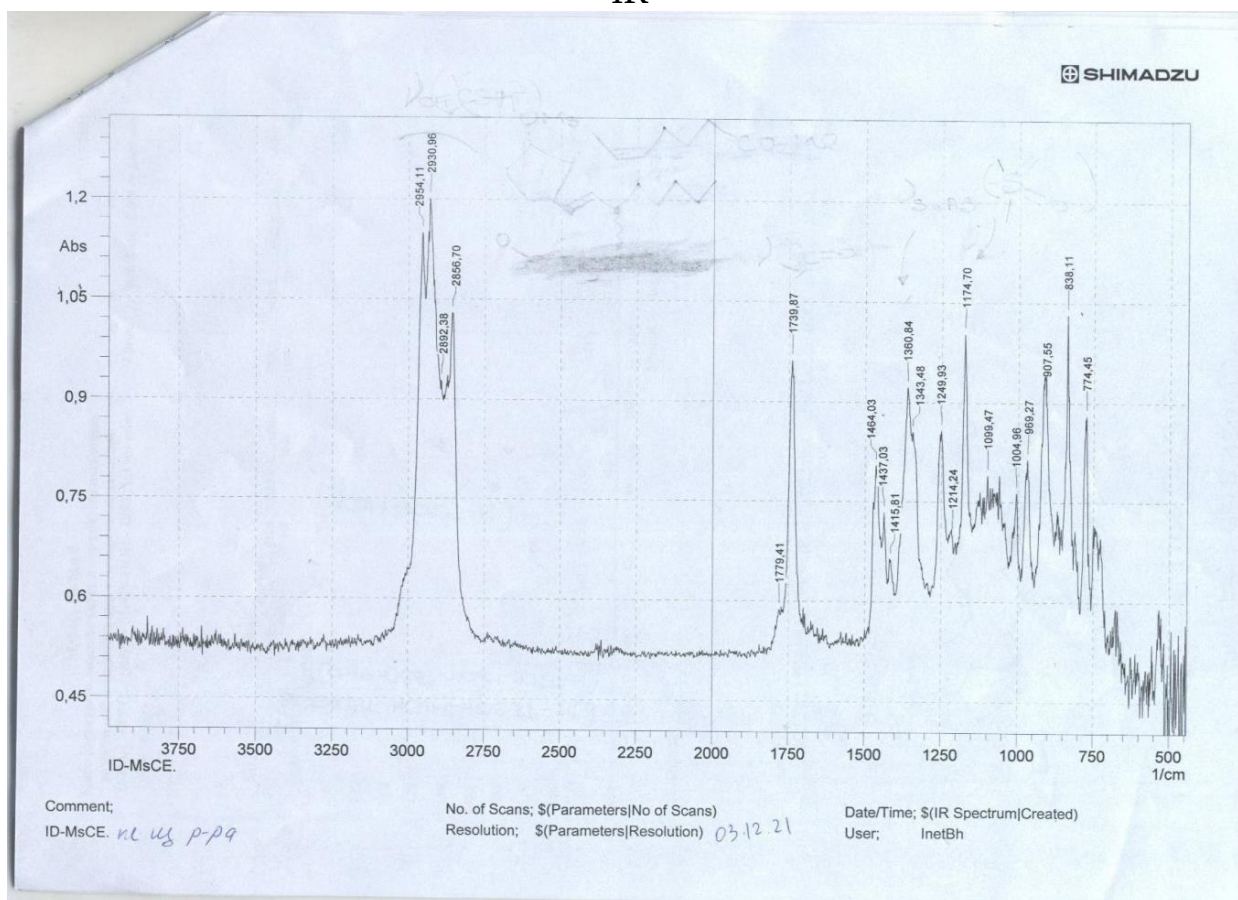


Sp-715 Makaev ID-MSCI-dept 20mg in Acetone, <sup>13</sup>C{<sup>1</sup>H} com AV500 02.12.2021 BIP

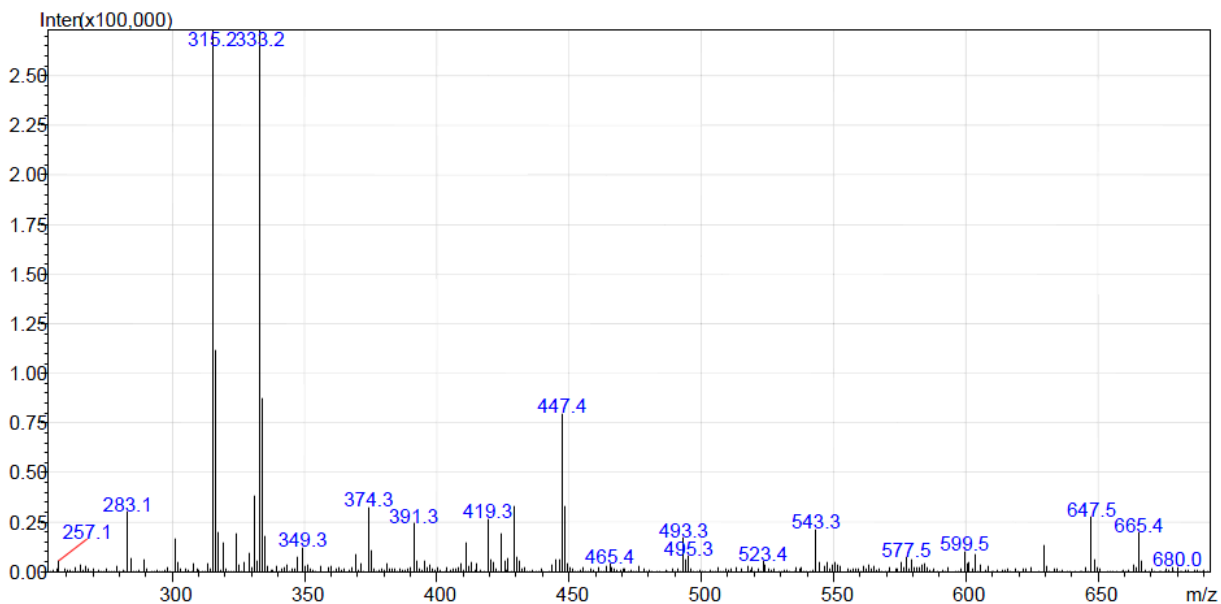
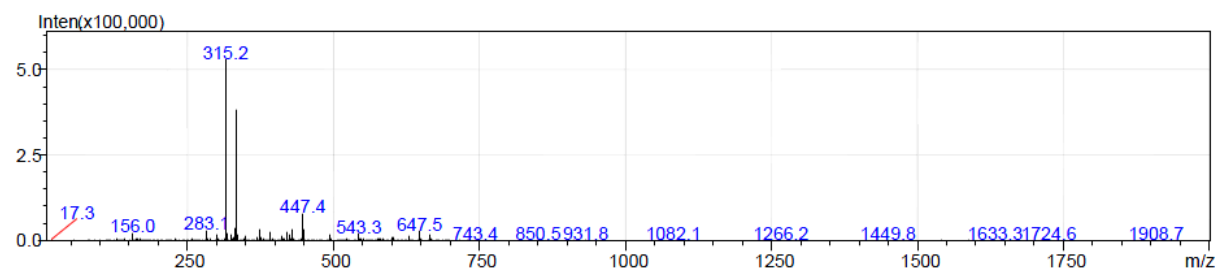
SW(13C)-236.63ppm; Q(13C)-110.00ppm; Obs.Freq.:125.76MHz; D1-0.9s; T-296.0K; Probe:BBQ; Exp.Time:13 min 31 sec; TimeDate: 13:49:20 02 Dec 2021.



## IR



# Mass

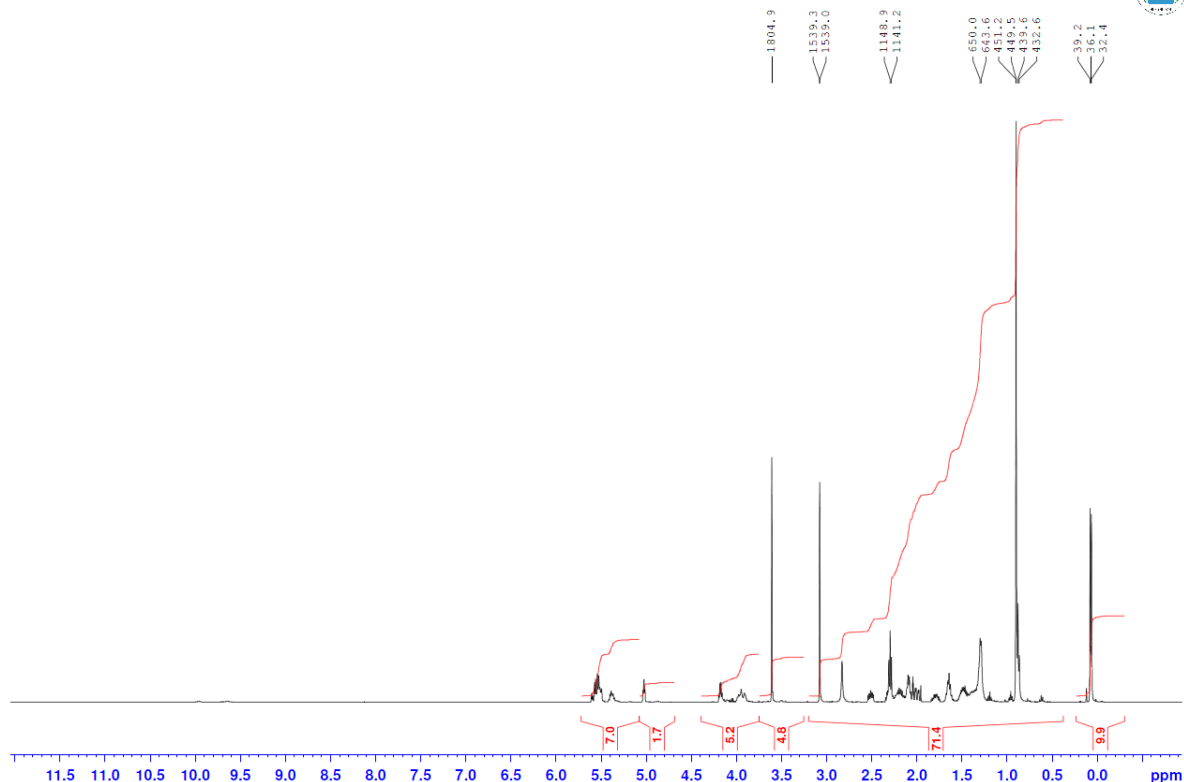


# Methyl (Z)-7-((1R\*,2R\*,3R\*,5S\*)-2-[(E)-3-(tert-butyldimethylsilyloxy)oct-1-en-1-yl]-3-hydroxy-5-(methylsulfonyloxy)cyclopentyl}hept-5-enoate (5)

## <sup>1</sup>H NMR

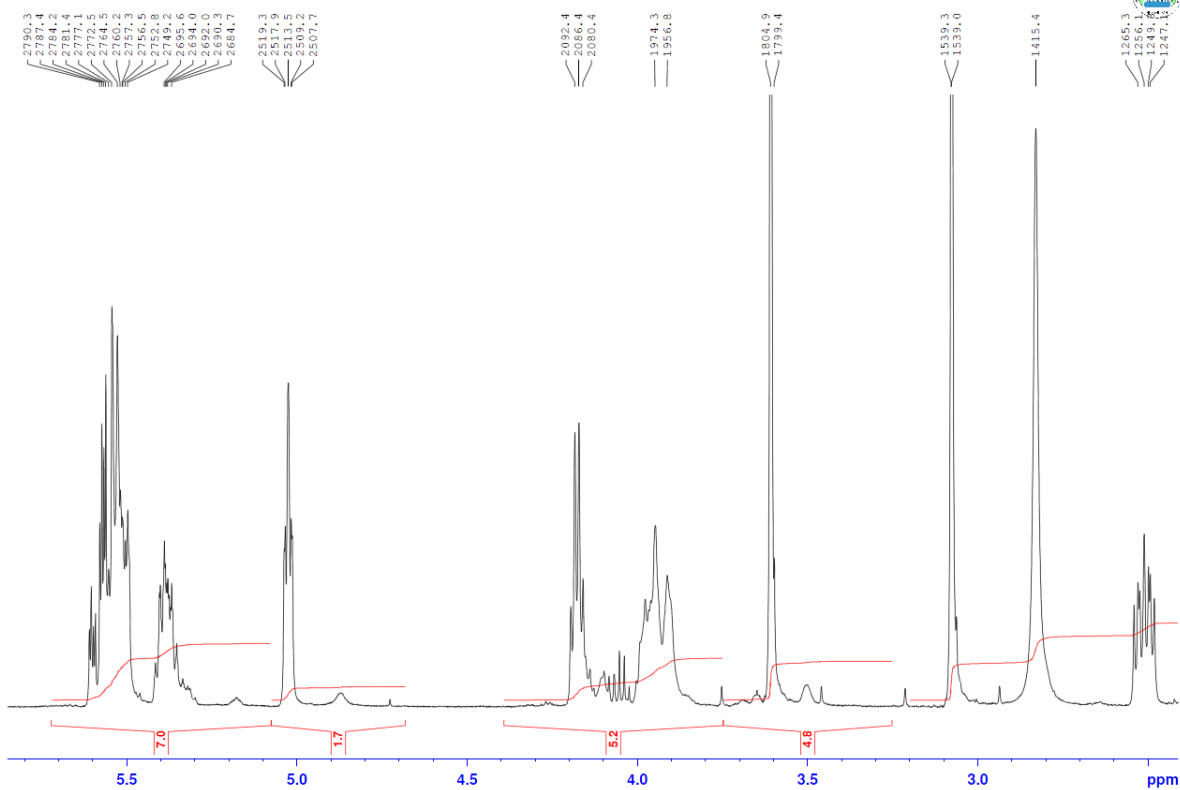
Sp-1323 Makaev NOTES 15mg in Acetone, 1H AV500 25.02.2022 NTR  
SW(1H)-19.99ppm; O1(1H)-7.00ppm; Obs.Freq.:500.13MHz; D1-2.0s; T-673.1K; Probe:BBG; Exp.Time:12 sec; TimeDate: 11:33:34 25 Feb 2022.

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Sp-1323 Makaev NOTES 15mg in Acetone, 1H AV500 25.02.2022 NTR  
SW(1H)-19.99ppm; O1(1H)-7.00ppm; Obs.Freq.:500.13MHz; D1-2.0s; T-673.1K; Probe:BBG; Exp.Time:12 sec; TimeDate: 11:33:34 25 Feb 2022.

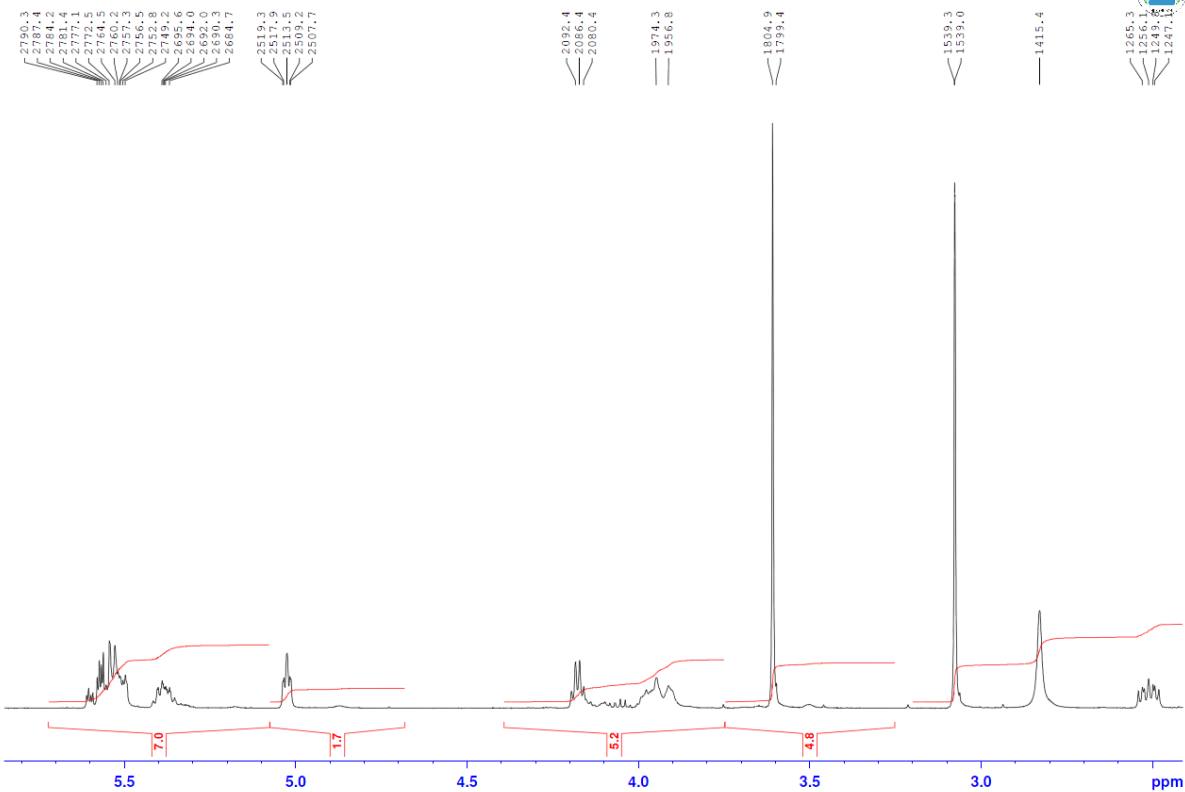
Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS). 2022



Sp-1323 Makaev NOTES 15mg in Acetone, 1H AV500 25.02.2022 NTR

Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS), 2022

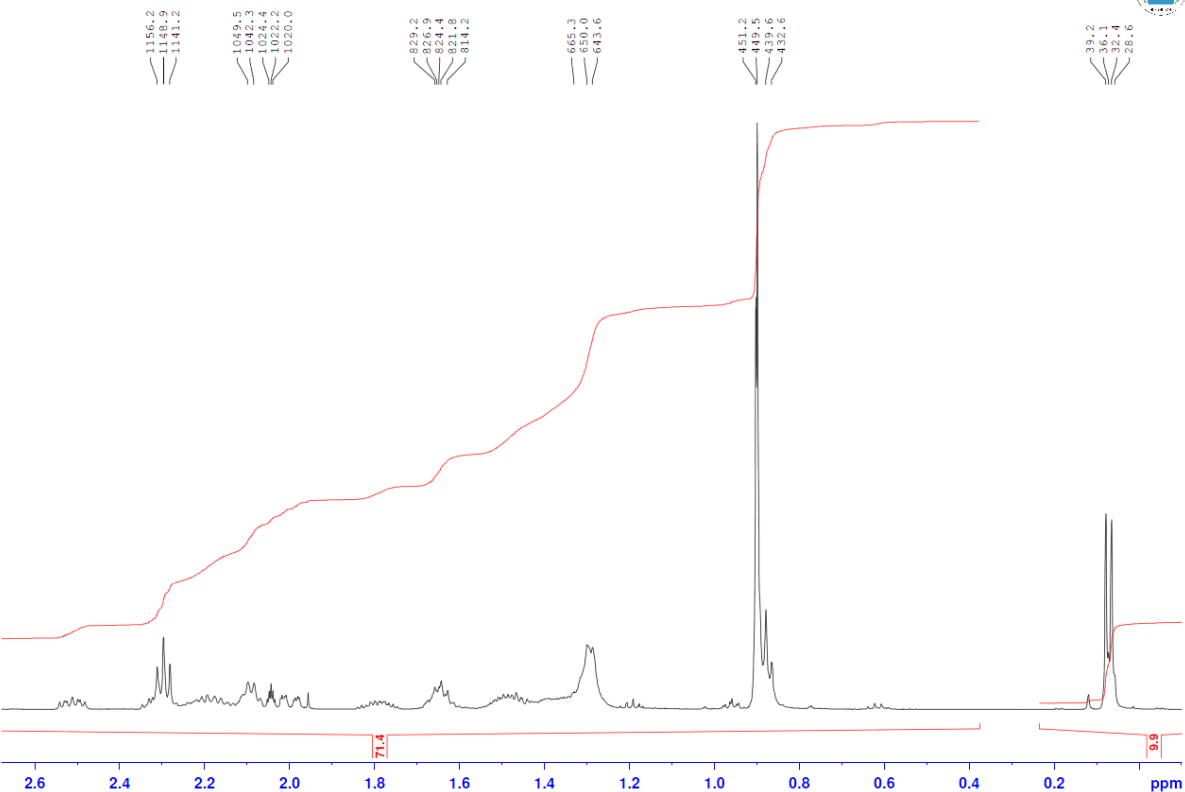
SW(H1)-19.99ppm; O1(H1)-7.00ppm; Obs.Freq:1500.13MHz; D1-2.0s; T-673.1K; Probe:BBG; Exp.Time:12 sec; TimeDate: 11:33:34 25 Feb 2022.



Sp-1323 Makaev NOTES 15mg in Acetone, 1H AV500 25.02.2022 NTR

Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS), 2022

SW(H1)-19.99ppm; O1(H1)-7.00ppm; Obs.Freq:1500.13MHz; D1-2.0s; T-673.1K; Probe:BBG; Exp.Time:12 sec; TimeDate: 11:33:34 25 Feb 2022.



# <sup>13</sup>C NMR

Sp-1323 Makaev NOTES 15mg in Acetone, <sup>13</sup>C(1H) dept135 AV500 25.02.2022 NTR

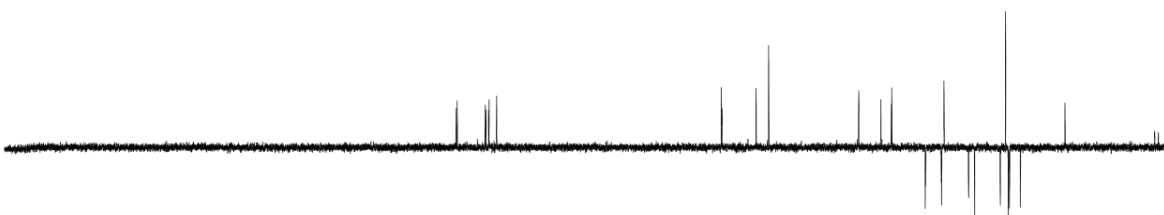
Ufa Institute of Chemistry of the Russian Academy of Sciences (IIC RAS), 2022

SW(13C)-236.63ppm; OI(13C)-110.00ppm; Obs.Freq.:125.70MHz; D1-1.0s; T-673.1K; Probe:BBBO; Exp.Time: 1 min 47 sec; TimeDate: 11:32:27 25 Feb 2022.

136.41  
135.42  
130.50  
129.86  
129.77  
129.64  
128.01  
127.95

82.85  
82.72  
75.84  
75.81  
73.30

55.13  
55.11  
54.94  
54.85  
48.43  
41.67  
38.43  
38.37  
32.96  
32.92  
32.88  
26.50  
26.45  
24.92  
24.90  
24.86  
24.64  
24.45  
23.44

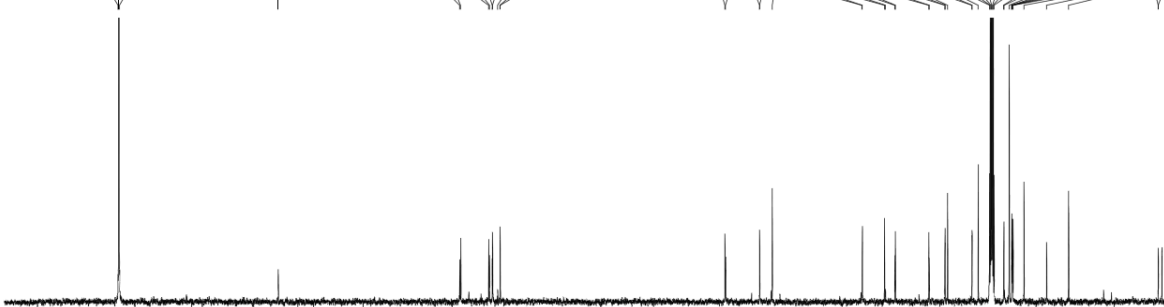


Sp-1323 Makaev NOTES 15mg in Acetone, <sup>13</sup>C(1H) com AV500 25.02.2022 NTR  
SW(13C)-236.63ppm; OI(13C)-110.00ppm; Obs.Freq.:125.70MHz; D1-0.9s; T-673.1K; Probe:BBBO; Exp.Time: 9 min 6 sec; TimeDate: 11:42:12 25 Feb 2022.

204.38  
204.36  
204.34  
172.35  
136.41  
135.42  
130.50  
129.86  
129.77  
129.64  
128.01  
127.95

82.85  
82.72  
75.84  
75.81  
73.30

54.43  
54.39  
48.43  
48.40  
40.87  
40.84  
38.43  
38.37  
32.96  
32.92  
32.88  
26.50  
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24.92  
24.90  
24.86  
24.64  
24.45  
23.44

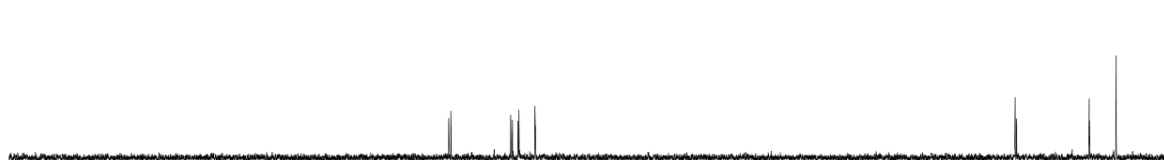


Sp-1323 Makaev NOTES 15mg in Acetone, <sup>13</sup>C(1H) dept135 AV500 25.02.2022 NTR  
SW(13C)-236.63ppm; OI(13C)-110.00ppm; Obs.Freq.:125.70MHz; D1-1.0s; T-673.1K; Probe:BBBO; Exp.Time: 1 min 47 sec; TimeDate: 11:32:27 25 Feb 2022.

136.41  
135.42  
130.50  
129.86  
129.77  
129.64  
128.01  
127.95

82.85  
82.72  
75.84  
75.81  
73.30

55.13  
55.11  
54.94  
54.85  
48.43  
41.67  
38.43  
38.37  
32.96  
32.92  
32.88  
26.50  
26.45  
24.92  
24.90  
24.86  
24.64  
24.45  
23.44

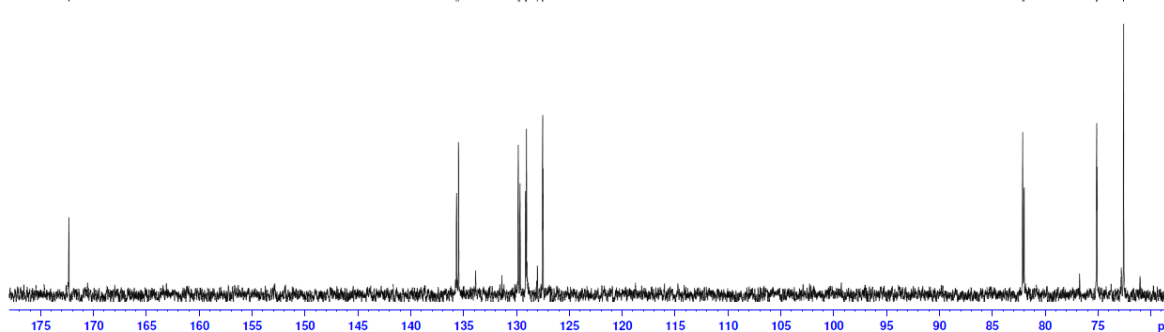


Sp-1323 Makaev NOTES 15mg in Acetone, <sup>13</sup>C(1H) com AV500 25.02.2022 NTR  
SW(13C)-236.63ppm; OI(13C)-110.00ppm; Obs.Freq.:125.70MHz; D1-0.9s; T-673.1K; Probe:BBBO; Exp.Time: 9 min 6 sec; TimeDate: 11:42:12 25 Feb 2022.

136.41  
135.42  
130.50  
129.86  
129.77  
129.64  
128.01  
127.95

82.85  
82.72  
75.84  
75.81  
73.30

55.13  
55.11  
54.94  
54.85  
48.43  
41.67  
38.43  
38.37  
32.96  
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24.92  
24.90  
24.86  
24.64  
24.45  
23.44

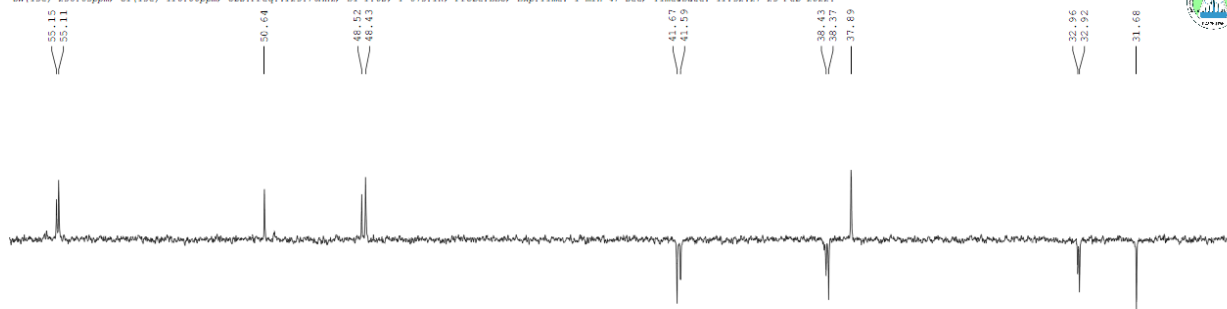


Sp-1323 Makaev NOTES 15mg in Acetone, <sup>13</sup>C(1H) dept135 AV500 25.02.2022 NTR

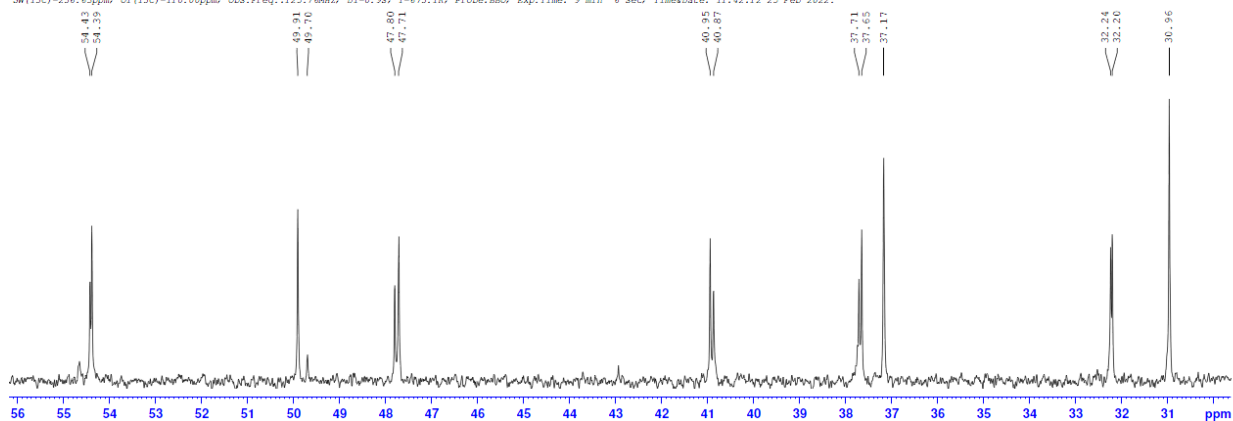
Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS), 2022



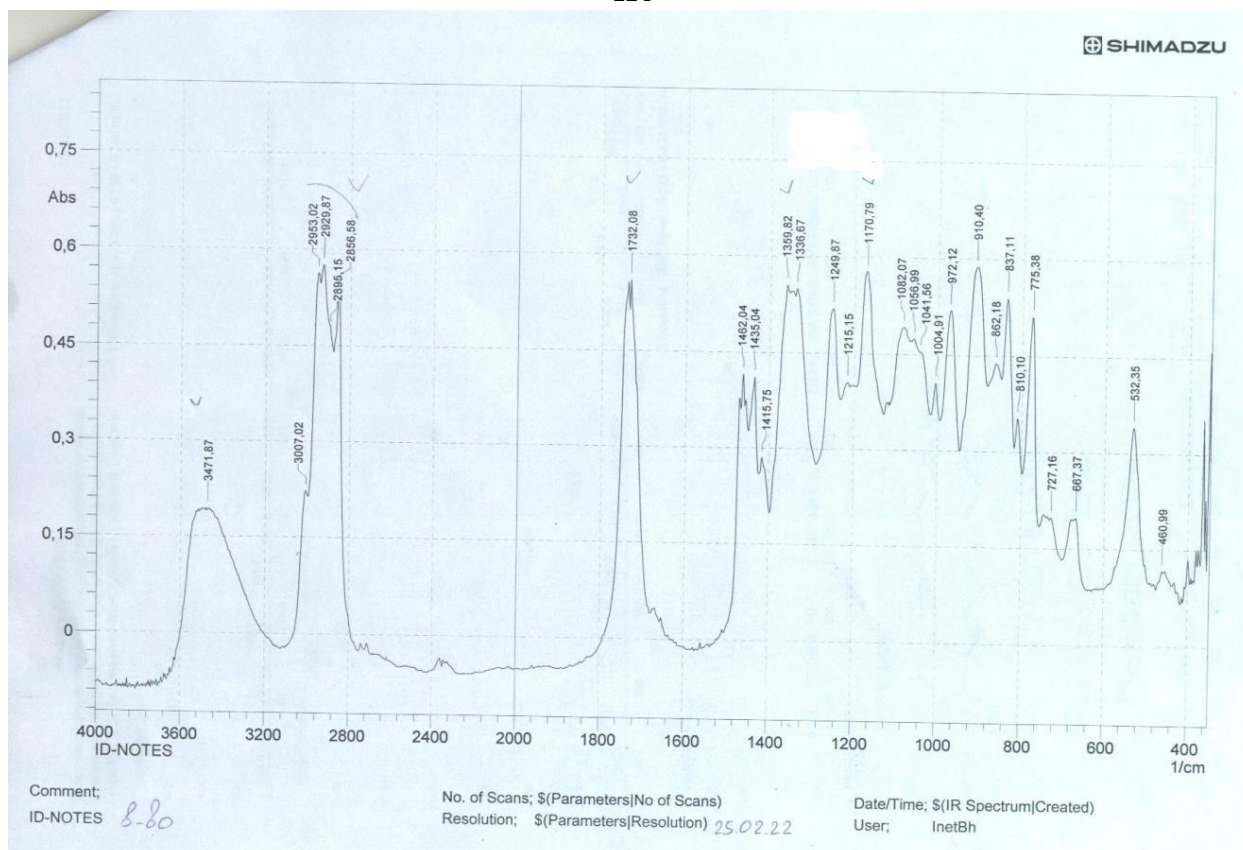
SW(13C)-236.63ppm; O1(13C)-110.00ppm; Obs.Freq.:125.76MHz; D1-1.0s; T-673.1K; Probe:BB0V Exp.Time: 1 min 47 sec; TimeDate: 11:32:27 25 Feb 2022.



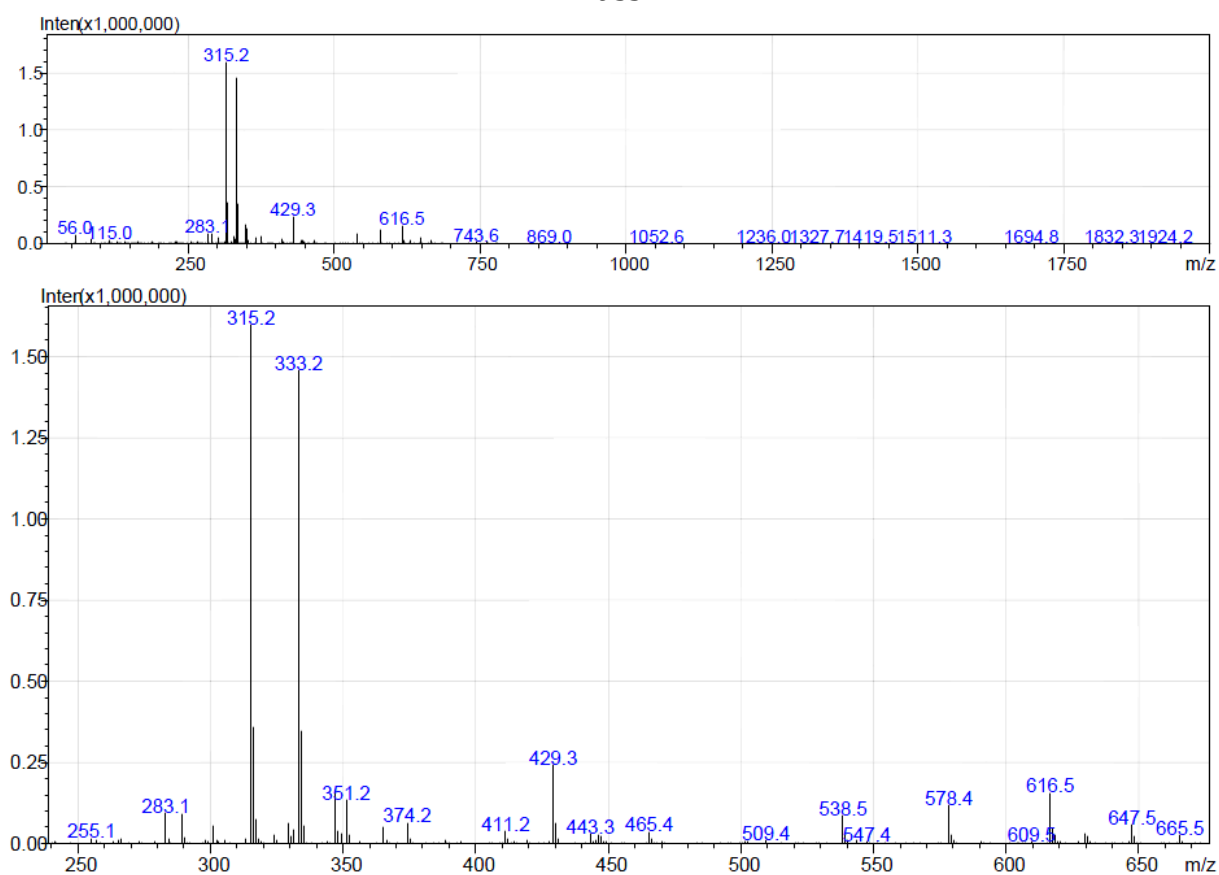
Sp-1323 Makaev NOTES 15mg in Acetone, <sup>13</sup>C(1H) com AV500 25.02.2022 NTR  
SW(13C)-236.63ppm; O1(13C)-110.00ppm; Obs.Freq.:125.76MHz; D1-0.9s; T-673.1K; Probe:BB0V Exp.Time: 9 min 6 sec; TimeDate: 11:42:12 25 Feb 2022.



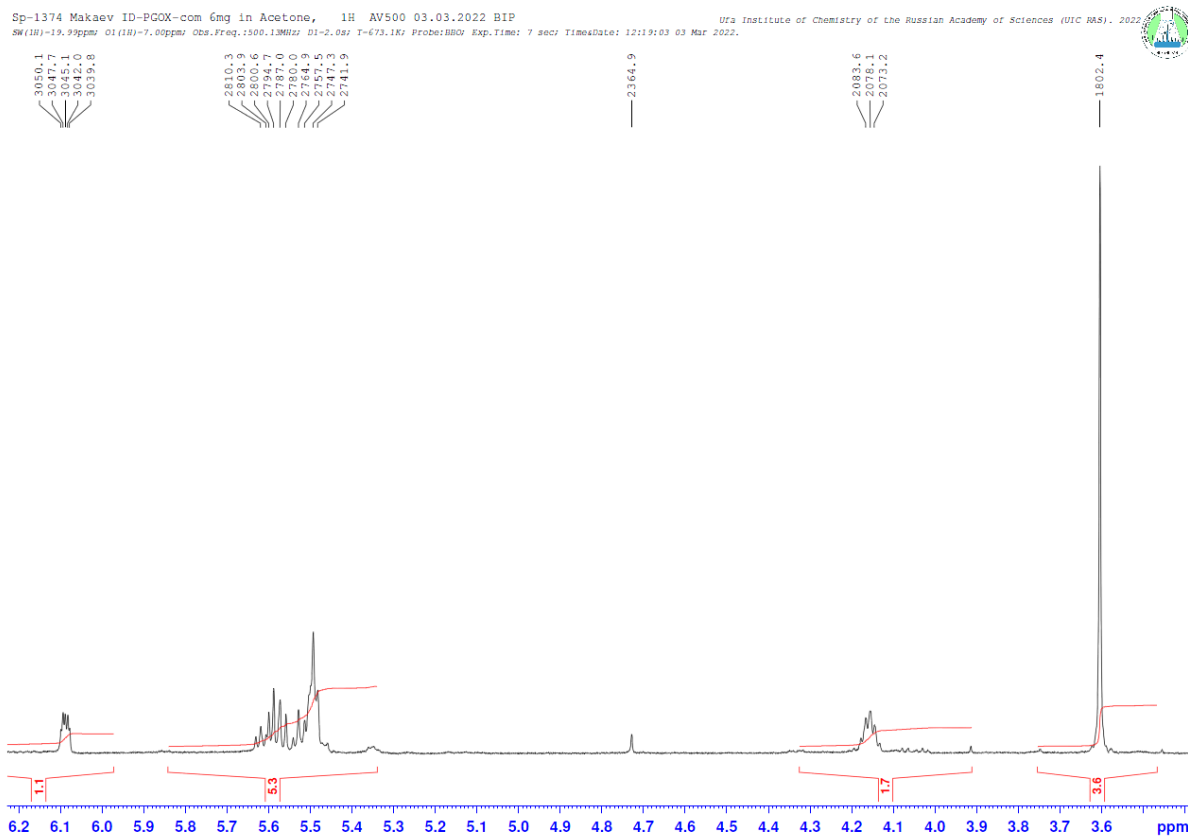
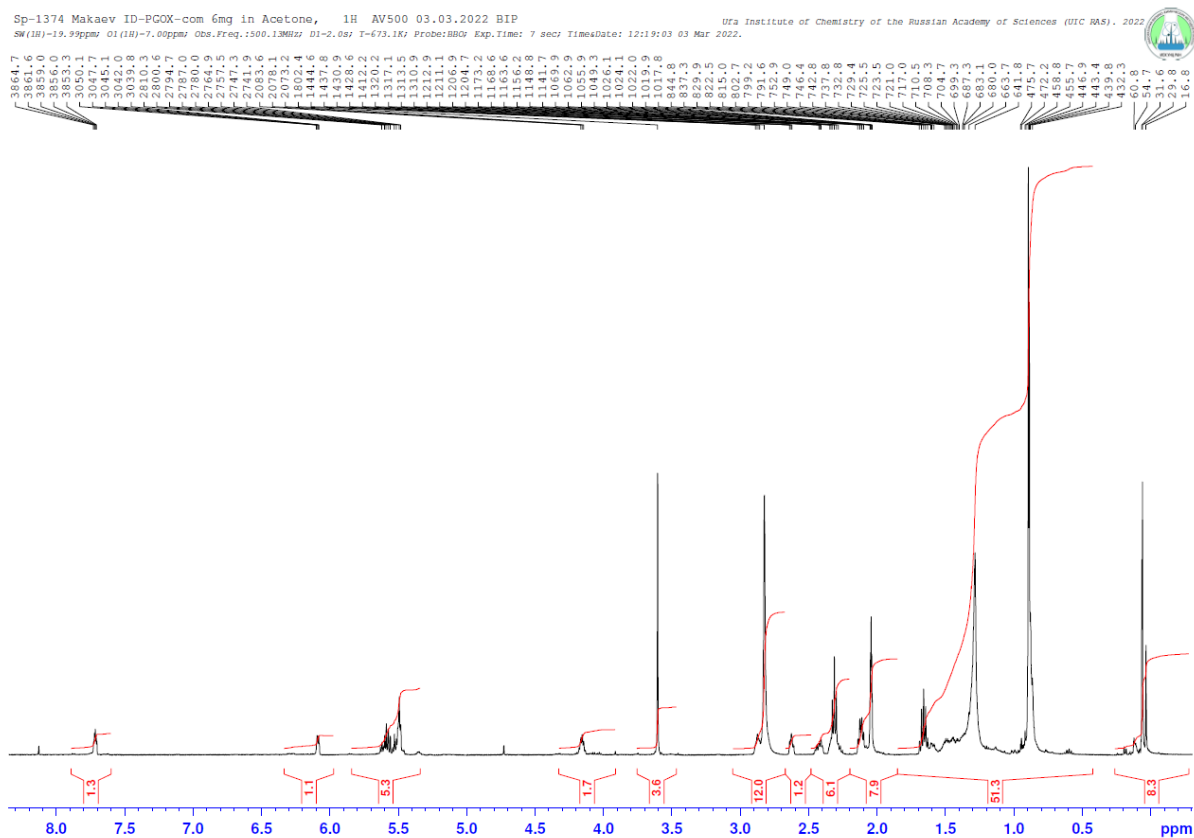
## IR



# Mass

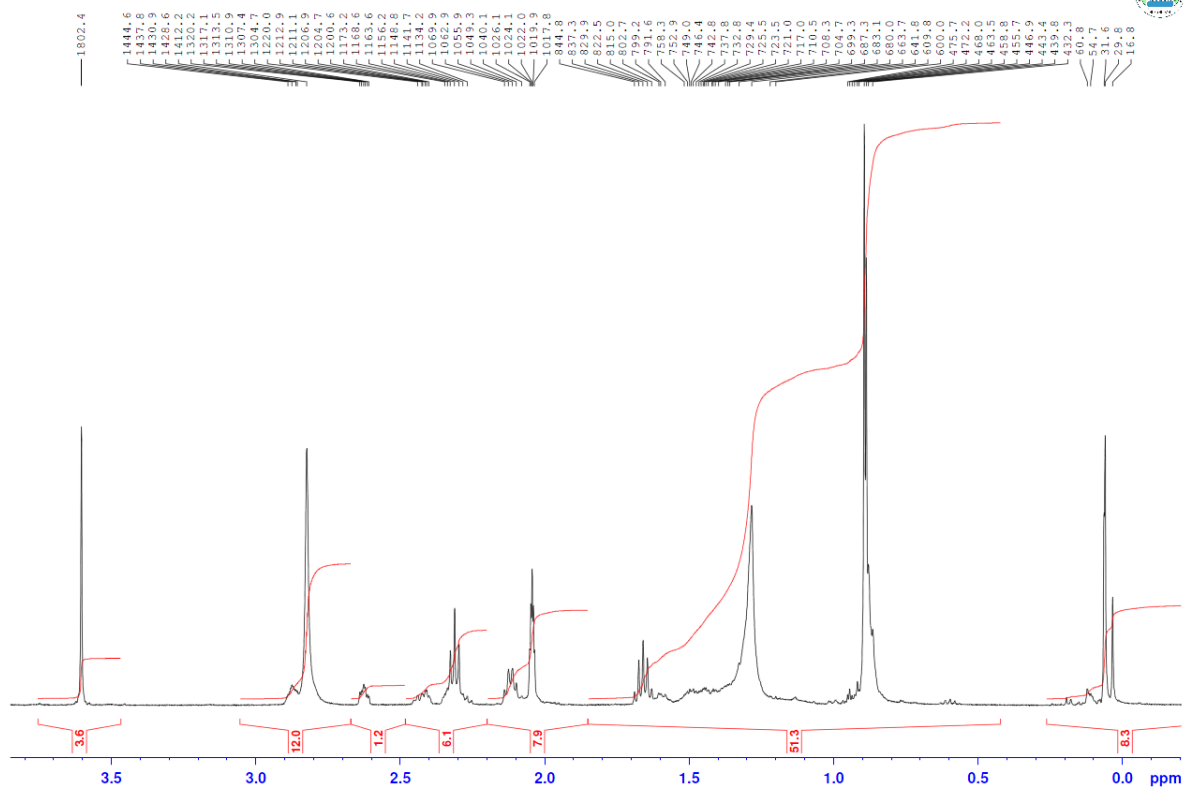


**Methyl (Z)-7-[(1S\*,5R\*)-5-[(E)-3-(tert-butyl dimethylsilyloxy)oct-1-en-1-yl]-4-oxocyclopent-2-en-1-yl]hept-5-enoate (13).**  
<sup>1</sup>H NMR



Sp-1374 Makaev ID-PG0X-com 6mg in Acetone, 1H AV500 03.03.2022 BIP  
 SW(1H)-19.99ppm; 01 (1H)-7.00ppm; Obs.Freq.:500.13MHz; D1-2.0s; T-673.1K; Probe:BB0; Exp.Time: 7 sec; TimesDate: 12:19:03 03 Mar 2022.

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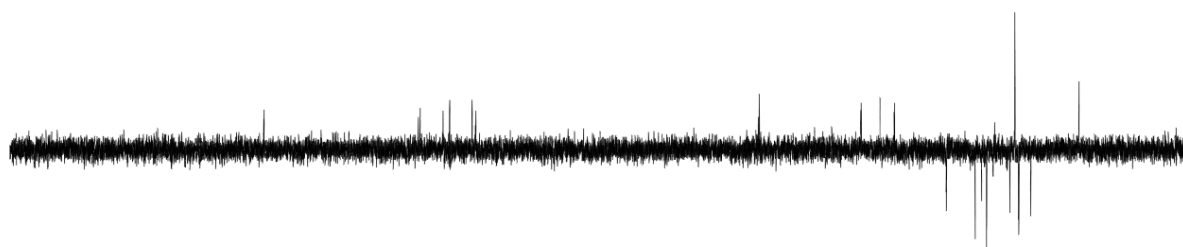
### $^{13}\text{C}$ NMR

Sp-1374 Makaev ID-PG0X-com 6mg in Acetone,  $^{13}\text{C}$ (1H) dept135 AV500 03.03.2022 BIP

Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS), 2022



SW(13C)-236.63ppm; 01 (13C)-110.00ppm; Obs.Freq.:125.76MHz; D1-1.0s; T-673.1K; Probe:BB0; Exp.Time: 7 min 2 sec; TimesDate: 12:49:36 03 Mar 2022.

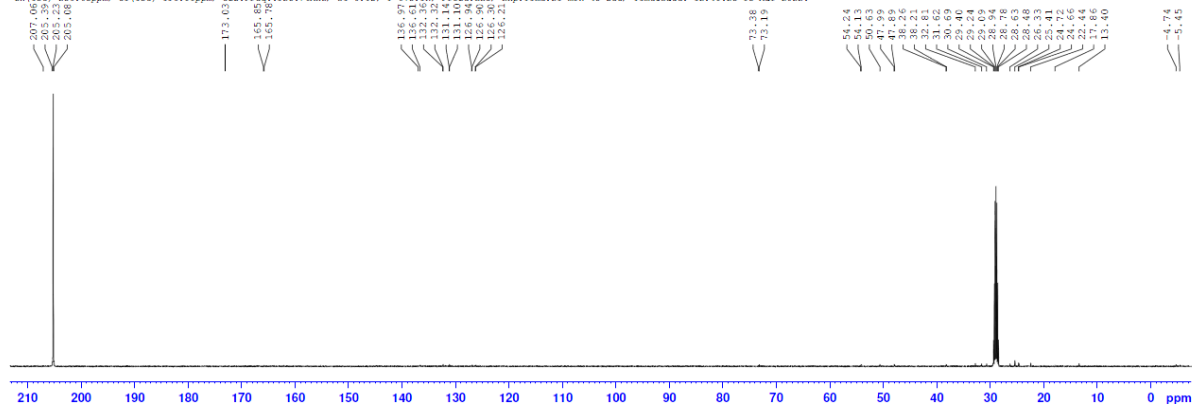


Sp-1374 Makaev ID-PG0X-com 6mg in Acetone,  $^{13}\text{C}$ (1H) com AV500 03.03.2022 BIP

Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS), 2022



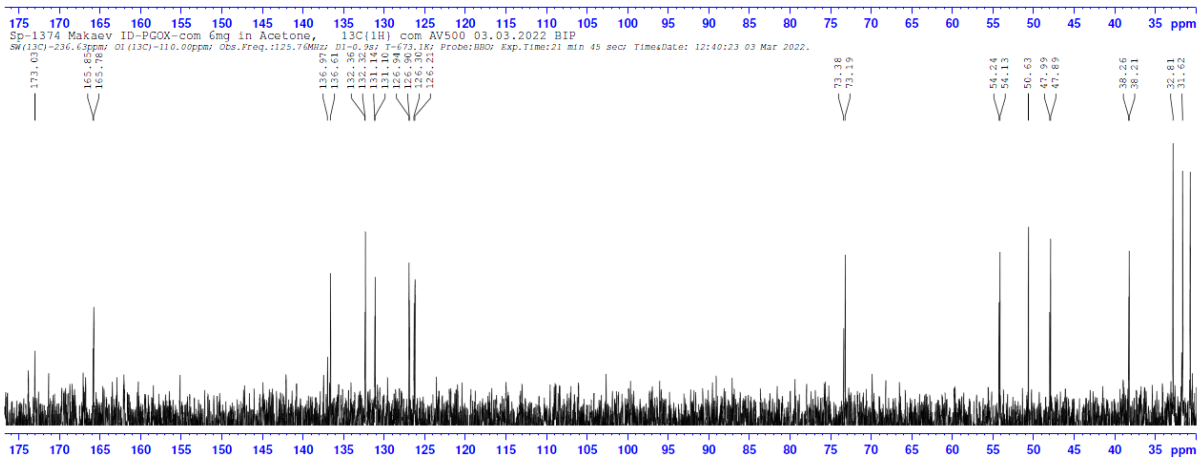
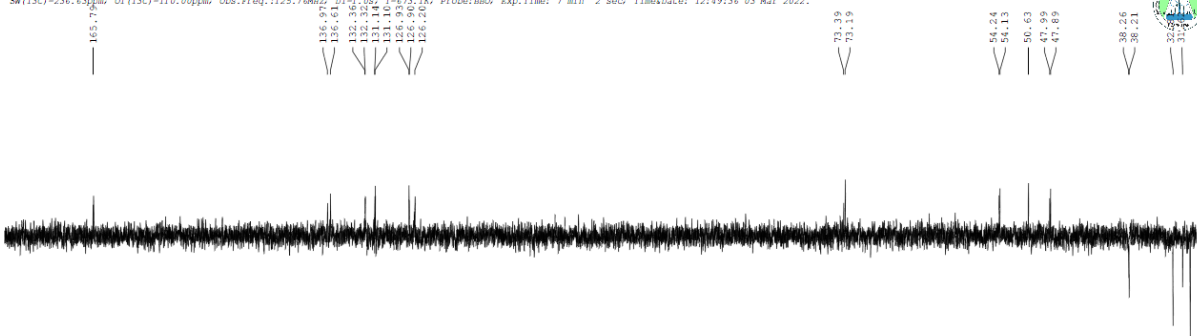
SW(13C)-236.63ppm; 01 (13C)-110.00ppm; Obs.Freq.:125.76MHz; D1-0.9s; T-673.1K; Probe:BB0; Exp.Time:21 min 45 sec; TimesDate: 12:40:23 03 Mar 2022.



Sp-1374 Makaev ID-PGQX-com 6mg in Acetone, <sup>13</sup>C{<sup>1</sup>H} dept135 AV500 03.03.2022 BIP

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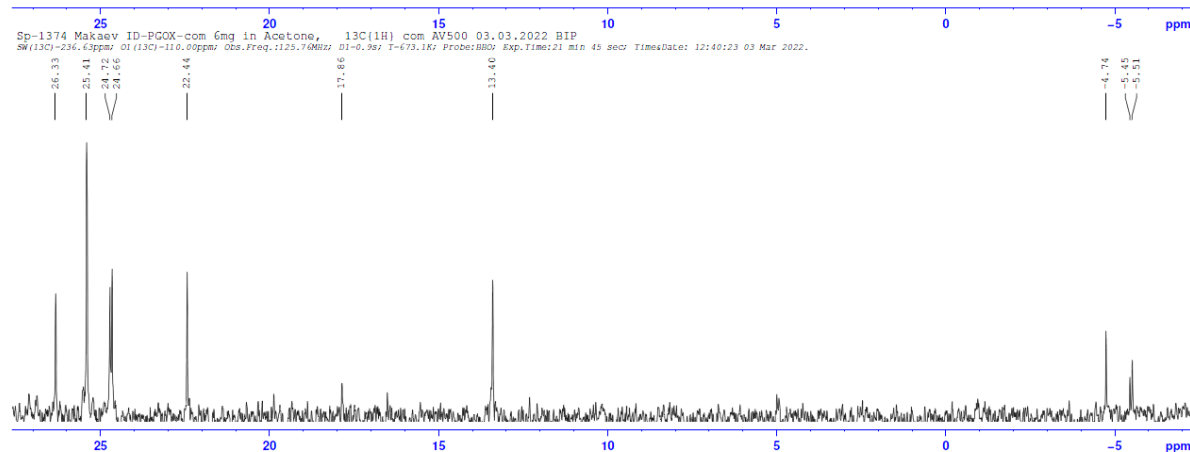
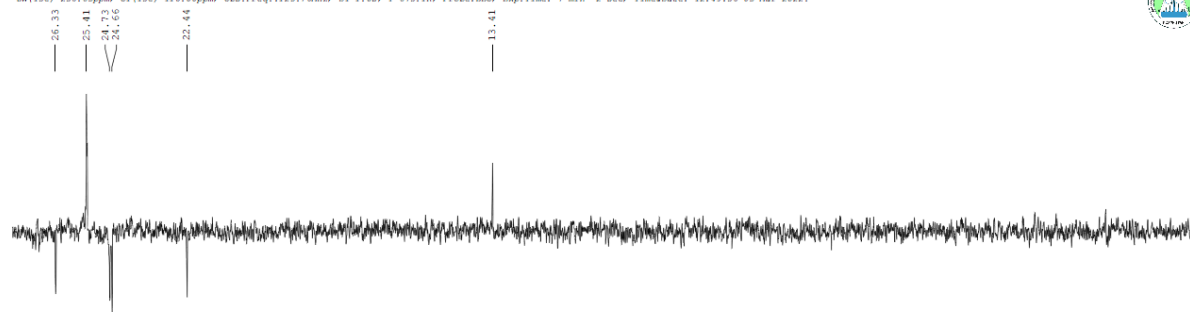
SW(13C)-236.63ppm; 01 (13C)-110.00ppm; Obs.Freq.:125.70MHz; D1=1.0s; T=673.1K; Probe:BBQ; Exp.Time: 7 min 2 sec; TimeDate: 12:49:36 03 Mar 2022.



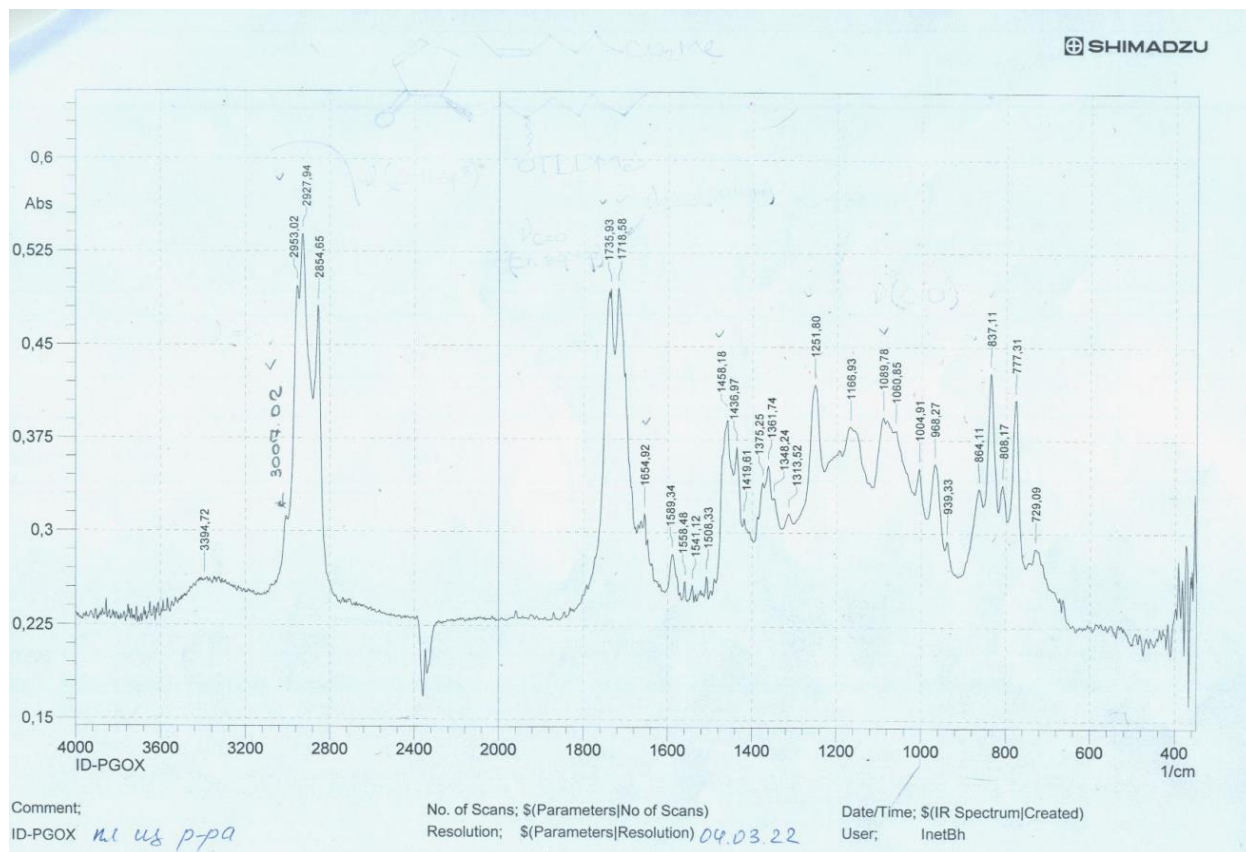
Sp-1374 Makaev ID-PGQX-com 6mg in Acetone, <sup>13</sup>C{<sup>1</sup>H} dept135 AV500 03.03.2022 BIP

Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS), 2022

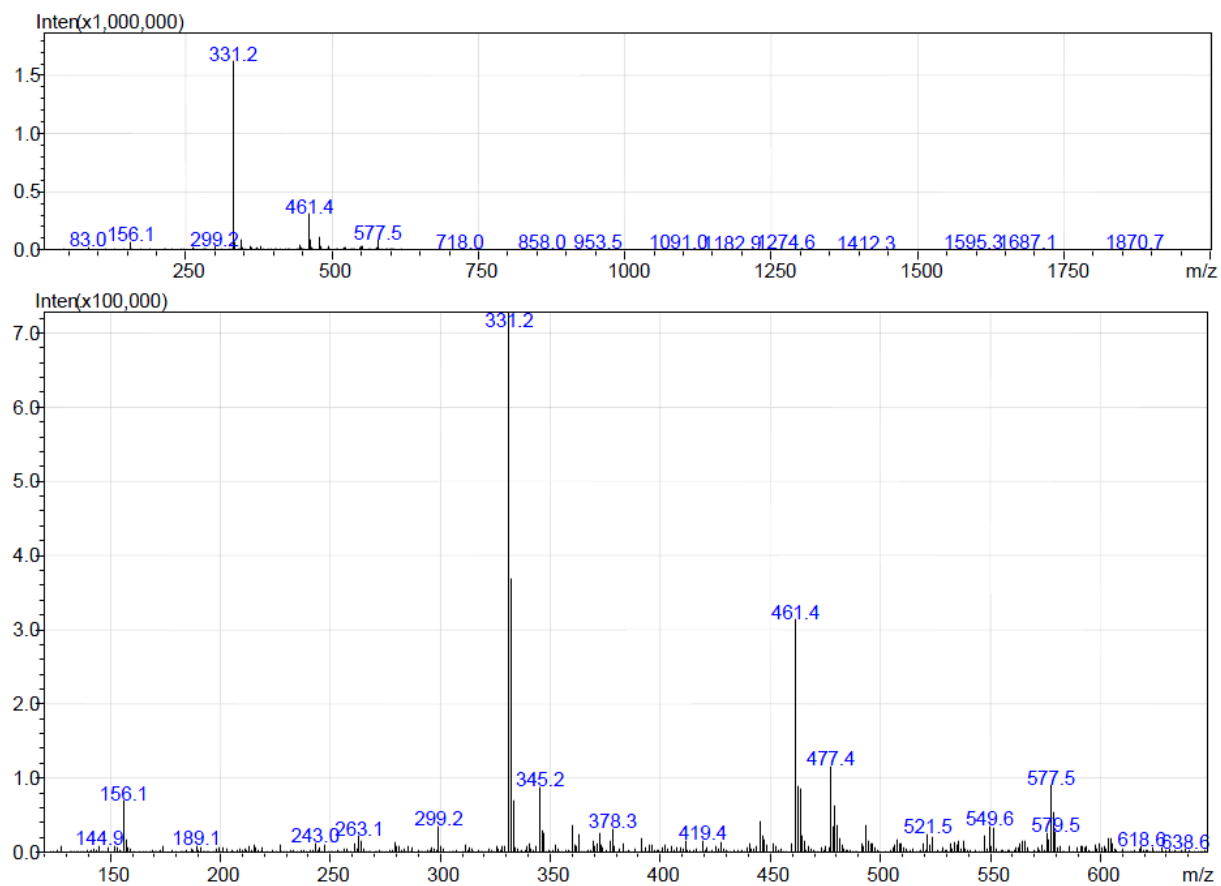
SW(13C)-236.63ppm; 01 (13C)-110.00ppm; Obs.Freq.:125.70MHz; D1=1.0s; T=673.1K; Probe:BBQ; Exp.Time: 7 min 2 sec; TimeDate: 12:49:36 03 Mar 2022.



## IR



## Mass

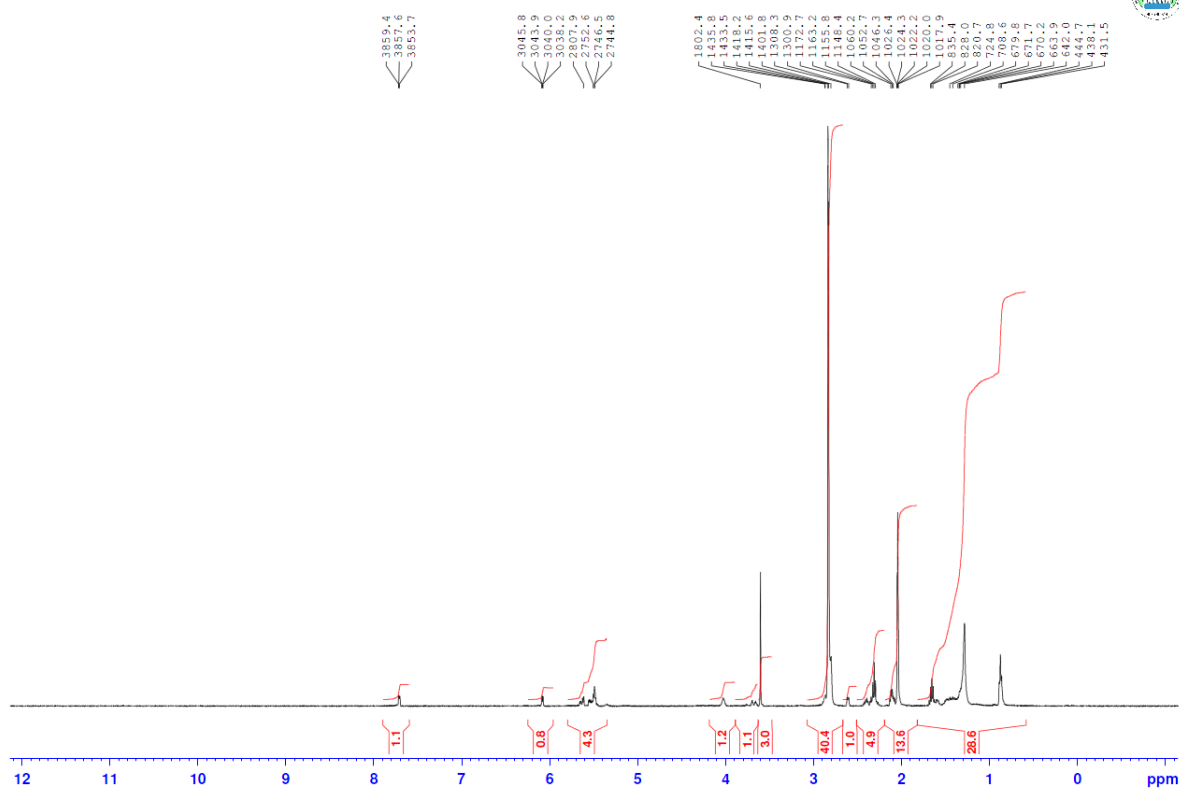


# Methyl (Z)-7-[(1S\*,5R\*)-5-((E)-3-hydroxyoct-1-en-1-yl)-4-oxocyclopent-2-en-1-yl]hept-5-enoate (14).

<sup>1</sup>H NMR

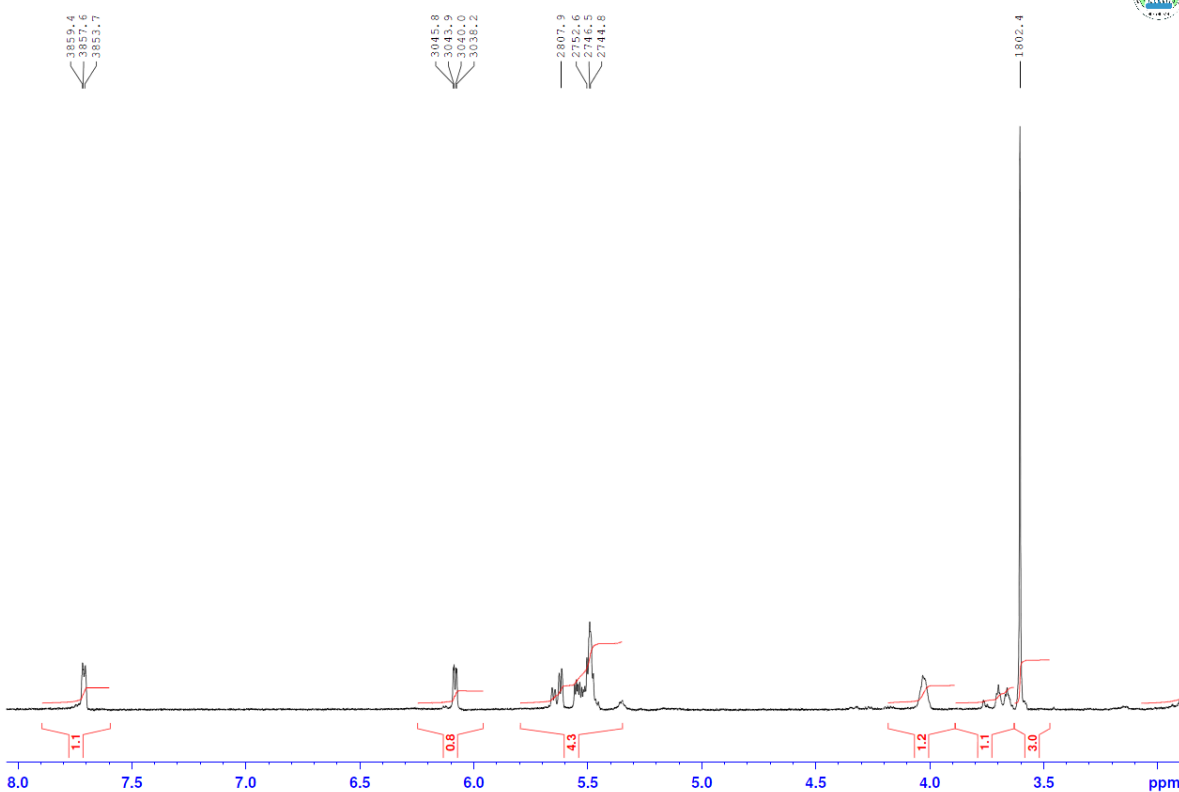
Sp-564 Makaev ID-PSPG 13mg in Acetone, 1H AV500 31.05.2022 NTR  
SW(1H)-19.99ppm; O1(1H)-7.00ppm; Obs.Freq.:500.13MHz; D1-2.0s; T-297.4K; Probe:BBG; Exp.Time:12 sec; TimeDate: 16:55:56 31 May 2022.

Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS). 2022



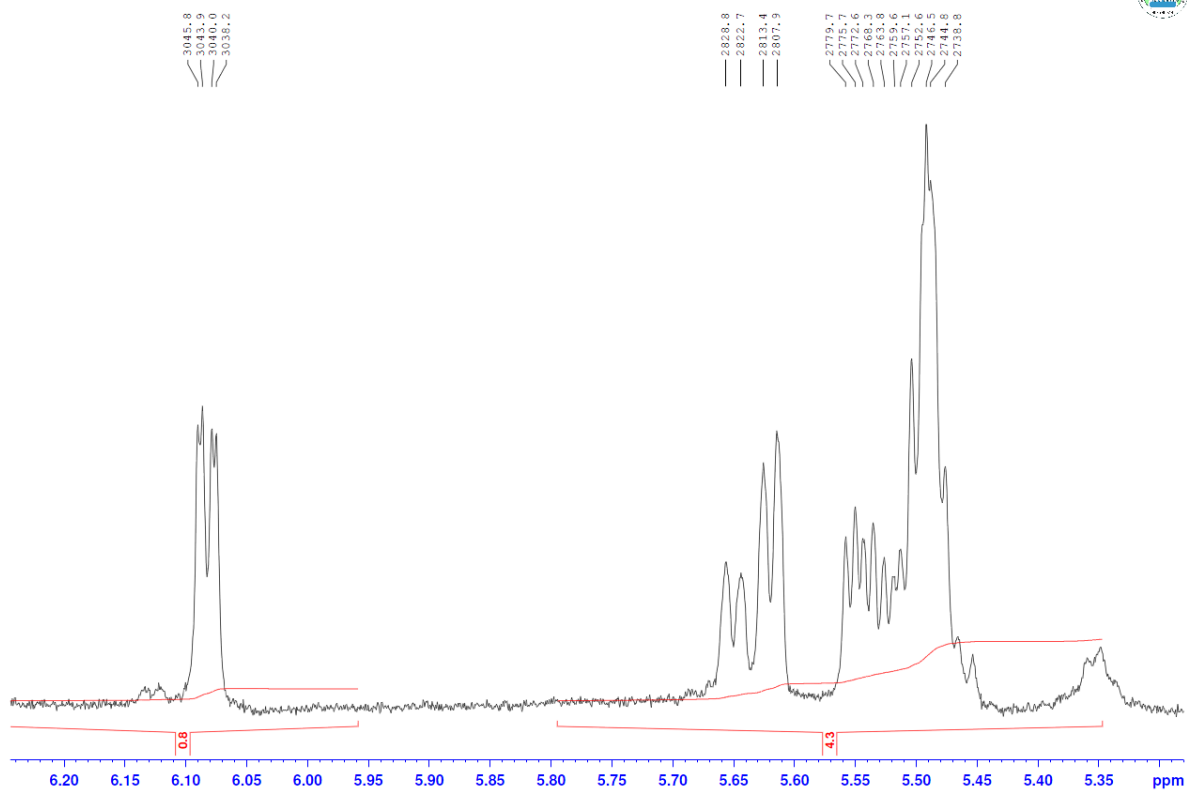
Sp-564 Makaev ID-PSPG 13mg in Acetone, 1H AV500 31.05.2022 NTR  
SW(1H)-19.99ppm; O1(1H)-7.00ppm; Obs.Freq.:500.13MHz; D1-2.0s; T-297.4K; Probe:BBG; Exp.Time:12 sec; TimeDate: 16:55:56 31 May 2022.

Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS). 2022



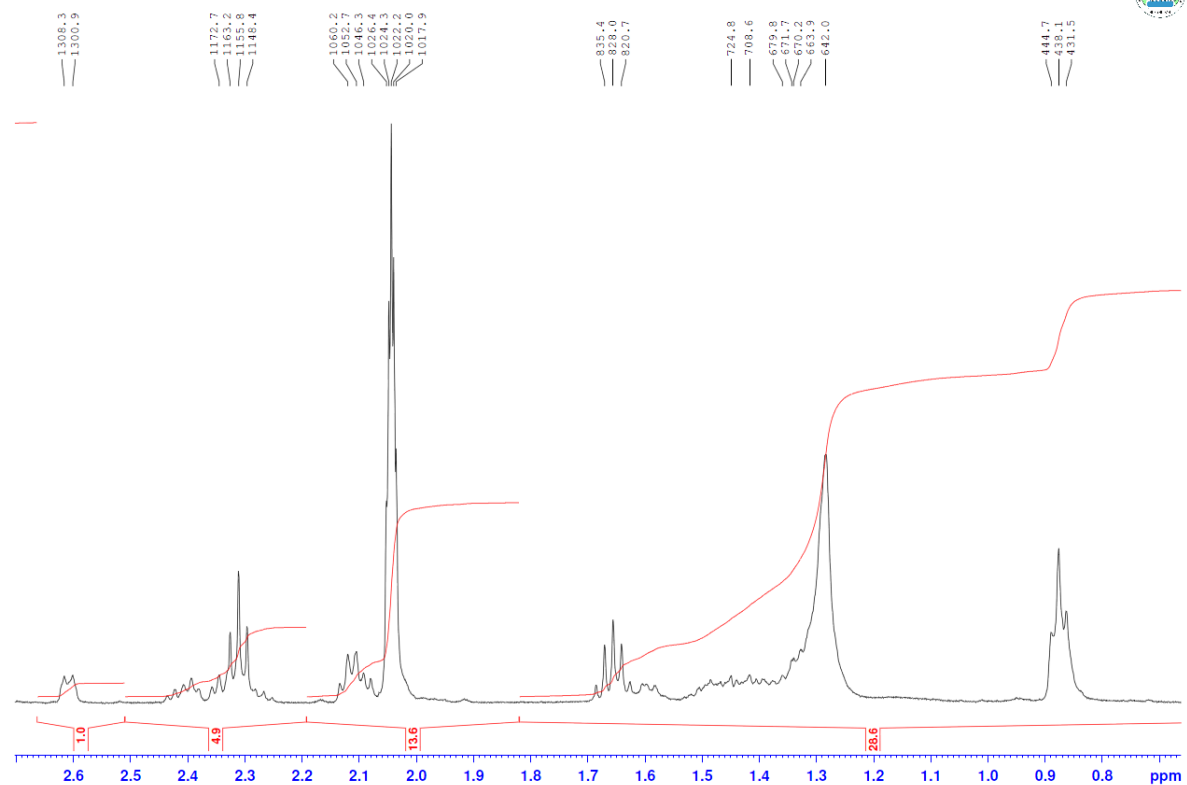
Sp-564 Makaev ID-PSPG 13mg in Acetone, 1H AV500 31.05.2022 NTR  
 SW(H1)=19.99ppm O1(H1)=7.00ppm Obs.Freq.:500.13MHz DI=2.09 T=297.4K Prober:BB0V Exp.Time:12 sec TimeDate: 16:55:56 31 May 2022.

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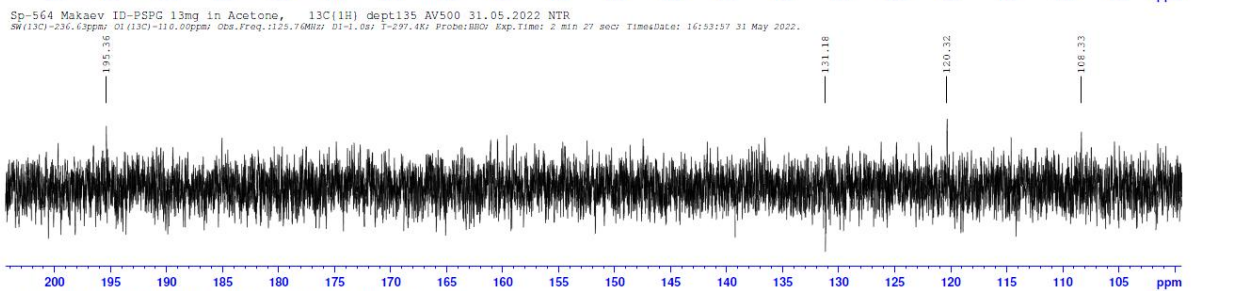
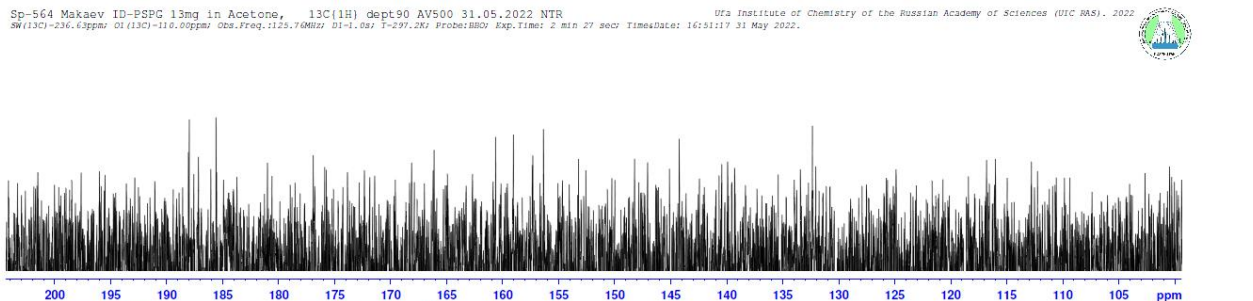
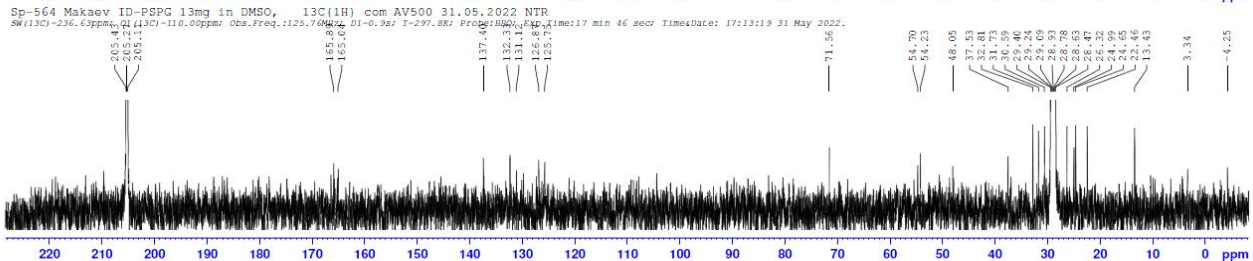
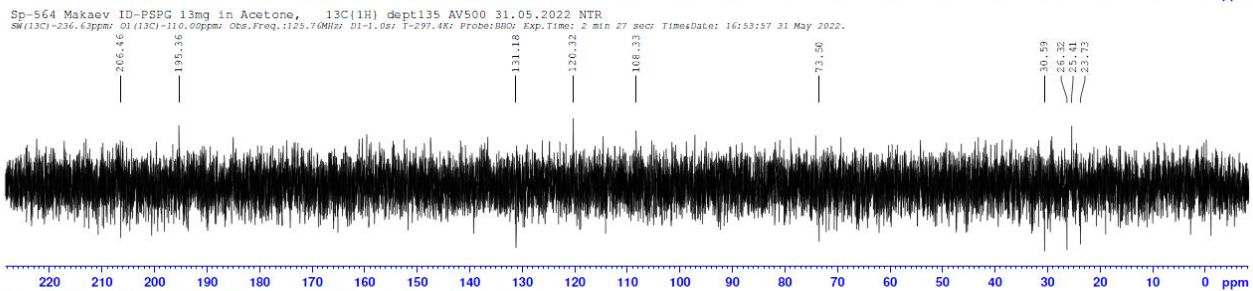
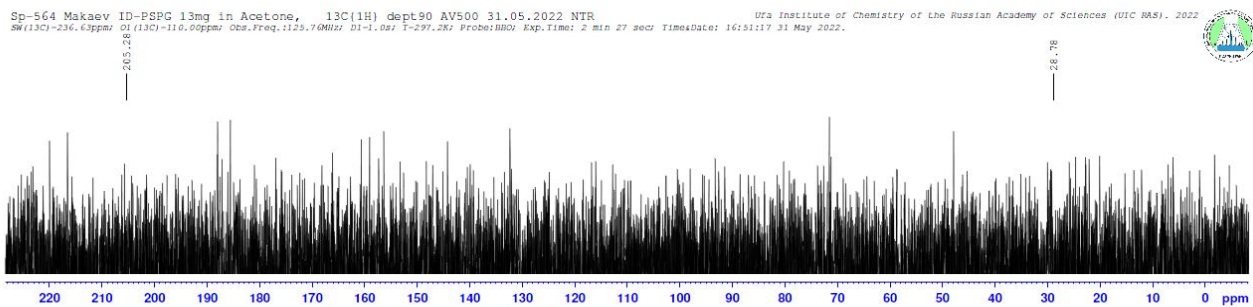


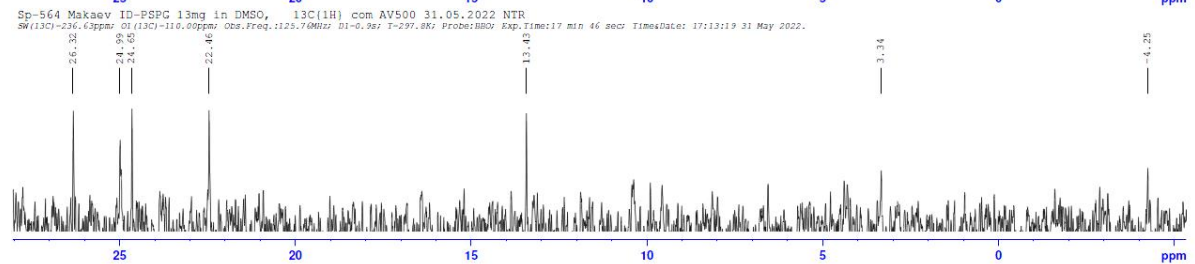
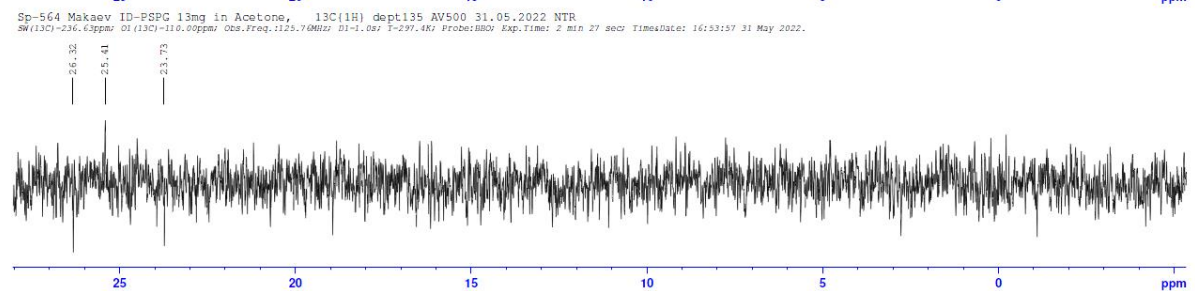
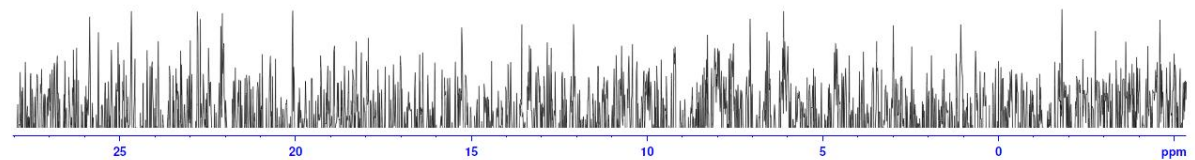
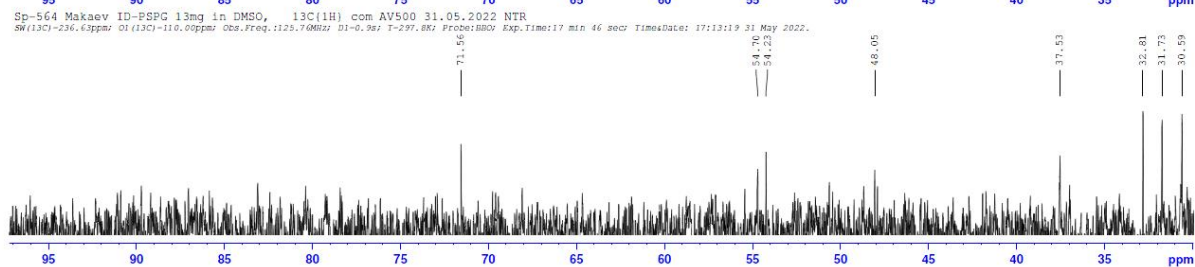
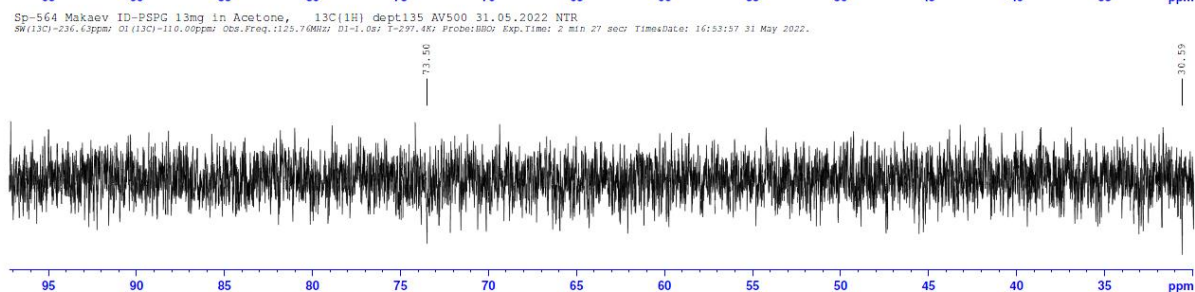
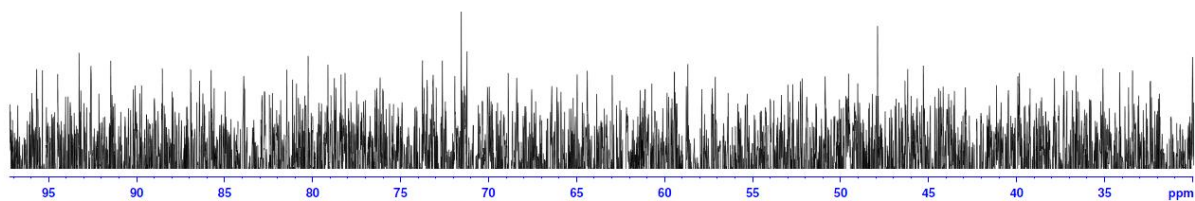
Sp-564 Makaev ID-PSPG 13mg in Acetone, 1H AV500 31.05.2022 NTR  
 SW(H1)=19.99ppm O1(H1)=7.00ppm Obs.Freq.:500.13MHz DI=2.09 T=297.4K Prober:BB0V Exp.Time:12 sec TimeDate: 16:55:56 31 May 2022.

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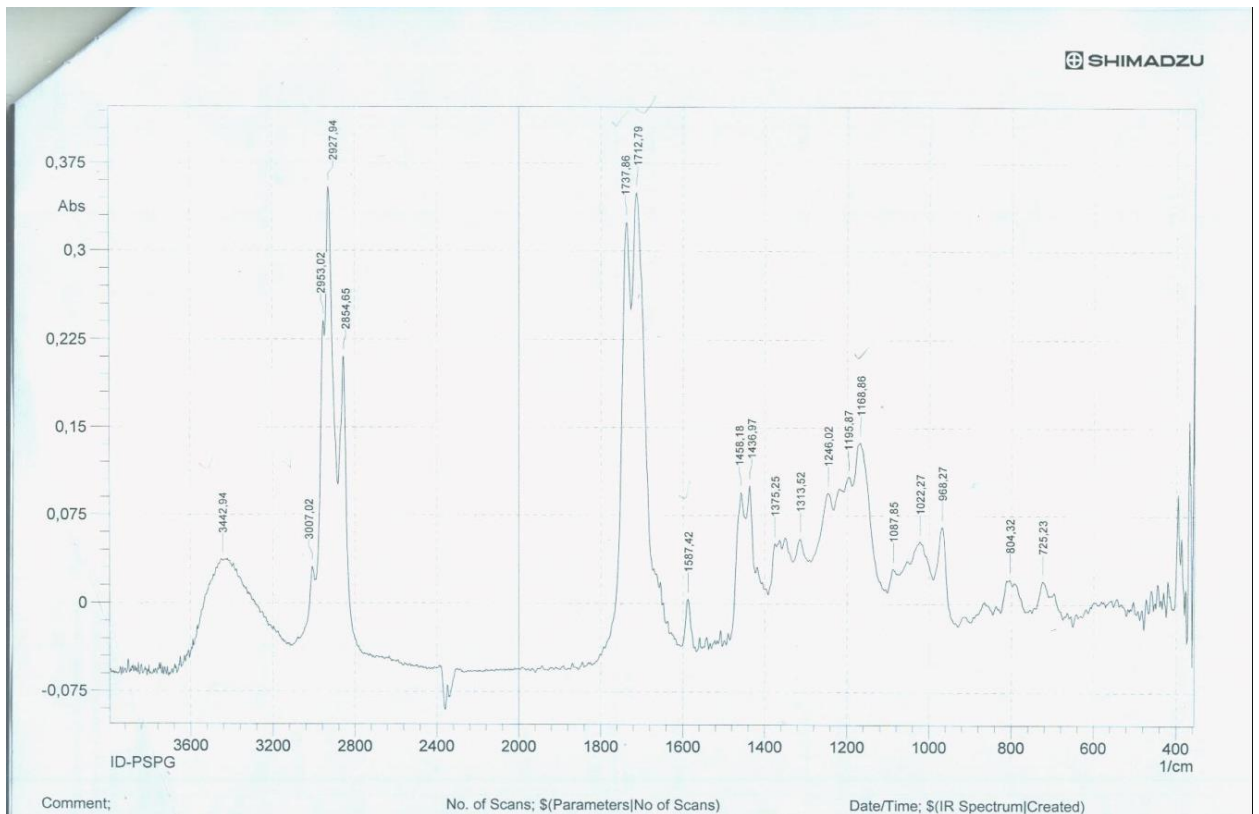


# <sup>13</sup>C NMR

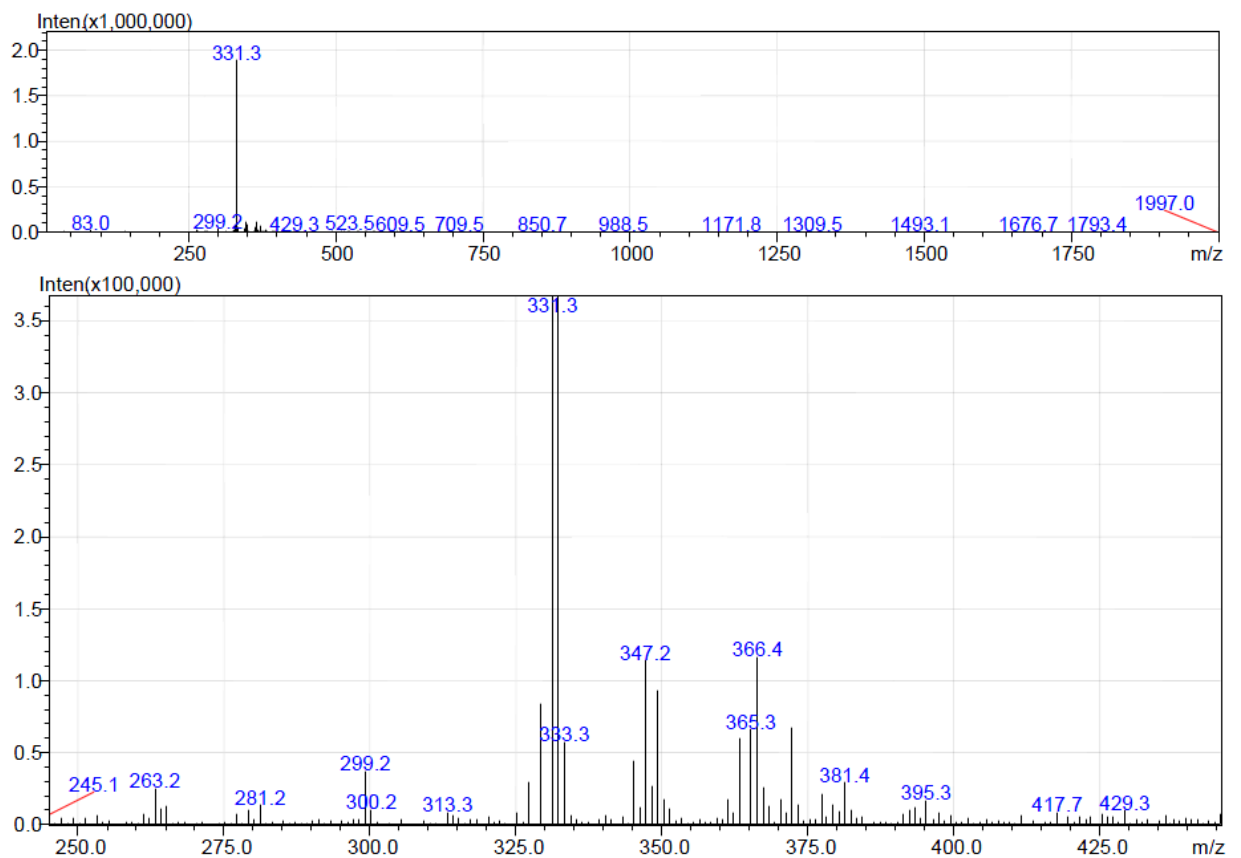




## IR



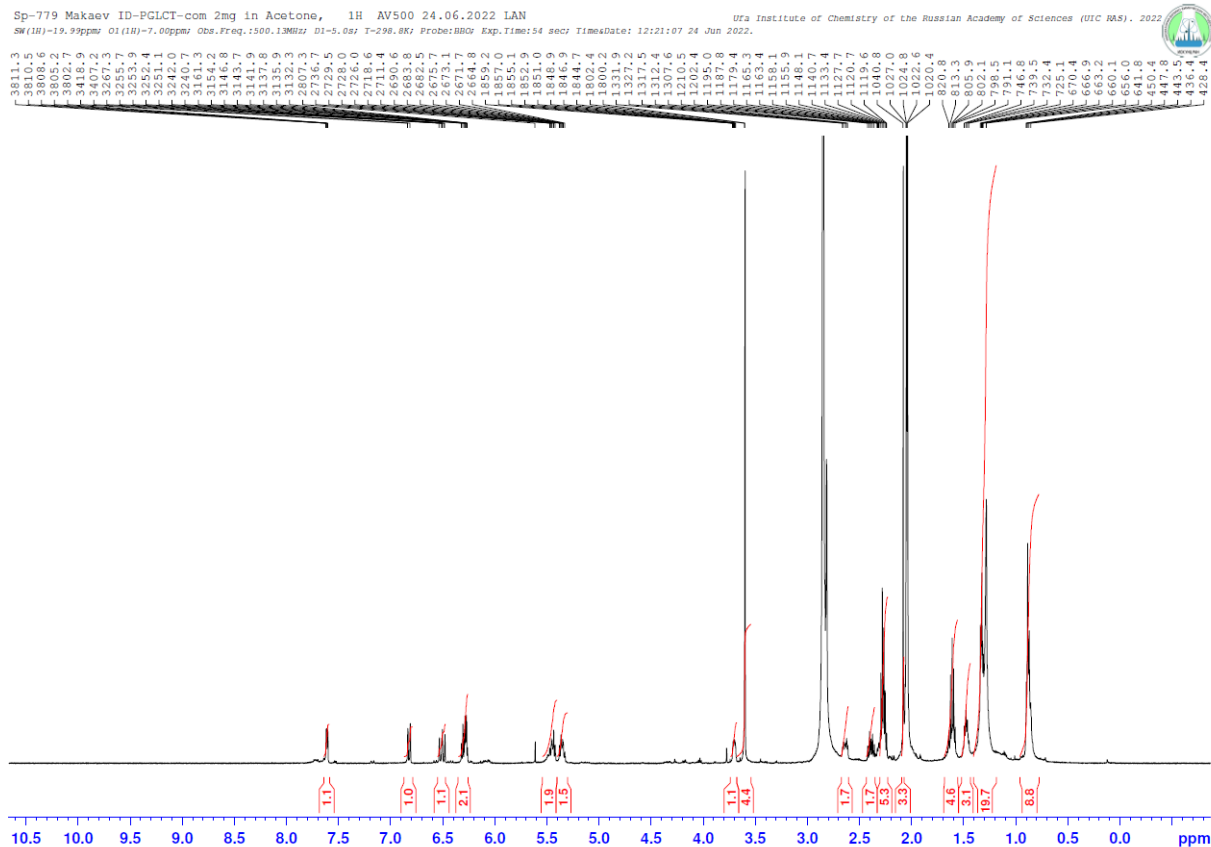
## Mass



# Methyl (±)-(5Z,12E,14E)-11-oxoprost-5,12,14-trien-1-oate (2).

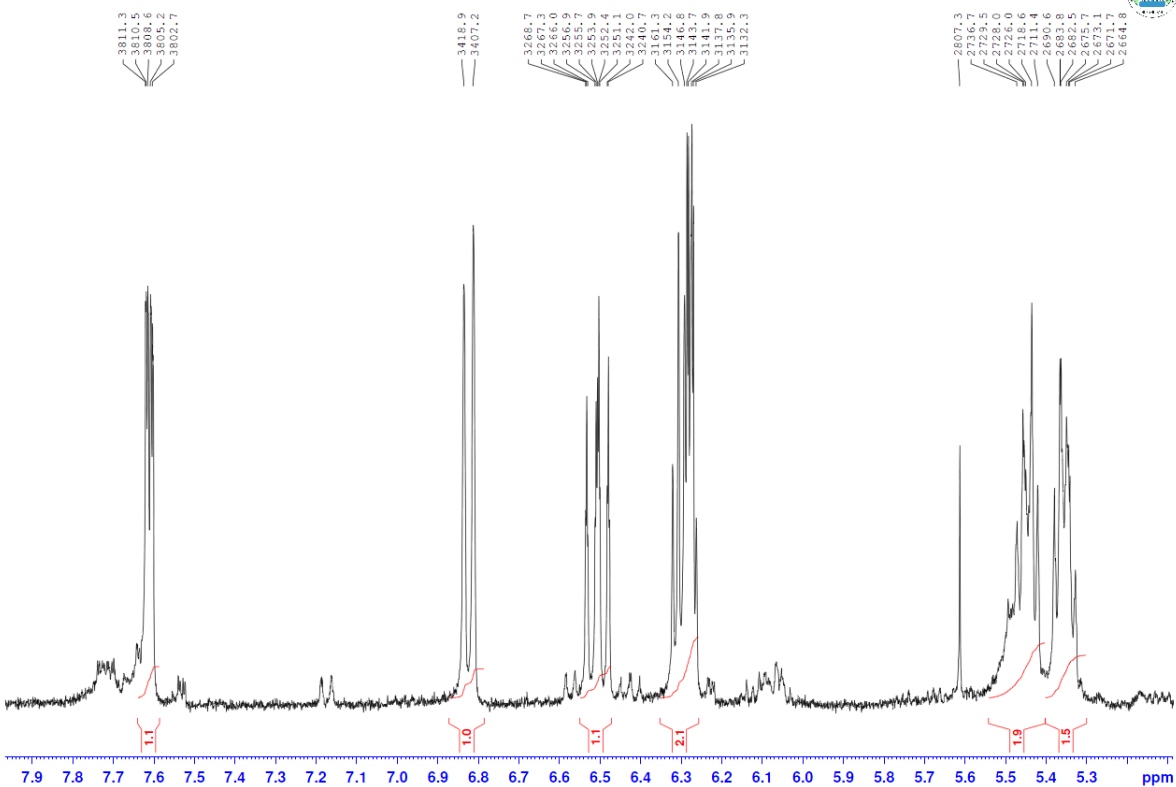
## (15-deoxy- $\Delta^{12,14}$ -Prostaglandin J<sub>2</sub>)

### <sup>1</sup>H NMR



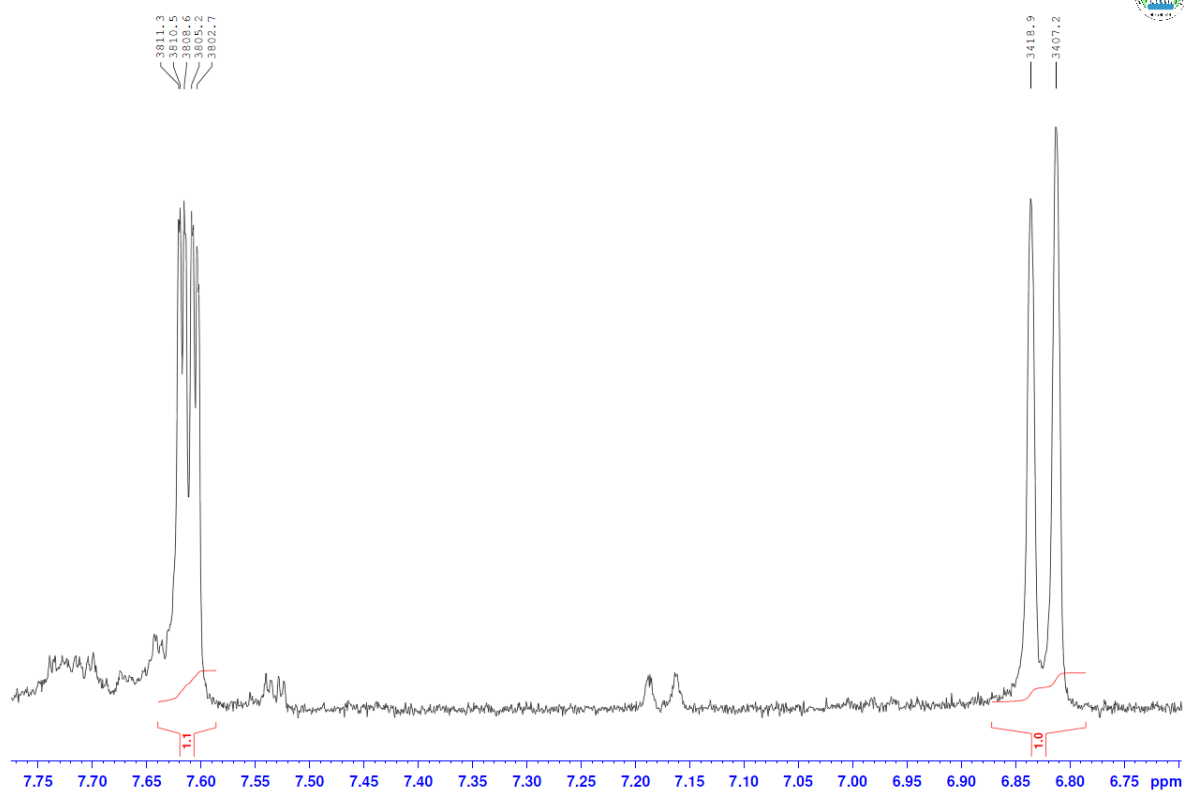
Sp-779 Makaev ID-PGLCT-com 2mg in Acetone, 1H AV500 24.06.2022 LAN

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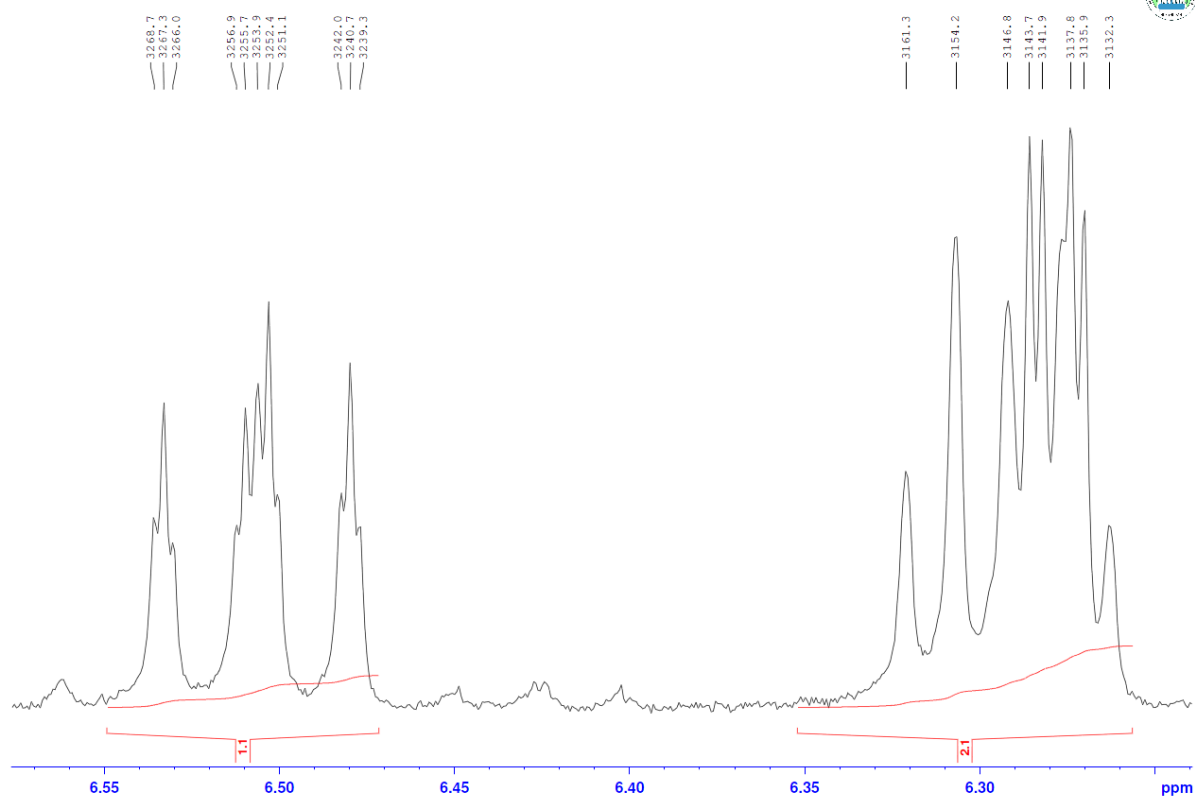
Sp-779 Makaev ID-PGLCT-com 2mg in Acetone, 1H AV500 24.06.2022 LAN

Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS), 2022



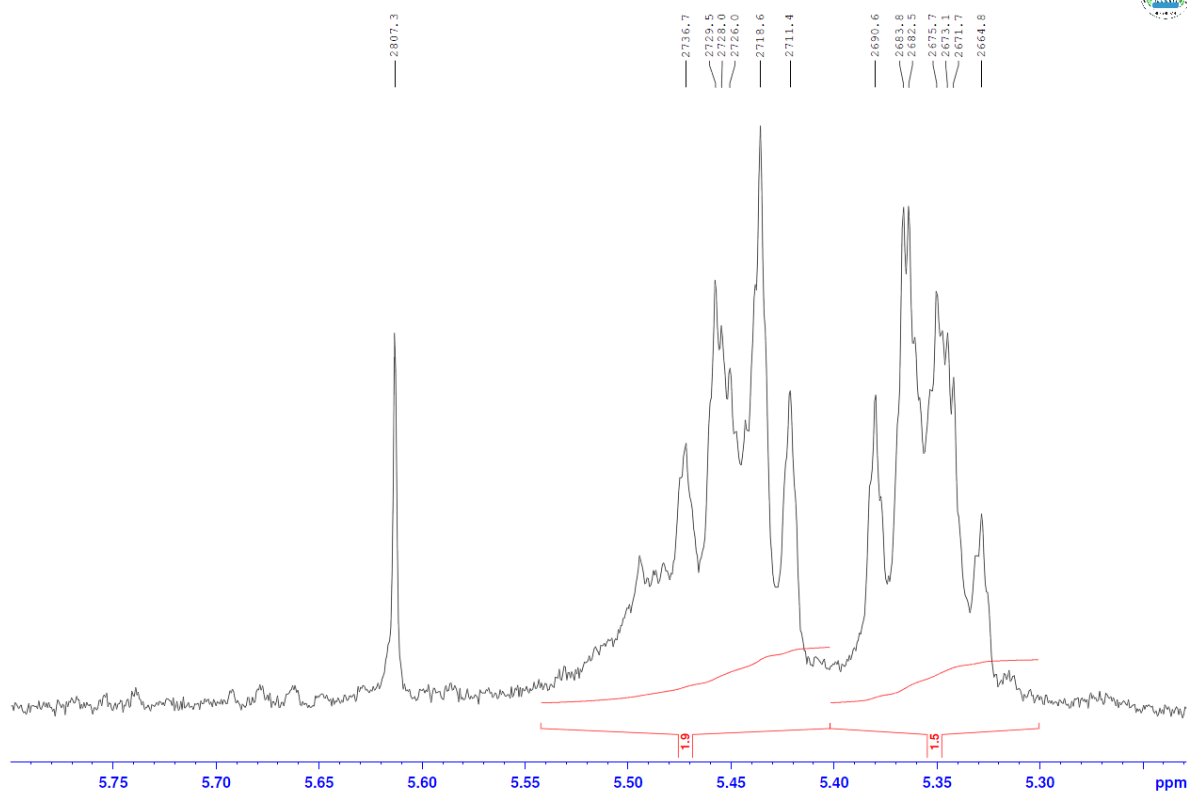
Sp-779 Makaev ID-PGLCT-com 2mg in Acetone, 1H AV500 24.06.2022 LAN  
 SW(1H)-19.99ppm O1(1H)-7.00ppm Obs.Freq.:500.13MHz D1-5.0s T-298.8K Probe:BBG Exp.Time:54 sec TimeDate: 12:21:07 24 Jun 2022.

Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS), 2022



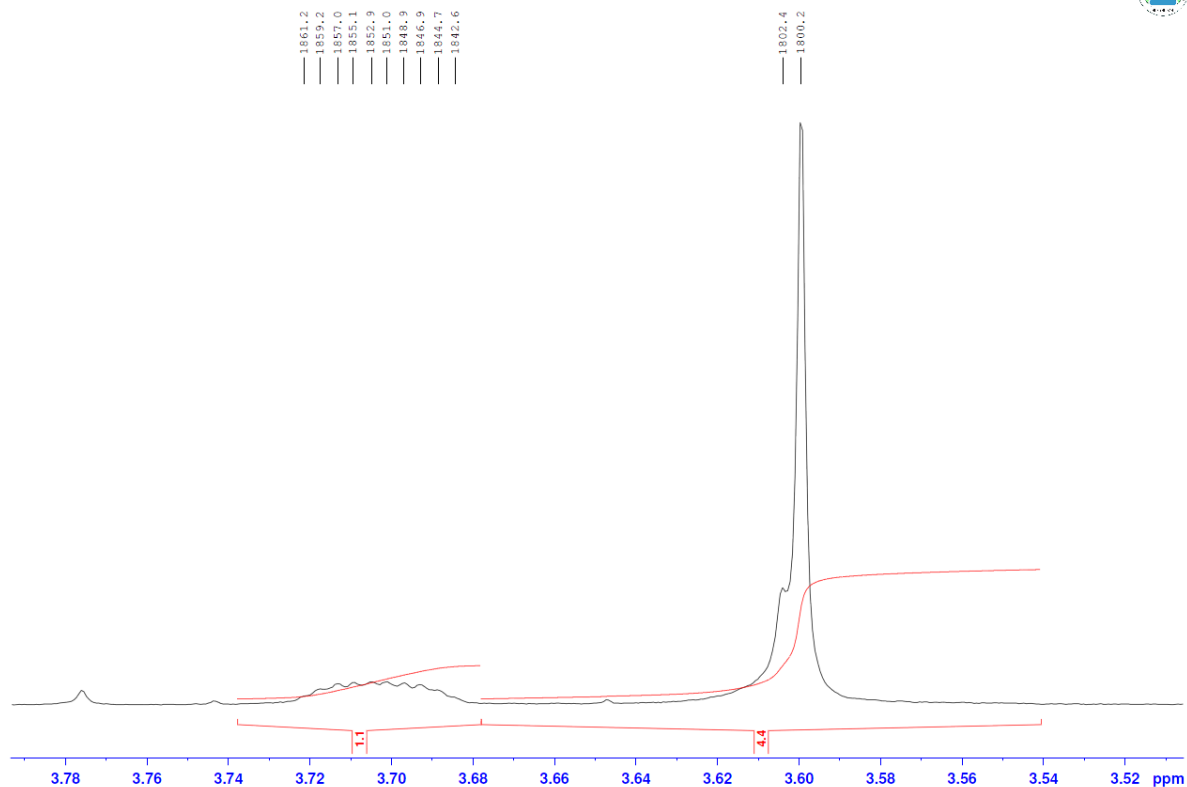
Sp-779 Makaev ID-PGLCT-com 2mg in Acetone, 1H AV500 24.06.2022 LAN  
 SW(1H)-19.99ppm O1(1H)-7.00ppm Obs.Freq.:500.13MHz D1-5.0s T-298.8K Probe:BBG Exp.Time:54 sec TimeDate: 12:21:07 24 Jun 2022.

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Sp-779 Makaev ID-PGLCT-com 2mg in Acetone, 1H AV500 24.06.2022 LAN  
 SW(1H)-19.99ppm; O1(1H)-7.00ppm; Obs.Freq.:500.13MHz; D1-5.0s; T-298.8K; Probe:BBQ; Exp.Time:54 sec; TimesDate: 12:21:07 24 Jun 2022.

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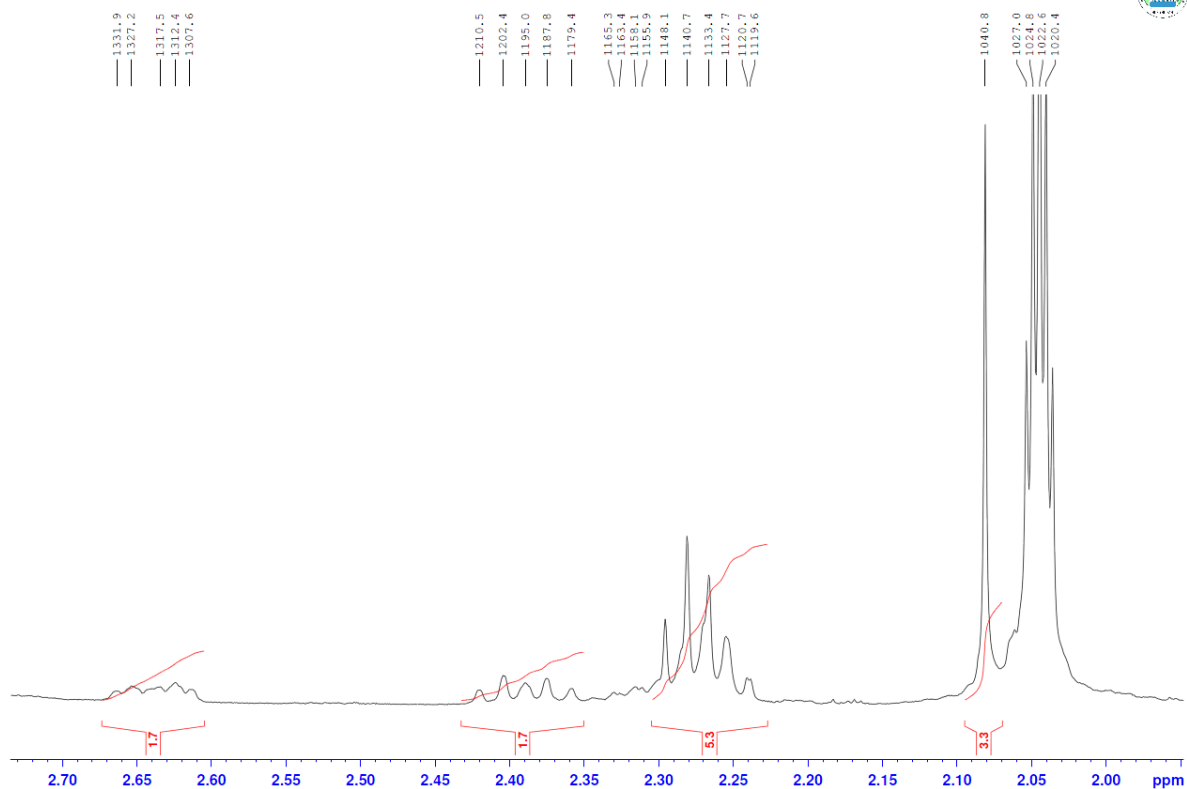


Sp-779 Makaev ID-PGLCT-com 2mg in Acetone, 1H AV500 24.06.2022 LAN

Ufa Institute of Chemistry of the Russian Academy of Sciences (UIC RAS), 2022



SW(1H)-19.99ppm; O1(1H)-7.00ppm; Obs.Freq.:500.13MHz; D1-5.0s; T-298.8K; Probe:BBG; Exp.Time:54 sec; TimeDate: 12:21:07 24 Jun 2022.

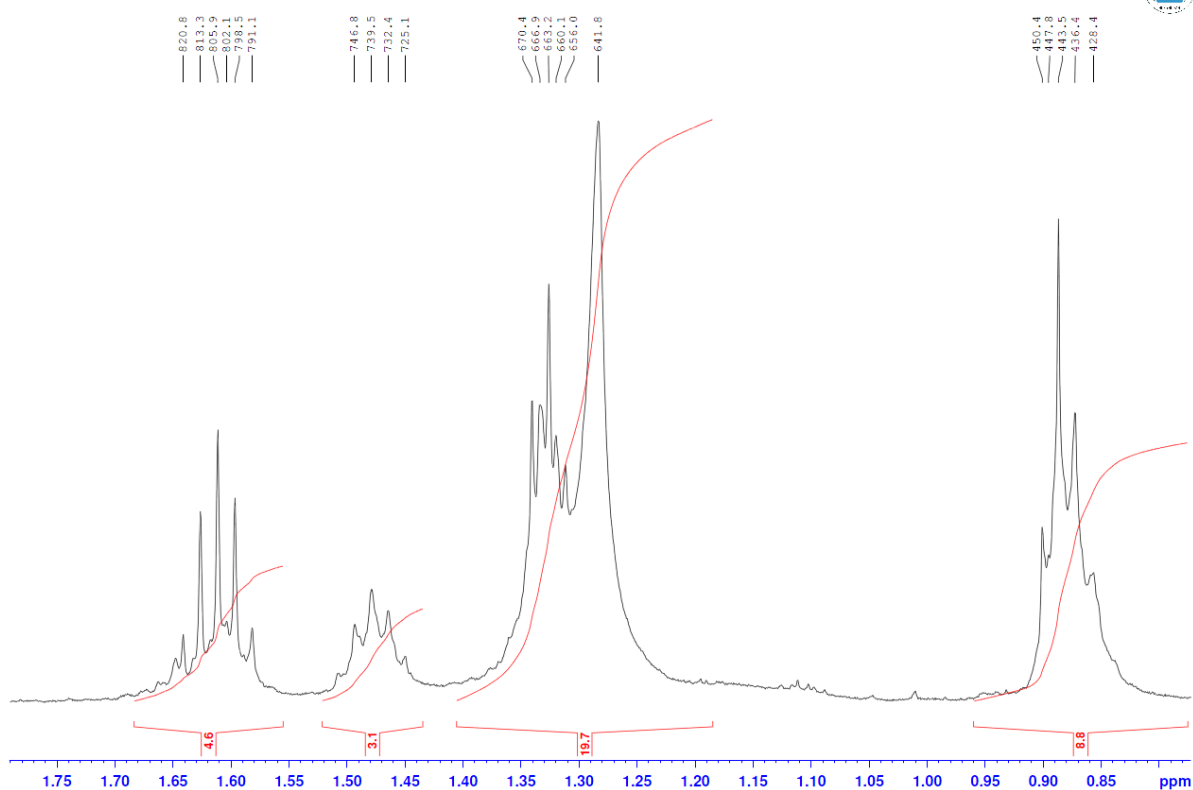


Sp-779 Makaev ID-PGLCT-com 2mg in Acetone, 1H AV500 24.06.2022 LAN

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# <sup>13</sup>C NMR

