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Synthesis and biological evaluation of novel cyanuric acid-tethered tris-pyridinium derivatives

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1. Biological Experiments

1.1 Samples

Samples, quaternary ammonium salts based on pyridine (n = 25) were obtained from the N. D. Zelinsky Institute of Organic Chemistry Russian Academy of Sciences (ZIOC RAS).

1.2 Antibacterial Assay

1.2.1 Strains of microorganisms

Reference strains of microorganisms *Escherichia coli* ATCC 25922, *Klebsiella pneumoniae* ATCC 700603, *Staphylococcus aureus* ATCC 43300, *Acinobacter Baumanii* ATCC 15308 *Pseudomonas aeruginosa* ATCC 27853 were obtained from the State Collection of Pathogenic Microorganisms "SCPM-Obolensk".

1.2.2 Species identification of microorganisms

The species identification of microorganisms was carried out on a MALDI-TOF Biotyper mass spectrometer (Bruker, Germany).

1.2.3 Cultivation of microorganisms

For the cultivation of microorganisms, agar and GRM broth (SRCAMB) were used. Cultivation of microorganisms was carried out for 20-24 hours at a temperature of 37 ºC.

1.2.4 Evaluation of the antibacterial activity of new tris-QACs

The MIC was determined by the micromethod of serial dilutions in broth in the presence of various stepwise concentrations of antibacterial drugs in sterile 96-well plates. Mueller-Hinton broth, Mueller-Hinton agar produced by SRCAMB were used as a nutrient medium.

In the course of the study, two-fold serial dilutions of the studied drugs were used. The mother liquor was prepared in 10% DMSO. Working solutions of the drug and its two-fold dilutions (500→1 mg ml^{-1}) were prepared from the stock solution with the addition of the GRM broth.

Nutrient broth with the appropriate concentration of the test drug was introduced into 10 holes in 10 holes in horizontal rows of 7 test bacterial strains. Broth was added to separate rows without a drug to control the growth of cultures.

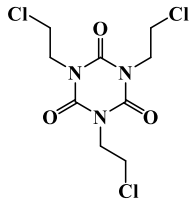
From single colonies grown on the GRM medium at 37 °C for 18 hours, a suspension was prepared with an optical density of 0.5 according to McFarland's standard in sterile saline, which corresponded to approximately 1-2 × 10^8 CFU*ml^{-1}. Then the suspension was diluted 1:100 by adding 0.2 ml of the suspension to a flask containing 19.8 ml of Mueller-Hinton broth. The concentration of microorganisms in this case was 106 CFU*ml^{-1}. 0.1 ml of the original suspension was introduced into the wells with the test drug and control wells with broth. The final concentration of microorganism in each well was 5x105 CFU*ml^{-1}. The plates were covered with lids and placed in a thermostat (37 °C) for 20 hours. The presence of bacterial growth was taken into account visually (by the presence of turbidity in the well). The minimum inhibitory concentration (MIC) was taken as the minimum concentration of the preparation at which the growth of bacteria was absent after 20 h of incubation. The minimum bactericidal concentration (MBC) was determined based on the results of seeding on solid nutrient media. For this, from all the wells in which there was no visible growth (by the presence of turbidity), 10 μl were seeded on Mueller-Hinton nutrient agar. The results were taken into account by the presence of culture growth at the site of application after 24 hours of incubation at 37 °C. If there was no growth in the well, but at the same time, the growth of the studied culture was observed when sowing from this well on a dense nutrient medium, then this concentration was taken as bacteriostatic. The MBC was taken as the lowest concentration at which cell growth was completely inhibited when plated on a solid nutrient medium.

2. Chemical Experimental Details

Reagents were purchased and used without further purification. Reactions were monitored by TLC on silica gel 60 F254 aluminum sheets under UV light. All products were analyzed by NMR spectroscopy on Bruker AM300, Bruker XWIND or Bruker AV600 spectrometers at ambient temperature in CD_3OD or DMSO-d_6 or $CDCl_3$. All melting points were determined on a Gallenkamp melting point apparatus in open capillaries and are uncorrected. IR spectra were recorded with a Bruker ALPHA-T FT-IR spectrometer in KBr pellets. The ESI contains detailed experimental protocols, summaries of spectral data, and spectra.

3. Synthesis and Characterization of Substrates and Products

Synthesis of 1,3,5-tris(2-chloroethyl)-1,3,5-triazinane-2,4,6-trione (2):

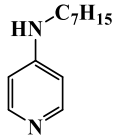


Thionyl chloride (7.85 g, 66 mmol) was slowly added to the solution of 1,3,5-tris(2-hydroxy-ethyl)-1,3,5-triazinane-2,4,6-trione (2.61 g, 20 mmol) in benzene (50 ml). The resulting mixture was heated under reflux for 10 hours. The solvent was removed under reduced pressure. The crude residue was purified by recrystallization from hexane to afford a white solid product (2.26 g, 19.2 mmol, 99% yield). $C_9H_{12}Cl_3N_3O_3$; M. w. 316.56; White solid; M. p. 96-97°C [1]; ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.78 (t, J = 6.4 Hz, 6H, $3CH_2$), 4.31 (t, J = 6.4 Hz, 6H, $3CH_2$) ppm.

Synthesis of 4-alkylaminopyridines 4a-f:

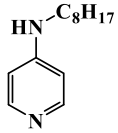
Thionyl chloride (4.72 ml, 65 mmol) was slowly added to a solution of alkanoic acid (50 mmol) and DMF (0.39 ml, 5 mmol) in toluene (70 ml). The resulting mixture was heated under reflux for 3 hours. The solvent was removed under reduced pressure. The obtained brown oil was dissolved in CH_2Cl_2 (10 ml) and then slowly added to a mixture of 4-aminopyridine (0.8 equiv.), triethylamine (1.2 equiv.) and CH_2Cl_2 (30 ml). The resulting mixture was heated under reflux for 2 hours, then cooled and washed with water (3×100 ml). The solvent from organic layer was removed under reduced pressure, and the residue was stripped to dryness. The obtained beige powder was dissolved in dry THF (60 ml) and slowly added to a suspension of lithium aluminium hydride (2 eq.) in dry THF (40 ml). The resulting mixture was heated under reflux for 8 hours. Water (3 ml), sodium hydroxide (25% water solution, 1 ml), then water (2 ml) were added successively while stirring. The precipitate was filtered, boiled in ethylacetate (50 ml) and filtered again. The solvent from combined organic filtrates was removed under reduced pressure. The crude residue was purified by recrystallization from hexane to provide yellowish solid product. The total yield was 52-86%.

4-Heptylaminopyridine (4a):



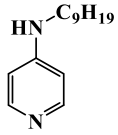
$C_{12}H_{20}N_2$; M. w. 192.31; Yellow solid (5.00 g, 26 mmol, 65% yield); M. p. 49-51°C [2]; ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.89 (t, J = 6.6 Hz, 3H, CH_3), 1.19-1.45 (m, 8H, $4CH_2$), 1.54-1.74 (m, 2H, CH_2), 3.11-3.21 (m, 2H, CH_2NH), 4.53 (t, J = 5.2 Hz, 3H, 3NH), 6.47 (d, J = 6.2 Hz, 2H, $2CH_{py}$), 8.16 (d, J = 6.2 Hz, 2H, $2CH_{py}$), ppm.

4-Octylaminopyridine (4b):



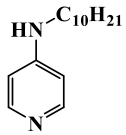
$C_{13}H_{22}N_2$; M. w. 206.33; Yellow solid (4.29 g, 20.8 mmol, 52% yield); M. p. 70-73°C [2]; ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.90 (t, J = 6.6 Hz, 3H, CH_3), 1.18-1.47 (m, 10H, $5CH_2$), 1.54-1.74 (m, 2H, CH_2), 3.14-3.20 (m, 2H, CH_2NH), 4.52 (t, J = 5.2 Hz, 3H, 3NH), 6.47 (d, J = 6.2 Hz, 2H, $2CH_{py}$), 8.16 (d, J = 6.2 Hz, 2H, $2CH_{py}$) ppm.

4-Nonylaminopyridine (4c):



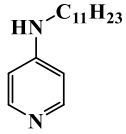
$C_{14}H_{24}N_2$; M. w. 220.36; Yellow solid (6.43 g, 29.2 mmol, 73% yield); M. p. 55-57°C [2]; ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.90 (t, J = 6.6 Hz, 3H, CH_3), 1.18-1.45 (m, 12H, $6CH_2$), 1.53-1.76 (m, 2H, CH_2), 3.11-3.21 (m, 2H, CH_2NH), 4.53 (t, J = 5.2 Hz, 3H, 3NH), 6.46 (d, J = 6.2 Hz, 2H, $2CH_{py}$), 8.15 (d, J = 6.2 Hz, 2H, $2CH_{py}$) ppm.

4-Decylaminopyridine (4d):



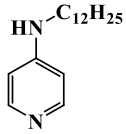
$C_{15}H_{26}N_2$; M. w. 234.39; Yellow solid (5.63 g, 24.0 mmol, 60% yield); M. p. 71-73°C [2]; ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.91 (t, J = 6.6 Hz, 3H, CH_3), 1.20-1.47 (m, 14H, $7CH_2$), 1.54-1.74 (m, 2H, CH_2), 3.13-3.19 (m, 2H, CH_2NH), 4.53 (t, J = 5.2 Hz, 3H, 3NH), 6.46 (d, J = 6.2 Hz, 2H, $2CH_{py}$), 8.16 (d, J = 6.2 Hz, 2H, $2CH_{py}$) ppm.

4-Undecylaminopyridine (4e):



$C_{16}H_{28}N_2$; M. w. 248.41; Yellow solid (8.55 g, 34.4 mmol, 86% yield); M. p. 47-50°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.89 (t, J = 6.6 Hz, 3H, CH_3), 1.19-1.47 (m, 16H, $8CH_2$), 1.57-1.75 (m, 2H, CH_2), 3.11-3.20 (m, 2H, CH_2NH), 4.53 (t, J = 5.2 Hz, 3H, 3NH), 6.46 (d, J = 6.2 Hz, 2H, $2CH_{py}$), 8.16 (d, J = 6.2 Hz, 2H, $2CH_{py}$) ppm.

4-Dodecylaminopyridine (4f):

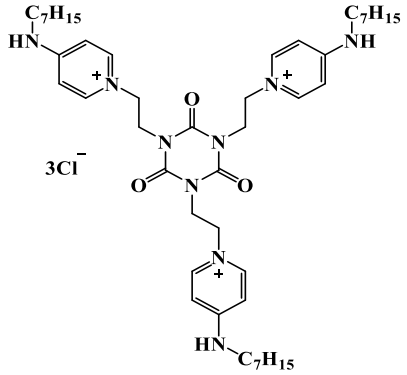


$C_{17}H_{29}N_2$; M. w. 262.44; Yellow solid (7.24 g, 27.6 mmol, 69% yield); M. p. 83-85°C [2]; ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.90 (t, J = 6.6 Hz, 3H, CH_3), 1.19-1.47 (m, 18H, $9CH_2$), 1.54-1.74 (m, 2H, CH_2), 3.09-3.20 (m, 2H, CH_2NH), 4.52 (t, J = 5.2 Hz, 3H, 3NH), 6.46 (d, J = 6.2 Hz, 2H, $2CH_{py}$), 8.16 (d, J = 6.2 Hz, 2H, $2CH_{py}$) ppm.

Synthesis of 1,1',1''-[(2,4,6-trioxo-1,3,5-triazinane-1,3,5-triyl)tris(ethane-2,1-diyl)]-tris(4-alkylaminopyridin-1-ium) trichlorides 5a-f.

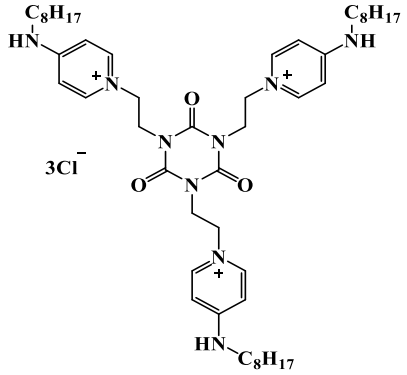
A mixture of 1,3,5-tris(2-chloroethyl)-1,3,5-triazinane-2,4,6-trione (0.32 g, 1 mmol), 4-alkylaminopyridine (3.3 mmol) and butan-1-ol (6 ml) was heated under reflux for 3 days. The solvent was removed under reduced pressure. The crude residue was washed with acetone and dried to provide a white solid product. The yield was 80-93%.

1,1',1''-[(2,4,6-Trioxo-1,3,5-triazinane-1,3,5-triyl)tris(ethane-2,1-diyl)]-tris(4-heptylamino-pyridin-1-ium) trichloride (5a):



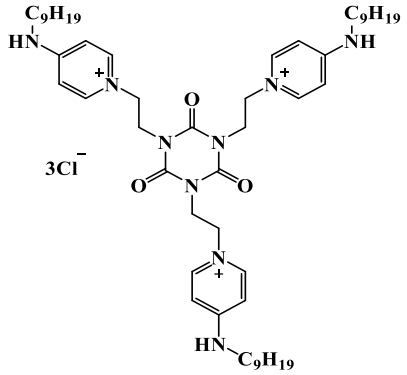
$C_{45}H_{72}Cl_3N_9O_3$; M. w. 893.48; White solid (0.79 g, 0.88 mmol, 88% yield); M. p. 116-119°C; ^{1}H NMR (300 MHz, DMSO-d_6): δ 0.86 (t, J = 6.9 Hz, 9H, $3CH_3$), 1.16-1.39 (m, 54H, $27CH_2$), 1.50-1.62 (m, 6, $3CH_2$), 3.21-3.33 (m, 6H, $3CH_2NH$), 4.01-4.15 (m, 6H, $3CH_2N$), 4.28-4.39 (m, 6H, $3CH_2N^+$), 6.89 (dd, J = 7.0, 2.6 Hz, 3H, $3CH_{py}$), 7.03 (dd, J = 7.0, 2.6 Hz, 3H, $3CH_{py}$), 8.26 (d, J = 7.0 Hz, 3H, $3CH_{py}$), 8.43 (d, J = 7.0 Hz, 3H, $3CH_{py}$), 9.20 (t, J = 5.2 Hz, 3H, 3NH) ppm; ^{13}C NMR (75 MHz, MeOD): δ 14.4, 23.6, 27.9, 29.4, 30.0, 32.9, 44.0, 44.4, 56.6, 106.6, 111.9, 142.9, 145.3, 150.4, 158.8 ppm; ν_{max}, (KBr): 3434, 2957, 2926, 2855, 1691, 1656, 1464, 1200, 842, 762 cm^{-1}

1,1',1''-[(2,4,6-Trioxo-1,3,5-triazinane-1,3,5-triyl)tris(ethane-2,1-diyl)]-tris(4-octylaminopyridin-1-ium) trichloride (5b):



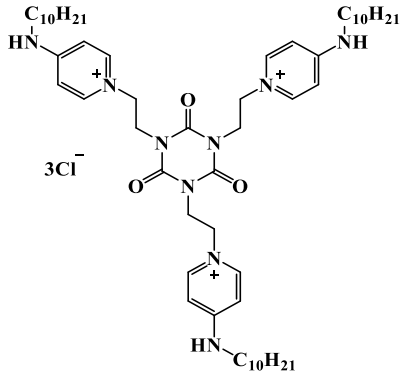
$C_{48}H_{78}Cl_3N_9O_3$; M. w. 935.56; White solid (0.87 g, 0.93 mmol, 93% yield); M. p. 130-133°C; ^{1}H NMR (300 MHz, DMSO-d_6): δ 0.86 (t, J = 6.9 Hz, 9H, $3CH_3$), 1.18-1.37 (m, 54H, $27CH_2$), 1.50-1.62 (m, 6, $3CH_2$), 3.19-3.30 (m, 6H, $3CH_2NH$), 4.01-4.11 (m, 6H, $3CH_2N$), 4.29-4.36 (m, 6H, $3CH_2N^+$), 6.89 (dd, J = 7.0, 2.6 Hz, 3H, $3CH_{py}$), 7.03 (dd, J = 7.0, 2.6 Hz, 3H, $3CH_{py}$), 8.26 (d, J = 7.0 Hz, 3H, $3CH_{py}$), 8.43 (d, J = 7.0 Hz, 3H, $3CH_{py}$), 9.18 (t, J = 5.2 Hz, 3H, 3NH) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): δ 13.8, 21.9, 26.2, 27.8, 28.5, 28.6, 31.1, 42.1, 43.1, 54.8, 104.9, 110.0, 141.6, 144.0, 148.7, 156.6 ppm; ν_{max}, (KBr): 3434, 2957, 2926, 2855, 1691, 1656, 1464, 1200, 842, 762 cm^{-1}

1,1',1''-[(2,4,6-Trioxo-1,3,5-triazinane-1,3,5-triyl)tris(ethane-2,1-diyl)]-tris(4-nonylaminopyridin-1-ium) trichloride (5c):



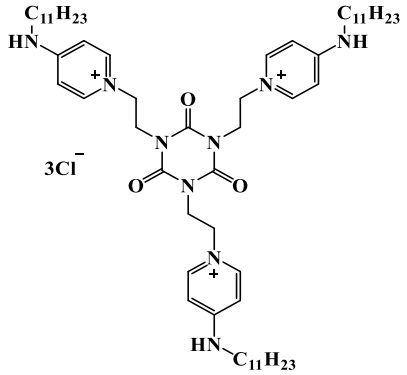
$C_{51}H_{84}Cl_3N_9O_3$; M. w. 977.64; White solid (0.82 g, 0.84 mmol, 84% yield); M. p. 172-175°C; ^{1}H NMR (300 MHz, DMSO-d_6): δ 0.86 (t, J = 6.9 Hz, 9H, $3CH_3$), 1.16-1.39 (m, 54H, $27CH_2$), 1.50-1.60 (m, 6, $3CH_2$), 3.15-3.33 (m, 6H, $3CH_2NH$), 4.00-4.18 (m, 6H, $3CH_2N$), 4.25-4.41 (m, 6H, $3CH_2N^+$), 6.89 (dd, J = 7.0, 2.6 Hz, 3H, $3CH_{py}$), 7.07 (dd, J = 7.0, 2.6 Hz, 3H, $3CH_{py}$), 8.30 (d, J = 7.0 Hz, 3H, $3CH_{py}$), 8.47 (d, J = 7.0 Hz, 3H, $3CH_{py}$), 9.31 (t, J = 5.2 Hz, 3H, 3NH) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): δ 13.8, 21.9, 26.2, 27.8, 28.5, 28.6, 28.8, 31.1, 42.1, 43.1, 54.8, 104.9, 110.0, 141.6, 144.0, 148.7, 156.6 ppm; ν_{max}, (KBr): 3434, 2957, 2926, 2855, 1691, 1656, 1464, 1200, 842, 762 cm^{-1}

1,1',1''-[(2,4,6-Trioxo-1,3,5-triazinane-1,3,5-triyl)tris(ethane-2,1-diyl)]-tris(4-decylaminopyridin-1-ium) trichloride (5d):



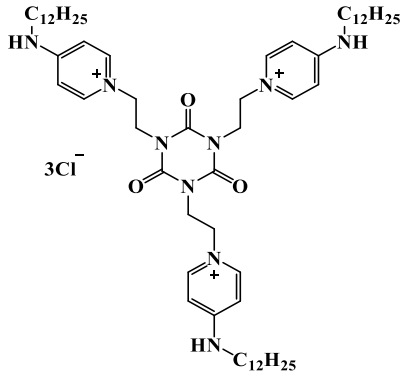
$C_{54}H_{90}Cl_3N_9O_3$; M. w. 1019.72; White solid (0.93 g, 0.91 mmol, 91% yield); M. p. 201-204°C; ^{1}H NMR (300 MHz, DMSO-d_6): δ 0.86 (t, J = 6.9 Hz, 9H, $3CH_3$), 1.16-1.39 (m, 54H, $27CH_2$), 1.50-1.60 (m, 6, $3CH_2$), 3.18-3.29 (m, 6H, $3CH_2NH$), 3.99-4.15 (m, 6H, $3CH_2N$), 4.25-4.39 (m, 6H, $3CH_2N^+$), 6.89 (dd, J = 7.0, 2.6 Hz, 3H, $3CH_{py}$), 7.06 (dd, J = 7.0, 2.6 Hz, 3H, $3CH_{py}$), 8.30 (d, J = 7.0 Hz, 3H, $3CH_{py}$), 8.46 (d, J = 7.0 Hz, 3H, $3CH_{py}$), 9.29 (t, J = 5.2 Hz, 3H, 3NH) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): δ 13.8, 21.9, 26.2, 27.8, 28.6, 28.6, 28.8, 28.9, 31.1, 42.1, 43.2, 54.8, 104.9, 110.0, 141.6, 144.0, 148.7, 156.6 ppm; ν_{max}, (KBr): 3434, 2957, 2926, 2855, 1691, 1656, 1464, 1200, 842, 762 cm^{-1}

1,1',1''-[(2,4,6-Trioxo-1,3,5-triazinane-1,3,5-triyl)tris(ethane-2,1-diyl)]-tris(4-undecylaminopyridin-1-ium) trichloride (5e):



$C_{57}H_{96}Cl_3N_9O_3$; M. w. 1061.81; White solid (0.85 g, 0.80 mmol, 80% yield); M. p. 239-242°C; ^{1}H NMR (300 MHz, DMSO-d_6): δ 0.86 (t, J = 6.9 Hz, 9H, $3CH_3$), 1.19-1.37 (m, 54H, $27CH_2$), 1.49-1.62 (m, 6, $3CH_2$), 3.18-3.29 (m, 6H, $3CH_2NH$), 4.01-4.15 (m, 6H, $3CH_2N$), 4.28-4.39 (m, 6H, $3CH_2N^+$), 6.89 (dd, J = 7.0, 2.6 Hz, 3H, $3CH_{py}$), 7.02 (dd, J = 7.0, 2.6 Hz, 3H, $3CH_{py}$), 8.24 (d, J = 7.0 Hz, 3H, $3CH_{py}$), 8.42 (d, J = 7.0 Hz, 3H, $3CH_{py}$), 9.13 (t, J = 5.2 Hz, 3H, 3NH) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): δ 13.8, 21.9, 25.0, 26.2, 27.8, 28.6, 28.6, 28.9, 29.0, 31.2, 42.1, 43.1, 54.8, 104.9, 110.0, 141.6, 144.0, 148.7, 156.6 ppm; ν_{max}, (KBr): 3434, 2957, 2926, 2855, 1691, 1656, 1464, 1200, 842, 762 cm^{-1}

1,1',1''-[(2,4,6-Trioxo-1,3,5-triazinane-1,3,5-triyl)tris(ethane-2,1-diyl)]-tris(4-dodecylaminopyridin-1-ium) trichloride (5f):



$C_{60}H_{102}Cl_3N_9O_3$; M. w. 1103.89; White solid (0.94 g, 0.85 mmol, 85% yield); M. p. 277-280°C; ^{1}H NMR (300 MHz, DMSO-d_6): δ 0.86 (t, J = 6.9 Hz, 9H, $3CH_3$), 1.16-1.39 (m, 54H, $27CH_2$), 1.50-1.60 (m, 6, $3CH_2$), 3.24 (m, 6H, $3CH_2NH$), 4.01-4.15 (m, 6H, $3CH_2N$), 4.28-4.39 (m, 6H, $3CH_2N^+$), 6.89 (dd, J = 7.0, 2.6 Hz, 3H, $3CH_{py}$), 7.03 (dd, J = 7.0, 2.6 Hz, 3H, $3CH_{py}$), 8.26 (d, J = 7.0 Hz, 3H, $3CH_{py}$), 8.43 (d, J = 7.0 Hz, 3H, $3CH_{py}$), 9.16 (t, J = 5.2 Hz, 3H, 3NH) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): δ 13.8, 21.9, 26.2, 27.8, 28.6, 28.6, 28.9, 29.0, 31.2, 42.2, 43.2, 54.8, 104.9, 110.0, 141.6, 144.0, 148.7, 156.6 ppm; ν_{max}, (KBr): 3434, 2957, 2926, 2855, 1691, 1656, 1464, 1200, 842, 762 cm^{-1}

4. References

1. D. A. Tomalia, N. D. Ojha, and B. P. Thill, *J. Org. Chem.*, 1969, **34**, 1400.
2. D. M. Bailey, C. G. DeGrazia, S. J. Hoff, P. L. Schulenberg, J. R. O'Connor, D. A. Paris, and A. M. Slee, *J. Med. Chem.*, 1984, **27**, 1457.

5. Figures

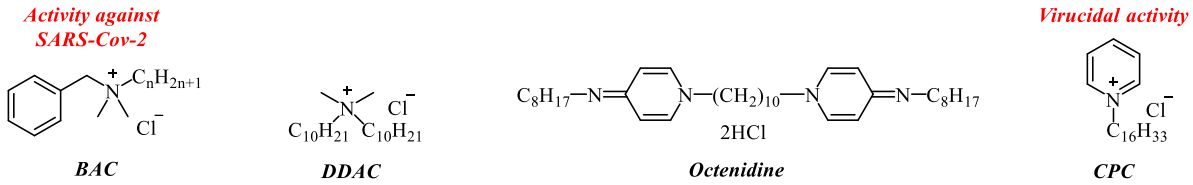


Figure S1. Main QACs on the market. *Note:* BAC – benzalkonium chloride, DDAC – didecyldimethylammonium chloride, CPC – cetylpyridinium chloride.

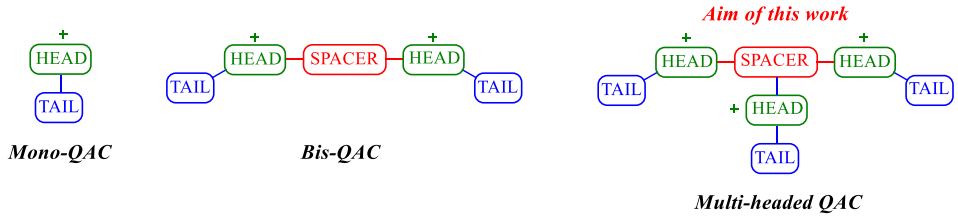


Figure S2. General structure of QACs.

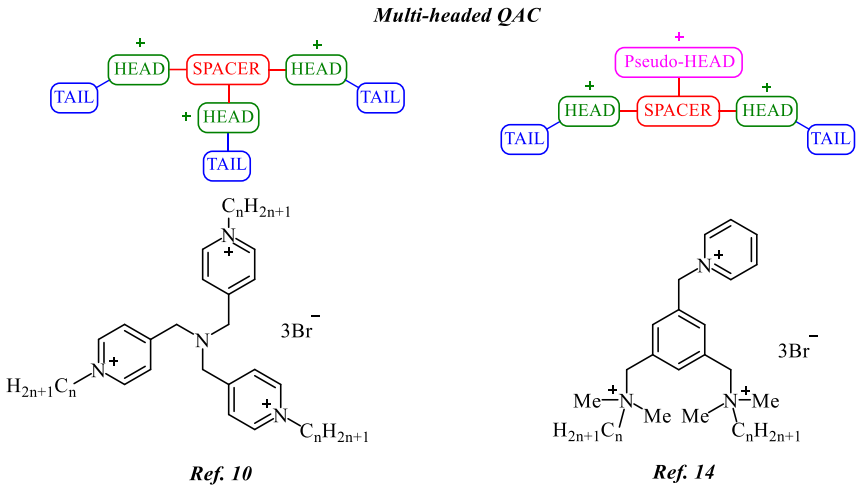
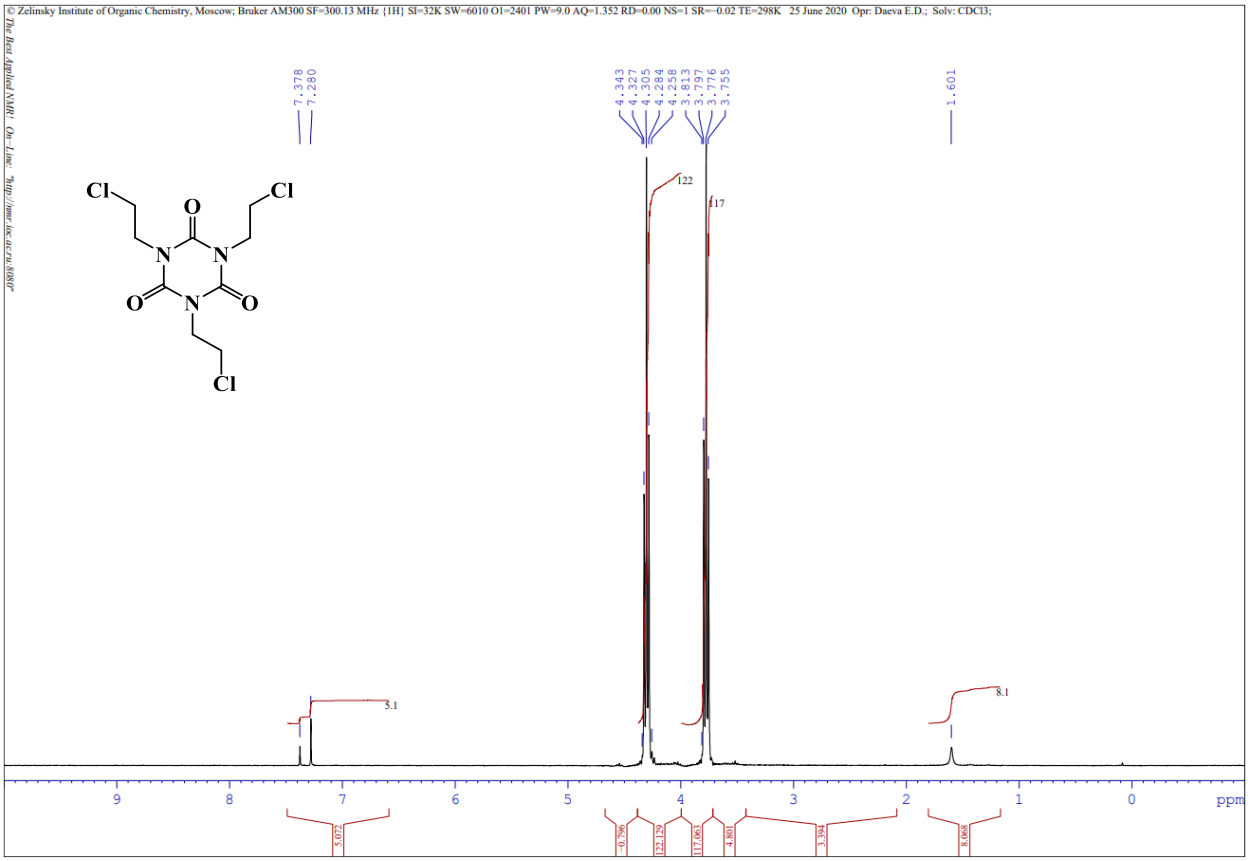


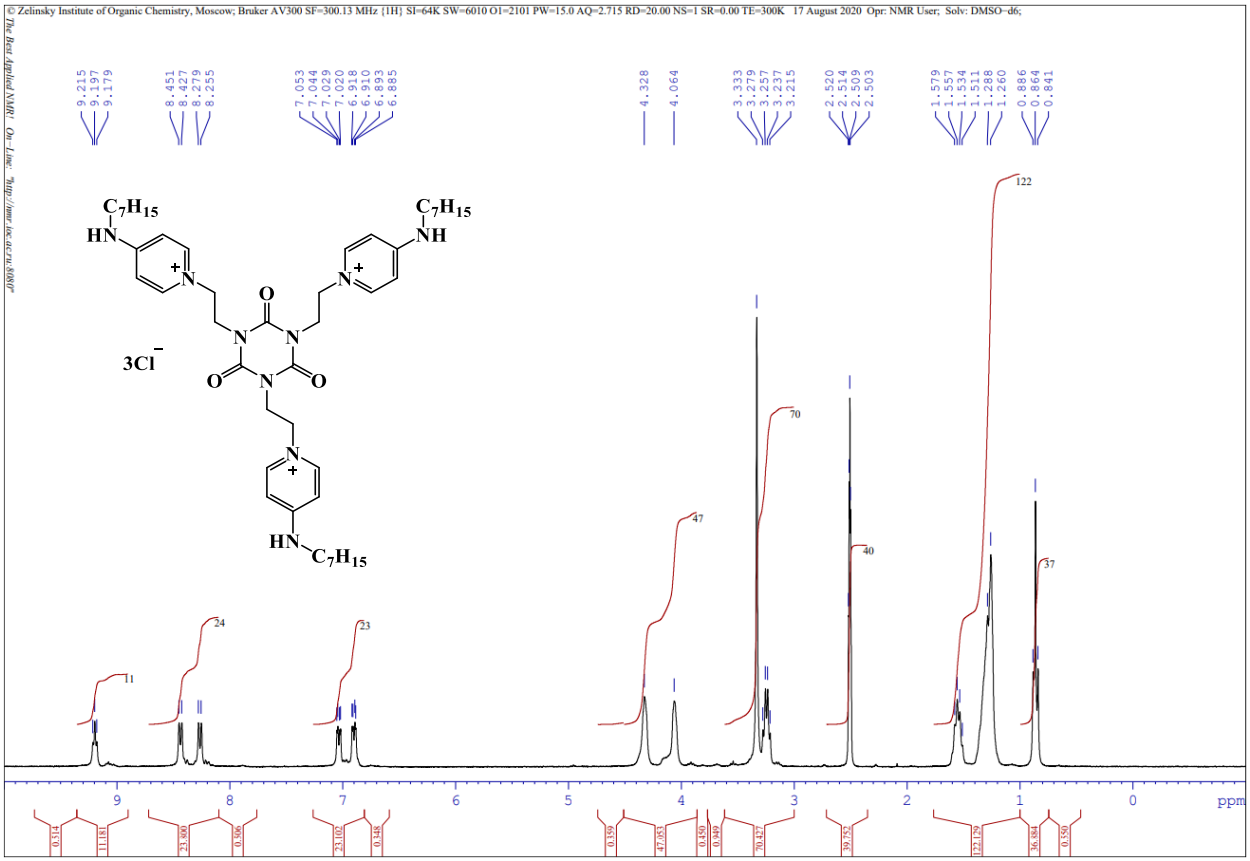
Figure S3. Multi-headed QACs.

6. ^{1}H and ^{13}C NMR spectra

2



5a – 7, Cl


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AV300 SF=300.13 MHz {1H} SI=64K SW=6010 O1=2101 PW=15.0 AQ=2.715 RD=20.00 NS=1 SR=0.00 TE=300K 17 August 2020 Opr: NMR User; Solv: DMSO-d6;
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9.197
9.179
8.451
8.427
8.279
8.255
7.053
7.044
7.029
7.020
6.918
6.910
6.893
6.885
4.328
4.064
3.333
3.279
3.257
3.237
3.215
2.520
2.514
2.509
2.503
1.579
1.557
1.534
1.511
1.288
1.260
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0.864
0.841
3Cl
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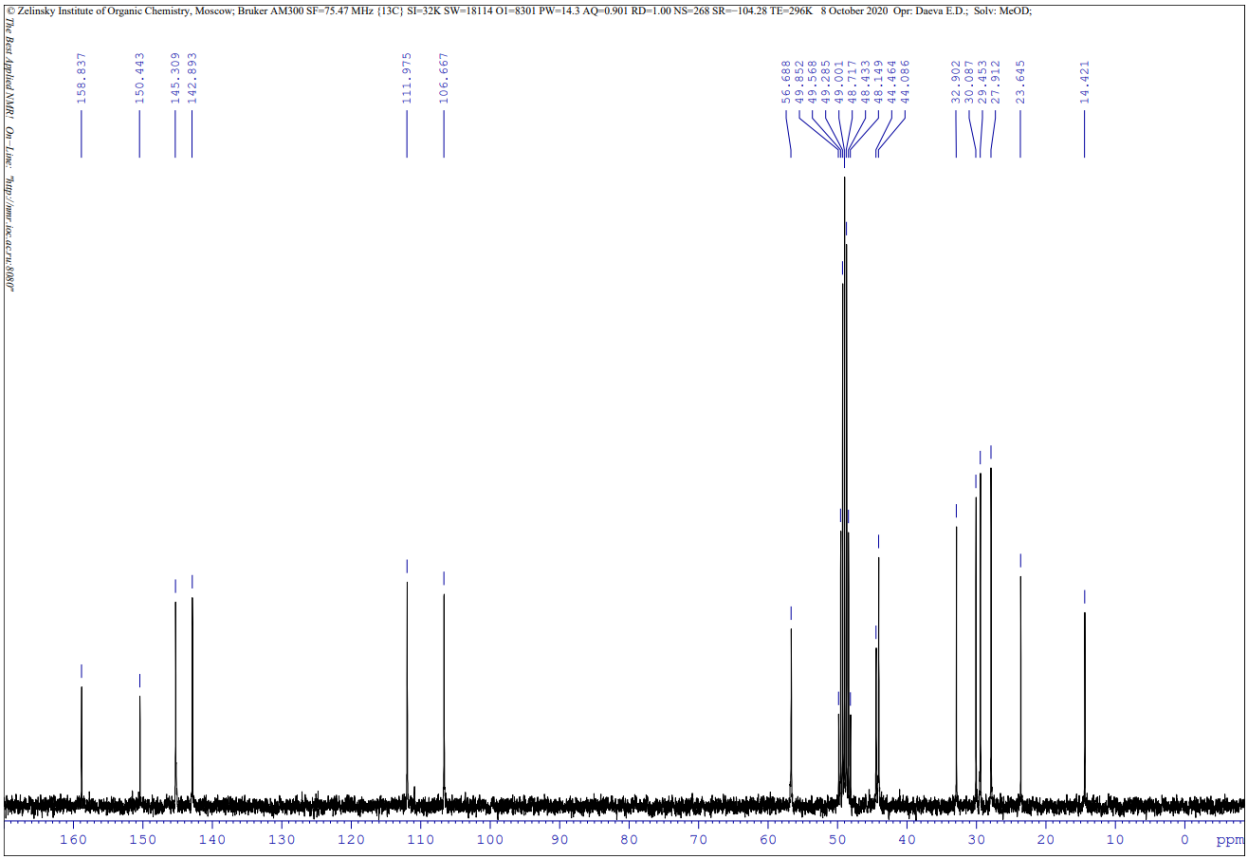
70
47
40
37
24
23
11
ppm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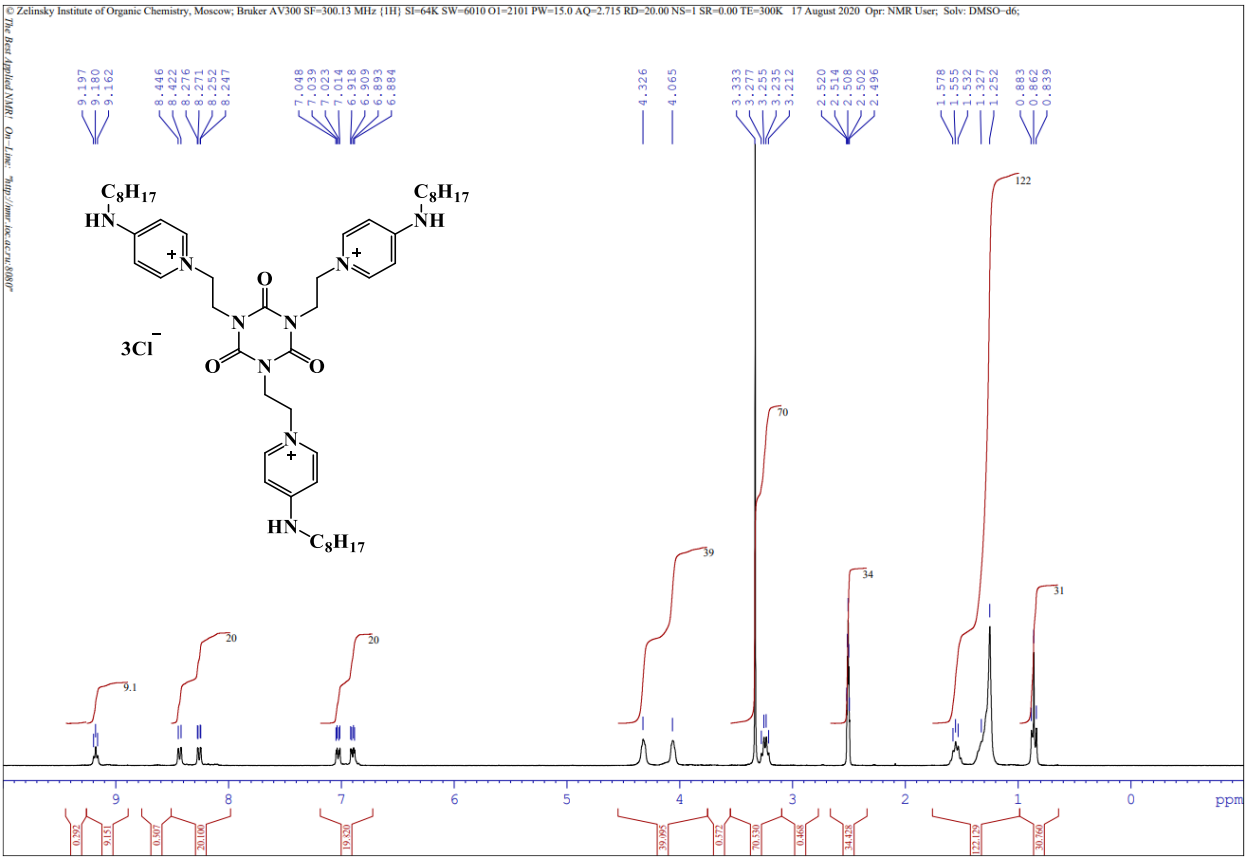
150.443
145.309
142.893
111.975
106.667
56.688
49.852
49.568
49.285
49.001
48.717
48.433
48.149
47.864
47.580
44.086
32.902
30.087
29.453
27.912
23.645
14.421
ppm

5b – 8, Cl


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AV300 SF=300.13 MHz {1H} SI=64K SW=6010 O1=2101 PW=15.0 AQ=2.715 RD=20.00 NS=1 SR=0.00 TE=300K 17 August 2020 Opr: NMR User; Solv: DMSO-d6;
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9.162
8.446
8.422
8.276
8.271
8.252
8.247
7.048
7.039
7.023
7.014
6.918
6.909
6.893
6.884
4.326
4.065
3.333
3.277
3.255
3.235
3.212
2.520
2.514
2.508
2.502
2.496
1.578
1.555
1.532
1.327
1.252
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0.862
0.839
3Cl⁻
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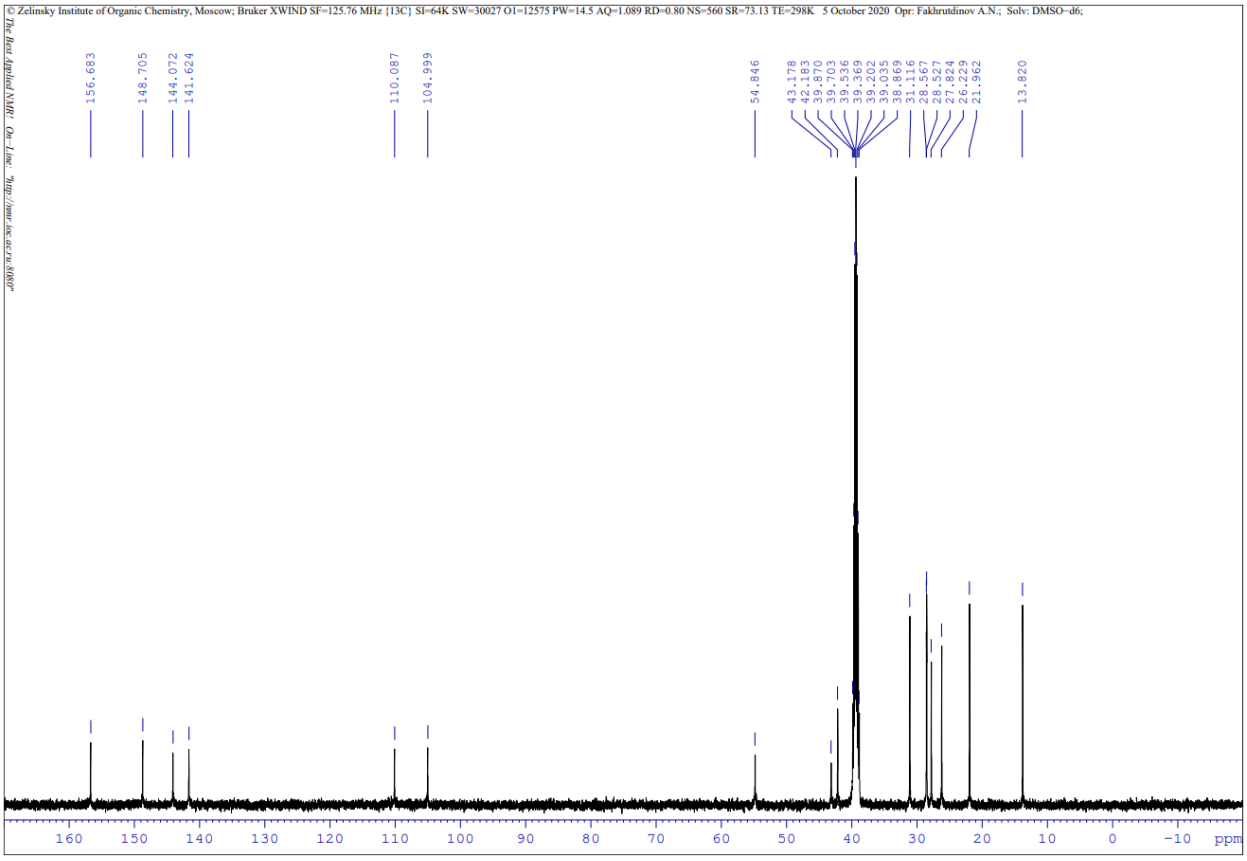
70
39
34
31
20
20
9.1
ppm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144.072
141.624
110.087
104.999
54.846
43.178
42.183
39.870
39.703
39.536
39.369
39.202
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31.116
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28.527
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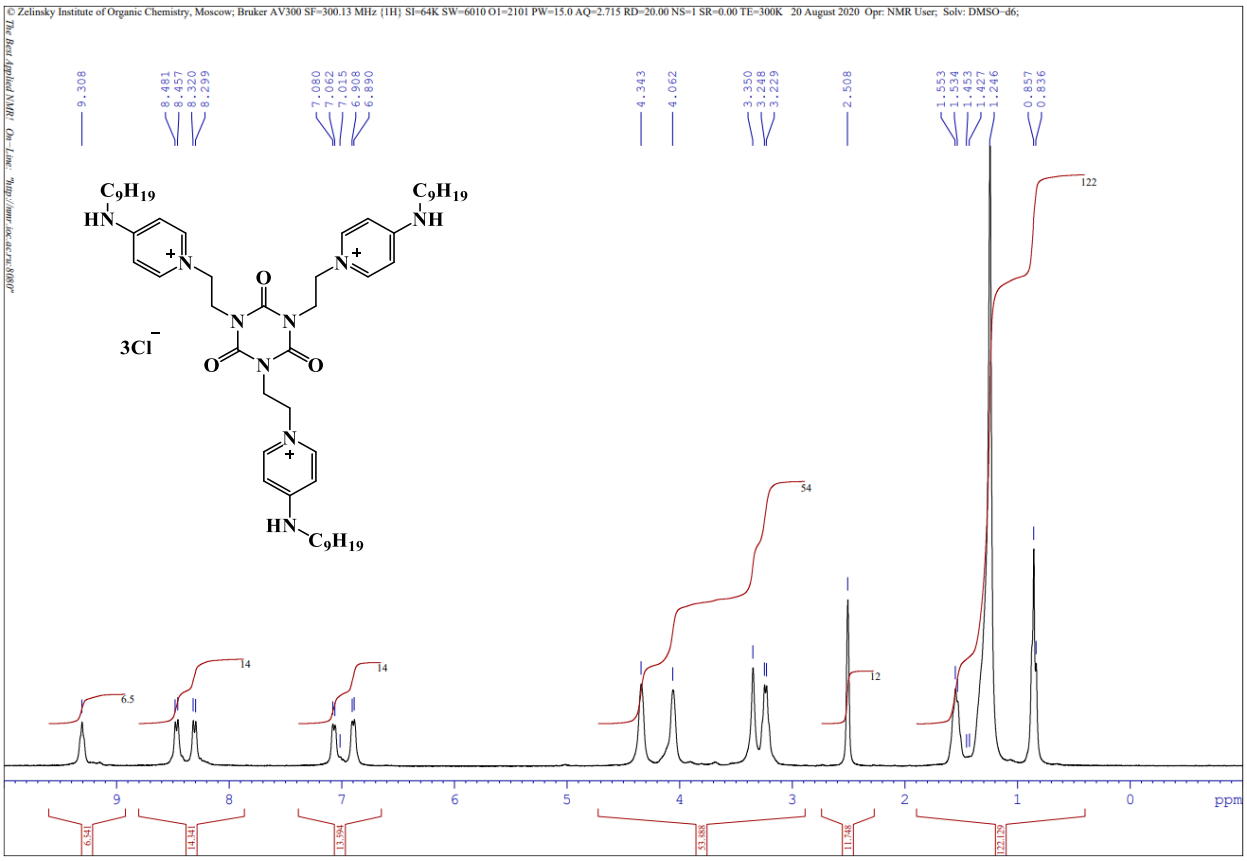
ppm

5c – 9, Cl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8.457
8.320
8.299
7.080
7.062
7.015
6.908
6.890
4.343
4.062
3.350
3.248
3.229
2.508
1.553
1.534
1.453
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1.246
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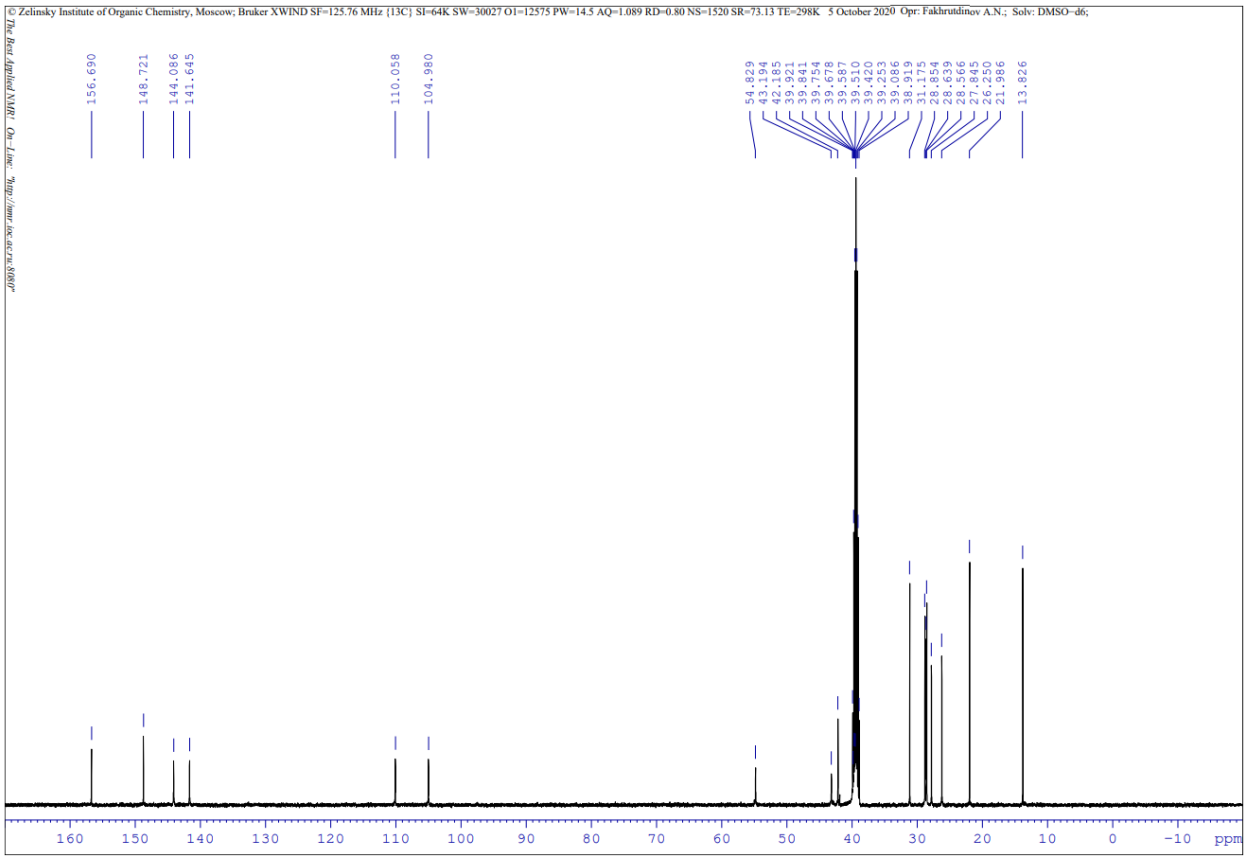
3Cl⁻
6.5
14
14
54
12
122
ppm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141.645
110.058
104.980
54.829
43.194
42.185
39.921
39.841
39.754
39.678
39.587
39.510
39.420
39.253
39.086
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21.986
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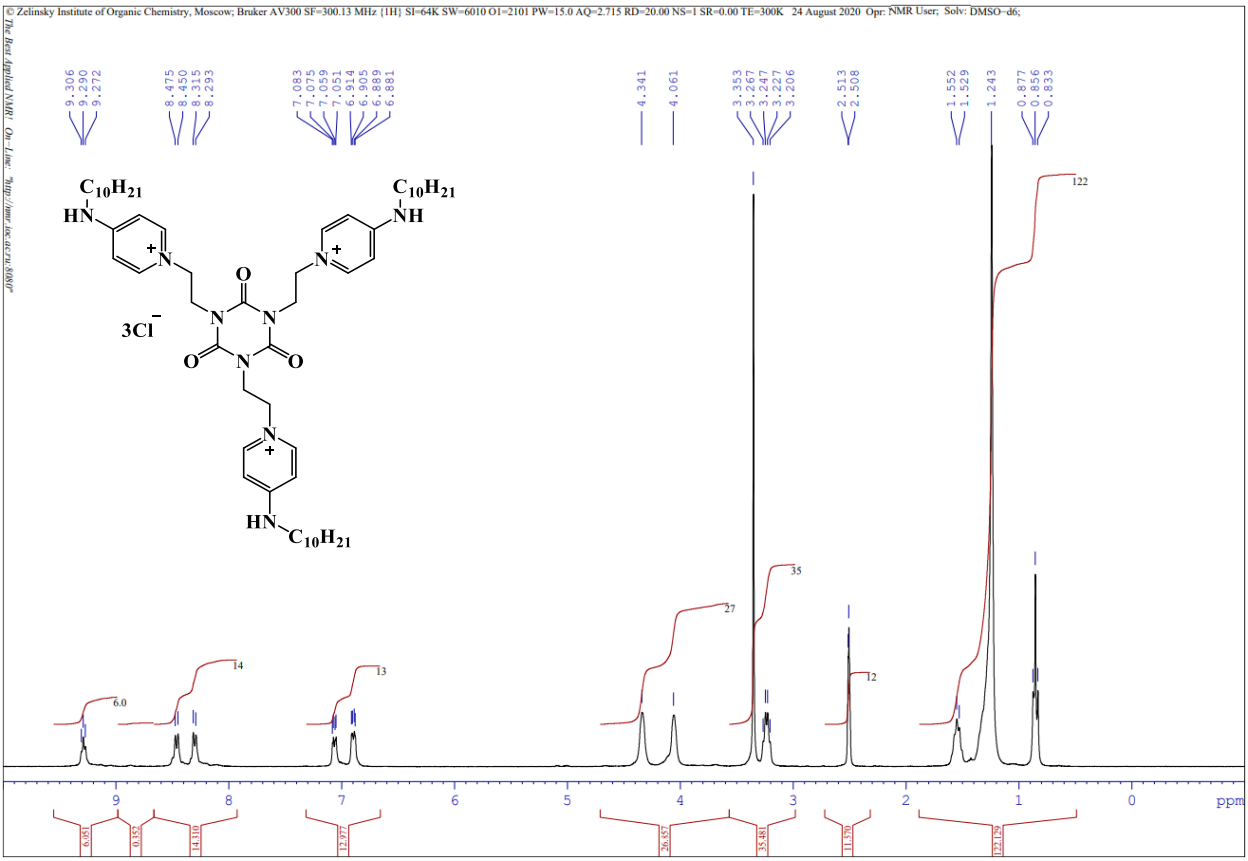
ppm

5d – 10, Cl


© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AV300 SF=300.13 MHz {1H} SI=64K SW=6010 O1=2101 PW=15.0 AQ=2.715 RD=20.00 NS=1 SR=0.00 TE=300K 24 August 2020 Opr: NMR User; Solv: DMSO-d6;
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8.315
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6.905
6.889
6.881
4.341
4.061
3.353
3.267
3.247
3.227
3.206
2.513
2.508
1.552
1.529
1.243
0.877
0.856
0.833
C10H21
HN
C10H21
NH
3Cl−
O
N
HN
C10H21
6.0
14
13
27
35
12
122
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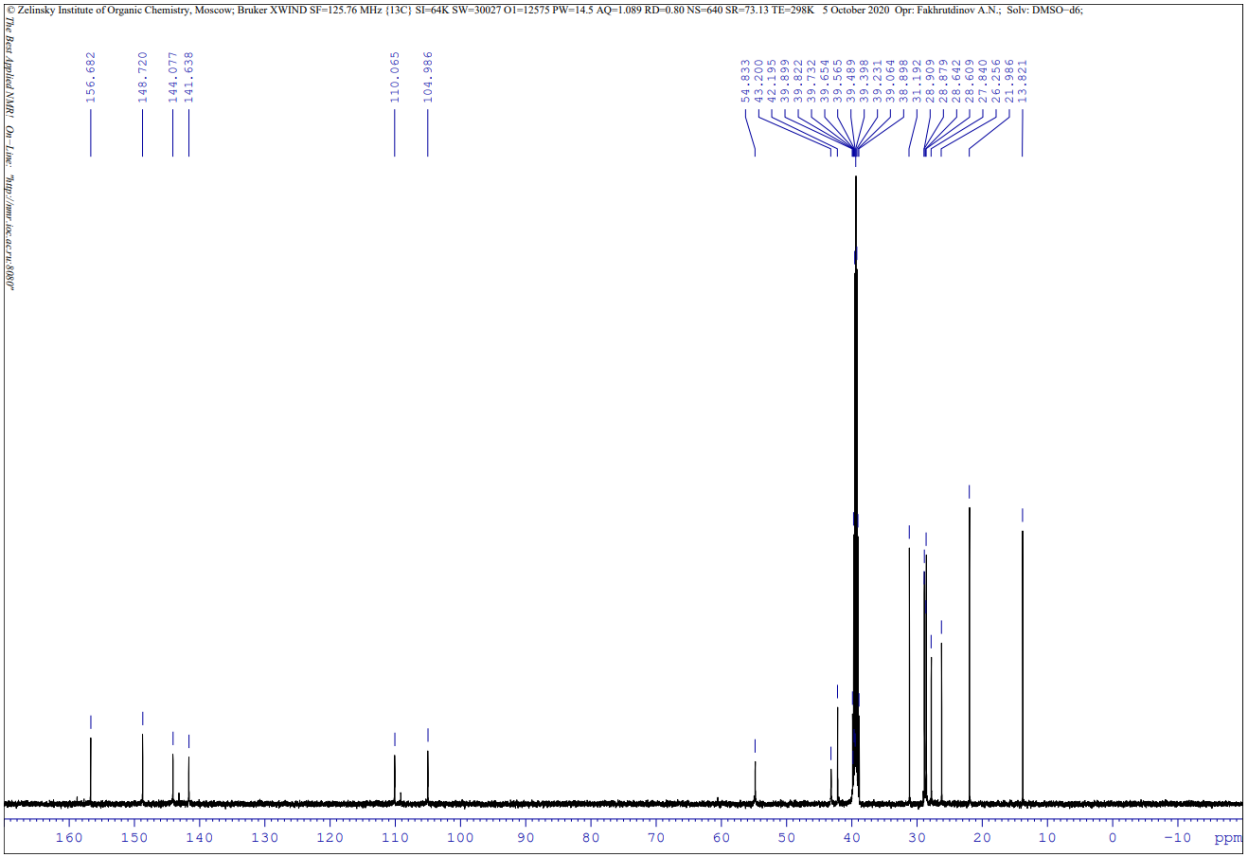
ppm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104.986
54.833
43.200
42.195
39.899
39.822
39.732
39.654
39.565
39.489
39.398
39.231
39.064
38.898
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28.879
28.642
28.609
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21.986
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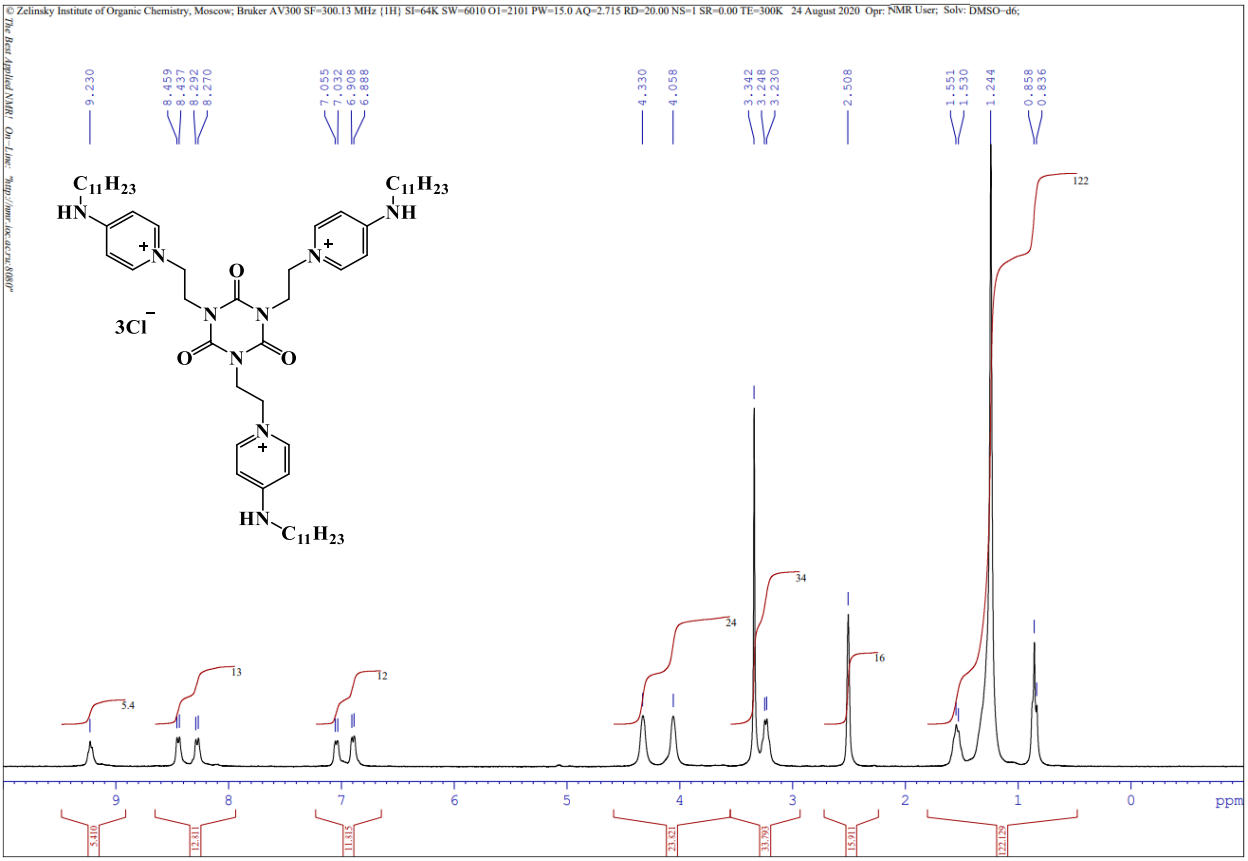
ppm

5e – 11, Cl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7.032
6.908
6.888
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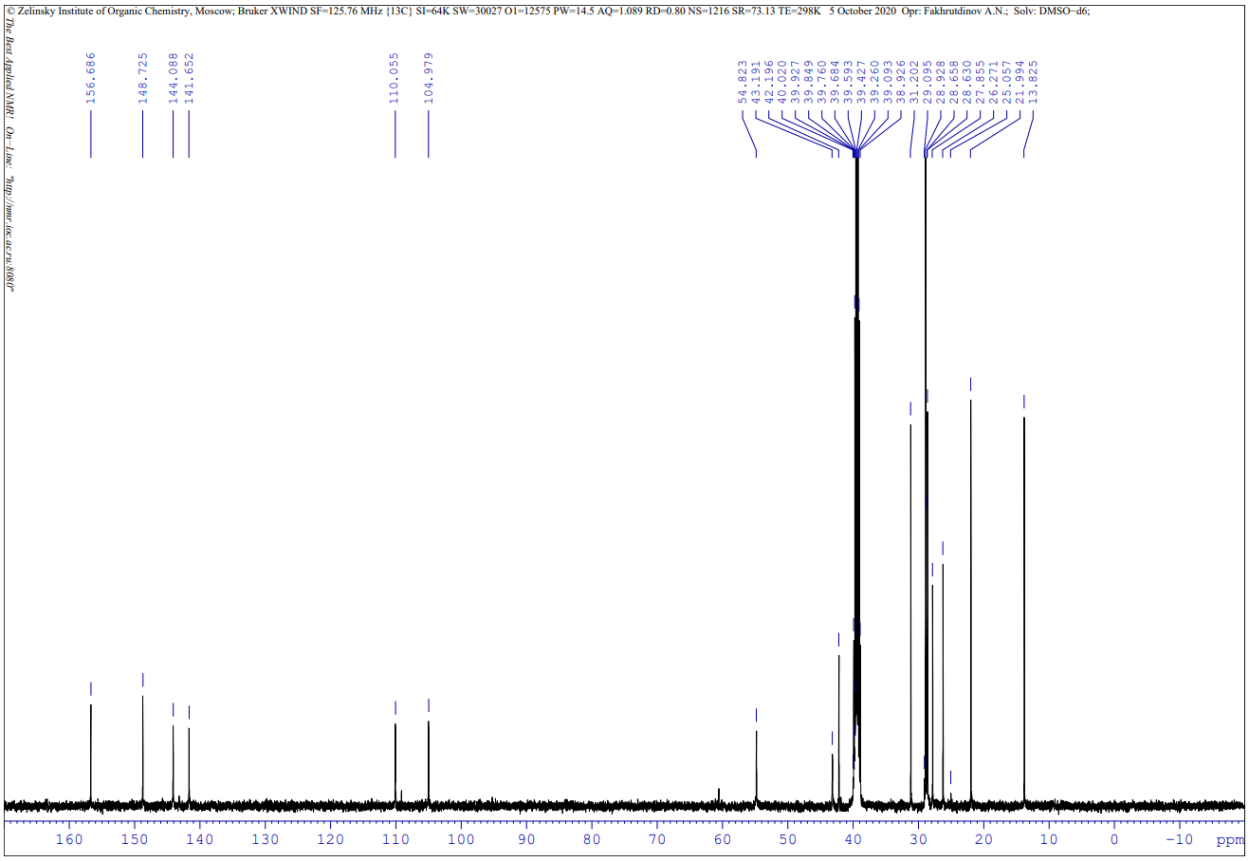
3.230
2.508
1.551
1.530
1.244
0.858
0.836
3Cl⁻
ppm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42.196
40.020
39.927
39.849
39.760
39.684
39.593
39.427
39.260
39.093
38.926
31.202
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21.994
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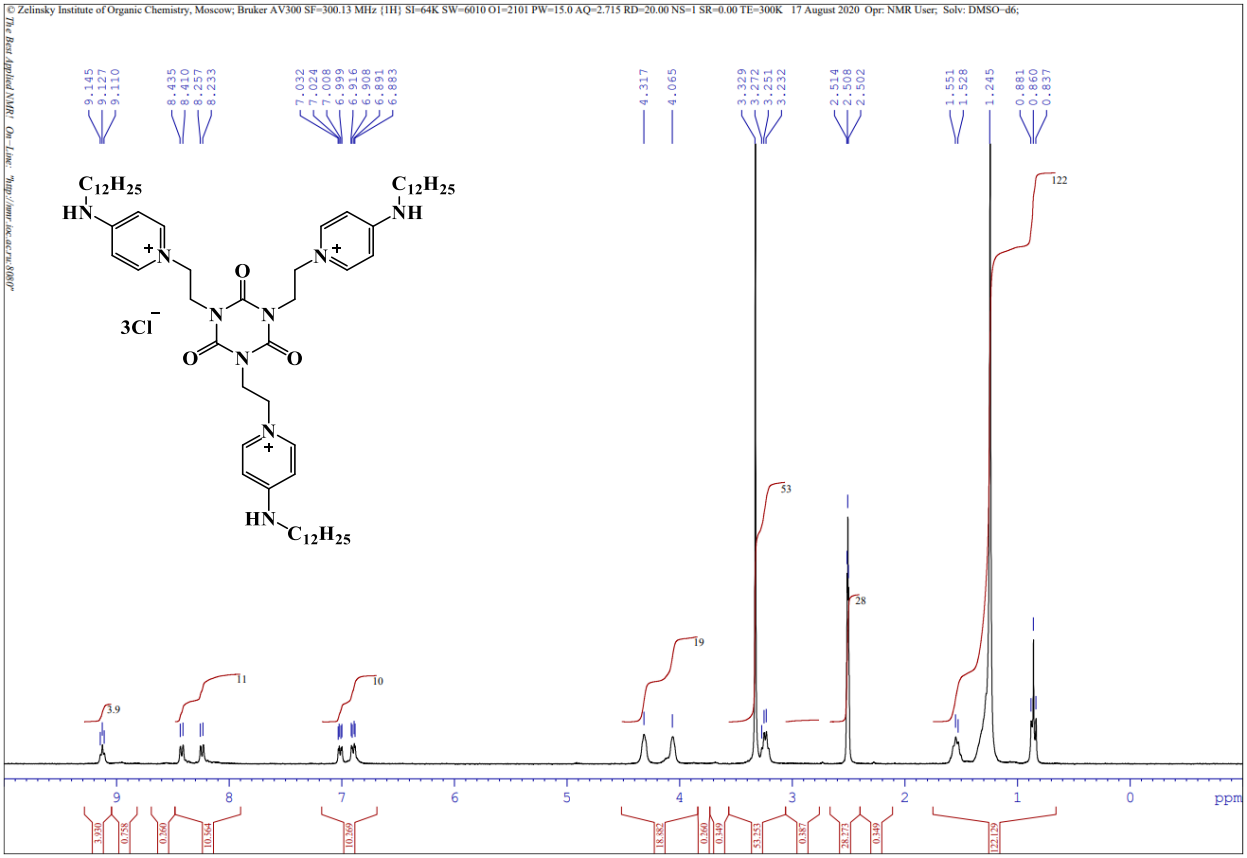
ppm

5f – 12, Cl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9.110
8.435
8.410
8.257
8.233
7.032
7.024
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6.891
6.883
4.317
4.065
3.329
3.272
3.251
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2.508
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1.528
1.245
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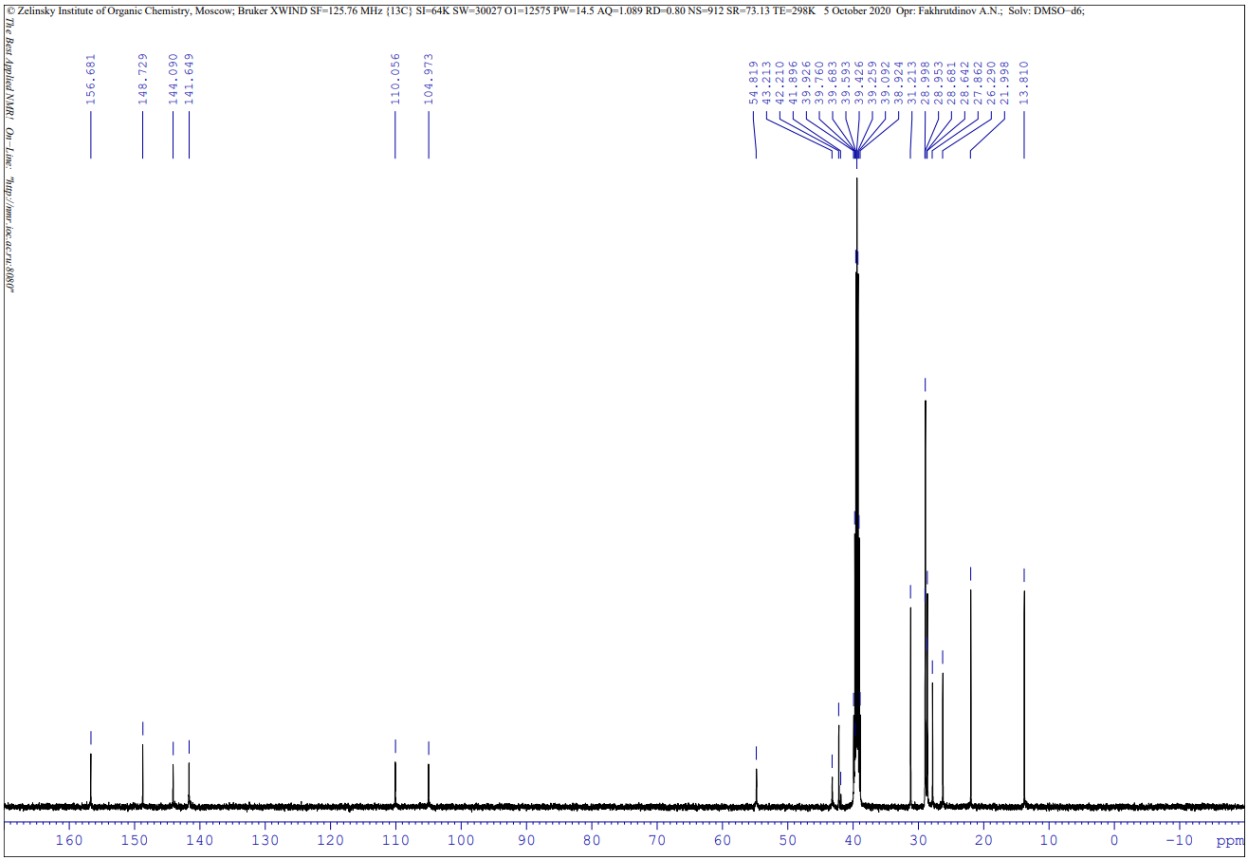
3Cl
ppm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141.649
110.056
104.973
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43.213
42.870
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39.760
39.683
39.593
39.426
39.259
39.092
38.924
31.213
28.998
28.953
28.681
28.642
27.862
26.290
21.998
13.810
ppm