

SUPPORTING INFORMATION

Chichibabin-type condensation of cyclic ketones with 3-R-1,2,4-triazin-5(4H)-ones

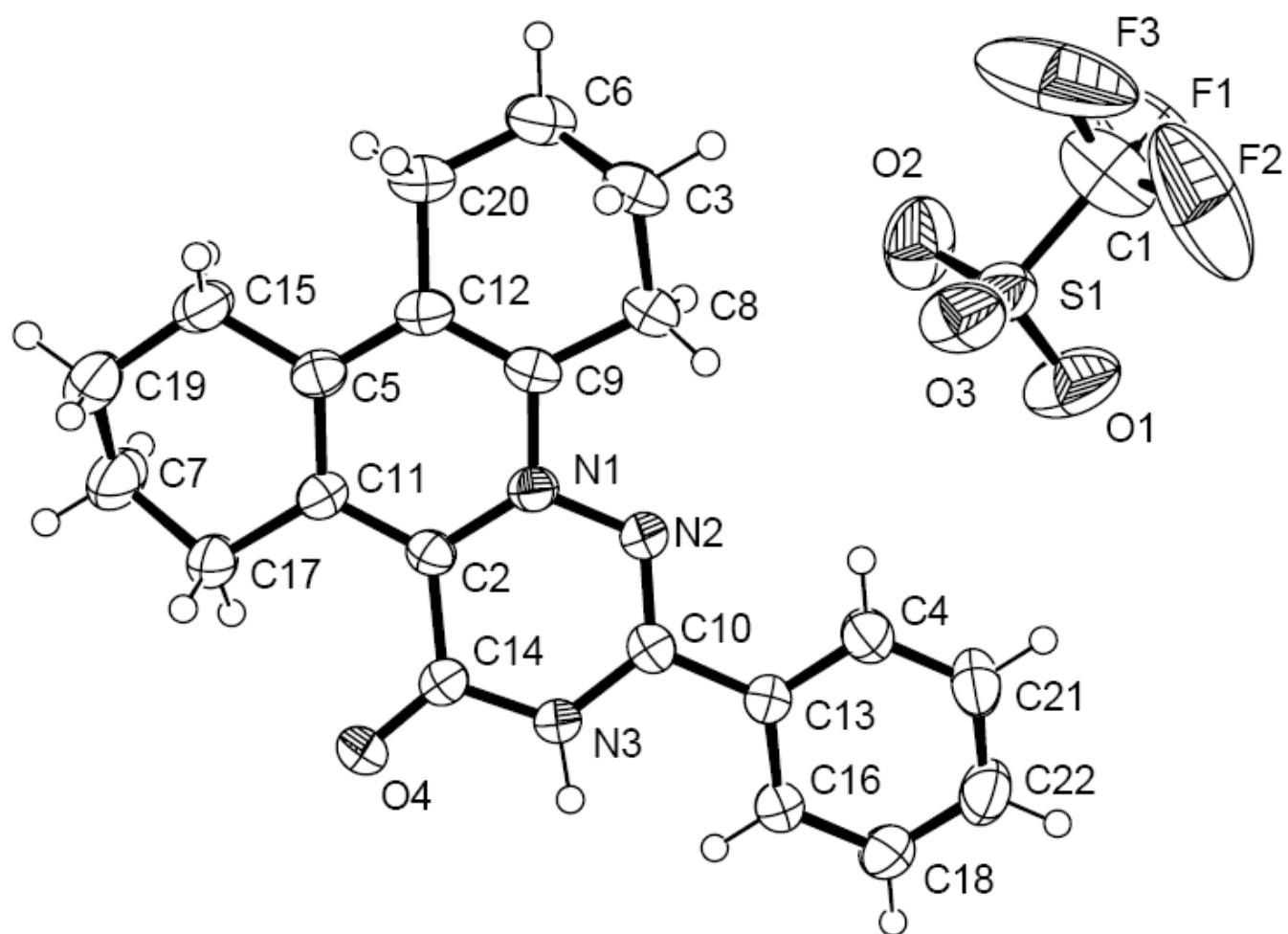
Ilya N. Egorov,^{a,*} Tatyana A. Tseitler,^a Igor S. Kovalev,^a Pavel A. Slepukhin,^b Vladimir L. Rusinov^{a,b} and Oleg N. Chupakhin^{a,b}

^a Ural Federal University, Department of Organic Chemistry,
Mira St. 19, Ekaterinburg, 620 002, Russia, e-mail: i.n.egorov@gmail.com

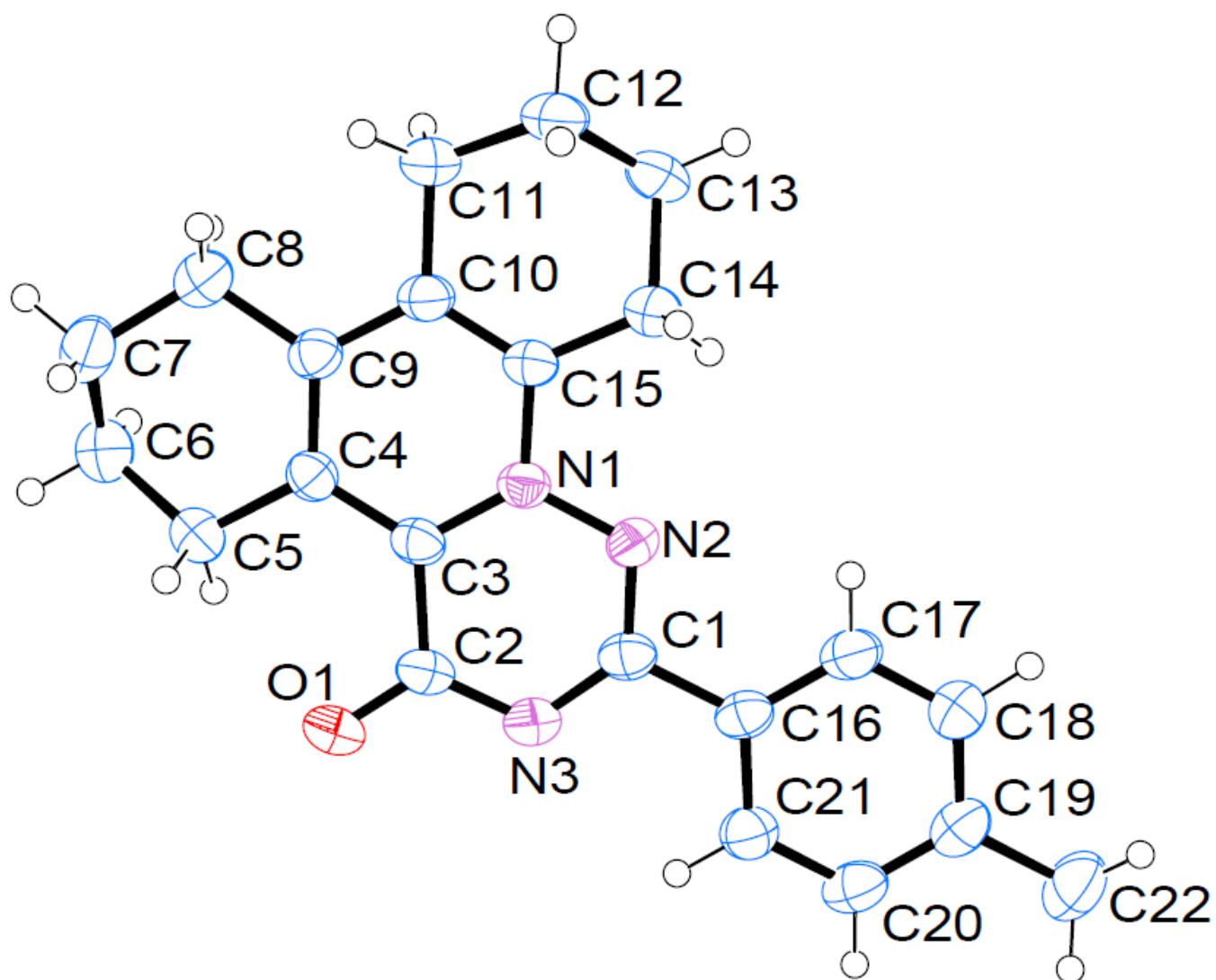
^b Institute of Organic Synthesis Ural Branch of Russian Academy of Sciences,
S. Kovalevskoy St. 22, Ekaterinburg, 620 041, Russia

Contents:

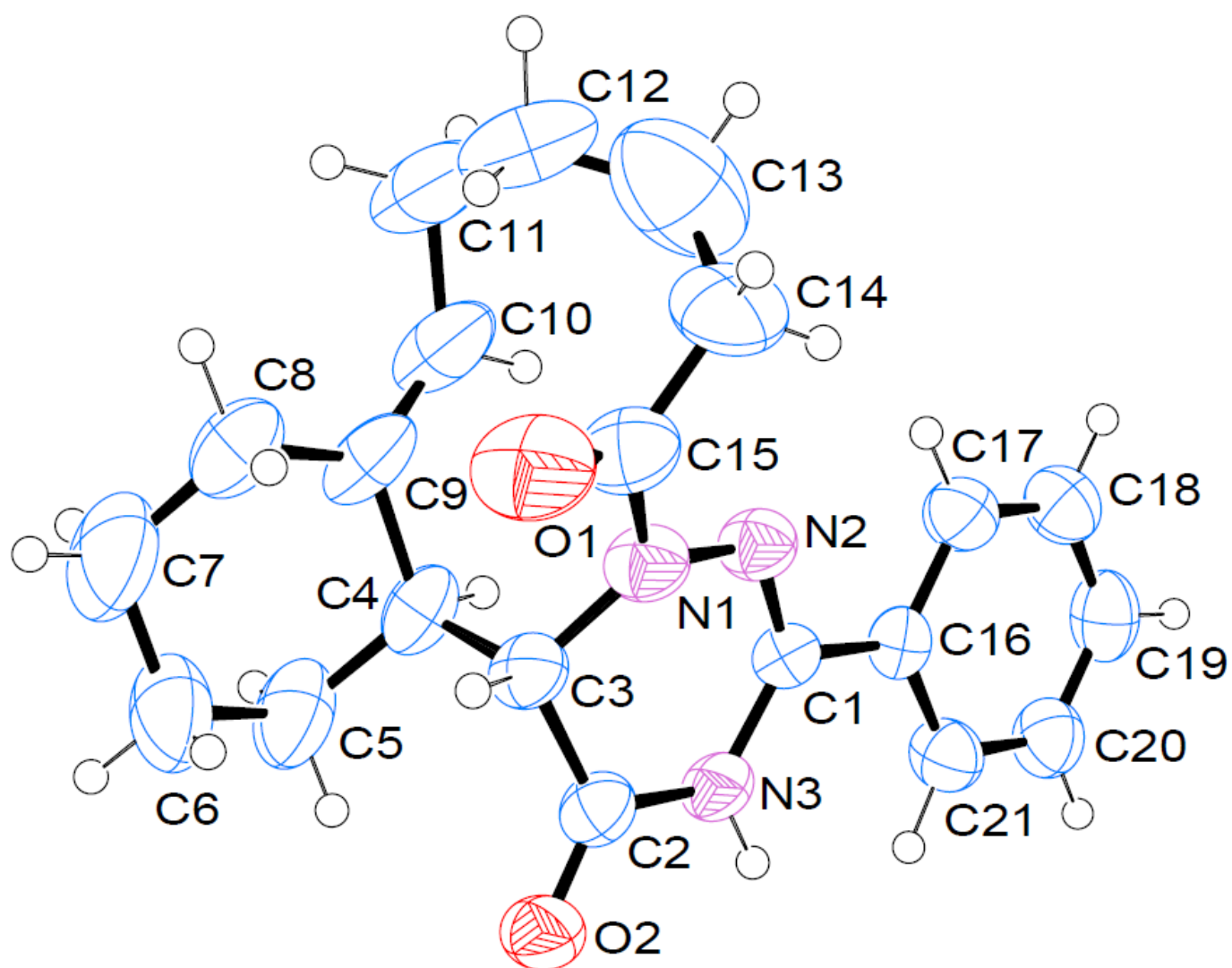
ORTEP diagrams	2-5pp
Copies of ¹ H NMR and ¹³ C NMR spectra	6-28pp



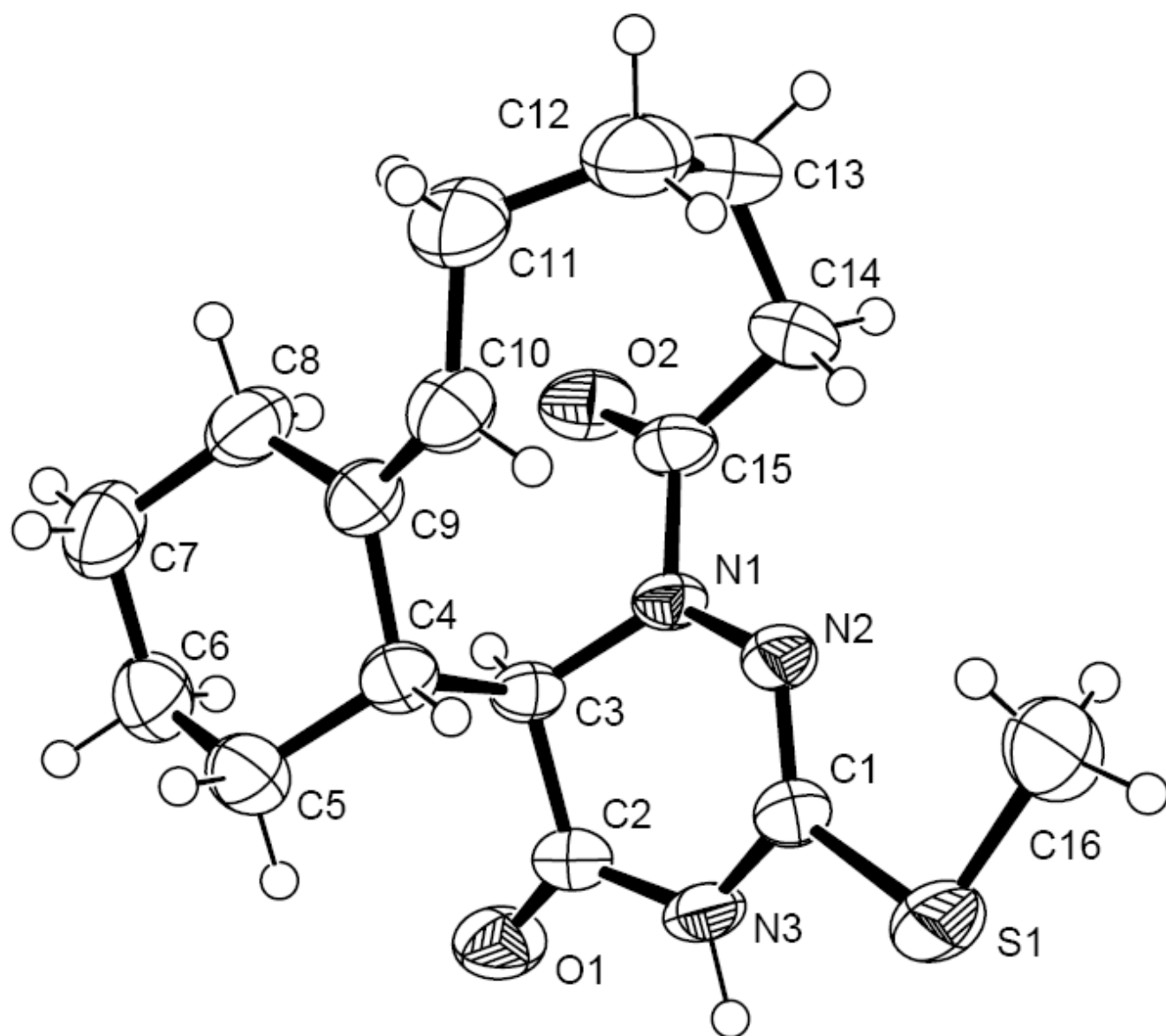
ORTEP diagram of **6aa**.



ORTEP diagram of **6c**.

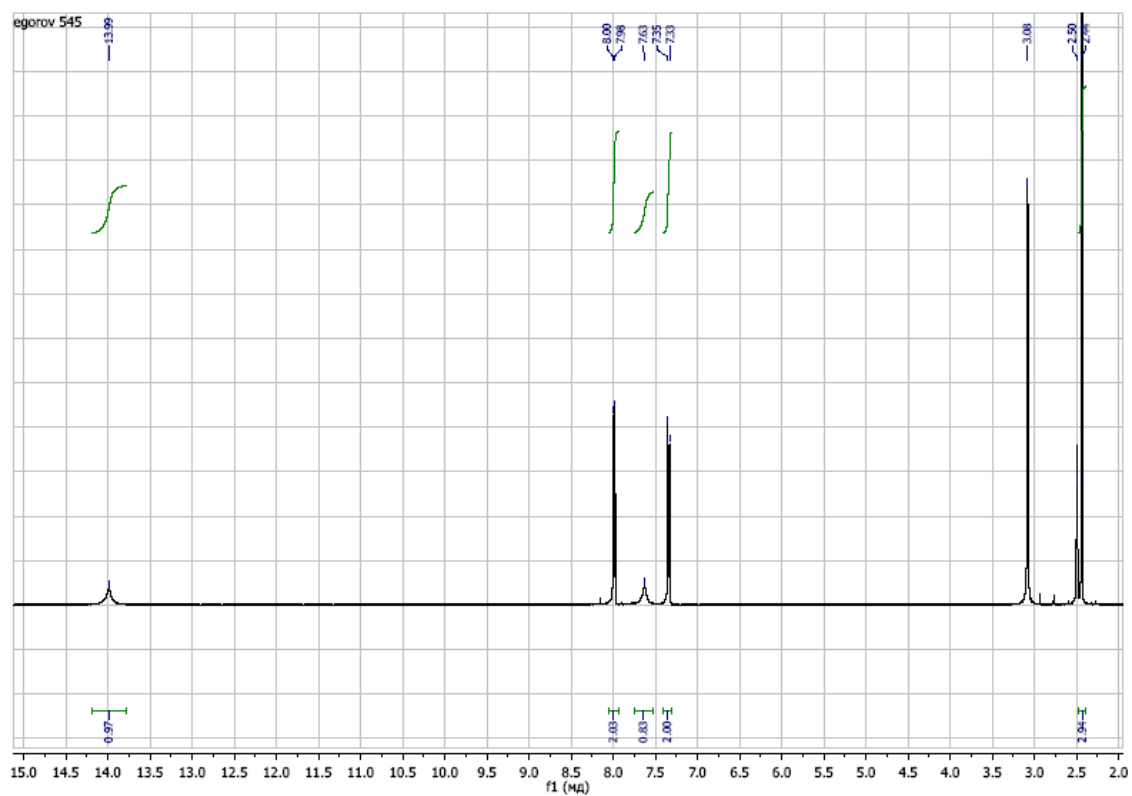


ORTEP diagram of **10a**.

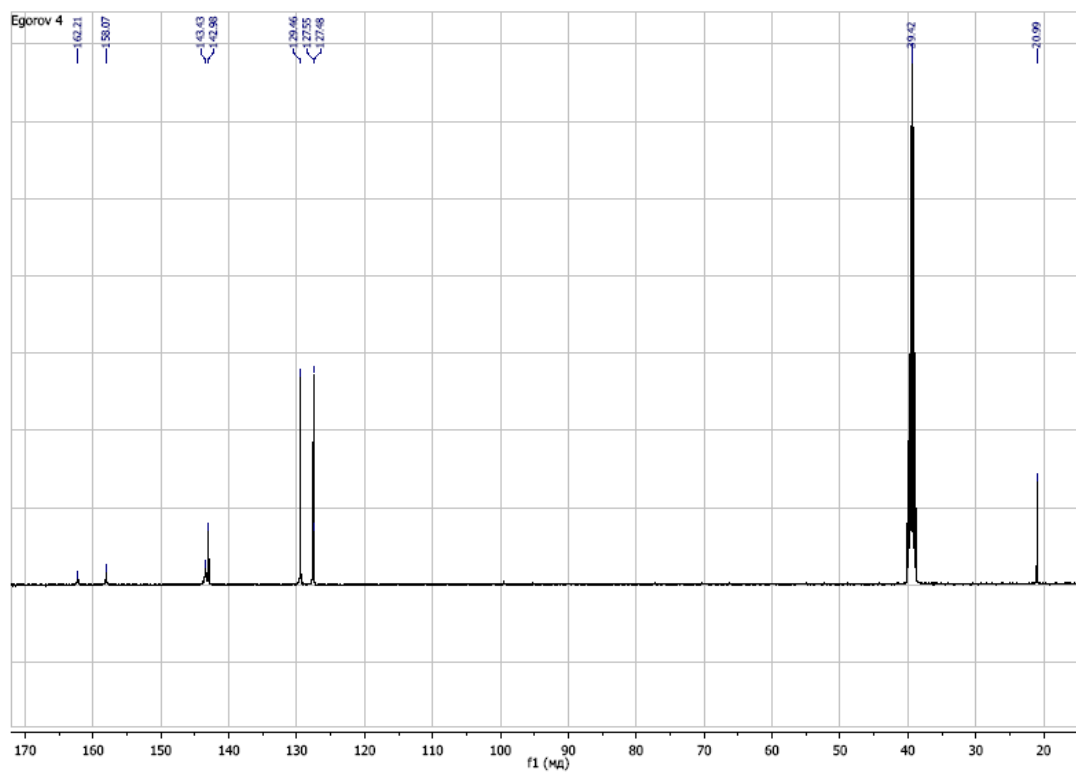


ORTEP diagram of **10b**.

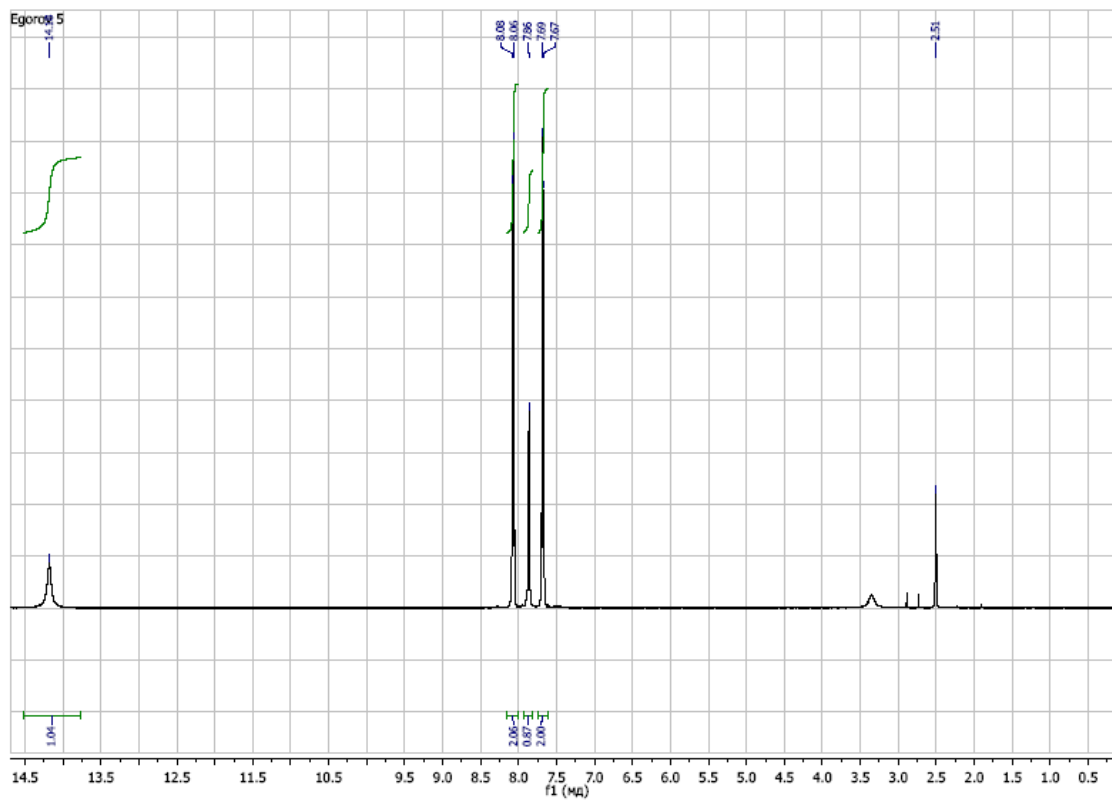
3-(4-Tolyl)-1,2,4-triazin-5(4H)-one (1c). ^1H NMR (400 MHz, DMSO-d_6)



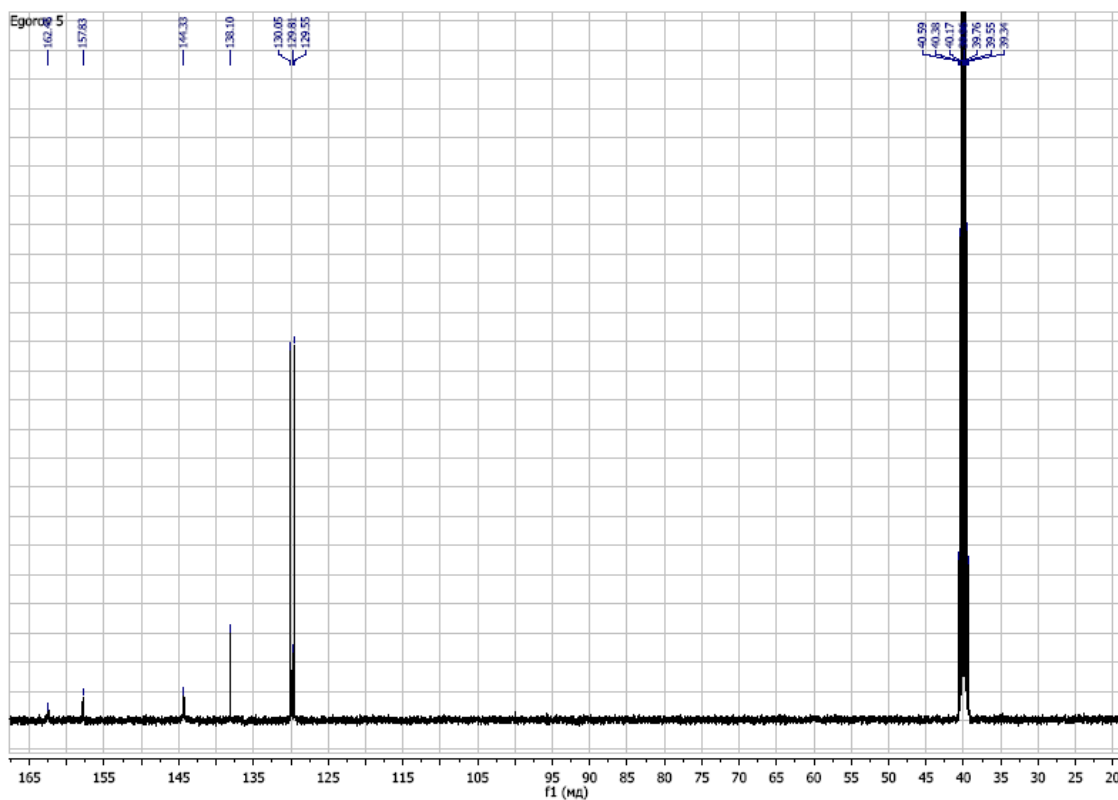
3-(4-Tolyl)-1,2,4-triazin-5(4H)-one (1c). ^{13}C NMR (100MHz, DMSO-d_6)



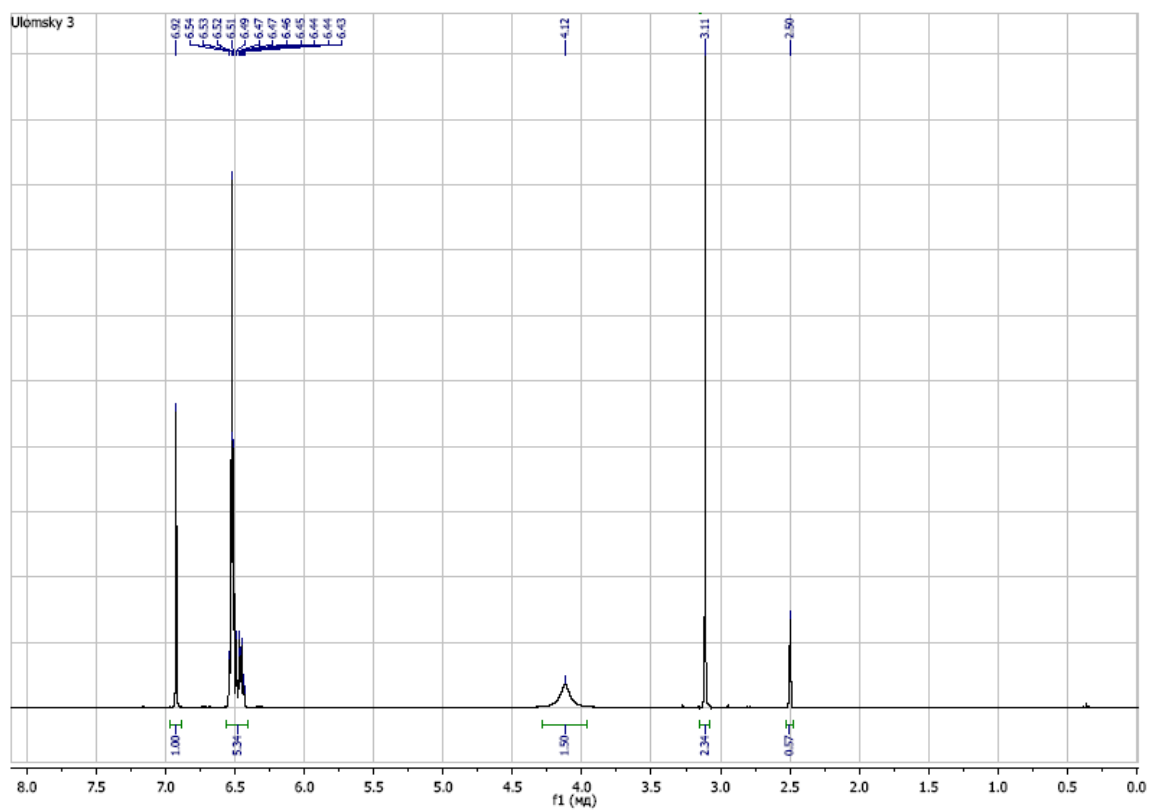
3-(4-Chlorophenyl)-1,2,4-triazin-5(4H)-one (1d) ^1H NMR (400 MHz, DMSO- d_6)



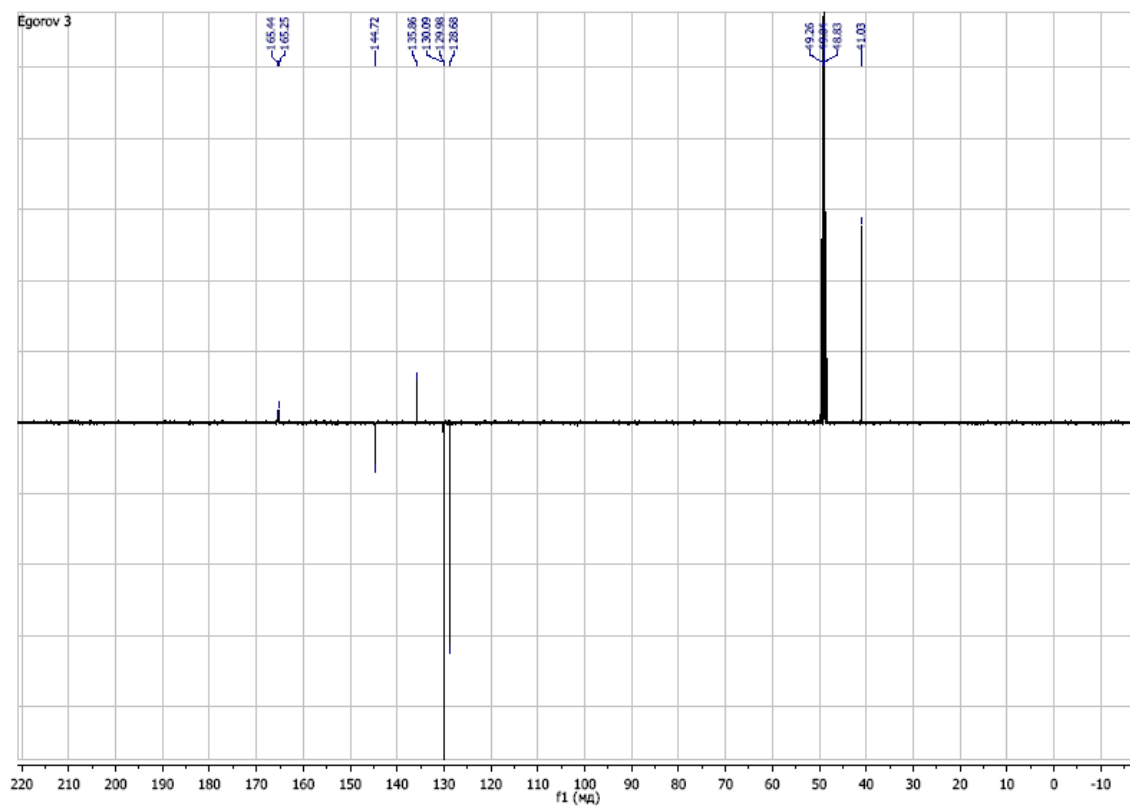
3-(4-Chlorophenyl)-1,2,4-triazin-5(4H)-one (1d) ^{13}C NMR (100MHz, DMSO- d_6)



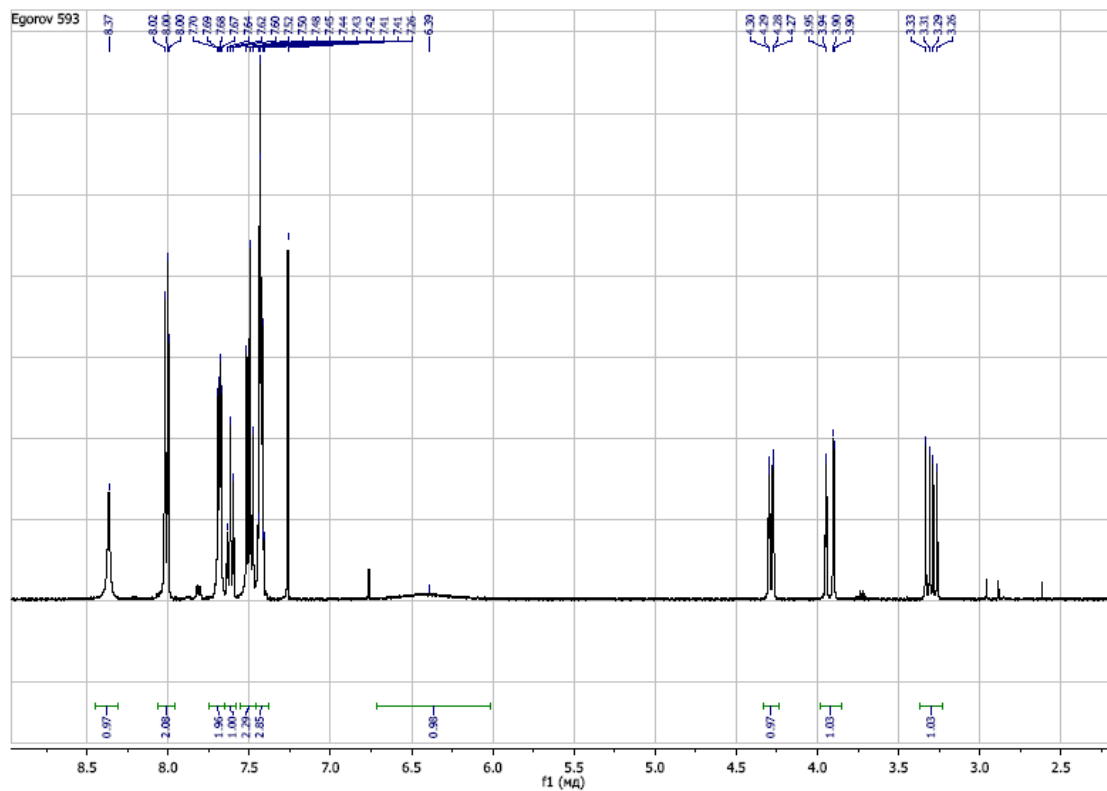
3-Benzyl-1,2,4-triazin-5(4H)-one (1e). ^1H NMR (400 MHz, DMSO-d_6)



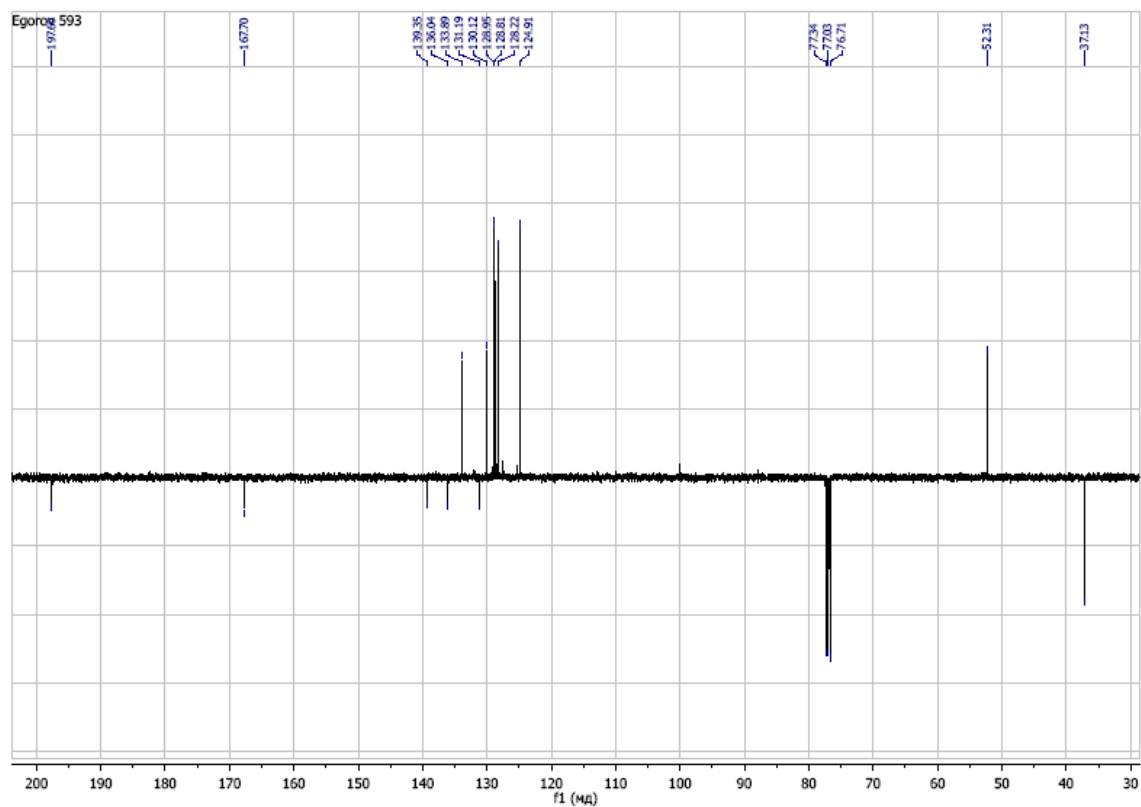
3-Benzyl-1,2,4-triazin-5(4H)-one (1e). ^{13}C NMR (100MHz, CD_3OD)



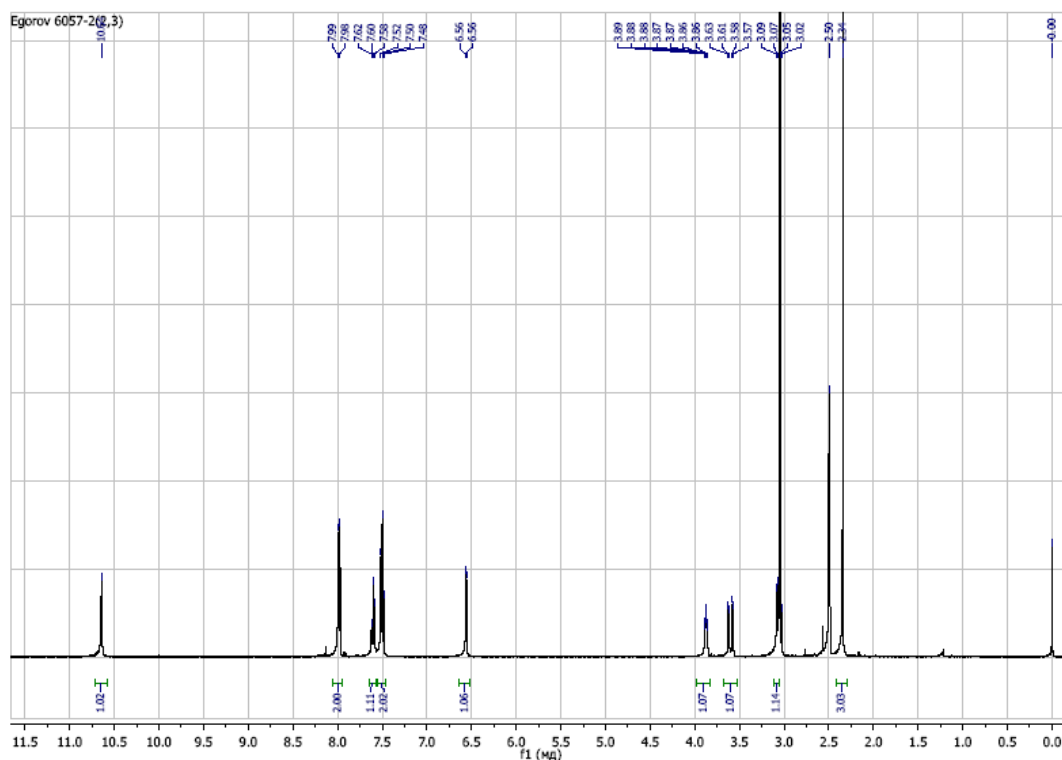
6-(2-Oxo-2-phenylethyl)-3-phenyl-1,6-dihydro-1,2,4-triazin-5(4H)-one (3a). ^1H NMR (400 MHz, CDCl_3)



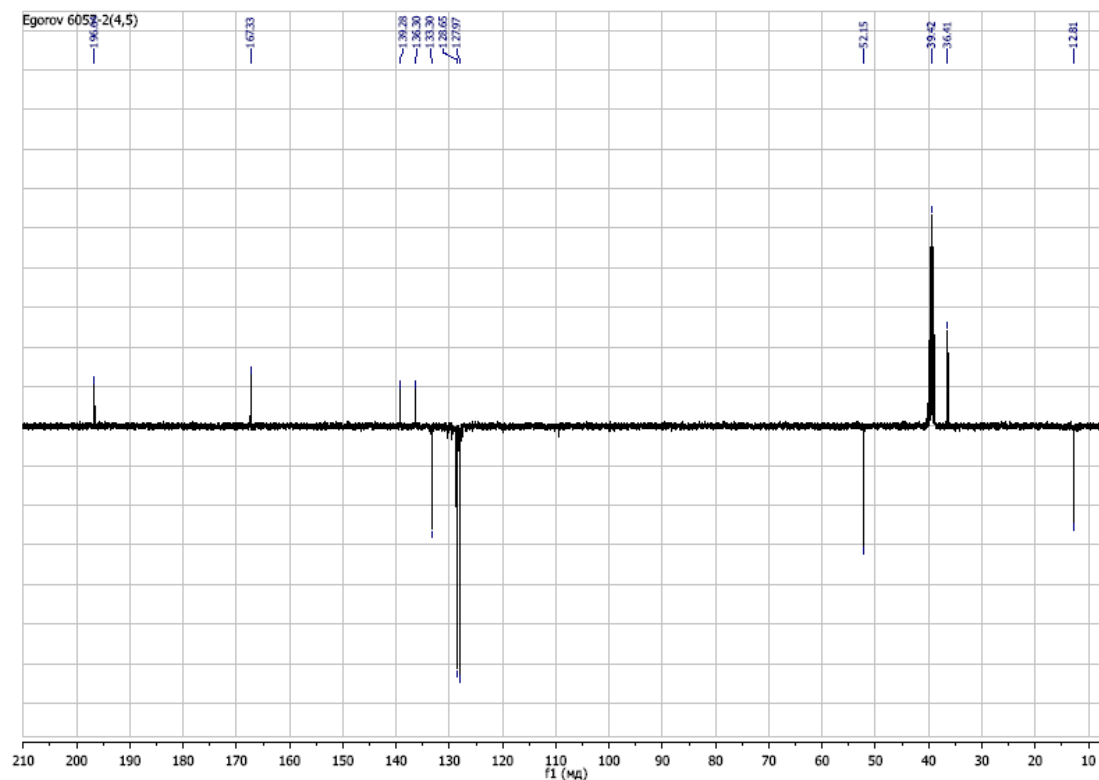
6-(2-Oxo-2-phenylethyl)-3-phenyl-1,6-dihydro-1,2,4-triazin-5(4H)-one (3a). ^{13}C NMR (100MHz, CDCl_3)



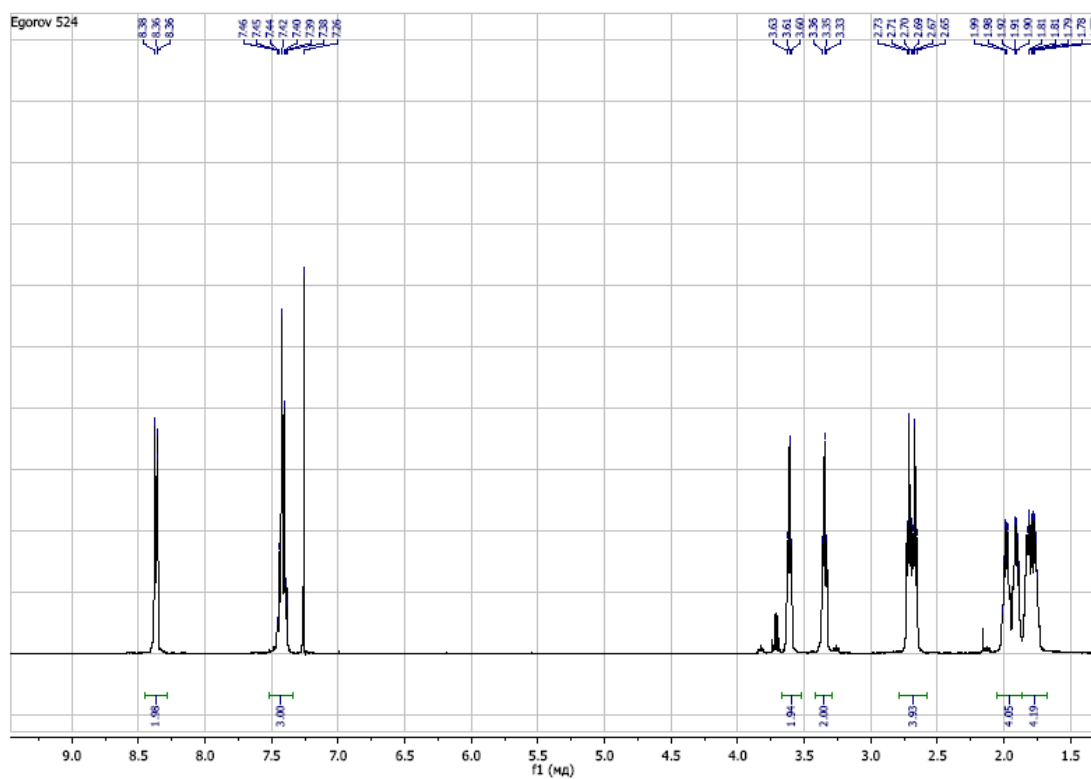
3-(Methylthio)-6-(2-oxo-2-phenylethyl)-1,6-dihydro-1,2,4-triazin-5(4H)-one (3b). ^1H NMR (400 MHz, DMSO- d_6)



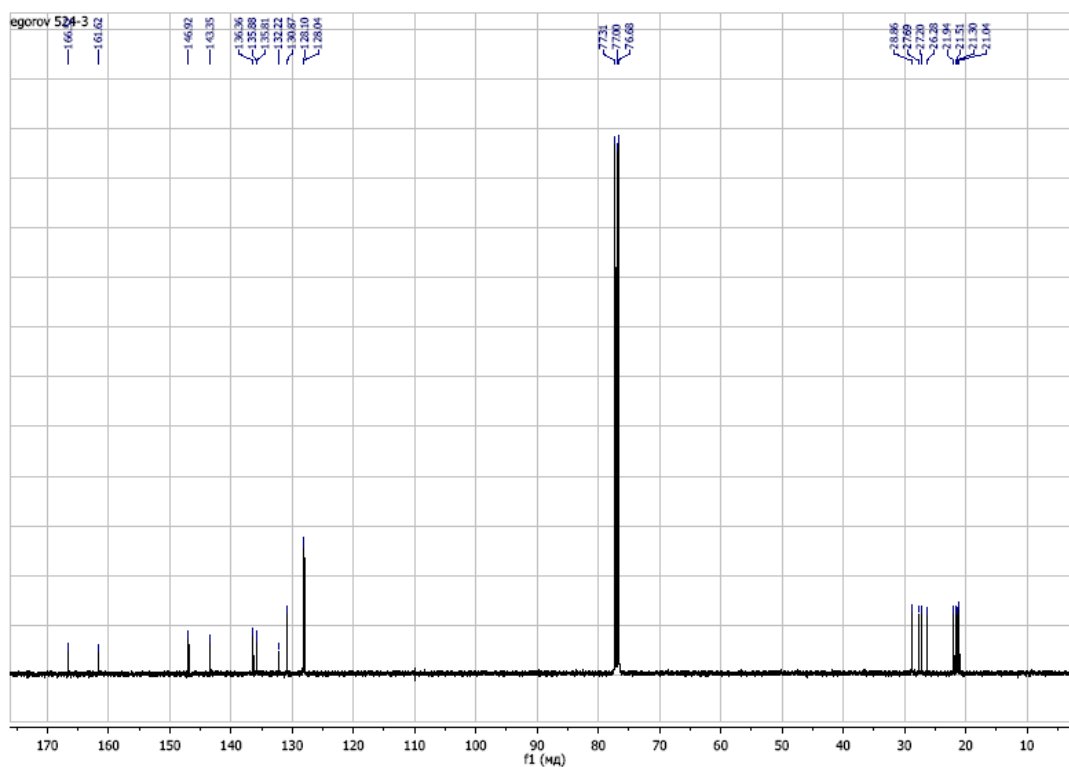
3-(Methylthio)-6-(2-oxo-2-phenylethyl)-1,6-dihydro-1,2,4-triazin-5(4H)-one (3b). ^{13}C NMR (100MHz, DMSO- d_6)



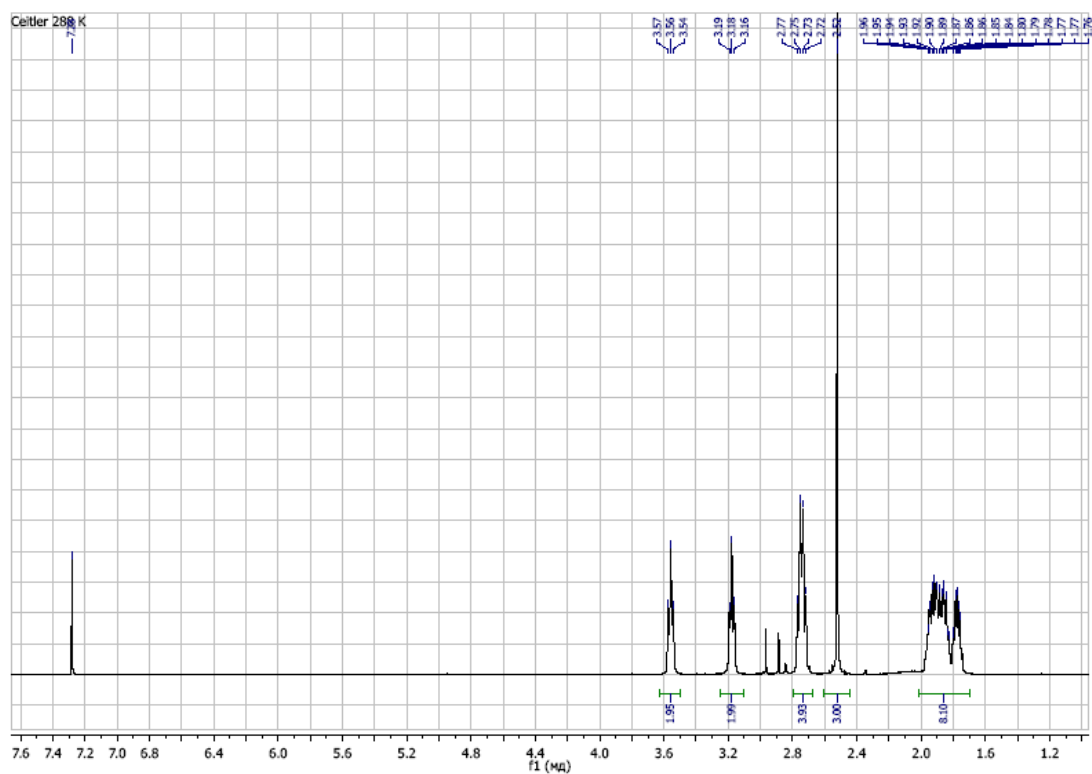
2-Phenyl-5,6,7,8,9,10,11,12-octahydro[1,2,4]triazino[1,6-f]phenanthridin-13-ium-4-olate (6a). ^1H NMR (400 MHz, CDCl_3)



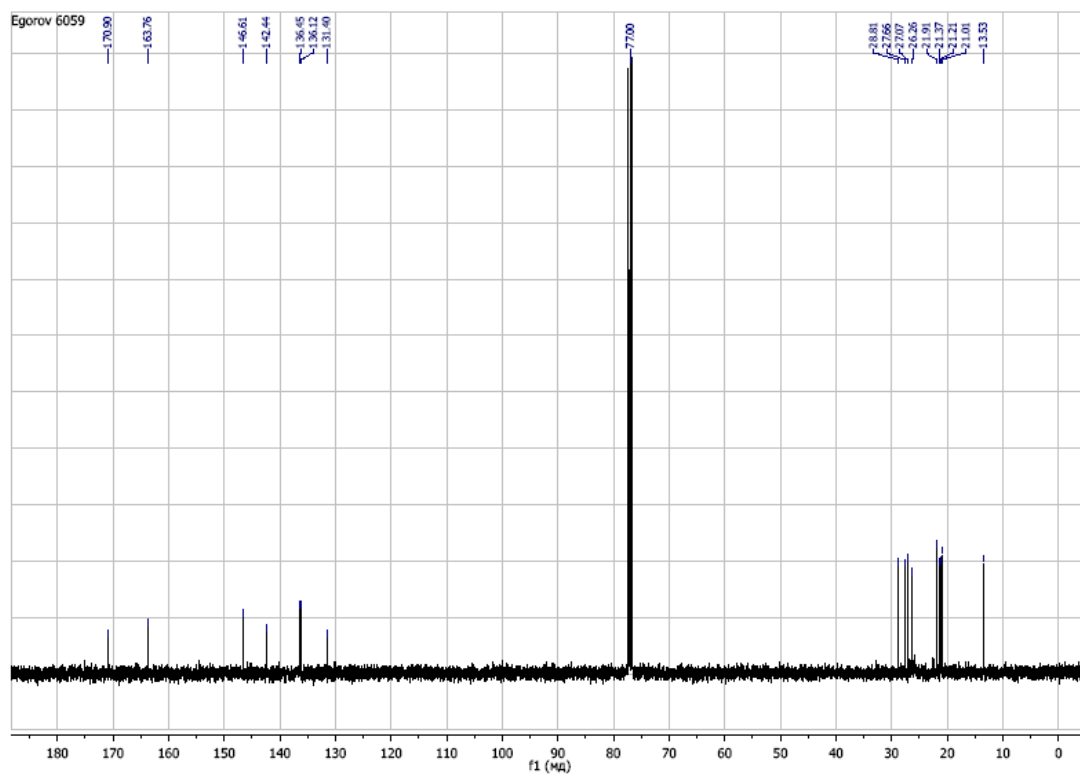
2-Phenyl-5,6,7,8,9,10,11,12-octahydro[1,2,4]triazino[1,6-f]phenanthridin-13-ium-4-olate (6a). ^{13}C NMR (100MHz, CDCl_3)



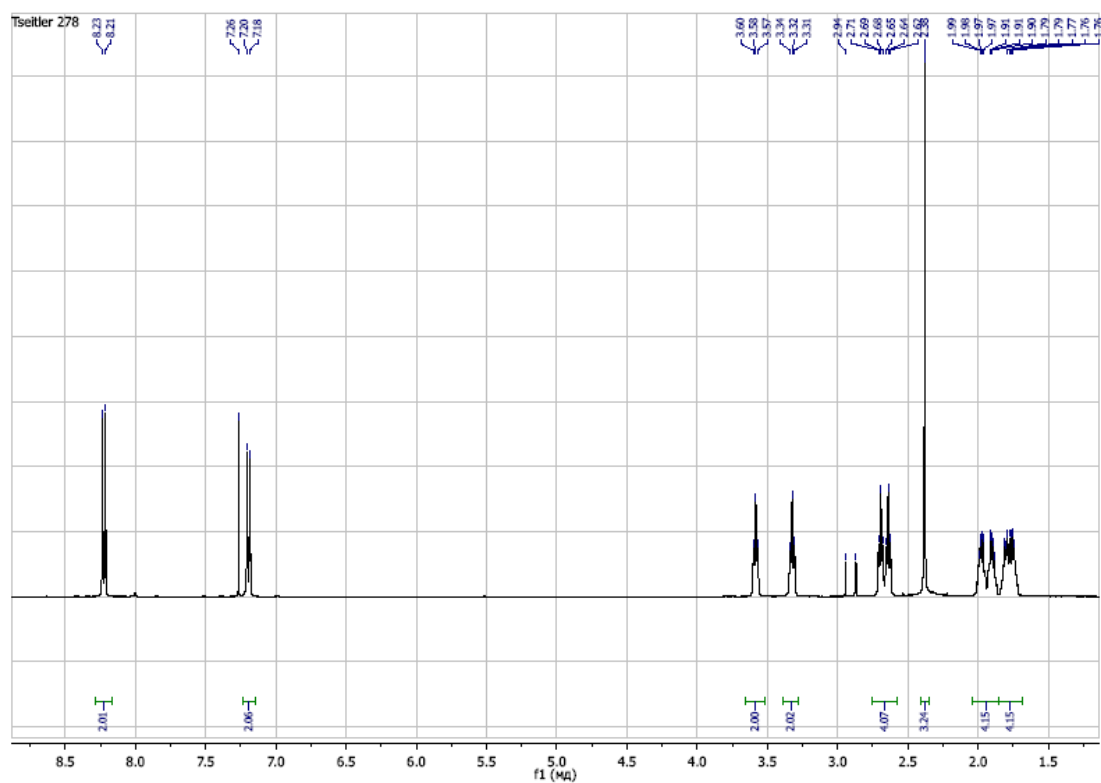
2-(Methylthio)-5,6,7,8,9,10,11,12-octahydro-[1,2,4]triazino[1,6-f]phenanthridin-13-ium-4-olate (6b). ^1H NMR (400 MHz, CDCl_3)



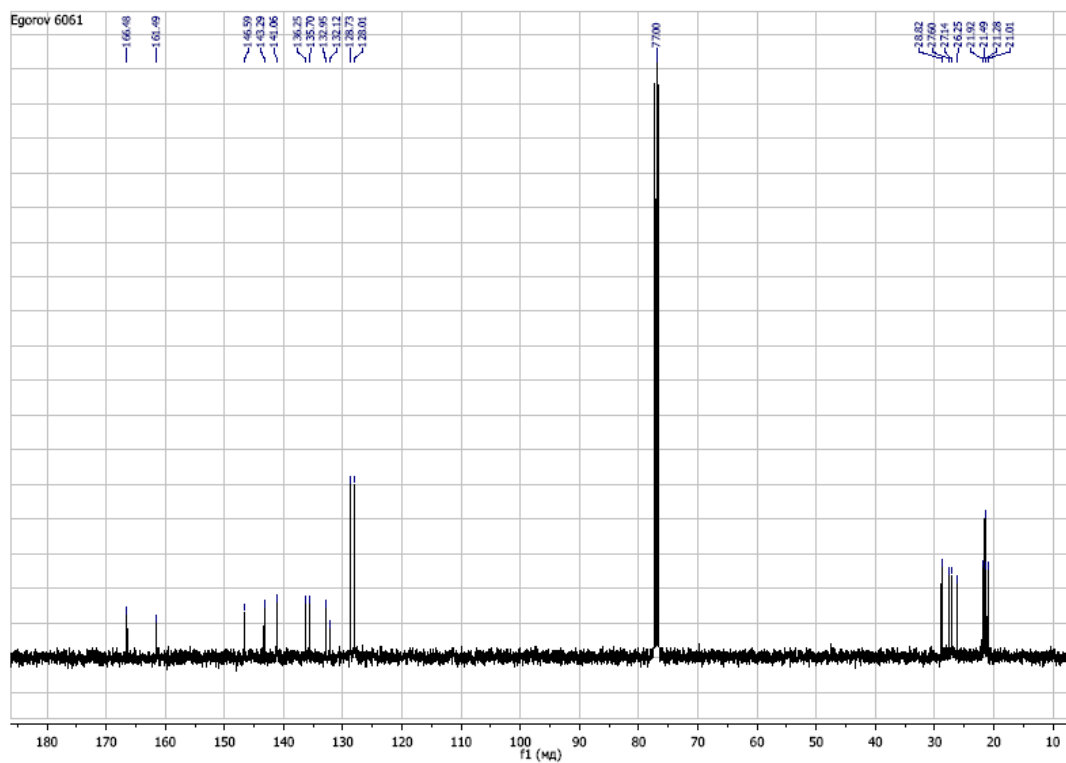
2-(Methylthio)-5,6,7,8,9,10,11,12-octahydro-[1,2,4]triazino[1,6-f]phenanthridin-13-ium-4-olate (6b). ^{13}C NMR (100MHz, CDCl_3)



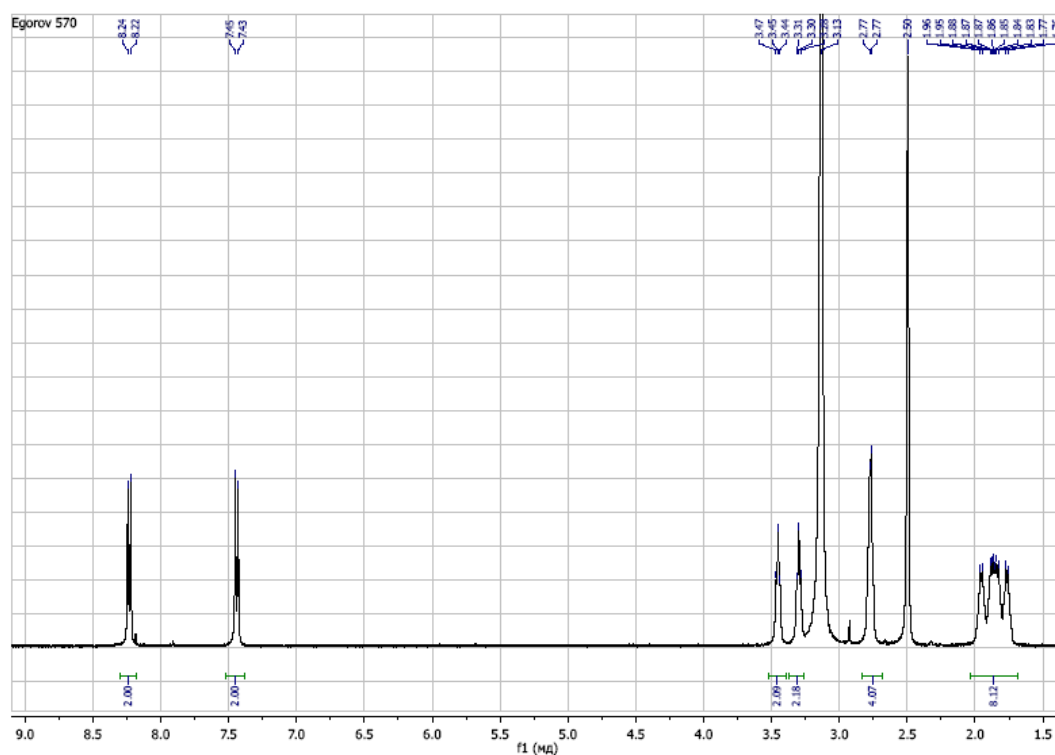
2-(p-Tolyl)-5,6,7,8,9,10,11,12-octahydro-[1,2,4]triazino[1,6-f]phenanthridin-13-ium-4-olate (6c). ^1H NMR (400 MHz, CDCl_3)



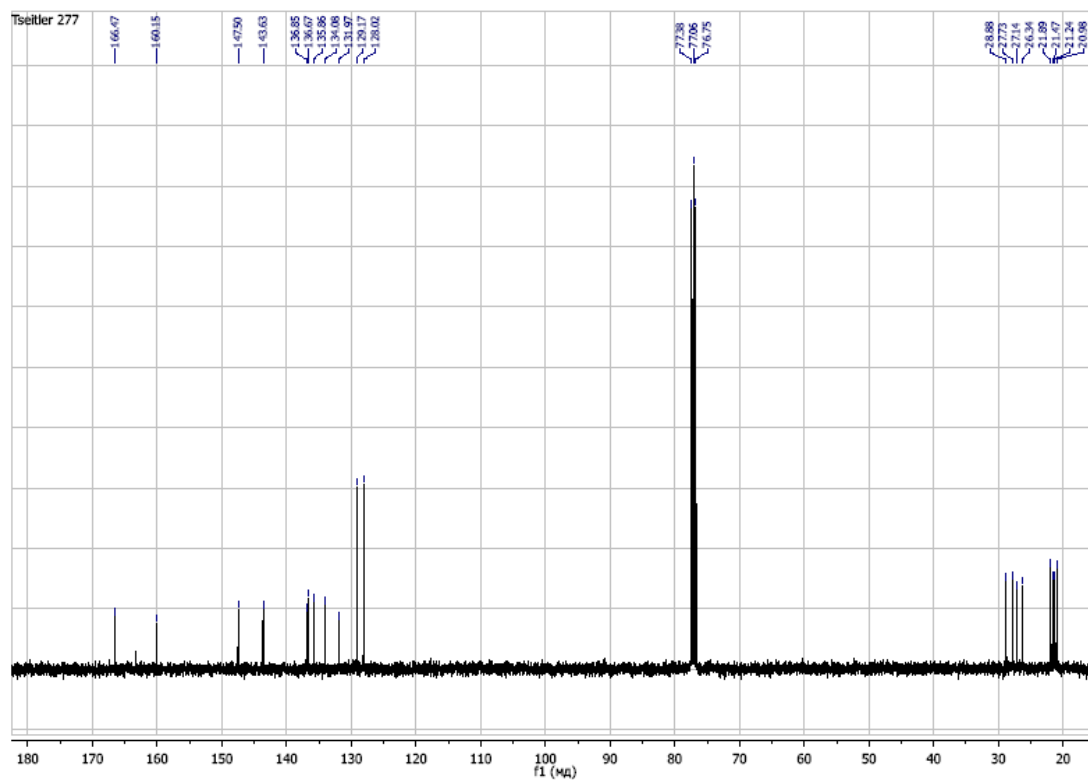
2-(p-Tolyl)-5,6,7,8,9,10,11,12-octahydro-[1,2,4]triazino[1,6-f]phenanthridin-13-ium-4-olate (6c). ^{13}C NMR (100MHz, DMSO-d_6)



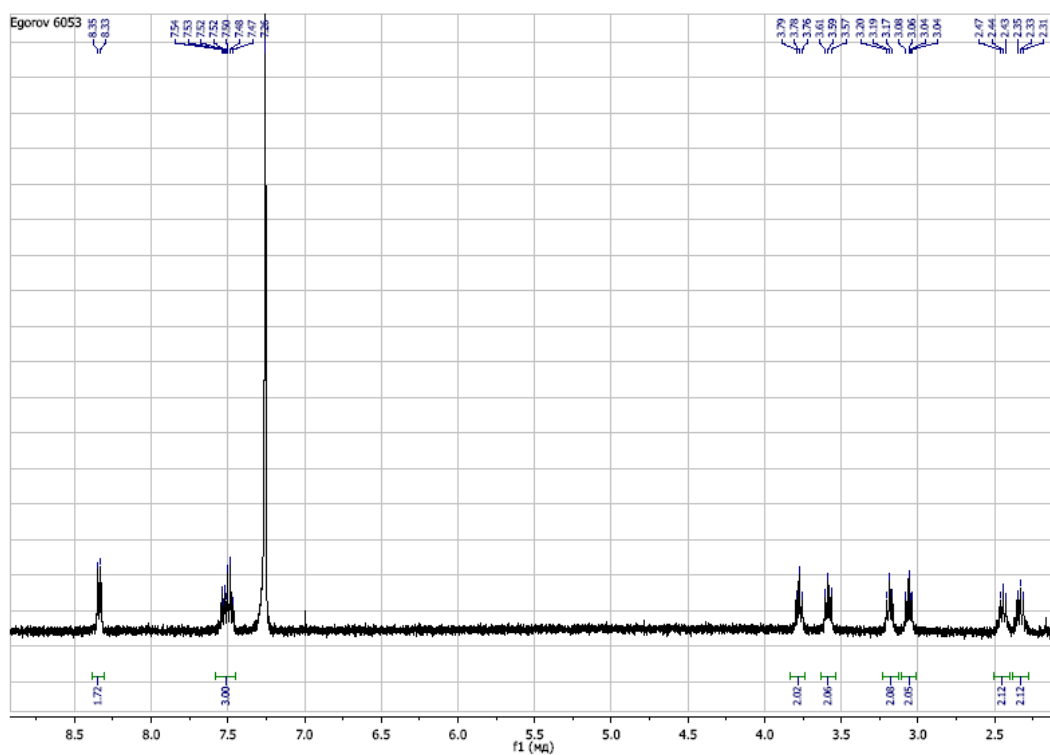
2-(4-Chlorophenyl)-5,6,7,8,9,10,11,12-octahydro-[1,2,4]triazino[1,6-f]phenanthridin-13-ium-4-olate (6d).
¹H NMR (400 MHz, CDCl₃)



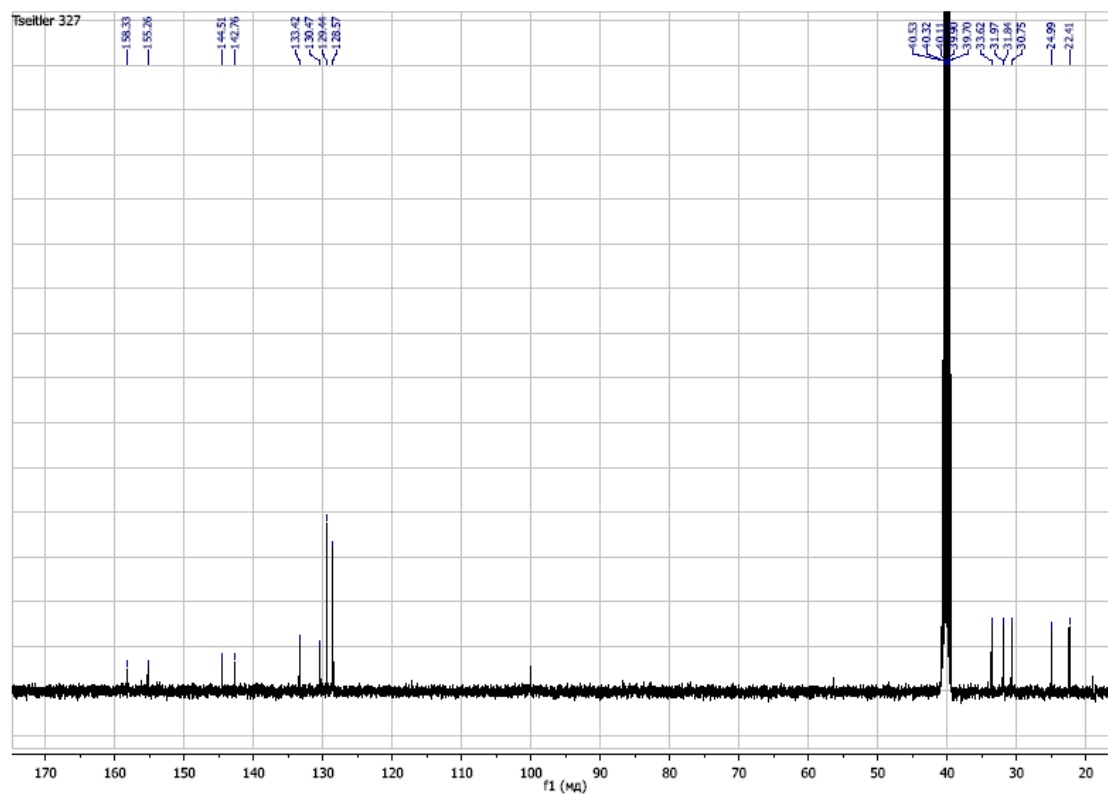
2-(4-Chlorophenyl)-5,6,7,8,9,10,11,12-octahydro-[1,2,4]triazino[1,6-f]phenanthridin-13-ium-4-olate (6d).
¹³C NMR (100MHz, CDCl₃)



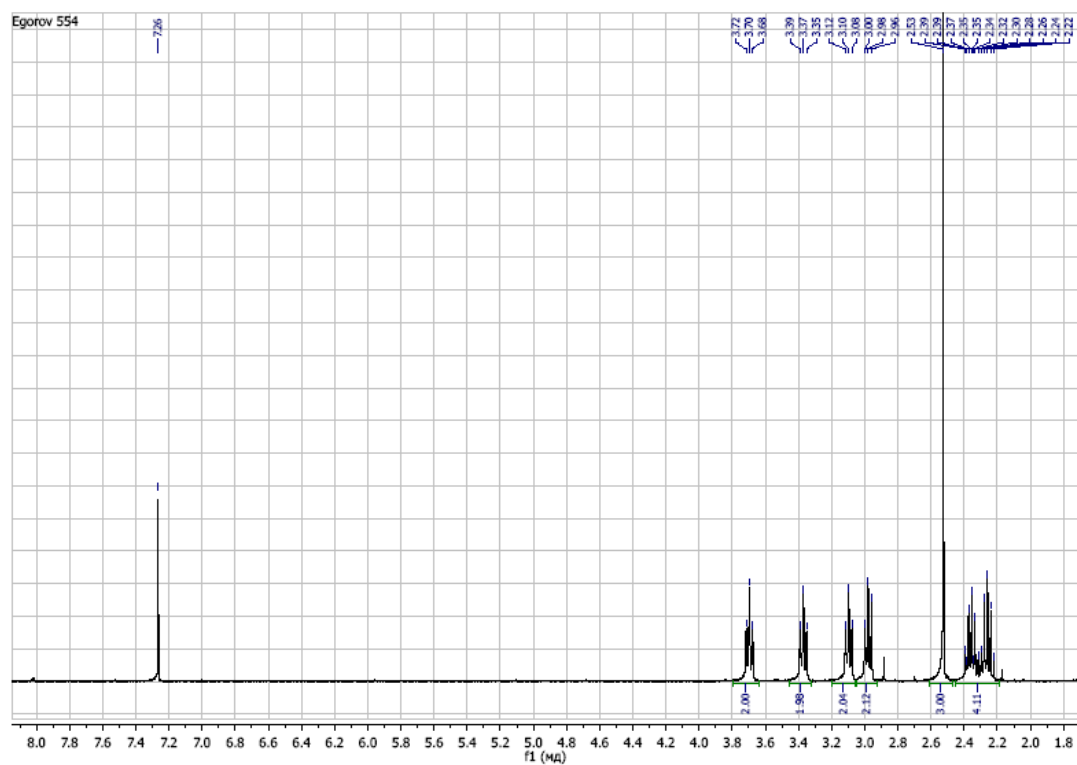
2-Phenyl-5,6,7,8,9,10-hexahydrodicyclopenta[3,4:5,6]pyrido[2,1-f][1,2,4]triazin-11-ium-4-olate (7a). ^1H NMR (400 MHz, CDCl_3)



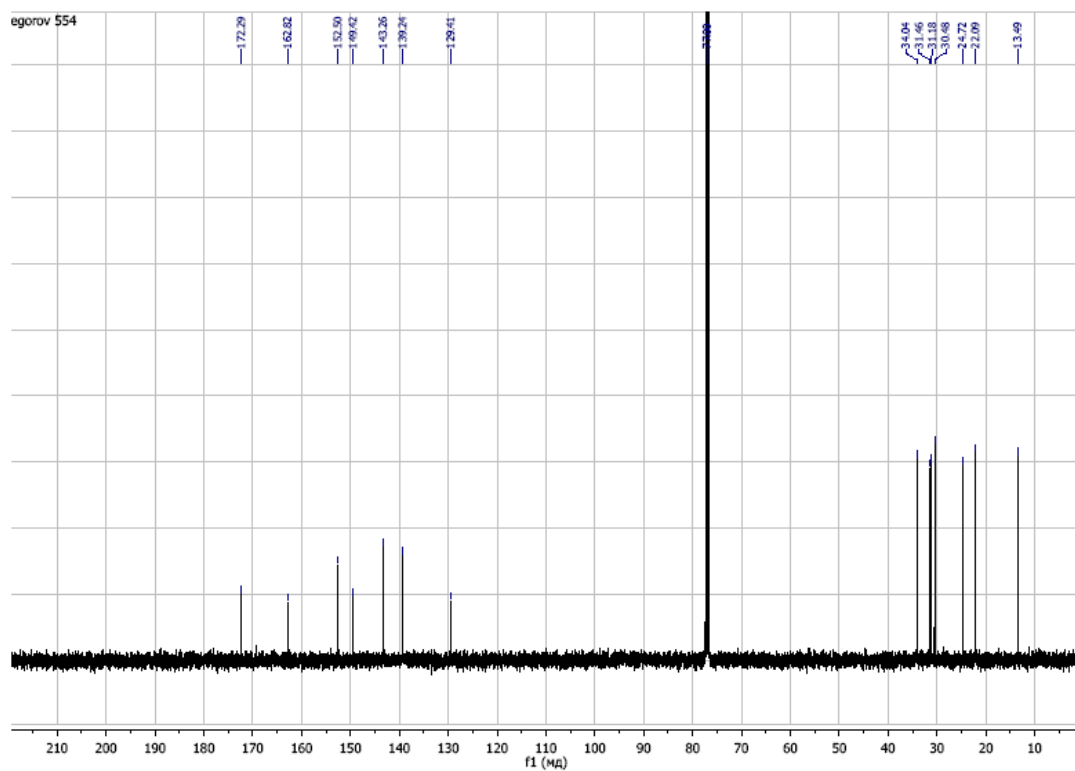
2-Phenyl-5,6,7,8,9,10-hexahydrodicyclopenta[3,4:5,6]pyrido[2,1-f][1,2,4]triazin-11-ium-4-olate (7a). ^{13}C NMR (100MHz, $\text{DMSO}-d_6$)



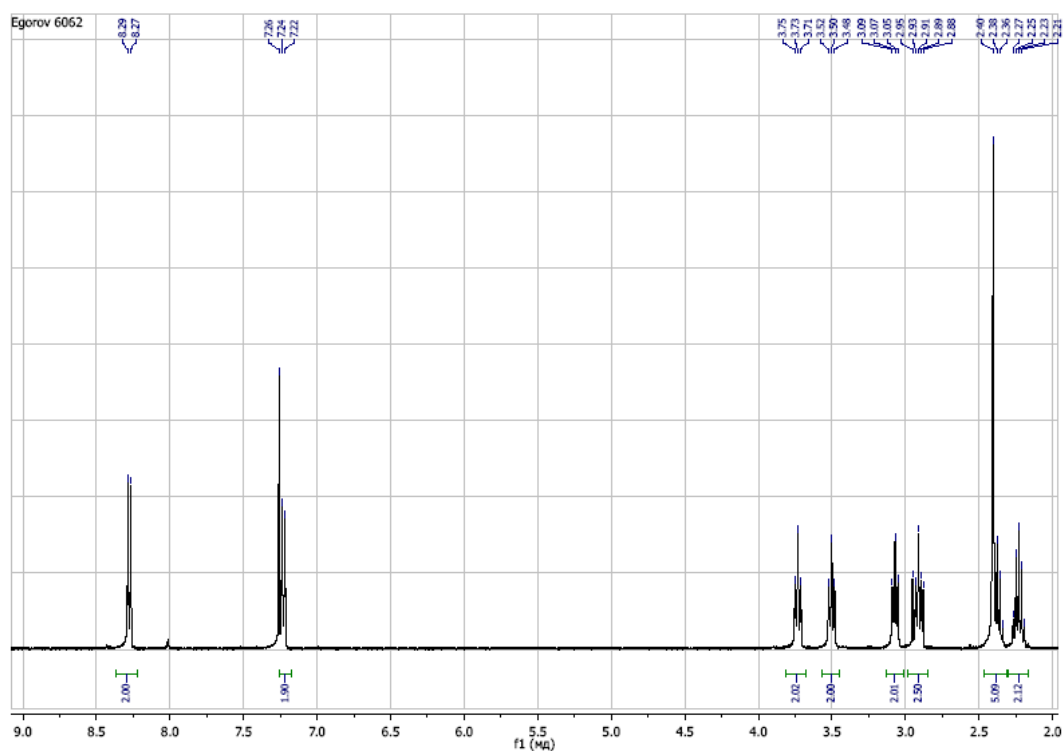
2-(Methylthio)-5,6,7,8,9,10-hexahydrodicyclopenta[3,4:5,6]pyrido[2,1-f][1,2,4]triazin-11-ium-4-olate (7b).
¹H NMR (400 MHz, CDCl₃)



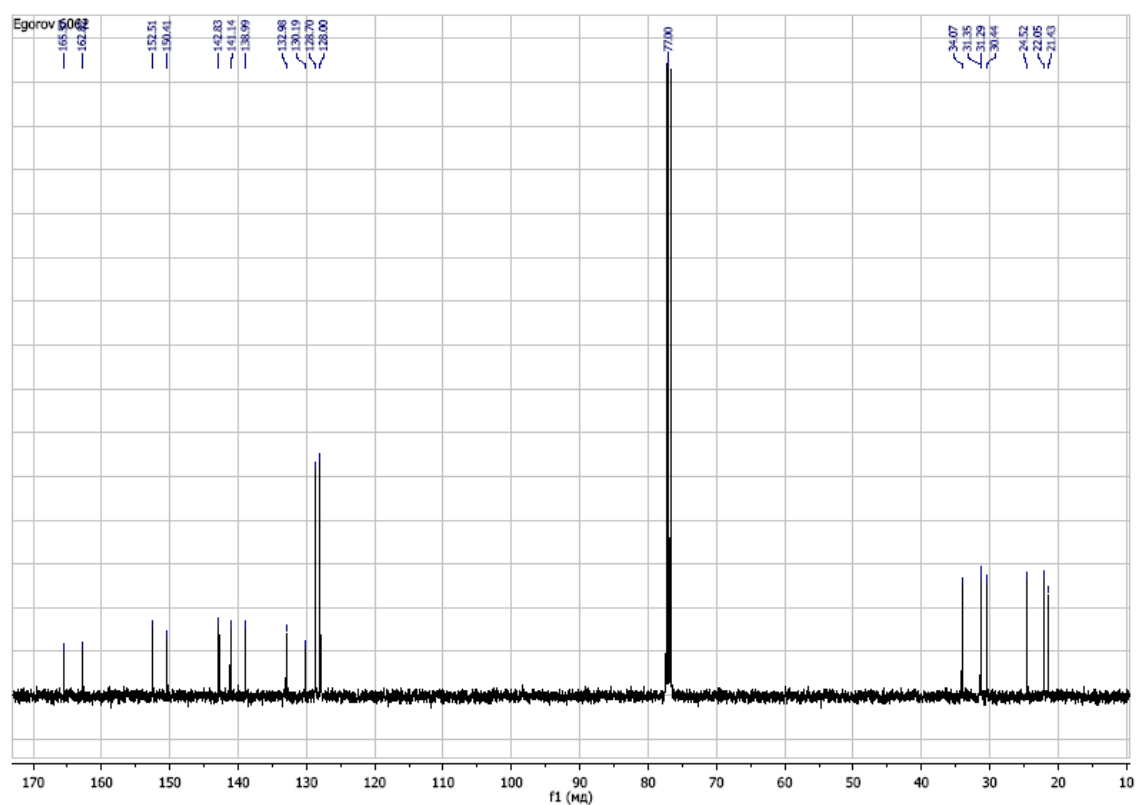
2-(Methylthio)-5,6,7,8,9,10-hexahydrodicyclopenta[3,4:5,6]pyrido[2,1-f][1,2,4]triazin-11-ium-4-olate (7b).
¹³C NMR (100MHz, CDCl₃)



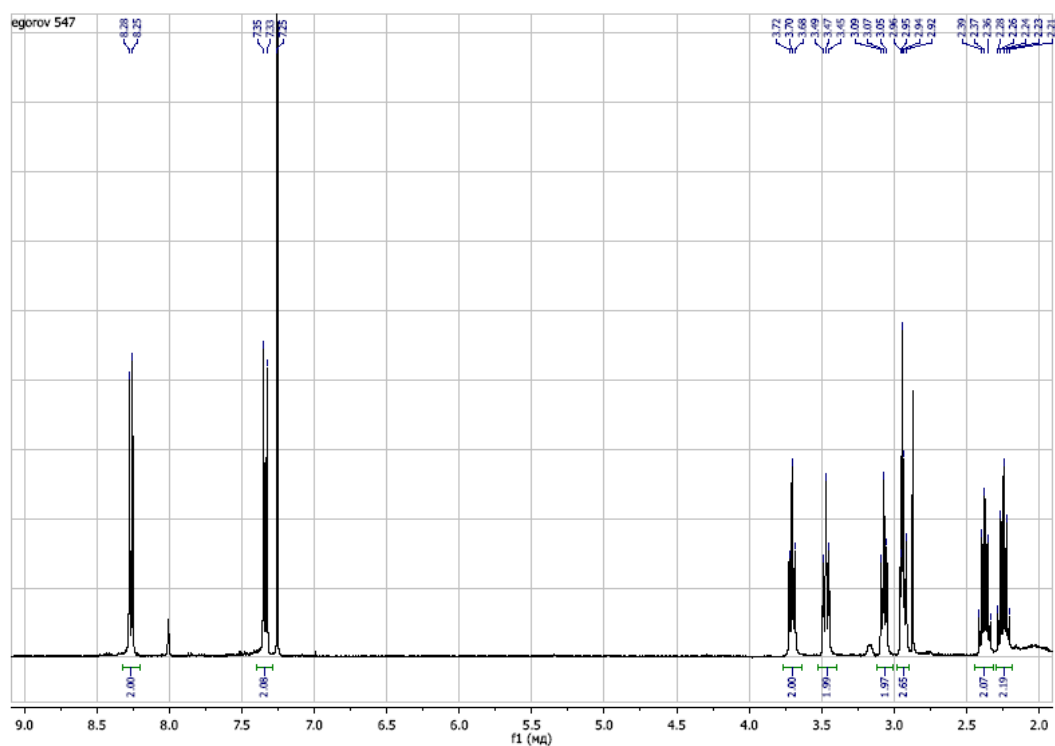
2-(p-Tolyl)-5,6,7,8,9,10-hexahydrodicyclopenta[3,4:5,6]pyrido[2,1-f][1,2,4]triazin-11-ium-4-olate (7c). ^1H NMR (400 MHz, CDCl_3)



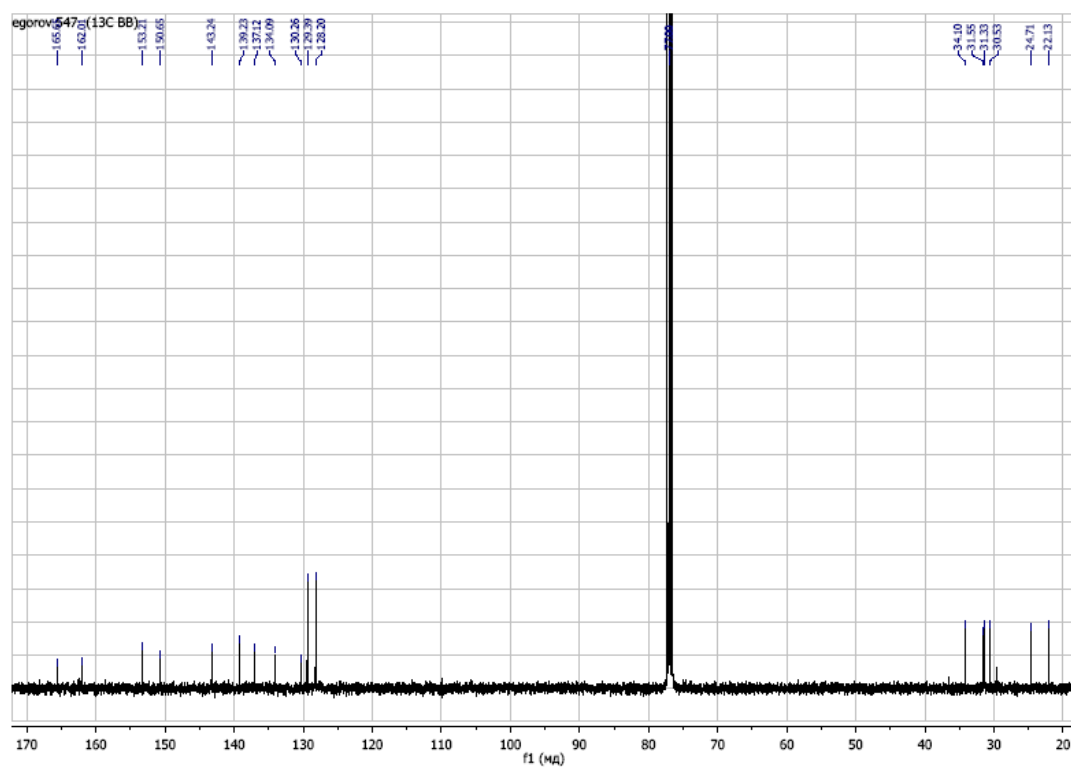
2-(p-Tolyl)-5,6,7,8,9,10-hexahydrodicyclopenta[3,4:5,6]pyrido[2,1-f][1,2,4]triazin-11-ium-4-olate (7c). ^{13}C NMR (100MHz, CDCl_3)



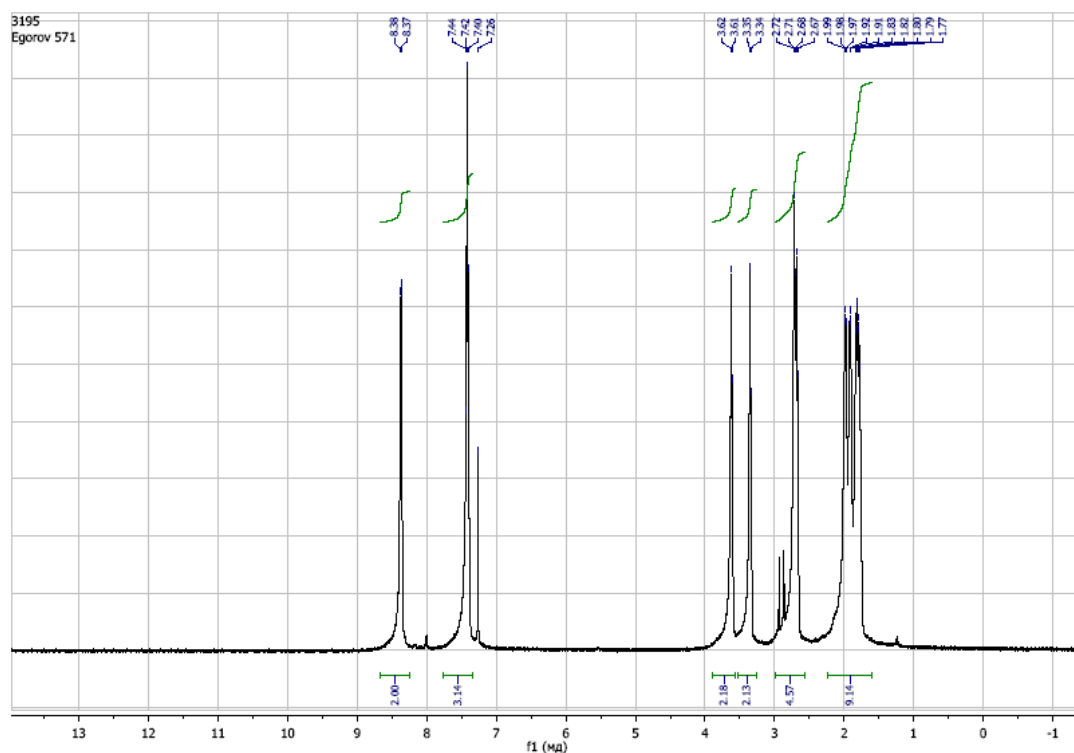
2-(4-Chlorophenyl)-5,6,7,8,9,10-hexahydrodicyclopenta[3,4:5,6]pyrido[2,1-f][1,2,4]triazin-11-ium-4-olate (7d). ^1H NMR (400 MHz, CDCl_3)



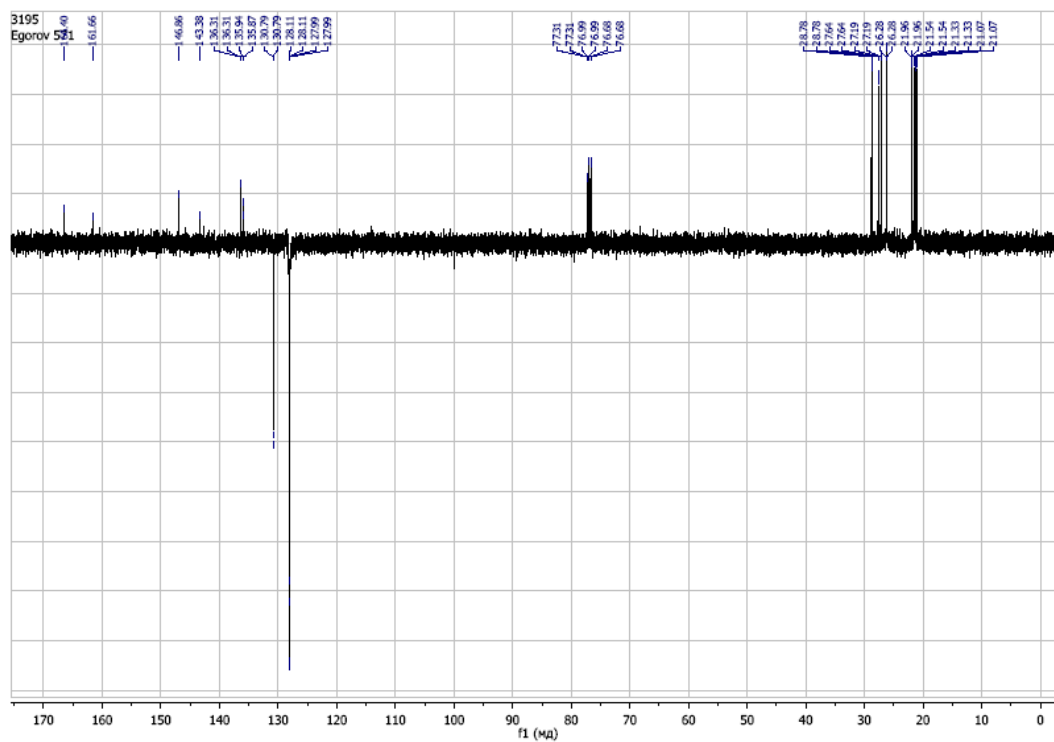
2-(4-Chlorophenyl)-5,6,7,8,9,10-hexahydrodicyclopenta[3,4:5,6]pyrido[2,1-f][1,2,4]triazin-11-ium-4-olate (7d). ^{13}C NMR (100MHz, CDCl_3)



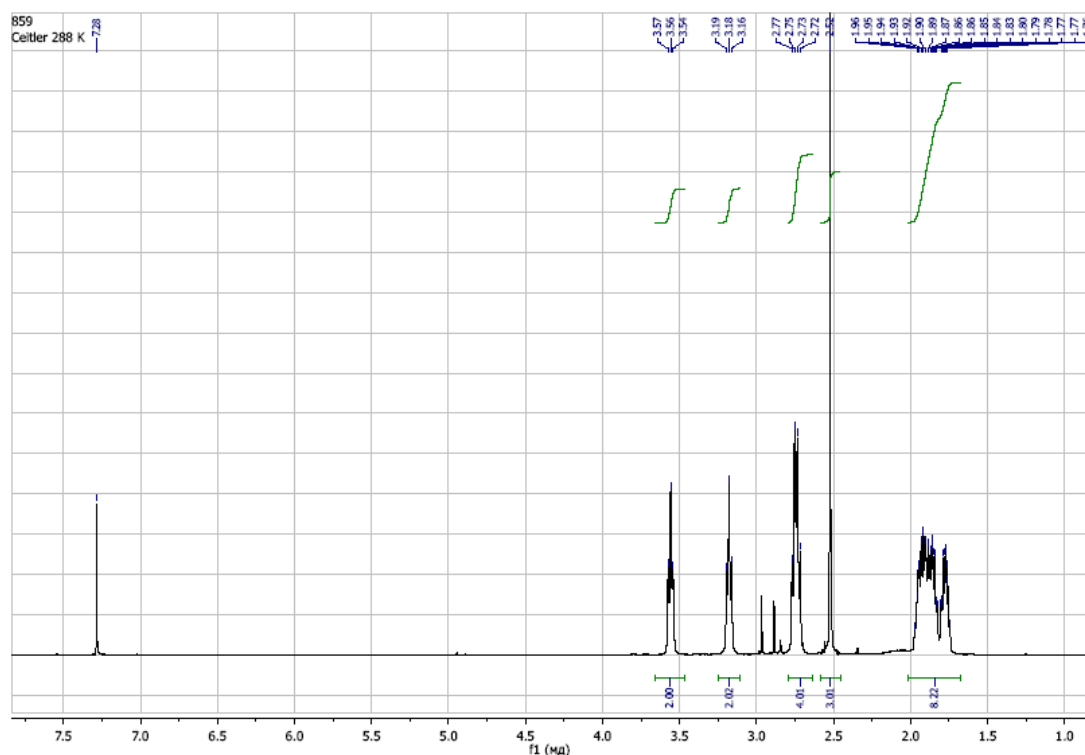
4-Oxo-2-phenyl-3,4,5,6,7,8,9,10,11,12-decahydro-[1,2,4]triazino[1,6-f]phenanthridin-13-ium trifluoromethanesulfonate (6aa). ^1H NMR (400 MHz, CDCl_3)



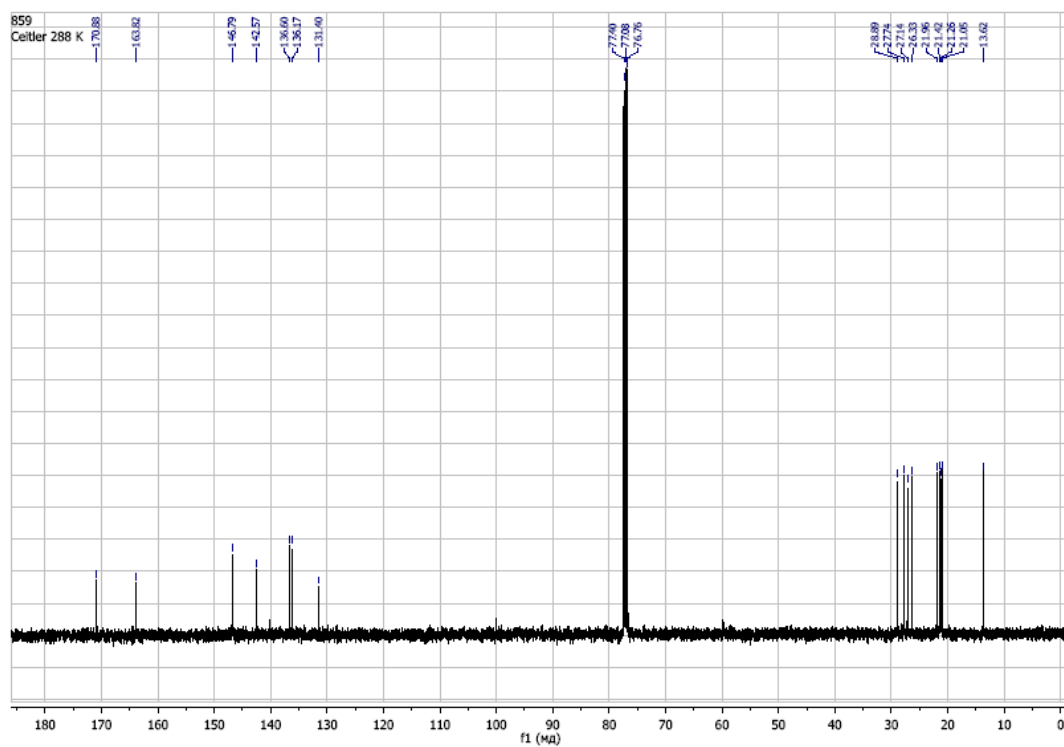
4-Oxo-2-phenyl-3,4,5,6,7,8,9,10,11,12-decahydro-[1,2,4]triazino[1,6-f]phenanthridin-13-ium trifluoromethanesulfonate (6aa). ^{13}C NMR (100MHz, CDCl_3)



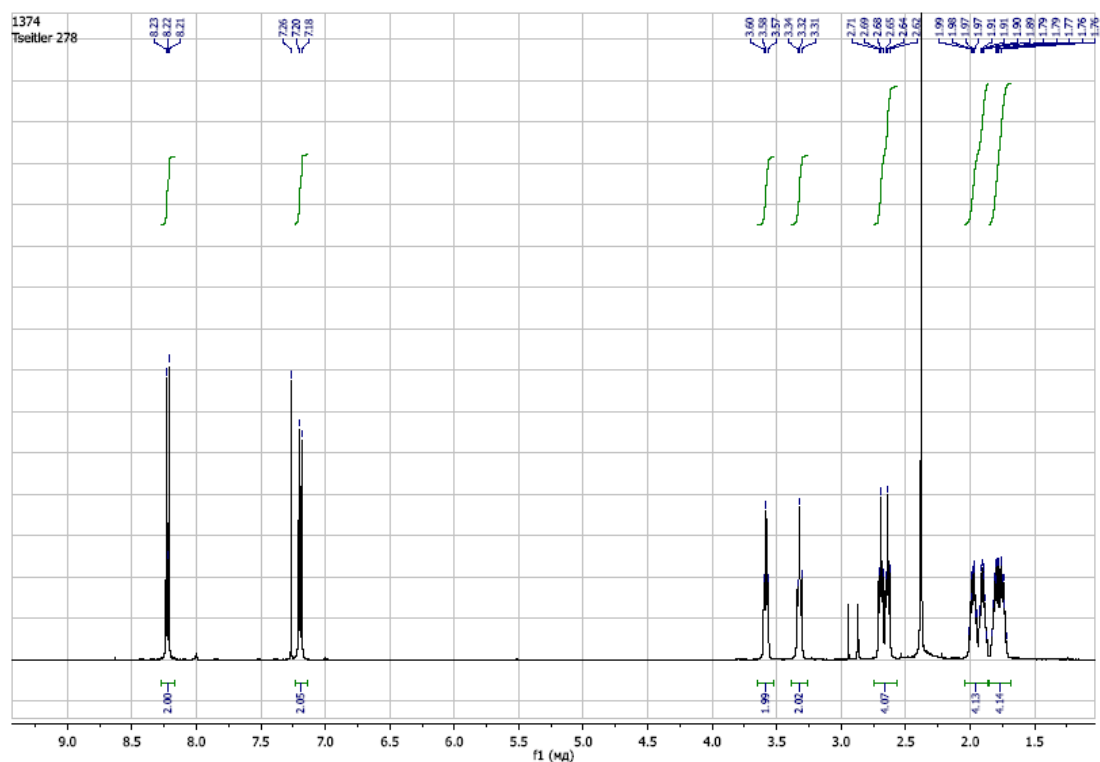
2-(Methylthio)-4-oxo-3,4,5,6,7,8,9,10,11,12-decahydro-[1,2,4]triazino[1,6-f]phenanthridin-13-ium trifluoromethanesulfonate (6ba). ^1H NMR (400MHz, CDCl_3)



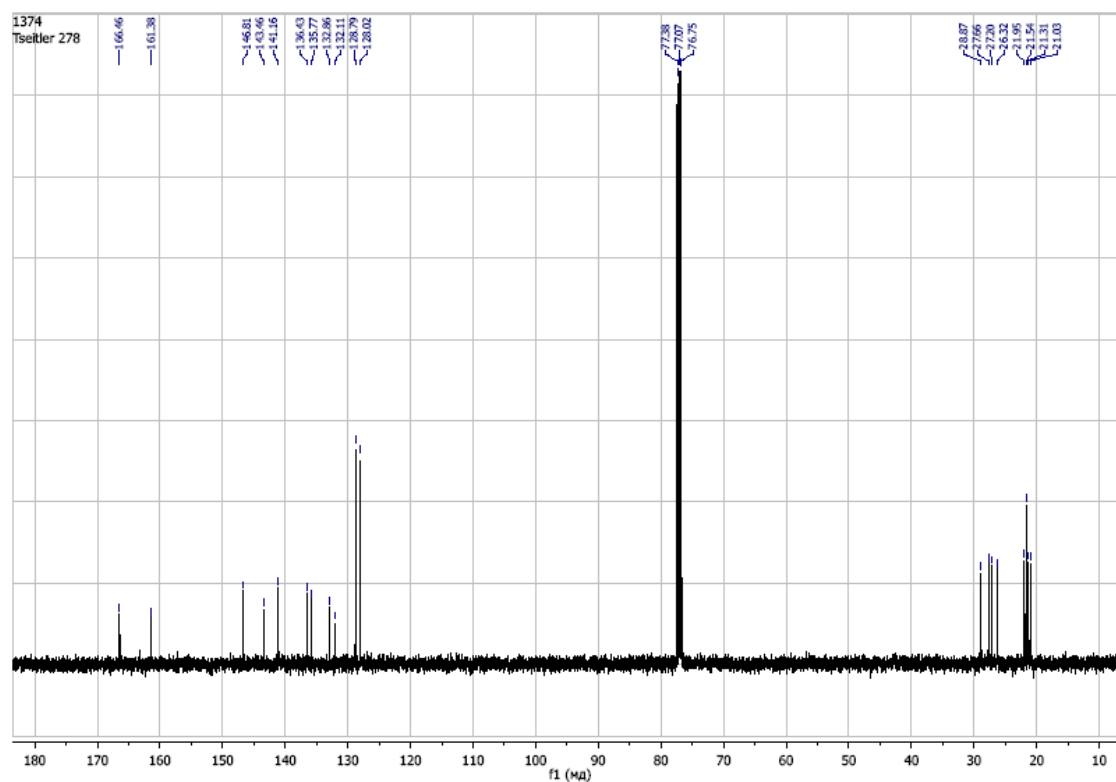
2-(Methylthio)-4-oxo-3,4,5,6,7,8,9,10,11,12-decahydro-[1,2,4]triazino[1,6-f]phenanthridin-13-ium trifluoromethanesulfonate (6ba). ^{13}C NMR (400MHz, CDCl_3)



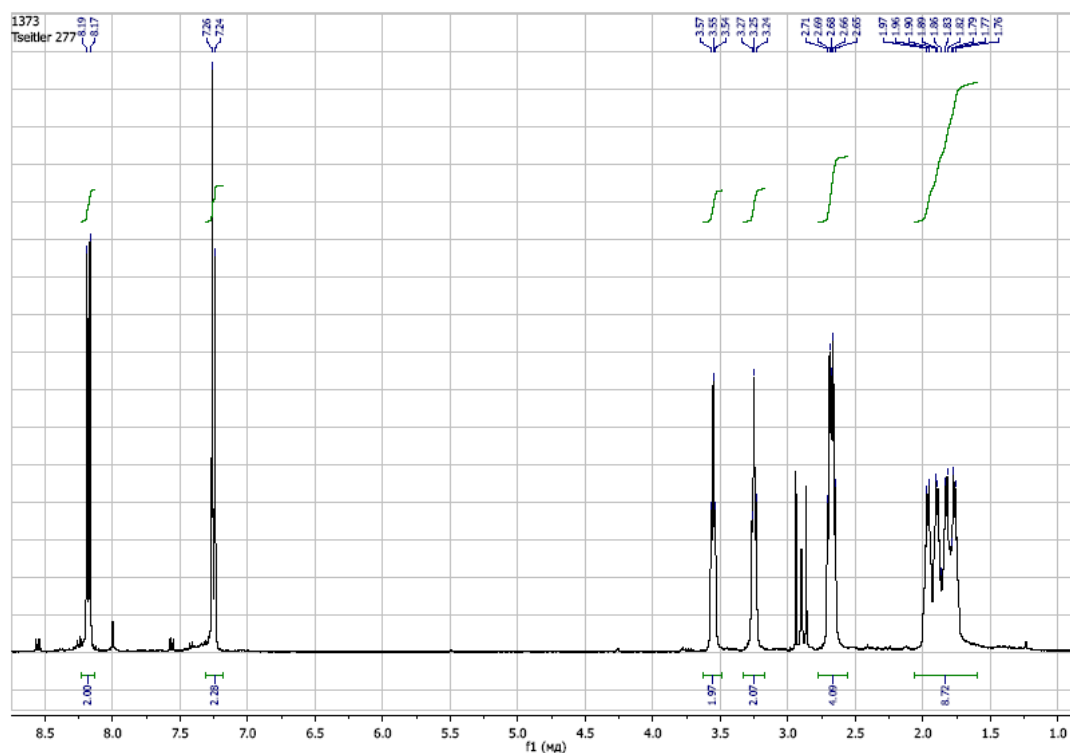
2-(4-Methylphenyl)-4-oxo-3,4,5,6,7,8,9,10,11,12-decahydro-[1,2,4]triazino[1,6-f]phenanthridin-13-ium trifluoromethanesulfonate (6ca). ^1H NMR (400MHz, CDCl_3)



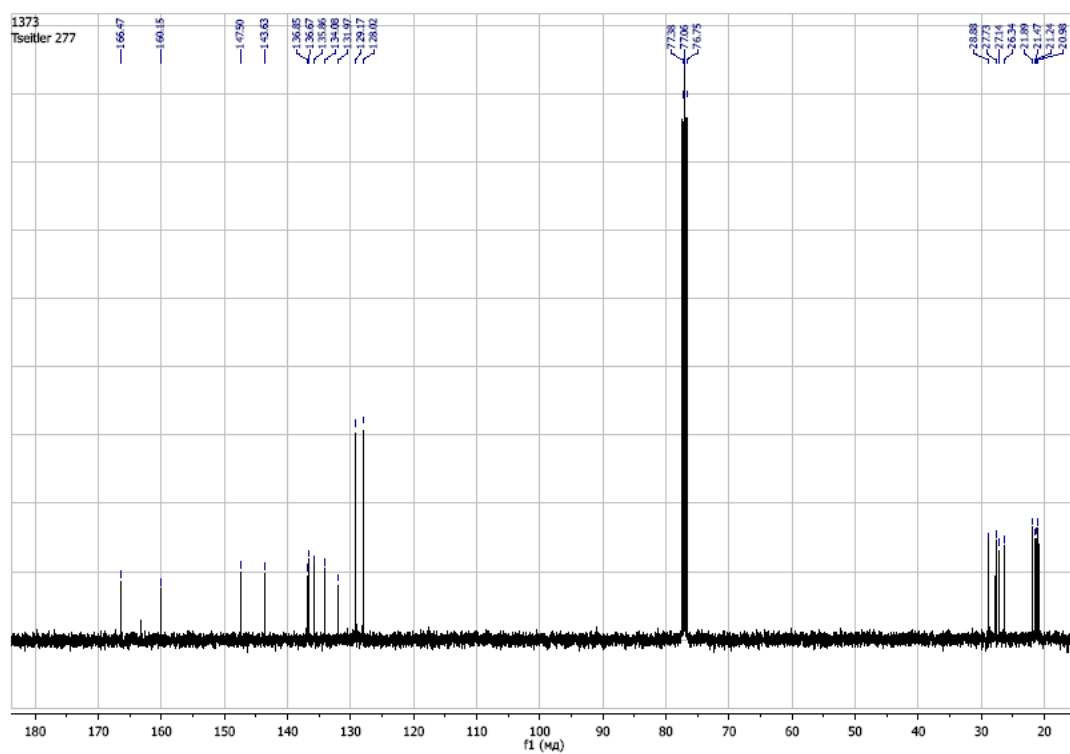
2-(4-Methylphenyl)-4-oxo-3,4,5,6,7,8,9,10,11,12-decahydro-[1,2,4]triazino[1,6-f]phenanthridin-13-ium trifluoromethanesulfonate (6ca). NMR ^{13}C (100MHz, CDCl_3)



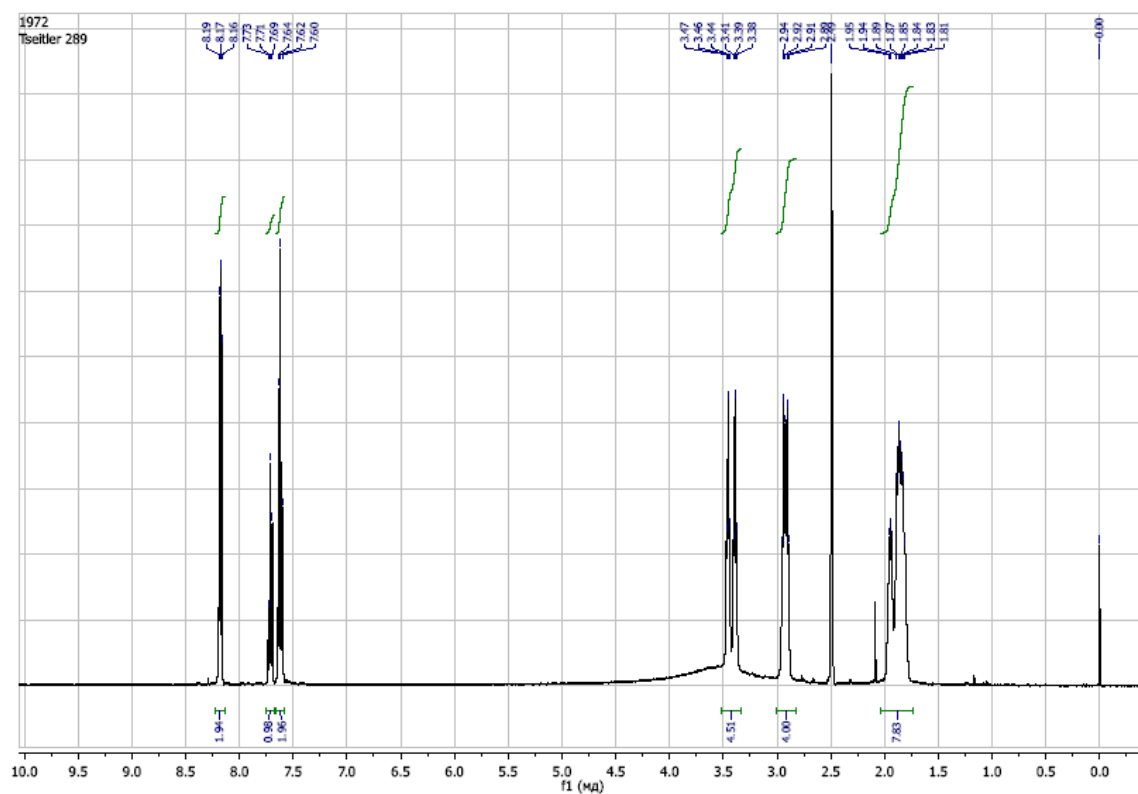
2-(4-Chlorophenyl)-4-oxo-3,4,5,6,7,8,9,10,11,12-decahydro-[1,2,4]triazino[1,6-f]phenanthridin-13-ium trifluoromethanesulfonate (6da). ¹H NMR (400MHz, CDCl₃)



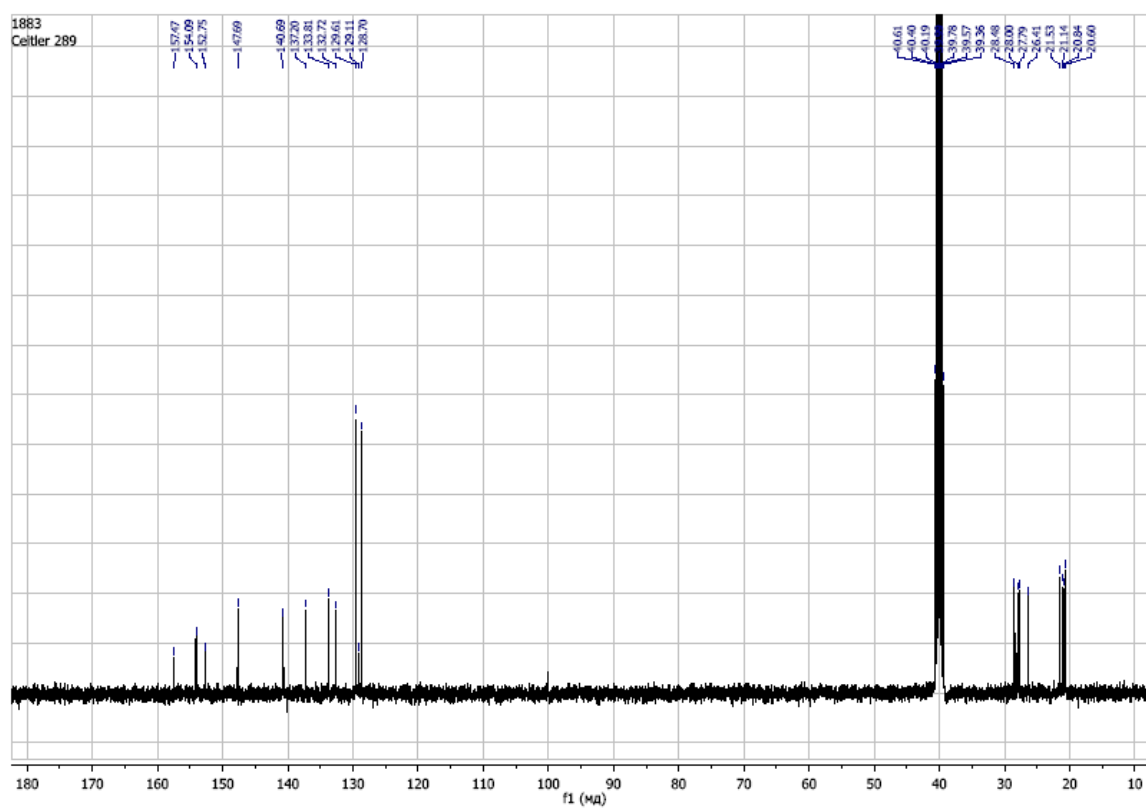
2-(4-Chlorophenyl)-4-oxo-3,4,5,6,7,8,9,10,11,12-decahydro-[1,2,4]triazino[1,6-f]phenanthridin-13-ium trifluoromethanesulfonate (6da). ¹³C NMR (100MHz, CDCl₃)



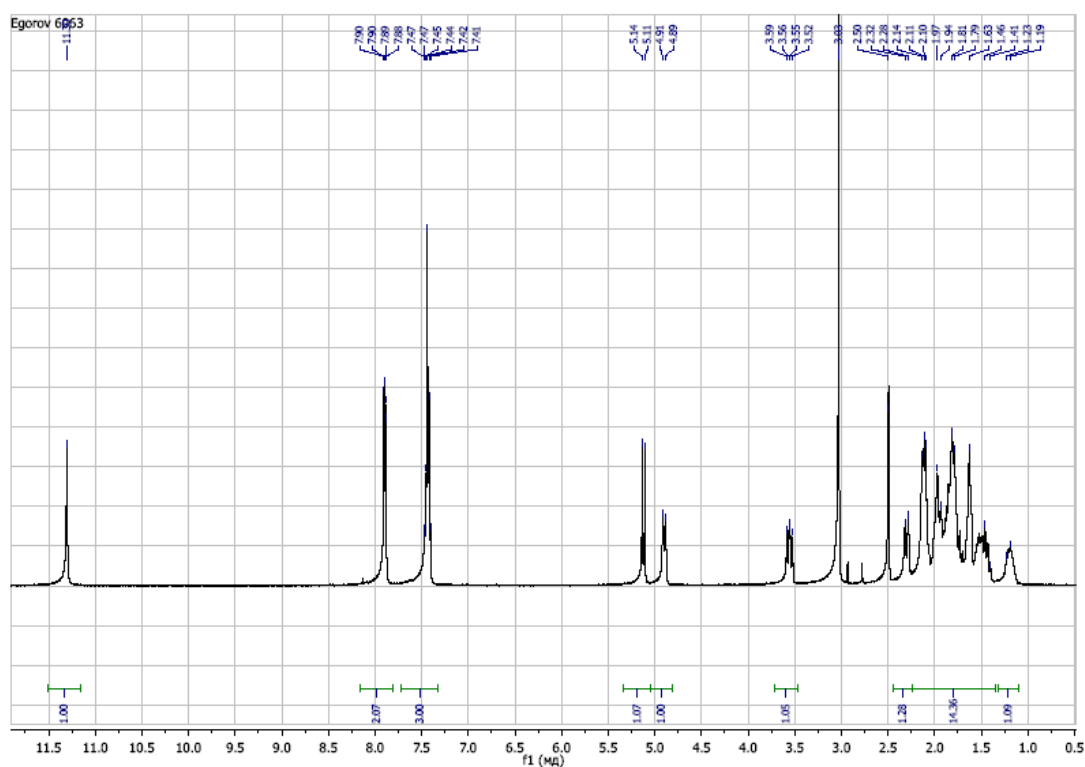
4-Oxo-2-phenyl-3,4,5,6,7,8,9,10,11,12-decahydro-[1,2,4]triazino[1,6-f]phenanthridin-13-ium chloride 6ab.



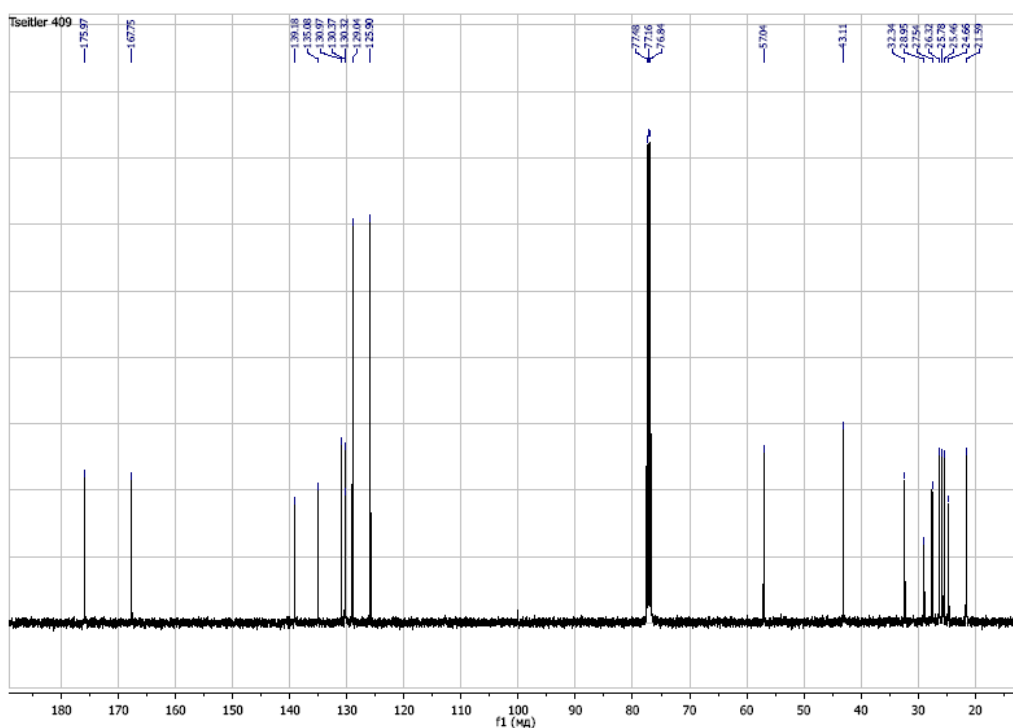
4-Oxo-2-phenyl-3,4,5,6,7,8,9,10,11,12-decahydro-[1,2,4]triazino[1,6-f]phenanthridin-13-ium chloride 6ab.



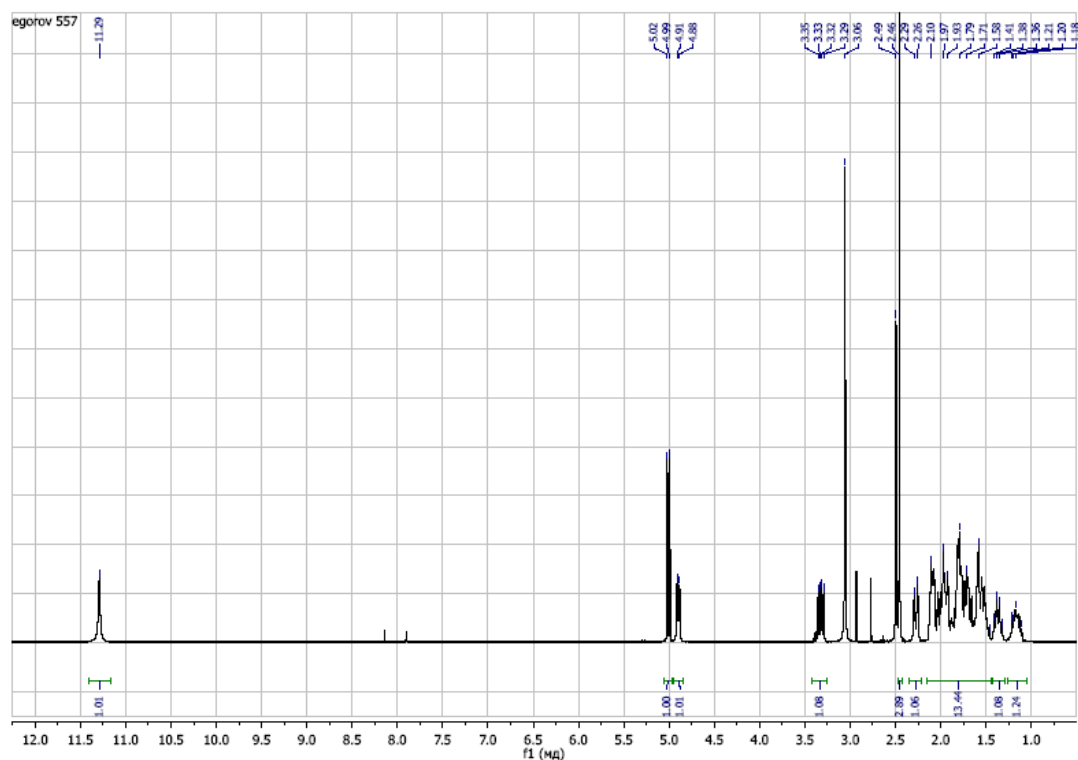
3-Phenyl-7,8,9,10,12,13,14,15,15a,15b-decahydro-1H-benzo[c][1,2,4]triazino[1,6-a]azecine-1,6(2H)-dione (10a). ^1H NMR (400 MHz, DMSO- d_6)



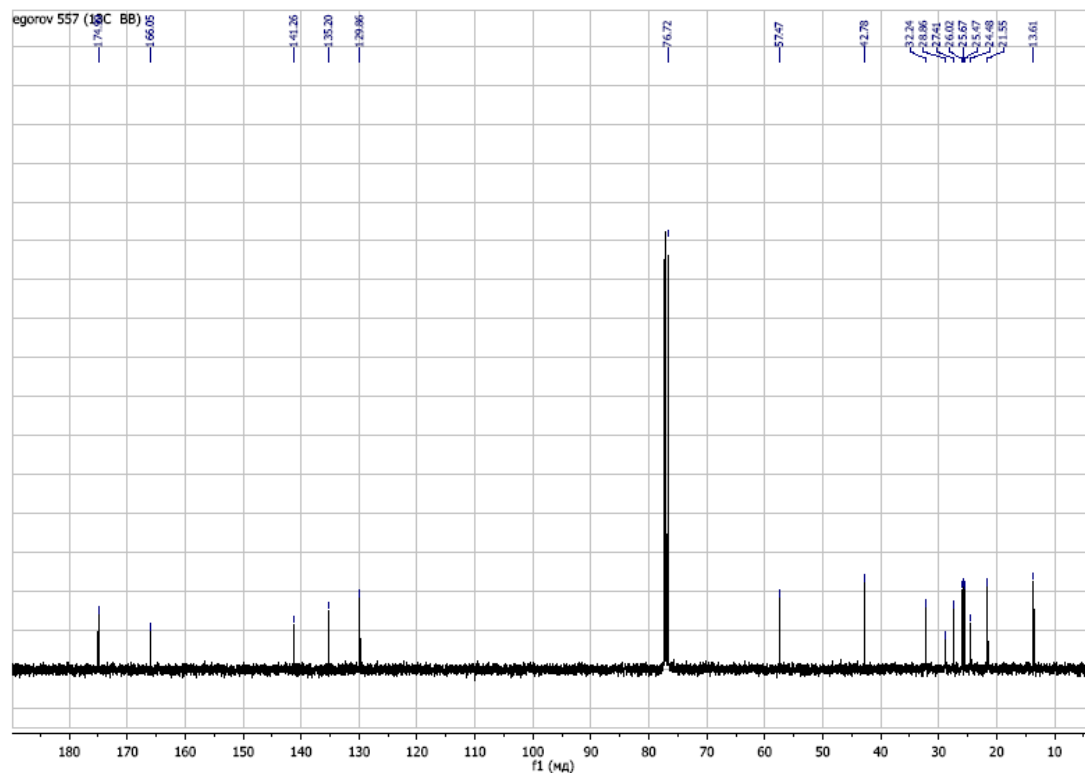
3-Phenyl-7,8,9,10,12,13,14,15,15a,15b-decahydro-1H-benzo[c][1,2,4]triazino[1,6-a]azecine-1,6(2H)-dione (10a). ^{13}C NMR (100MHz, CDCl_3)



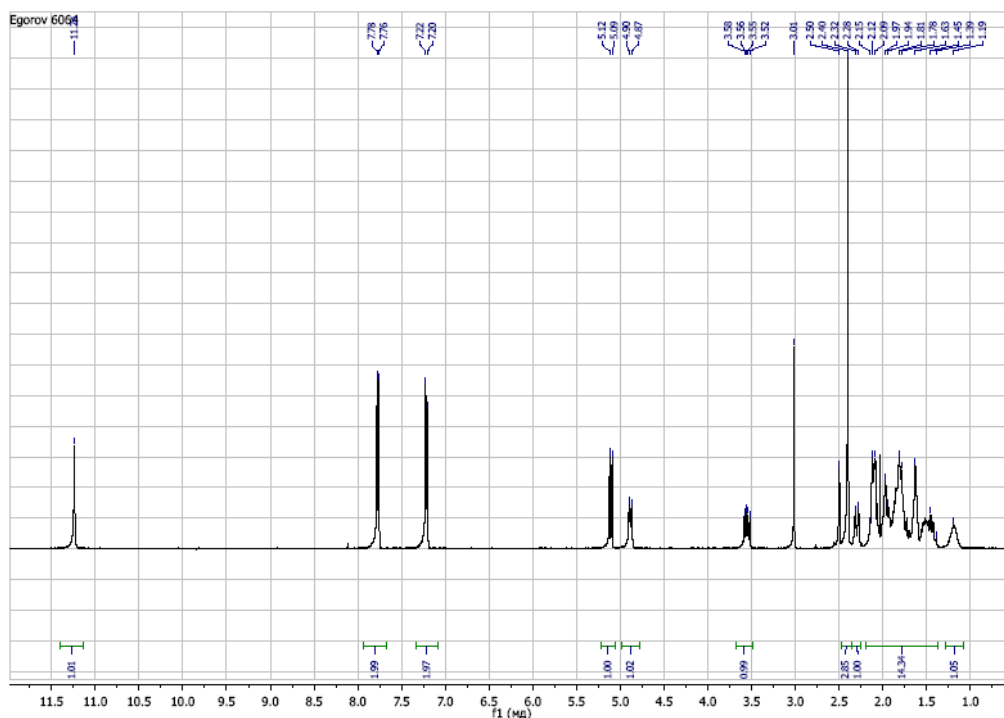
3-(Methylthio)-7,8,9,10,12,13,14,15,15a,15b-decahydro-1H-benzo[c][1,2,4]triazino[1,6-a]azecine-1,6(2H)-dione (10b). ^1H NMR (400 MHz, DMSO- d_6)



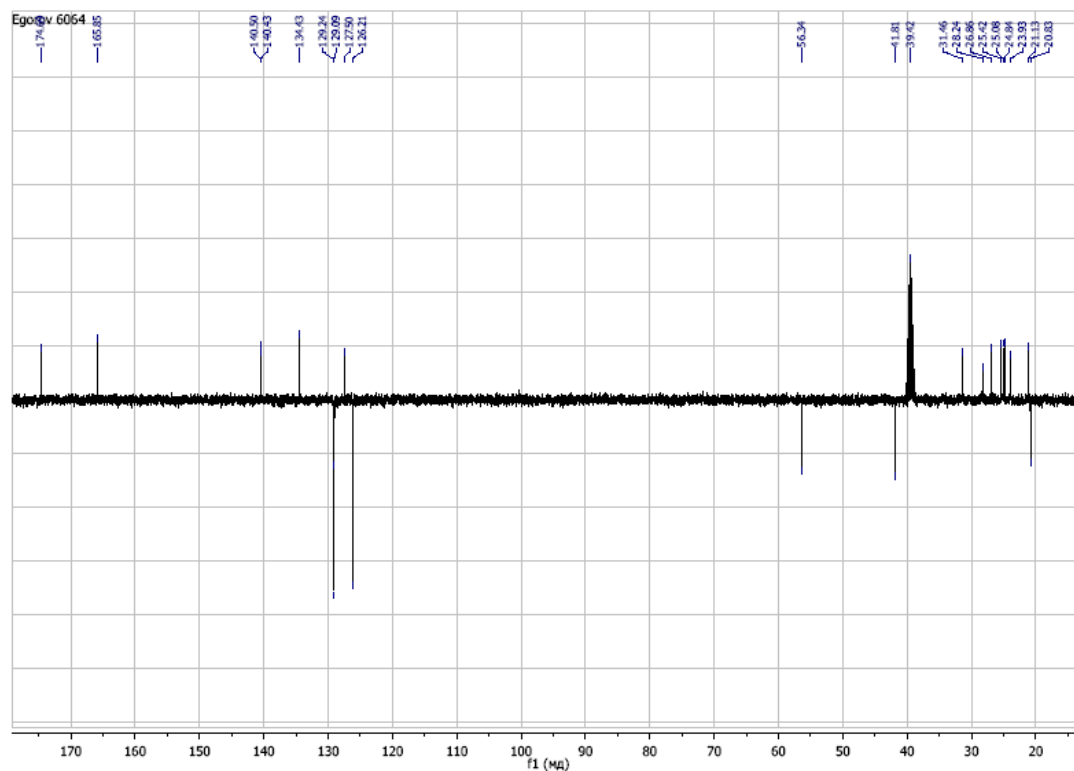
3-(Methylthio)-7,8,9,10,12,13,14,15,15a,15b-decahydro-1H-benzo[c][1,2,4]triazino[1,6-a]azecine-1,6(2H)-dione (10b). ^{13}C NMR (100MHz, DMSO- d_6)



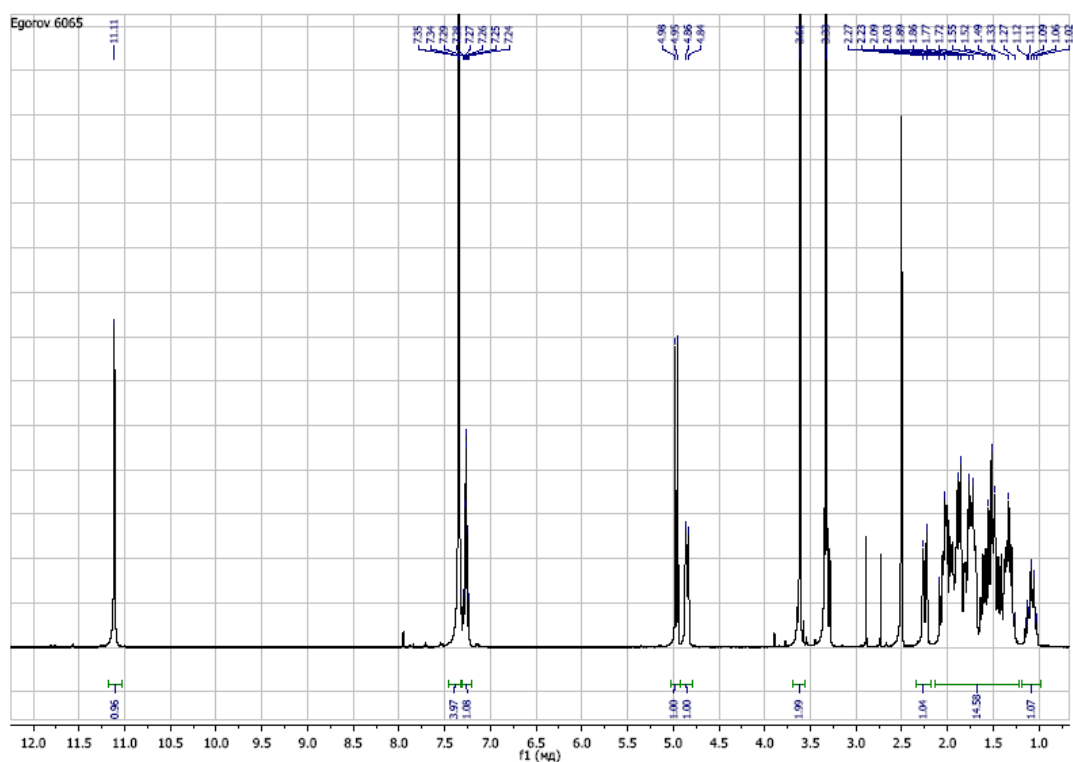
3-p-Tolyl-7,8,9,10,12,13,14,15,15a,15b-decahydro-1H-benzo[c][1,2,4]triazino[1,6-a]azecine-1,6(2H)-dione (10c). ^1H NMR (400 MHz, DMSO- d_6)



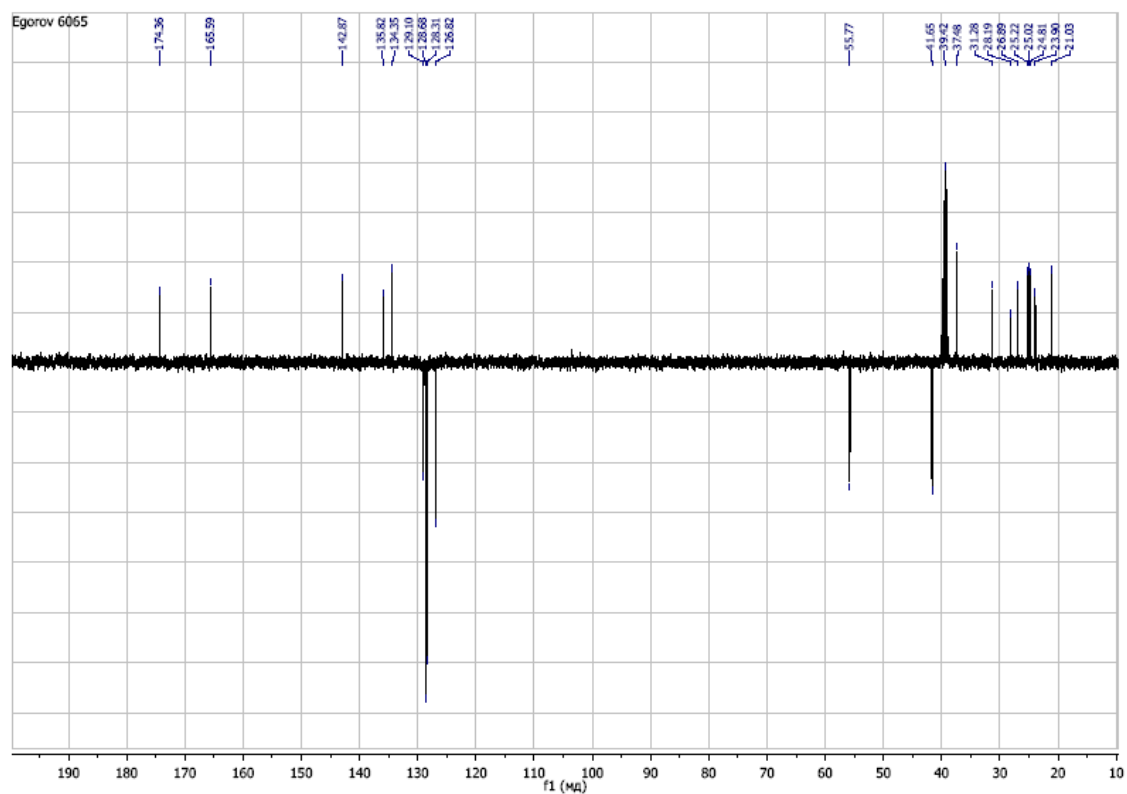
3-p-Tolyl-7,8,9,10,12,13,14,15,15a,15b-decahydro-1H-benzo[c][1,2,4]triazino[1,6-a]azecine-1,6(2H)-dione (10c). ^{13}C NMR (100MHz, DMSO- d_6)



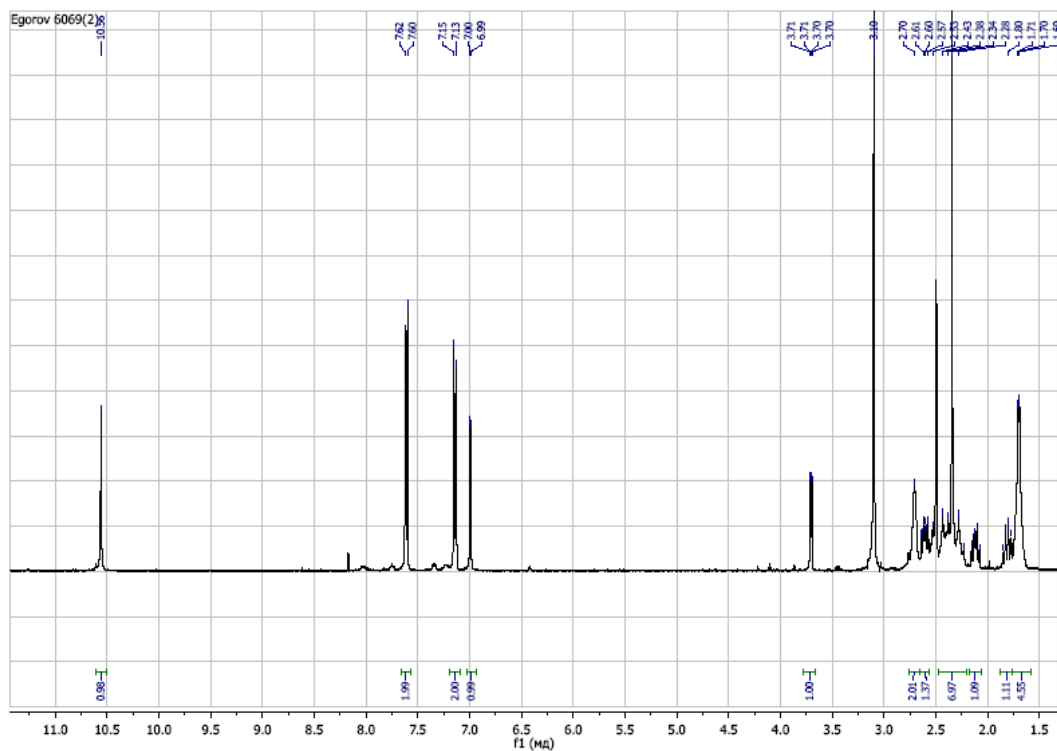
3-Benzyl-7,8,9,10,12,13,14,15,15a,15b-decahydro-1H-benzo[c][1,2,4]triazino[1,6-a]azecine-1,6(2H)-dione (10e). ^1H NMR (400 MHz, DMSO- d_6)



3-Benzyl-7,8,9,10,12,13,14,15,15a,15b-decahydro-1H-benzo[c][1,2,4]triazino[1,6-a]azecine-1,6(2H)-dione (10e). ^{13}C NMR (100MHz, DMSO- d_6)



3-(p-Tolyl)-7,8,9,11,12,13,13a,13b-octahydro-1H-cyclopenta[c][1,2,4]triazino[1,6-a]azonine-1,6(2H)-dione (12c). ^1H NMR (400 MHz, DMSO- d_6)



3-(p-Tolyl)-7,8,9,11,12,13,13a,13b-octahydro-1H-cyclopenta[c][1,2,4]triazino[1,6-a]azonine-1,6(2H)-dione (12c). ^{13}C NMR (100MHz, DMSO- d_6)

