Supplementary Information

Efficient Synthesis of Chloro-aminocyclooctanediol and Aminocyclooctanetriol: Unexpected Acetolysis Product

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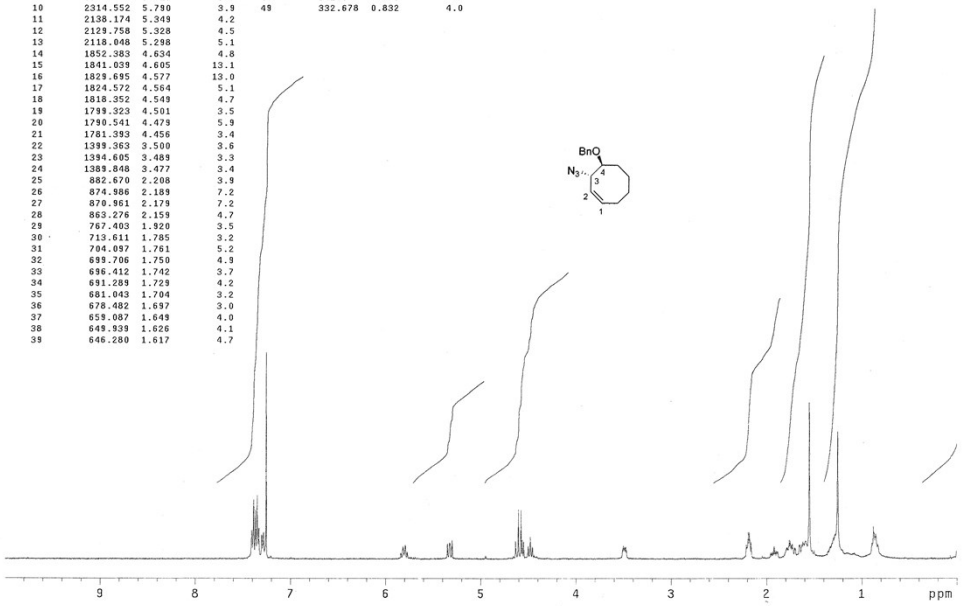
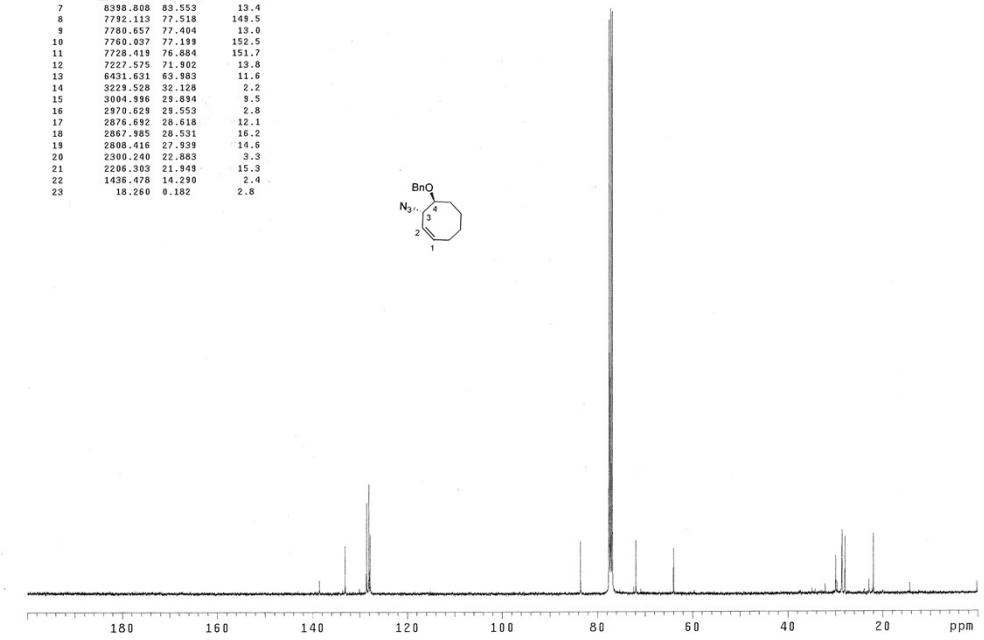
^{1}H NMR and ^{13}C NMR Spectra

(3*S*(*R*),4*S*(*R*),*Z*)-3-Azido-4-(benzyloxy)cyclooct-1-ene (6): $CDCl_3$

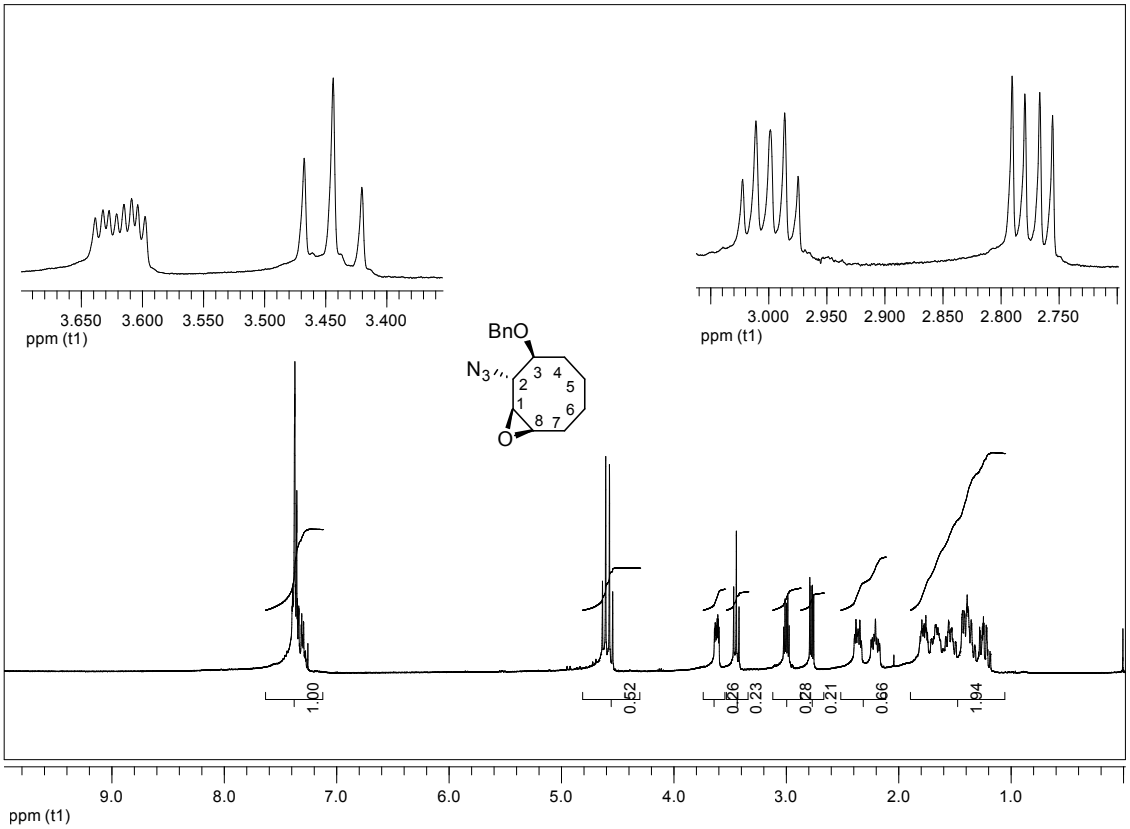
INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	2961.515	7.408	7.7	40	635.668	1.590	5.0
2	2954.196	7.390	15.7	41	620.299	1.552	40.9
3	2946.146	7.370	13.1	42	533.208	1.334	3.6
4	2938.827	7.351	16.9	43	529.182	1.324	3.7
5	2931.143	7.332	8.3	44	517.839	1.295	5.8
6	2919.433	7.303	6.6	45	501.006	1.253	33.3
7	2912.115	7.285	7.2	46	357.196	0.894	4.3
8	2901.503	7.258	53.9	47	350.975	0.878	8.7
9	2325.164	5.816	3.5	48	341.095	0.853	6.7
10	2314.552	5.790	3.9	49	332.678	0.832	4.0
11	2138.174	5.349	4.2				
12	2129.758	5.328	4.5				
13	2118.048	5.298	5.1				
14	1852.383	4.634	4.8				
15	1841.039	4.605	13.1				
16	1829.695	4.577	13.0				
17	1824.572	4.564	5.1				
18	1818.352	4.549	4.7				
19	1799.323	4.501	3.5				
20	1790.541	4.479	5.9				
21	1781.393	4.456	3.4				
22	1399.363	3.500	3.6				
23	1394.605	3.488	3.3				
24	1389.848	3.477	3.4				
25	882.670	2.208	3.9				
26	874.986	2.189	7.2				
27	870.961	2.179	7.2				
28	863.276	2.159	4.7				
29	767.403	1.920	3.5				
30	713.611	1.785	3.2				
31	704.097	1.761	5.2				
32	699.706	1.750	4.9				
33	696.412	1.742	3.7				
34	691.289	1.729	4.2				
35	681.043	1.704	3.2				
36	678.482	1.697	3.0				
37	659.087	1.649	4.0				
38	649.939	1.626	4.1				
39	646.280	1.617	4.7				

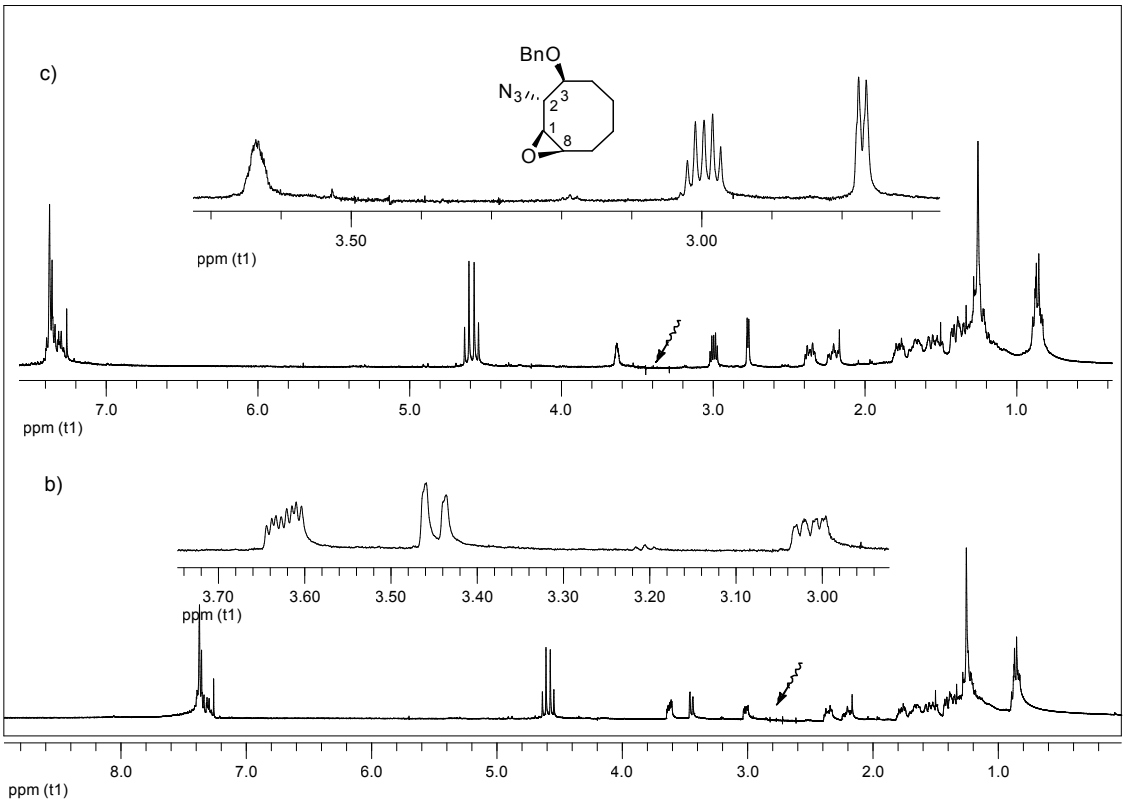


INDEX	FREQUENCY	PPM	HEIGHT
1	13927.340	138.553	3.3
2	13380.214	133.110	12.3
3	12923.360	128.565	23.6
4	12875.704	128.091	28.6
5	12864.707	127.981	4.4
6	12848.669	127.822	15.3
7	8398.808	83.553	13.4
8	7792.113	77.518	149.5
9	7780.657	77.404	13.0
10	7760.037	77.199	152.5
11	7728.419	76.884	151.7
12	7227.575	71.902	13.8
13	6431.631	63.983	11.6
14	3229.528	32.128	2.2
15	3004.996	29.894	9.5
16	2970.629	29.553	2.8
17	2876.692	28.618	12.1
18	2867.985	28.531	16.2
19	2808.416	27.939	14.6
20	2300.240	22.883	3.3
21	2206.303	21.949	15.3
22	1436.478	14.290	2.4
23	18.260	0.182	2.8

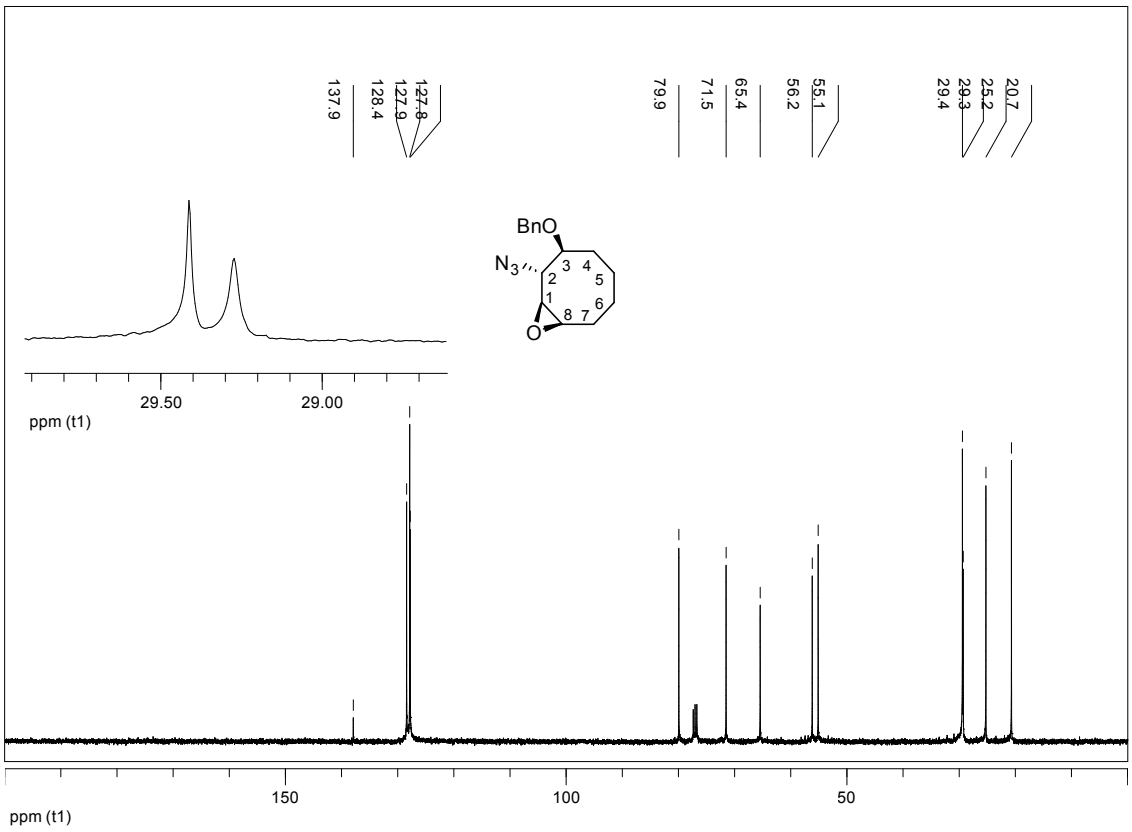


(1*S*(*R*),2*R*(*S*),3*S*(*R*),8*R*(*S*))-2-Azido-3-(benzyloxy)-9-oxabicyclo[6.1.0]nonane (7): $CDCl_3$ (^{1}H NMR and Double Resonance)

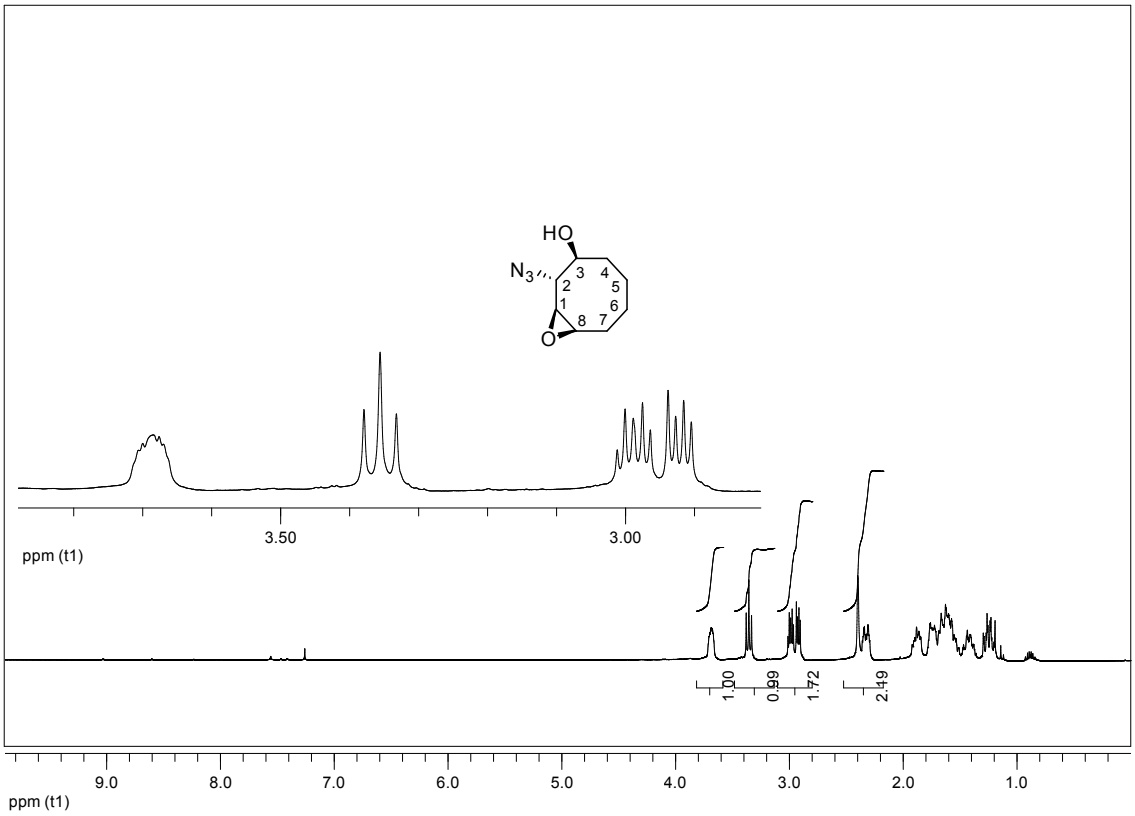


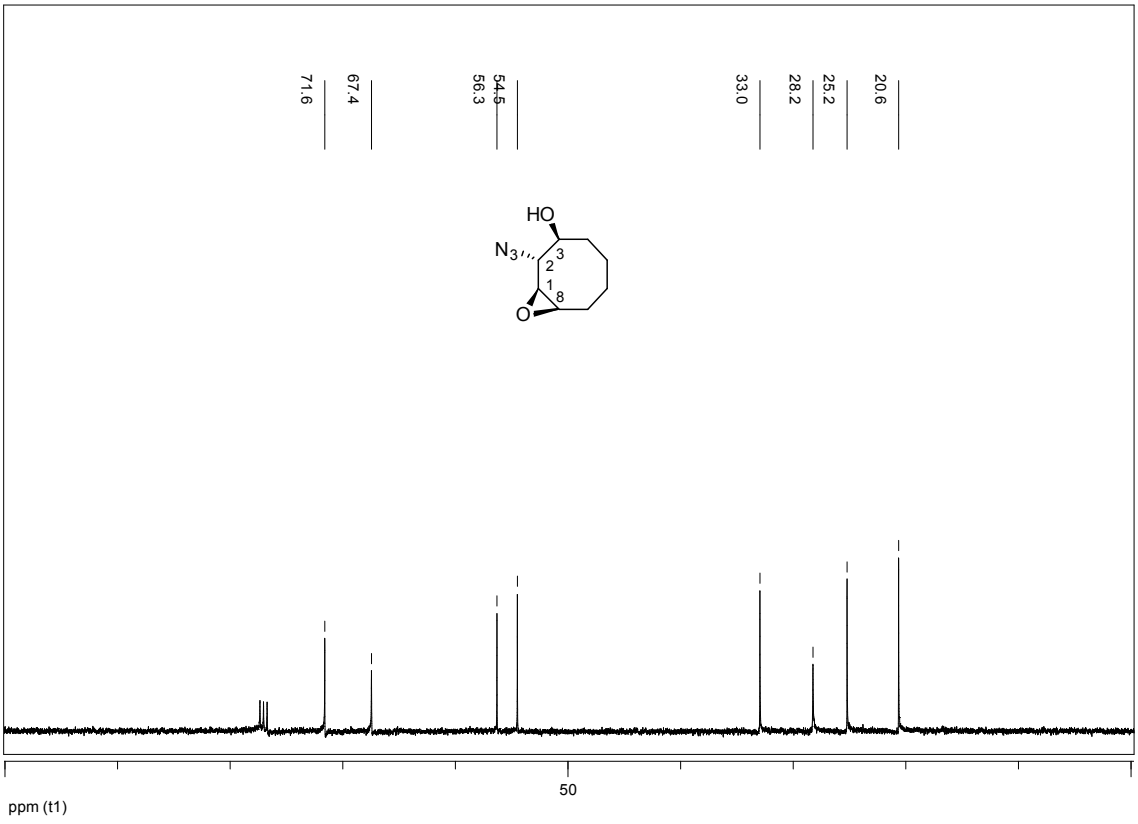


^{13}C NMR of compound 7

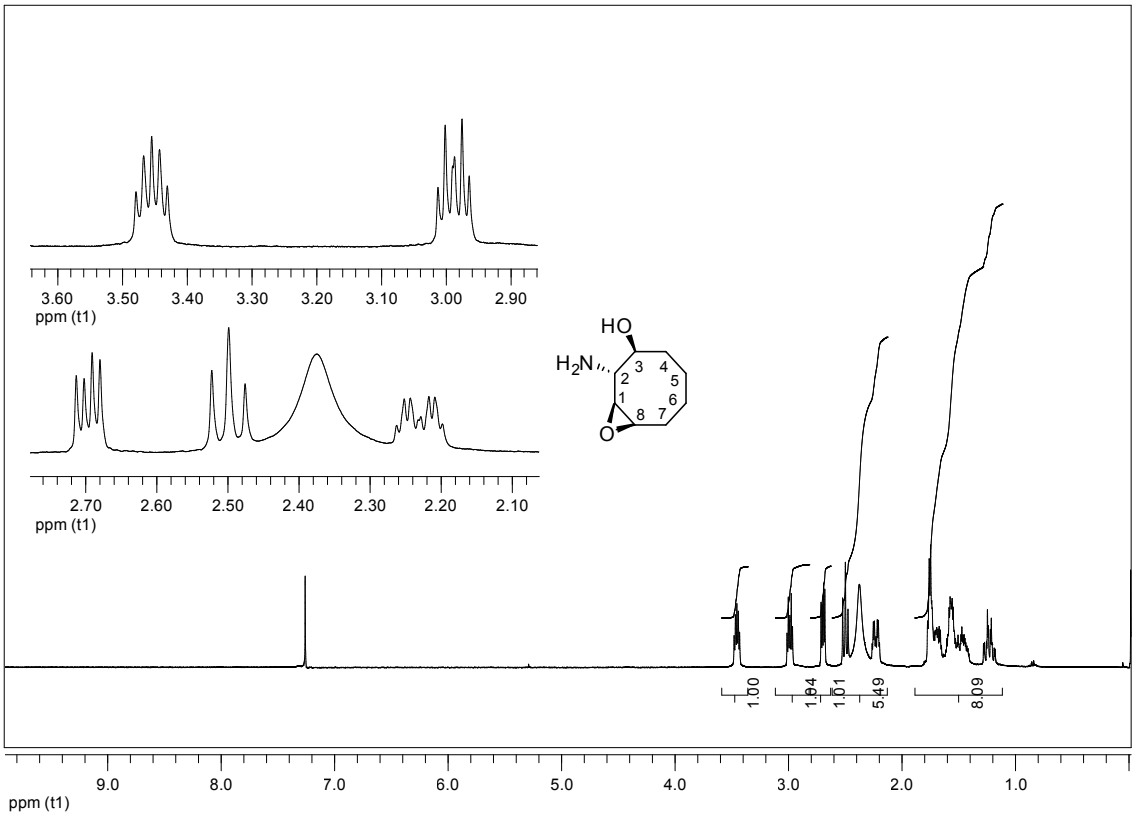


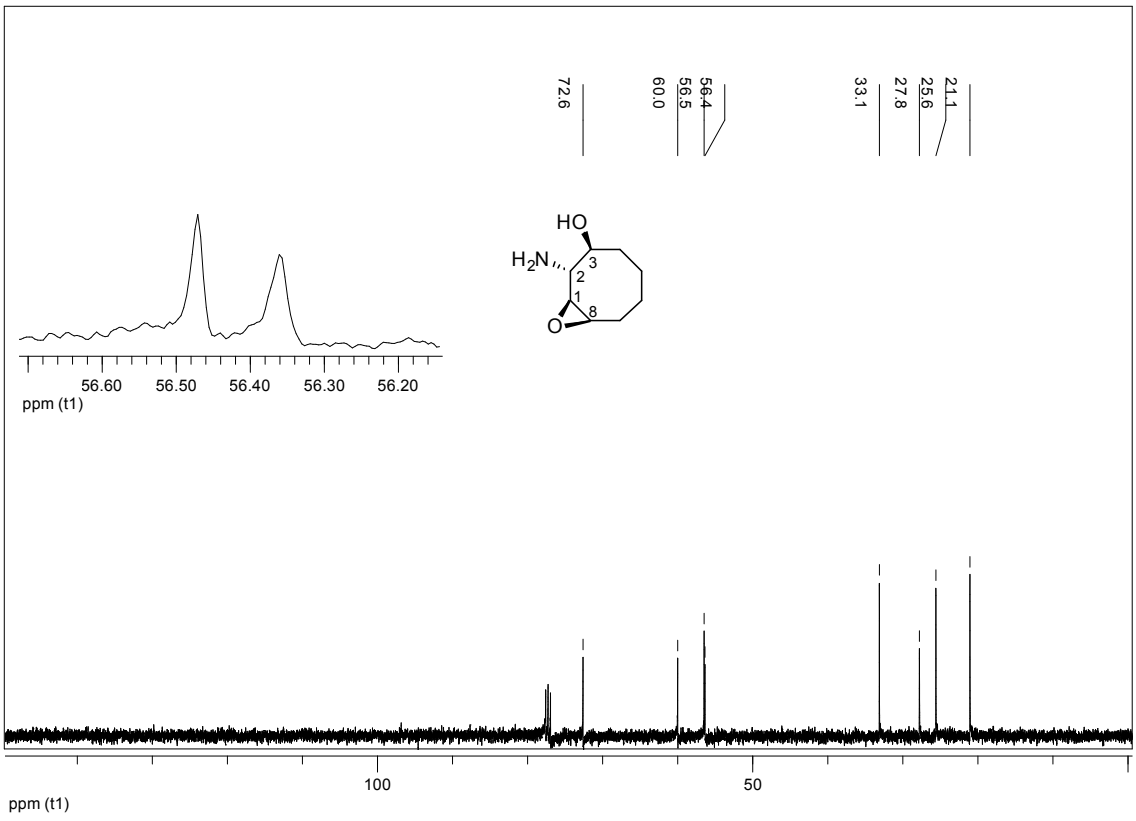
(1*S*(*R*),2*R*(*S*),3*S*(*R*),8*R*(*S*))-2-Azido-9-oxabicyclo[6.1.0]nonan-3-ol (8): $CDCl_3$



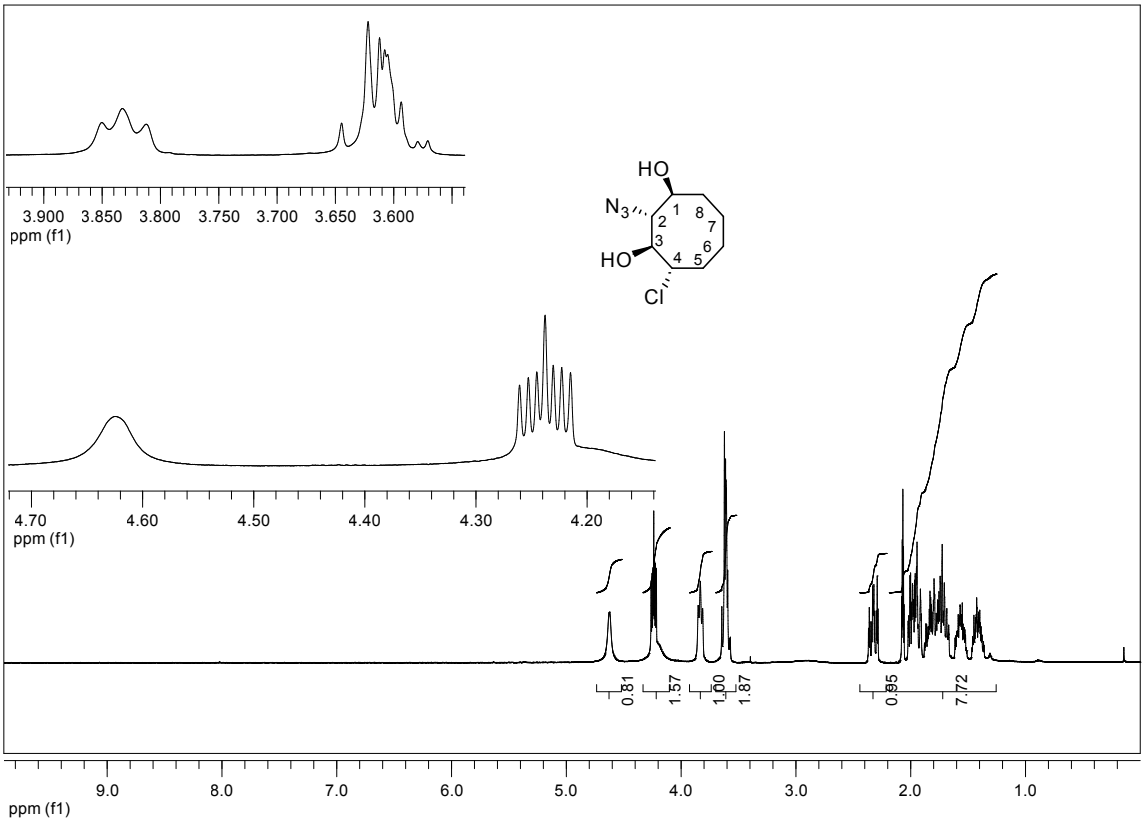


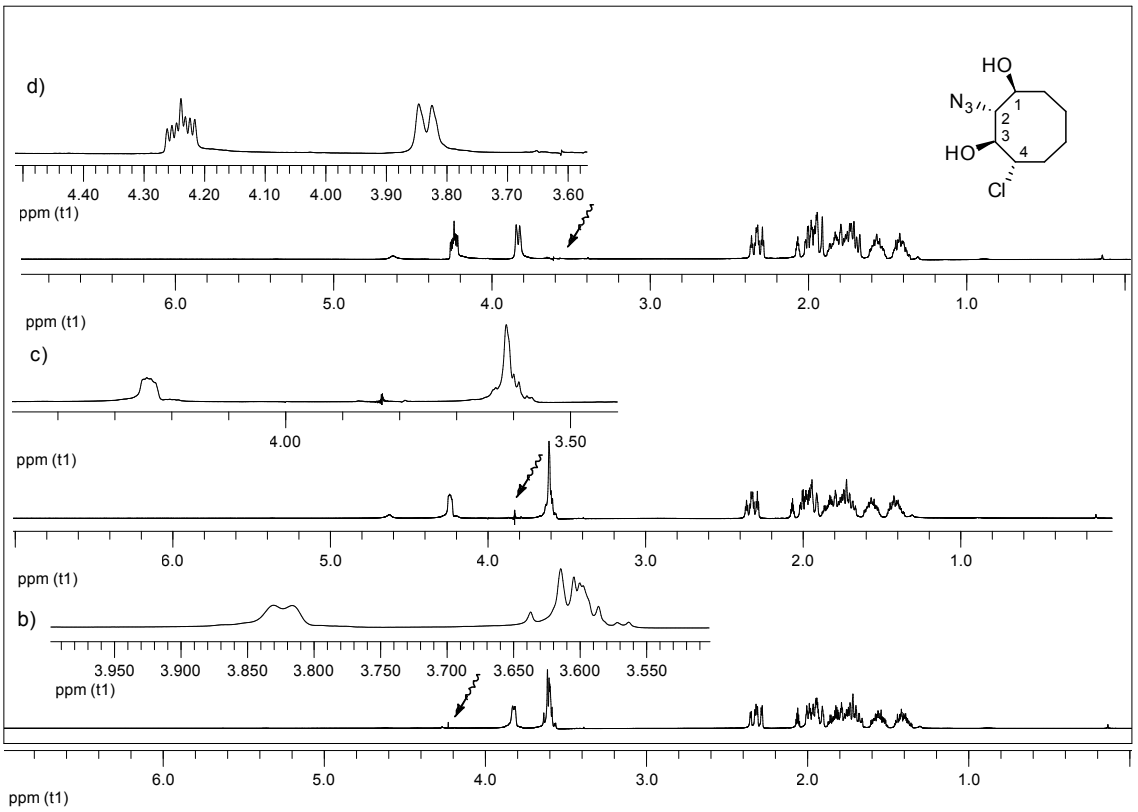
(1*S*(*R*),2*R*(*S*),3*S*(*R*),8*R*(*S*))-2-Amino-9-oxabicyclo[6.1.0]nonan-3-ol (9): $CDCl_3$



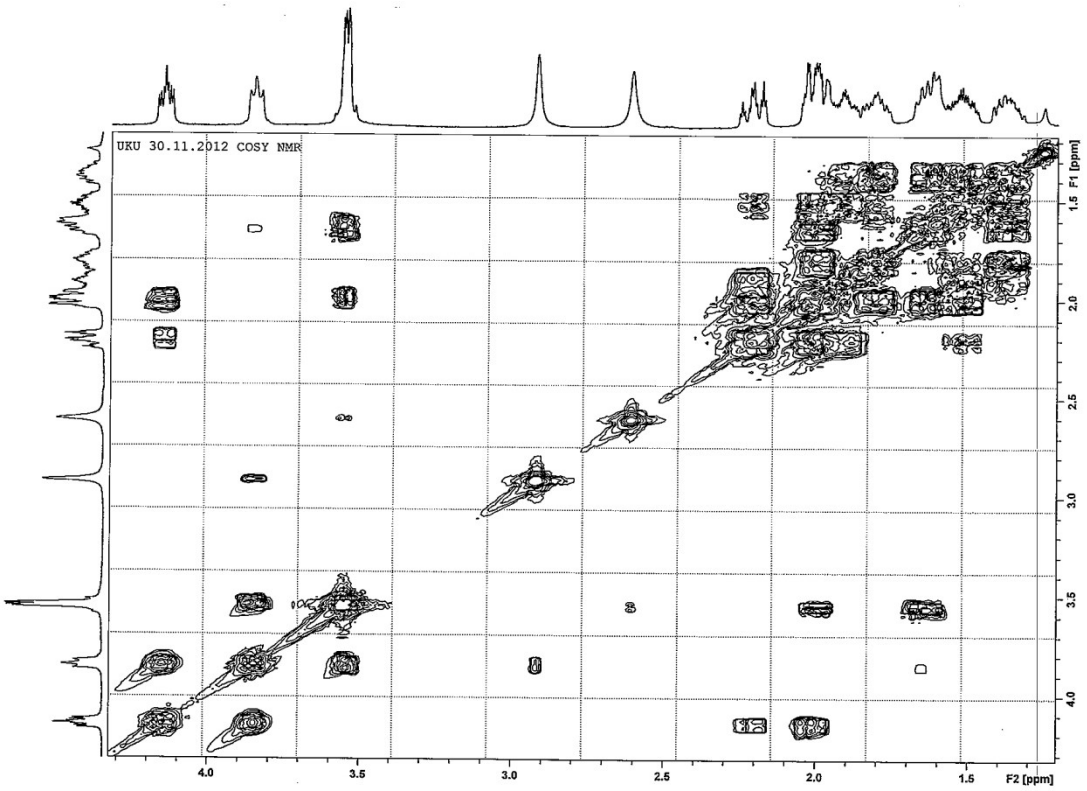


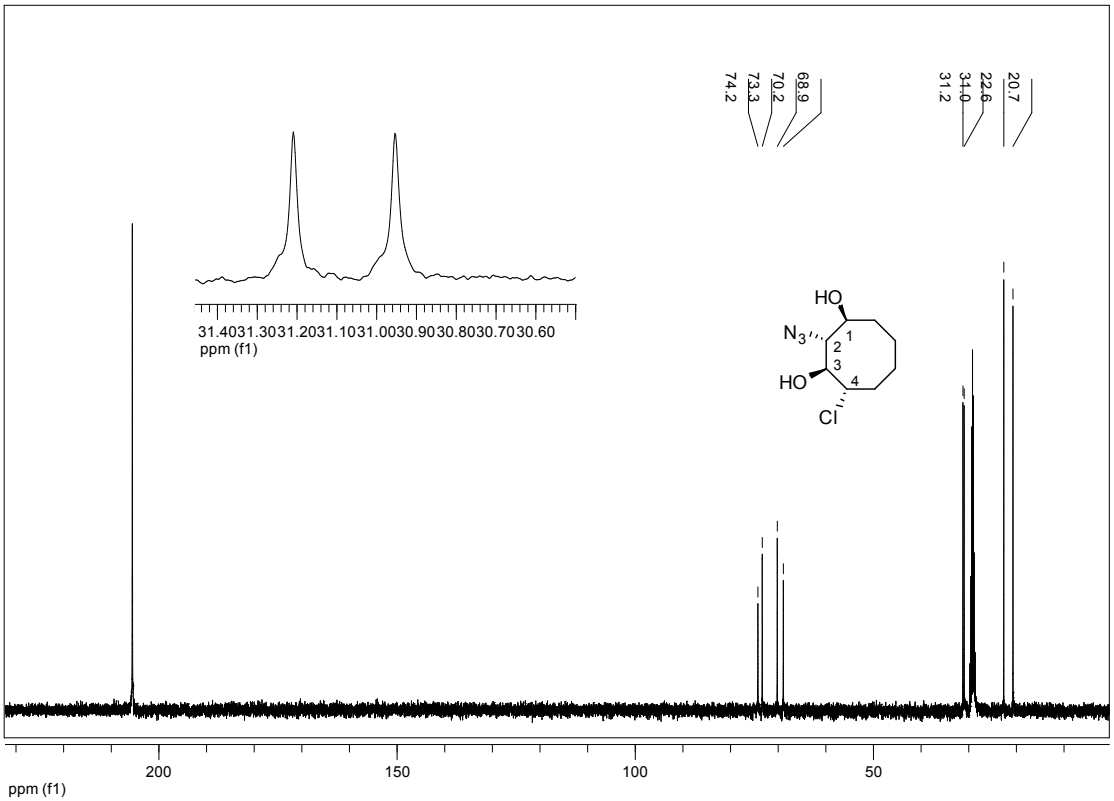
(1*S*(*R*),2*R*(*S*),3*S*(*R*),4*S*(*R*))-2-Azido-4-chlorocyclooctane-1,3-diol (10): acetone-d_6 (^{1}H NMR and Double Resonance)



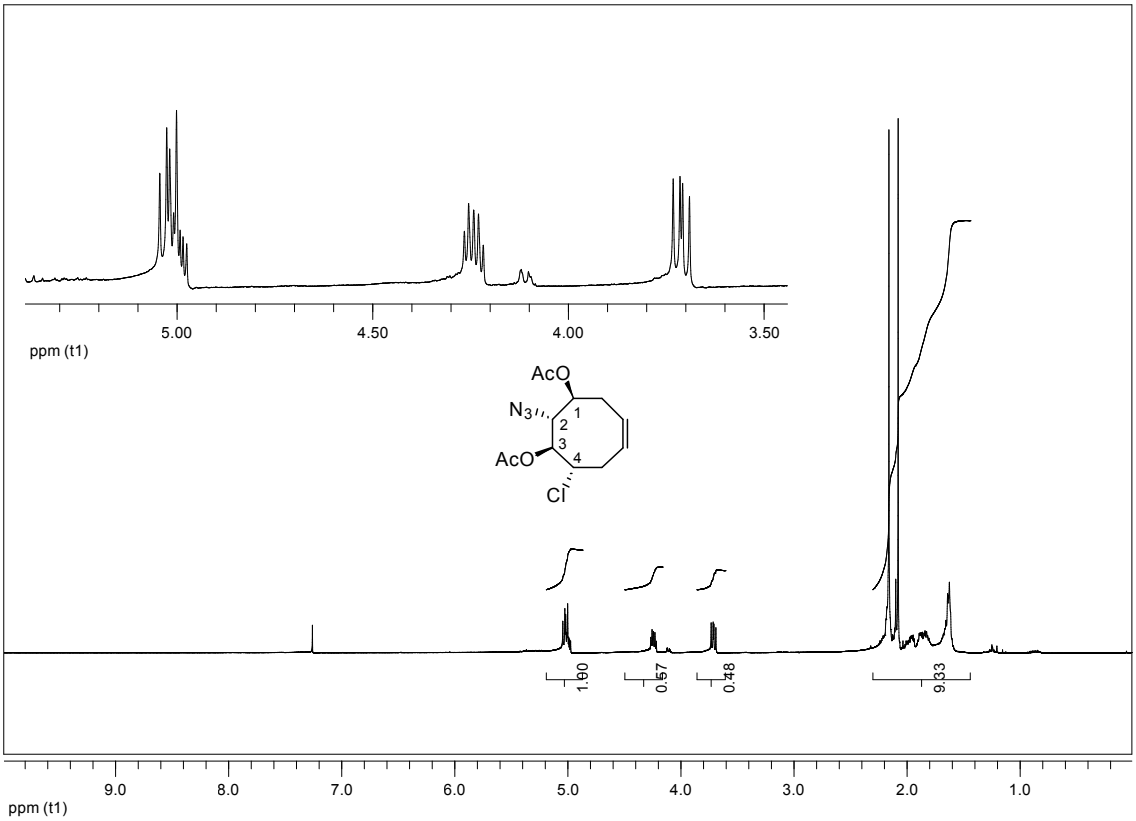


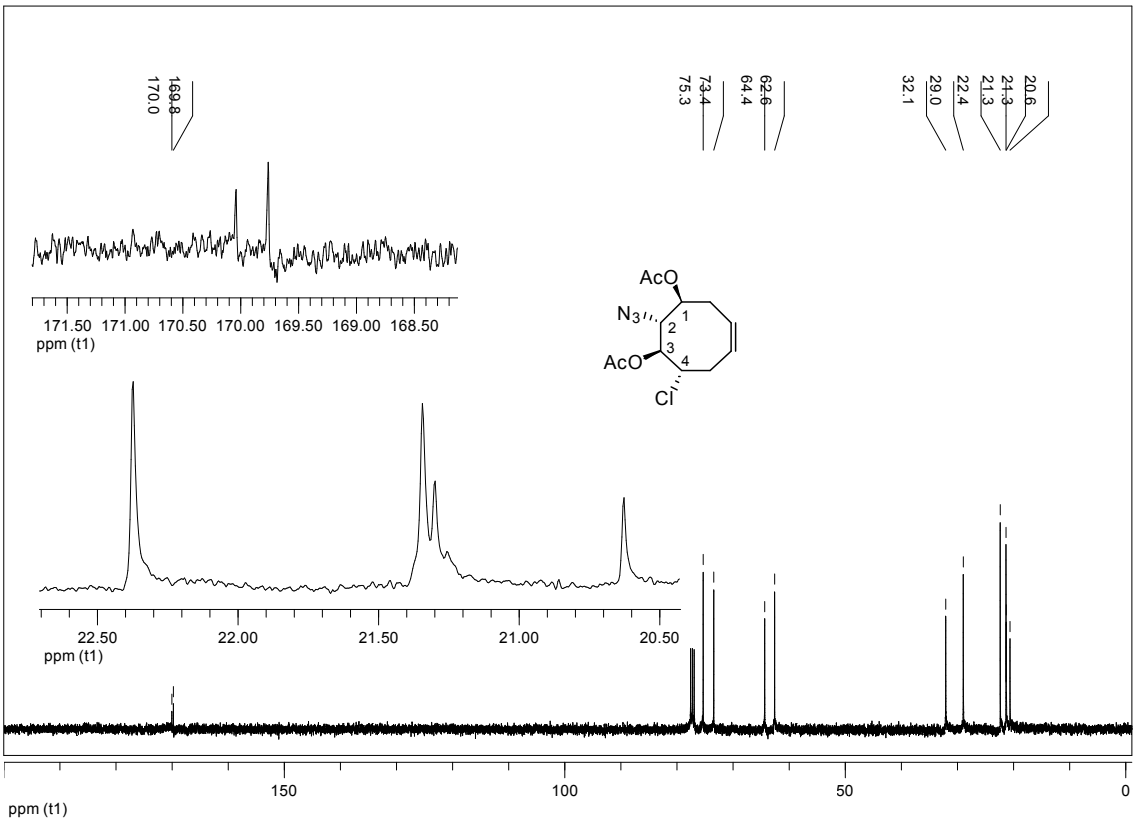
COSY and ^{13}C NMR of compound 10



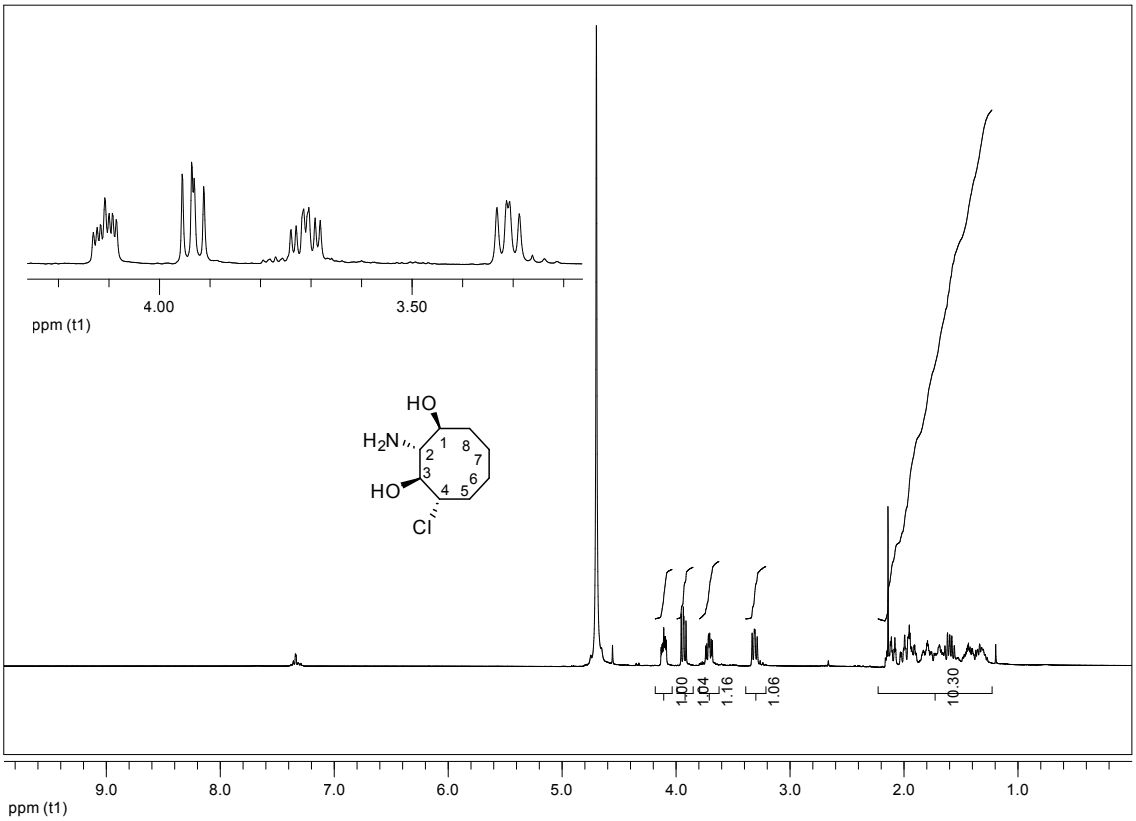


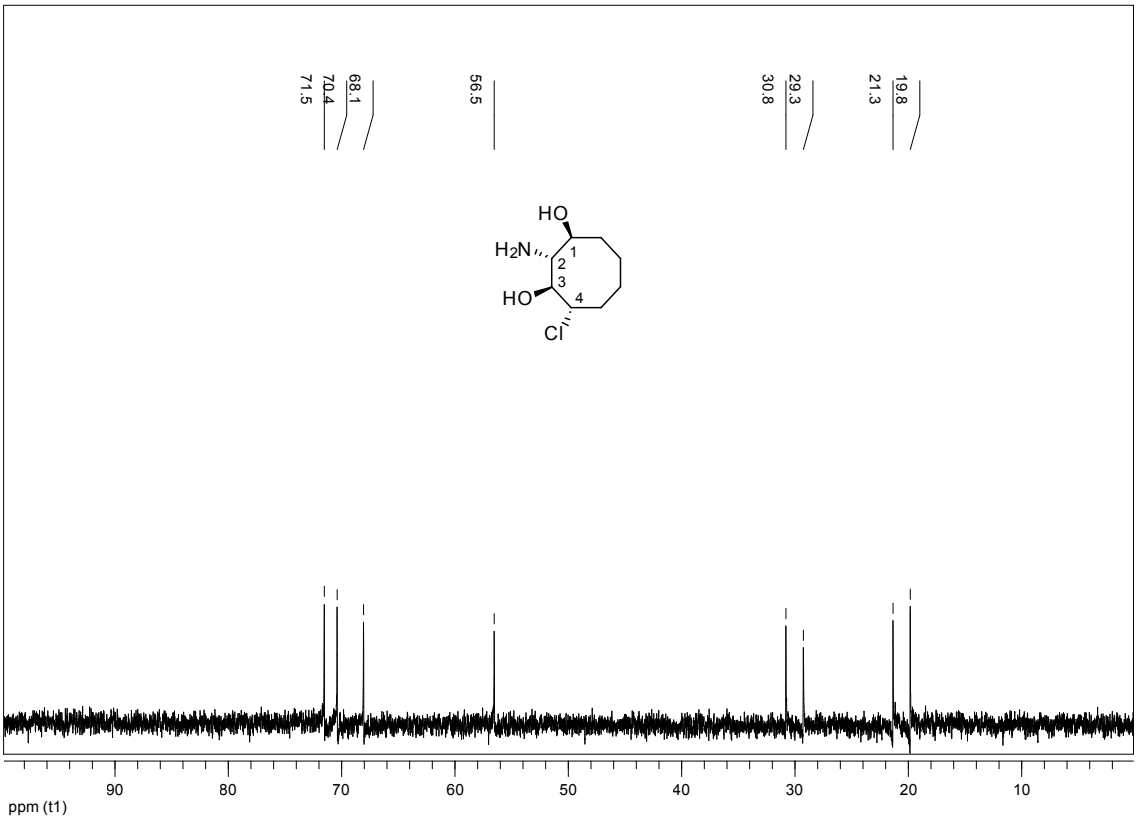
(1*S*(*R*),2*R*(*S*),3*S*(*R*),4*S*(*R*))-2-Azido-4-chlorocyclooctane-1,3-diyl diacetate (11): $CDCl_3$



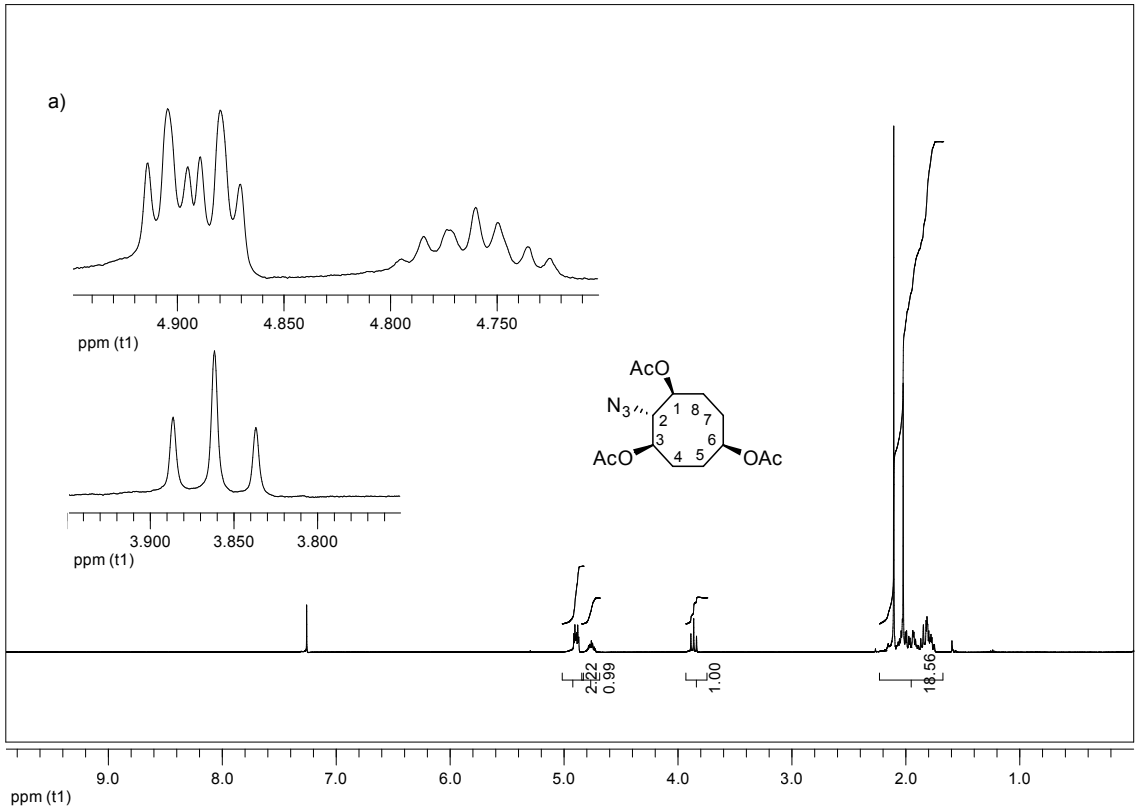


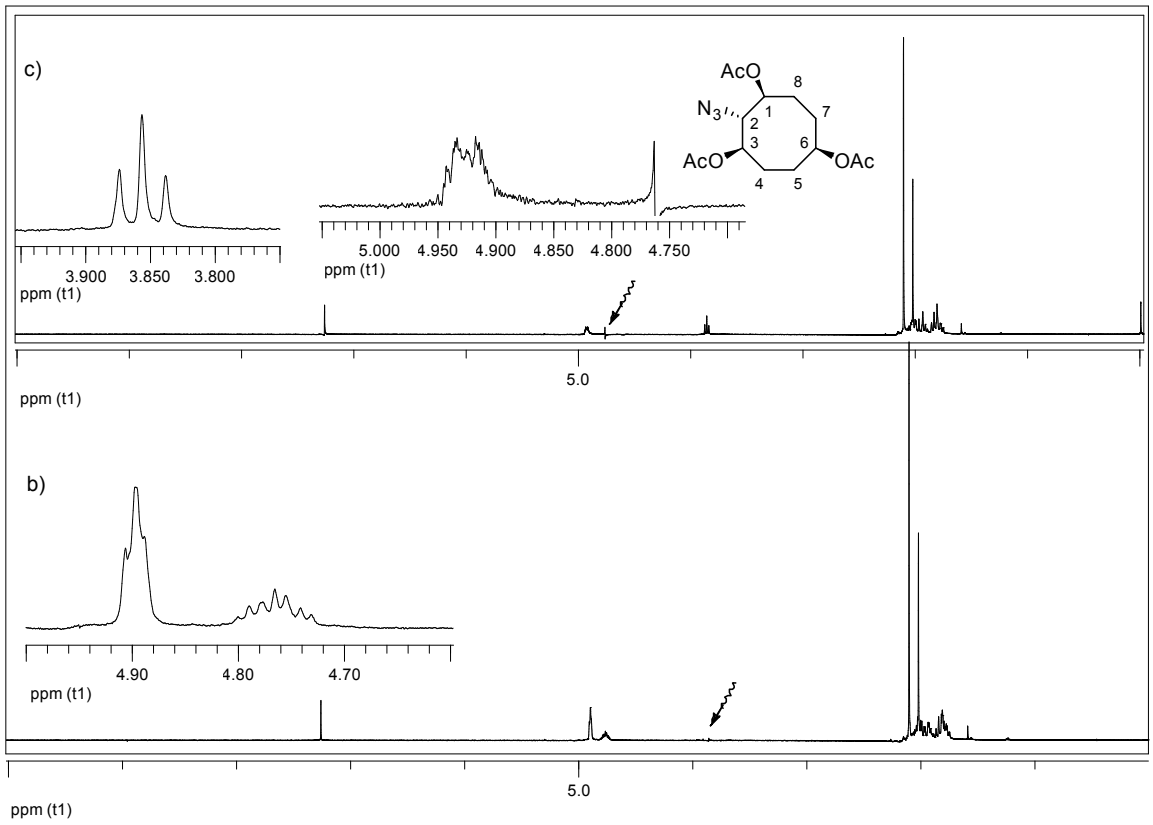
(1*S*(*R*),2*R*(*S*),3*S*(*R*),4*S*(*R*))-2-Amino-4-chlorocyclooctane-1,3-diol (12): D_2O



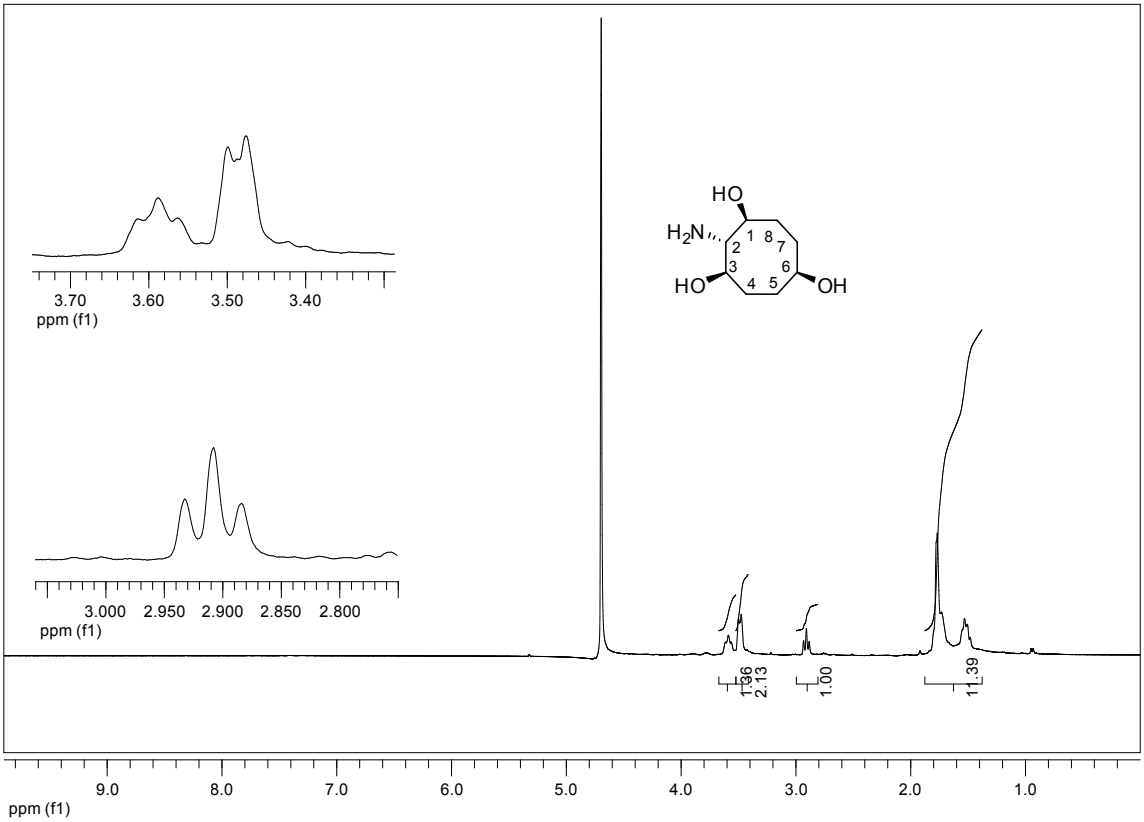


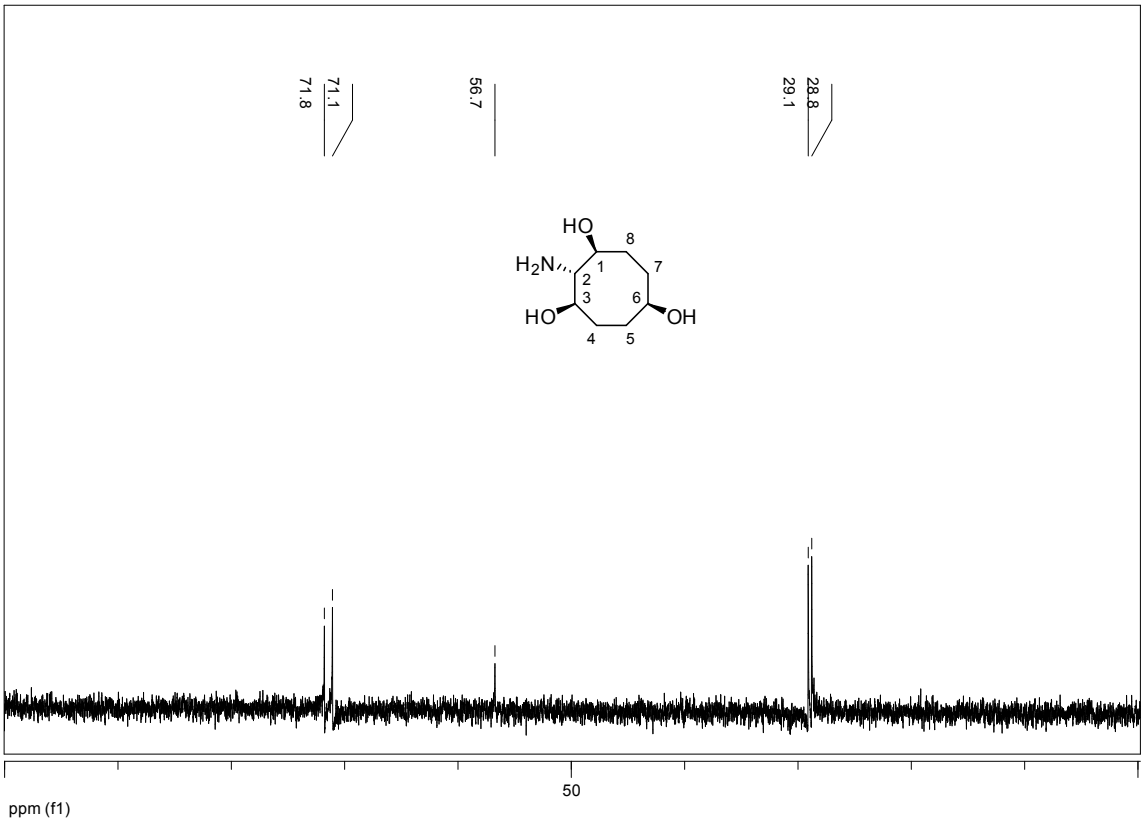
(1*R*(*S*),2*R*(*S*),3*S*(*R*),6*R*(*S*))-2-Azidocyclooctane-1,3,6-triyl triacetate (16): $CDCl_3$ (^{1}H NMR and Double Resonance)



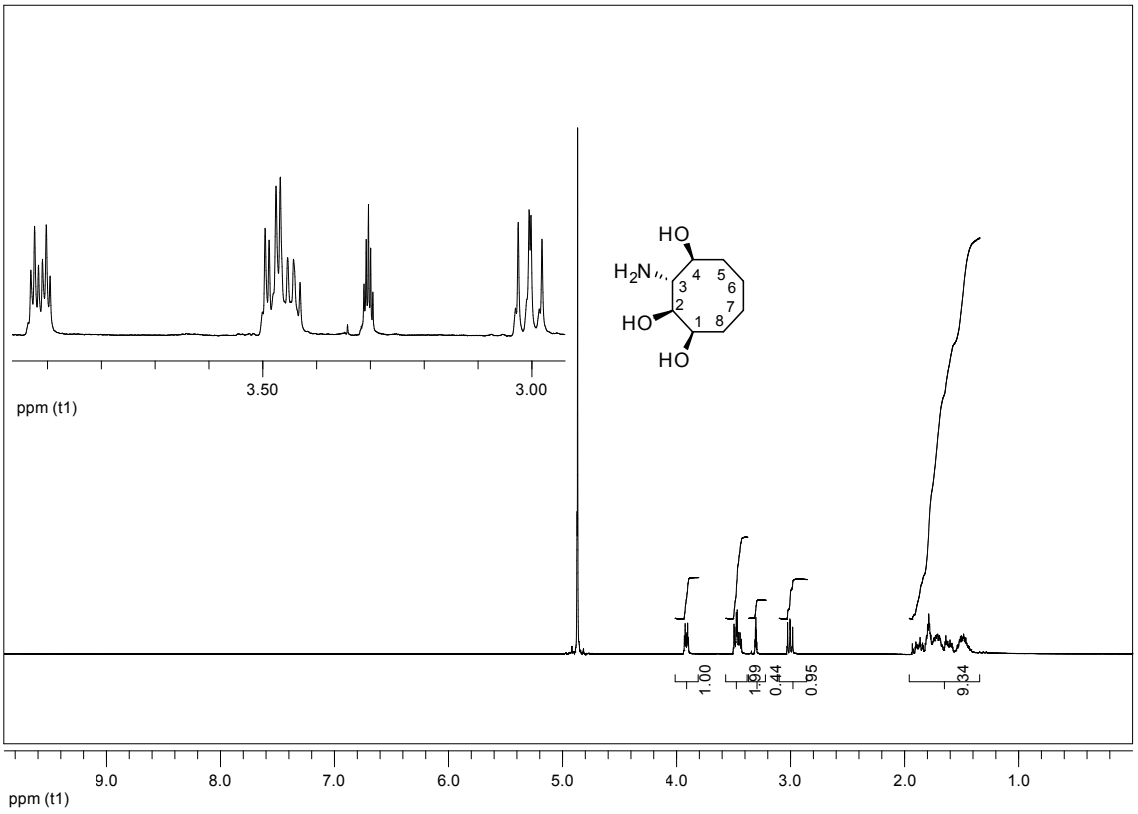


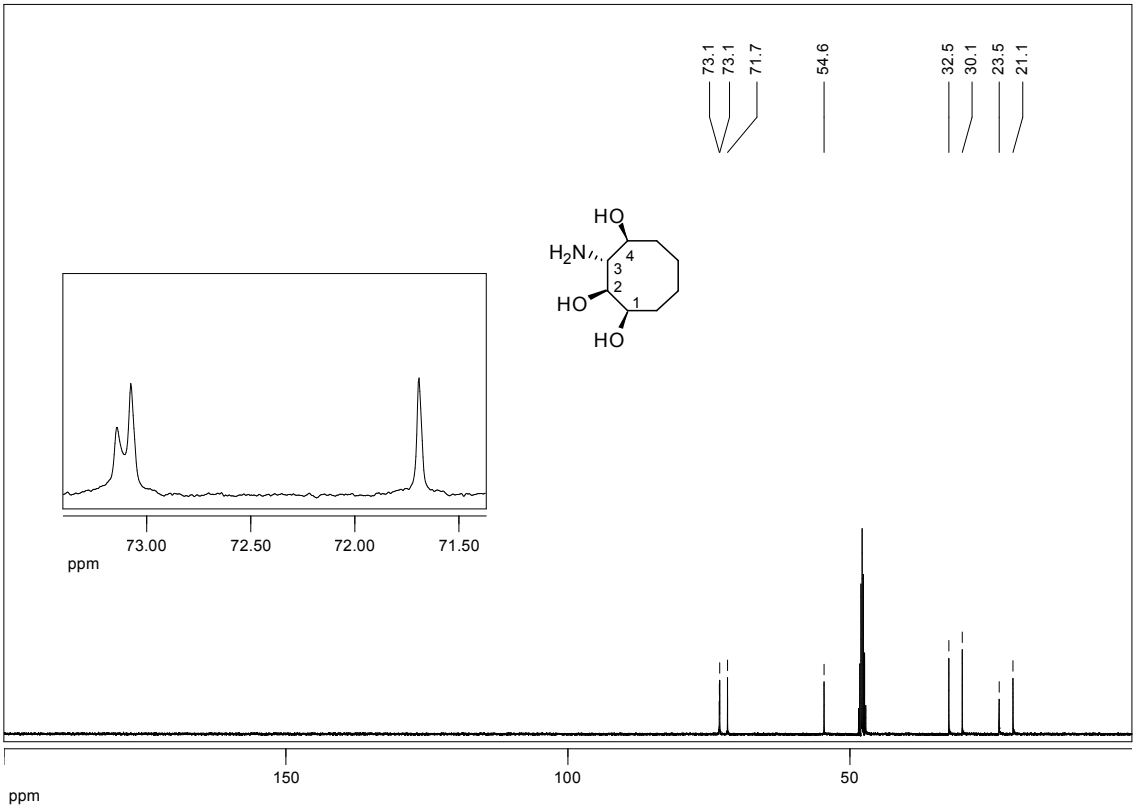
(1*R*(*S*),2*R*(*S*),3*S*(*R*),6*R*(*S*))-2-Aminocyclooctane-1,3,6-triol (18): D_2O



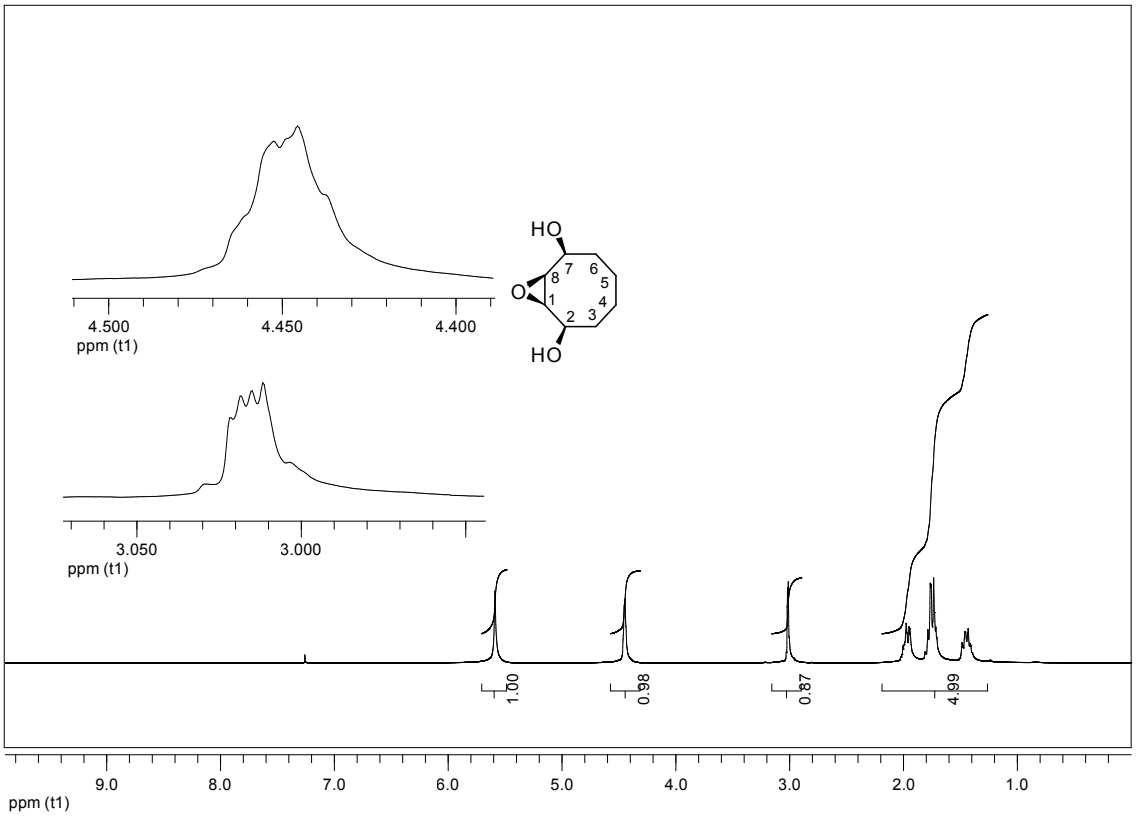


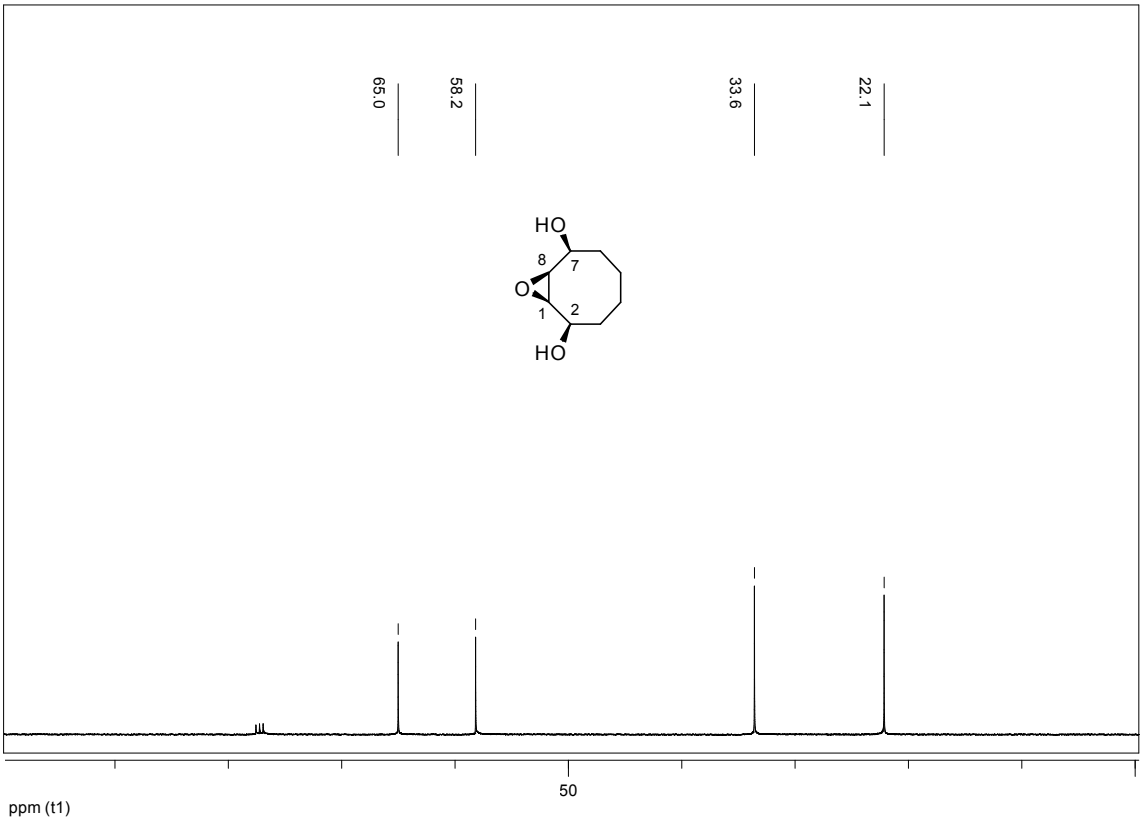
(1*R*(*S*),2*S*(*R*),3*R*(*S*),4*S*(*R*))-3-Aminocyclooctane-1,2,4-triol (17): CD_3OD



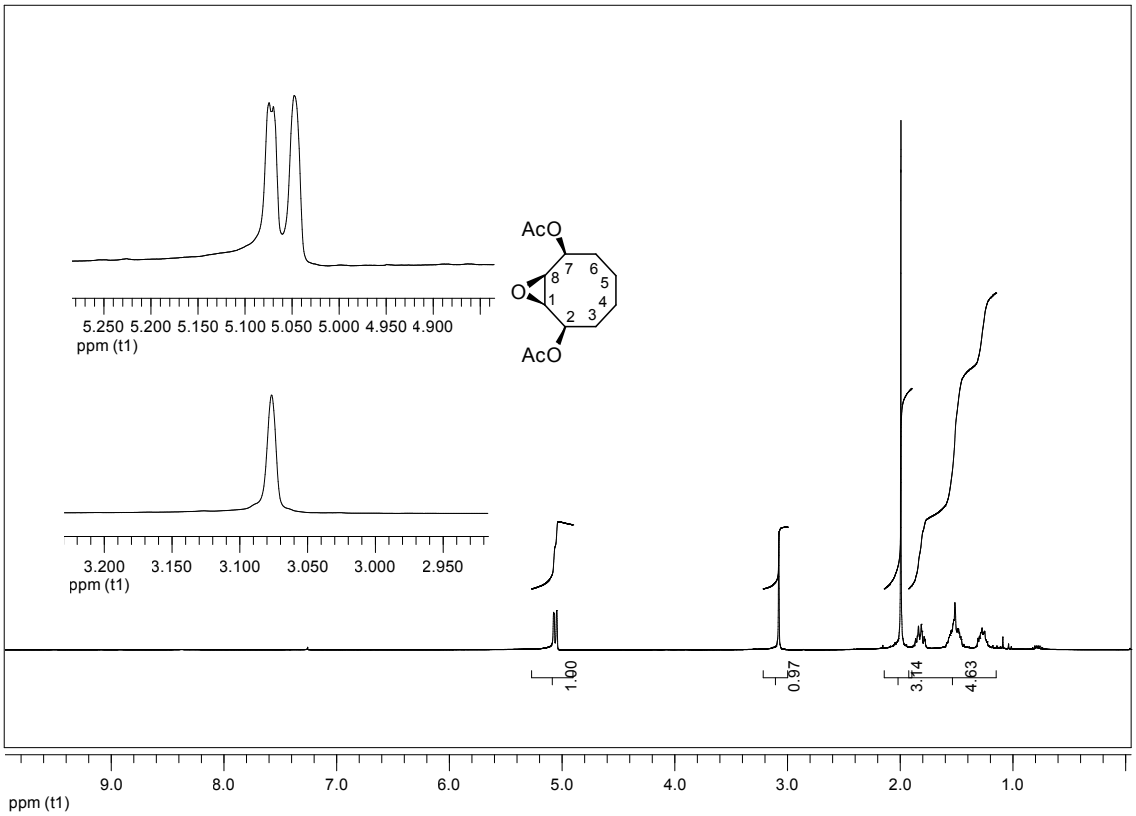


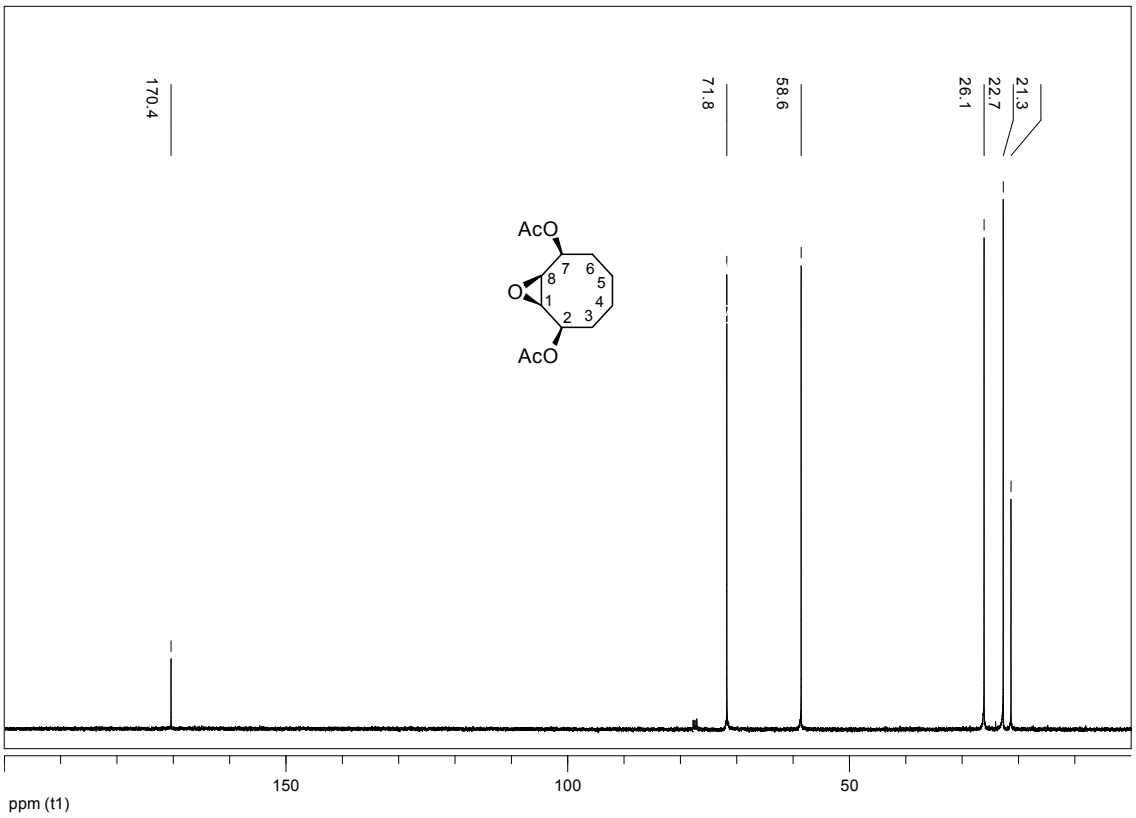
(1*R*(*S*),2*R*(*S*),7*S*(*R*),8*S*(*R*))-9-Oxabicyclo[6.1.0]nonane-2,7-diol (32): $CDCl_3$





(1*R*(*S*),2*R*(*S*),7*S*(*R*),8*S*(*R*))-9-Oxabicyclo[6.1.0]nonane-2,7-diyl diacetate (33): $CDCl_3$





X-ray Crystallographic Data

For the crystal structure determination, single-crystal of the compounds **10** and **16** was used for data collection on a four-circle Rigaku R-AXIS RAPID-S diffractometer (equipped with a two-dimensional area IP detector). Graphite-monochromated Mo-K_α radiation (λ = 0.71073 Å) and oscillation scans technique with Δw = 5º for one image were used for data collection. The lattice parameters were determined by the least-squares methods based on all reflections with $F^2 > 2\sigma(F^2)$. Integration of the intensities, correction for Lorentz and polarization effects and cell refinement were performed using CrystalClear (Rigaku/MSC Inc., 2005) software.* The structure was solved by direct methods using SHELXS-97[2] and non-hydrogen atoms were refined using anisotropic displacement parameters by full-matrix least-squares procedure using the program SHELXL-97.[2] Hydrogen atoms were positioned geometrically and refined using a riding model. The final difference Fourier maps showed no peaks of chemical significance. Details about the analysed crystal and data collection are presented in Tables S1 and S2, respectively.

X-ray crystallographic data of compound 10

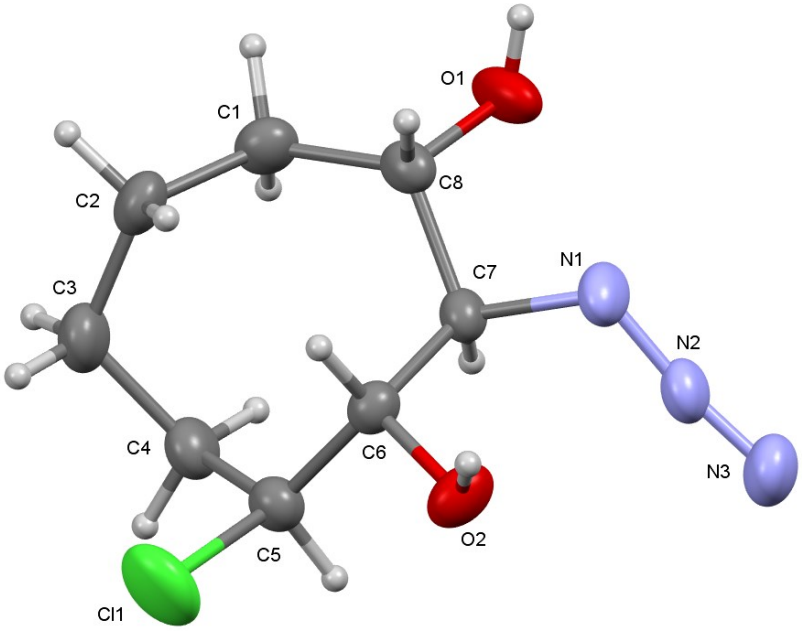


Figure S1 X-ray crystal structure of **10**

Table S1 Crystal data and structure refinement for compound **10**

* Rigaku/MSC, Inc., 9009 new Trails Drive, The Woodlands, TX 77381-5209, USA, 2005.

[2] G.M. Sheldrick, SHELXS-97, SHELXL-97 Program for Crystal Structure Solution and refinement, University of Gottingen, Göttingen, Germany, 1997.

Empirical formula	$C_8H_{14}ClN_3O_2$
Formula weight	219.67
Temperature	296 K
Wavelength	0.71073 A
Crystal system, space group	orthorhombic, *P*bca; (no: 61)
Unit cell dimensions	a = 20.7755(3), b = 8.7032(1), c = 22.9649(4) Å, α= 90, β = 90, γ=90º
Volume	4152.36(11) Å^3
Z, calculated density	16, 1.406 g/cm^3
absorption coefficient	0.348 mm^{-1}
F(000)	1856
θ-range for data collection	1.7-26.4º
refinement method	full matrix least-square on F^2
data/parameters	3607/258
goodness-of-fit on F^2	1.051
final R-indices [$I > 2\sigma(I)$]	R_1 = 0.043, wR_2 = 0.114
largest diff. peak and hole	0.471 and -0.367 e Å^{-3}

X-ray crystallographic data of compound 16

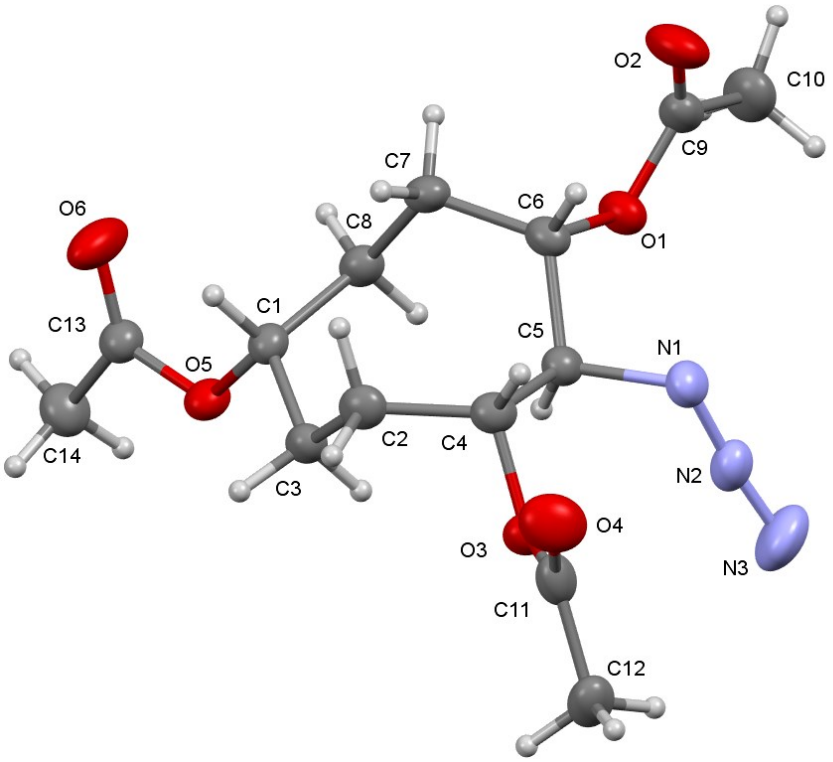


Figure S2 X-ray crystal structure of **16**

Table S2 Crystal data and structure refinement for compound **16**

Empirical formula	$C_{14}H_{21}N_3O_6$
Formula weight	327.34
Temperature	296 K
Wavelength	0.71073 A
Crystal system, space group	tetragonal, $P4_12_12$; (no: 92)
Unit cell dimensions	a = 12.7747(4), b = 12.7747(4), c = 20.7228(9) Å, α= 90, β = 97.176(3), γ=90º
Volume	3381.8(3) Å^3
Z, calculated density	8, 1.286 g/cm^3
absorption coefficient	0.101 mm^{-1}
F(000)	1392
θ-range for data collection	2.2-26.4º
refinement method	full matrix least-square on F^2
data/parameters	2870/212
goodness-of-fit on F^2	1.067
final R-indices [$I > 2\sigma(I)$]	R_1 = 0.044, wR_2 = 0.107
largest diff. peak and hole	0.189 and -0.146 e Å^{-3}