

Supplementary data for article:

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Electronic Supplementary Information for the paper:

A short stereoselective synthesis of (+)-aza-galacto-fagomine (AGF)

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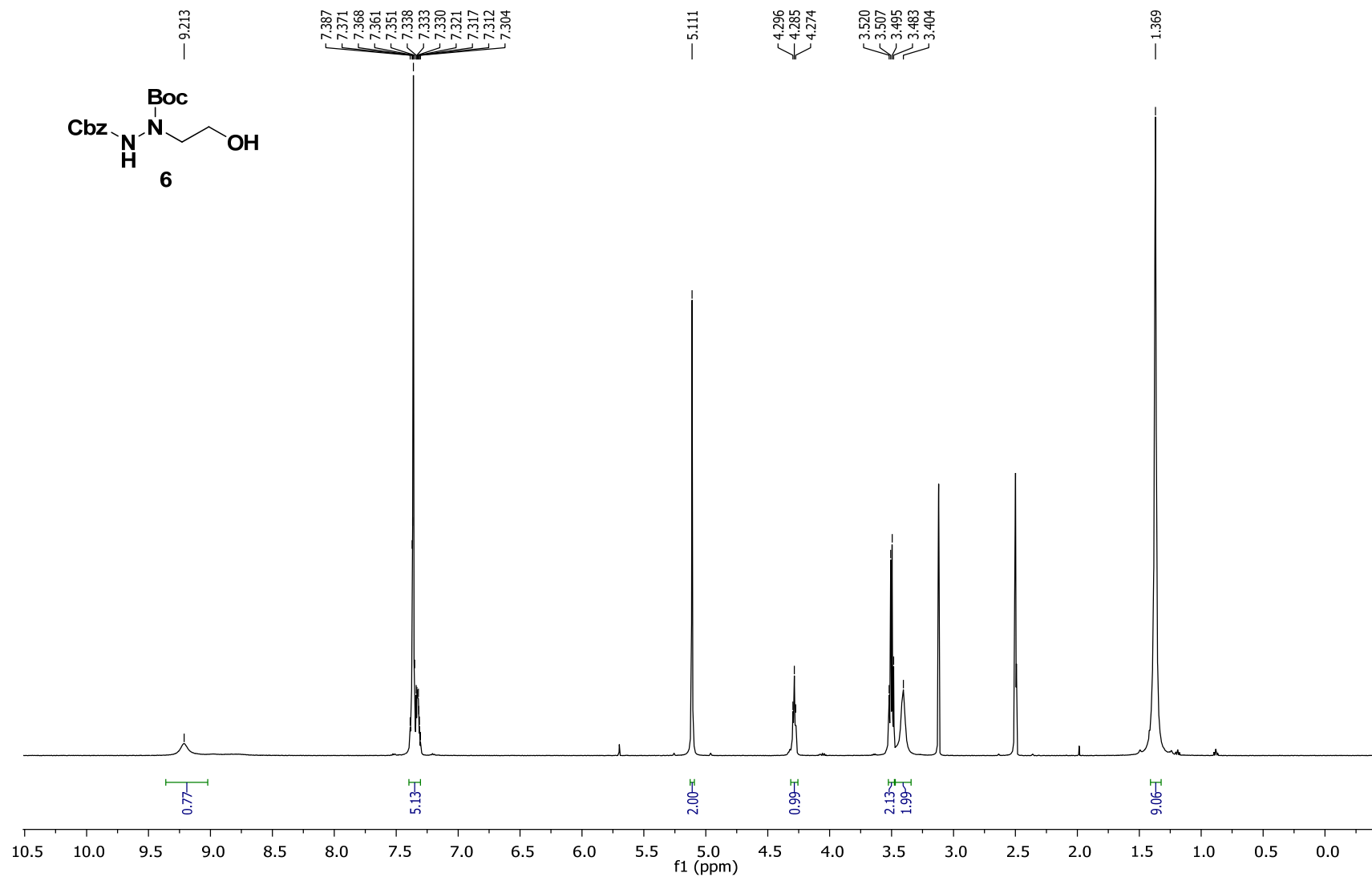
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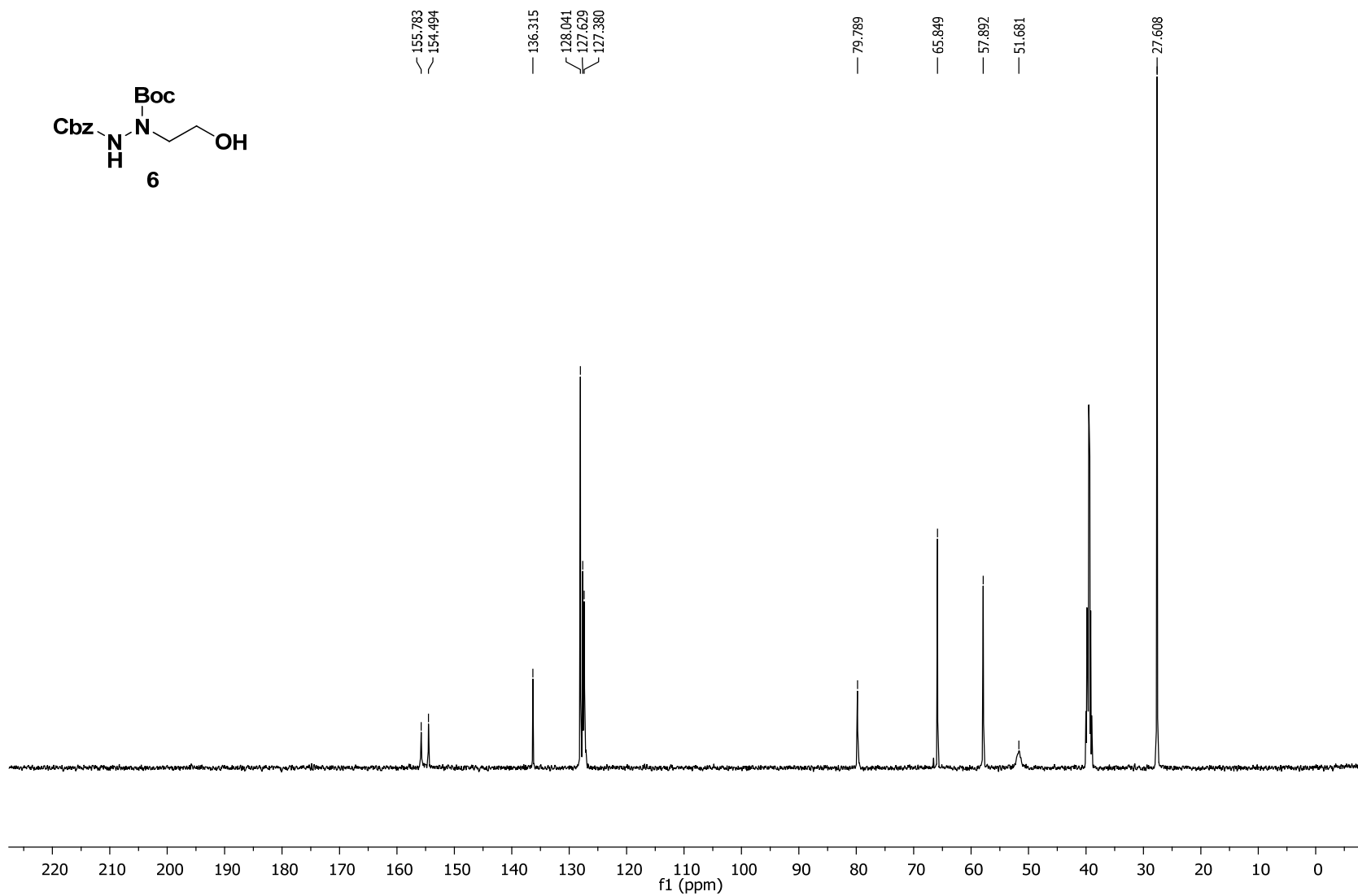
Content:

¹H and ¹³C NMR Spectra for compounds 6, 7, 8, 9 and 1	S2-11
Determinatin of optical purity of compound 8 by chiral HPLC	S12-13

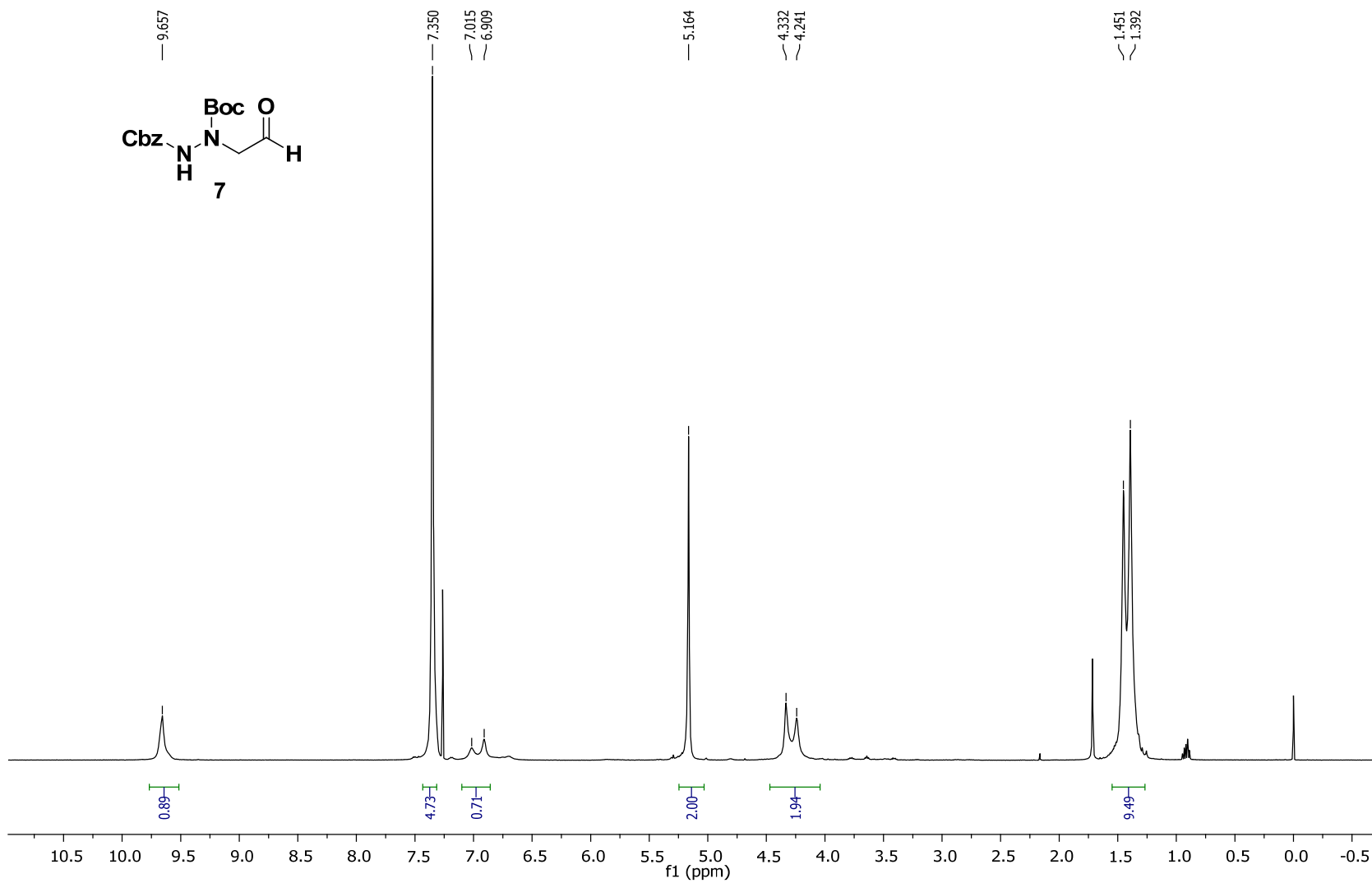
500 MHz ^1H NMR spectrum of 2-Benzyl 1-*tert*-butyl 1-(2-hydroxyethyl)hydrazine-1,2-dicarboxylate (**6**) in $\text{DMSO-}d_6$, 65 $^\circ\text{C}$



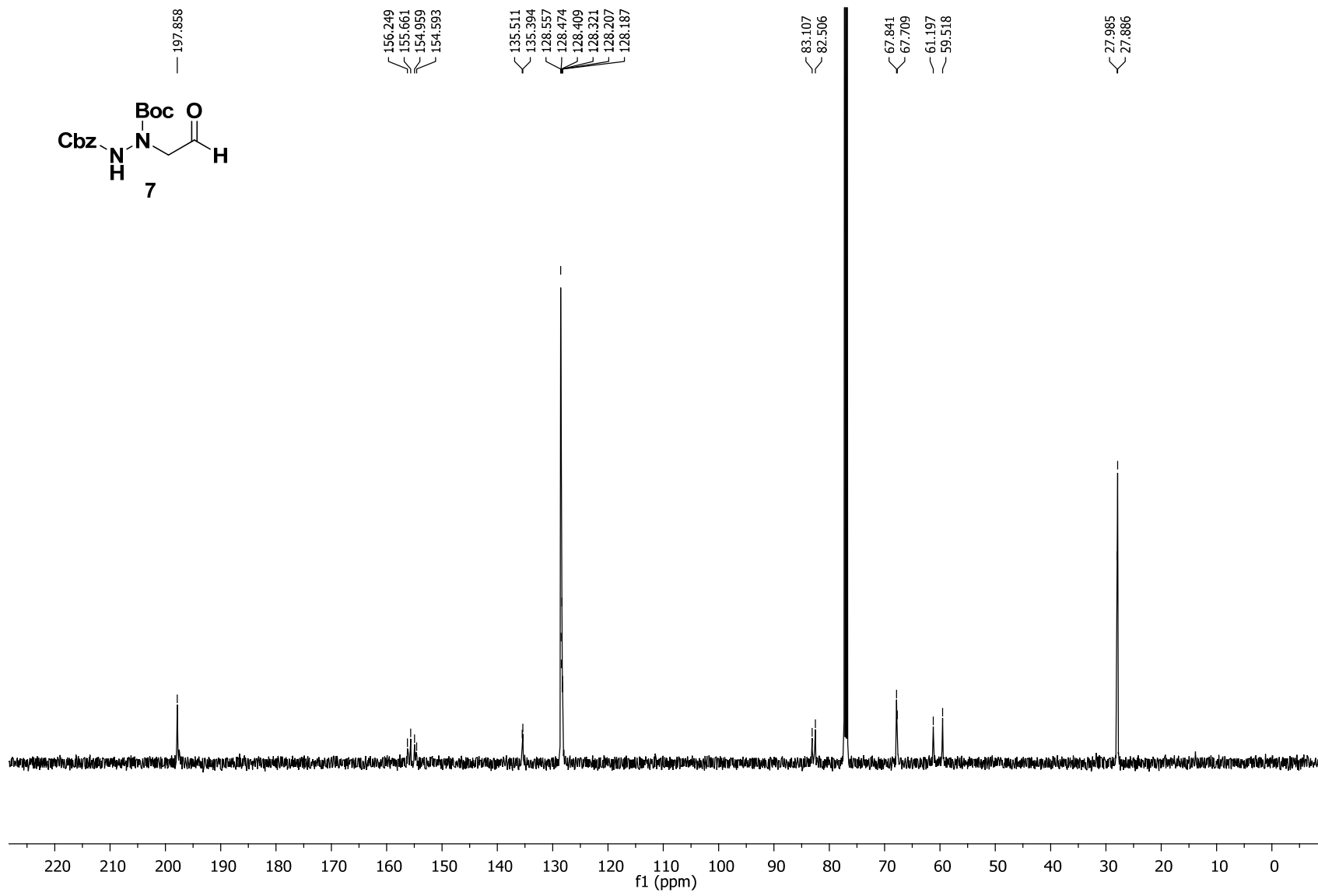
125 MHz ^{13}C NMR spectrum of 2-Benzyl 1-*tert*-butyl 1-(2-hydroxyethyl)hydrazine-1,2-dicarboxylate (**6**) in $\text{DMSO-}d_6$, 65 °C



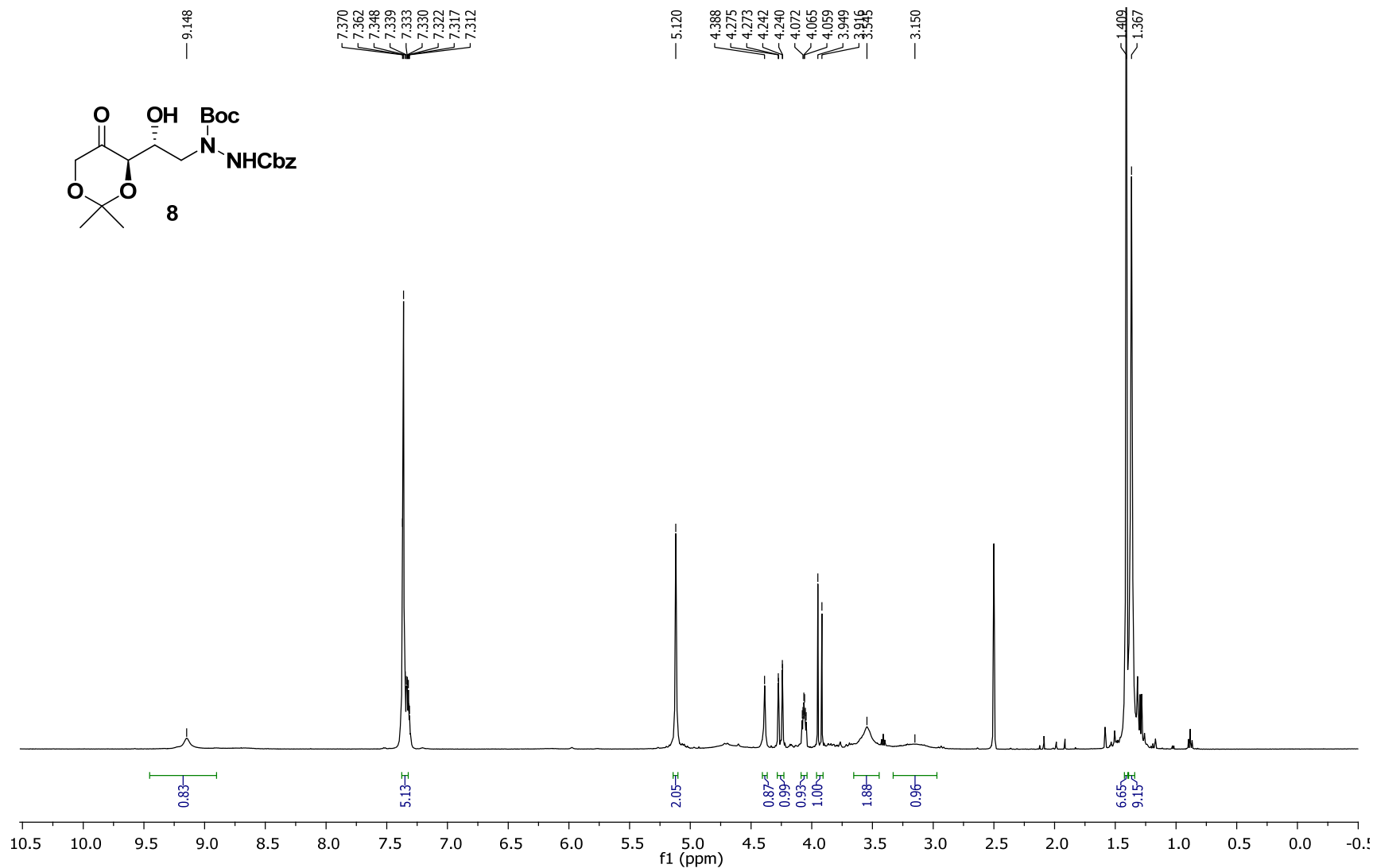
500 MHz ^1H NMR spectrum of 2-Benzyl 1-*tert*-butyl 1-(2-oxoethyl)hydrazine-1,2-dicarboxylate (**7**) in CDCl_3



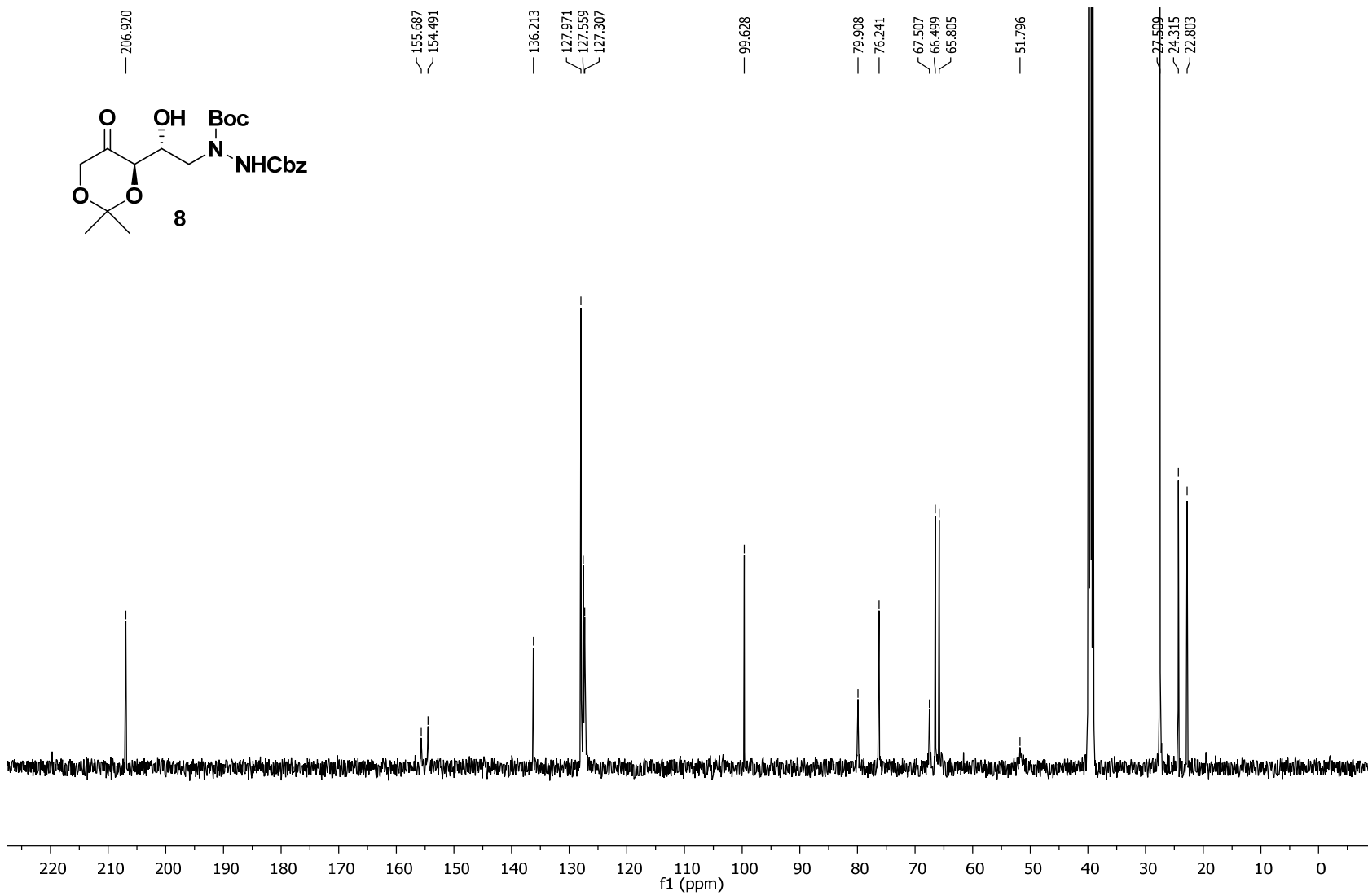
125 MHz ^{13}C NMR spectrum of 2-Benzyl 1-*tert*-butyl 1-(2-oxoethyl)hydrazine-1,2-dicarboxylate (**7**) in CDCl_3



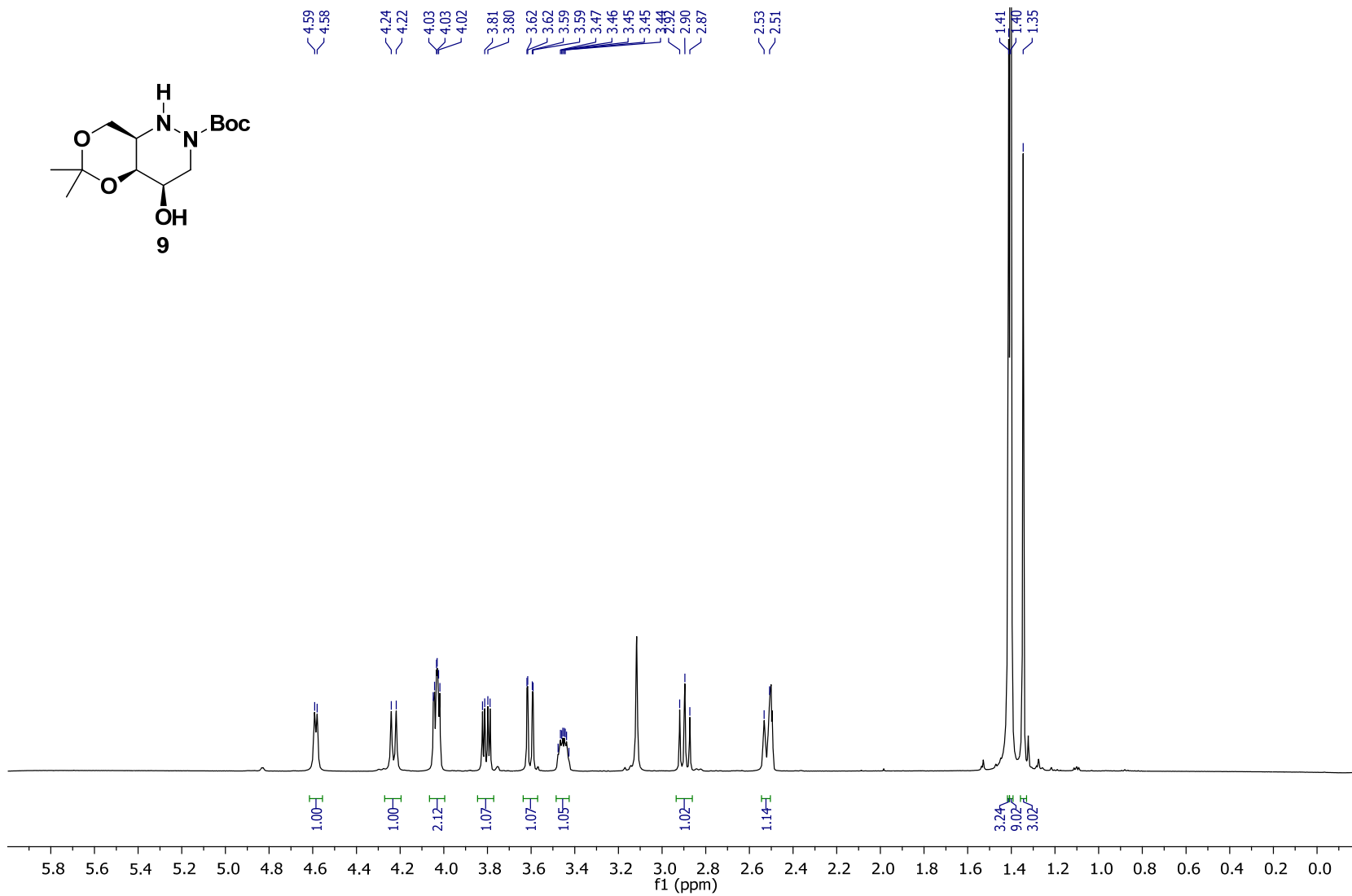
500 MHz ^1H NMR spectrum of 2-Benzyl 1-*tert*-butyl 1-((*R*)-2-((*R*)-2,2-dimethyl-5-oxo-1,3-dioxan-4-yl)-2-hydroxyethyl)hydrazine-1,2-dicarboxylate (**8**) in $\text{DMSO-}d_6$, 65 $^\circ\text{C}$



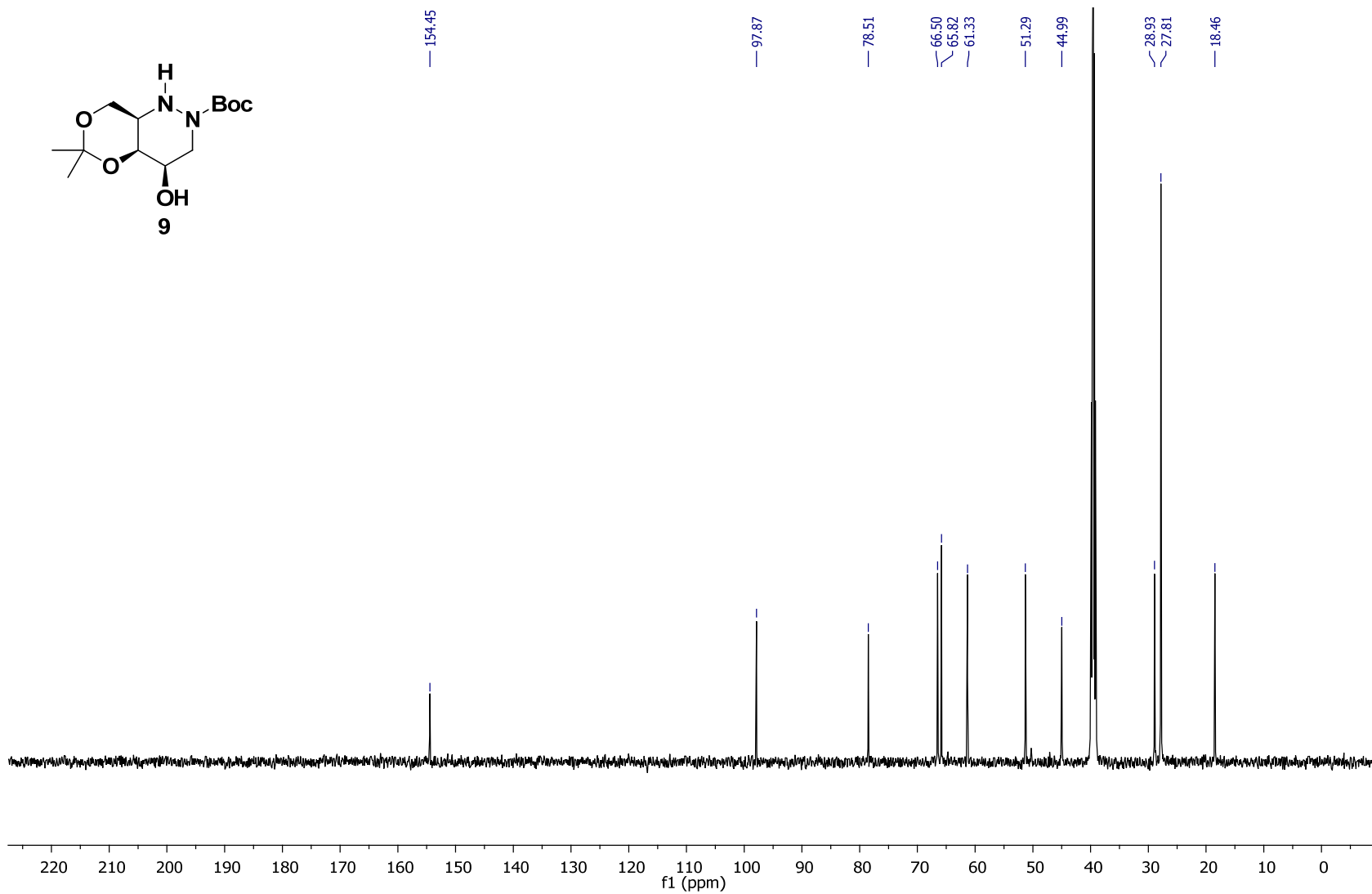
125 MHz ^{13}C NMR spectrum of 2-Benzyl 1-*tert*-butyl 1-((*R*)-2-((*R*)-2,2-dimethyl-5-oxo-1,3-dioxan-4-yl)-2-hydroxyethyl)hydrazine-1,2-dicarboxylate (**8**) in $\text{DMSO-}d_6$, 65 °C



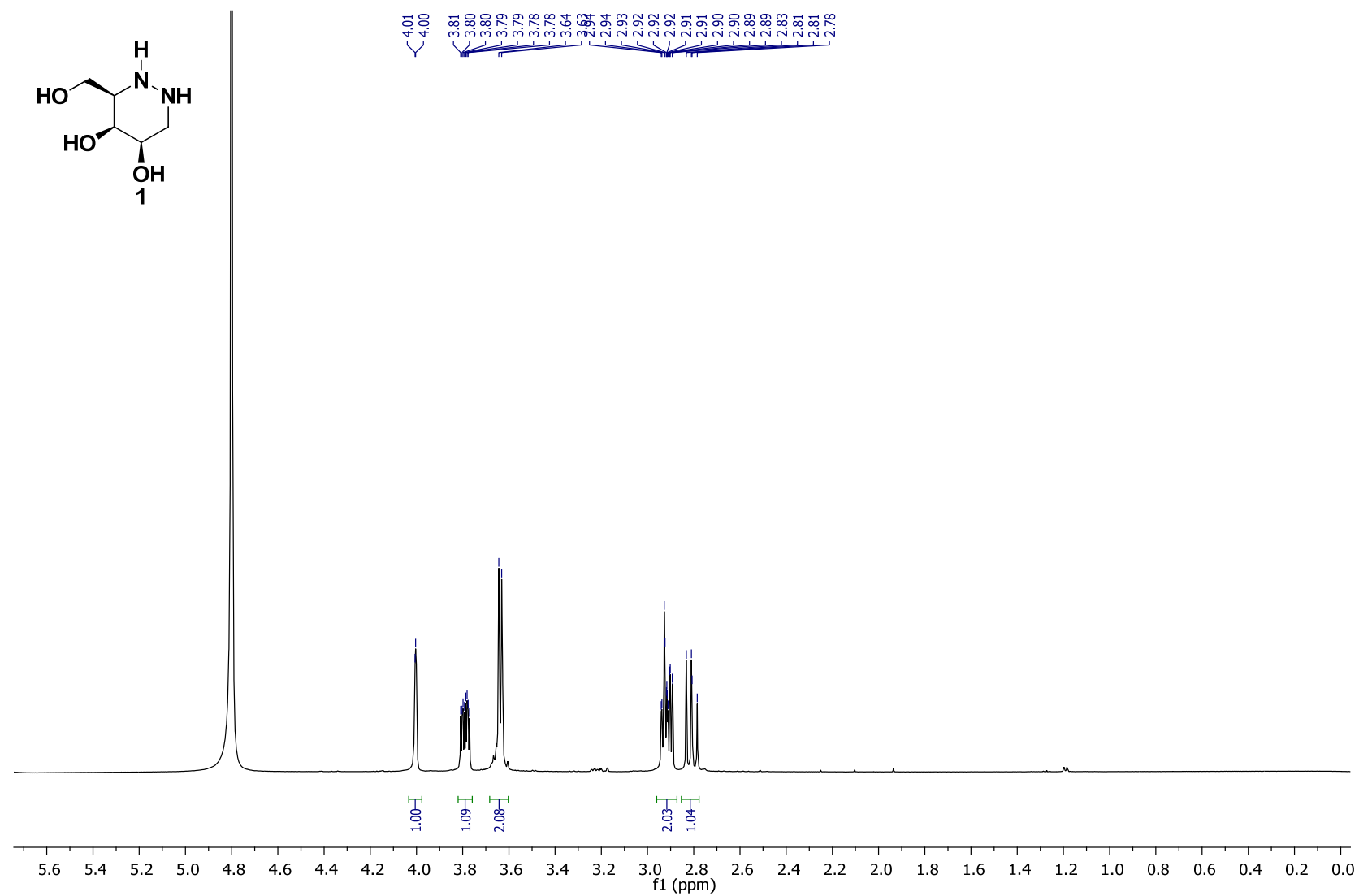
500 MHz ^1H NMR spectrum of (4*R*,4*aS*,8*aR*)-*tert*-butyl 4-hydroxy-6,6-dimethyltetrahydro-1*H*-[1,3]dioxino[5,4-*c*]pyridazine-2(3*H*)-carboxylate (**9**) in $\text{DMSO-}d_6$, 65 $^\circ\text{C}$



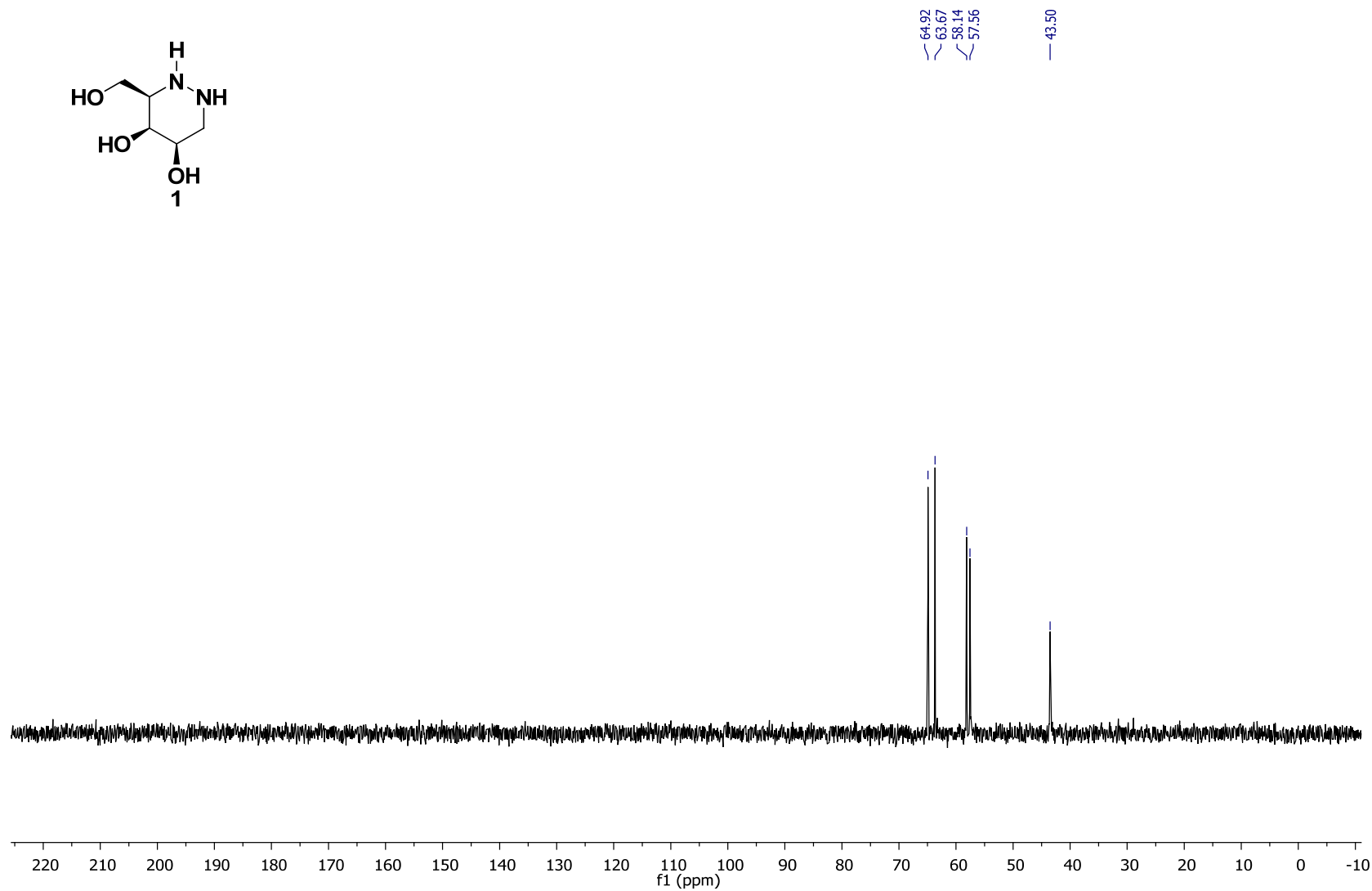
125 MHz ^{13}C NMR spectrum of (4*R*,4*aS*,8*aR*)-*tert*-butyl 4-hydroxy-6,6-dimethyltetrahydro-1*H*-[1,3]dioxino[5,4-*c*]pyridazine-2(3*H*)-carboxylate (**9**) in $\text{DMSO-}d_6$, 65 °C



500 MHz ^1H NMR spectrum of (3*R*,4*S*,5*R*)-3-(hydroxymethyl)piperazine-4,5-diol (aza-galacto-fagomine) (**1**) in D_2O



125 MHz ^{13}C NMR spectrum of (3*R*,4*S*,5*R*)-3-(hydroxymethyl)piperazine-4,5-diol (aza-galacto-fagomine) (**1**) in D_2O



Determination of the optical purity of aldol 8

98% ee; HPLC: Chiracel OZ-H (250 x 46 mm), *n*-heptane/*i*PrOH = 80/20, 0.5 mL/min, $t_{R1} = 19.63$ min i $t_{R2} = 22.39$ min

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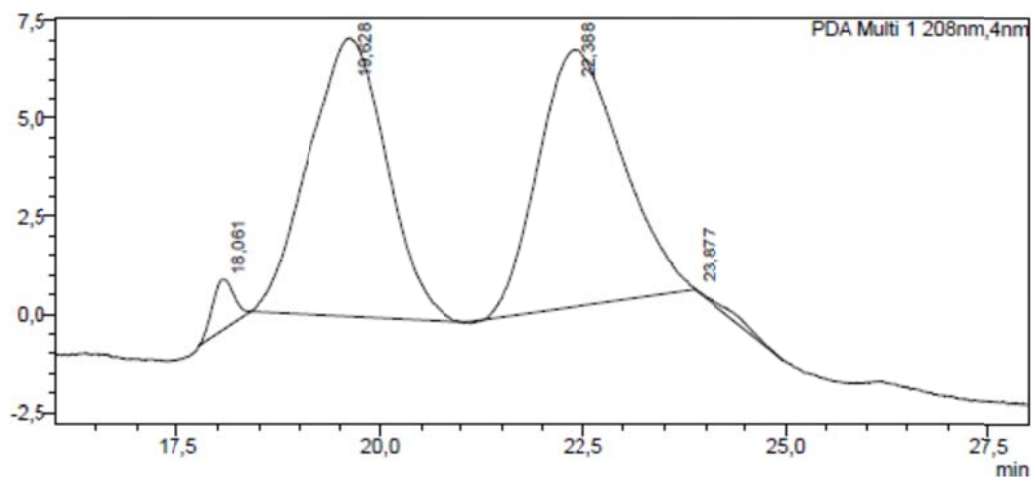
HPLC chromatogram of the racemic aldol **8-rac** for control

<Sample Information>

Sample Name	: 462-dl	Sample Type	: Unknown
Sample ID	: 462-dl_6	Acquired by	: System Administrator
Data Filename	: 462-dl_6.lcd	Processed by	: System Administrator
Method Filename	: C6 80_20 60 min fl 0.5.lcm		
Batch Filename	: 20170212.lcb		
Vial #	: 1-94		
Injection Volume	: 5 uL		
Date Acquired	: 18-2-2017 0:35:55		
Date Processed	: 18-2-2017 1:35:57		

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 208nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	18,061	23555	1288	0,000		M	
2	19,628	472928	7120	0,000		M	
3	22,388	474170	6580	0,000		M	
4	23,877	5383	9	0,000		M	
Total		976037	14997				

#

Peak	Retention time (min)	Area	Area %
1	19.628	472928	49.9
2	22.388	474170	50.1
Total		947098	100

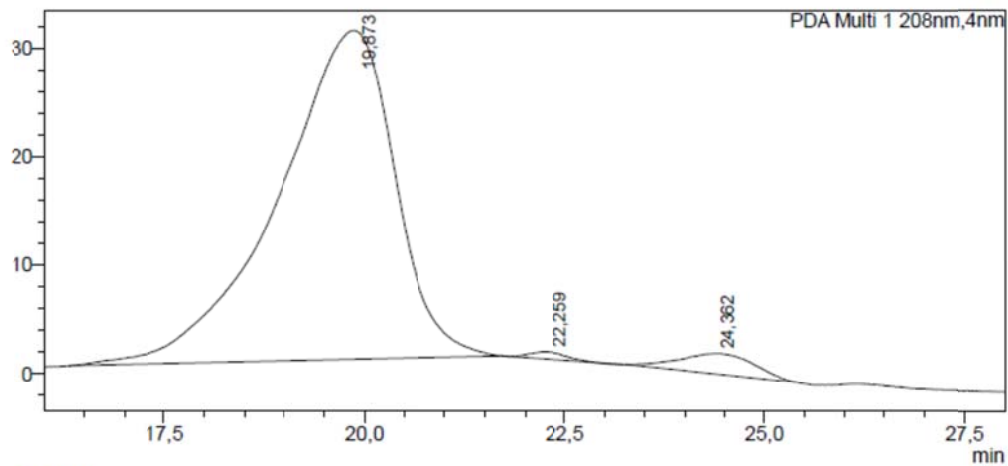
HPLC Chromatogram of aldol 8

<Sample Information>

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Sample ID : 462-d_6	Acquired by : System Administrator
Data Filename : 462-d_6.lcd	Processed by : System Administrator
Method Filename : C6_80_20_60 min fl 0.5.lcm	
Batch Filename : 20170212.lcb	
Vial # : 1-26	
Injection Volume : 5 uL	
Date Acquired : 20-2-2017 5:46:11	
Date Processed : 20-2-2017 6:46:14	

<Chromatogram>

mAU



<Peak Table>

PDA Ch1 208nm							
Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	19.873	3061673	30273	0,000		M	
2	22.259	21510	692	0,000		M	
3	24.362	123388	1858	0,000		M	
Total		3206570	32824				

Peak	Retention time (min)	Area	Area %
1	19.873	3061673	99.3
2	22.259	21510	0.7
Total		3083183	100