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SUPPORTING INFORMATION

DOI: 10.1002/ejoc.201600141
Title: Ring-Opening of Carbamate-Protected Aziridines and Azetidines in Liquid Sulfur Dioxide
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1. Safety information on sulfur dioxide

Symbol	 GHS04 [1]	 GHS05	 GHS06 (Category 3)
Signal word	Danger		
Hazard statements	H280-H314-H331		
Precautionary statements	P261-P280-P305 + P351 + P338-P310-P410 + P403		
Personal Protective Equipment	Faceshields, full-face respirator (US), Gloves, Goggles, multi-purpose combination respirator cartridge (US)		
RIDADR	UN 1079 8(2.3)		
WGK Germany	1		
RTECS	WS4550000		

It should be mentioned that also other frequently used laboratory chemicals have similar levels of safety issues and should be treated with appropriate care.
Some examples are:
Formic acid: GHS02, GHS05, GHS06
HCl in dioxane: GHS02, GHS05, GHS07, GHS08
Thiophenol: GHS02, GHS05, GHS06, GHS08, GHS09

[1] See the phase diagram of SO_2 : it is easy to liquefy gas which develops relatively low pressure.

2. Additional example on ionizing and dissociating ability of liquid sulfur dioxide

An excerpt from Reichardt, C. Solvents and Solvent Effects in Organic Chemistry, Third Edition. WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim, 2003, Chapter 2.6. Ionization and Dissociation (pages 49-50):

Chloro-triphenylmethane constitutes a classical example for distinguishing the ionizing and dissociating ability of a solvent. In 1902, Walden used it in liquid sulfur dioxide in the first demonstration of the existence of carbenium ions [147]. The colourless chloro-triphenylmethane dissolves in liquid sulfur dioxide ($\varepsilon_r = 15.6$ at 0 °C), giving an intense yellow colour ($\lambda_{max} = 430$ nm). This is caused by a partial formation of ion pairs, which do not conduct electricity. At low concentrations, the ion pairs partially dissociate into free ions, which do conduct electricity [148, 149].

$$[(C_6H_5)_3C\text{-}Cl]_{SO_2} \overset{K_{Ion}}{\rightleftharpoons} [(C_6H_5)_3C^{\oplus}\ Cl^{\ominus}]_{SO_2} \overset{K_{Dissoc}}{\rightleftharpoons} [(C_6H_5)_3C^{\oplus}]_{SO_2} + [Cl^{\ominus}]_{SO_2} \qquad (2\text{-}18)$$

colorless yellow yellow

$K_{Ion} = 1.46 \cdot 10^{-2}$ (0 °C); $K_{Dissoc} = 2.88 \cdot 10^{-3}$ mol/L (0 °C);

$K_{exp} = 4.1 \cdot 10^{-5}$ mol/L (0 °C) [148].

Table 2-11. Ionization equilibrium constants K_{Ion} of chloro-triphenylmethane in various solvents at 0...25 °C [150]. *Cf.* also [282].

Solvents	ε_r[a] (at 0...25 °C)	$K_{Ion} \cdot 10^4$	References
Nitrobenzene	34.8 (25 °C)	Too low to measure (25 °C)[c]	[151]
Acetonitrile	35.9 (25 °C)	Too low to measure (25 °C)	[152]
Dichloromethane	8.9 (25 °C)	0.07	[153]
1,1,2,2-Tetrachloroethane	8.2 (20 °C)	0.48 (18.5 °C)	[154]
1,2-Dichloroethane	10.4 (25 °C)	0.56 (20 °C)	[154]
Nitromethane	35.9 (25 °C)	2.7 (25 °C)	[155]
Sulfur dioxide	15.6 (0 °C)[b]	146 (0 °C)	[148]
Formic acid	58.5 (16 °C)	3100 (20.5 °C)	[156]
m-Cresol	11.8 (25 °C)	5600[d] (18 °C)	[157]

[a] J. A. Riddick, W. B. Bunger, and T. K. Sakano: *Organic Solvents*, 4th edition, in A. Weissberger (ed.), *Techniques of Chemistry*, Vol. II, Wiley-Interscience, New York, 1986.
[b] A. A. Maryott and E. R. Smith: *Table of Dielectric Constants of Pure Liquids*, NBS Circular 514, Washington, 1951.
[c] Because nitrobenzene absorbs strongly at the wavelength of the carbenium ion maximum from chlorotriphenylmethane, this result was obtained with chloro-diphenyl-4-tolylmethane.
[d] This K_{Ion} value corresponds to 36 ± 4% ionization of chlorotriphenylmethane in *m*-cresol [157].

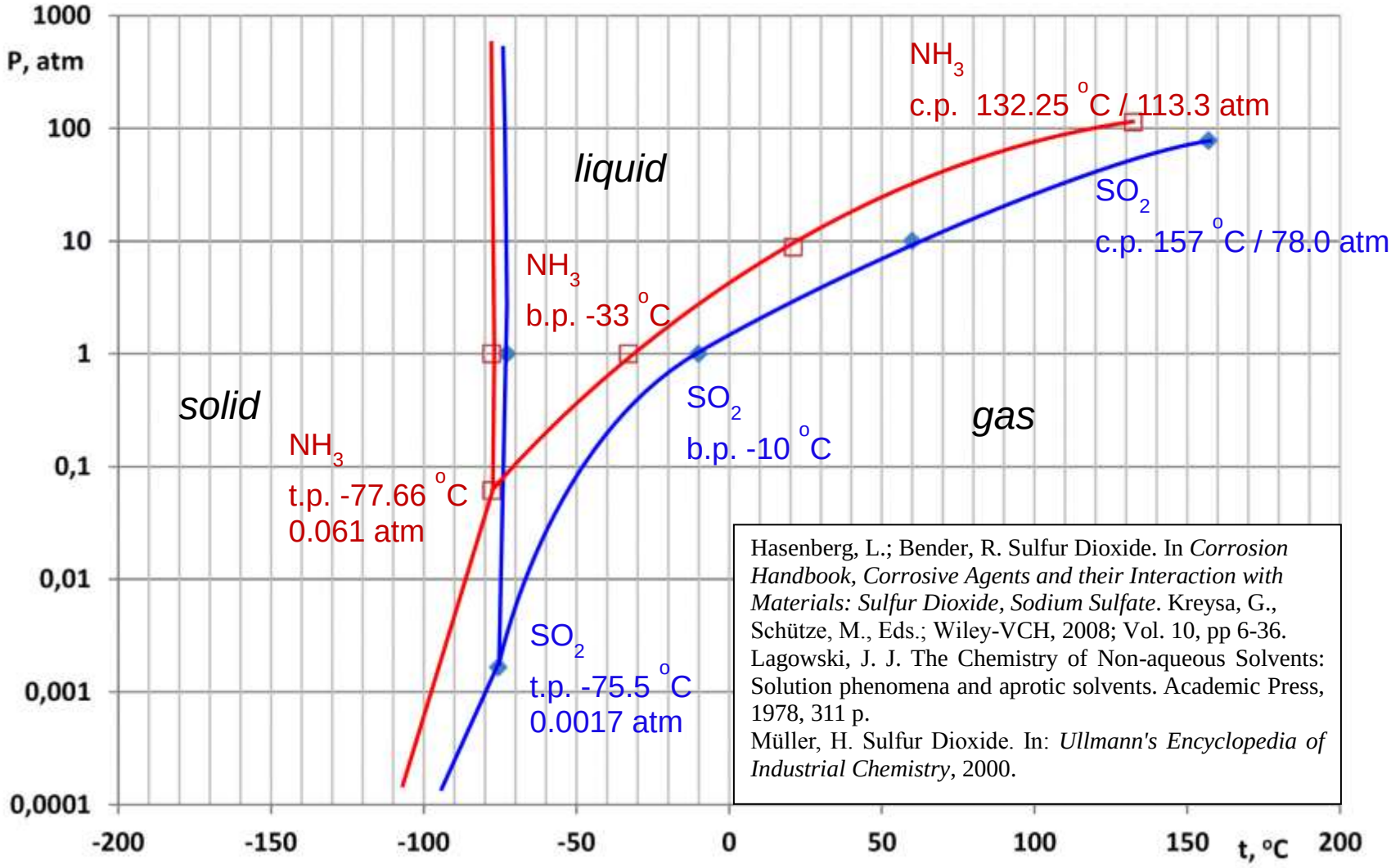
Sulfur dioxide is a π-electron-pair acceptor. The standard explanation for the strong ionizing power of SO_2 is the formation of an EPD—EPA complex between the halide anion and the sulfur dioxide molecules [148]. Table 2-11 summarizes some of the available data concerning the comparative efficiencies of various solvents in promoting the ionization of chloro-triphenylmethane [150].

[147] P. Walden, Ber. Dtsch. Chem. Ges. 35, 2018 (1902).
[148] N. N. Lichtin: Ionization and Dissociation Equilibria in Solution in Liquid Sulfur Dioxide, Progr. Phys. Org. Chem. 1, 75 (1963).
[149] W. Karcher and H. Hecht: Chemie in flussigem Schwefeldioxid, in G. Jander, H. Spandau, and C. C. Addison (eds.): Chemie in nichtwaßrigen ionisierenden Losungsmitteln, Vieweg, Braunschweig, Pergamon Press, Oxford, 1967, Vol. III, Part 2, p. 79ff.
[150] H. H. Freedman: Arylcarbonium Ions, in G. A. Olah and P. v. R. Schleyer (eds.): Carbonium Ions, Wiley-Interscience, New York, 1973, Vol. IV, p. 1501ff.

Additionally, dielectric constants of sulfur dioxide at various temperatures were determined by Nickerson, J. D.; McIntosh, R. *Can. J. Chem.* **1957**, *35*, 1325-1331:

Temperature, ° C.	Frequency, Mc./second	Dielectric constant
	A. Sulphur dioxide	
6.4	64.0	14.65
−5.5	91.5	15.50
−10.4	48.8	15.90
−18.2	10.3	16.41
−22.0	91.5	16.70
−25.0	64.0	16.95
−30.0	91.5	17.34
−30.0	10.3	17.32
−30.1	48.8	17.35

3. Comparison of phase diagrams of sulfur dioxide (blue) and ammonia (red)



Hasenberg, L.; Bender, R. Sulfur Dioxide. In *Corrosion Handbook, Corrosive Agents and their Interaction with Materials: Sulfur Dioxide, Sodium Sulfate*. Kreysa, G., Schütze, M., Eds.; Wiley-VCH, 2008; Vol. 10, pp 6-36.
Lagowski, J. J. The Chemistry of Non-aqueous Solvents: Solution phenomena and aprotic solvents. Academic Press, 1978, 311 p.
Müller, H. Sulfur Dioxide. In: *Ullmann's Encyclopedia of Industrial Chemistry*, 2000.

4. Comparison of catalogue prices for anhydrous solvents

Entry	Catalog Nr	Name	Purity	Water content	Price			Webpage
					Quantity	Catalog price, EUR	Price per kg or L, EUR	
1.	AGA, The Linde Group	**Sulfur dioxide, 3.8**	**99.98%**	**50 ppm**	61,6 kg 50 L	315.00	5.11 €/kg 6.30 €/L	http://hiq.linde-gas.com/en/specialty_gases/pure_gas_finder.html
2.	Acros 42325010 0	Acetonitrile, 99.6%, ACS reagent	≥99.5%	≤0.3%	10 L	343.80	34.38 €/L	http://www.acros.com/DesktopModules/Acros_Search_Results/Acros_Search_Results.aspx?search_type=CAS&SearchString=75-05-8
3.	Ficher A/0620/ 27	Acetonitrile, extra pure, SLR	≥99%	≤0.3%	200 L	740.80	3.70 €/L	http://www.acros.com/DesktopModules/Acros_Search_Results/Acros_Search_Results.aspx?search_type=CAS&SearchString=75-05-8
4.	Acros 29552025 0	Methyl sulfoxide, 99.8+%, extra pure	≥99.8%	≤0.1%	25 L	587.40	23.50 €/L	http://www.acros.com/DesktopModules/Acros_Search_Results/Acros_Search_Results.aspx?search_type=PartOfName&SearchString=dimethylsulfoxide
5.	Acros 12779025 0	Methyl sulfoxide, 99.7%, pure	≥99.6%	≤0.2%	25 L	196.00	7.84 €/L	http://www.acros.com/DesktopModules/Acros_Search_Results/Acros_Search_Results.aspx?search_type=PartOfName&SearchString=dimethylsulfoxide
6.	Acros 34844002 5	Methyl sulfoxide, 99.7+%, Extra Dry over Molecular Sieve, AcroSeal®	≥99.7%	≤0.005 % **(≤ 50 ppm)**	2.5 L	140.80	56.32 €/L	http://www.acros.com/DesktopModules/Acros_Search_Results/Acros_Search_Results.aspx?search_type=PartOfName&SearchString=dimethylsulfoxide

continued

Entry	Catalog Nr	Name	Purity	Water content	Price			Webpage
					Quantity	Catalog price, EUR	Price per kg or L, EUR	
7.	Acros 176630250	Tetrahydrofuran, 99+%, extra pure, stabilized with BHT	≥99.0%	≤0.05%	25 L	421.70	16.87 €/L	http://www.acros.com/DesktopModules/Acros_Search_Results/Acros_Search_Results.aspx?search_type=CAS&SearchString=109-99-9
8.	Fisher T/0700/27	Tetrahydrofuran, extra pure, SLR, stabilized with 0.025% BHT	≥99.5%	≤0.05%	200 L	1549.40	7.75 €/L	http://www.acros.com/DesktopModules/Acros_Search_Results/Acros_Search_Results.aspx?search_type=CAS&SearchString=109-99-9
9.	Acros 326970025	Tetrahydrofuran, 99.85%, Extra Dry, stabilized, AcroSeal®	≥99.80 %	≤0.005 % **(≤ 50 ppm)**	2.5 L	193.90	77.56 €/L	http://www.acros.com/DesktopModules/Acros_Search_Results/Acros_Search_Results.aspx?search_type=CAS&SearchString=109-99-9
10.	Fisher T/0702/PB17	Tetrahydrofuran, Extra Dry, for synthesis, unstabilized	n.a.	≤0.01%	2.5 L	159.90	63.96 €/L	http://www.acros.com/DesktopModules/Acros_Search_Results/Acros_Search_Results.aspx?search_type=CAS&SearchString=109-99-9
11.	Acros 326870025	N,N-Dimethylformamide, 99.8%, Extra Dry, AcroSeal®	≥99.75 %	≤0.005 % **(≤ 50 ppm)**	2.5 L	240.70	96.28 €/L	http://www.acros.com/DesktopModules/Acros_Search_Results/Acros_Search_Results.aspx?search_type=PartOfName&SearchString=dimethylformamide
12.	Fisher D/3841/27	Dimethylformamide, for analysis	≥99.5%	≤0.05%	200 L	953.20	4.77 €/L	http://www.acros.com/DesktopModules/Acros_Search_Results/Acros_Search_Results.aspx?search_type=PartOfName&SearchString=dimethylformamide

continued

Entry	Catalog Nr	Name	Purity	Water content	Price			Webpage
					Quantity	Catalog price, EUR	Price per kg or L, EUR	
13.	Acros 116220250	N,N-Dimethylformamide, 99+%, extra pure	≥99.0%	≤0.2%	25 L	307.30	12.29 €/L	http://www.acros.com/DesktopModules/Acros_Search_Results/Acros_Search_Results.aspx?search_type=PartOfName&SearchString=dimethylformamide
14.	Acros 131780250	Pyridine, 99+%, extra pure	≥99.0%	≤0.1%	25 L	972.20	38.89 €/L	http://www.acros.com/DesktopModules/Acros_Search_Results/Acros_Search_Results.aspx?search_type=1&SearchString=C5H5N

5. Visualisation of sulfur dioxide condensation process

4.1. Condensing of SO_2 into a pressure reactor, reaction and removal of SO_2.

Degassed vessel containing the substrate and the reagent

Filling of SO_2 into the vessel from storage cylinder

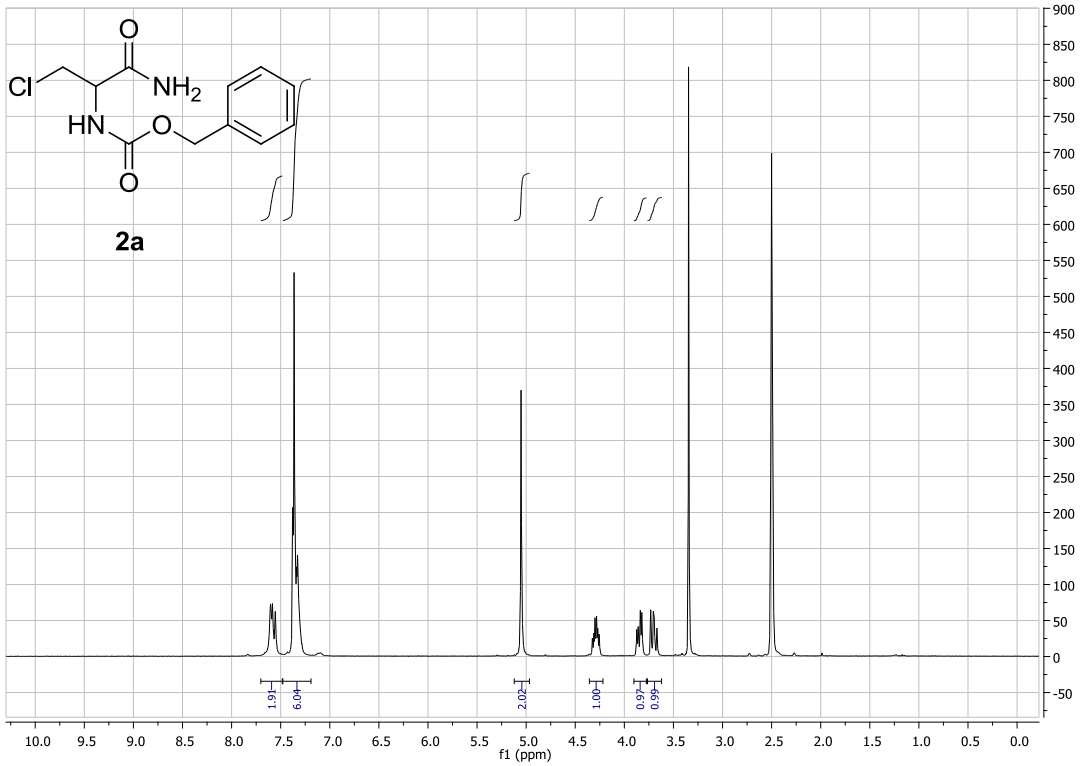
Performance of the reaction at the given temperature

Transfer of SO_2 back to the storage cylinder

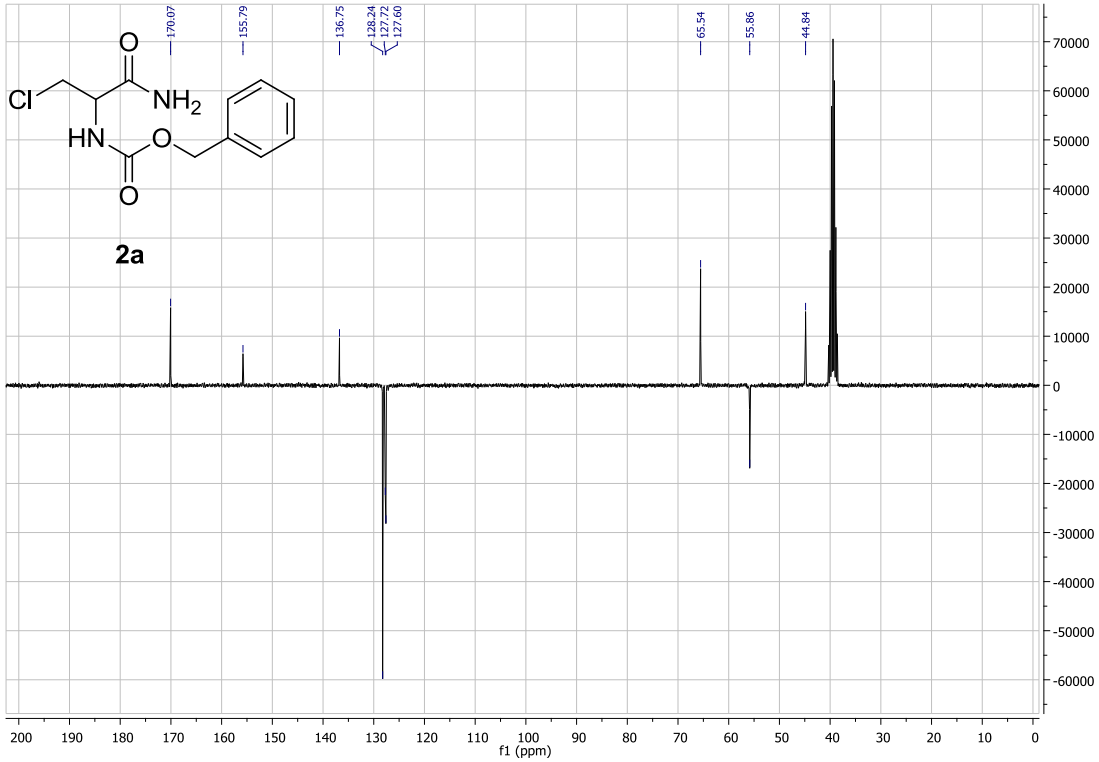
4.2. Condensing of SO_2 into a round bottom flask for reactions at ambient pressure and at temperatures $\leq -10\ ^{o}C$.

6. NMR Spectra

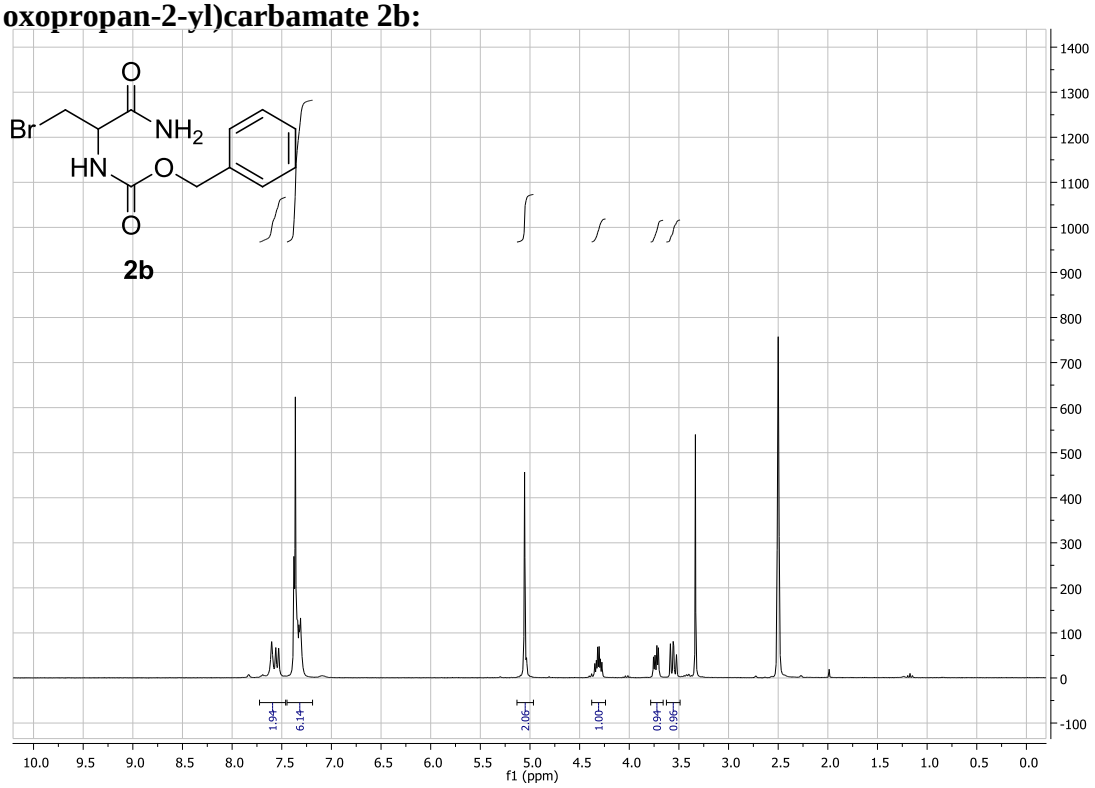
^{1}H-NMR (DMSO$_{d6}$, 300 MHz) spectrum of benzyl (1-amino-3-chloro-1-oxopropan-2-yl)carbamate 2a:



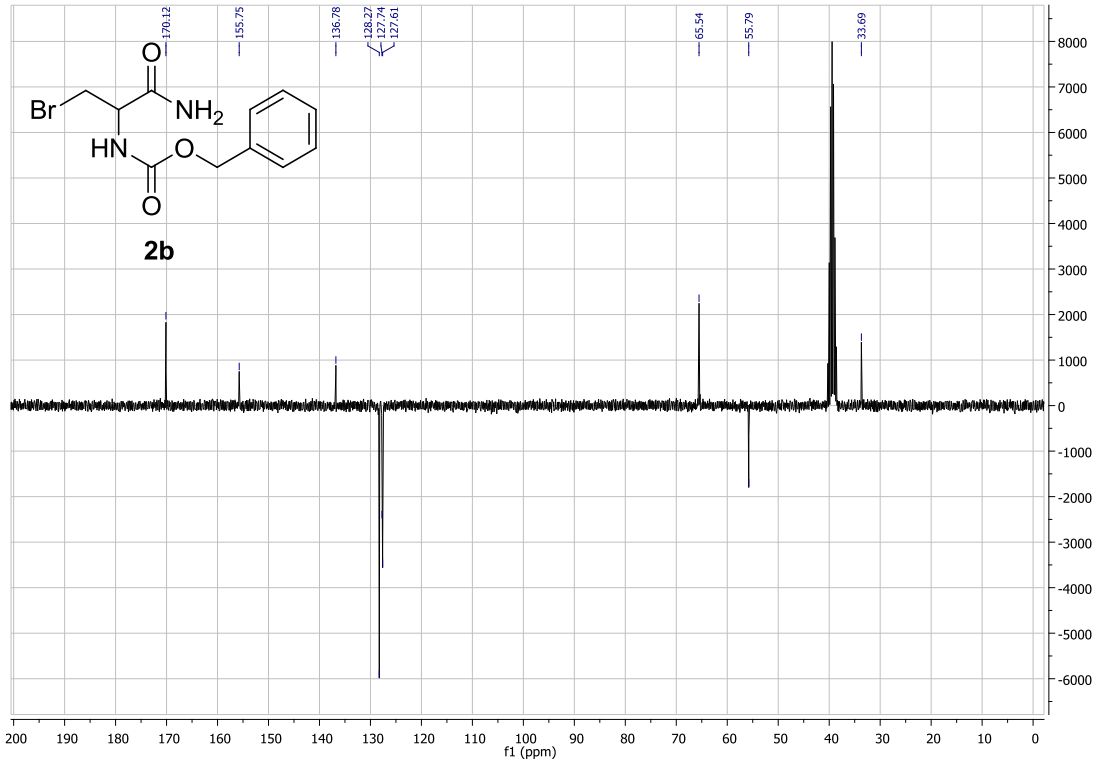
^{13}C-NMR (DMSO$_{d6}$, 75.5 MHz) spectrum of benzyl (1-amino-3-chloro-1-oxopropan-2-yl)carbamate 2a:



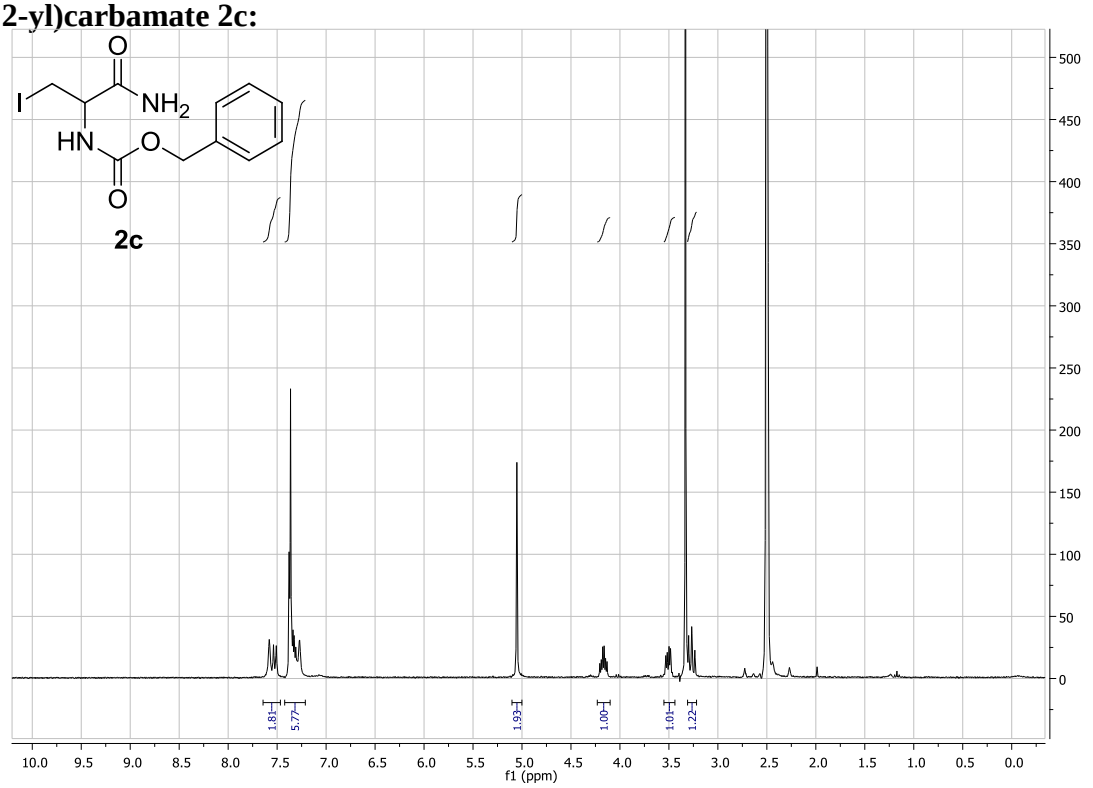
^{1}H-NMR (DMSO$_{d6}$, 300 MHz) spectrum of benzyl (1-amino-3-bromo-1-oxopropan-2-yl)carbamate 2b:



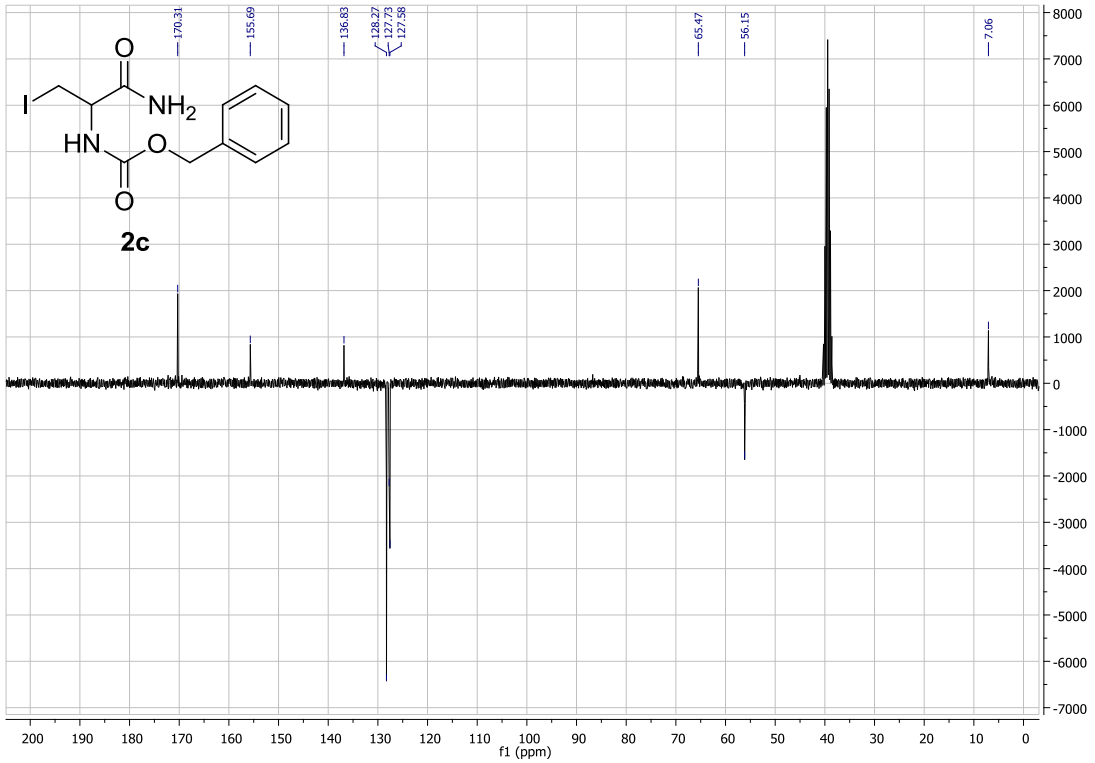
^{13}C-NMR (DMSO$_{d6}$, 75.5 MHz) spectrum of benzyl (1-amino-3-bromo-1-oxopropan-2-yl)carbamate 2b:



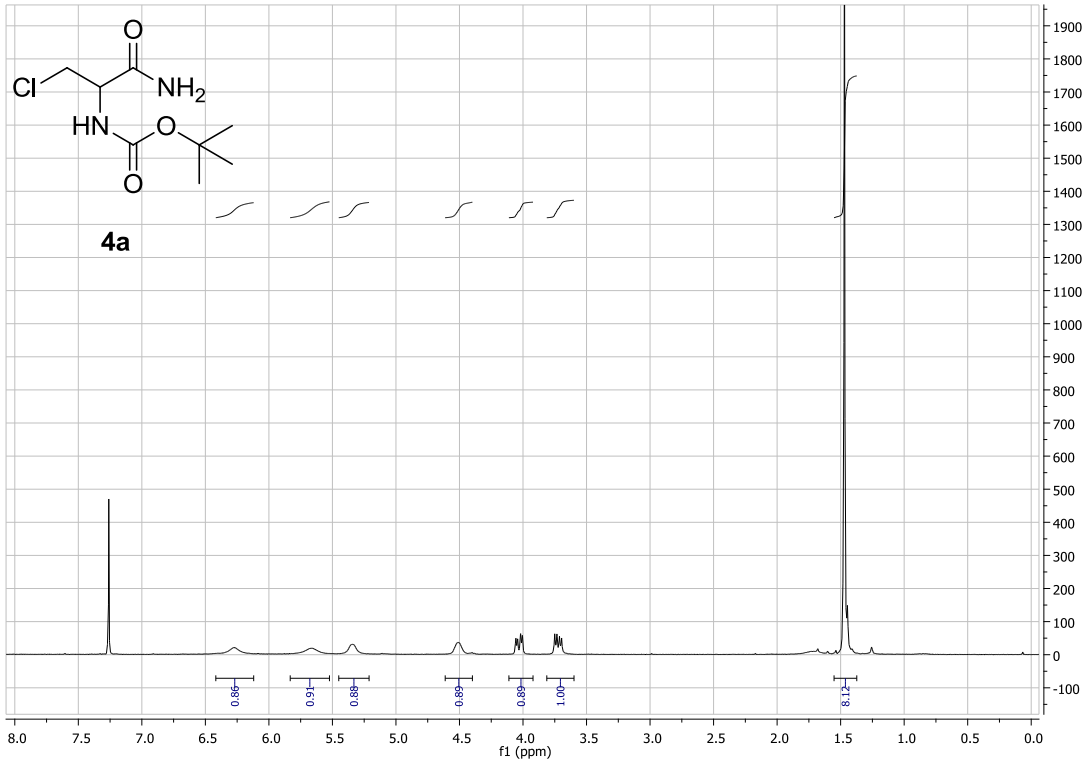
^{1}H-NMR (DMSO$_{d6}$, 300 MHz) spectrum of benzyl (1-amino-3-iodo-1-oxopropan-2-yl)carbamate 2c:



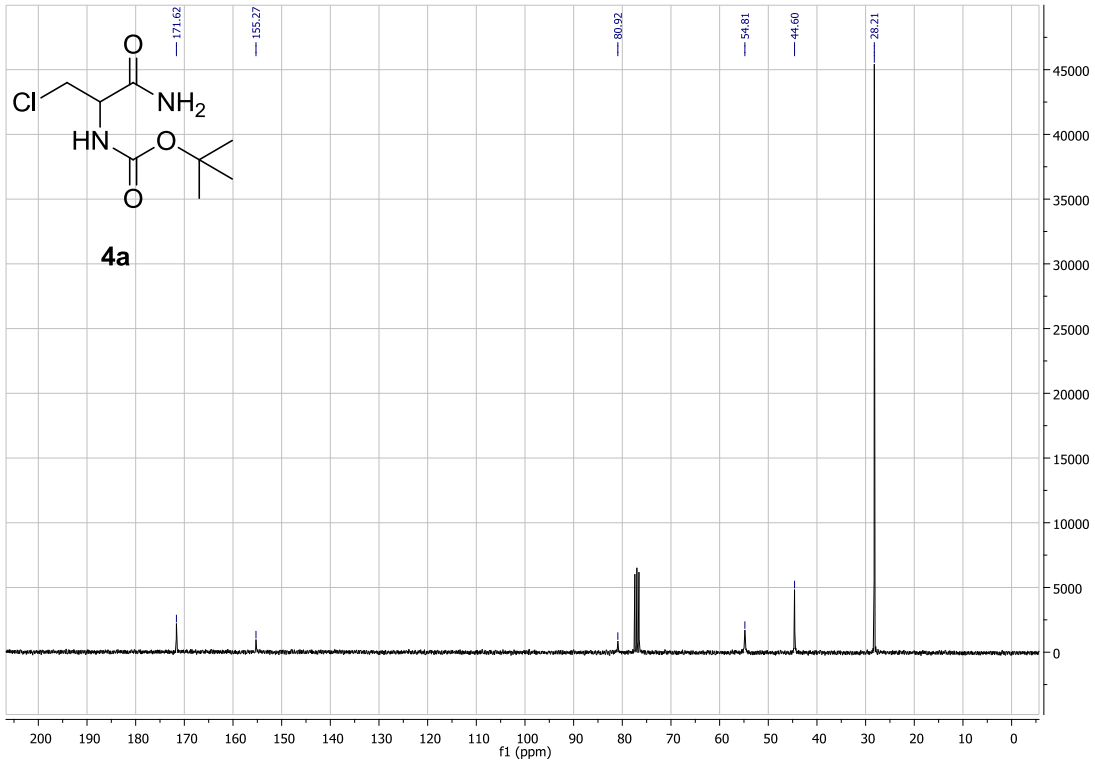
^{13}C-NMR (DMSO$_{d6}$, 75.5 MHz) spectrum of benzyl (1-amino-3-iodo-1-oxopropan-2-yl)carbamate 2c:



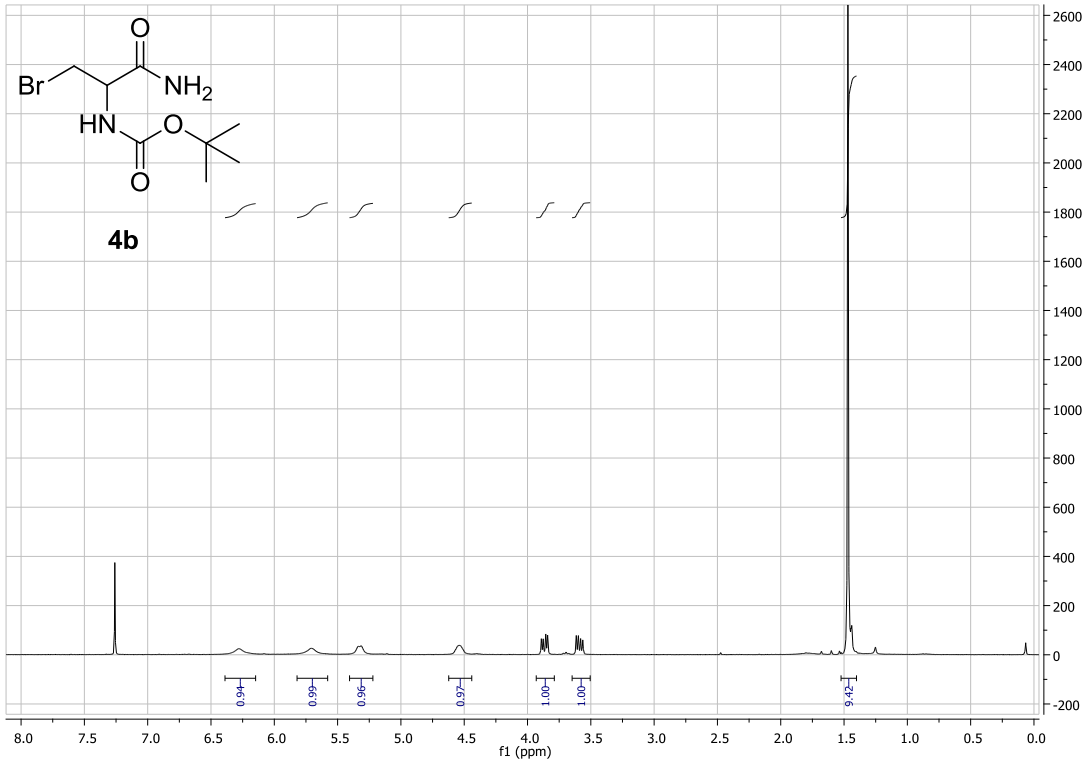
^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of *tert*-butyl (1-amino-3-chloro-1-oxopropan-2-yl)carbamate 4a:



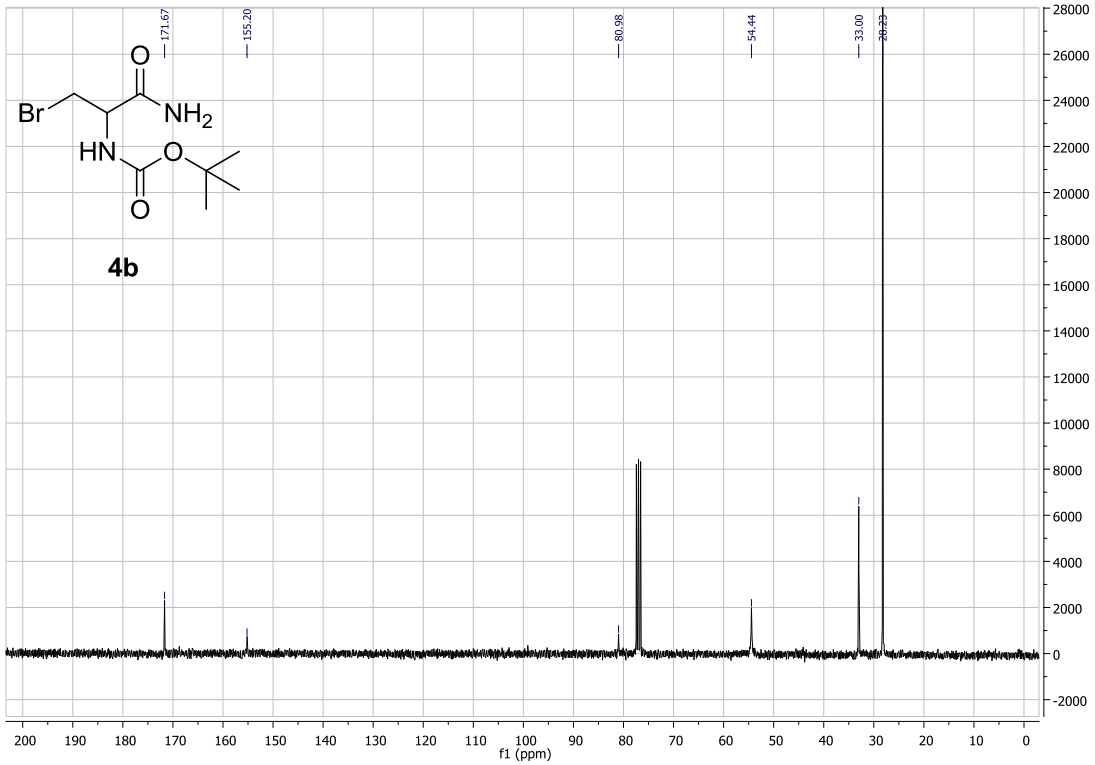
^{13}C-NMR ($CDCl_3$, 75.5 MHz) spectrum of *tert*-butyl (1-amino-3-chloro-1-oxopropan-2-yl)carbamate 4a:



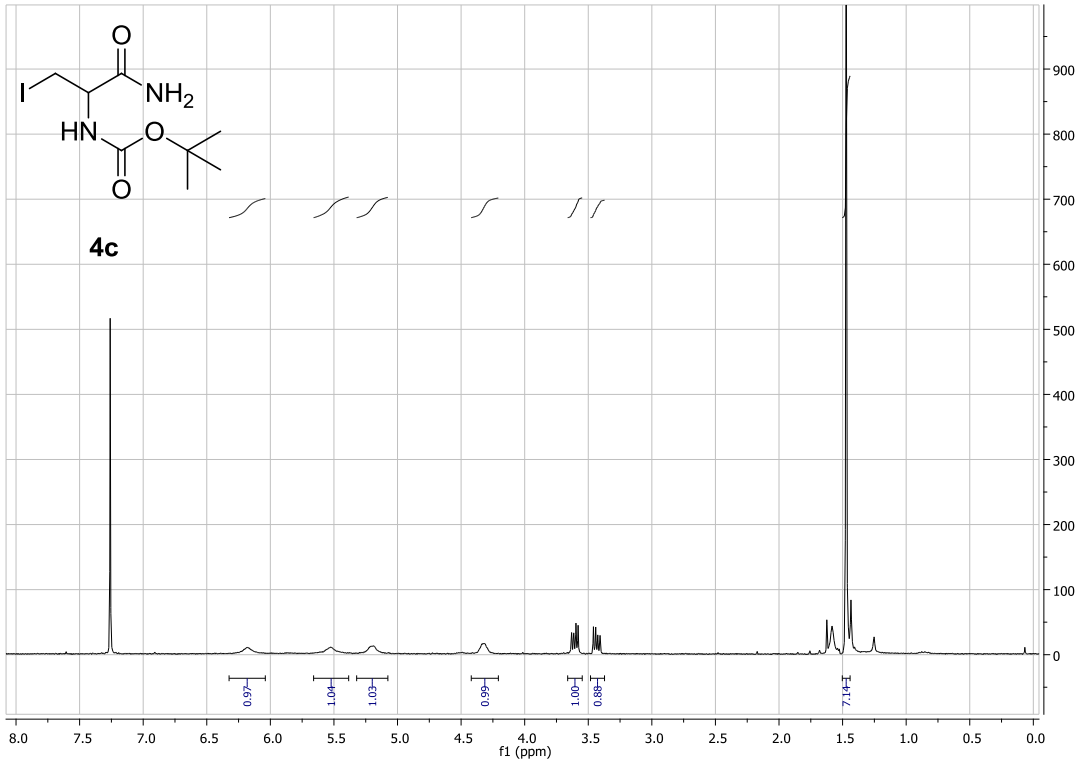
^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of *tert*-butyl (1-amino-3-bromo-1-oxopropan-2-yl)carbamate 4b:



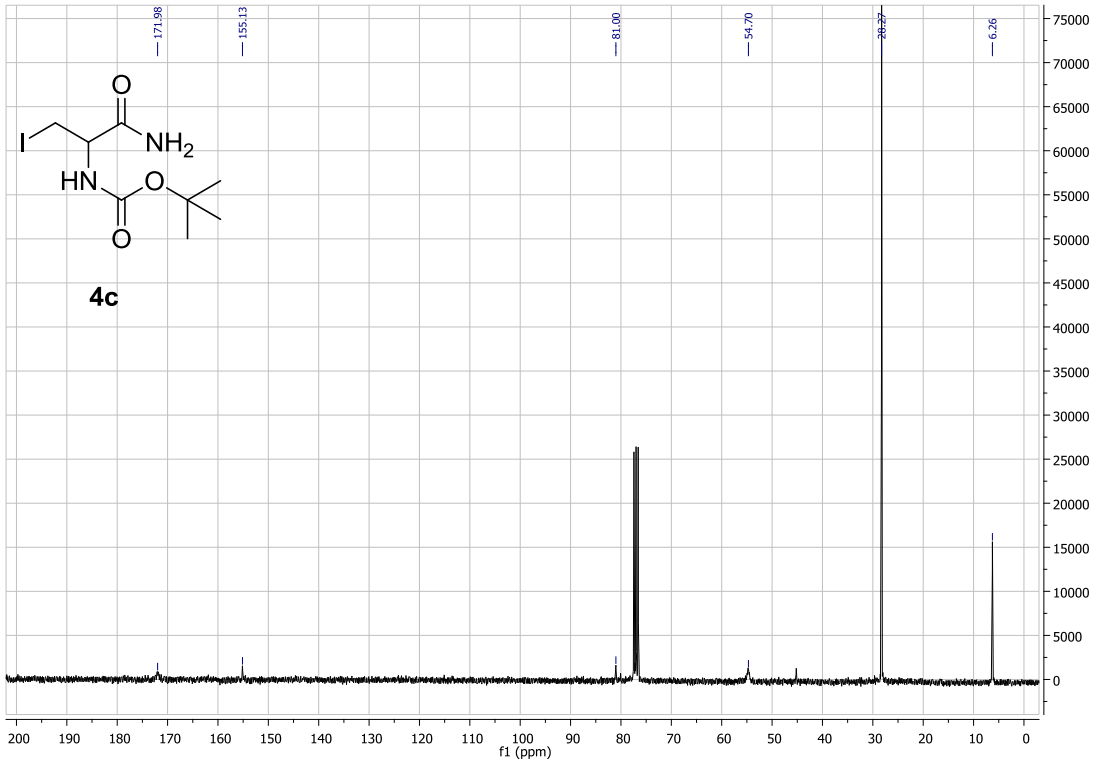
^{13}C-NMR ($CDCl_3$, 75.5 MHz) spectrum of *tert*-butyl (1-amino-3-bromo-1-oxopropan-2-yl)carbamate 4b:



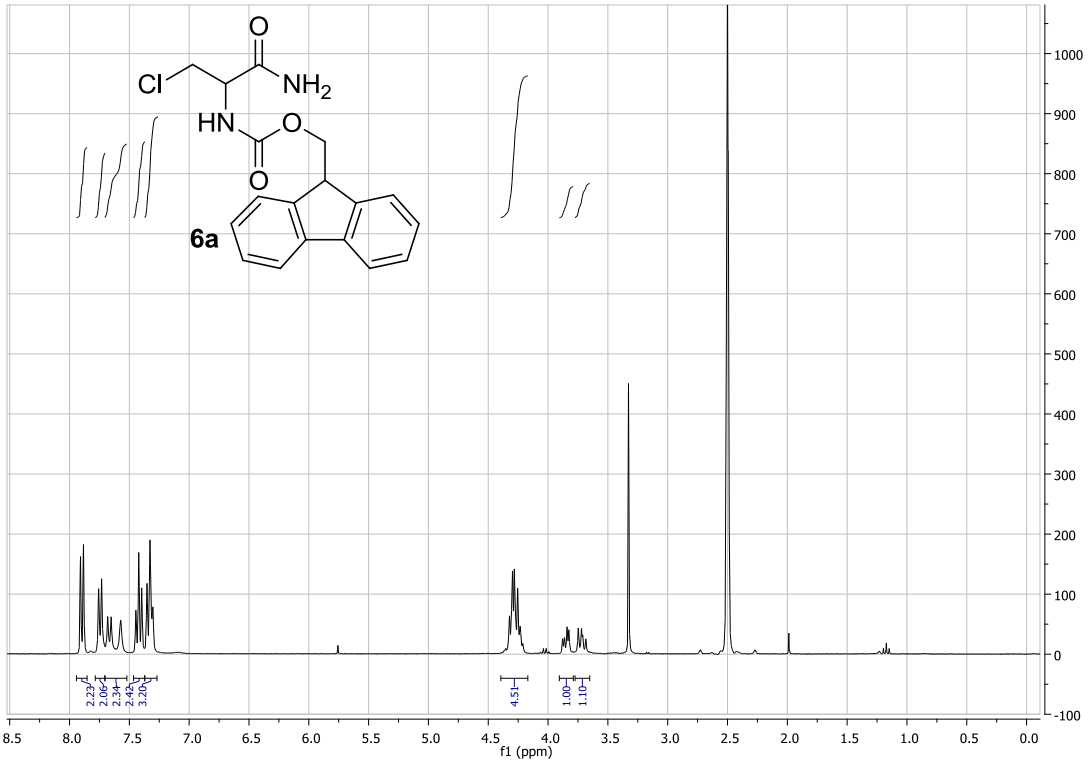
^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of *tert*-butyl (1-amino-3-iodo-1-oxopropan-2-yl)carbamate 4c:



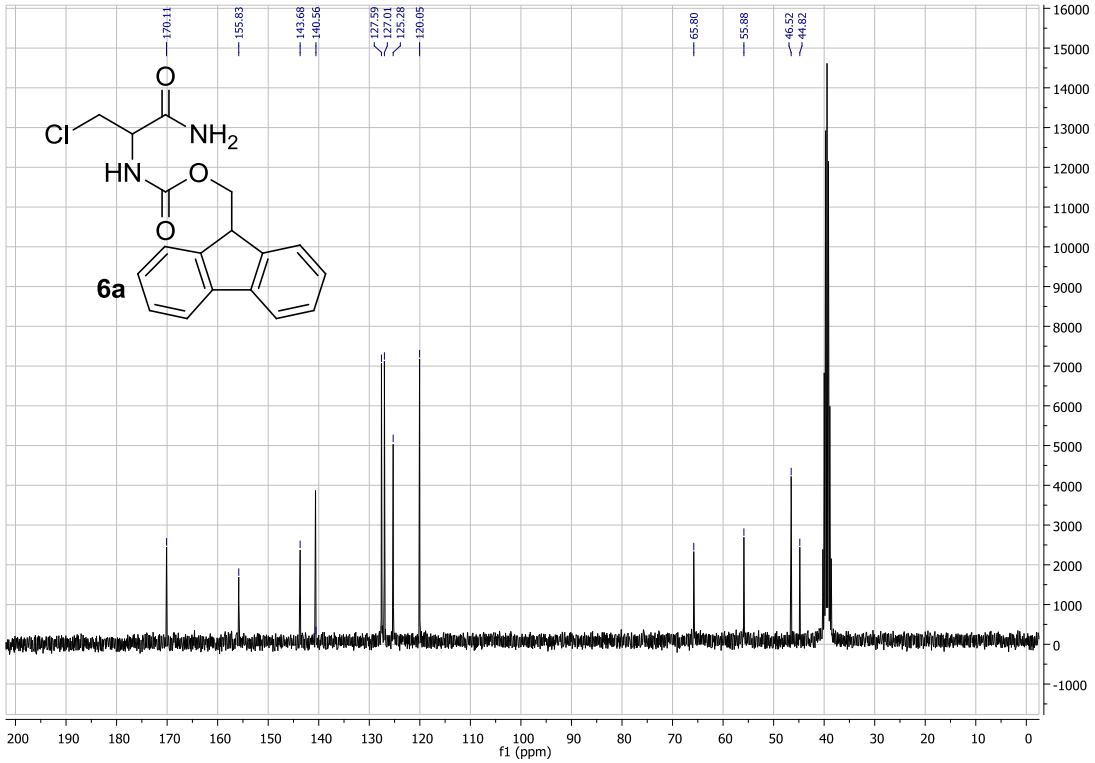
^{13}C-NMR ($CDCl_3$, 75.5 MHz) spectrum of *tert*-butyl (1-amino-3-iodo-1-oxopropan-2-yl)carbamate 4c:



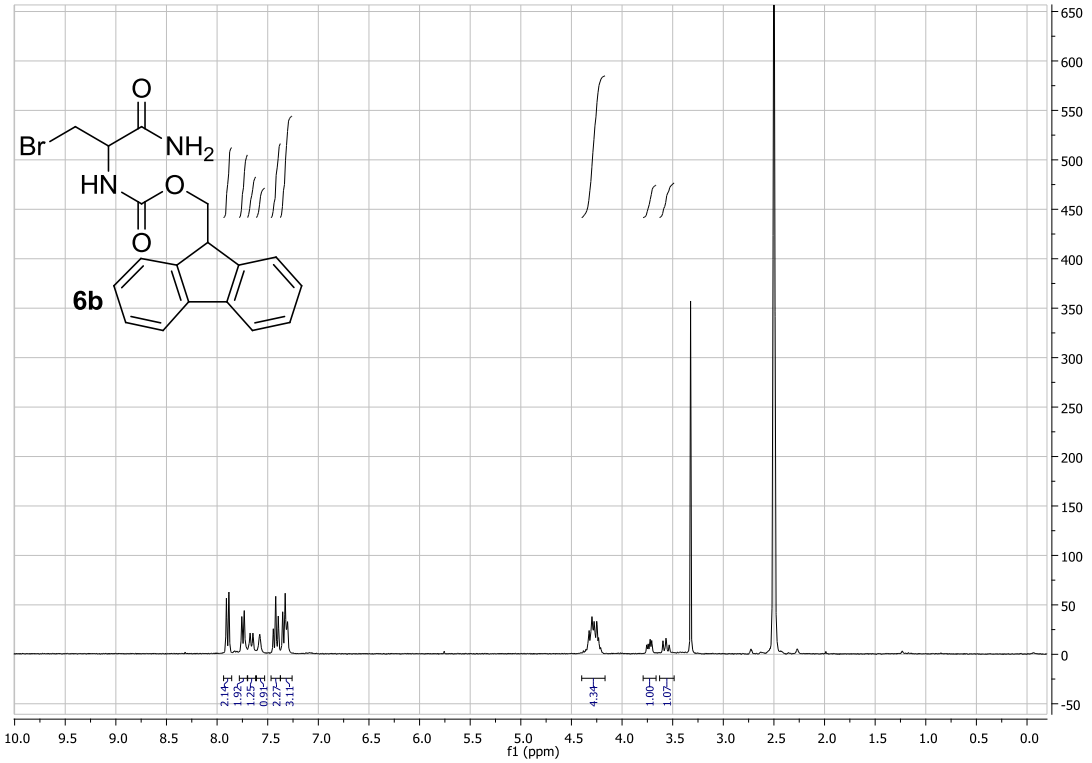
^{1}H-NMR (DMSO$_{d6}$, 300 MHz) spectrum of (*9H*-fluoren-9-yl)methyl(1-amino-3-chloro-1-oxopropan-2-yl) carbamate 6a:



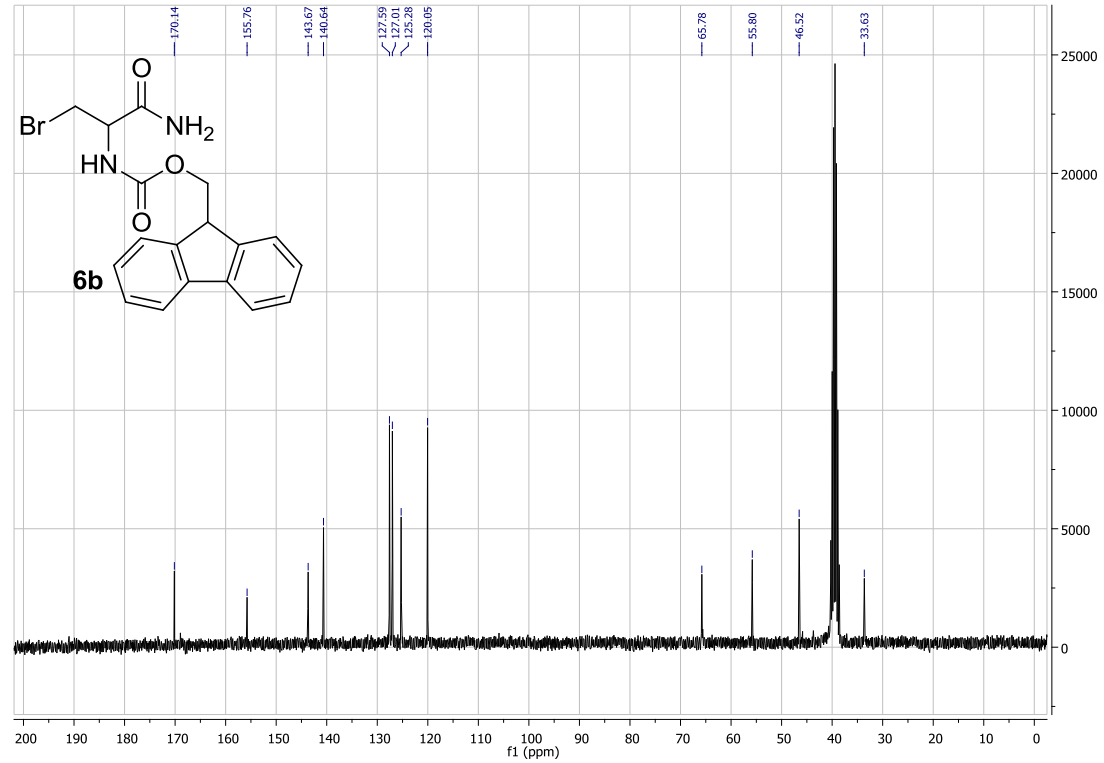
^{13}C-NMR (DMSO$_{d6}$, 75.5 MHz) spectrum of (*9H*-fluoren-9-yl)methyl(1-amino-3-chloro-1-oxopropan-2-yl) carbamate 6a:



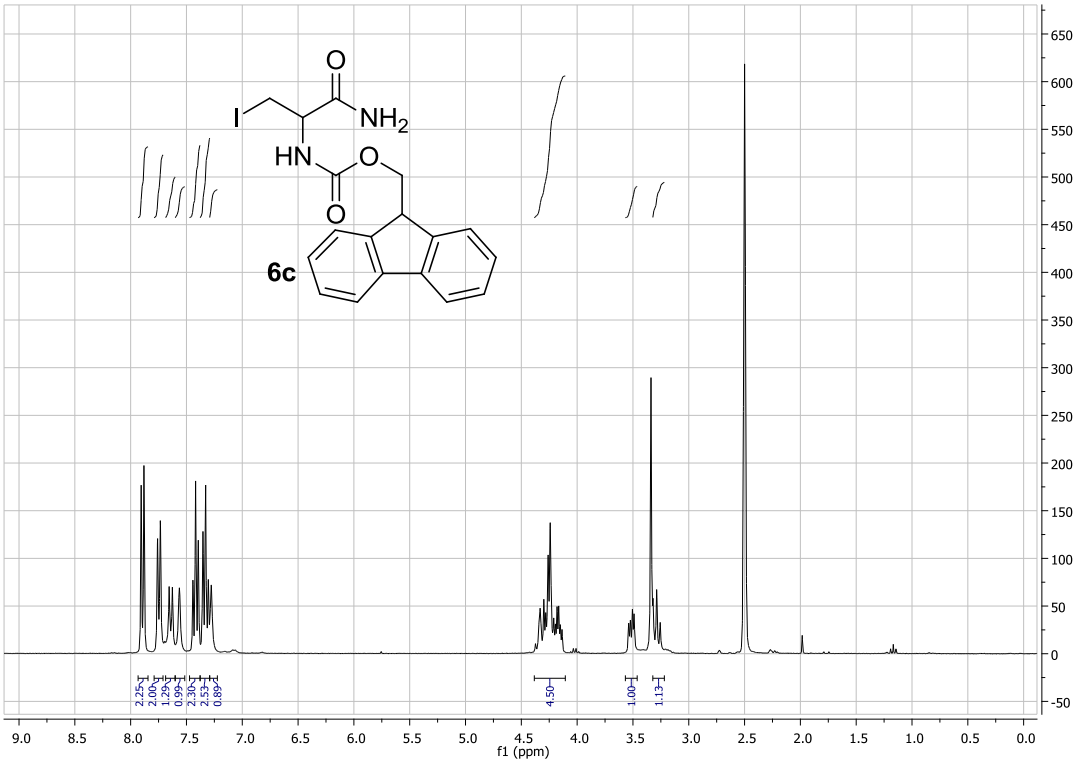
^{1}H-NMR (DMSO$_{d6}$, 300 MHz) spectrum of (*9H*-fluoren-9-yl)methyl(1-amino-3-bromo-1-oxopropan-2-yl) carbamate 6b:



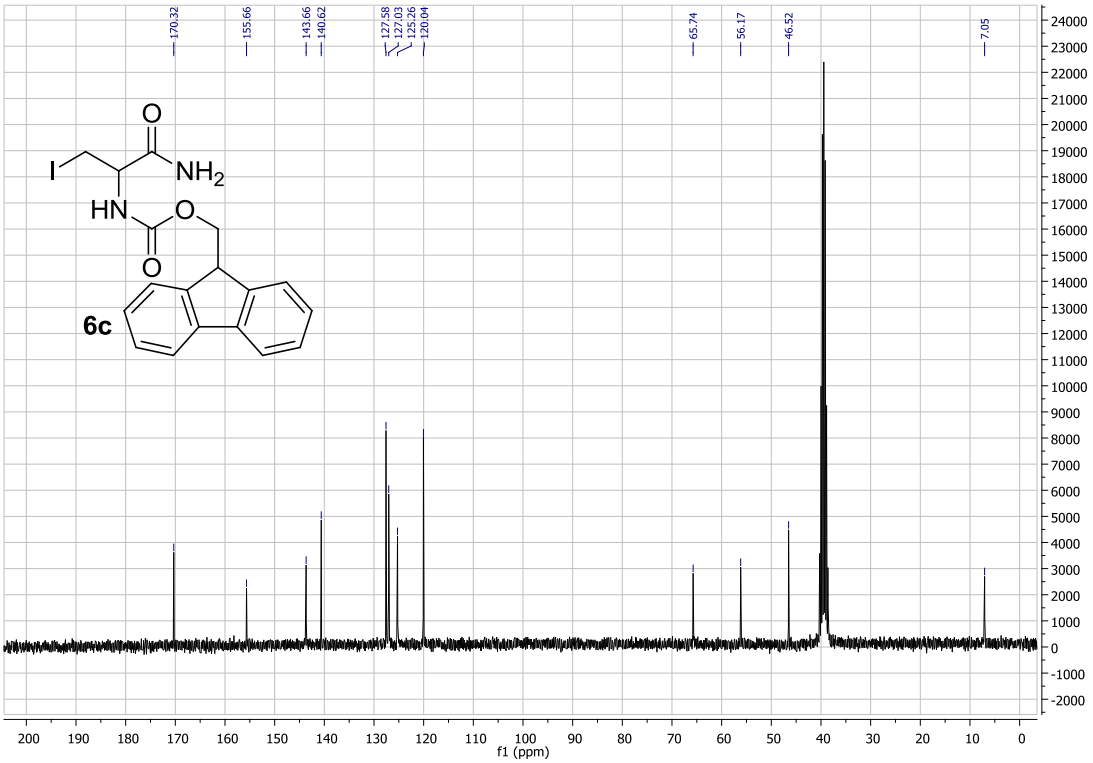
^{13}C-NMR (DMSO$_{d6}$, 75.5 MHz) spectrum of (*9H*-fluoren-9-yl)methyl(1-amino-3-bromo-1-oxopropan-2-yl) carbamate 6b:



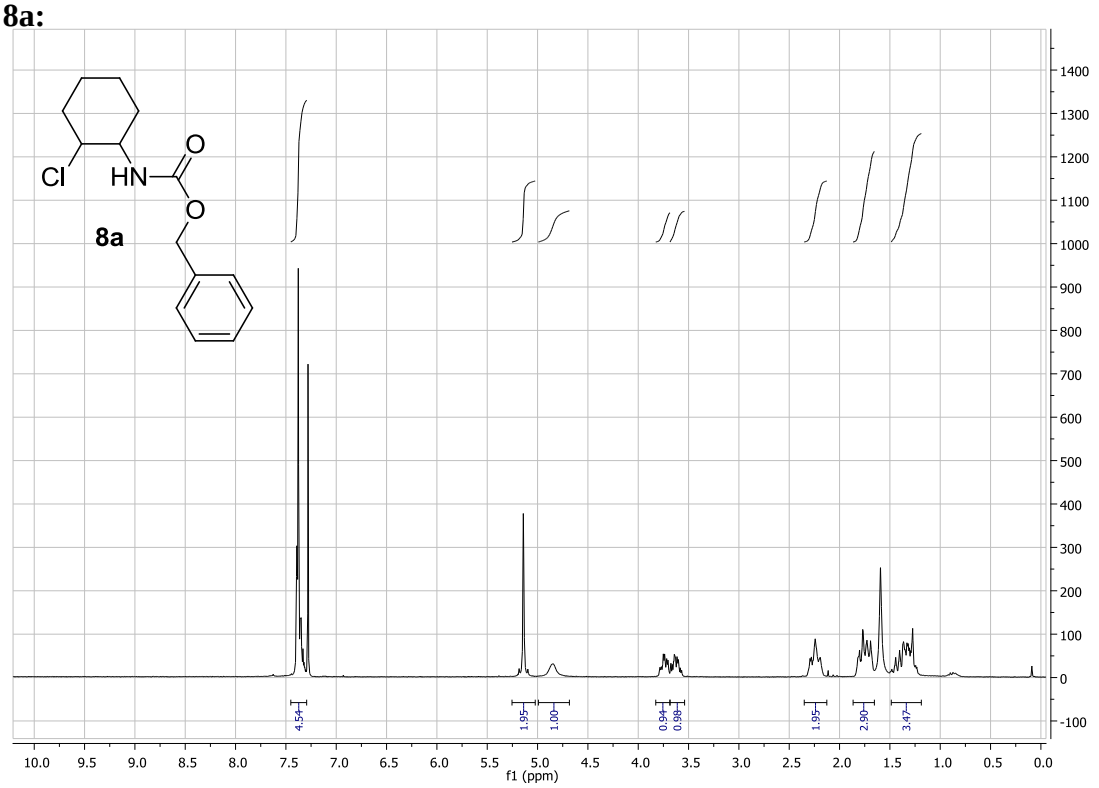
^{1}H-NMR (DMSO$_{d6}$, 300 MHz) spectrum of (*9H*-fluoren-9-yl)methyl(1-amino-3-iodo-1-oxopropan-2-yl) carbamate 6c:



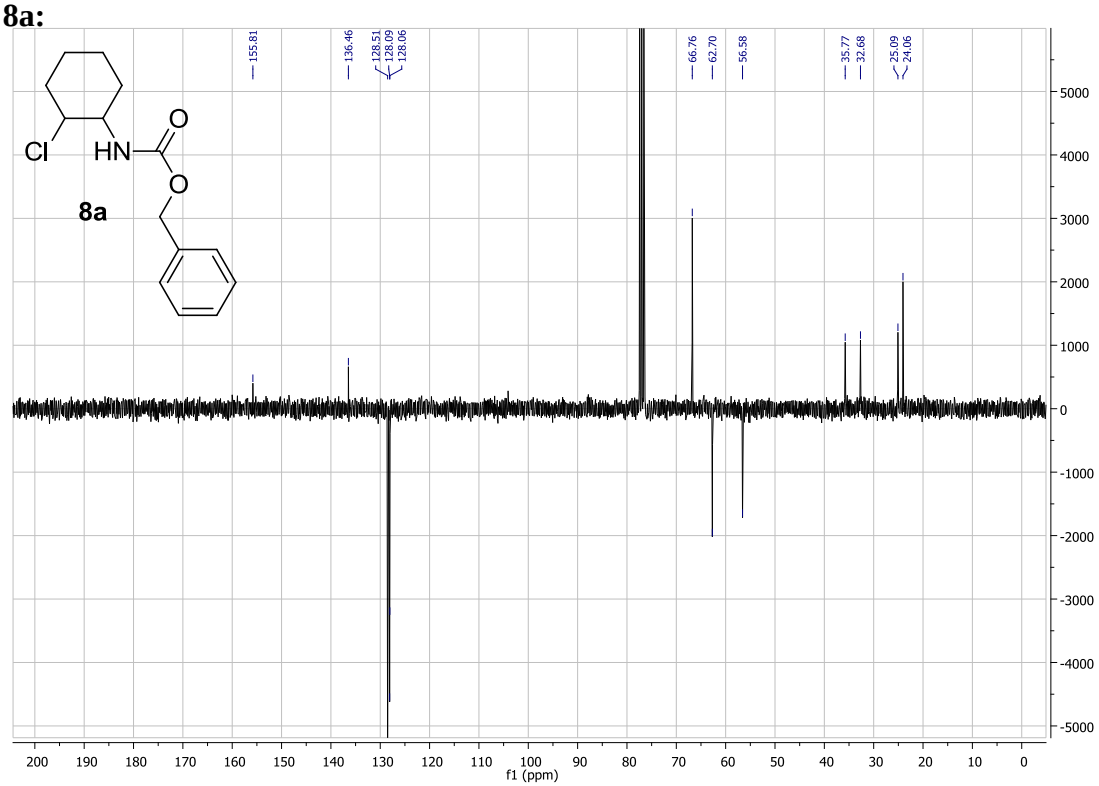
^{13}C-NMR (DMSO$_{d6}$, 75.5 MHz) spectrum of (*9H*-fluoren-9-yl)methyl(1-amino-3-iodo-1-oxopropan-2-yl) carbamate 6c:



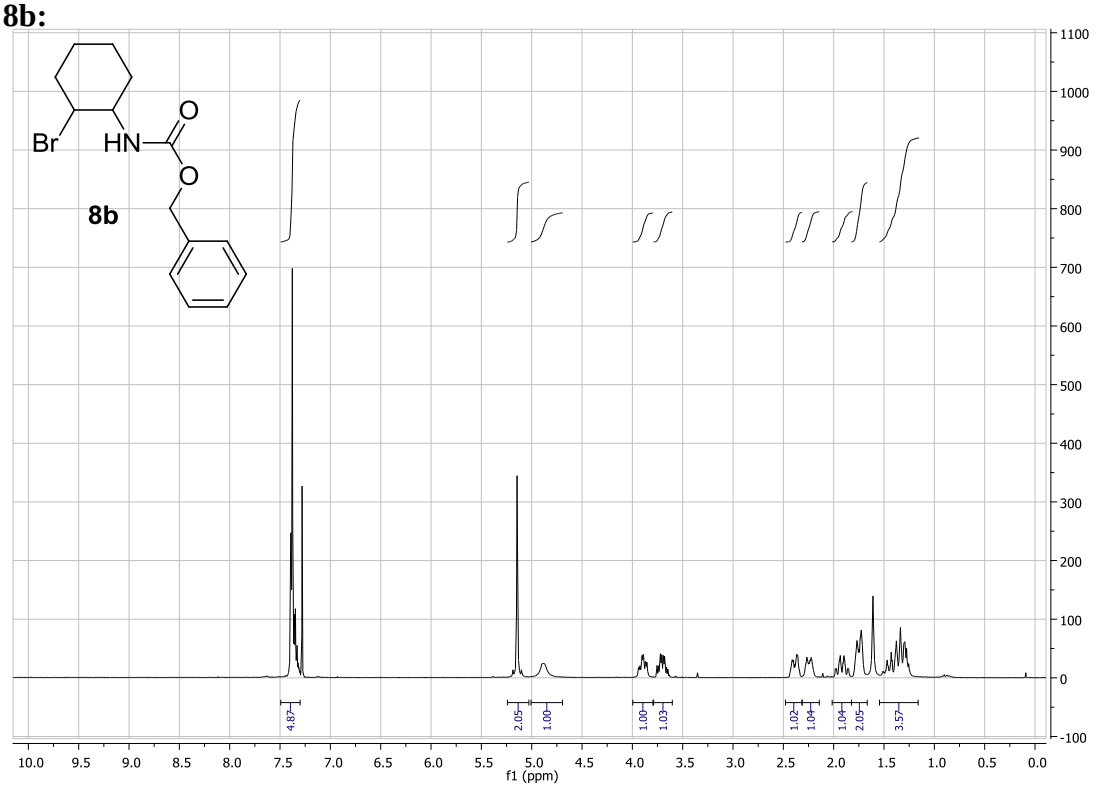
^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of benzyl (2-chlorocyclohexyl)carbamate 8a:



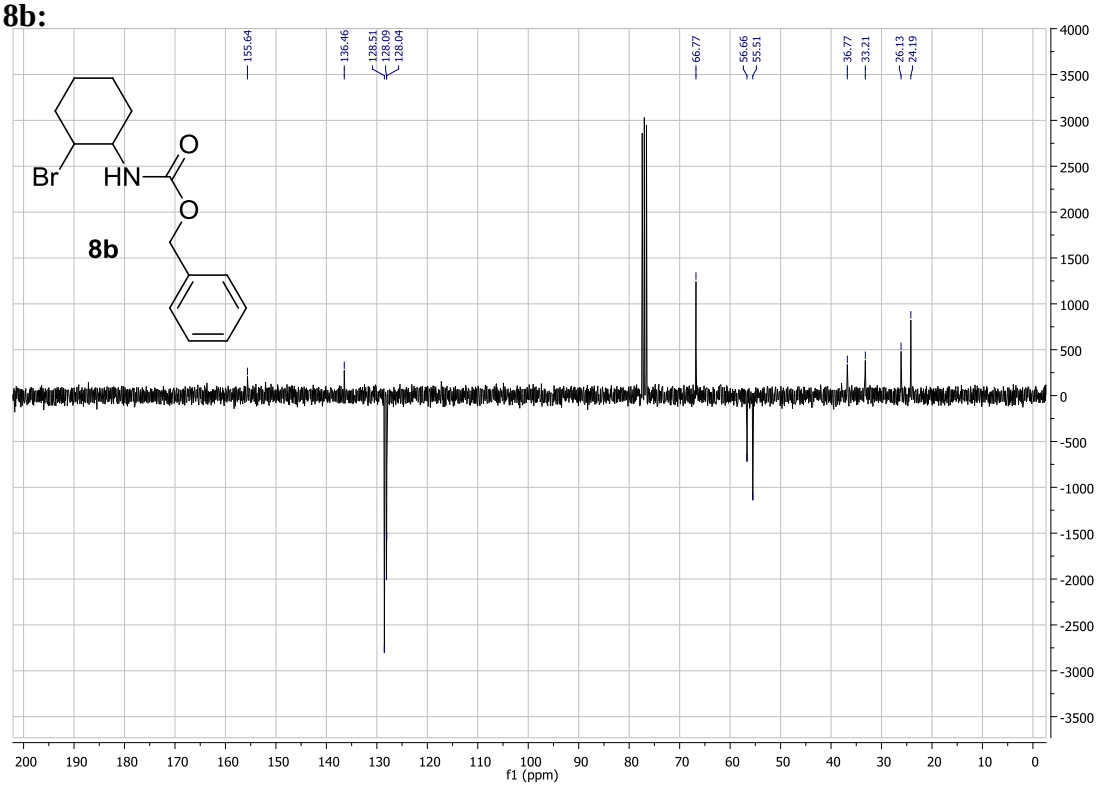
^{13}C-NMR ($CDCl_3$, 75.5 MHz) spectrum of benzyl (2-chlorocyclohexyl)carbamate 8a:



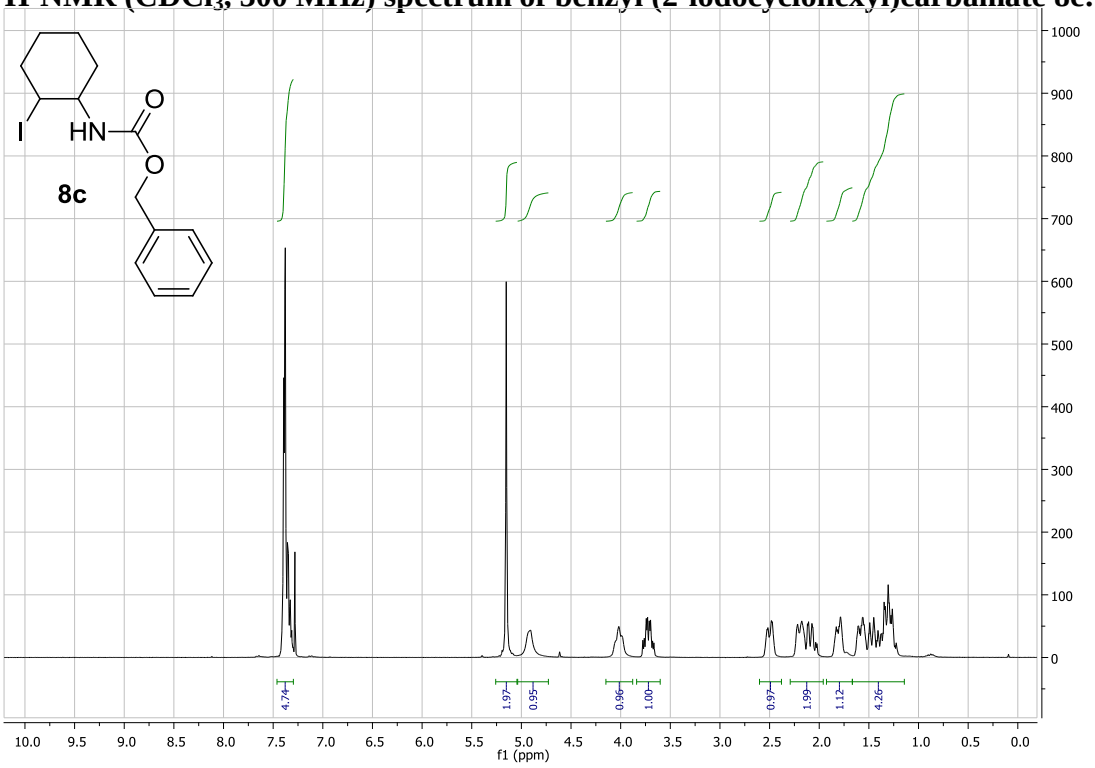
^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of benzyl (2-bromocyclohexyl)carbamate 8b:



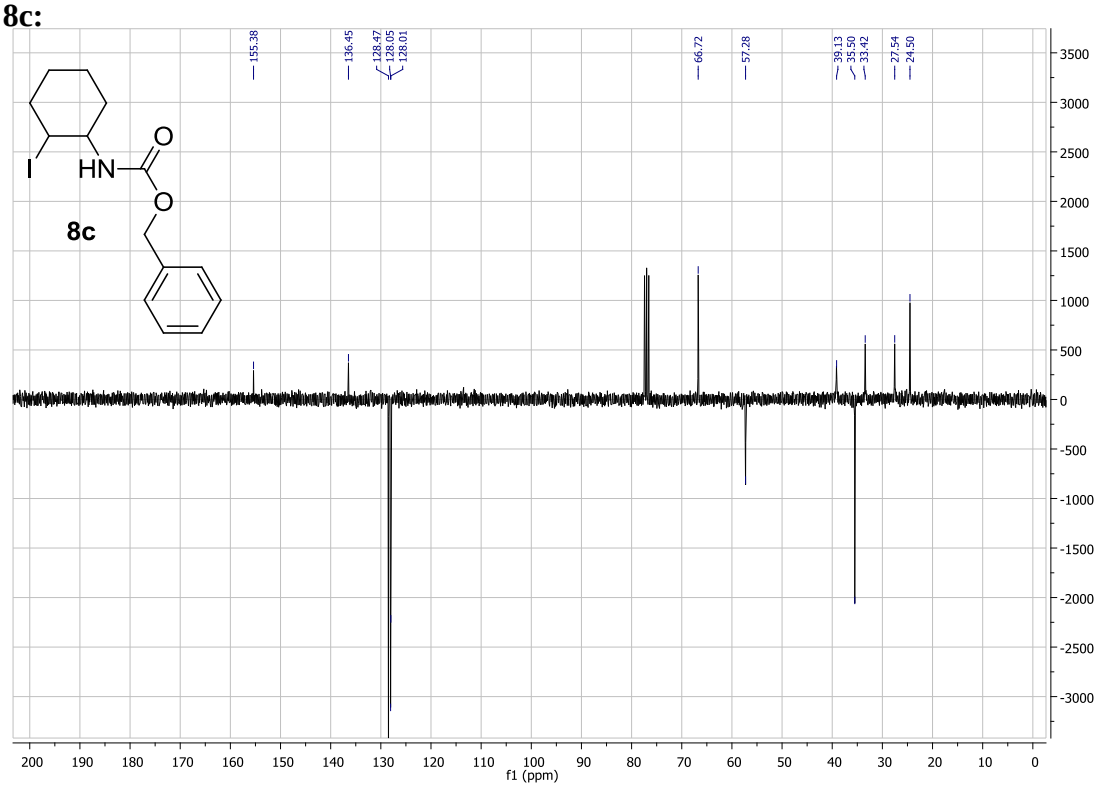
^{13}C-NMR ($CDCl_3$, 75.5 MHz) spectrum of benzyl (2-bromocyclohexyl)carbamate 8b:



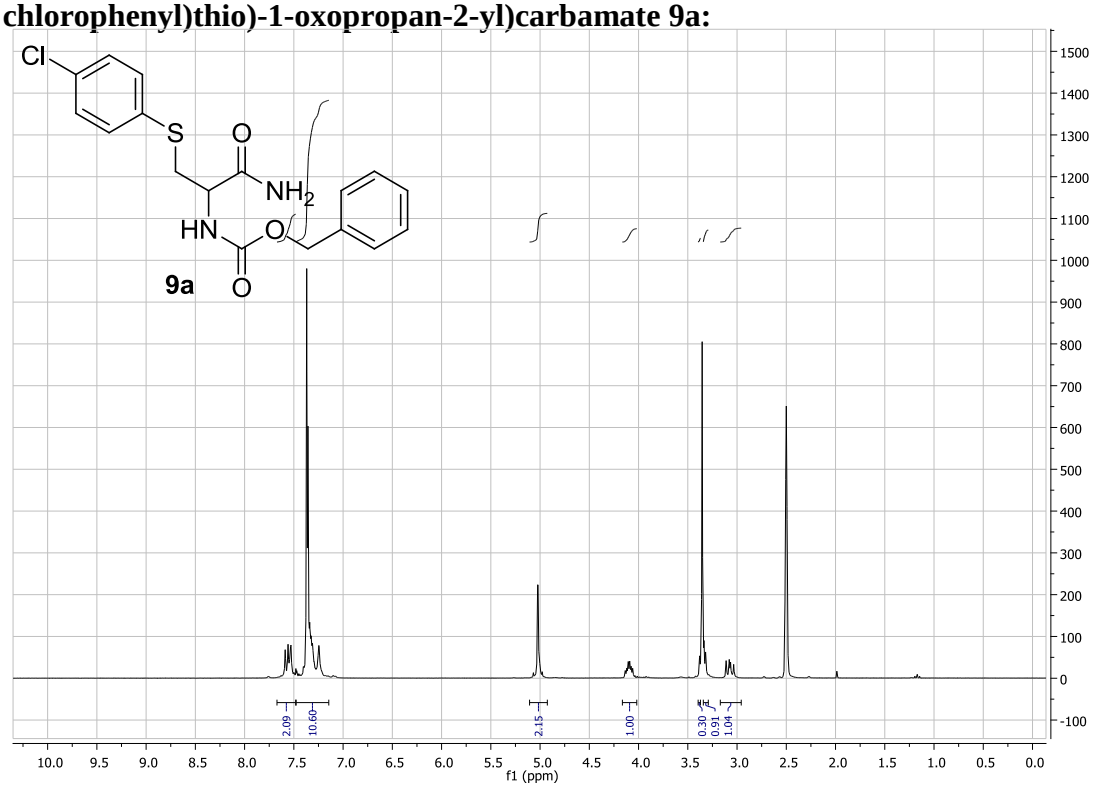
^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of benzyl (2-iodocyclohexyl)carbamate 8c:



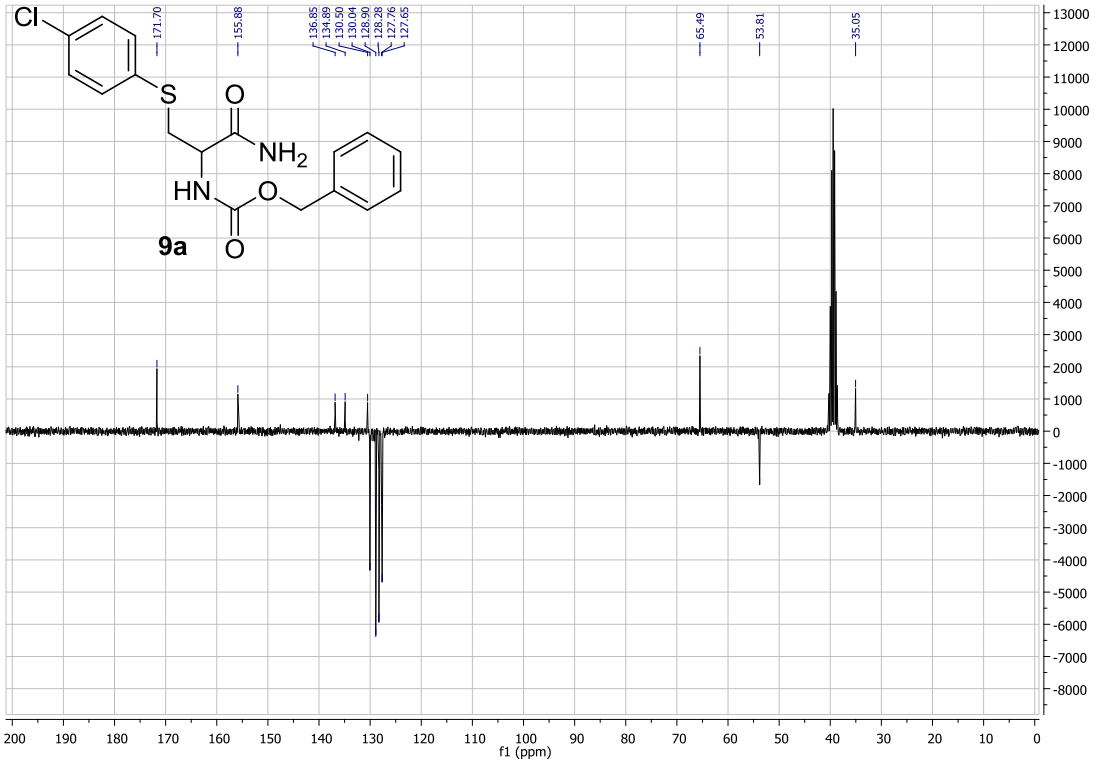
^{13}C-NMR ($CDCl_3$, 75.5 MHz) spectrum of benzyl (2-iodocyclohexyl)carbamate 8c:



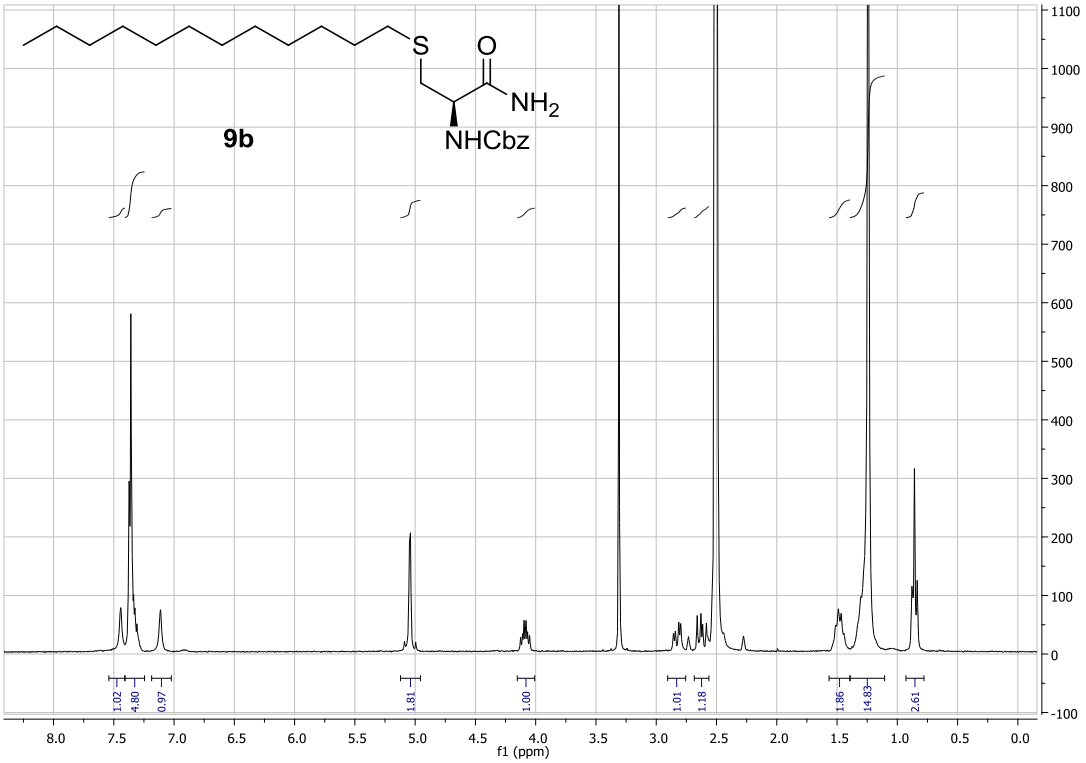
^{1}H-NMR (DMSO$_{d6}$, 300 MHz) spectrum of benzyl (1-amino-3-((4-chlorophenyl)thio)-1-oxopropan-2-yl)carbamate 9a:



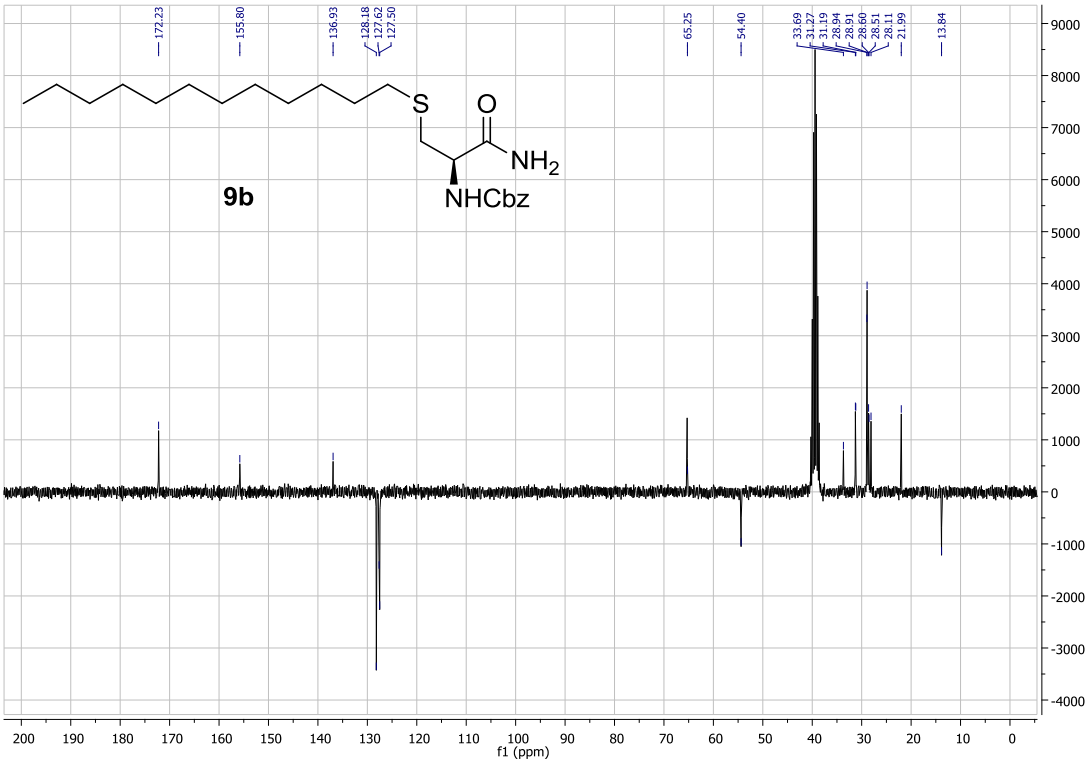
^{13}C-NMR (DMSO$_{d6}$, 75.5 MHz) spectrum of benzyl (1-amino-3-((4-chlorophenyl)thio)-1-oxopropan-2-yl)carbamate 9a:



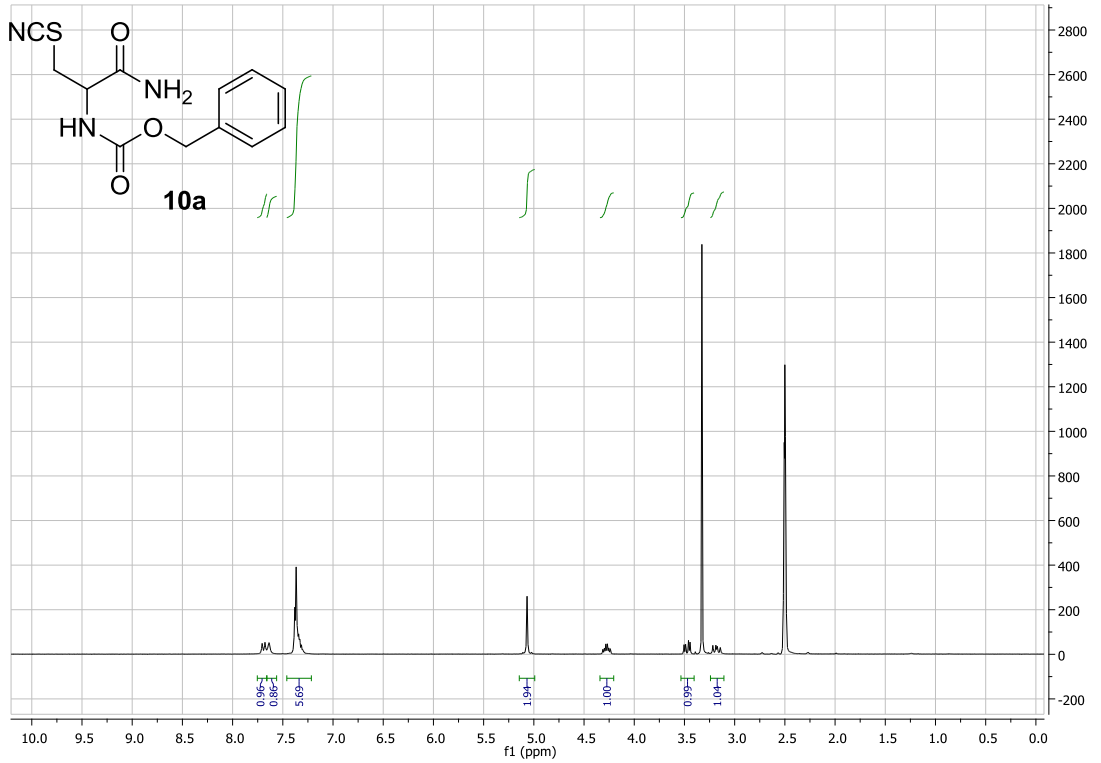
^{1}H-NMR (DMSO$_{d6}$, 300 MHz) spectrum of (*R*)-benzyl (1-amino-3-(dodecylthio)-1-oxopropan-2-yl)carbamate 9b:



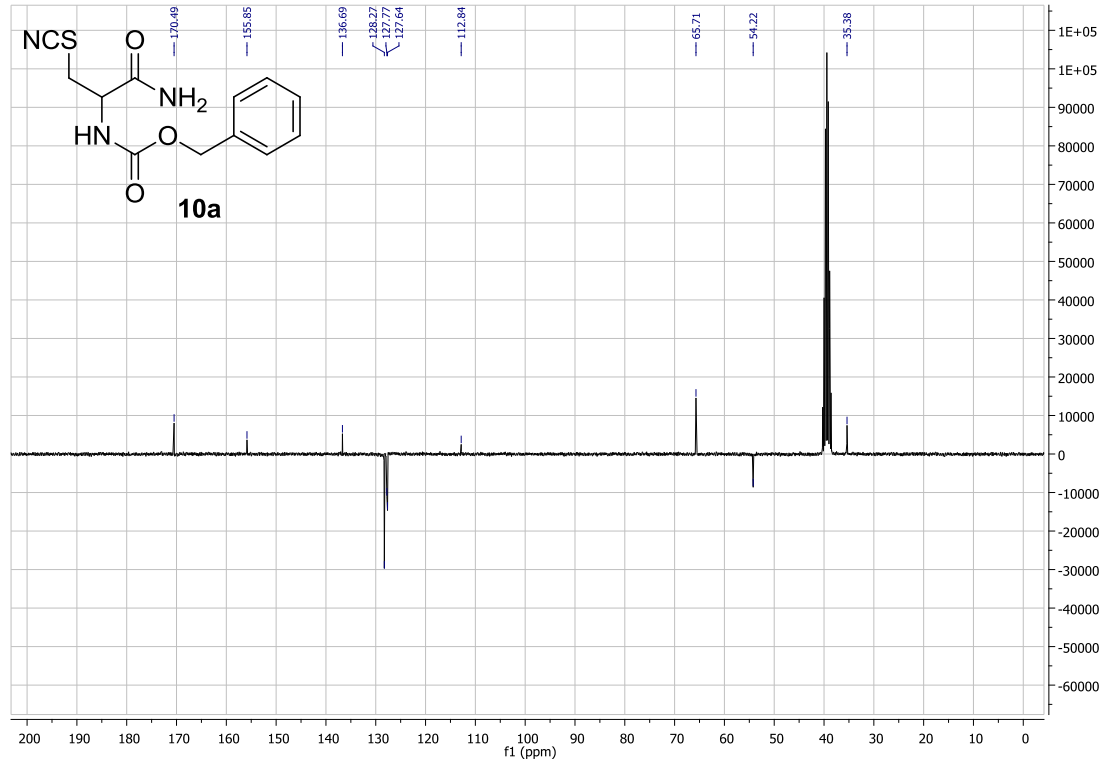
^{13}C-NMR (DMSO$_{d6}$, 75.5 MHz) spectrum of (*R*)-benzyl (1-amino-3-(dodecylthio)-1-oxopropan-2 yl)carbamate 9b:



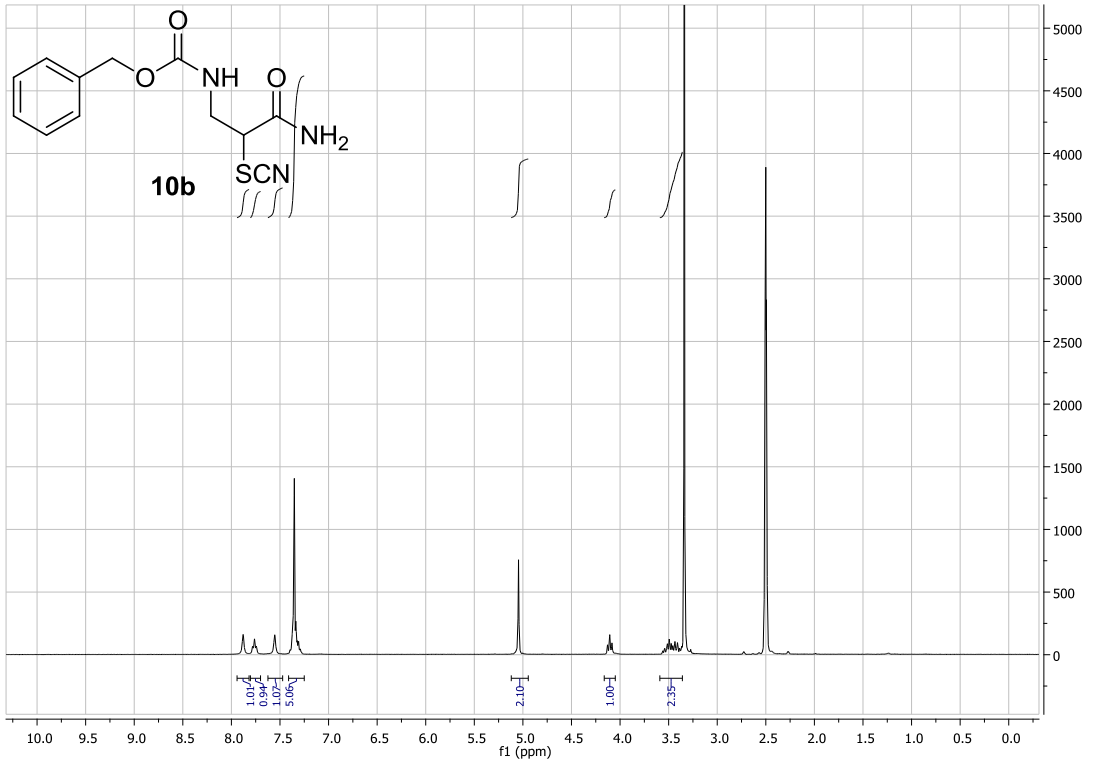
^{1}H-NMR (DMSO$_{d6}$, 300 MHz) spectrum of benzyl (1-amino-3-isothiocyanato-1-oxopropan-2-yl)carbamate 10a:



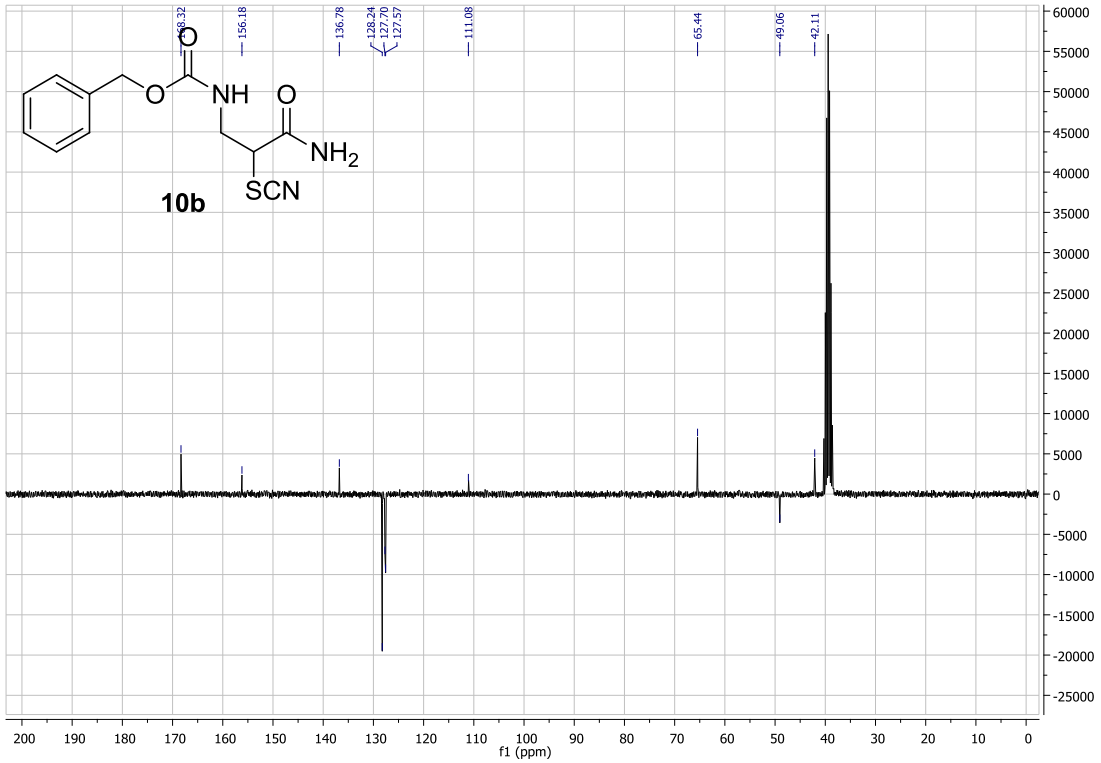
^{13}C-NMR (DMSO$_{d6}$, 75.5 MHz) spectrum of benzyl (1-amino-3-isothiocyanato-1-oxopropan-2-yl)carbamate 10a:



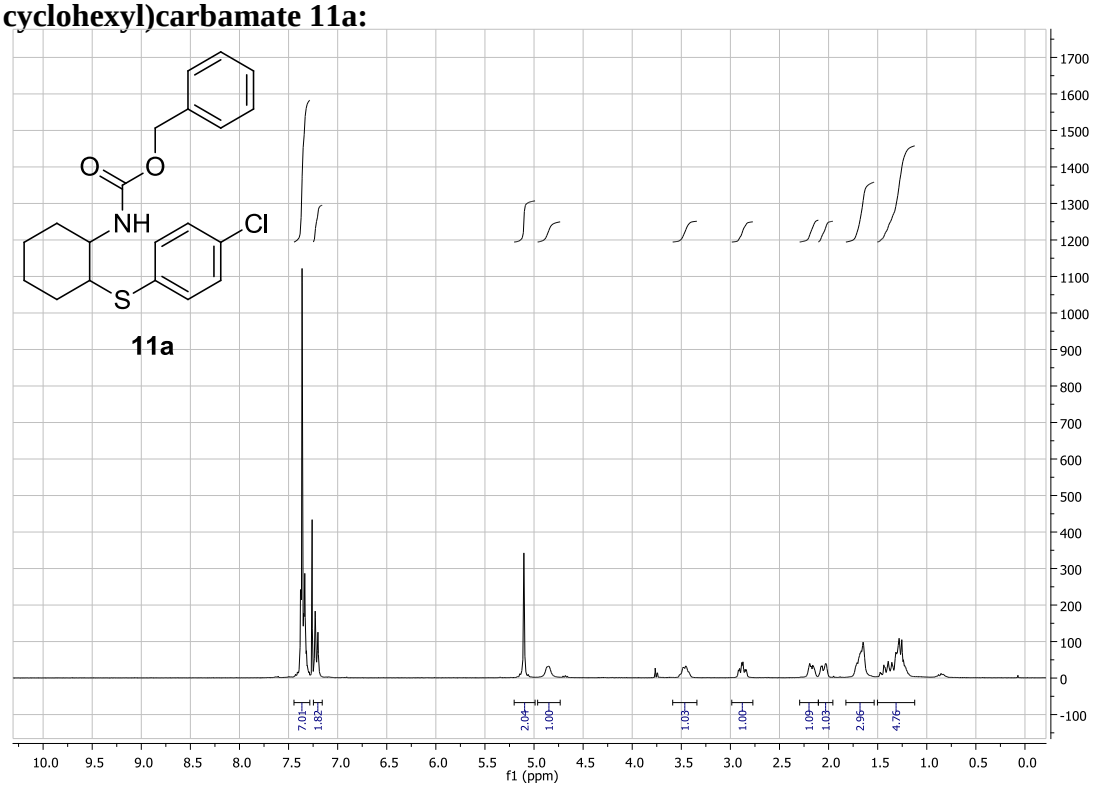
^{1}H-NMR (DMSO$_{d6}$, 300 MHz) spectrum of benzyl (3-amino-3-oxo-2-thiocyanatopropyl)carbamate 10b:



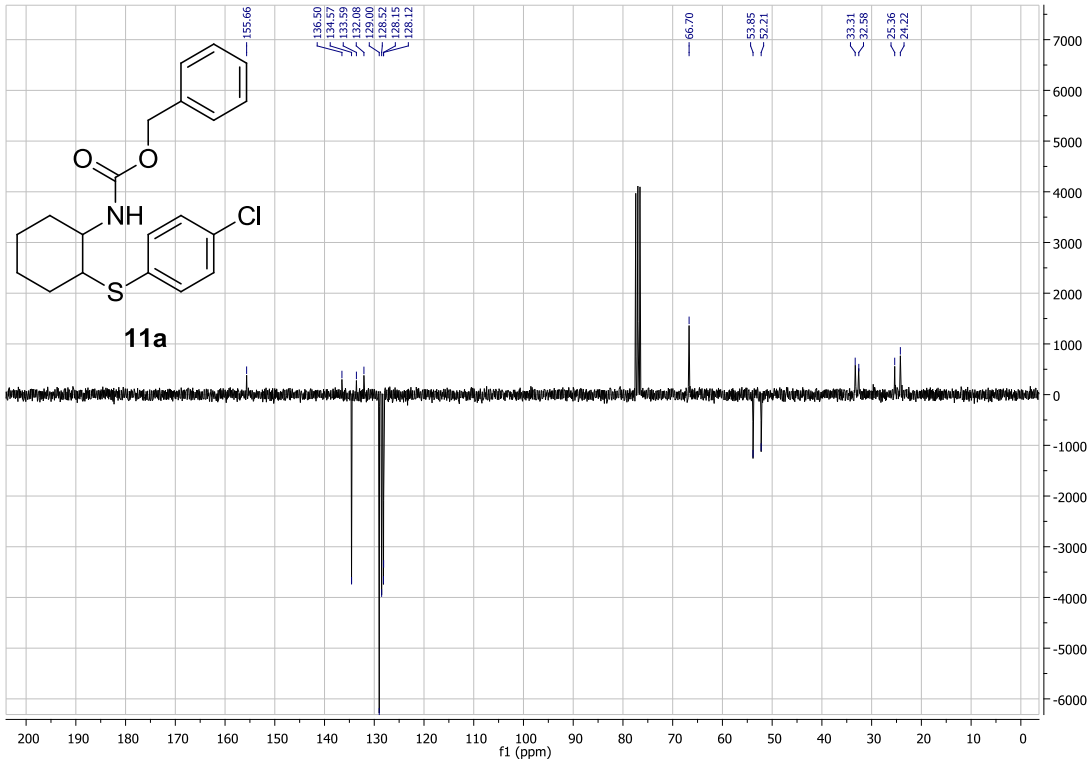
^{13}C-NMR (DMSO$_{d6}$, 75.5 MHz) spectrum of benzyl (3-amino-3-oxo-2-thiocyanatopropyl)carbamate 10b:



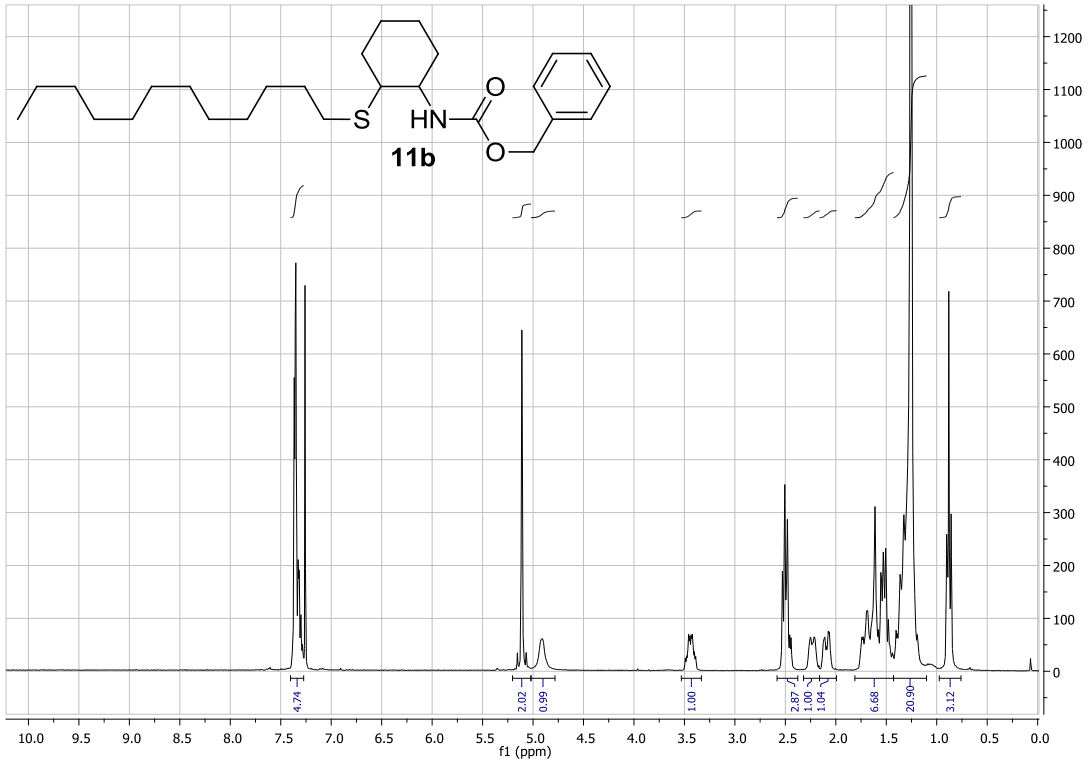
^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of benzyl (2-((4-chlorophenyl) thio) cyclohexyl)carbamate 11a:



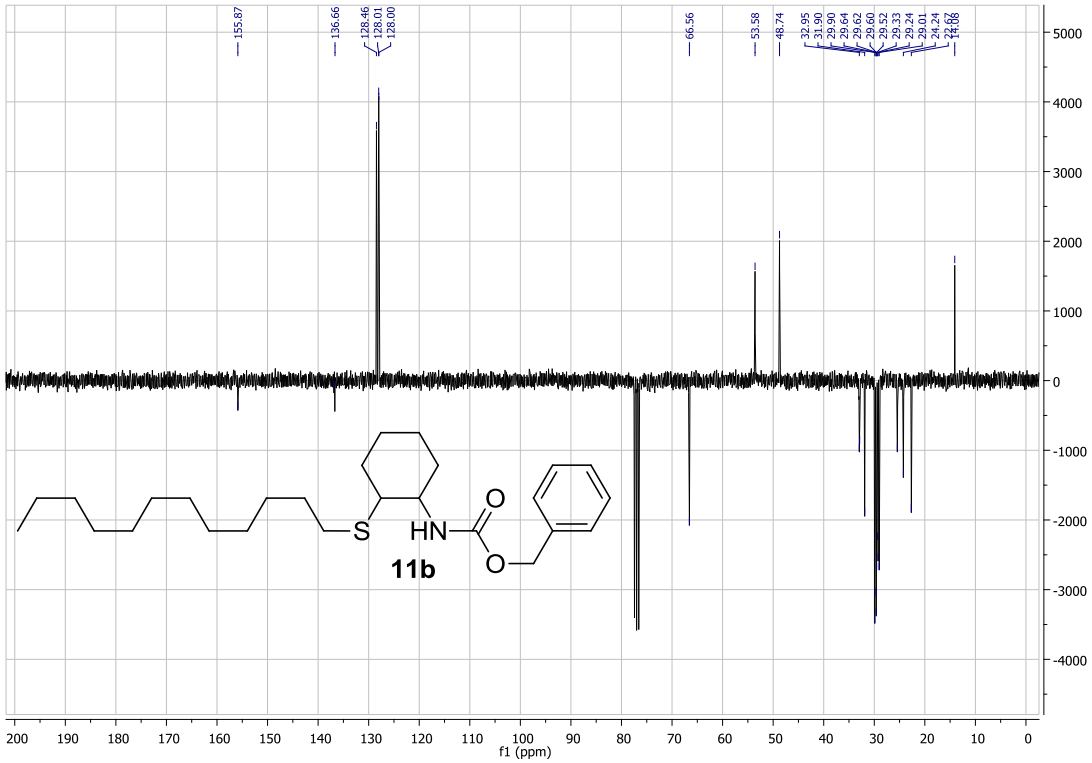
^{13}C-NMR ($CDCl_3$, 75.5 MHz) spectrum of benzyl (2-((4-chlorophenyl)thio)cyclohexyl)carbamate 11a:



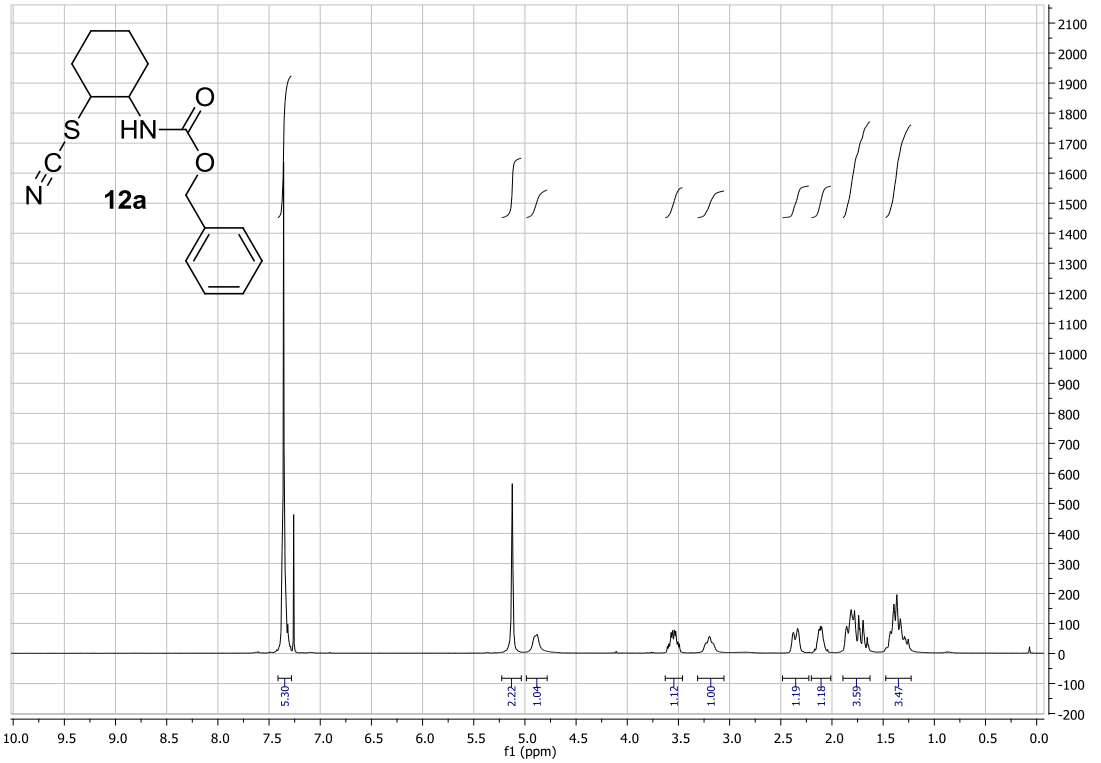
^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of benzyl (2-(dodecylthio)cyclohexyl)carbamate 11b:



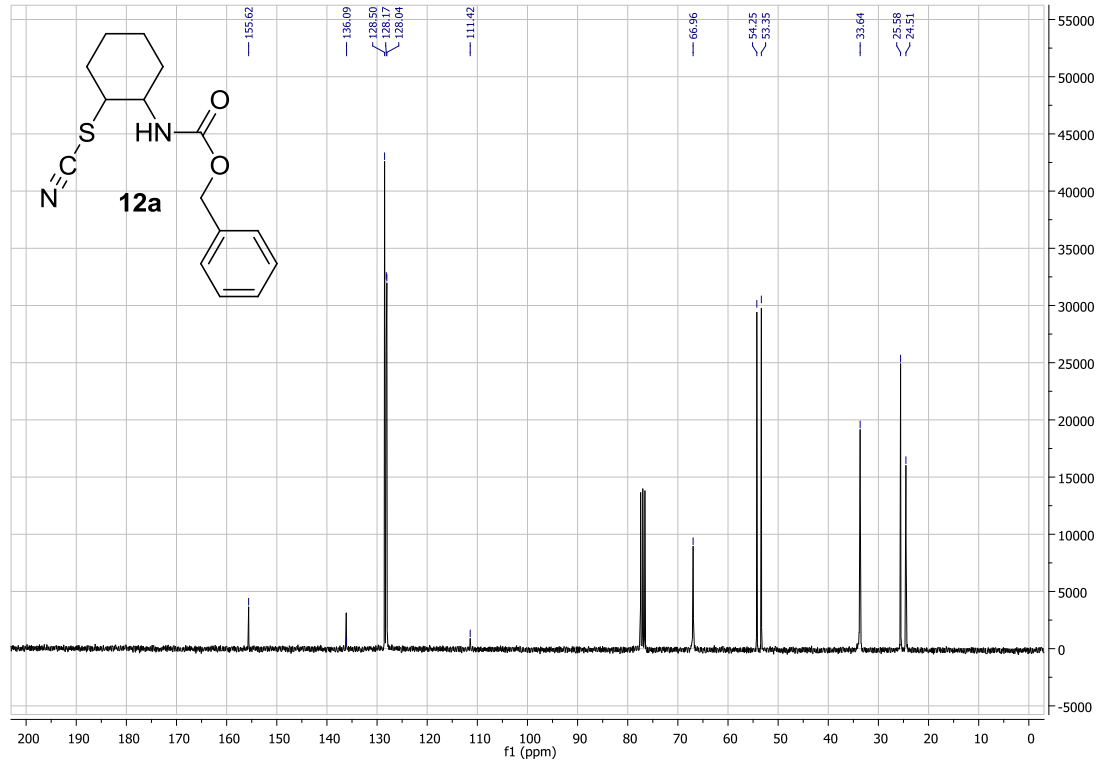
^{13}C-NMR ($CDCl_3$, 75.5 MHz) spectrum of benzyl (2-(dodecylthio)cyclohexyl)carbamate 11b:



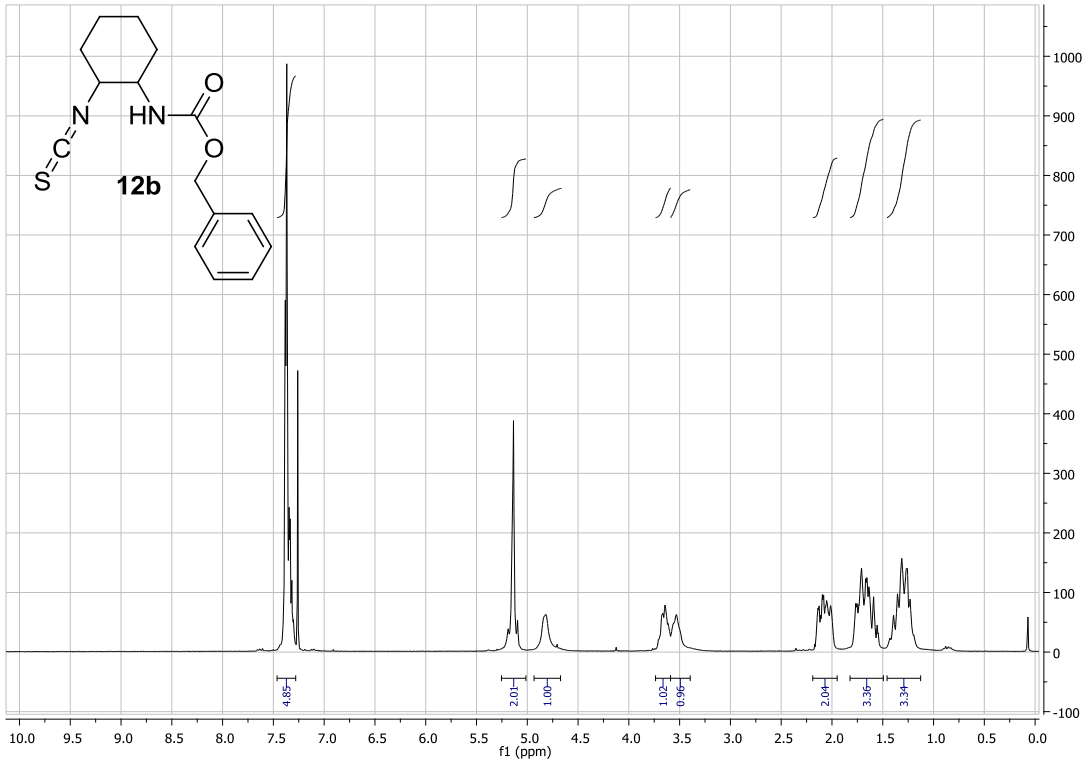
^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of benzyl (2-thiocyanatocyclohexyl) carbamate 12a:



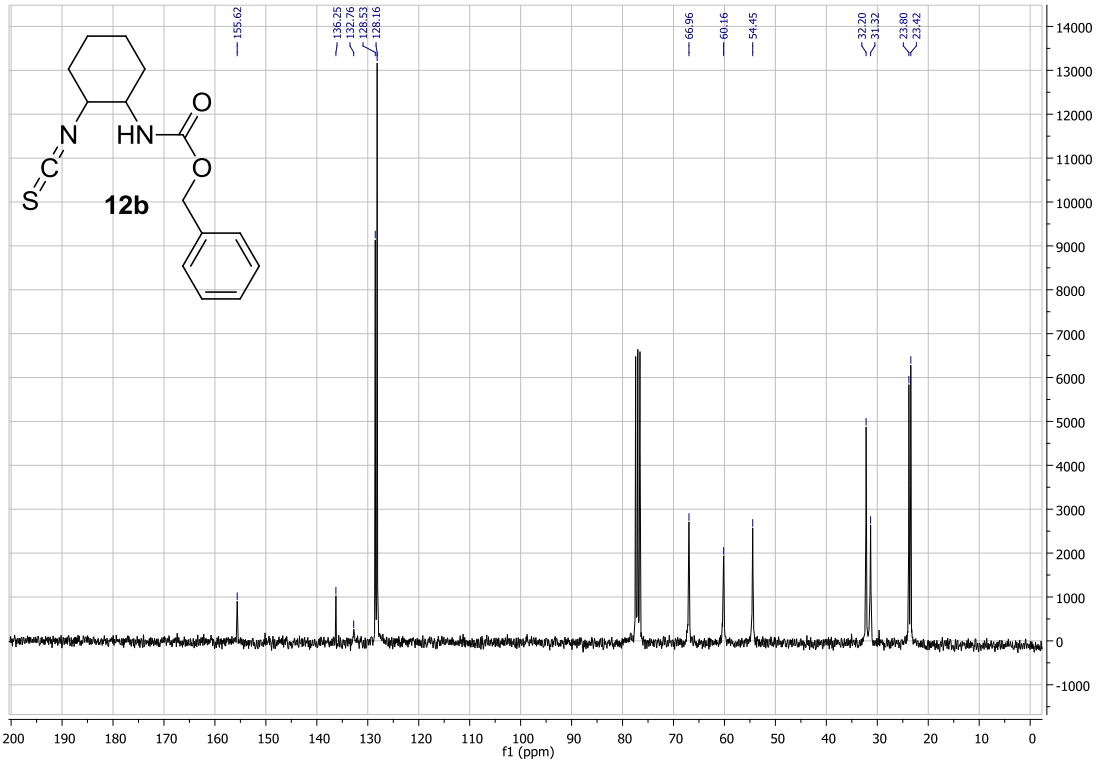
^{13}C-NMR ($CDCl_3$, 75.5 MHz) spectrum of benzyl (2-thiocyanatocyclohexyl) carbamate 12a:



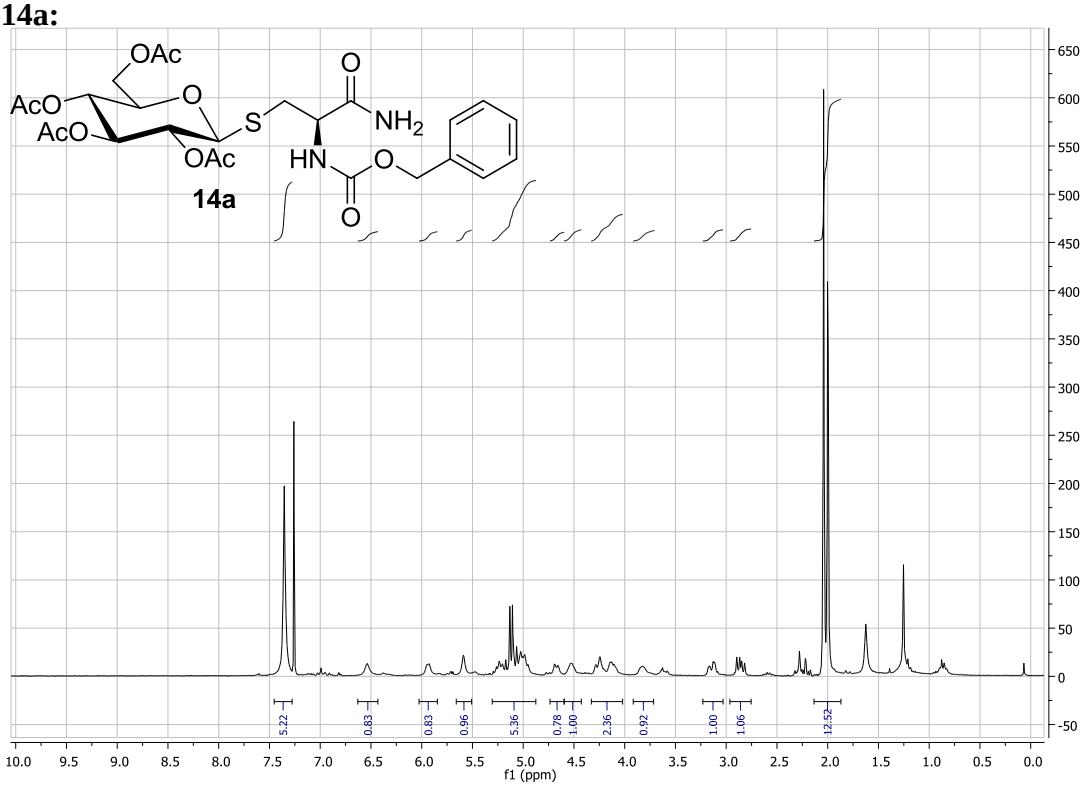
^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of benzyl (2-isothiocyanatocyclohexyl) carbamate 12b:



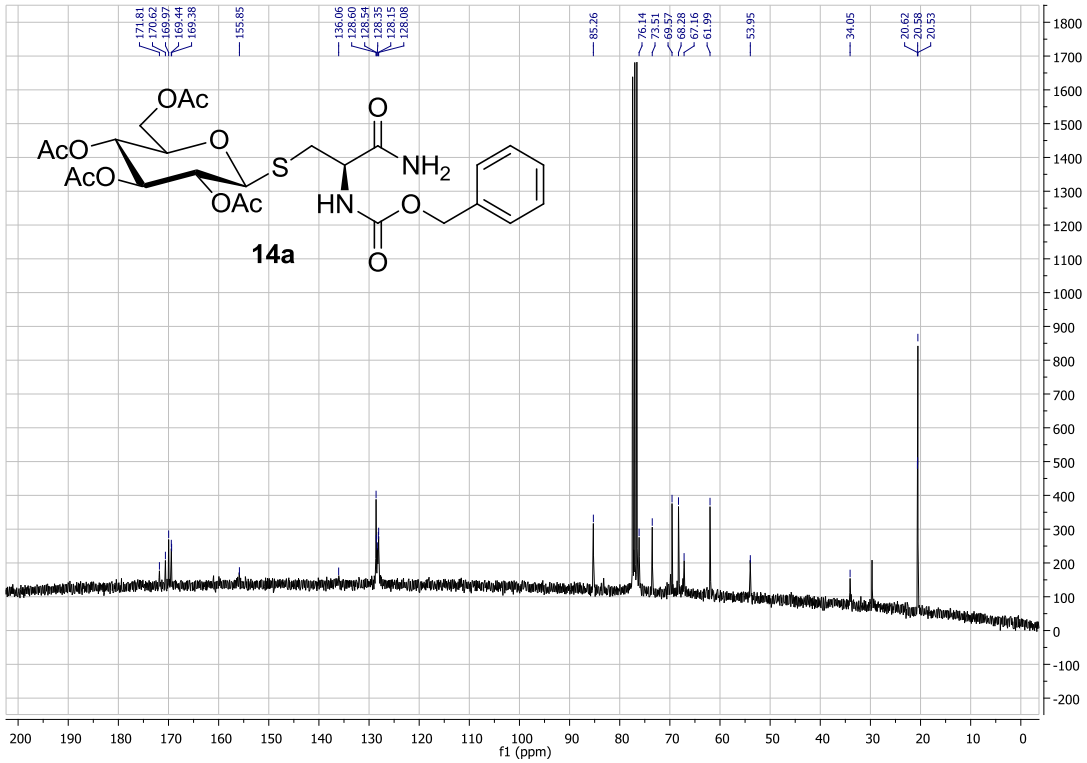
^{13}C-NMR ($CDCl_3$, 75.5 MHz) spectrum of benzyl (2-isothiocyanatocyclohexyl) carbamate 12b:



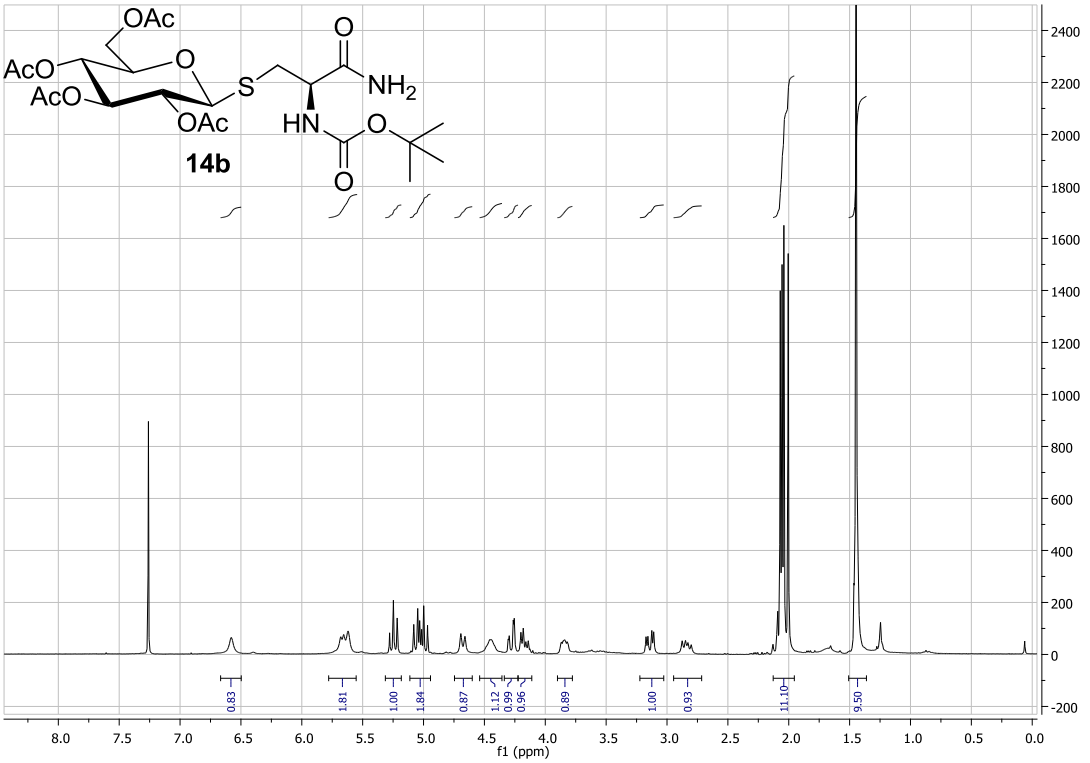
^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of amino – acid carbohydrate conjugate 14a:



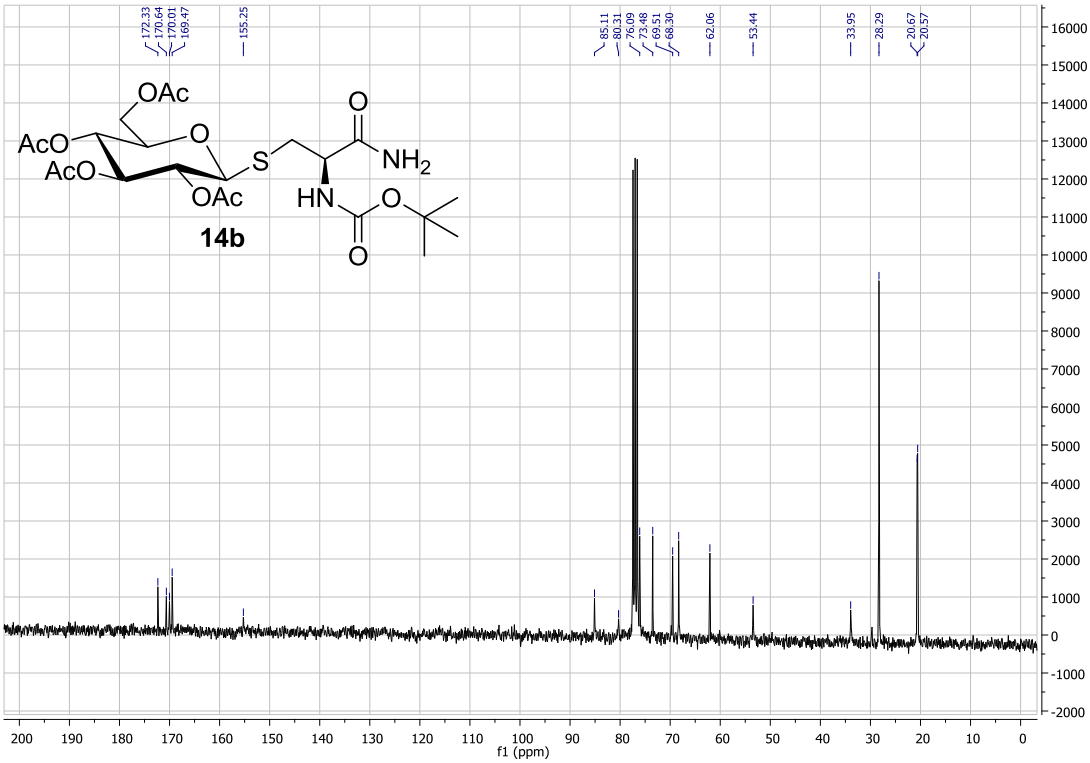
^{13}C-NMR ($CDCl_3$, 75.5 MHz) spectrum of of amino – acid carbohydrate conjugate 14a:



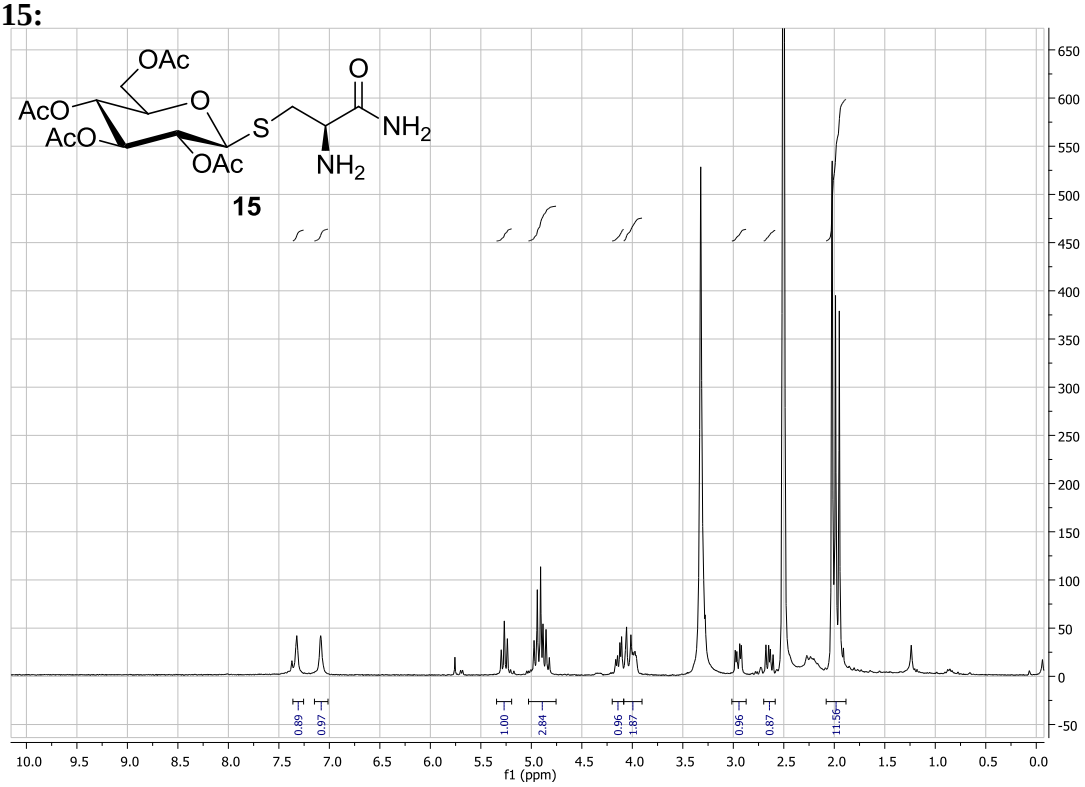
^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of amino – acid carbohydrate conjugate 14b:



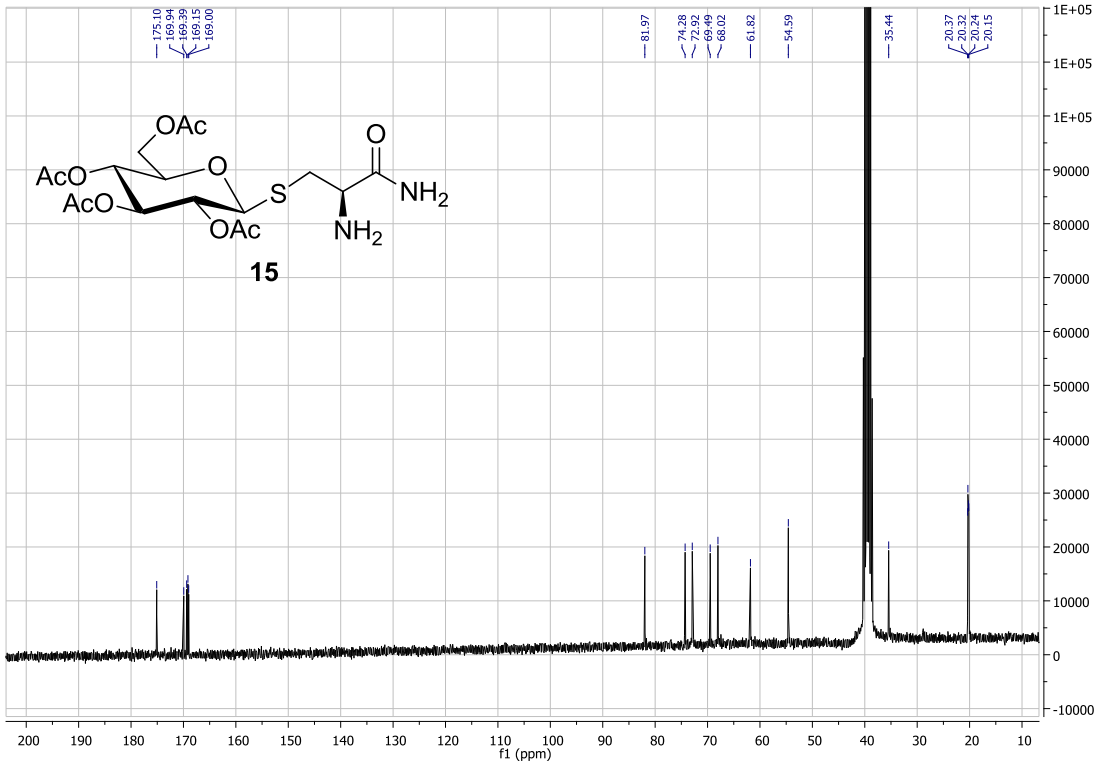
^{13}C-NMR ($CDCl_3$, 75.5 MHz) spectrum of of amino – acid carbohydrate conjugate 14b:



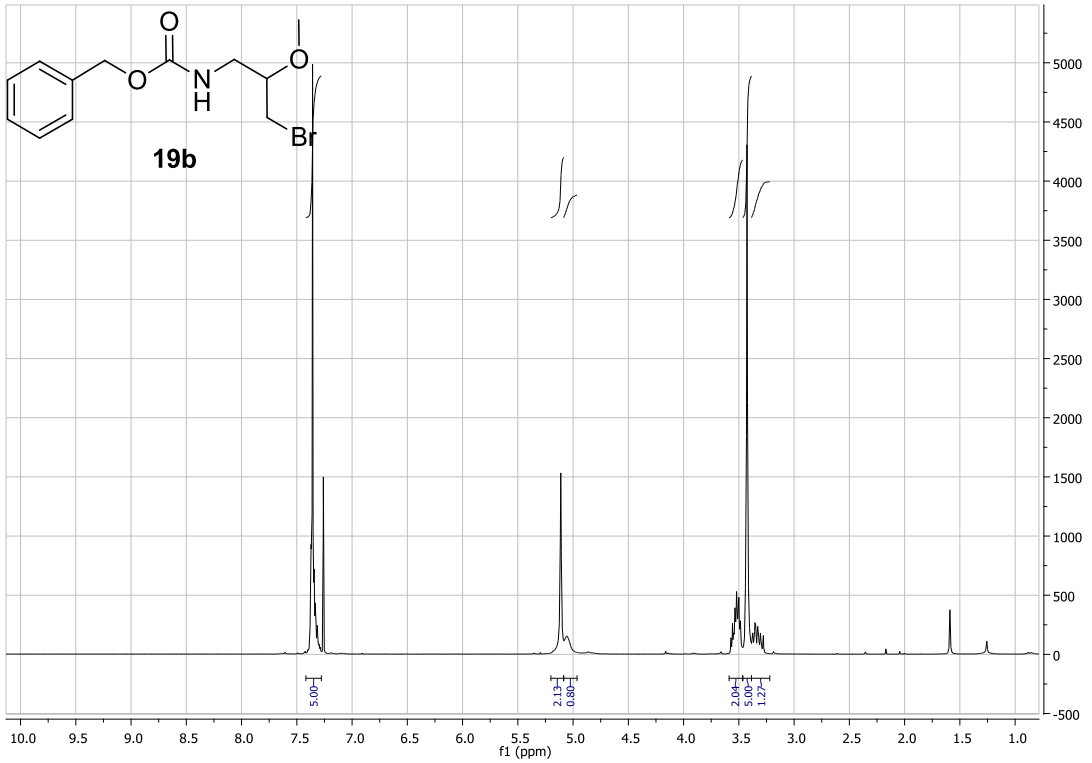
^{1}H-NMR (DMSO$_{d6}$, 300 MHz) spectrum of amino – acid carbohydrate conjugate 15:



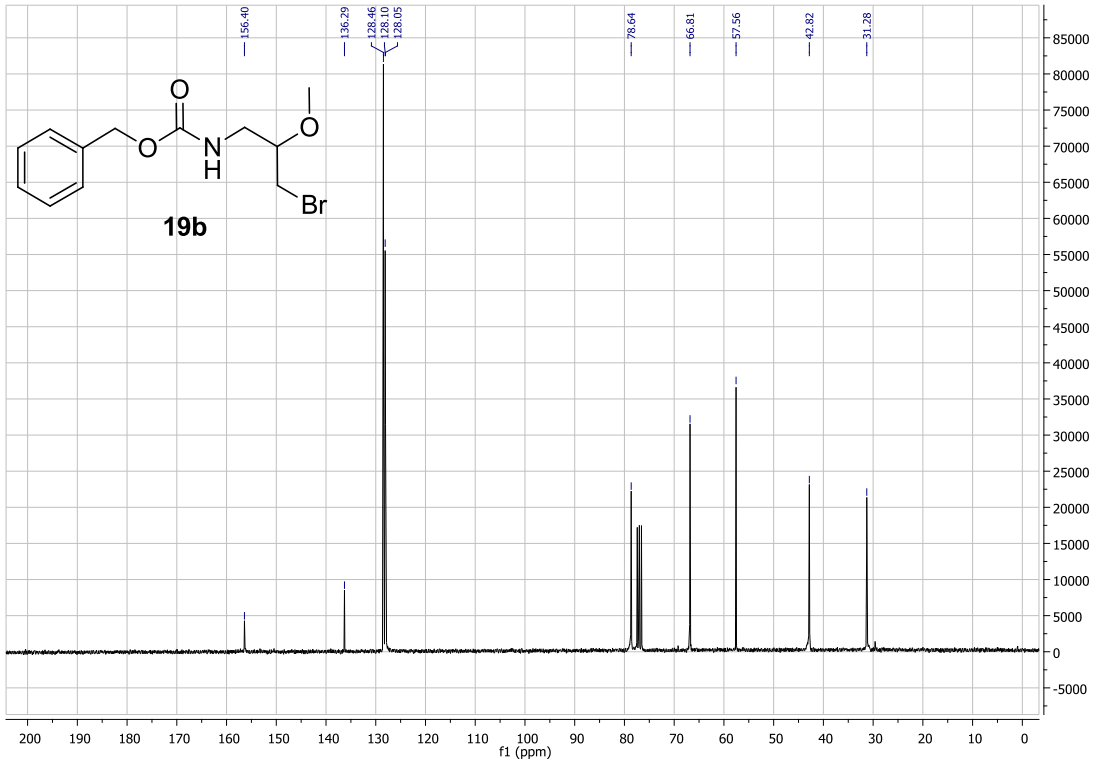
^{13}C-NMR (DMSO$_{d6}$, 75.5 MHz) spectrum of amino – acid carbohydrate conjugate 15:



^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of benzyl (3-bromo-2-methoxypropyl) carbamate 19b:

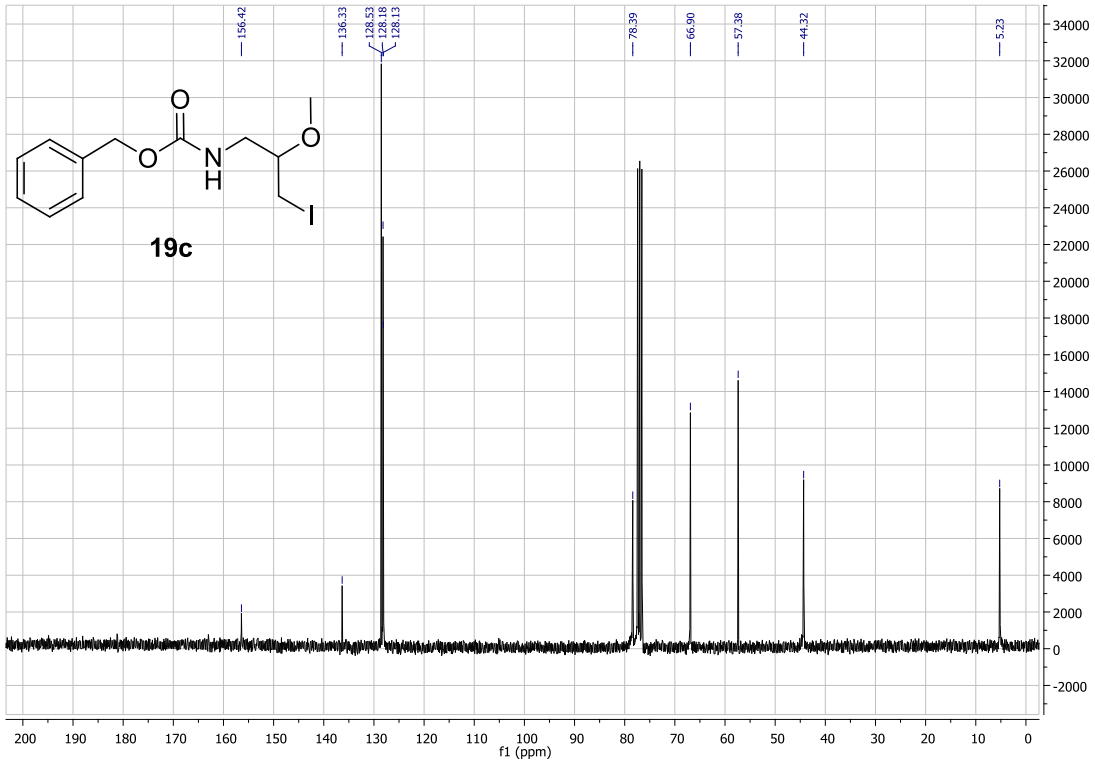


^{13}C-NMR ($CDCl_3$, 75.5 MHz) spectrum of benzyl (3-bromo-2-methoxypropyl) carbamate 19b:

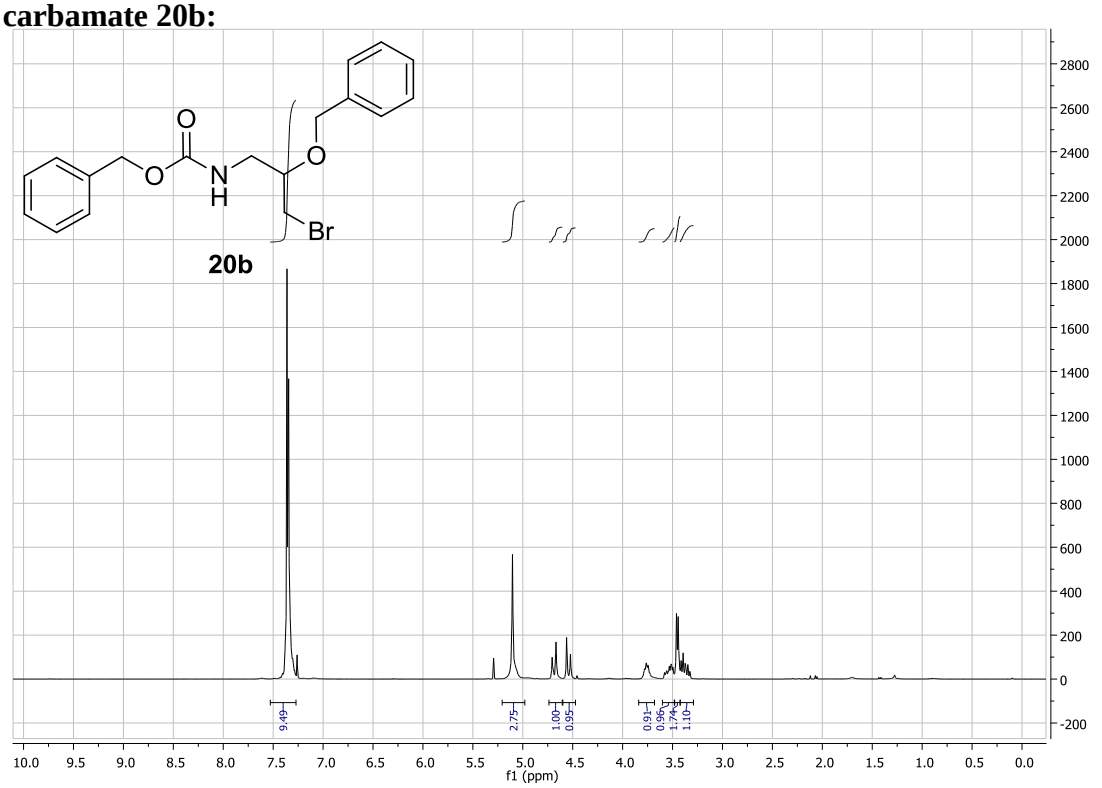


^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of benzyl (3-iodo-2-methoxypropyl) carbamate 19c:

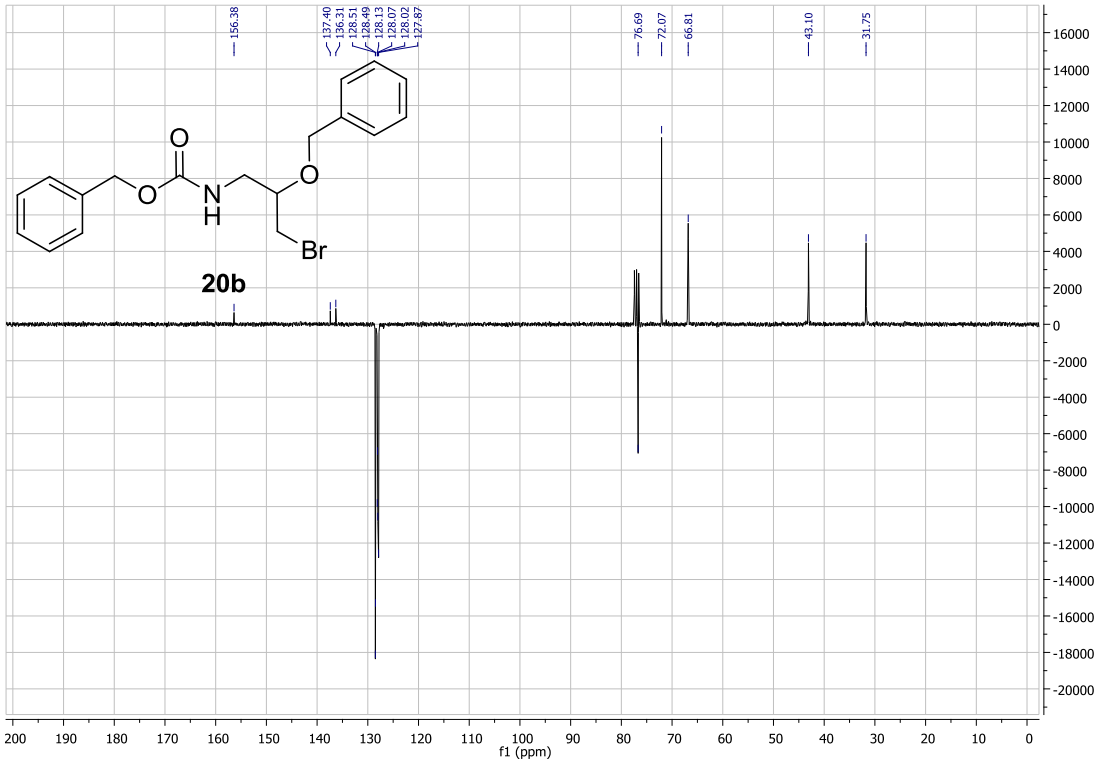
^{13}C-NMR ($CDCl_3$, 75.5 MHz) spectrum of benzyl (3-iodo-2-methoxypropyl) carbamate 19c:



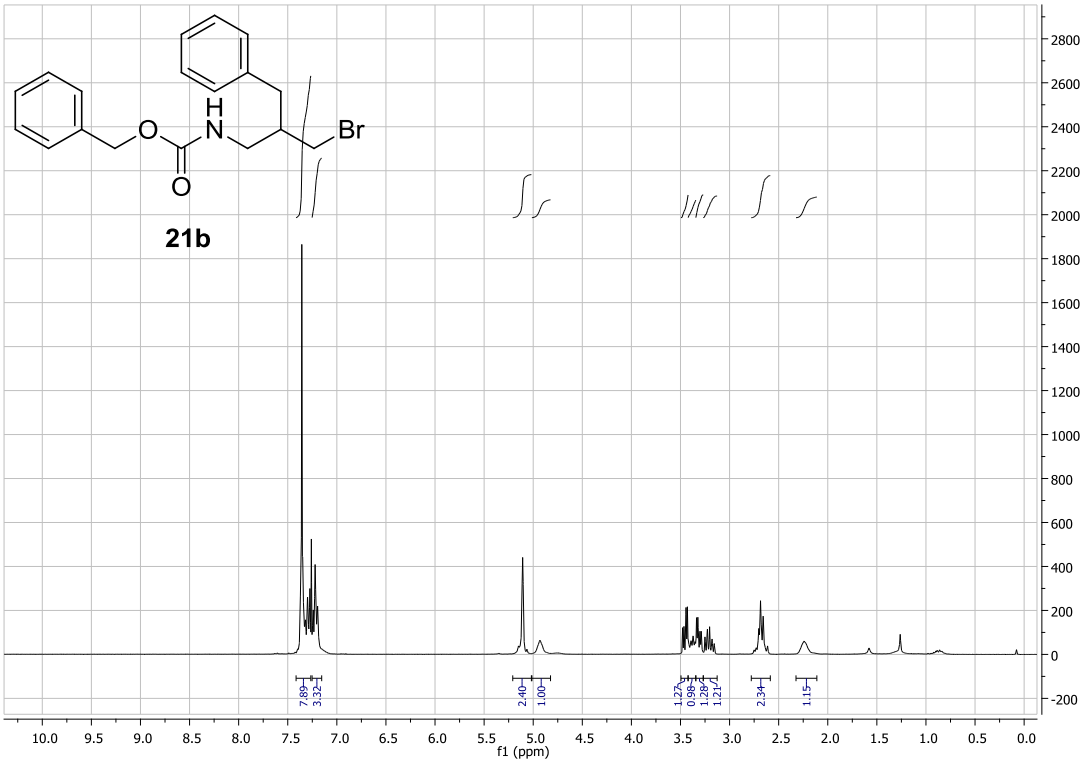
^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of benzyl (2-(benzyloxy)-3-bromopropyl) carbamate 20b:



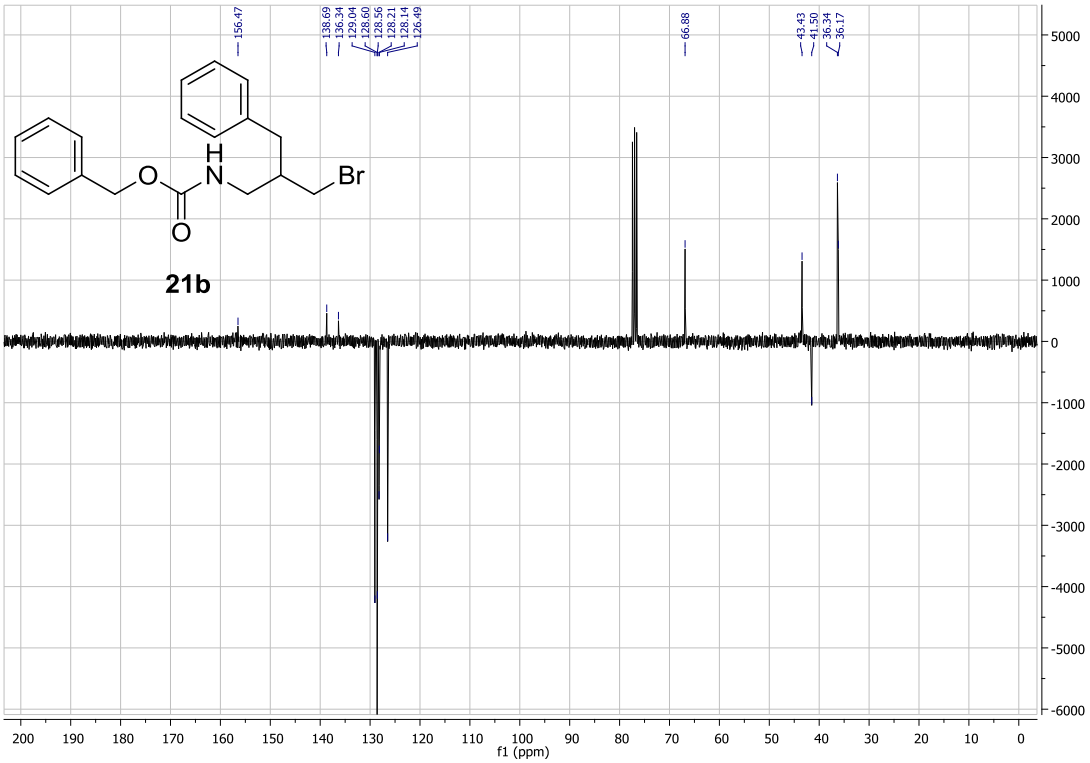
^{13}C-NMR ($CDCl_3$, 75.5 MHz) spectrum of benzyl (2-(benzyloxy)-3-bromopropyl) carbamate 20b:



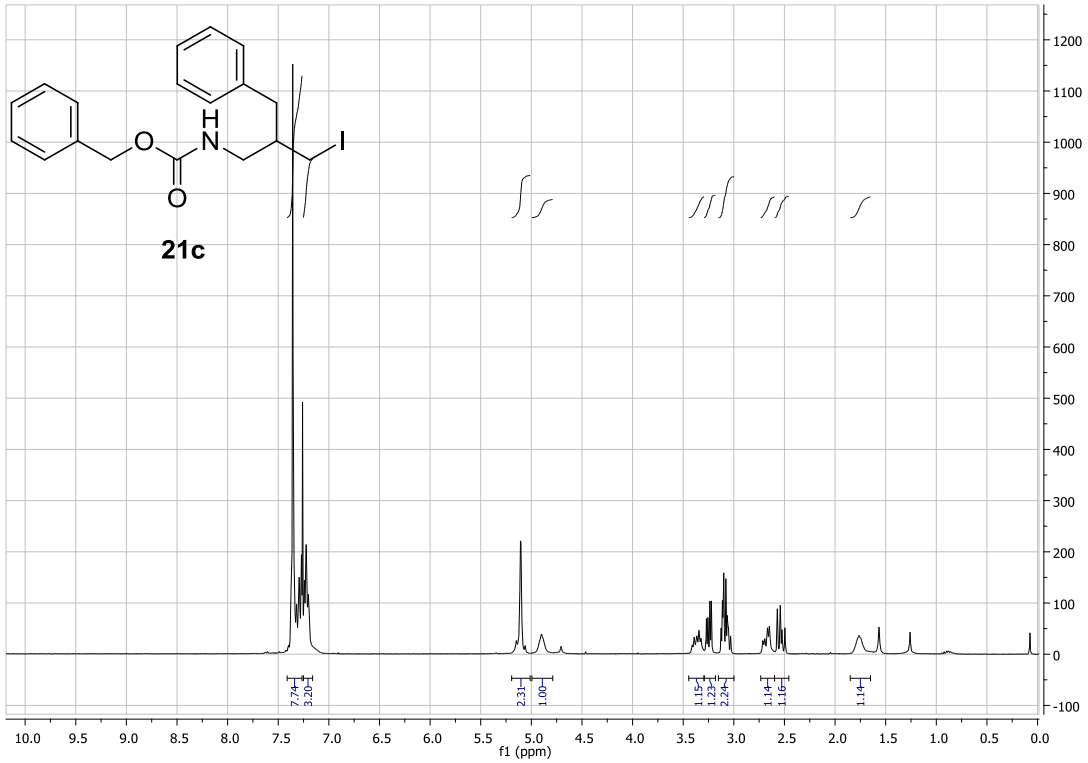
^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of benzyl (2-benzyl-3-bromopropyl) carbamate 21b:



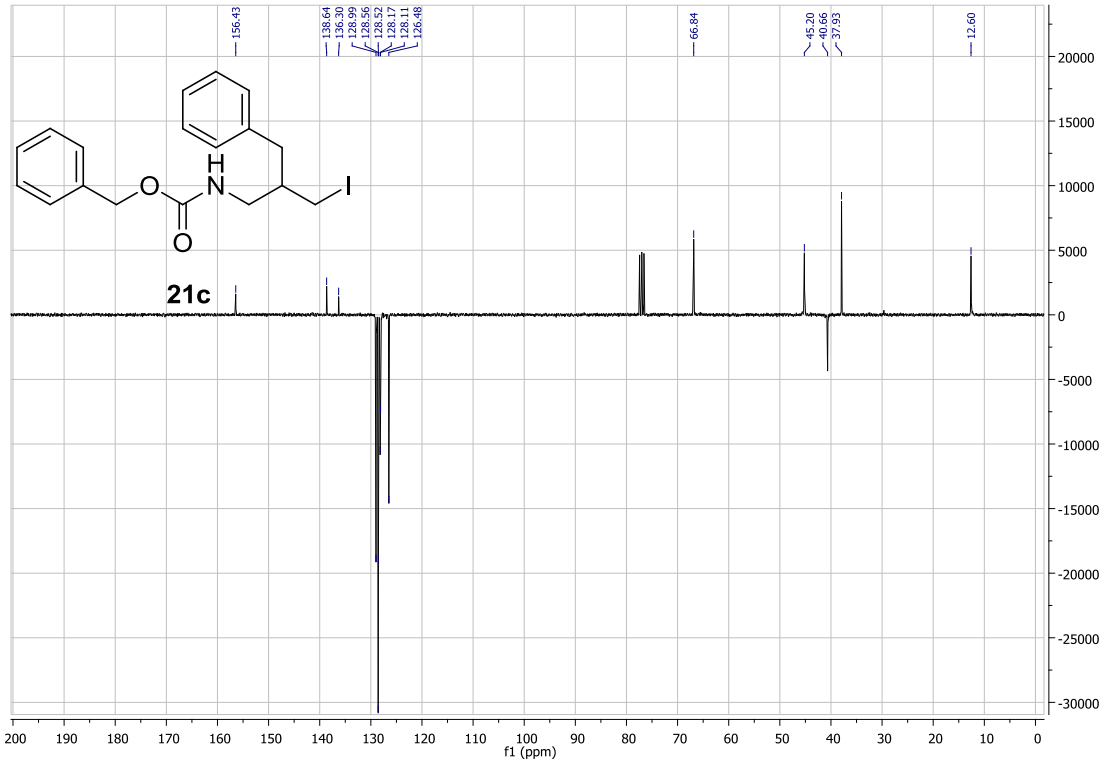
^{13}C-NMR ($CDCl_3$, 75.5 MHz) spectrum of benzyl (2-benzyl-3-bromopropyl) carbamate 21b:



^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of benzyl (2-benzyl-3-iodopropyl) carbamate 21c:



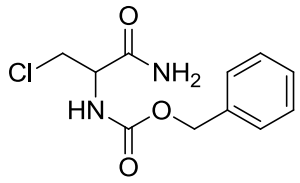
^{13}C-NMR ($CDCl_3$, 75.5 MHz) spectrum of benzyl (2-benzyl-3-iodopropyl) carbamate 21c:

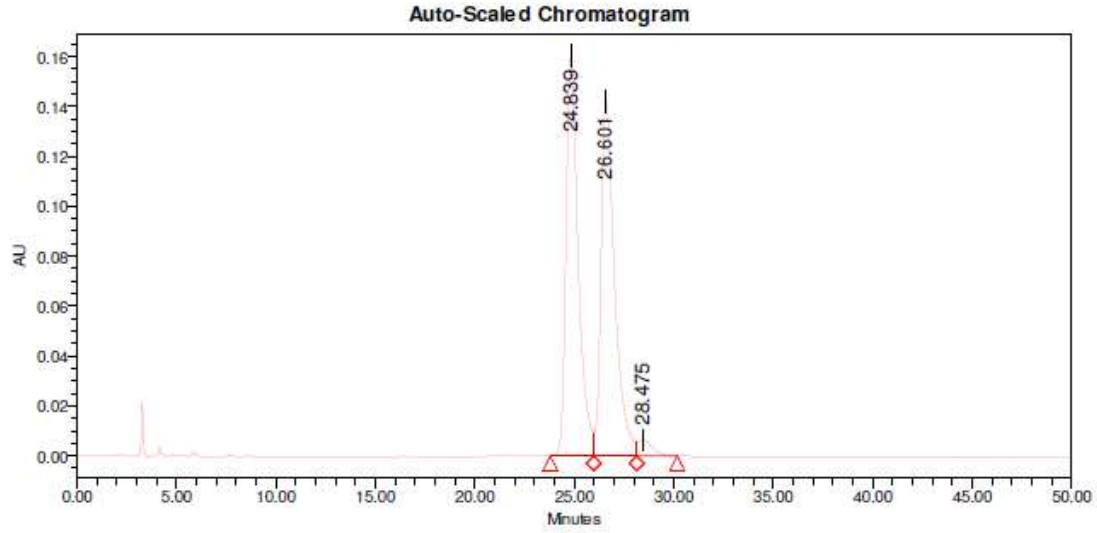


7. HPLC Chromatograms

HPLC analysis for the estimation of enantiomeric purity was performed with Chiralpak IA column (0.46 × 25 cm); isocratic regime: 6% isopropanol in hexanes (prepared by parallel pumping of 40% hexanes and 60% of an eluent containing 10% isopropanol in hexanes); flow rate 1.0 ml/min; sample: 1 mg/ml in 10 % isopropanol in hexanes, injection volume 5 μL, detector wavelength 220 nm.

7.1. HPLC Traces for racemic **2a**

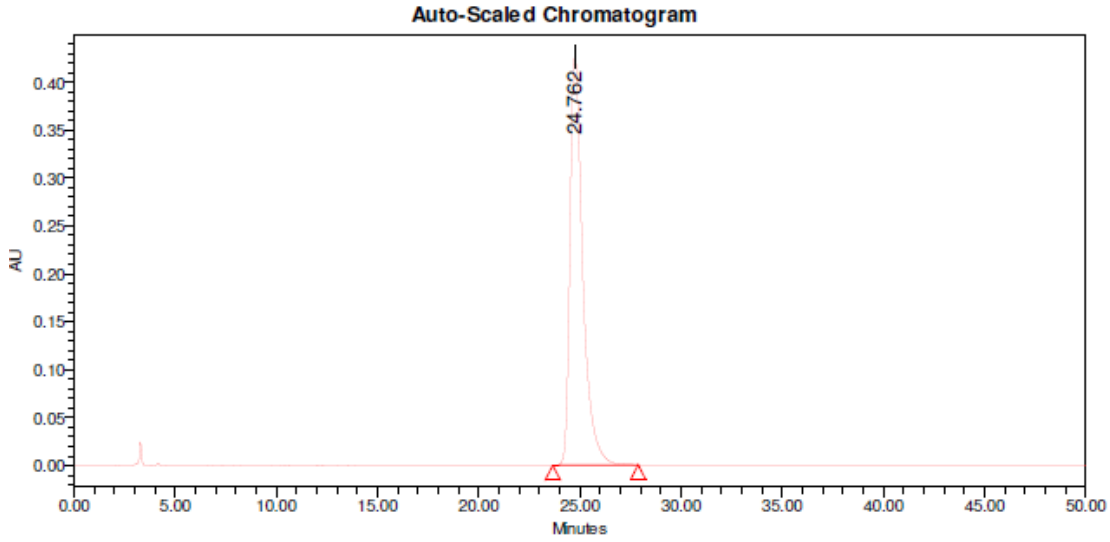




Peak Results

	RT	Area	Height	% Area	Start Time (min)	End Time (min)
1	24.839	6844739	160731	49.01	23.800	25.983
2	26.601	6794300	141863	48.65	25.983	28.150
3	28.475	327721	6314	2.35	28.150	30.183

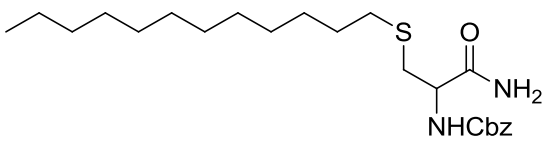
7.2. HPLC Traces for enantioenriched (*R*)-**2a**

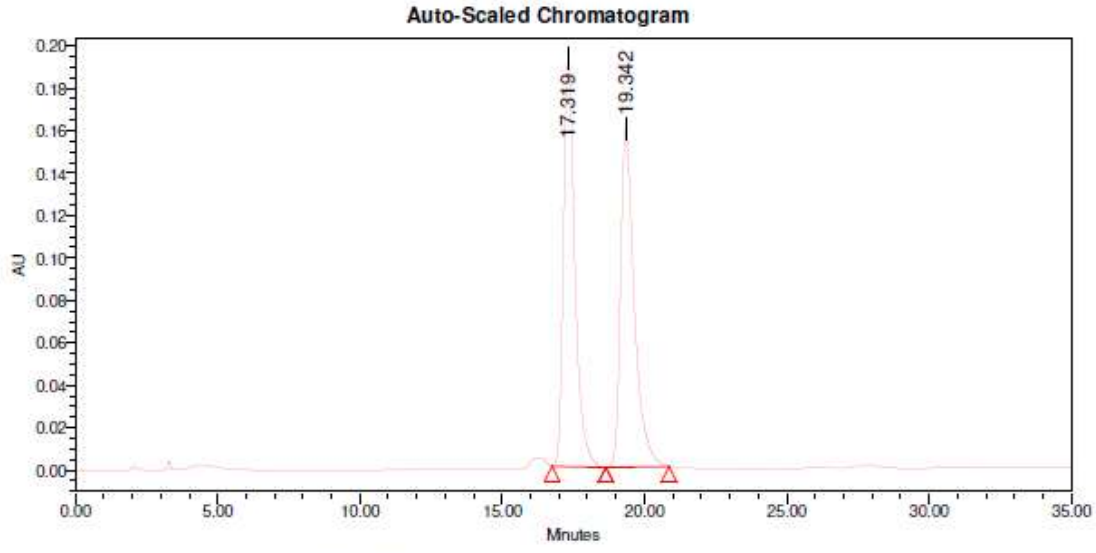


Peak Results

	RT	Area	Height	% Area	Start Time (min)	End Time (min)
1	24.762	18755207	426637	100.00	23.667	27.883

7.3. HPLC Traces for racemic **9b**

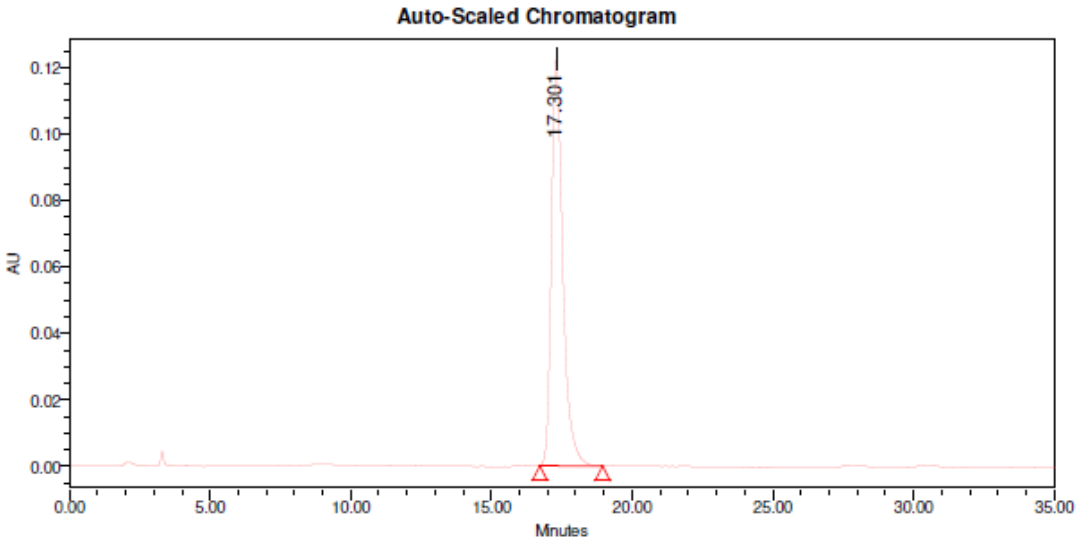




Peak Results

	RT	Area	Height	% Area	Start Time (min)	End Time (min)
1	17.319	5392657	192465	50.15	16.750	18.633
2	19.342	5360895	159550	49.85	18.650	20.867

7.4. HPLC Traces for enantioenriched (*R*)-**9b**



Peak Results

	RT	Area	Height	% Area	Start Time (min)	End Time (min)
1	17.301	3454413	122482	100.00	16.717	18.950