Supporting Information

Alkene Oxyamination using Malonoyl Peroxides: Preparation of Pyrrolidines and Isoxazolidines

Carla Alamillo-Ferrer,[†] Jonathan M. Curle,[†] Stuart C. Davidson,[†] Simon C. C. Lucas,[†,‡] Stephen J. Atkinson,[‡] Matthew Campbell,[‡] Alan R. Kennedy[†] and Nicholas C. O. Tomkinson[†,*]

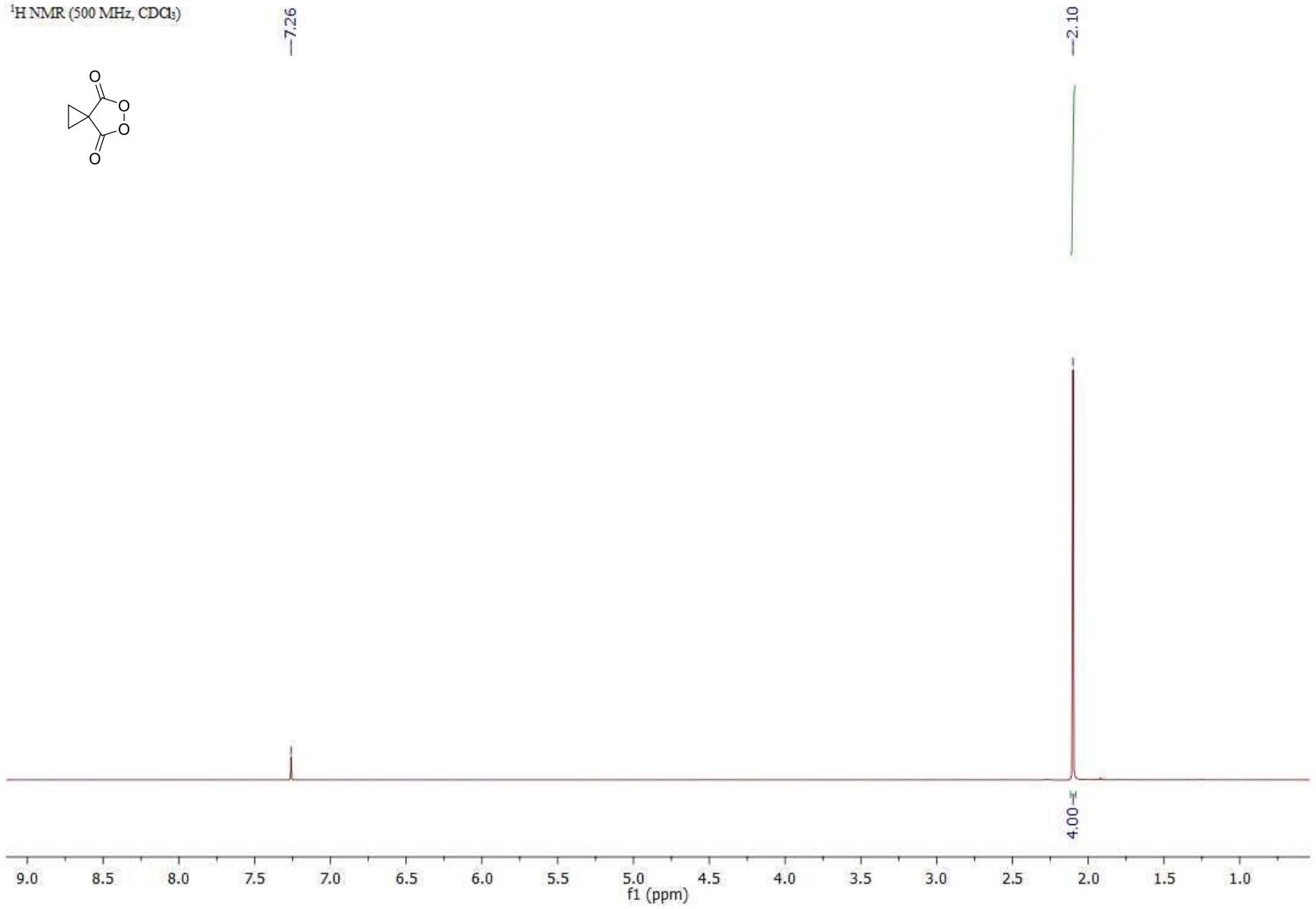
[†]*WestCHEM, Department of Pure and Applied Chemistry, Thomas Graham Building, University of Strathclyde, 295 Cathedral Street, Glasgow, G1 1XL, United Kingdom*

[‡]*GlaxoSmithKline Medicines Research Centre, Gunnels Wood Road, Stevenage, SG1 2NY, United Kingdom*

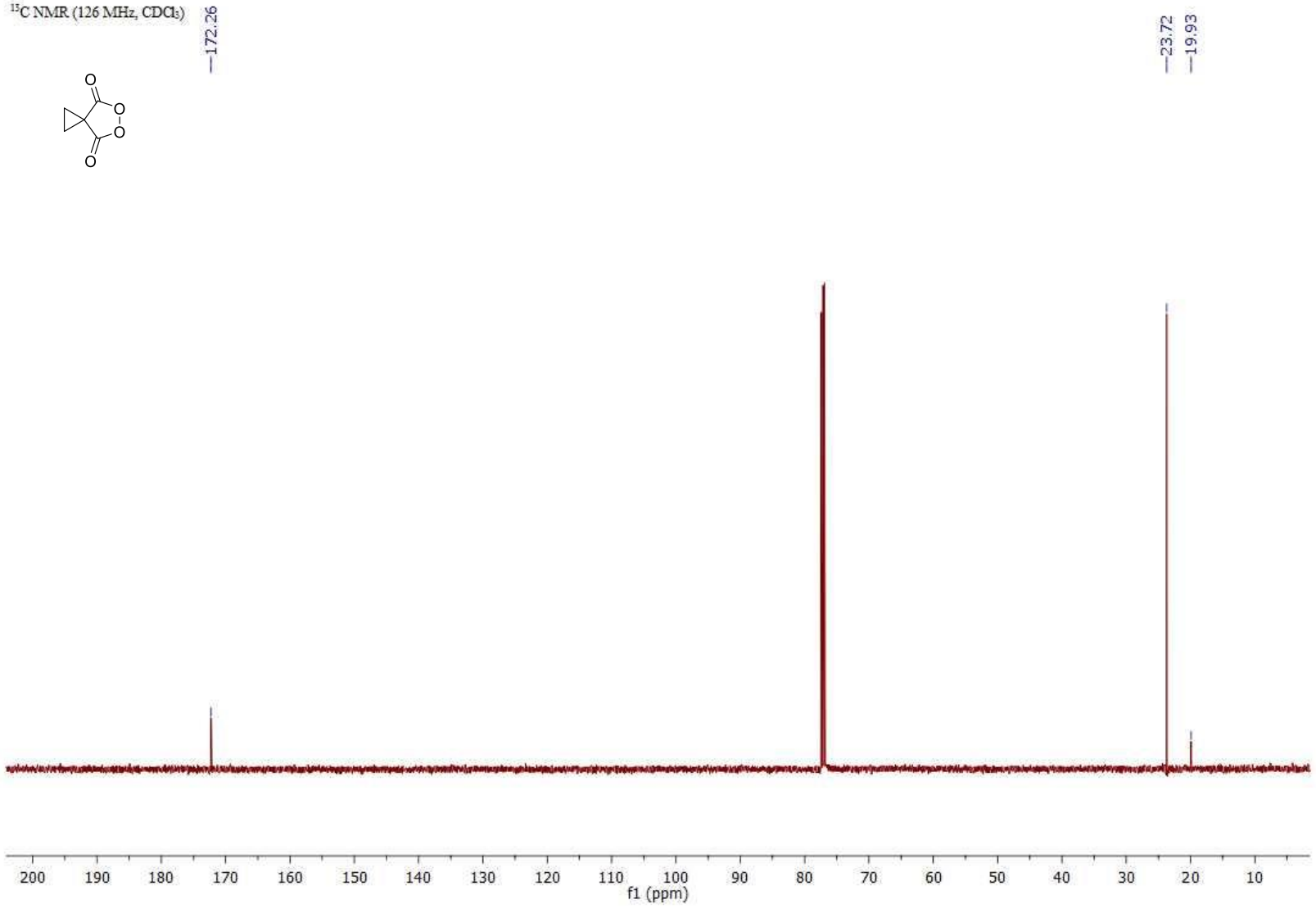
Nicholas.Tomkinson@strath.ac.uk

Contents

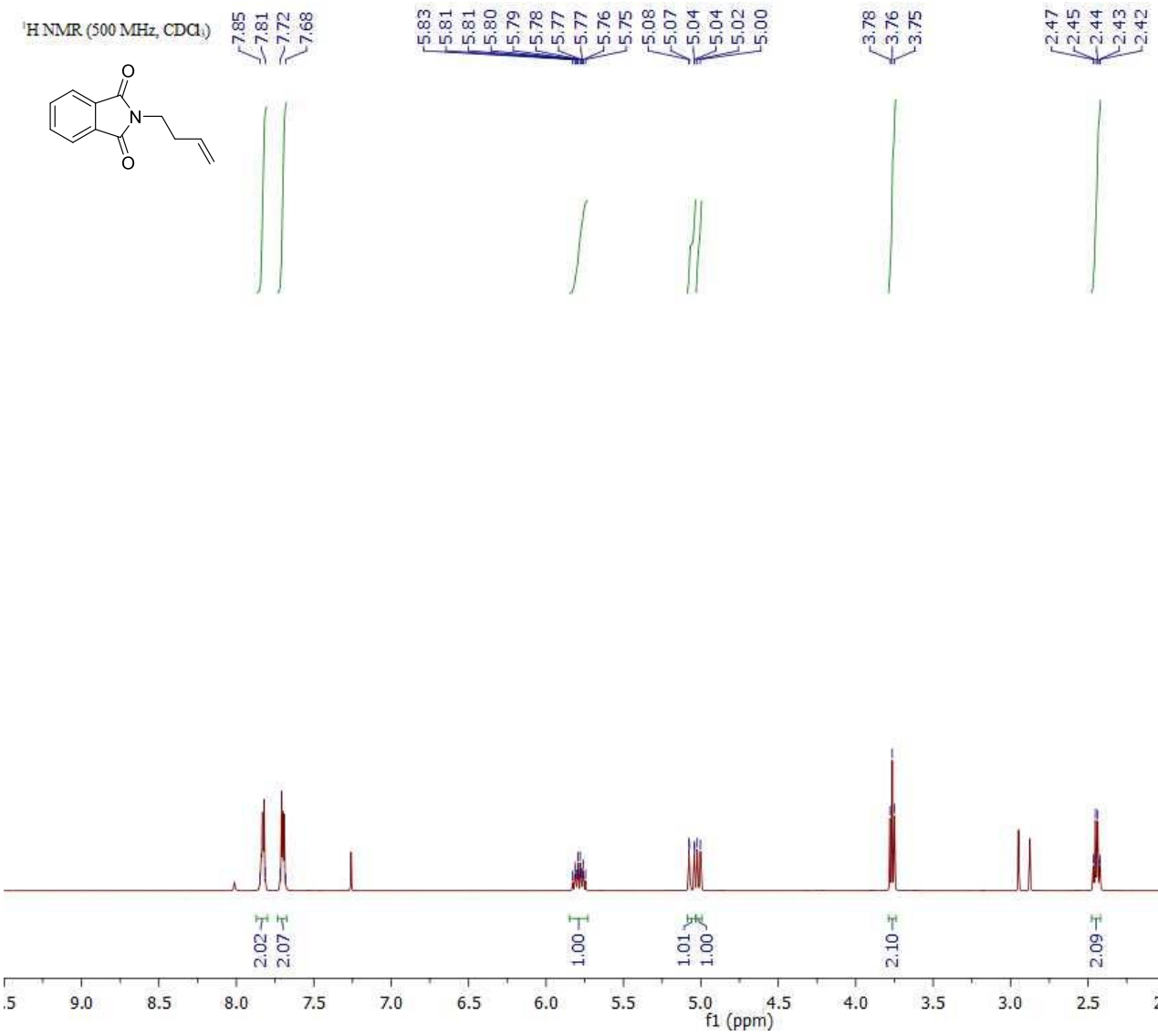
Malonoyl peroxide 1, ^{1}H NMR 500 MHz $CDCl_3$



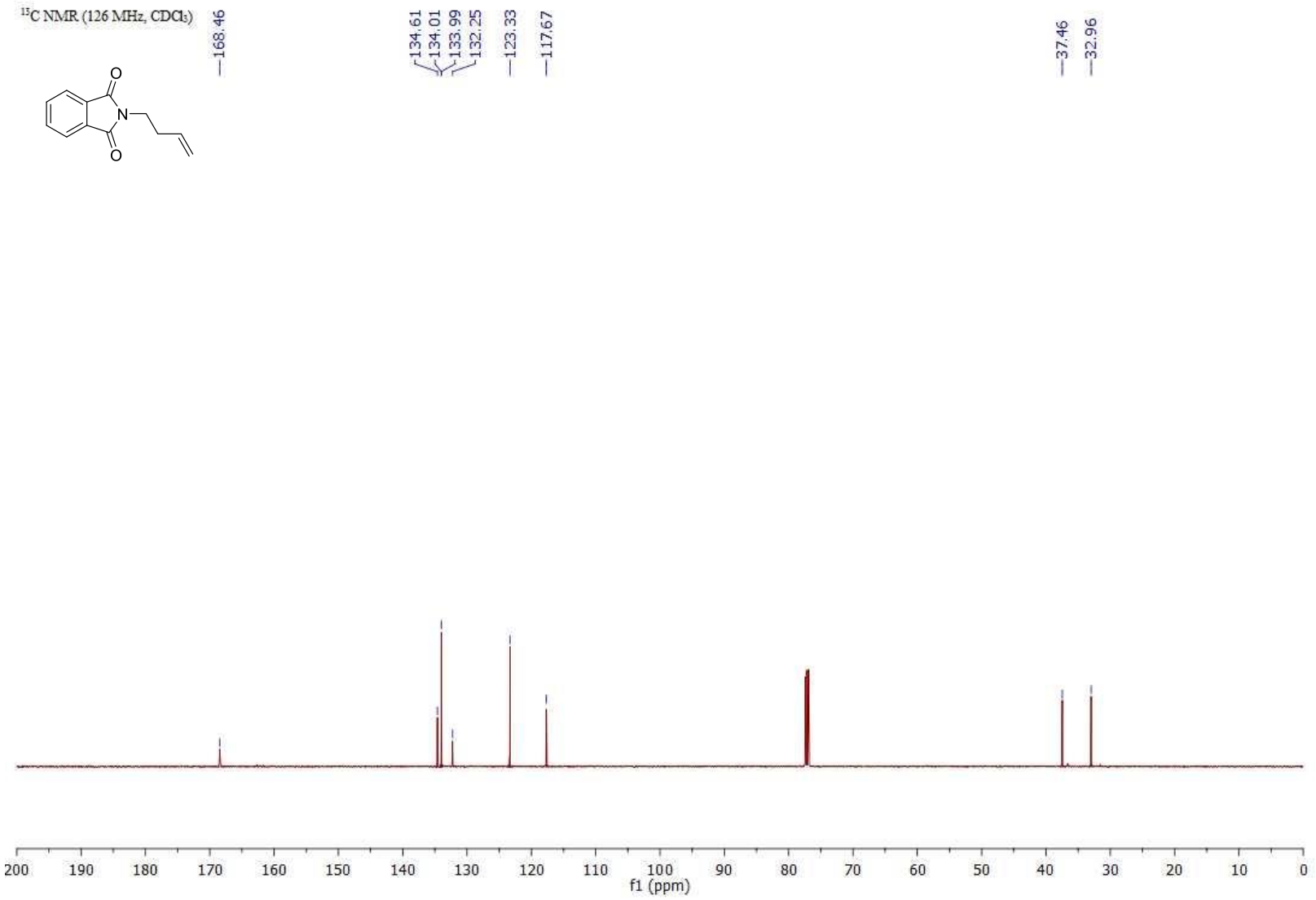
Malonoyl peroxide 1, ^{13}C NMR 126 MHz $CDCl_3$



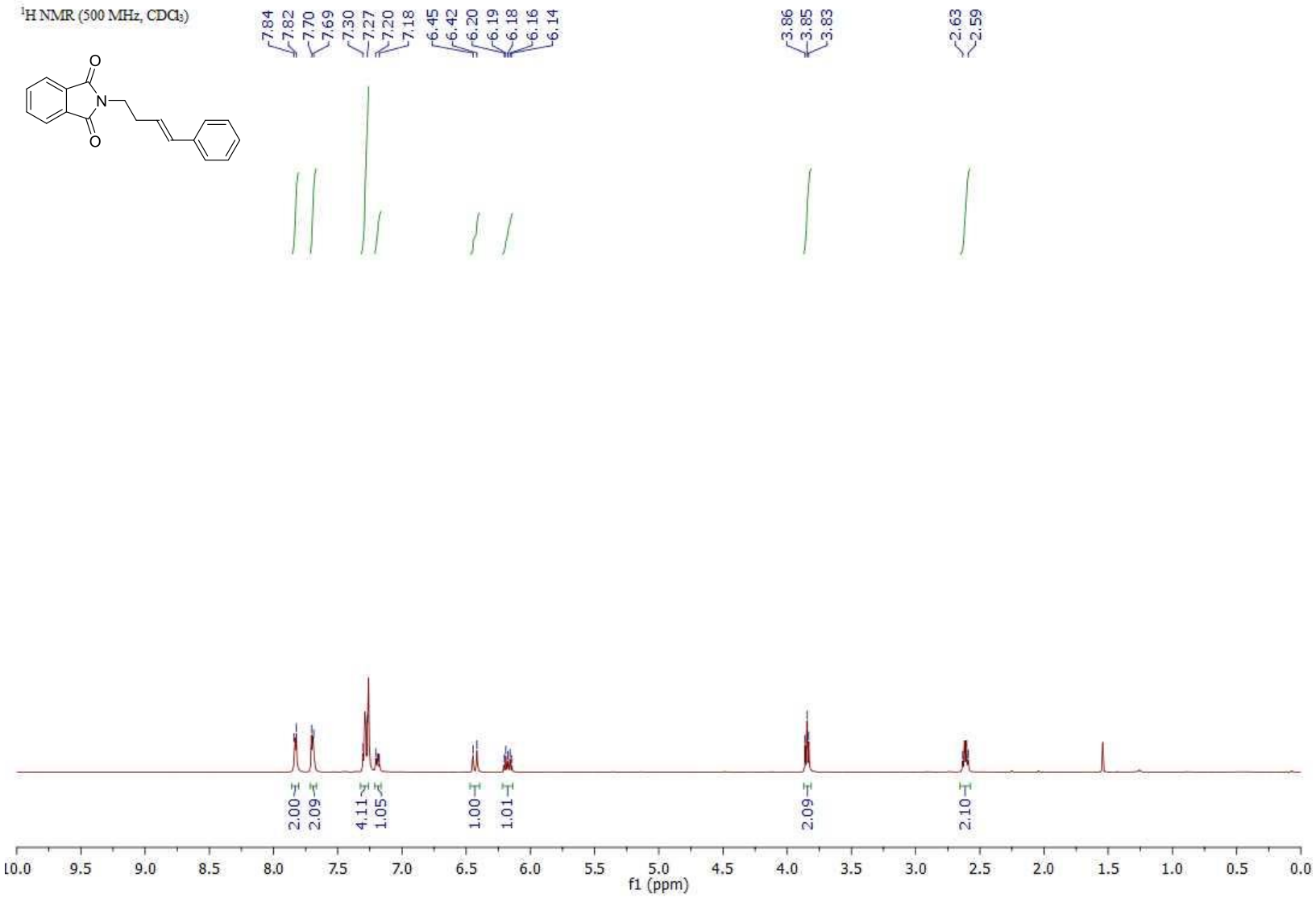
2-(But-3-en-1-yl)isoindoline-1,3-dione, ^{1}H NMR 500 MHz $CDCl_3$



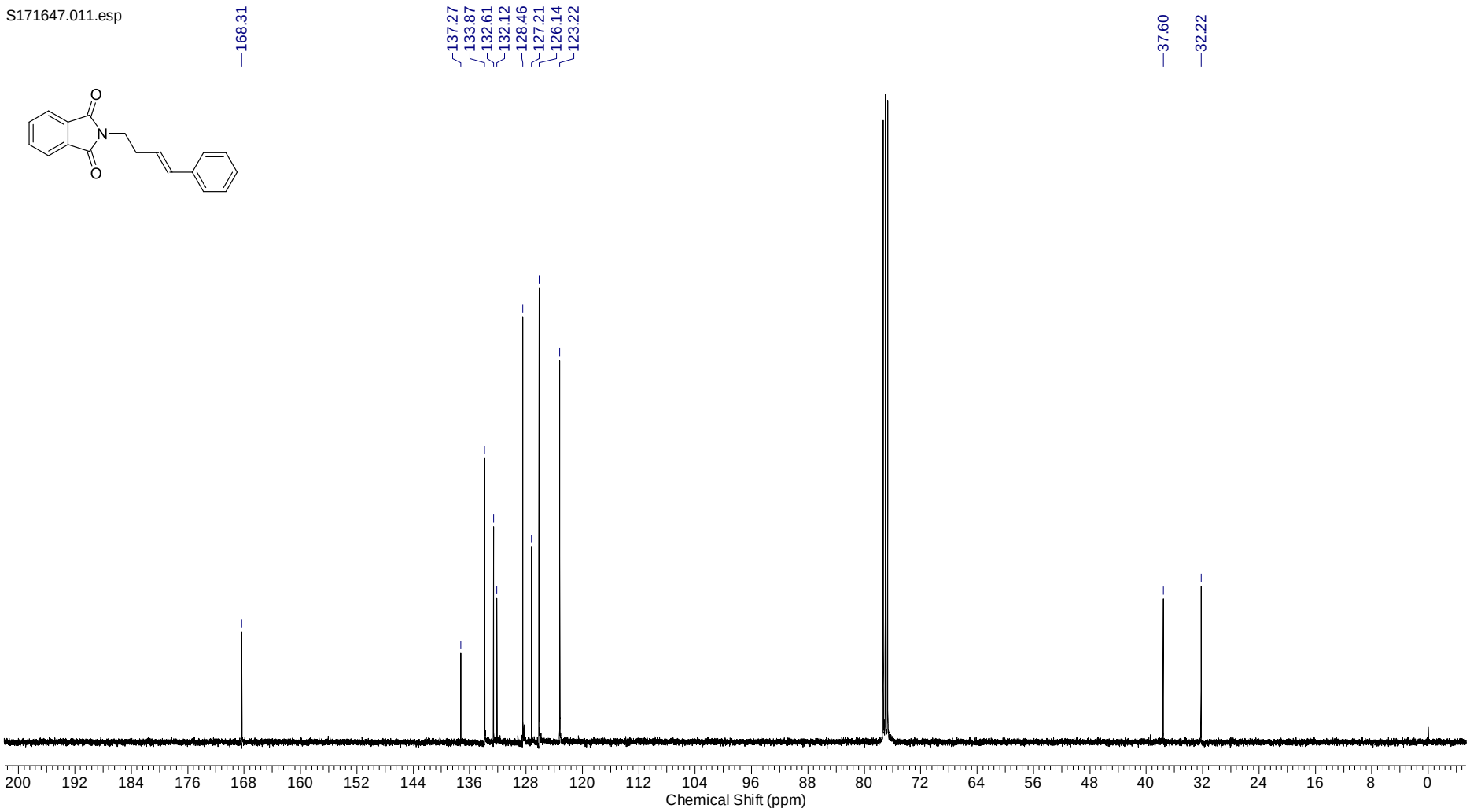
2-(But-3-en-1-yl)isoindoline-1,3-dione, ^{13}C NMR 126 MHz $CDCl_3$



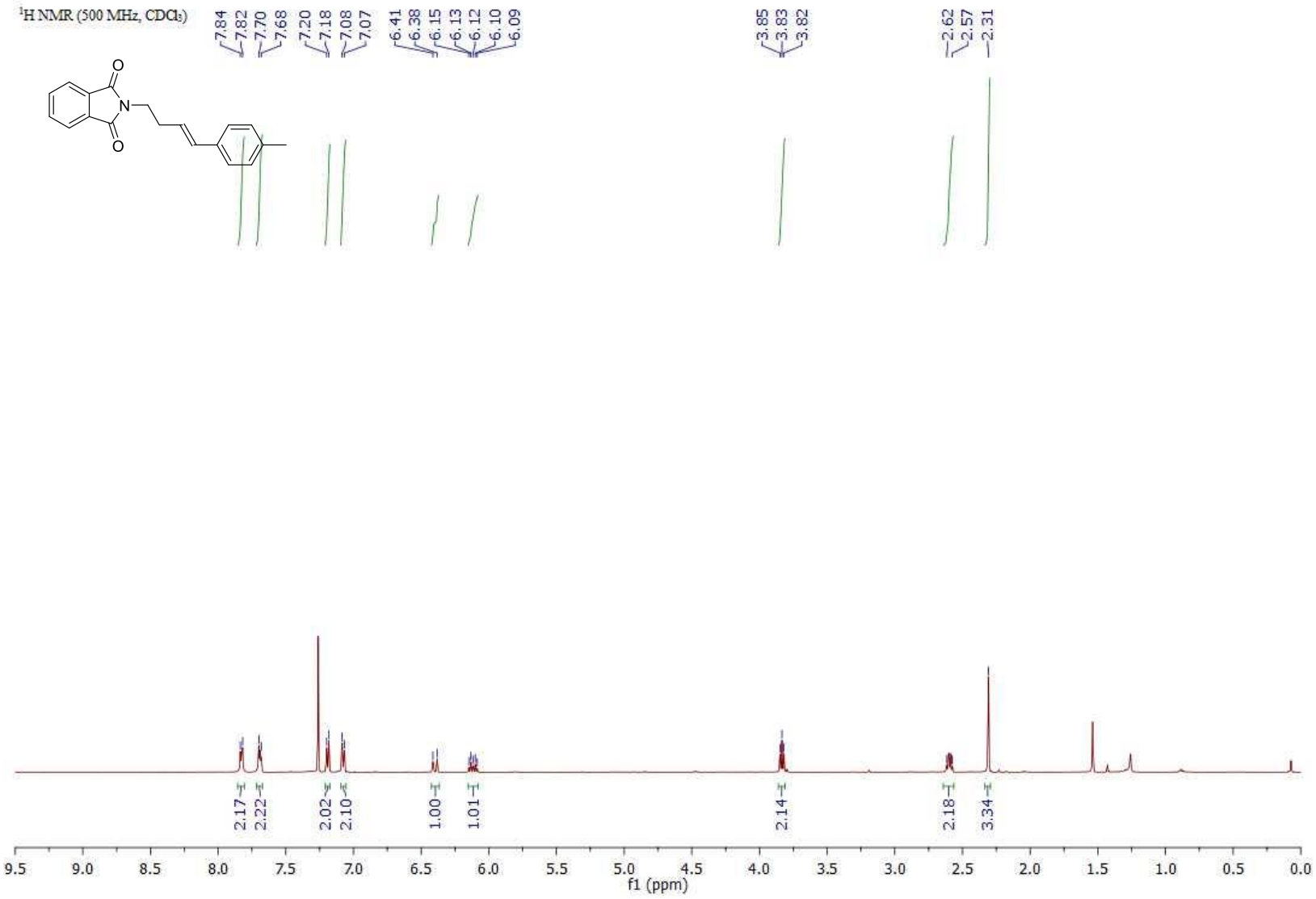
(*E*)-2-(4-Phenylbut-3-en-1-yl)isoindoline-1,3-dione, ^{1}H NMR 500 MHz $CDCl_3$



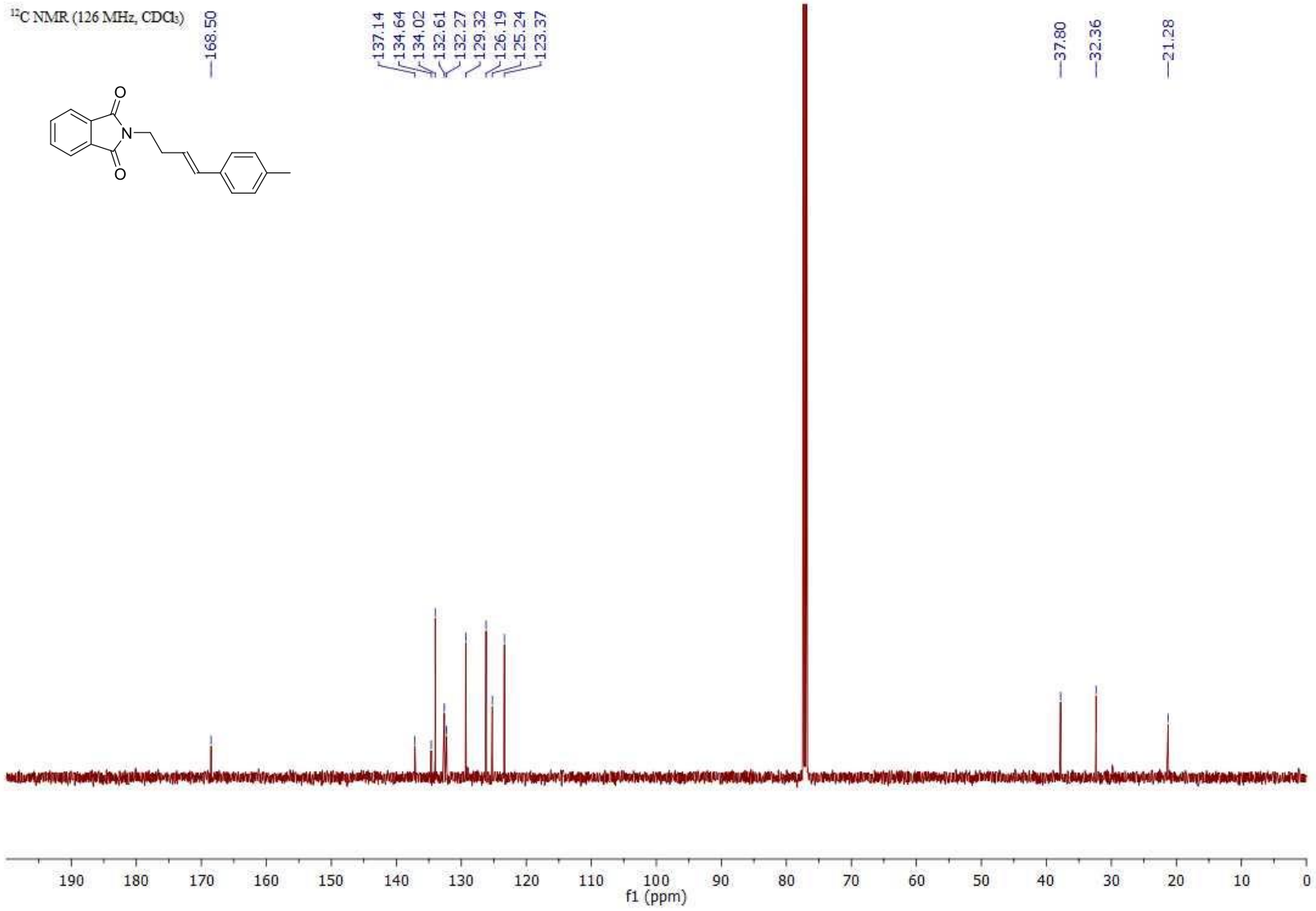
(*E*)-2-(4-Phenylbut-3-en-1-yl)isoindoline-1,3-dione,^{13}C NMR 126 MHz $CDCl_3$



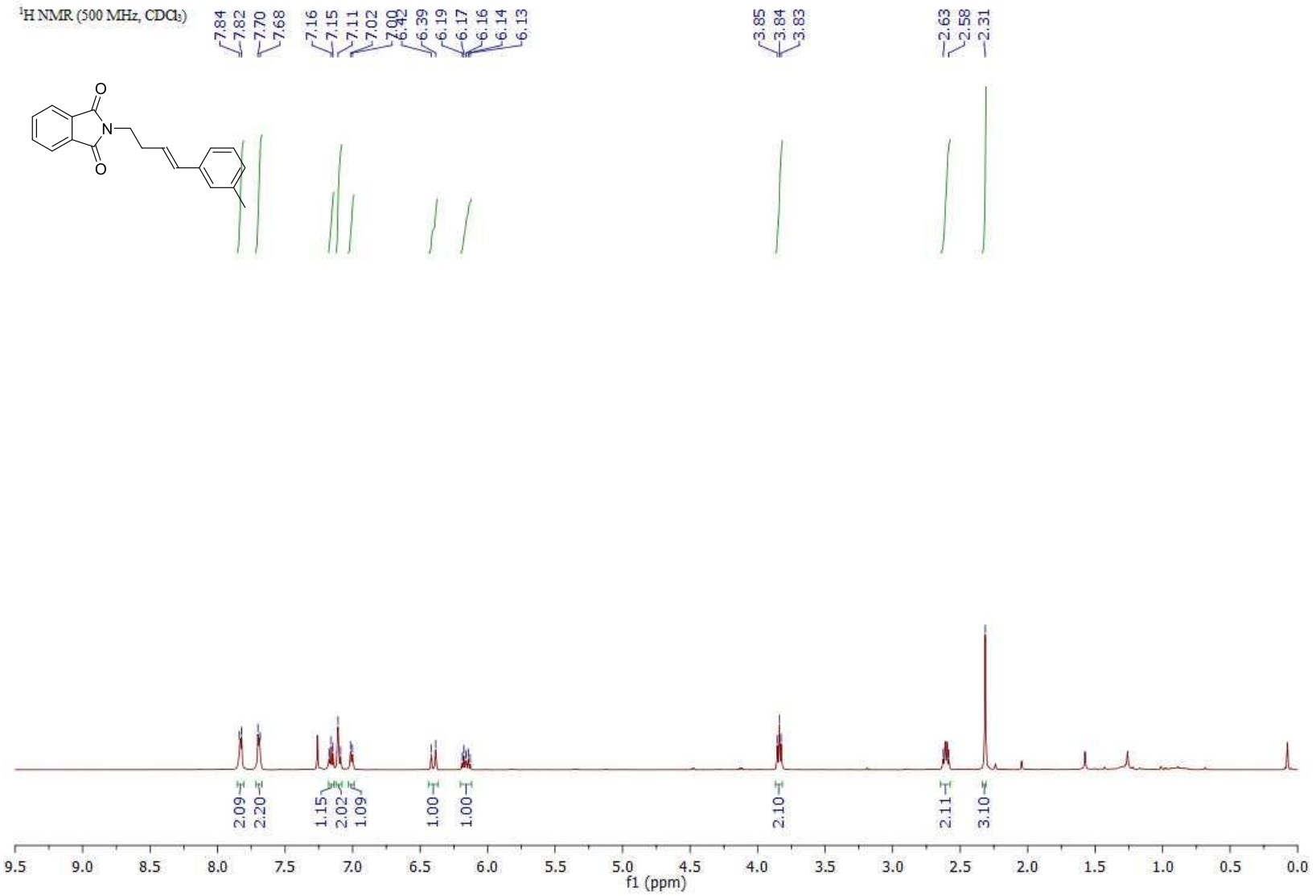
(*E*)-2-(4-*p*-Tolyl)but-3-en-1-yl)isoindoline-1,3-dione, ^{1}H NMR 500 MHz $CDCl_3$



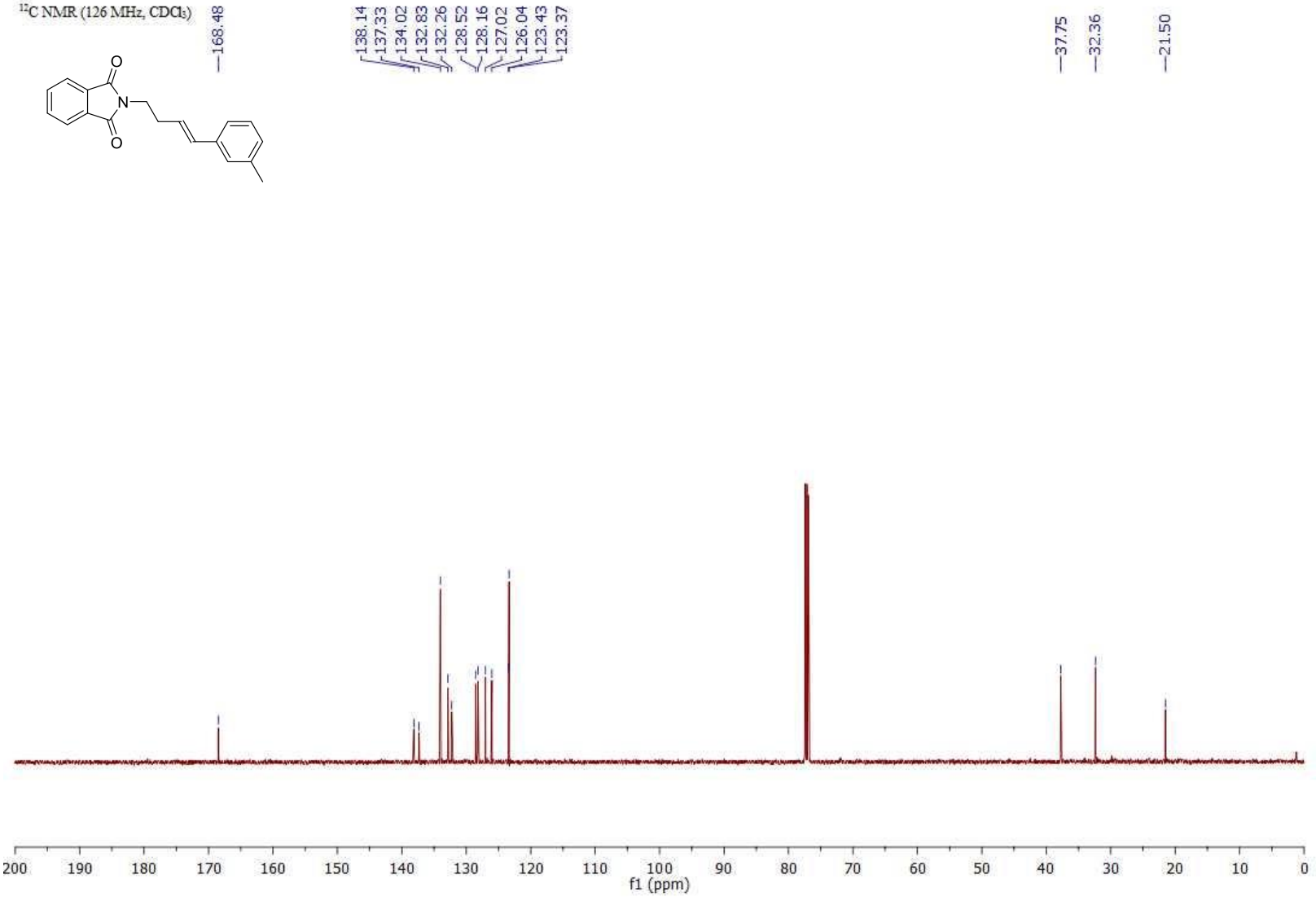
(*E*)-2-(4-*p*-Tolyl)but-3-en-1-yl)isoindoline-1,3-dione, ^{13}C NMR 126 MHz $CDCl_3$



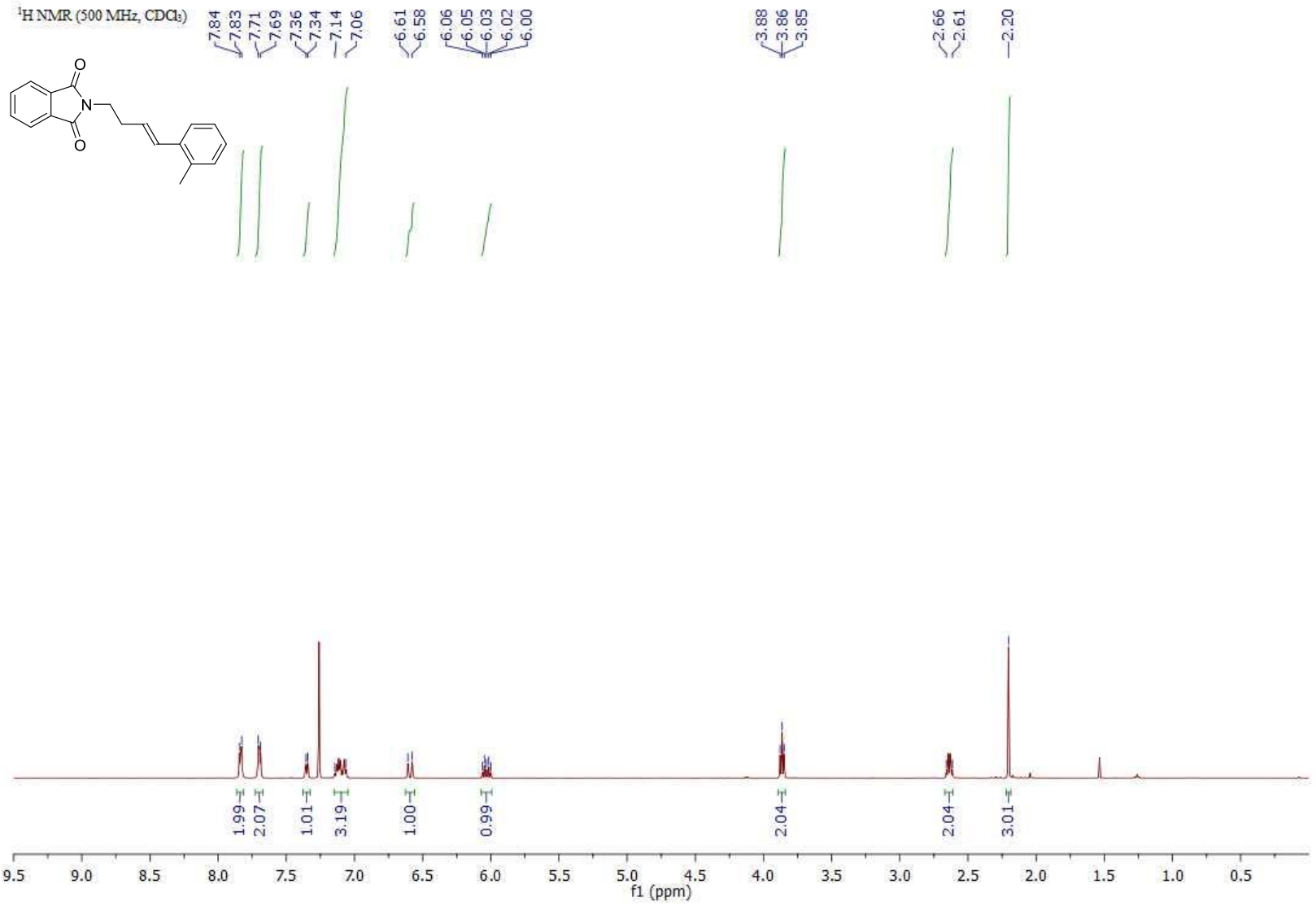
(*E*)-2-(4-*m*-Tolyl)but-3-en-1-yl)isoindoline-1,3-dione, ^{1}H NMR 500 MHz $CDCl_3$



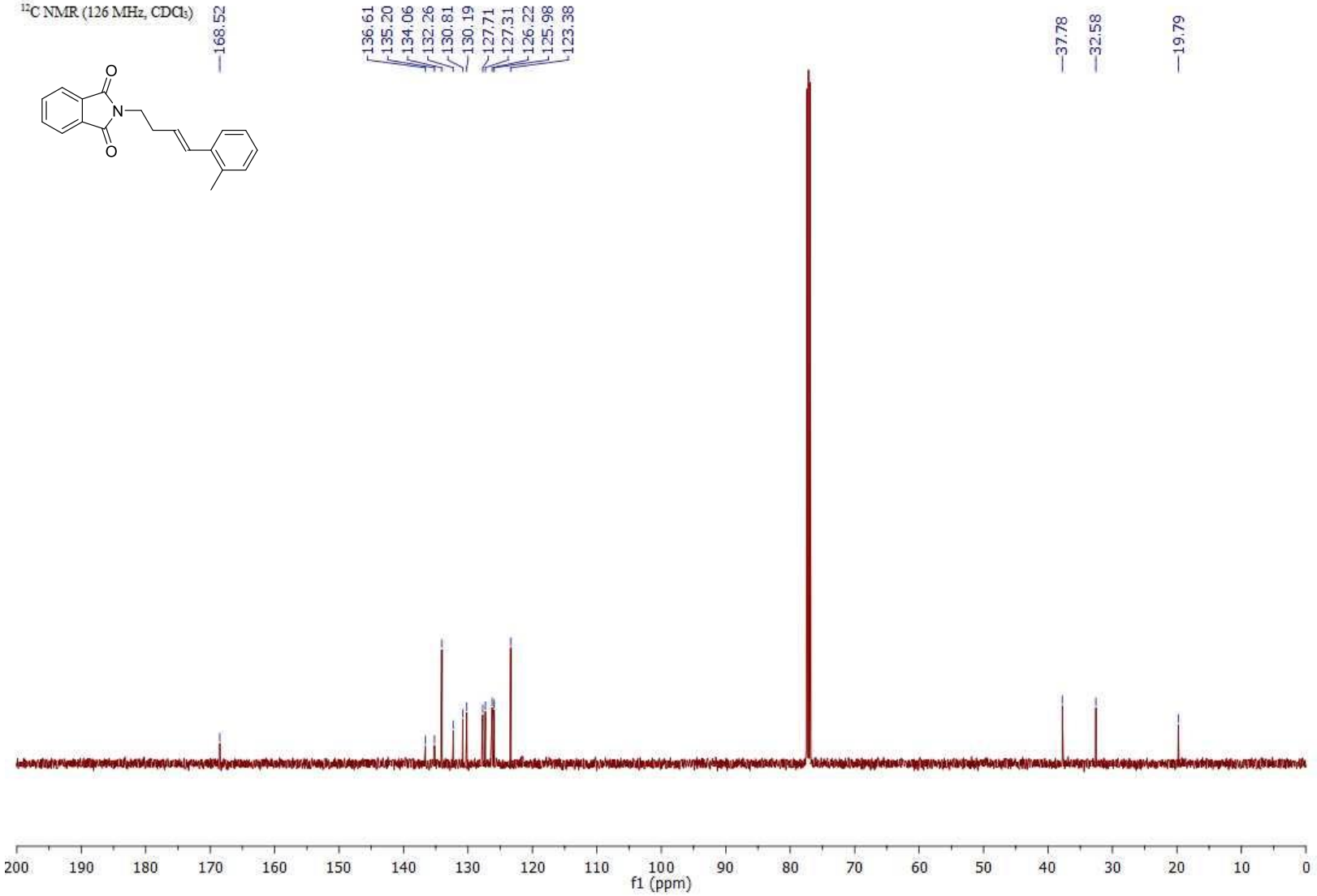
(*E*)-2-(4-*m*-Tolyl)but-3-en-1-yl)isoindoline-1,3-dione, ^{13}C NMR 126 MHz $CDCl_3$



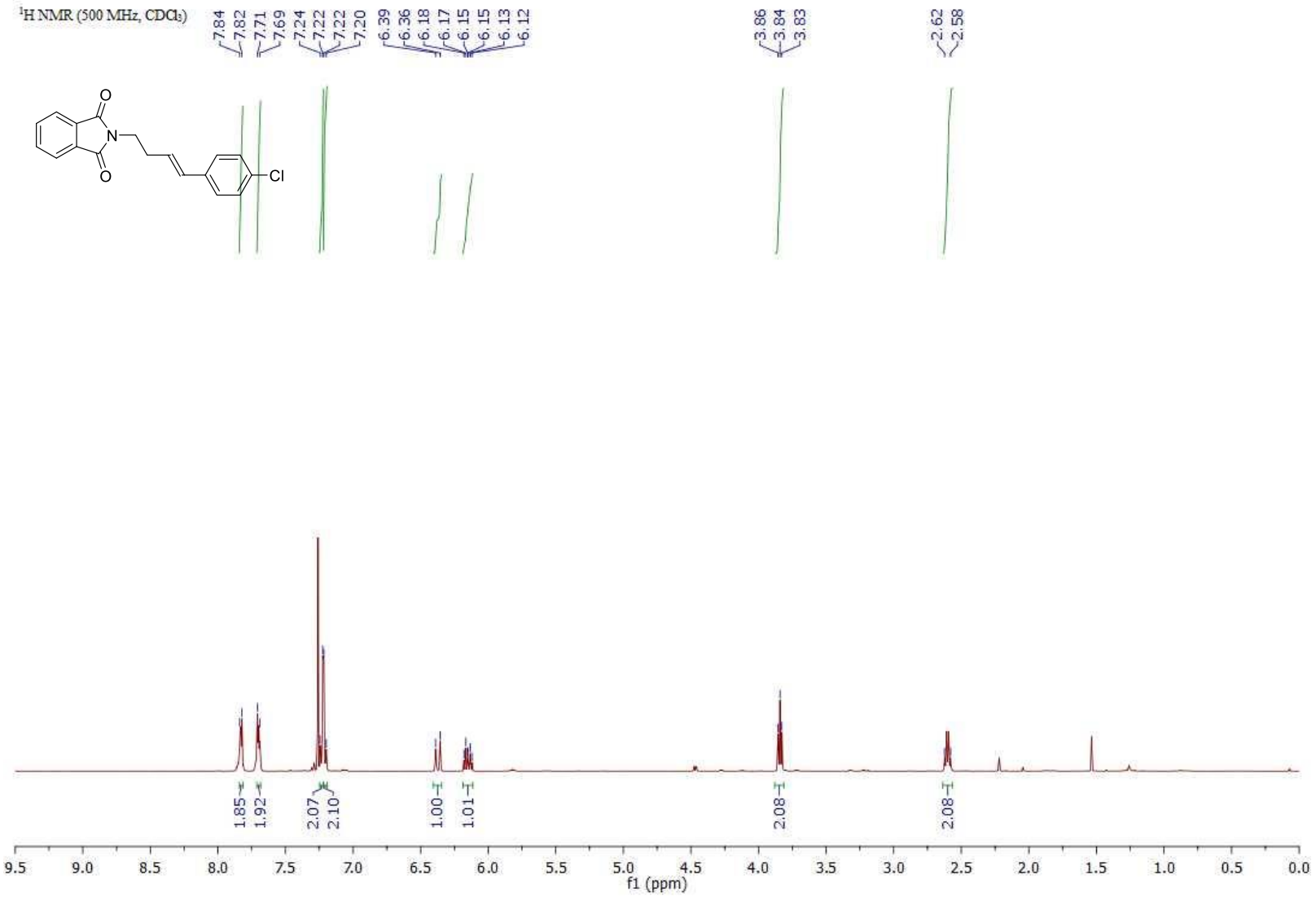
(*E*)-2-(4-*o*-Tolyl)but-3-en-1-yl)isoindoline-1,3-dione, ^{1}H NMR 500 MHz $CDCl_3$



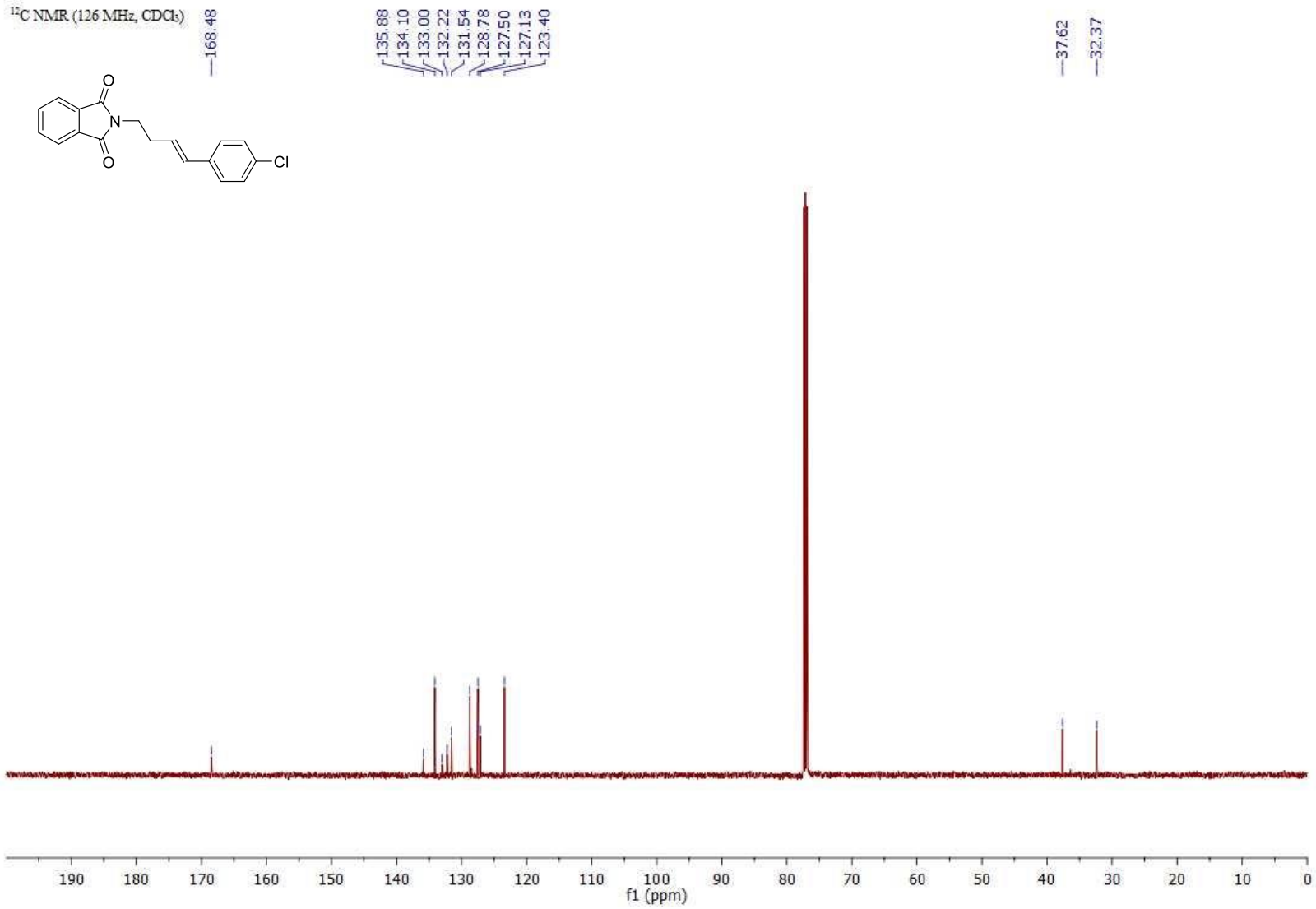
(*E*)-2-(4-*o*-Tolyl)but-3-en-1-yl)isoindoline-1,3-dione, ^{13}C NMR 126 MHz $CDCl_3$



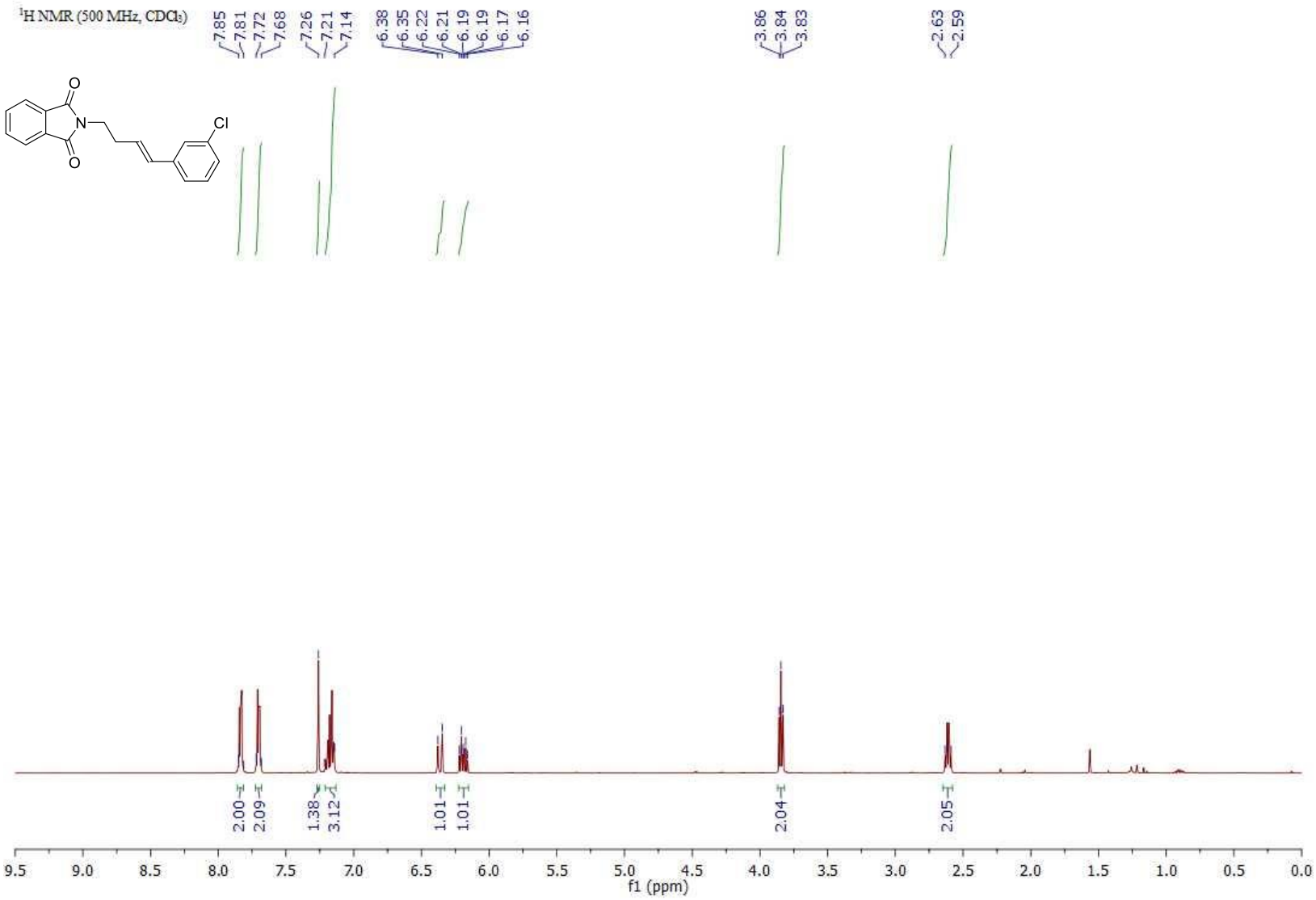
(*E*)-2-(4-(4-Chlorophenyl)but-3-en-1-yl)isoindoline-1,3-dione, ^{1}H NMR 500 MHz $CDCl_3$



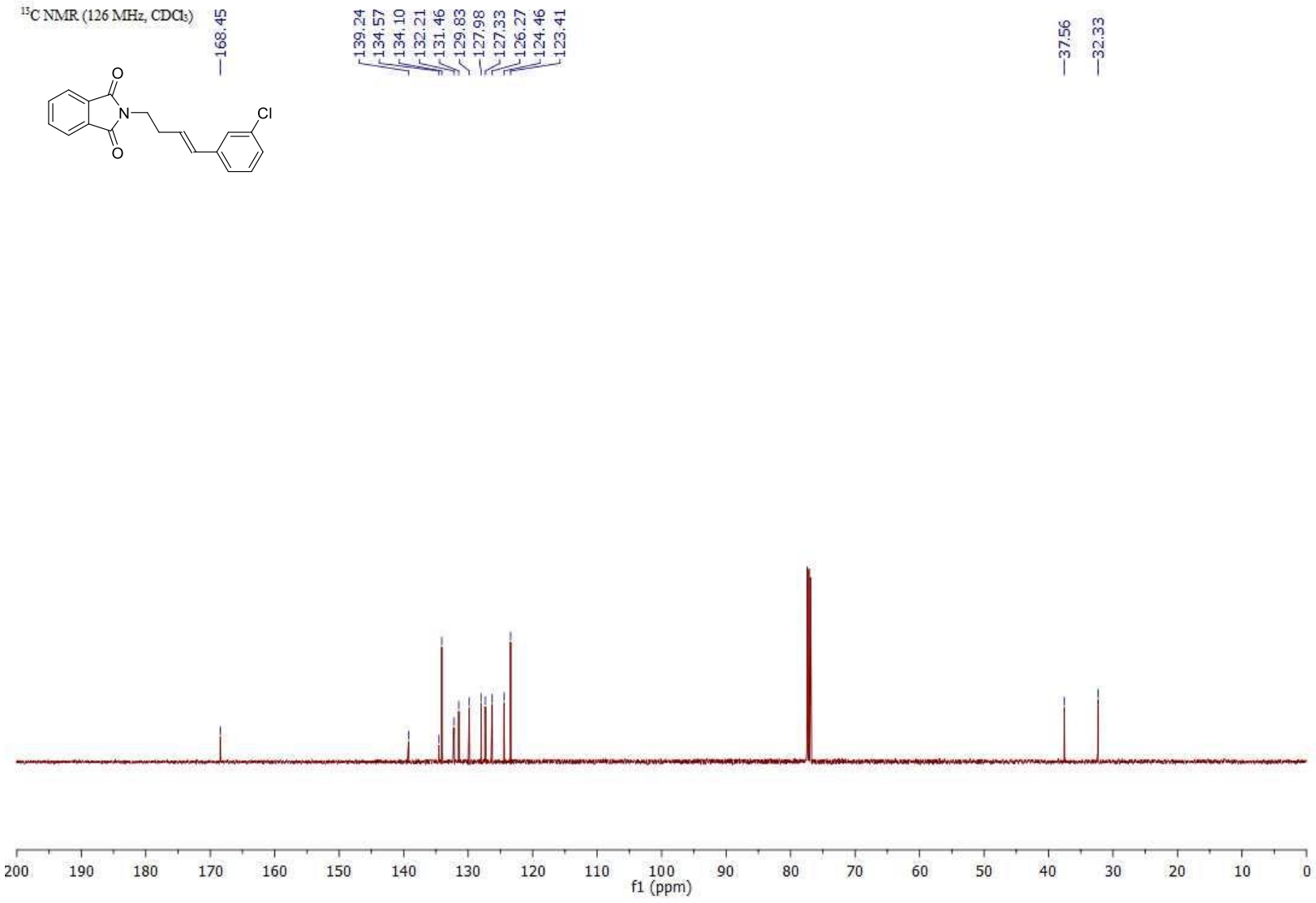
(*E*)-2-(4-(4-Chlorophenyl)but-3-en-1-yl)isoindoline-1,3-dione, ^{13}C NMR 126 MHz $CDCl_3$



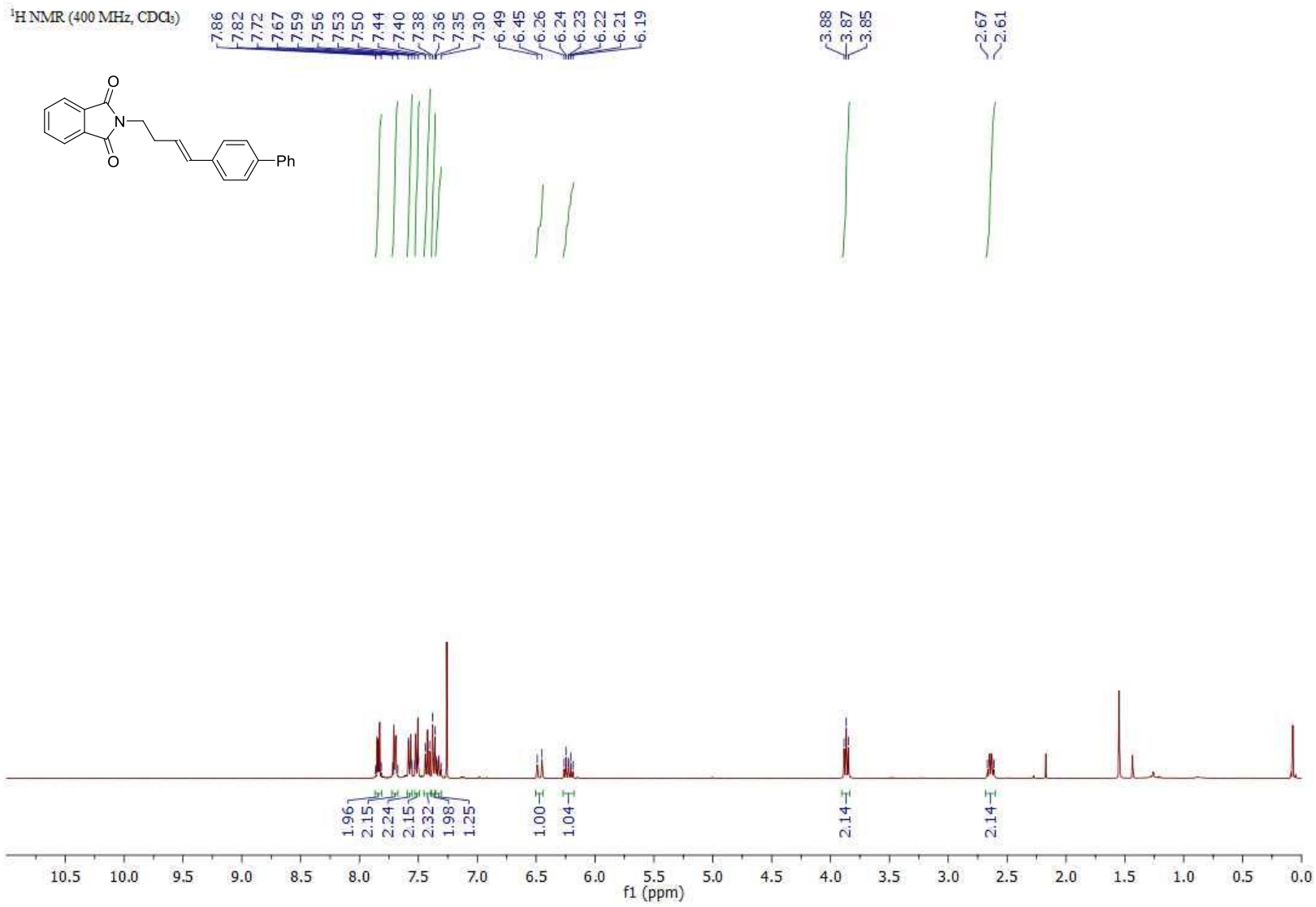
(*E*)-2-(4-(3-Chlorophenyl)but-3-en-1-yl)isoindoline-1,3-dione, ^{1}H NMR 500 MHz $CDCl_3$



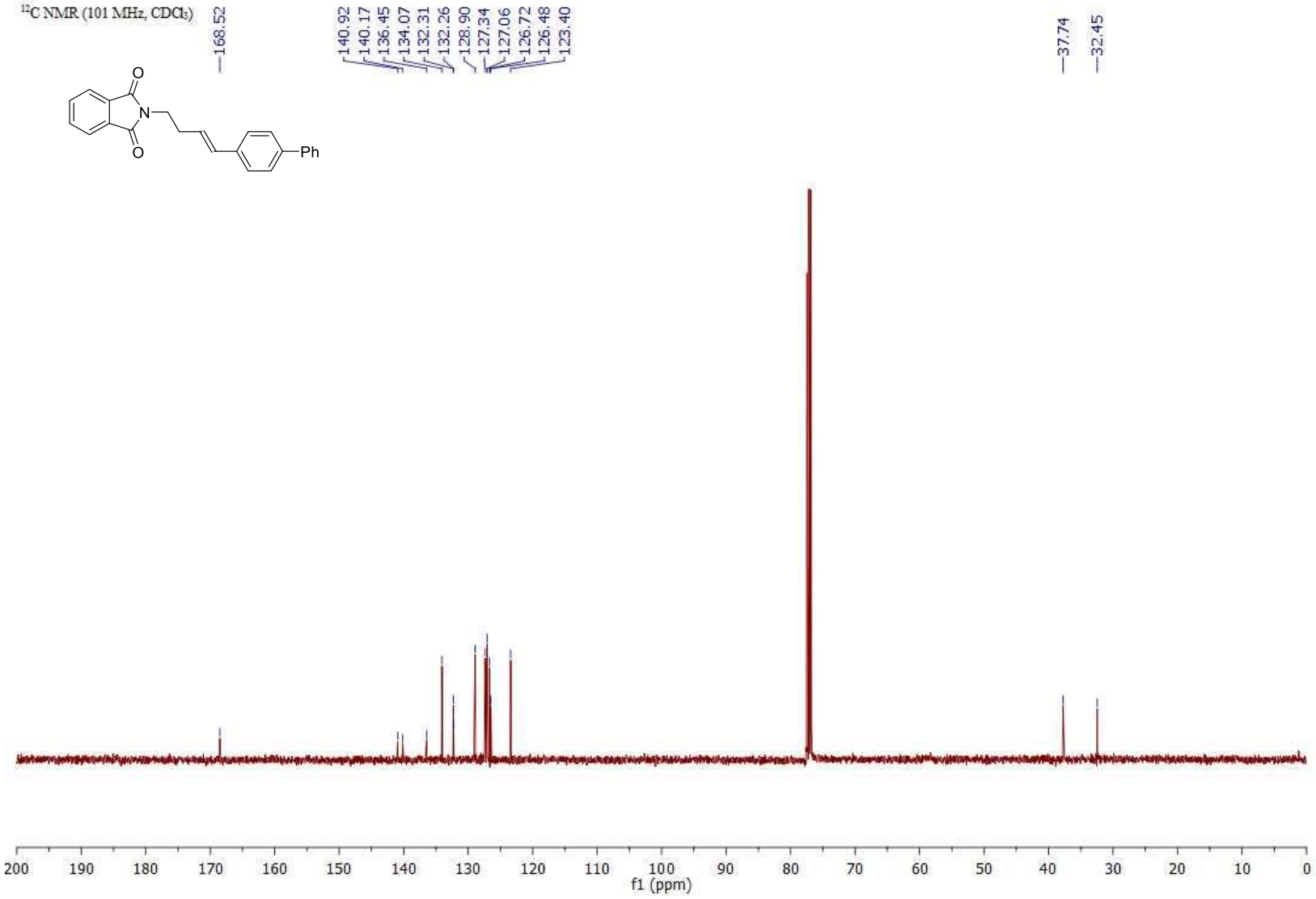
(*E*)-2-(4-(3-Chlorophenyl)but-3-en-1-yl)isoindoline-1,3-dione, ^{13}C NMR 126 MHz $CDCl_3$



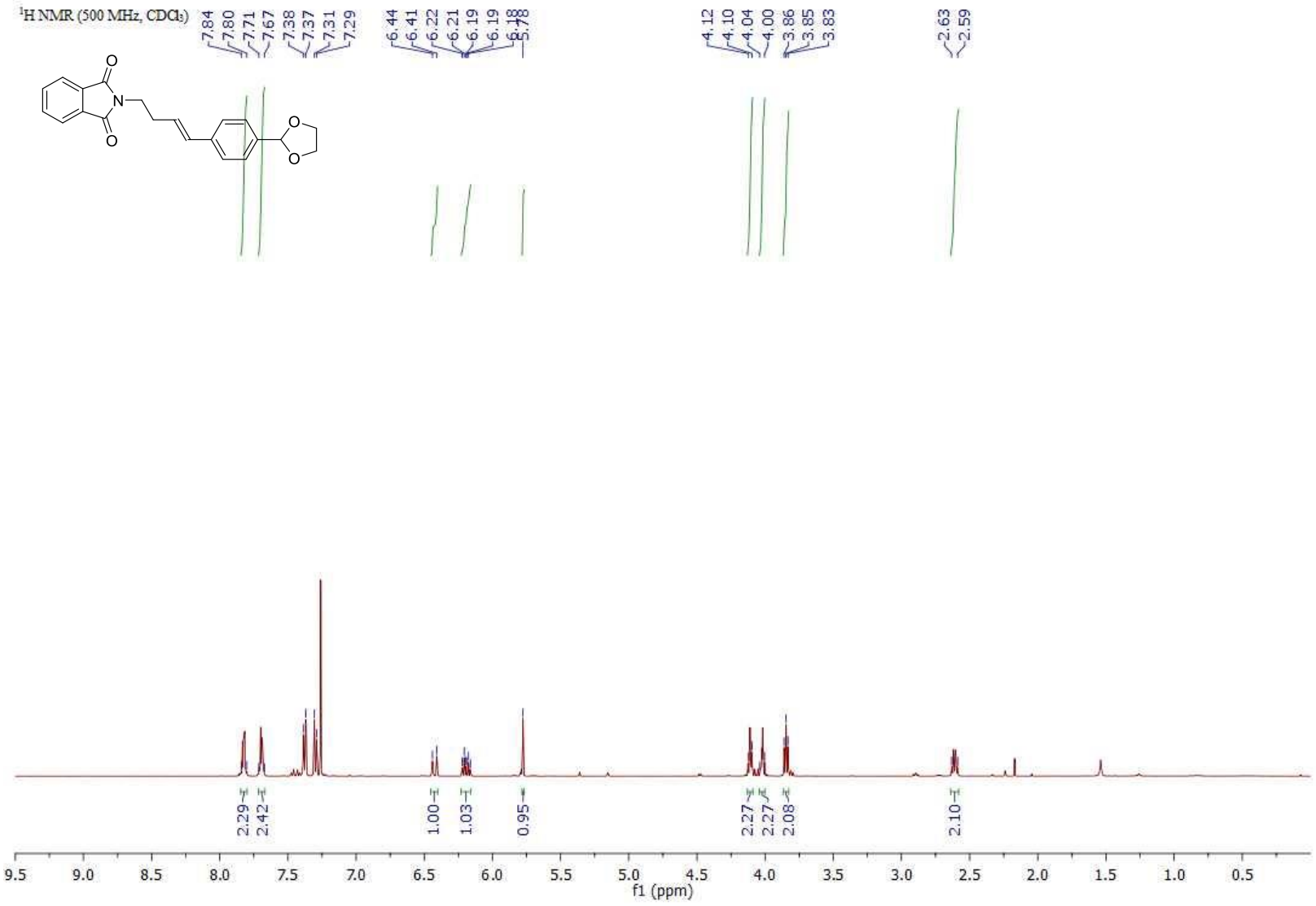
(*E*)-2-(4-([1,1'-Biphenyl]-4-yl)but-3-en-1-yl)isoindoline-1,3-dione, ^{1}H NMR 400 MHz $CDCl_3$



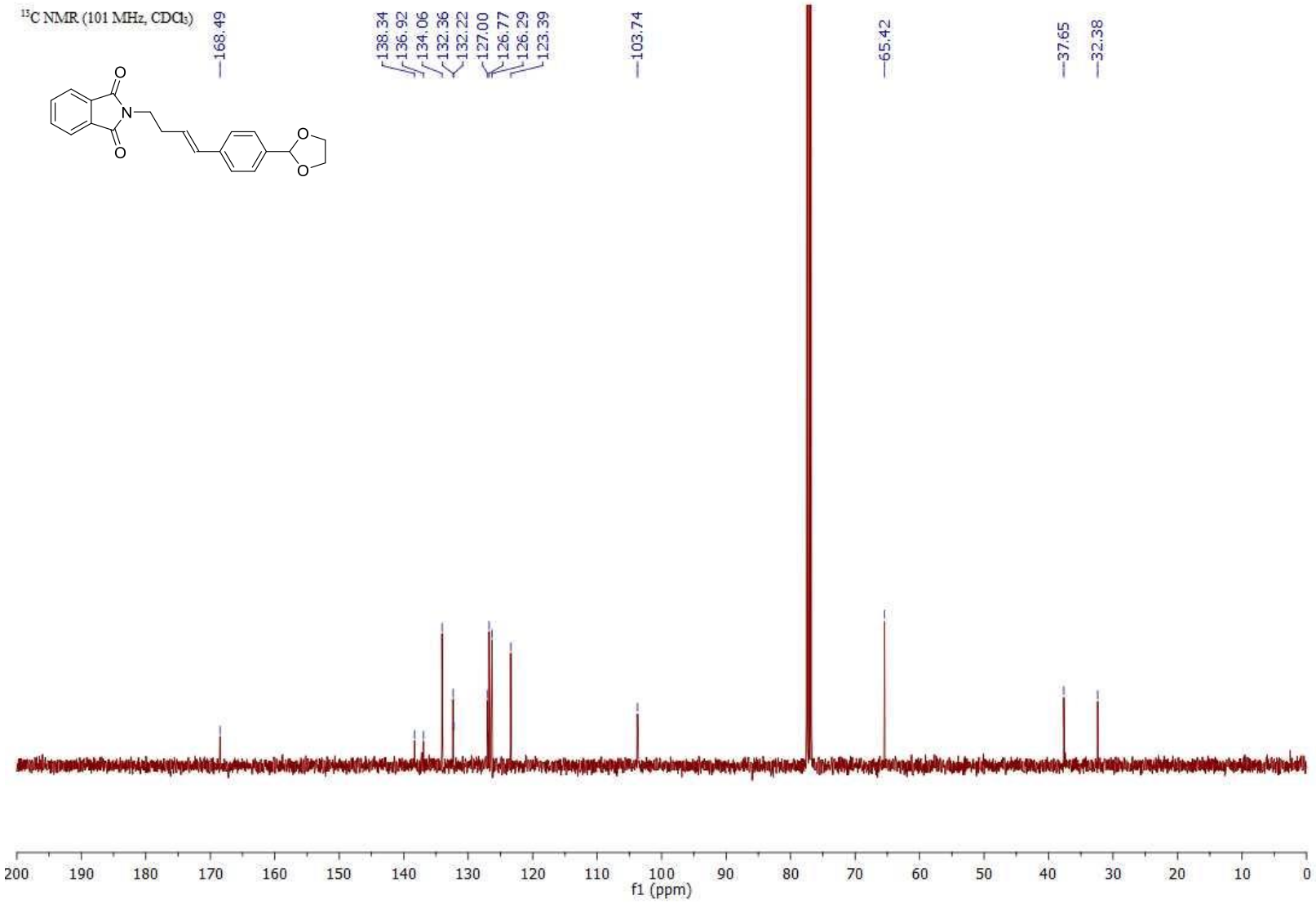
(*E*)-2-(4-([1,1'-Biphenyl]-4-yl)but-3-en-1-yl)isoindoline-1,3-dione, ^{13}C NMR 101 MHz $CDCl_3$



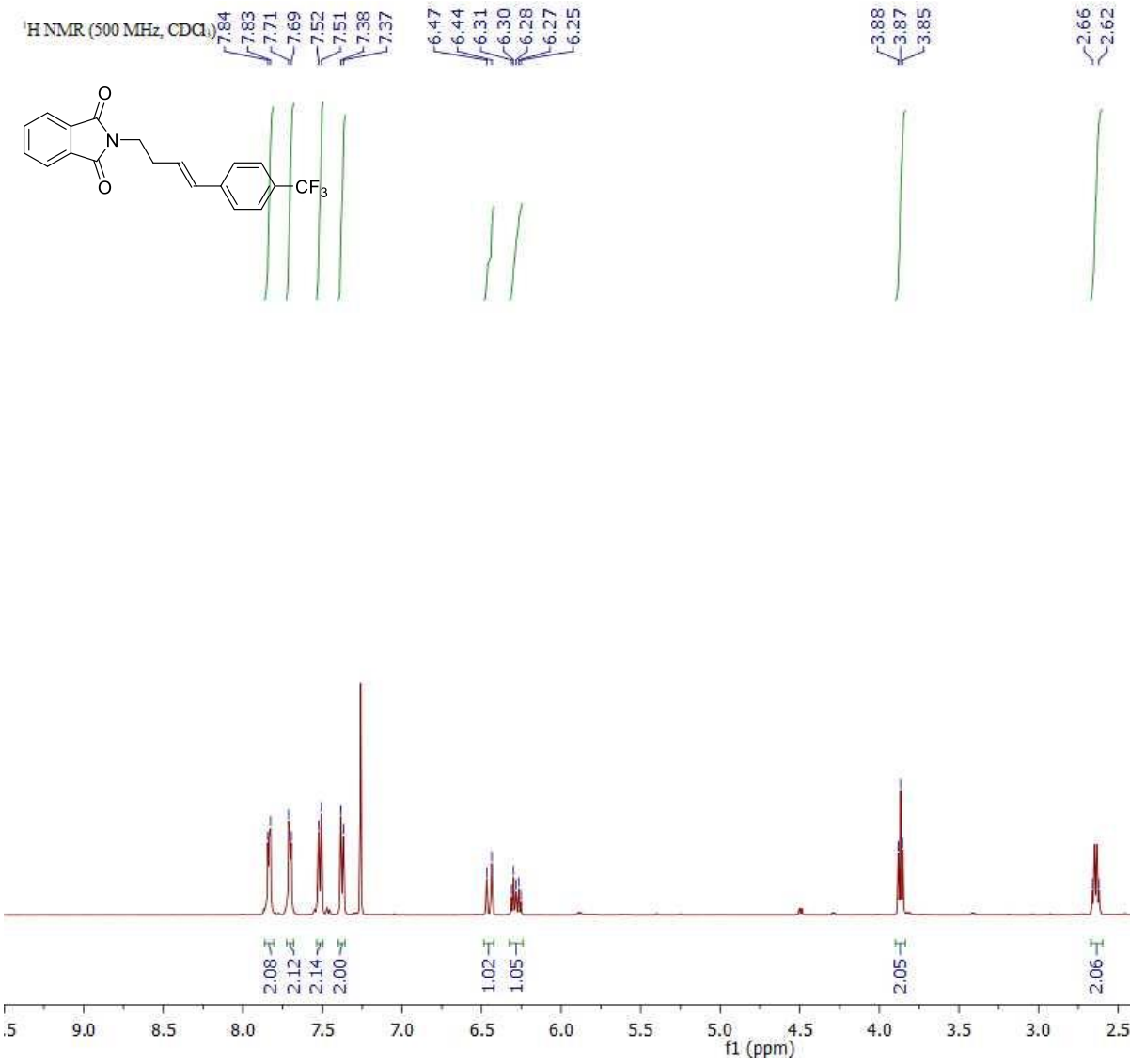
(*E*)-2-(4-(4-(1,3-Dioxalan-2-yl)phenyl)but-3-en-1-yl)isoindoline-1,3-dione, ^{1}H NMR 500 MHz $CDCl_3$



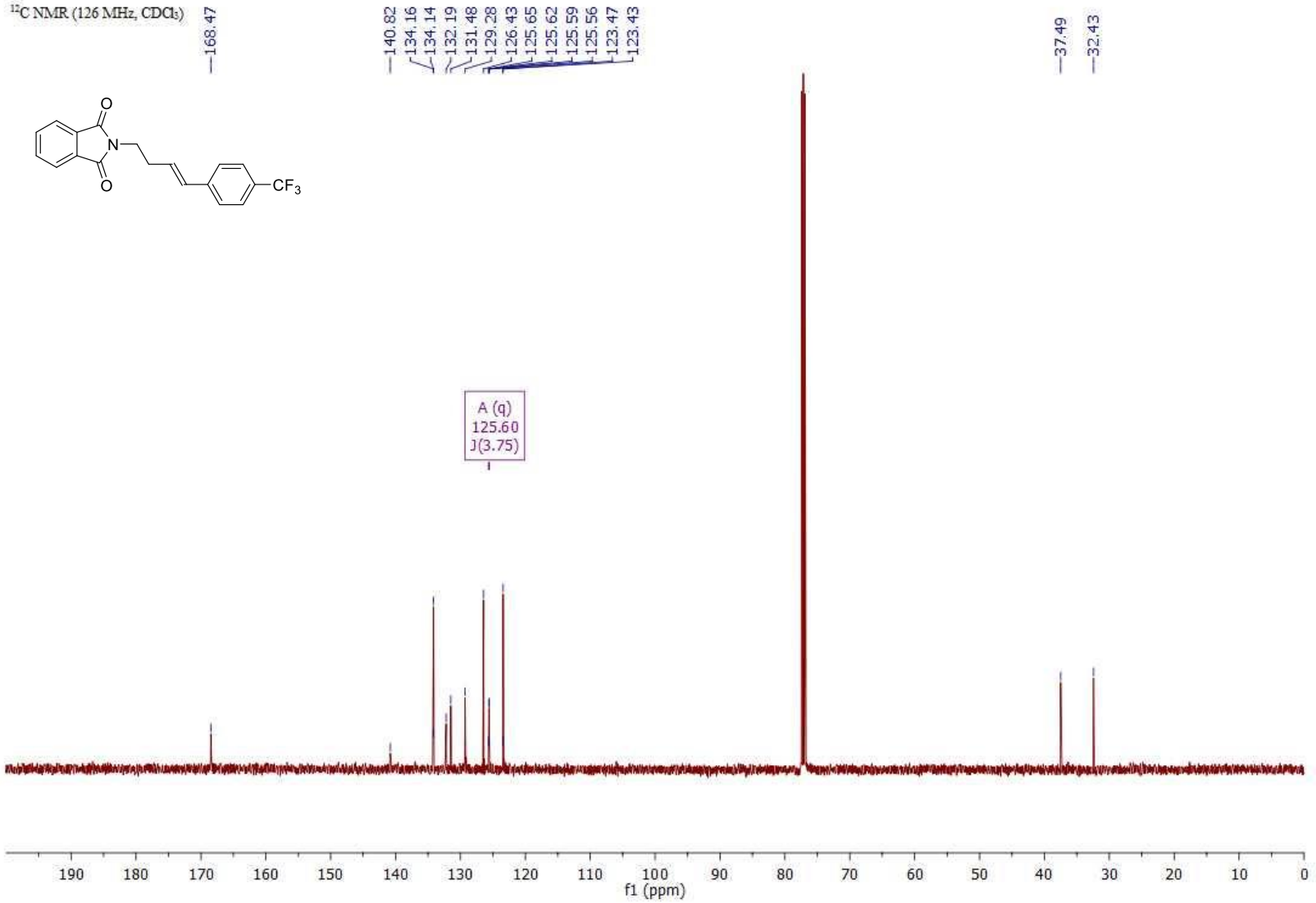
(*E*)-2-(4-(4-(1,3-Dioxalan-2-yl)phenyl)but-3-en-1-yl)isoindoline-1,3-dione, ^{13}C NMR 101 MHz $CDCl_3$



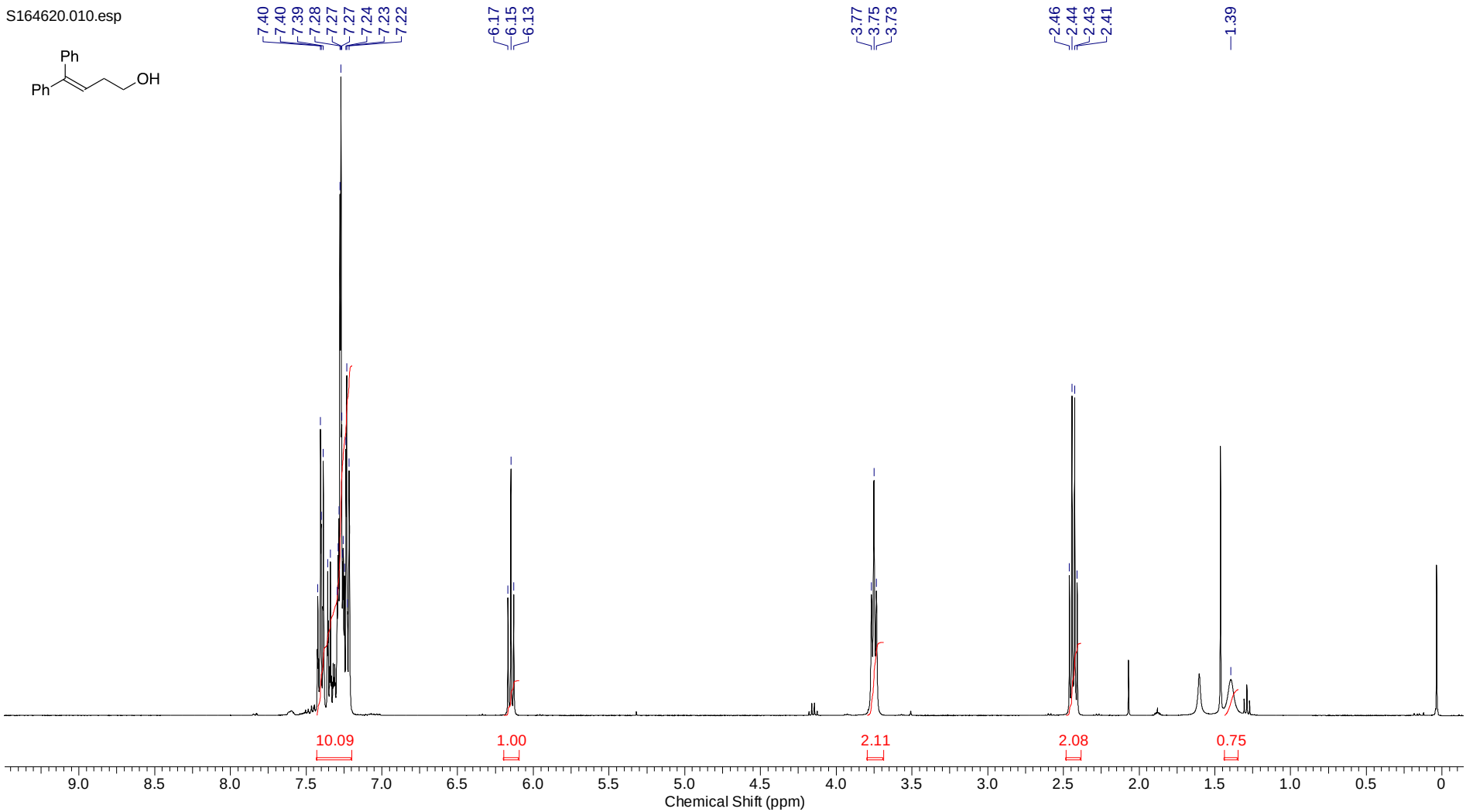
(*E*)-2-(4-(4-Trifluoromethyl)phenyl)but-3-en-1-yl)isoindoline-1,3-dione, ^{1}H NMR 500 MHz $CDCl_3$



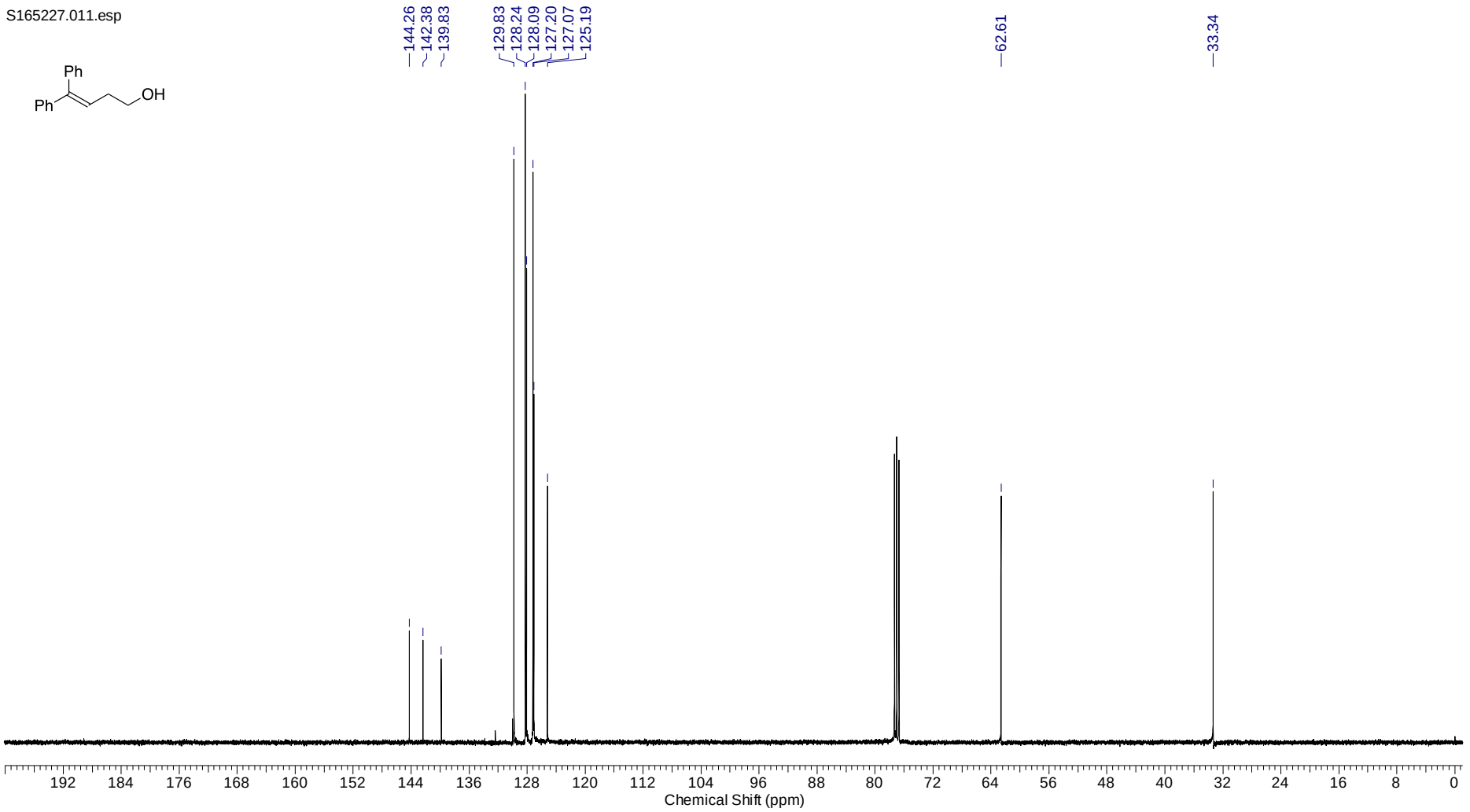
(*E*)-2-(4-(4-Trifluoromethyl)phenyl)but-3-en-1-yl)isoindoline-1,3-dione, ^{13}C NMR 126 MHz $CDCl_3$



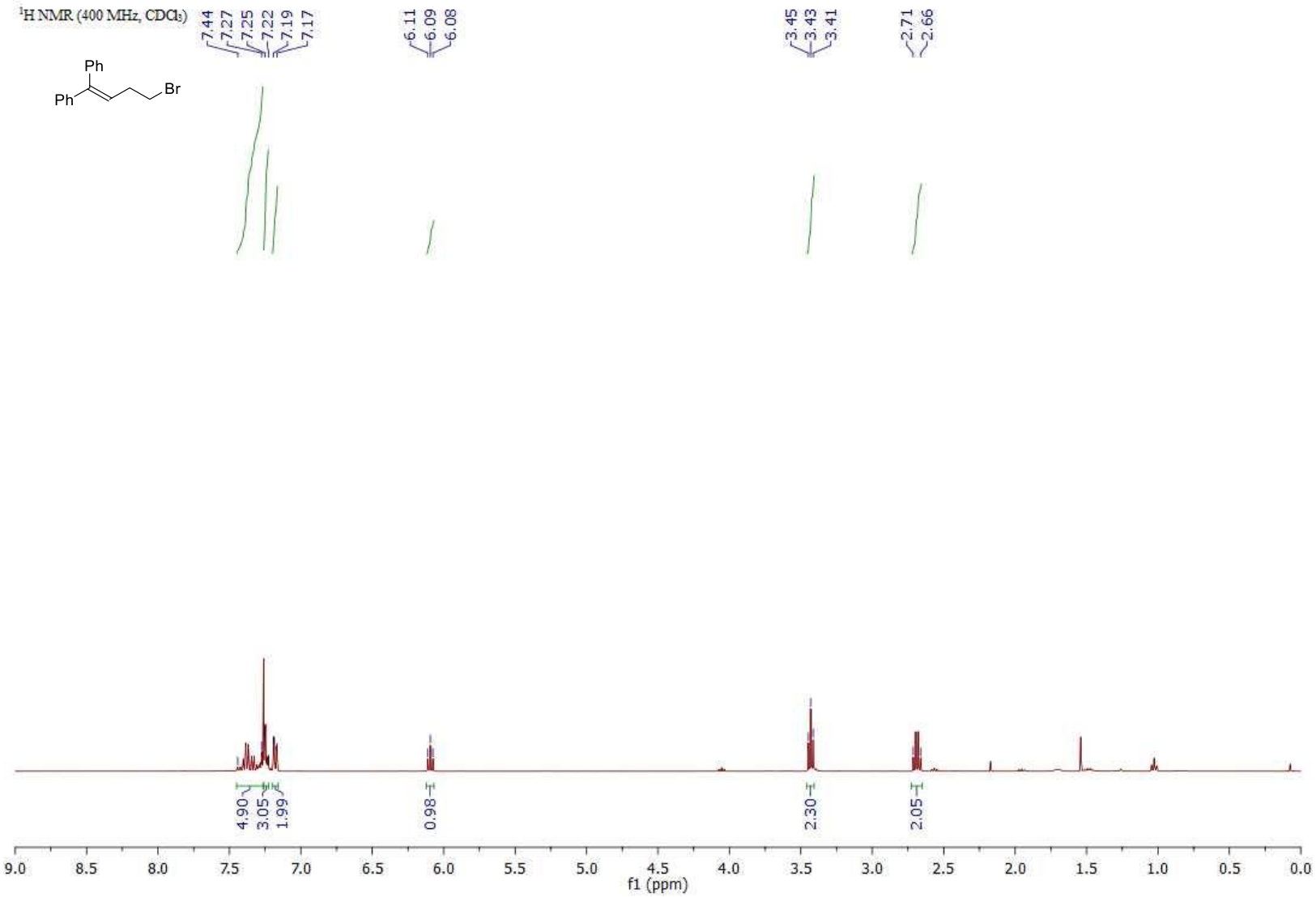
4,4-Diphenylbut-3-en-1-ol, ^{1}H NMR 400 MHz $CDCl_3$



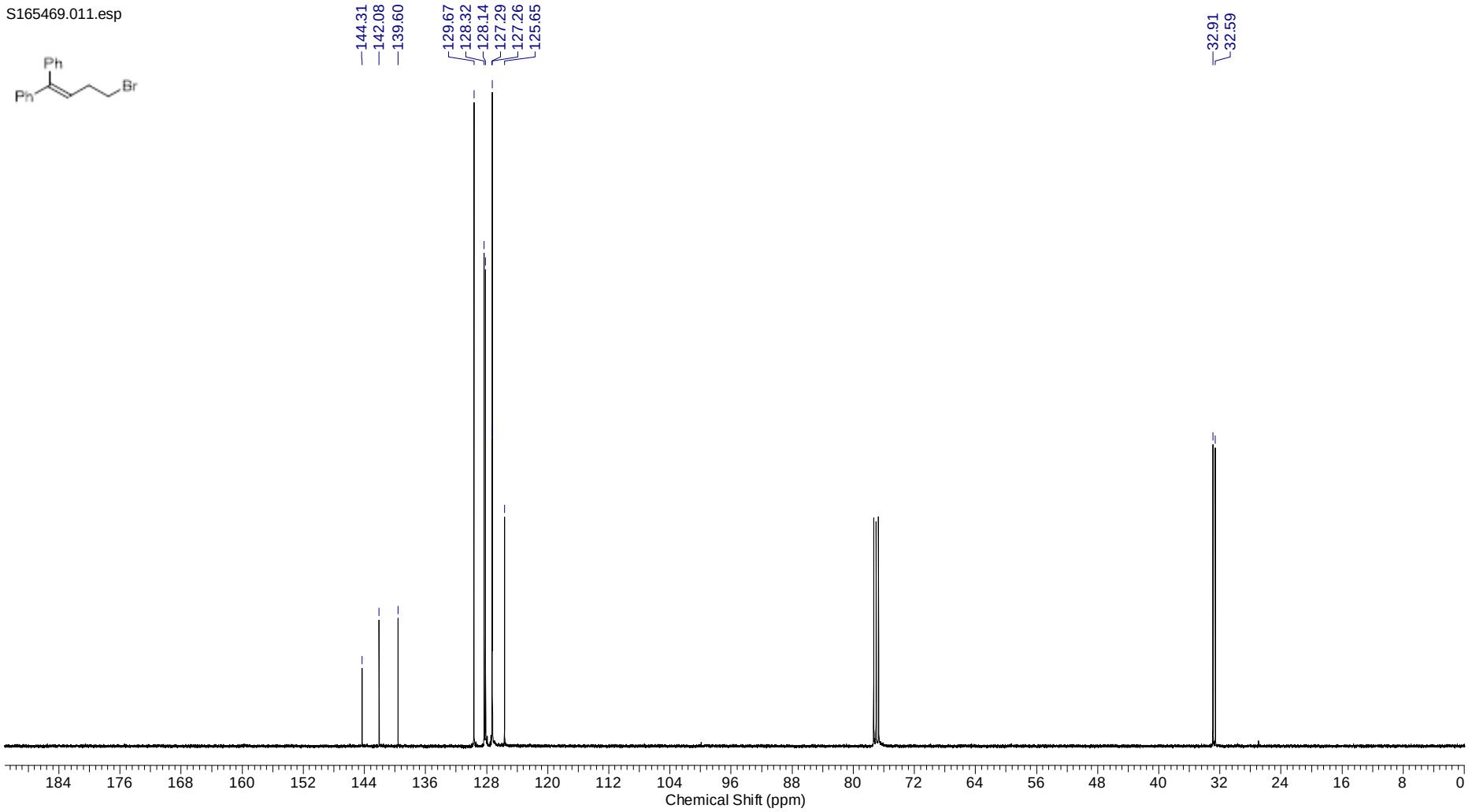
4,4-Diphenylbut-3-en-1-ol, ^{13}C NMR 100 MHz $CDCl_3$



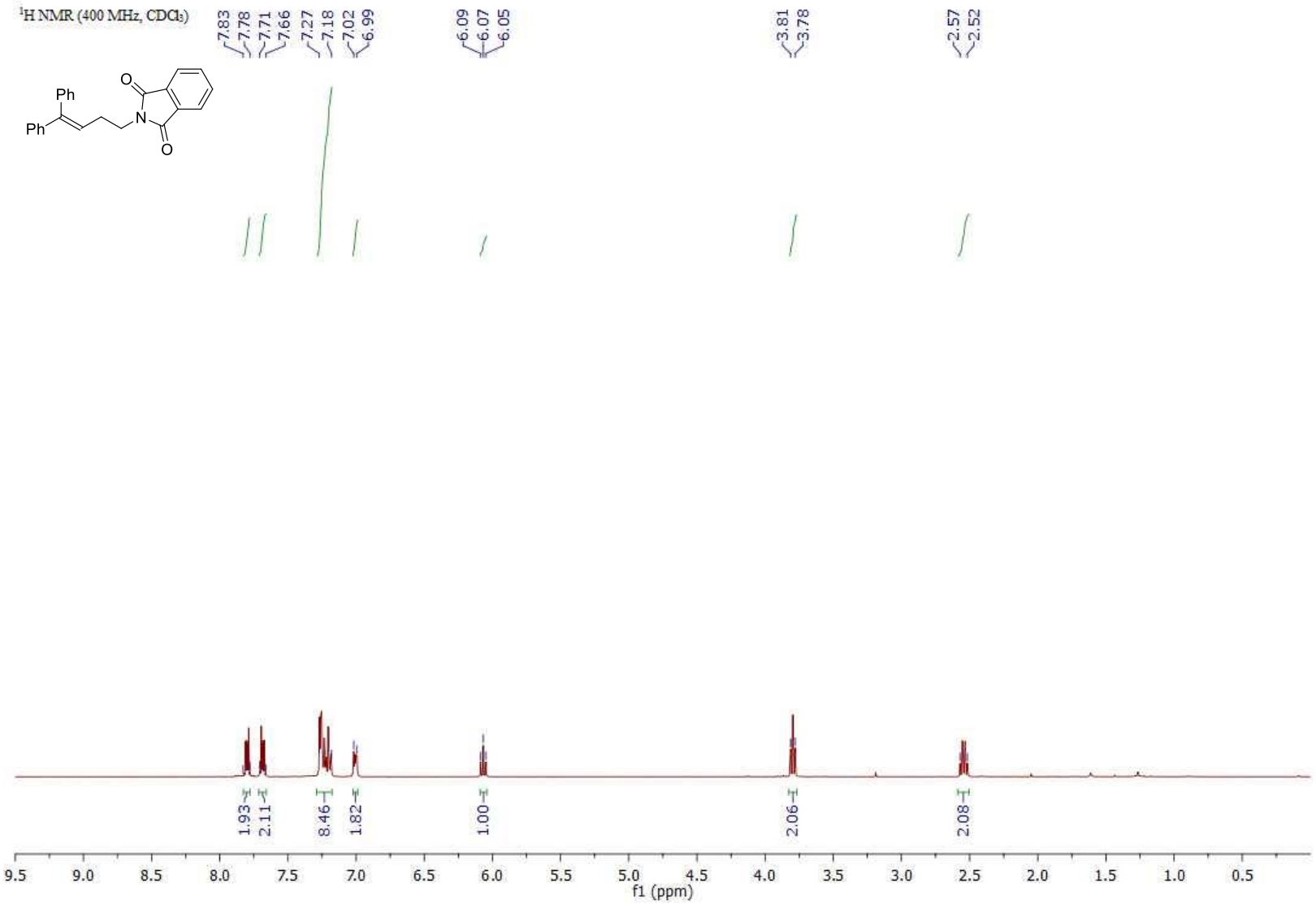
(4-Bromobut-1-ene-1,1diyl)dibenzene, ^{1}H NMR 400 MHz $CDCl_3$



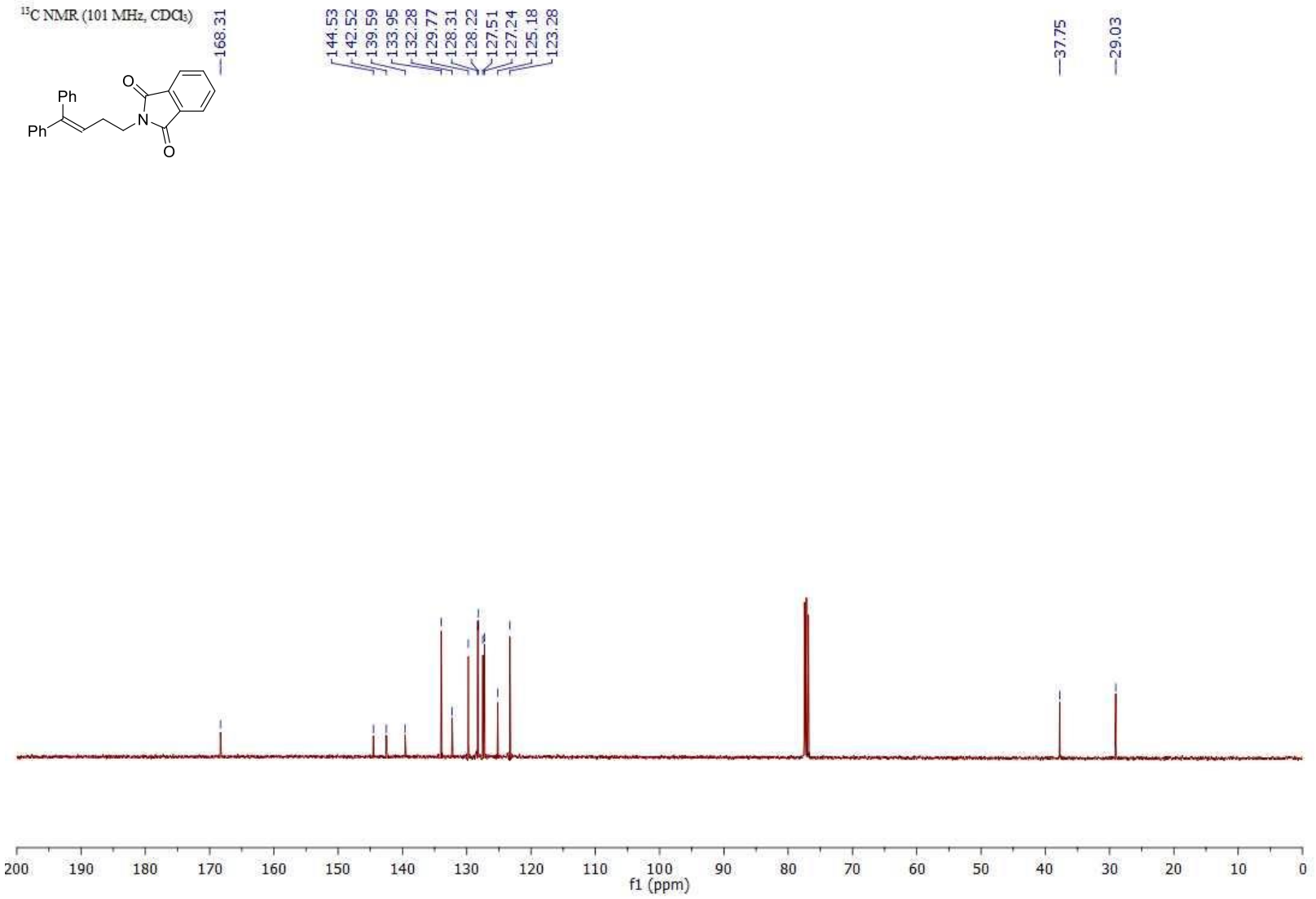
(4-Bromobut-1-ene-1,1diyl)dibenzene, ^{13}C NMR 101 MHz $CDCl_3$



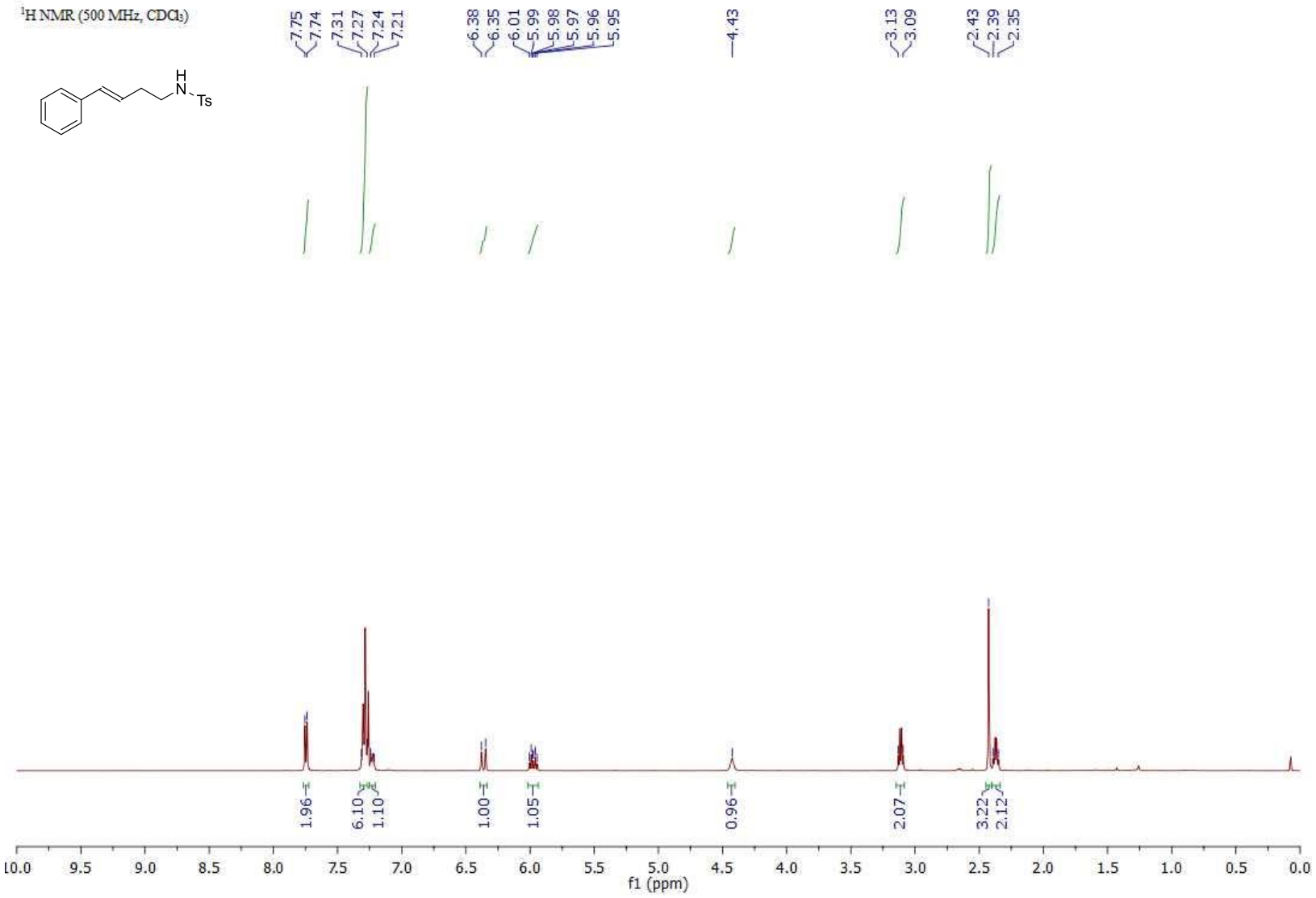
2-(4,4-Diphenylbut-3-en-1-yl)isoindoline-1,3-dione, ^{1}H NMR 400 MHz $CDCl_3$



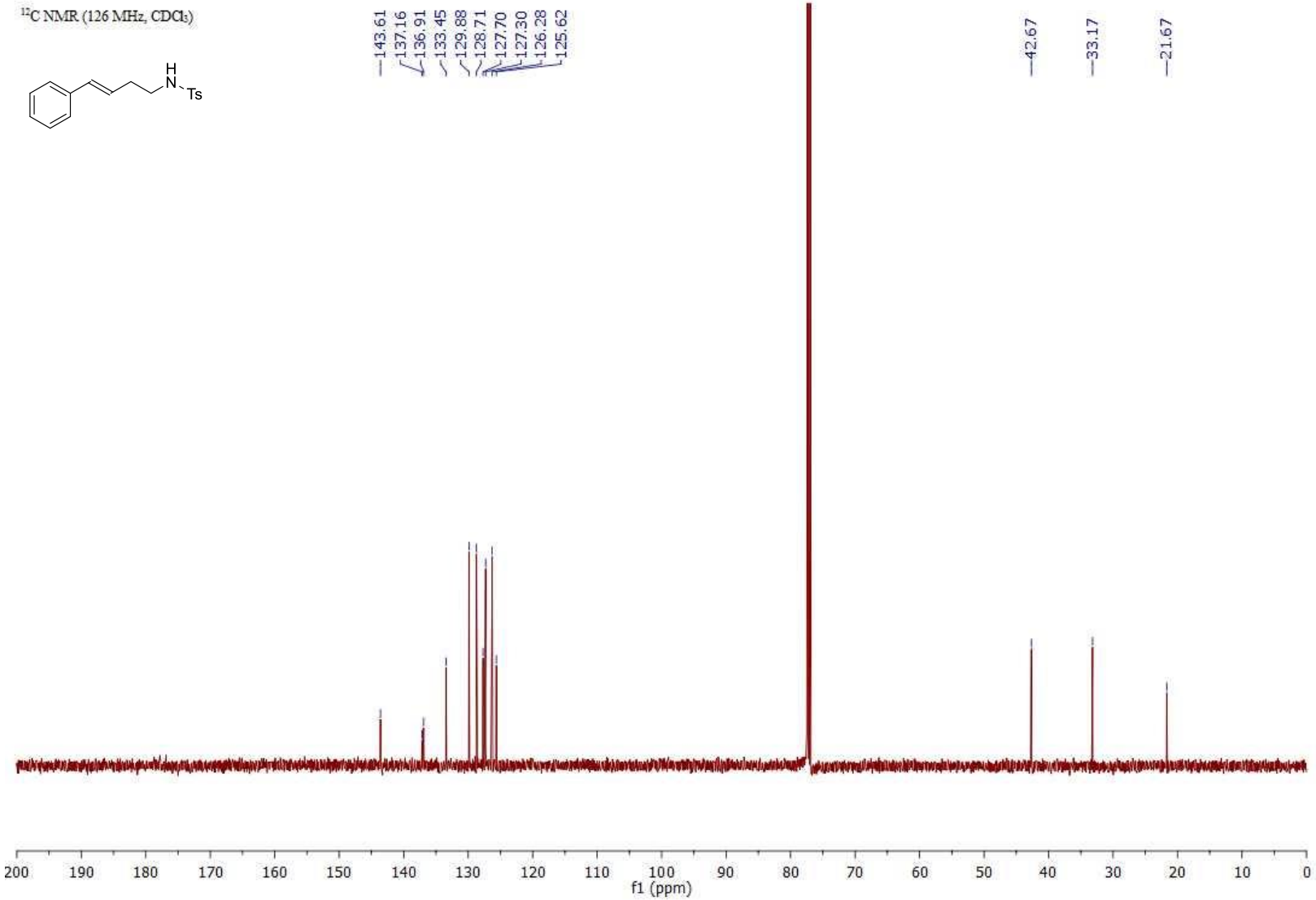
2-(4,4-Diphenylbut-3-en-1-yl)isoindoline-1,3-dione, ^{13}C NMR 101 MHz $CDCl_3$



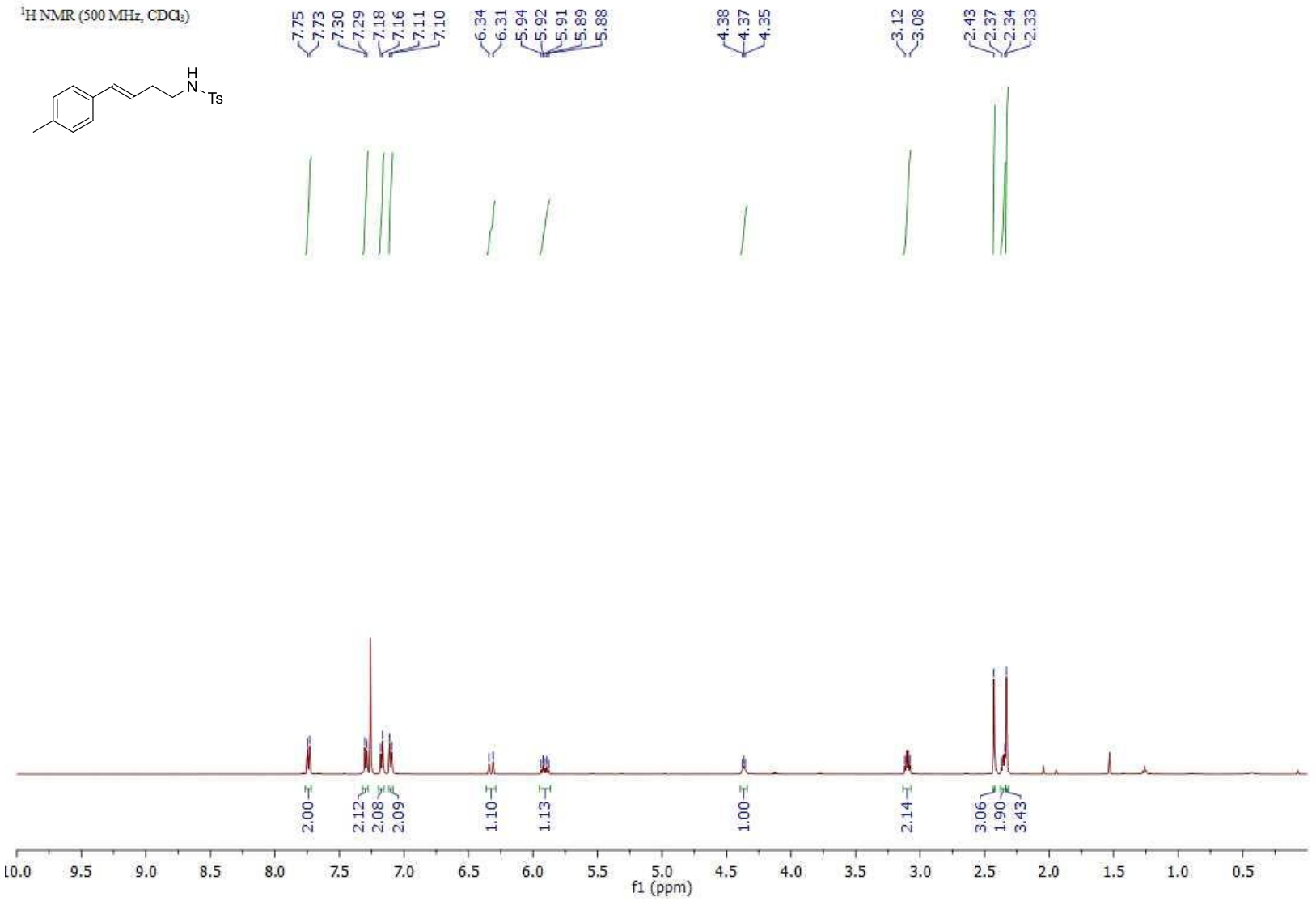
(*E*)-4-Methyl-*N*-(4-phenylbut-3-en-1-yl)benzenesulfonamide 8a, ^{1}H NMR 500 MHz $CDCl_3$



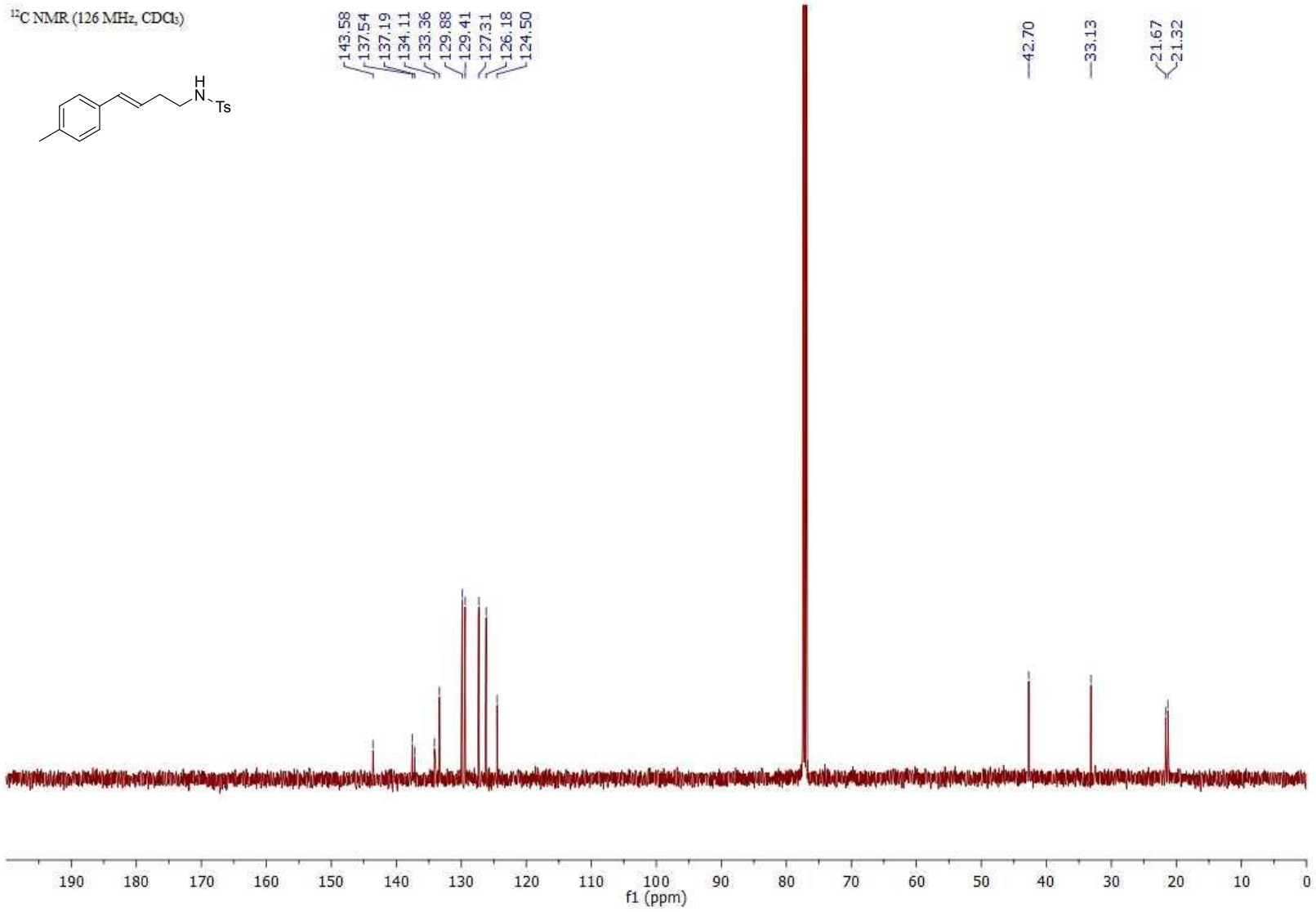
(*E*)-4-Methyl-*N*-(4-phenylbut-3-en-1-yl)benzenesulfonamide 8a, ^{13}C NMR 126 MHz $CDCl_3$



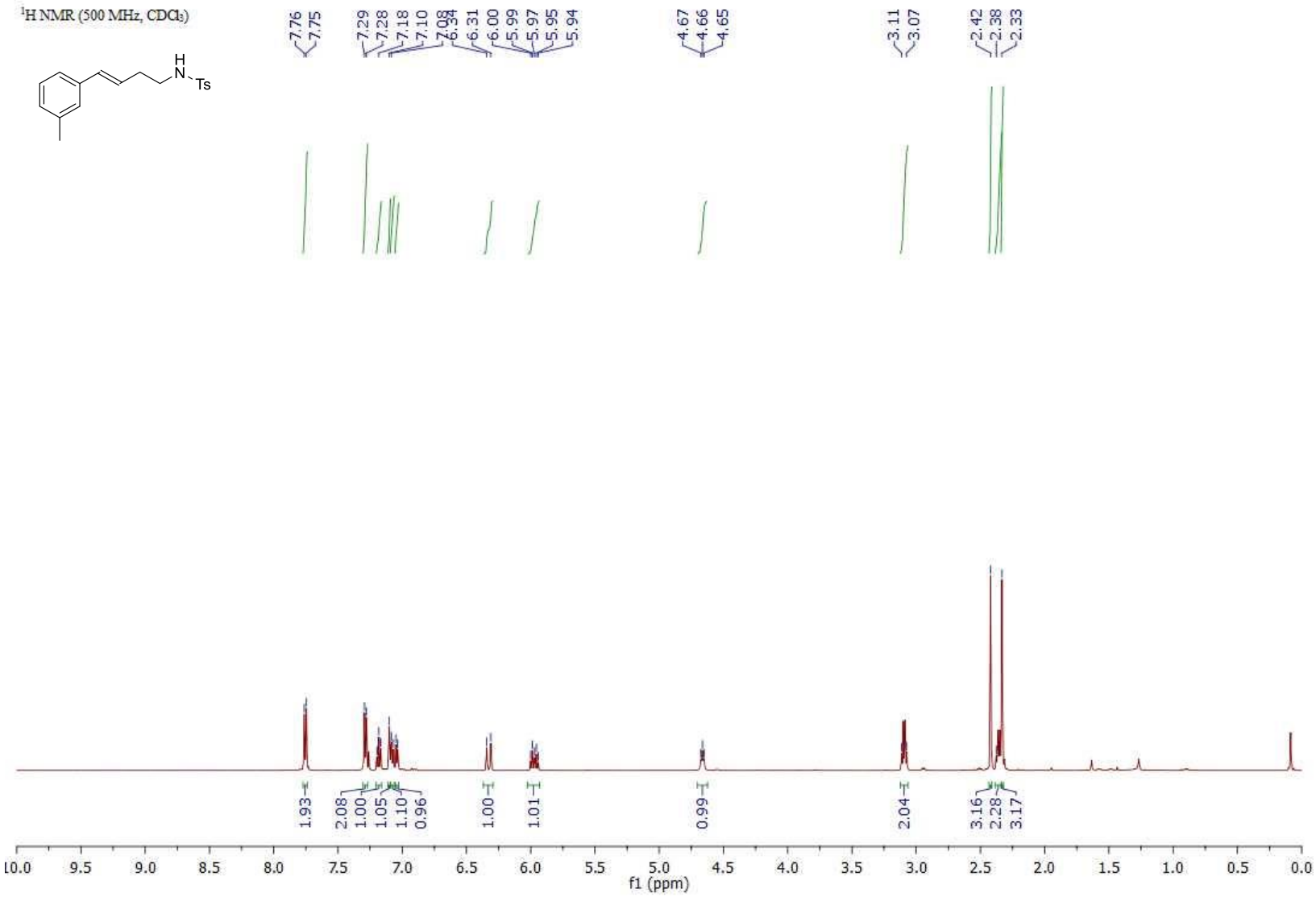
(*E*)-4-Methyl-*N*-(4-(*p*-tolyl)but-3-en-1-yl)benzenesulfonamide, ^{1}H NMR 500 MHz $CDCl_3$



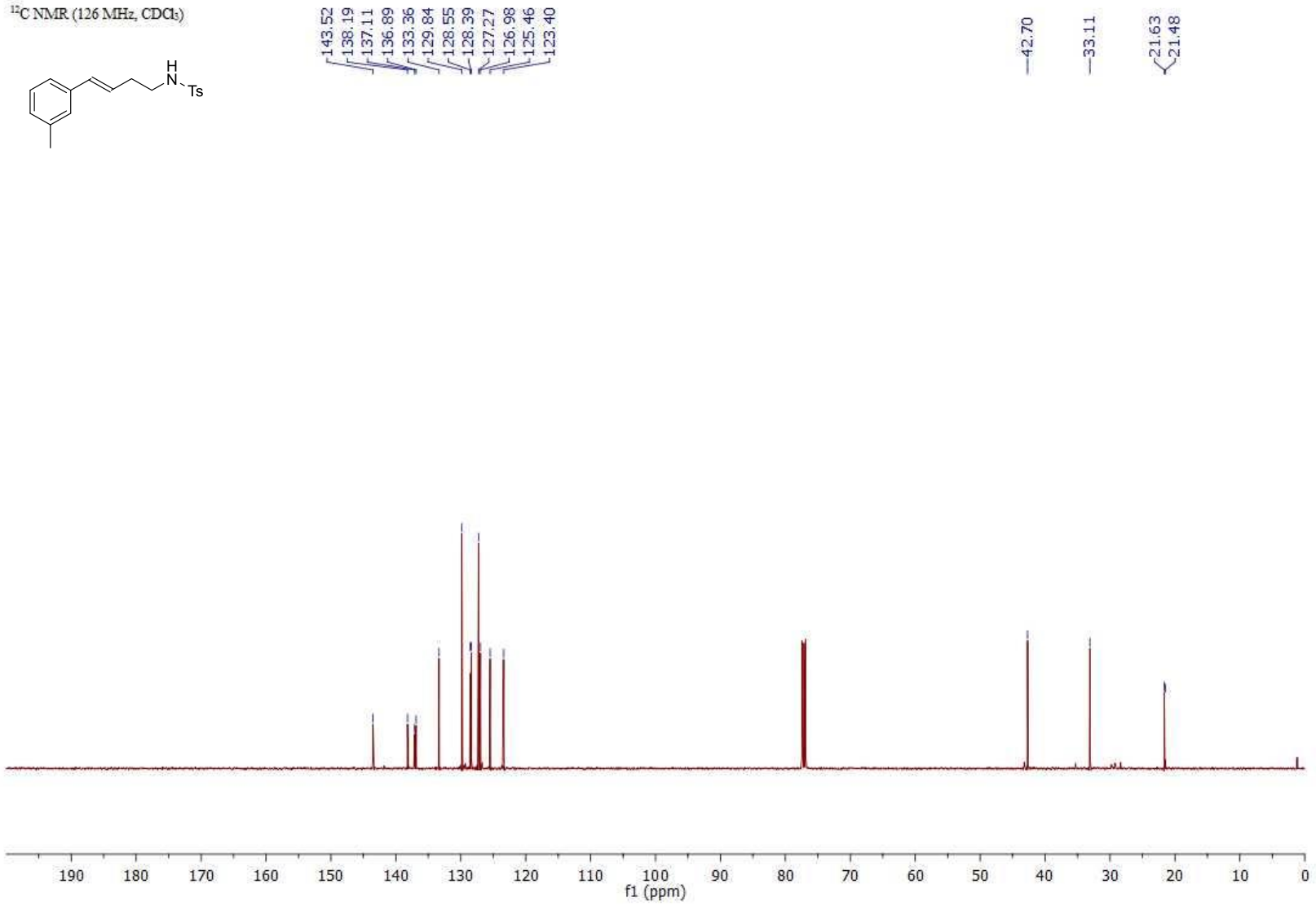
(*E*)-4-Methyl-*N*-(4-(*p*-tolyl)but-3-en-1-yl)benzenesulfonamide, ^{13}C NMR 126 MHz $CDCl_3$



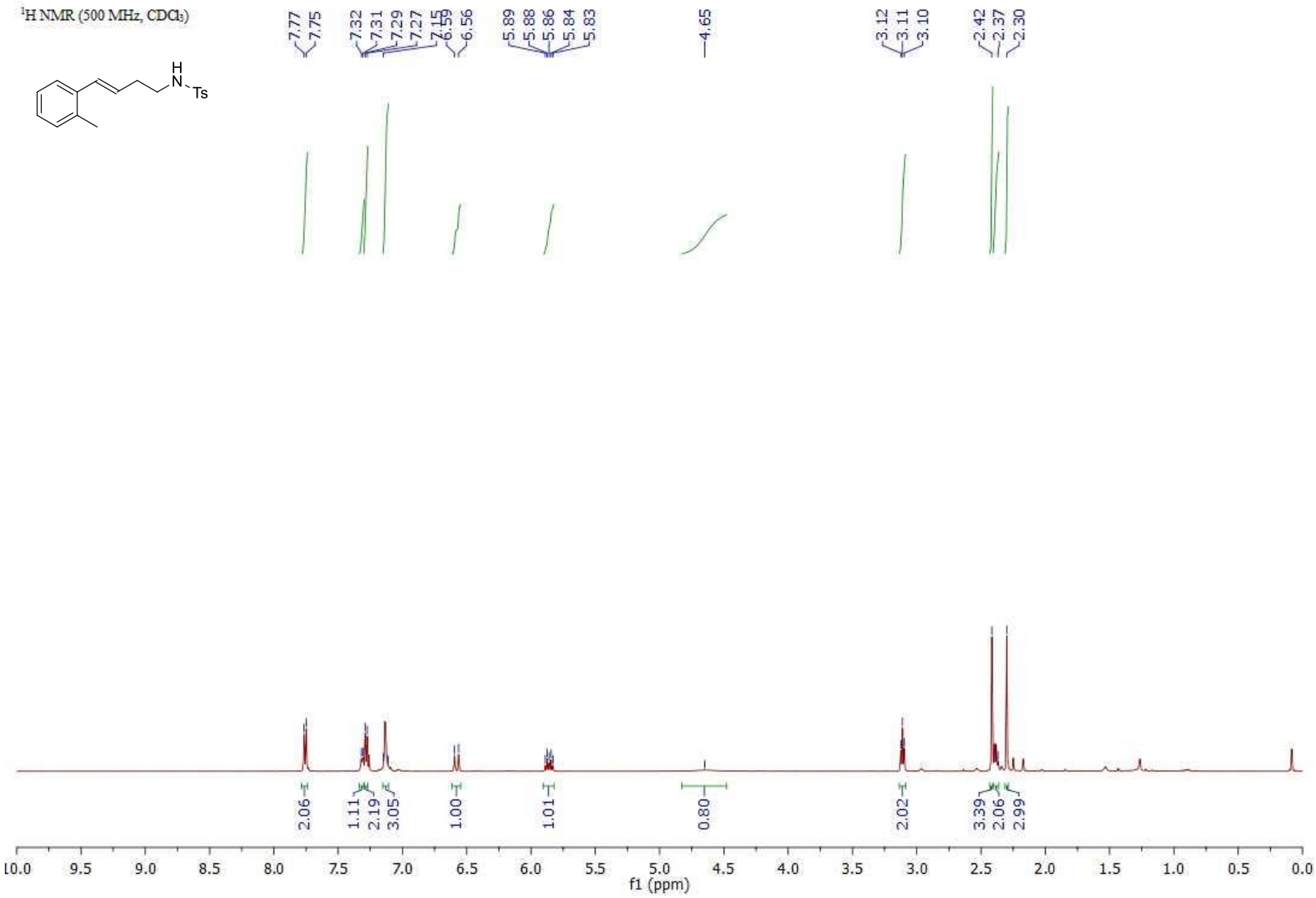
(*E*)-4-Methyl-*N*-(4-(*m*-tolyl)but-3-en-1-yl)benzenesulfonamide, ^{1}H NMR 500 MHz $CDCl_3$



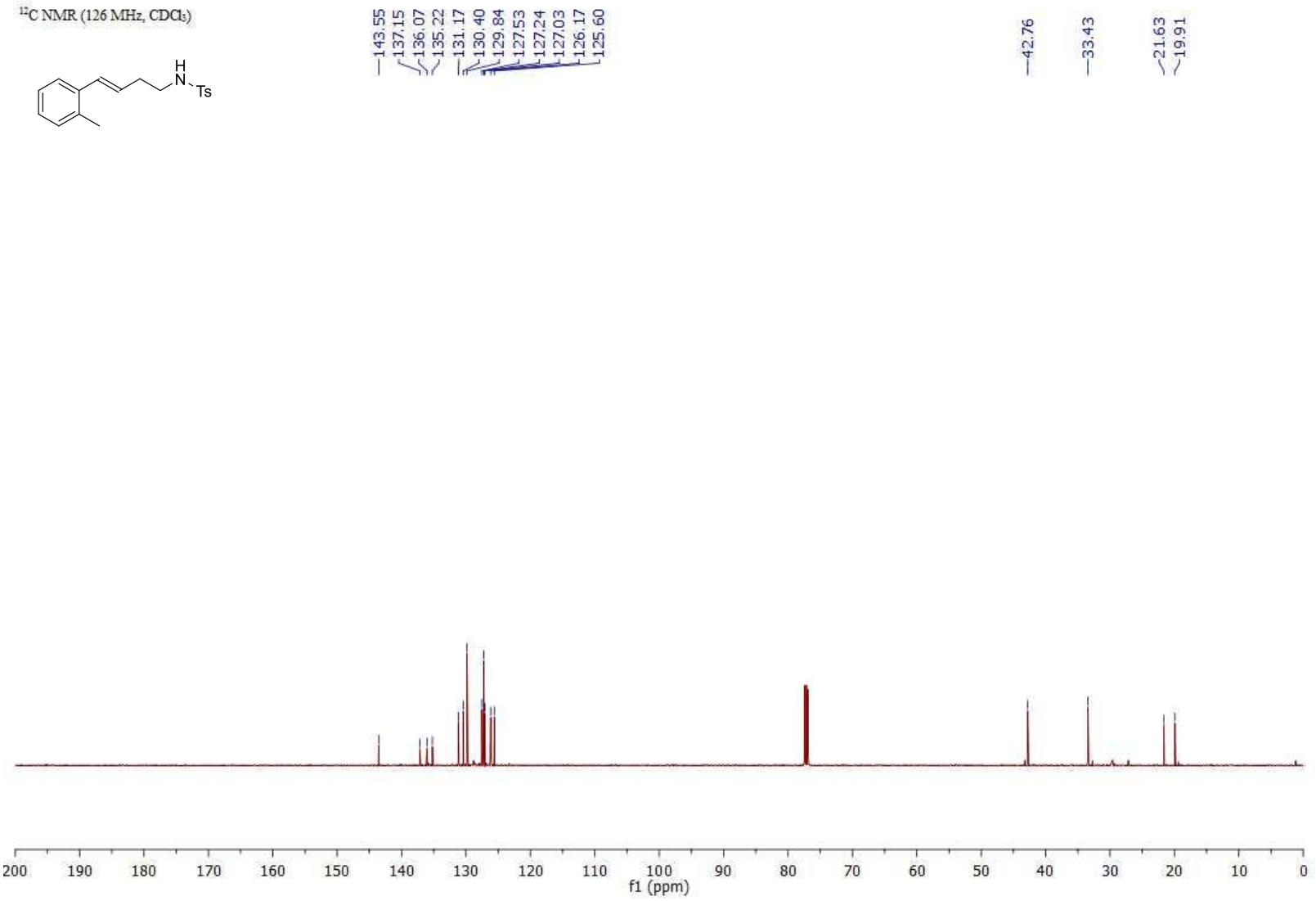
(*E*)-4-Methyl-*N*-(4-(*m*-tolyl)but-3-en-1-yl)benzenesulfonamide, ^{13}C NMR 126 MHz $CDCl_3$



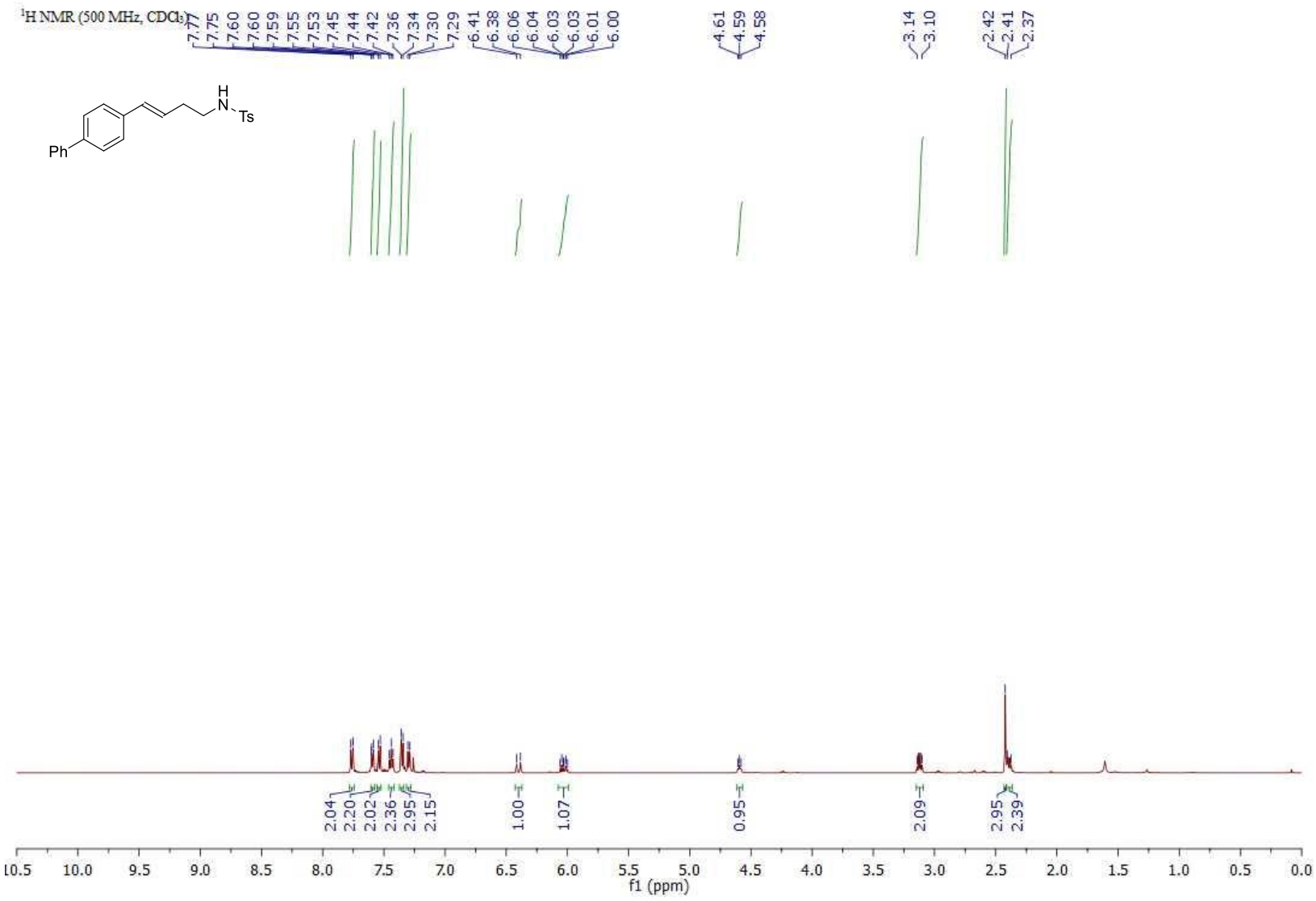
(*E*)-4-Methyl-*N*-(4-(*o*-tolyl)but-3-en-1-yl)benzenesulfonamide, ^{1}H NMR 500 MHz $CDCl_3$



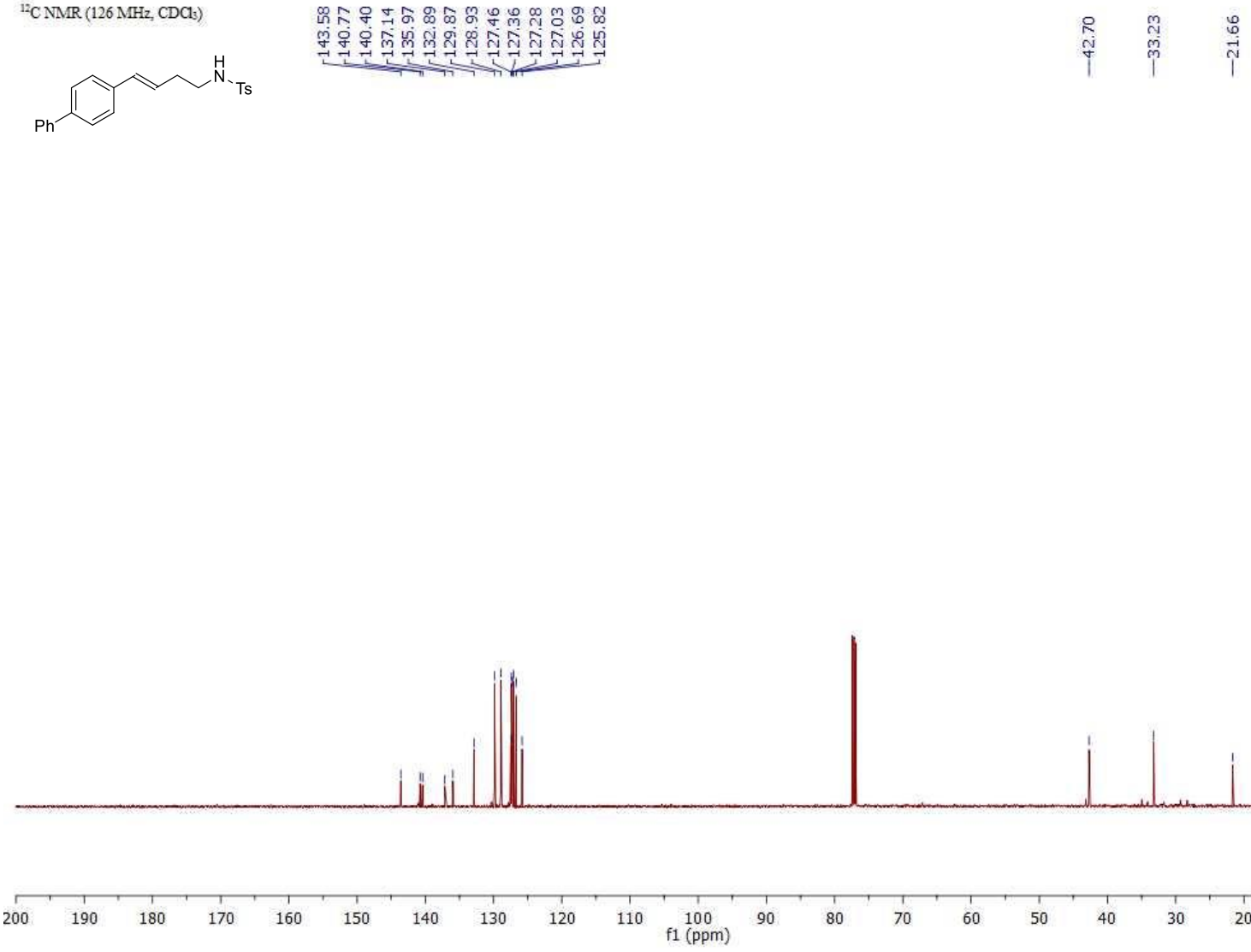
(*E*)-4-Methyl-*N*-(4-(*o*-tolyl)but-3-en-1-yl)benzenesulfonamide, ^{13}C NMR 126 MHz $CDCl_3$



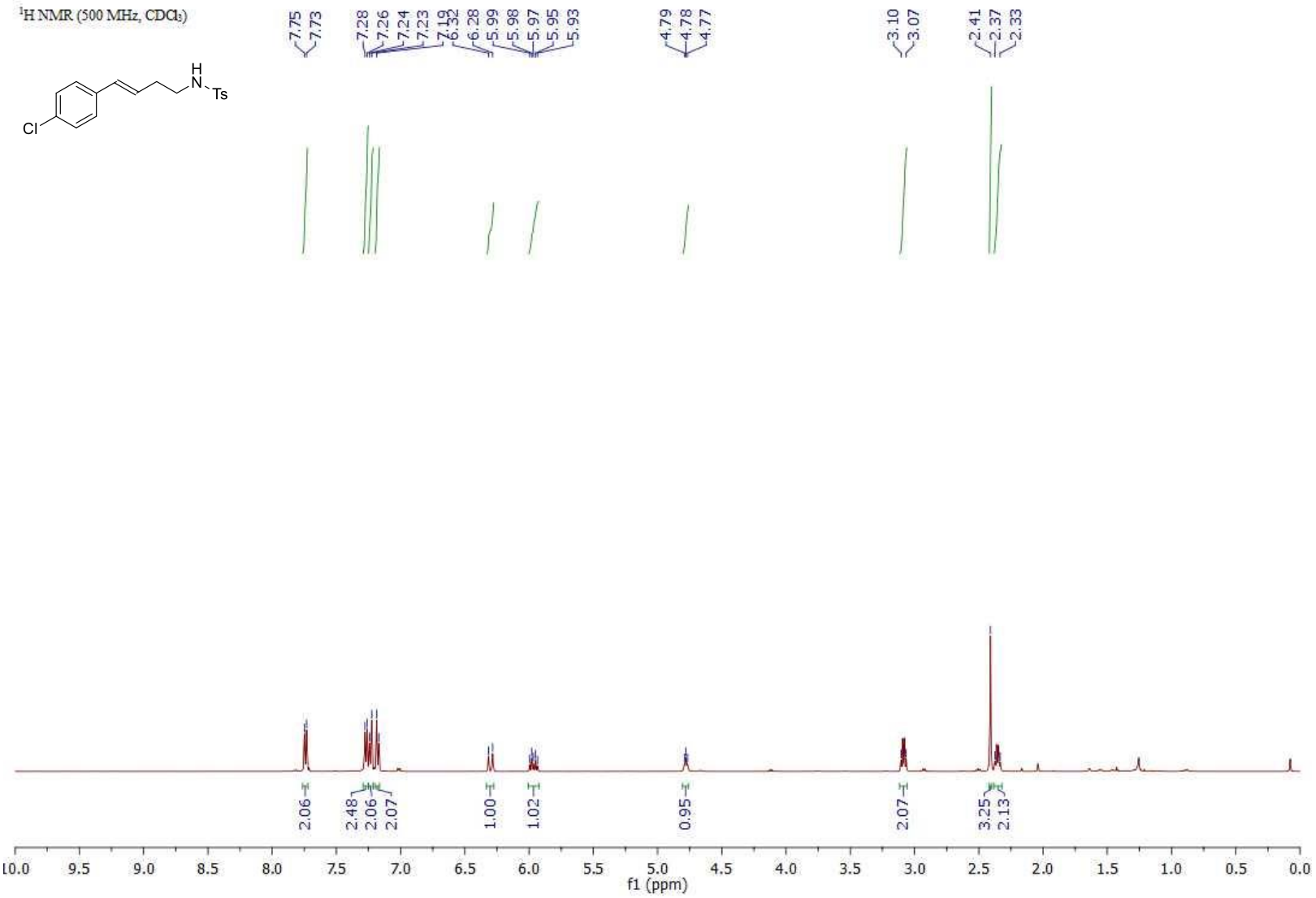
(*E*)-2-(4-([1,1'-Biphenyl]-4-yl)but-3-en-1-yl)-4-methylbenzenesulfonamide, ^{1}H NMR 500 MHz $CDCl_3$



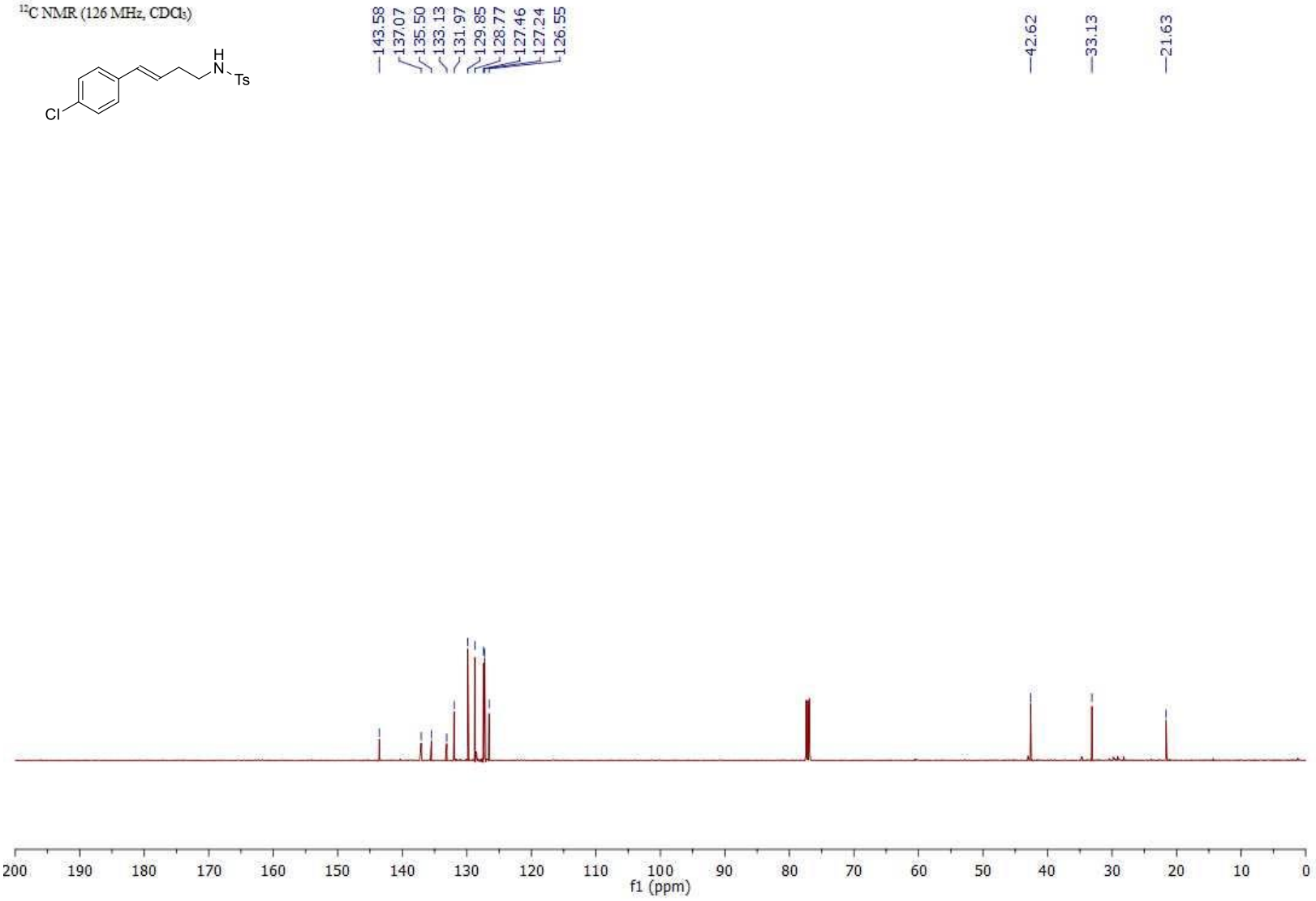
(*E*)-2-(4-([1,1'-Biphenyl]-4-yl)but-3-en-1-yl)-4-methylbenzenesulfonamide, ^{13}C NMR 126 MHz $CDCl_3$



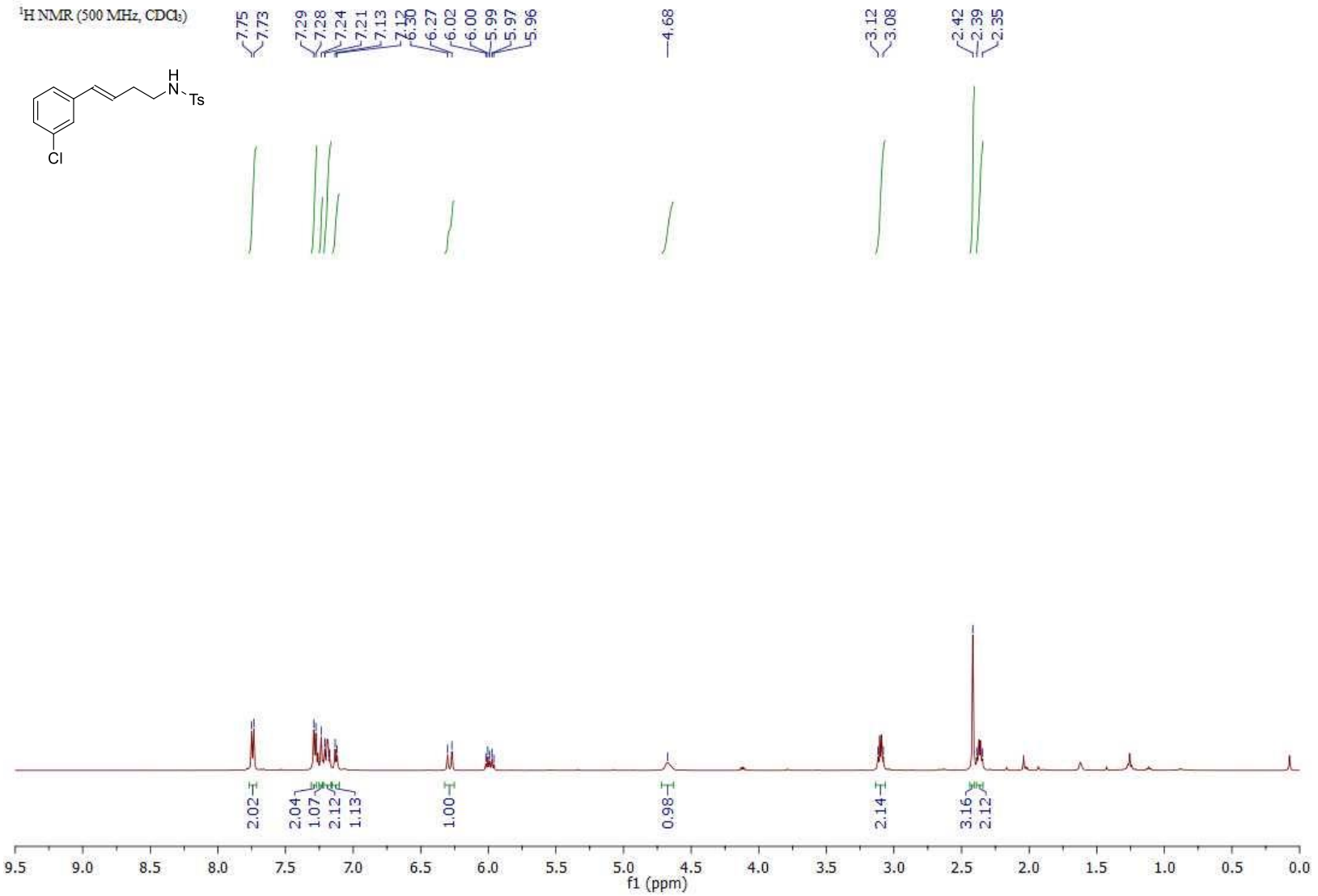
(*E*)-*N*-(4-(4-Chlorophenyl)but-3-en-1-yl)-4-methylbenzenesulfonamide, ^{1}H NMR 500 MHz $CDCl_3$



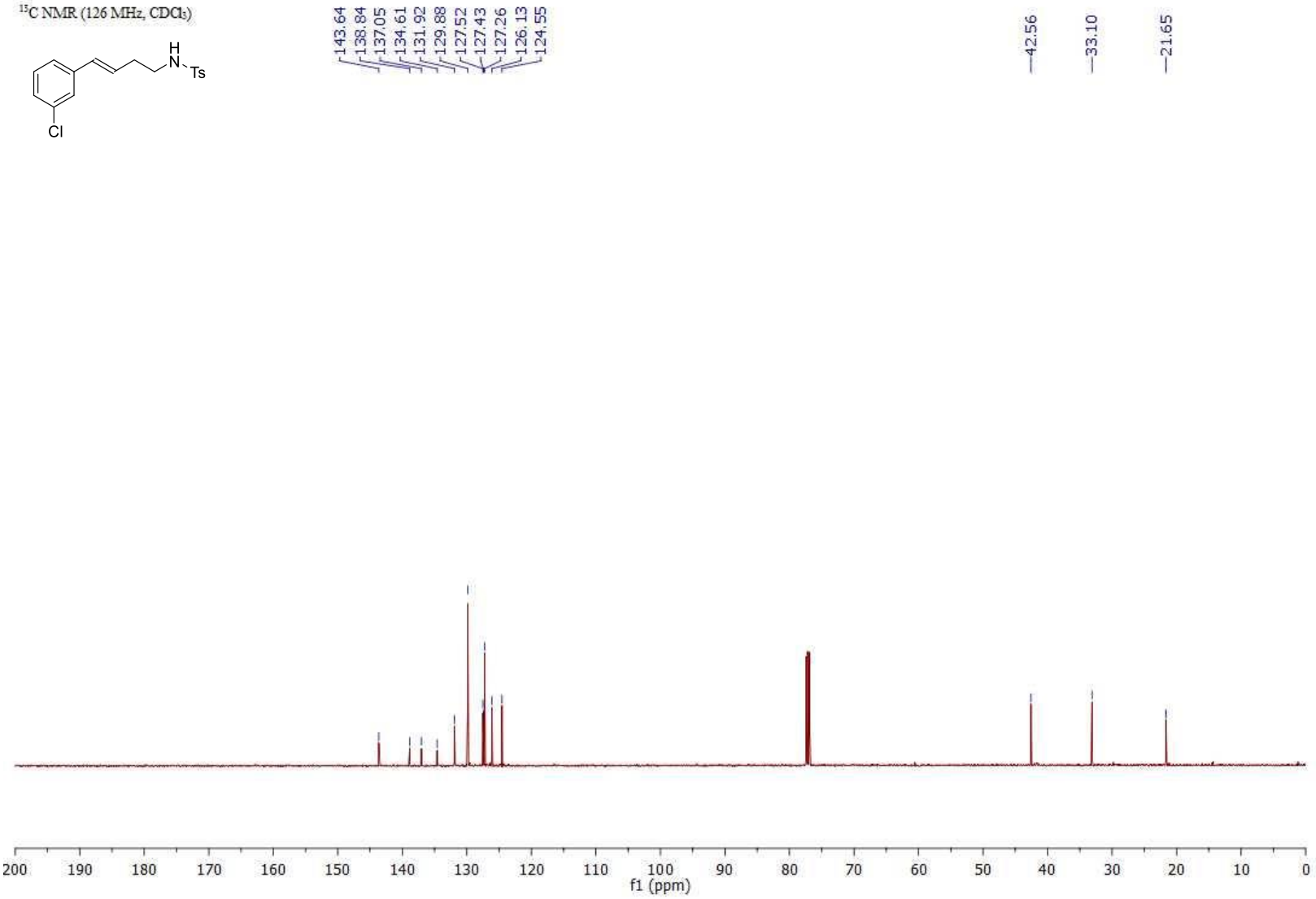
(*E*)-*N*-(4-(4-Chlorophenyl)but-3-en-1-yl)-4-methylbenzenesulfonamide, ^{13}C NMR 126 MHz $CDCl_3$



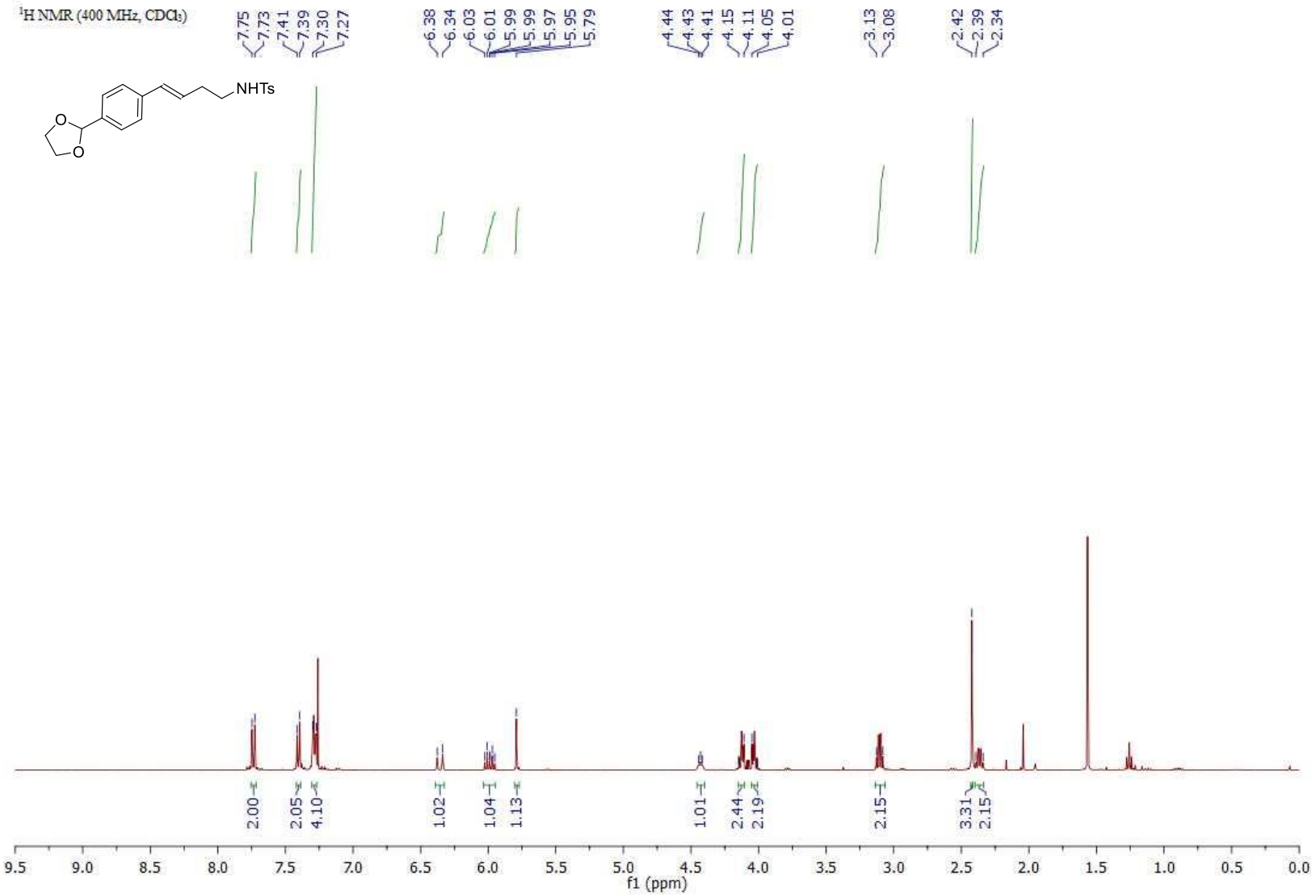
(*E*)-*N*-(4-(3-Chlorophenyl)but-3-en-1-yl)-4-methylbenzenesulfonamide, ^{1}H NMR 500 MHz $CDCl_3$



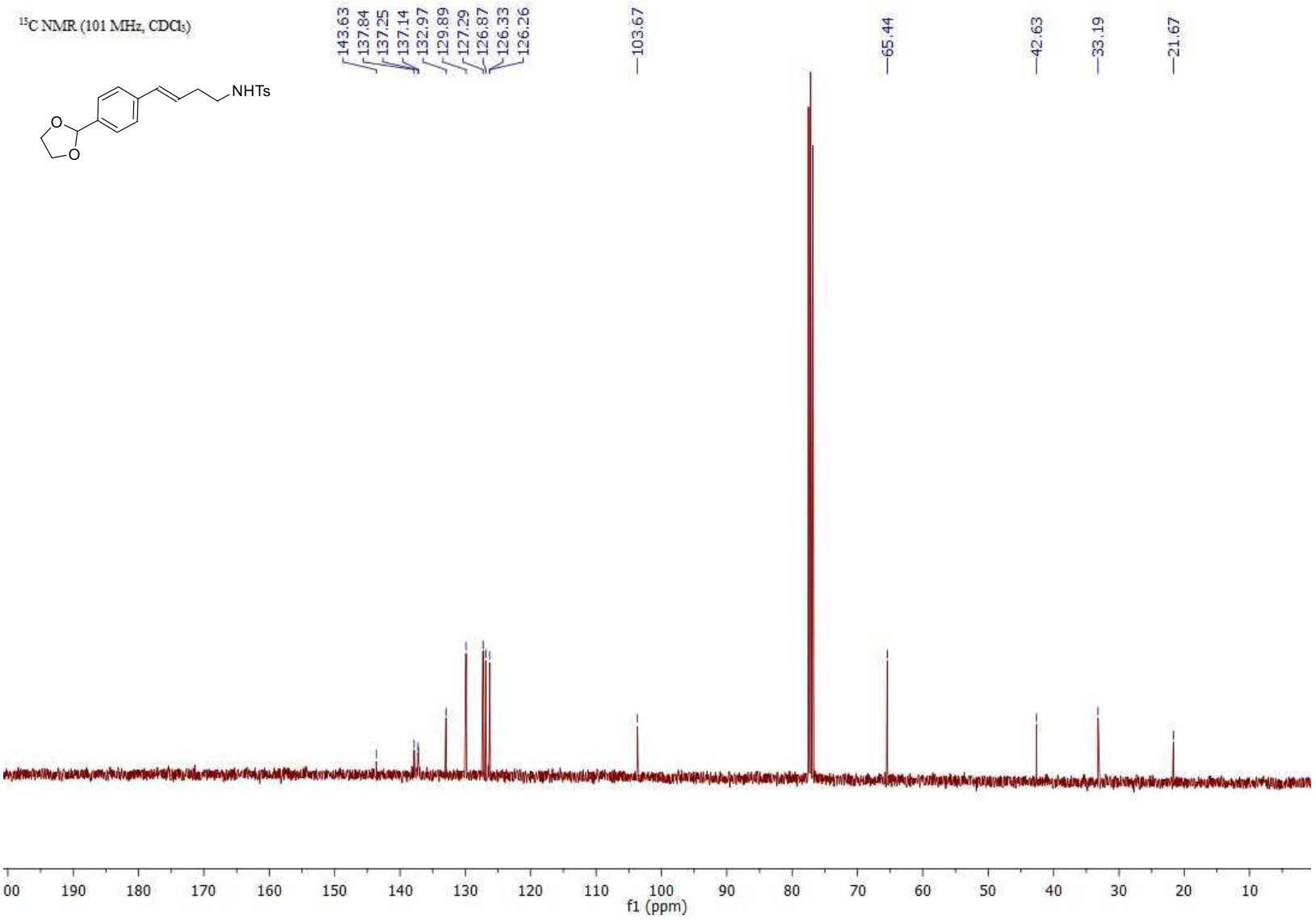
(*E*)-*N*-(4-(3-Chlorophenyl)but-3-en-1-yl)-4-methylbenzenesulfonamide, ^{13}C NMR 126 MHz $CDCl_3$



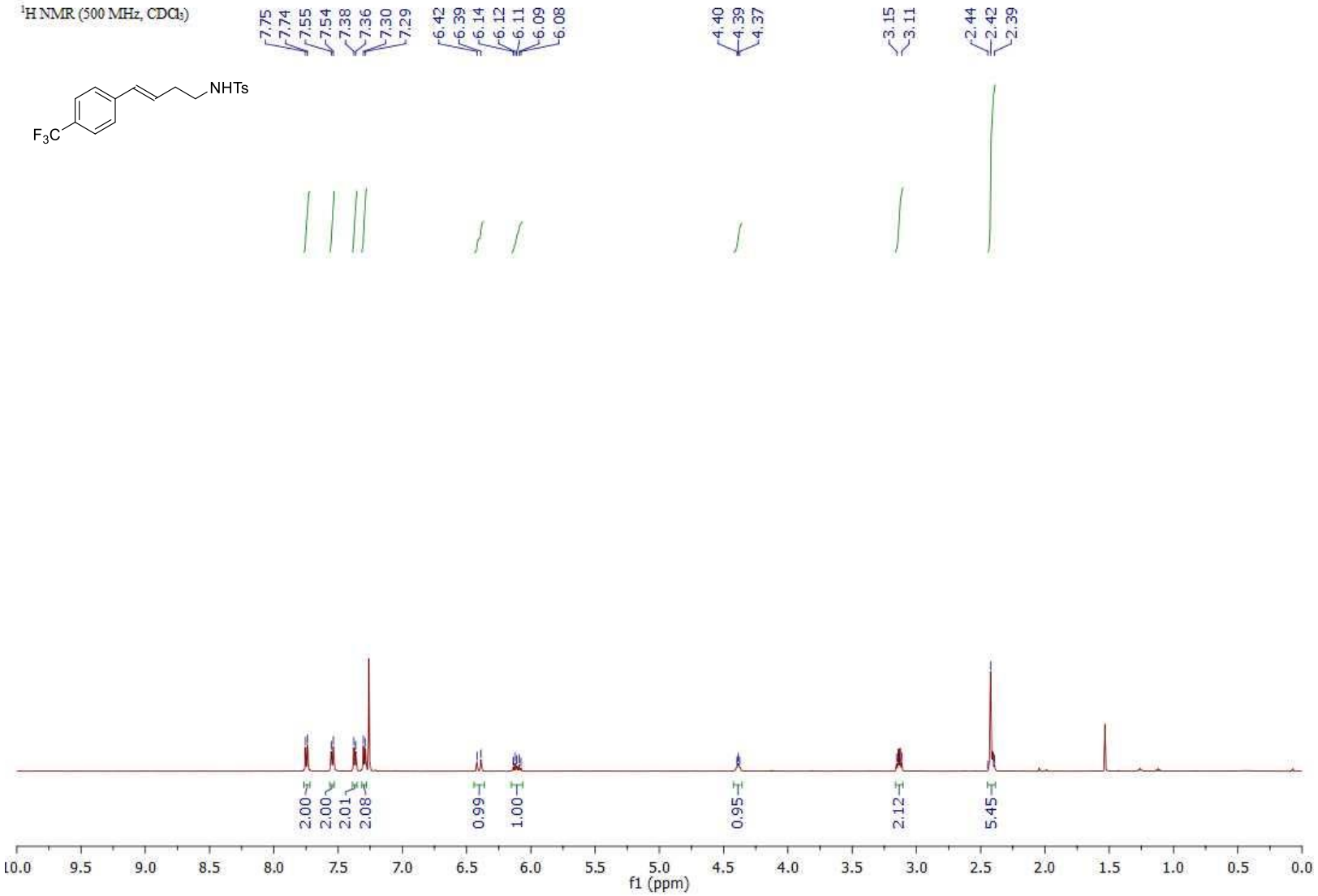
(*E*)-*N*-(4-(4-(1,3-Dioxalan-2-yl)phenyl)but-3-en-1-yl)-4-methylbenzenesulfonamide, ^{1}H NMR 400 MHz $CDCl_3$



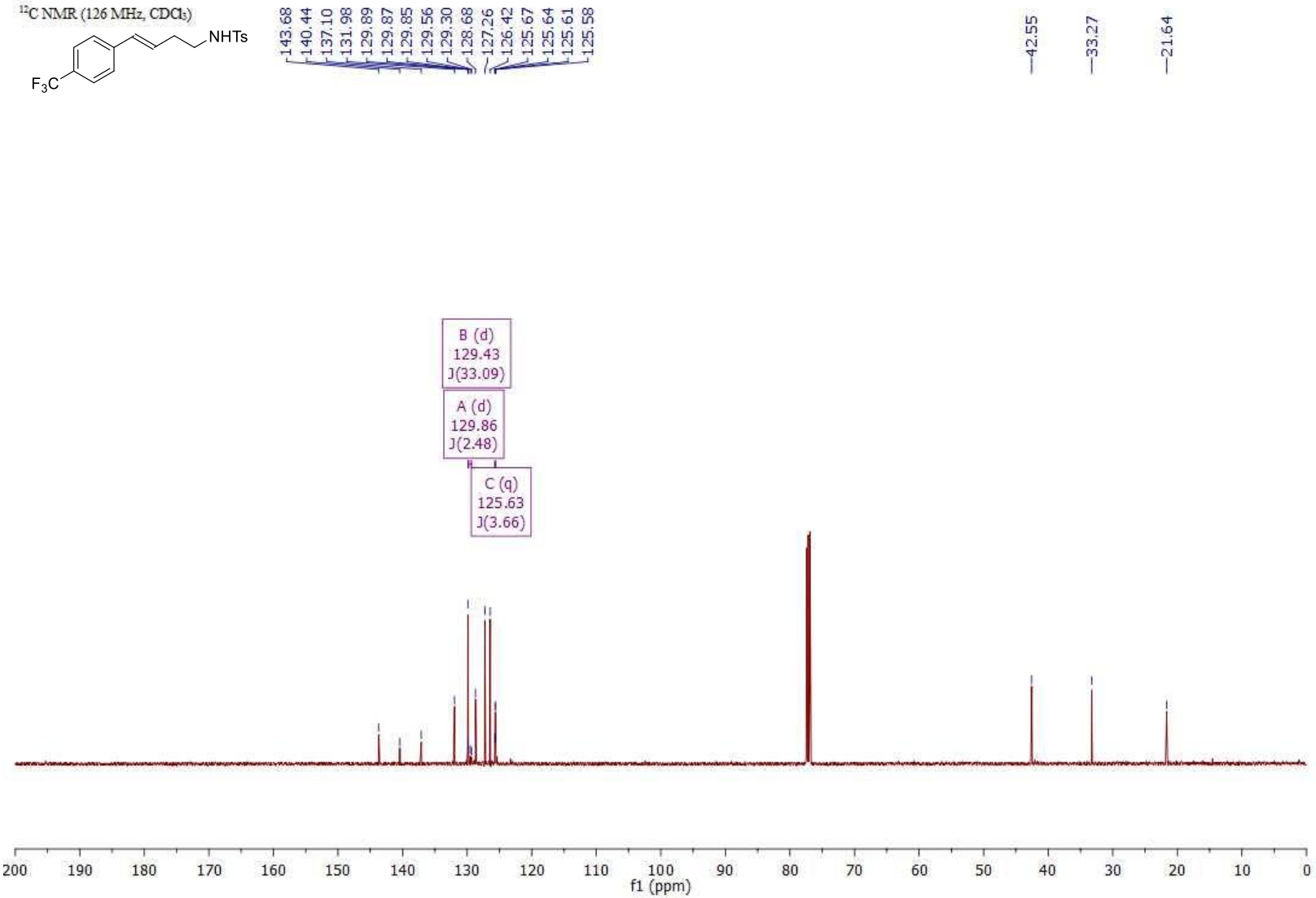
(*E*)-*N*-(4-(4-(1,3-Dioxalan-2-yl)phenyl)but-3-en-1-yl)-4-methylbenzenesulfonamide, ^{13}C NMR 101 MHz $CDCl_3$



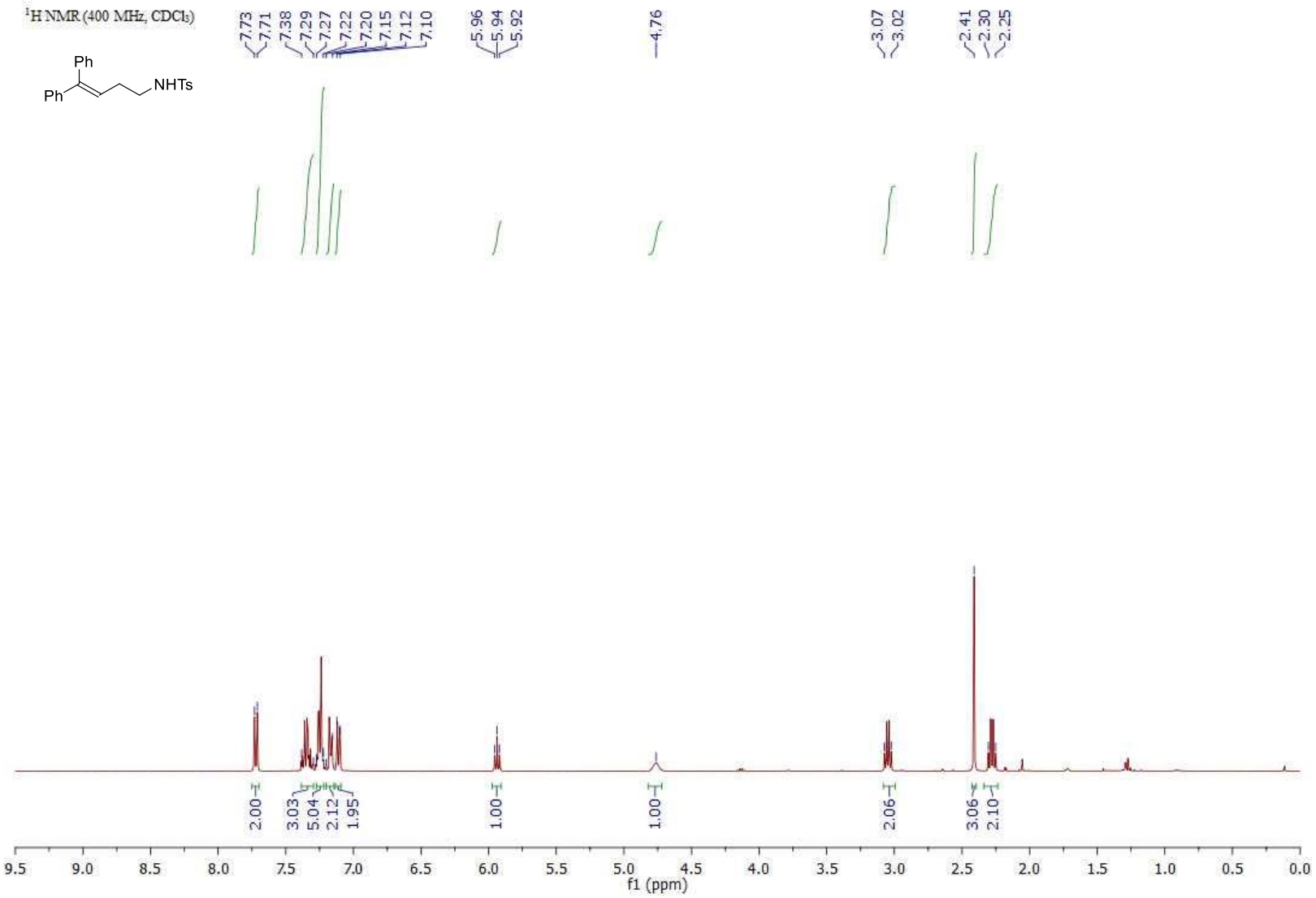
(*E*)-4-Methyl-*N*-(4-(4-(trifluoromethyl)phenyl)but-3-en-1-yl)benzenesulfonamide, ^{1}H NMR 500 MHz $CDCl_3$



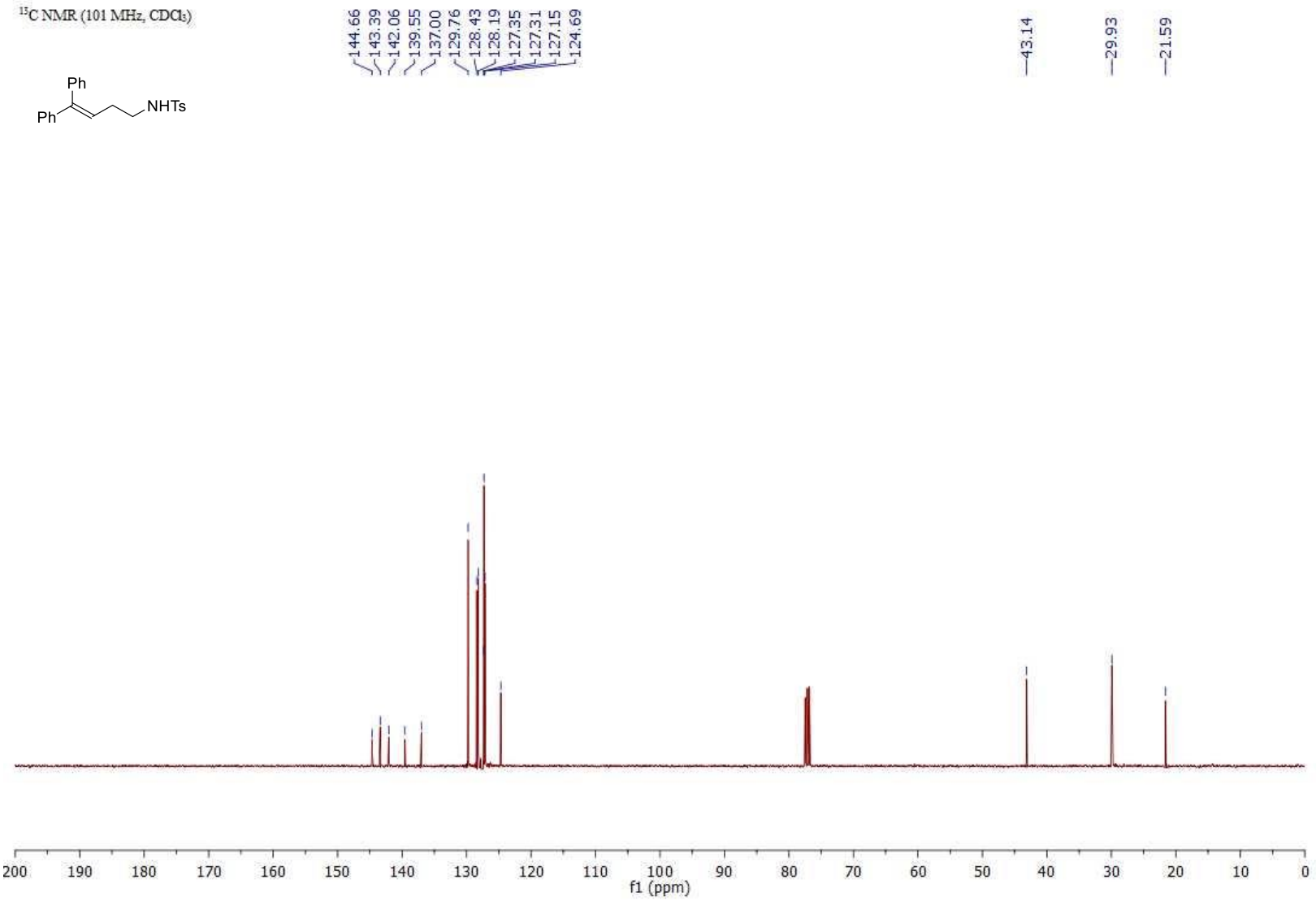
(*E*)-4-Methyl-*N*-(4-(4-(trifluoromethyl)phenyl)but-3-en-1-yl)benzenesulfonamide, ^{13}C NMR 126 MHz $CDCl_3$



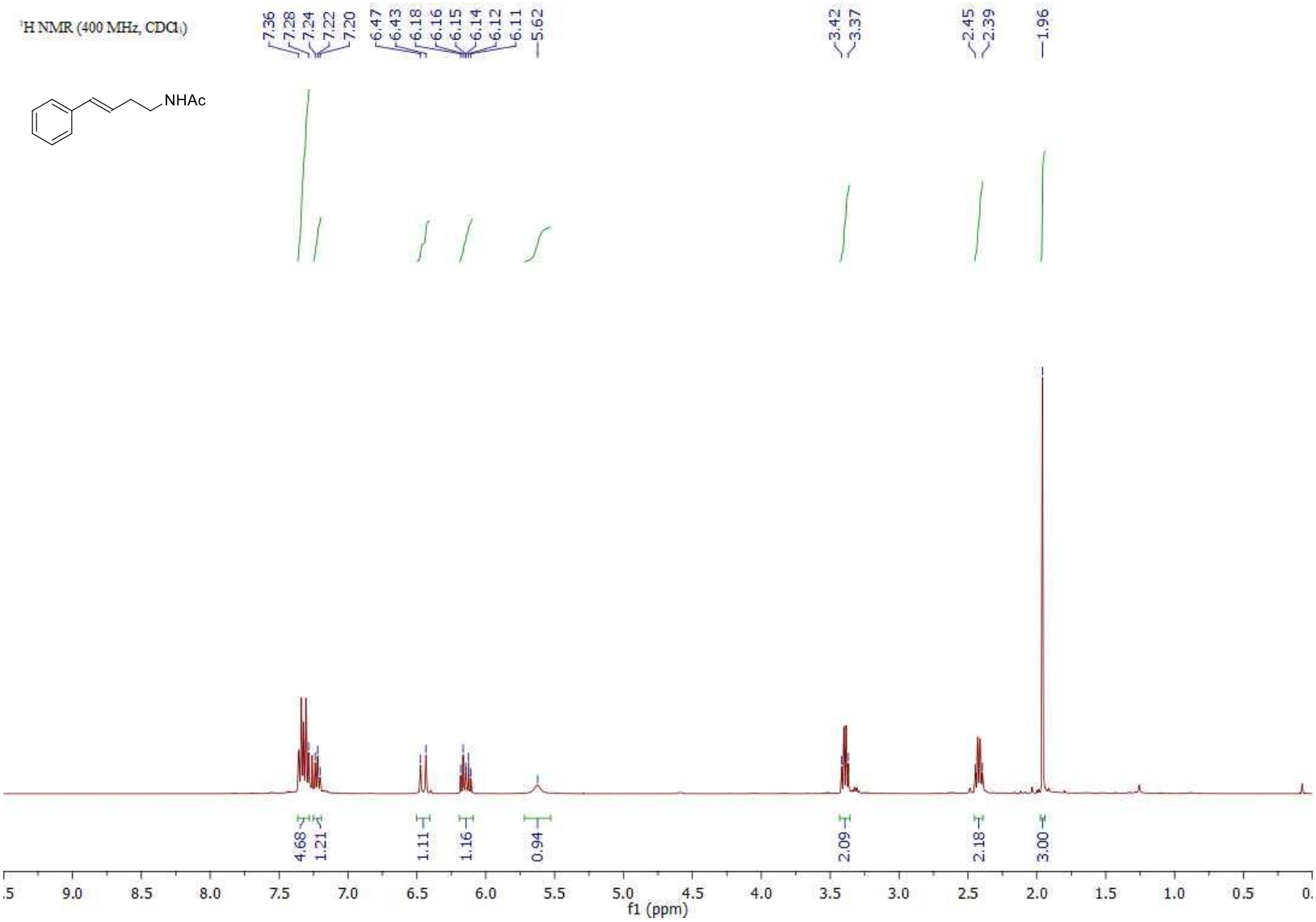
***N*-(4,4-Diphenylbut-3-en-1-yl)-4-methylbenzenesulfonamide, ^{1}H NMR 400 MHz $CDCl_3$**



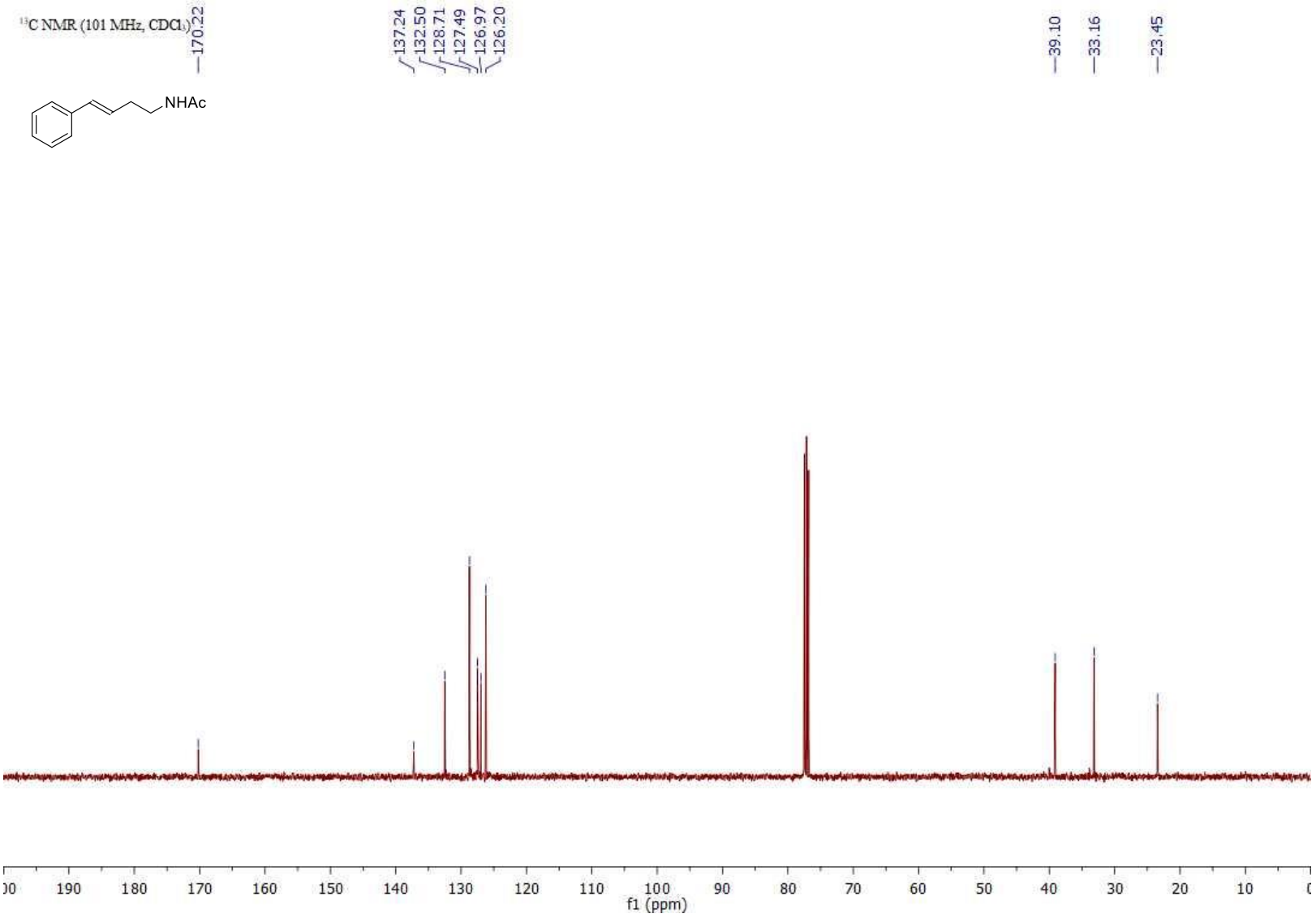
***N*-(4,4-Diphenylbut-3-en-1-yl)-4-methylbenzenesulfonamide, ^{13}C NMR 101 MHz $CDCl_3$**



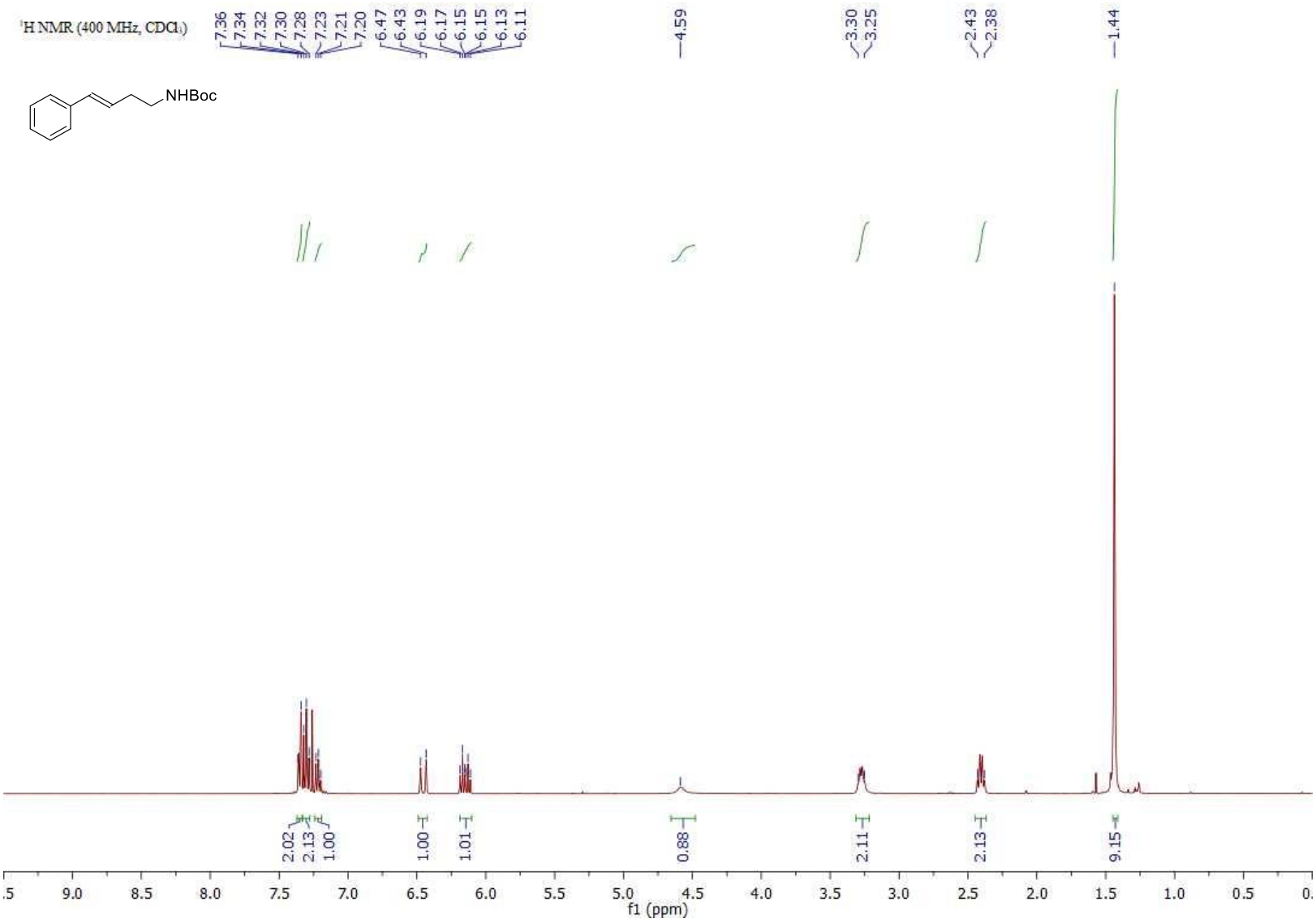
(*E*)-(4-phenylbut-3-en-1-yl)acetamide 8b, ^{1}H NMR 400 MHz $CDCl_3$



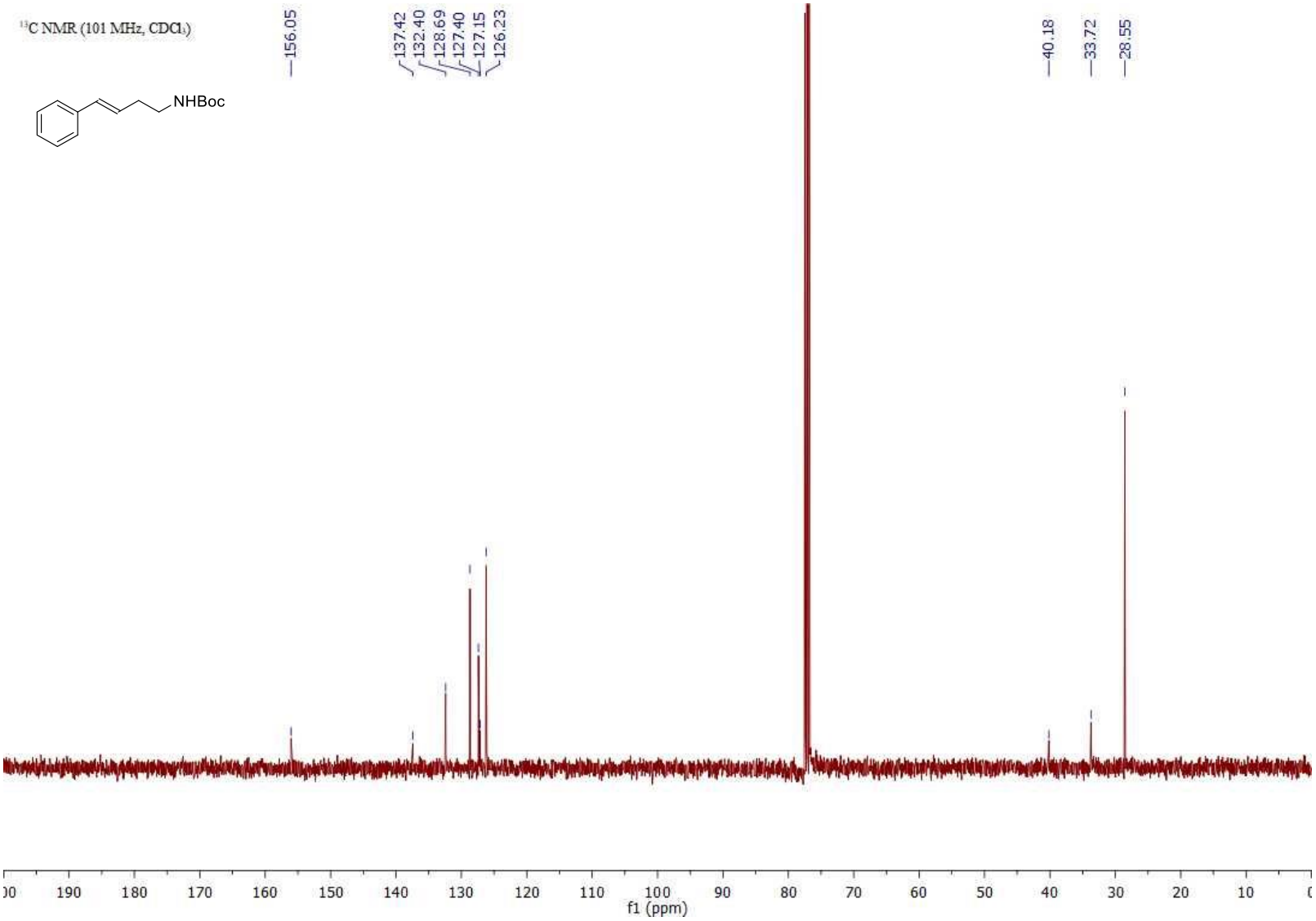
(*E*)-(4-phenylbut-3-en-1-yl)acetamide 8b, ^{13}C NMR 101 MHz $CDCl_3$



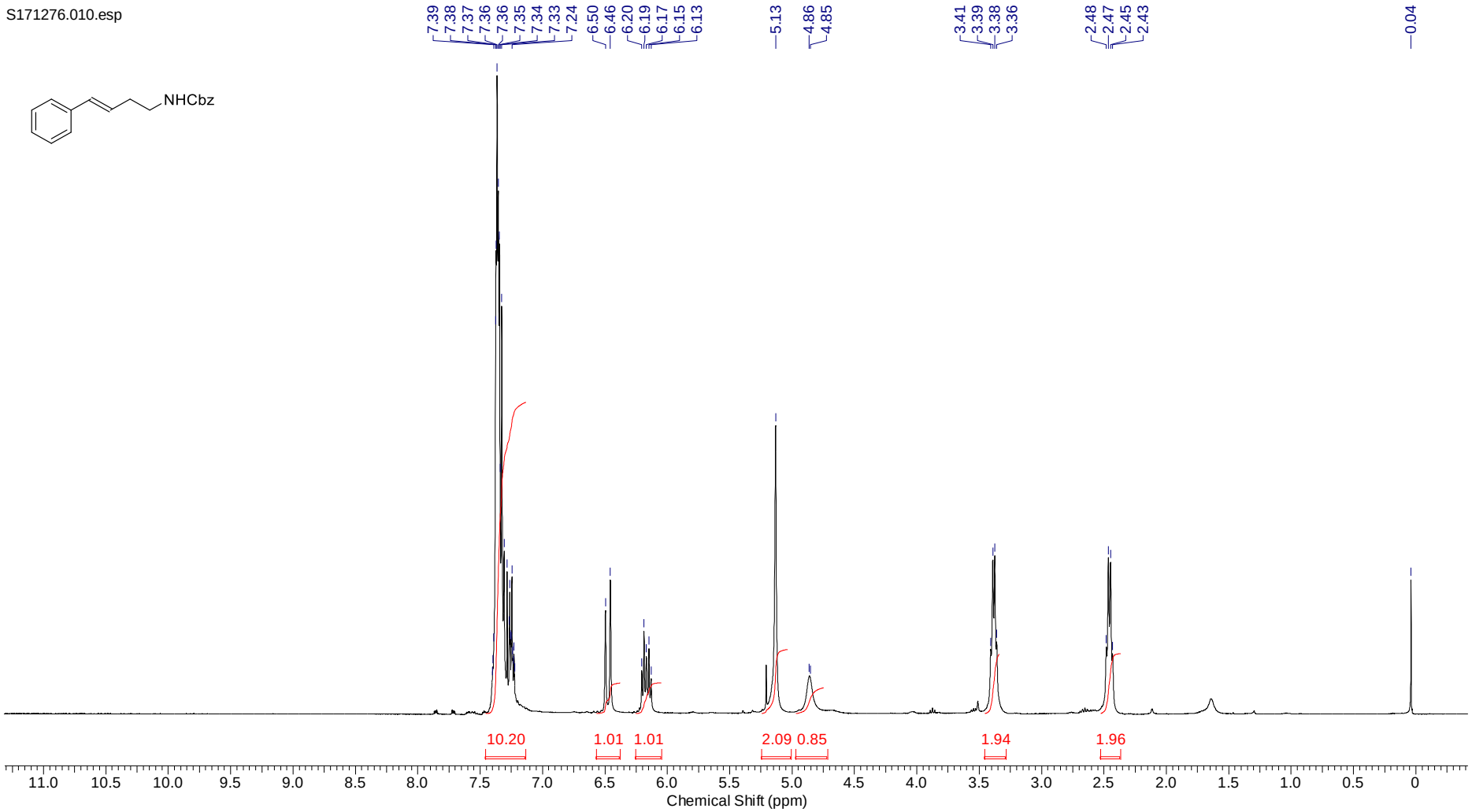
***tert*-Butyl (*E*)-(4-phenylbut-3-en-1-yl)carbamate 8c, ^{1}H NMR400 MHz $CDCl_3$**



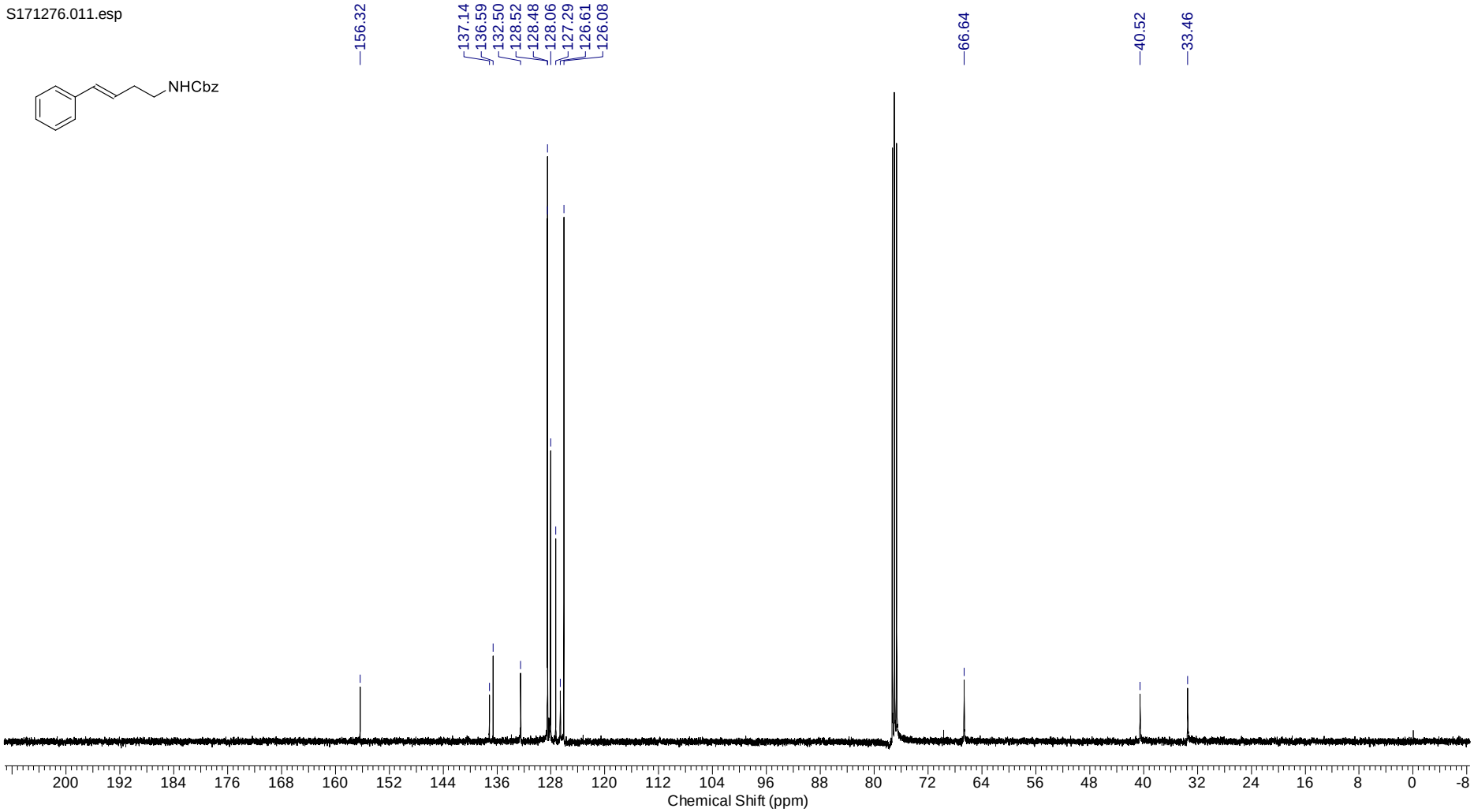
***tert*-Butyl (*E*)-(4-phenylbut-3-en-1-yl)carbamate 8c, ^{13}C NMR 101 MHz $CDCl_3$**



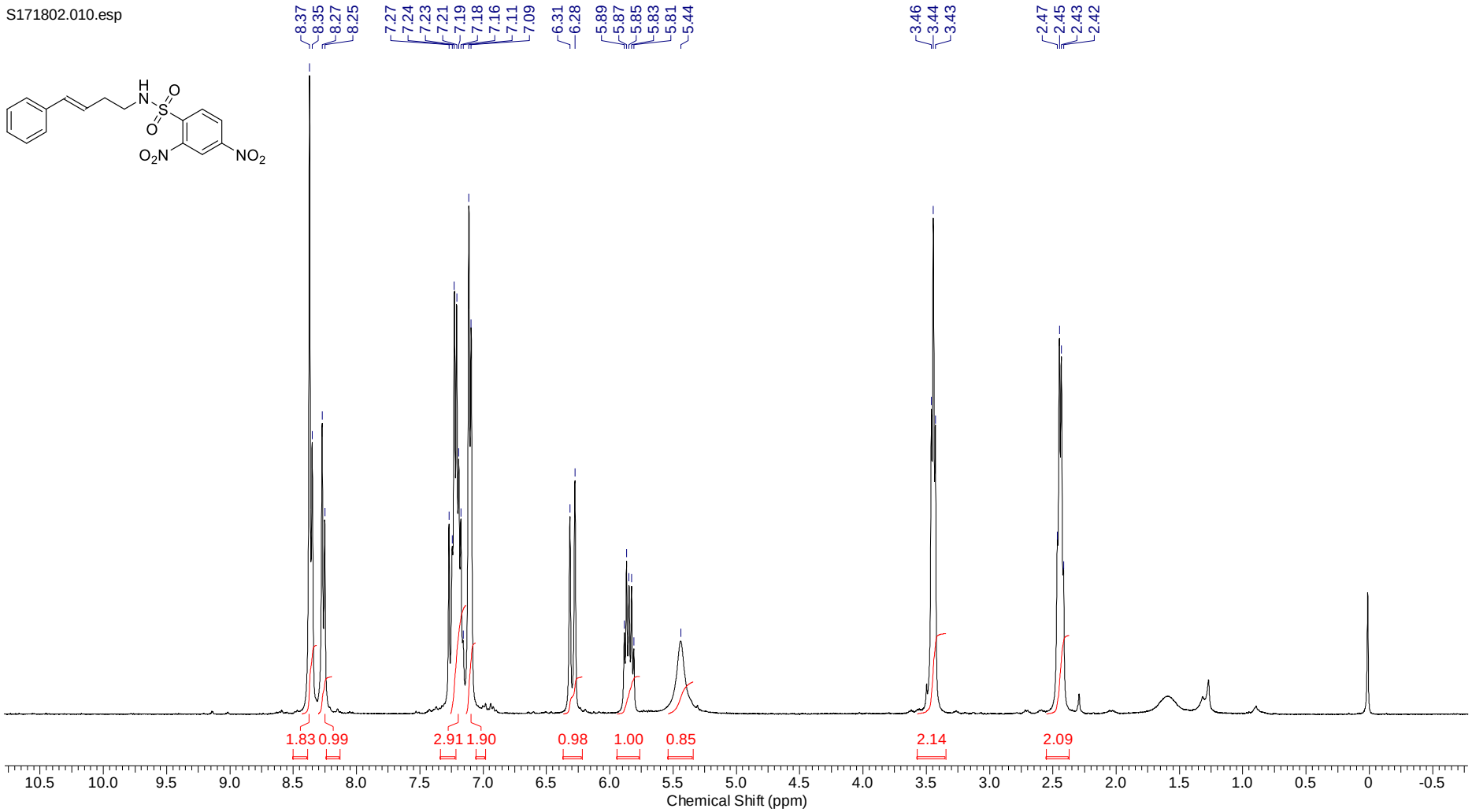
Benzyl (*E*)-(4-phenylbut-3-en-1-yl)carbamate 8d, ^{1}H NMR 400 MHz $CDCl_3$



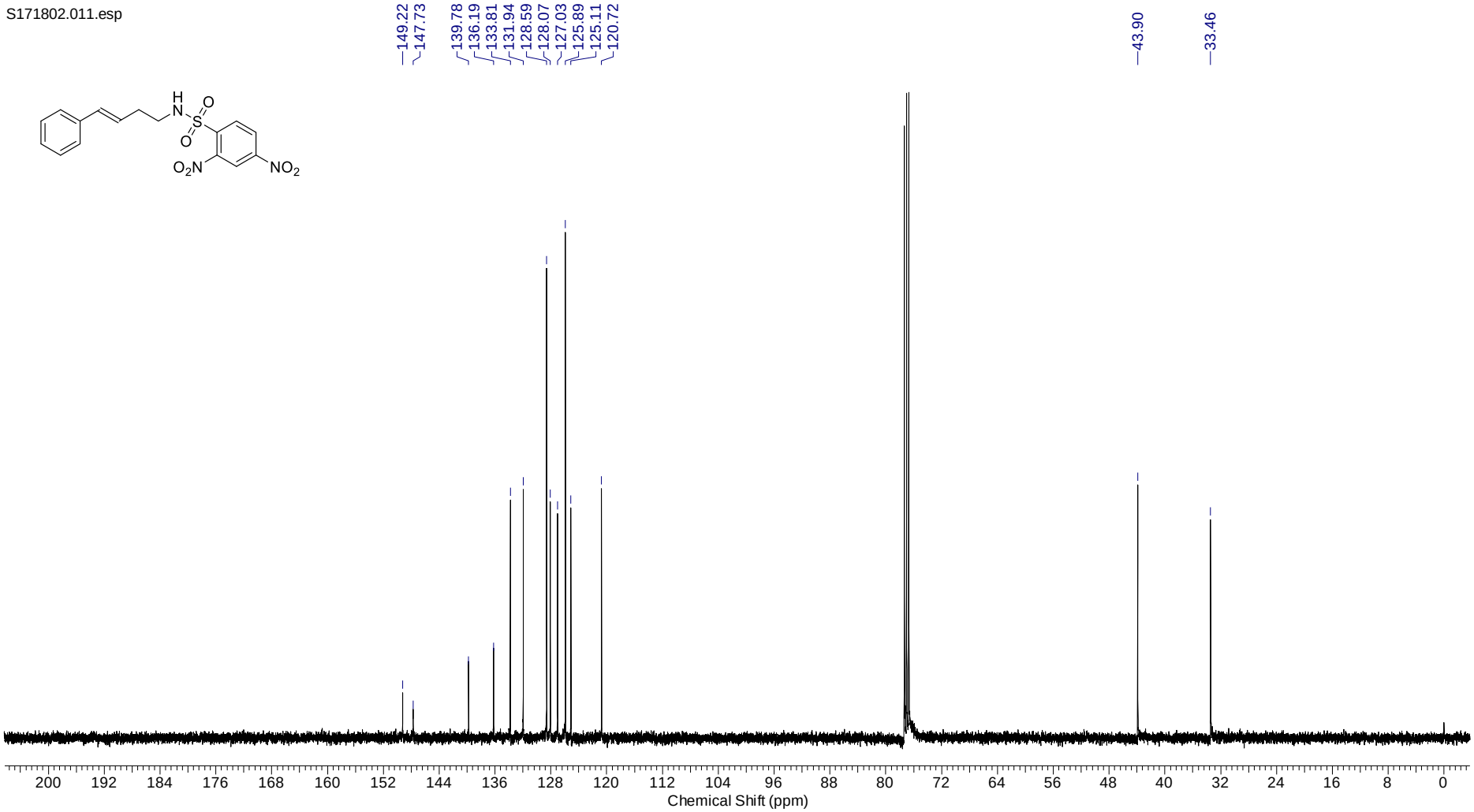
Benzyl (*E*)-(4-phenylbut-3-en-1-yl)carbamate 8d, ^{13}C NMR 101 MHz $CDCl_3$



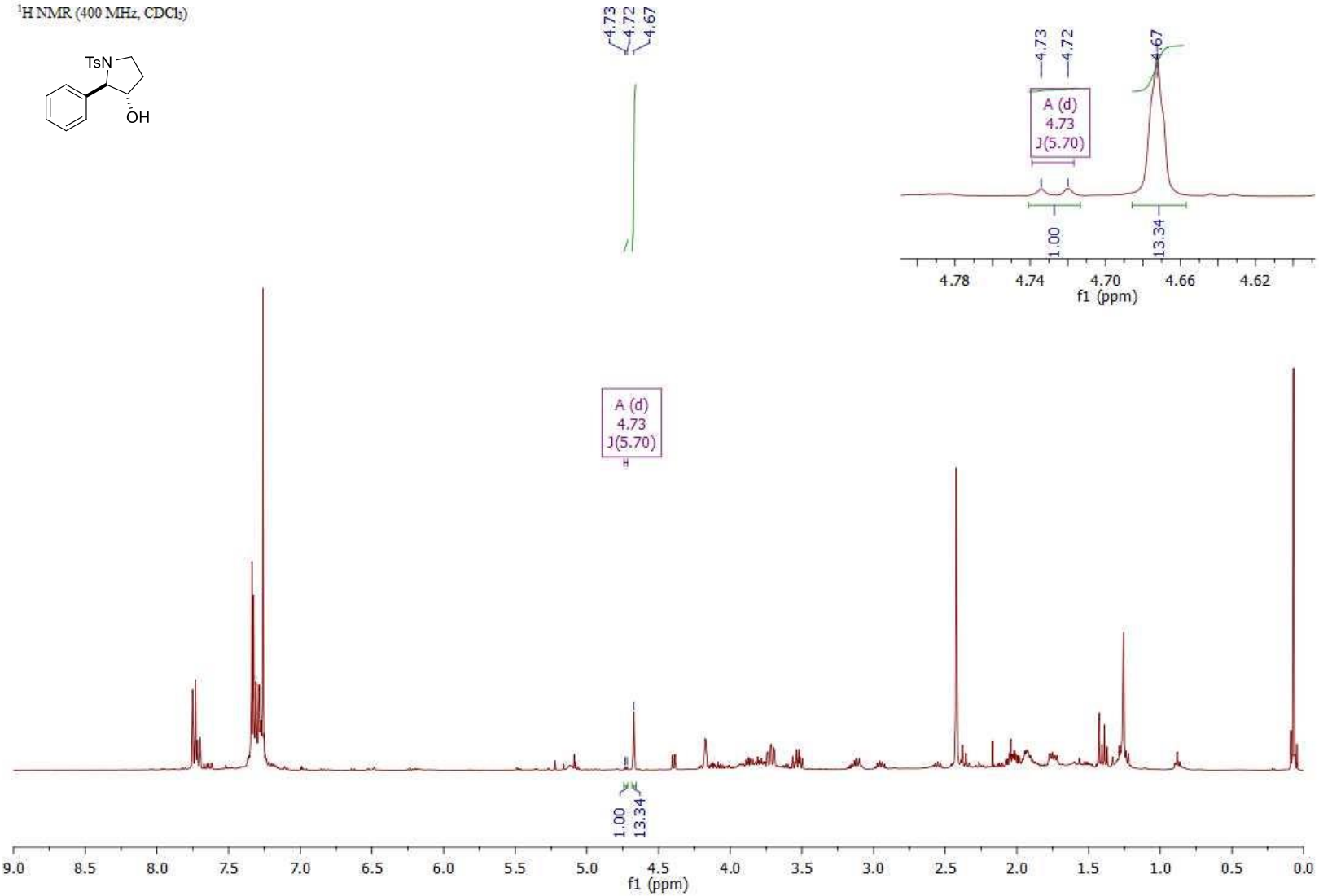
(*E*)-2,4-Dinitro-*N*-(4-phenylbut-3-en-1-yl)benzenesulfonamide 8e, ^{1}H NMR 400 MHz $CDCl_3$



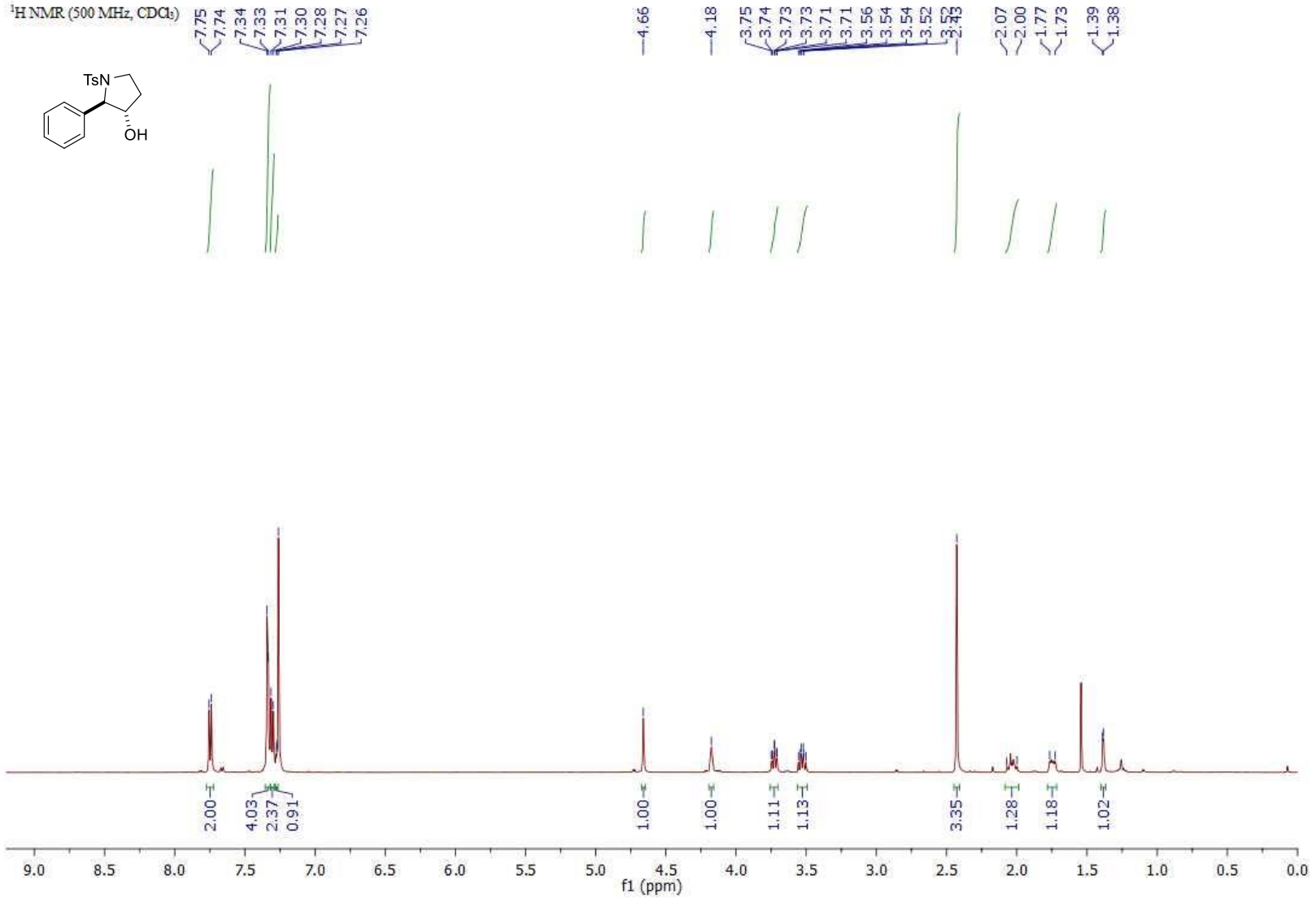
(*E*)-2,4-Dinitro-*N*-(4-phenylbut-3-en-1-yl)benzenesulfonamide 8e, ^{13}C NMR 101 MHz $CDCl_3$



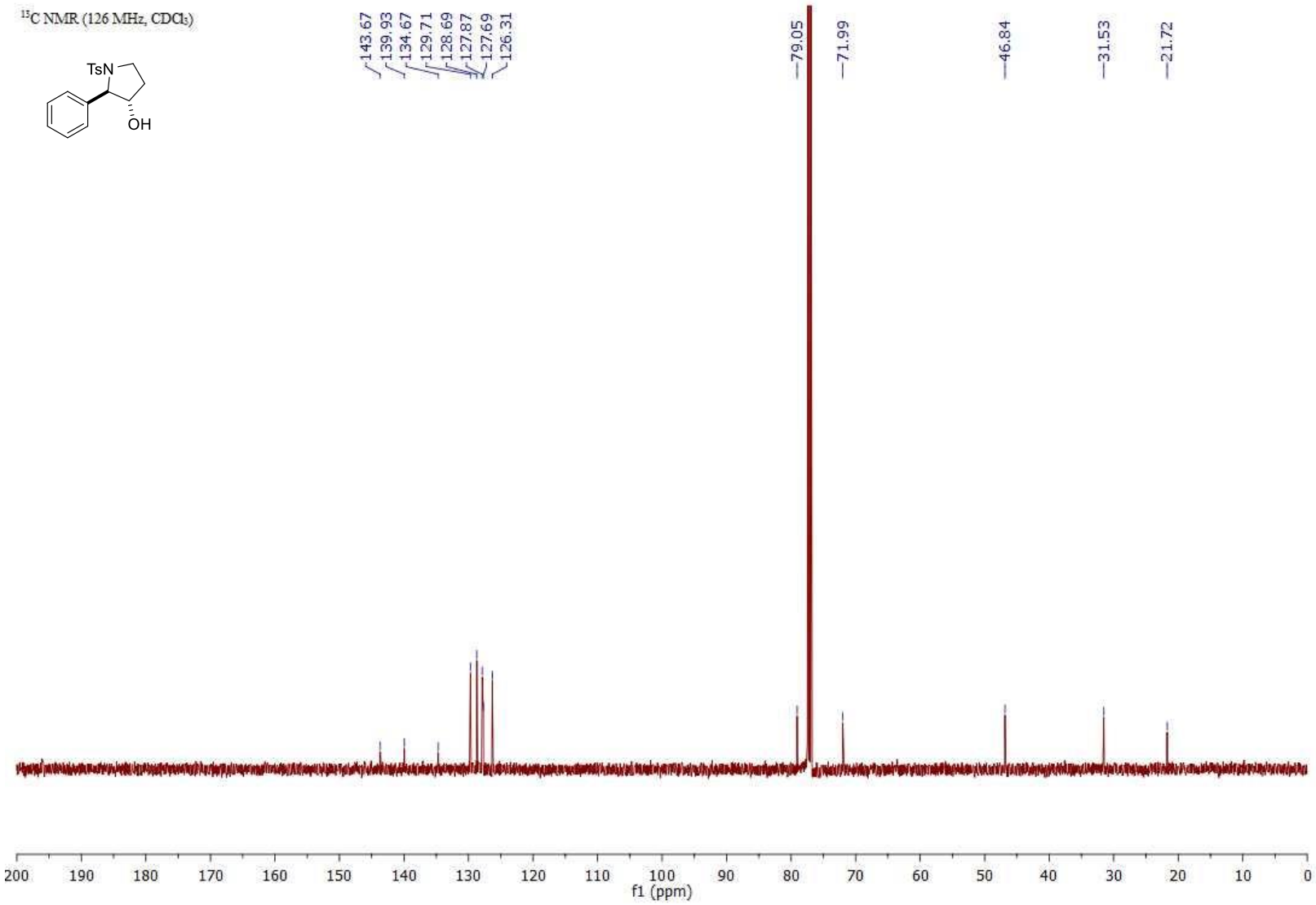
(±)-2-Phenyl-1-tosylpyrrolidin-3-ol 9a, crude reaction mixture ^{1}H NMR 400 MHz $CDCl_3$



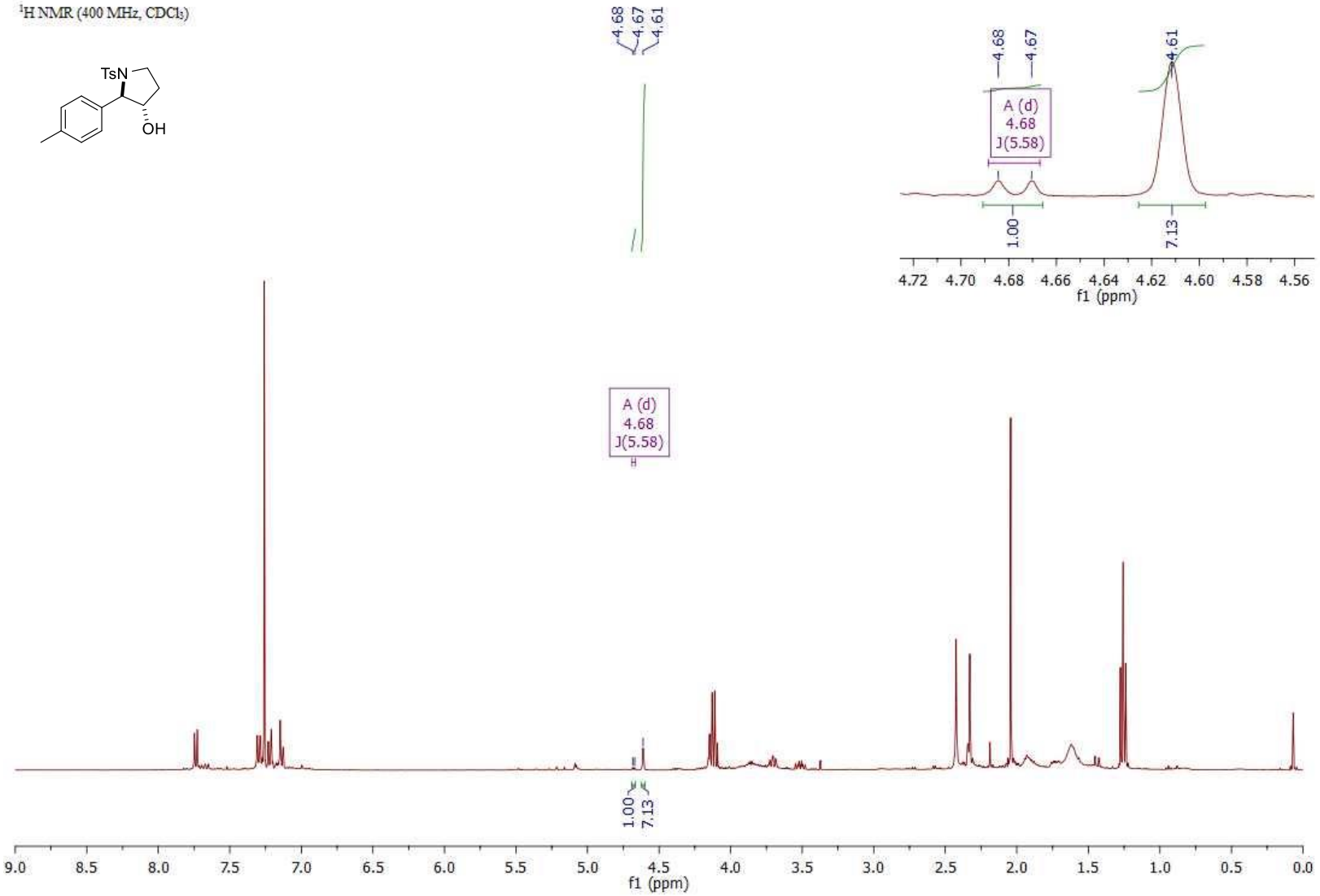
(±)-2-Phenyl-1-tosylpyrrolidin-3-ol 9a, purified sample ^{1}H NMR 500 MHz $CDCl_3$



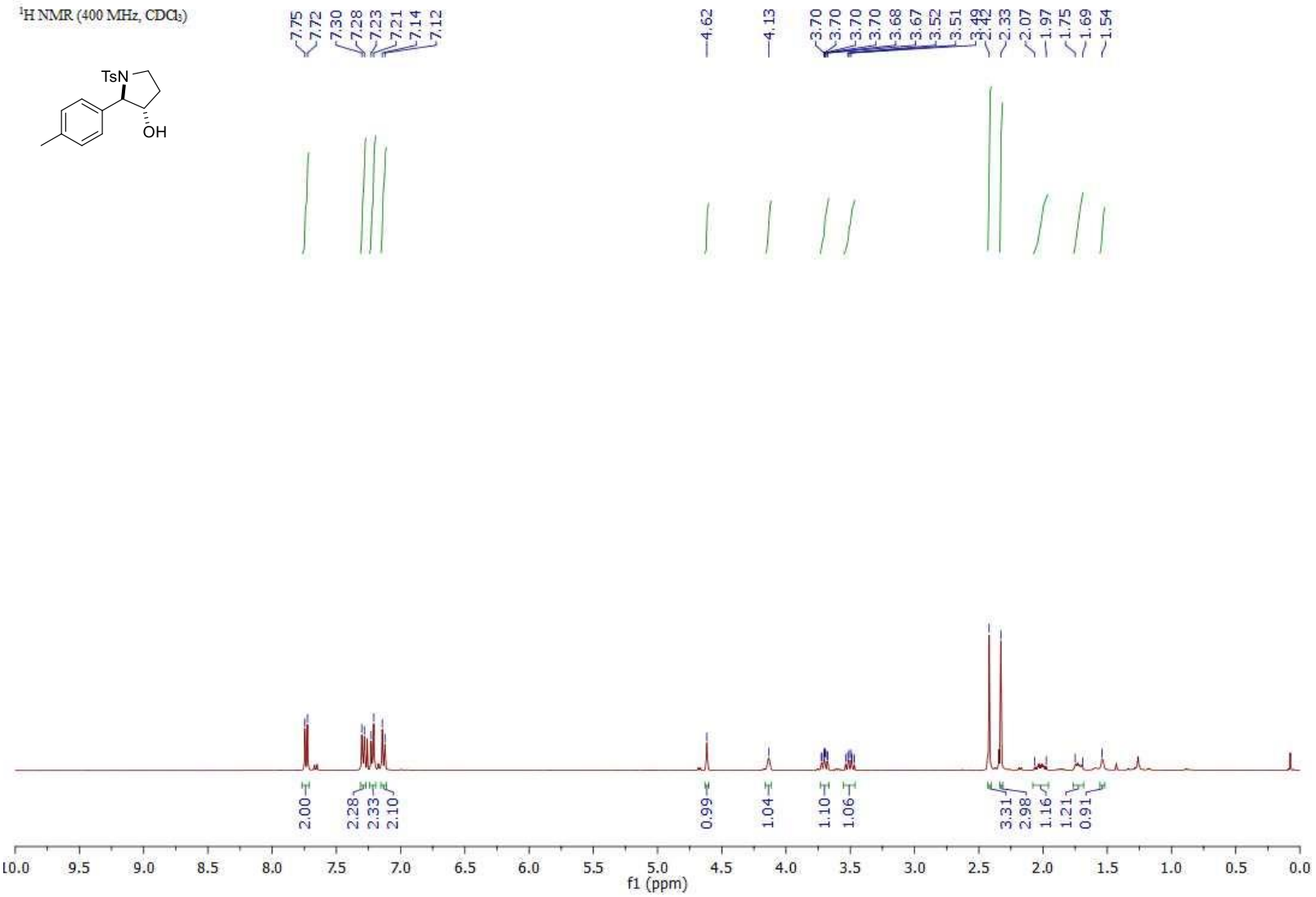
(±)-2-Phenyl-1-tosylpyrrolidin-3-ol 9a, purified sample ^{13}C NMR 126 MHz $CDCl_3$



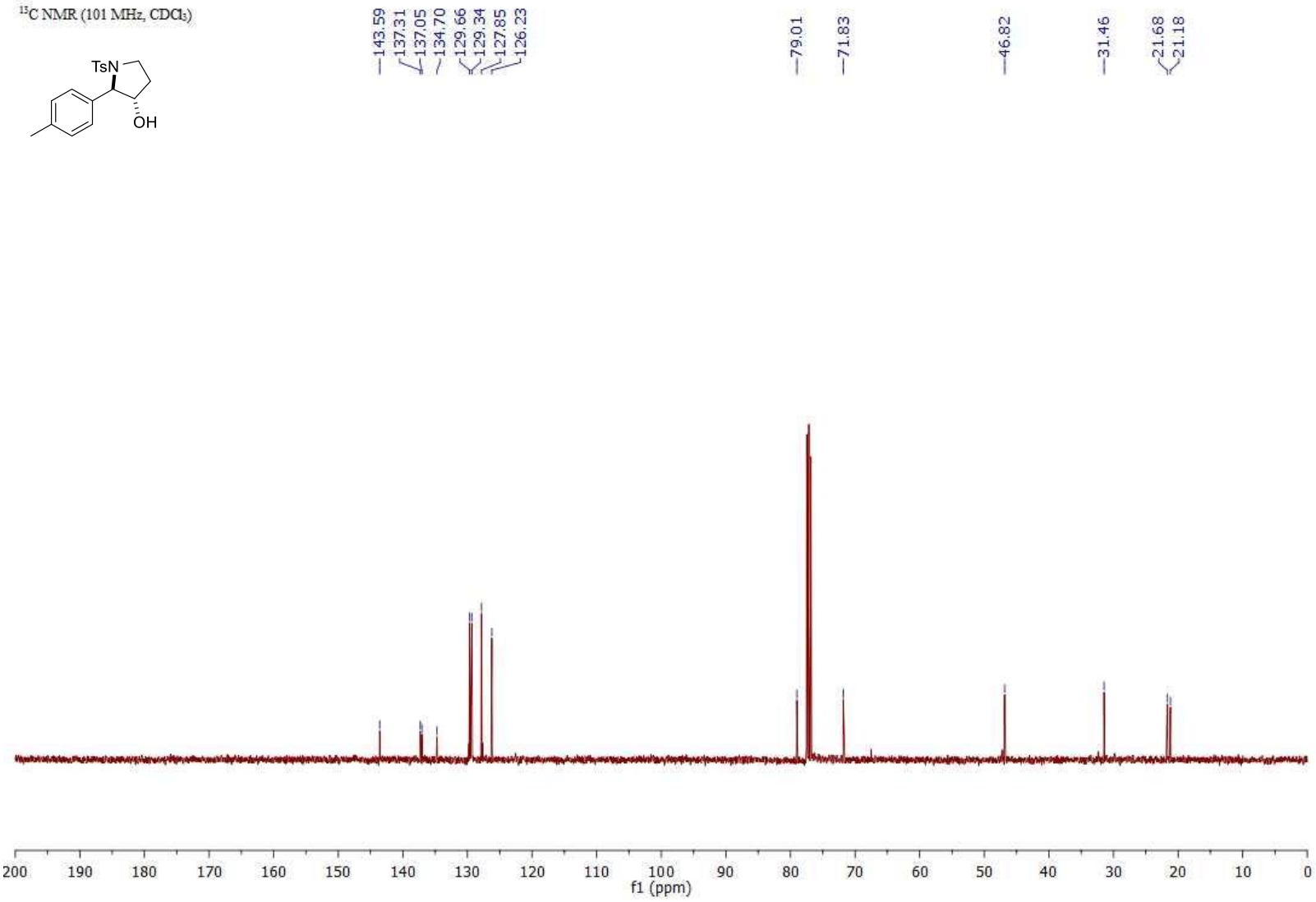
(±)-2-(*p*-Tolyl)-1-tosylpyrrolidin-3-ol 13, crude reaction mixture ^{1}H NMR 400 MHz $CDCl_3$



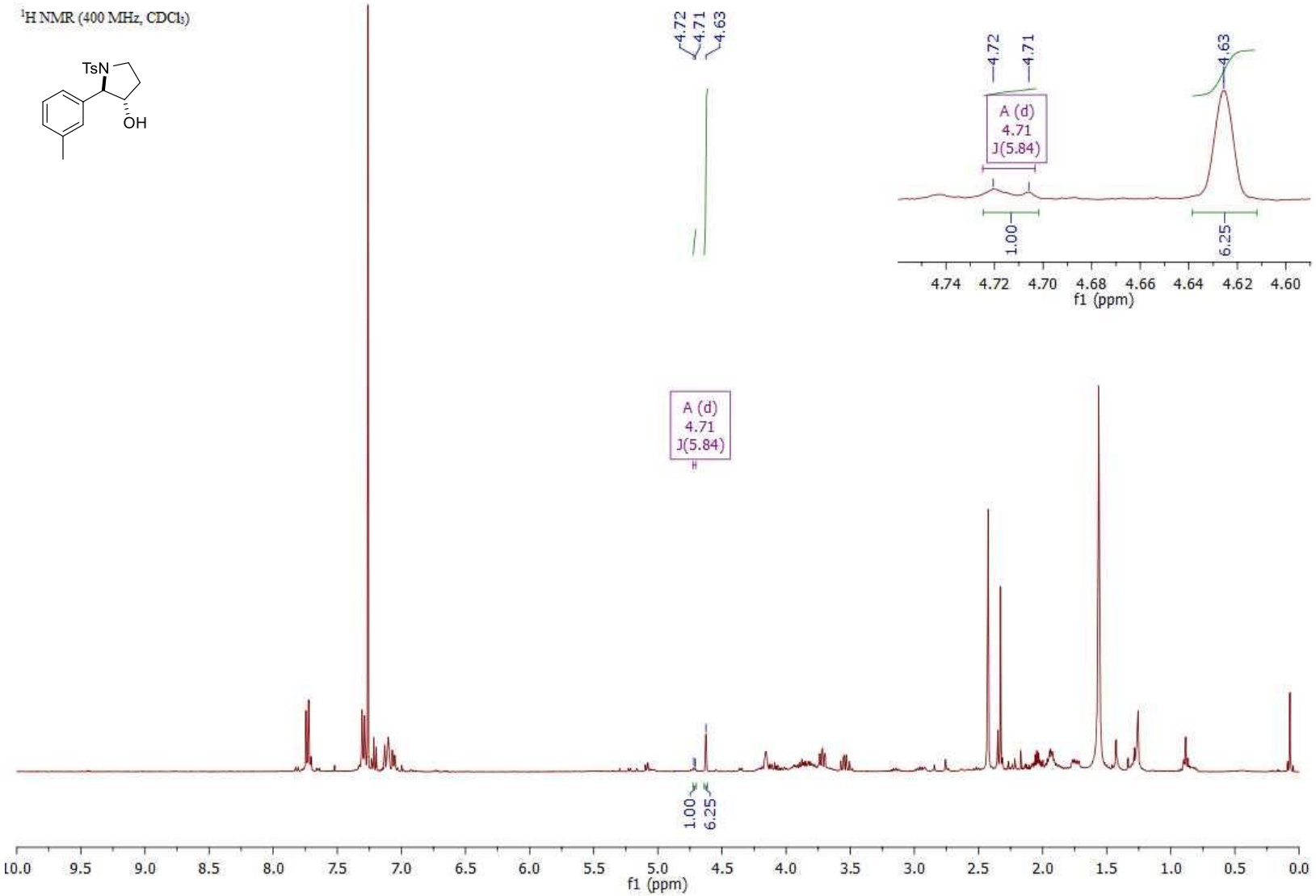
(±)-2-(*p*-Tolyl)-1-tosylpyrrolidin-3-ol 13, purified sample ^{1}H NMR 400 MHz $CDCl_3$



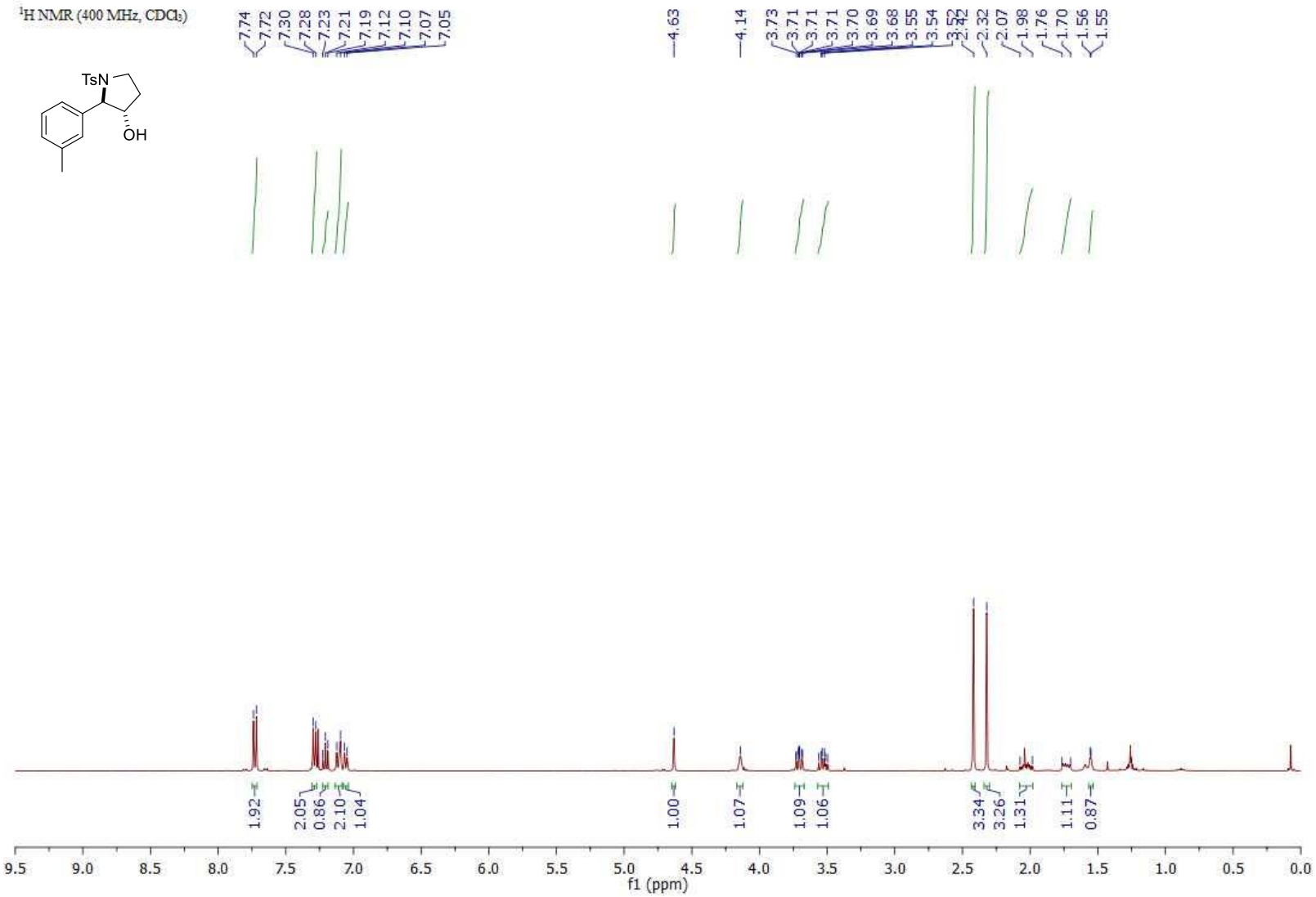
(±)-2-(*p*-Tolyl)-1-tosylpyrrolidin-3-ol 13, purified sample ^{13}C NMR 101 MHz $CDCl_3$



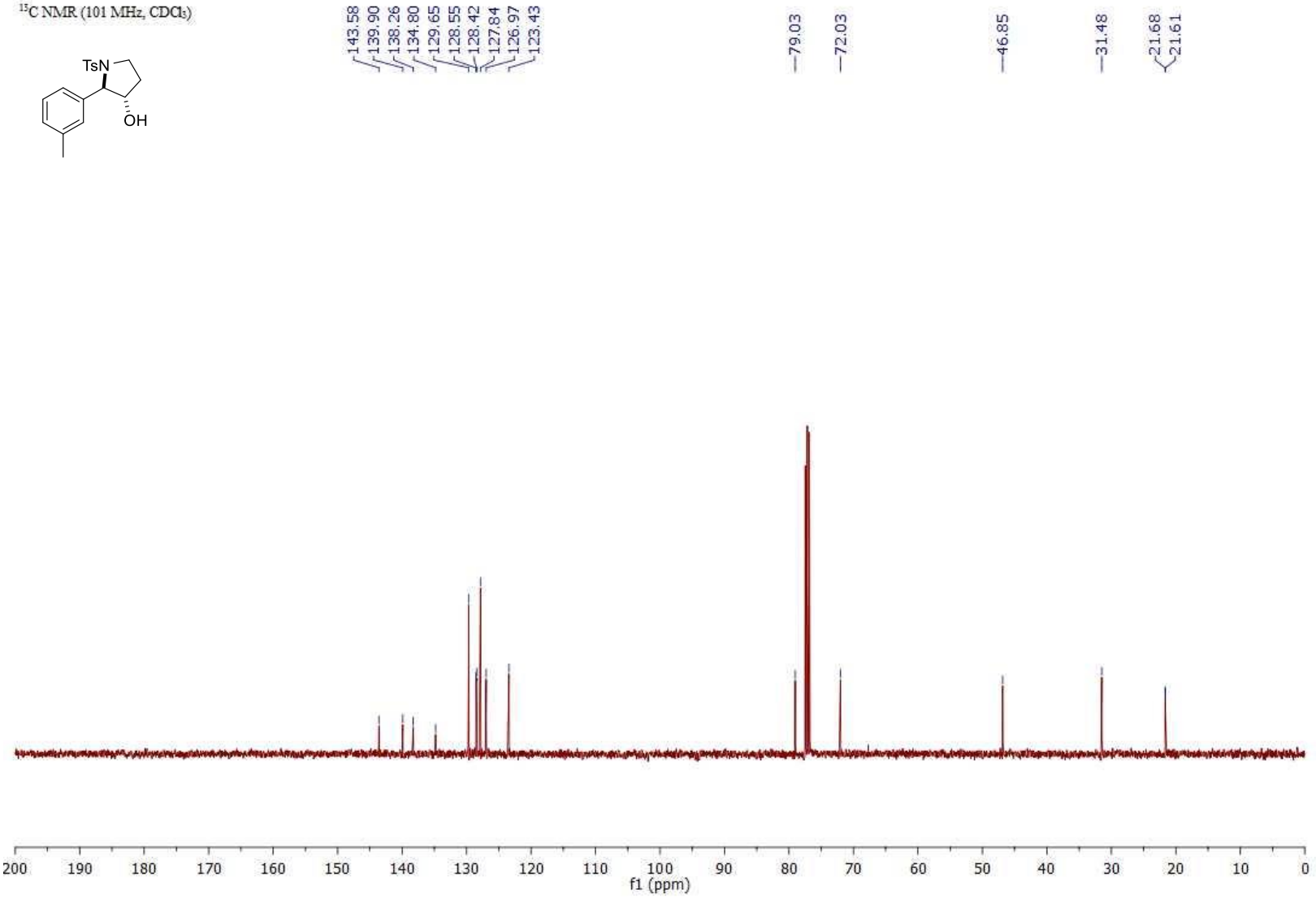
(±)-2-(*m*-Tolyl)-1-tosylpyrrolidin-3-ol 14, crude reaction mixture ^{1}H NMR 400 MHz $CDCl_3$



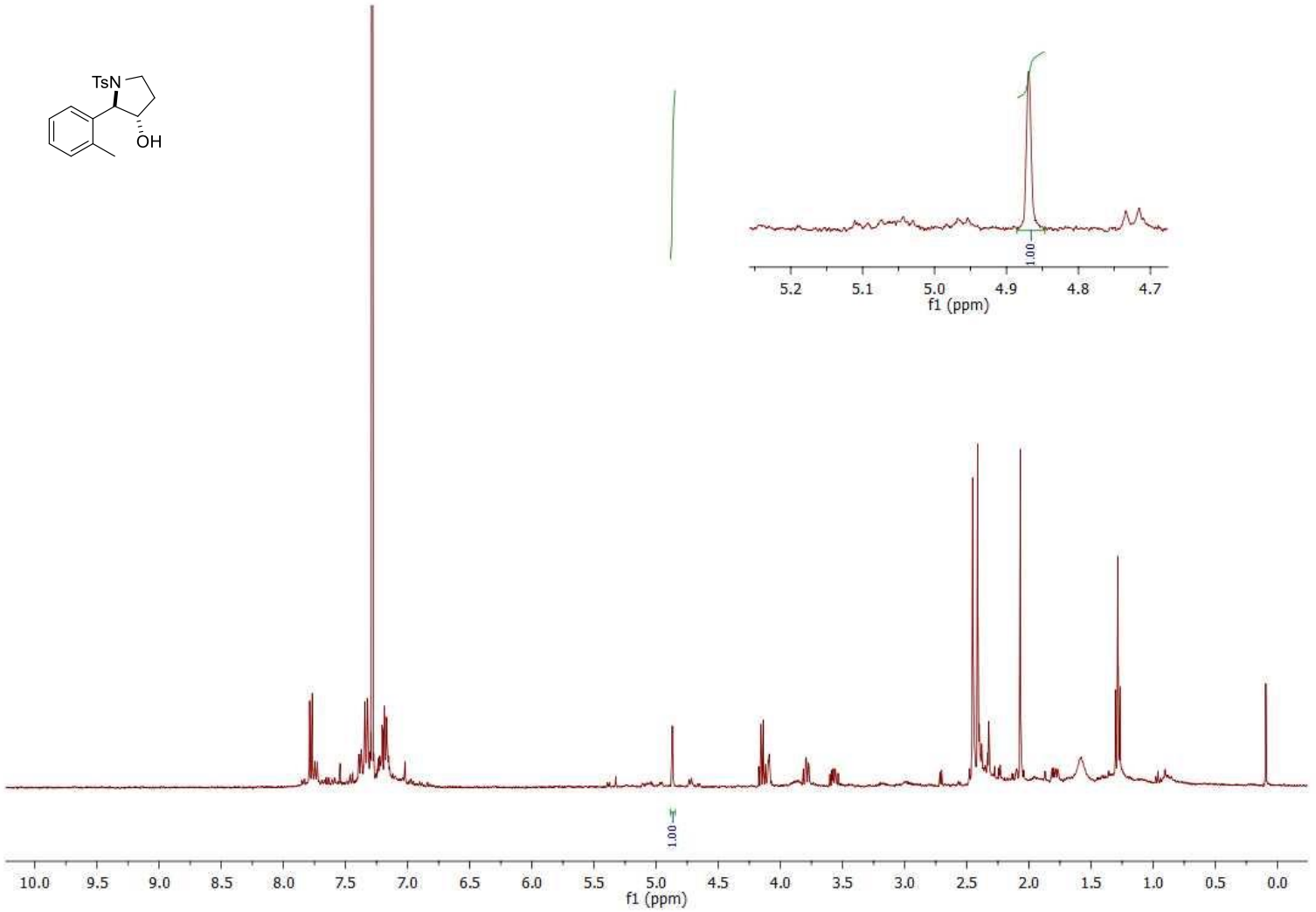
(±)-2-(*m*-Tolyl)-1-tosylpyrrolidin-3-ol 14, purified sample ^{1}H NMR 400 MHz $CDCl_3$



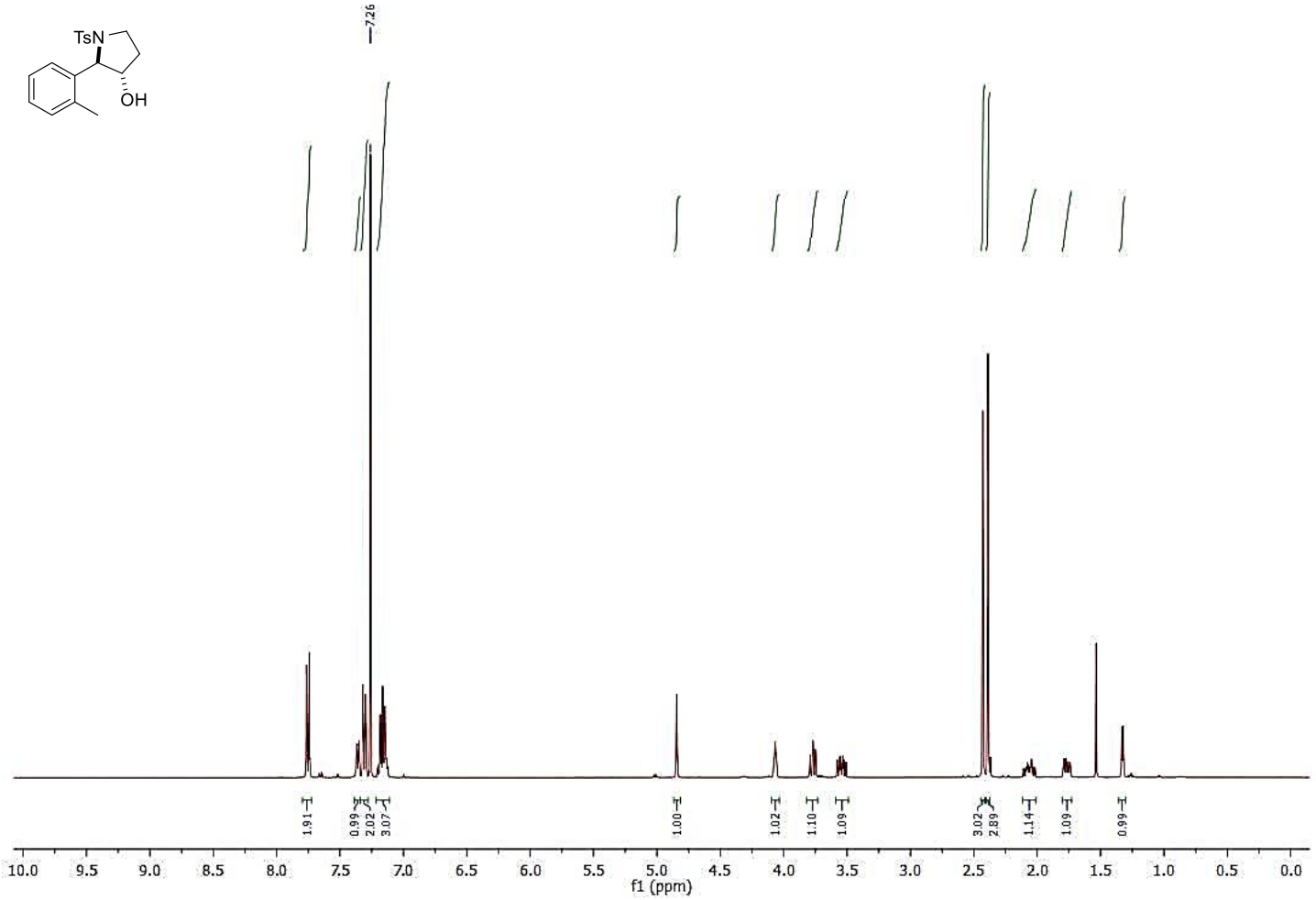
(±)-2-(*m*-Tolyl)-1-tosylpyrrolidin-3-ol 14, purified sample ^{13}C NMR 101 MHz $CDCl_3$



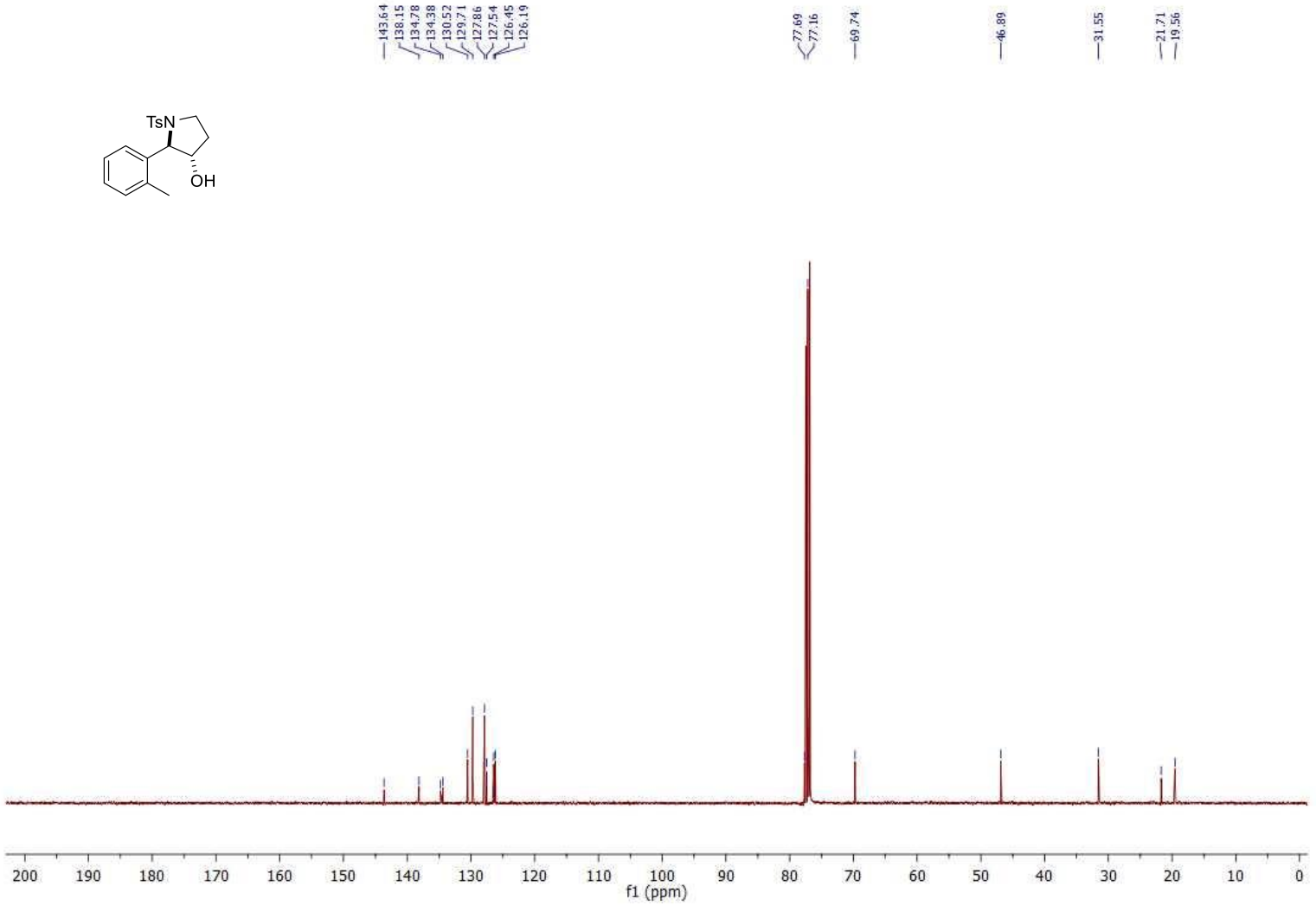
(±)-2-(*o*-Tolyl)-1-tosylpyrrolidin-3-ol 15, crude reaction mixture ^{1}H NMR 500 MHz $CDCl_3$ diastereomeric ratio not assigned.



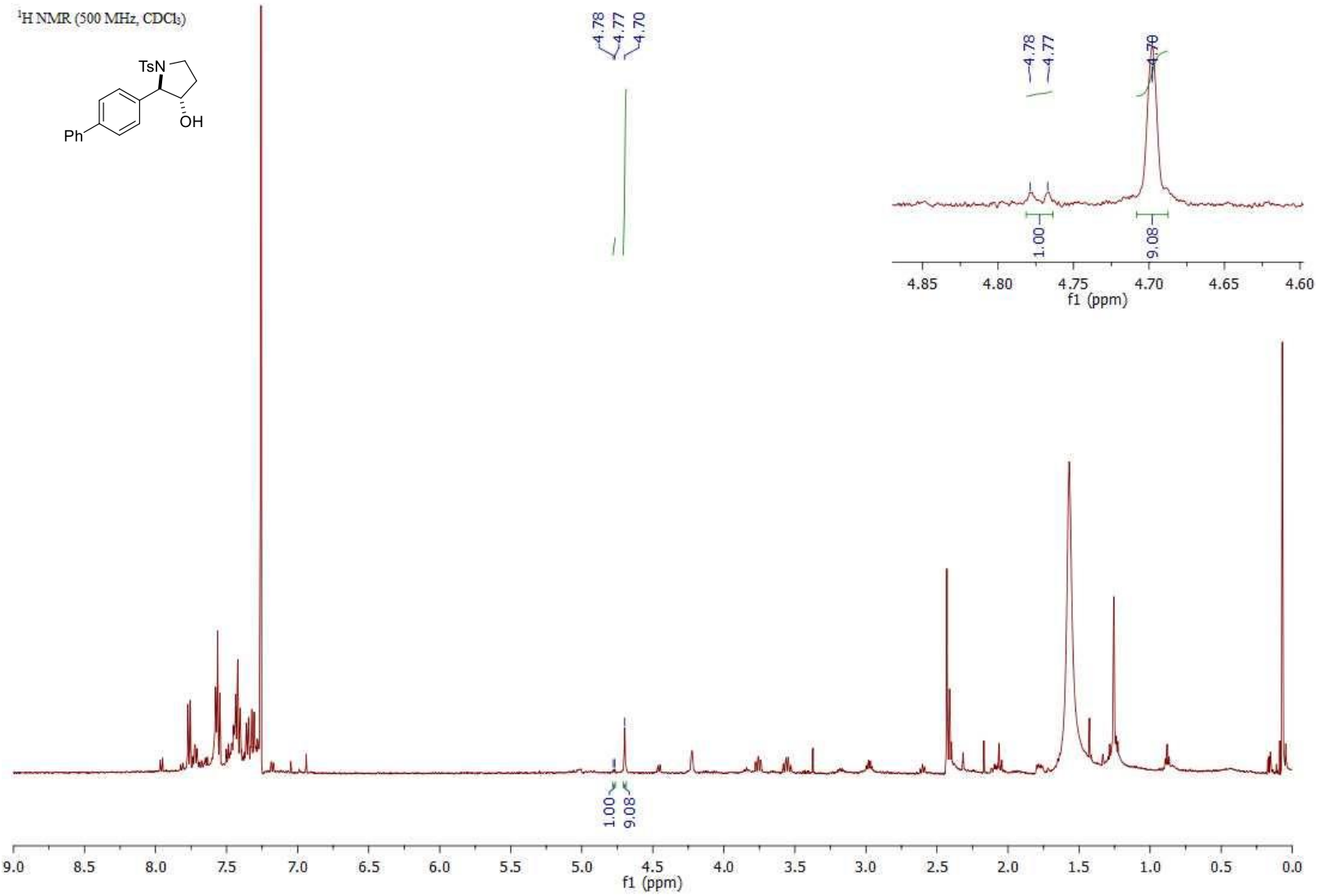
(±)-2-(*o*-Tolyl)-1-tosylpyrrolidin-3-ol 15, purified sample ^{1}H NMR 400 MHz $CDCl_3$



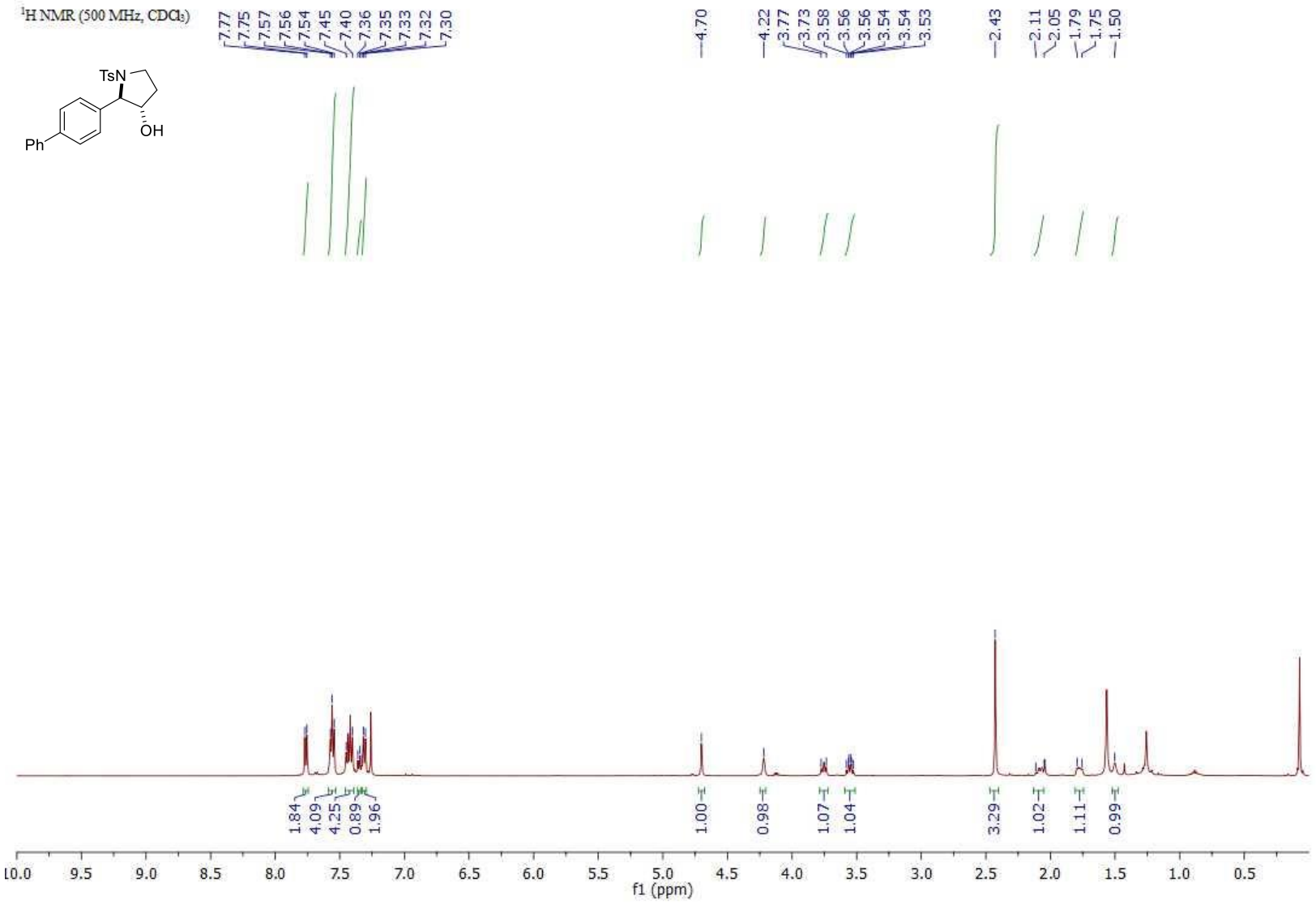
(±)-2-(*o*-Tolyl)-1-tosylpyrrolidin-3-ol 15, purified sample ^{13}C NMR 101 MHz $CDCl_3$



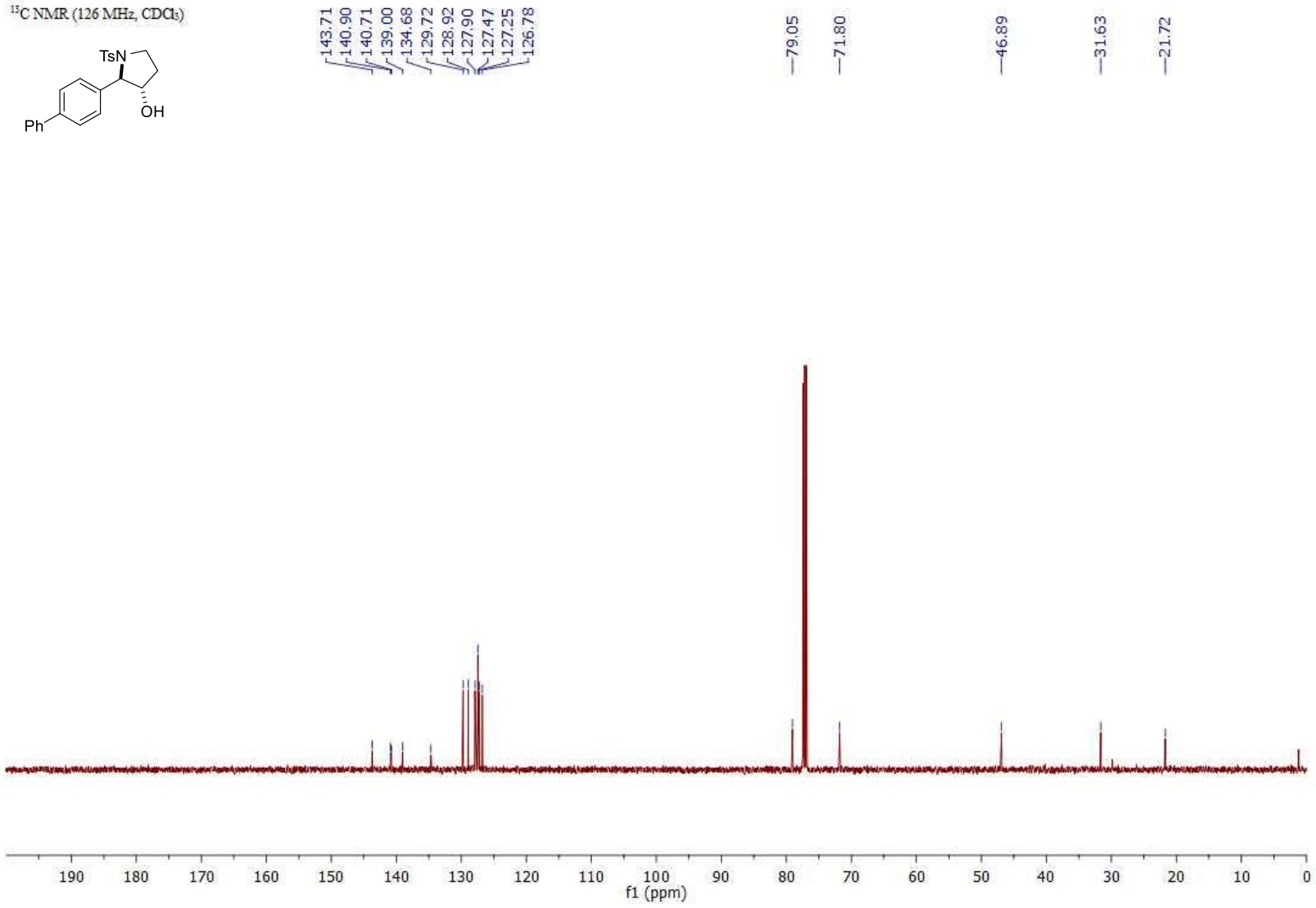
(±)-2-([1,1'-Biphenyl]-4-yl)-1-tosylpyrrolidin-3-ol 16, crude reaction mixture ^{1}H NMR 500 MHz $CDCl_3$



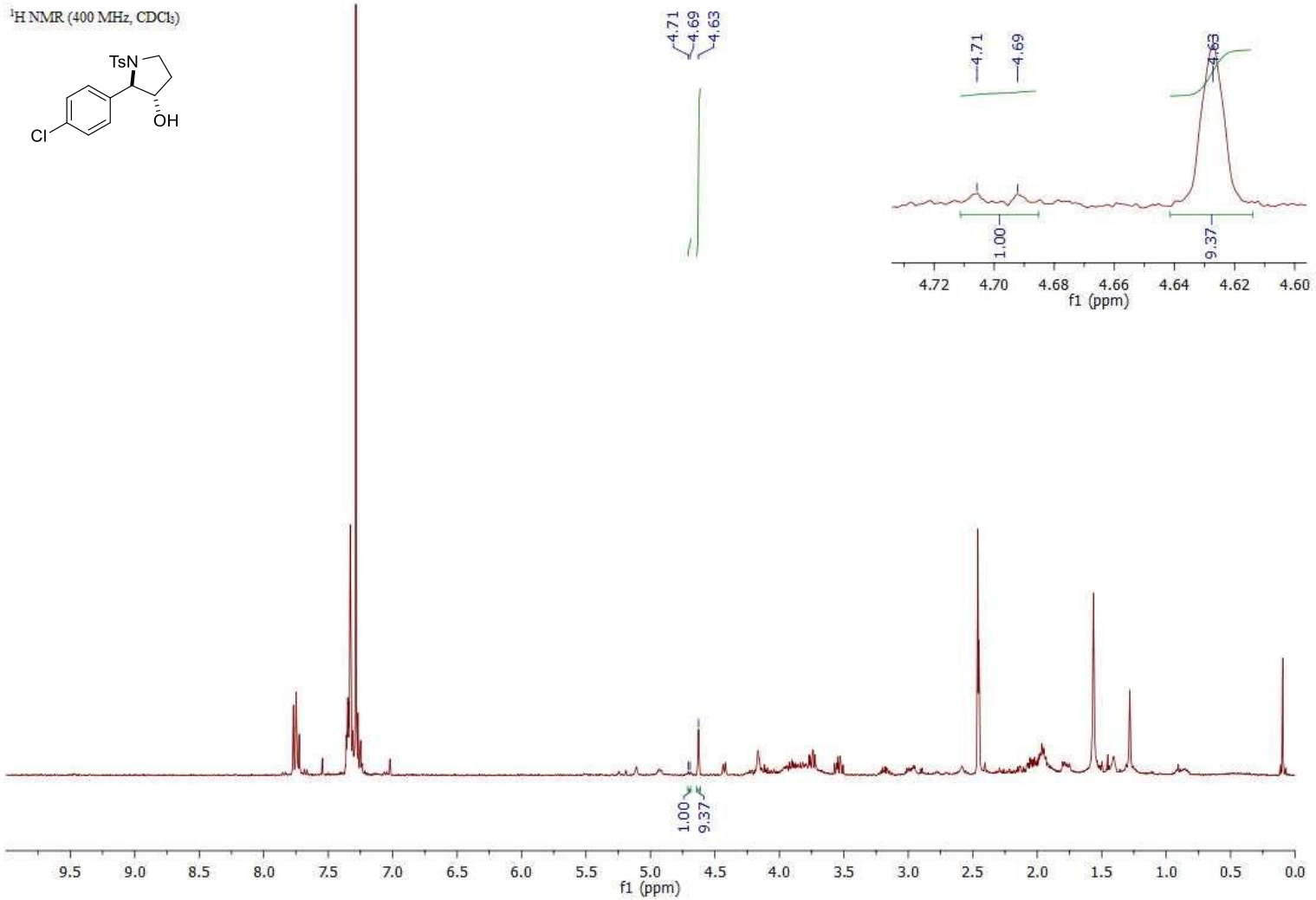
(±)-2-([1,1'-Biphenyl]-4-yl)-1-tosylpyrrolidin-3-ol 16, purified sample ^{1}H NMR 500 MHz $CDCl_3$



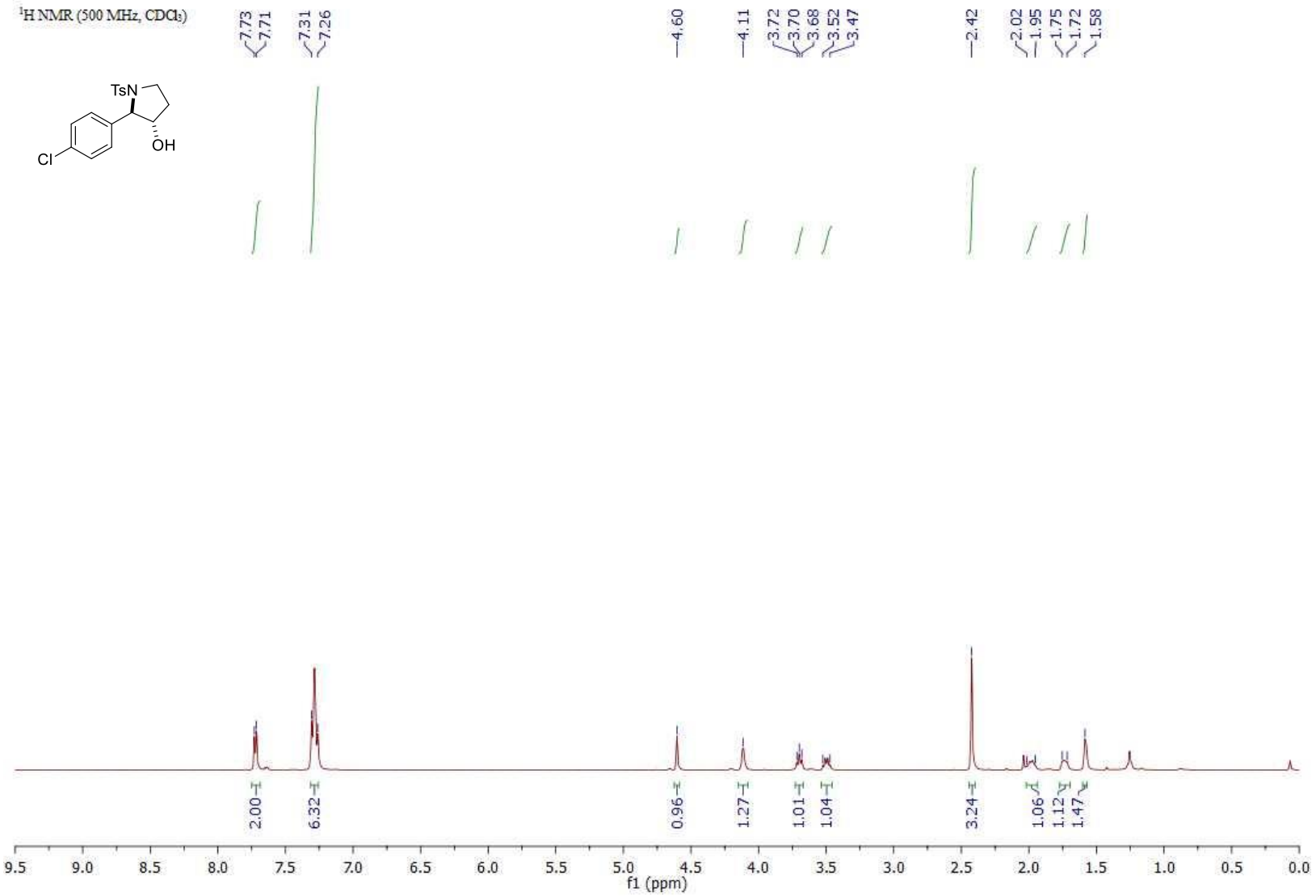
(±)-2-([1,1'-Biphenyl]-4-yl)-1-tosylpyrrolidin-3-ol 16, purified sample ^{13}C NMR 126 MHz $CDCl_3$



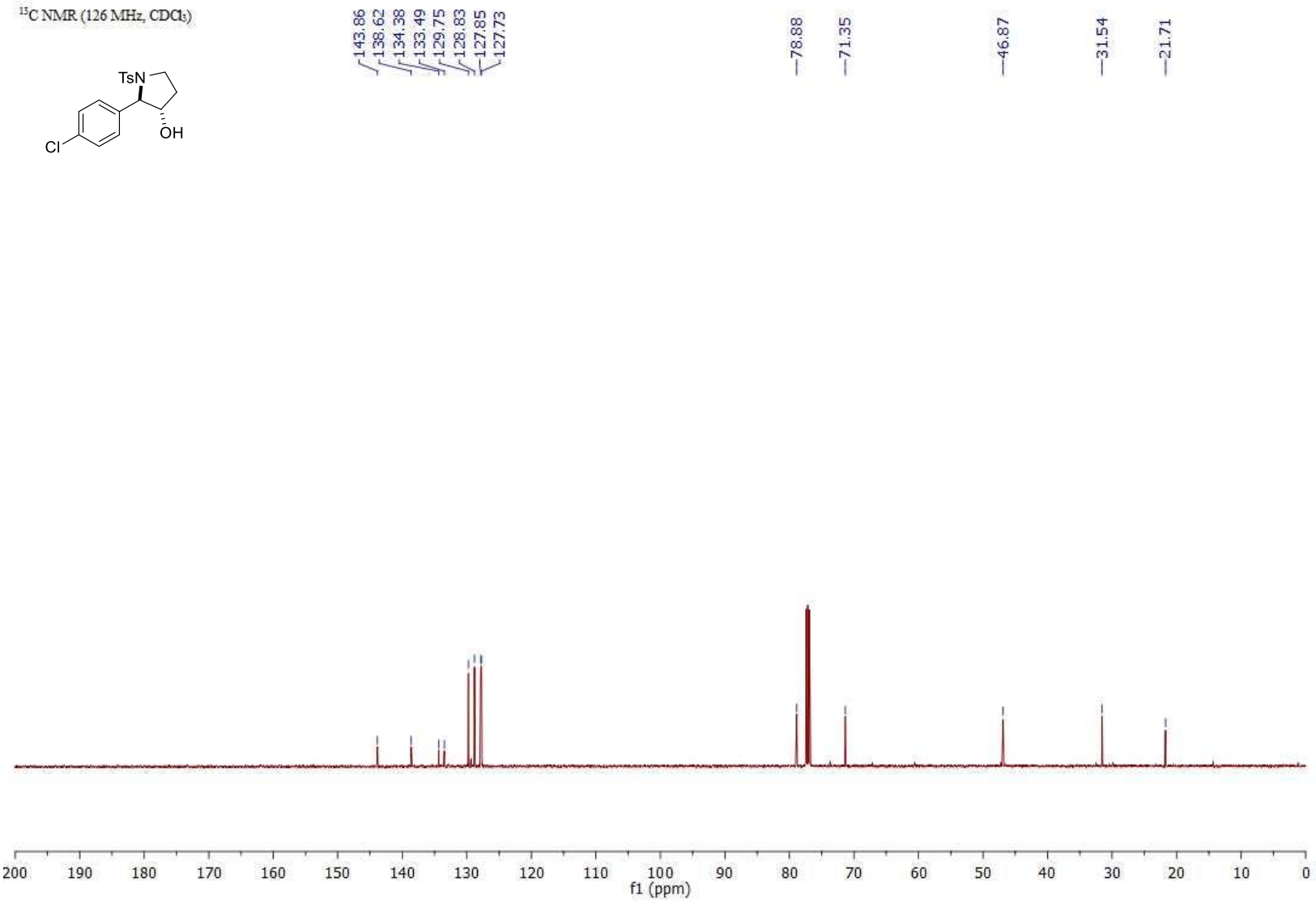
(±)-2-(4-Chlorophenyl)-1-tosylpyrrolidin-3-ol 17, crude reaction mixture ^{1}H NMR 400 MHz $CDCl_3$



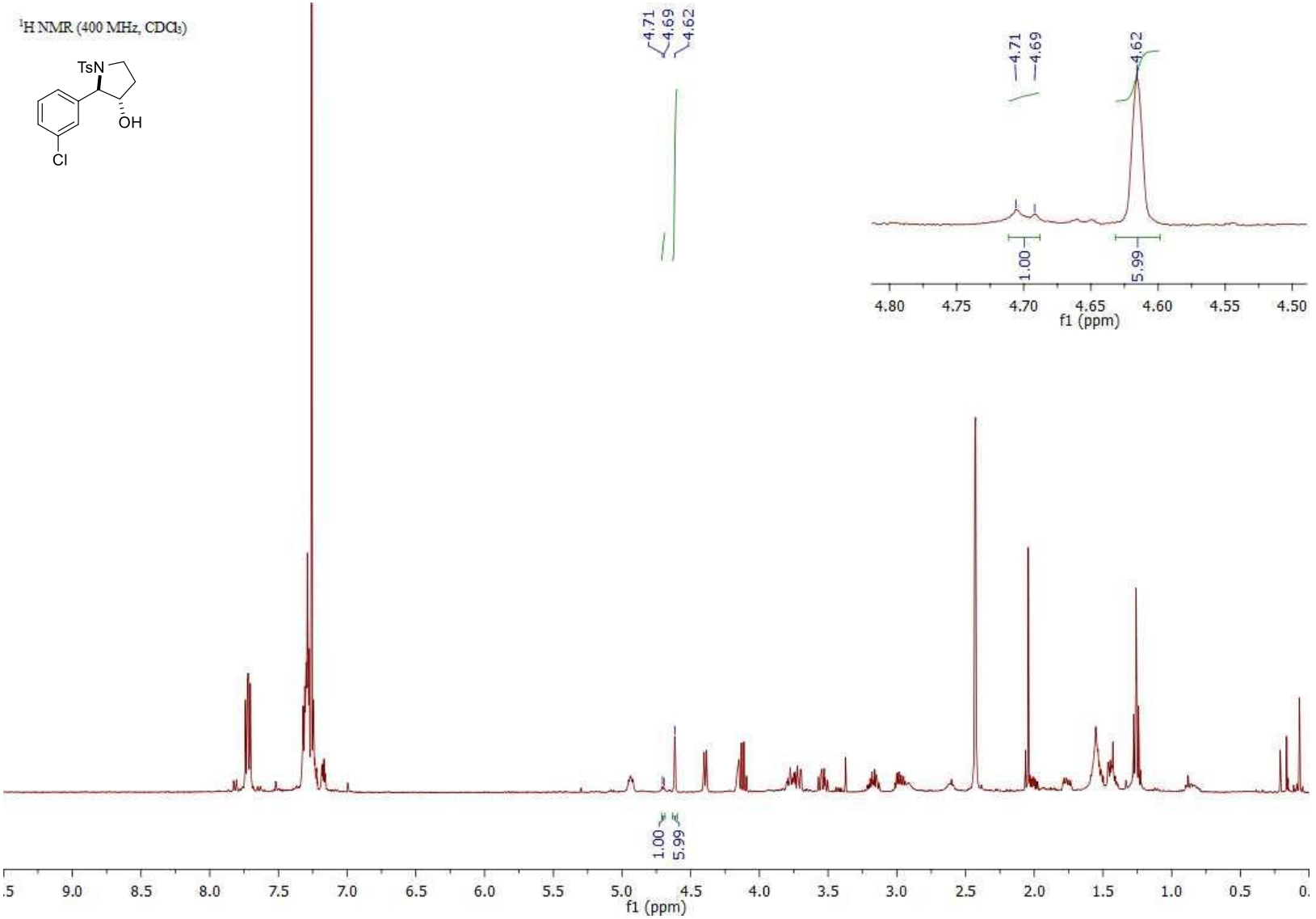
(±)-2-(4-Chlorophenyl)-1-tosylpyrrolidin-3-ol 17, purified sample ^{1}H NMR 500 MHz $CDCl_3$



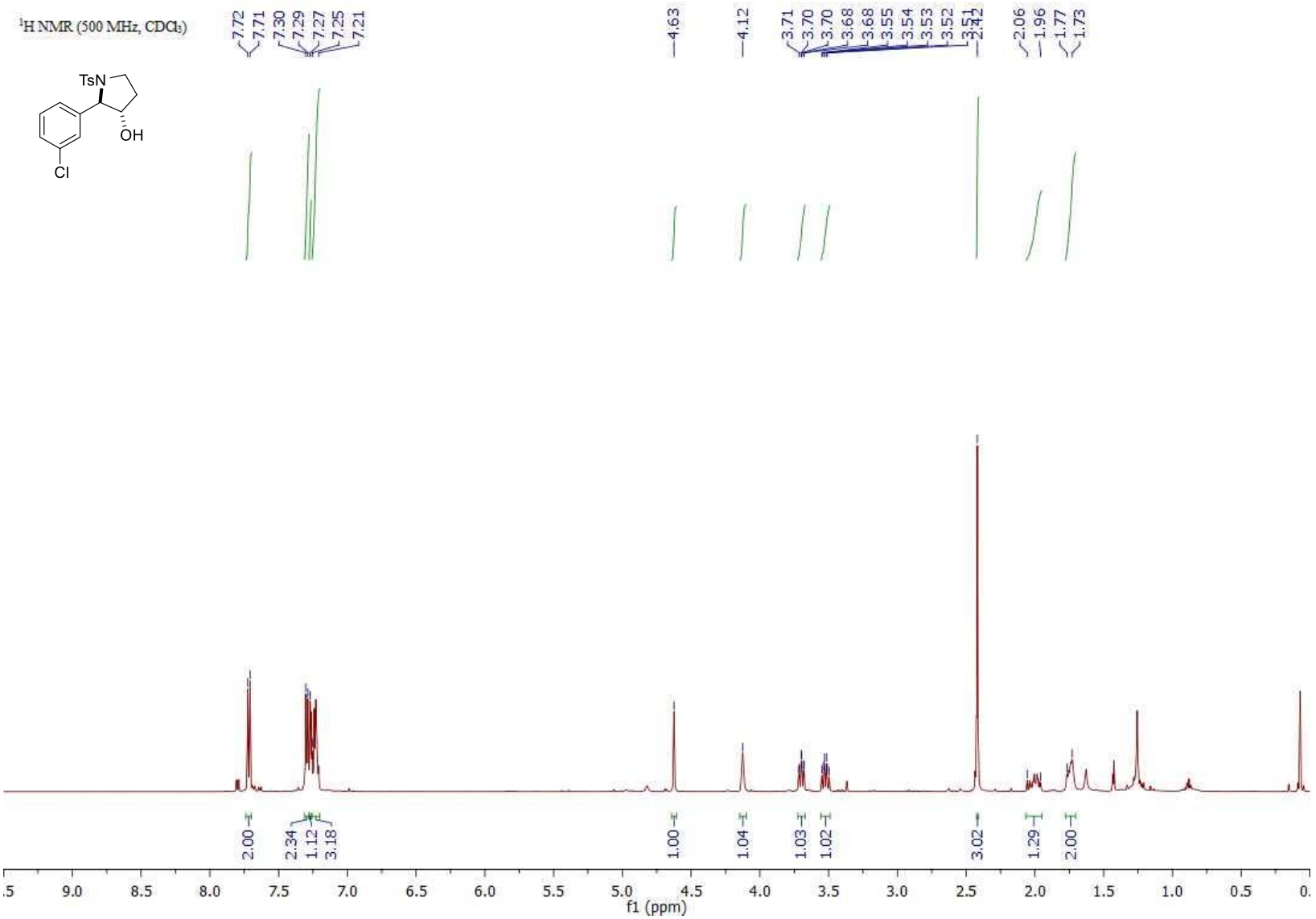
(±)-2-(4-Chlorophenyl)-1-tosylpyrrolidin-3-ol 17, purified sample ^{13}C NMR 126 MHz $CDCl_3$



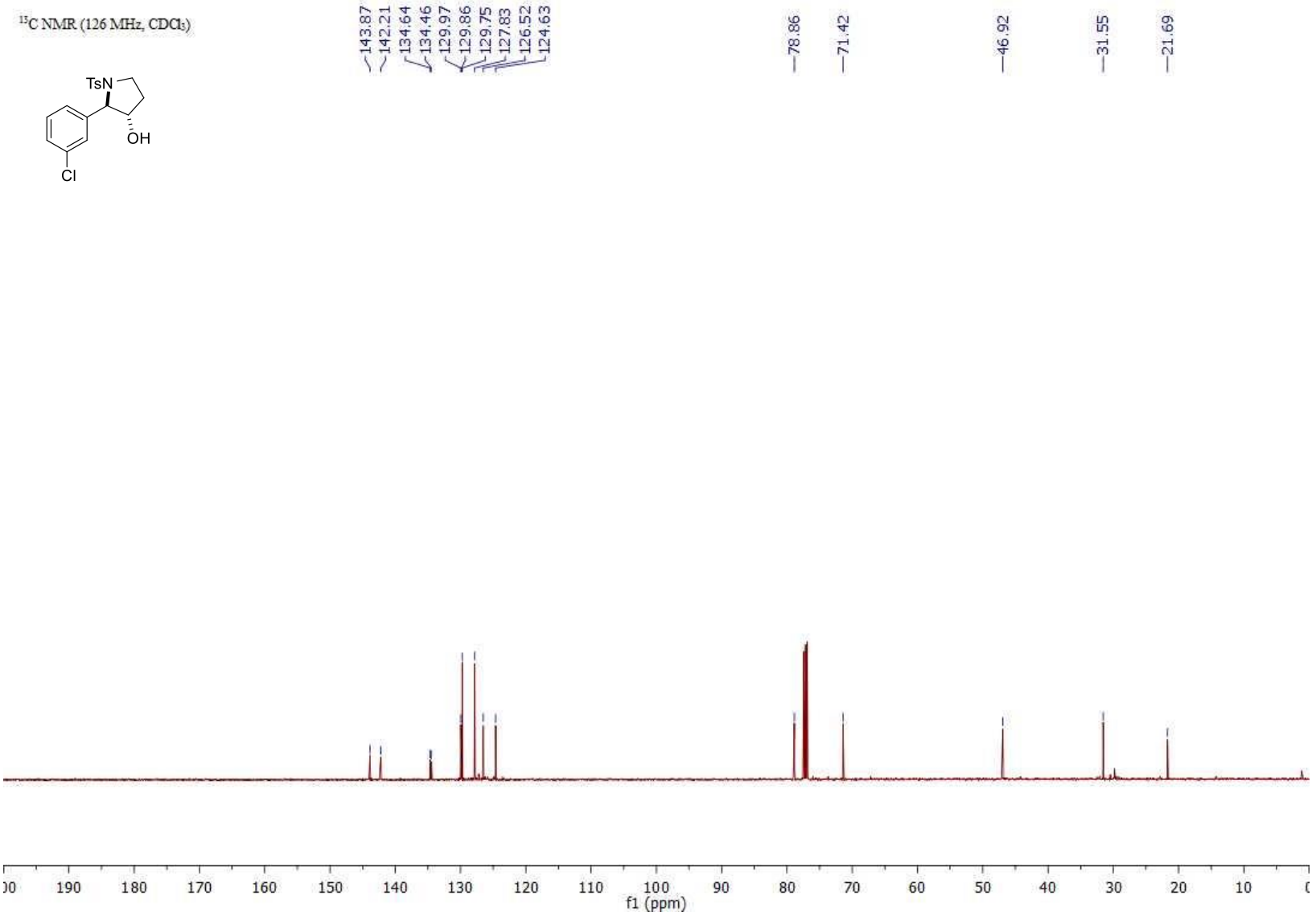
(±)-2-(3-Chlorophenyl)-1-tosylpyrrolidin-3-ol 18, crude reaction mixture ^{1}H NMR 400 MHz $CDCl_3$



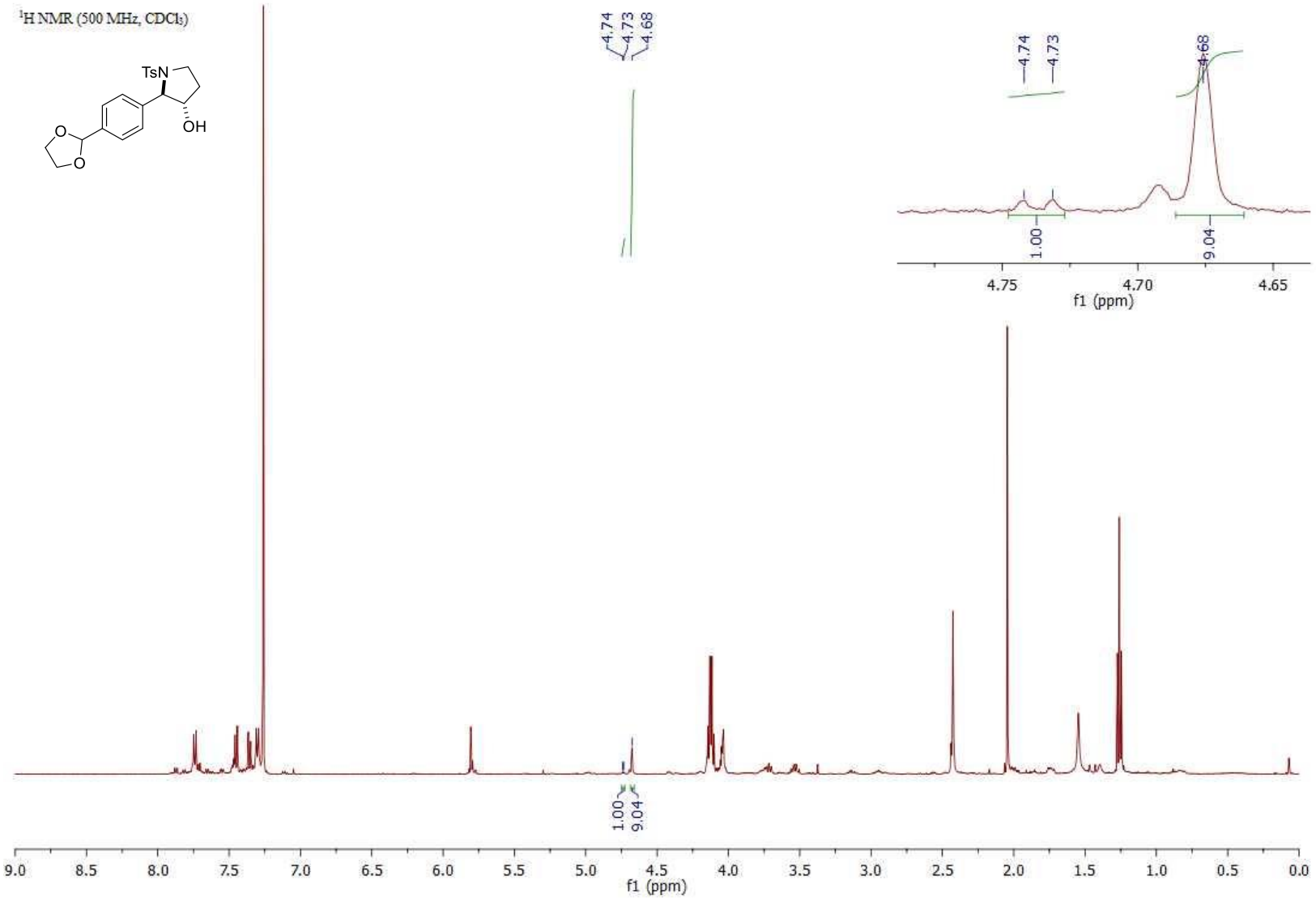
(±)-2-(3-Chlorophenyl)-1-tosylpyrrolidin-3-ol 18, purified sample ^{1}H NMR 500 MHz $CDCl_3$



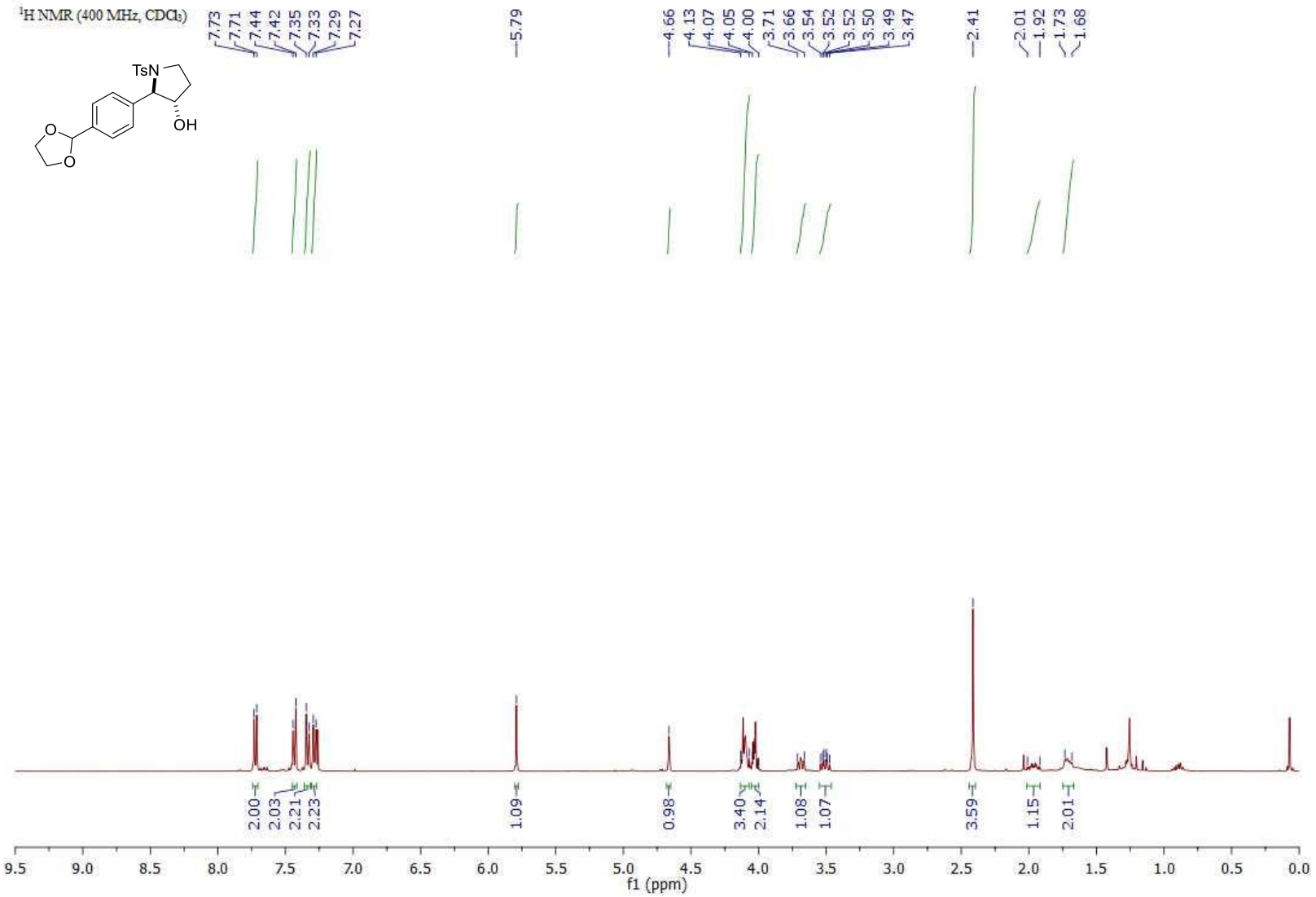
(±)-2-(3-Chlorophenyl)-1-tosylpyrrolidin-3-ol 18, purified sample ^{13}C NMR 126 MHz $CDCl_3$



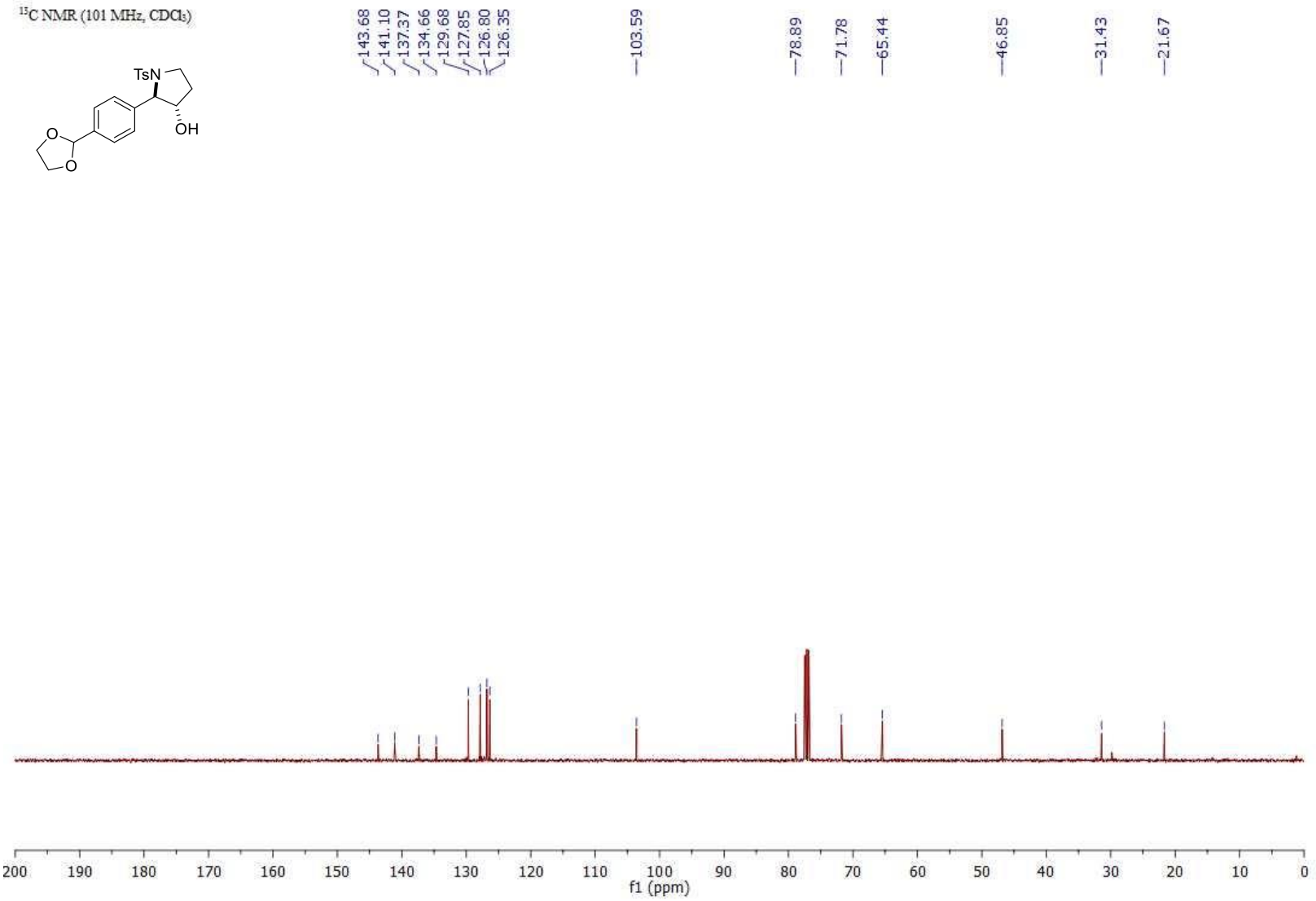
(±)-2-(4-(1,3-Dioxalan-2-yl)phenyl)-1-tosylpyrrolidin-3-ol 19, crude reaction mixture ^{1}H NMR 500 MHz $CDCl_3$



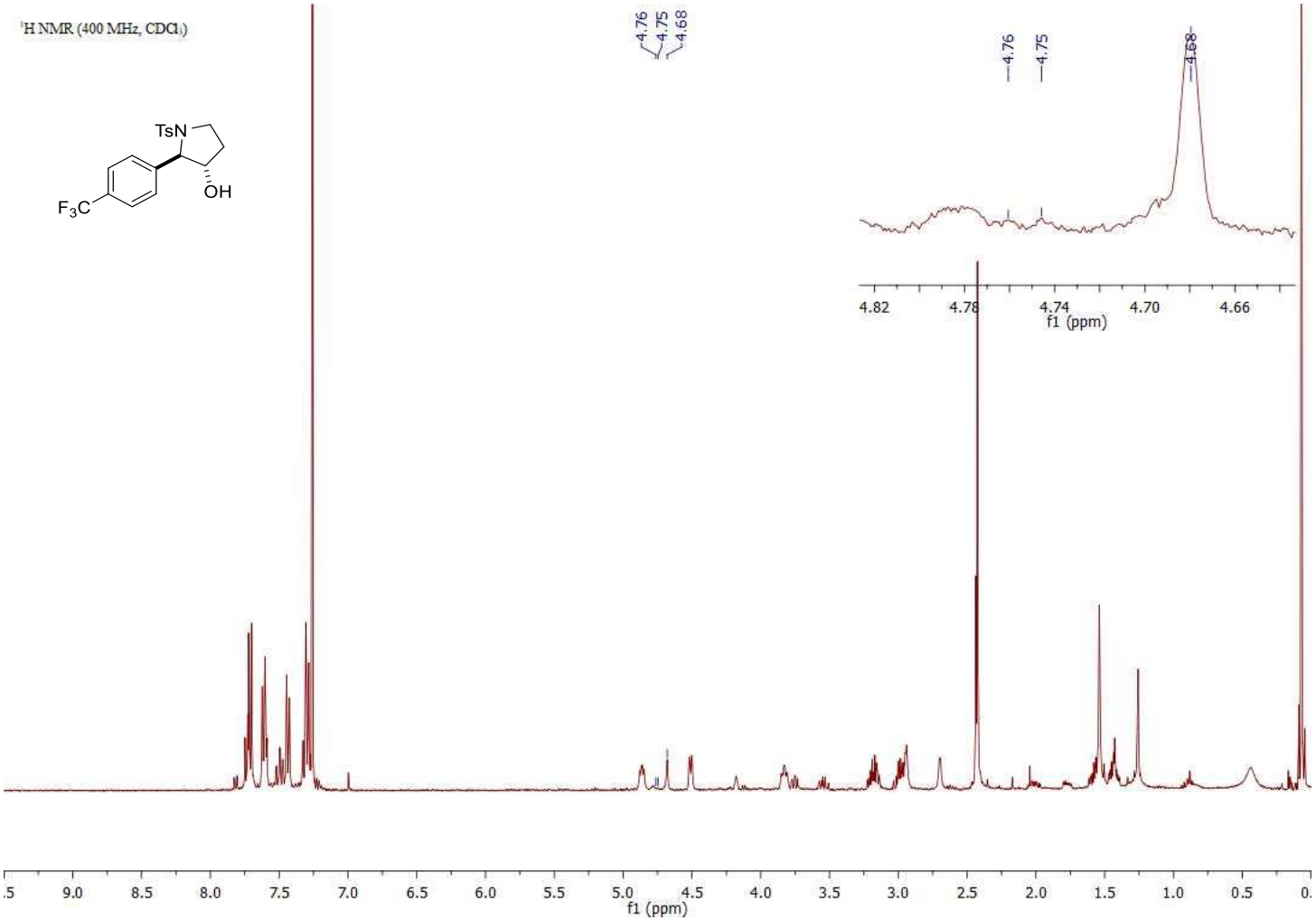
(±)-2-(4-(1,3-Dioxalan-2-yl)phenyl)-1-tosylpyrrolidin-3-ol 19, purified sample ^{1}H NMR 400 MHz $CDCl_3$



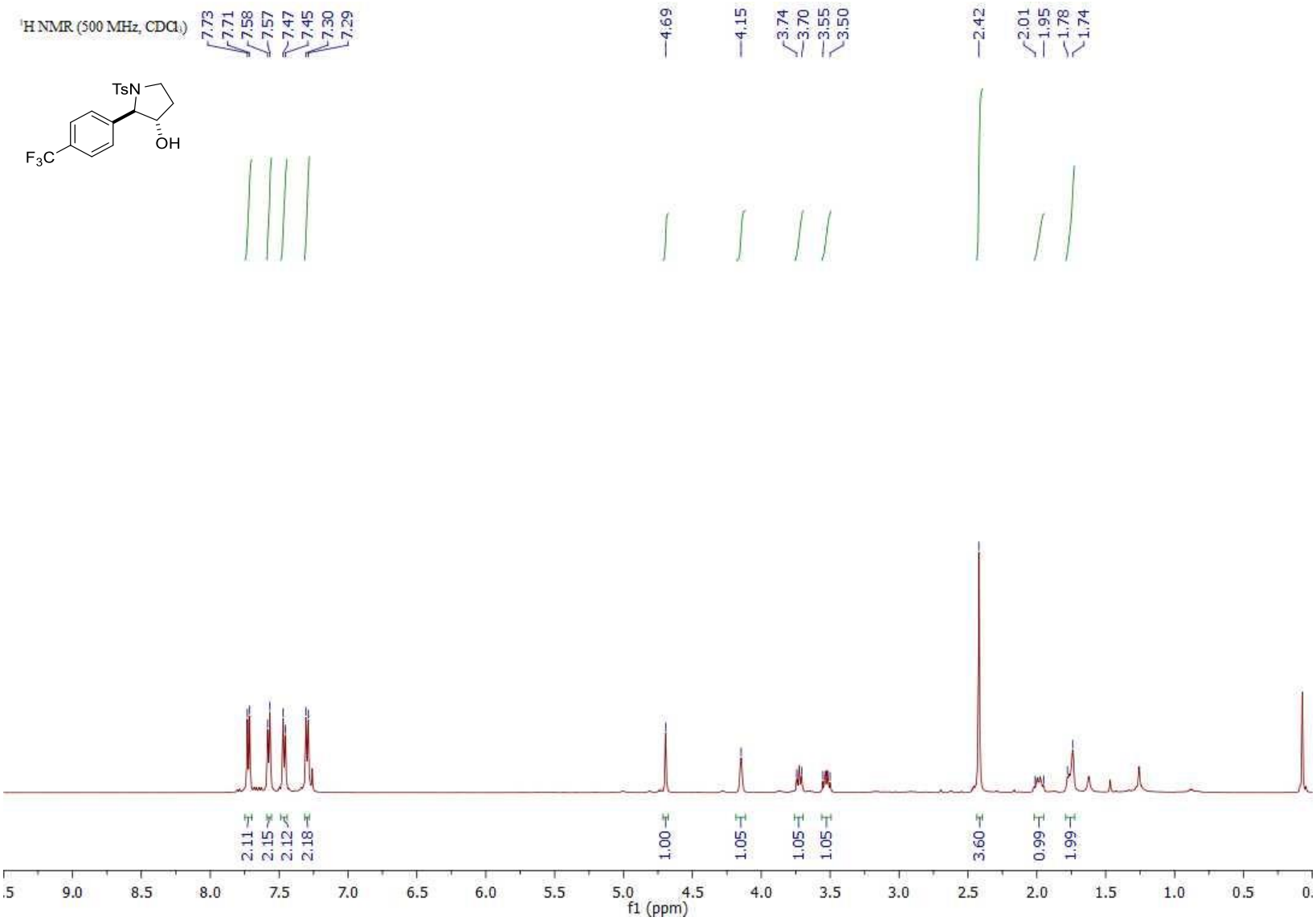
(±)-2-(4-(1,3-Dioxalan-2-yl)phenyl)-1-tosylpyrrolidin-3-ol 19, purified sample ^{13}C NMR 101 MHz $CDCl_3$



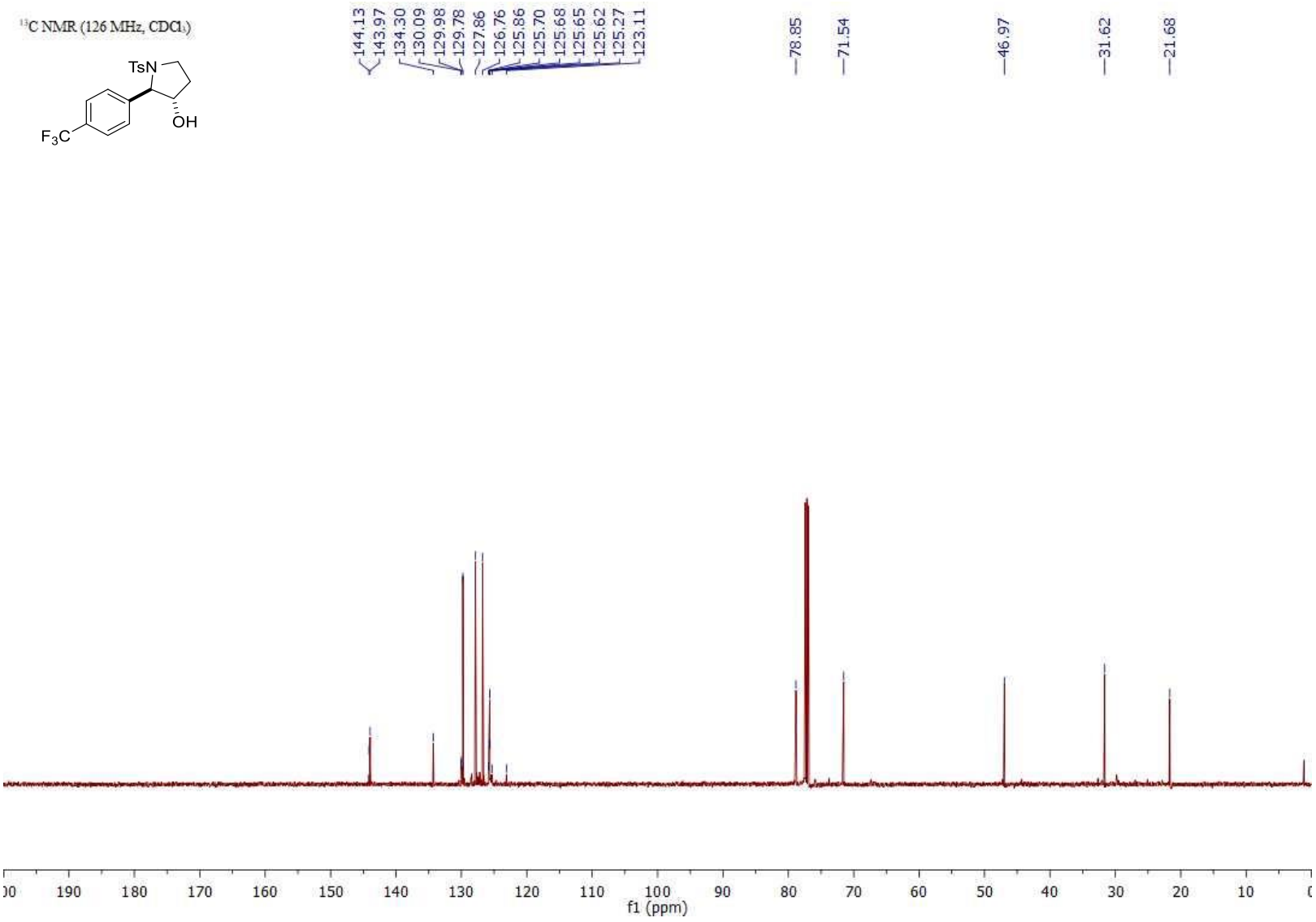
(±)-(2*R*,3*S*)-1-Tosyl-2-(4-(trifluoromethyl)phenyl)pyrrolidine-3-ol 20, crude reaction mixture ^{1}H NMR 400 MHz $CDCl_3$ diastereomeric ratio not assigned.



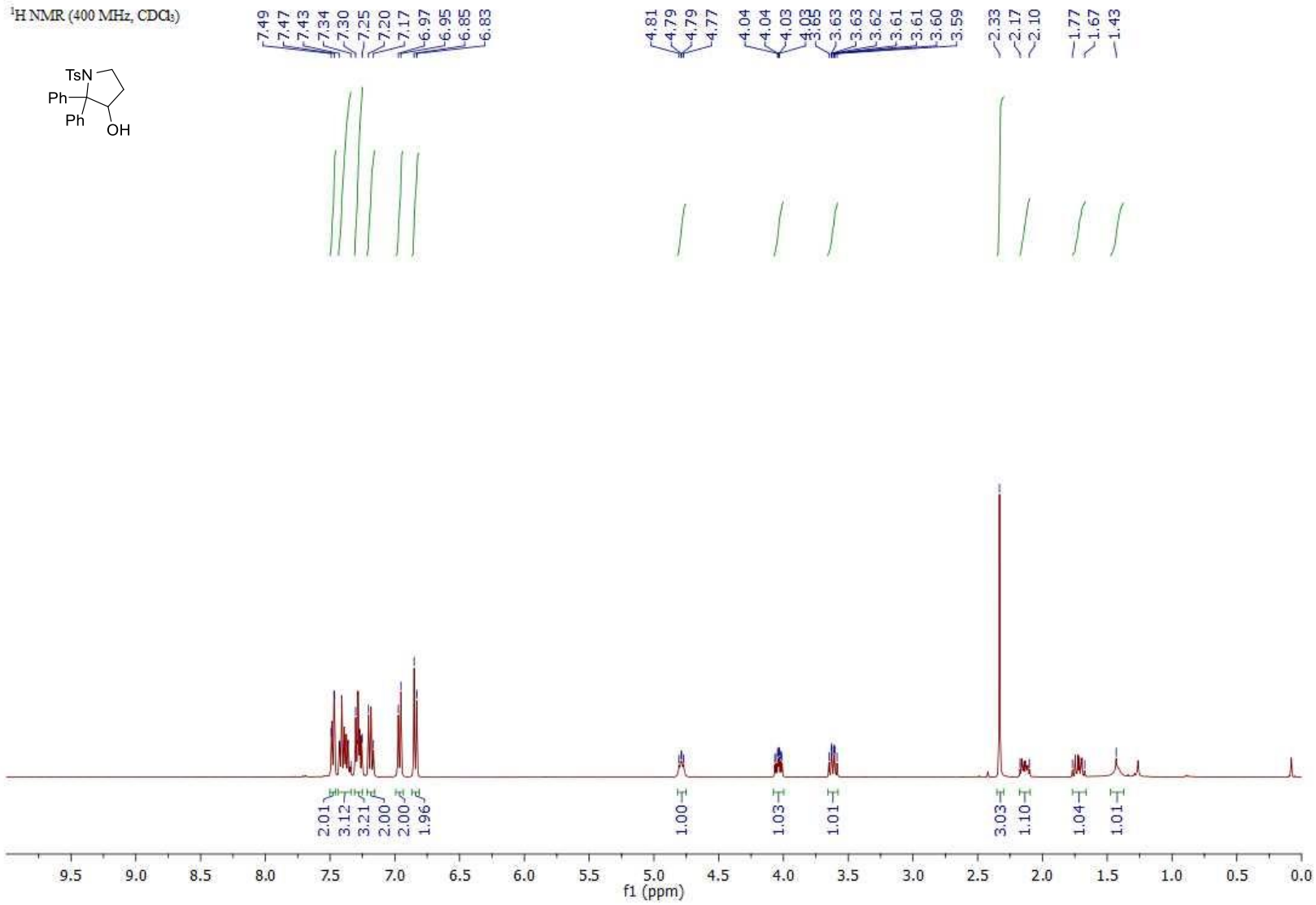
(±)-1-Tosyl-2-(4-(trifluoromethyl)phenyl)pyrrolidine-3-ol 20, purified sample ^{1}H NMR 500 MHz $CDCl_3$



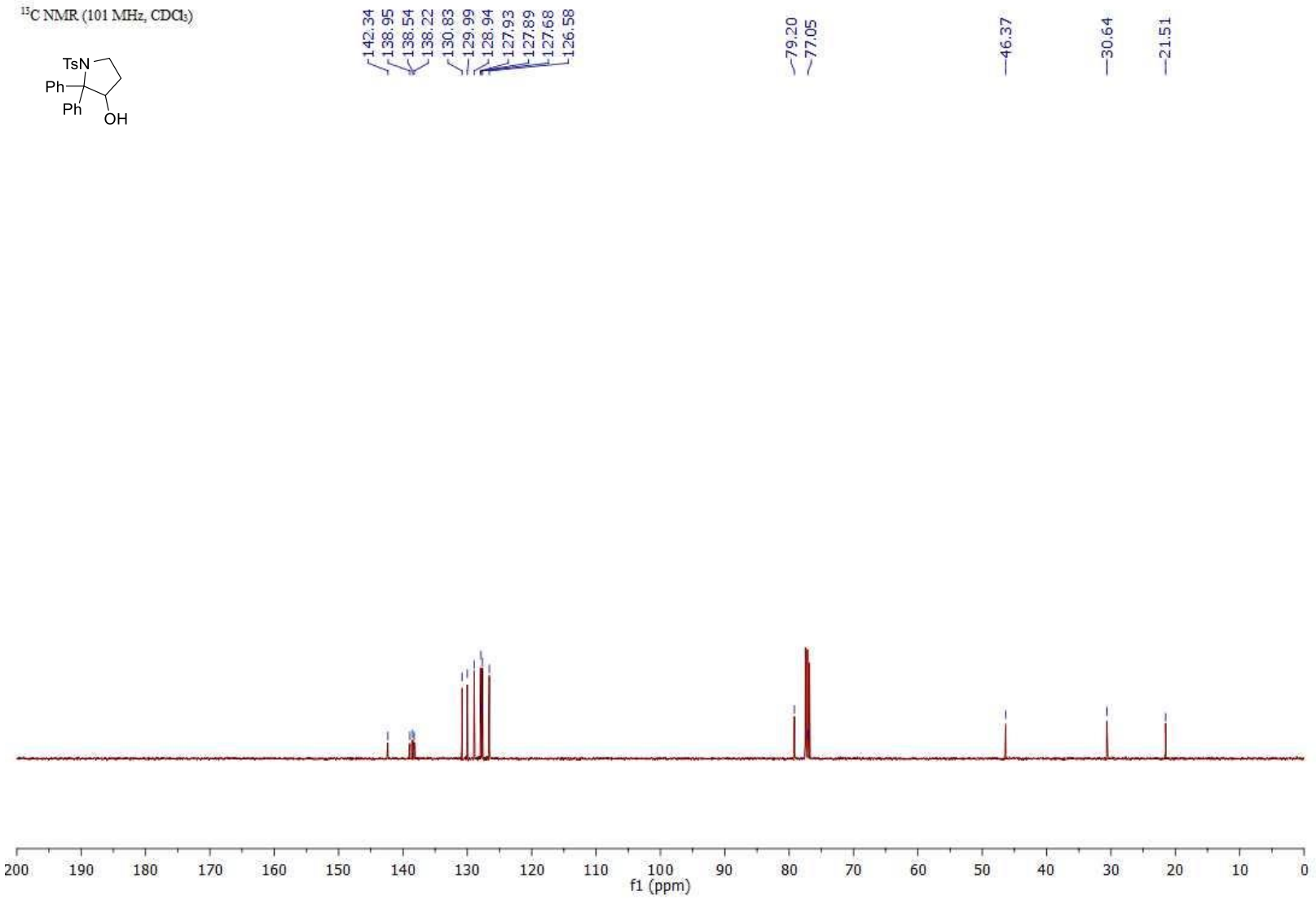
(±)-1-Tosyl-2-(4-(trifluoromethyl)phenyl)pyrrolidine-3-ol 20, purified sample ^{13}C NMR 126 MHz $CDCl_3$



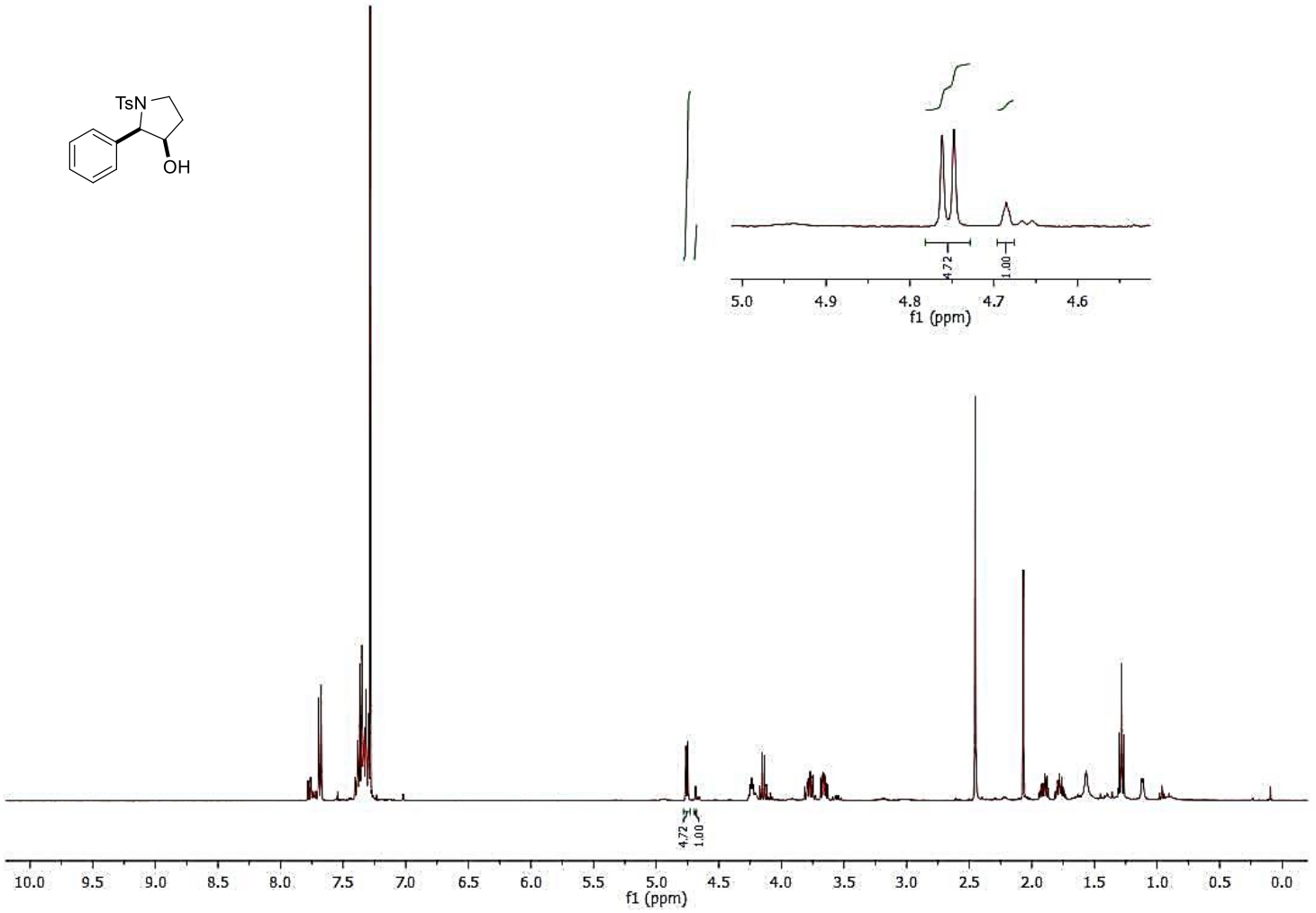
(±)-2,2-Diphenyl-1-tosylpyrrolidin-3-ol 21, purified sample ^{1}H NMR 400 MHz $CDCl_3$



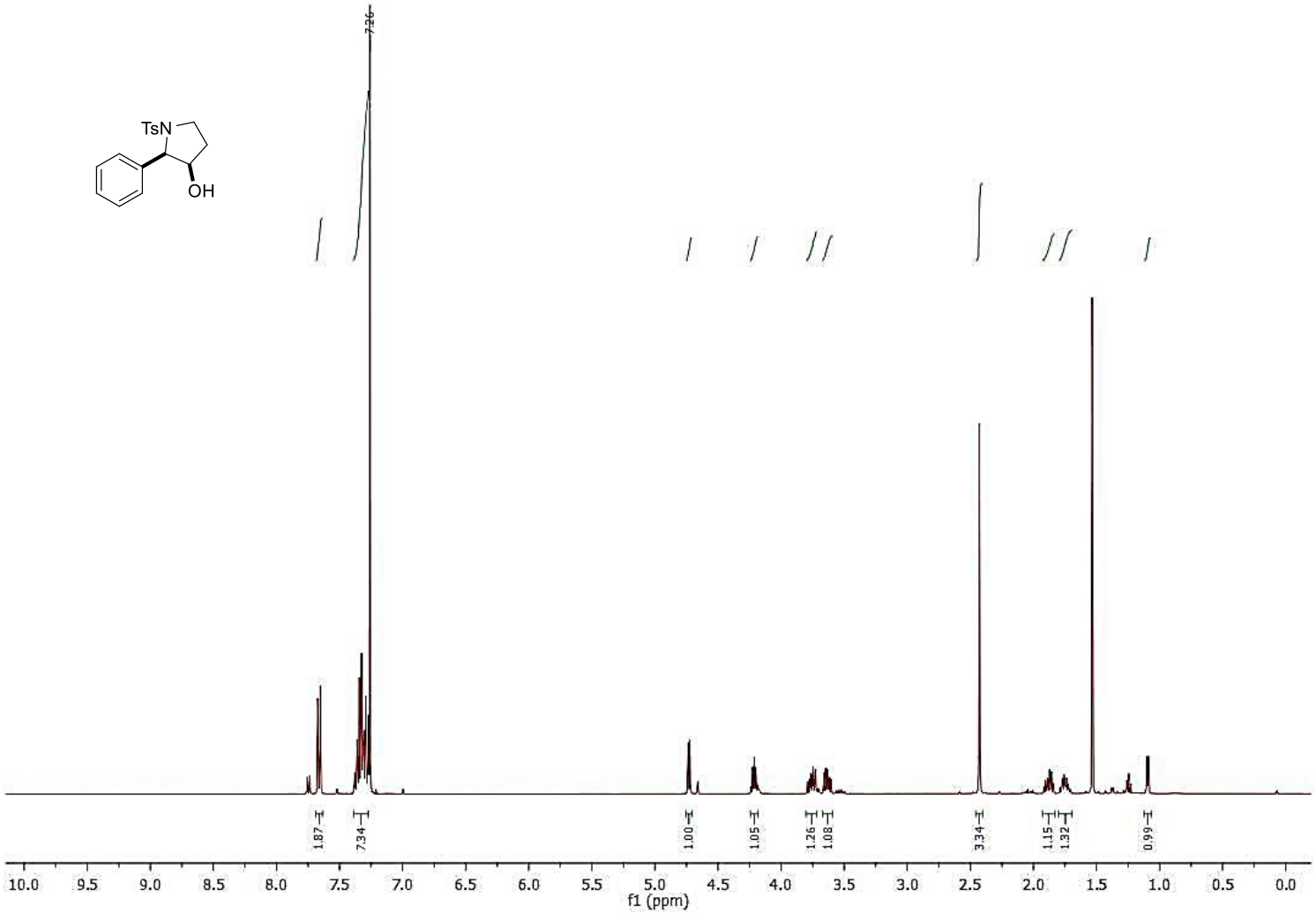
(±)-2,2-Diphenyl-1-tosylpyrrolidin-3-ol 21, purified sample ^{13}C NMR 101 MHz $CDCl_3$



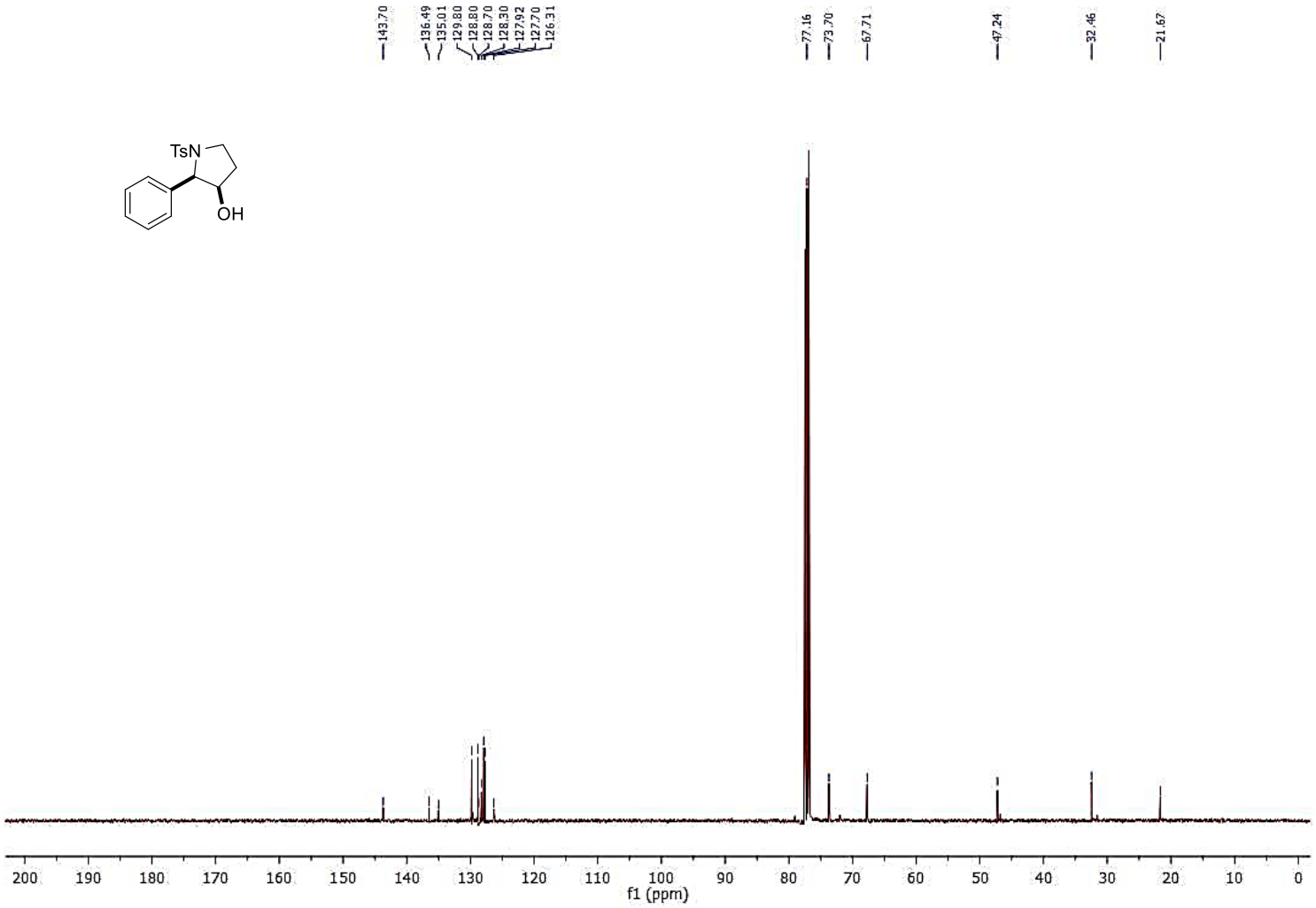
(±)-2-Phenyl-1-tosylpyrrolidin-3-ol (22) crude reaction mixture ^{1}H NMR 400 MHz $CDCl_3$



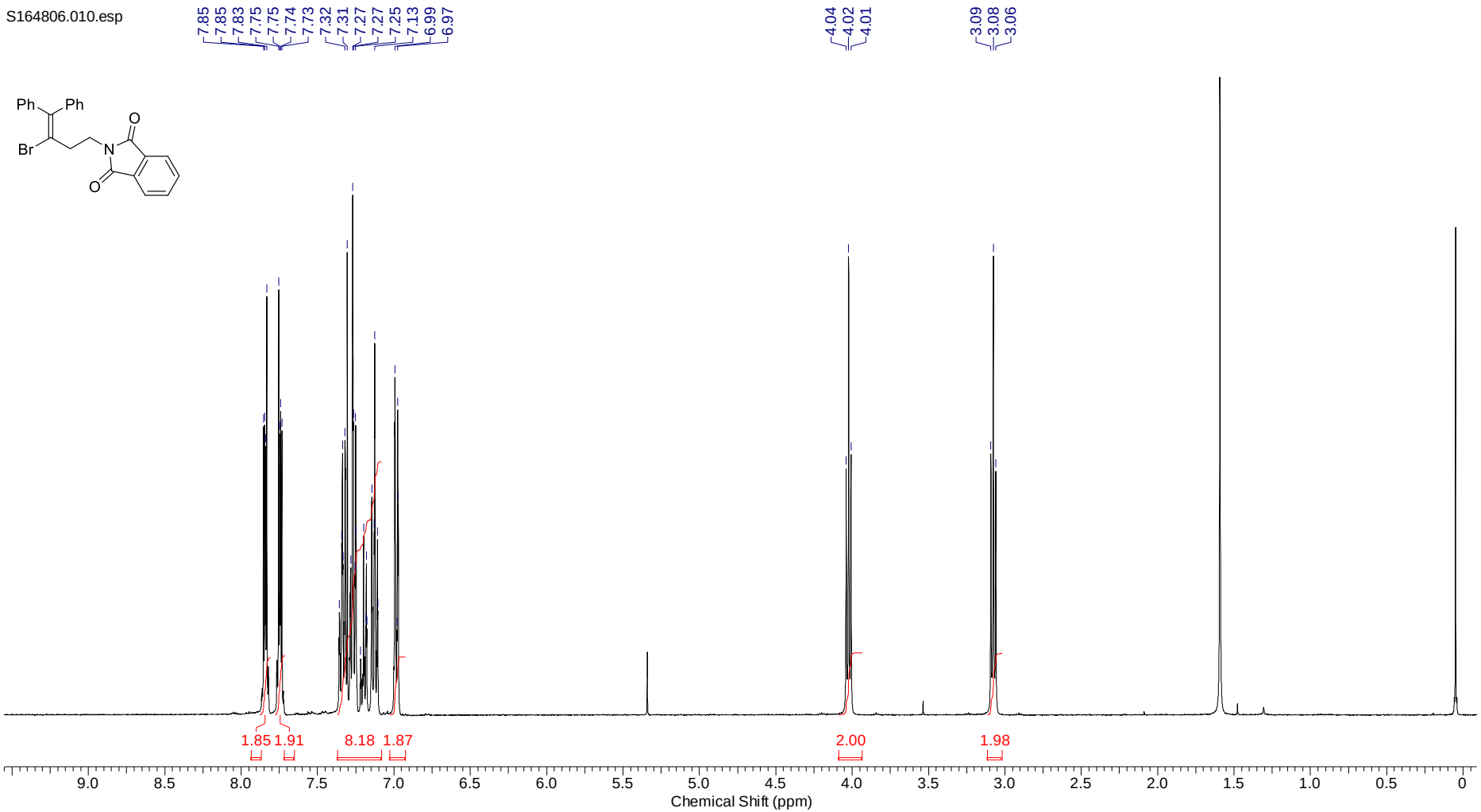
(±)-2-Phenyl-1-tosylpyrrolidin-3-ol (22) purified material ^{1}H NMR 400 MHz $CDCl_3$



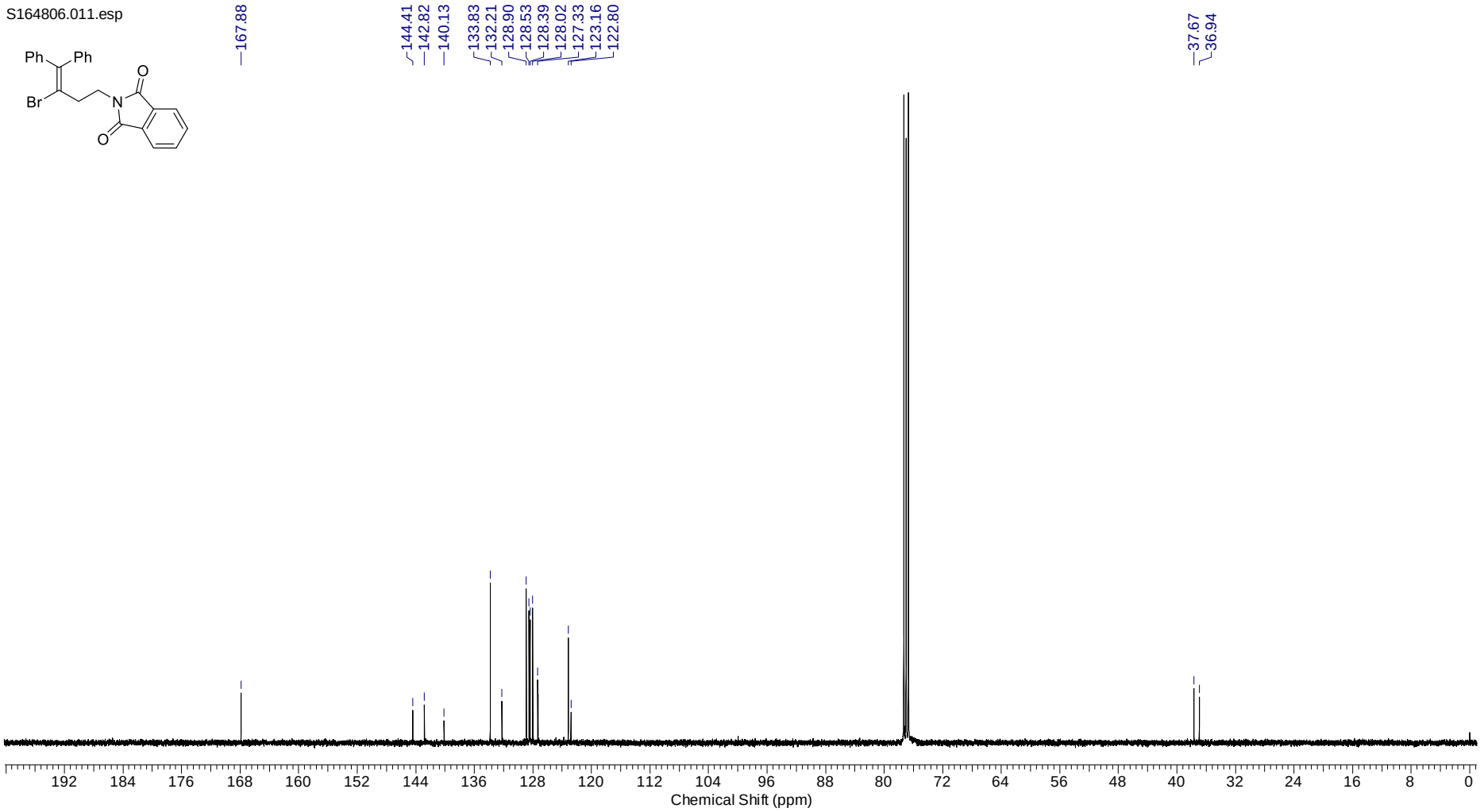
(±)-2-Phenyl-1-tosylpyrrolidin-3-ol (22) purified material ^{13}C NMR 101 MHz $CDCl_3$



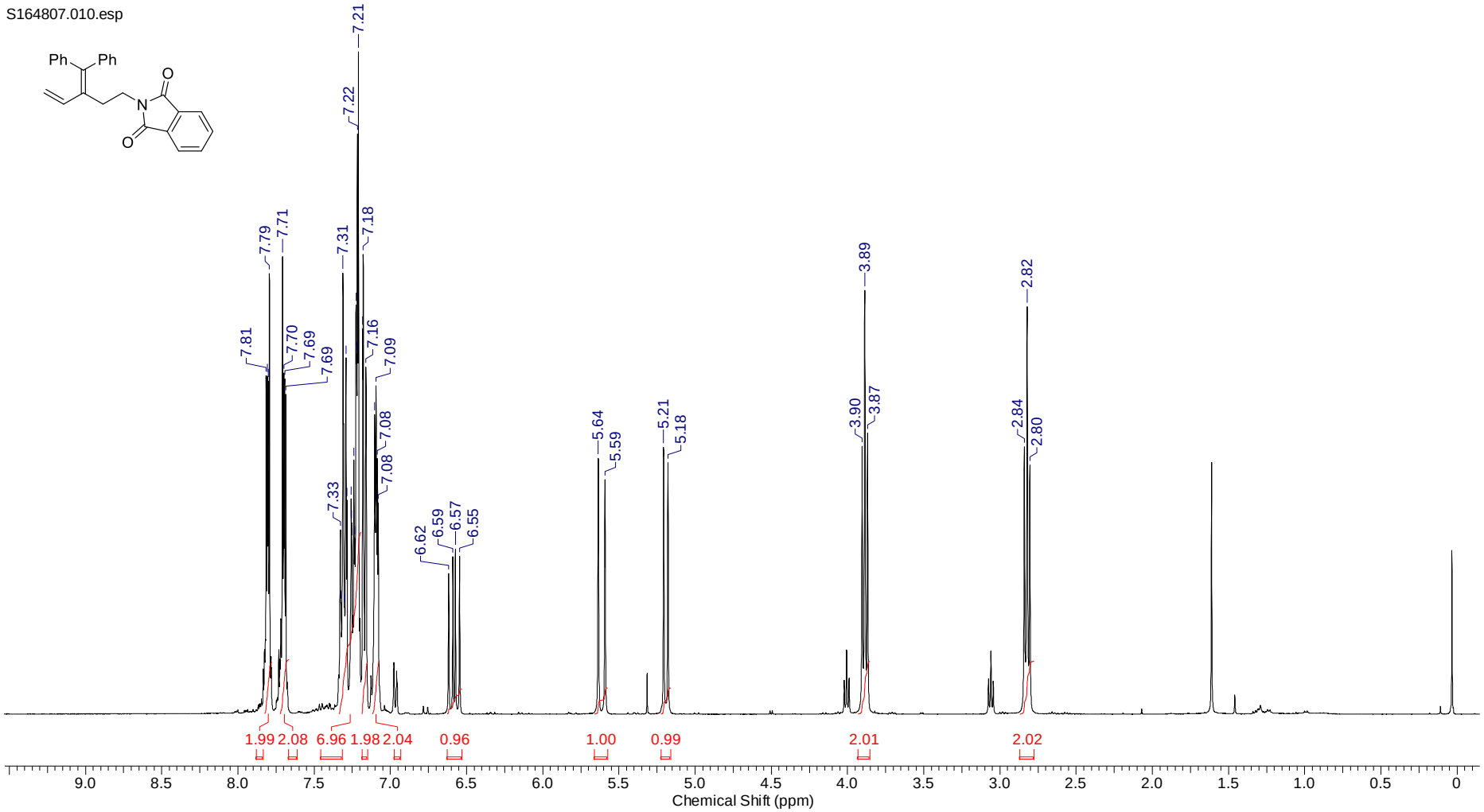
2-(3-Bromo-4,4-diphenylbut-3-en-1-yl)isoindoline-1,3-dione, ^{1}H NMR 400 MHz $CDCl_3$



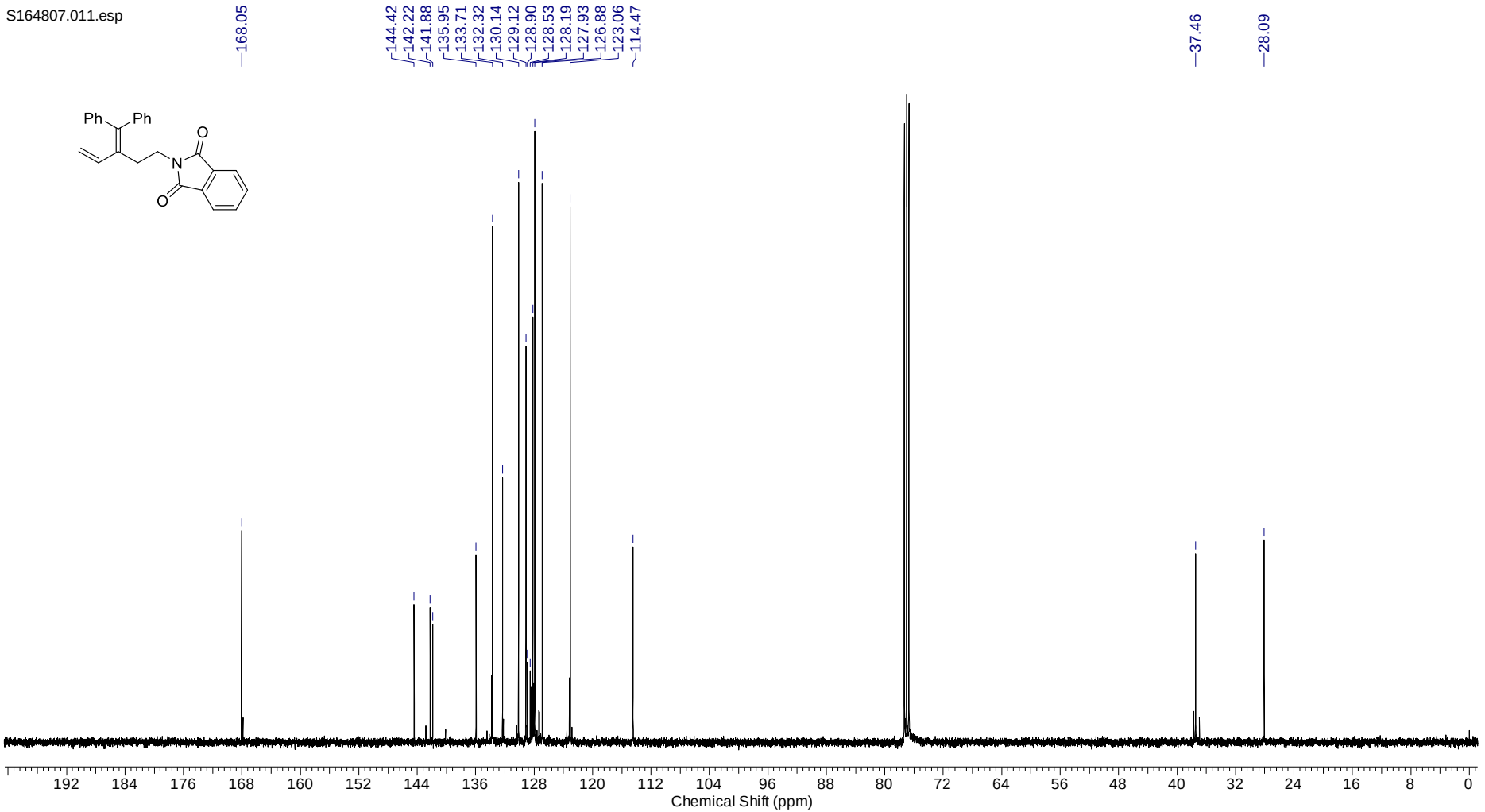
2-(3-Bromo-4,4-diphenylbut-3-en-1-yl)isoindoline-1,3-dione, ^{13}C NMR 101 MHz $CDCl_3$



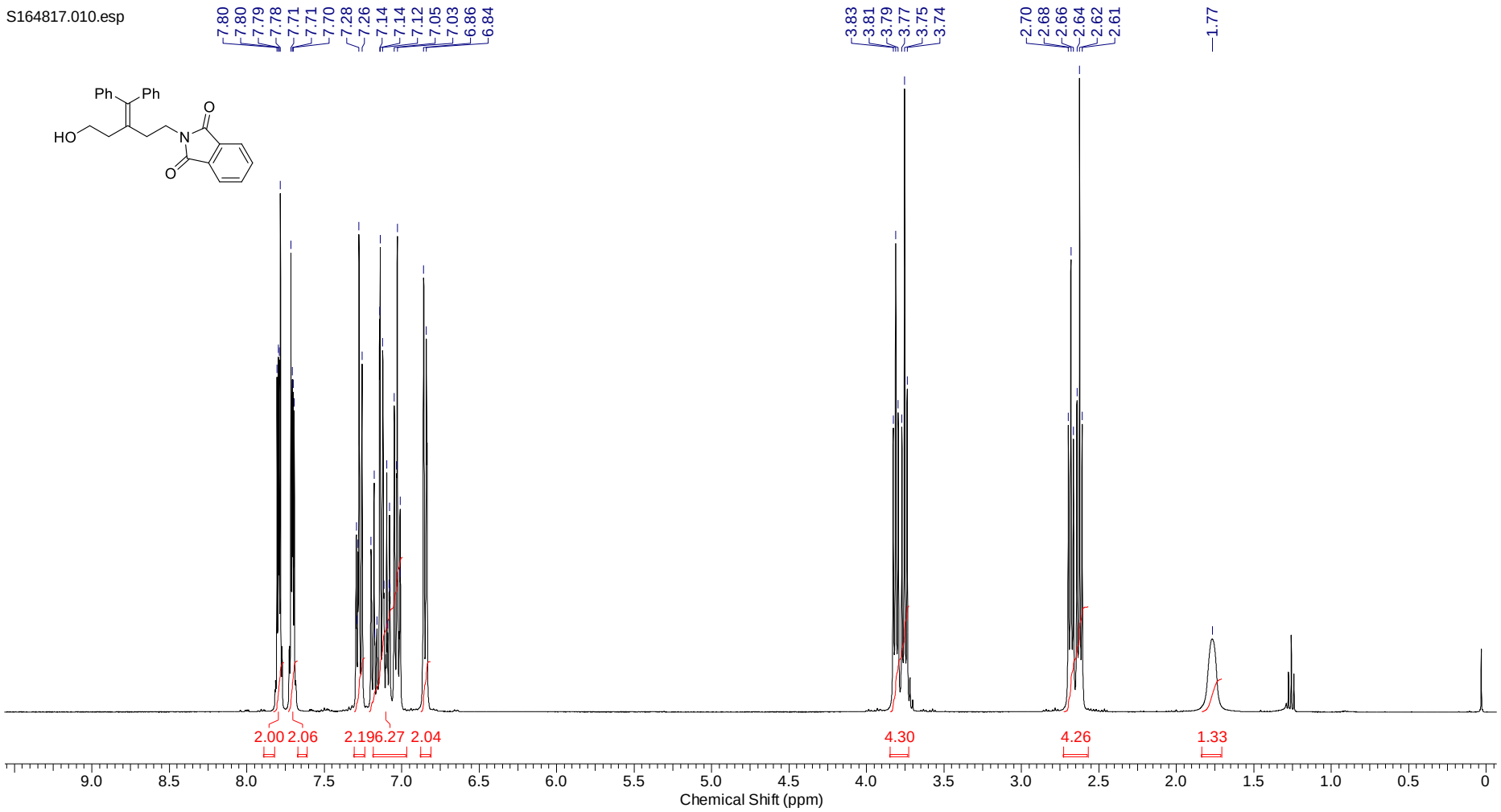
2-(3-(Diphenylmethylene)pent-4-en-1-yl)isoindoline-1,3-dione, ^{1}H NMR 400 MHz $CDCl_3$



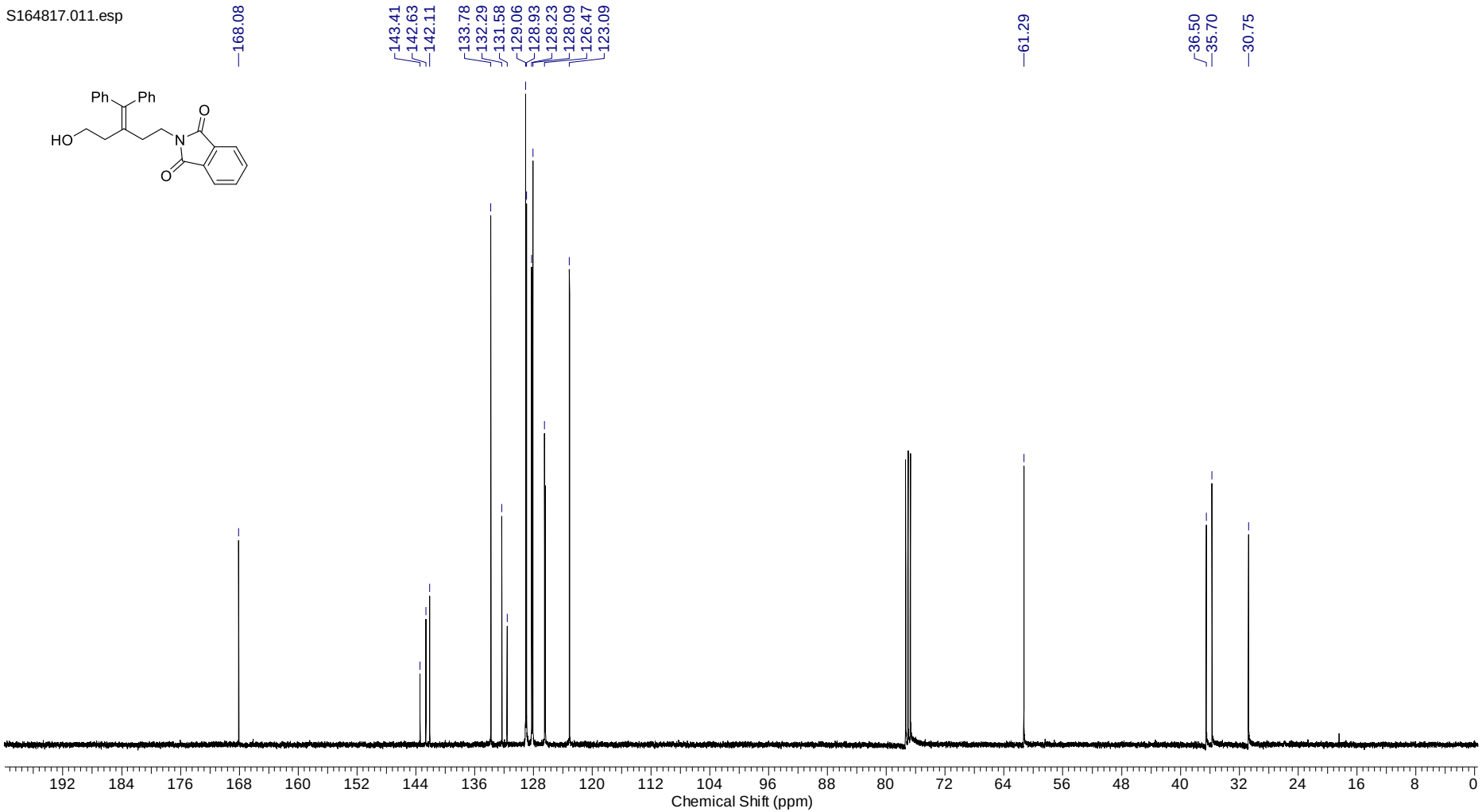
2-(3-(Diphenylmethylene)pent-4-en-1-yl)isoindoline-1,3-dione, ^{13}C NMR 101 MHz $CDCl_3$



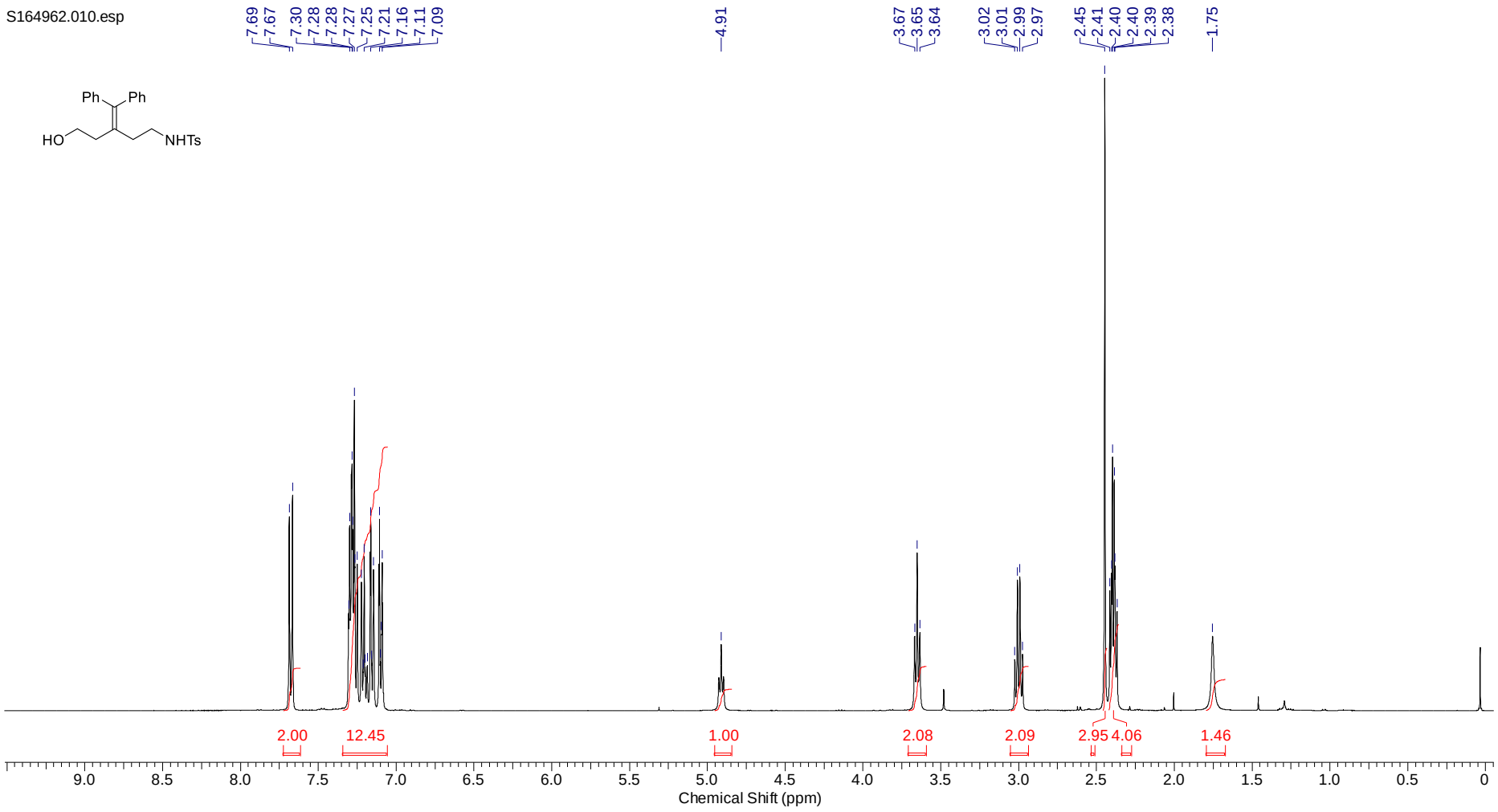
2-(3-(Diphenylmethylene)-5-hydroxypentyl)isoindoline-1,3-dione, ^{1}H NMR 400 MHz $CDCl_3$



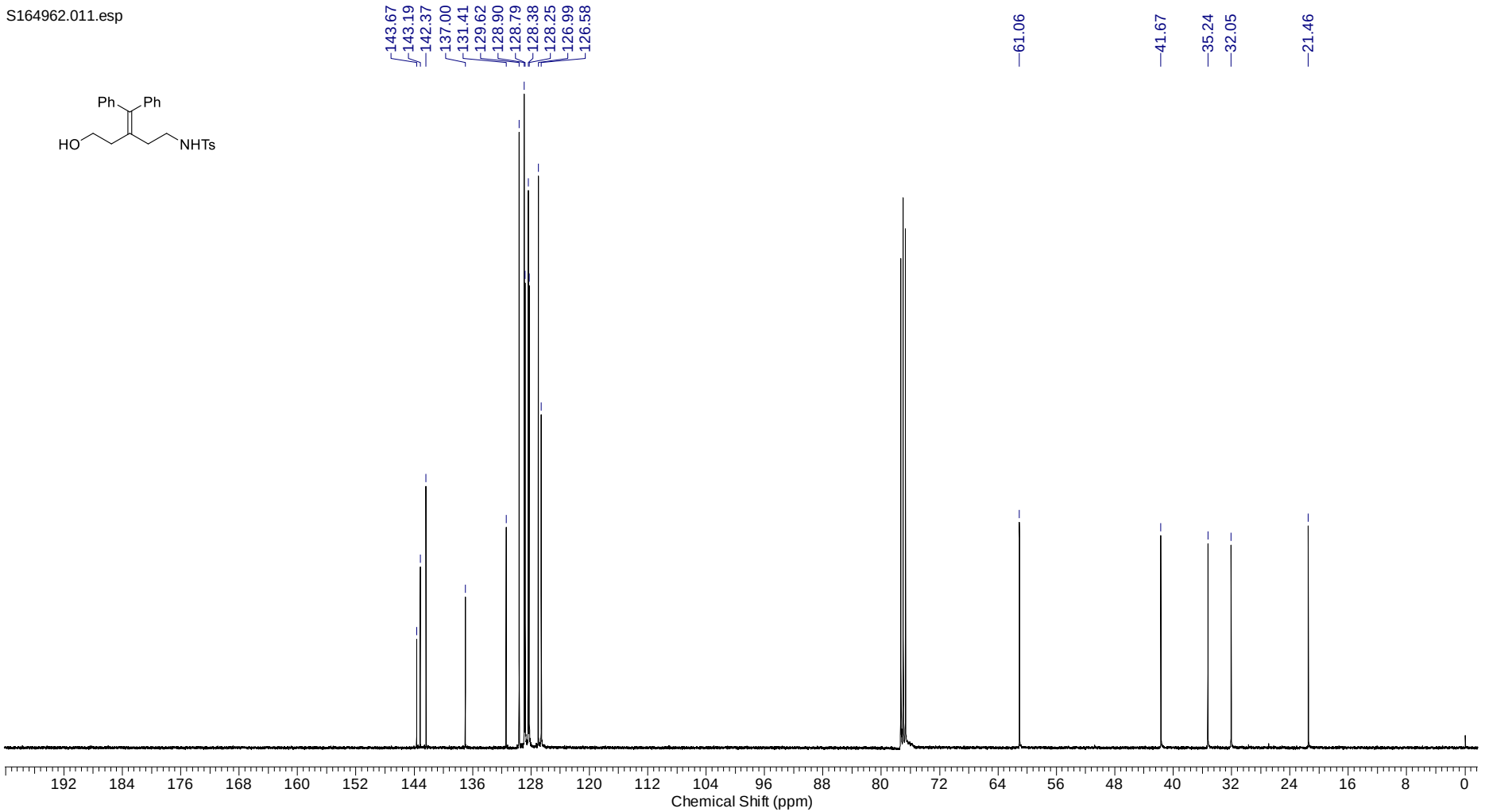
2-(3-(Diphenylmethylene)-5-hydroxypentyl)isoindoline-1,3-dione, ^{13}C NMR 101 MHz $CDCl_3$



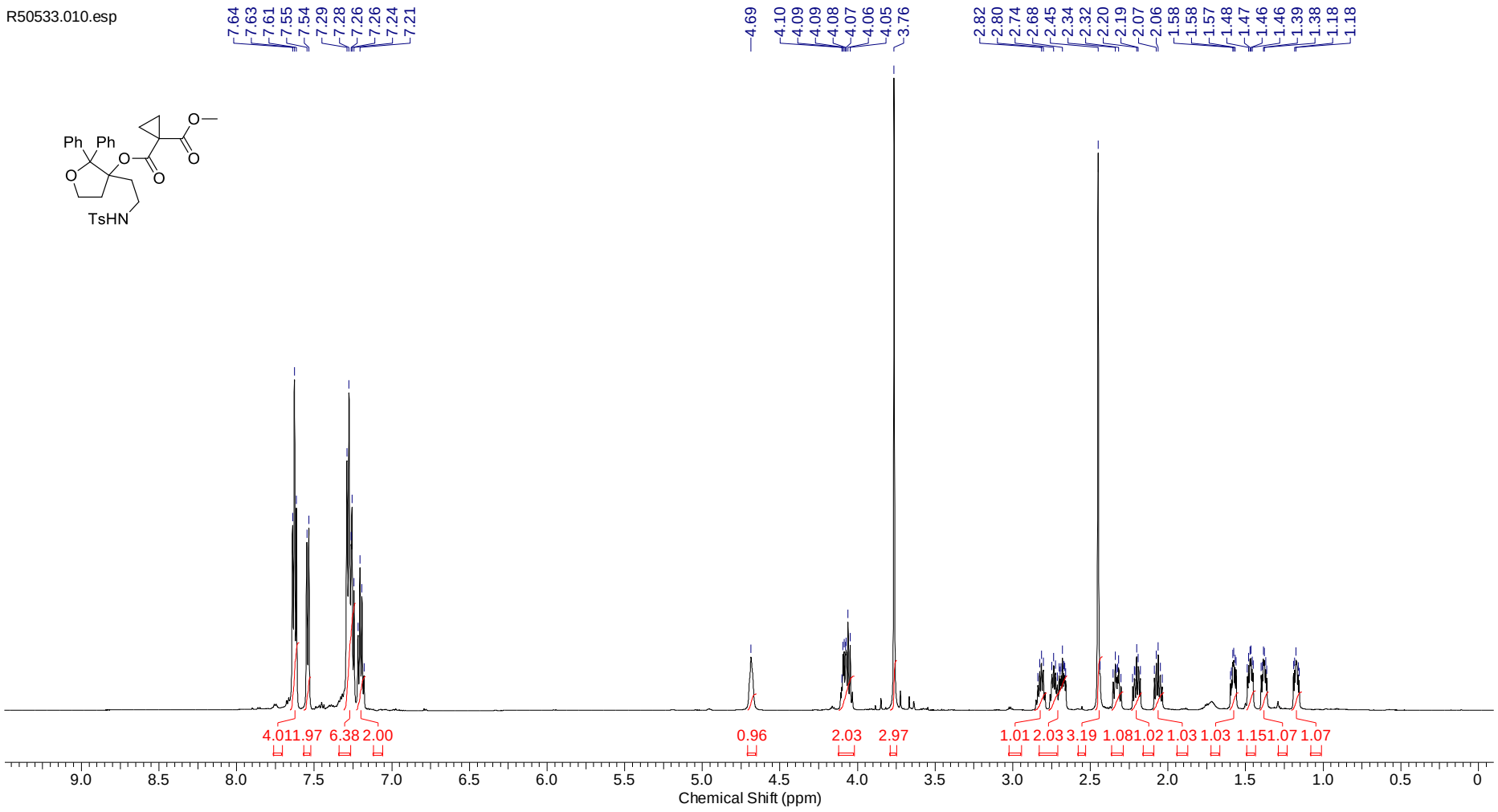
_N_-(3-(Diphenylmethylene)-5-hydroxypentyl)-4-methylbenzenesulfonamide 28, ^{1}H NMR 600 MHz $CDCl_3$



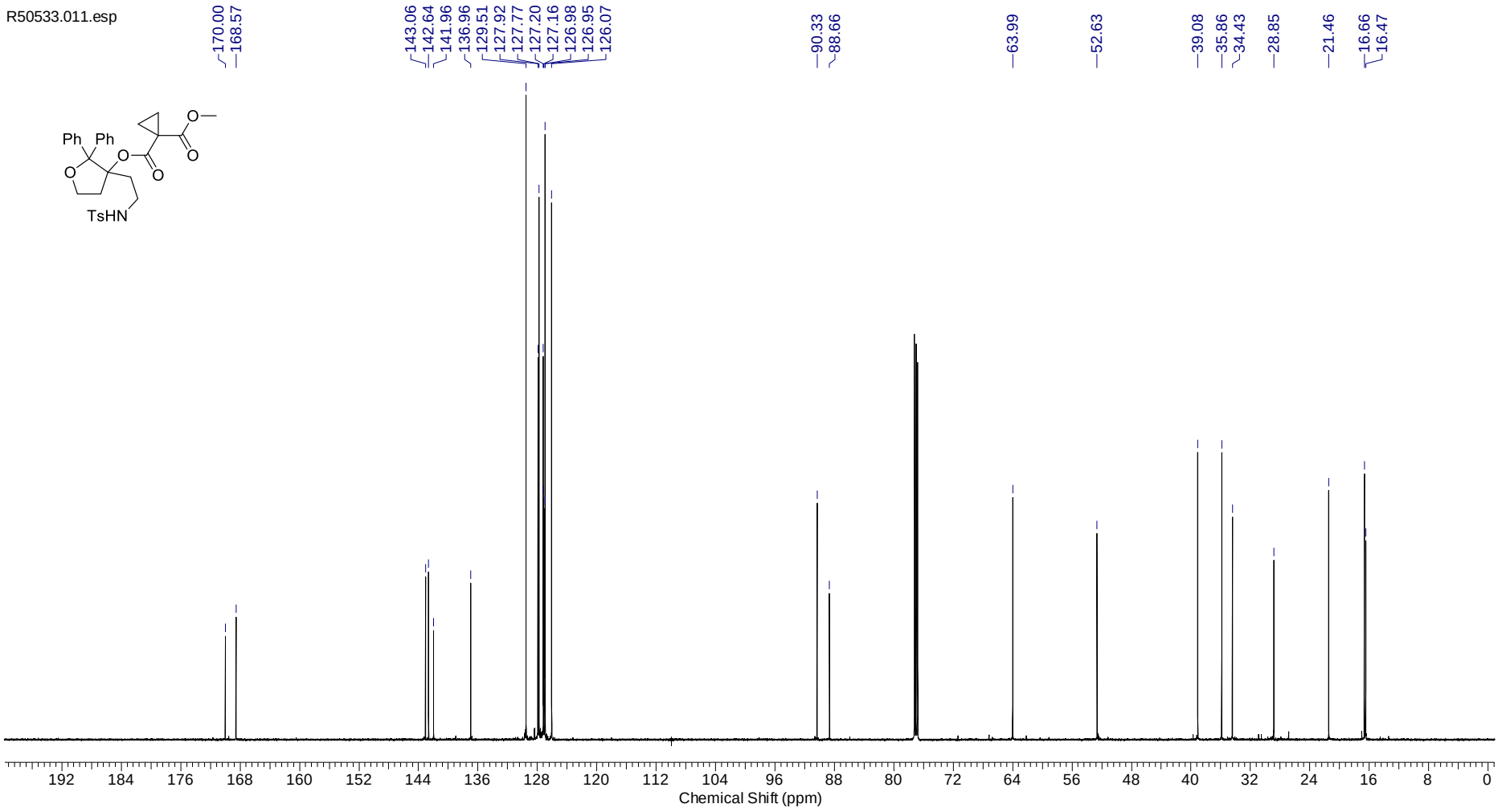
_N_-(3-(Diphenylmethylene)-5-hydroxypentyl)-4-methylbenzenesulfonamide 28, ^{13}C NMR 157 MHz $CDCl_3$



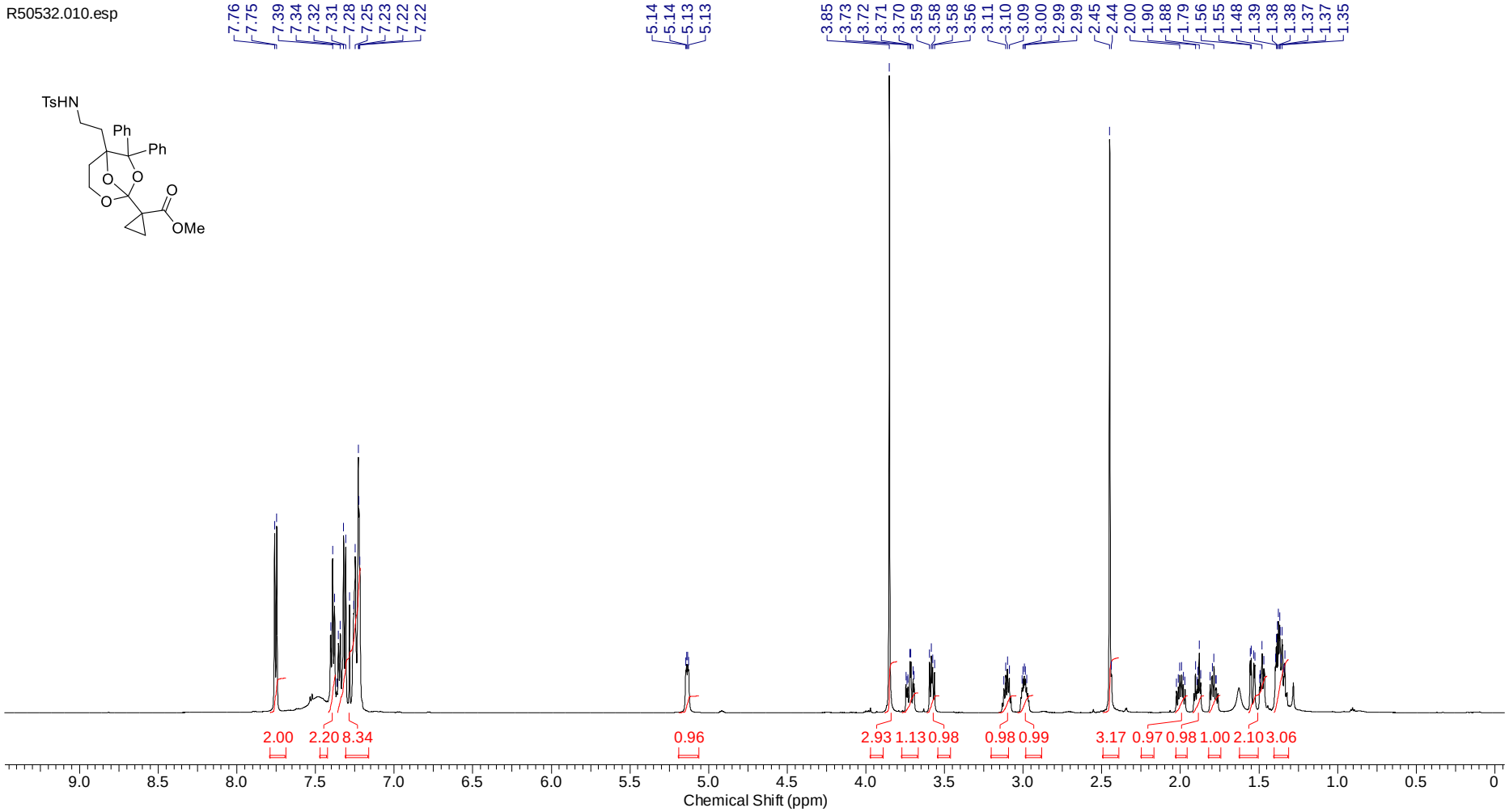
1-Methyl 1-(3-(2-((4-methylphenyl)sulfonamido)ethyl)-2,2-diphenyltetrahydrofuran-3-yl) cyclopropane-1,1-dicarboxylate 29, ^{1}H NMR 600 MHz $CDCl_3$



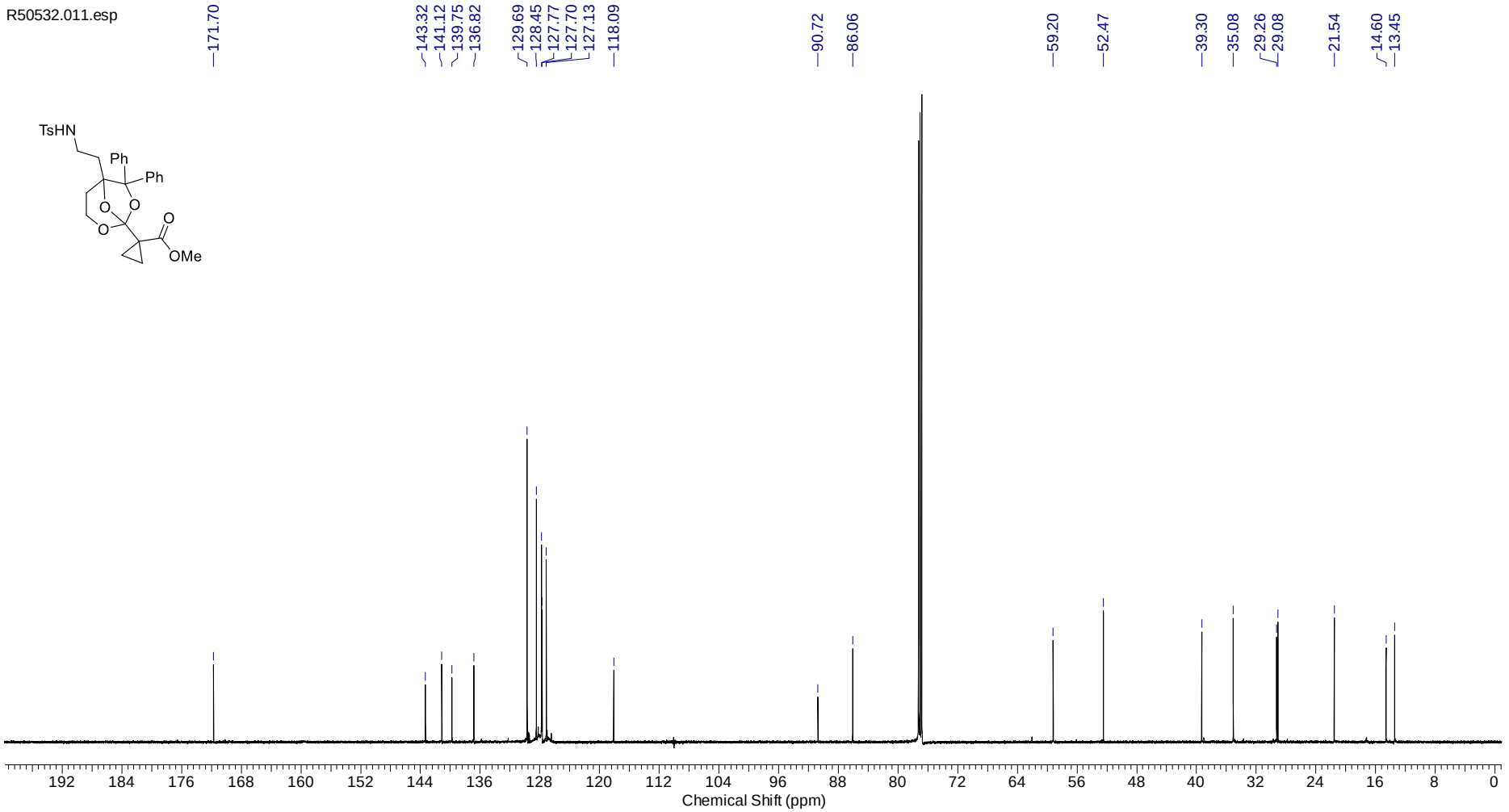
1-Methyl 1-(3-(2-((4-methylphenyl)sulfonamido)ethyl)-2,2-diphenyltetrahydrofuran-3-yl) cyclopropane-1,1-dicarboxylate 29, ^{13}C NMR 157 MHz $CDCl_3$



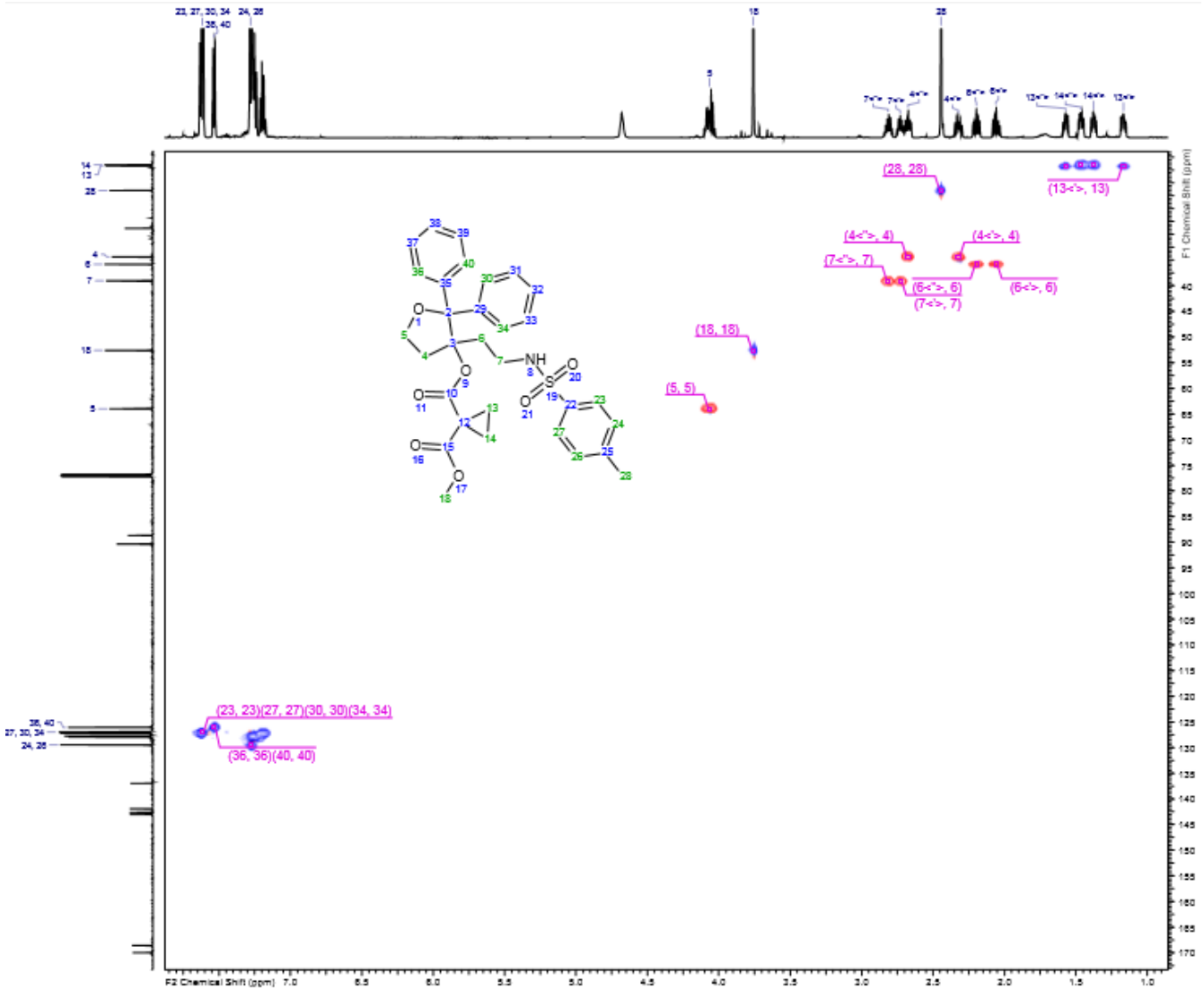
Methyl 1-(5-(2-((4-methylphenyl)sulphonamide)ethyl)-6,6-diphenyl-2,7,8-trioxabicyclo[3.2.1]octan-1-yl)cyclopropane-1-carboxylate 31, ^{1}H NMR 600 MHz $CDCl_3$



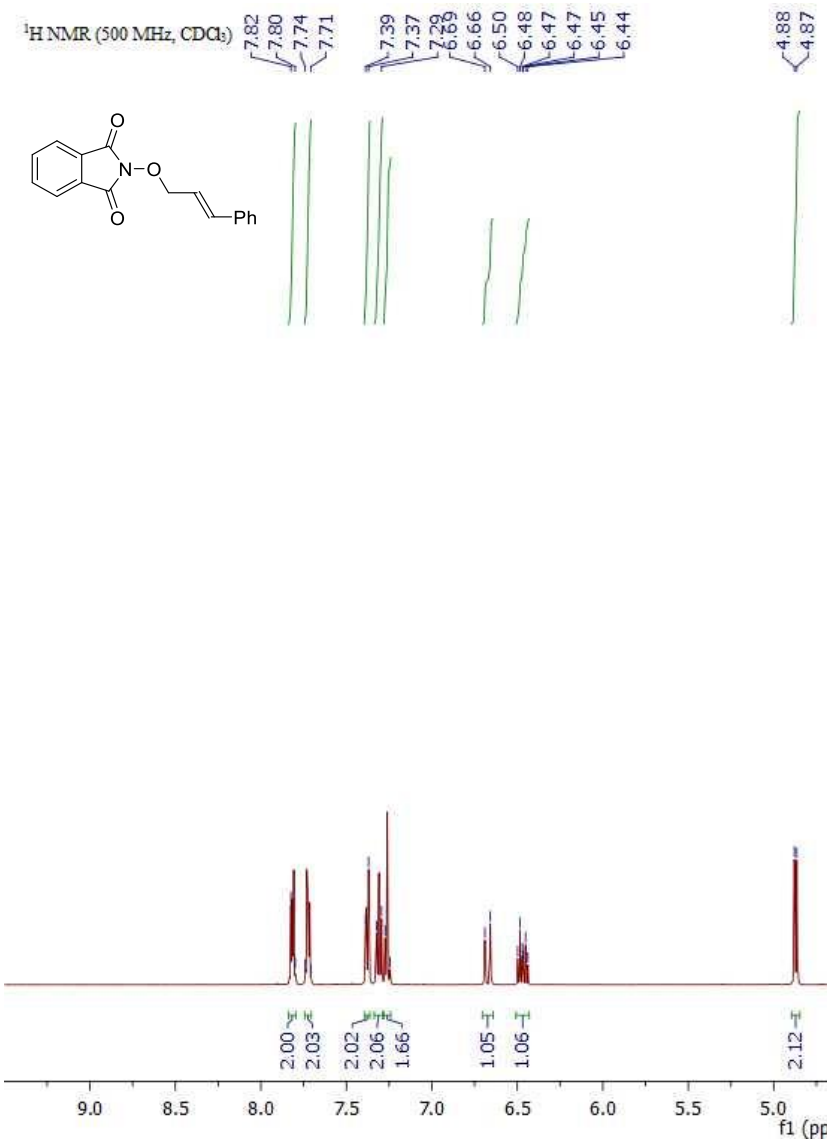
Methyl 1-(5-(2-((4-methylphenyl)sulphonamide)ethyl)-6,6-diphenyl-2,7,8-trioxabicyclo[3.2.1]octan-1-yl)cyclopropane-1-carboxylate 31, ^{13}C NMR 157 MHz $CDCl_3$



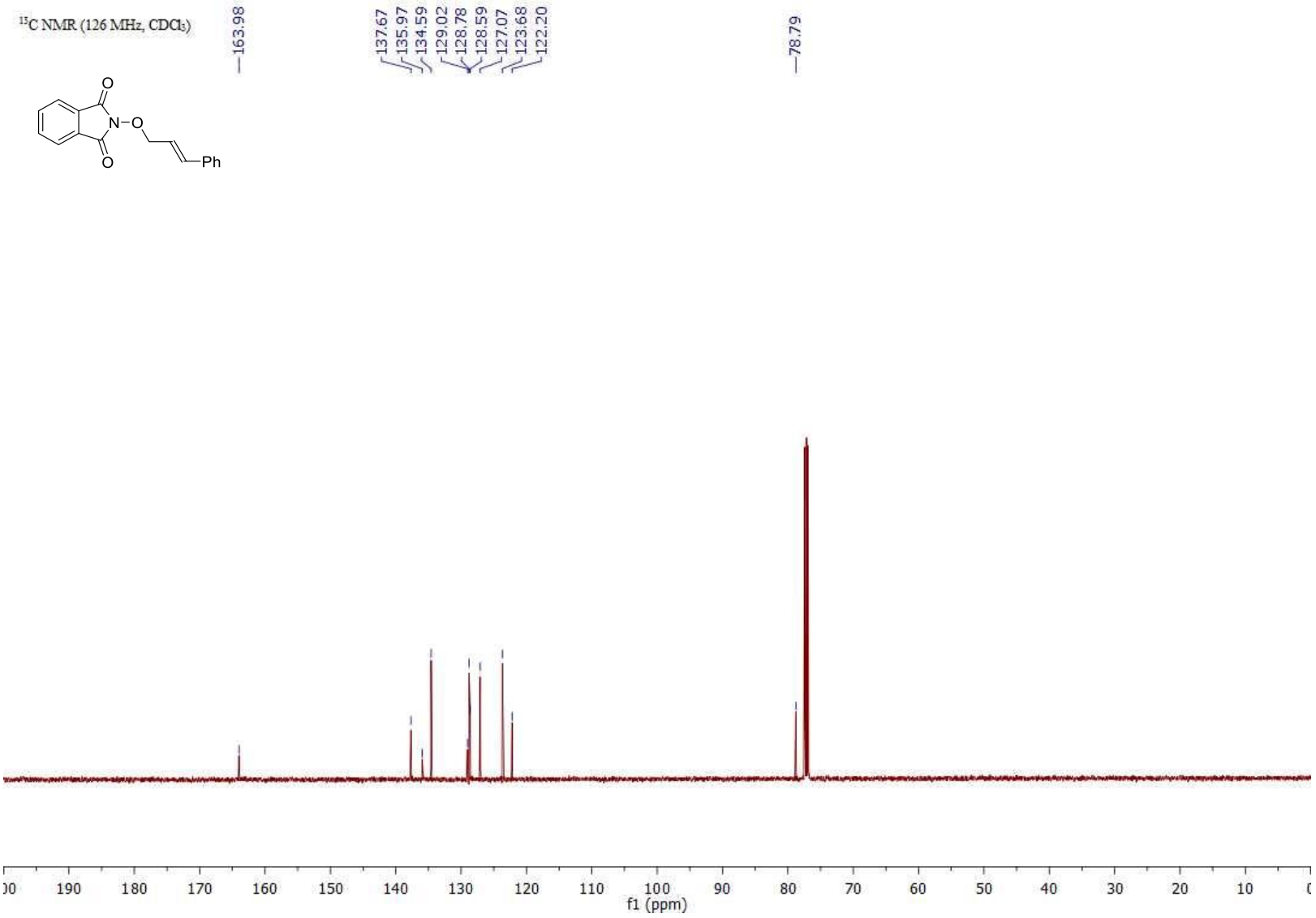
Data confirmation of furan compound 29. HSQC



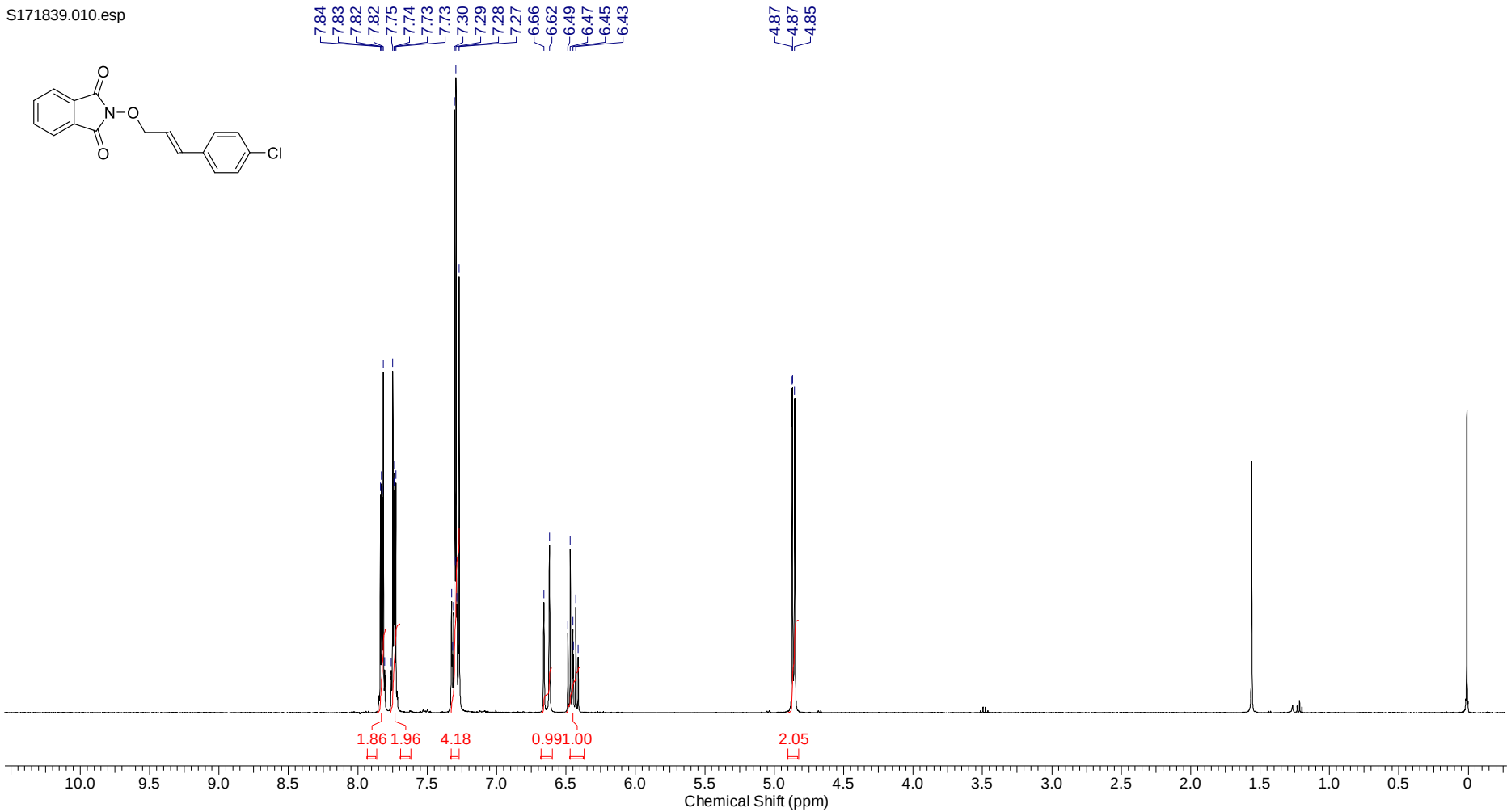
2-(Cinnamyloxy)isoindoline-1,3-dione, ^{1}H NMR 500 MHz $CDCl_3$



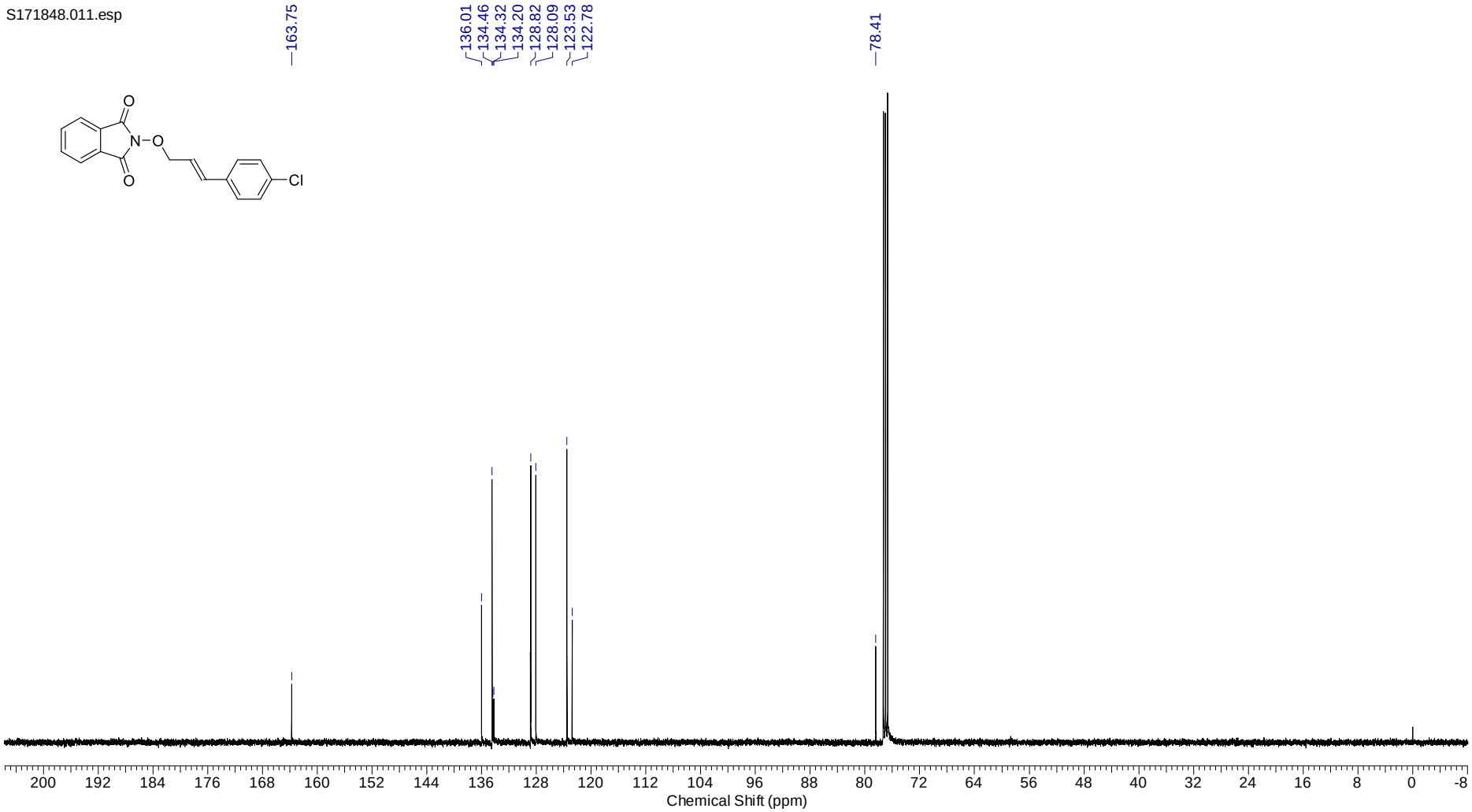
2-(Cinnamyloxy)isoindoline-1,3-dione, ^{13}C NMR 126 MHz $CDCl_3$



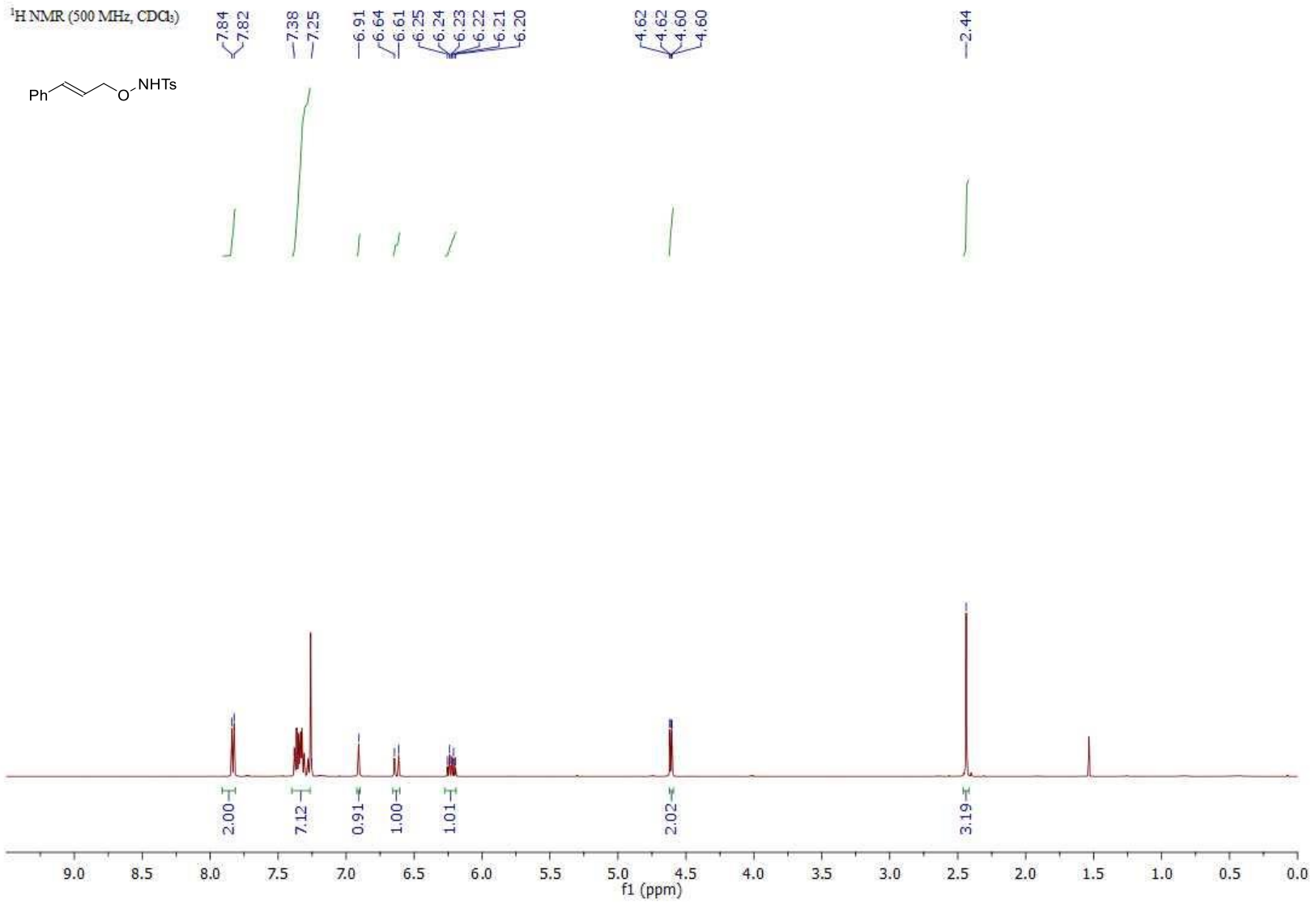
(*E*)-2-((3-(4-Chlorophenyl)allyl)oxy)isoindoline-1,3-dione, ^{1}H NMR 400 MHz $CDCl_3$



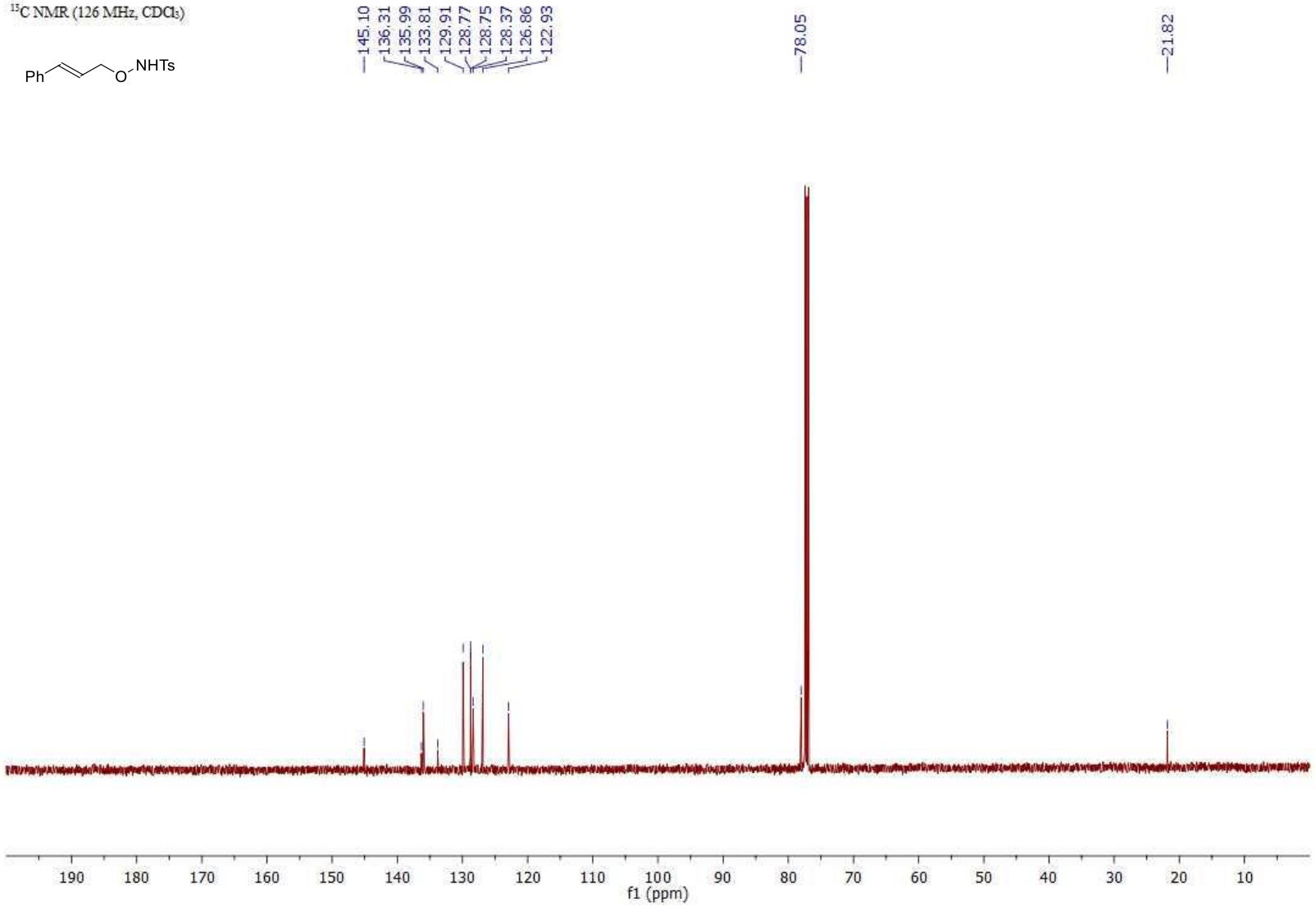
(*E*)-2-((3-(4-Chlorophenyl)allyl)oxy)isoindoline-1,3-dione, ^{13}C NMR 101 MHz $CDCl_3$



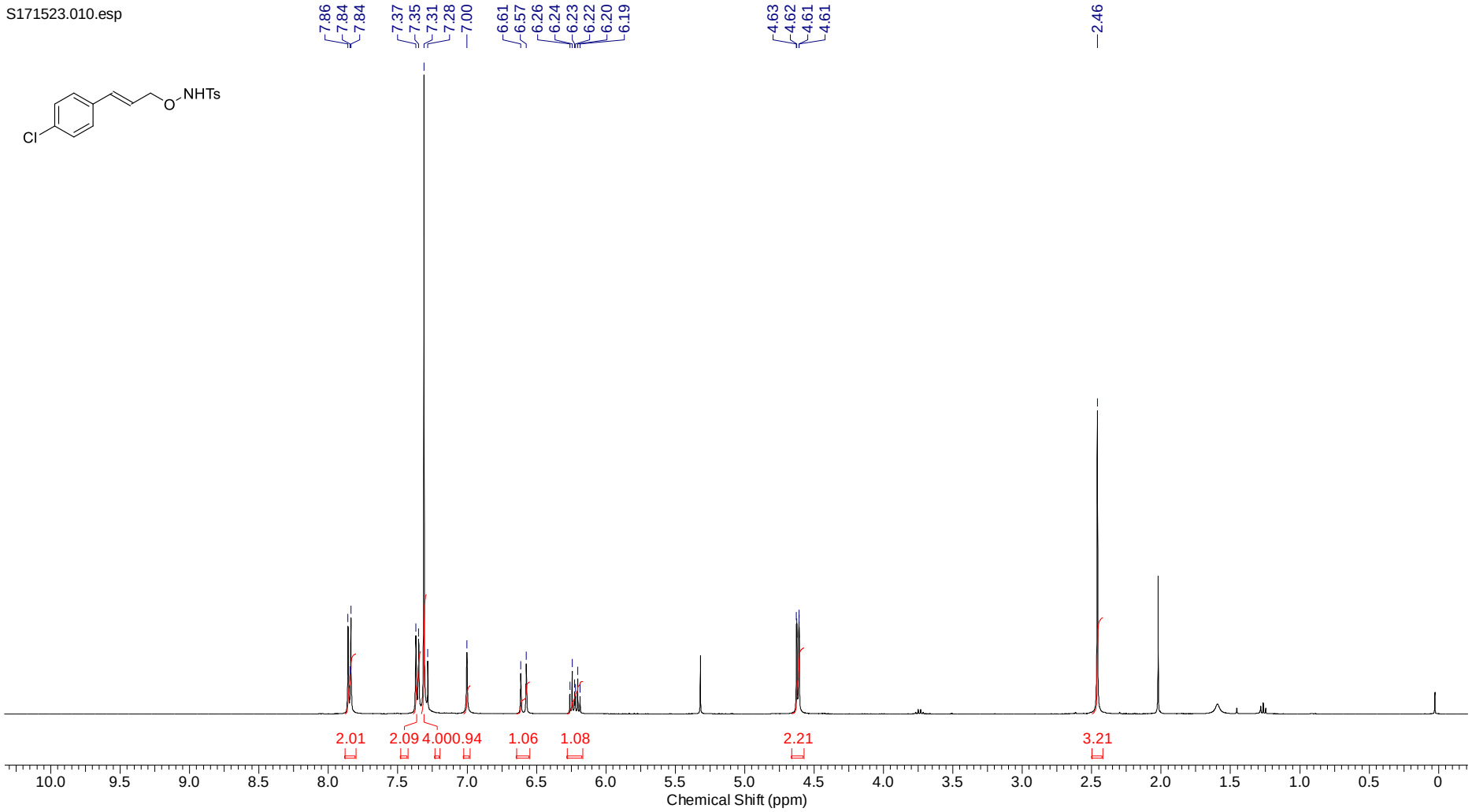
***N*-(Cinnamyloxy)-4-methylbenzenesulfonamide 33, ^{1}H NMR 500 MHz $CDCl_3$**



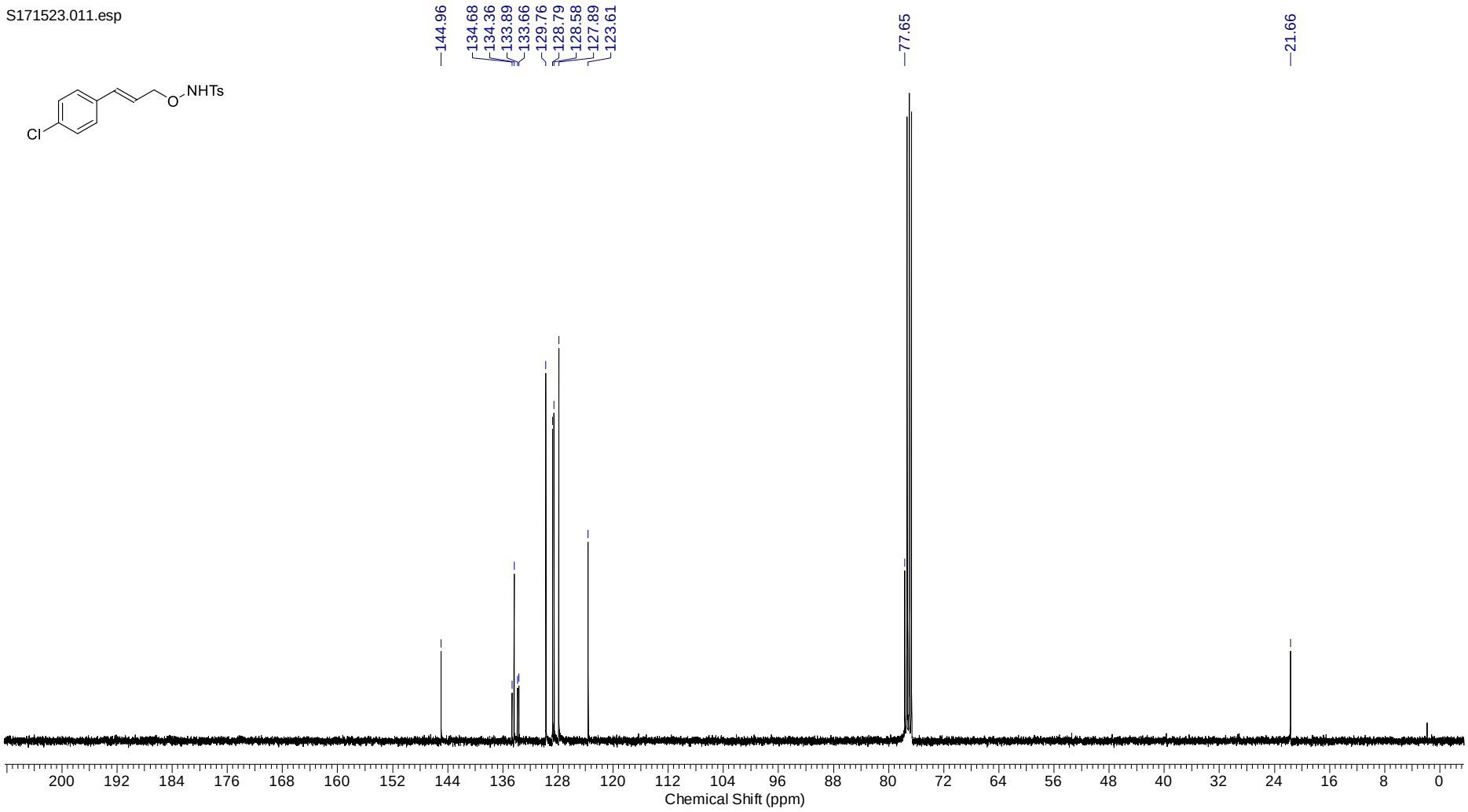
***N*-(Cinnamyloxy)-4-methylbenzenesulfonamide 33, ^{13}C NMR 126 MHz $CDCl_3$**



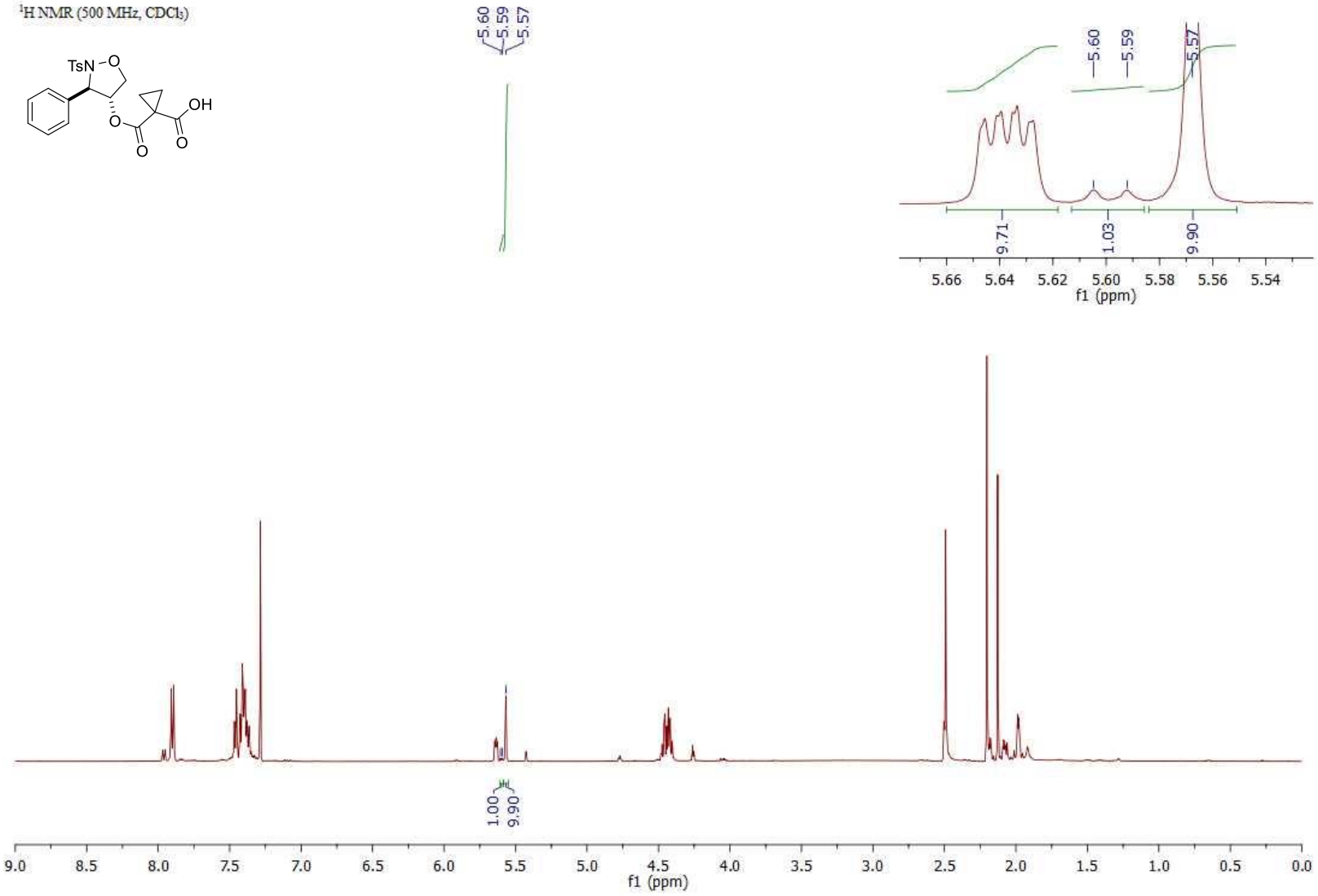
(*E*)-*N*-((3-(4-Chlorophenyl)allyl)oxy)-4-methylbenzenesulfonamide 35, ^{1}H NMR 500 MHz $CDCl_3$



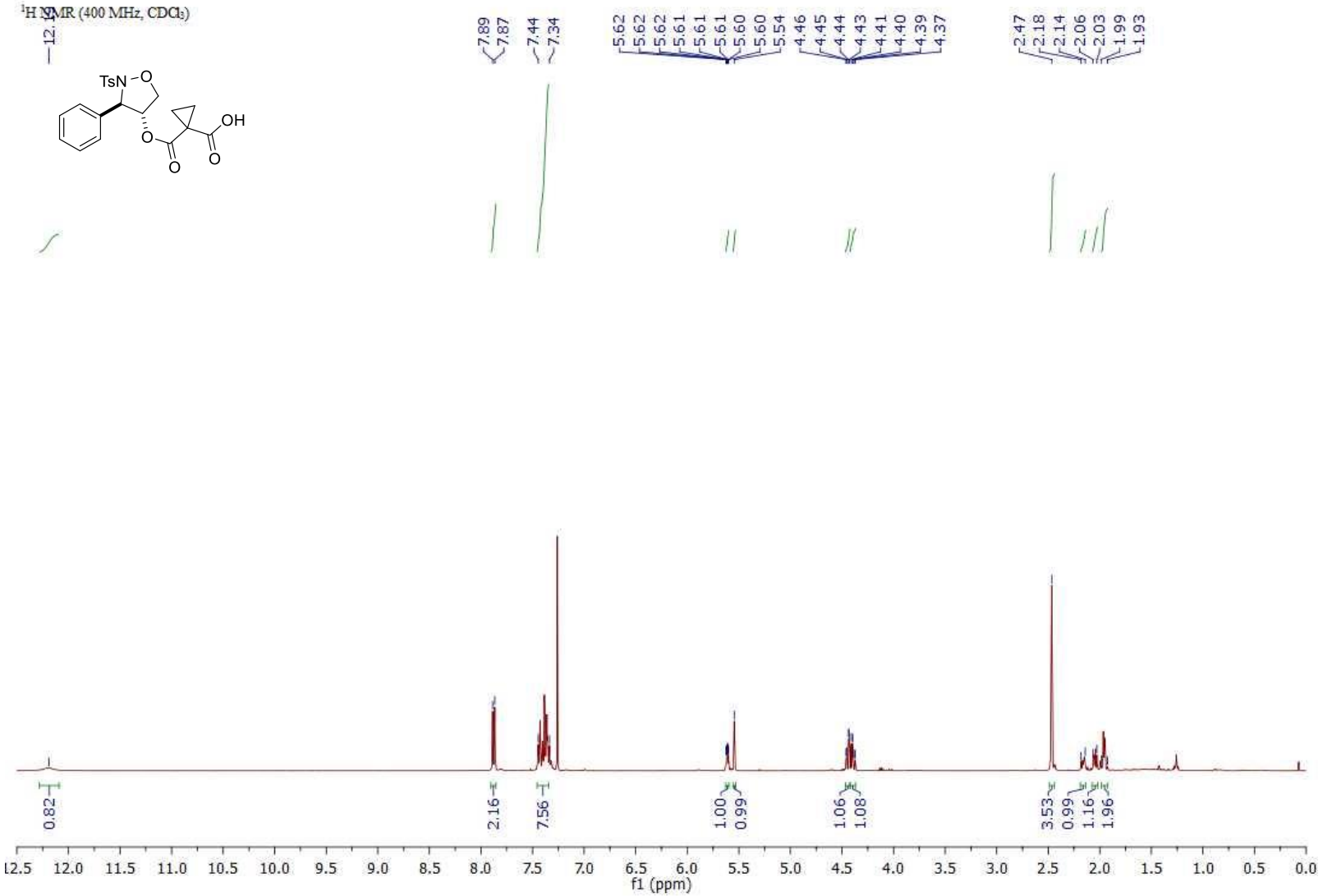
(*E*)-*N*-((3-(4-Chlorophenyl)allyl)oxy)-4-methylbenzenesulfonamide 35, ^{13}C NMR 126 MHz $CDCl_3$



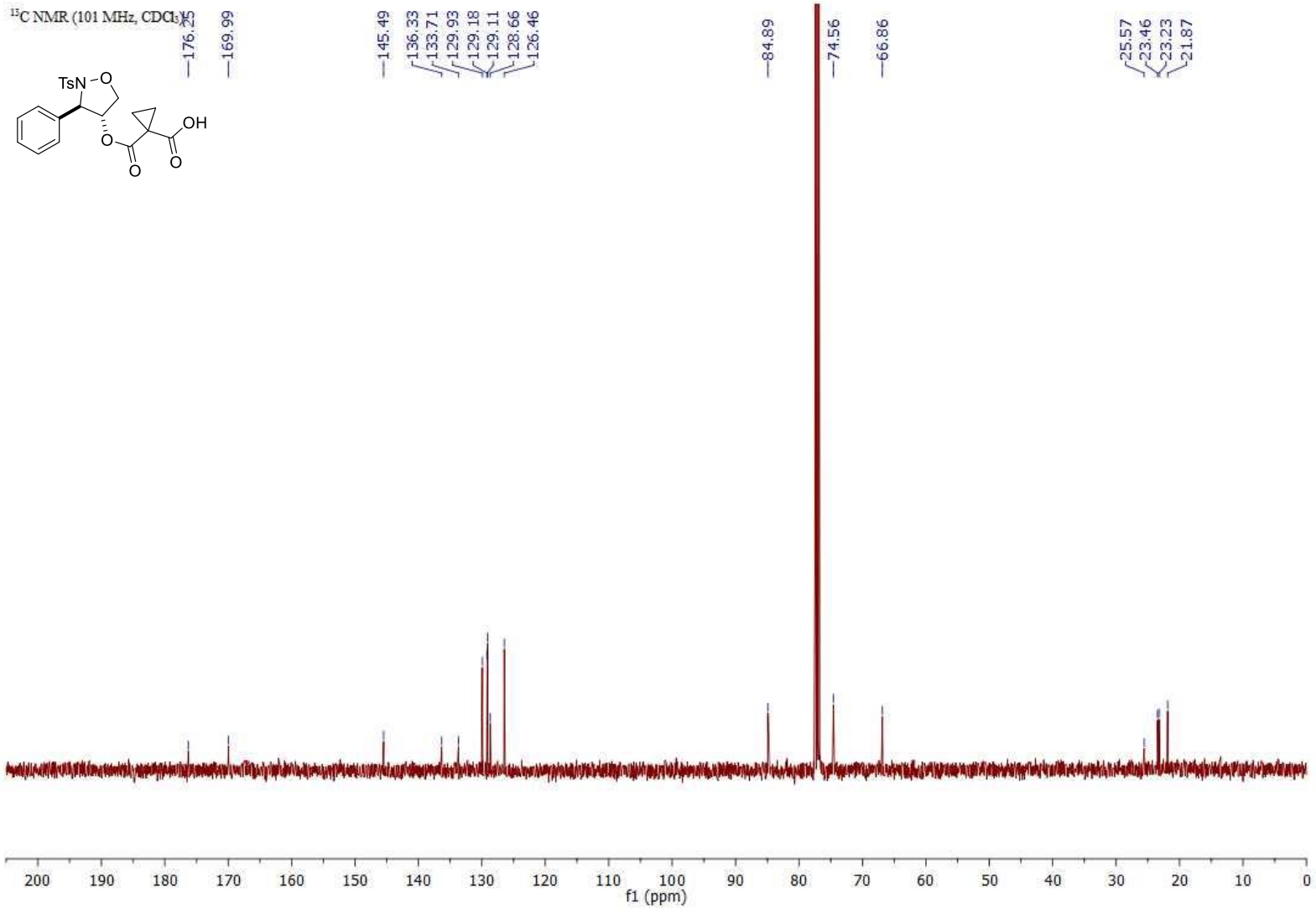
(±)-1-(((-3-Phenyl-2-tosylisoxazolidin-4-yl)oxy)carbonyl)cyclopropane-1-carboxylic acid 37, crude reaction mixture ^{1}H NMR 500 MHz $CDCl_3$



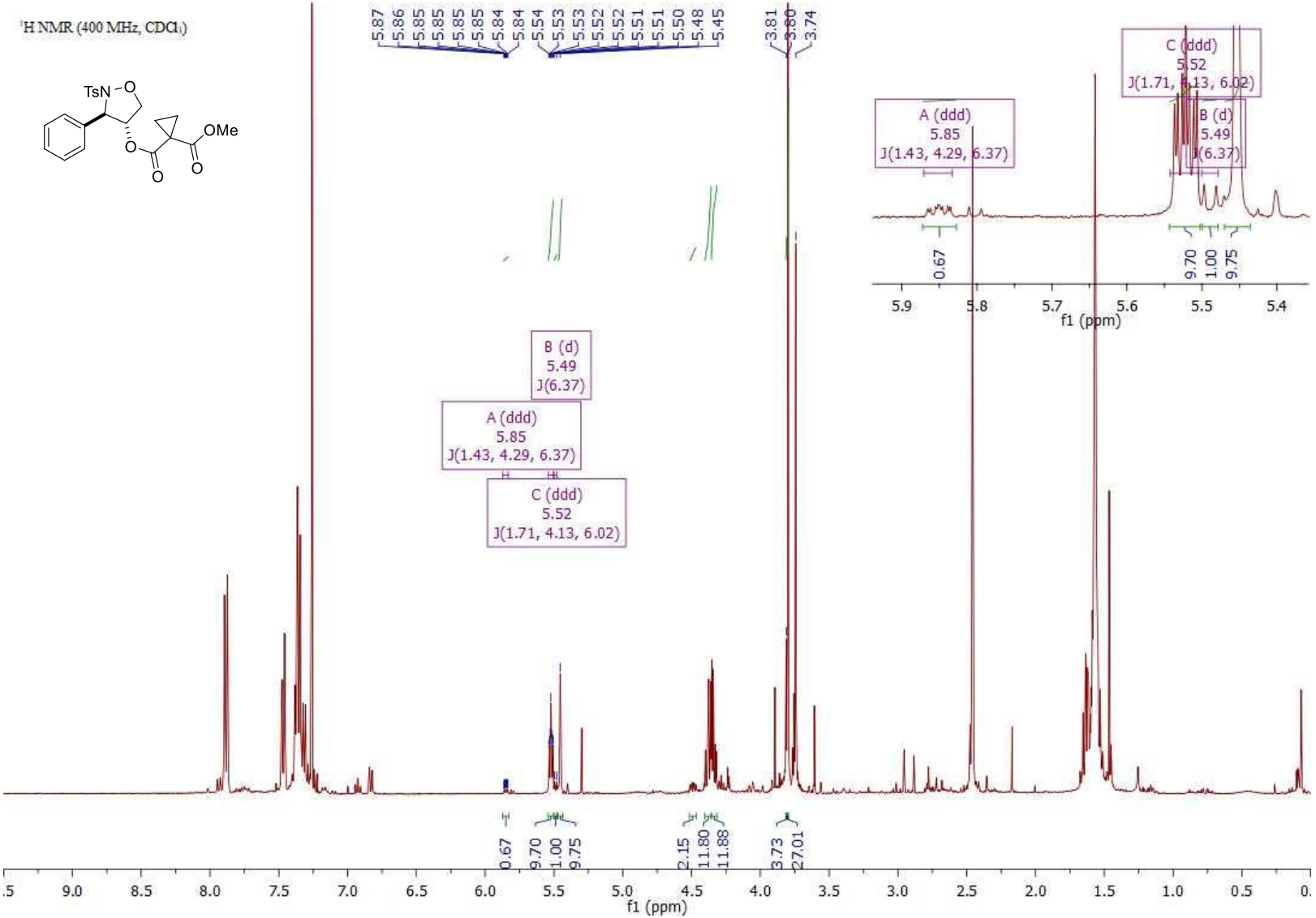
(±)-1-(((-3-Phenyl-2-tosylisoxazolidin-4-yl)oxy)carbonyl)cyclopropane-1-carboxylic acid 37, purified sample ^{1}H NMR 400 MHz $CDCl_3$



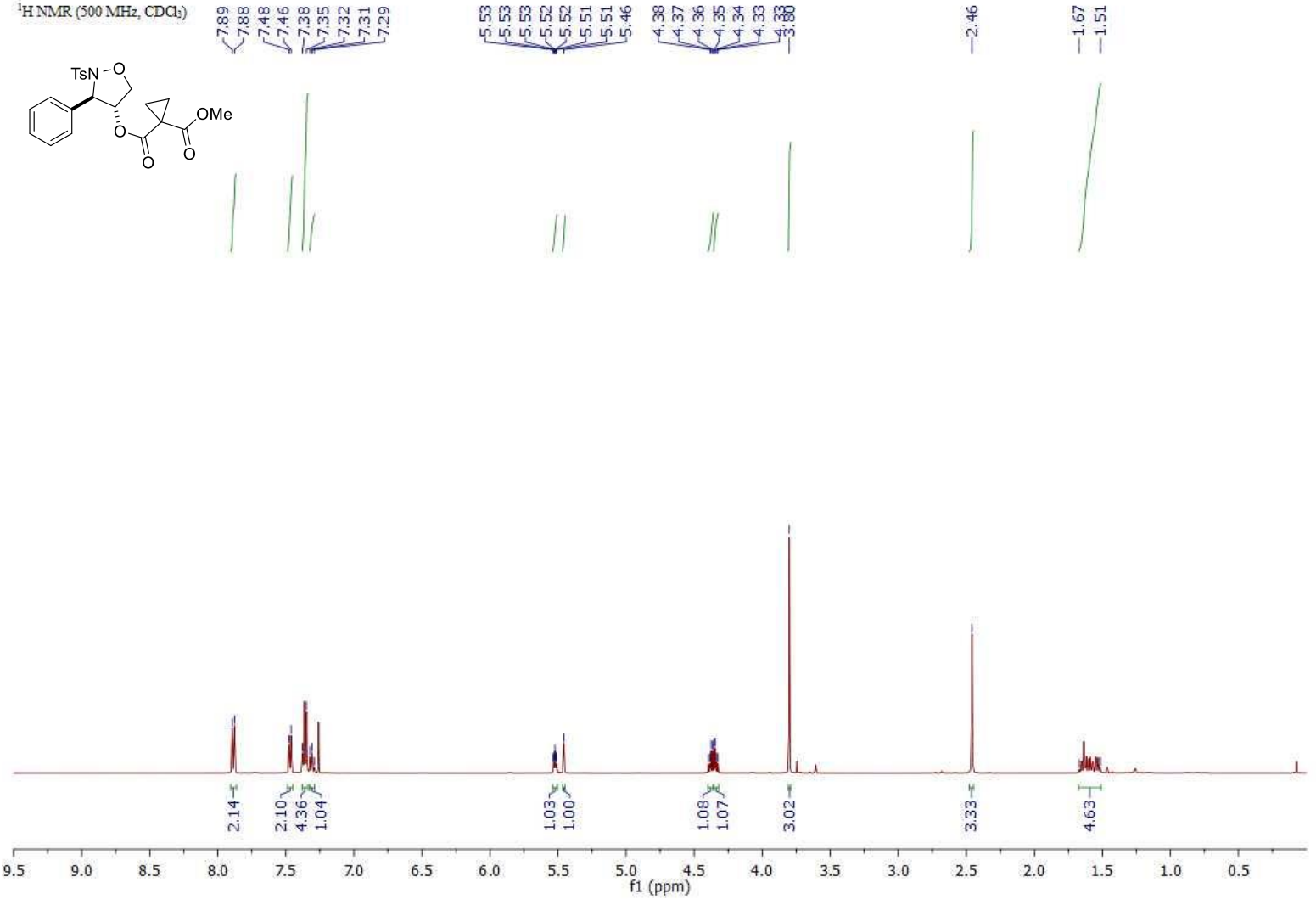
(±)-1-(((-3-Phenyl-2-tosylisoxazolidin-4-yl)oxy)carbonyl)cyclopropane-1-carboxylic acid 37, purified sample ^{13}C NMR 101 MHz $CDCl_3$



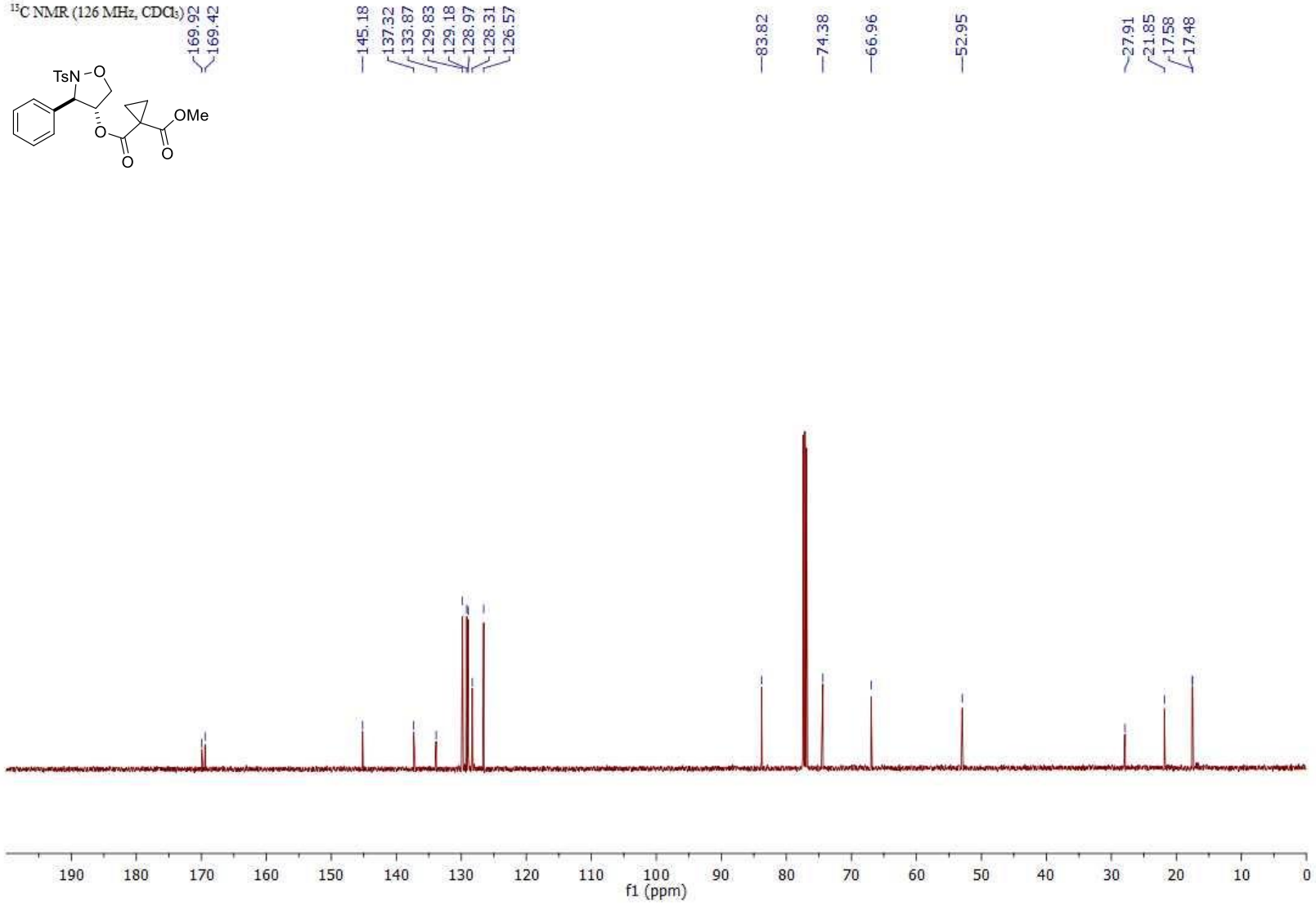
(±)-1-Methyl 1-(3-phenyl-2-tosylisoxazolidin-4-yl) cyclopropane-1,1-dicarboxylate 34, crude reaction mixture ^{1}H NMR 400 MHz $CDCl_3$



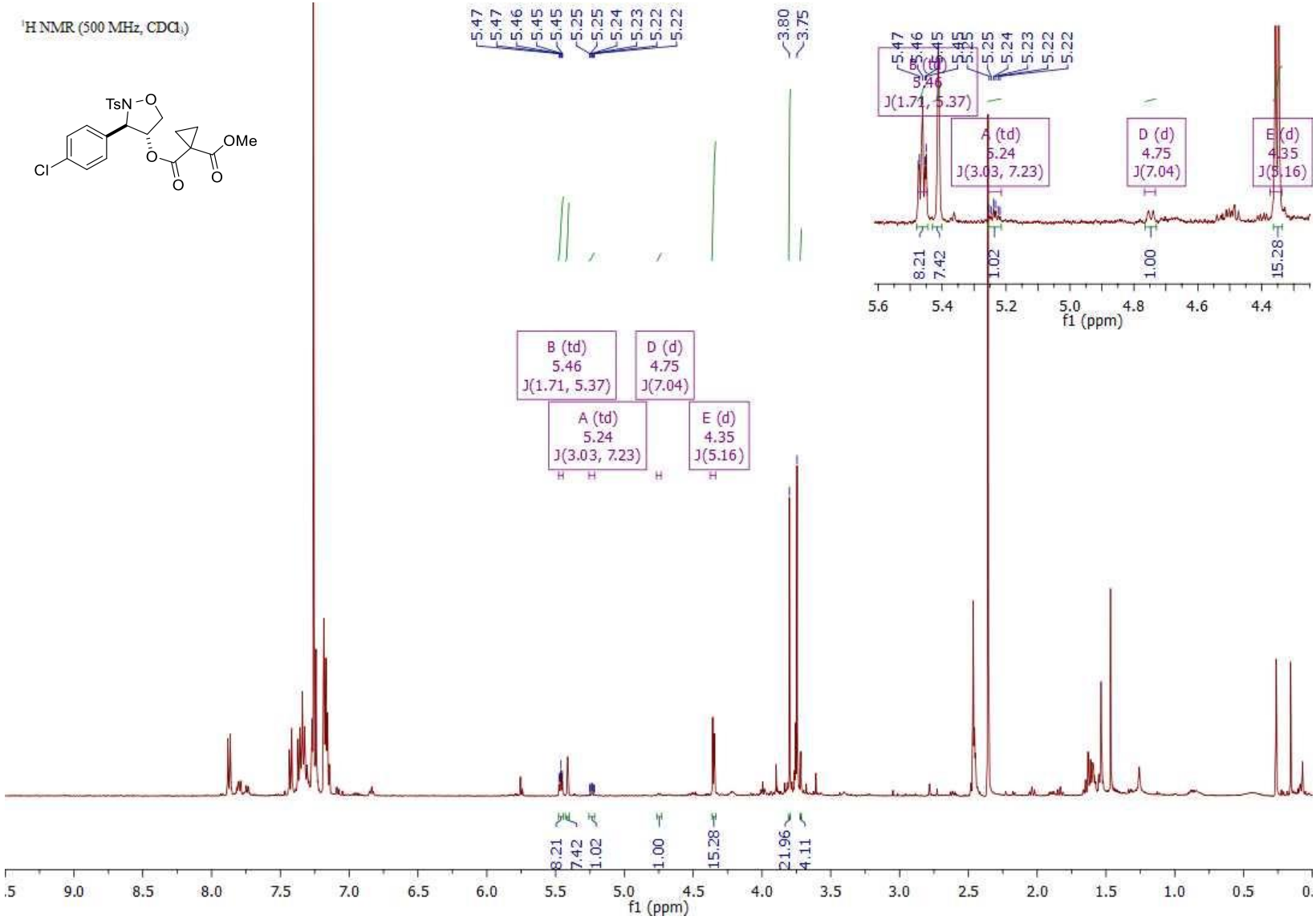
(±)-1-Methyl 1-(3-phenyl-2-tosylisoxazolidin-4-yl) cyclopropane-1,1-dicarboxylate 34, purified sample ^{1}H NMR 500 MHz $CDCl_3$



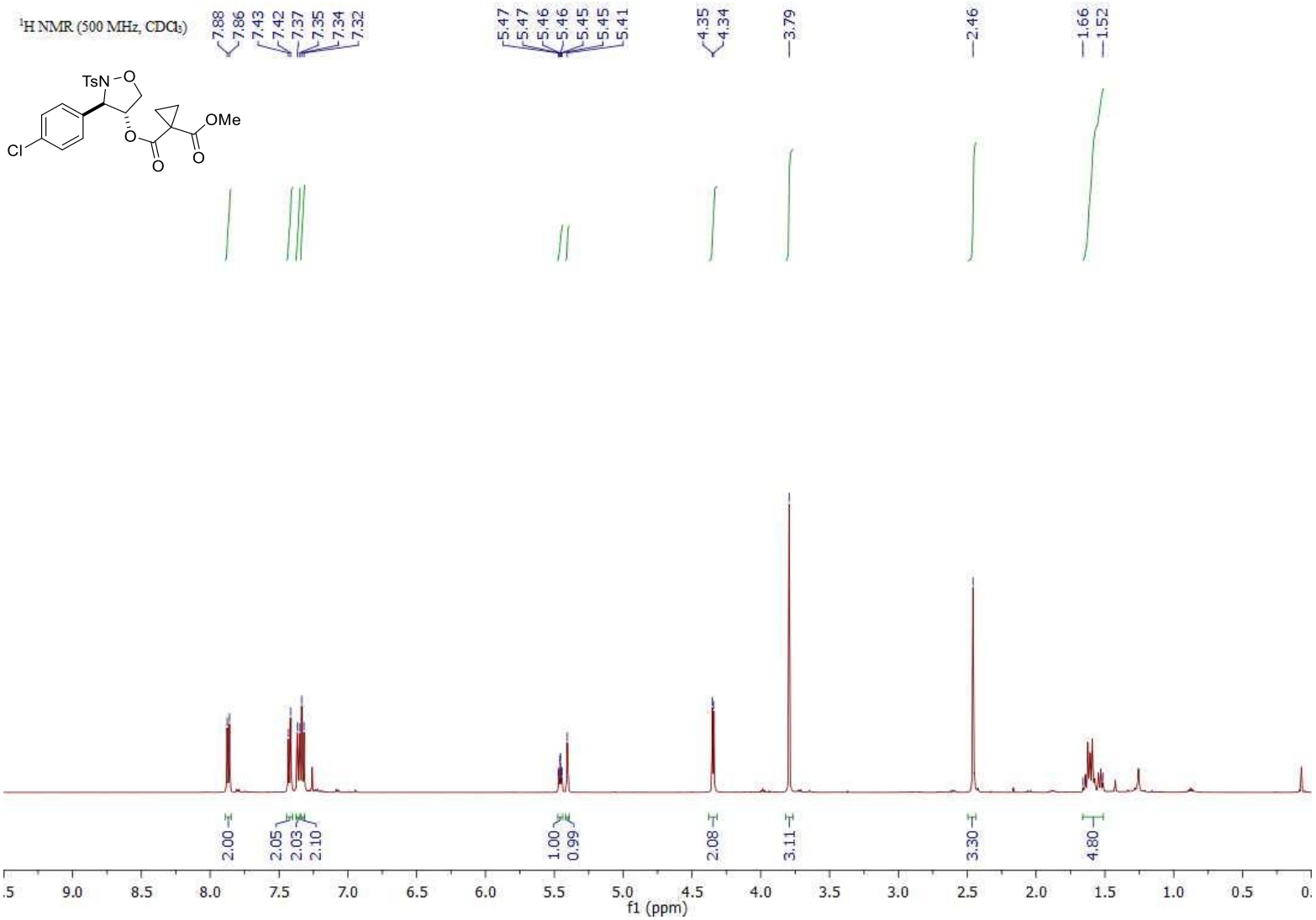
(±)-1-Methyl 1-(3-phenyl-2-tosylisoxazolidin-4-yl) cyclopropane-1,1-dicarboxylate 34, purified sample ^{13}C NMR 126 MHz $CDCl_3$



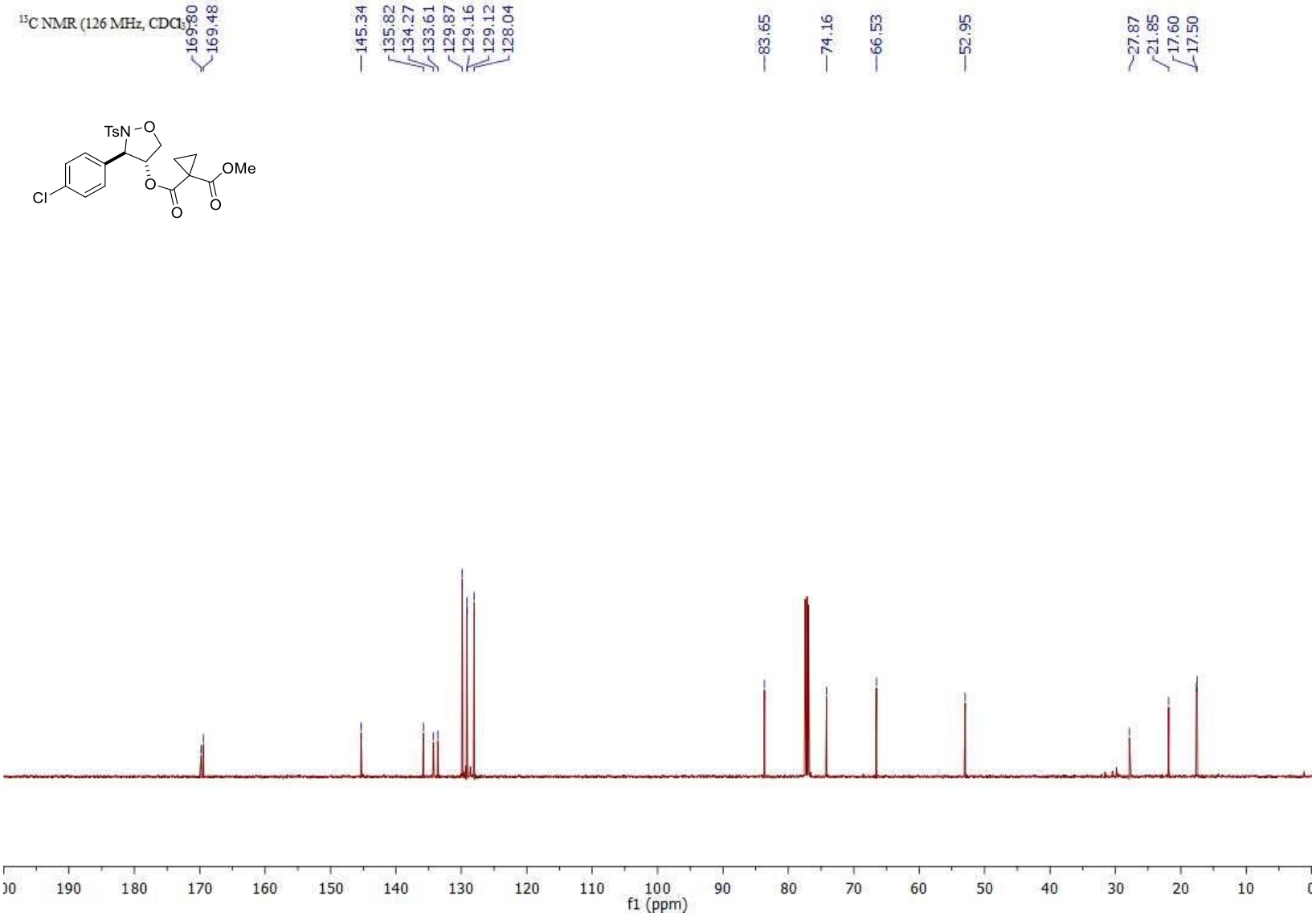
(±)-1-(3-(4-Chlorophenyl)-2-tosylisoxazolidin-4-yl) 1-methylcyclopropane-1,1-dicarboxylate 36, crude reaction mixture ^{1}H NMR 500 MHz $CDCl_3$



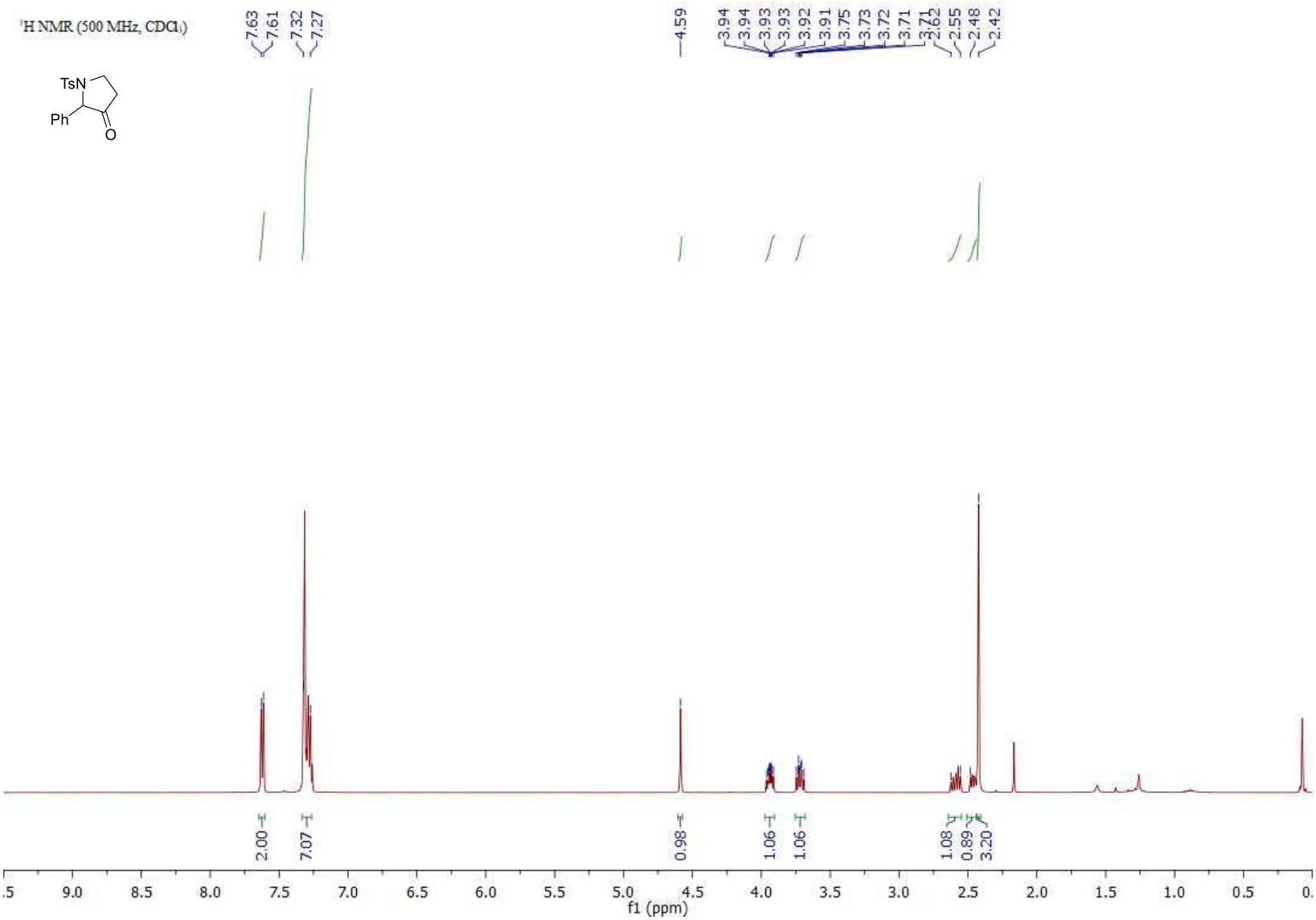
(±)-1-(3-(4-Chlorophenyl)-2-tosylisoxazolidin-4-yl) 1-methylcyclopropane-1,1-dicarboxylate 36, purified sample ^{1}H NMR 500 MHz $CDCl_3$



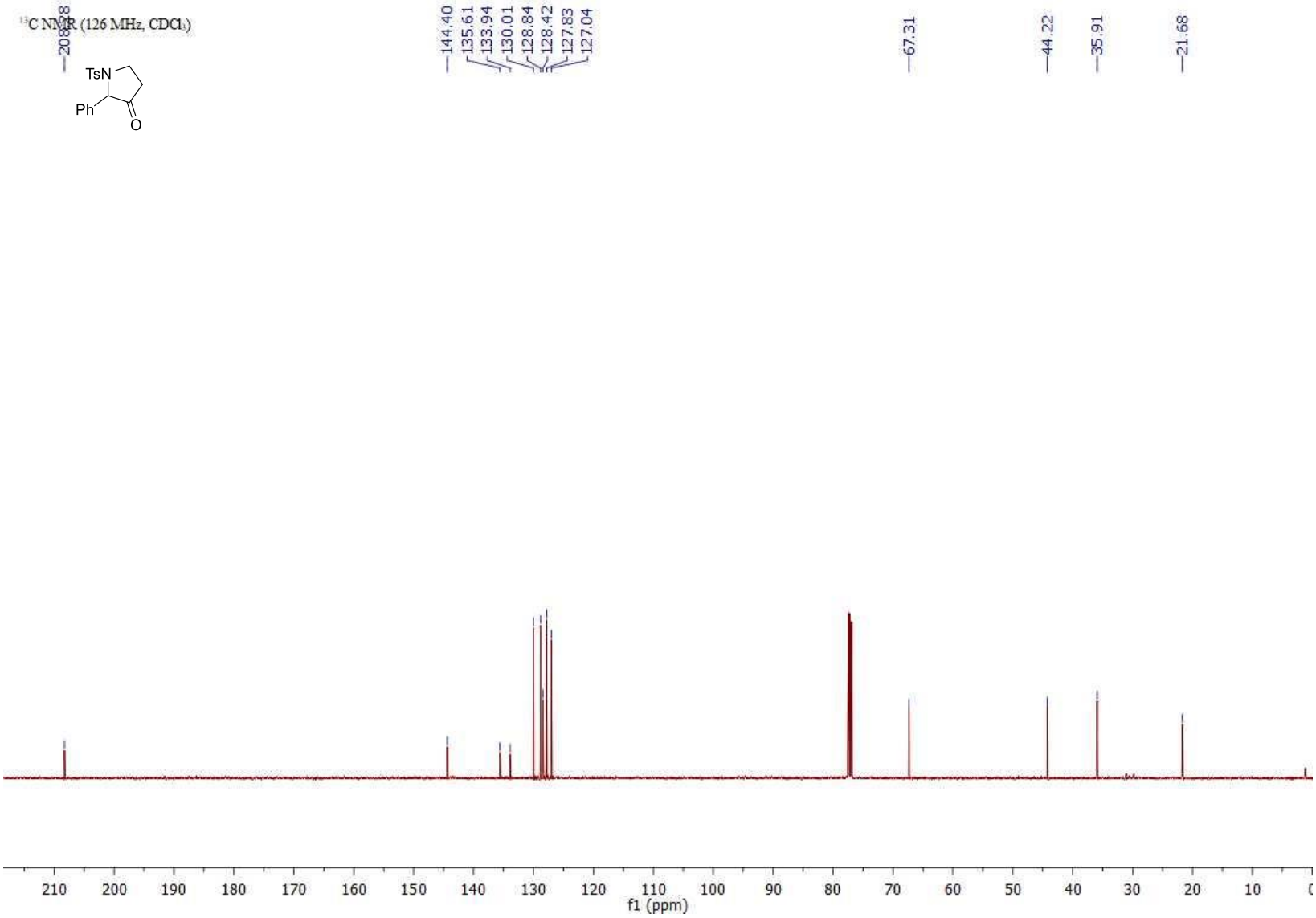
(±)-1-(3-(4-Chlorophenyl)-2-tosylisoxazolidin-4-yl) 1-methylcyclopropane-1,1-dicarboxylate 36, purified sample ^{13}C NMR 126 MHz $CDCl_3$



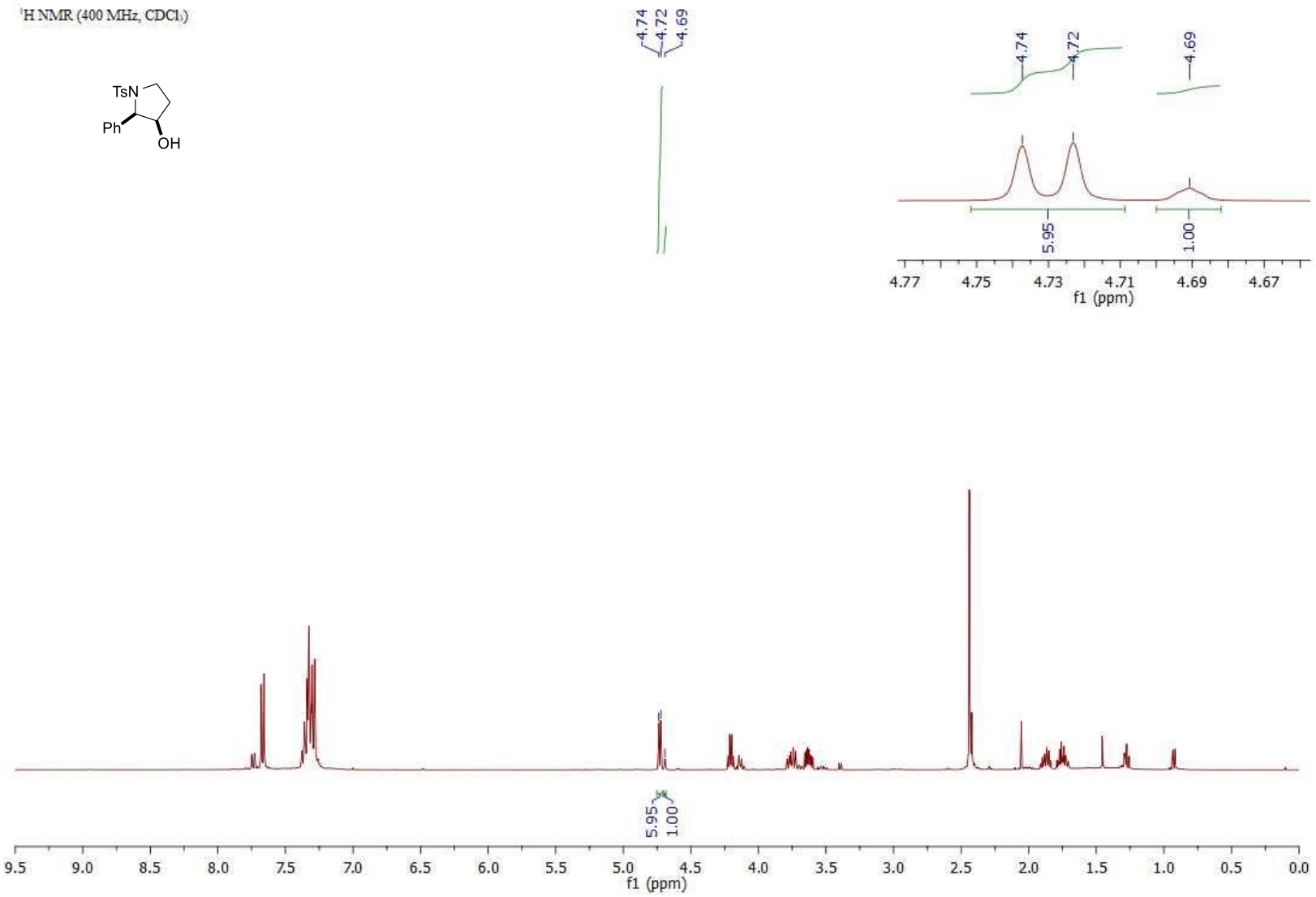
(±)-2-Phenyl-1-tosylpyrrolidin-3-one 38, ^{1}H NMR 500 MHz $CDCl_3$



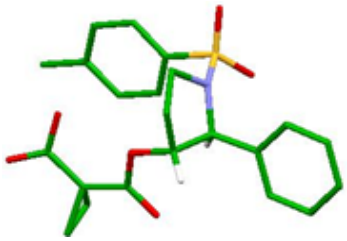
(±)-2-Phenyl-1-tosylpyrrolidin-3-one 38, ^{13}C NMR 126 MHz $CDCl_3$



(±)-2-Phenyl-1-tosylpyrrolidin-3-ol 22, Dibal-H reduction crude reaction mixture ^{1}H NMR 500 MHz $CDCl_3$

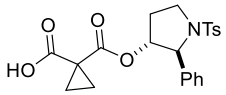


X-Ray data for (±)-1-(((-3-Phenyl-2-tosylisoxazolidin-4-yl)oxy)carbonyl)cyclopropane-1-carboxylic acid 10



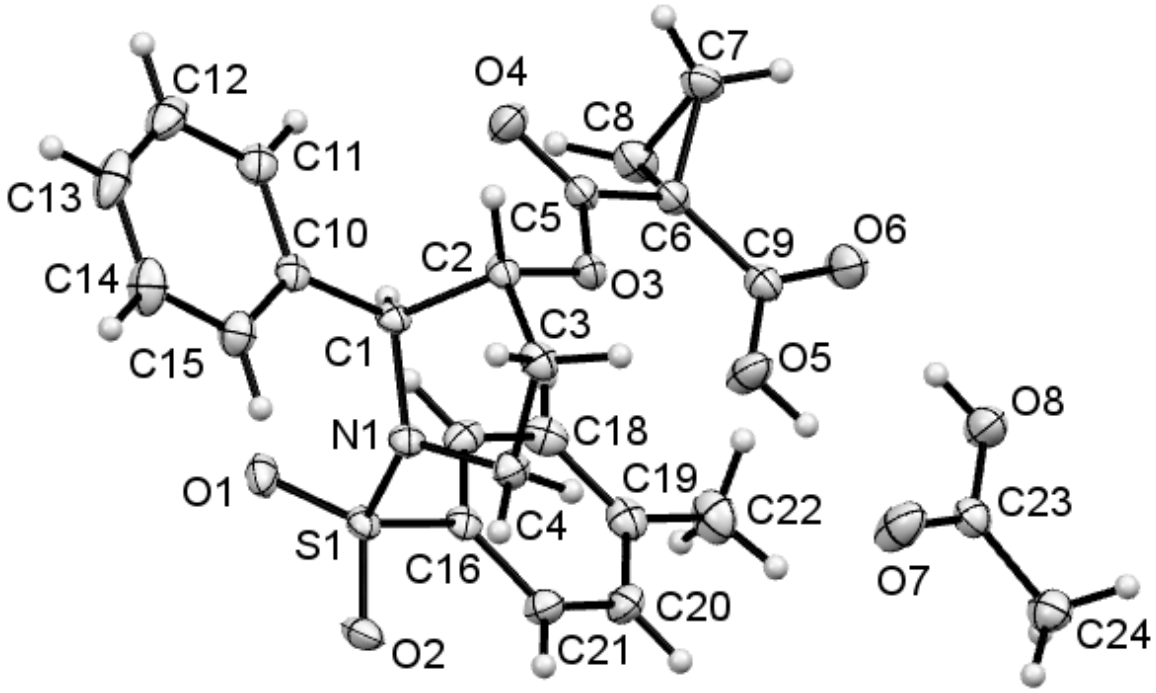
CCDC 1569729

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

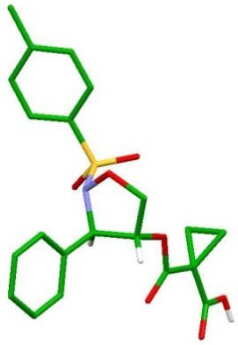


Identification code	tom_stuart_needles	
Empirical formula	C24 H27 N O8 S	
Formula weight	489.52	
Temperature	123(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 2_1/n	
Unit cell dimensions	a = 14.2564(10) Å	α= 90°.
	b = 7.5171(5) Å	β= 92.211(7)°.
	c = 21.4885(16) Å	γ = 90°.
Volume	2301.1(3) $Å^3$	
Z	4	
Density (calculated)	1.413 Mg/m^3	
Absorption coefficient	0.192 mm^{-1}	
F(000)	1032	
Crystal size	0.33 x 0.16 x 0.05 mm^3	
Theta range for data collection	3.234 to 27.995°.	
Index ranges	-18<=h<=17, -9<=k<=9, -27<=l<=27	
Reflections collected	15728	
Independent reflections	5410 [R(int) = 0.0285]	
Completeness to theta = 27.000°	99.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.00000 and 0.86786	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5410 / 0 / 315	
Goodness-of-fit on F^2	1.110	
Final R indices [I>2sigma(I)]	R1 = 0.0541, wR2 = 0.1081	
R indices (all data)	R1 = 0.0691, wR2 = 0.1166	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.429 and -0.441 $e.Å^{-3}$	

Figure S1. Molecular structure of compound **(±)-1-(((-3-Phenyl-2-tosylisoxazolidin-4-yl)oxy)carbonyl)cyclopropane-1-carboxylic acid acetic acid salt 10·AcOH**. Non-H atoms have been drawn with 50 % probability ellipsoids. H atoms are drawn as small spheres of arbitrary size.



X-Ray data for (±)-1-(((-3-Phenyl-2-tosylisoxazolidin-4-yl)oxy)carbonyl)cyclopropane-1-carboxylic acid 37



CCDC 1569728

Table S2. Crystal data and structure refinement for **37**.

Identification code	ferrer_caf652	
Empirical formula	C21 H21 N O7 S	
Formula weight	431.45	
Temperature	123(2) K	
Wavelength	1.54180 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 16.6619(6) Å	α = 90°.
	b = 10.7864(3) Å	β = 101.846(3)°.
	c = 11.6368(3) Å	γ = 90°.
Volume	2046.85(11) $Å^3$	
Z	4	
Density (calculated)	1.400 Mg/m^3	
Absorption coefficient	1.793 mm^{-1}	
F(000)	904	
Crystal size	0.18 x 0.14 x 0.04 mm^3	
Theta range for data collection	4.92 to 73.18°.	
Index ranges	-20<=h<=20, -12<=k<=13, -14<=l<=14	
Reflections collected	18649	
Independent reflections	4075 [R(int) = 0.0344]	
Completeness to theta = 70.00°	99.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.00000 and 0.74640	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4075 / 0 / 276	
Goodness-of-fit on F^2	1.036	
Final R indices [I>2sigma(I)]	R1 = 0.0456, wR2 = 0.1218	
R indices (all data)	R1 = 0.0534, wR2 = 0.1296	
Largest diff. peak and hole	0.412 and -0.250 $e.Å^{-3}$	

Figure S2. Molecular structure of compound **(±)-1-(((-3-Phenyl-2-tosylisoxazolidin-4-yl)oxy)carbonyl)cyclopropane-1-carboxylic acid 37**. Non-H atoms have been drawn with 50 % probability ellipsoids. H atoms are drawn as small spheres of arbitrary size.

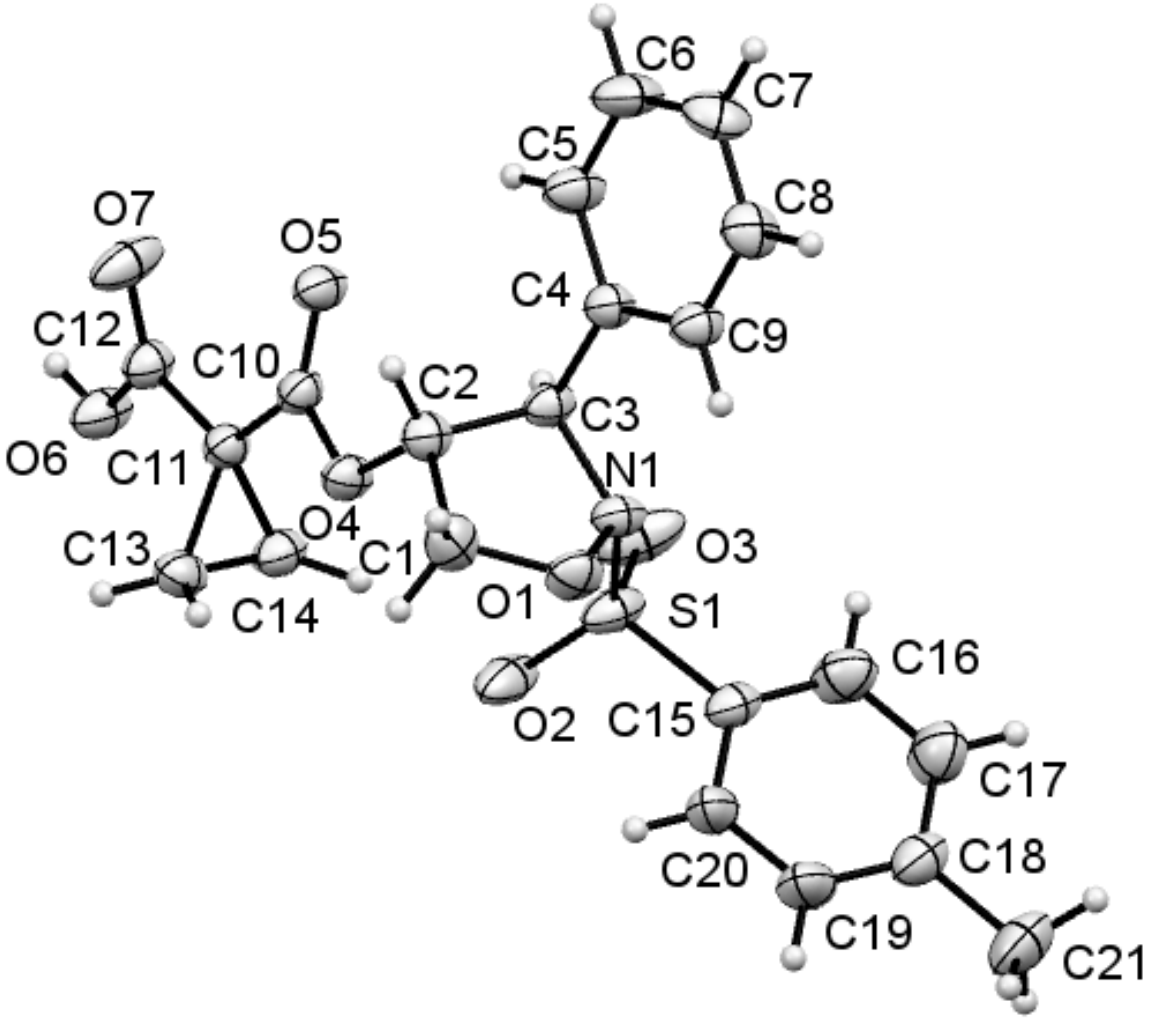
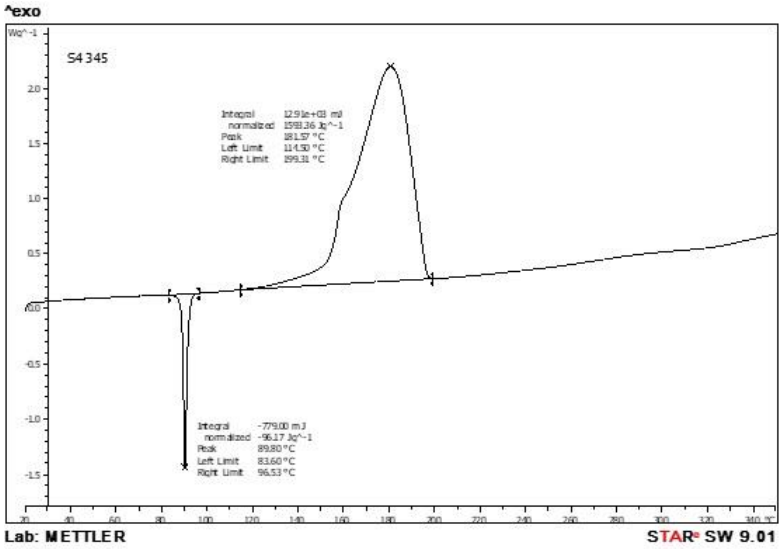


Figure S3. DSC data for malonoyl peroxide **1**.



DSC data showing melting point of malonoyl peroxide **1** at 89.8 °C and decomposition of peroxide at 181.57 °C.