Supplementary Material

Sterically controlled rhenium-catalyzed hydroxyl transposition

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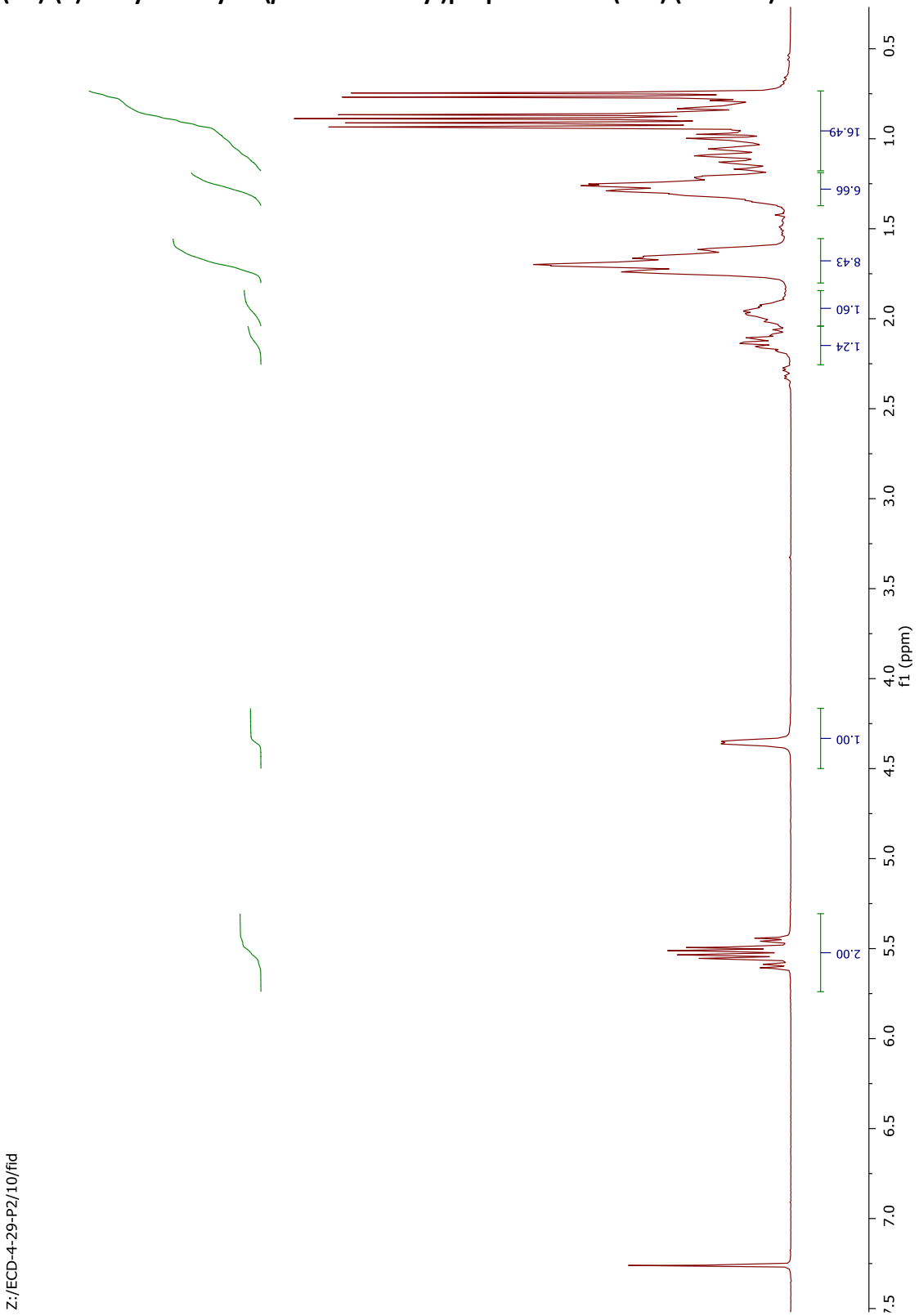
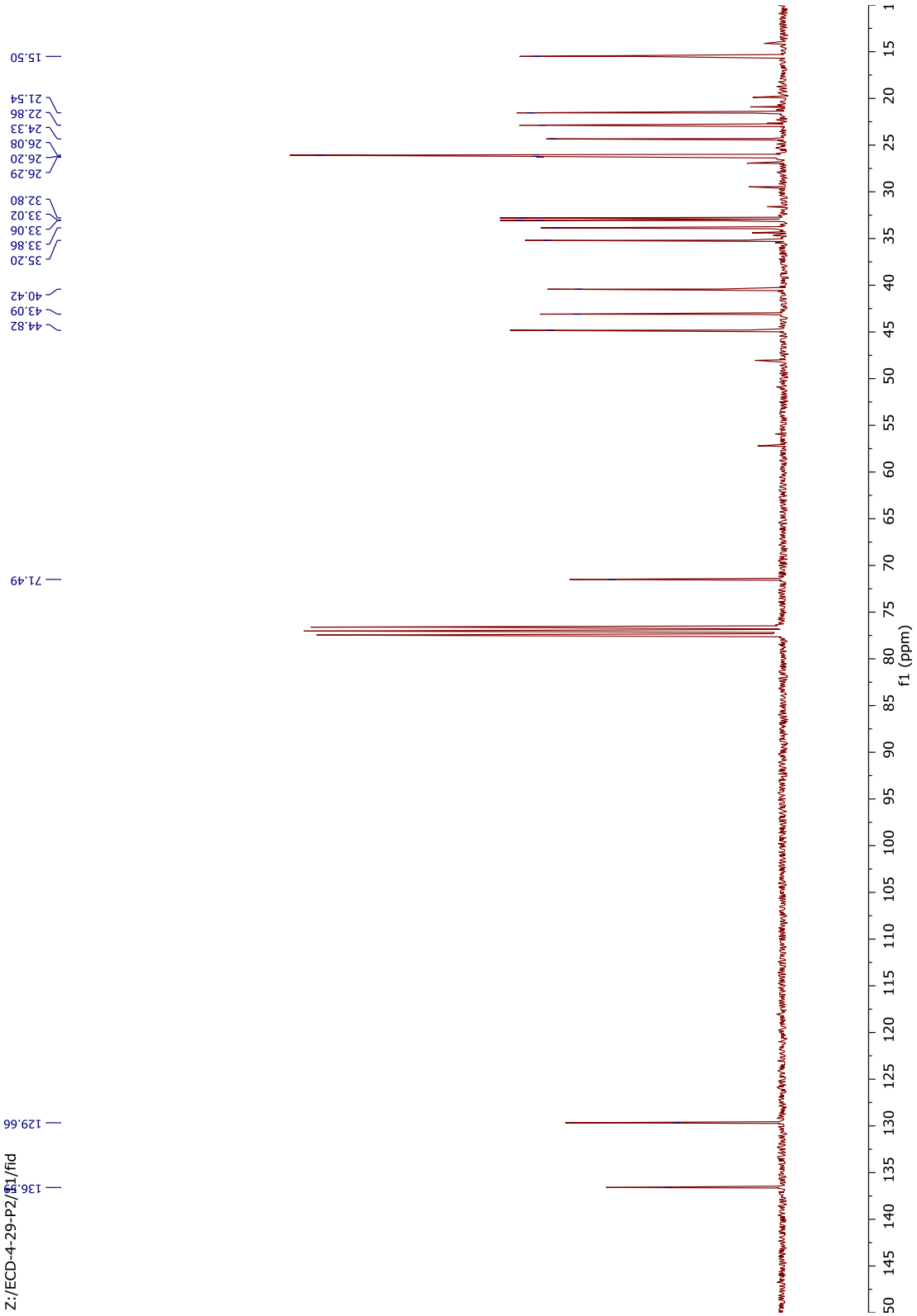
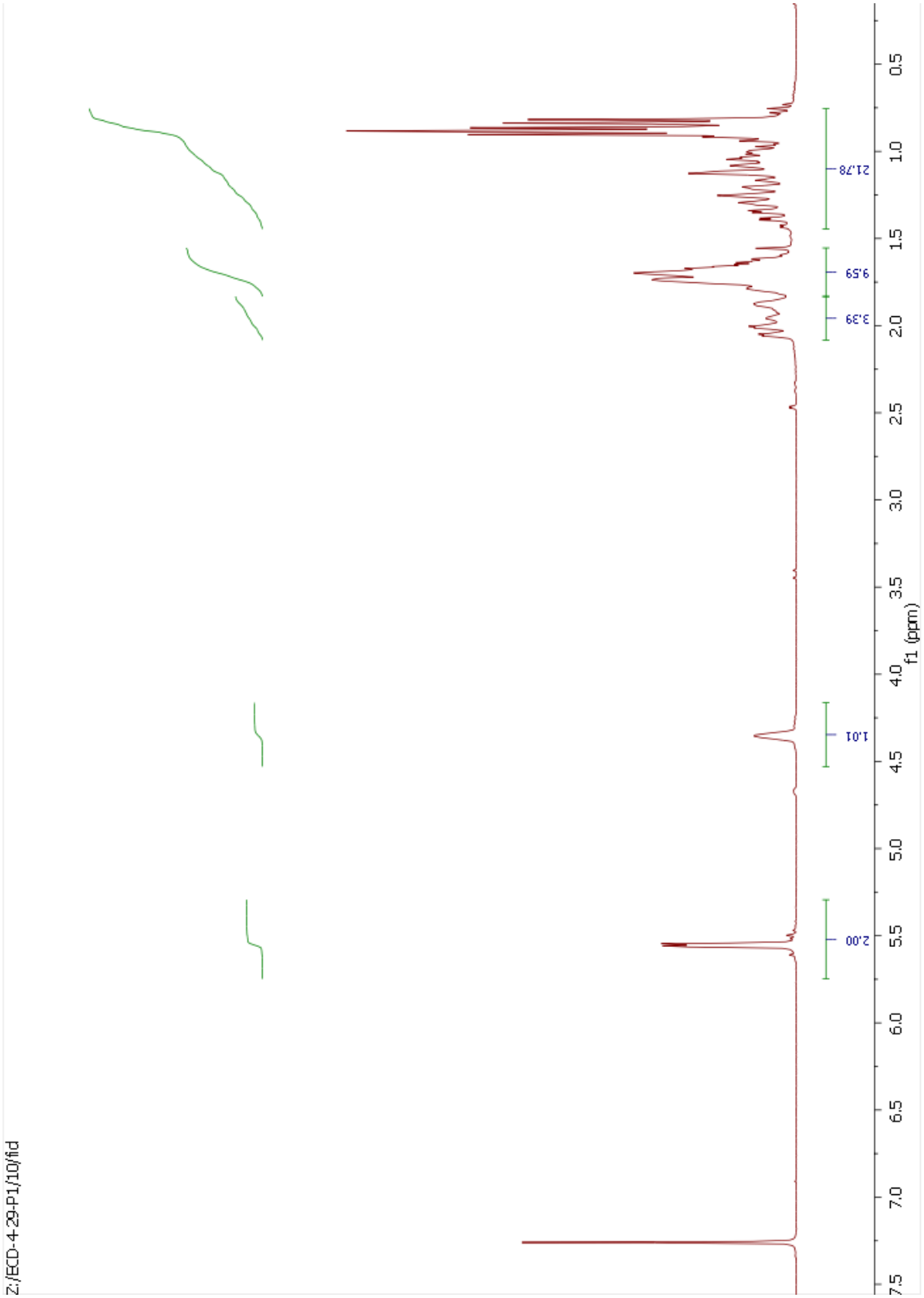
(1*R*)-(*E*)- 3-Cyclohexyl-1-(*p*-menthan-3-yl)prop-2-en-1-ol (16e) (^{1}H NMR)



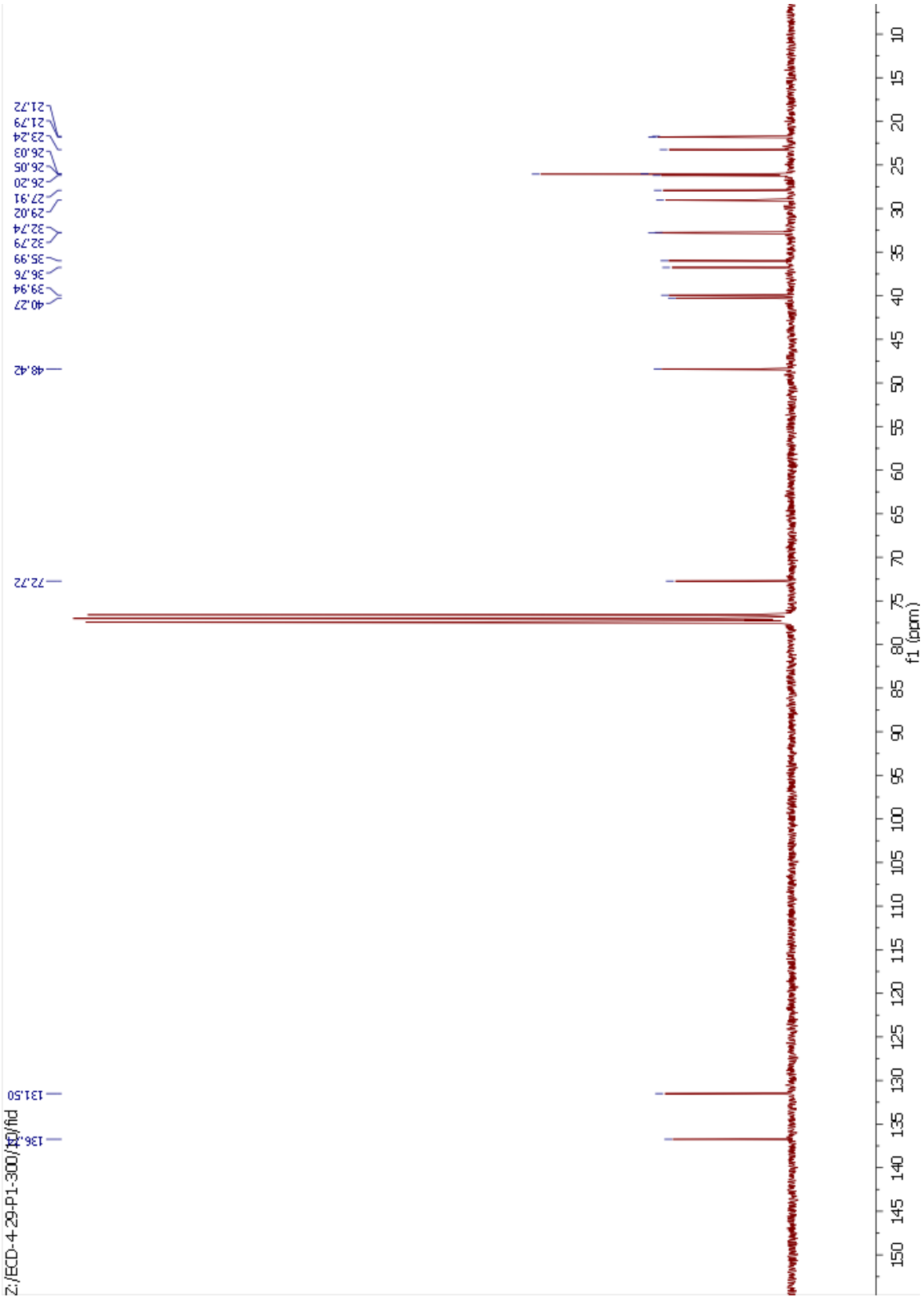
(1*R*)-(*E*)- 3-Cyclohexyl-1-(*p*-menthan-3-yl)prop-2-en-1-ol (16e) (^{13}C NMR)



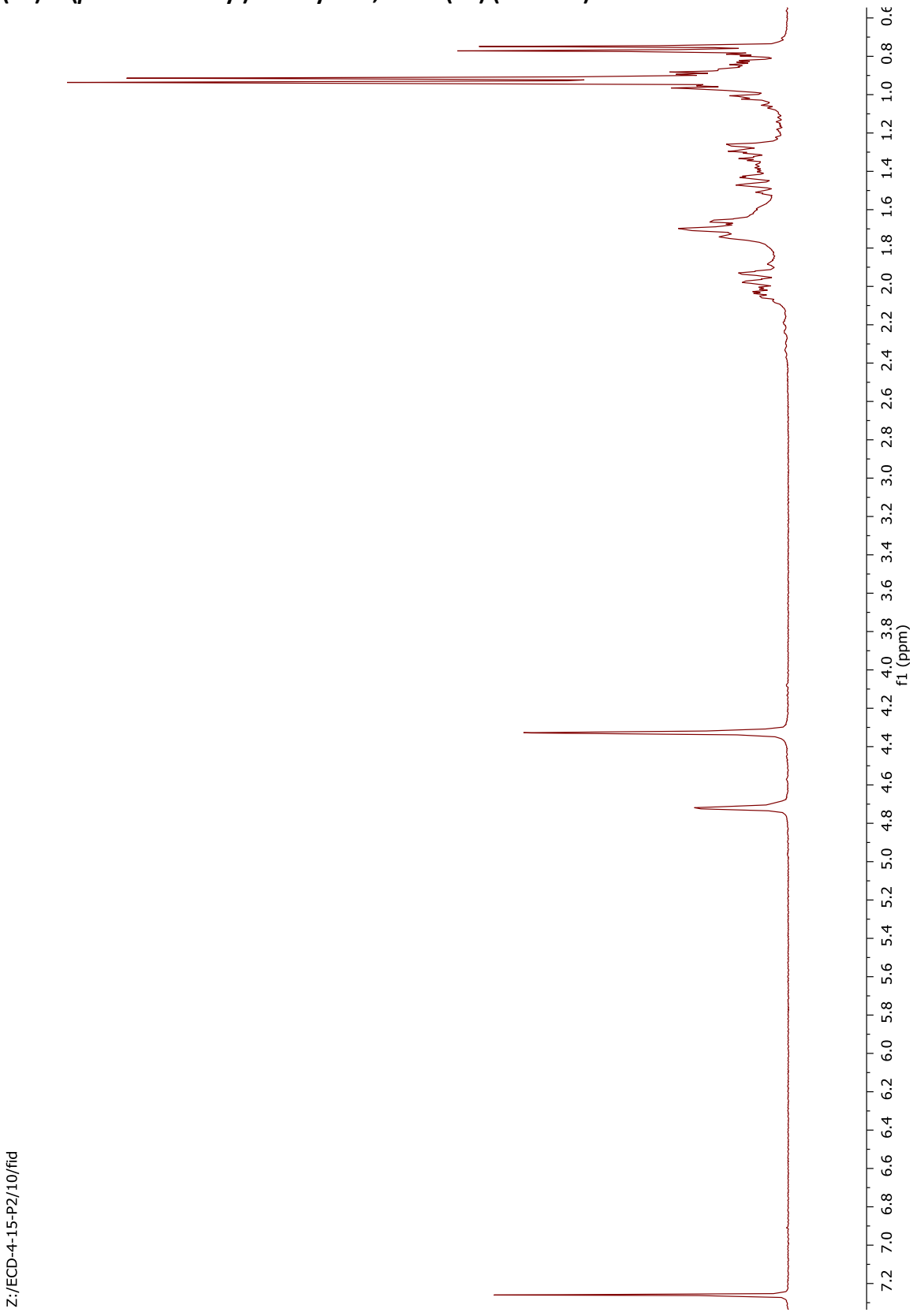
(1*S*)-(*E*)- 3-Cyclohexyl-1-(*p*-menthan-3-yl)prop-2-en-1-ol (19e) (^{1}H NMR)



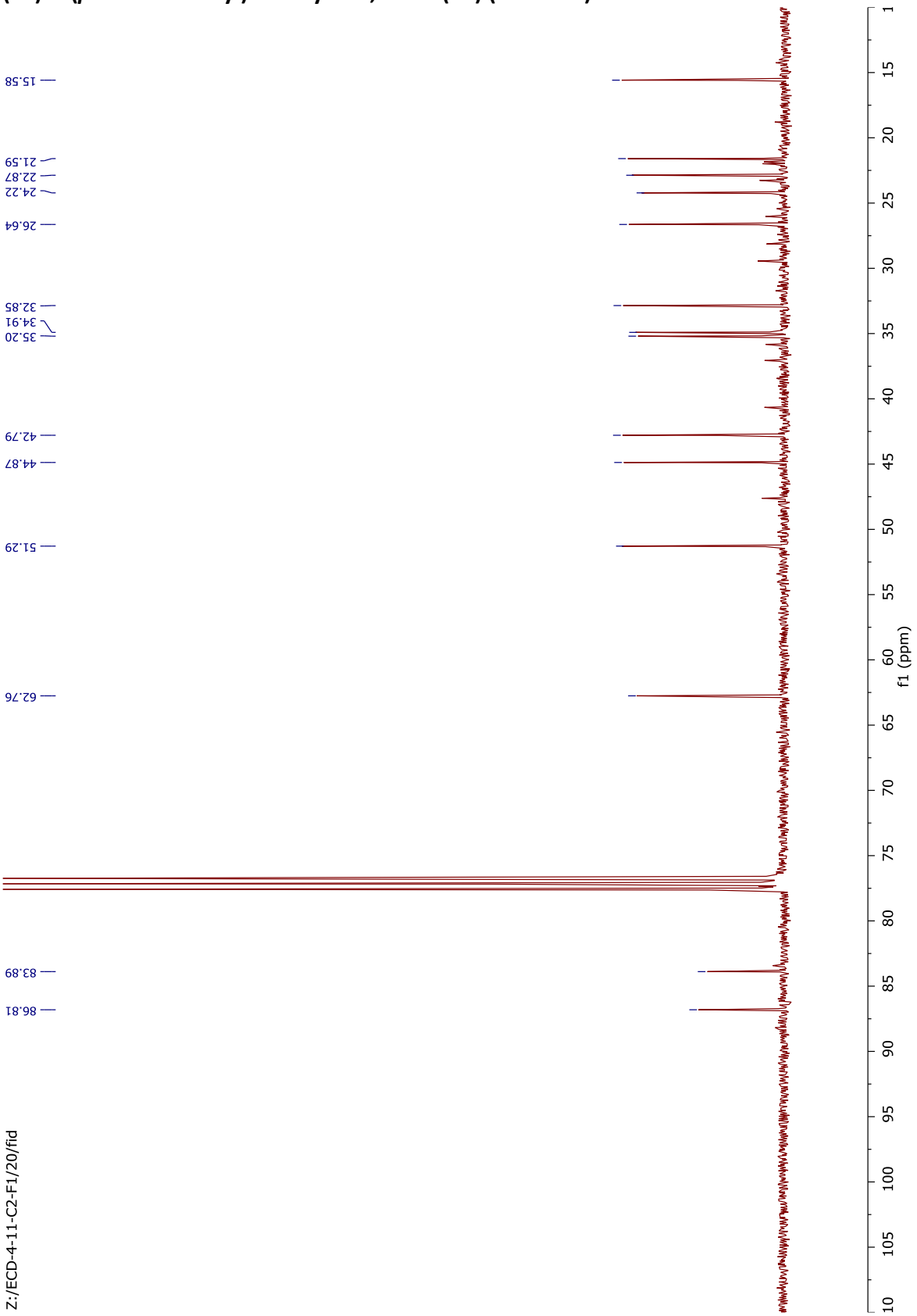
(1*S*)-(*E*)- 3-Cyclohexyl-1-(*p*-menthan-3-yl)prop-2-en-1-ol (19e) (^{13}C NMR)



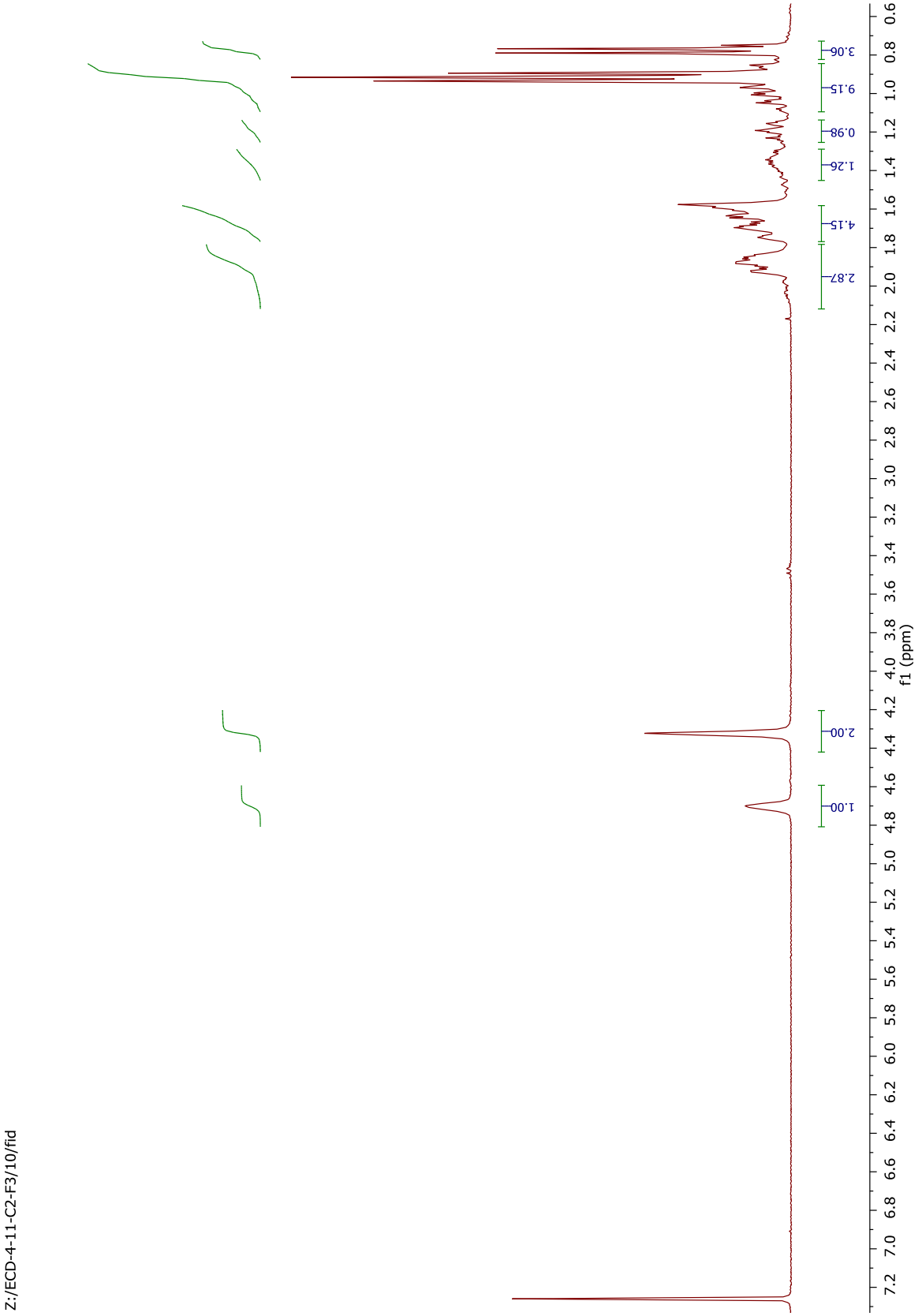
(1*S*)-1-(*p*-Menthan-3-yl)but-2-yne-1,4-diol (23) (^{1}H NMR)



(1*S*)-1-(*p*-Menthan-3-yl)but-2-yne-1,4-diol (23) (^{13}C NMR)



(1*R*)-1-(*p*-Menthan-3-yl)but-2-yne-1,4-diol (24) (^{1}H NMR)



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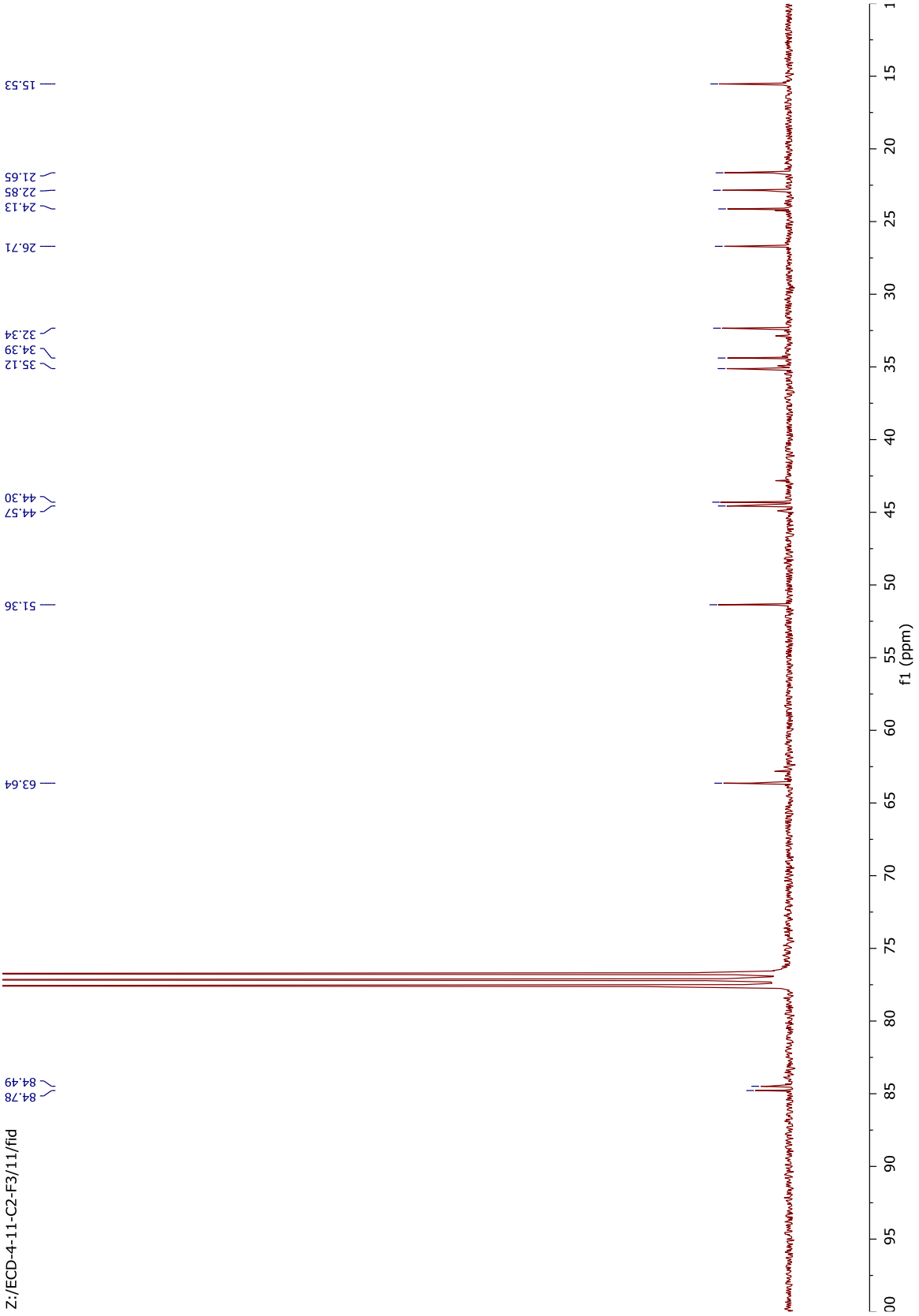
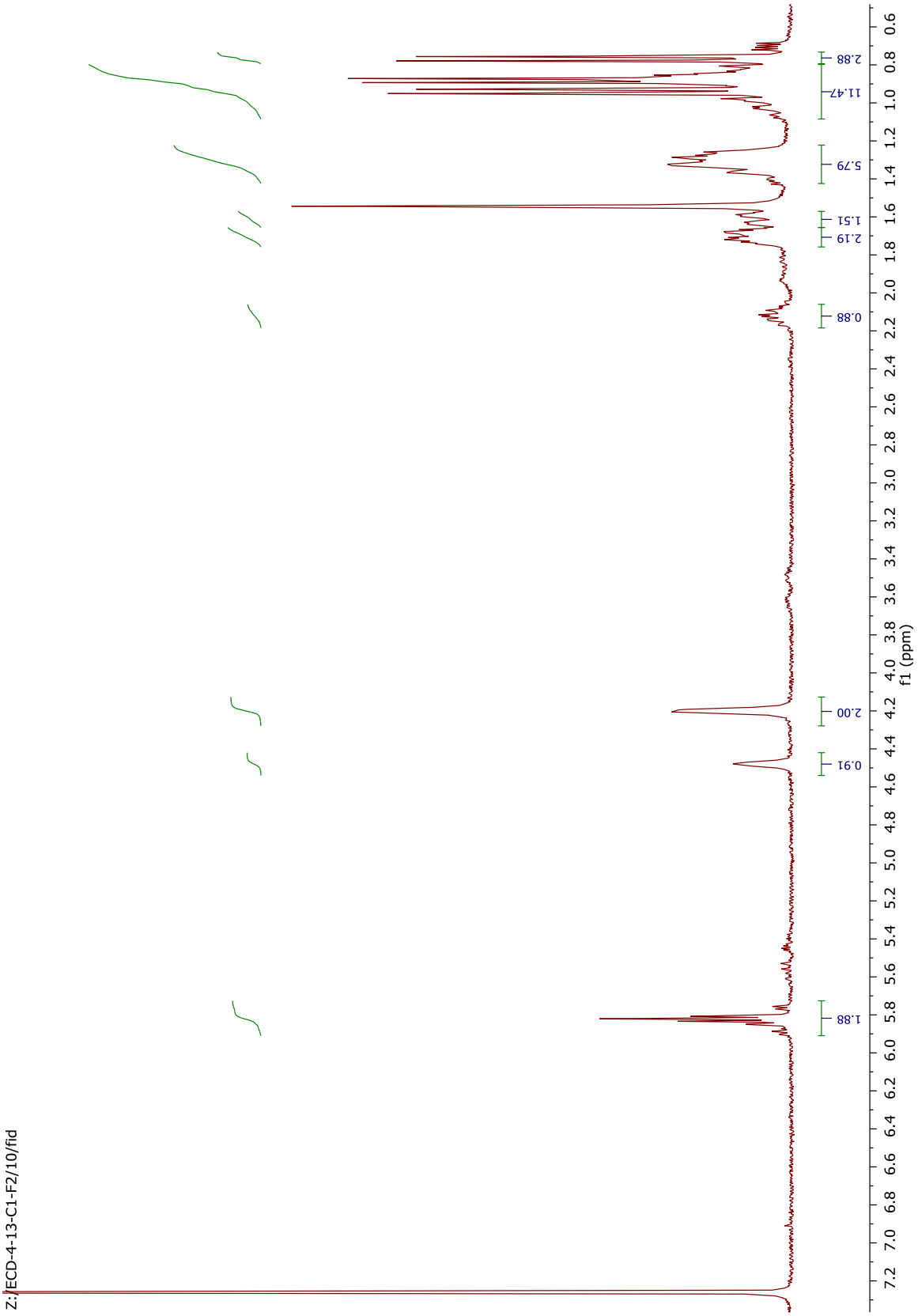
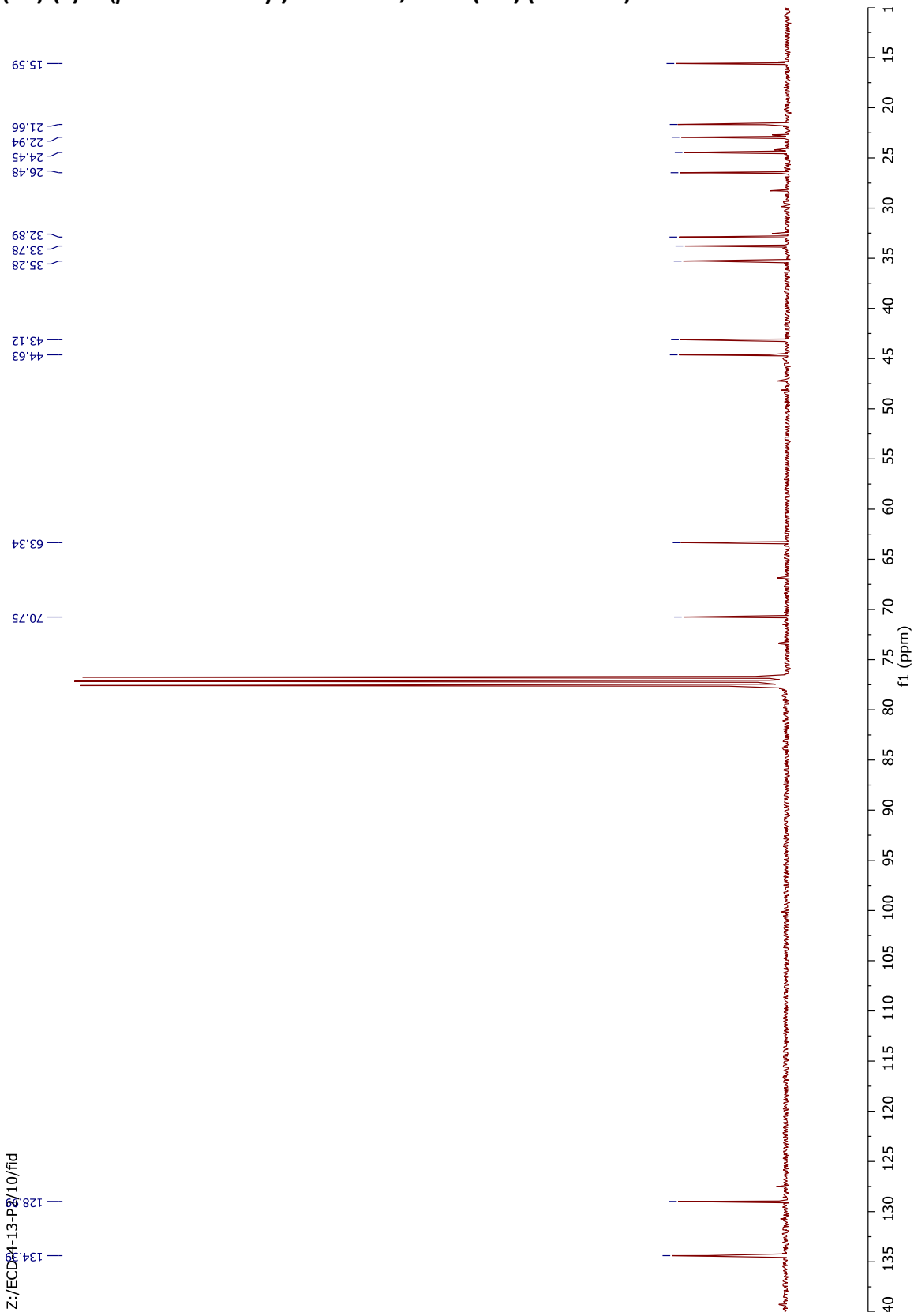


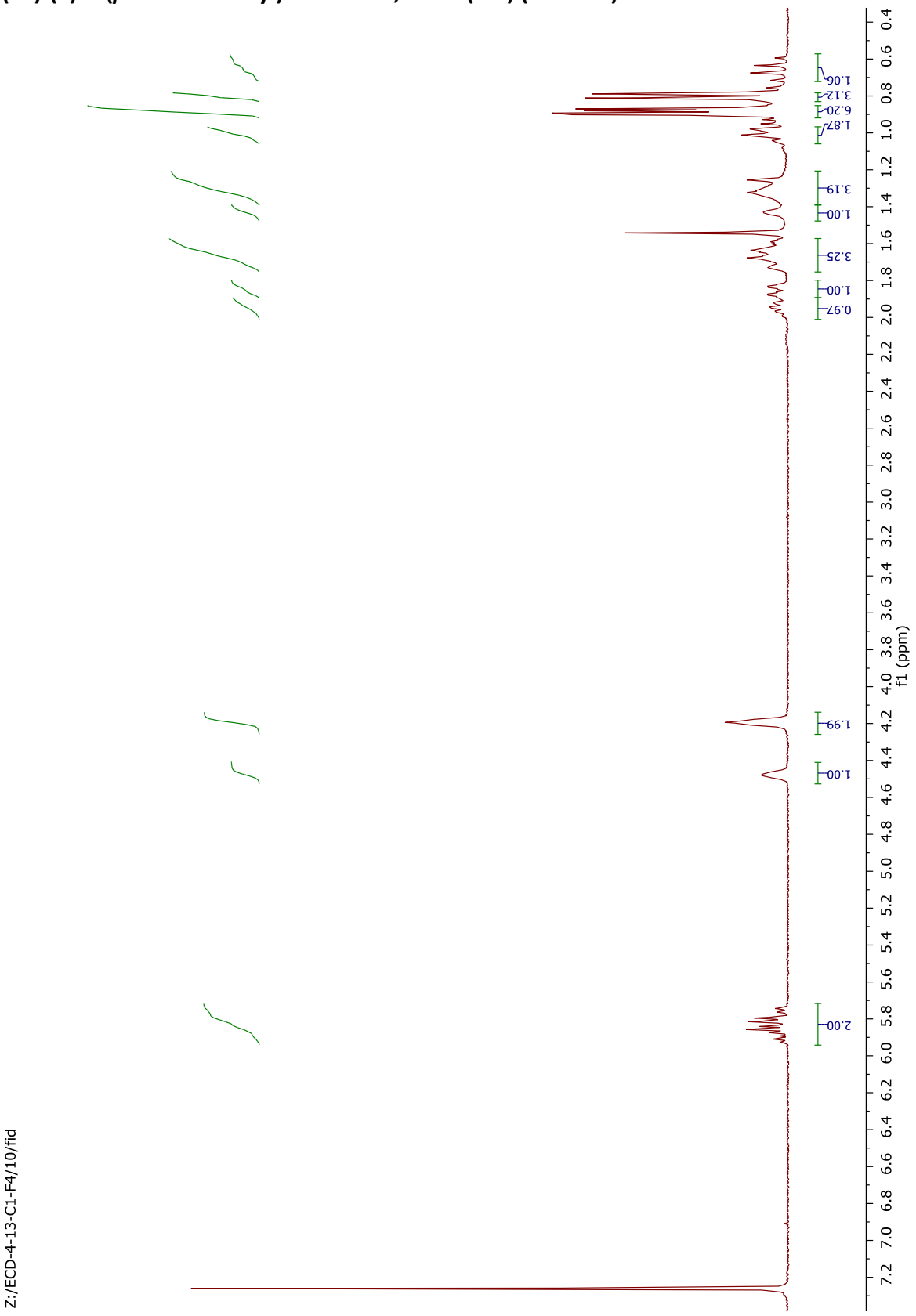
(1*R*)-(*E*)-1-(*p*-Menthan-3-yl)but-2-en-1,4-diol (16f) (^{1}H NMR)



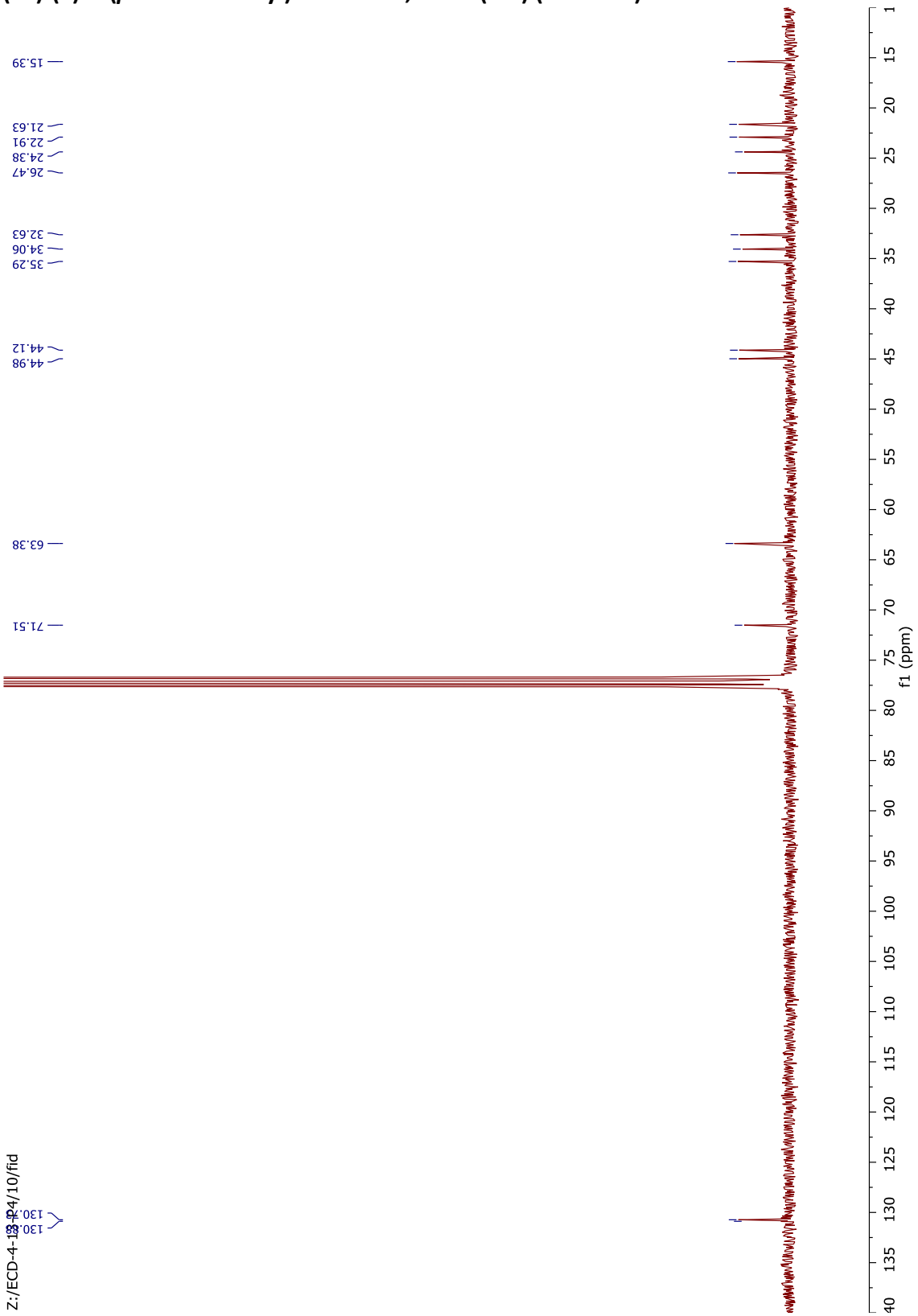
(1*R*)-(*E*)-1-(*p*-Menthan-3-yl)but-2-en-1,4-diol (16f) (^{13}C NMR)



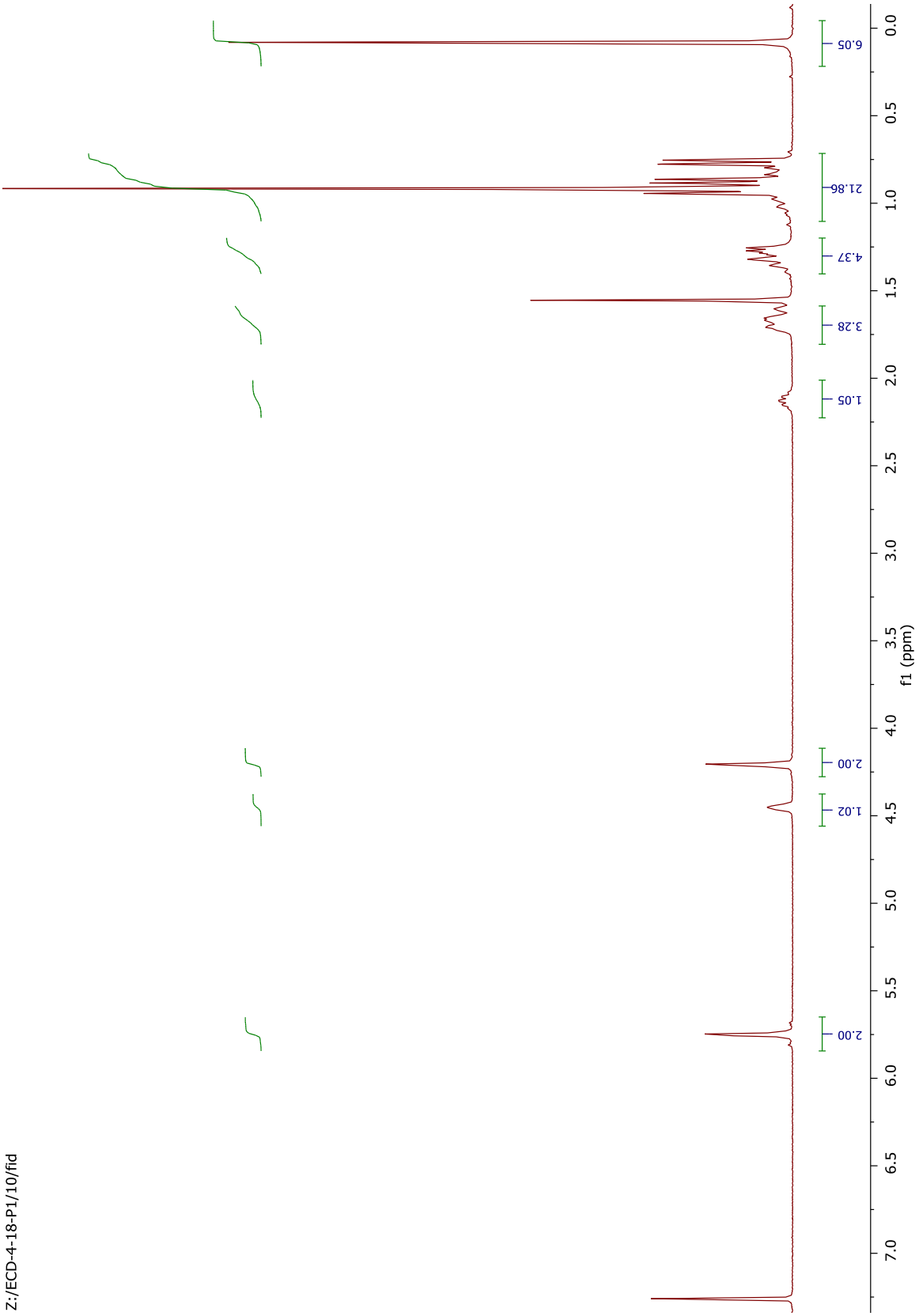
(1*S*)-(*E*)-1-(*p*-Menthan-3-yl)but-2-en-1,4-diol (19f) (^{1}H NMR)



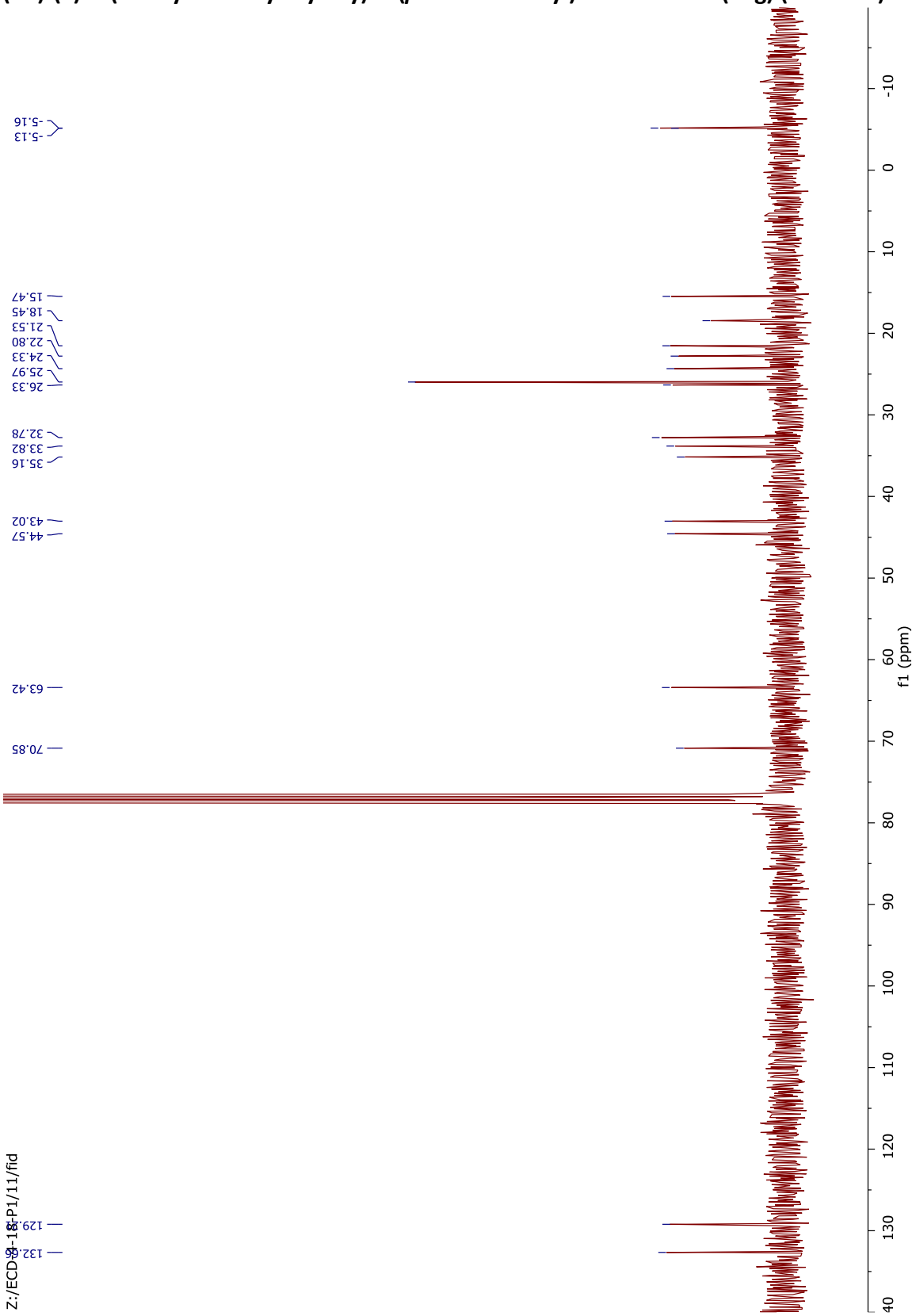
(1*S*)-(*E*)-1-(*p*-Menthan-3-yl)but-2-en-1,4-diol (19f) (^{13}C NMR)



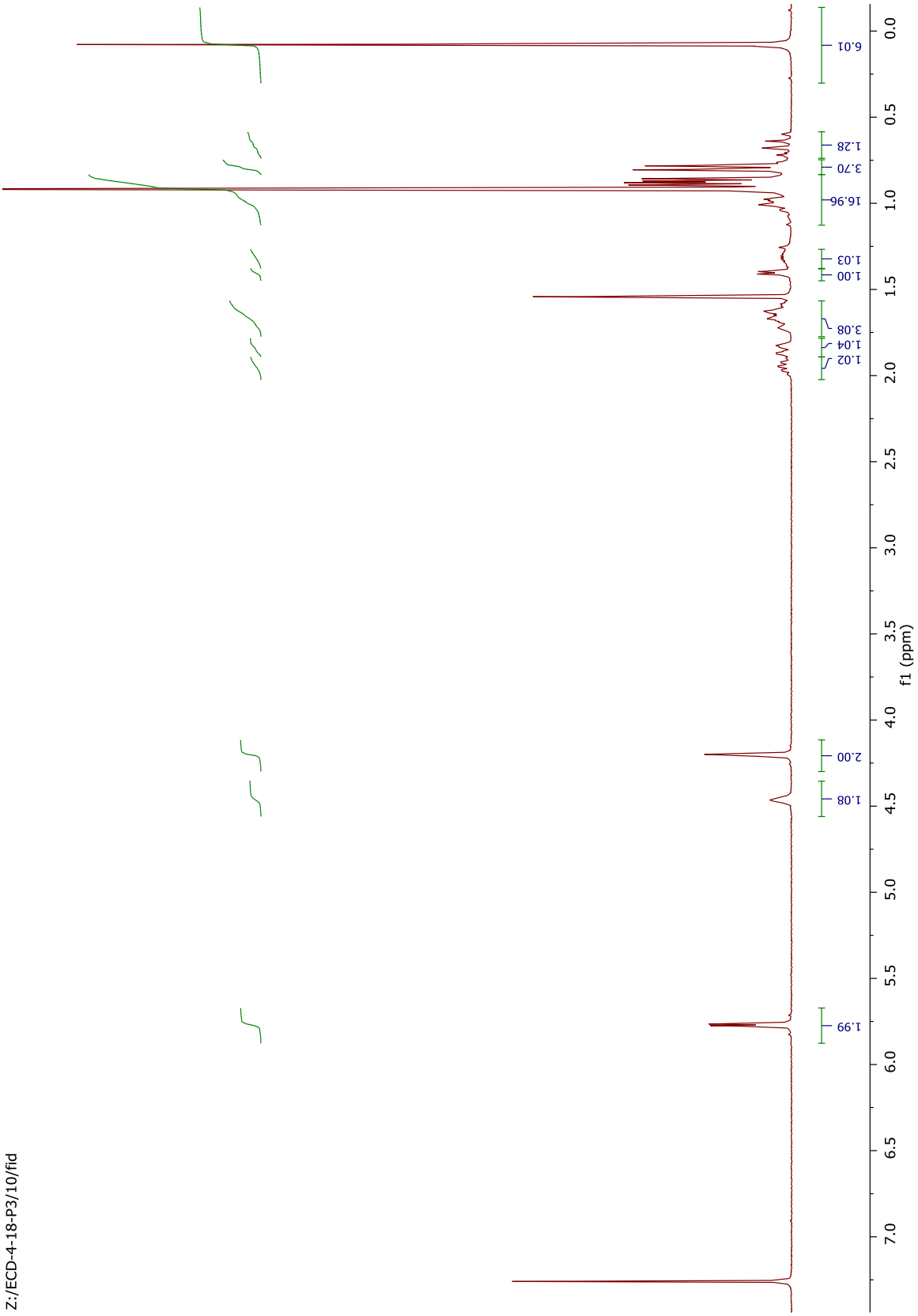
(1*R*)-(*E*)-4-(*t*-Butyldimethylsilyloxy)-1-(*p*-menthan-3-yl)but-2-en-1-ol (16g) (^{1}H NMR)



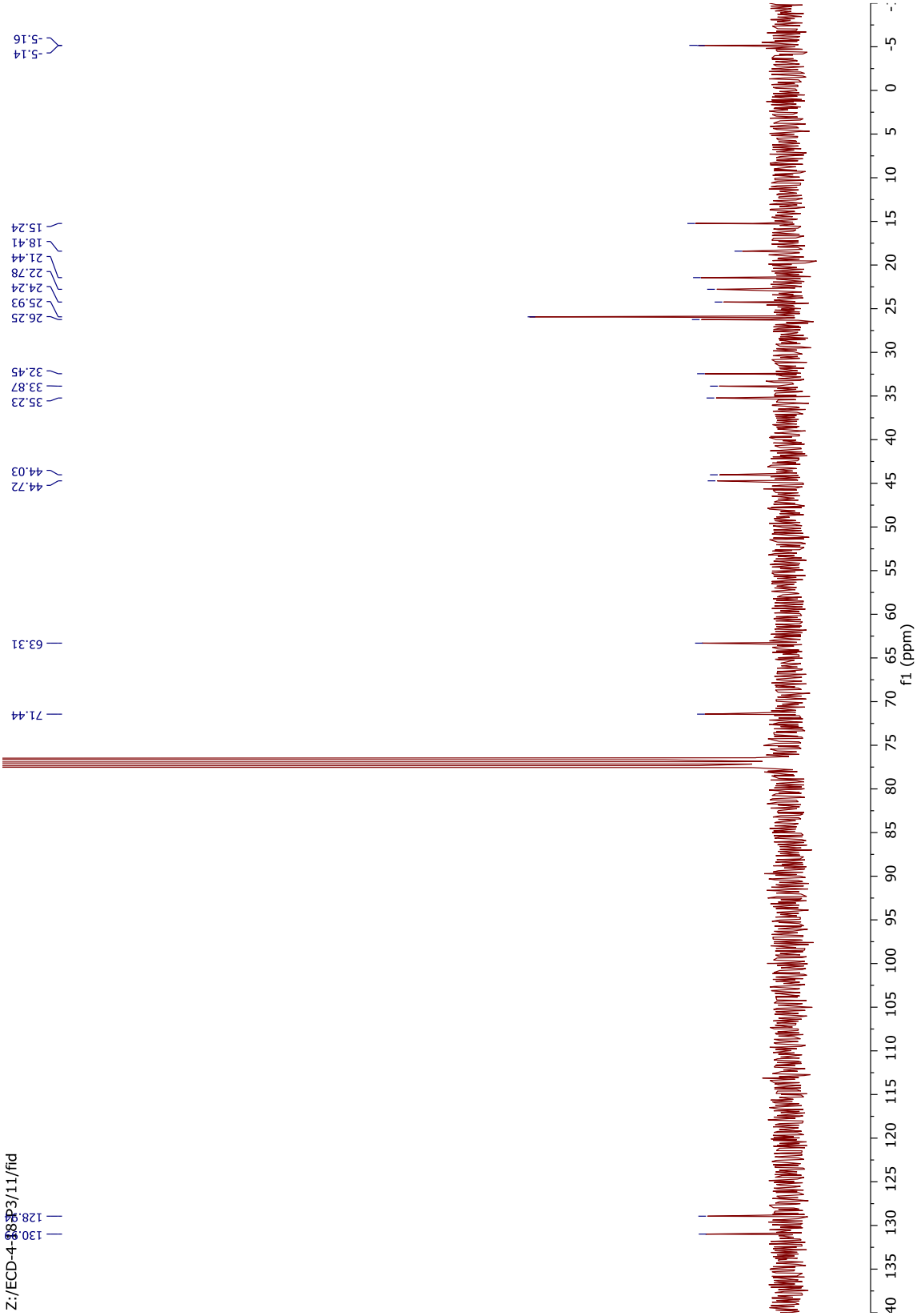
(1*R*)-(*E*)-4-(*t*-Butyldimethylsilyloxy)-1-(*p*-menthan-3-yl)but-2-en-1-ol (16g) (^{13}C NMR)



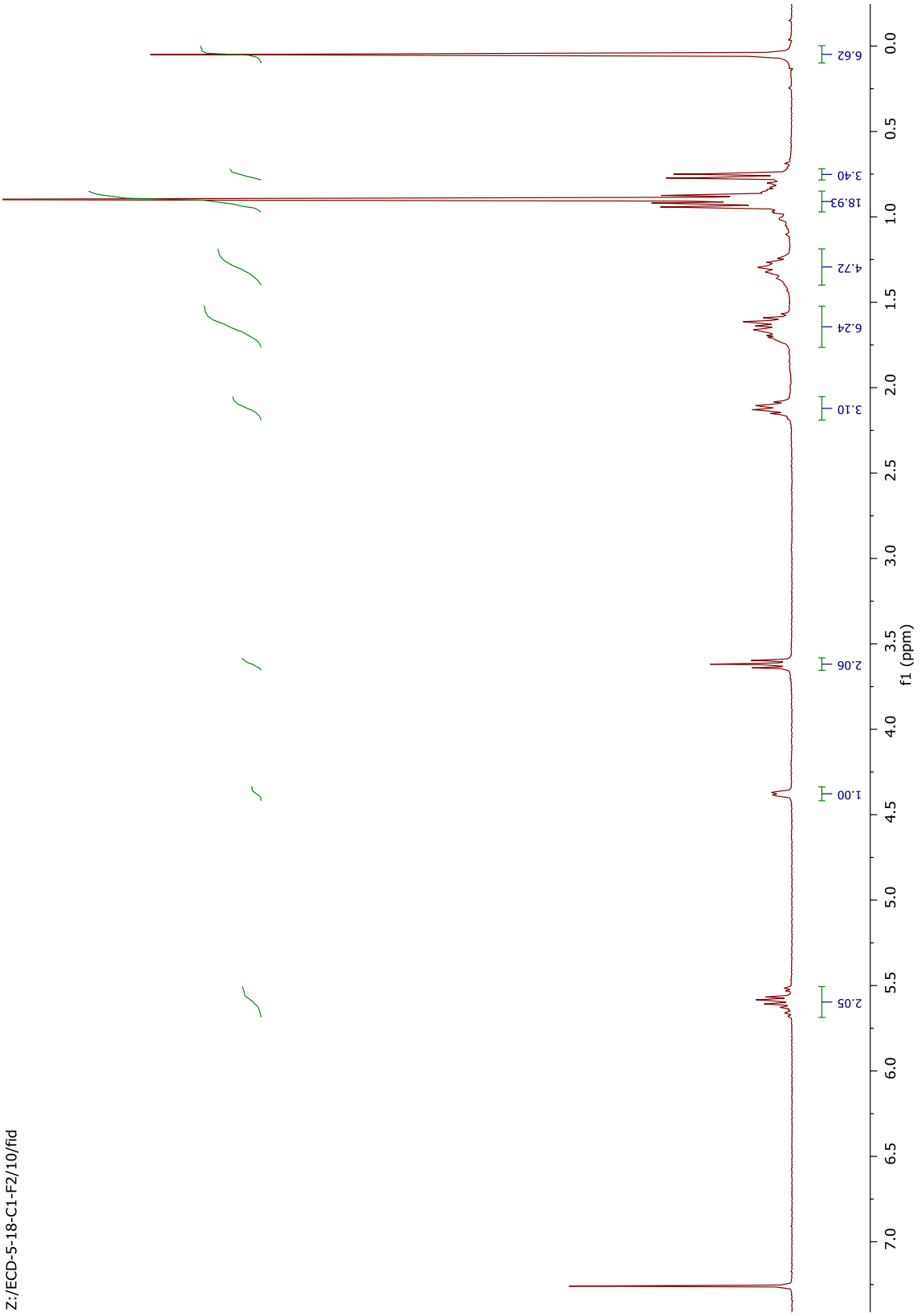
(1*S*)-(*E*)-4-(*t*-Butyldimethylsilyloxy)-1-(*p*-menthan-3-yl)but-2-en-1-ol (19g) (^{1}H NMR)



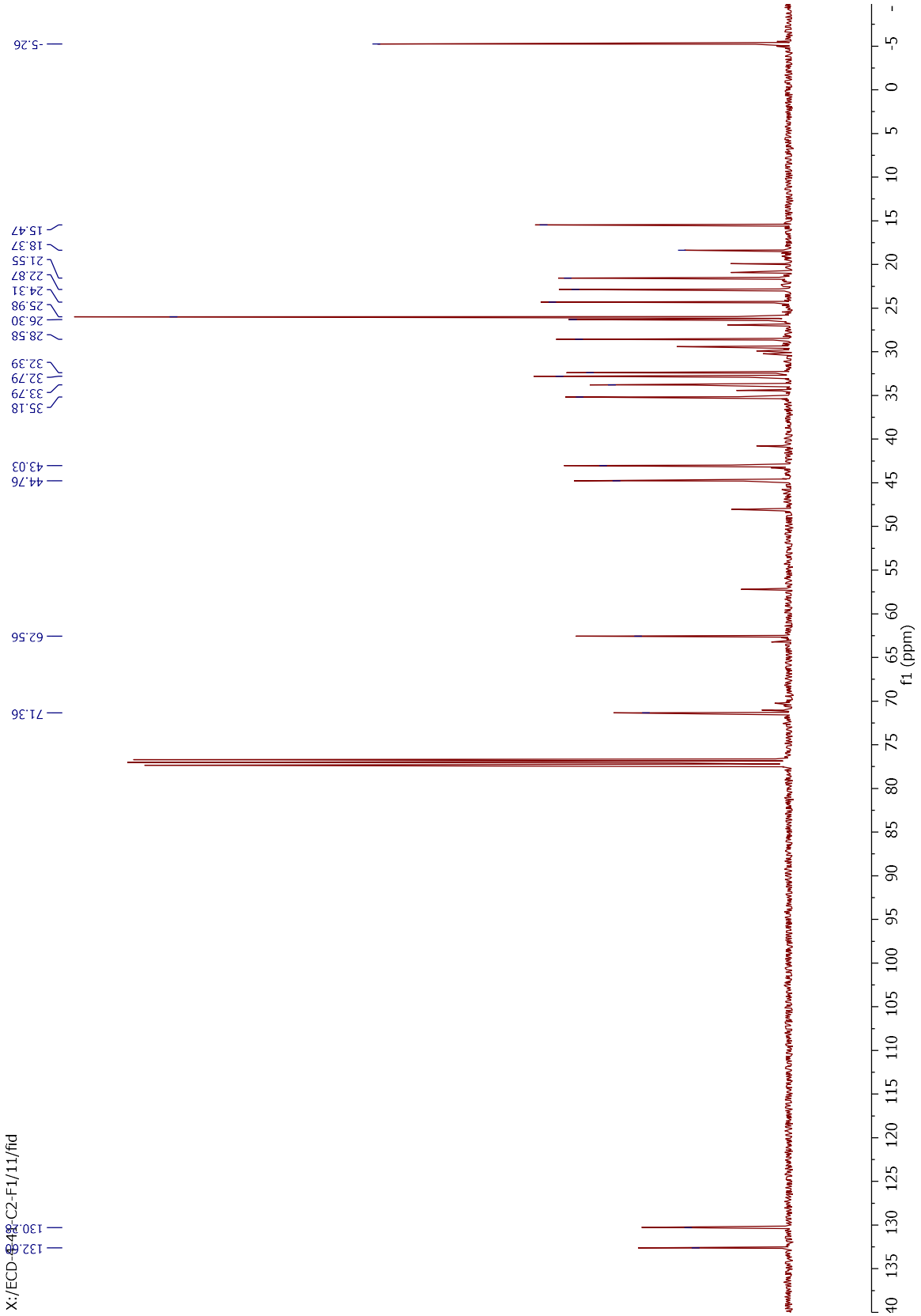
(1*S*)-(*E*)-4-(*t*-Butyldimethylsilyloxy)-1-(*p*-menthan-3-yl)but-2-en-1-ol (19g) (^{13}C NMR)



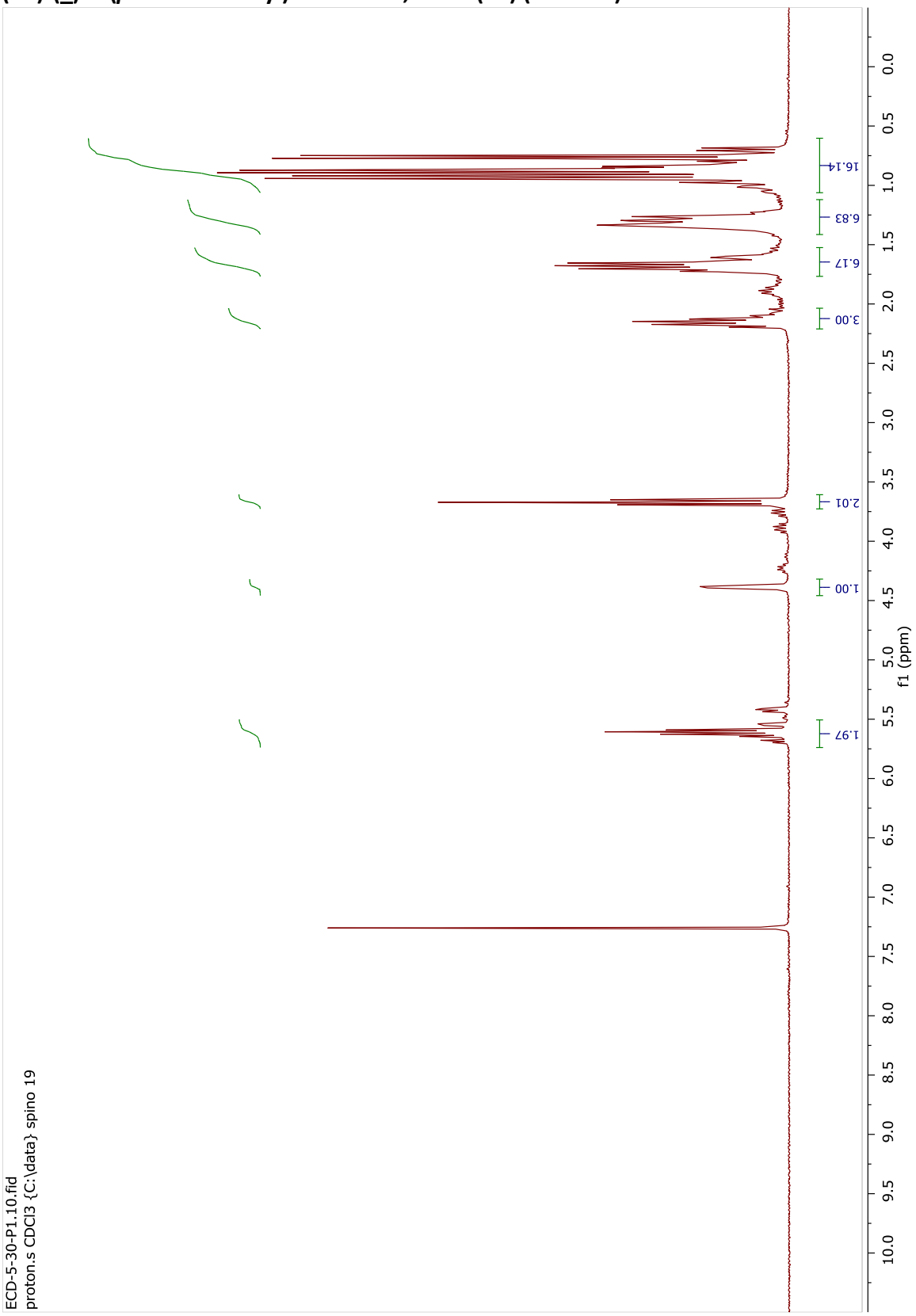
(1*R*)-(*E*)-6-(*t*-Butyldimethylsilyloxy)-1-(*p*-menthan-3-yl)hex-2-en-1-ol (26) (^{1}H NMR)



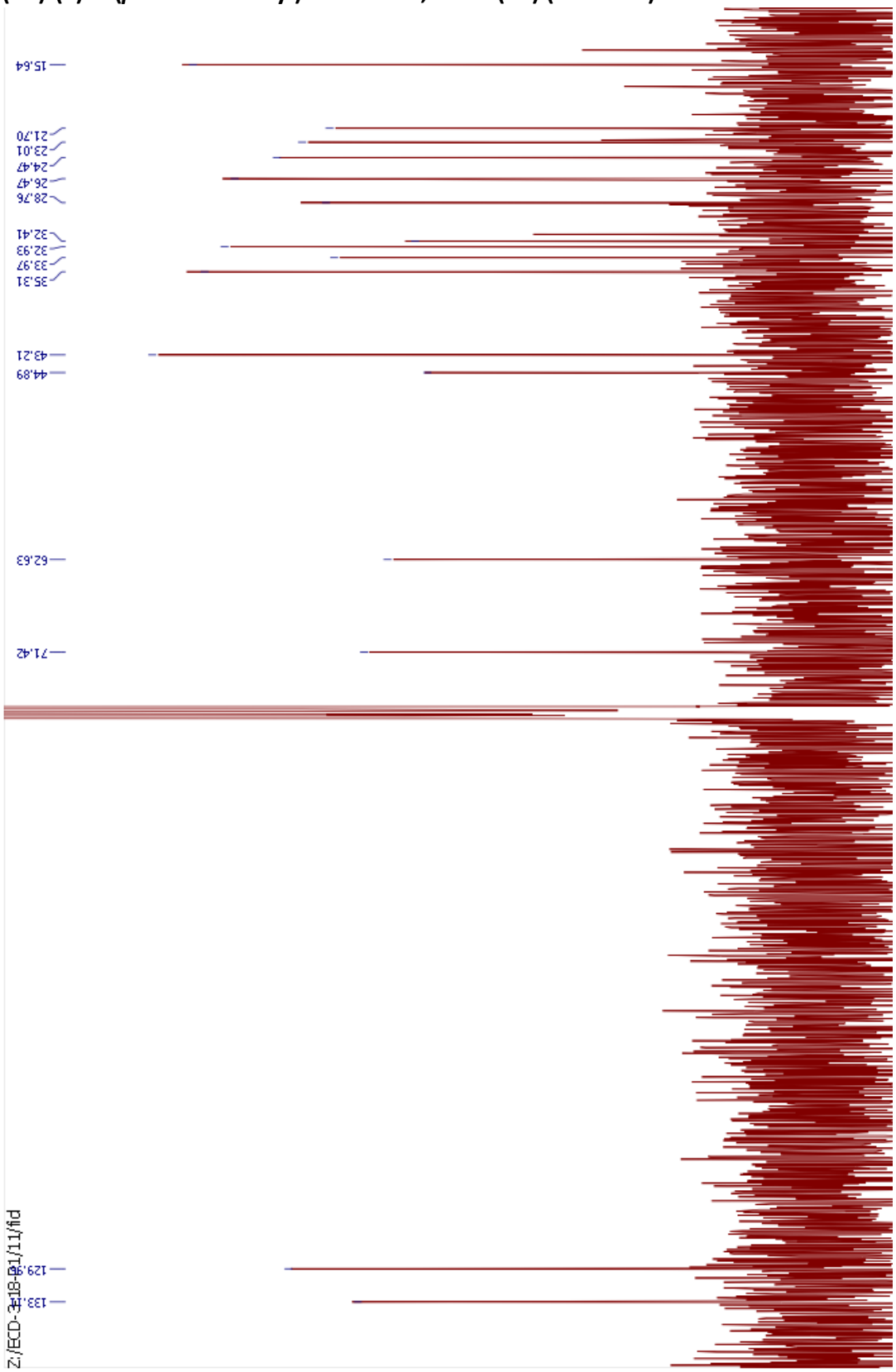
(1*R*)-(*E*)-6-(*t*-Butyldimethylsilyloxy)-1-(*p*-menthan-3-yl)hex-2-en-1-ol (26) (^{13}C NMR)



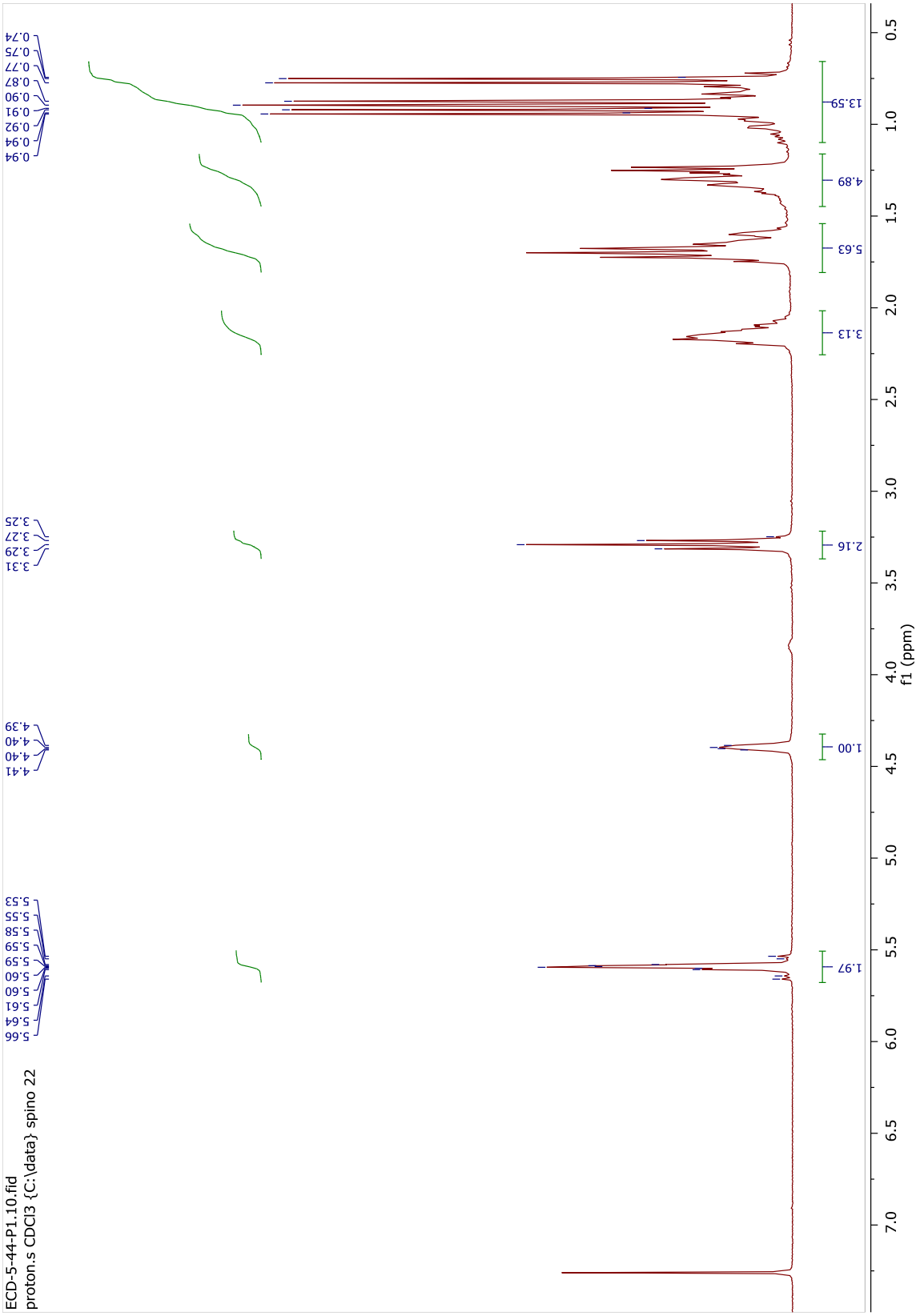
(1*R*)-(*E*)-1-(*p*-Menthan-3-yl)hex-2-en-1,6-diol (27) (^{1}H NMR)



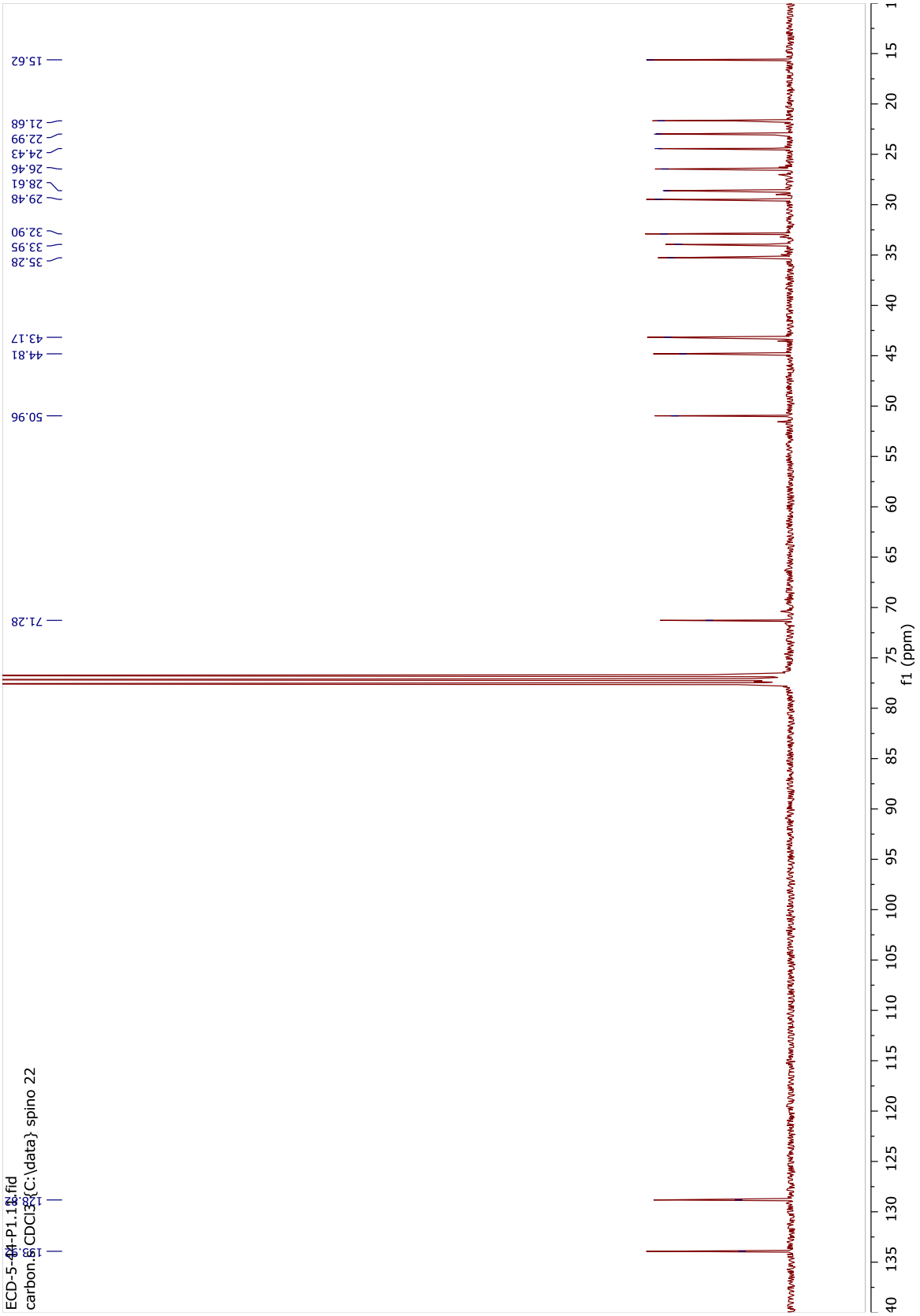
(1*R*)-(*E*)-1-(*p*-Menthan-3-yl)hex-2-en-1,6-diol (27) (^{13}C NMR)



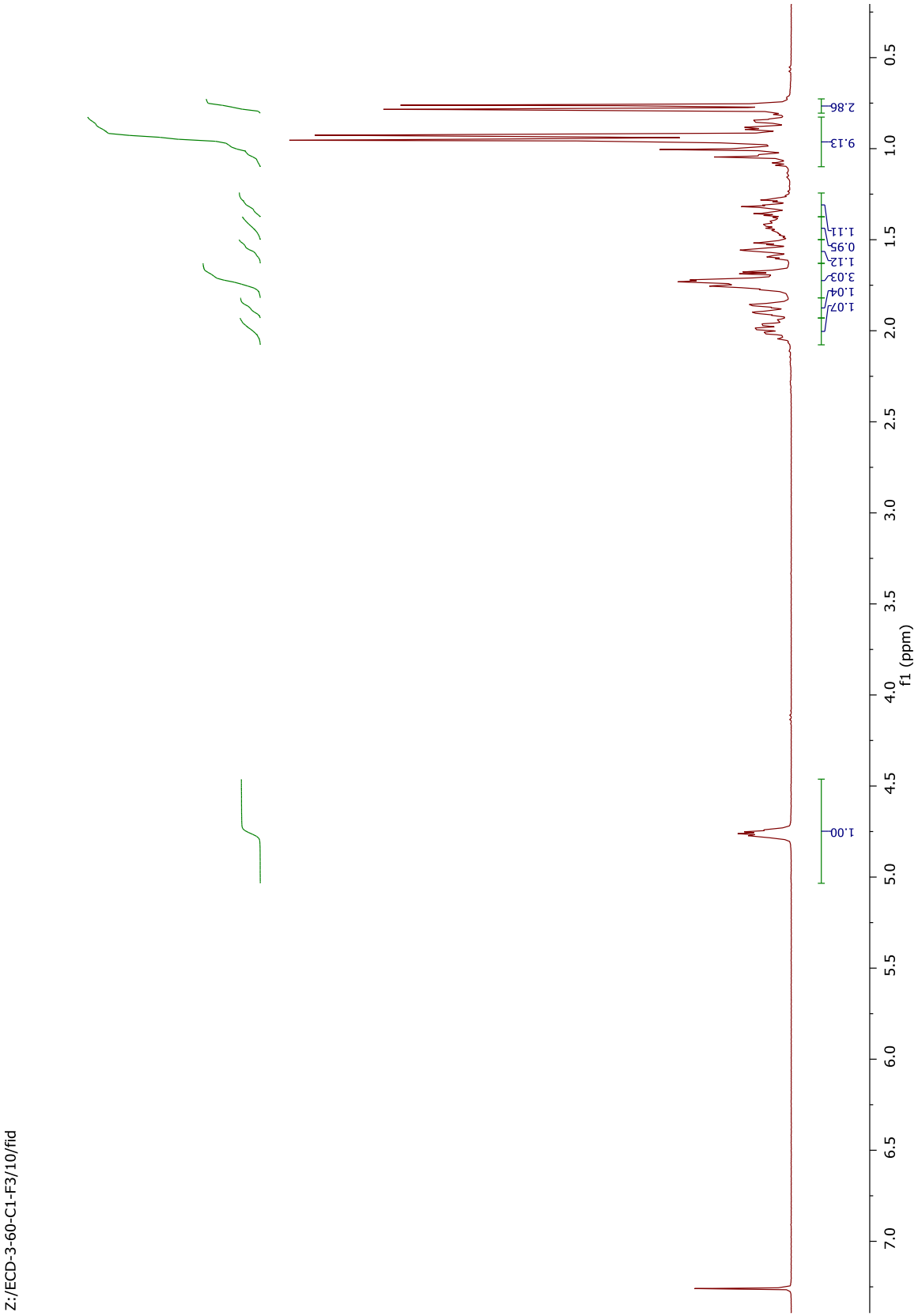
(1*R*)-(*E*)-6-Azido-1-(*p*-menthan-3-yl)hex-2-en-1-ol (16h) (^{1}H NMR)



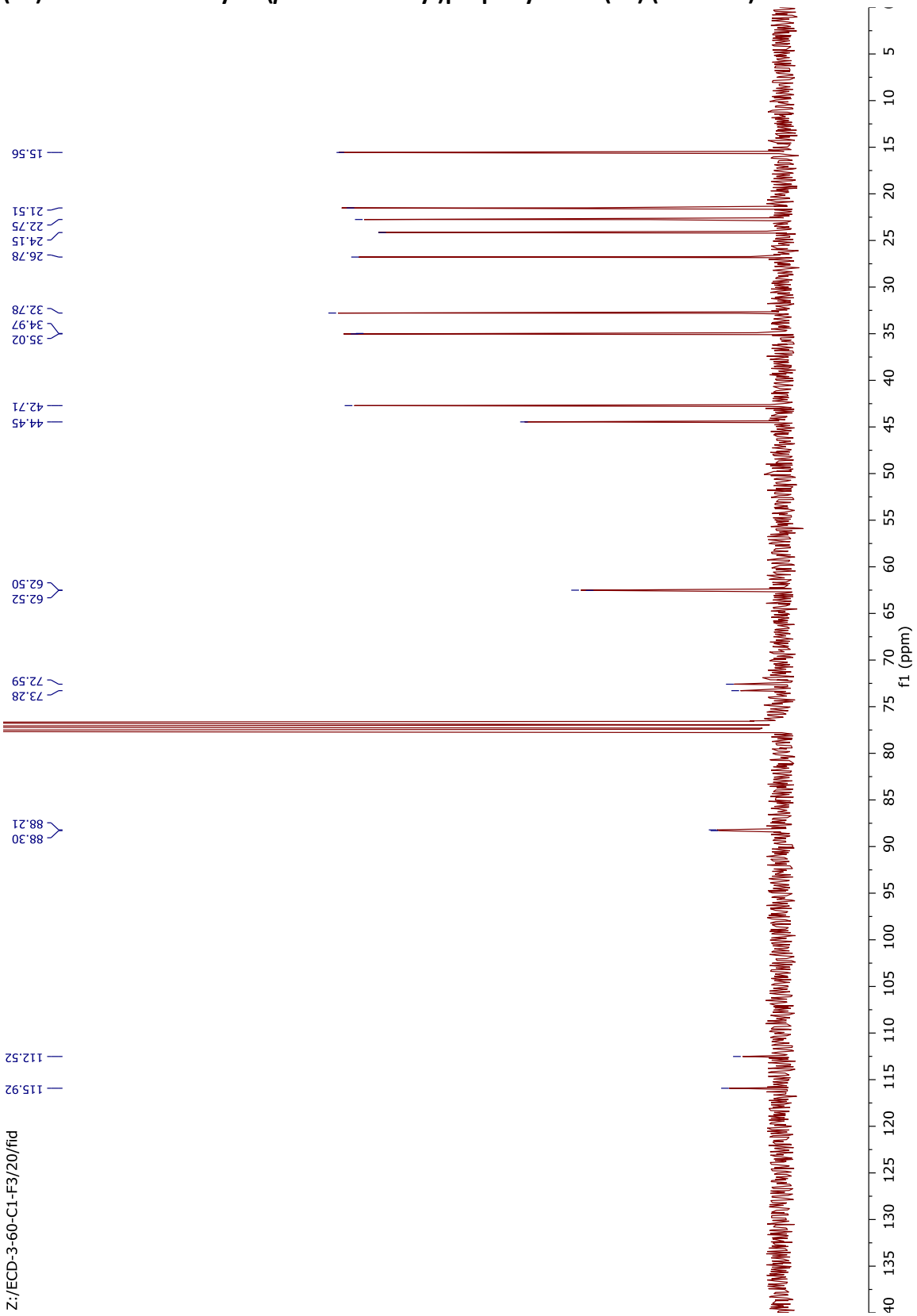
(1*R*)-(*E*)-6-Azido-1-(*p*-menthan-3-yl)hex-2-en-1-ol (16h) (^{13}C NMR)



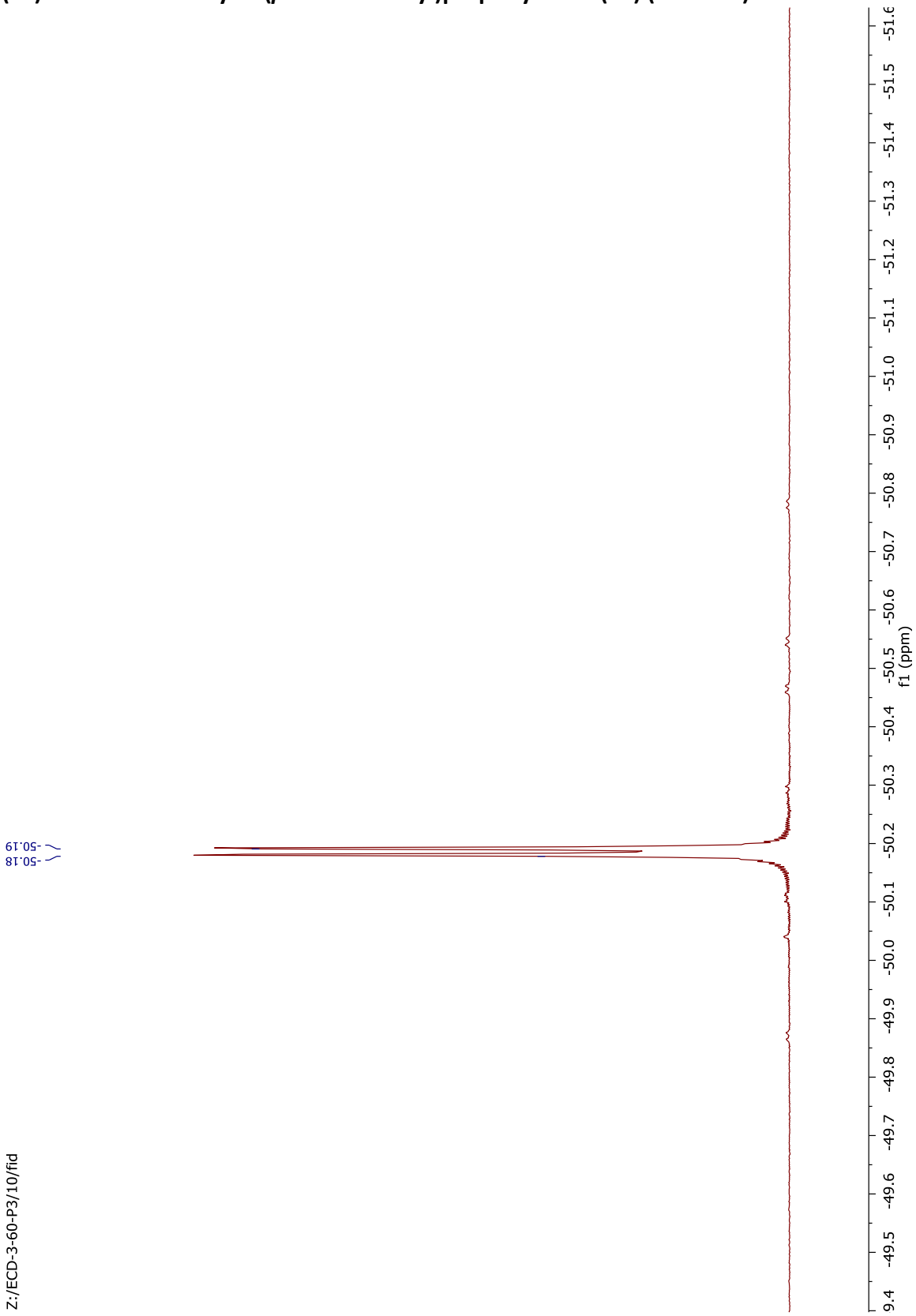
(1*S*)-1-Trifluoromethyl-4-(*p*-menthan-3-yl)prop-2-yn-1-ol (28) (^{1}H NMR)



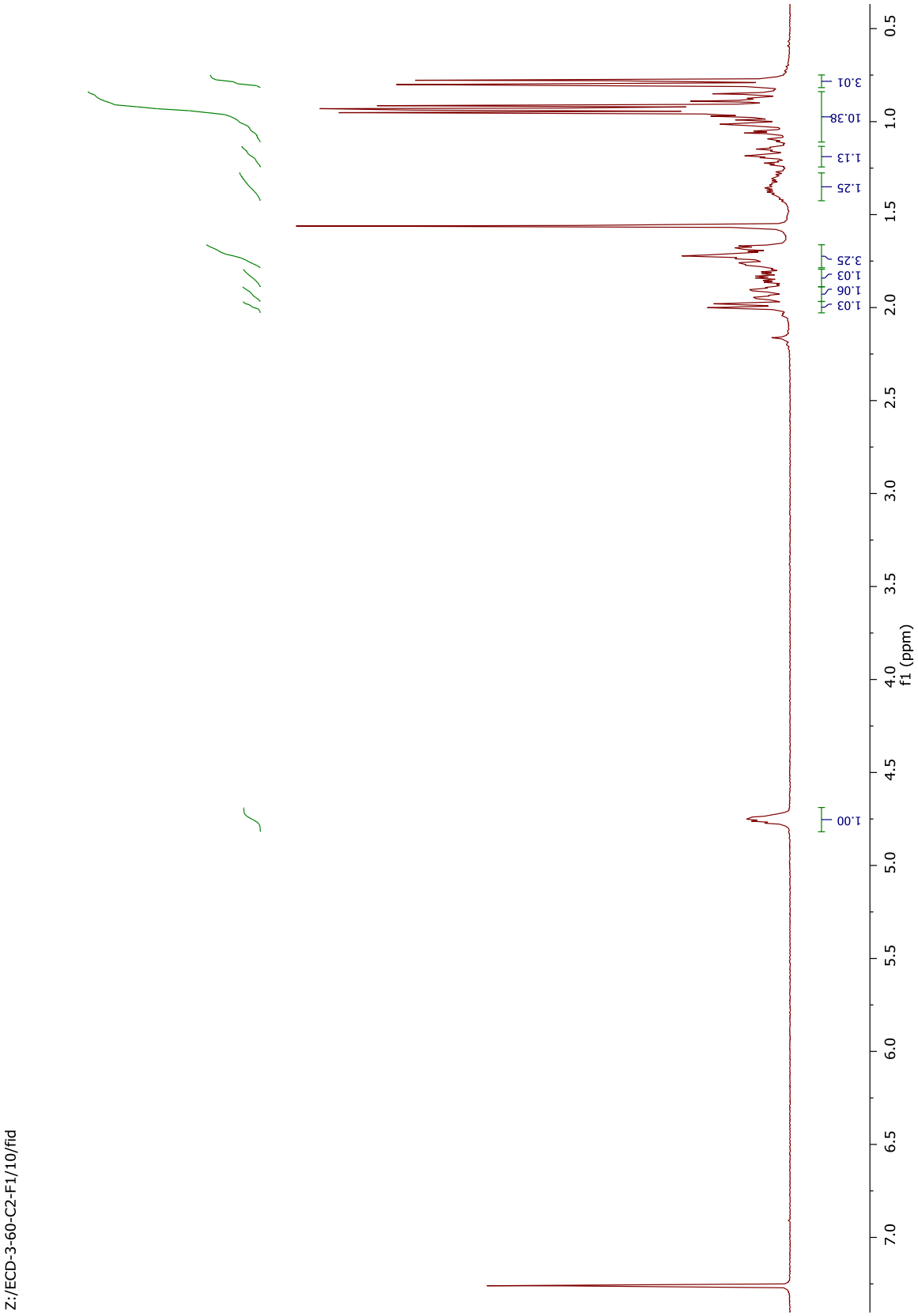
(1*S*)-1-Trifluoromethyl-4-(*p*-menthan-3-yl)prop-2-yn-1-ol (28) (^{13}C NMR)



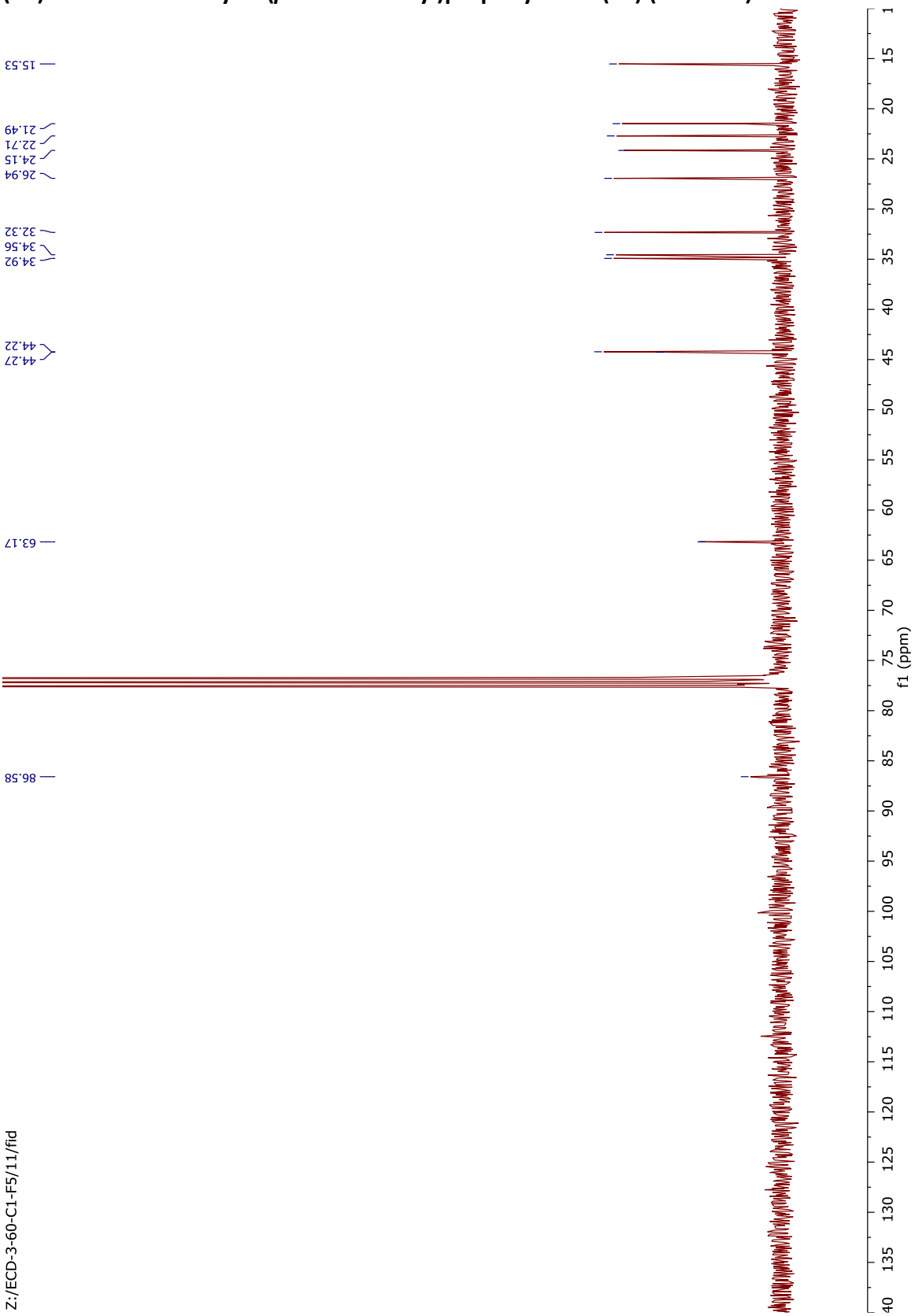
(1*S*)-1-Trifluoromethyl-4-(*p*-menthan-3-yl)prop-2-yn-1-ol (28) (^{19}F NMR)



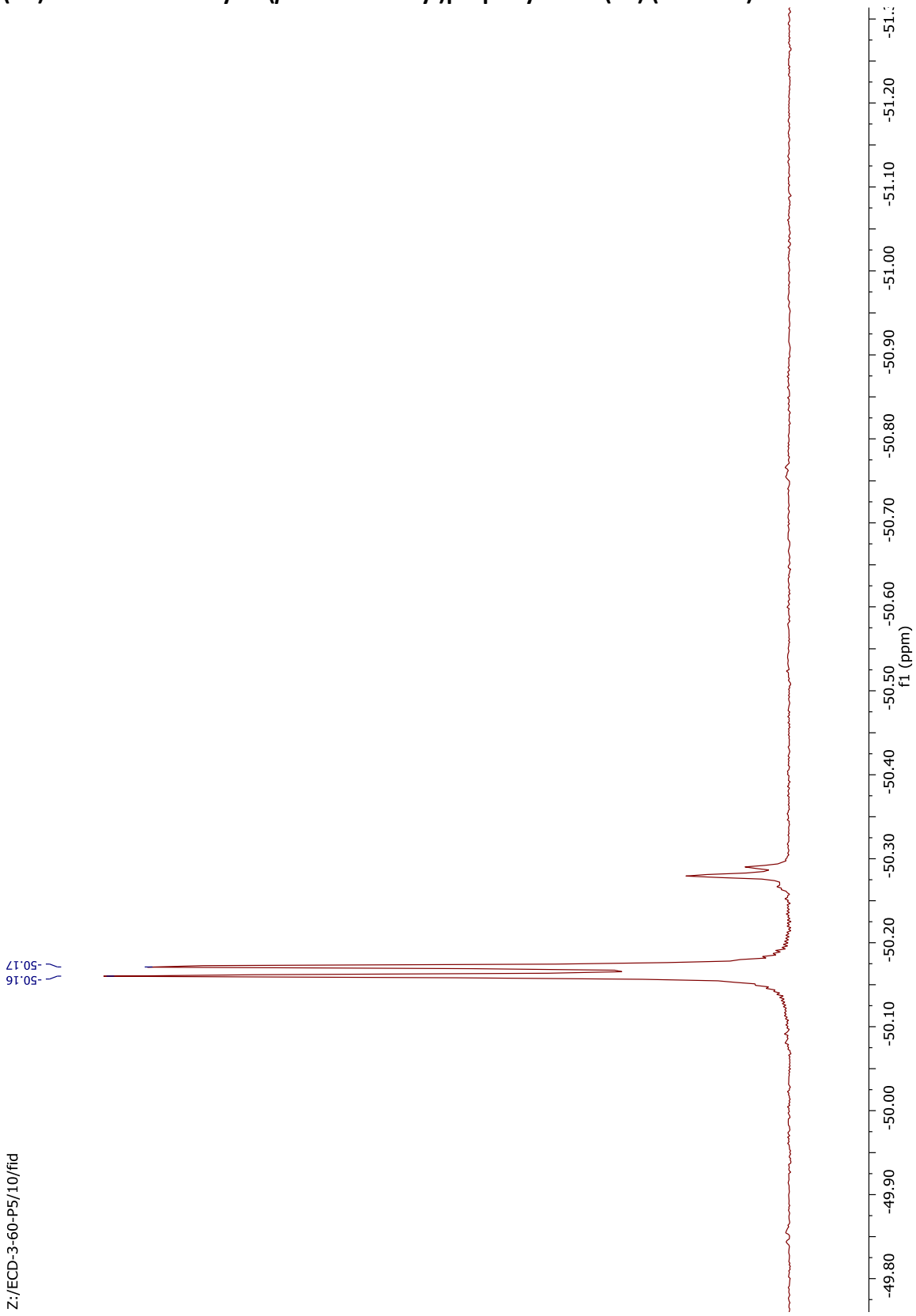
(1*R*)-1-Trifluoromethyl-4-(*p*-menthan-3-yl)prop-2-yn-1-ol (29) (^{1}H NMR)



(1*R*)-1-Trifluoromethyl-4-(*p*-menthan-3-yl)prop-2-yn-1-ol (29) (^{13}C NMR)



(1*R*)-1-Trifluoromethyl-4-(*p*-menthan-3-yl)prop-2-yn-1-ol (29) (^{19}F NMR)



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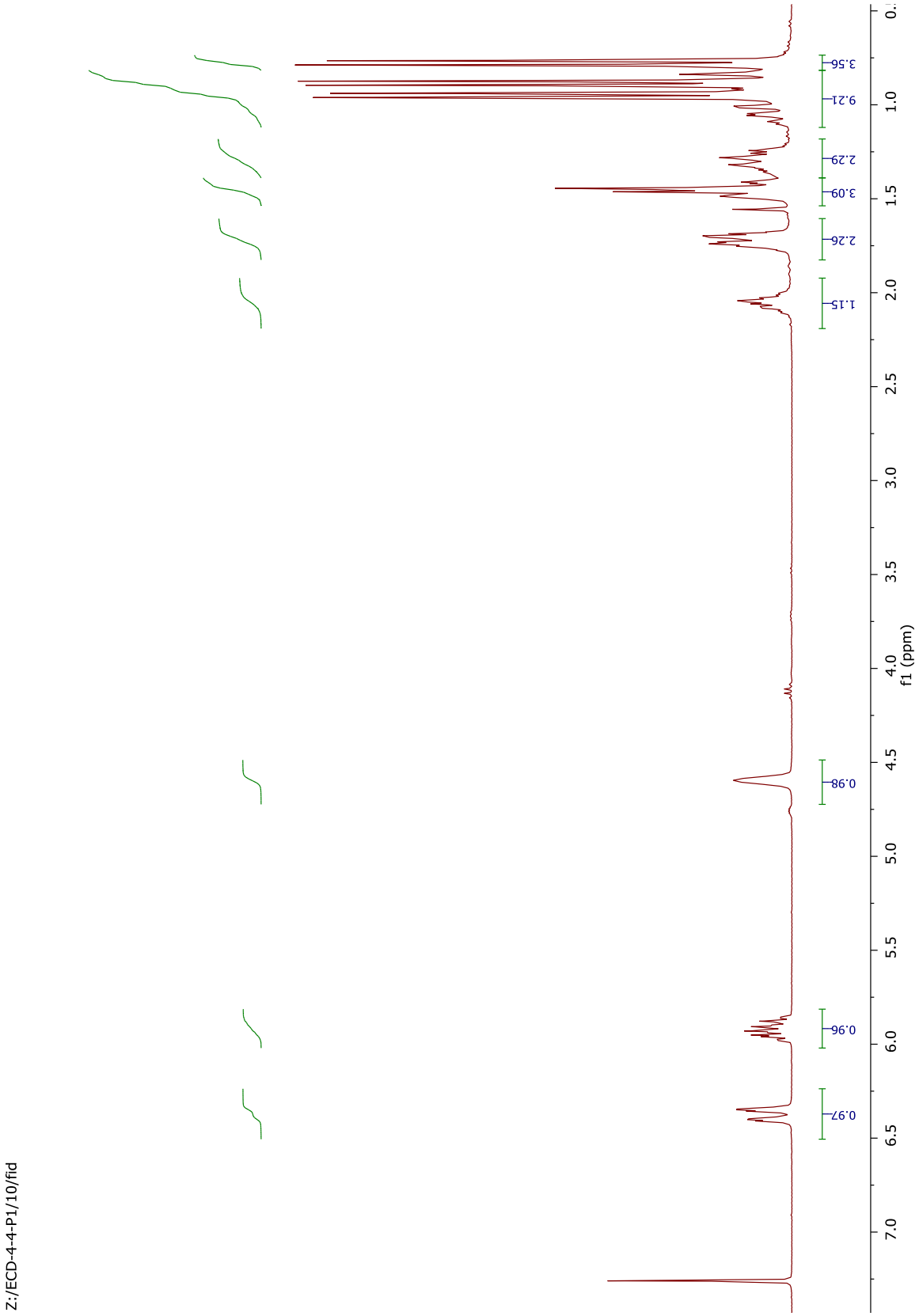

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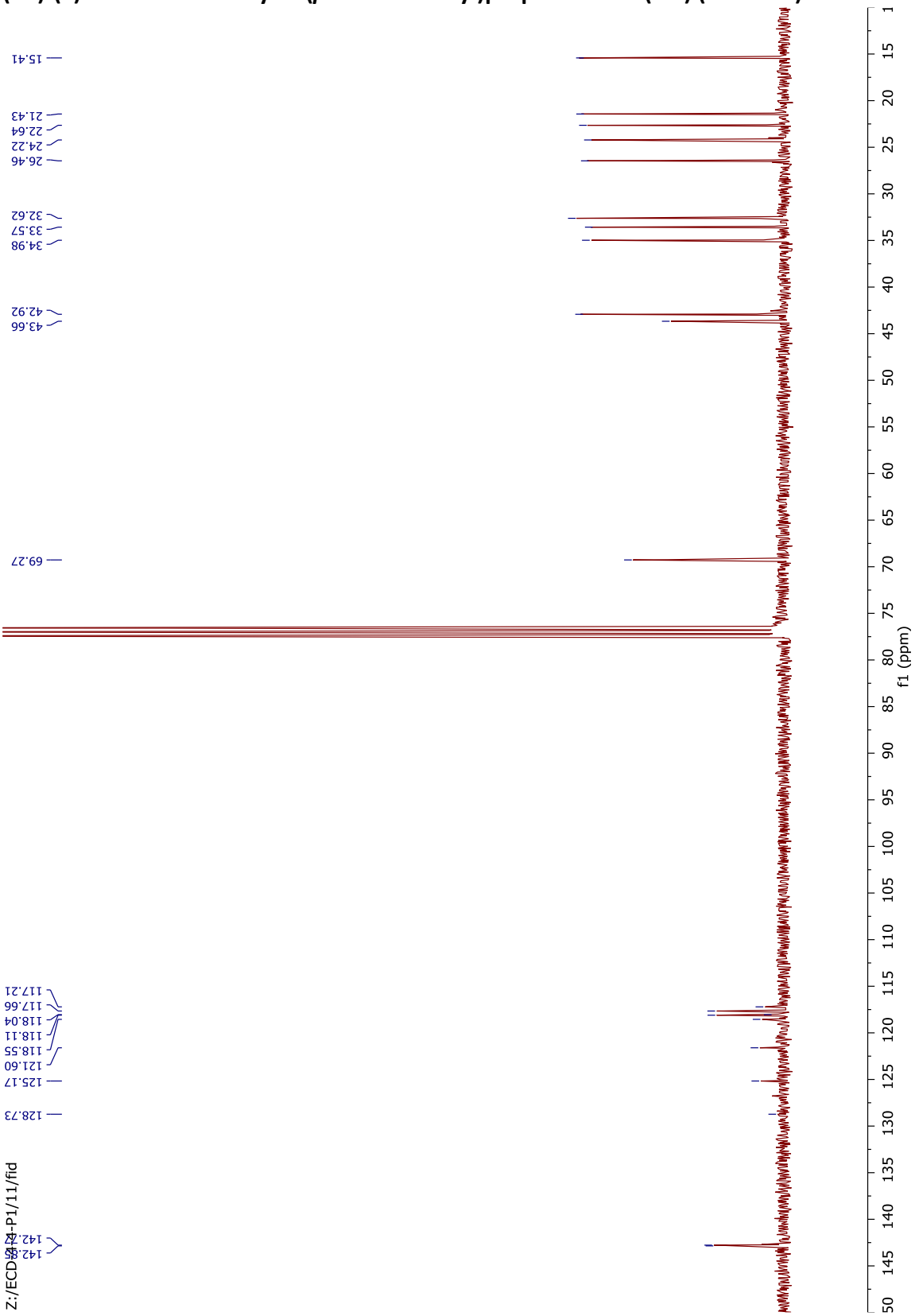
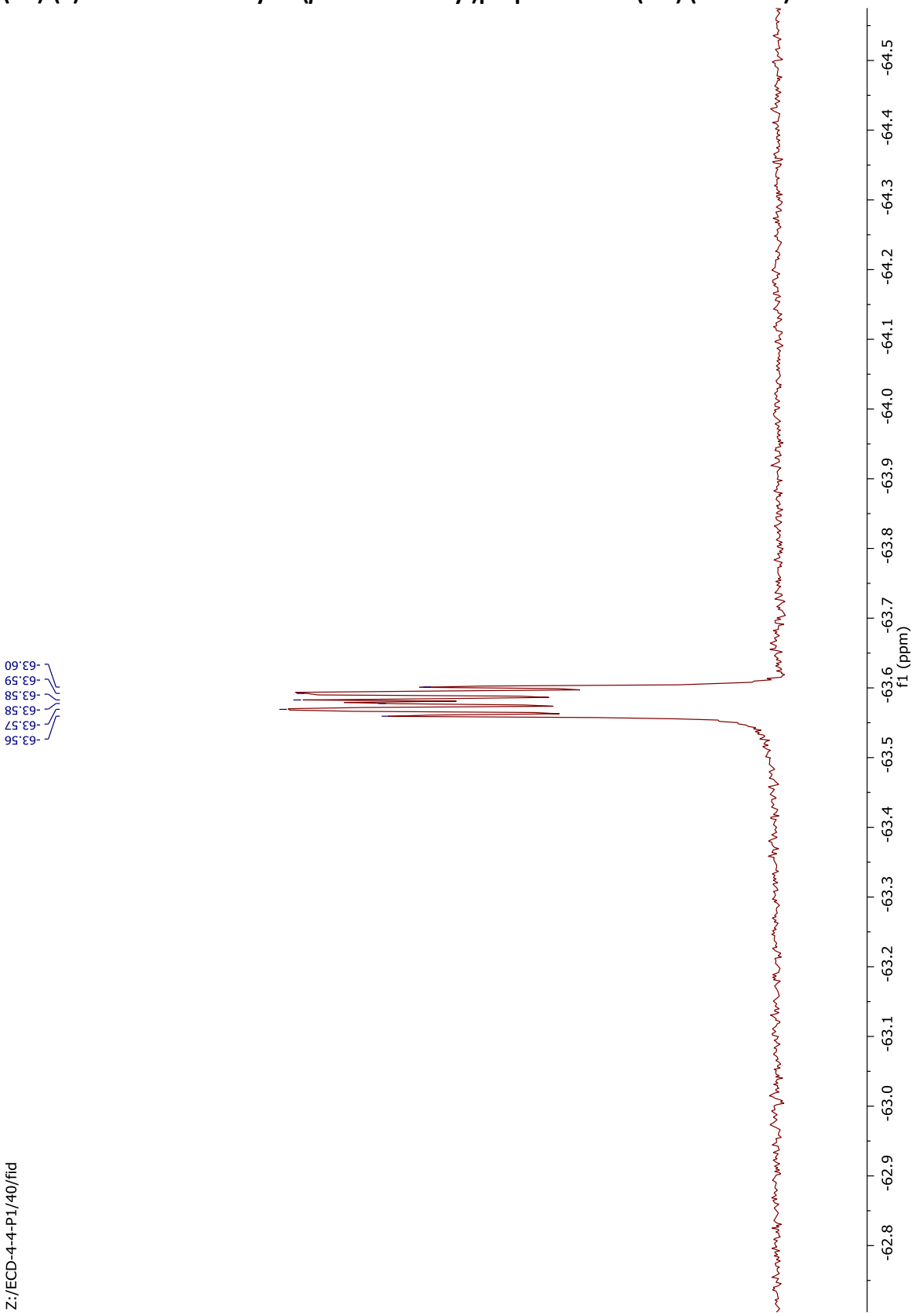
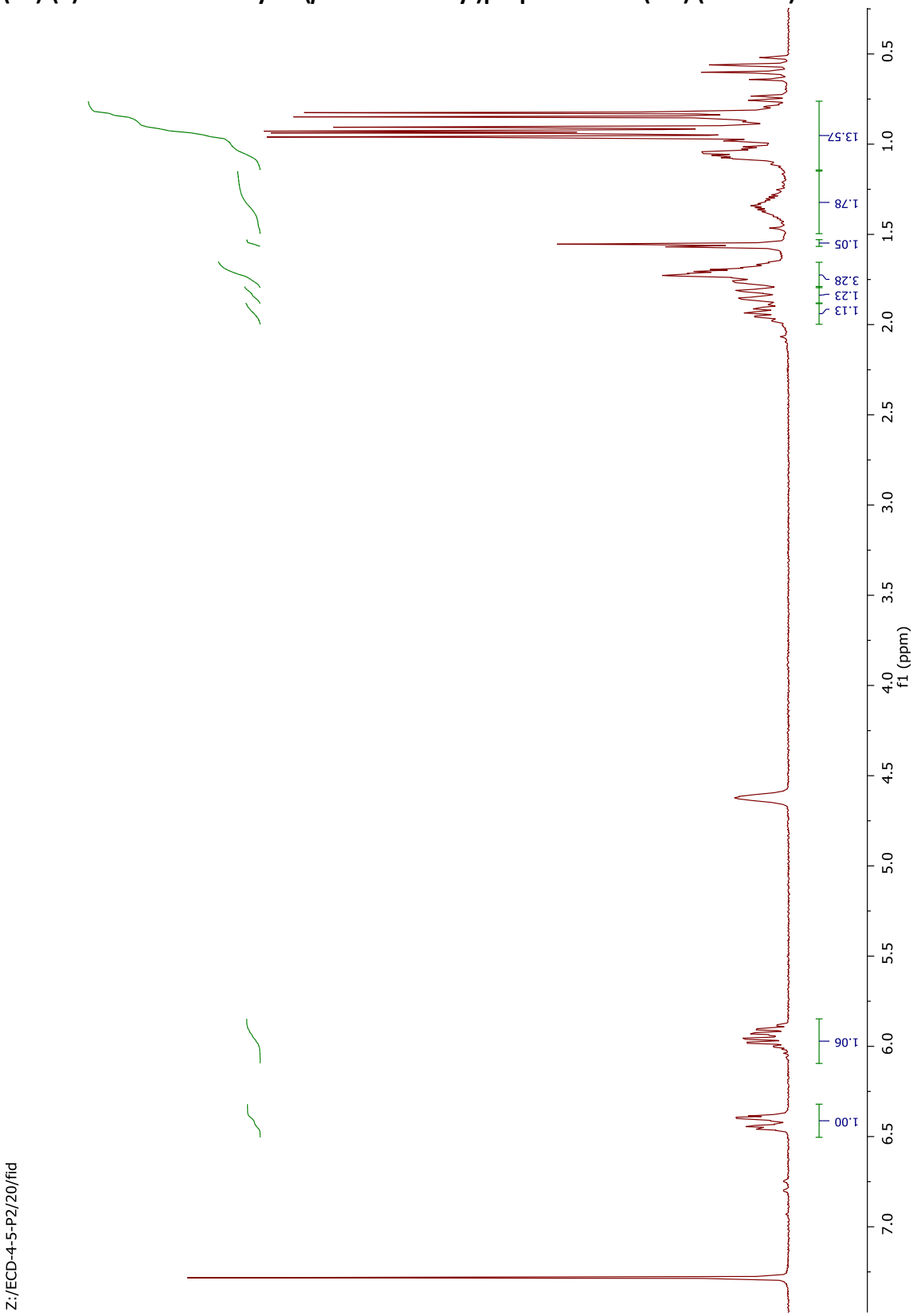


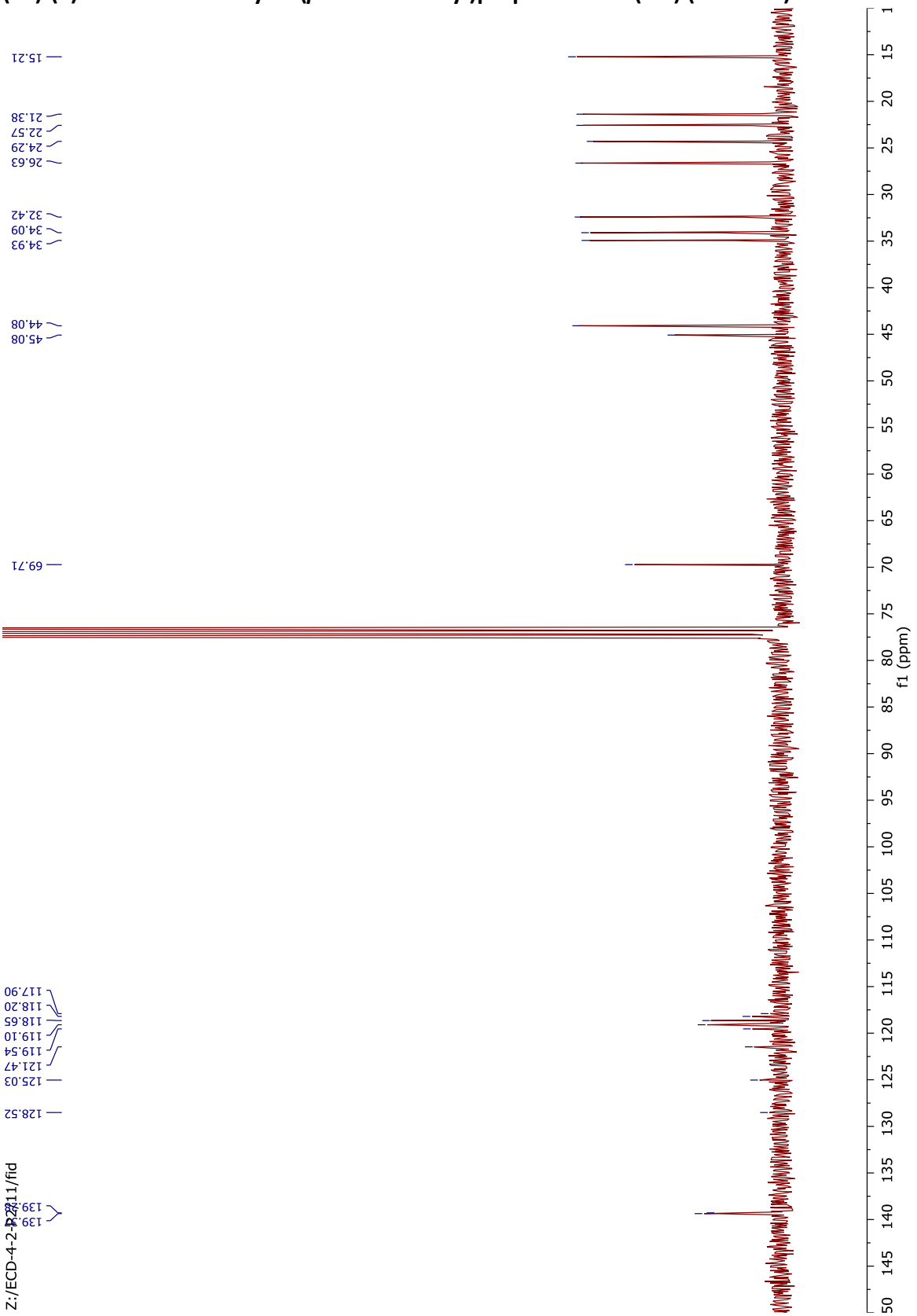
(1*R*)-(*E*)-1-Trifluoromethyl-4-(*p*-menthan-3-yl)prop-2-en-1-ol (16i) (^{19}F NMR)



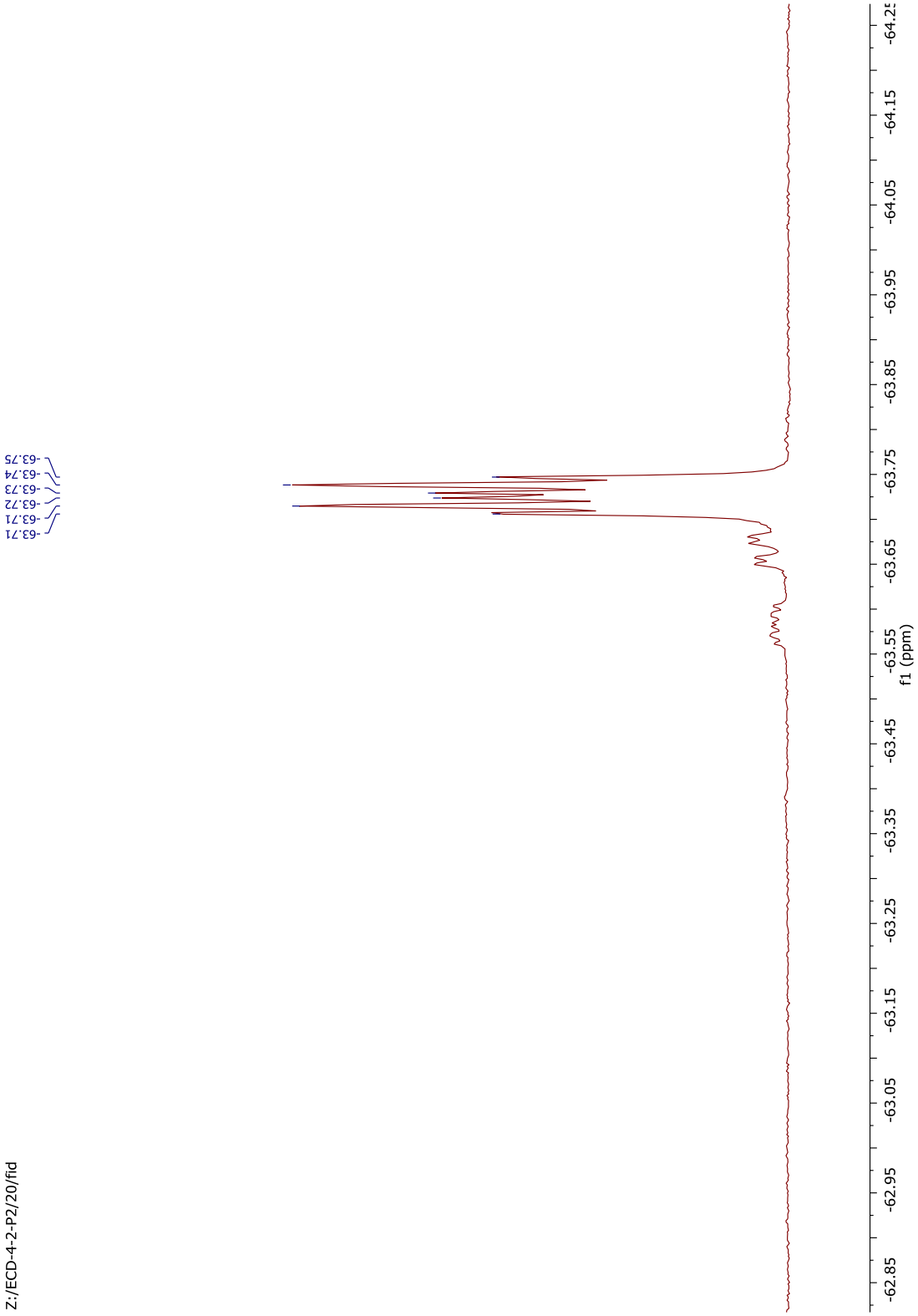
(1*S*)-(*E*)-1-Trifluoromethyl-4-(*p*-menthan-3-yl)prop-2-en-1-ol (19i) (^{1}H NMR)



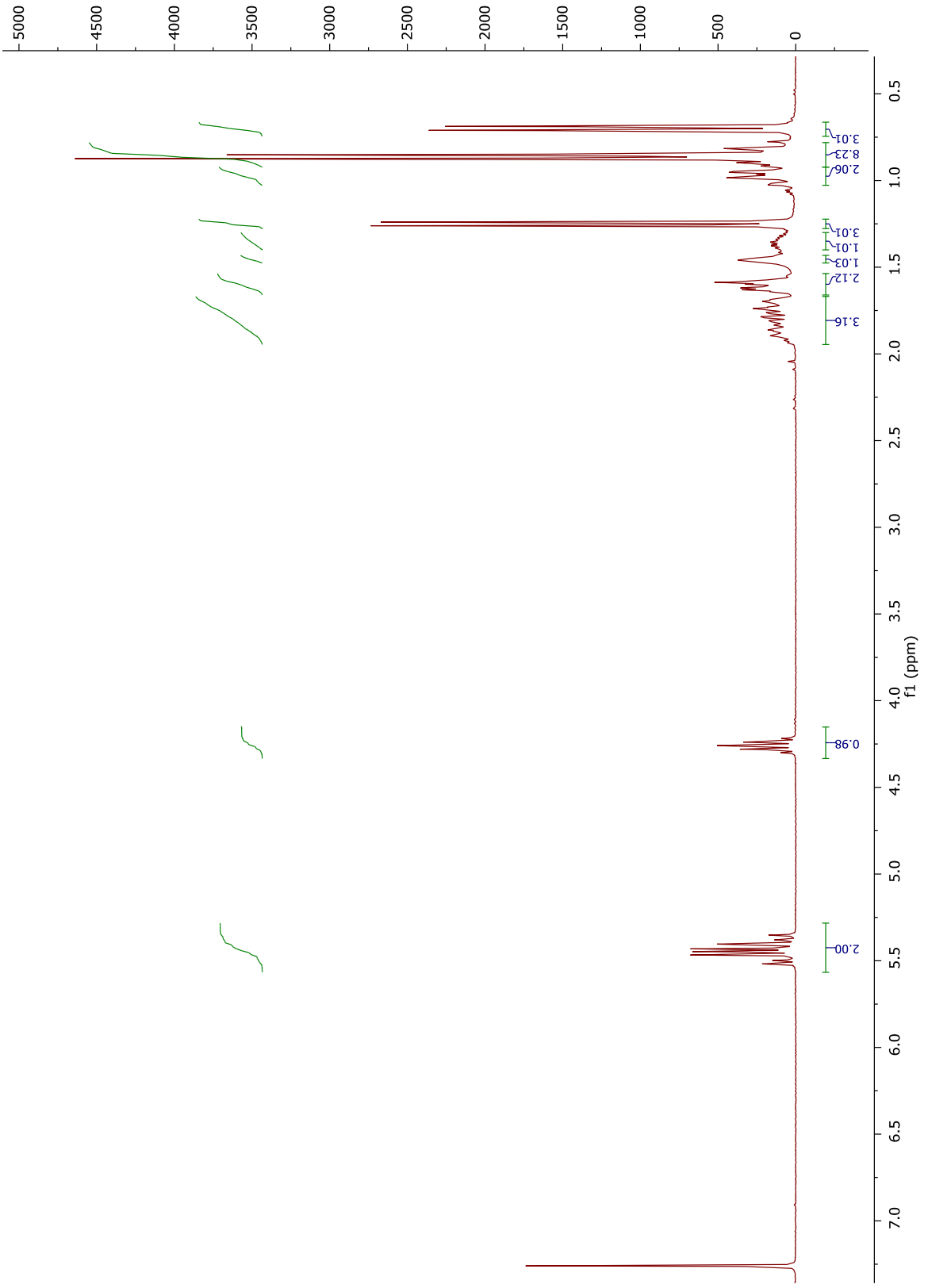
(1*S*)-(*E*)-1-Trifluoromethyl-4-(*p*-menthan-3-yl)prop-2-en-1-ol (19i) (^{13}C NMR)



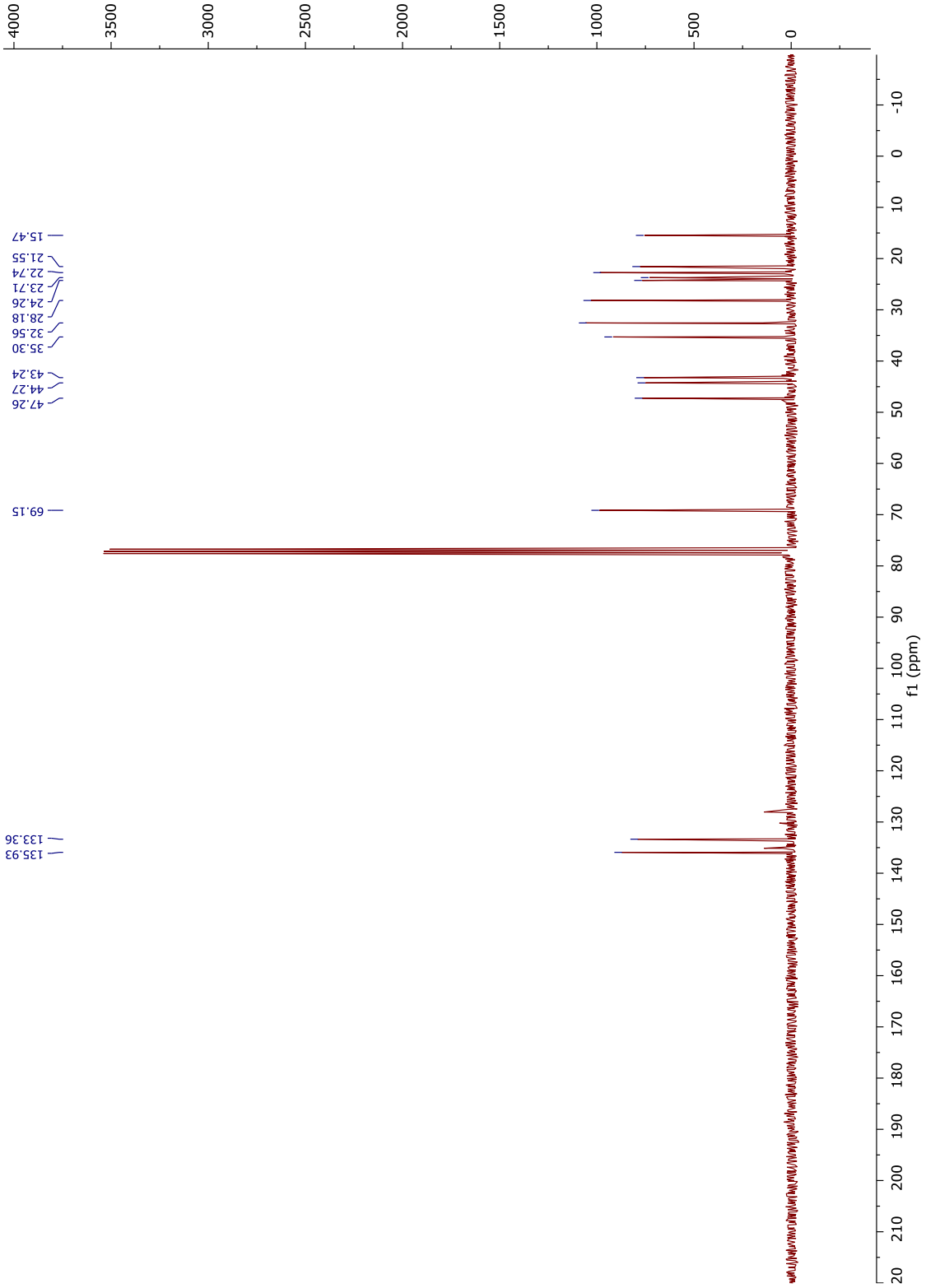
(1*S*)-(*E*)-1-Trifluoromethyl-4-(*p*-menthan-3-yl)prop-2-en-1-ol (19i) (^{19}F NMR)



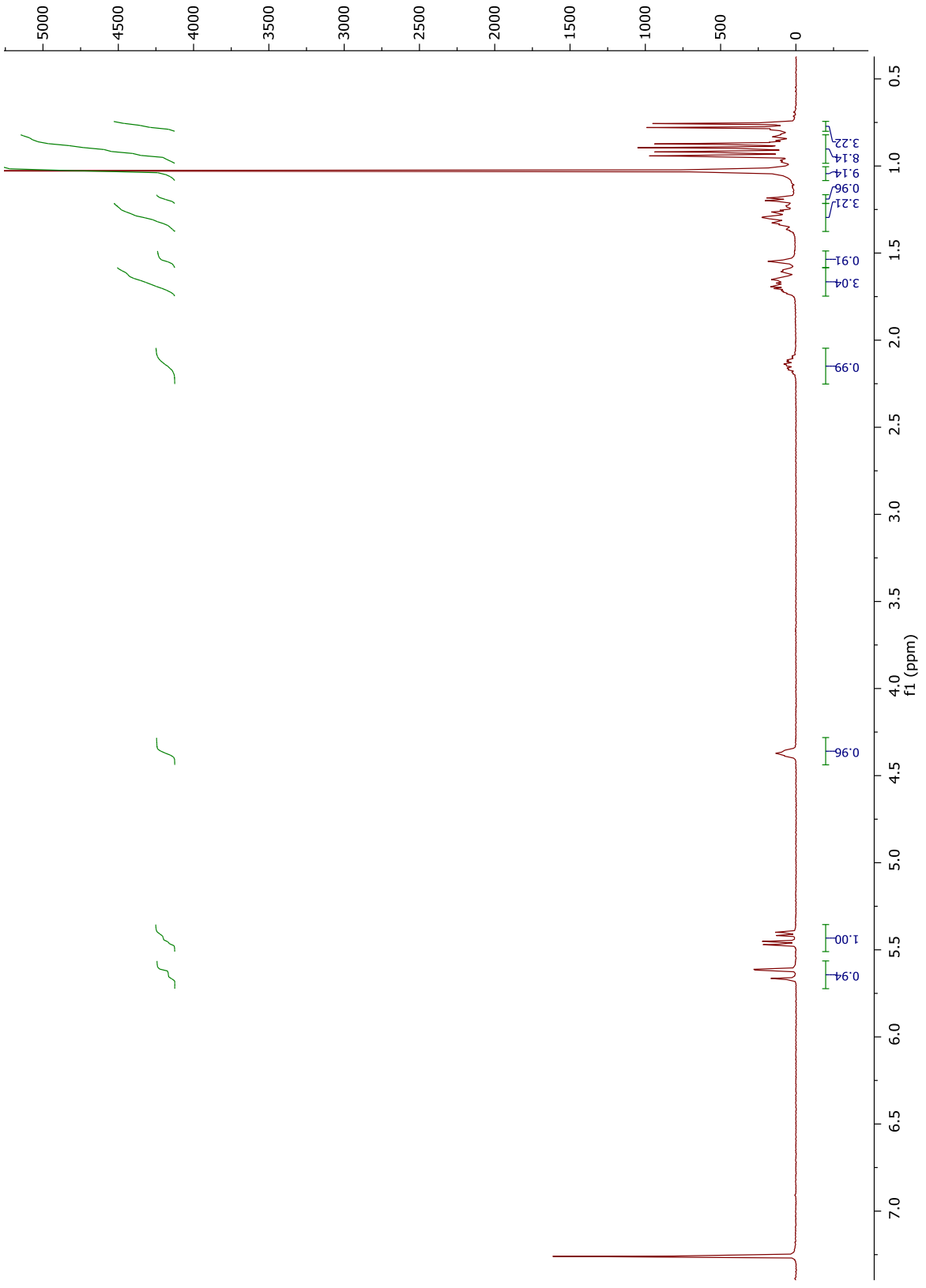
(2*R*)-(*E*)-4-(*p*-Menthan-3-yl)but-3-en-2-ol (17a) (^{1}H NMR)



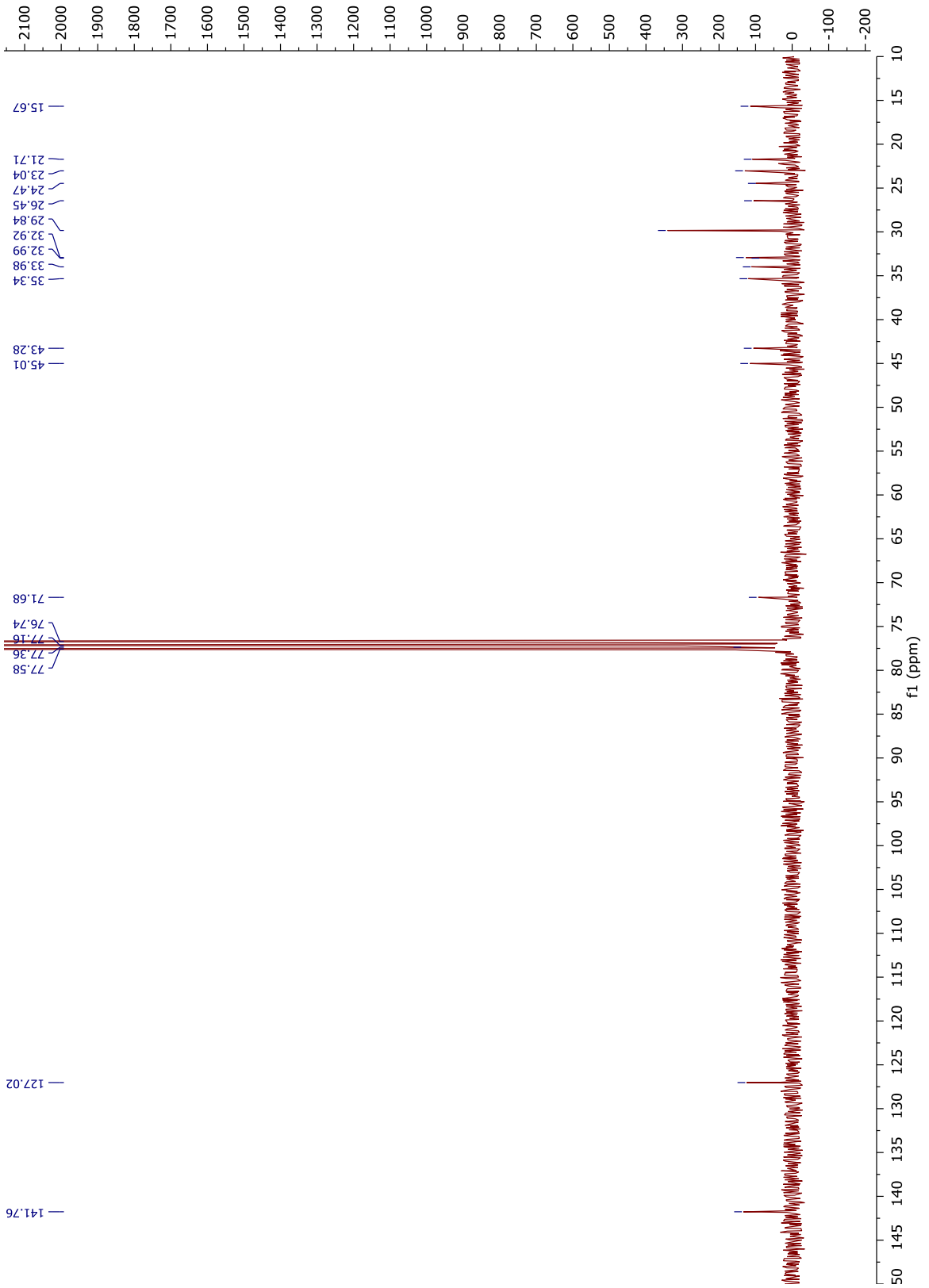
(2*R*)-(*E*)-4-(*p*-Menthan-3-yl)but-3-en-2-ol (17a) (^{13}C NMR)



(3*S*)-(*E*)-5-(*p*-Menthan-3-yl)-2,2-dimethylpent-4-en-3-ol (17b) (^{1}H NMR)

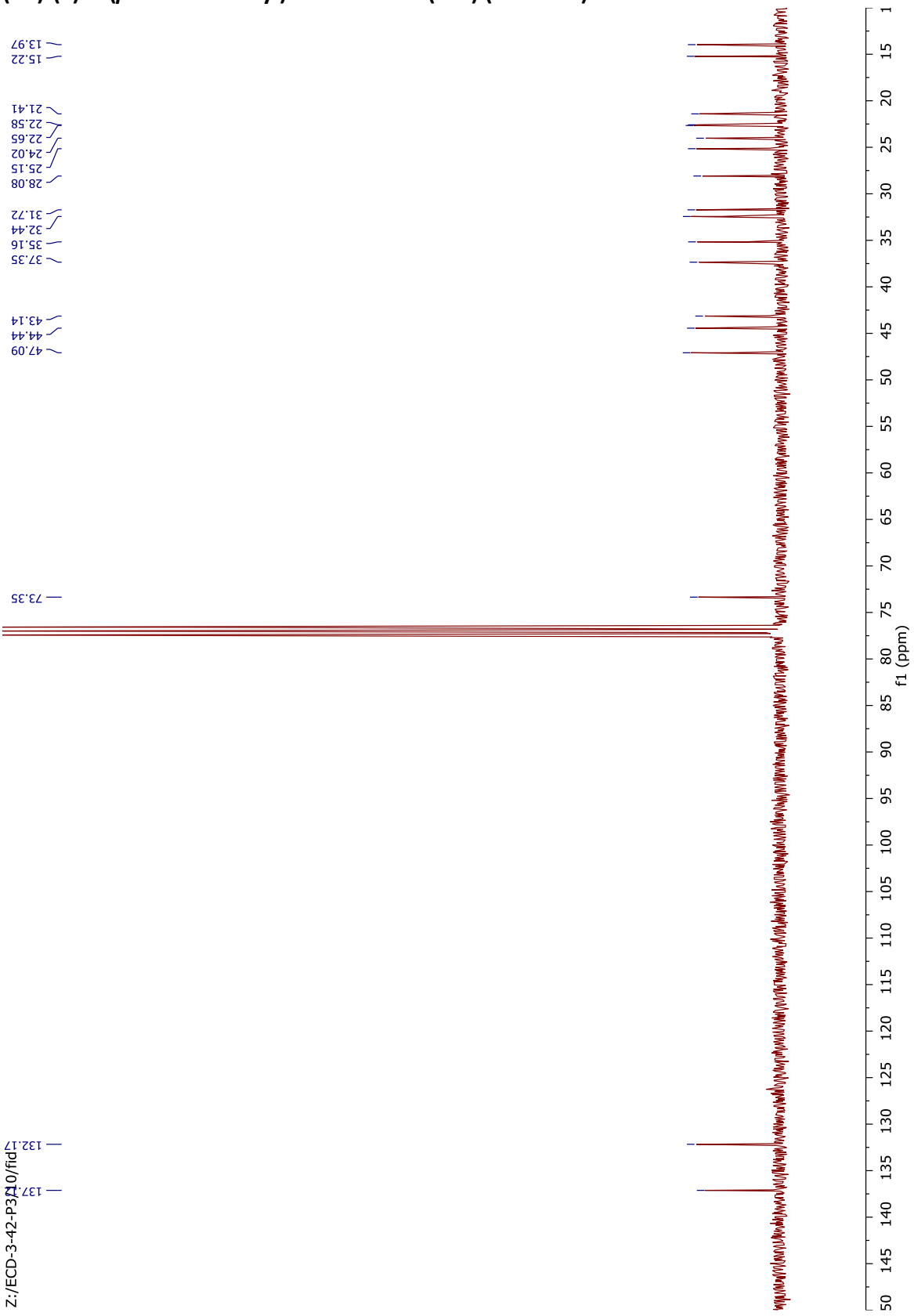


(3*S*)-(*E*)-5-(*p*-Menthan-3-yl)-2,2-dimethylpent-4-en-3-ol (17b) (^{13}C NMR)

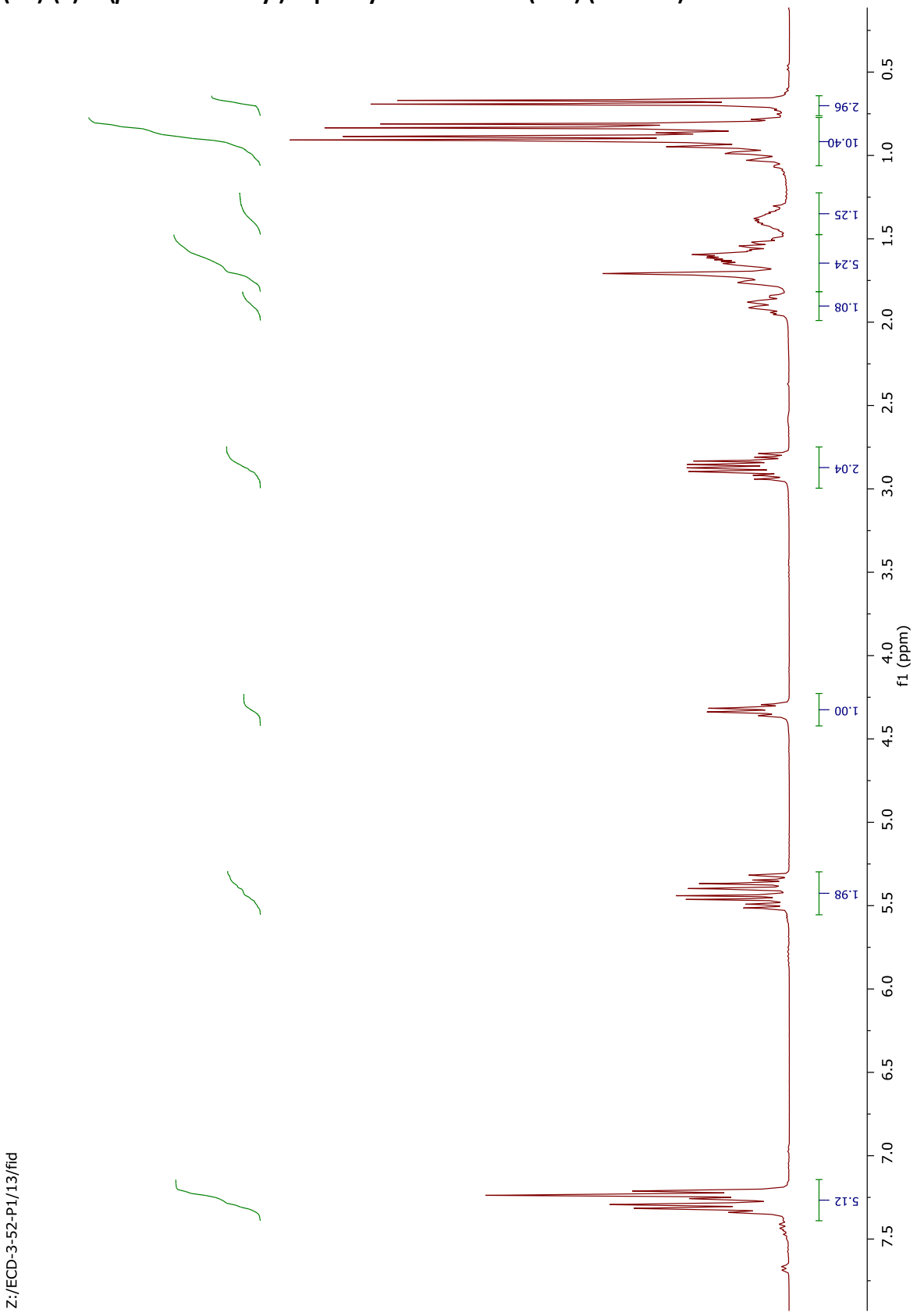


(6*R*)-(*E*)-8-(*p*-Menthan-3-yl)oct-7-en-6-ol (17c) (^{1}H NMR)

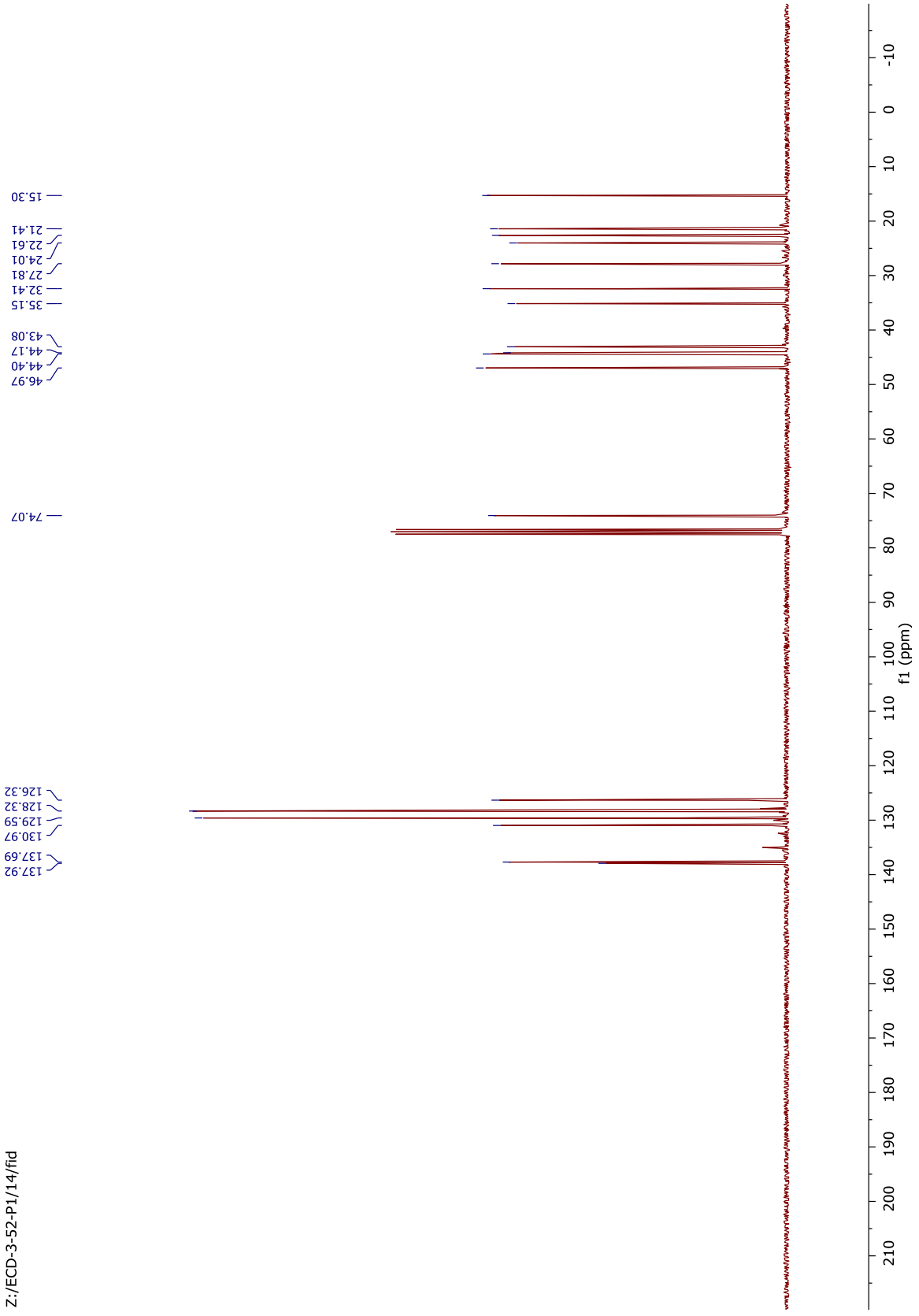
(6*R*)-(*E*)-8-(*p*-Menthan-3-yl)oct-7-en-6-ol (17c) (^{13}C NMR)



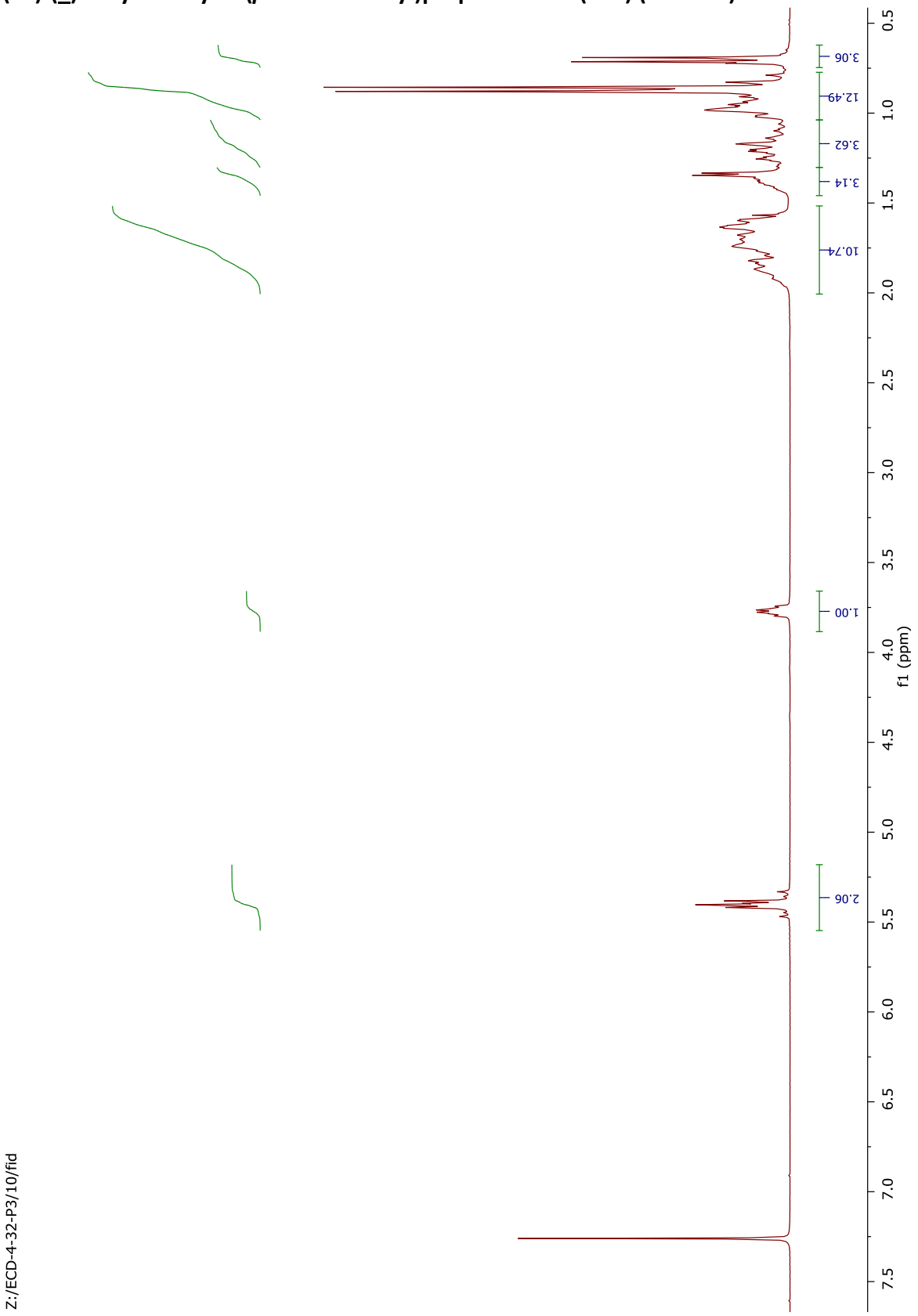
(2*R*)-(*E*)-4-(*p*-Menthan-3-yl)-1-phenylbut-3-en-2-ol (17d) (^{1}H NMR)



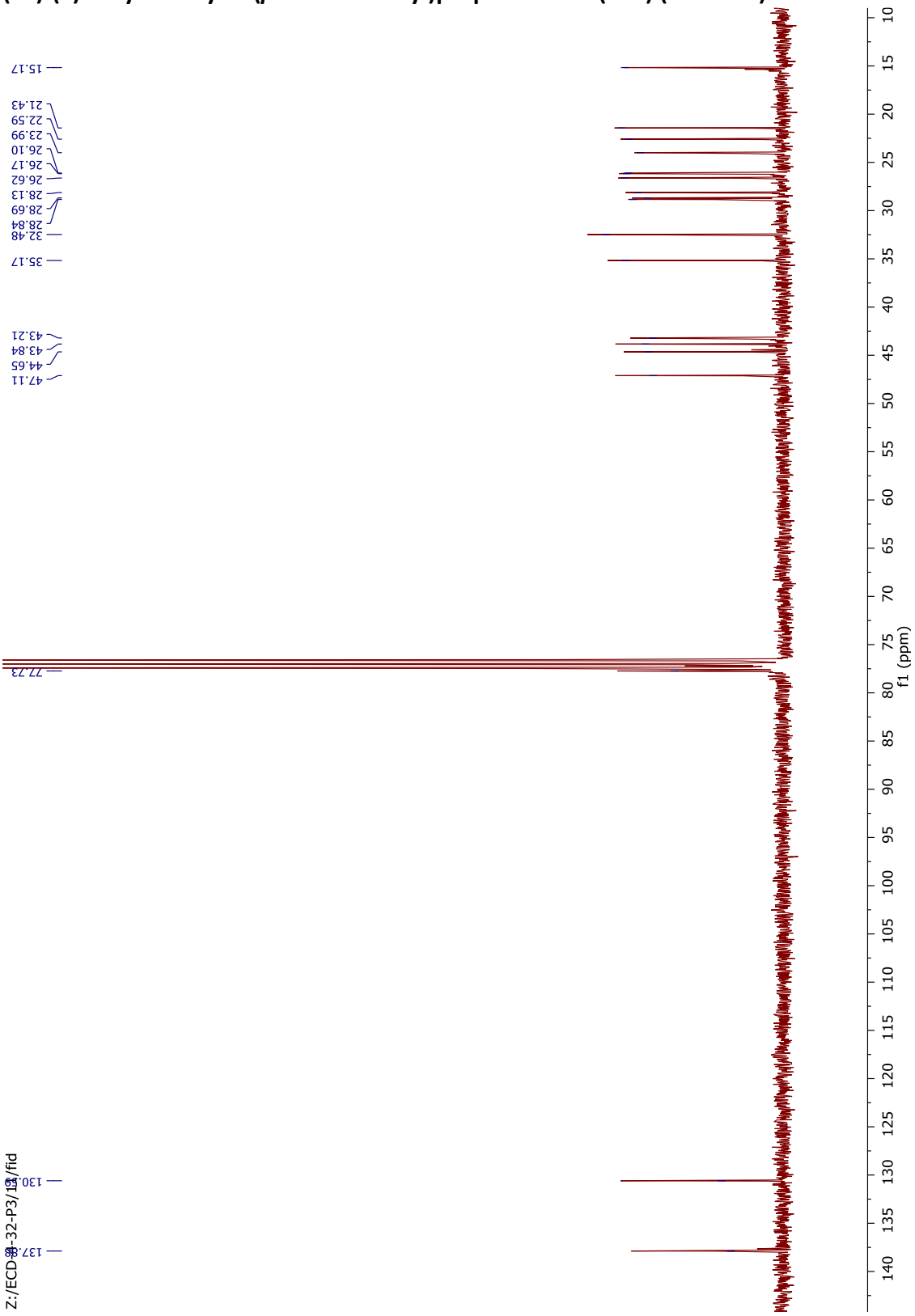
(2*R*)-(*E*)-4-(*p*-Menthan-3-yl)-1-phenylbut-3-en-2-ol (17d) (^{13}C NMR)



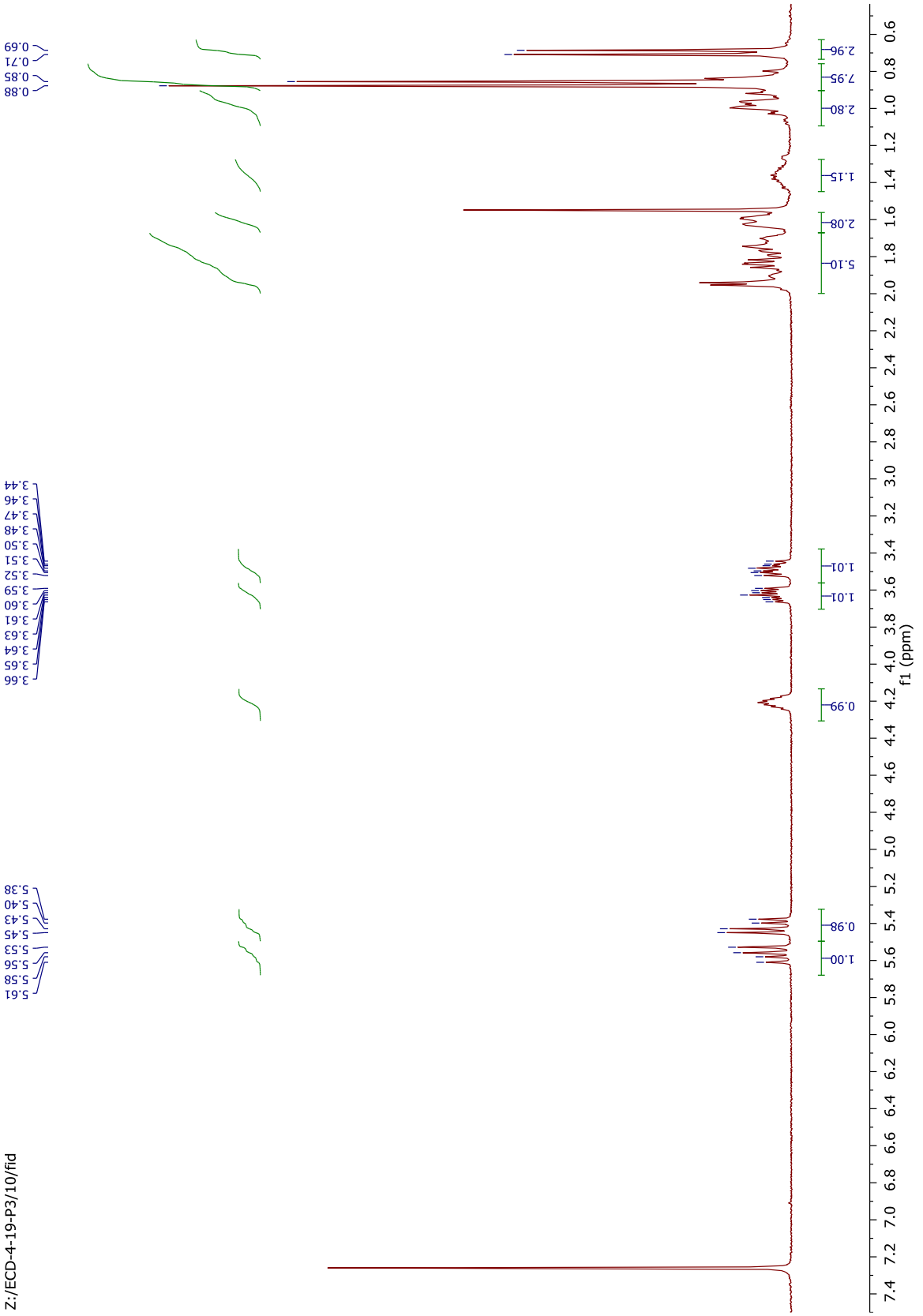
(1*S*)-(*E*)-1-Cyclohexyl-3-(*p*-menthan-3-yl)prop-2-en-1-ol (17e) (^{1}H NMR)



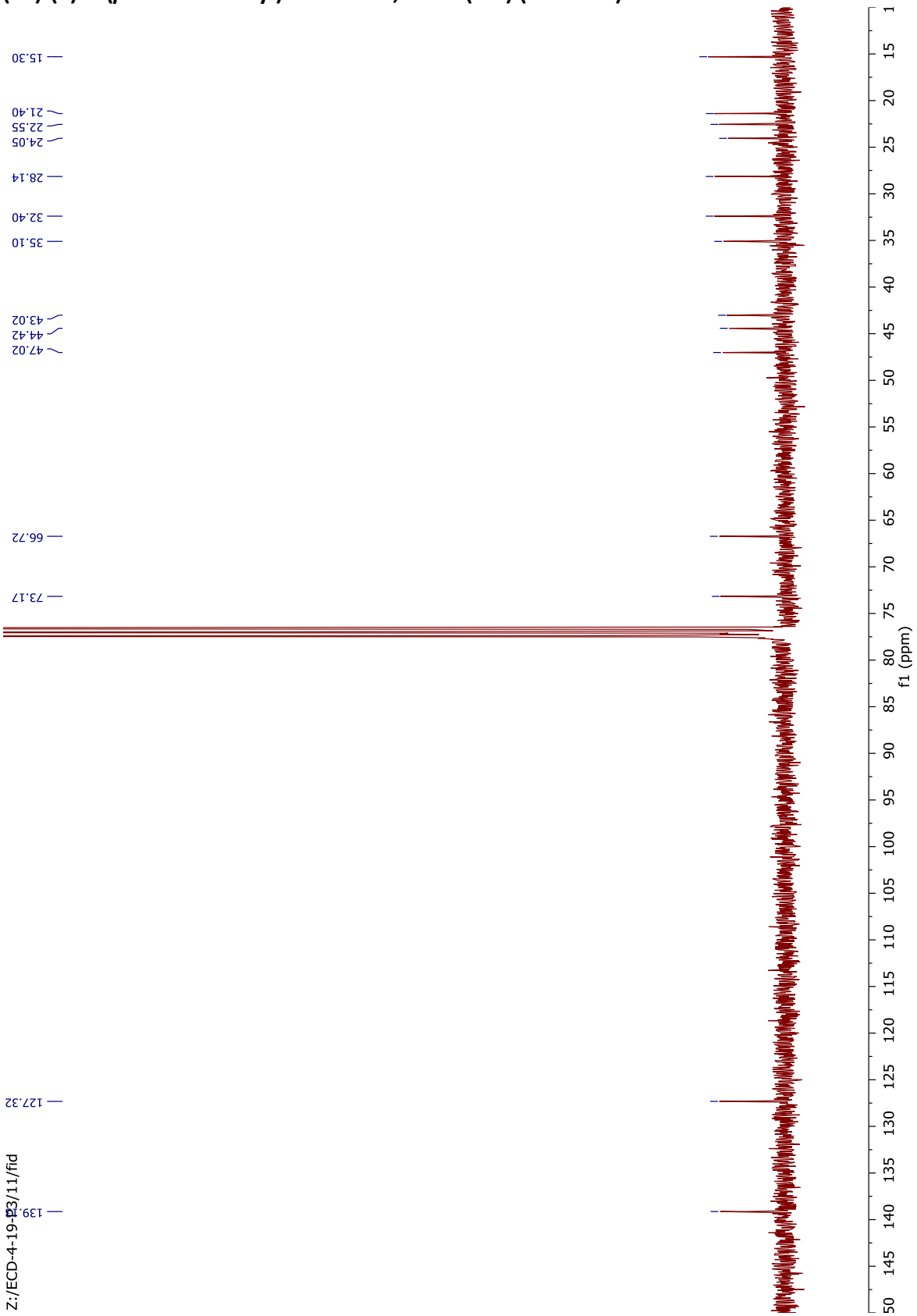
(1*S*)-(*E*)-1-Cyclohexyl-3-(*p*-menthan-3-yl)prop-2-en-1-ol (17e) (^{13}C NMR)



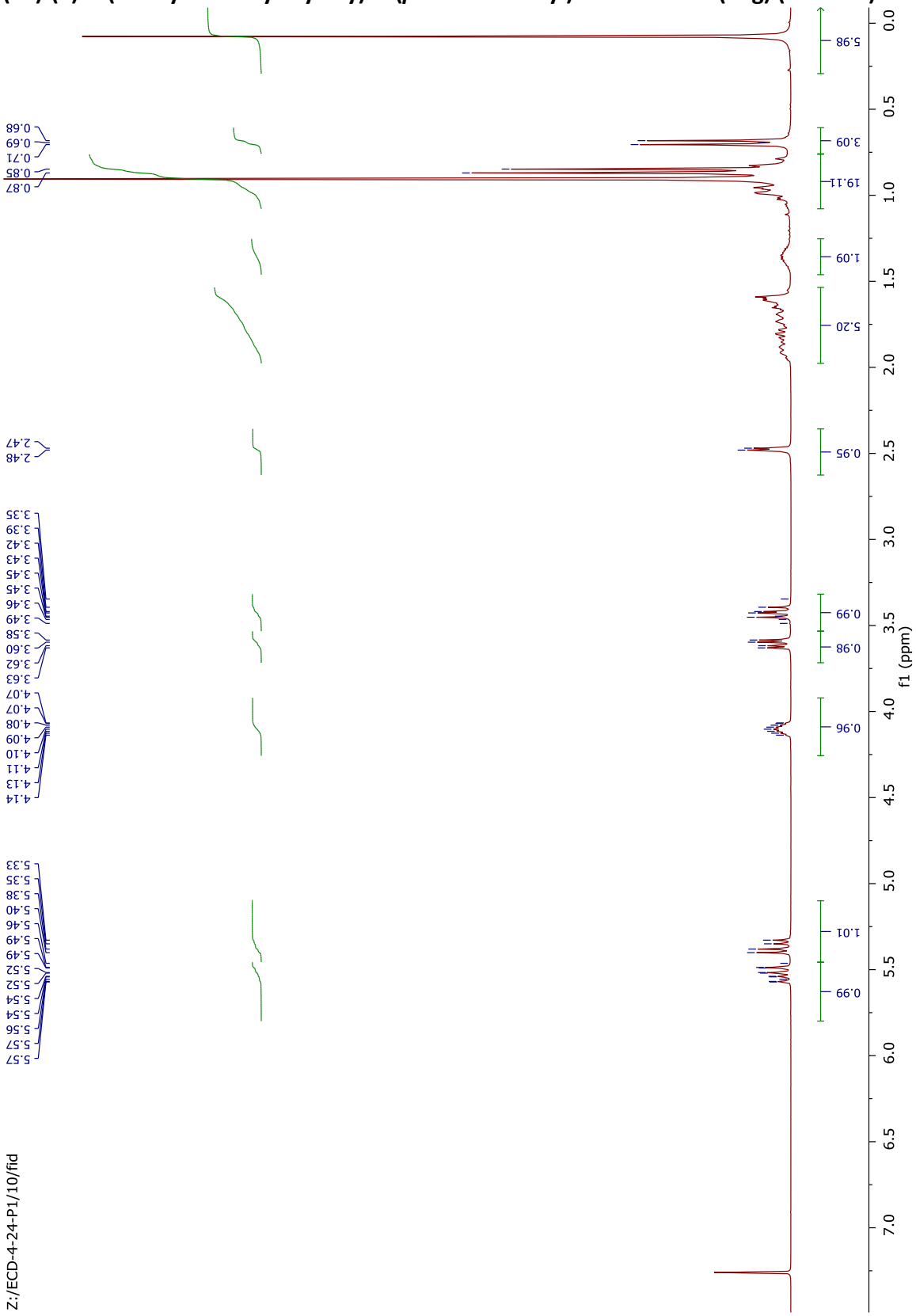
(2*S*)-(*E*)-4-(*p*-Menthan-3-yl)but-3-en-1,2-diol (17f) (^{1}H NMR)



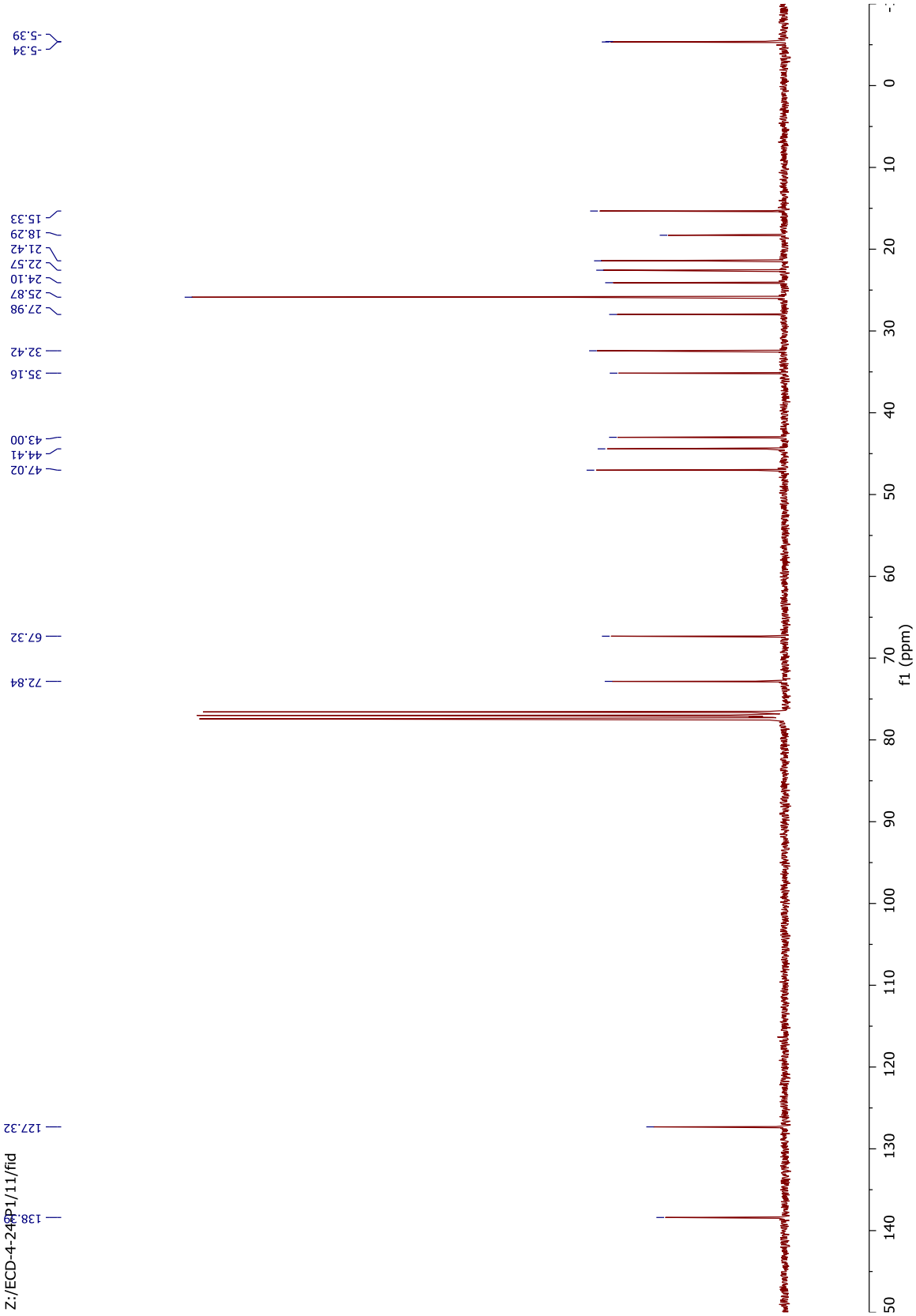
(2*S*)-(*E*)-4-(*p*-Menthan-3-yl)but-3-en-1,2-diol (17f) (^{13}C NMR)



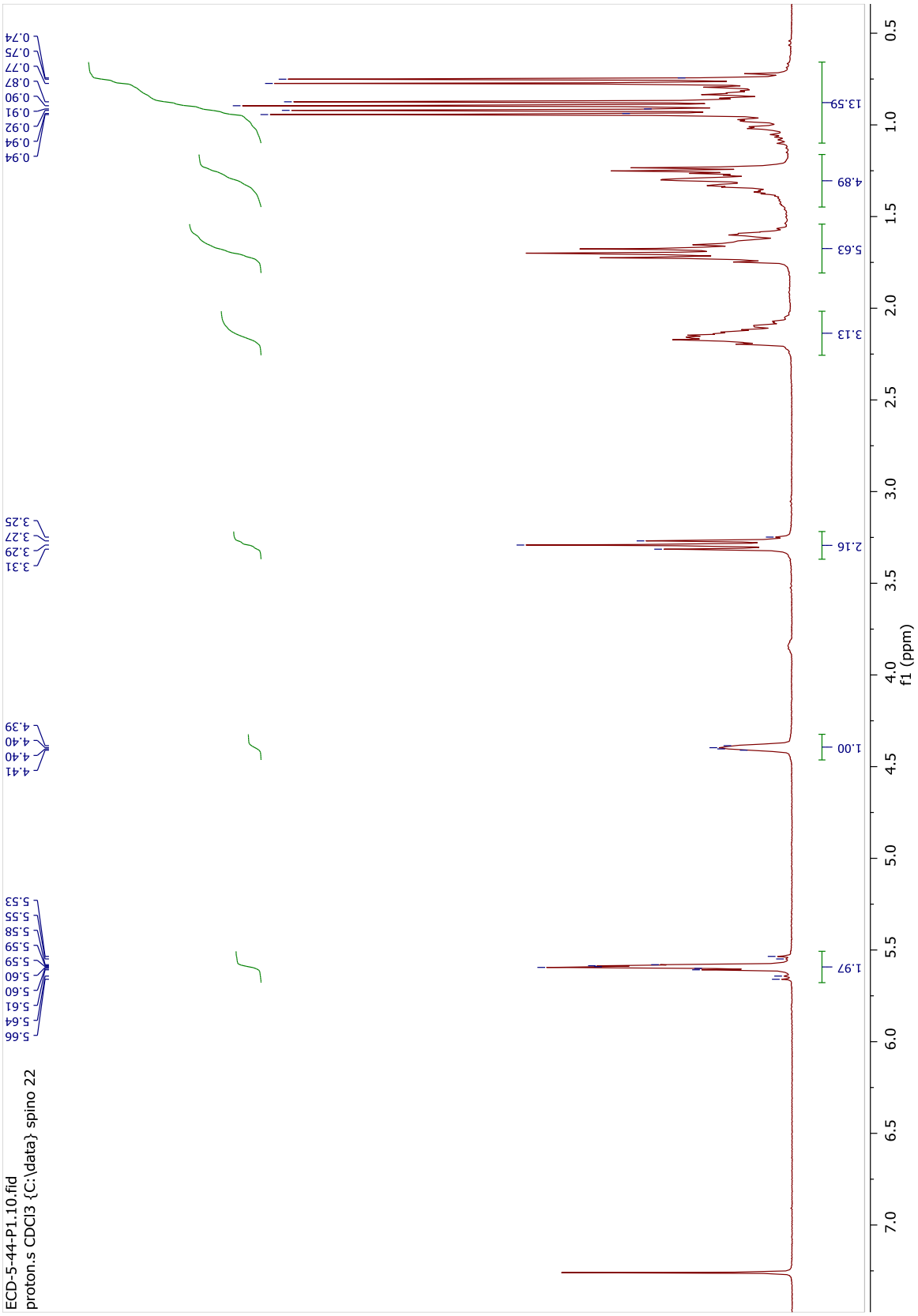
(2*S*)-(*E*)-1-(*t*-Butyldimethylsilyloxy)-4-(*p*-menthan-3-yl)but-3-en-2-ol (17g) (^{1}H NMR)



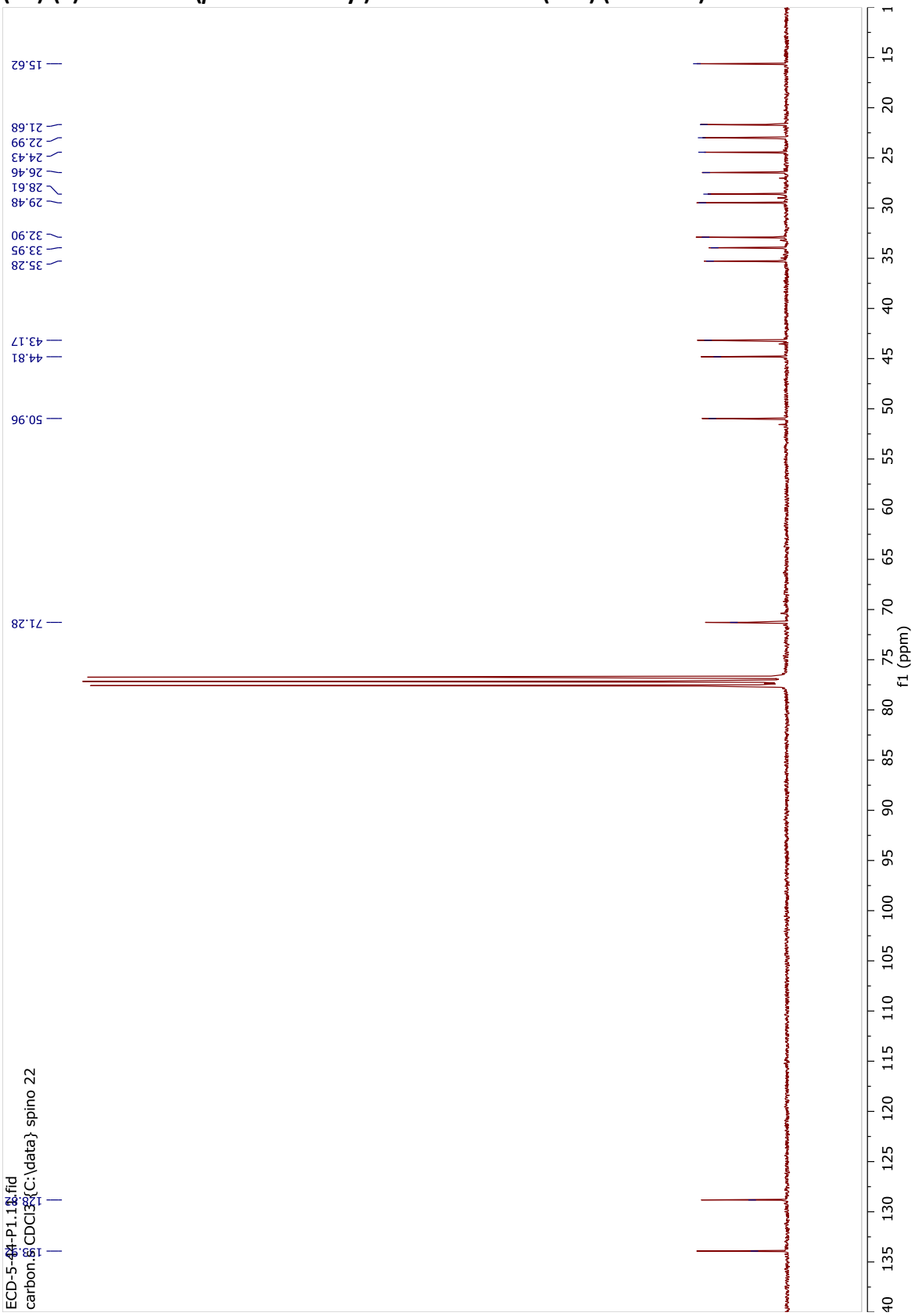
(2*S*)-(*E*)-1-(*t*-Butyldimethylsilyloxy)-4-(*p*-menthan-3-yl)but-3-en-2-ol (17g) (^{13}C NMR)



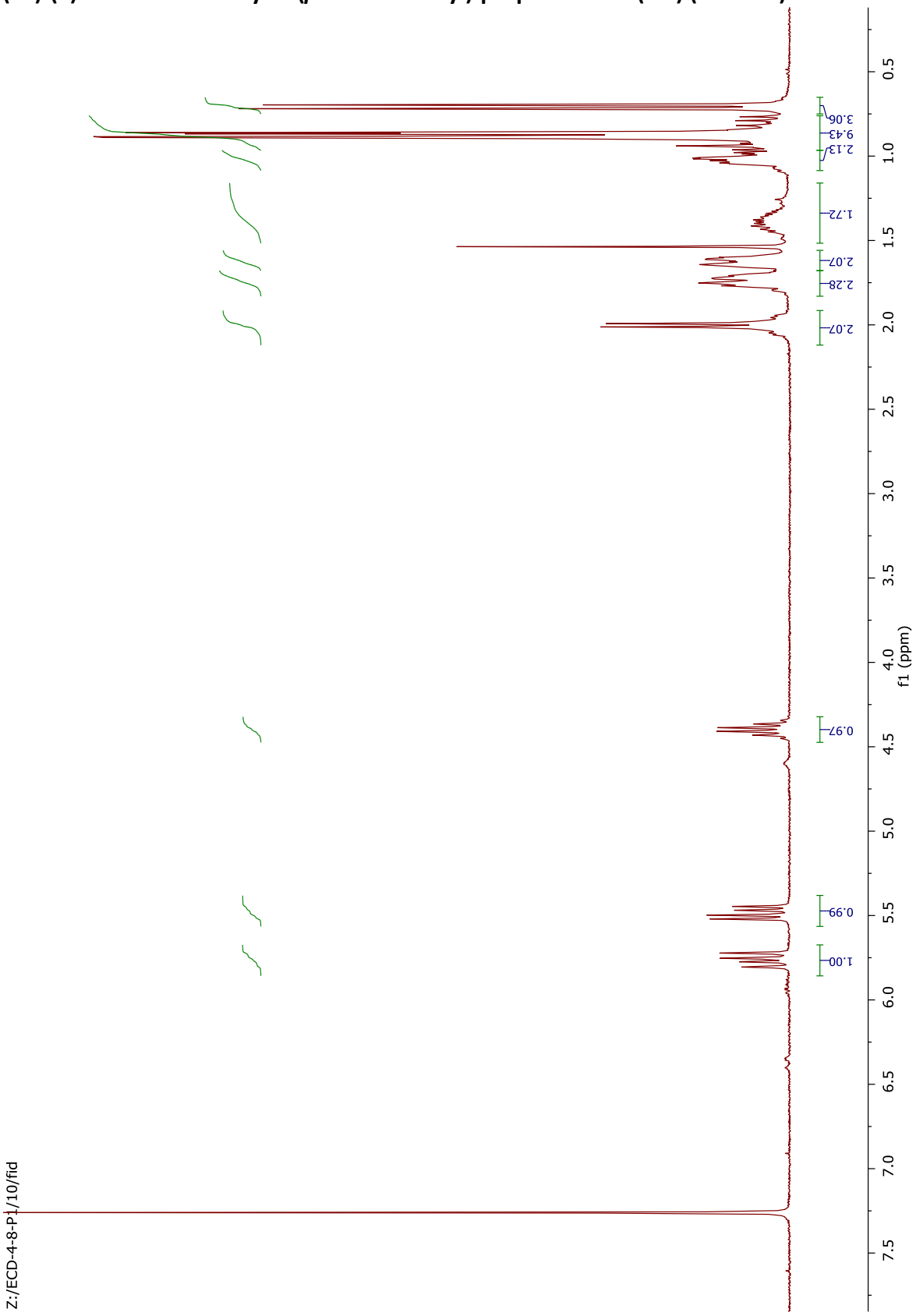
(4*R*)-(*E*)-1-Azido-6-(*p*-menthan-3-yl)hexen-5-en-4-ol (17h) (^{1}H NMR)



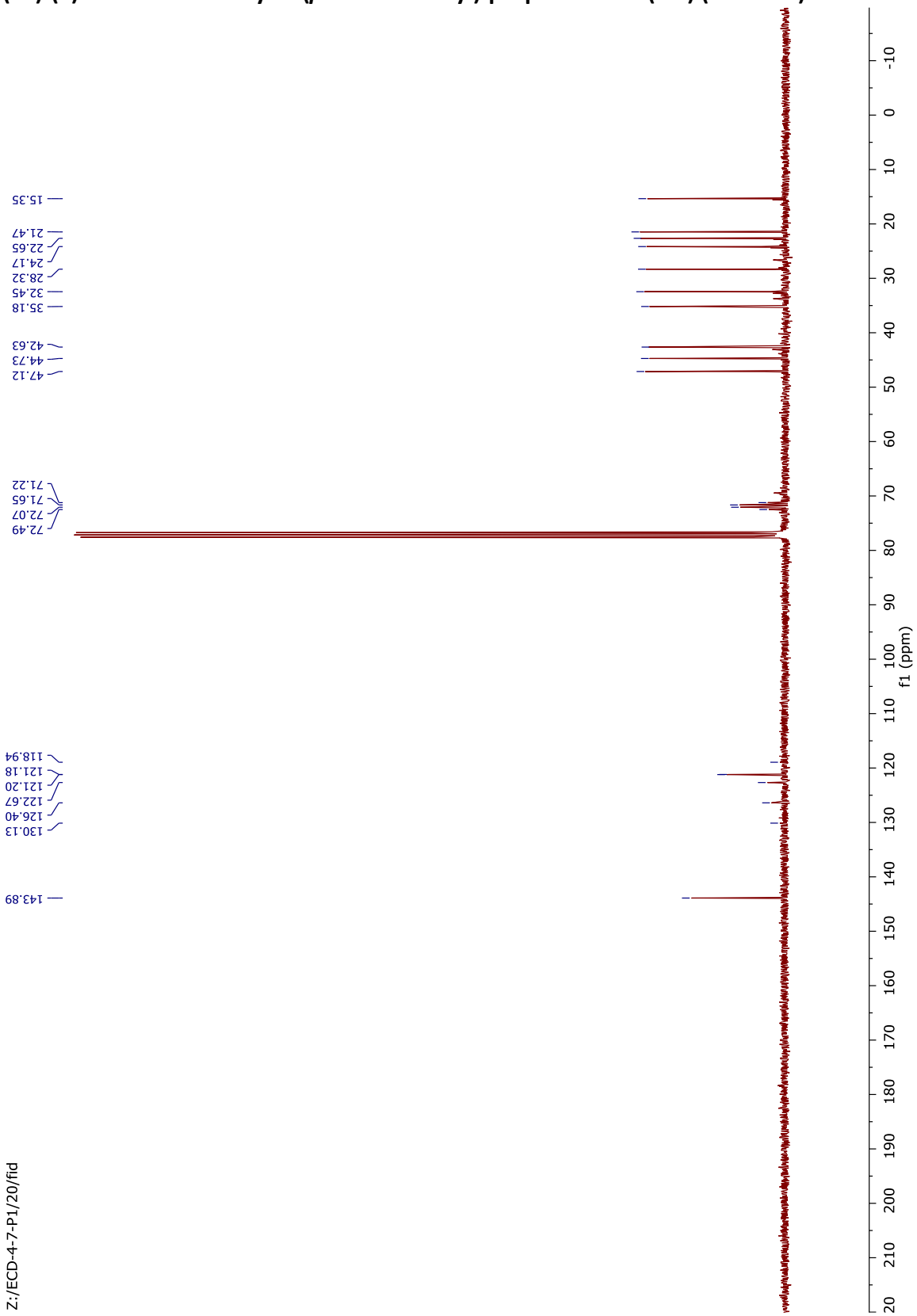
(4*R*)-(*E*)-1-Azido-6-(*p*-menthan-3-yl)hexen-5-en-4-ol (17h) (^{13}C NMR)



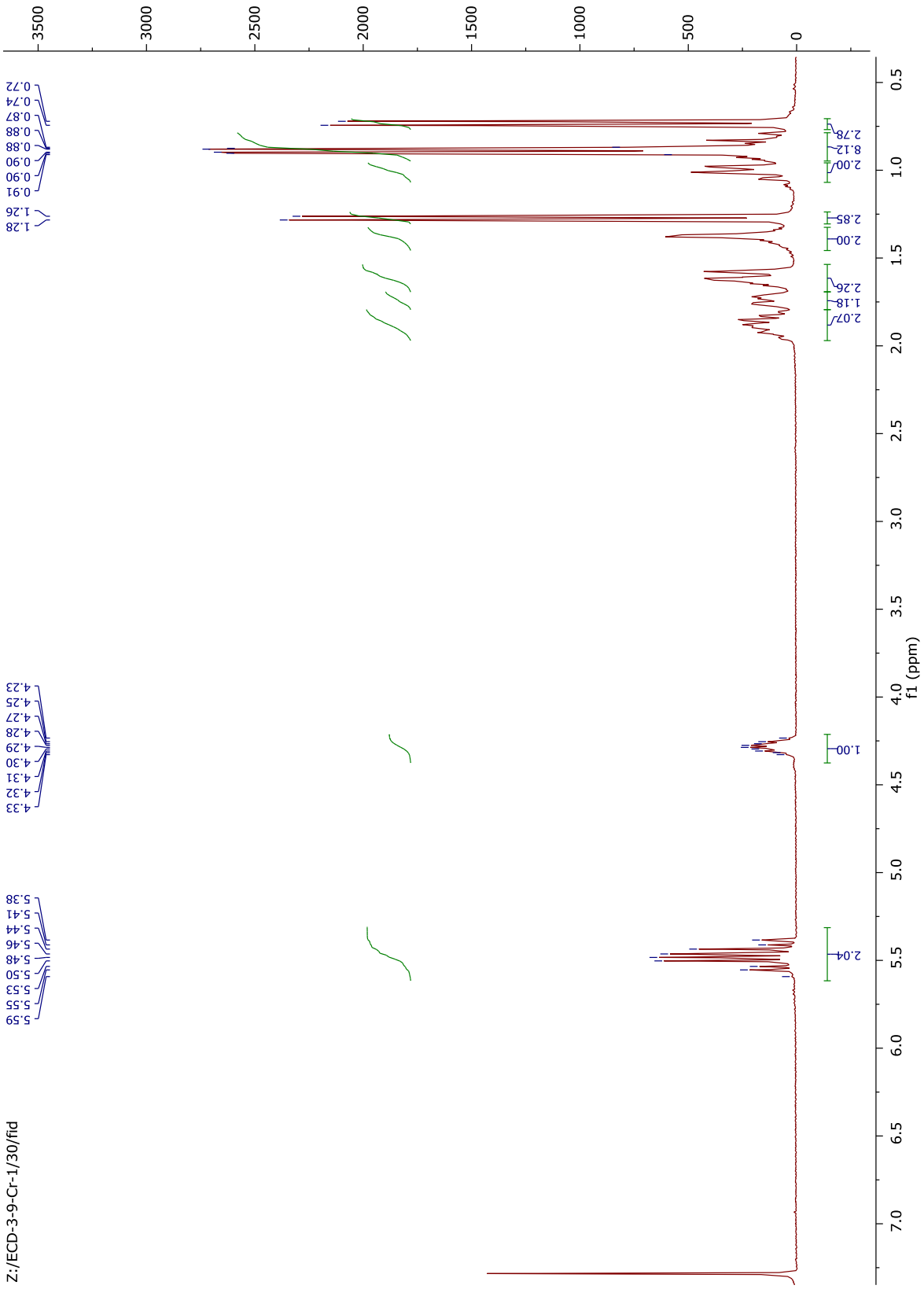
(1*S*)-(*E*)-1-Trifluoromethyl-4-(*p*-menthan-3-yl)-prop-2-en-1-ol (17i) (^{1}H NMR)



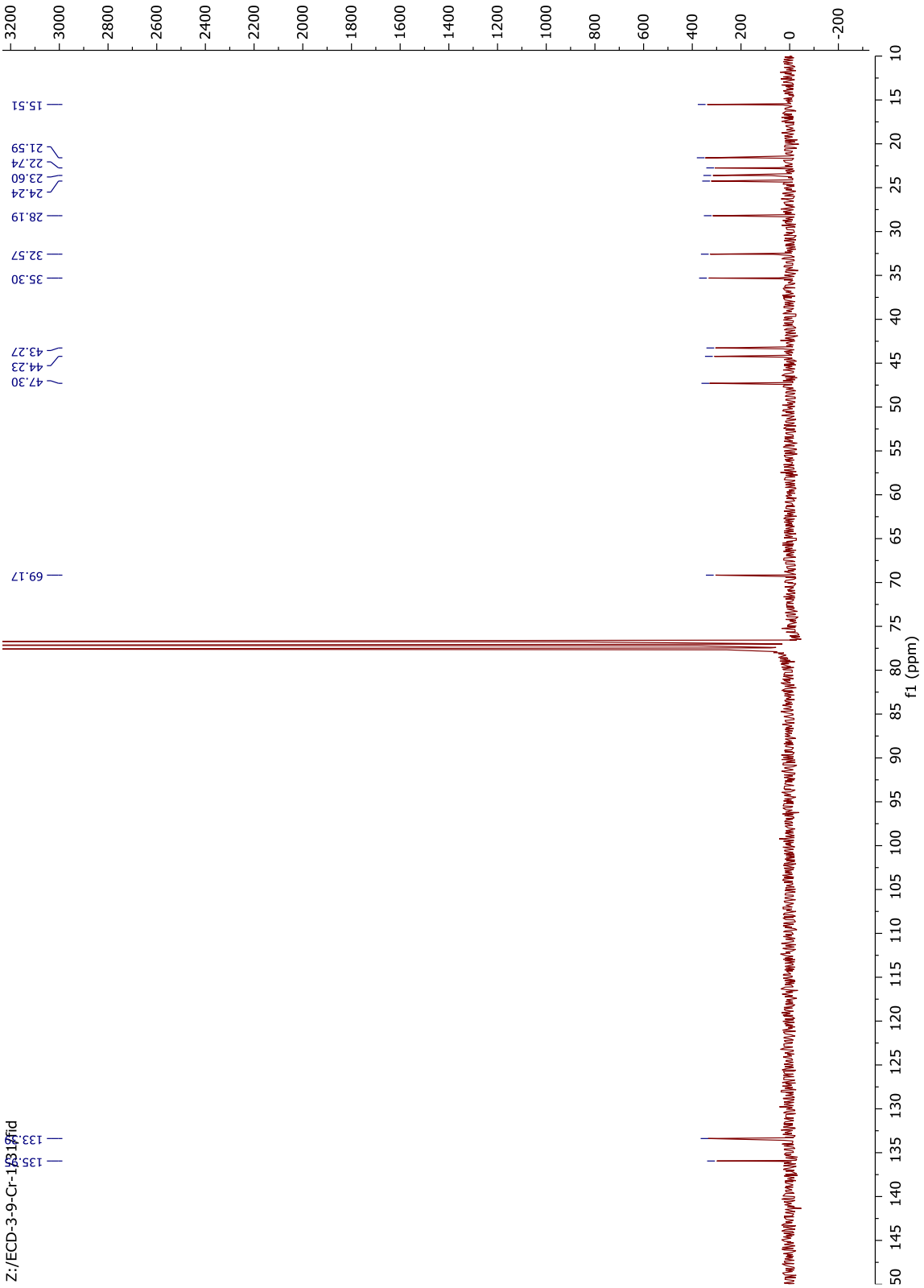
(1*S*)-(*E*)-1-Trifluoromethyl-4-(*p*-menthan-3-yl)-prop-2-en-1-ol (17i) (^{13}C NMR)



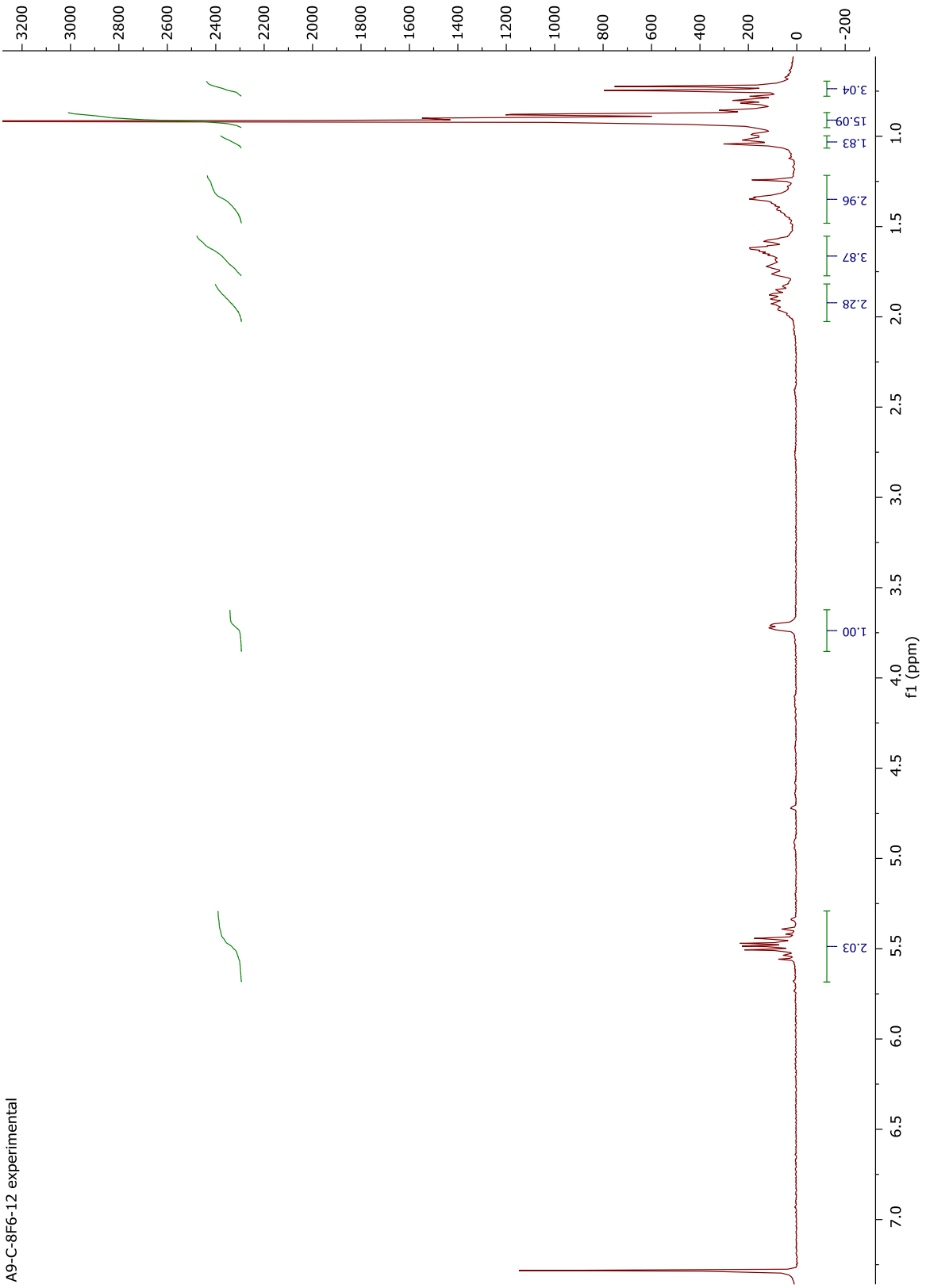
(2*S*)-(*E*)-4-(*p*-Menthan-3-yl)but-3-en-2-ol (18a) (^{1}H NMR)



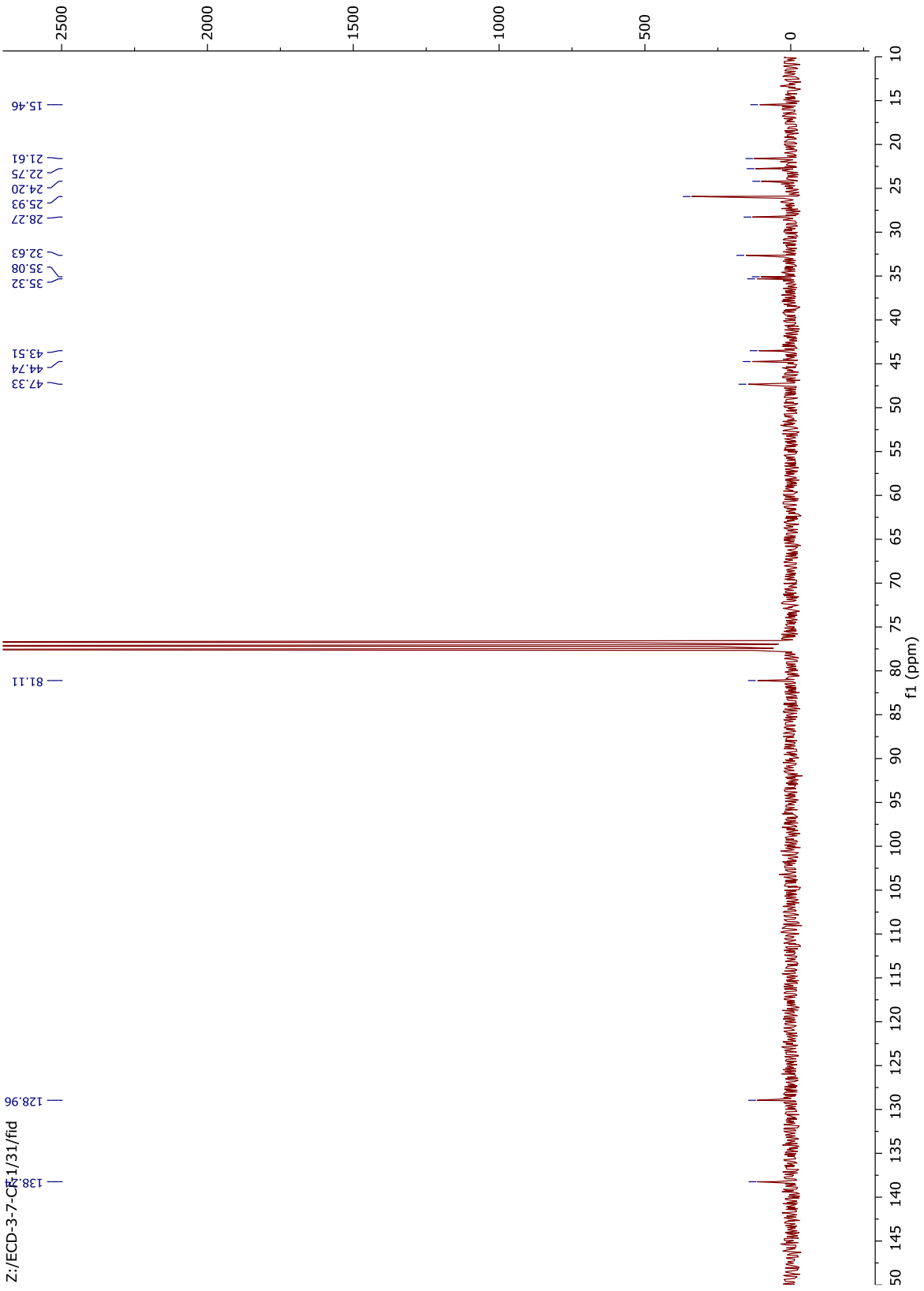
(2*S*)-(*E*)-4-(*p*-Menthan-3-yl)but-3-en-2-ol (18a) (^{13}C NMR)



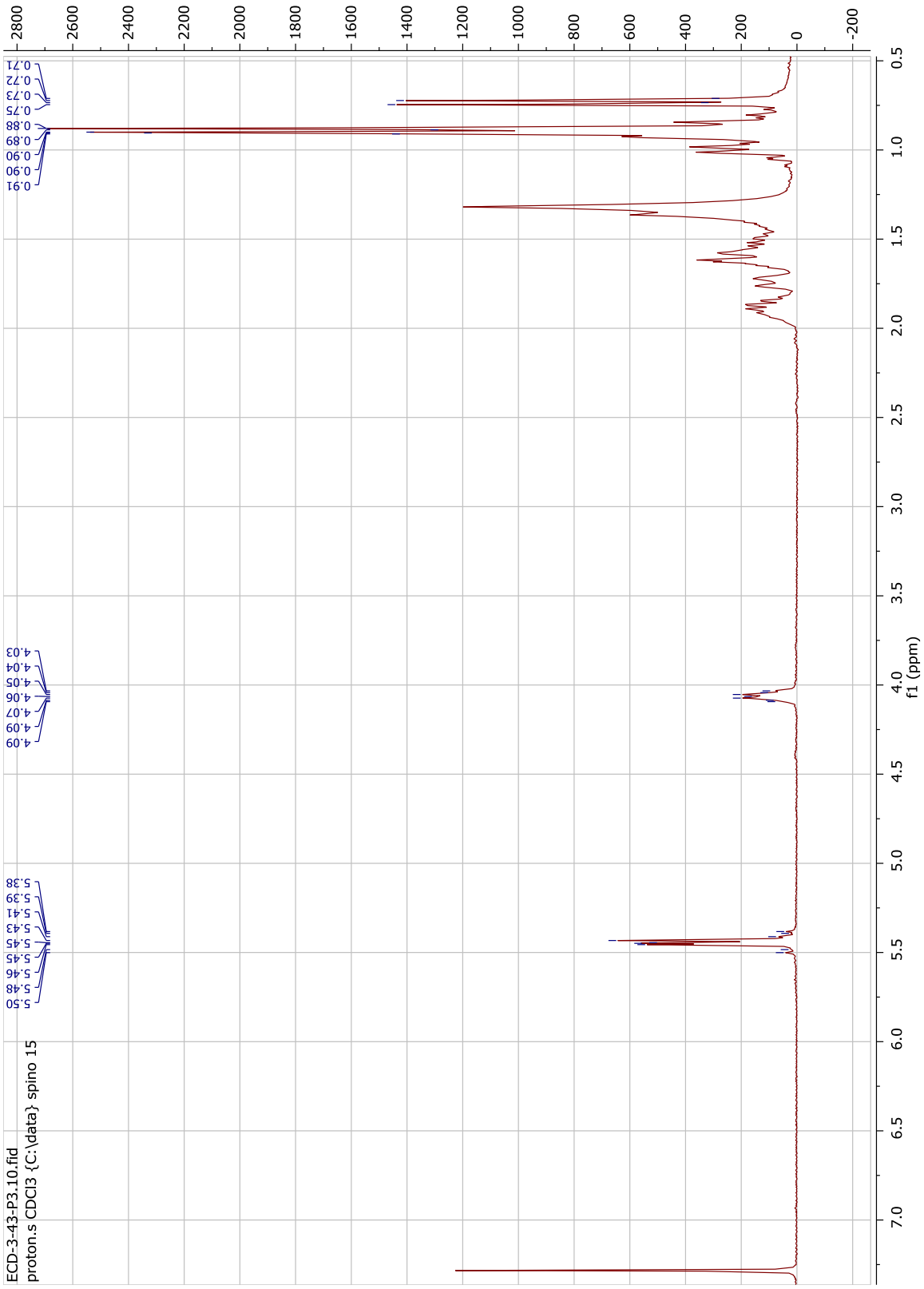
(3*R*)-(*E*)-5-(*p*-Menthan-3-yl)-2,2-dimethylpent-4-en-3-ol (18b) (^{1}H NMR)



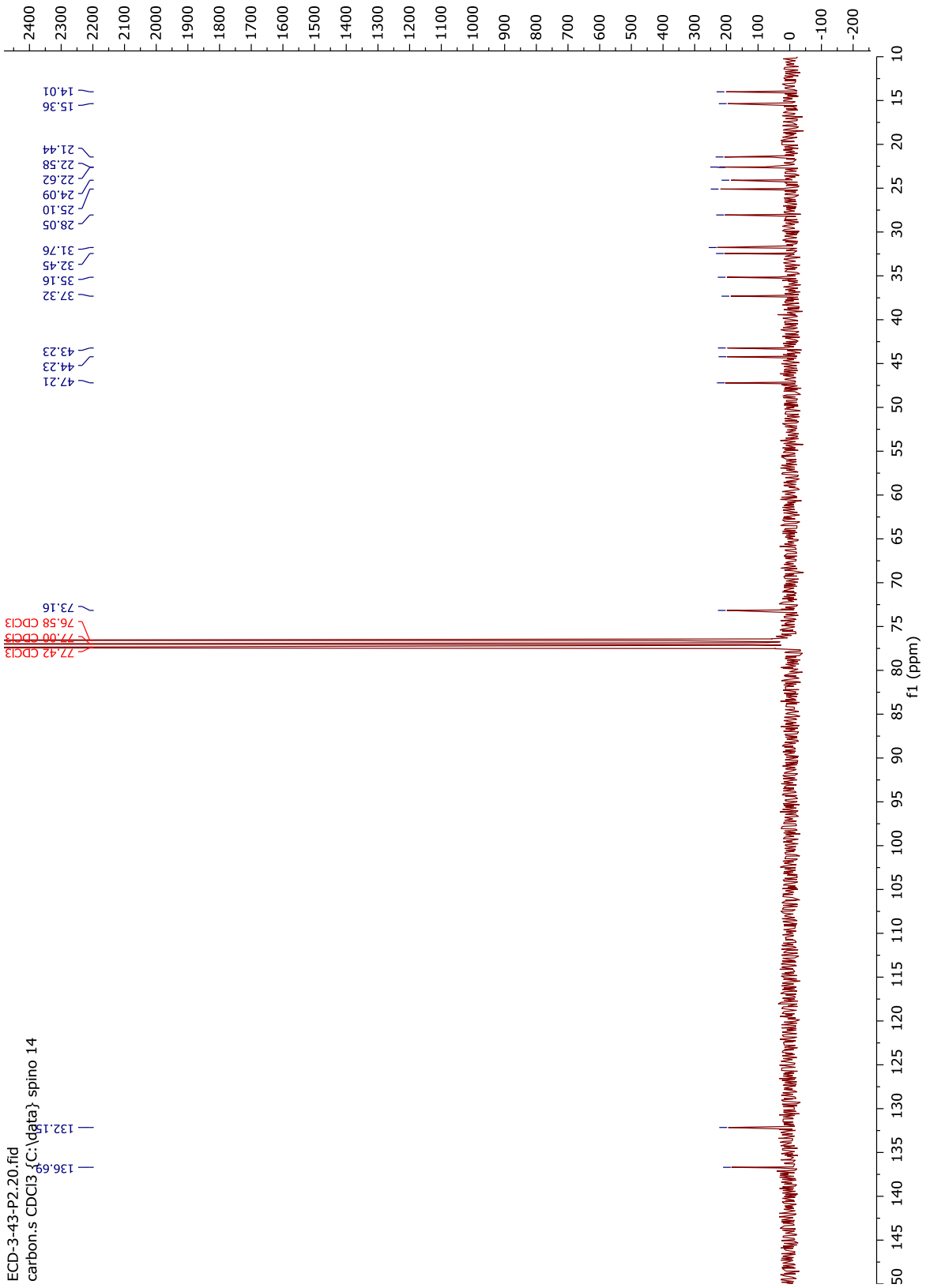
(3*R*)-(*E*)-5-(*p*-Menthan-3-yl)-2,2-dimethylpent-4-en-3-ol (18b) (^{13}C NMR)



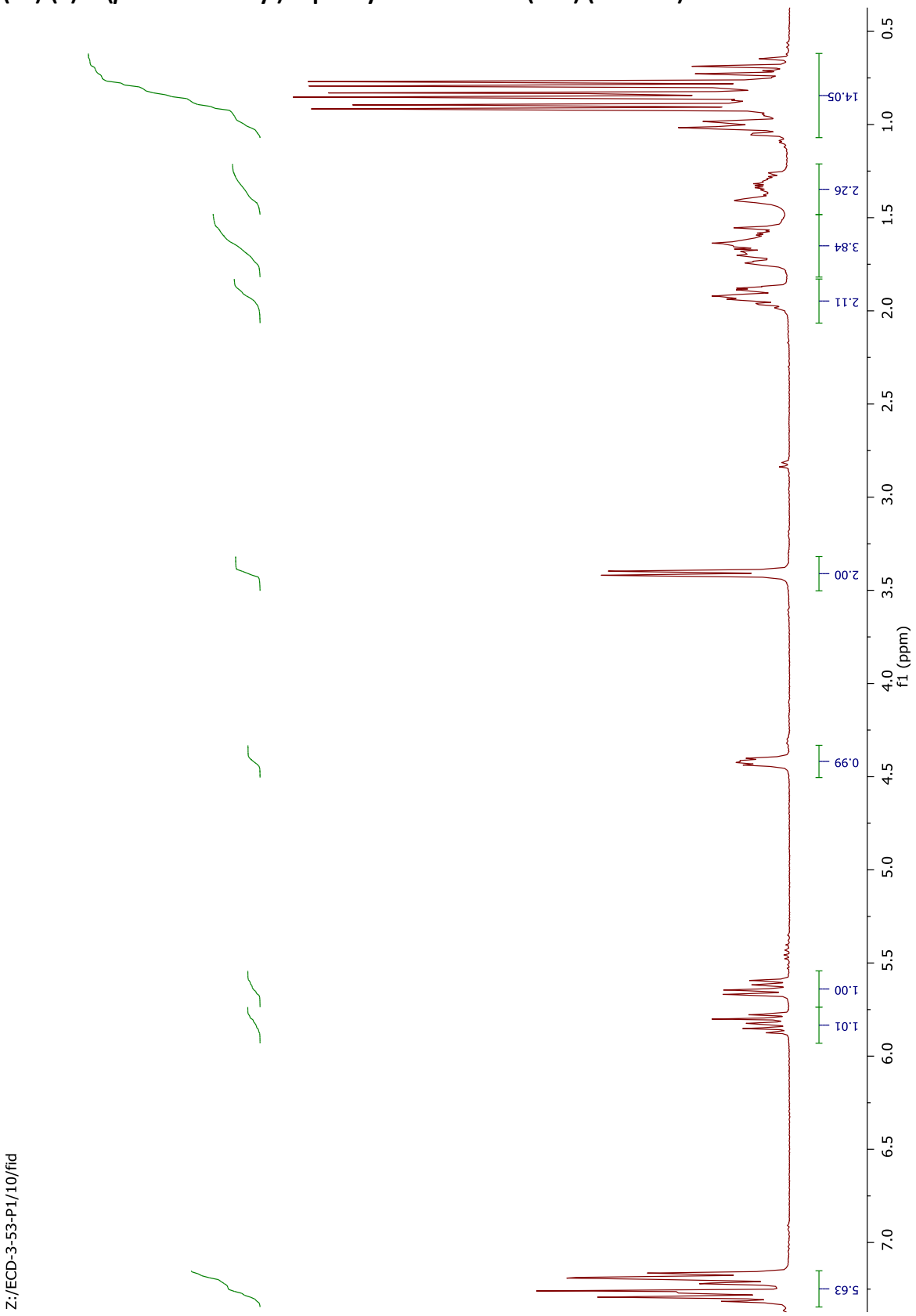
(6*S*)-(*E*)-8-(*p*-Menthan-3-yl)oct-7-en-6-ol (18c) (^{1}H NMR)



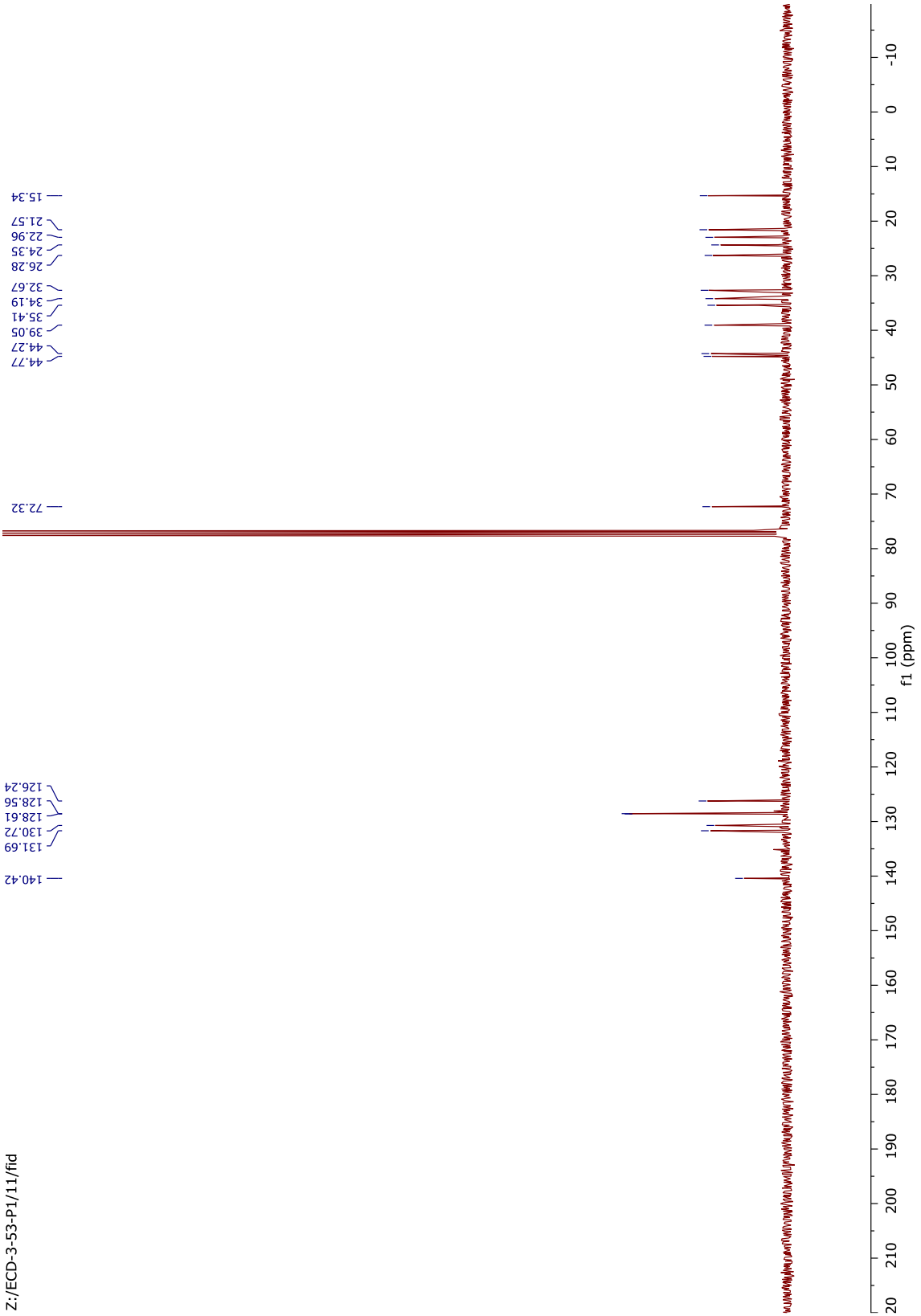
(6*S*)-(*E*)-8-(*p*-Menthan-3-yl)oct-7-en-6-ol (18c) (^{13}C NMR)



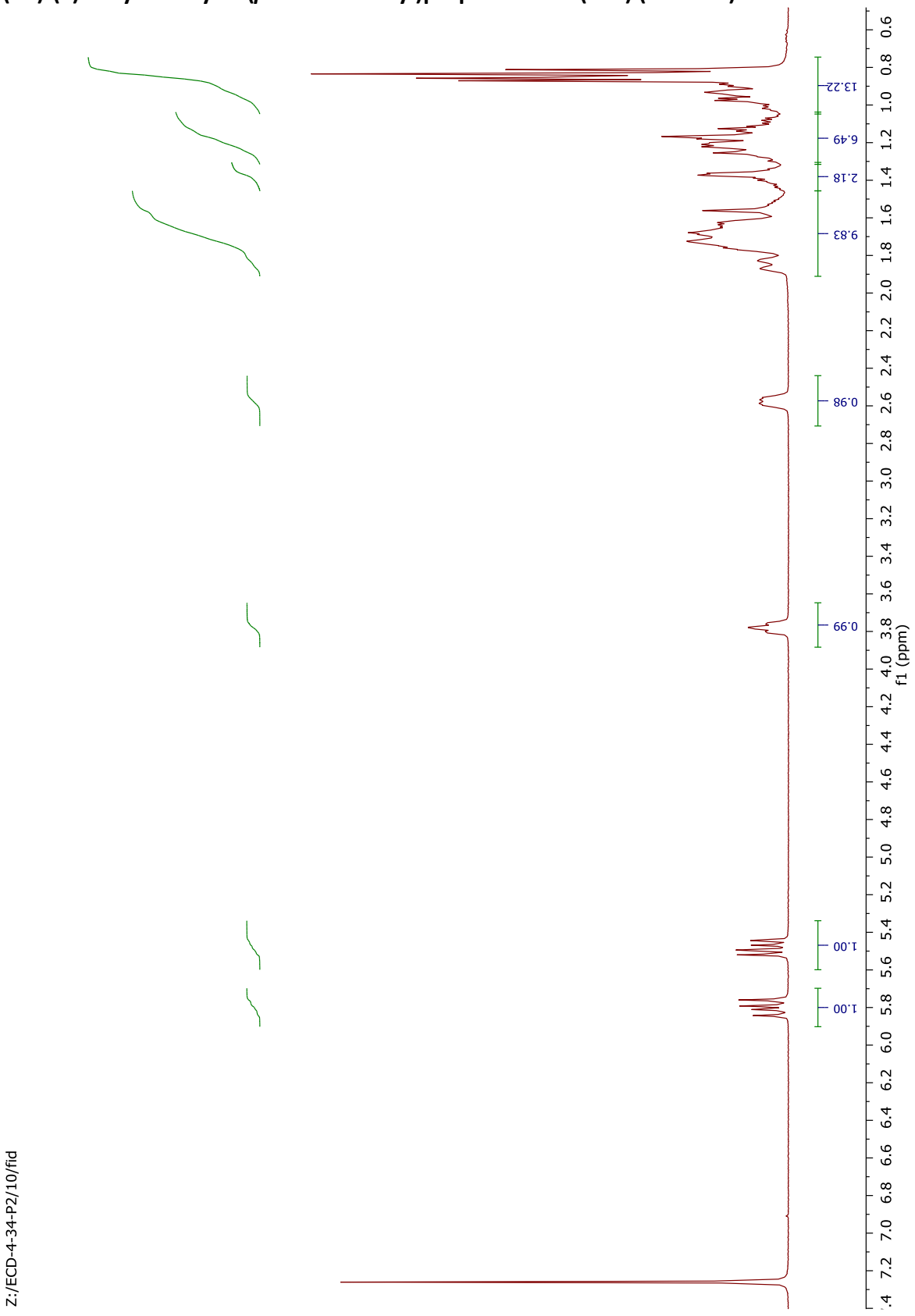
(2*S*)-(*E*)-4-(*p*-Menthan-3-yl)-1-phenylbut-3-en-2-ol (18d) (^{1}H NMR)



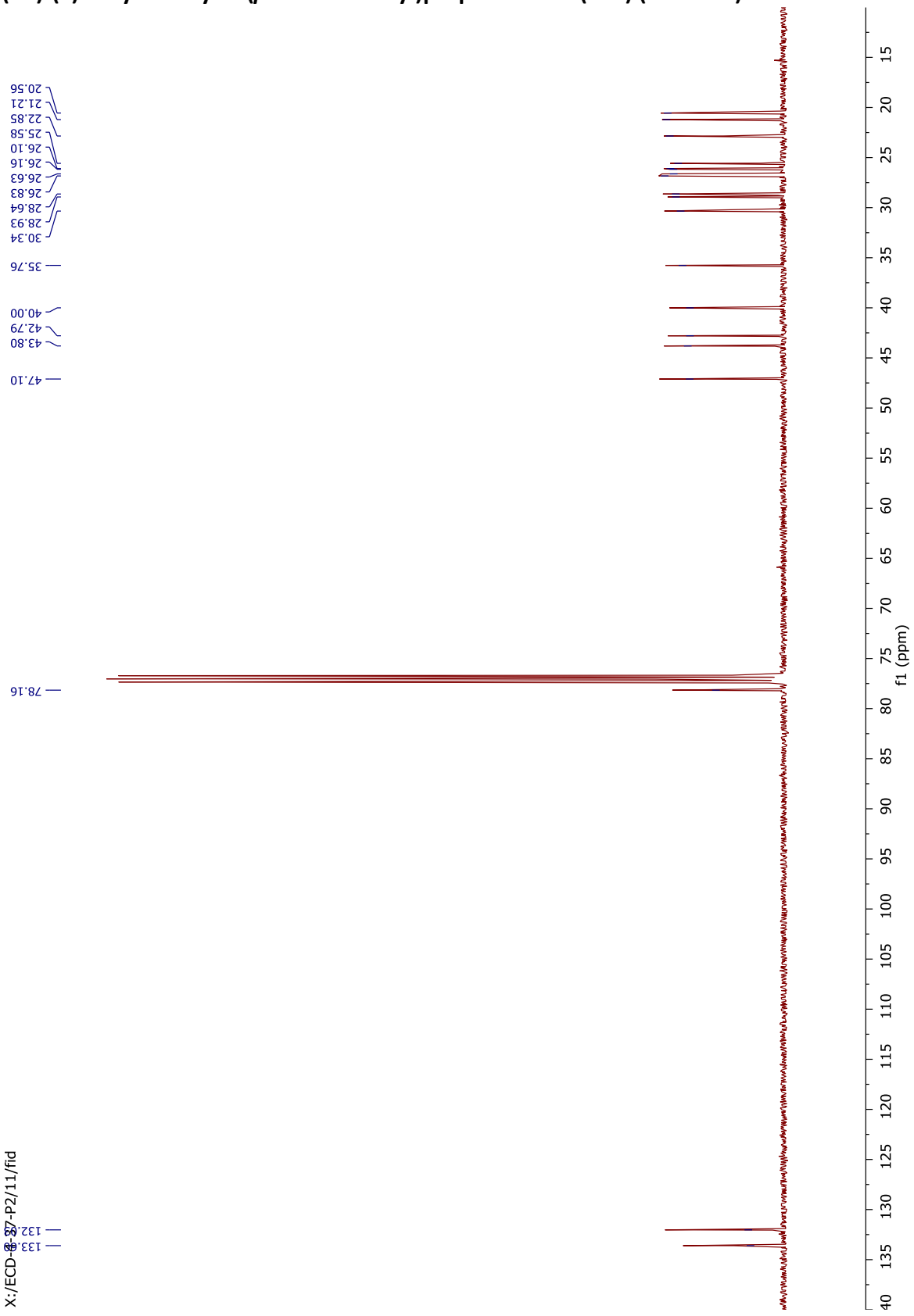
(2*S*)-(*E*)-4-(*p*-Menthan-3-yl)-1-phenylbut-3-en-2-ol (18d) (^{13}C NMR)



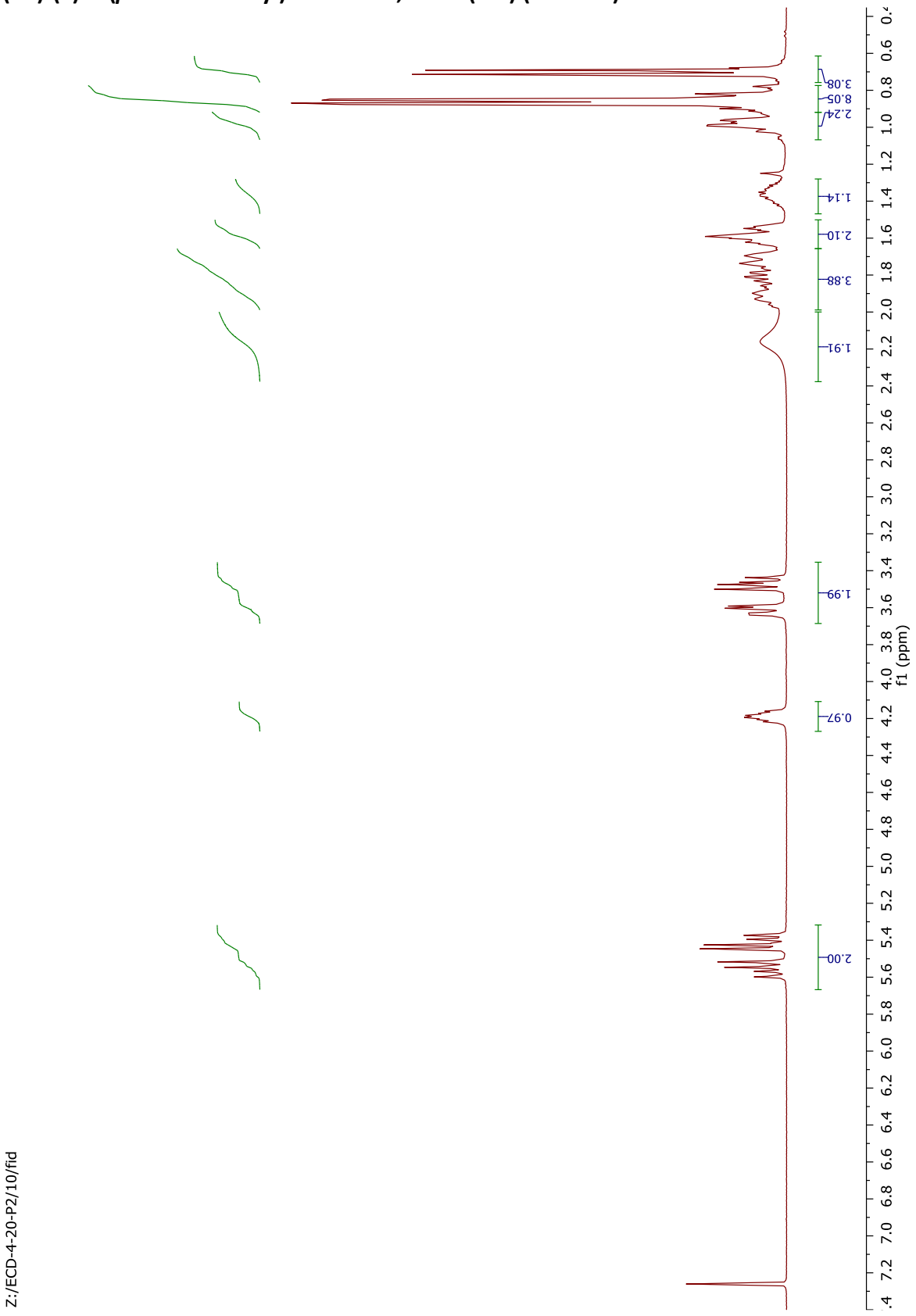
(1*R*)-(*E*)-1-Cyclohexyl-3-(*p*-menthan-3-yl)prop-2-en-1-ol (18e) (^{1}H NMR)



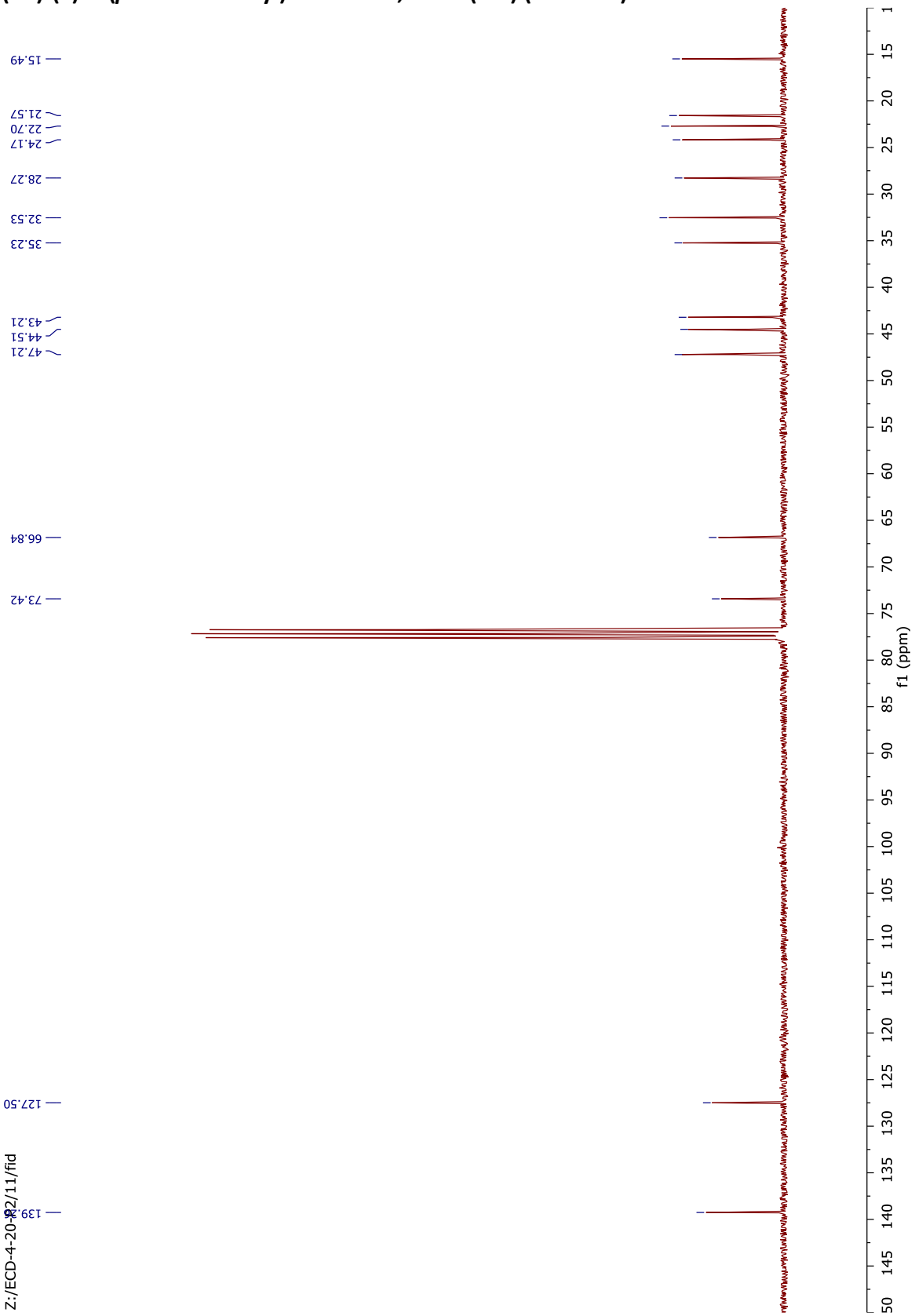
(1*R*)-(*E*)-1-Cyclohexyl-3-(*p*-menthan-3-yl)prop-2-en-1-ol (18e) (^{13}C NMR)



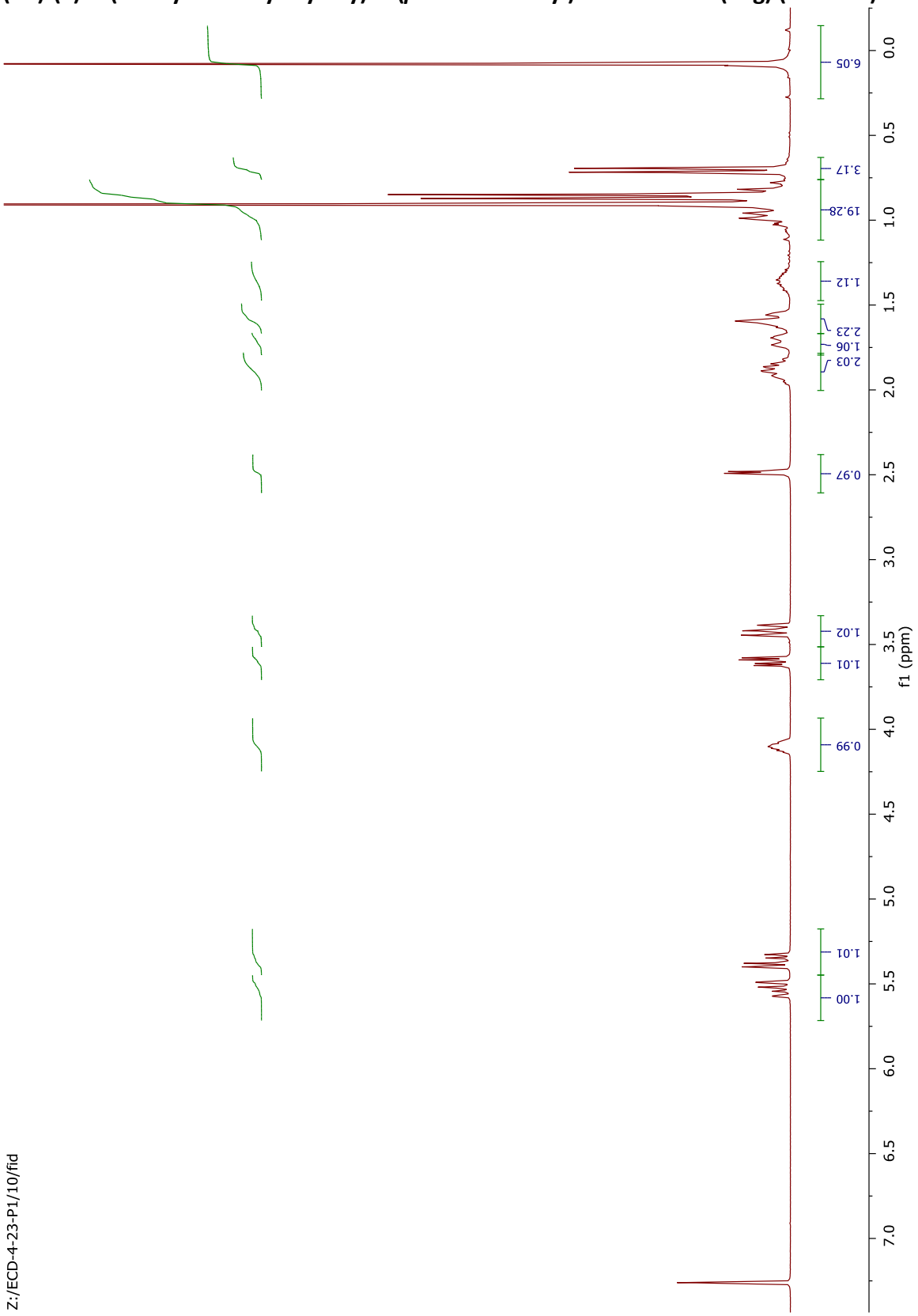
(2*R*)-(*E*)-4-(*p*-Menthan-3-yl)but-3-en-1,2-diol (18f) (^{1}H NMR)



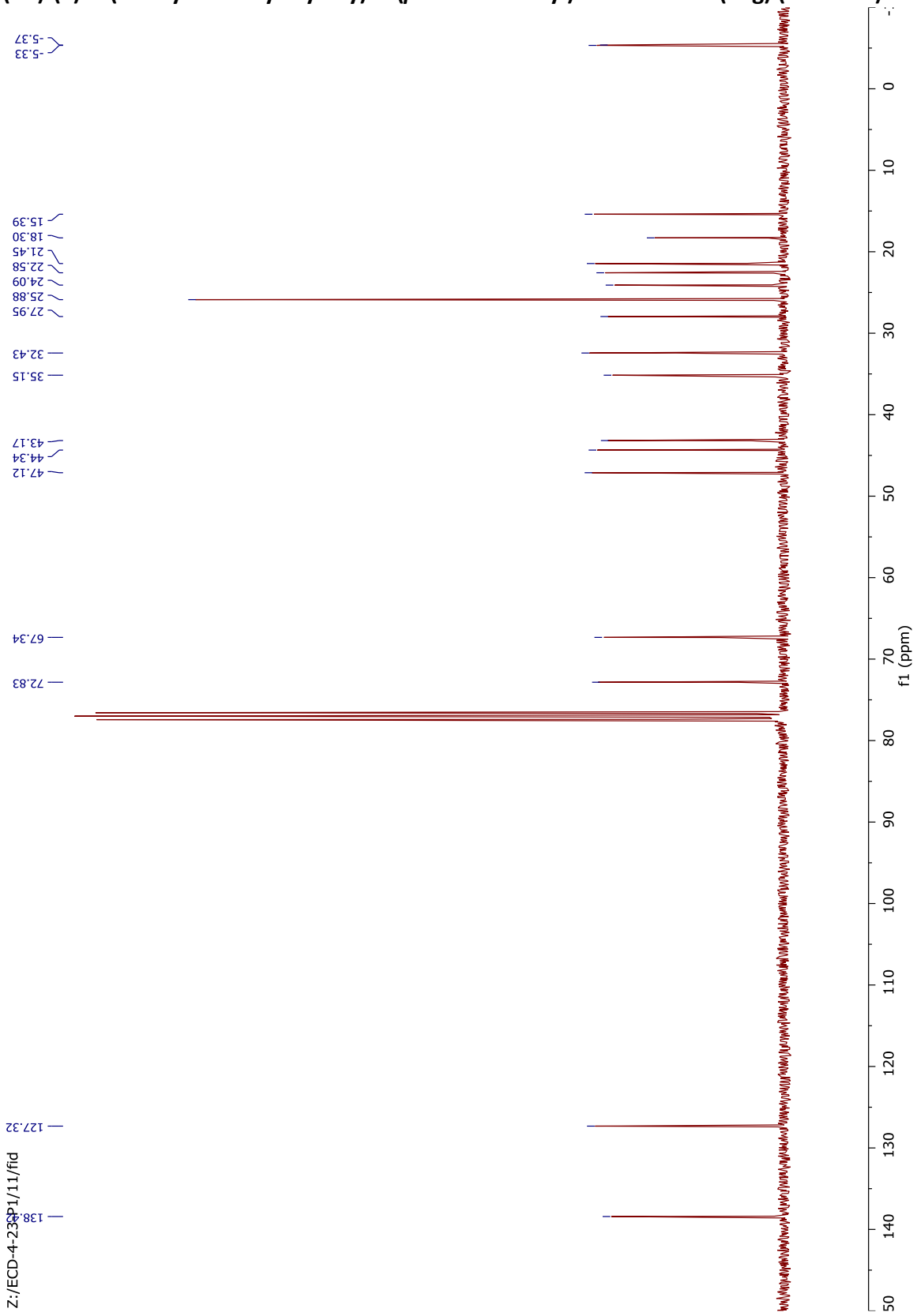
(2*R*)-(*E*)-4-(*p*-Menthan-3-yl)but-3-en-1,2-diol (18f) (^{13}C NMR)



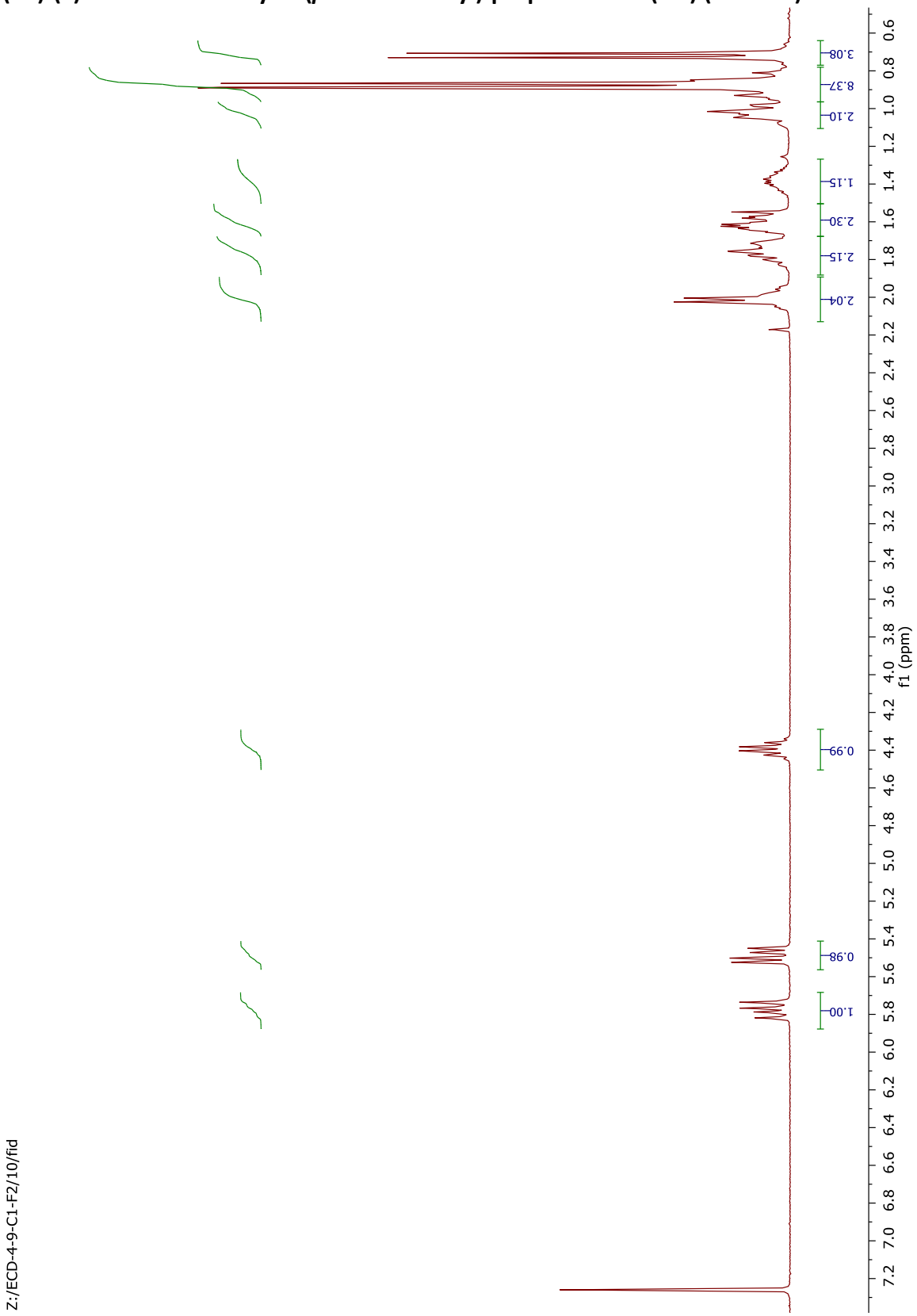
(2*R*)-(*E*)-1-(*t*-Butyldimethylsilyloxy)-4-(*p*-menthan-3-yl)but-3-en-2-ol (18g) (^{1}H NMR)



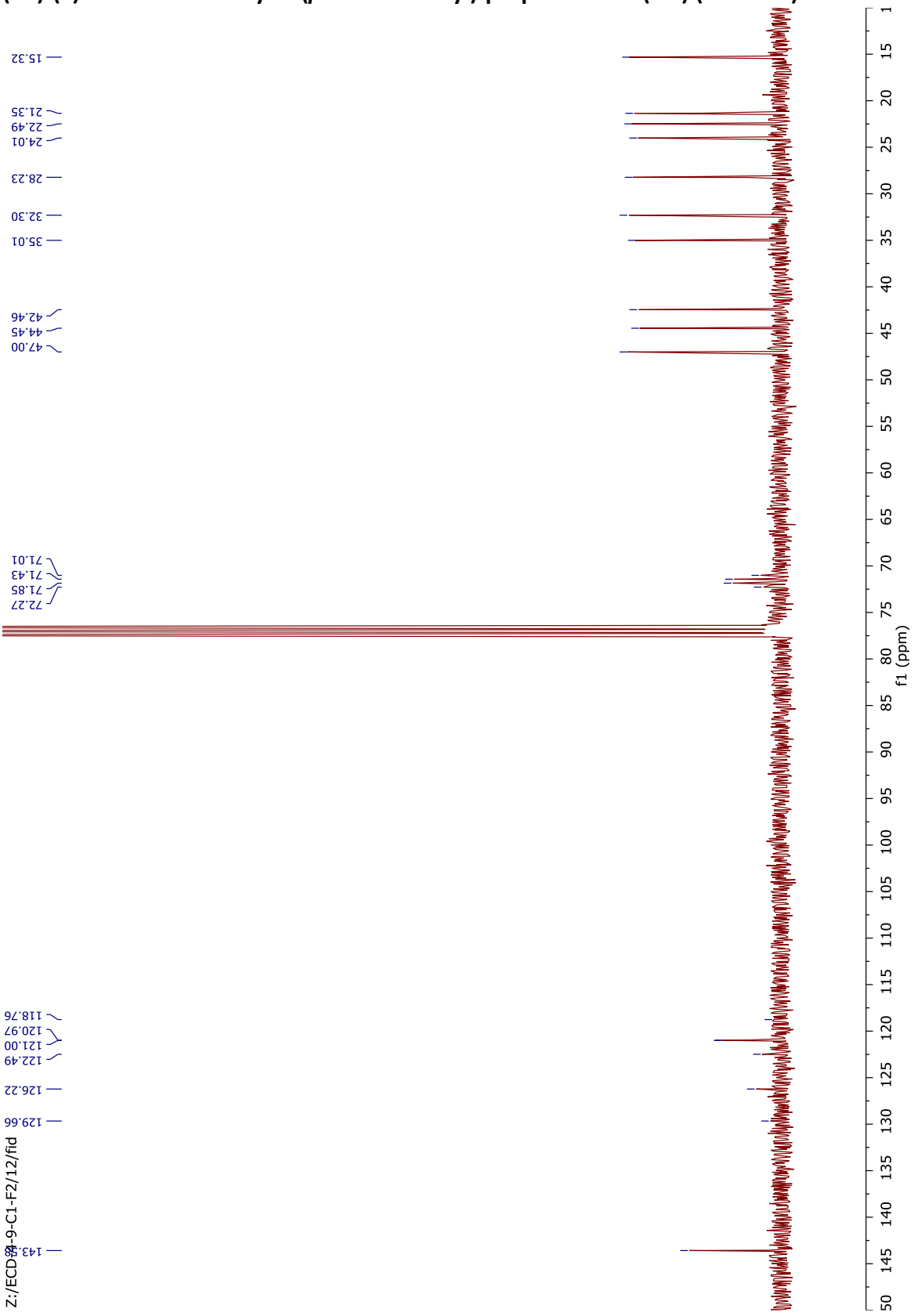
(2*R*)-(*E*)-1-(*t*-Butyldimethylsilyloxy)-4-(*p*-menthan-3-yl)but-3-en-2-ol (18g) (^{13}C NMR)



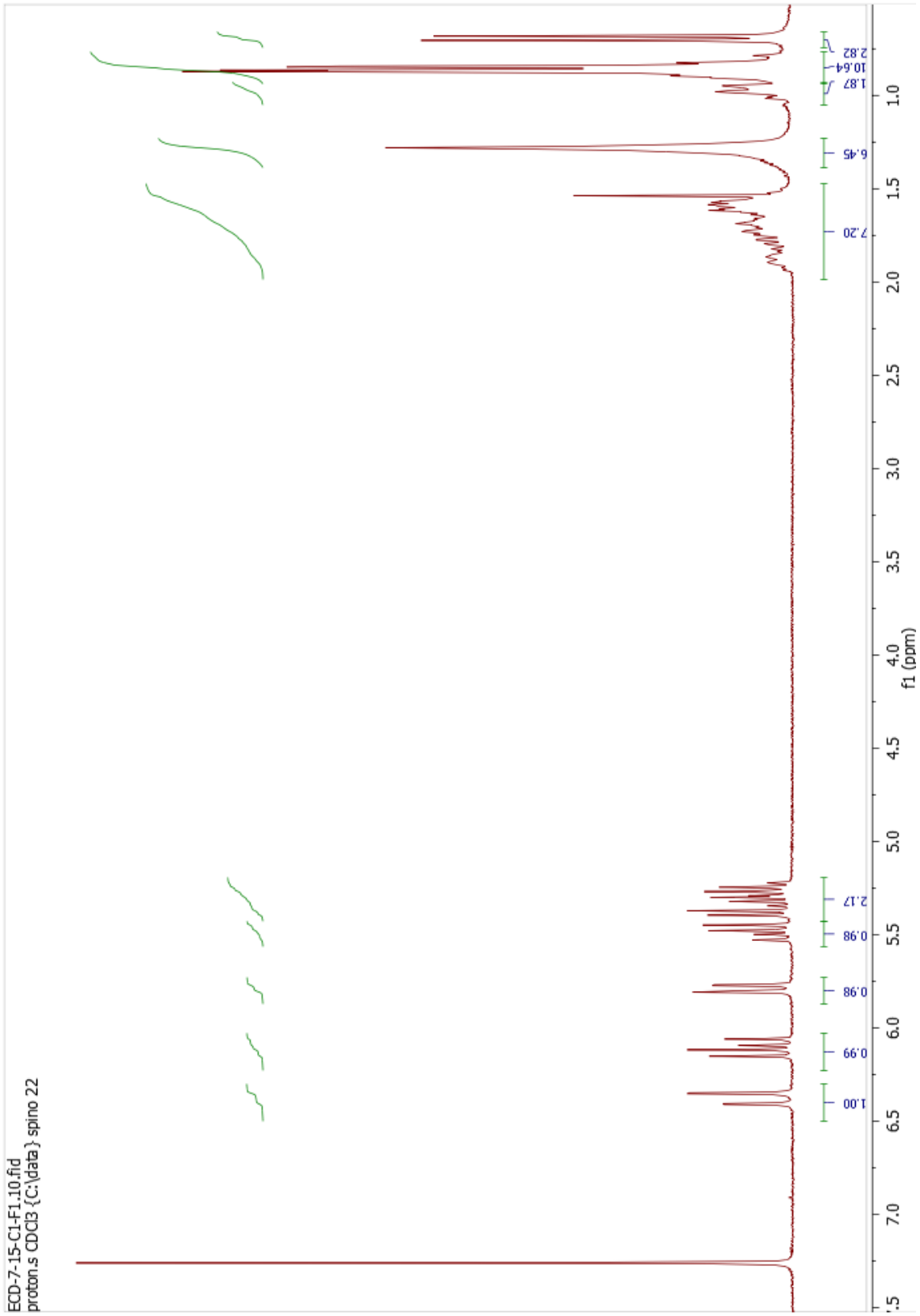
(1*R*)-(*E*)-1-Trifluoromethyl-4-(*p*-menthan-3-yl)-prop-2-en-1-ol (18i) (^{1}H NMR)



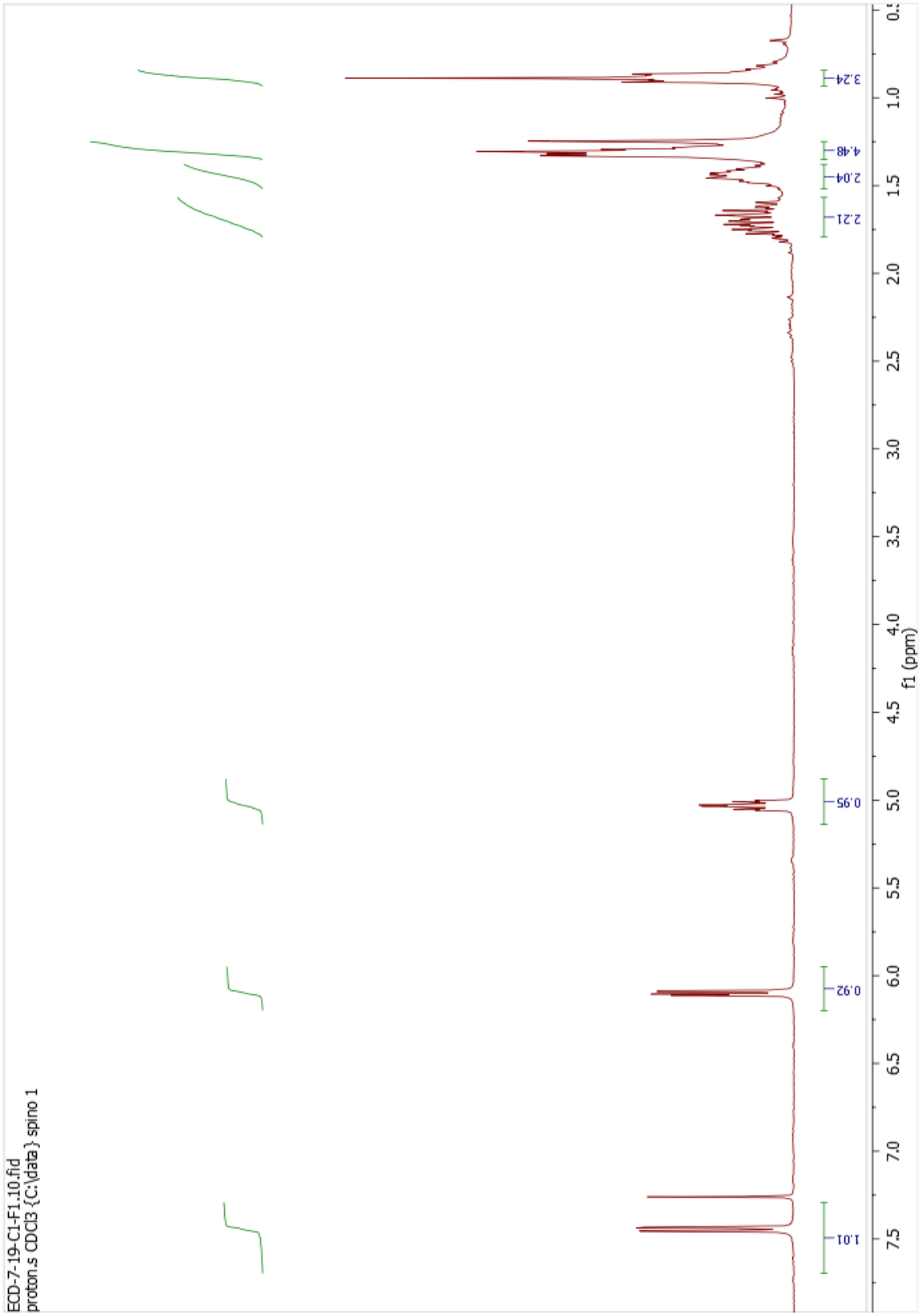
(1*R*)-(*E*)-1-Trifluoromethyl-4-(*p*-menthan-3-yl)-prop-2-en-1-ol (18i) (^{13}C NMR)



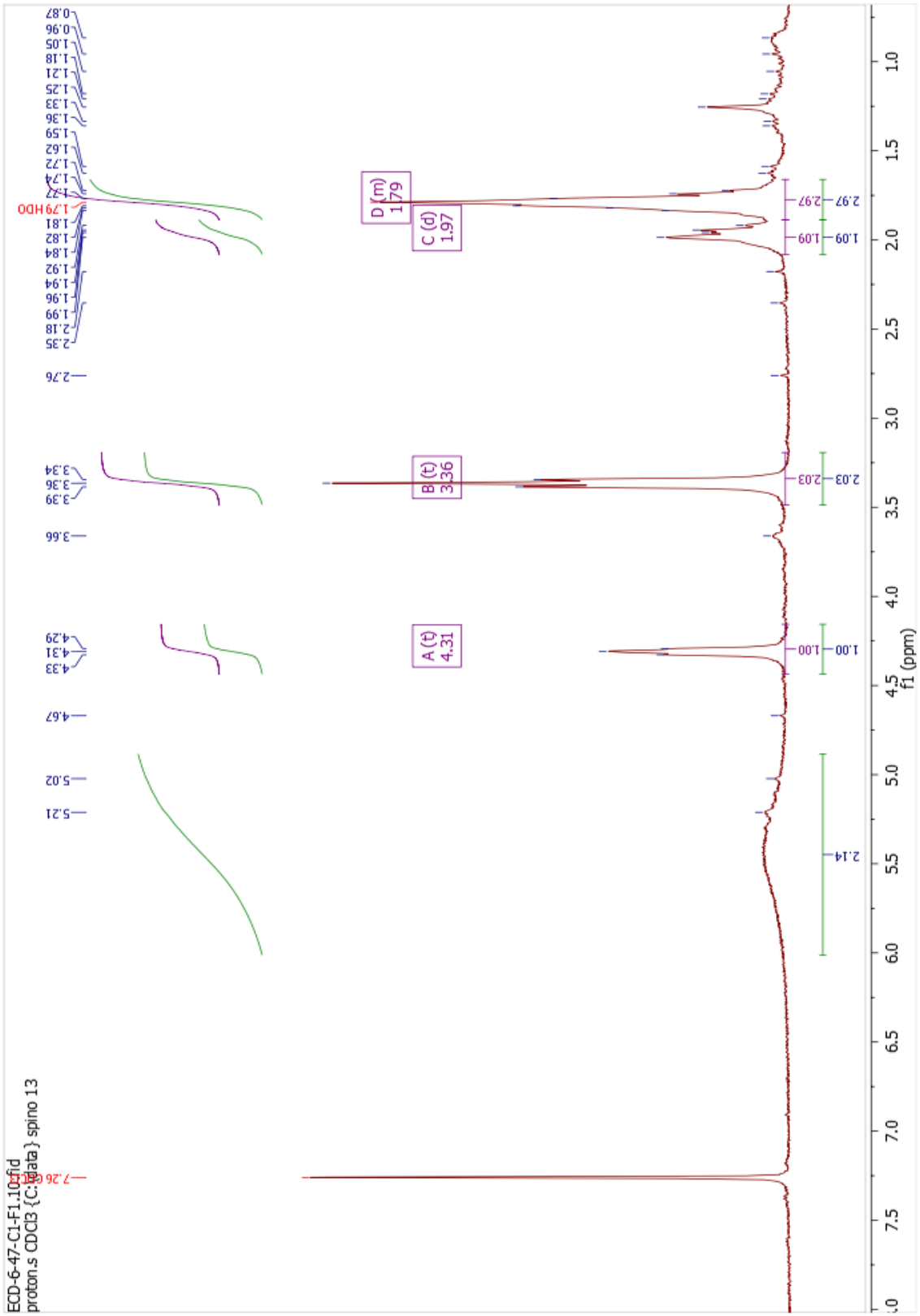
(6*R*)-(*E*)-8-(p-Menthan-3-yl)oct-7-en-6-ol, acryloyl ester (30) (^{1}H NMR)



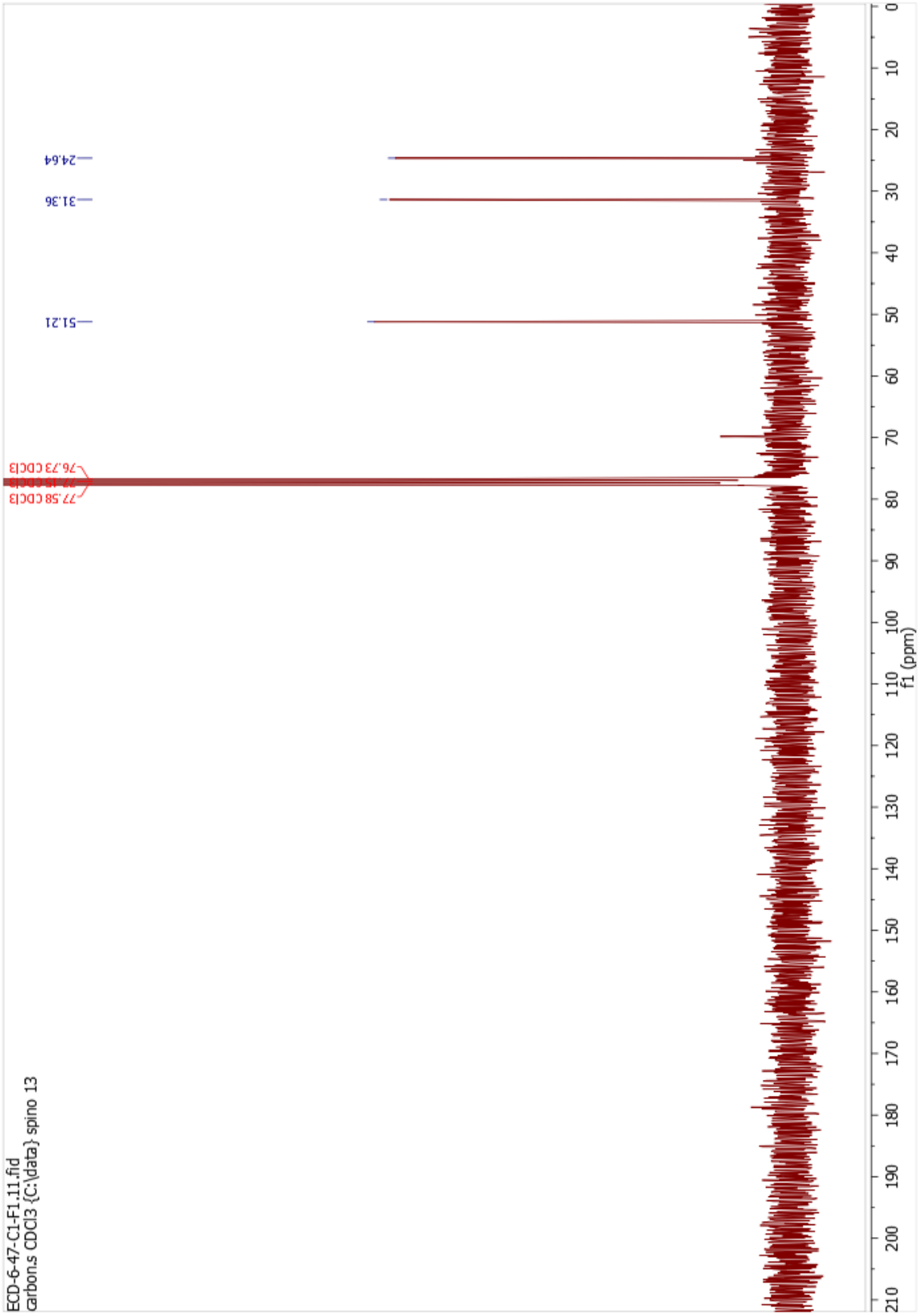
(5*R*)-5-Pentylfuran-2(5H)-one (20) (^{1}H NMR)



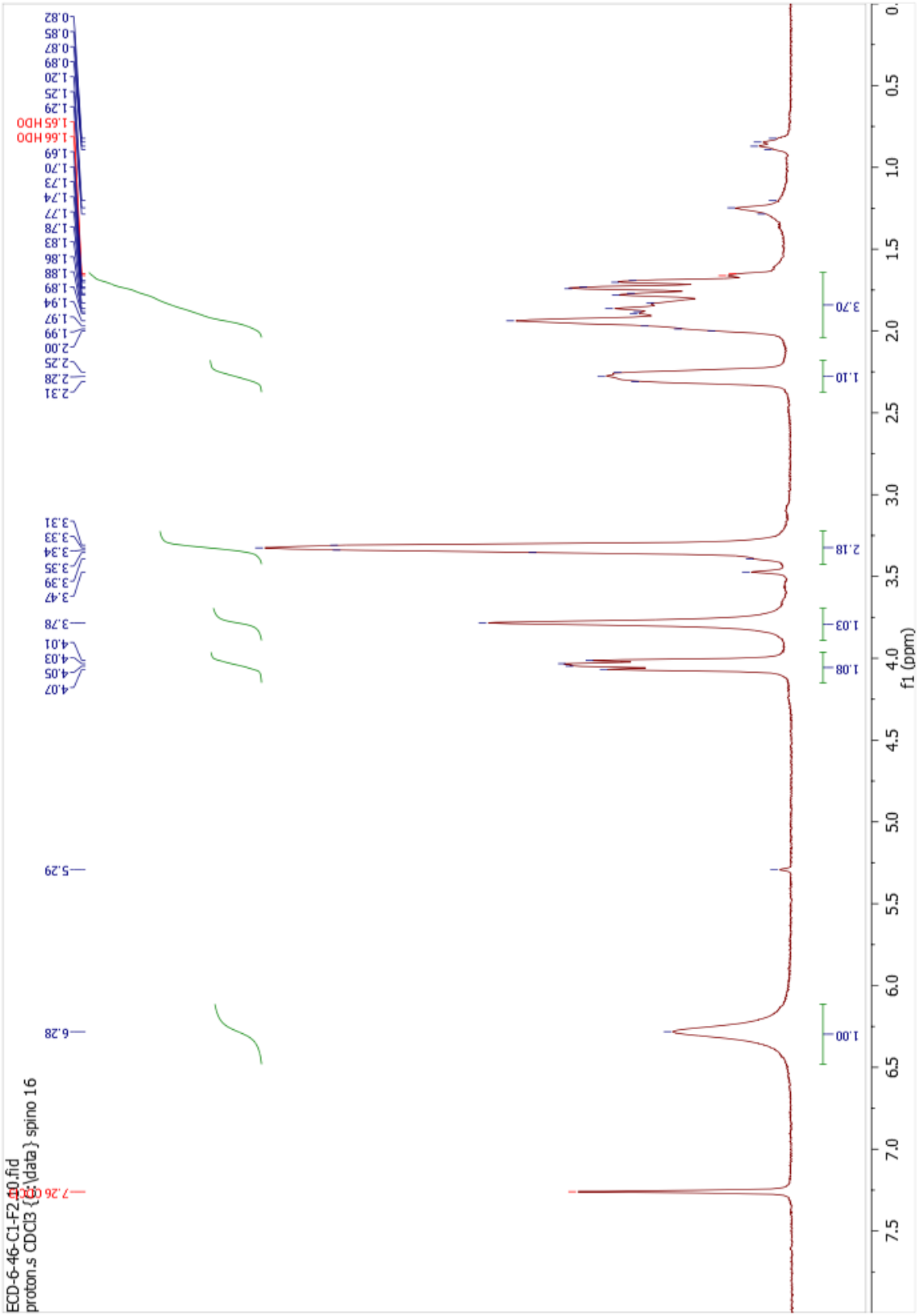
5-Azido-2-hydroxypentanoic acid 21 (^{1}H NMR)



5-Azido-2-hydroxypentanoic acid 21 (^{13}C NMR)



(3*R*)-3-Hydroxypiperidin-2-one (22) (^{1}H NMR)

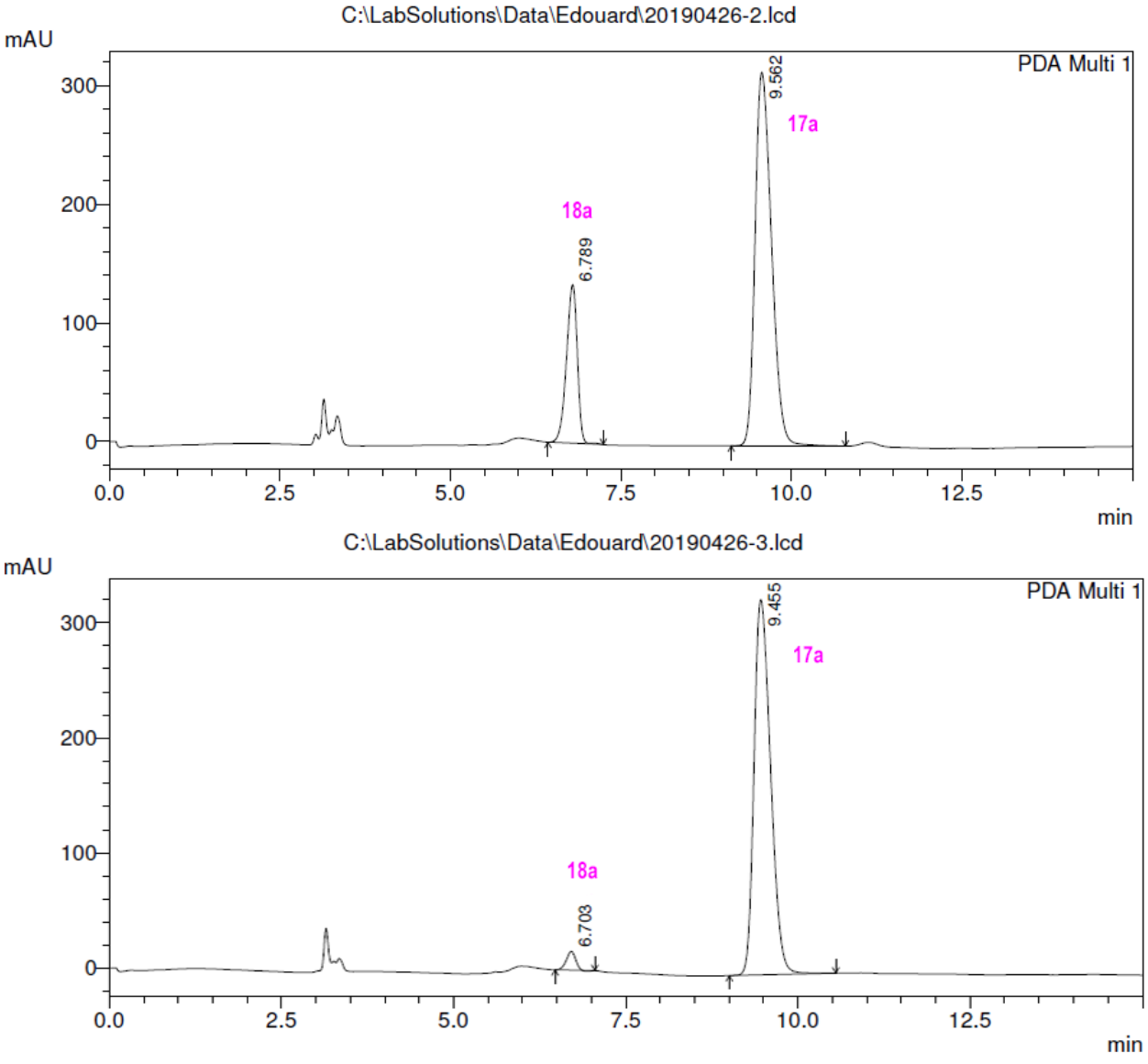


Copies of HPLC traces

All HPLCs were run on a Shimadzu LC using the same column and the same method : OZ_H_1_IPA_1.3 mL_min.lcm

HPLC of rearrangement of 16a.

1st spectrum, mixture of **17a** and **18a**. 2nd spectrum, rearrangement of **16a** (crude)



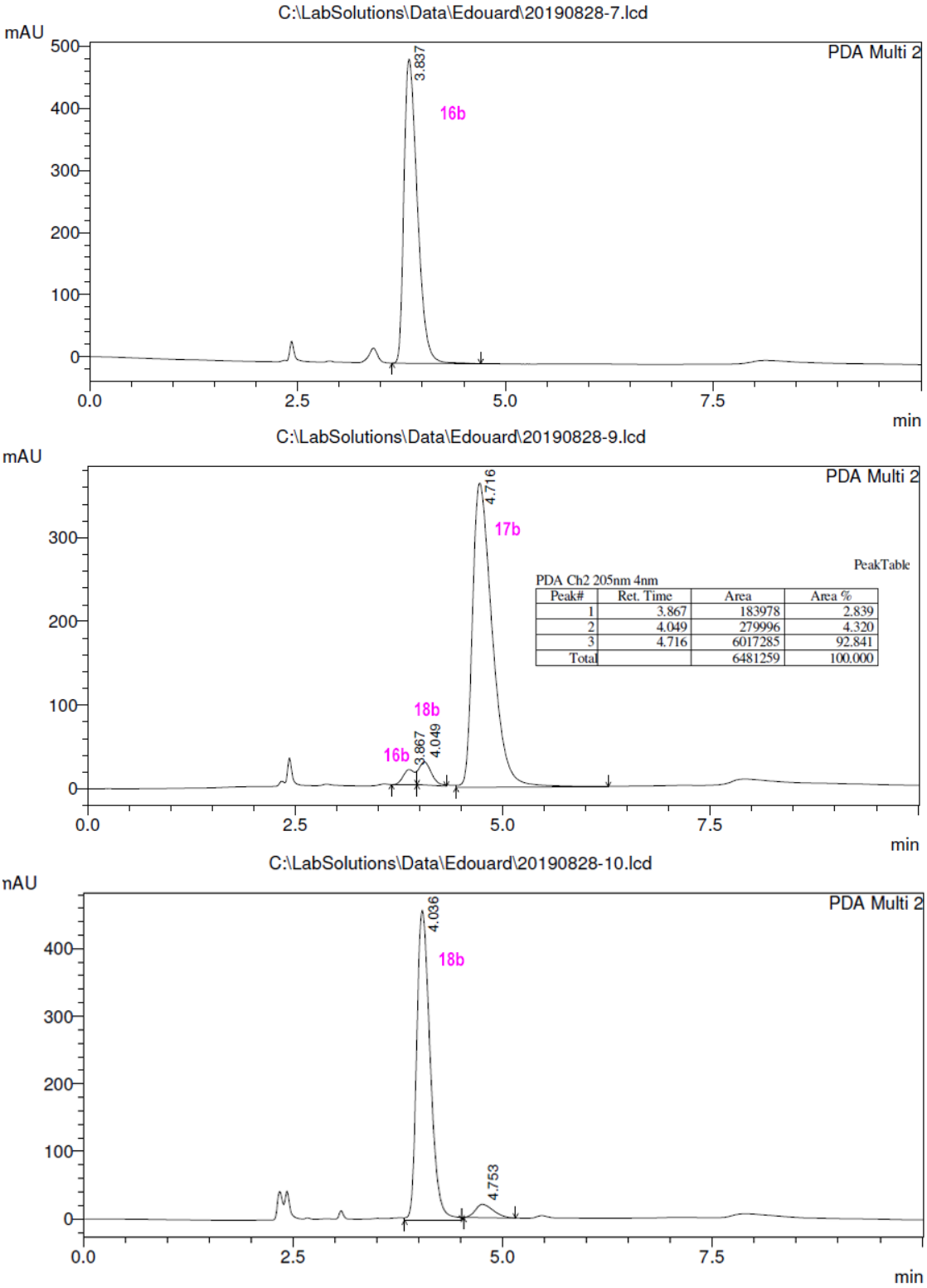
PeakTable

PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Area %
1	6.703	148395	2.649
2	9.455	5454583	97.351
Total		5602979	100.000

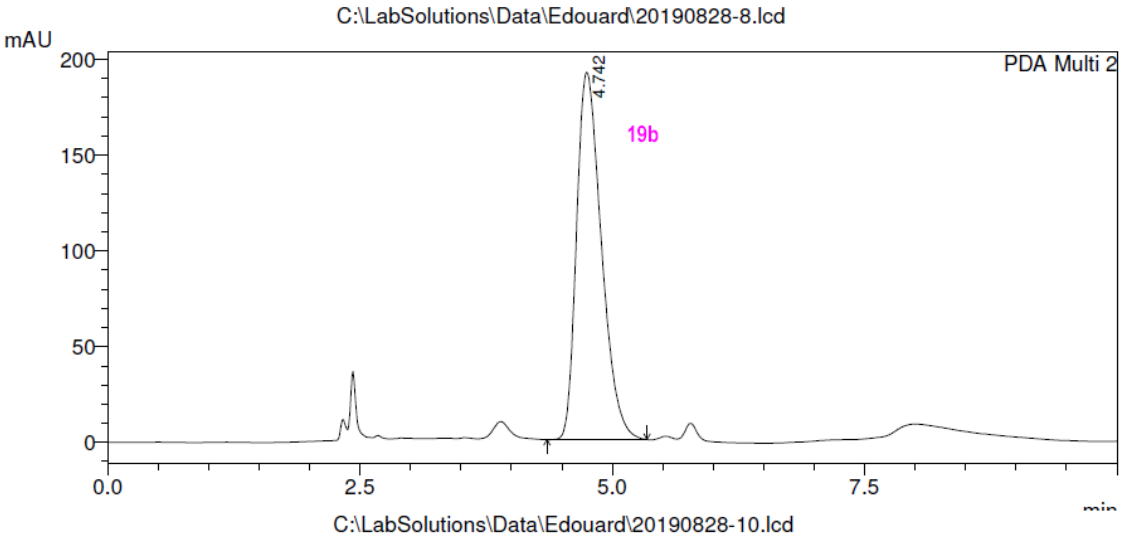
HPLC of rearrangement of 16b.

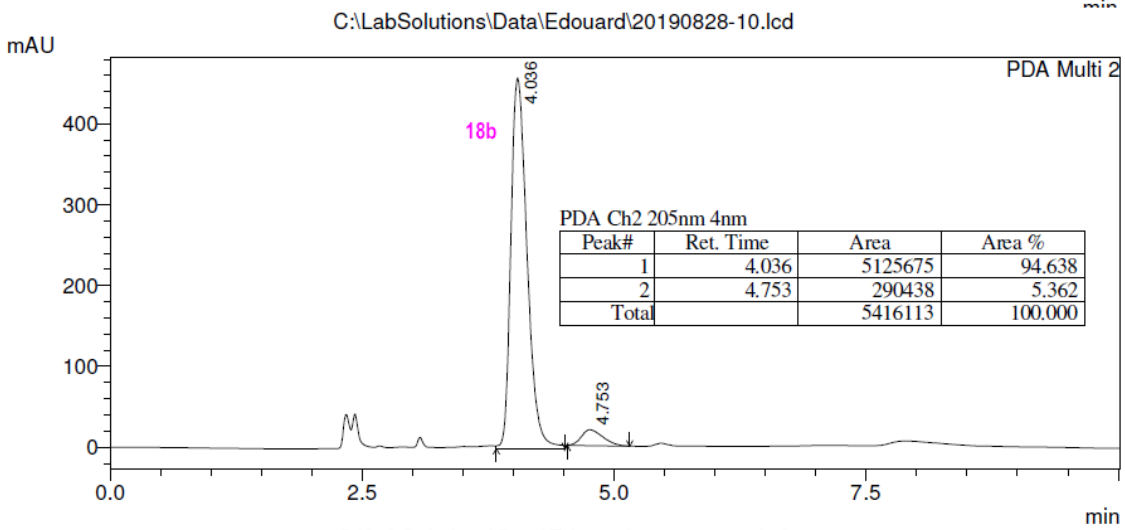
1st spectrum, **16b**. 2nd spectrum rearrangement of **16b**. 3rd spectrum **18b**



HPLC of rearrangement of 19b.

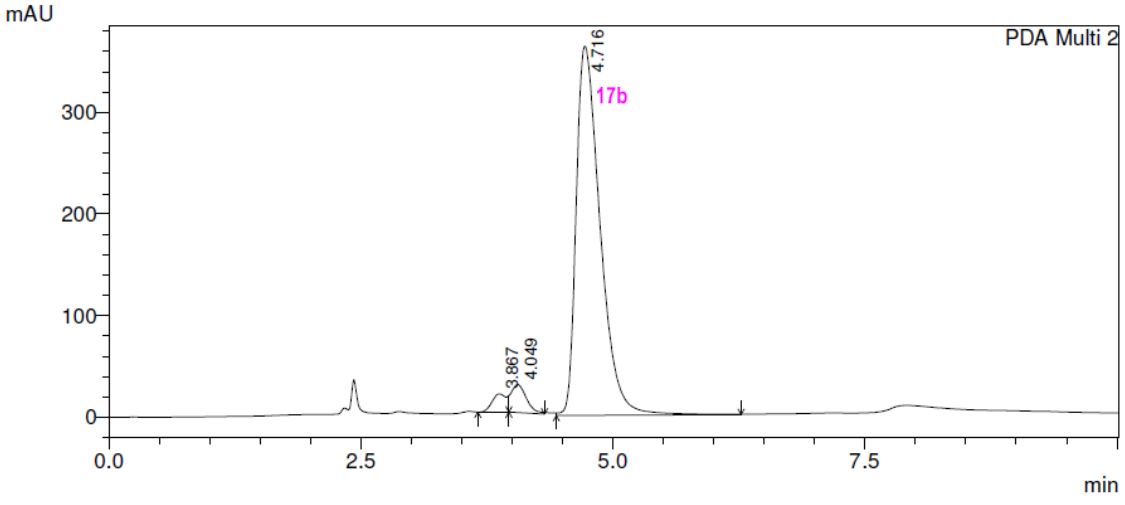
1st spectrum **19b**. 2nd spectrum rearrangement of **19b.** 3rd spectrum **17b**.





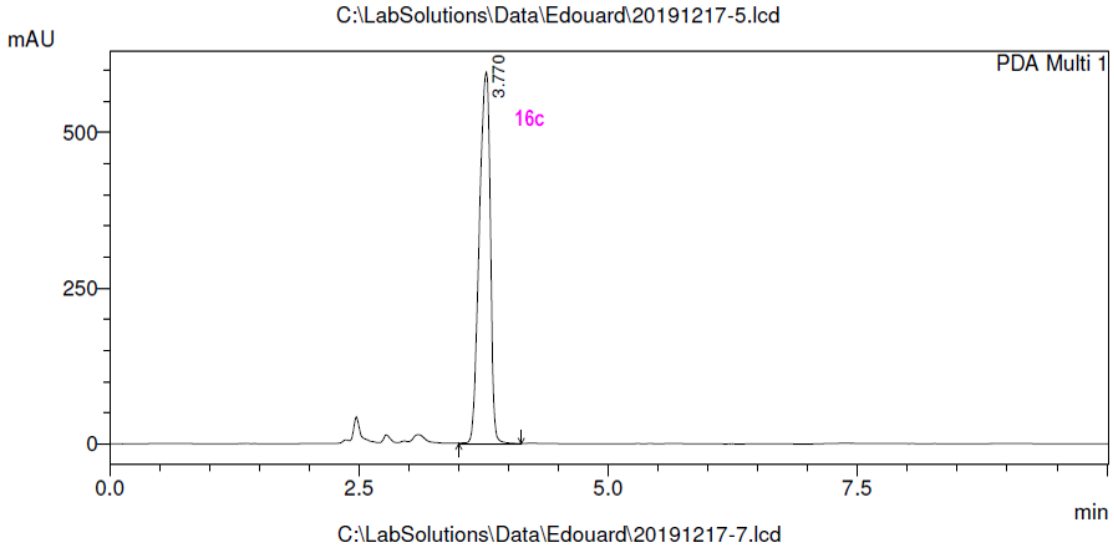
PDA Ch2 205nm 4nm

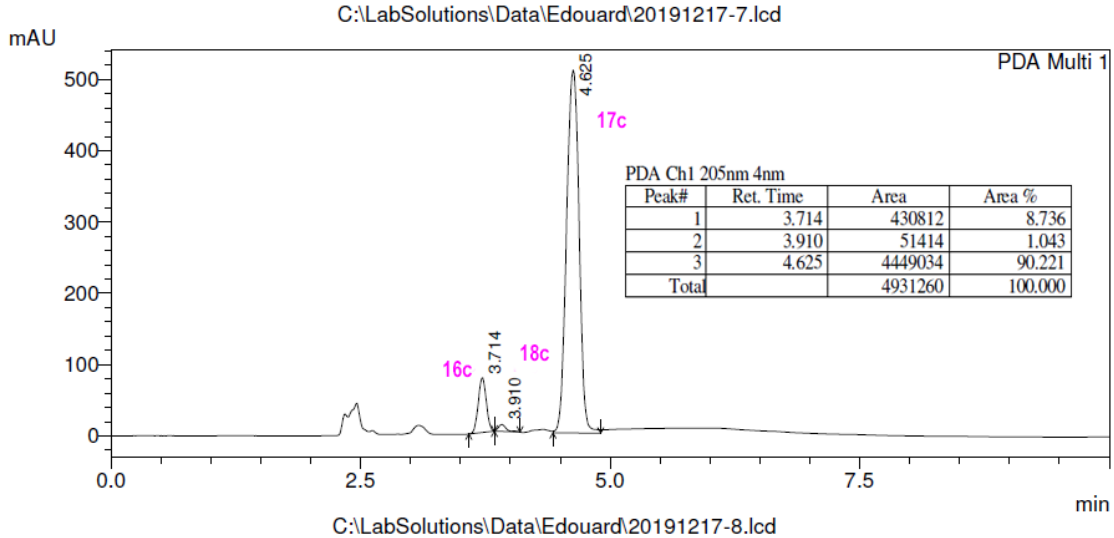
Peak#	Ret. Time	Area	Area %
1	4.036	5125675	94.638
2	4.753	290438	5.362
Total		5416113	100.000



HPLC of rearrangement of 16c.

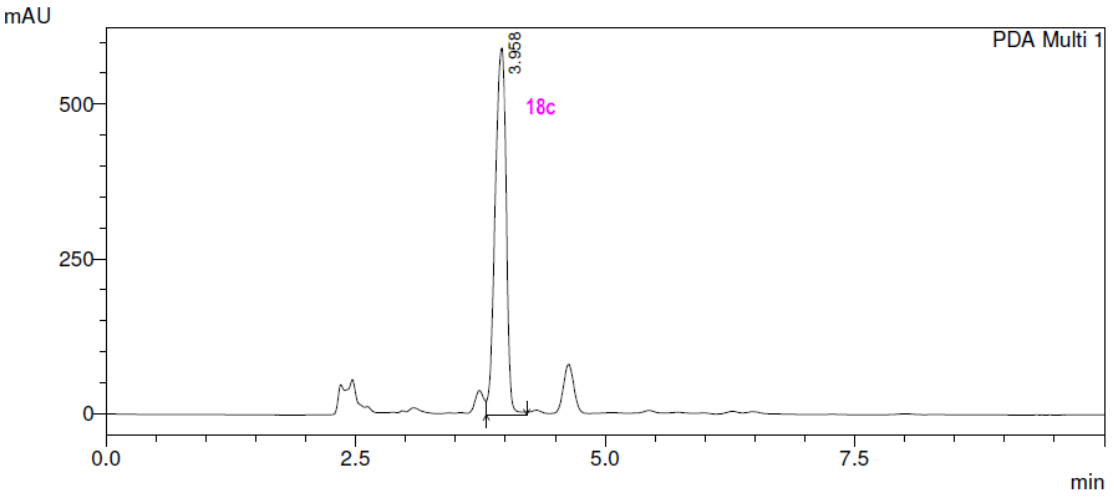
1st spectrum **16c**. 2nd spectrum rearrangement of **16c.** 3rd spectrum **18c**.





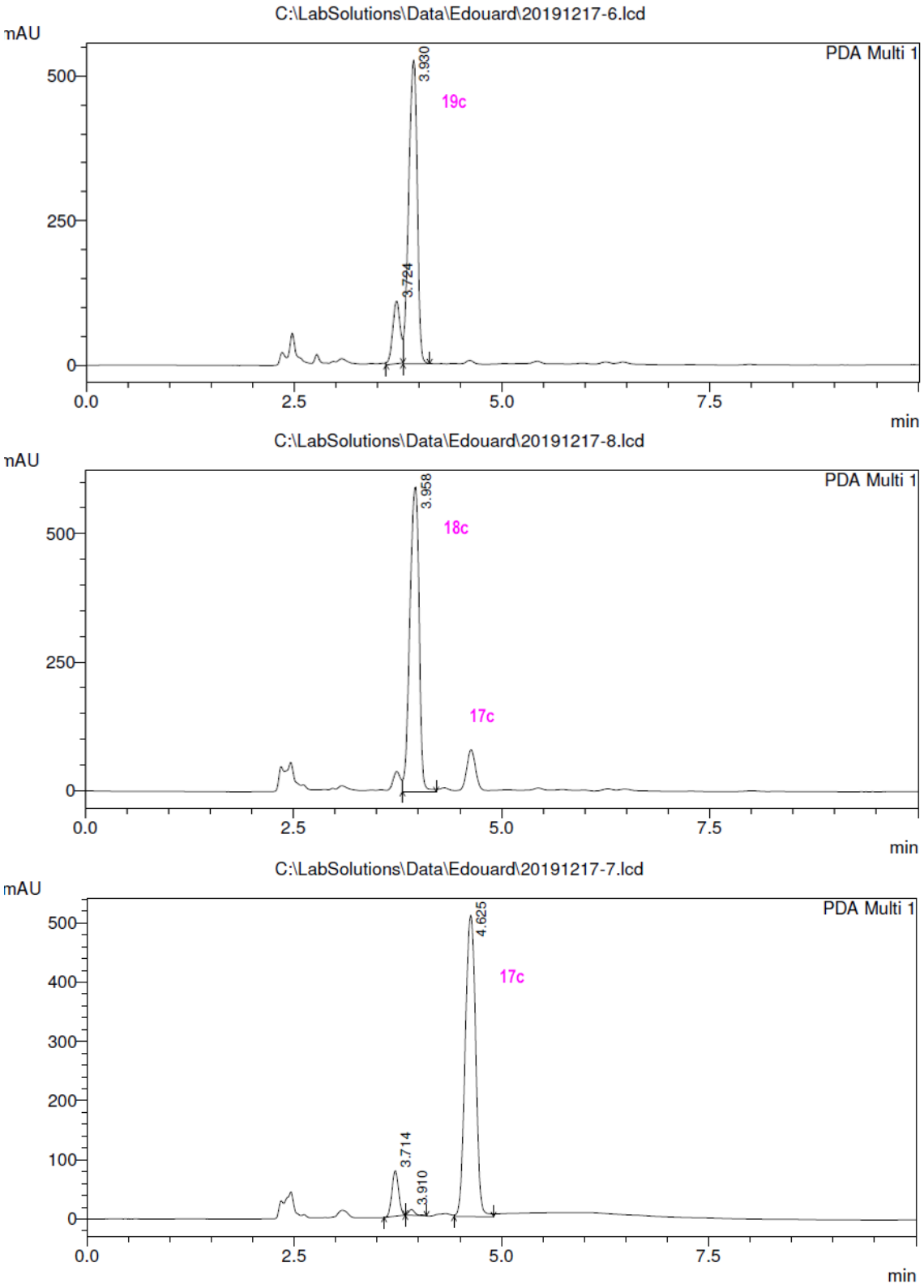
PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Area %
1	3.714	430812	8.736
2	3.910	51414	1.043
3	4.625	4449034	90.221
Total		4931260	100.000



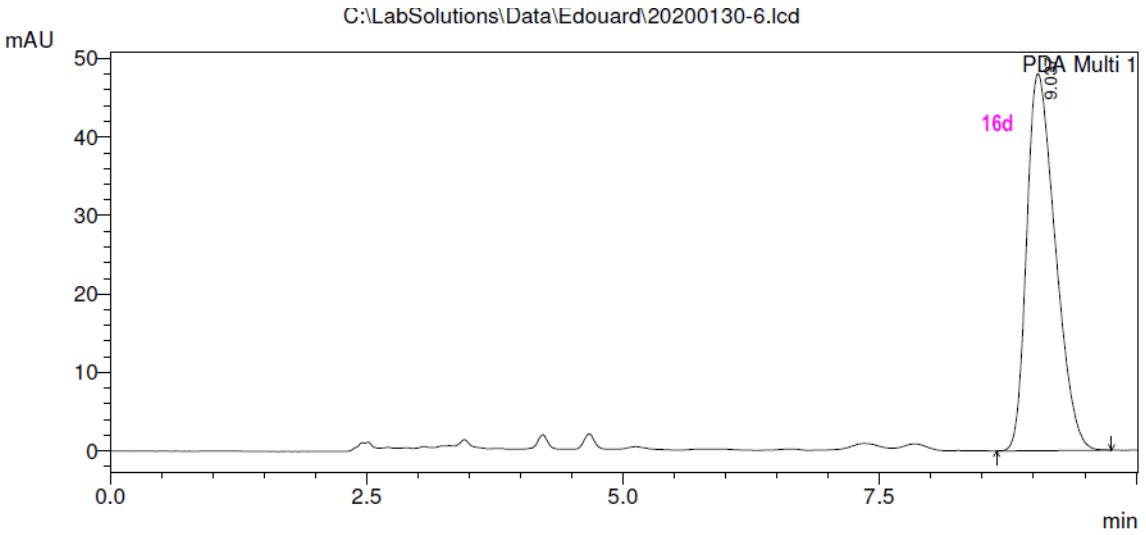
HPLC of rearrangement of 19c.

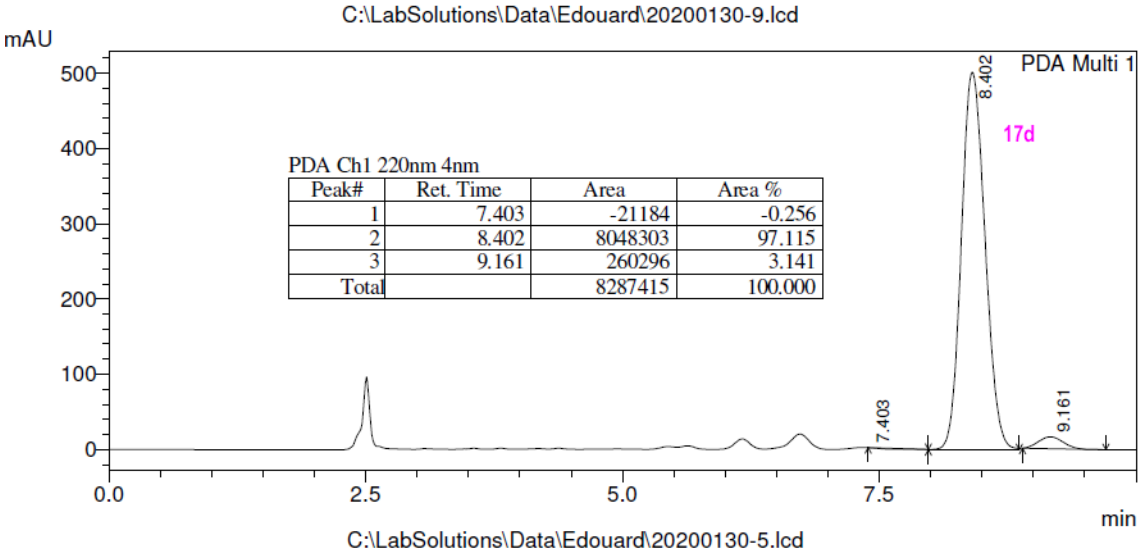
1st spectrum **19c**. 2nd spectrum rearrangement of **19c.** 3rd spectrum **17c**.



HPLC of rearrangement of 16d.

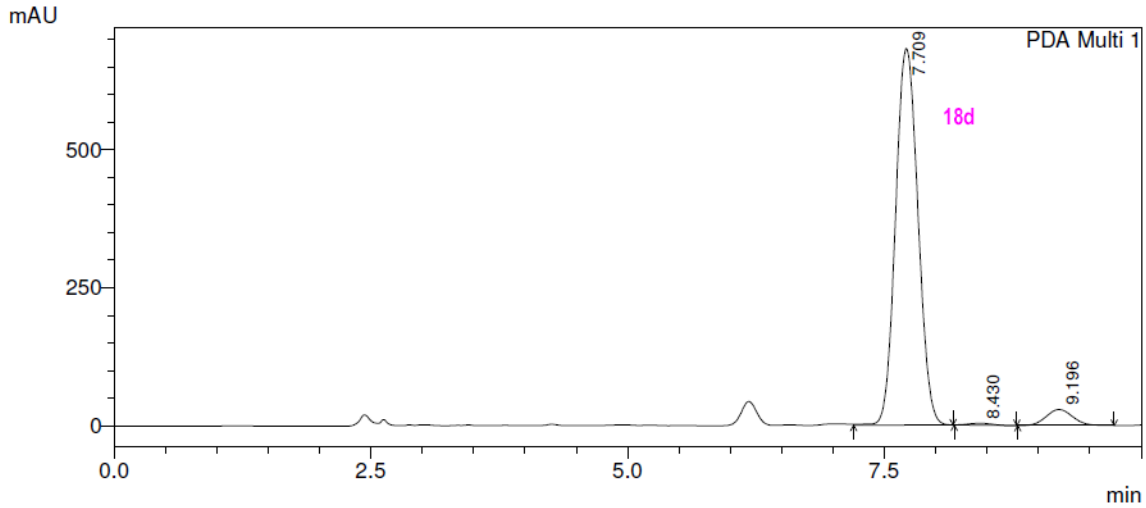
1st spectrum **16d**. 2nd spectrum rearrangement of **16d.** 3rd spectrum **18d**.





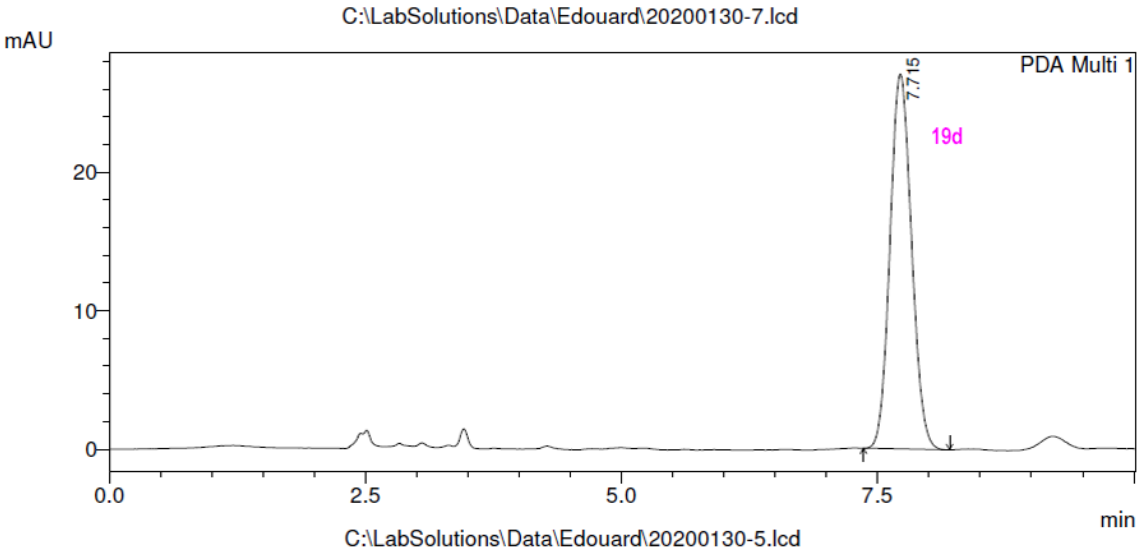
PDA Ch1 220nm 4nm

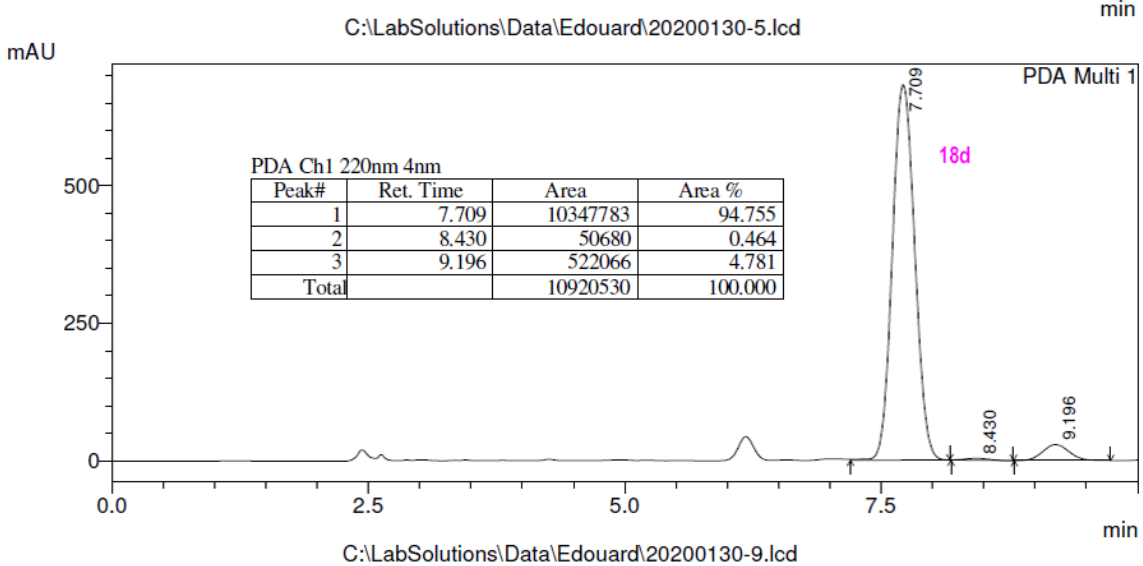
Peak#	Ret. Time	Area	Area %
1	7.403	-21184	-0.256
2	8.402	8048303	97.115
3	9.161	260296	3.141
Total		8287415	100.000



HPLC of rearrangement of 19d.

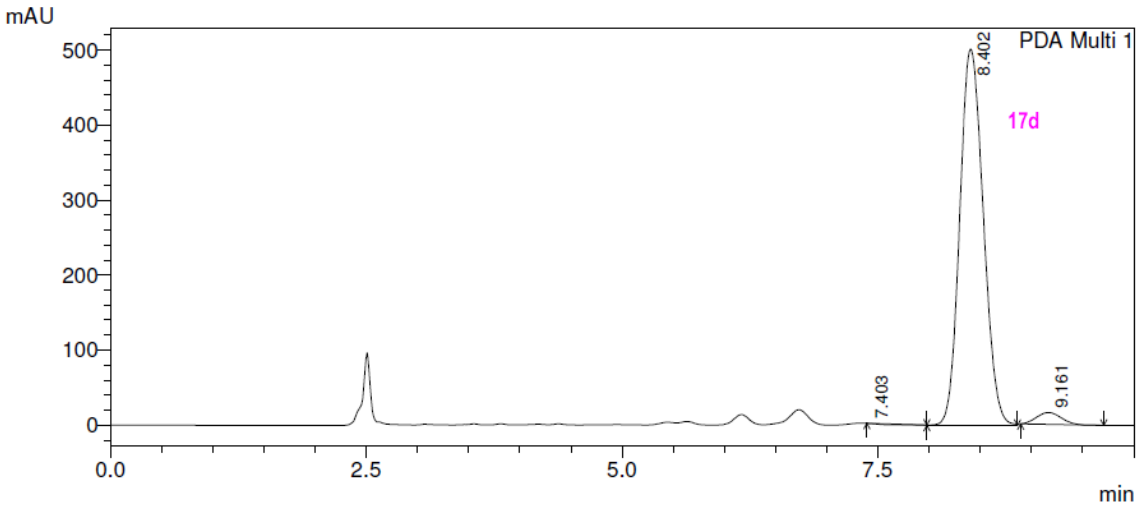
1st spectrum **19d**. 2nd spectrum rearrangement of **19d.** 3rd spectrum **17d**.





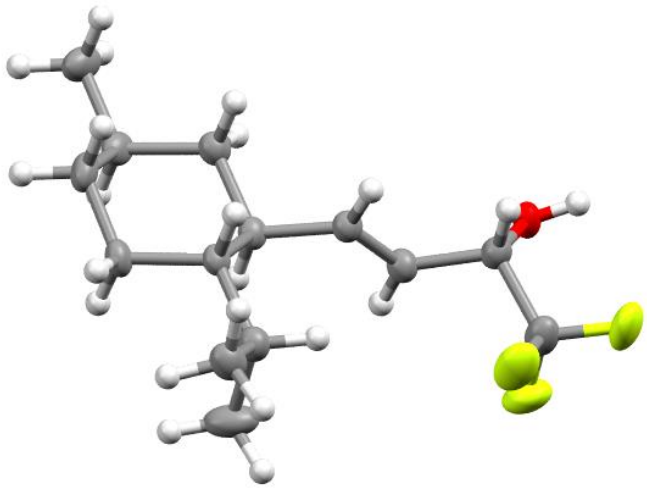
PDA Ch1 220nm 4nm

Peak#	Ret. Time	Area	Area %
1	7.709	10347783	94.755
2	8.430	50680	0.464
3	9.196	522066	4.781
Total		10920530	100.000



X-Ray data

(*E*)-(1*S*)-4-(*p*-Menthan-3-yl)-1-trifluoromethylprop-2-en-1-ol (17i)
Sample **ECD_4_12_P1.**
Deposition number : CCDC 2290613



A Needle-like specimen of $C_{14}H_{23}F_3O$, approximate dimensions 0.060 mm x 0.110 mm x 0.700 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured (λ = 1.54178 Å).

The total exposure time was 4.80 hours. The frames were integrated with the Bruker SAINT software package using a wide-frame algorithm. The integration of the data using a trigonal unit cell yielded a total of 14386 reflections to a maximum θ angle of 70.86° (0.82 Å resolution), of which 2648 were independent (average redundancy 5.433, completeness = 98.0%, R_{int} = 10.20%, R_{sig} = 8.16%) and 2237 (84.48%) were greater than $2\sigma(F^2)$. The final cell constants of a = 14.3818(2) Å, b = 14.3818(2) Å, c = 6.23960(10) Å, volume = 1117.67(4) $Å^3$, are based upon the refinement of the XYZ-centroids of 9751 reflections above 20 σ(I) with 7.097° < 2θ < 141.5°. Data were corrected for absorption effects using the Multi-Scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.621. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.4680 and 0.7530.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group P 31, with Z = 3 for the formula unit, $C_{14}H_{23}F_3O$. The final anisotropic full-matrix least-squares refinement on F^2 with 167 variables converged at R1 = 5.11%, for the observed data and wR2 = 13.75% for all data. The goodness-of-fit was 1.061. The largest peak in the final difference electron density synthesis was 0.215 $e^-/Å^3$ and the largest hole was -0.209 $e^-/Å^3$ with an RMS deviation of 0.049 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.178 g/cm^3 and F(000), 426 e^-.

Table 1. Sample and crystal data.

Identification code	Spino_ECD_4_12_P1	
Chemical formula	$C_{14}H_{23}F_3O$	
Formula weight	264.32 g/mol	
Temperature	173(2) K	
Wavelength	1.54178 Å	
Crystal size	0.060 x 0.110 x 0.700 mm	
Crystal system	trigonal	
Space group	P 31	
Unit cell dimensions	a = 14.3818(2) Å	α = 90°
	b = 14.3818(2) Å	β = 90°
	c = 6.23960(10) Å	γ = 120°
Volume	1117.67(4) $Å^3$	
Z	3	
Density (calculated)	1.178 g/cm^3	
Absorption coefficient	0.824 mm^{-1}	
F(000)	426	

Table 2. Data collection and structure refinement.

Theta range for data collection	3.55 to 70.86°
Index ranges	-17<=h<=17, -15<=k<=17, -6<=l<=7
Reflections collected	14386
Independent reflections	2648 [R(int) = 0.1020]
Coverage of independent reflections	98.0%
Absorption correction	Multi-Scan
Max. and min. transmission	0.7530 and 0.4680
Structure solution technique	direct methods
Structure solution program	XT, VERSION 2014/5
Refinement method	Full-matrix least-squares on F^2
Refinement program	SHELXL-2018/3 (Sheldrick, 2018)
Function minimized	$\Sigma\ w(F_o^2 - F_c^2)^2$
Data / restraints / parameters	2648 / 1 / 167
Goodness-of-fit on F^2	1.061
Final R indices	2237 data; I>2σ(I) R1 = 0.0511, wR2 = 0.1292
	all data R1 = 0.0633, wR2 = 0.1375
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0722P)^2]$ where $P=(F_o^2+2F_c^2)/3$
Absolute structure parameter	0.13(16)
Largest diff. peak and hole	0.215 and -0.209 eÅ^{-3}
R.M.S. deviation from mean	0.049 eÅ^{-3}

Table 3. Atomic coordinates and equivalent isotropic atomic displacement parameters ($Å^2$).

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x/a	y/b	z/c	U(eq)
C1	0.2180(3)	0.3149(3)	0.5275(5)	0.0228(7)
C2	0.3209(3)	0.3188(3)	0.6146(5)	0.0235(7)
C3	0.2936(3)	0.2184(3)	0.7444(5)	0.0281(8)
C4	0.2150(3)	0.1982(3)	0.9267(5)	0.0294(8)
C5	0.1137(3)	0.1910(3)	0.8348(6)	0.0329(8)
C6	0.1388(3)	0.2924(3)	0.7119(5)	0.0295(8)
C7	0.2410(3)	0.4134(3)	0.3925(6)	0.0308(8)
C8	0.1408(4)	0.3996(3)	0.2806(6)	0.0350(9)
C9	0.2940(4)	0.5183(4)	0.5198(9)	0.0512(13)
C10	0.1905(4)	0.0986(3)	0.0559(6)	0.0416(10)
C11	0.3951(3)	0.3305(3)	0.4358(5)	0.0249(7)
C12	0.4978(3)	0.4056(3)	0.4172(5)	0.0271(8)
C13	0.5657(3)	0.4082(3)	0.2341(5)	0.0264(8)
C14	0.6075(3)	0.5114(3)	0.1052(7)	0.0357(9)
F1	0.5263(2)	0.5188(2)	0.0201(5)	0.0558(8)
F2	0.6649(2)	0.5991(2)	0.2255(5)	0.0552(7)
F3	0.6704(2)	0.5167(2)	0.9437(4)	0.0541(8)
O1	0.65421(19)	0.4008(2)	0.3155(4)	0.0279(6)

Table 4. Bond lengths (Å).

C1-C7	1.535(5)	C1-C6	1.536(5)
C1-C2	1.551(5)	C1-H1	1.0
C2-C11	1.493(5)	C2-C3	1.525(5)
C2-H2	1.0	C3-C4	1.526(5)
C3-H3A	0.99	C3-H3B	0.99
C4-C5	1.521(5)	C4-C10	1.524(5)
C4-H4	1.0	C5-C6	1.522(6)
C5-H5A	0.99	C5-H5B	0.99
C6-H6A	0.99	C6-H6B	0.99
C7-C8	1.522(5)	C7-C9	1.529(6)
C7-H7	1.0	C8-H8A	0.98
C8-H8B	0.98	C8-H8C	0.98
C9-H9A	0.98	C9-H9B	0.98
C9-H9C	0.98	C10-H10A	0.98
C10-H10B	0.98	C10-H10C	0.98
C11-C12	1.329(5)	C11-H11	0.95
C12-C13	1.492(5)	C12-H12	0.95
C13-O1	1.422(4)	C13-C14	1.523(5)
C13-H13	1.0	C14-F3	1.331(5)
C14-F1	1.335(5)	C14-F2	1.340(6)
O1-H14	0.84		

Table 5. Bond angles (°).

C7-C1-C6	113.6(3)	C7-C1-C2	113.5(3)
C6-C1-C2	109.8(3)	C7-C1-H1	106.5
C6-C1-H1	106.5	C2-C1-H1	106.5
C11-C2-C3	109.6(3)	C11-C2-C1	110.9(3)
C3-C2-C1	111.2(3)	C11-C2-H2	108.3
C3-C2-H2	108.3	C1-C2-H2	108.3
C2-C3-C4	113.1(3)	C2-C3-H3A	109.0
C4-C3-H3A	109.0	C2-C3-H3B	109.0
C4-C3-H3B	109.0	H3A-C3-H3B	107.8
C5-C4-C10	111.9(3)	C5-C4-C3	109.0(3)
C10-C4-C3	112.0(3)	C5-C4-H4	107.9
C10-C4-H4	107.9	C3-C4-H4	107.9
C4-C5-C6	111.4(3)	C4-C5-H5A	109.4
C6-C5-H5A	109.4	C4-C5-H5B	109.4
C6-C5-H5B	109.4	H5A-C5-H5B	108.0
C5-C6-C1	112.0(3)	C5-C6-H6A	109.2
C1-C6-H6A	109.2	C5-C6-H6B	109.2
C1-C6-H6B	109.2	H6A-C6-H6B	107.9
C8-C7-C9	109.8(3)	C8-C7-C1	112.3(3)
C9-C7-C1	113.3(3)	C8-C7-H7	107.0
C9-C7-H7	107.0	C1-C7-H7	107.0
C7-C8-H8A	109.5	C7-C8-H8B	109.5
H8A-C8-H8B	109.5	C7-C8-H8C	109.5
H8A-C8-H8C	109.5	H8B-C8-H8C	109.5
C7-C9-H9A	109.5	C7-C9-H9B	109.5
H9A-C9-H9B	109.5	C7-C9-H9C	109.5
H9A-C9-H9C	109.5	H9B-C9-H9C	109.5
C4-C10-H10A	109.5	C4-C10-H10B	109.5
H10A-C10-H10B	109.5	C4-C10-H10C	109.5
H10A-C10-H10C	109.5	H10B-C10-H10C	109.5
C12-C11-C2	126.7(3)	C12-C11-H11	116.6
C2-C11-H11	116.6	C11-C12-C13	122.3(3)
C11-C12-H12	118.8	C13-C12-H12	118.8
O1-C13-C12	108.9(3)	O1-C13-C14	109.2(3)
C12-C13-C14	111.3(3)	O1-C13-H13	109.1

C12-C13-H13	109.1	C14-C13-H13	109.1
F3-C14-F1	106.9(3)	F3-C14-F2	106.8(3)
F1-C14-F2	107.5(3)	F3-C14-C13	112.4(3)
F1-C14-C13	110.7(3)	F2-C14-C13	112.3(3)
C13-O1-H14	109.5		

Table 6. Torsion angles (°).

C7-C1-C2-C11	-56.9(4)	C6-C1-C2-C11	174.8(3)
C7-C1-C2-C3	-179.1(3)	C6-C1-C2-C3	52.5(4)
C11-C2-C3-C4	-178.1(3)	C1-C2-C3-C4	-55.1(4)
C2-C3-C4-C5	56.6(4)	C2-C3-C4-C10	-179.1(3)
C10-C4-C5-C6	178.2(3)	C3-C4-C5-C6	-57.4(4)
C4-C5-C6-C1	58.3(4)	C7-C1-C6-C5	177.0(3)
C2-C1-C6-C5	-54.7(4)	C6-C1-C7-C8	-62.6(4)
C2-C1-C7-C8	171.0(3)	C6-C1-C7-C9	62.5(4)
C2-C1-C7-C9	-63.9(4)	C3-C2-C11-C12	-111.0(4)
C1-C2-C11-C12	125.8(4)	C2-C11-C12-C13	178.2(3)
C11-C12-C13-O1	-120.6(4)	C11-C12-C13-C14	118.9(4)
O1-C13-C14-F3	58.3(4)	C12-C13-C14-F3	178.6(3)
O1-C13-C14-F1	177.8(3)	C12-C13-C14-F1	-61.9(4)
O1-C13-C14-F2	-62.1(4)	C12-C13-C14-F2	58.2(4)

Table 7. Anisotropic atomic displacement parameters (Å^2)

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0257(17)	0.0265(17)	0.0157(14)	-0.0033(13)	-0.0011(13)	0.0127(14)
C2	0.0242(17)	0.0289(18)	0.0168(15)	-0.0031(13)	-0.0027(13)	0.0129(15)
C3	0.0319(19)	0.032(2)	0.0226(17)	-0.0017(14)	-0.0023(14)	0.0178(16)
C4	0.041(2)	0.0297(19)	0.0150(16)	-0.0025(13)	0.0000(14)	0.0152(17)
C5	0.0292(19)	0.037(2)	0.0230(16)	-0.0006(15)	0.0063(14)	0.0097(17)
C6	0.0302(19)	0.039(2)	0.0218(16)	-0.0024(15)	0.0031(14)	0.0195(17)
C7	0.031(2)	0.034(2)	0.0292(18)	0.0005(15)	-0.0001(15)	0.0179(17)
C8	0.044(2)	0.043(2)	0.0262(17)	-0.0004(16)	-0.0043(16)	0.028(2)
C9	0.056(3)	0.028(2)	0.063(3)	-0.0045(19)	-0.022(2)	0.017(2)
C10	0.062(3)	0.033(2)	0.0238(18)	0.0036(16)	0.0034(18)	0.020(2)
C11	0.0278(18)	0.0275(18)	0.0197(16)	-0.0007(13)	-0.0014(14)	0.0139(16)
C12	0.0255(18)	0.0313(19)	0.0252(17)	-0.0017(14)	-0.0014(14)	0.0148(16)
C13	0.0251(17)	0.0284(19)	0.0254(18)	0.0031(14)	-0.0002(14)	0.0132(16)
C14	0.034(2)	0.046(3)	0.0323(19)	0.0109(18)	0.0021(17)	0.024(2)
F1	0.0500(16)	0.0696(19)	0.0592(15)	0.0310(14)	0.0043(13)	0.0385(15)
F2	0.0542(17)	0.0303(13)	0.0718(18)	0.0108(12)	0.0029(14)	0.0142(12)
F3	0.0550(17)	0.0676(18)	0.0463(14)	0.0323(13)	0.0246(12)	0.0356(14)
O1	0.0273(13)	0.0384(15)	0.0233(12)	0.0051(11)	0.0036(10)	0.0205(11)

Table 8. Hydrogen atomic coordinates and isotropic atomic displacement parameters (Å^2) for Spino_ECD_4_12_P1.

	x/a	y/b	z/c	U(eq)
H1	0.1830	0.2515	0.4295	0.027
H2	0.3588	0.3826	0.7108	0.028
H3A	0.3606	0.2257	0.8049	0.034
H3B	0.2622	0.1554	0.6475	0.034
H4	0.2488	0.2614	1.0253	0.035
H5A	0.0632	0.1800	0.9528	0.039
H5B	0.0783	0.1283	0.7376	0.039
H6A	0.1696	0.3542	0.8117	0.035
H6B	0.0713	0.2846	0.6533	0.035
H7	0.2927	0.4205	0.2778	0.037
H8A	0.1039	0.3296	0.2091	0.053
H8B	0.1613	0.4567	0.1740	0.053
H8C	0.0927	0.4037	0.3866	0.053
H9A	0.3143	0.5788	0.4221	0.077
H9B	0.3582	0.5265	0.5920	0.077
H9C	0.2434	0.5167	0.6269	0.077
H10A	0.1596	0.0357	0.9613	0.062
H10B	0.1392	0.0879	1.1699	0.062
H10C	0.2570	0.1080	1.1196	0.062
H11	0.3658	0.2790	0.3238	0.03
H12	0.5290	0.4596	0.5243	0.032
H13	0.5221	0.3454	0.1383	0.032
H14	0.6858	0.3898	0.2139	0.042

Table 9. Hydrogen bond distances (Å) and angles (°) for Spino_ECD_4_12_P1.

	Donor-H	Acceptor-H	Donor-Acceptor	Angle
O1-H14···O1#1	0.84	1.95	2.788(3)	173.0

Symmetry transformations used to generate equivalent atoms:
#1 -x+1, y+1, z-1/3

