

Supplementary Information

Site-Selective Sequential Coupling Reactions Controlled by “Electrochemical Reaction Site Switching”: a Straightforward Approach to 1,4-Bis(diaryl)buta-1,3-diyne

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1. X-ray Crystallography

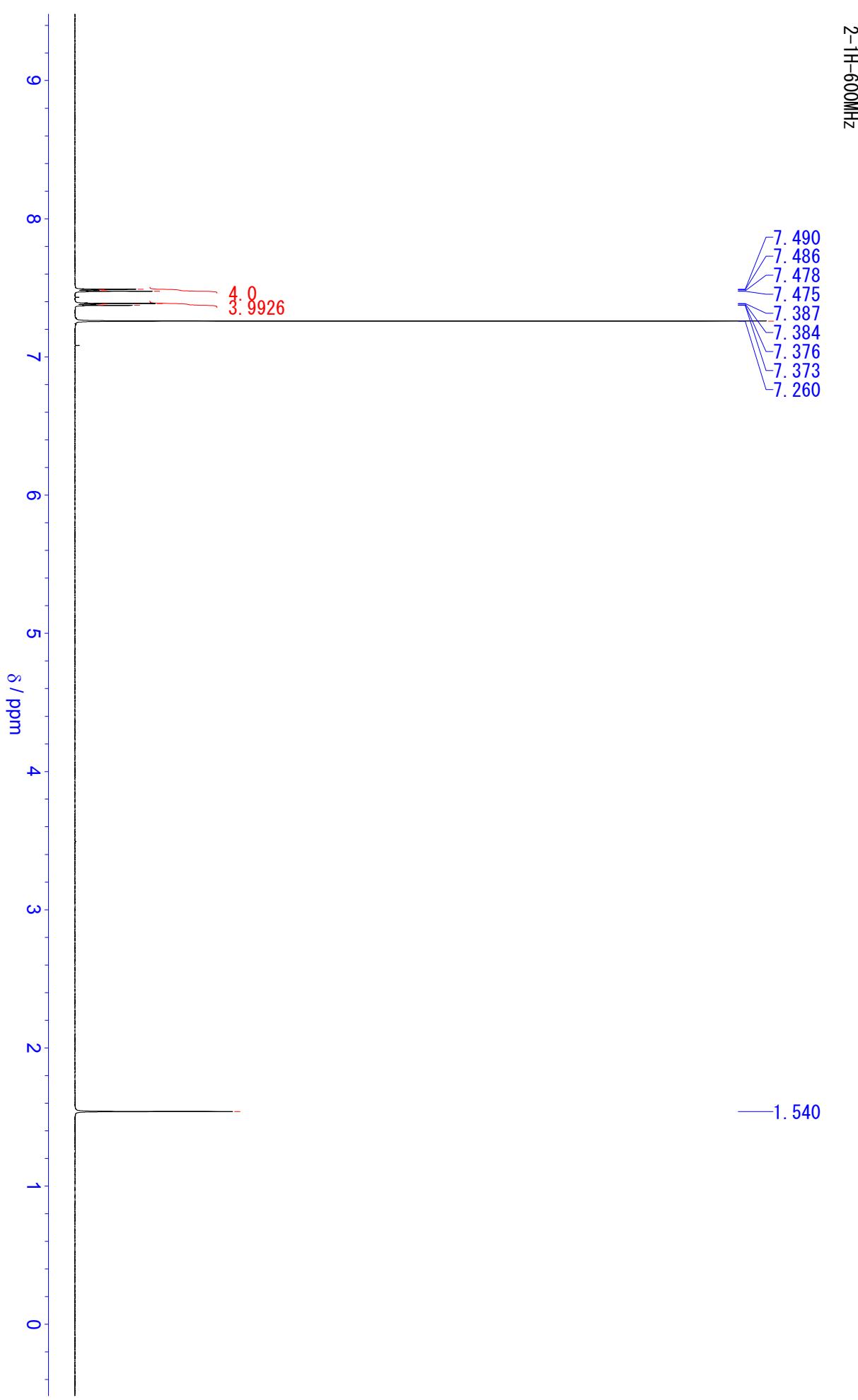
Details of the crystal data and a summary of the intensity data collection parameters for bis(diaryl)butadiyne **3g** are listed in Table S1. X-ray single crystal analysis was conducted with a Rigaku VariMax with Saturn. Graphite-monochromated Mo K α radiation ($\lambda = 0.71075 \text{ \AA}$) was used. The structures were solved by direct methods with (SHELXS-97)¹ and refined by full-matrix least-squares techniques against F^2 (SHELXL-97). The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were placed using AFIX instructions. In the subsequent refinement, the function $\Sigma w(F_o^2 - F_c^2)^2$ was minimized, where $|F_o|$ and $|F_c|$ are the observed and calculated structure factor amplitudes, respectively. The agreement indices are defined as $R_1 = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|$ and $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2 / \Sigma(wF_o^4)]^{1/2}$. All calculations were performed by using Yadokari-XG 2009² and illustrations were drawn by using ORTEP-3.

Table S1. Crystallographic data and structure refinement details for **3g**.

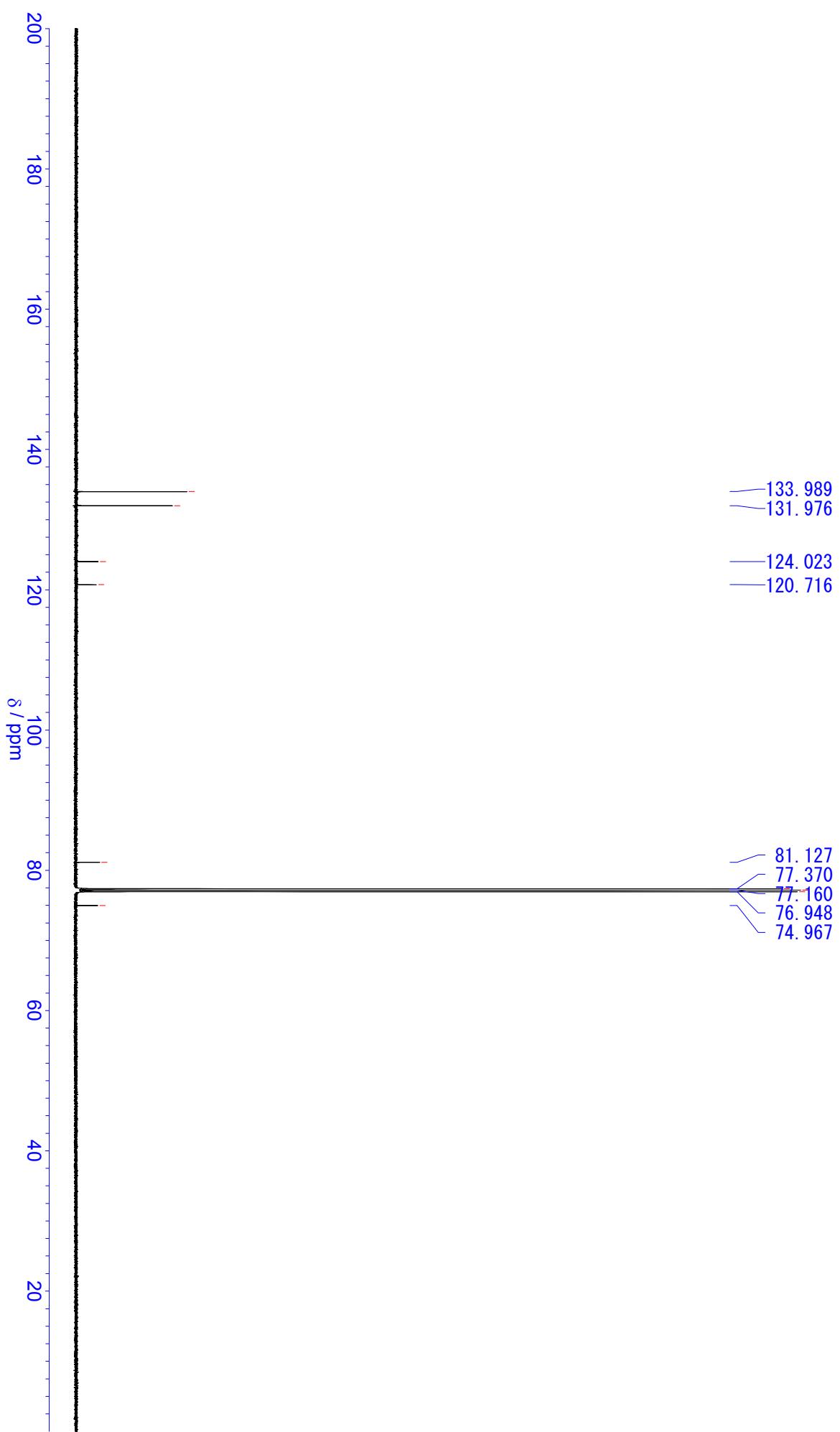
Empirical formula	C ₃₂ H ₂₂ O ₂		
Formula weight	438.50		
Temperature	93(2) K		
Wavelength	0.71075 Å		
Crystal system	Triclinic		
Space group	P-1 (#2)		
Unit cell dimensions	$a = 9.271(7) \text{ \AA}$	$\alpha = 90.570(11)^\circ$	
	$b = 9.707(7) \text{ \AA}$	$\beta = 97.203(5)^\circ$	
	$c = 20.928(14) \text{ \AA}$	$\gamma = 114.198(13)^\circ$	
Volume	1700(2) Å ³		
Z	3		
Density (calculated)	1.285 Mg/m ³		
Absorption coefficient	0.079 mm ⁻¹		
F(000)	690		
Crystal size	0.68 × 0.20 × 0.05 mm ³		
Theta range for data collection	3.12 to 27.47°		
Index ranges	-12≤h≤12, -12≤k≤12, -27≤l≤27		
Reflections collected	20748		
Independent reflections	7713 [R(int) = 0.0871]		
Completeness to theta = 27.47°	99.1%		
Max. and min. transmission	0.9961 and 0.9484		
Refinement method	Full-matrix least-squares on F^2		
Data / restraints / parameters	7713 / 71 / 483		
Goodness-of-fit on F^2	0.912		
Final R indices [I > 2sigma(I)]	$R_1 = 0.0581, wR_2 = 0.1153$		
R indices (all data)	$R_1 = 0.1239, wR_2 = 0.1405$		

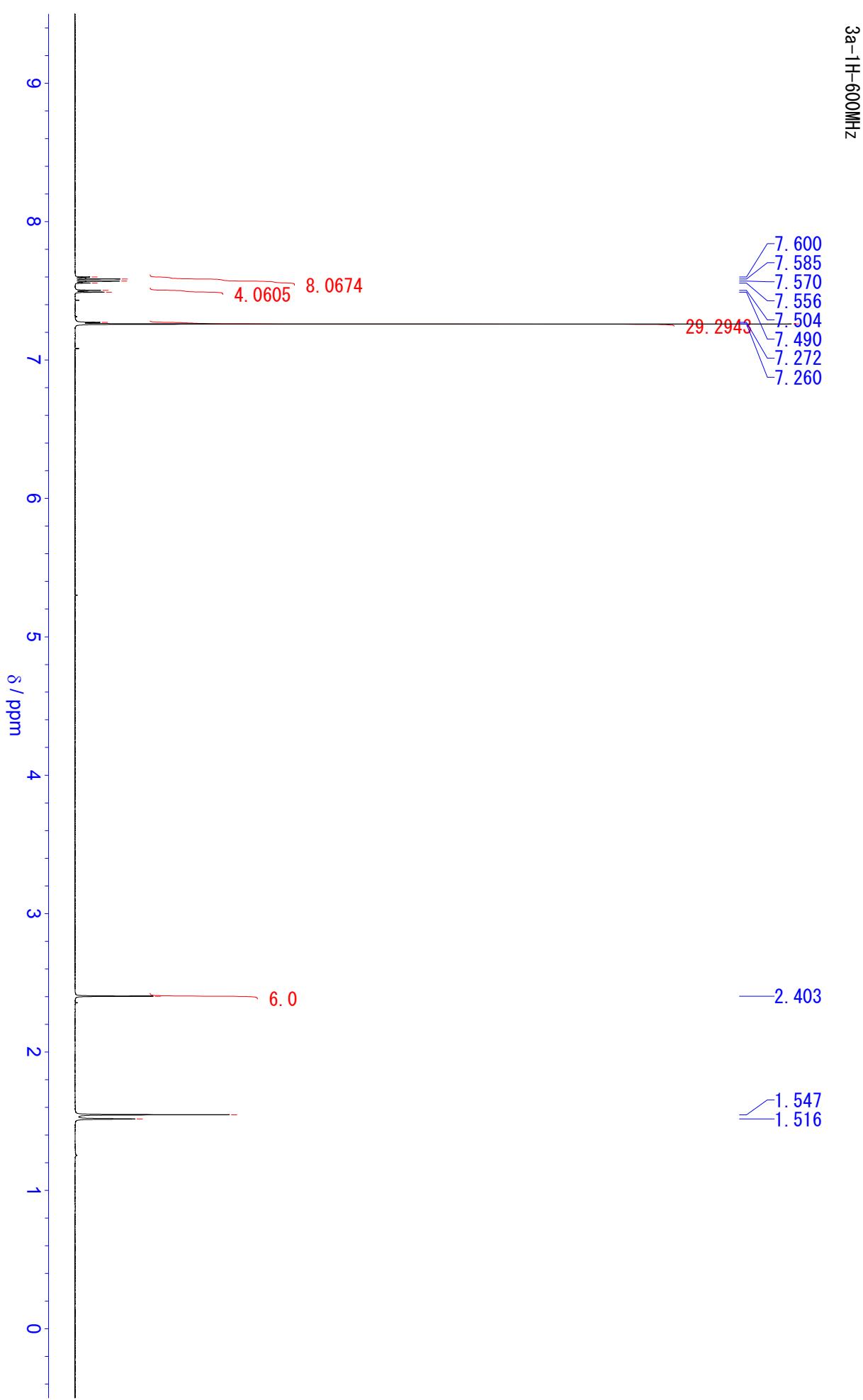
(1) SHELX, Program for the Solution of Crystal Structures (a) Sheldrick, G. M.; University of Göttingen: Göttingen, Germany, 1997. (b) Sheldrick, G.M. *Acta Cryst.* **2008**, A64, 112–122.

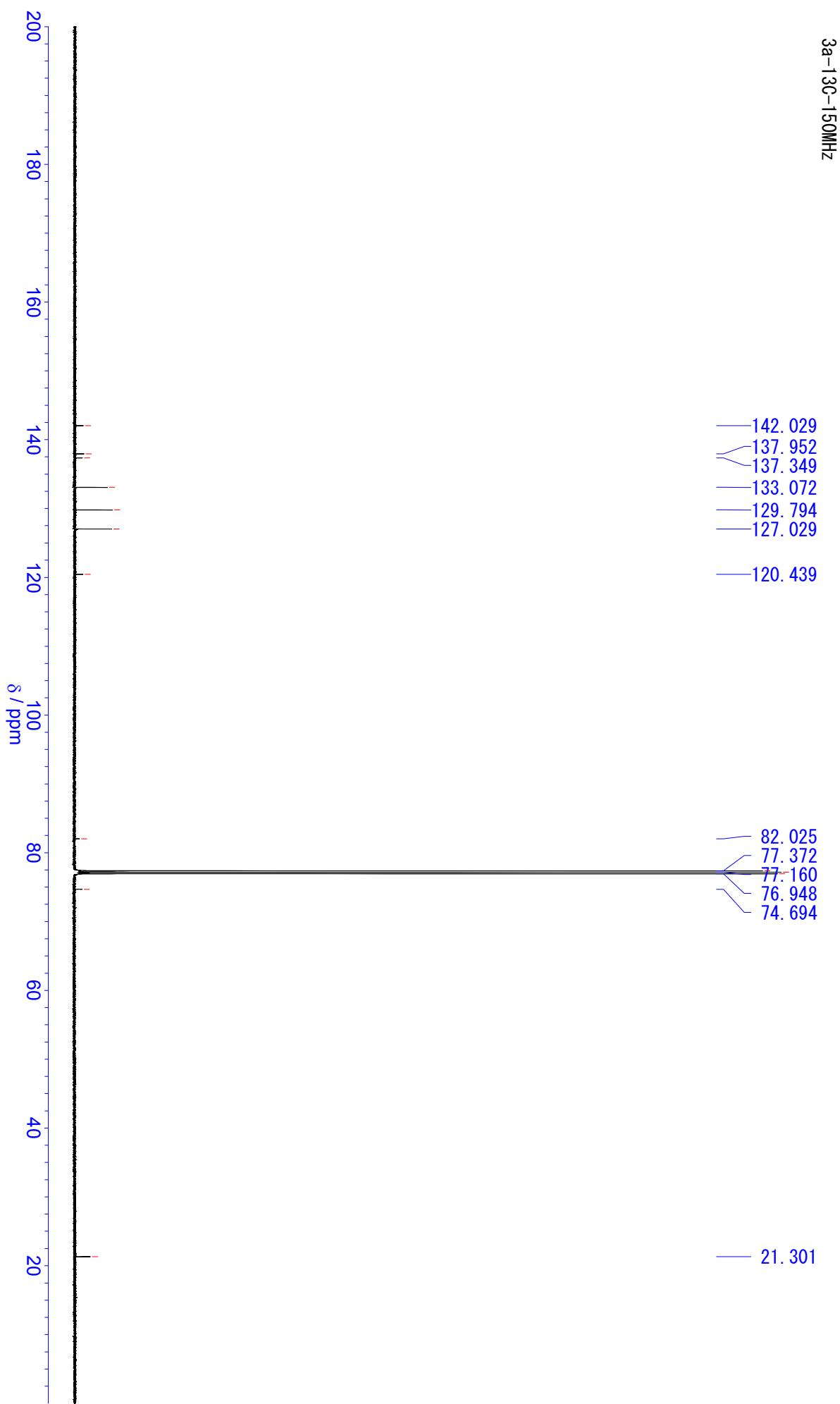
(2) Yadokari-XG, Software for Crystal Structure Analyses, K. Wakita (2001); Release of Software (Yadokari-XG 2009) for Crystal Structure Analyses, Kabuto, C.; Akine, S.; Nemoto, T.; Kwon, E. *J. Cryst. Soc. Jpn.* **2009**, 51, 218–224.

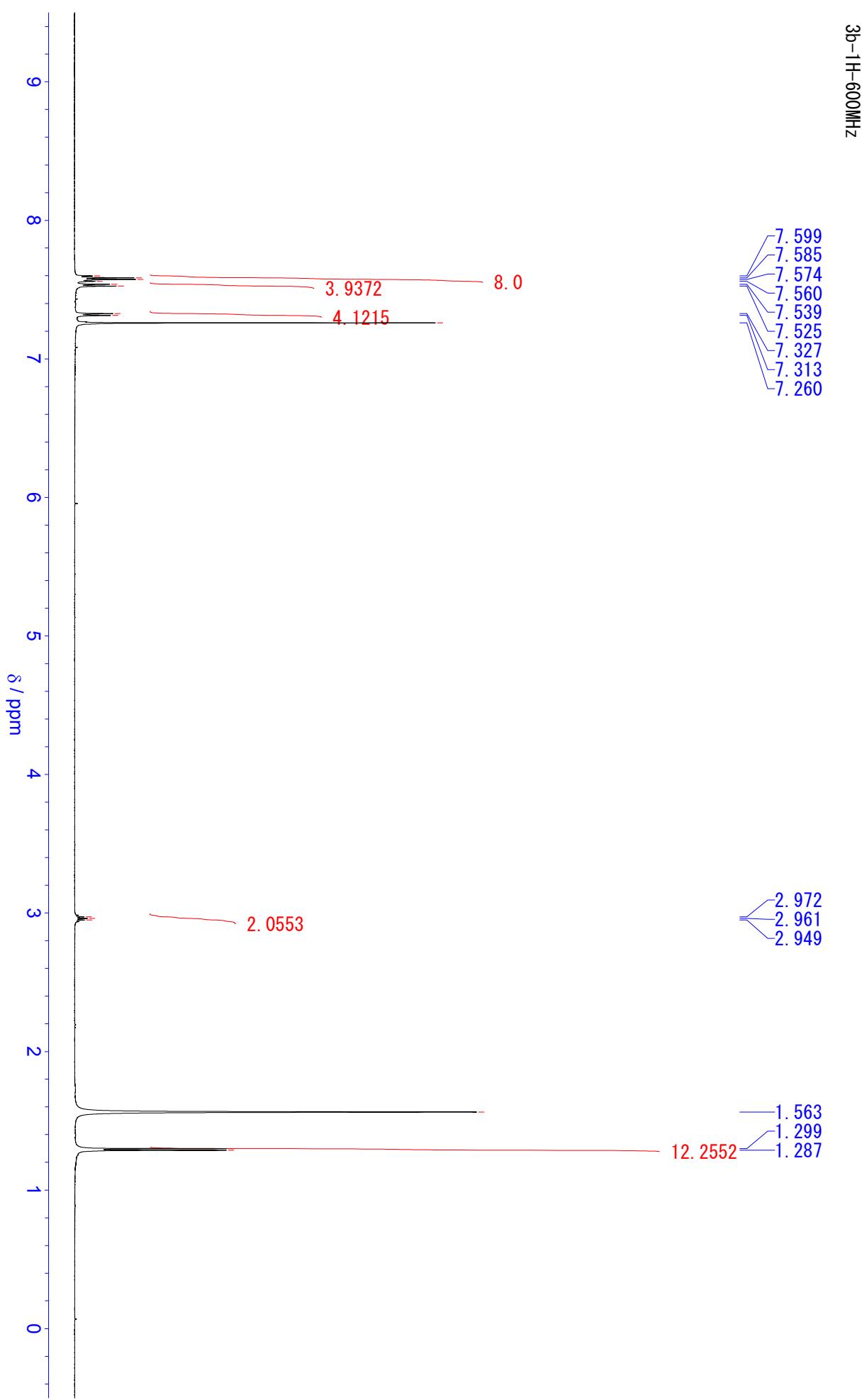


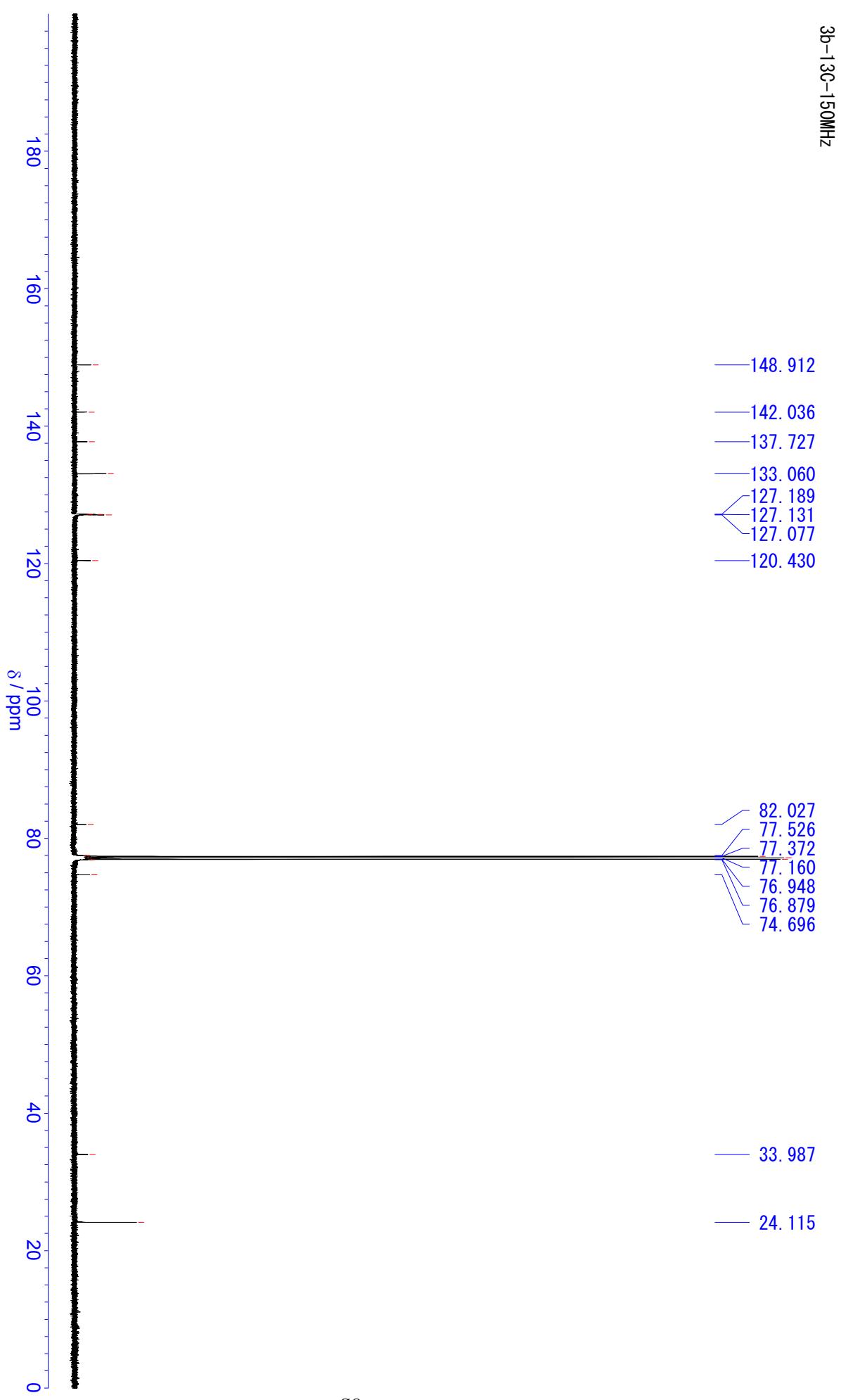
2-13C-150MHz

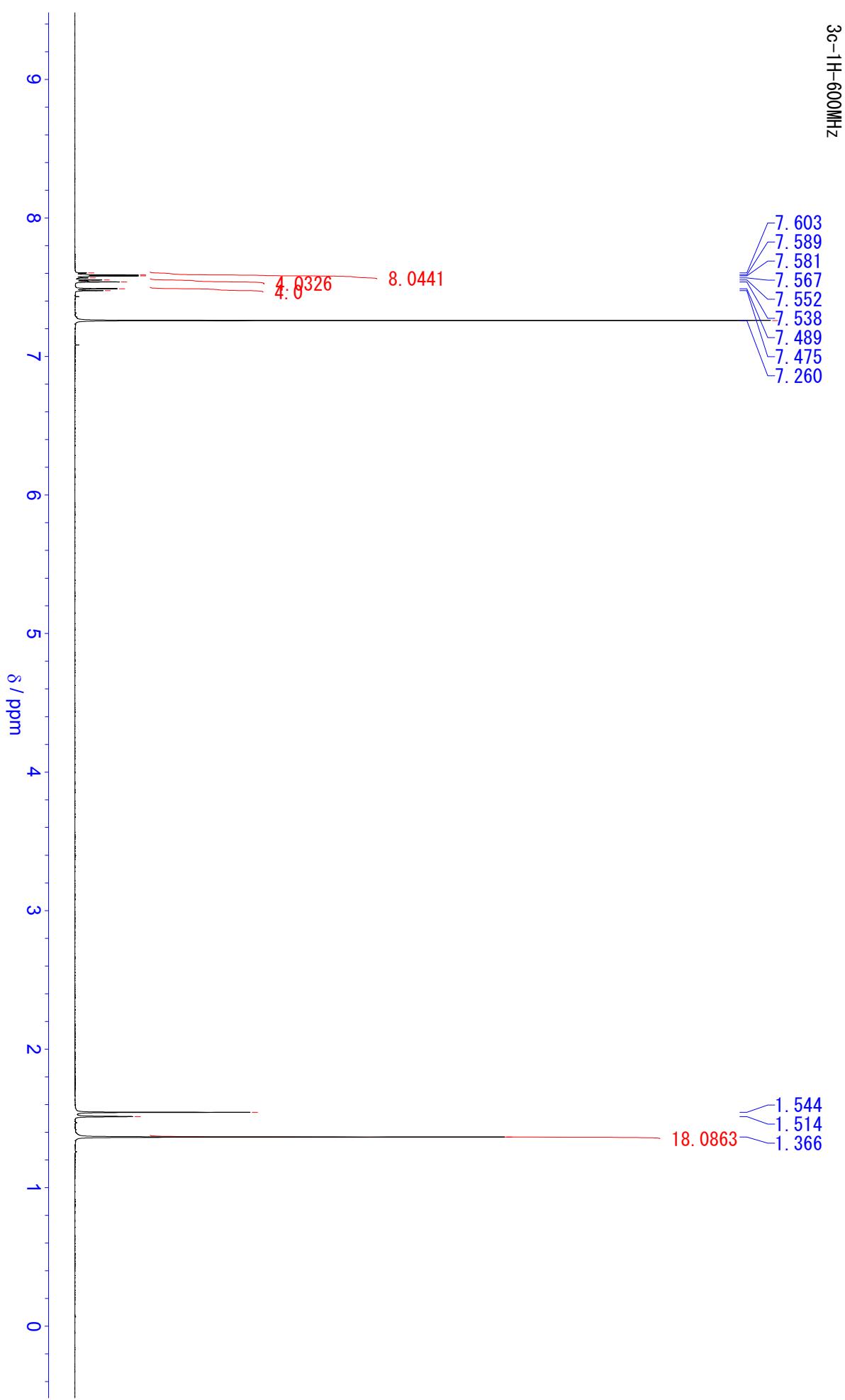


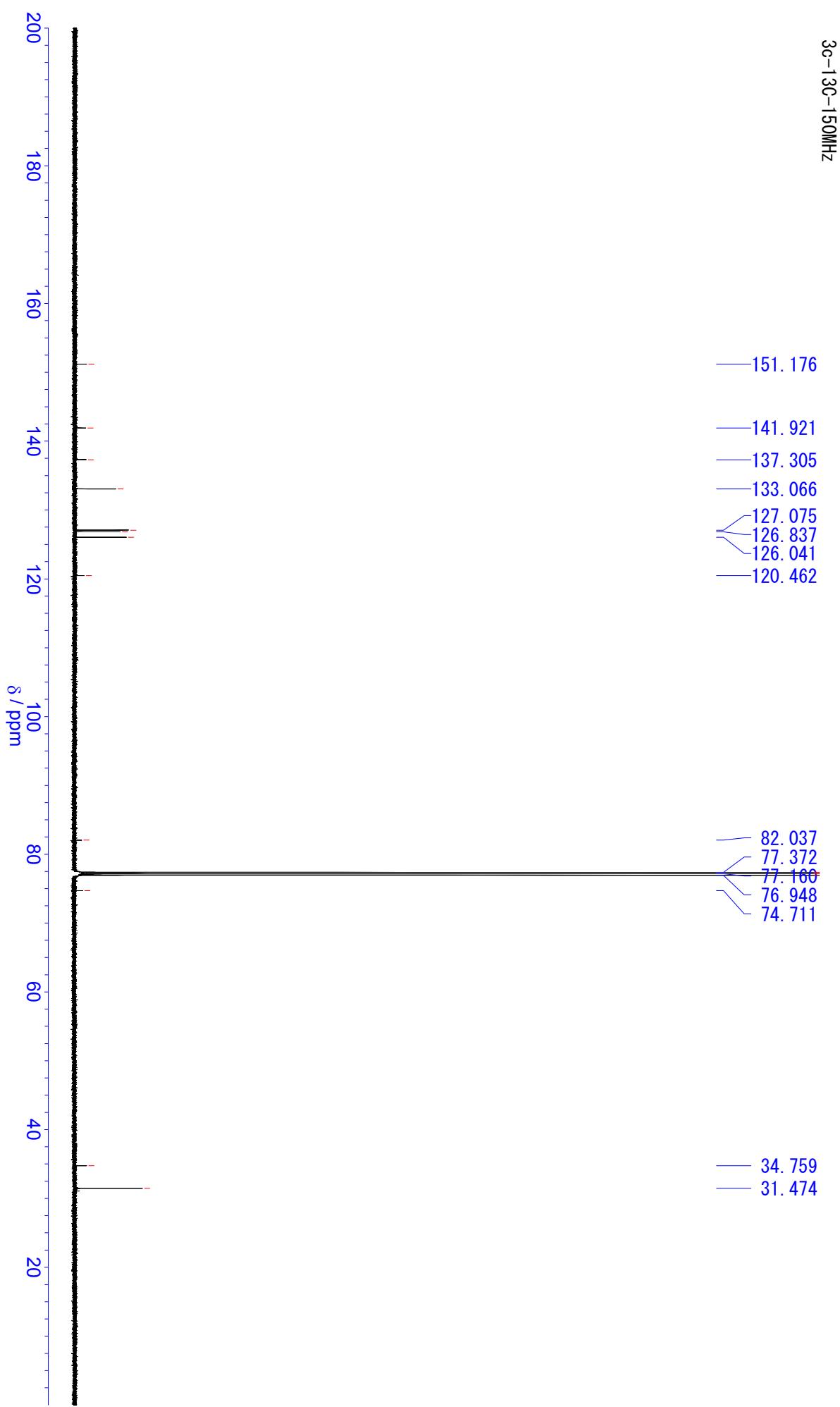


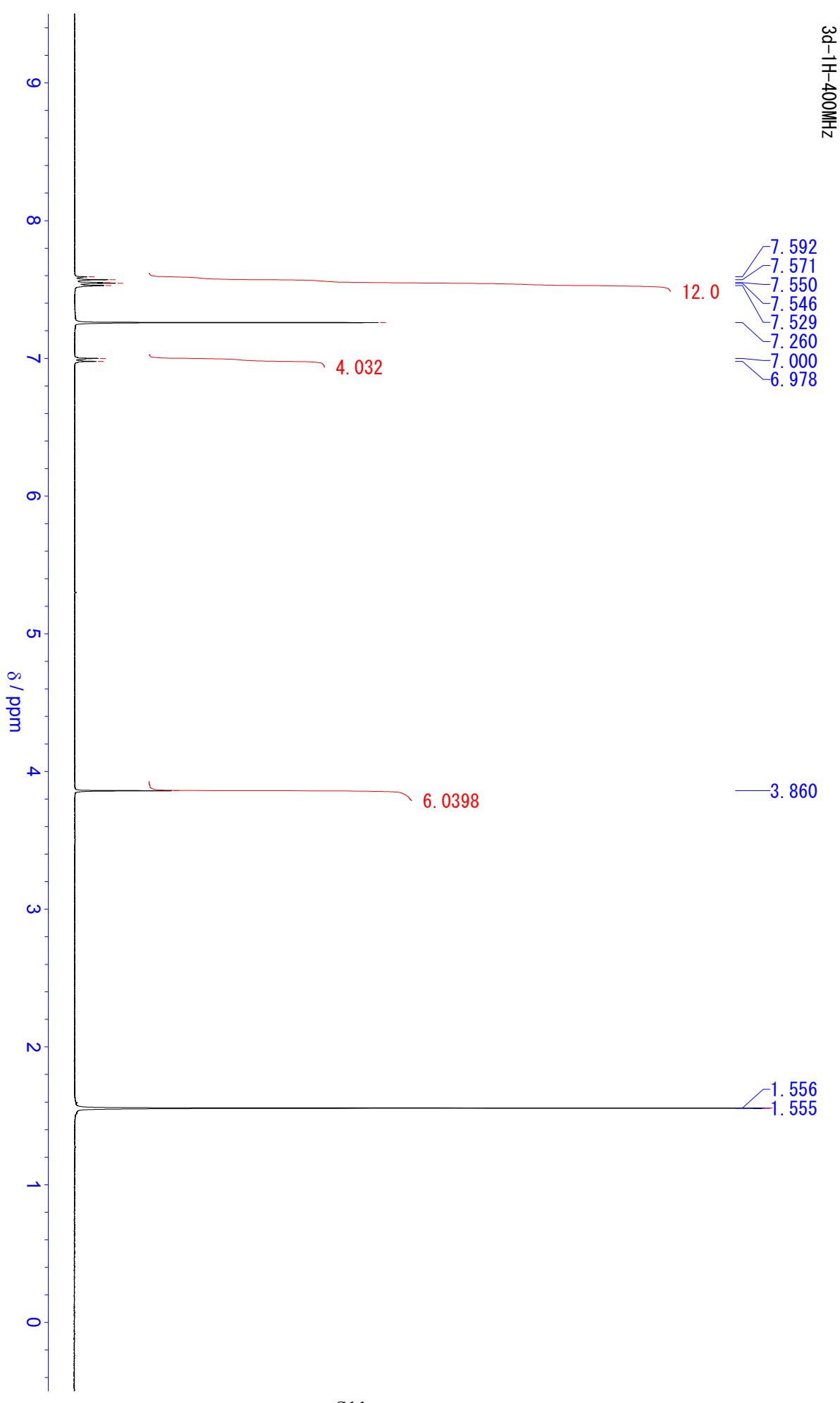




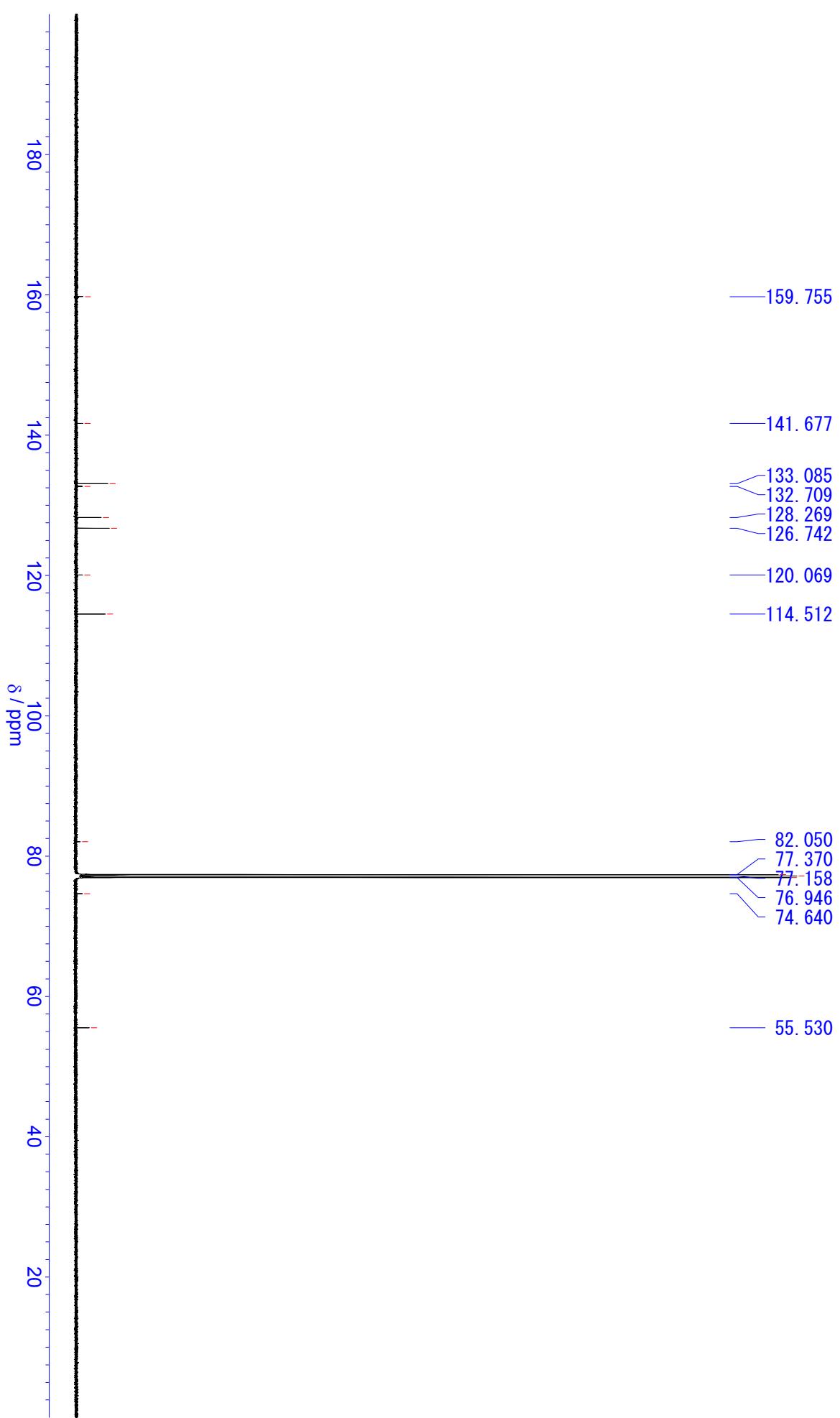


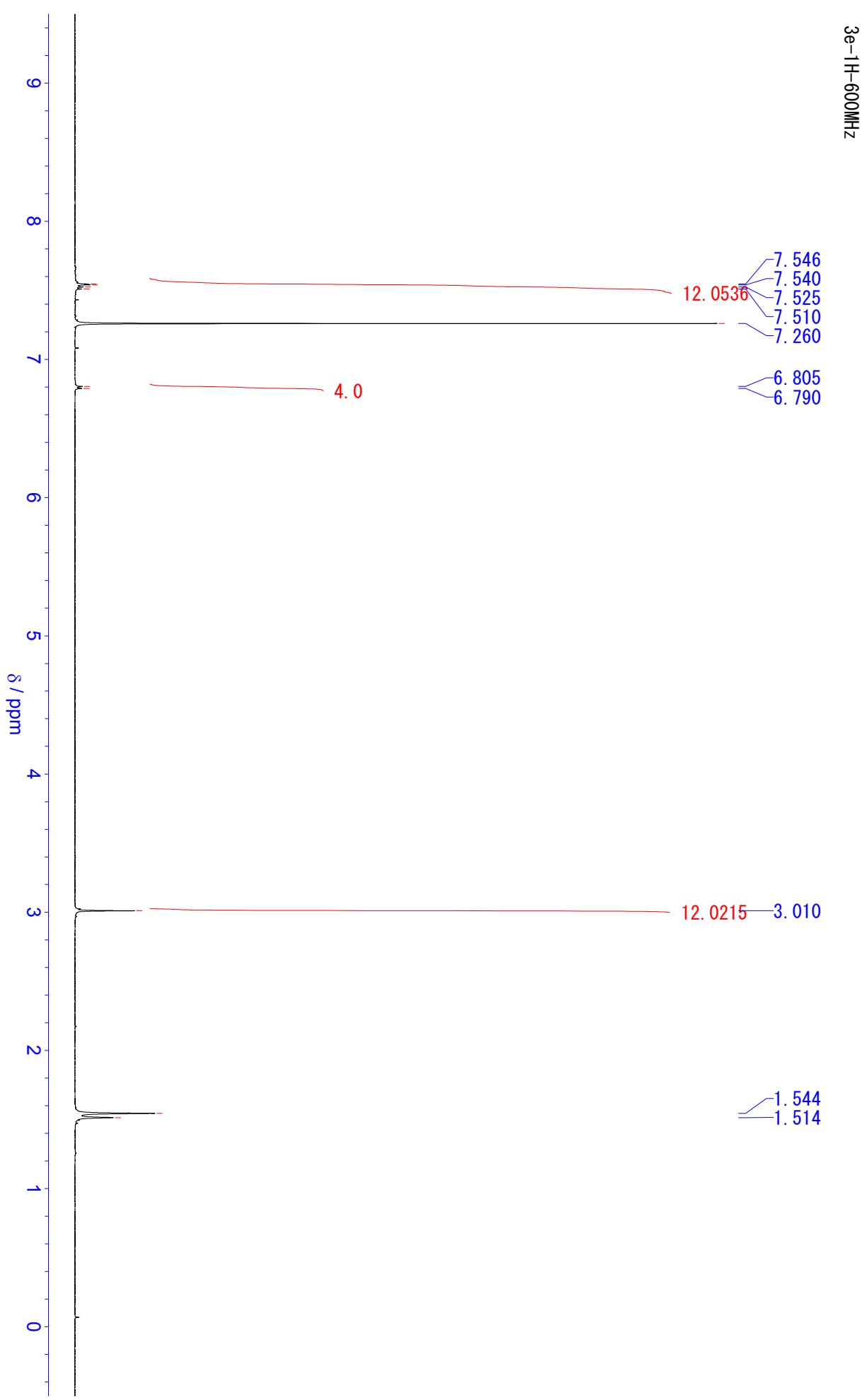


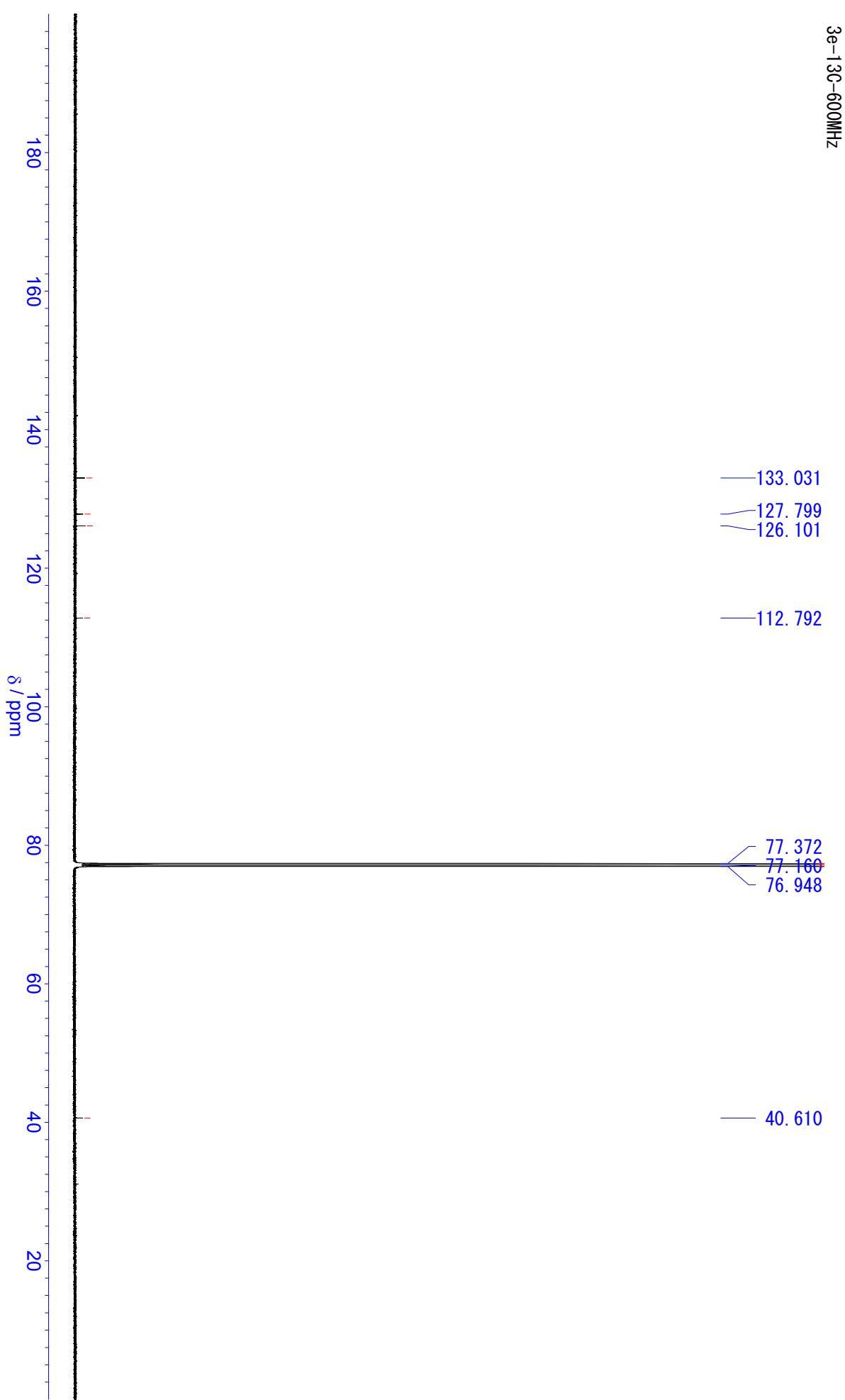


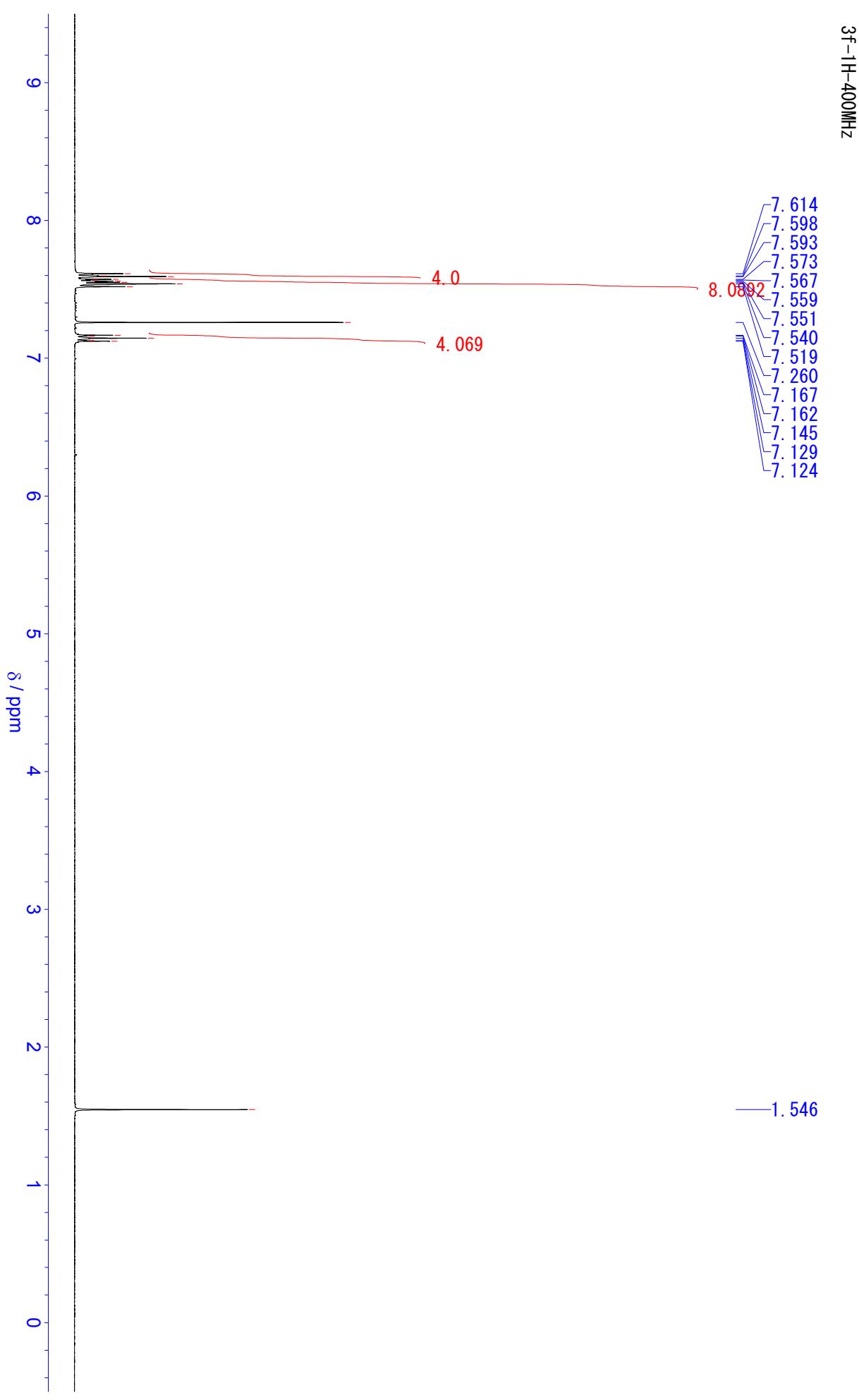


3d-¹³C-150MHz

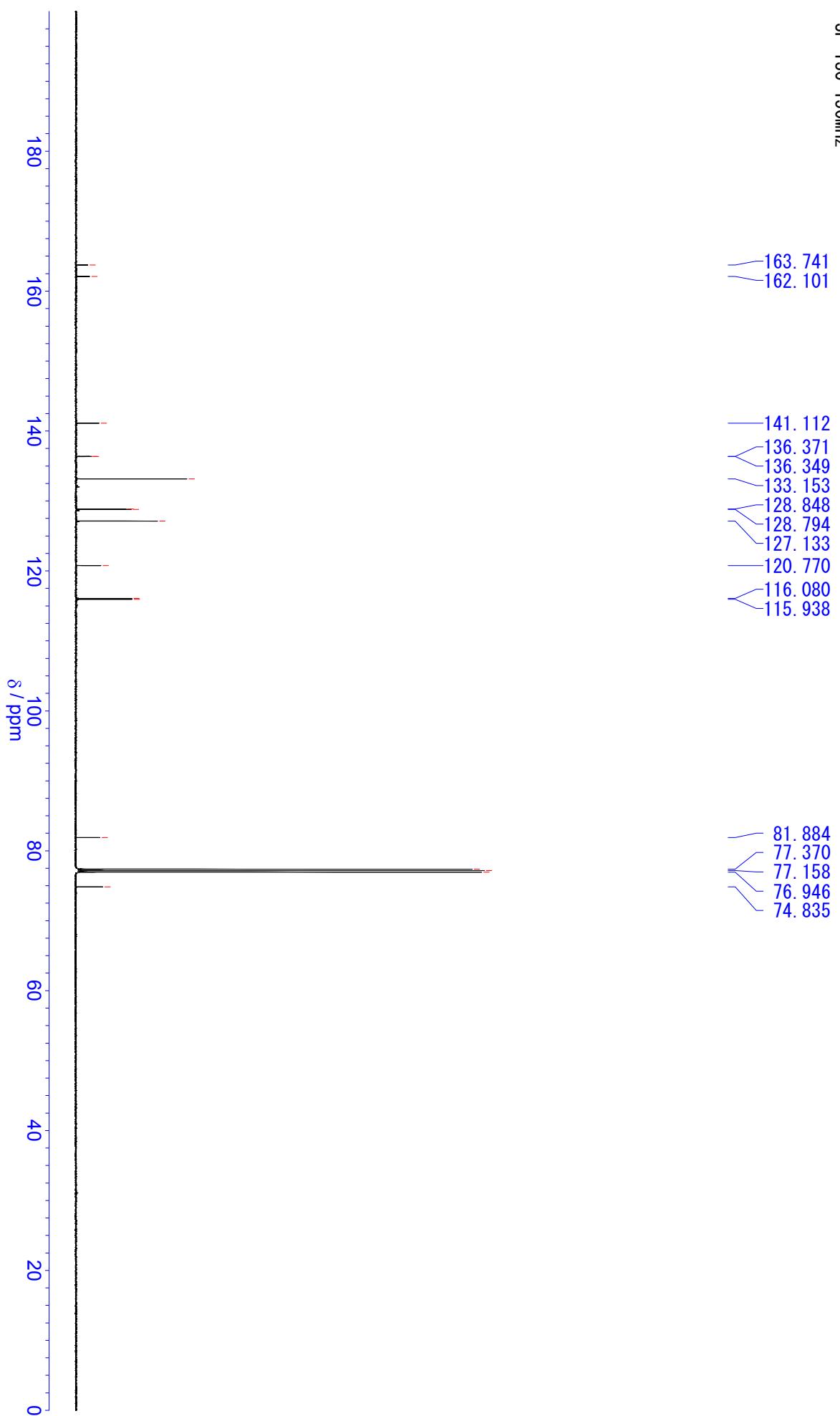


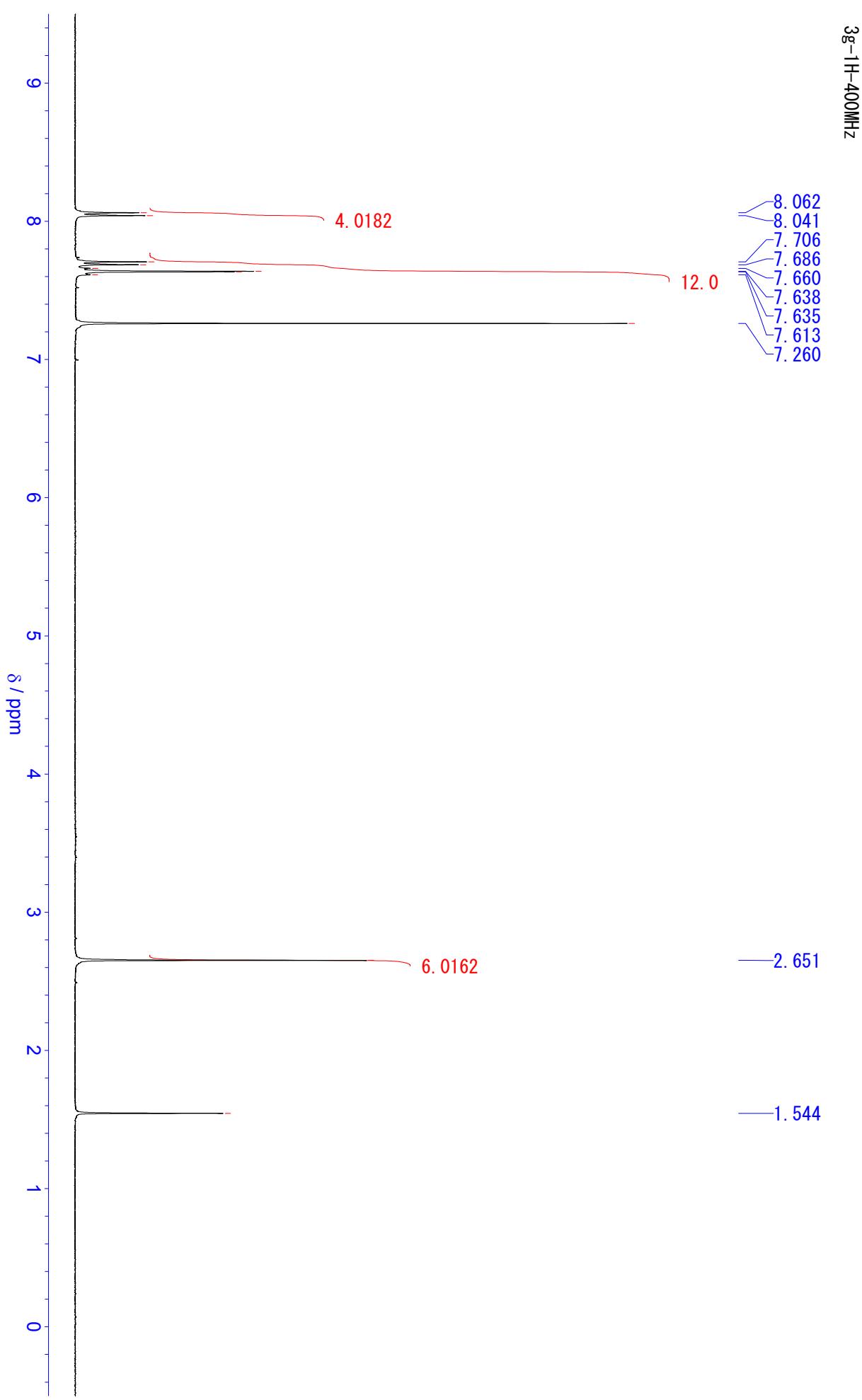


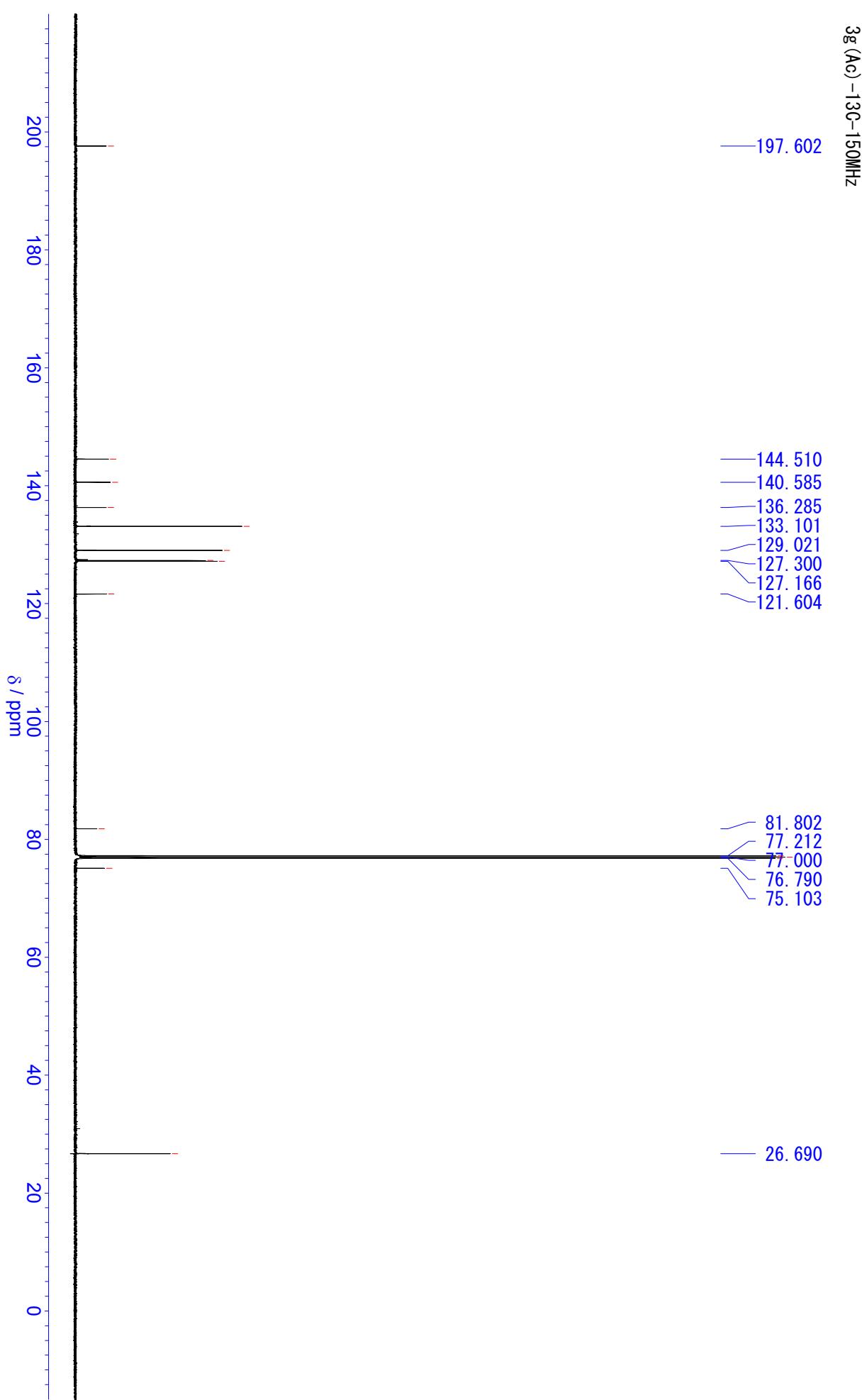


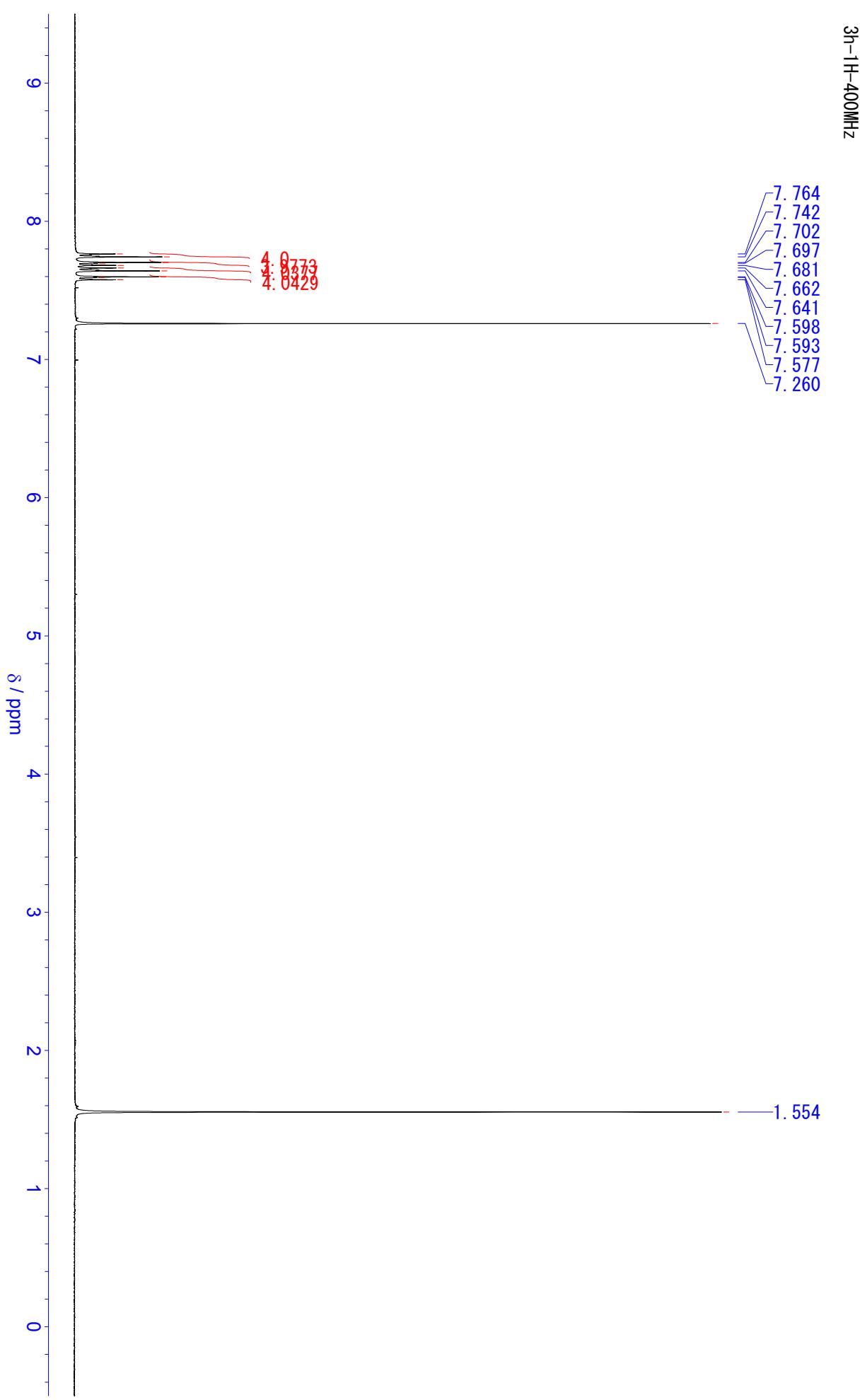


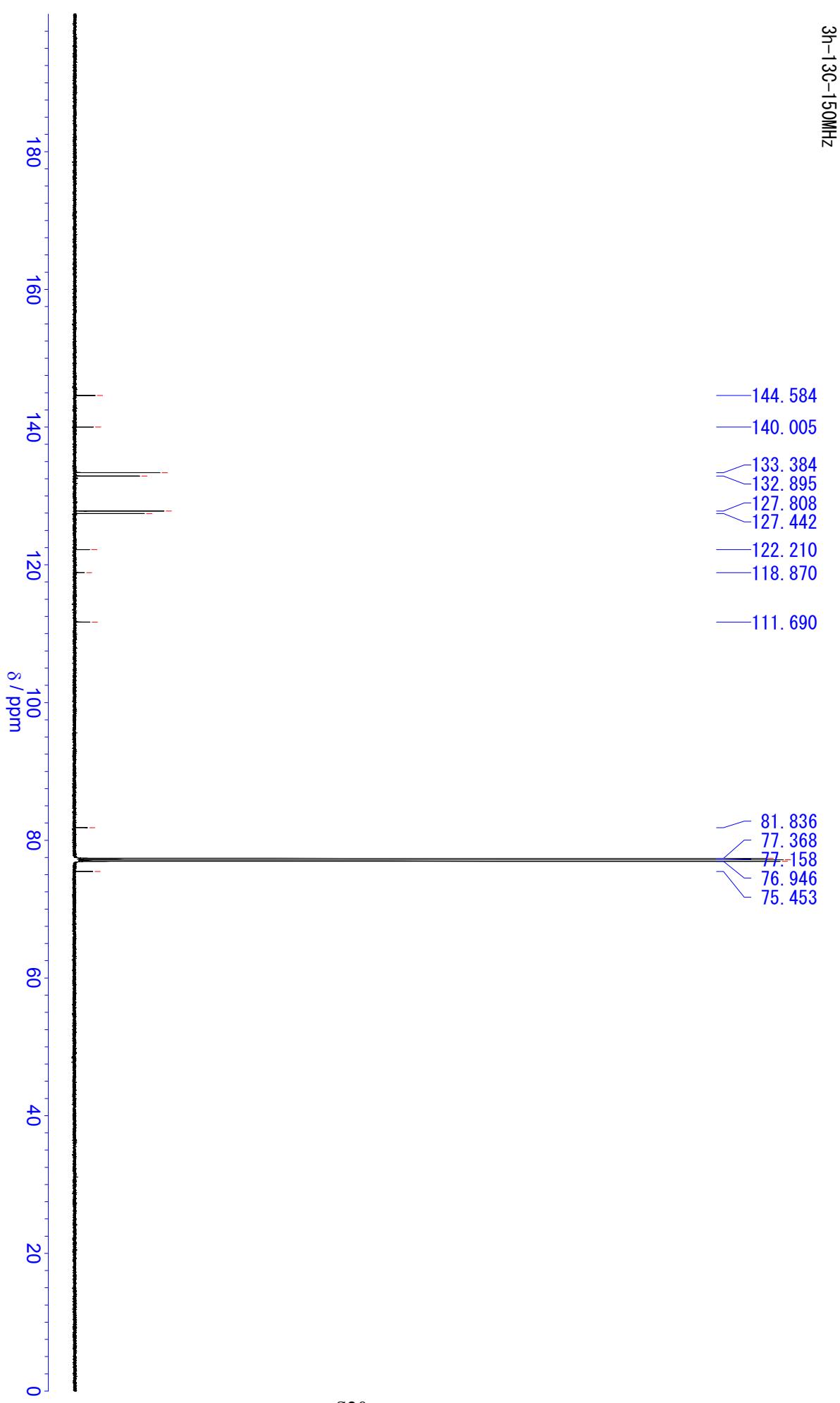
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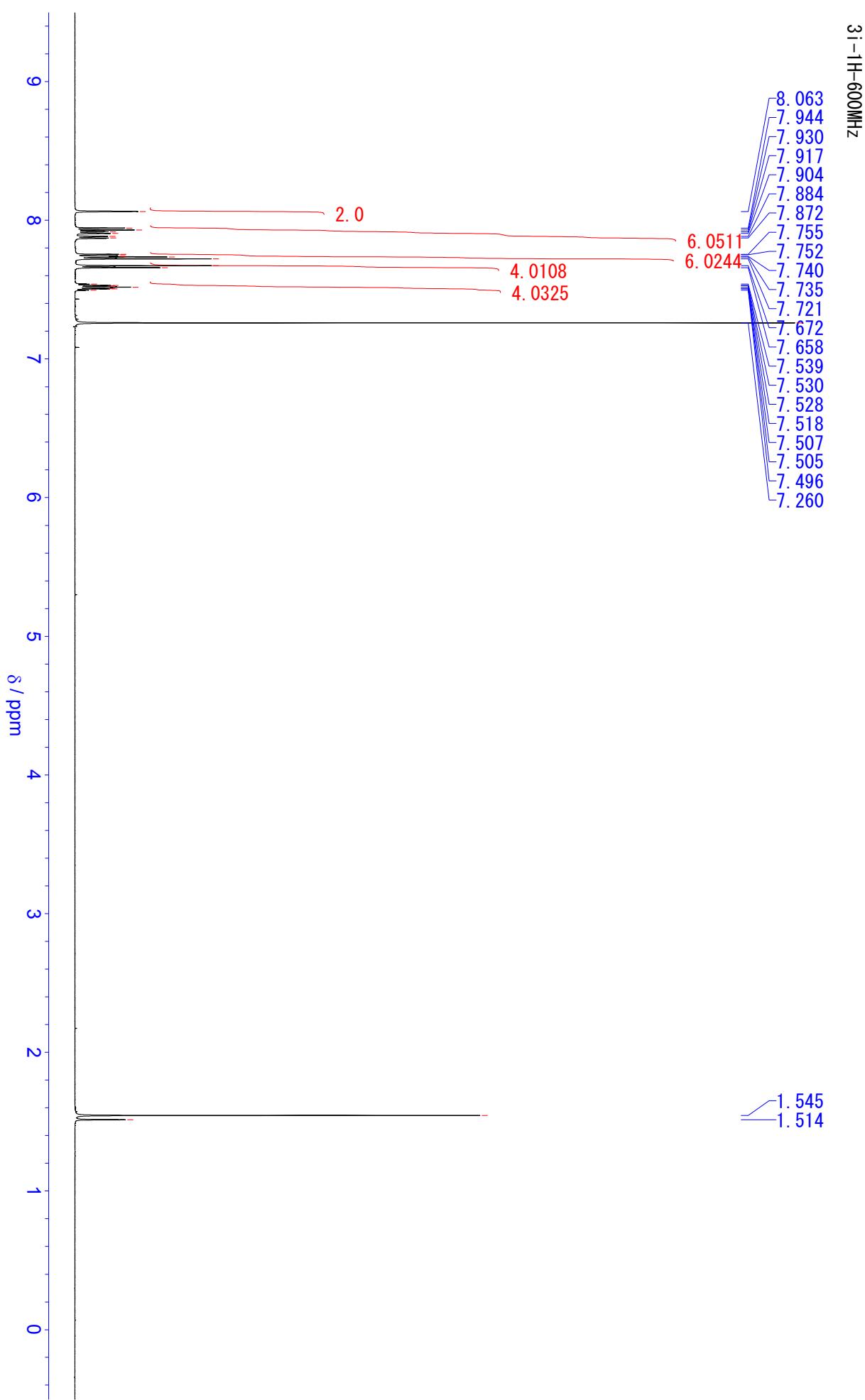




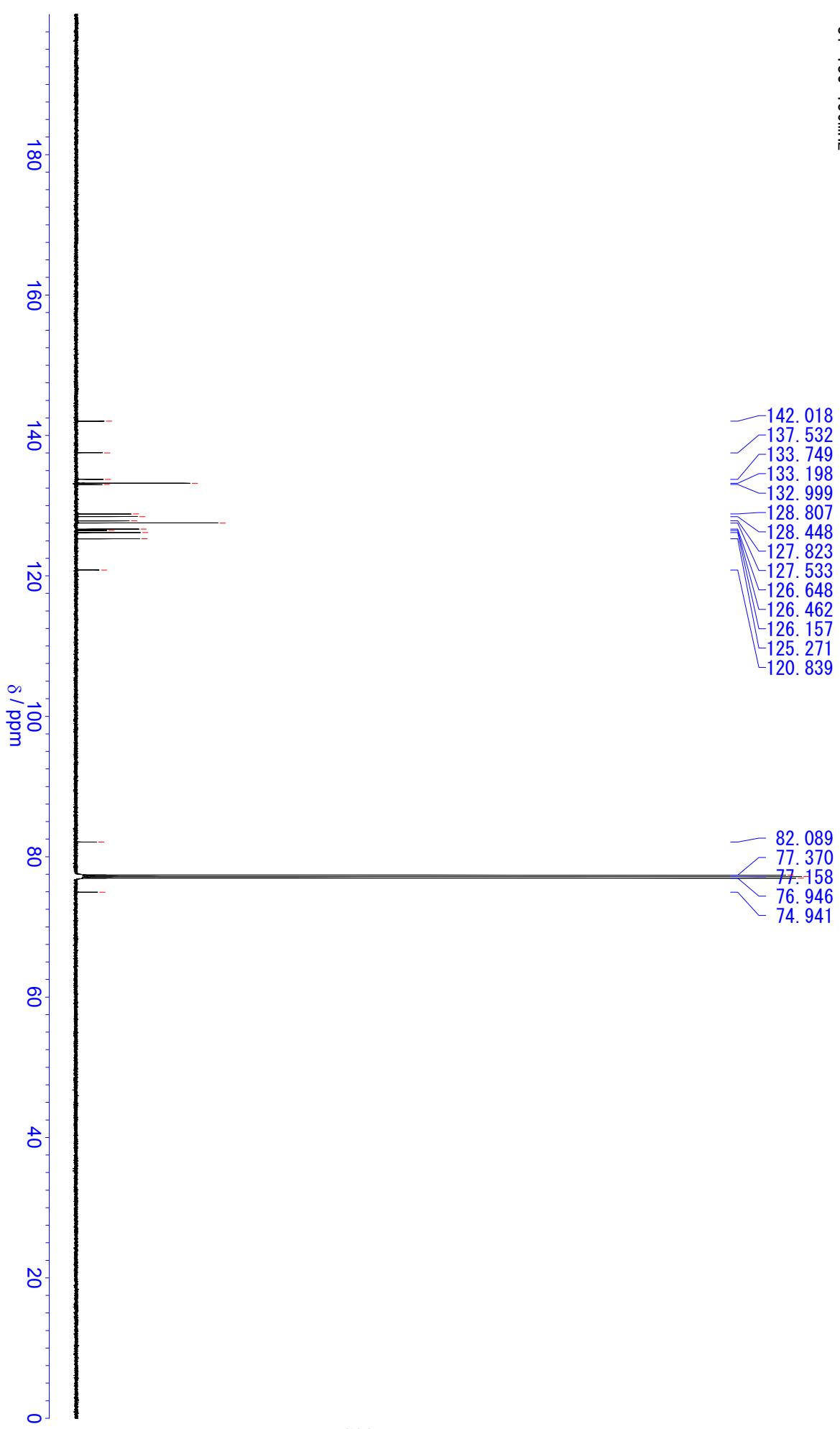


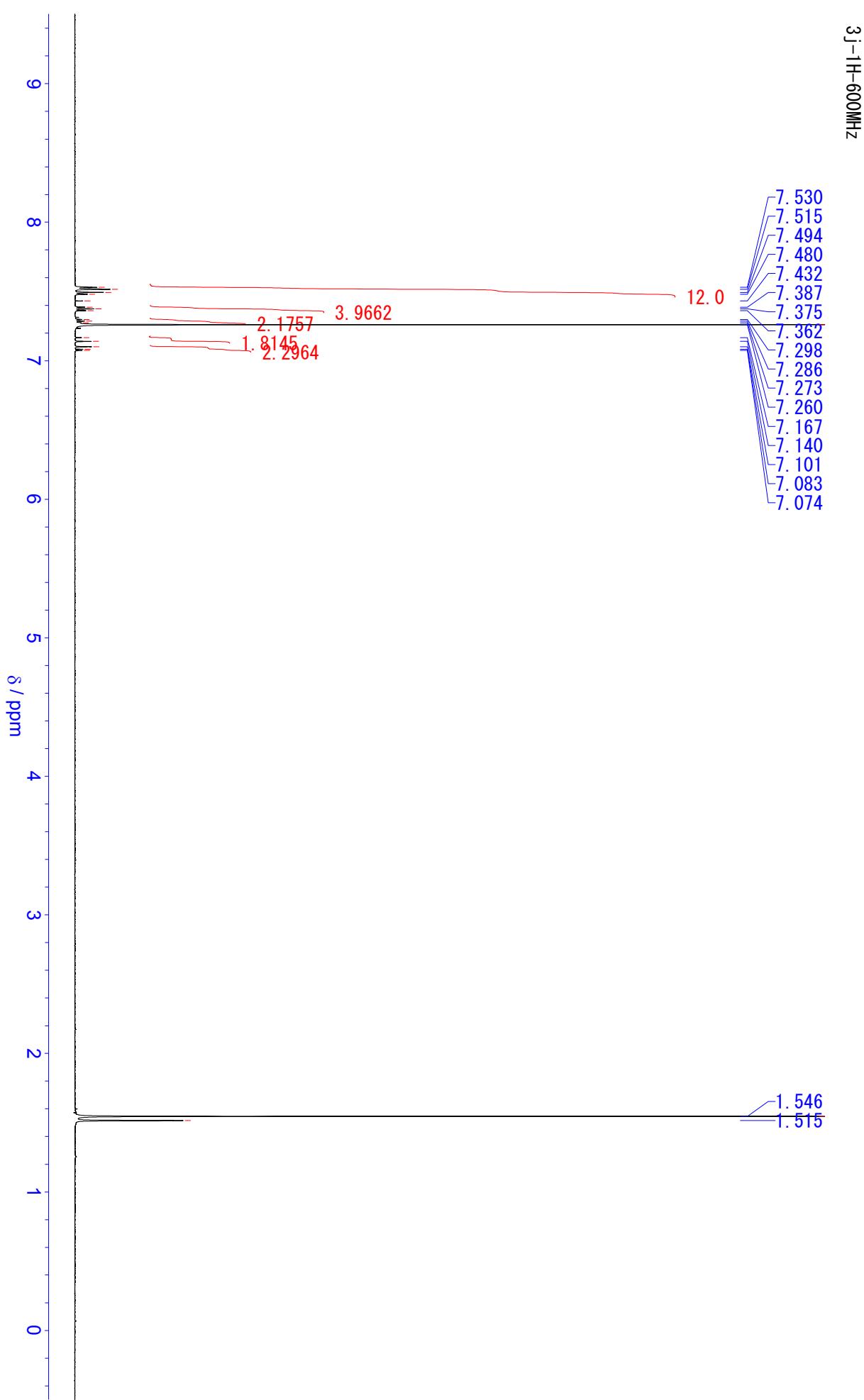




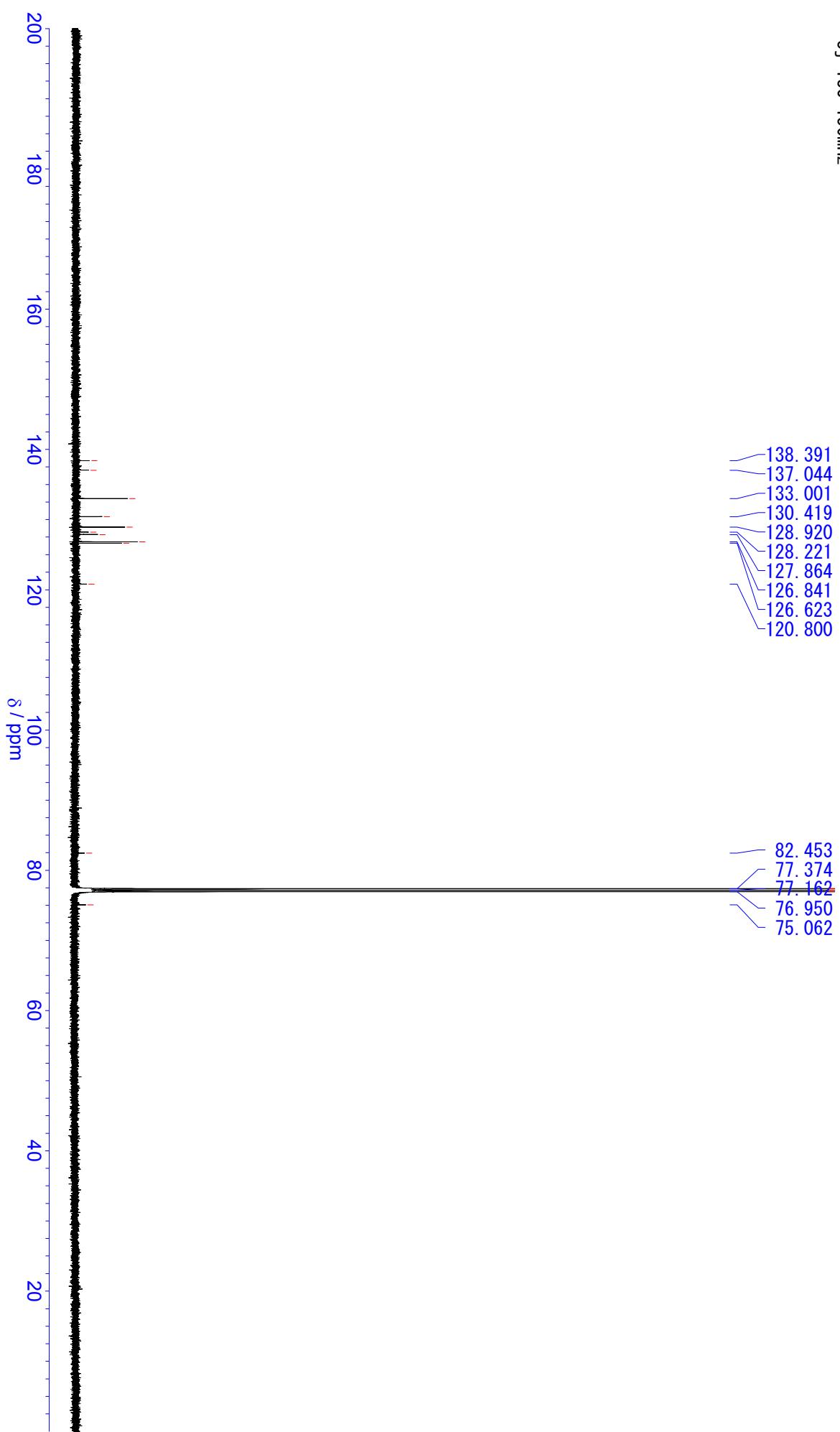


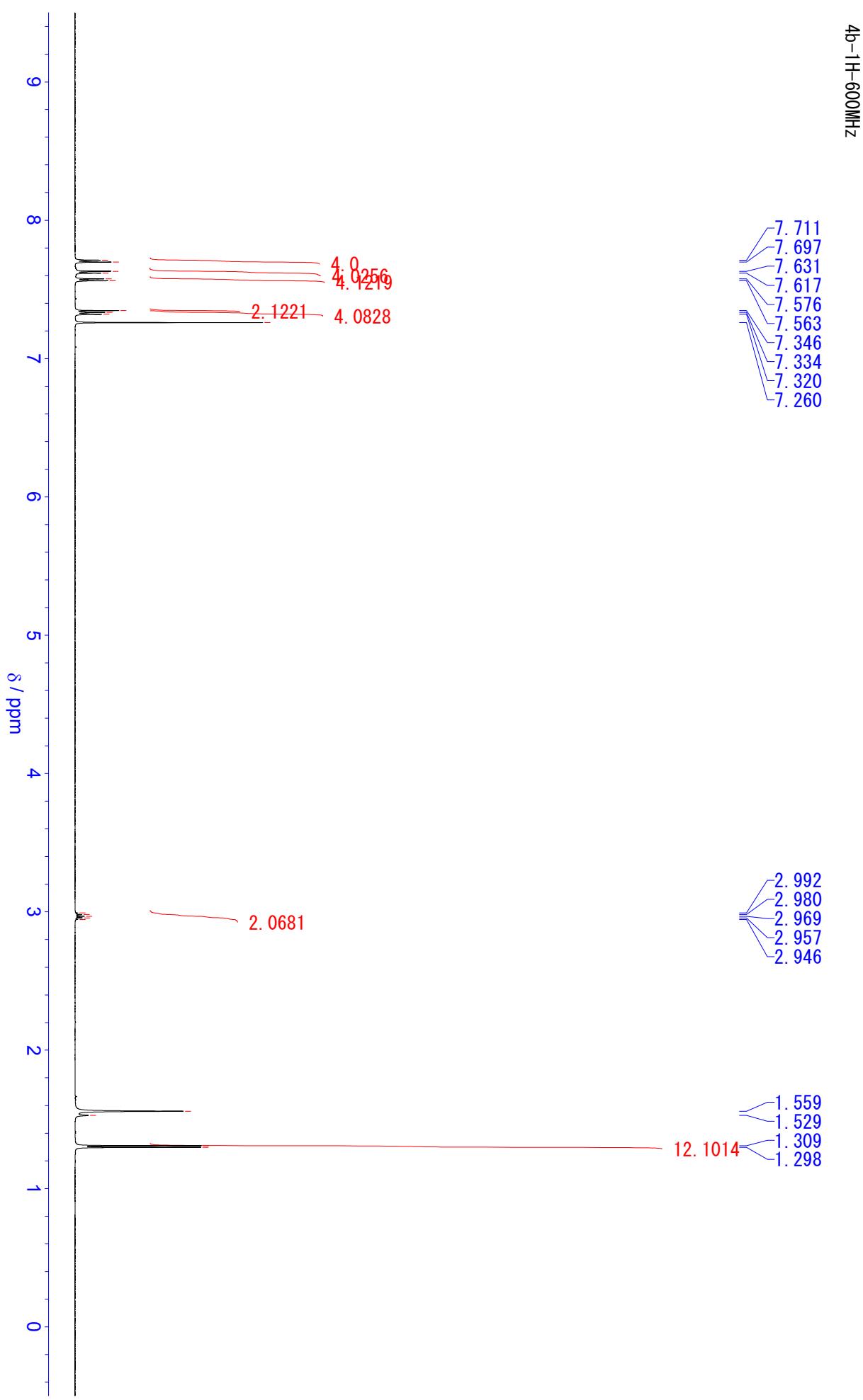
3i-¹³C-150MHz





3J-¹³C-150MHz





4b-¹³C-150MHz

