

Supporting Information

One-Pot Syntheses of Immunostimulatory Glycolipids

*Matthew Schombs, Francine E. Park, Wenjun Du, Suvarn S. Kulkarni and Jacquelyn Gervay-Hague**

Department of Chemistry, University of California, Davis, One Shields Avenue, Davis, CA 95616, USA.

E-mail: gervay@chem.ucdavis.edu

Contents

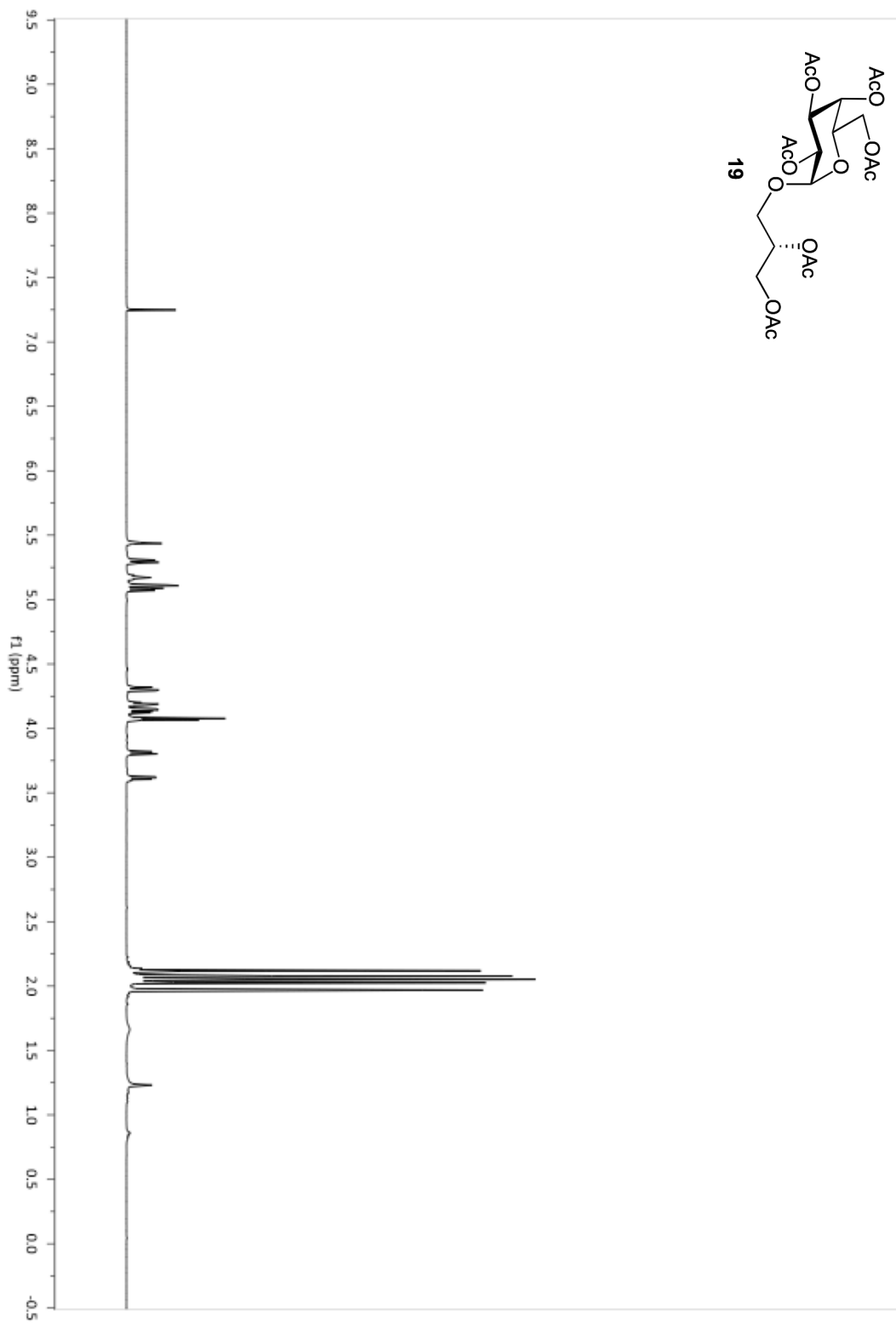
Title	Page
General Experimental	S3
¹ H-NMR spectra of compound 19	S4
¹³ C-NMR spectra of compound 19	S5
COSY-NMR spectra of compound 19	S6
HSQC-NMR spectra of compound 19	S7
HMBC-NMR spectra of compound 19	S8
DEPT-NMR spectra of compound 19	S9
¹ H-NMR spectra of compound 20	S10
¹³ C-NMR spectra of compound 20	S11
COSY-NMR spectra of compound 20	S12
HSQC-NMR spectra of compound 20	S13

HMBC-NMR spectra of compound 20	S14
DEPT-NMR spectra of compound 20	S15
¹ H-NMR spectra of competition experiment	S16
¹³ C-NMR spectra of competition experiment	S17
COSY-NMR spectra of competition experiment	S18
HSQC-NMR spectra of competition experiment	S19
DEPT-NMR spectra of competition experiment	S20
¹ H-NMR spectra of compound 22	S21
¹³ C-NMR spectra of compound 22	S22
COSY-NMR spectra of compound 22	S23
HSQC-NMR spectra of compound 22	S24
DEPT-NMR spectra of compound 22	S25
¹ H-NMR spectra of compound 23	S26
¹³ C-NMR spectra of compound 23	S27
COSY-NMR spectra of compound 23	S28
HSQC-NMR spectra of compound 23	S29
DEPT-NMR spectra of compound 23	S30
¹ H-NMR spectra of compound 24	S31
¹³ C-NMR spectra of compound 24	S32
COSY-NMR spectra of compound 24	S33
HSQC-NMR spectra of compound 24	S34
HMBC-NMR spectra of compound 24	S35

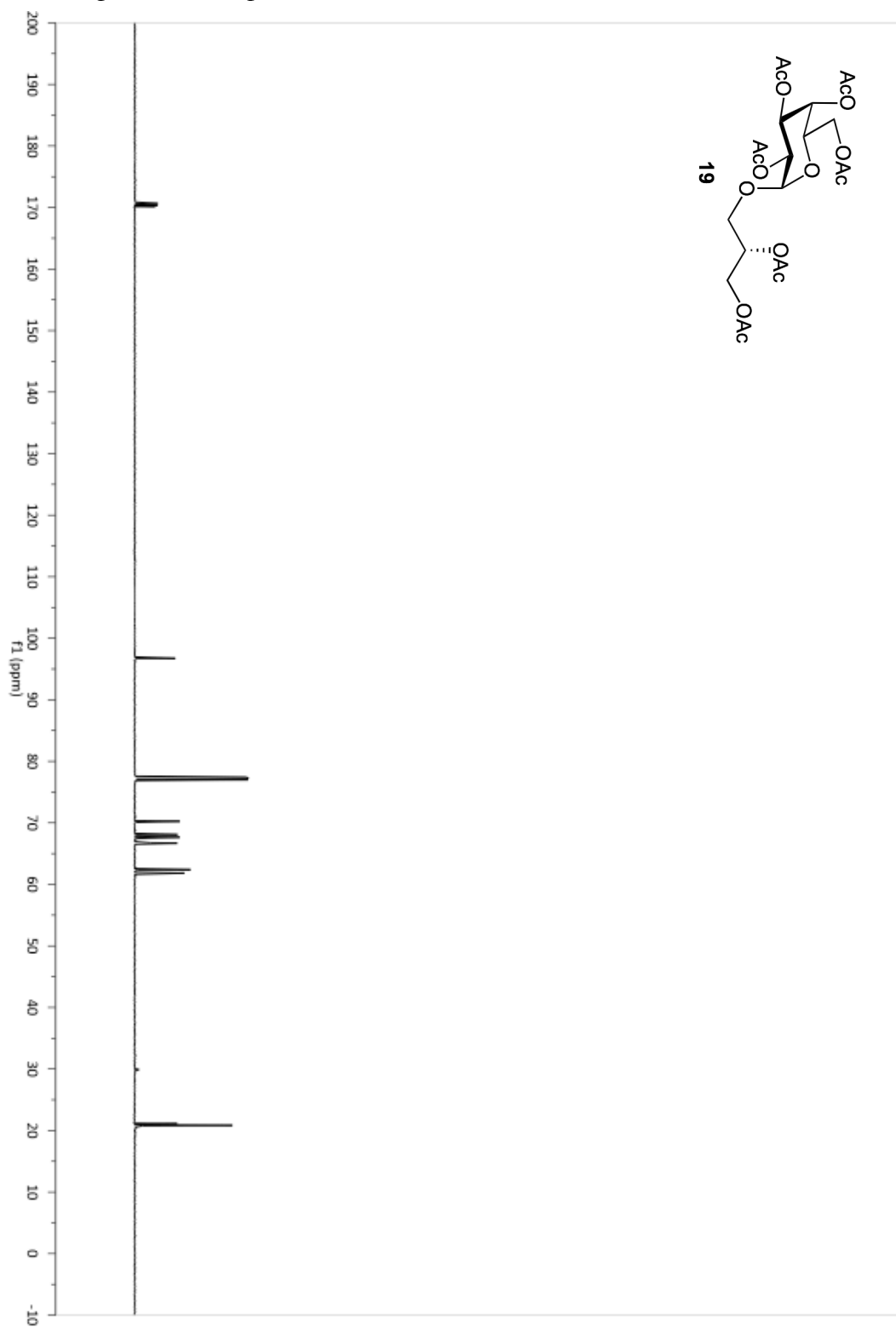
General Experimental

All reactions were conducted under a dried argon stream. The solvents (CH_2Cl_2 99.8%, benzene 99.8%) were purchased in capped DriSolv™ bottles and used without further purification and stored under argon. TMSI was stored at $-20\text{ }^\circ\text{C}$ under a desiccated atmosphere. All other solvents and reagents were purchased from commercial sources and used without further purification. All glassware utilized was flame-dried before use. Glass-backed TLC plates (Silica Gel 60 with a 254 nm fluorescent indicator) were used without further manipulation and stored over desiccant. TLC plates were visualized using a short-wave UV lamp, stained with an $\text{I}_2\text{-SiO}_2$ mixture, and /or by heating plates that were dipped in a solution of ammonium molybdate/cerium (IV) sulfate. Flash column chromatography was performed using a silica gel (32-63 μm) stationary phase with a variable mobile phase correlated with TLC mobility. Optical rotations were measured at 598 nm using a 100 mm cell. NMR experiments were conducted on a 600 MHz instrument using CDCl_3 (99.9% D), CD_3OD (99.9% D) or pyridine d_5 (99.9% D) as the solvent. Chemical shifts are referenced to the appropriate deuterated solvent peak and are in parts per million (ppm). Samples were analyzed by electrospray ionization in both the negative and positive mode using flow-injection analysis on the IonMax source. Standard source conditions were employed and NaI was used as an additive when necessary for cation generation. The microwave-assisted reaction was conducted in a Discover Labmate® (CEM Co., Matthews, NC) microwave reactor.

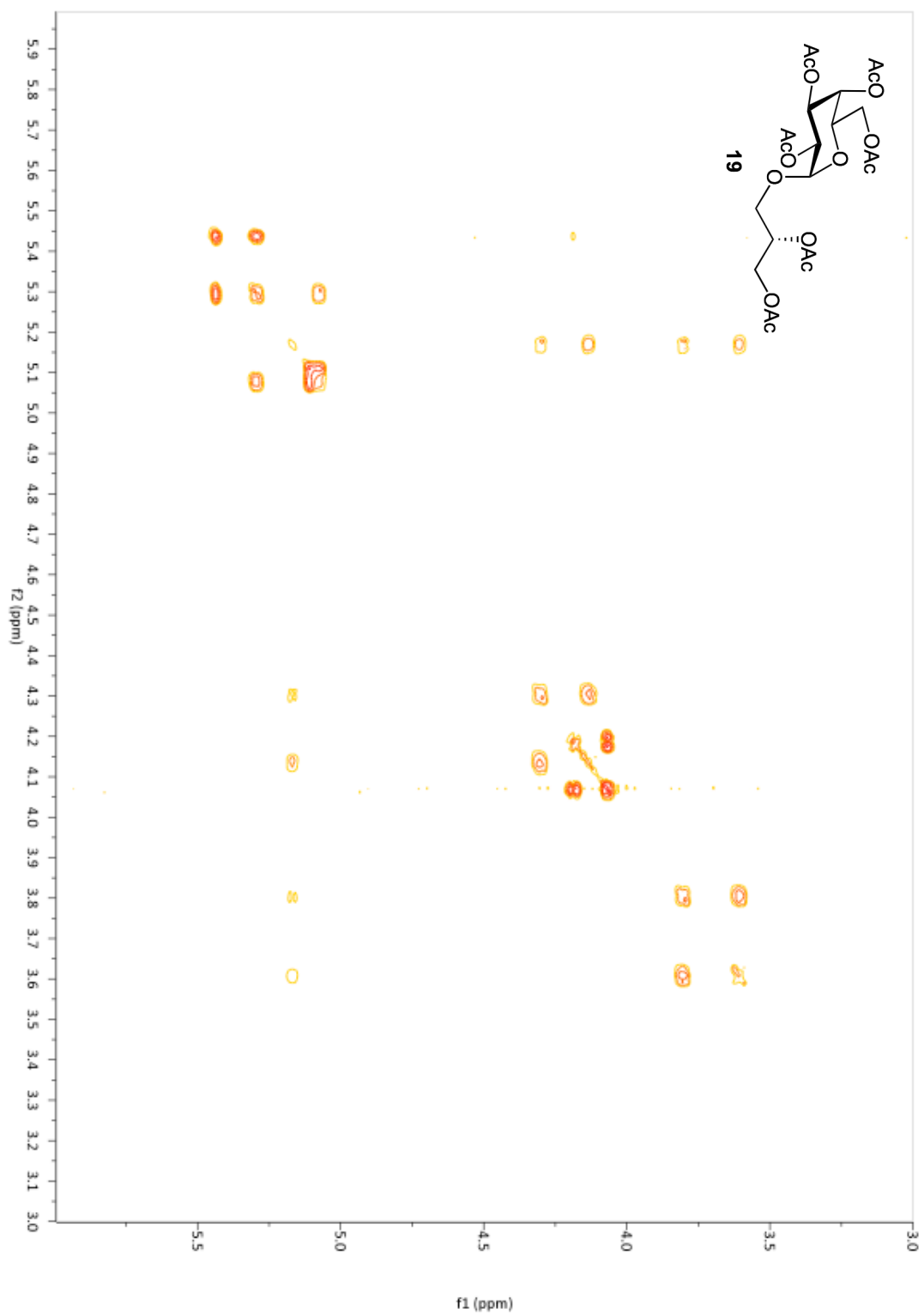
¹H-NMR spectra of compound **19**



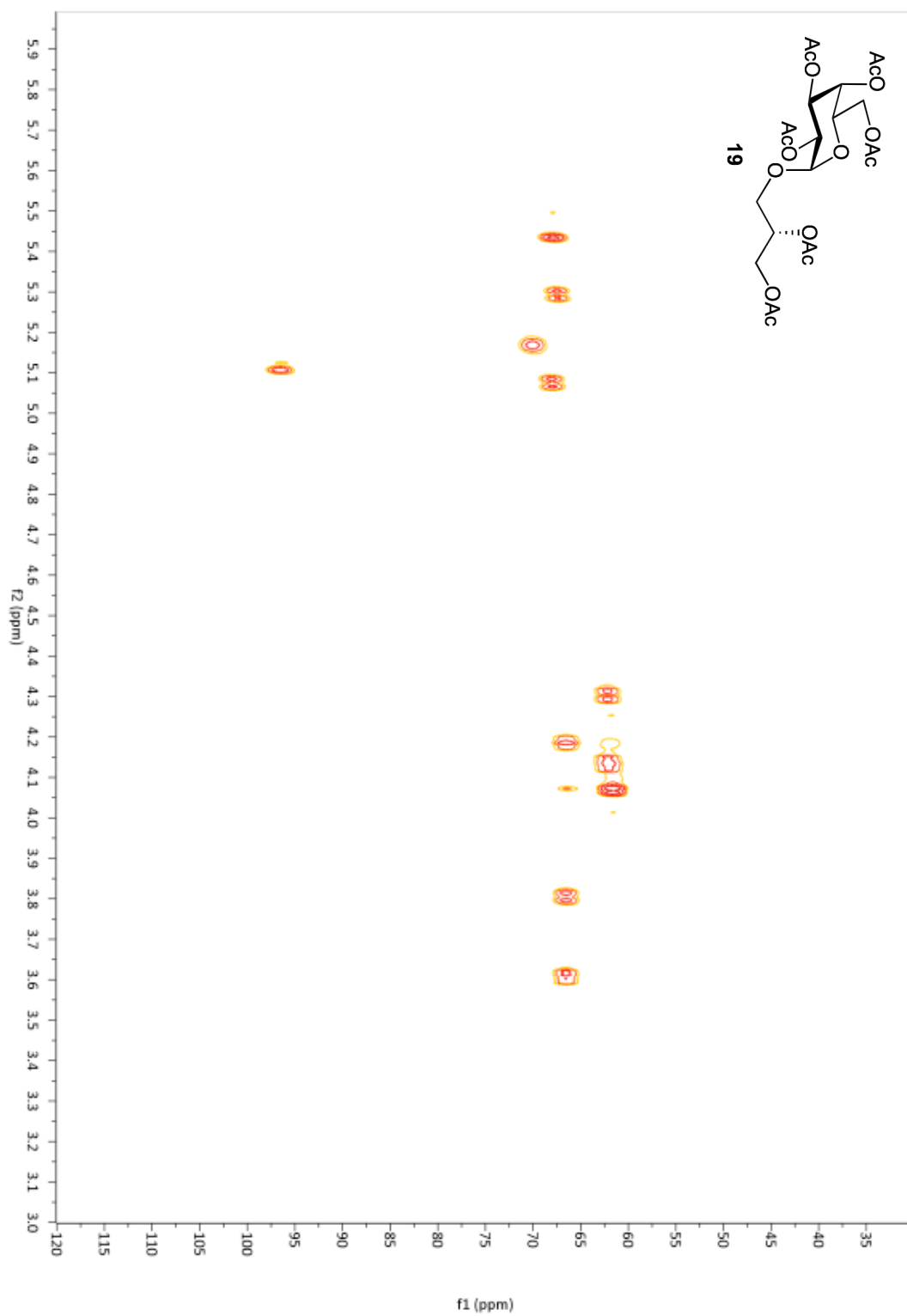
^{13}C -NMR spectra of compound **19**



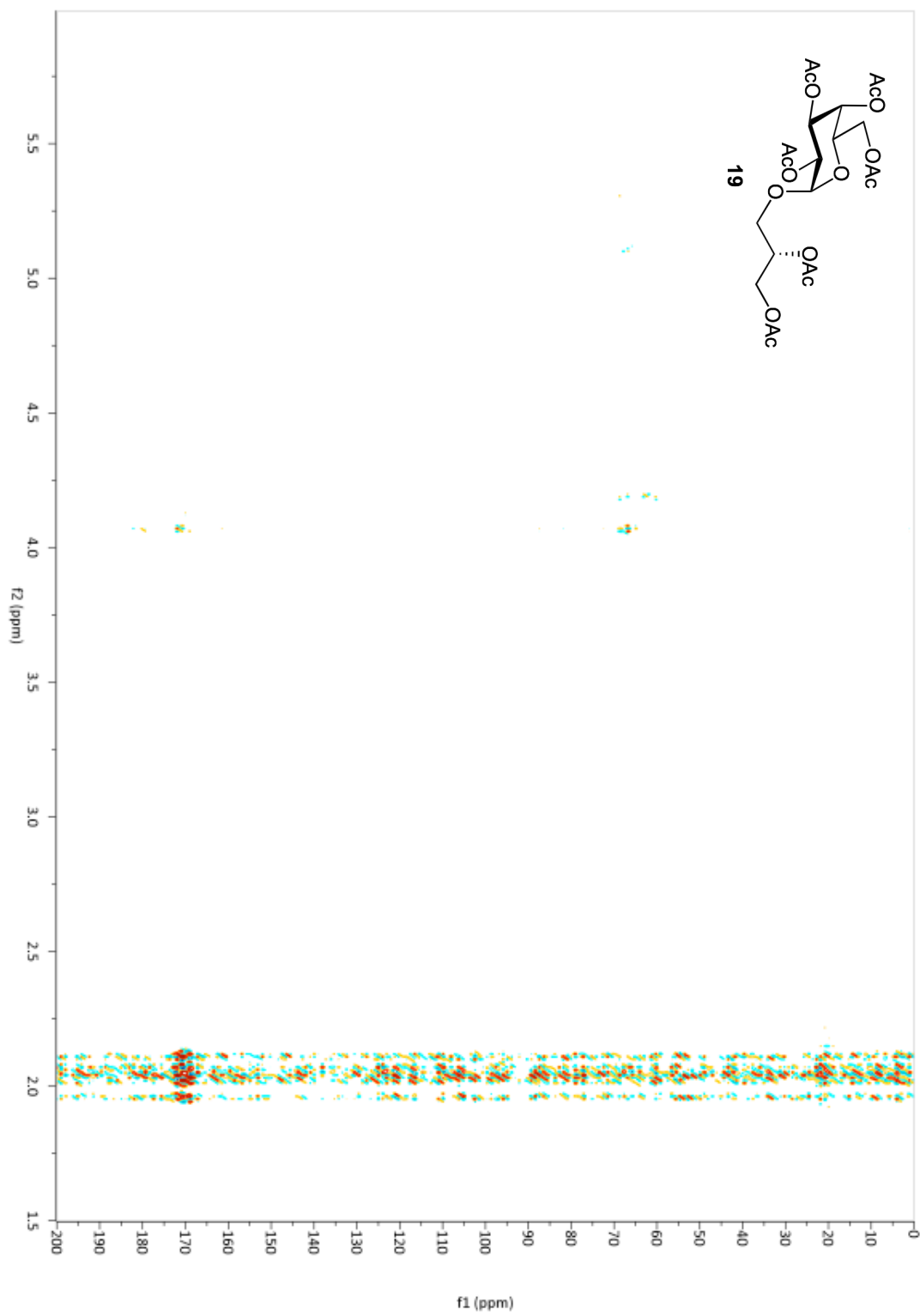
COSY-NMR spectra of compound **19**



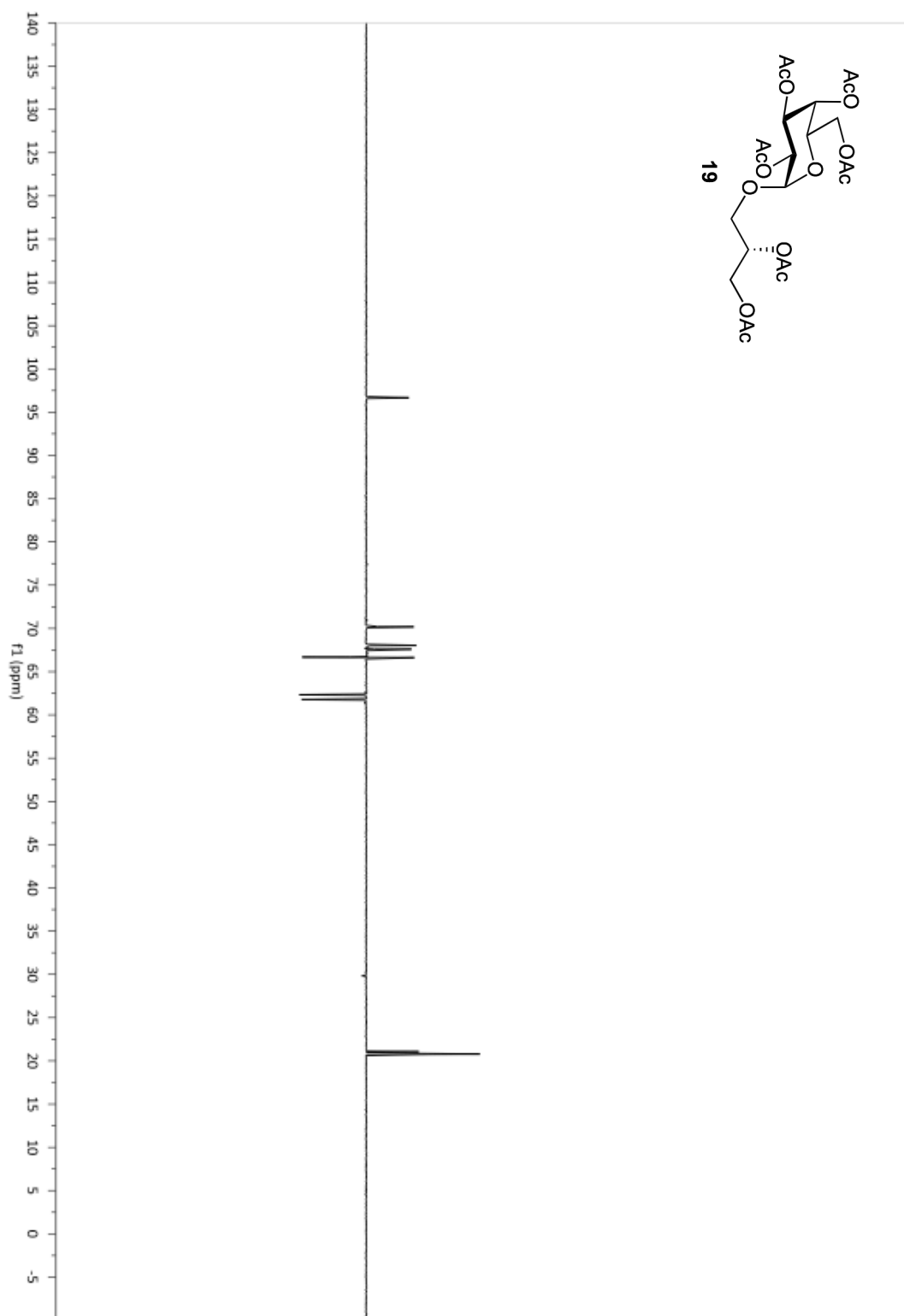
HSQC-NMR spectra of compound **19**



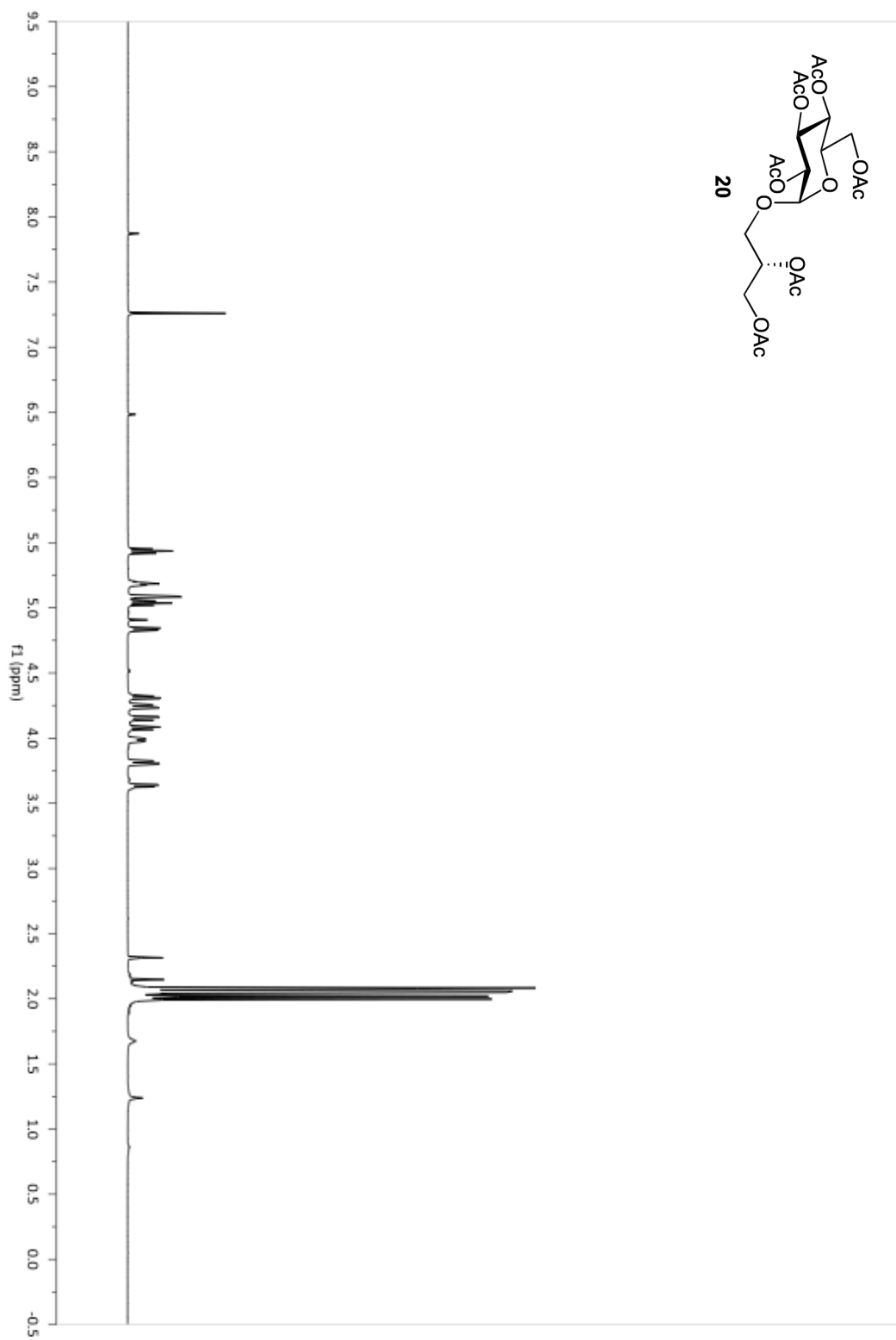
HMBC-NMR spectra of compound **19**



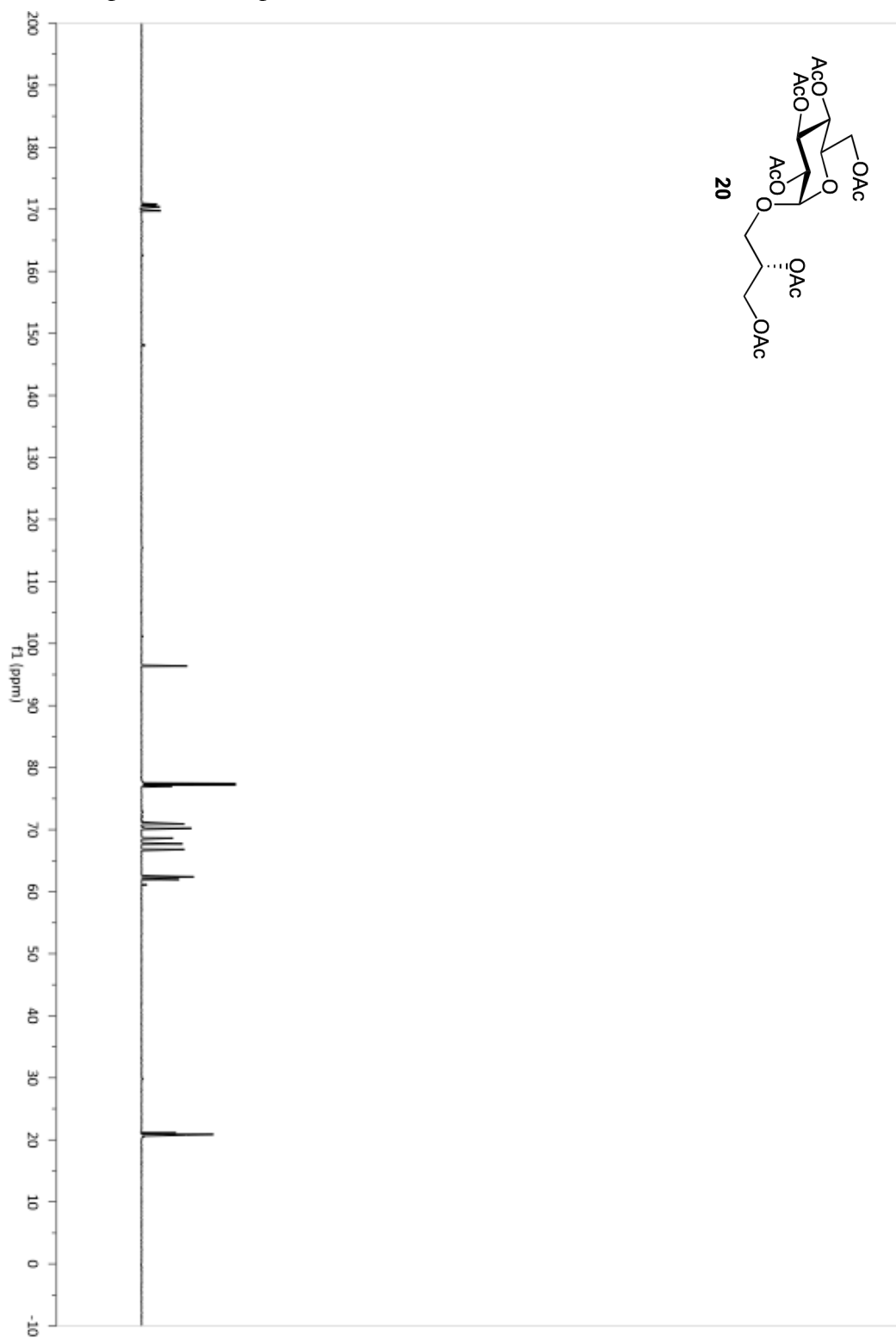
DEPT-NMR spectra of compound **19**



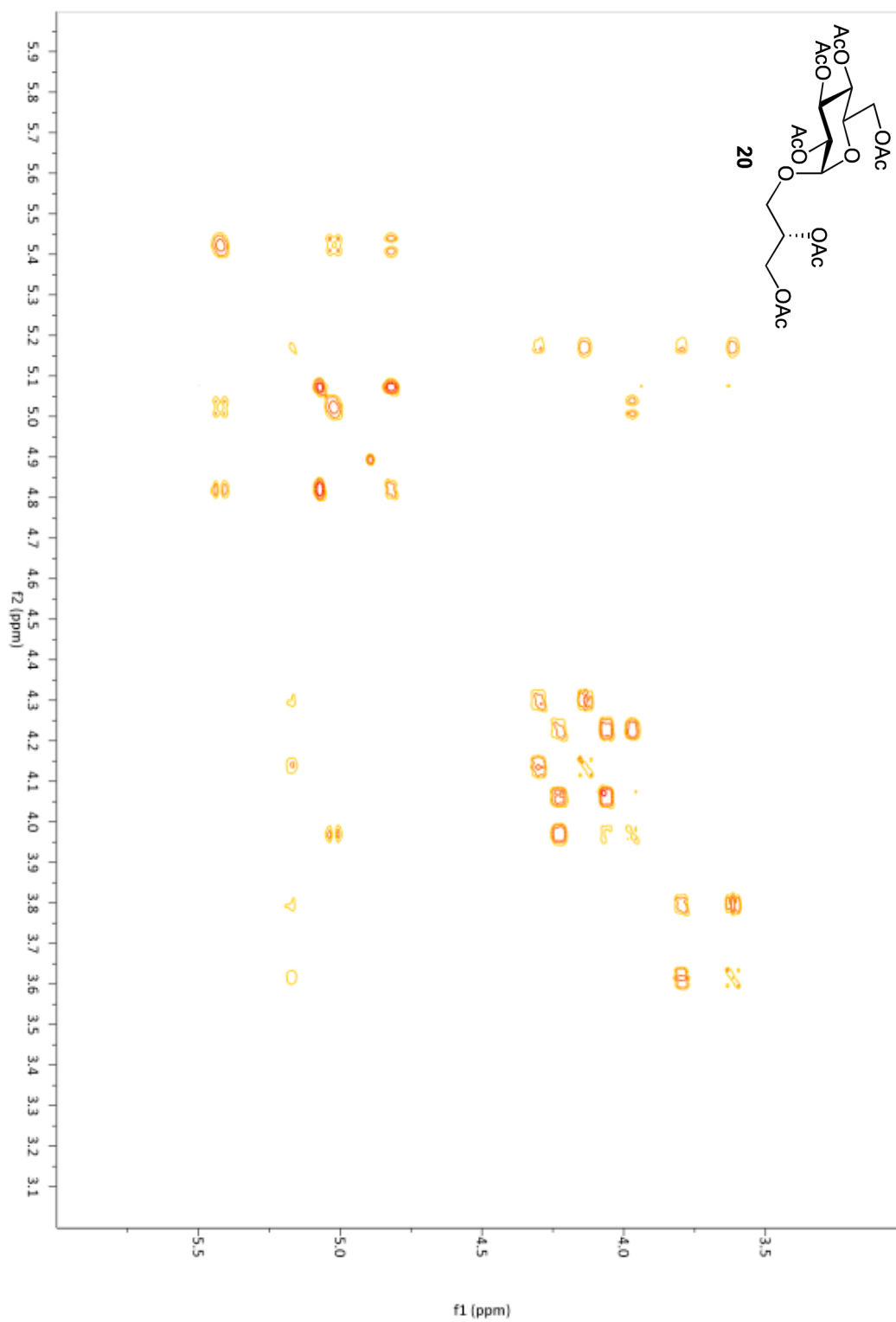
¹H-NMR spectra of compound **20**



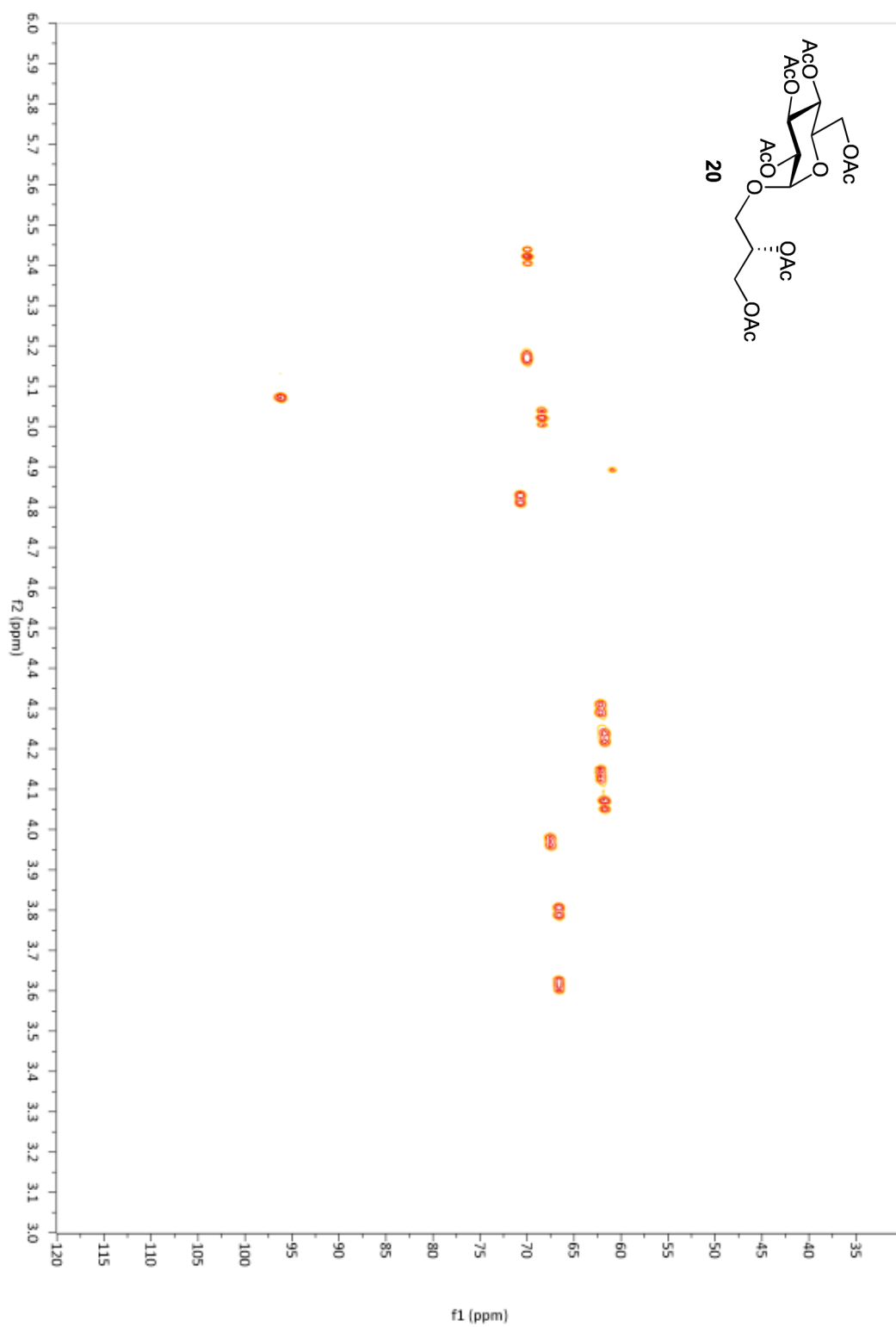
¹³C-NMR spectra of compound **20**



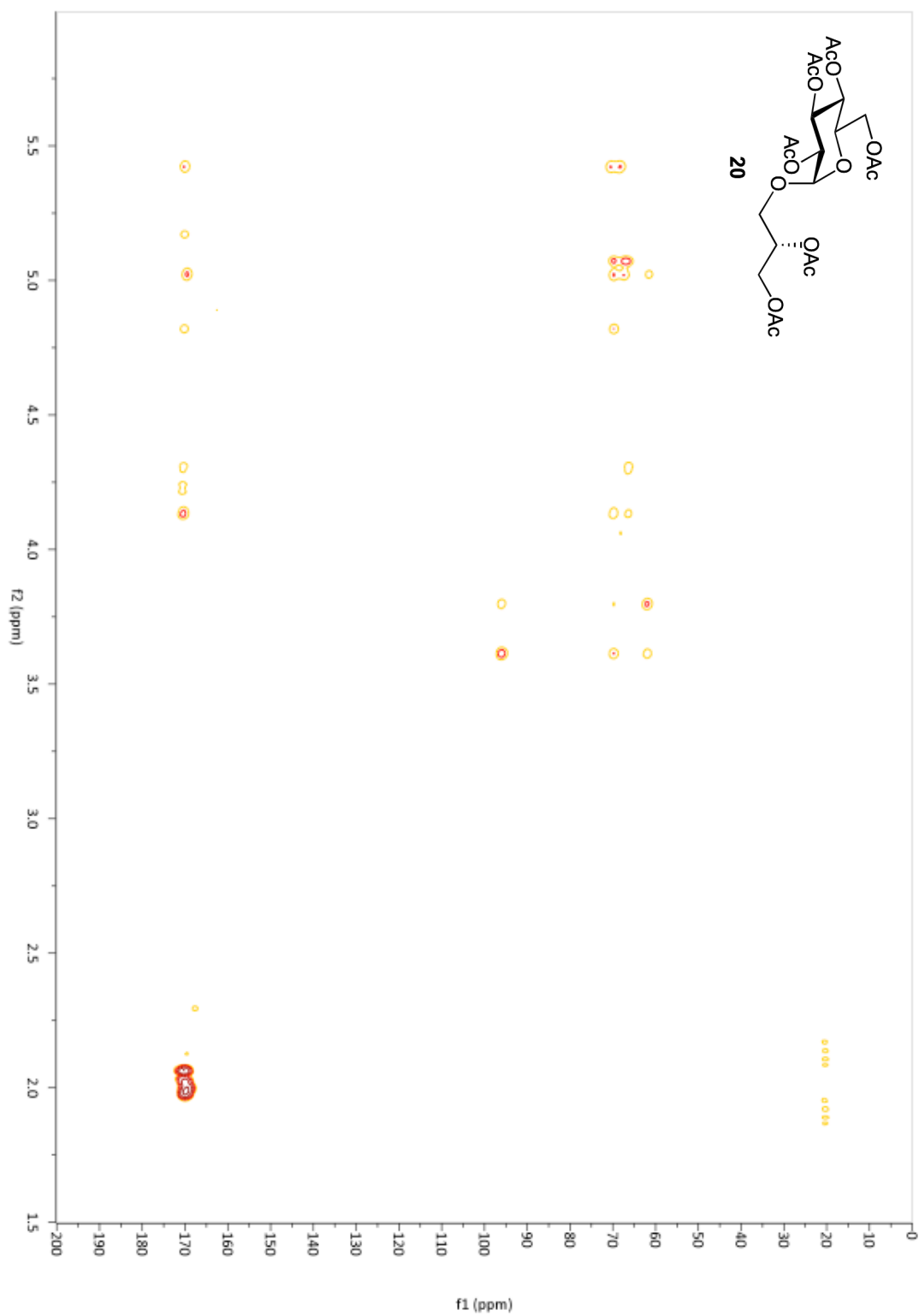
COSY-NMR spectra of compound **20**



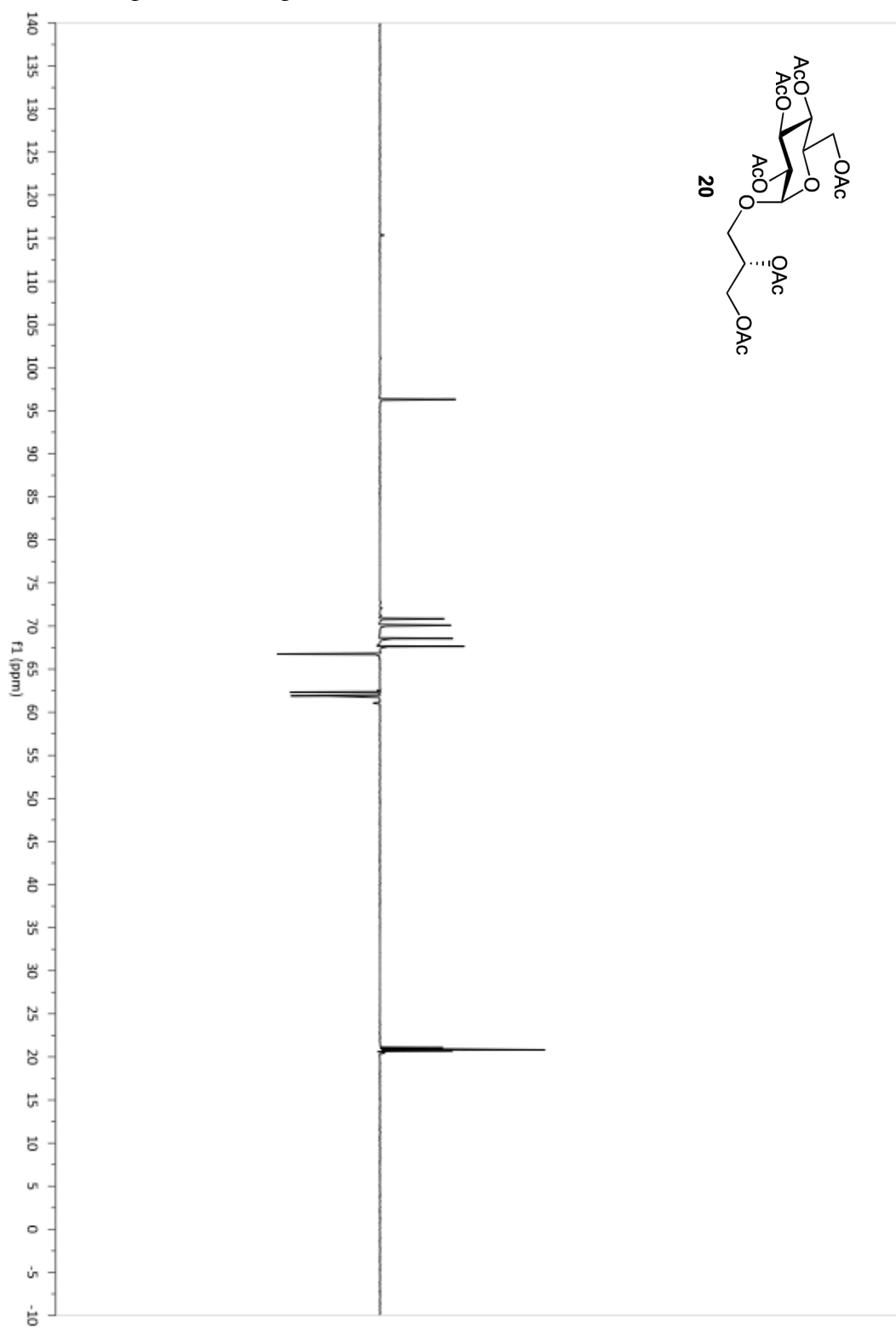
HSQC-NMR spectra of compound **20**



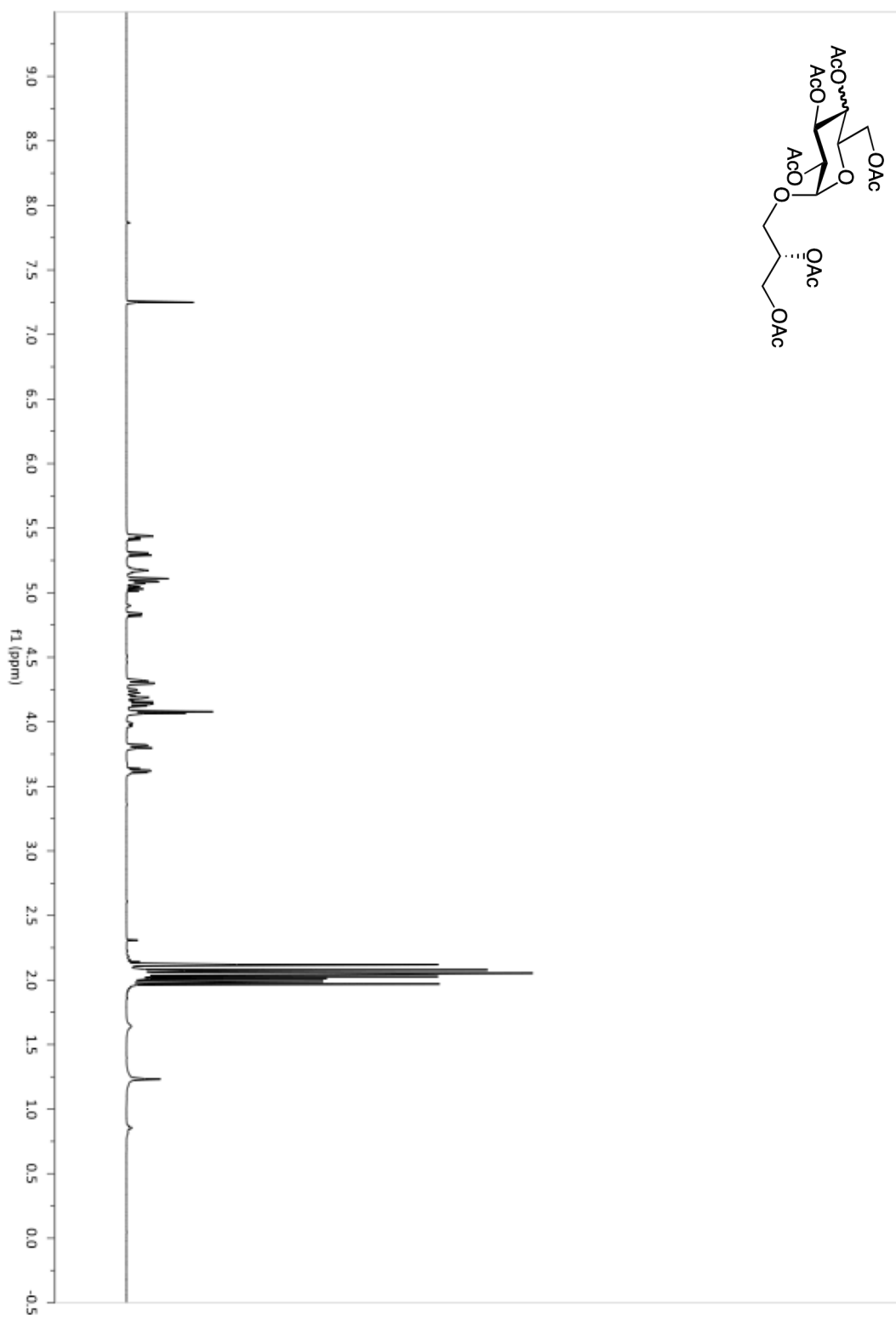
HMBC-NMR spectra of compound **20**



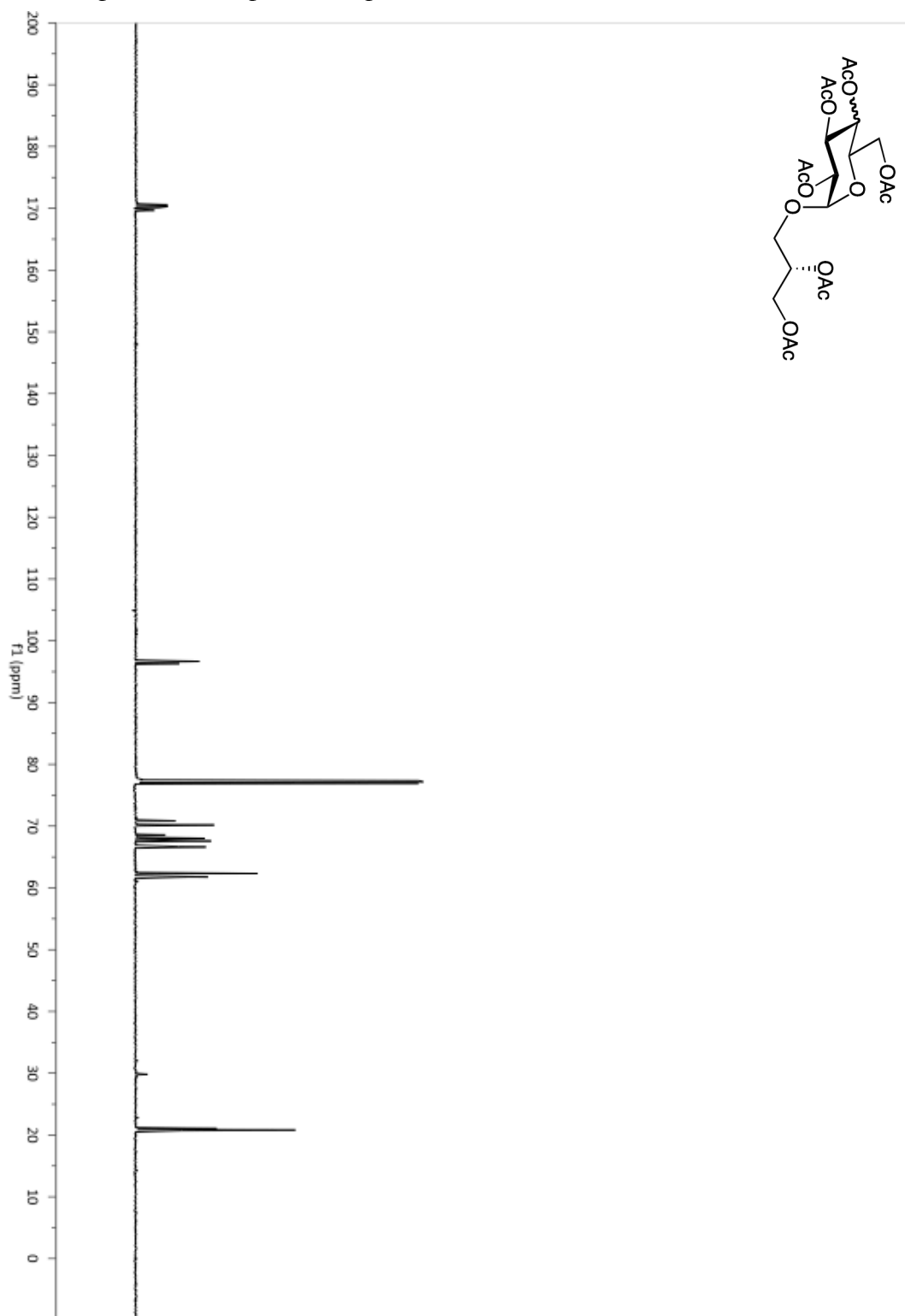
DEPT-NMR spectra of compound **20**



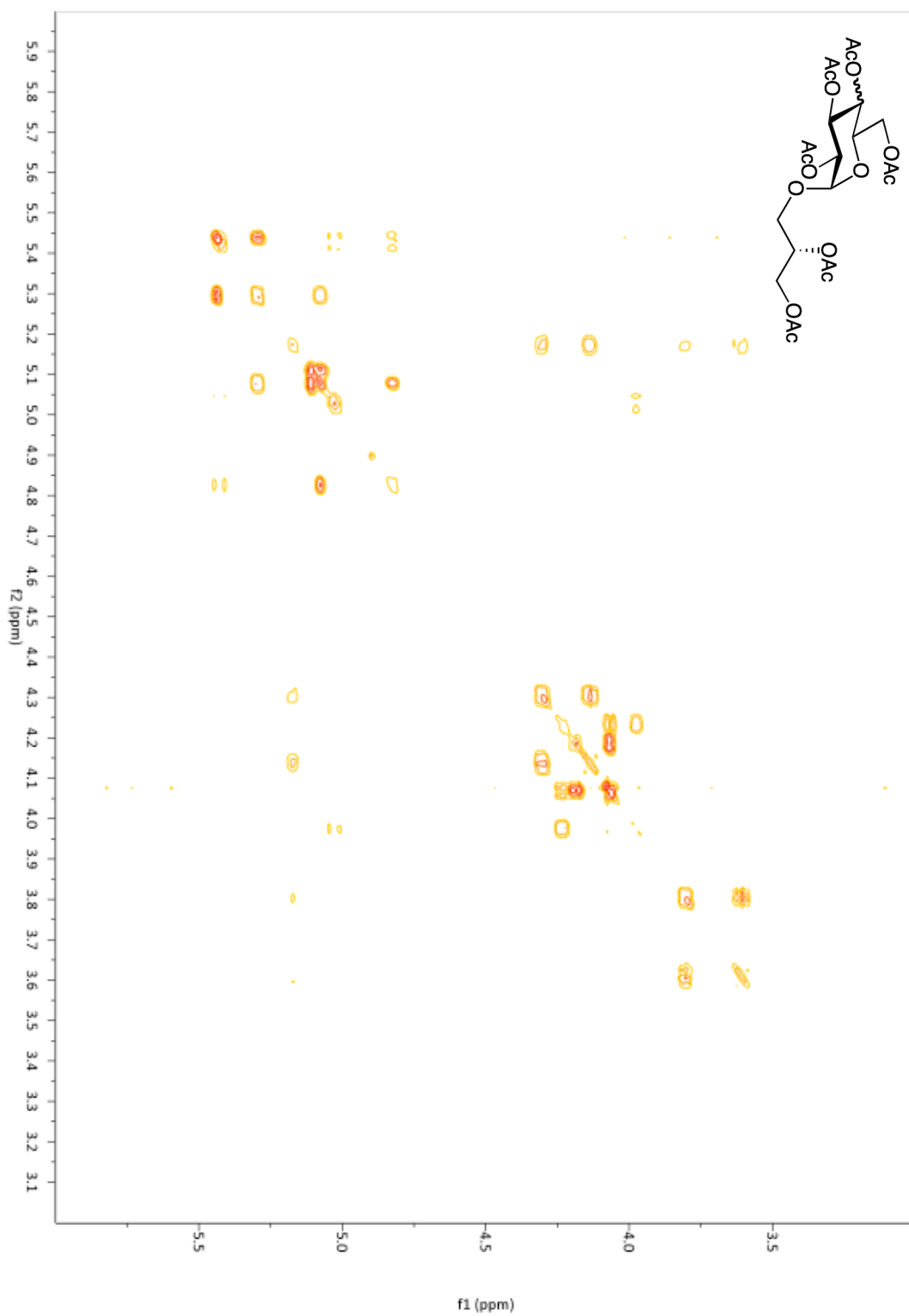
^1H -NMR spectra of competition experiment



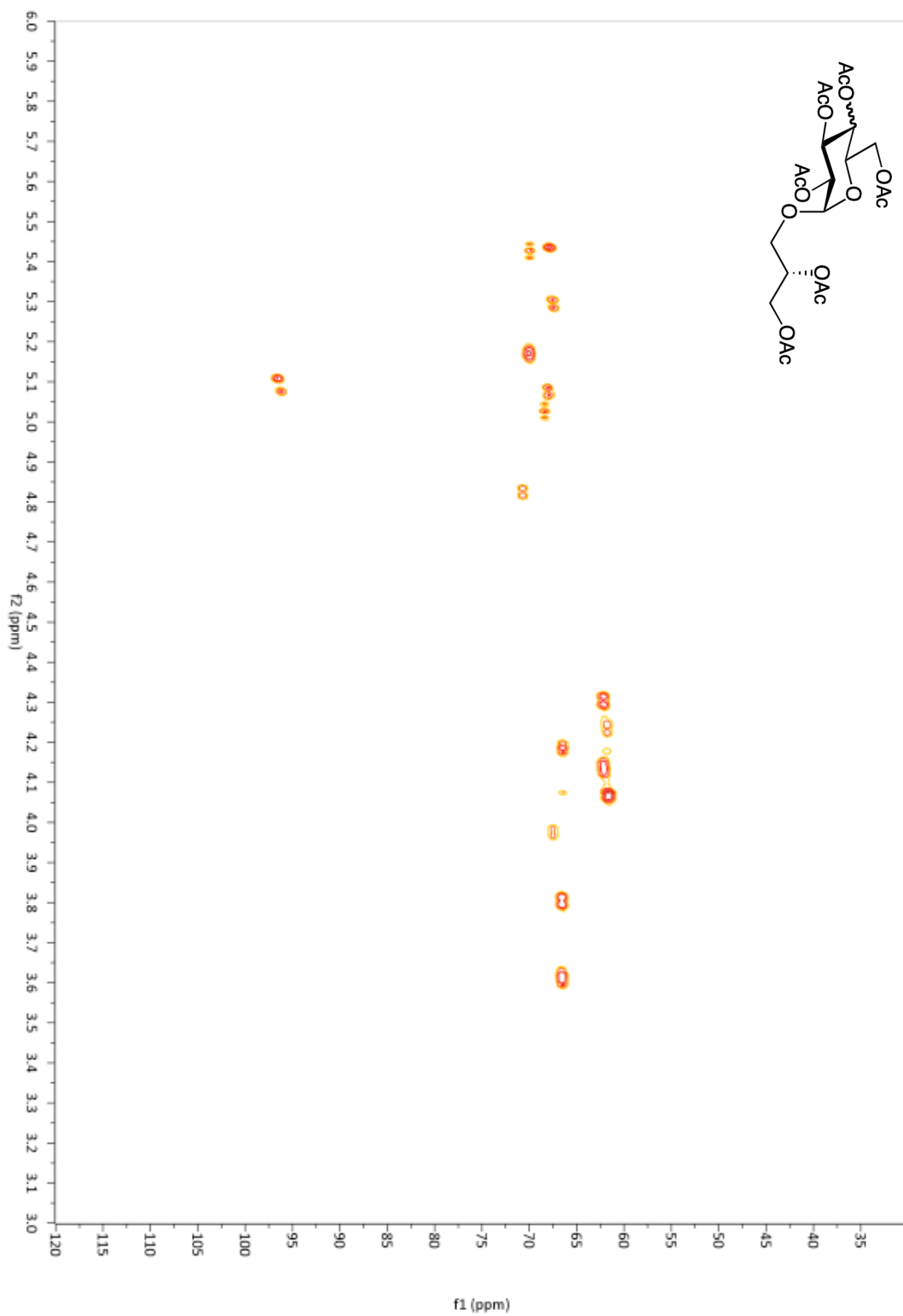
^{13}C -NMR spectra of competition experiment



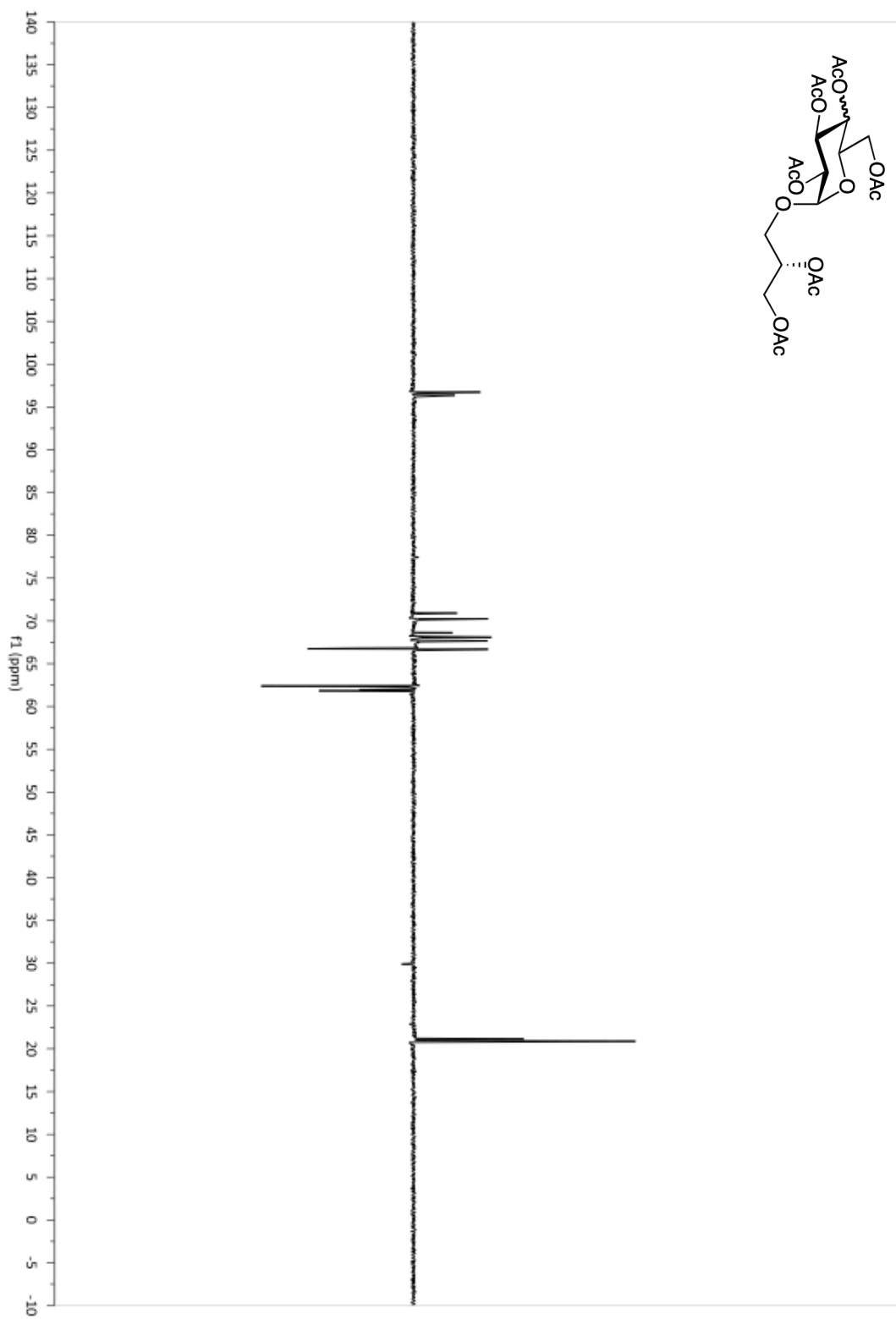
COSY-NMR spectra of competition experiment



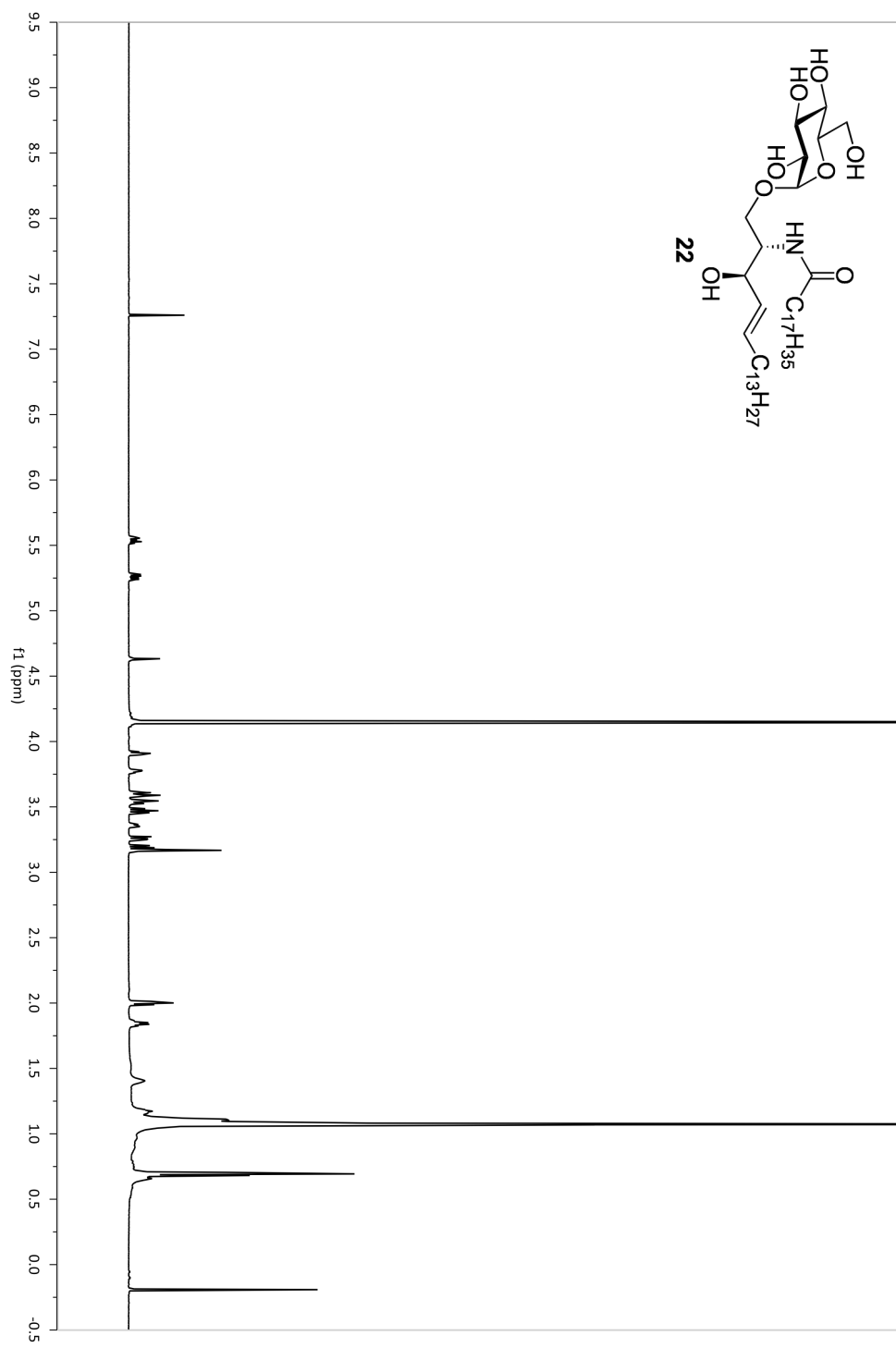
HSQC-NMR spectra of competition experiment



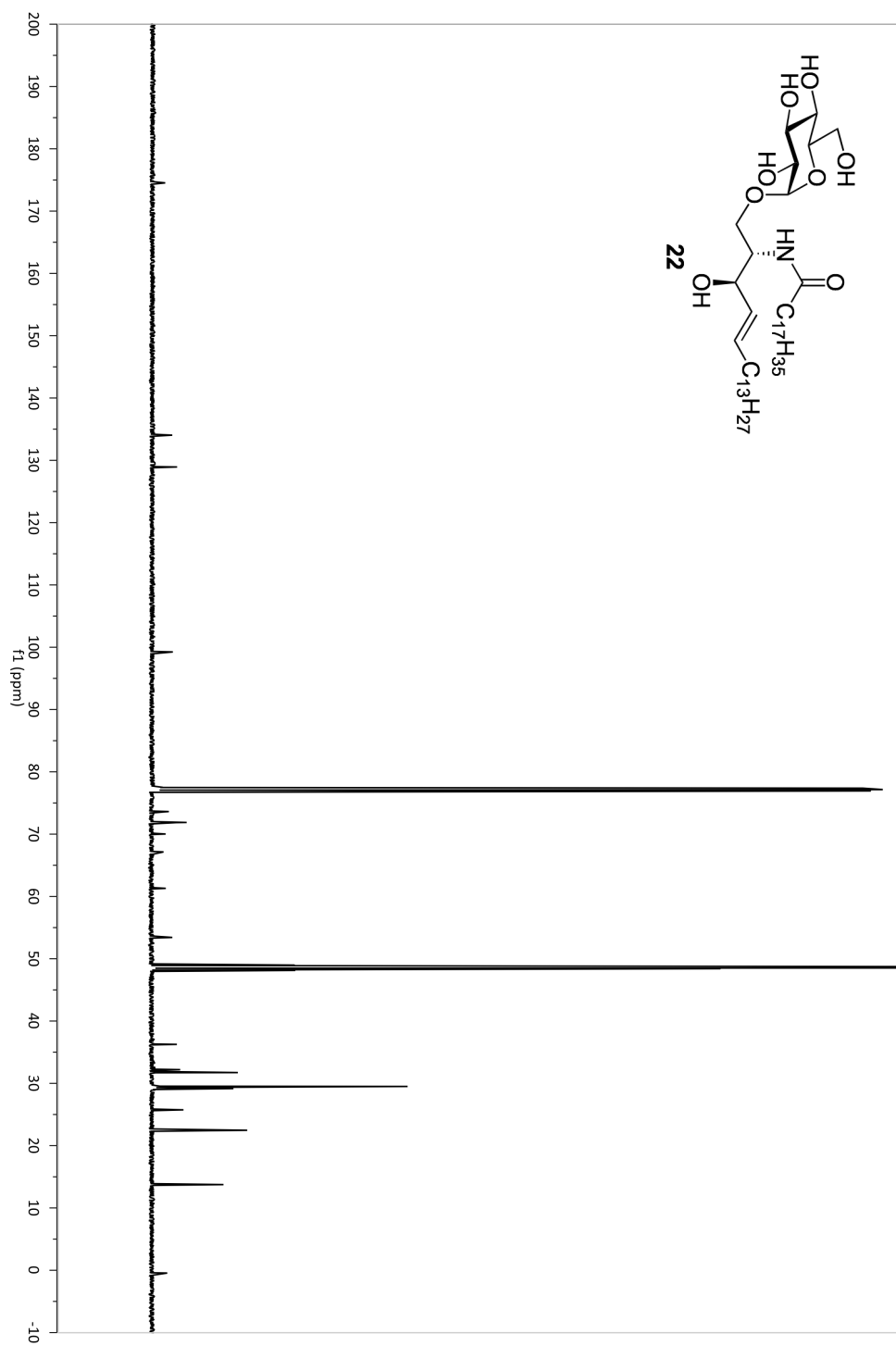
DEPT-NMR spectra of competition experiment



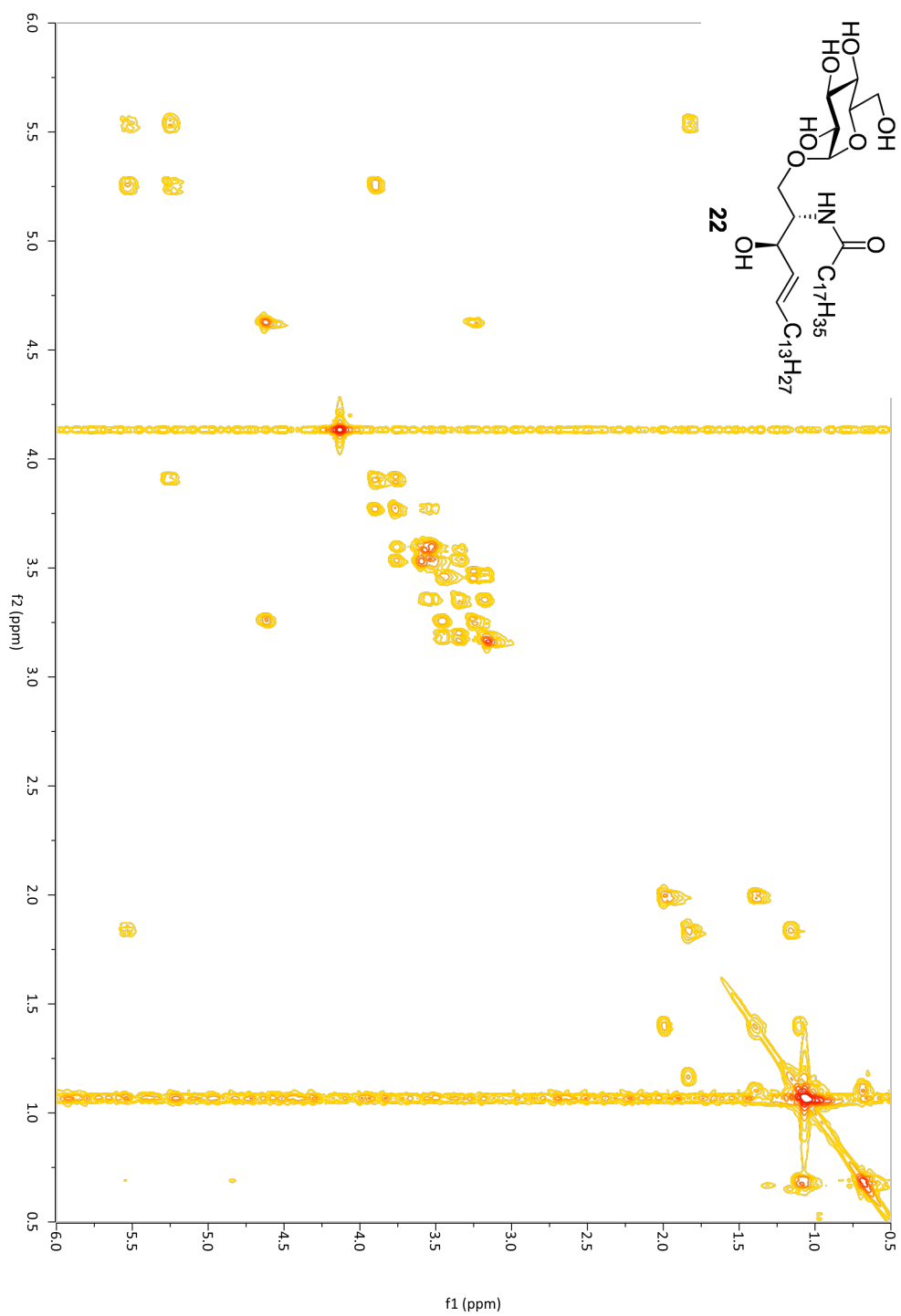
¹H-NMR spectra of compound **22**



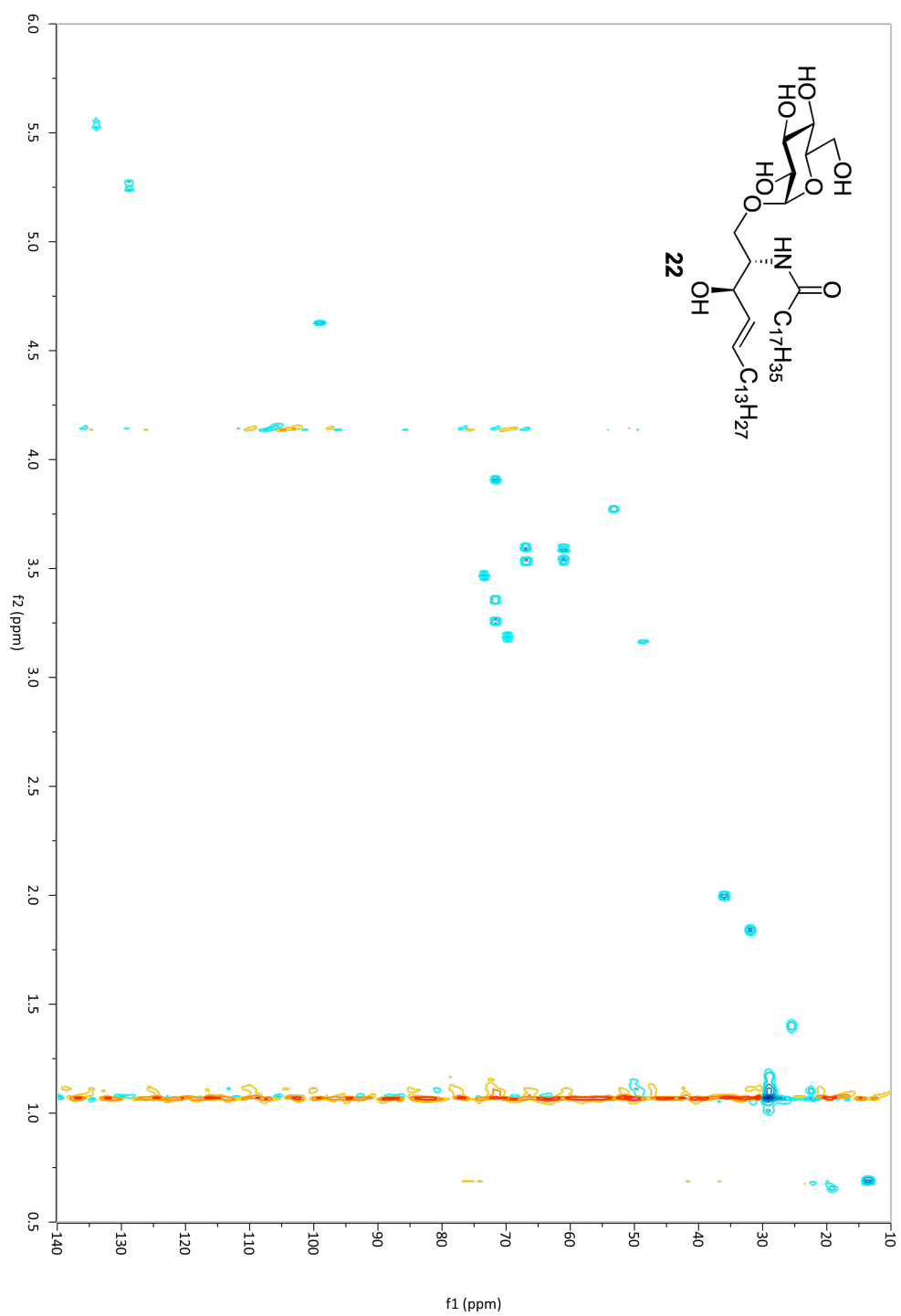
¹³C-NMR spectra of compound **22**



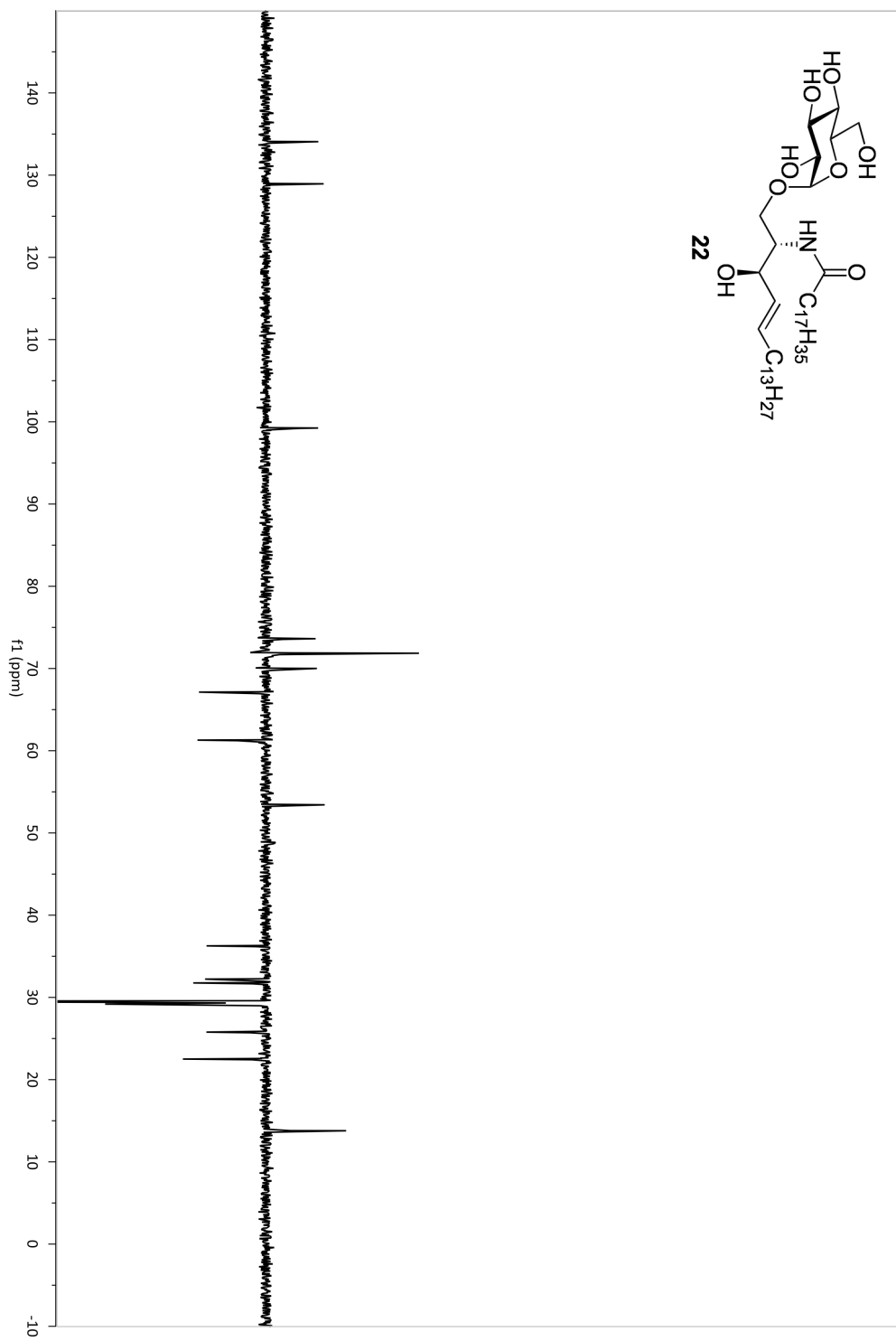
COSY-NMR spectra of compound **22**



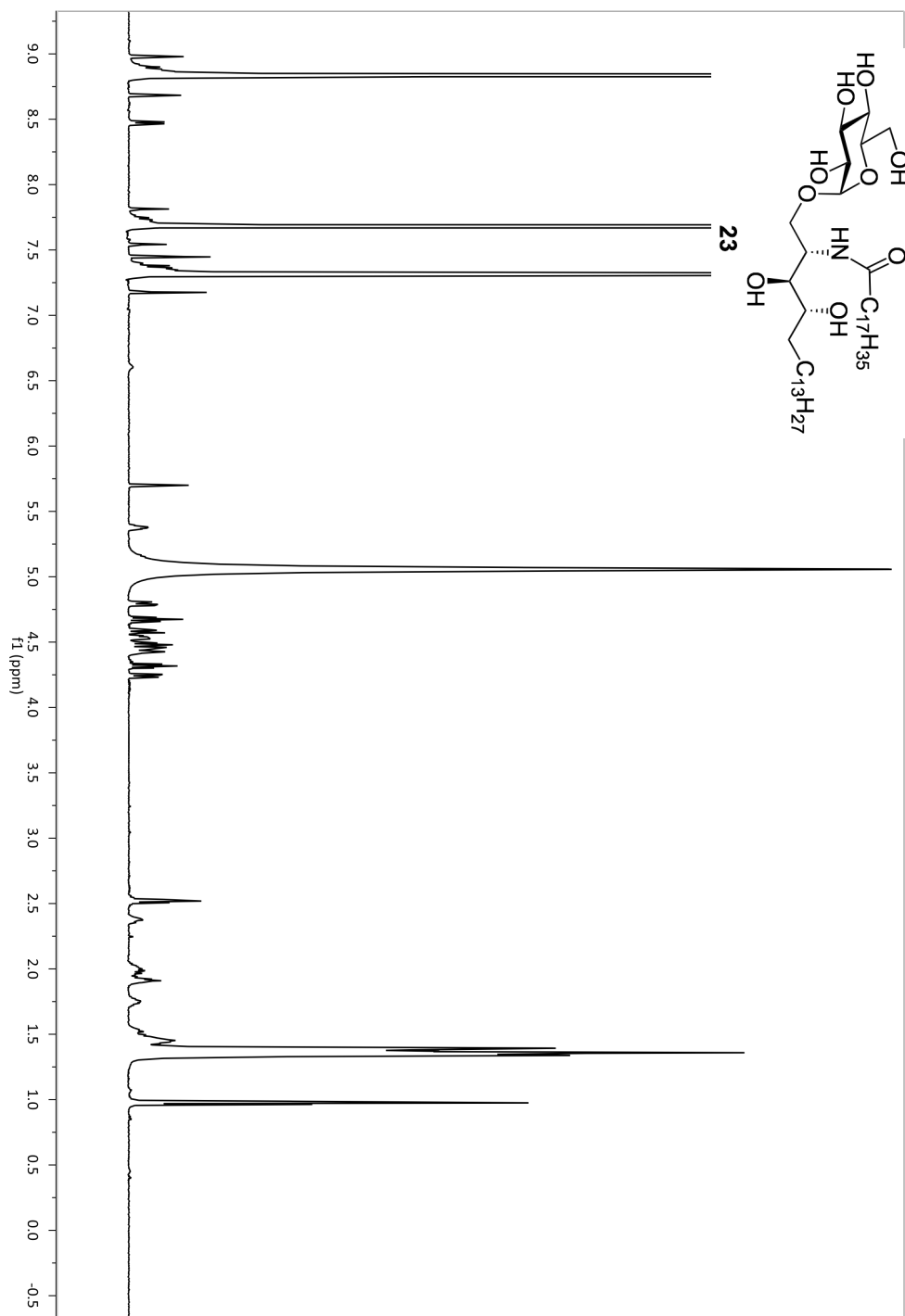
HSQC-NMR spectra of compound **22**



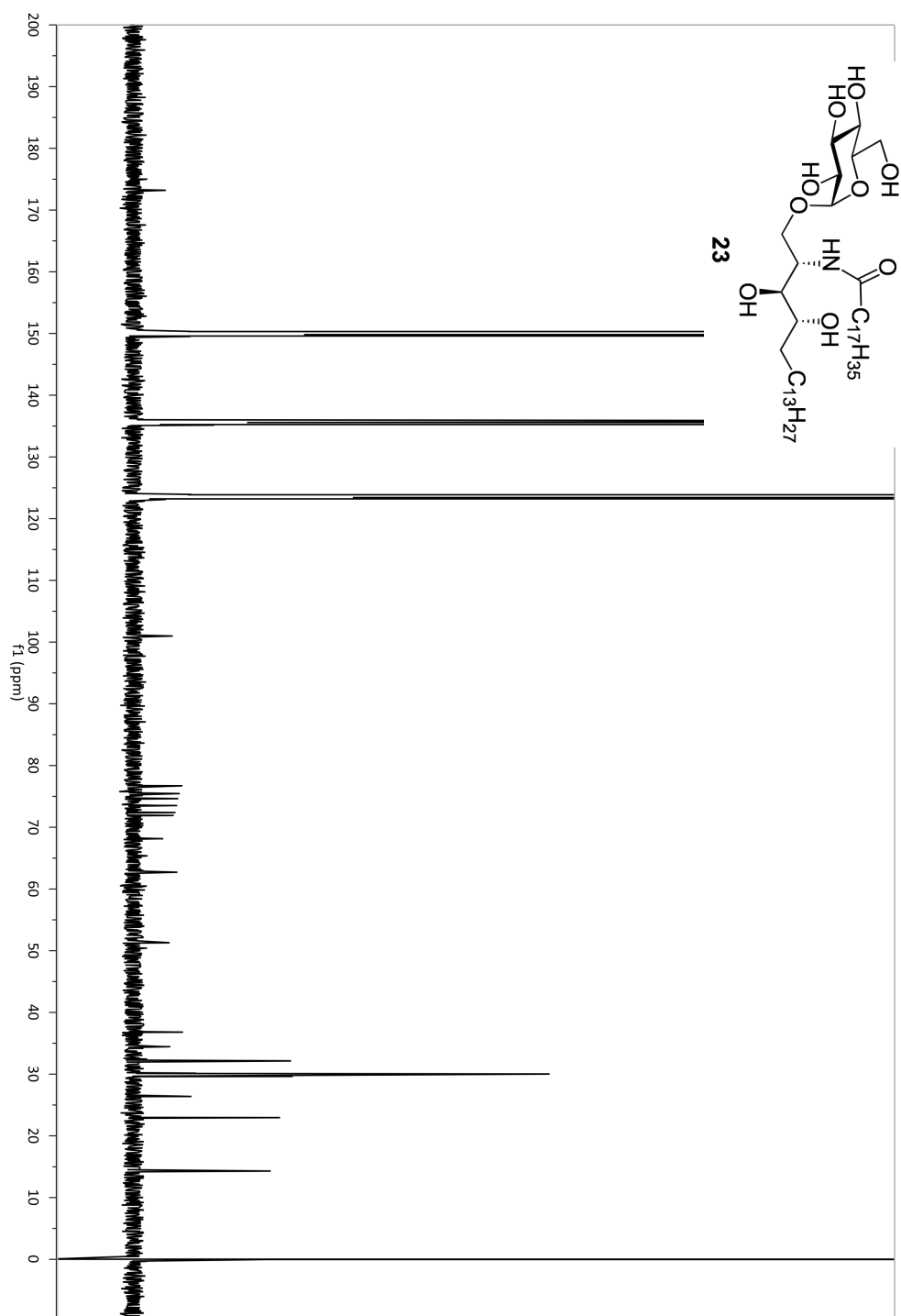
DEPT-NMR spectra of compound **22**



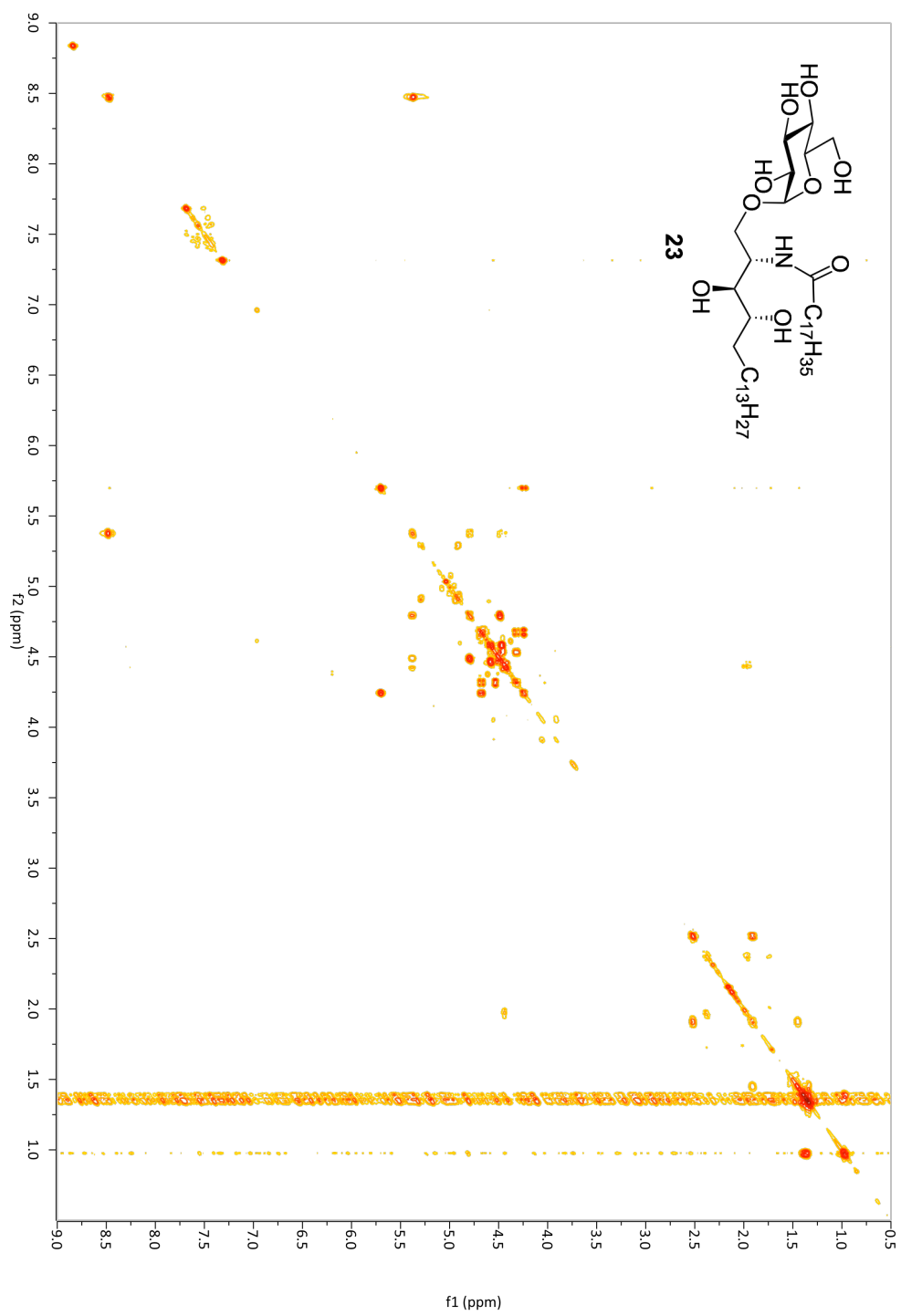
¹H-NMR spectra of compound **23**



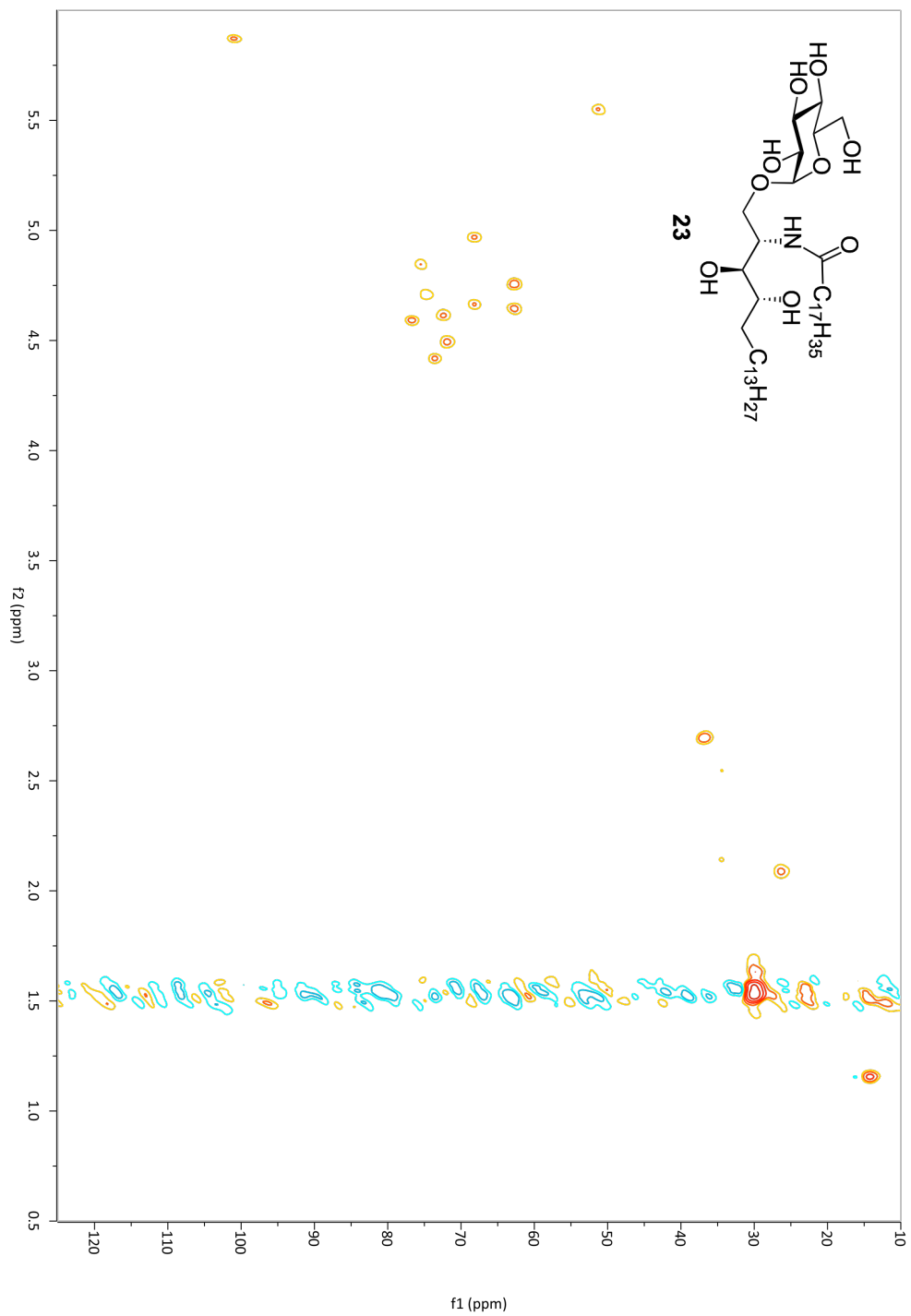
^{13}C -NMR spectra of compound **23**



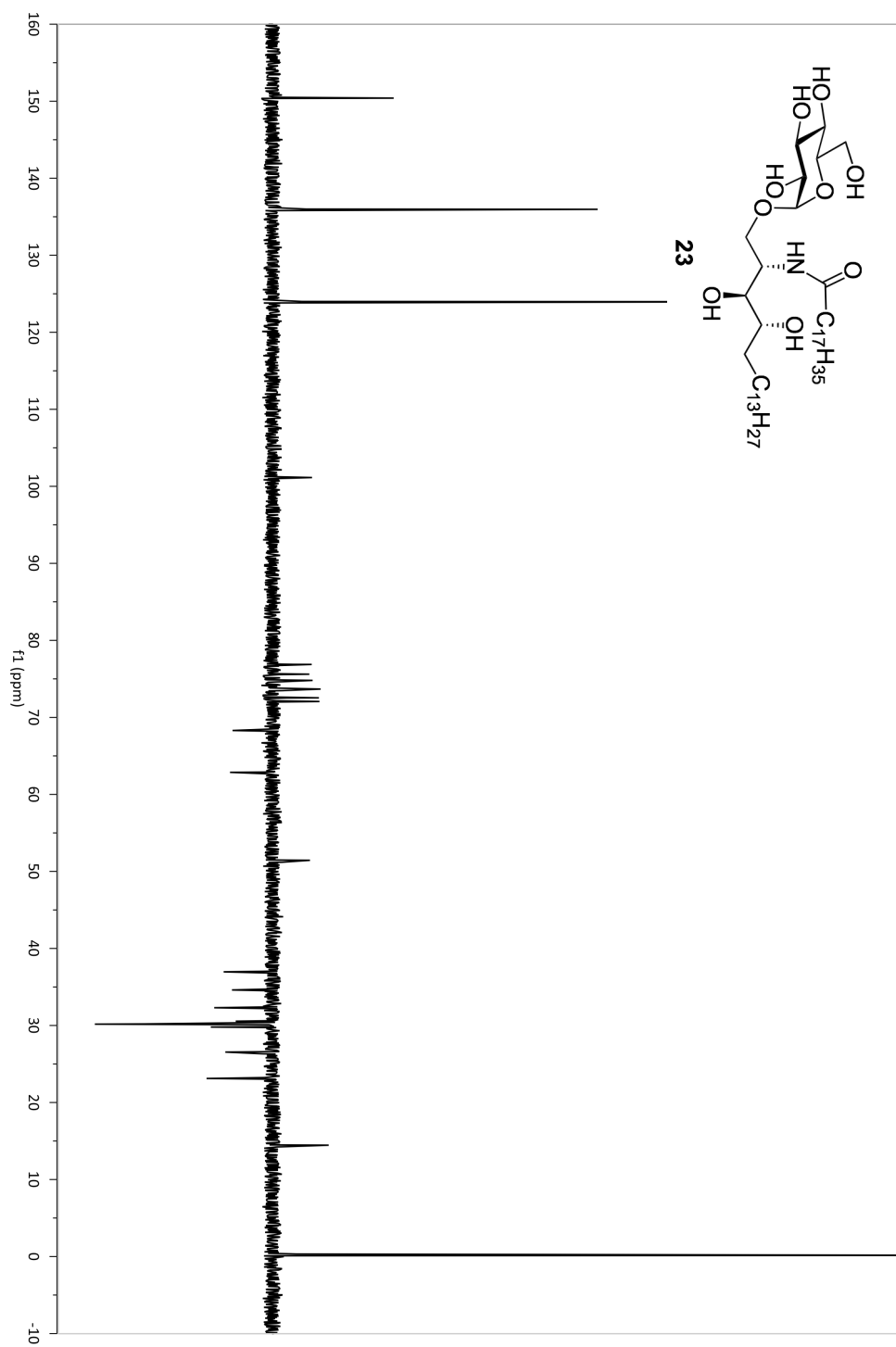
COSY-NMR spectra of compound **23**



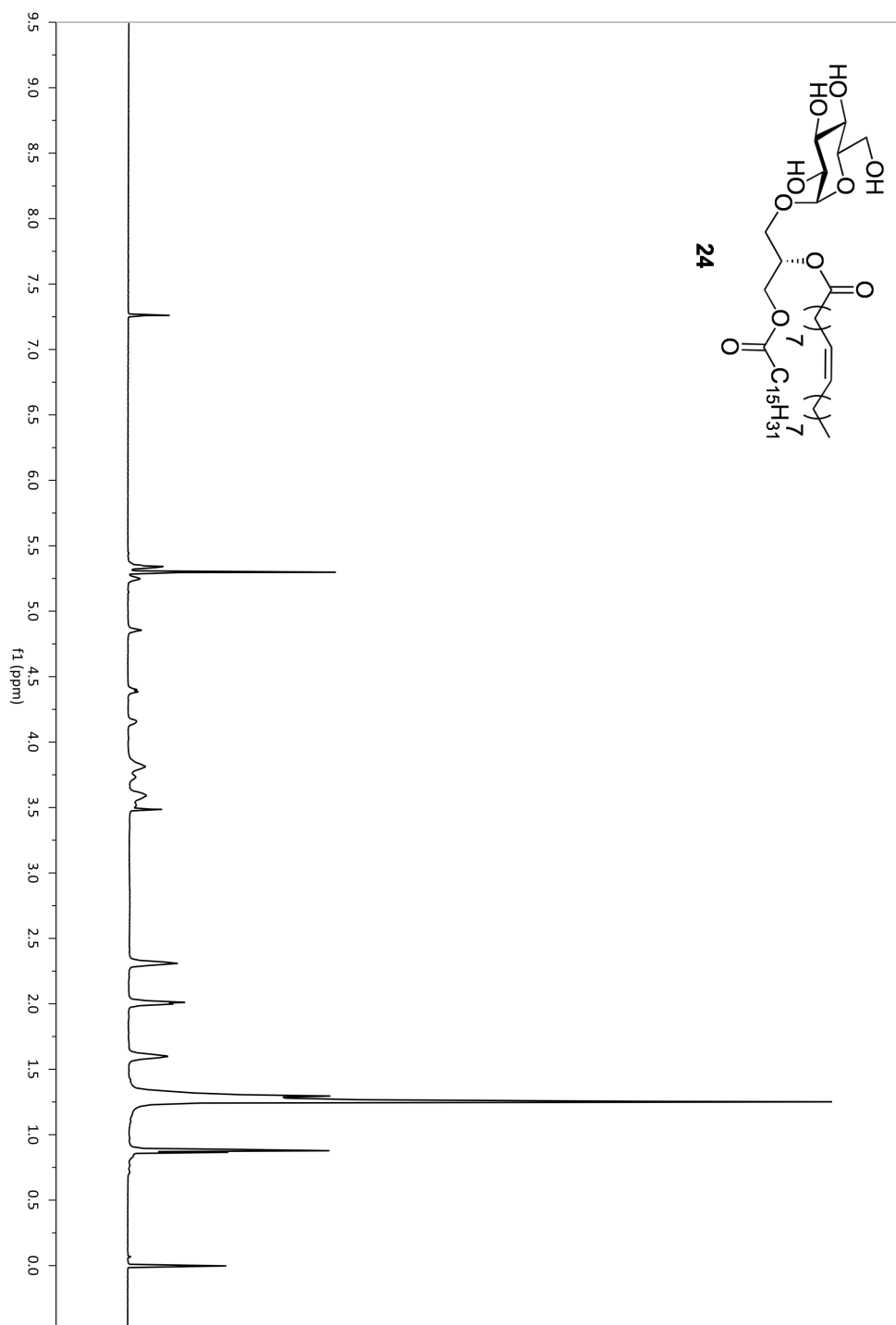
HSQC-NMR spectra of compound **23**



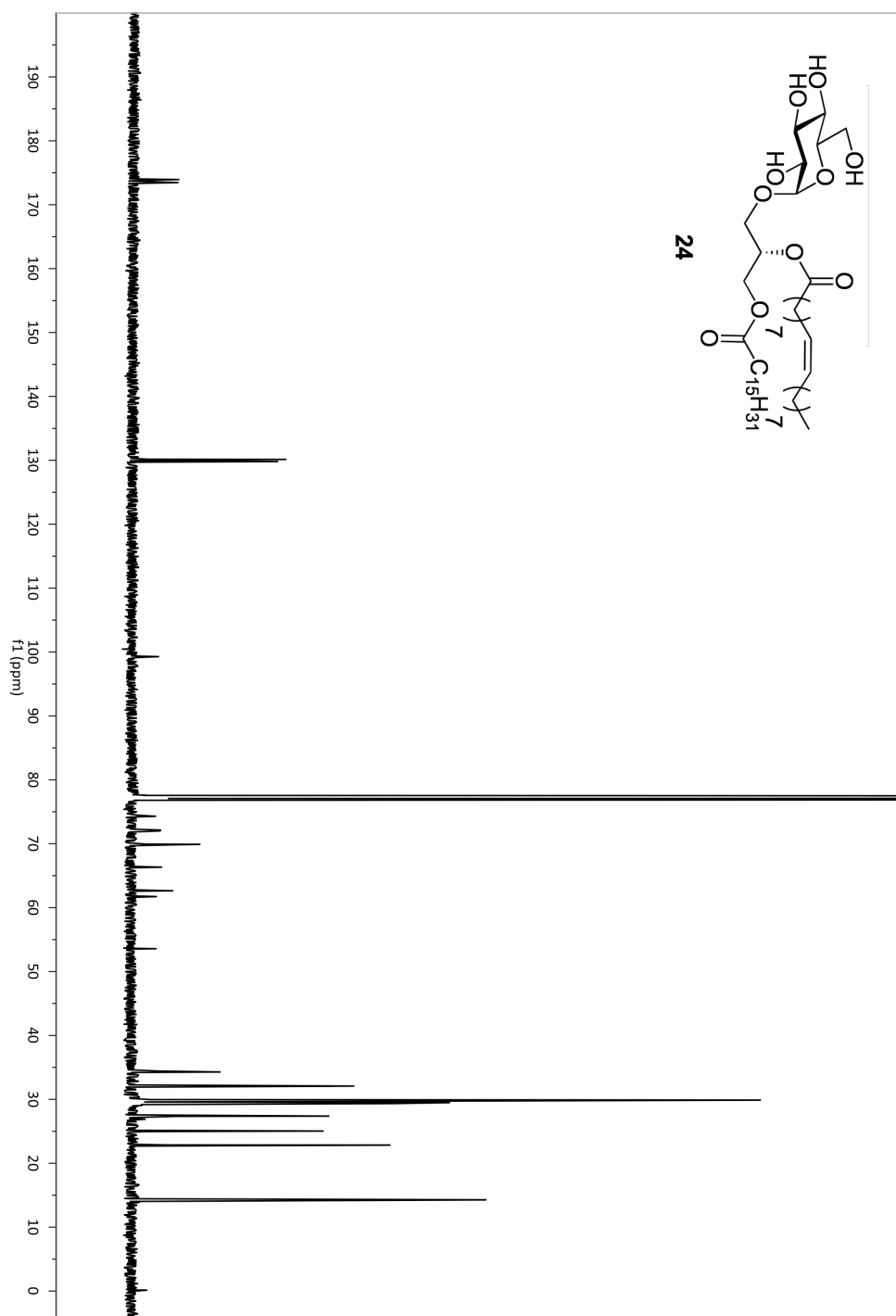
DEPT-NMR spectra of compound **23**



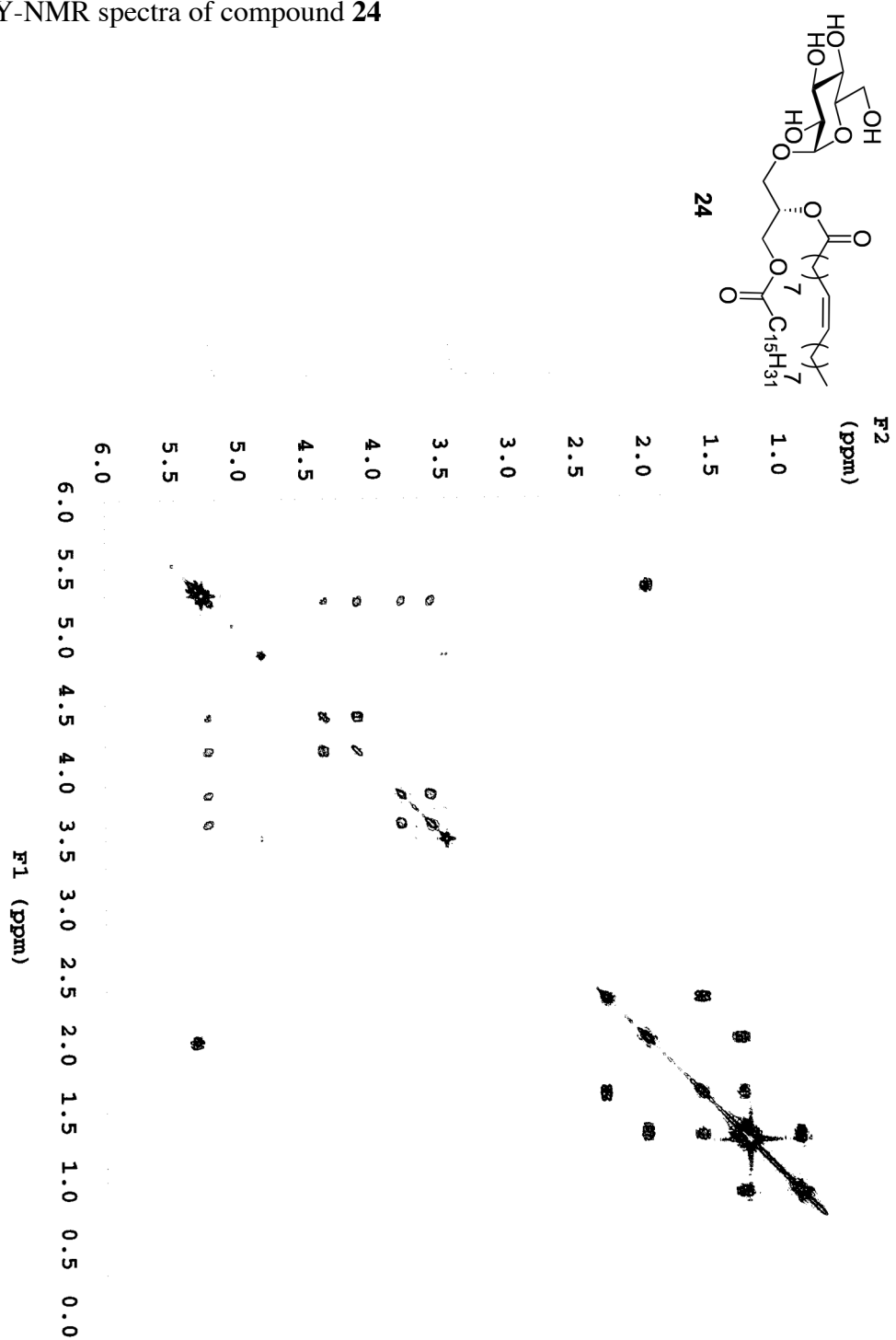
¹H-NMR spectra of compound **24**



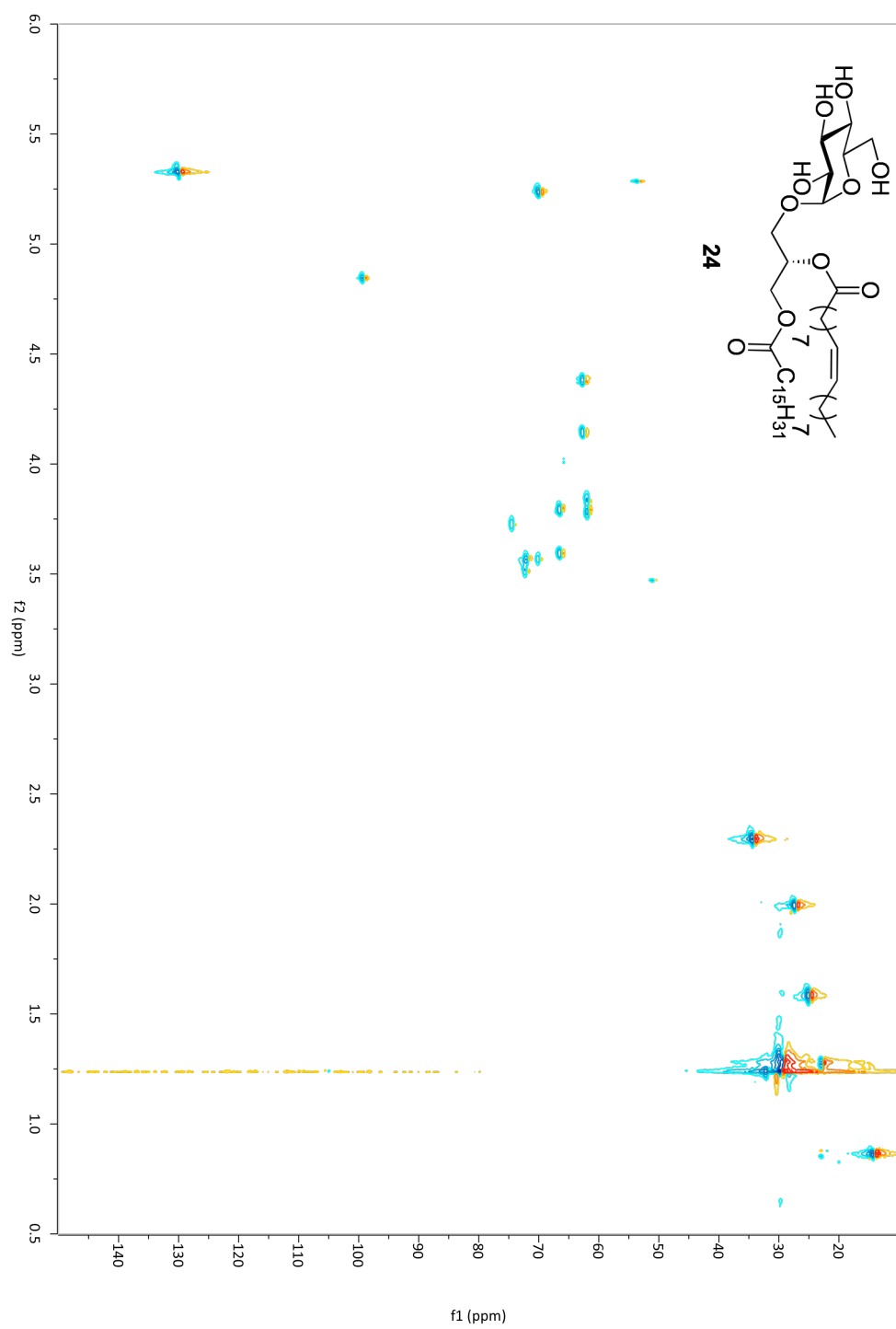
¹³C-NMR spectra of compound **24**



COSY-NMR spectra of compound **24**



HSQC-NMR spectra of compound **24**



HMBC-NMR spectra of compound **24**

