Supplementary Information

Streamlining routine organic chemistry reactions by the employment of high shear mixers.

Reyna Zeferino-Díaz,[a] J. Ciciolil Hilario-Martínez,[a] Manuel Sánchez-Cantú,*[b] María A. Fernández-Herrera[c] and Jesus Sandoval-Ramirez*[a]

a. Facultad de Ciencias Químicas. FCQ-8. Ciudad Universitaria. 72595 Puebla, Pue.
b. Facultad de Ingeniería Químicas. FIQ. Ciudad Universitaria. 72595 Puebla, Pue.
c. Departamento de Física Aplicada. Laboratorio de Biomoléculas. CINVESTAV-Mérida. 97310 Mérida, Yuc.
Corresponding authors: manuel.sanchez@correo.buap.mx; jesus.sandoval@correo.buap.mx

Table of Contents

General Information

NMR

Nuclear Magnetic Resonance spectra of ^{1}H and ^{13}C were recorded in a Bruker Avance spectrometer (500 MHz). NMR spectra were obtained in deuterated chloroform ($CDCl_3$. The chemical shifts are expressed in ppm (δ), referenced to internal C**H**Cl_3 (δ 7.26), ^{13}**C**DCl_3 (δ 77.16). and the coupling constants (J) are expressed in Hz.

HSM

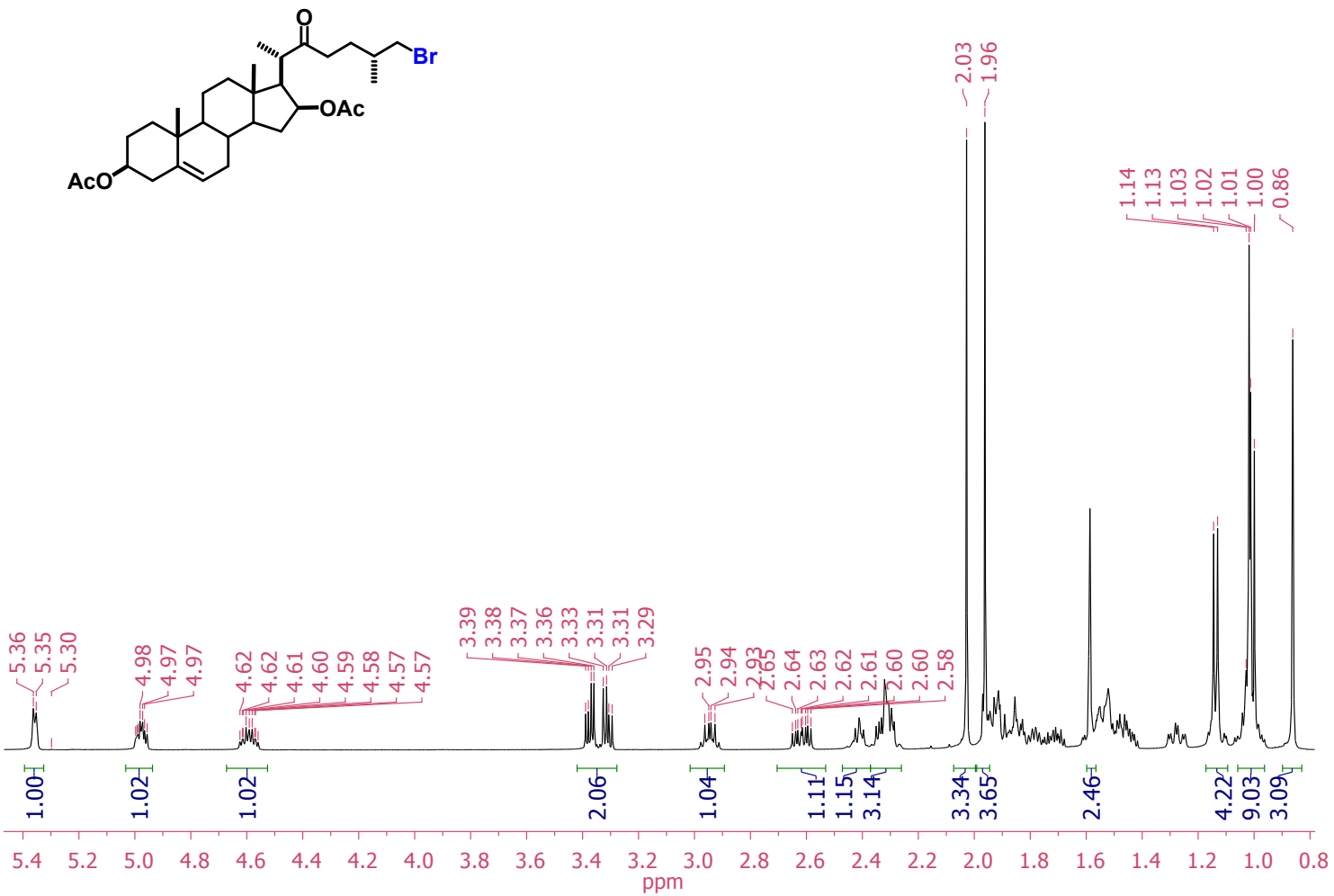
High Shear Mixing experiments were carried out using an IKA ULTRA-TURRAX® T 18 digital apparatus at 6000, 8000 and 10000 rpm.

SYNTHESIS OF AZIDOSTEROIDS

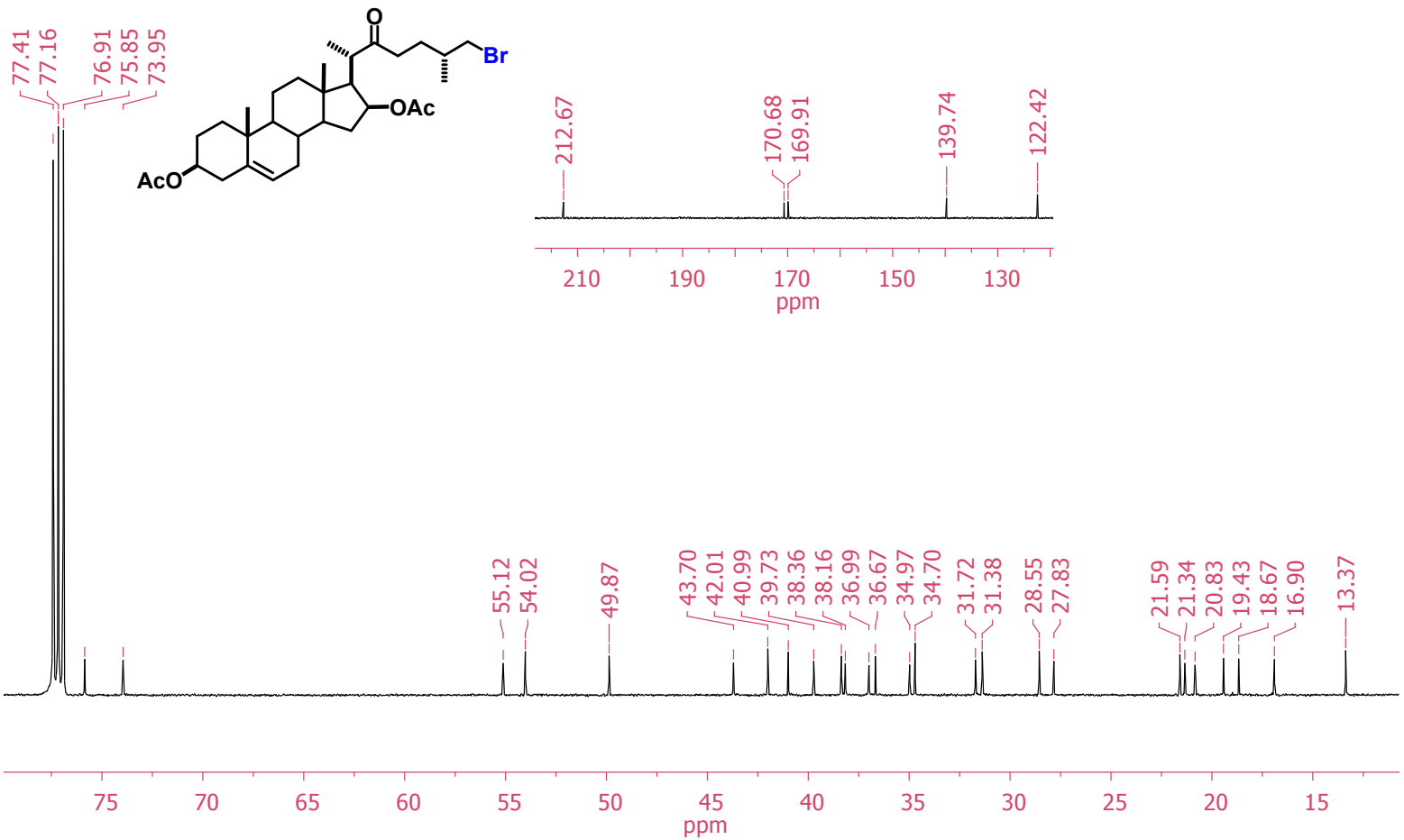
For the synthesis of azidosteroids, sodium azide (1.5 eq) was added to a solution of 26-substituted-22-oxocholestanes **1a** or **1b** (500 mg, 0.8 mmol) in DMF (6 mL). The reaction mixture was stirred using a magnetic stirrer (300 rpm) or the HSM (6000, and 8000 rpm) at 25 °C and 65 ºC. DMF was removed from aliquots of the reaction mixtures, and dried samples were analyzed by ^{1}H-NMR

SYNTHESIS OF PENTA AND TETRAACETYLATED GLUCOSE

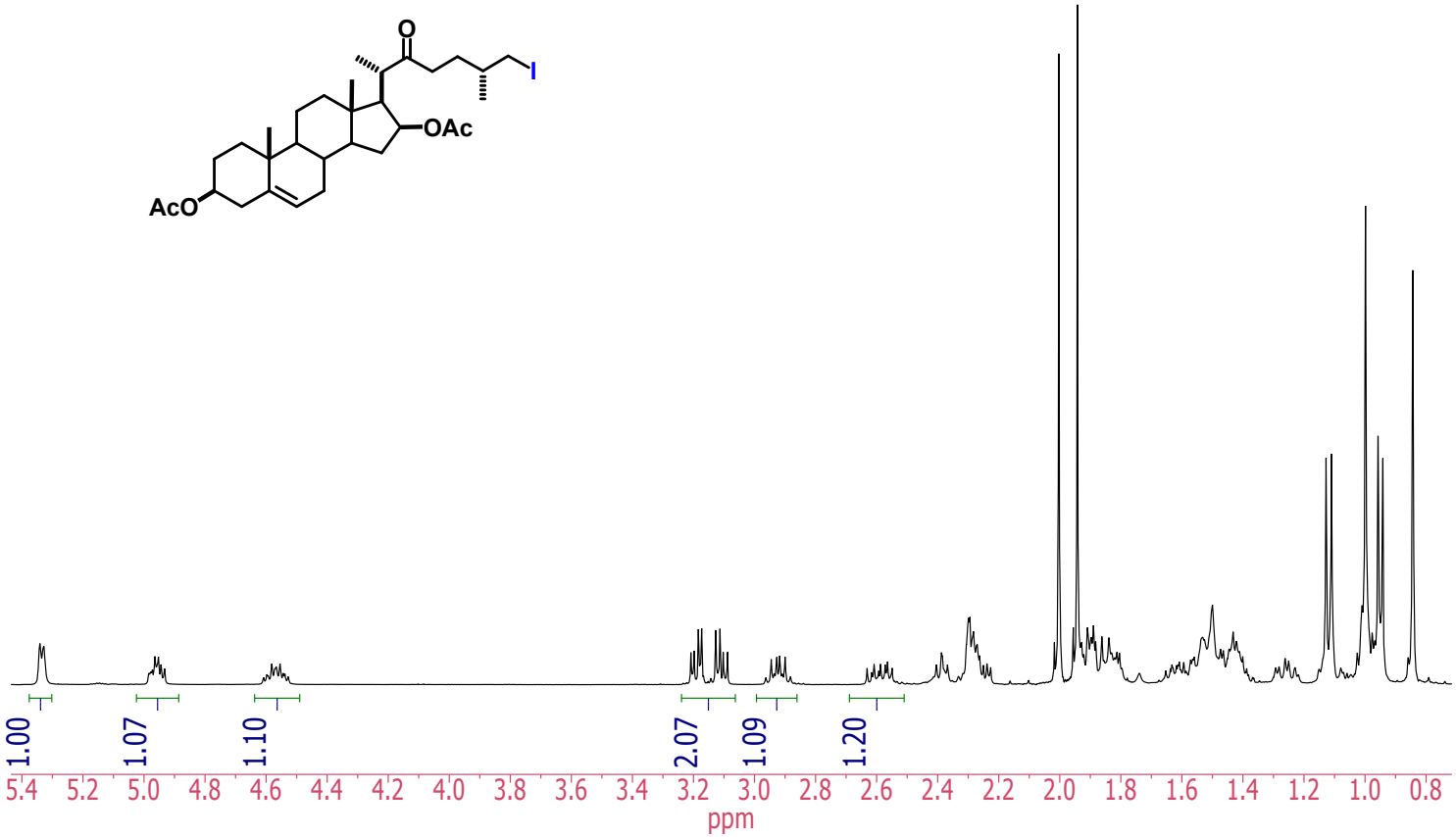
Glucose (2 g, 11.1 mmol) was suspended in 5 ml of CH_2Cl_2 and 5.8 ml of Ac_2O (5.5 eq); then 5% mol of DMAP was added and stirred at 300, 8000, and 10000 rpm. Reactions were followed by ^{1}H-NMR. After complete conversion, the organic phase was extracted with CH_2Cl_2 and the extract was washed with a saturated solution of $NaHCO_3$ (5 x 100 mL), water (3 x 100 mL), dried over Na_2SO_4, and concentrated under vacuum. The pentaacetylated glucose (**4**) was obtained quantitatively, as a syrup. For a selective deprotection reaction, the peracetylated glucose **4** (2 g, 5.12 mmol) was dissolved in 10 ml DMF, and 1.5 eq of hydrazine acetate were added. The reaction mixture was stirred at 300, 8000, and 10000 rpm. The organic phase was extracted with AcOEt (100 mL) and washed with saturated $NaHCO_3$ solution (3 x 100 mL), water (3 x 100 mL), dried over anhydrous Na_2SO_4 and concentrated under reduced pressure to obtain a syrup of the tetraacetylated glucose (**5**).



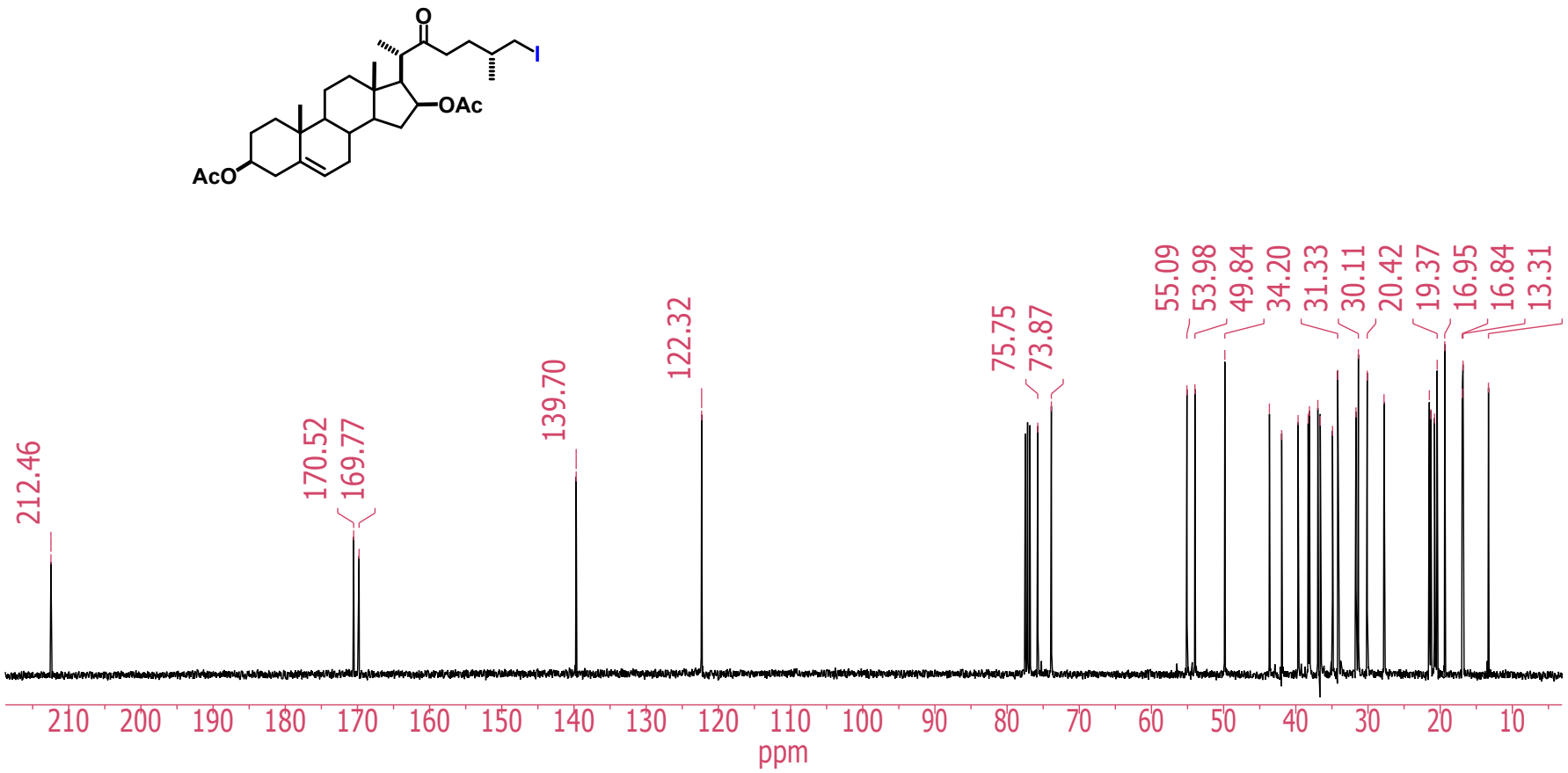
^{1}H NMR Spectrum of (25*R*)-26-bromo-22-oxocholest-5-ene-3β,16β-diyl diacetate (**1a**, 500 MHz, $CDCl_3$).



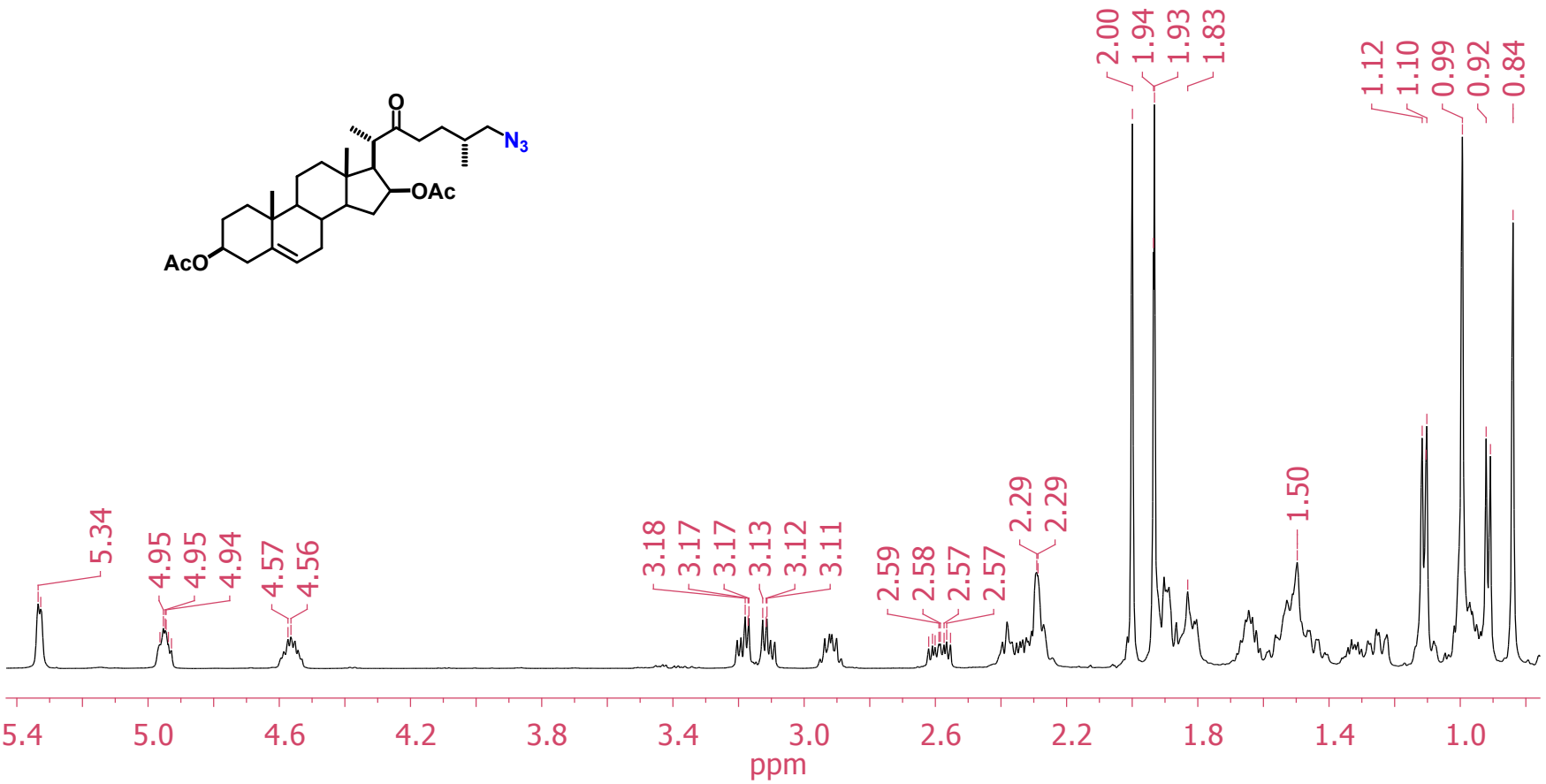
^{13}C NMR Spectrum of (25*R*)-26-bromo-22-oxocholest-5-ene-3β,16β-diyl diacetate (**1a**, 125 MHz, $CDCl_3$).



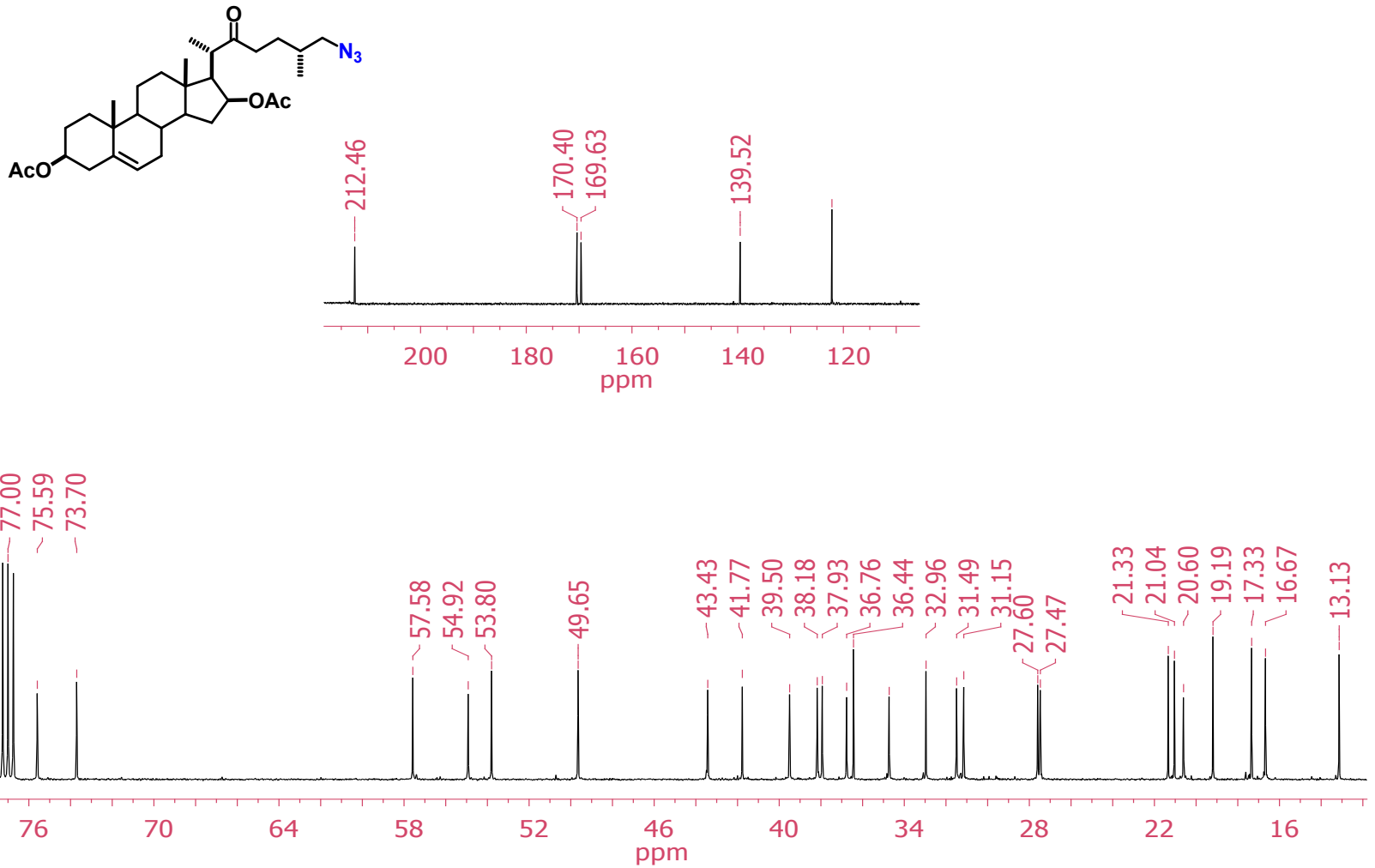
^{1}H Spectrum of (25*R*)-26-iodo-22-oxocholest-5-ene-3β,16β-diyl diacetate (**1b**, 500 MHz, $CDCl_3$).



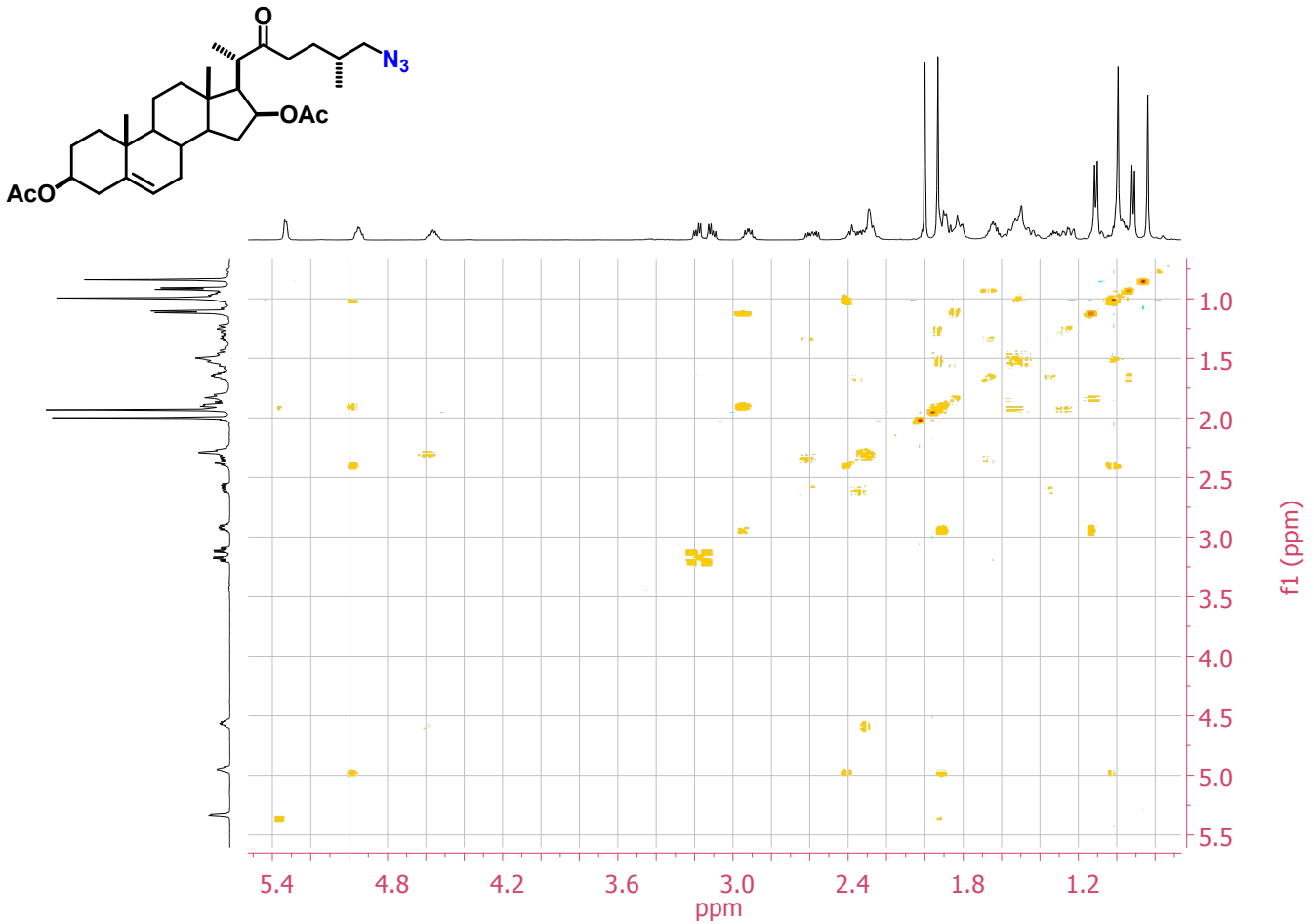
^{13}C NMR Spectrum of (25*R*)-26-iodo-22-oxocholest-5-ene-3β,16β-diyl diacetate (**1b**, 125 MHz, $CDCl_3$).



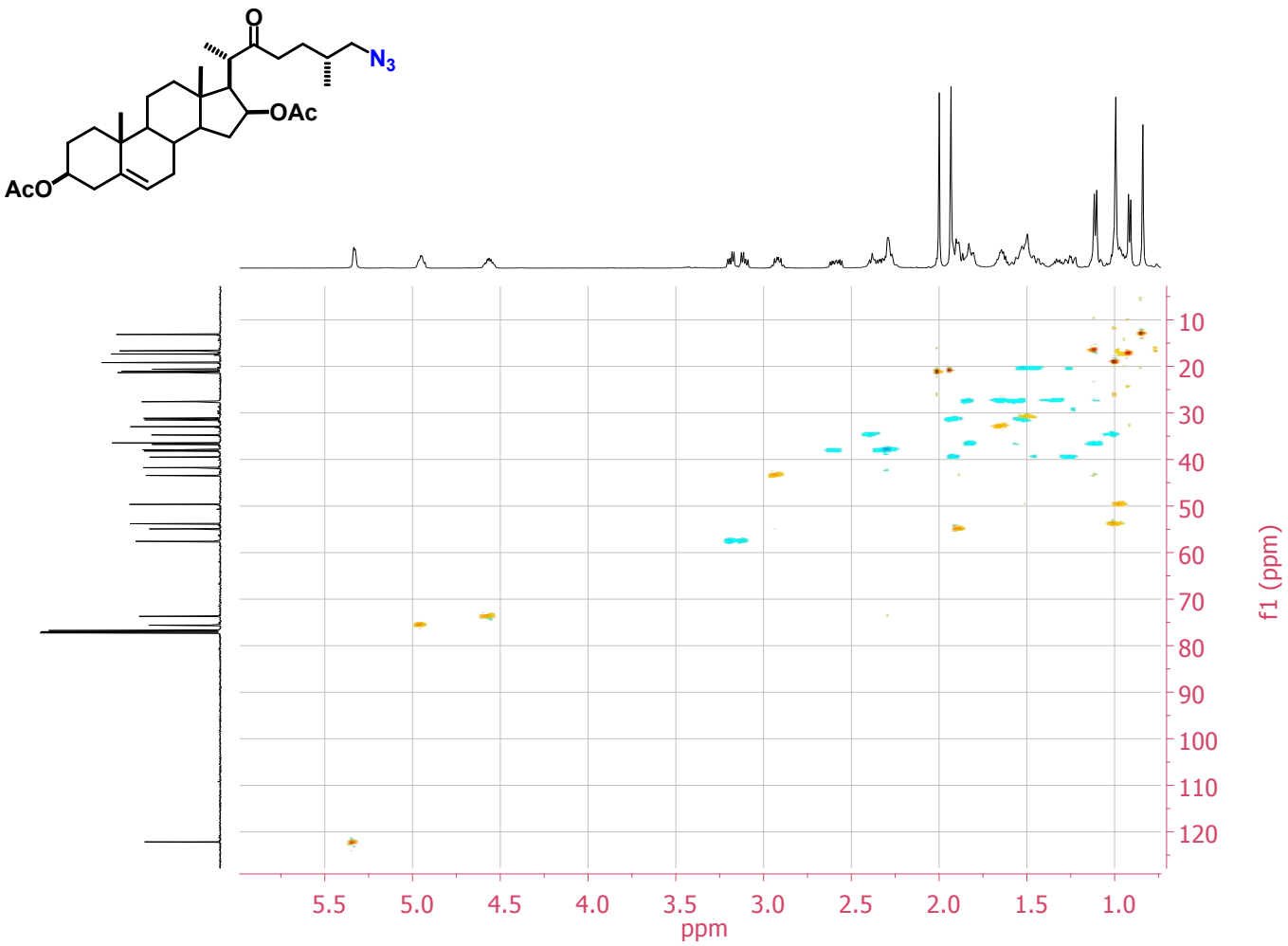
^{1}H NMR Spectrum of (25*R*)-26-azido-22-oxocholest-5-ene-3β,16β-diyl diacetate (**2**, 500 MHz, $CDCl_3$).



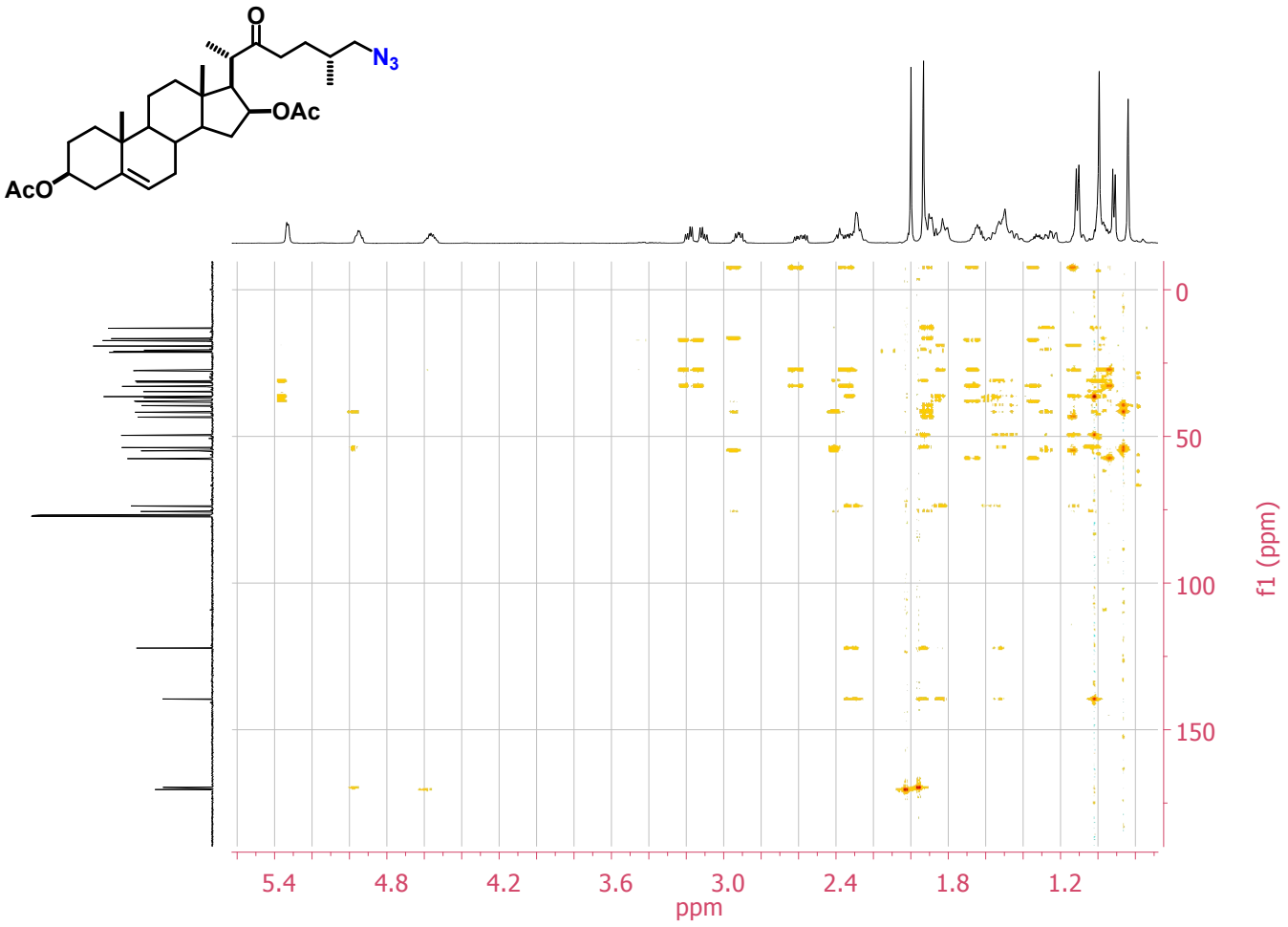
^{13}C NMR Spectrum of (25*R*)-26-azido-22-oxocholest-5-ene-3β,16β-diyl diacetate (**2**, 125 MHz, $CDCl_3$).



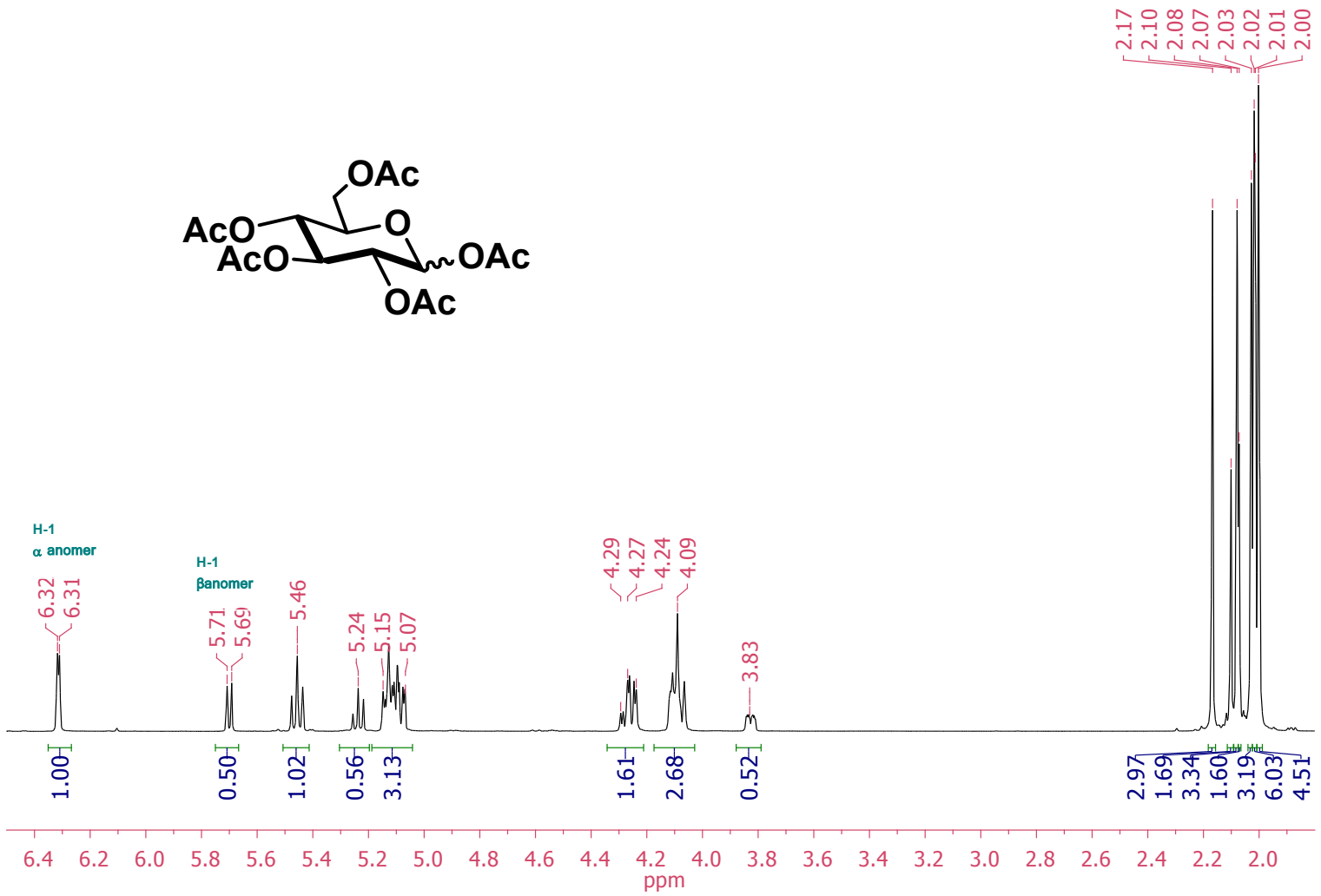
COSY 2D NMR experiment of (25*R*)-26-azido-22-oxocholest-5-ene-3β,16β-diyl diacetate (**2**, $CDCl_3$).



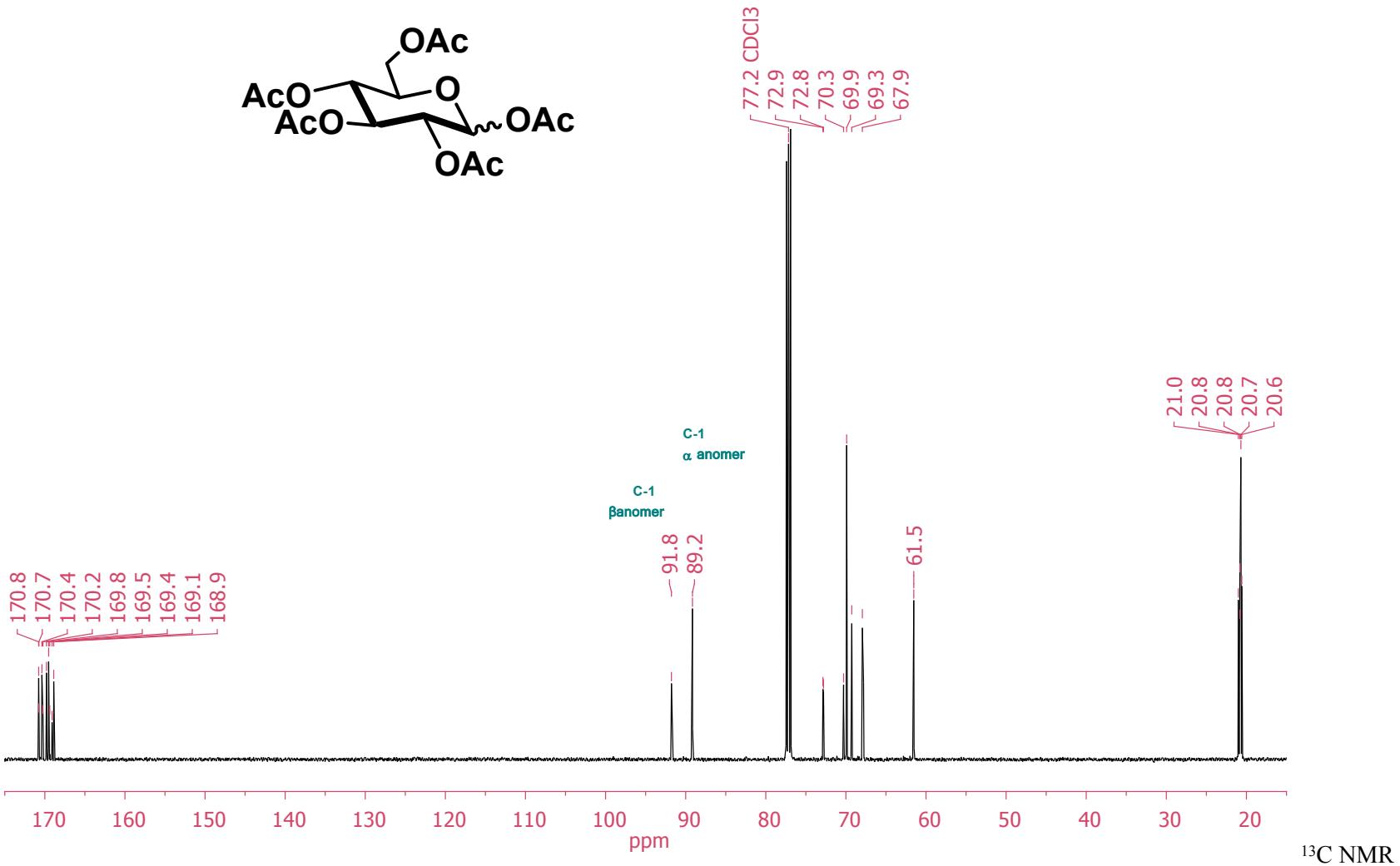
HSQC 2D NMR experiment of (25*R*)-26-azido-22-oxocholest-5-ene-3β,16β-diyl diacetate (**2**, $CDCl_3$).



HMBC 2D NMR experiment of (25*R*)-26-azido-22-oxocholest-5-ene-3β,16β-diyl diacetate (**2**, $CDCl_3$).

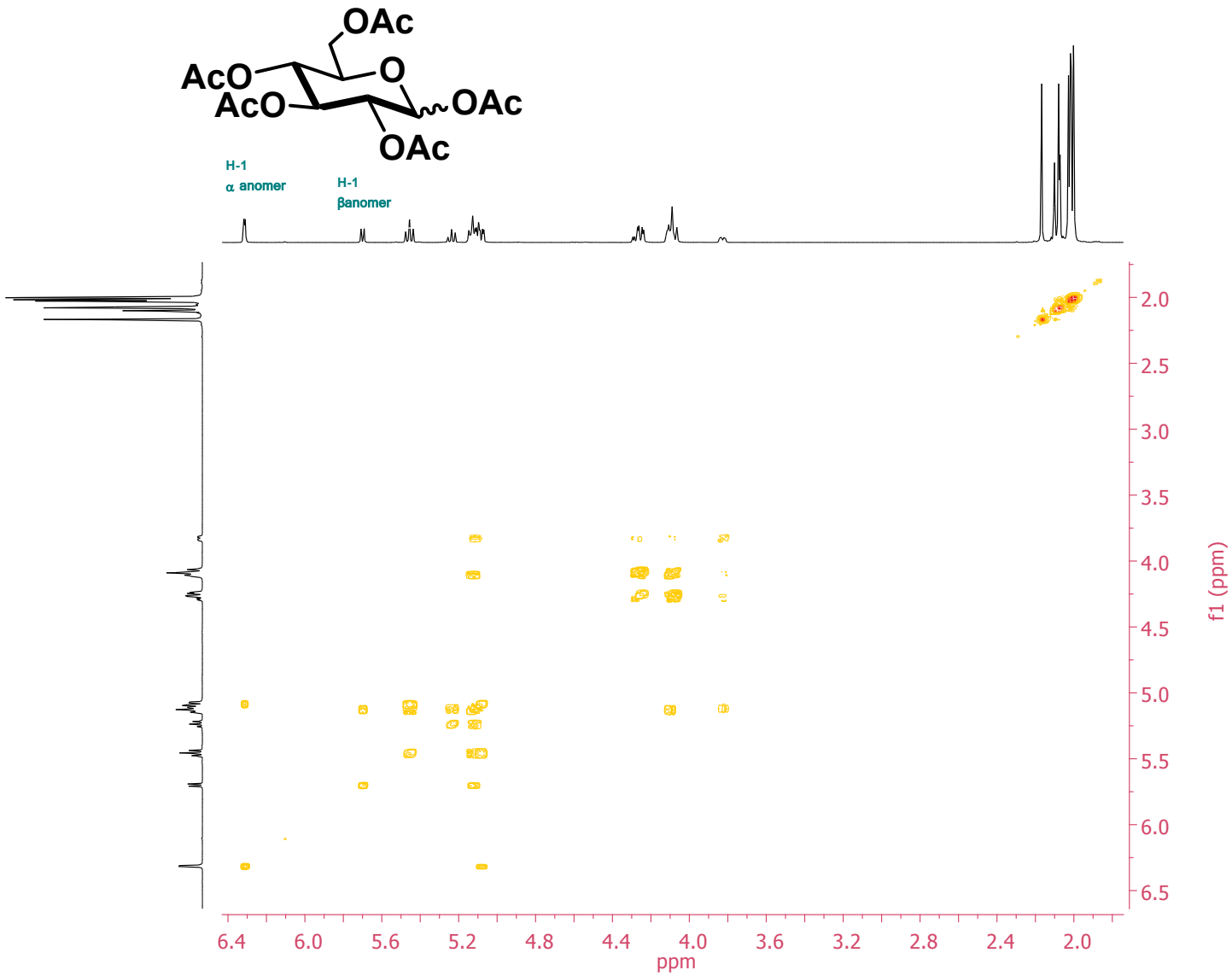


^{1}H NMR Spectrum of ξ-D-glucopyranose pentaacetate (**4**, 500 MHz, $CDCl_3$).

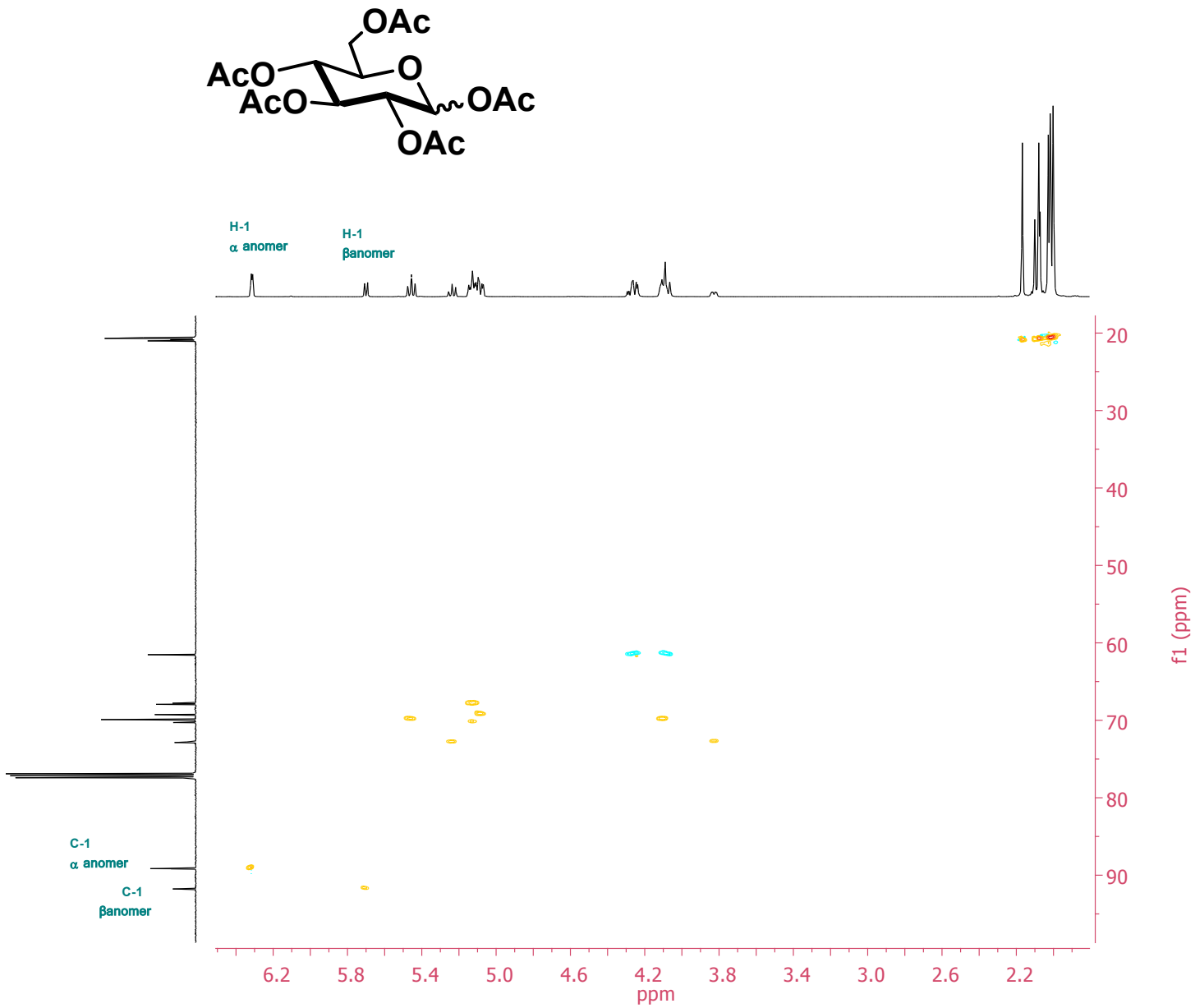


^{13}C NMR

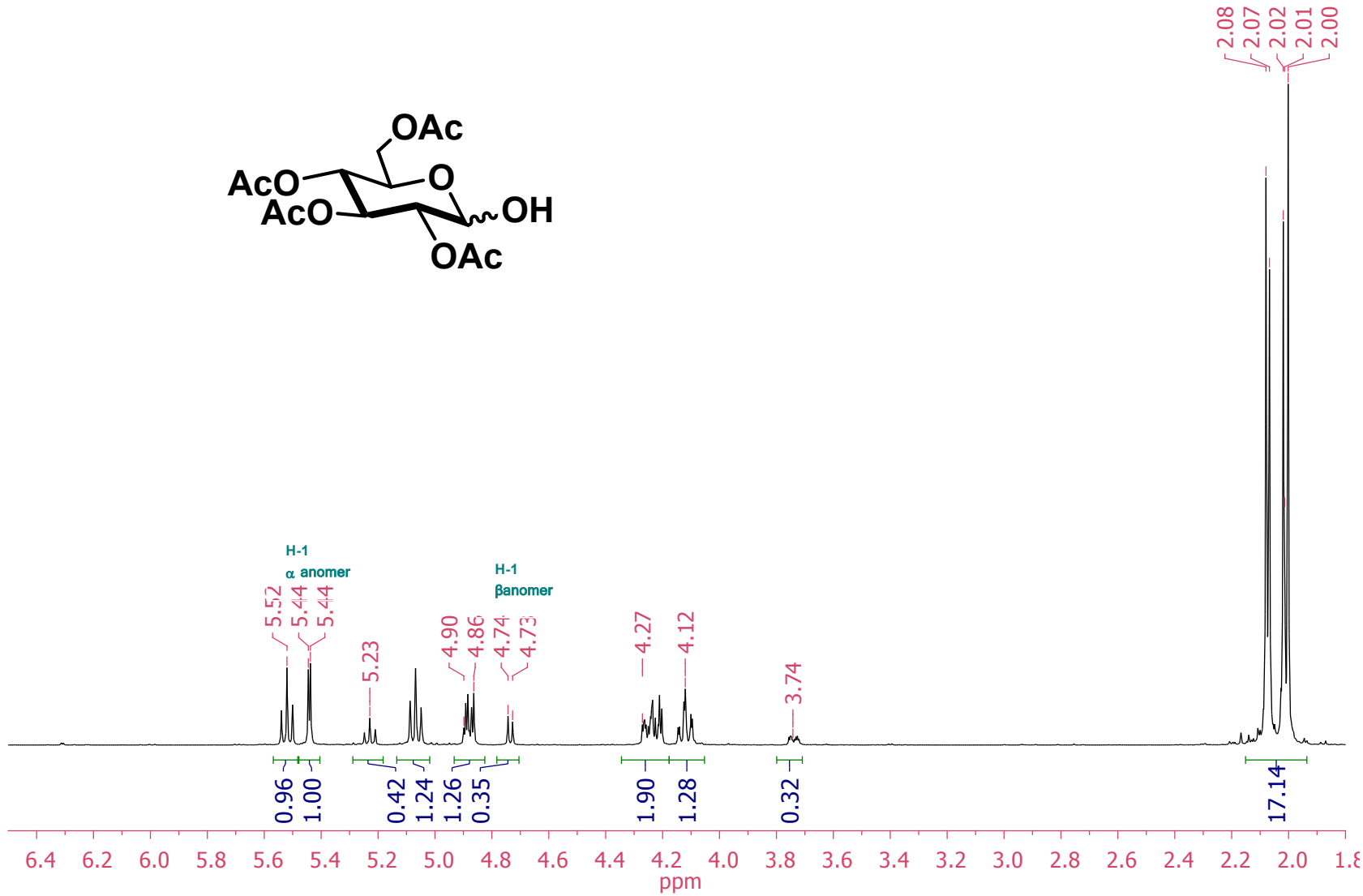
Spectrum of ξ-D-glucopyranose pentaacetate (**4**, 125 MHz, $CDCl_3$).



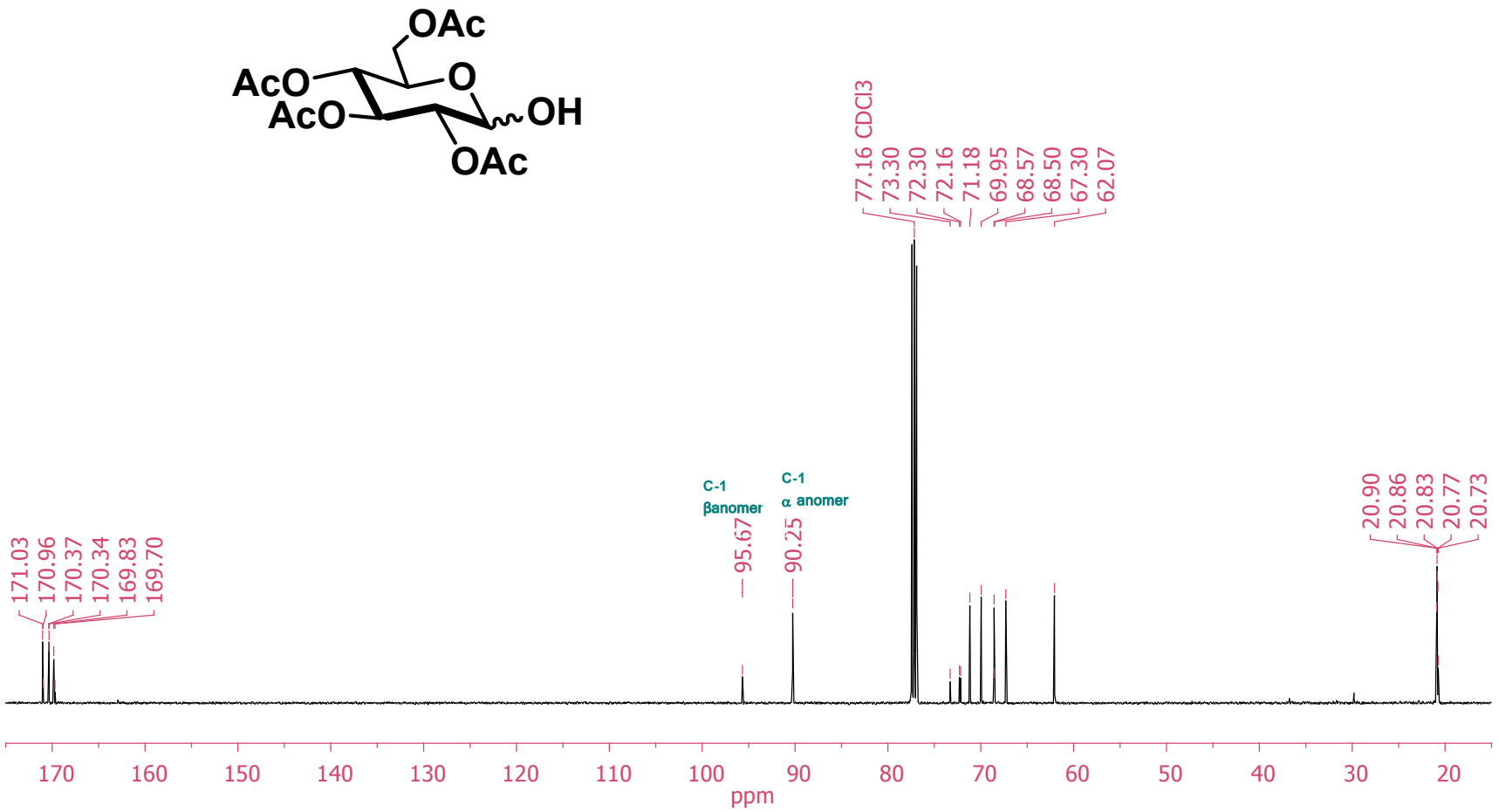
COSY 2D NMR experiment of ξ-D-glucopyranose pentaacetate (**4**, $CDCl_3$).



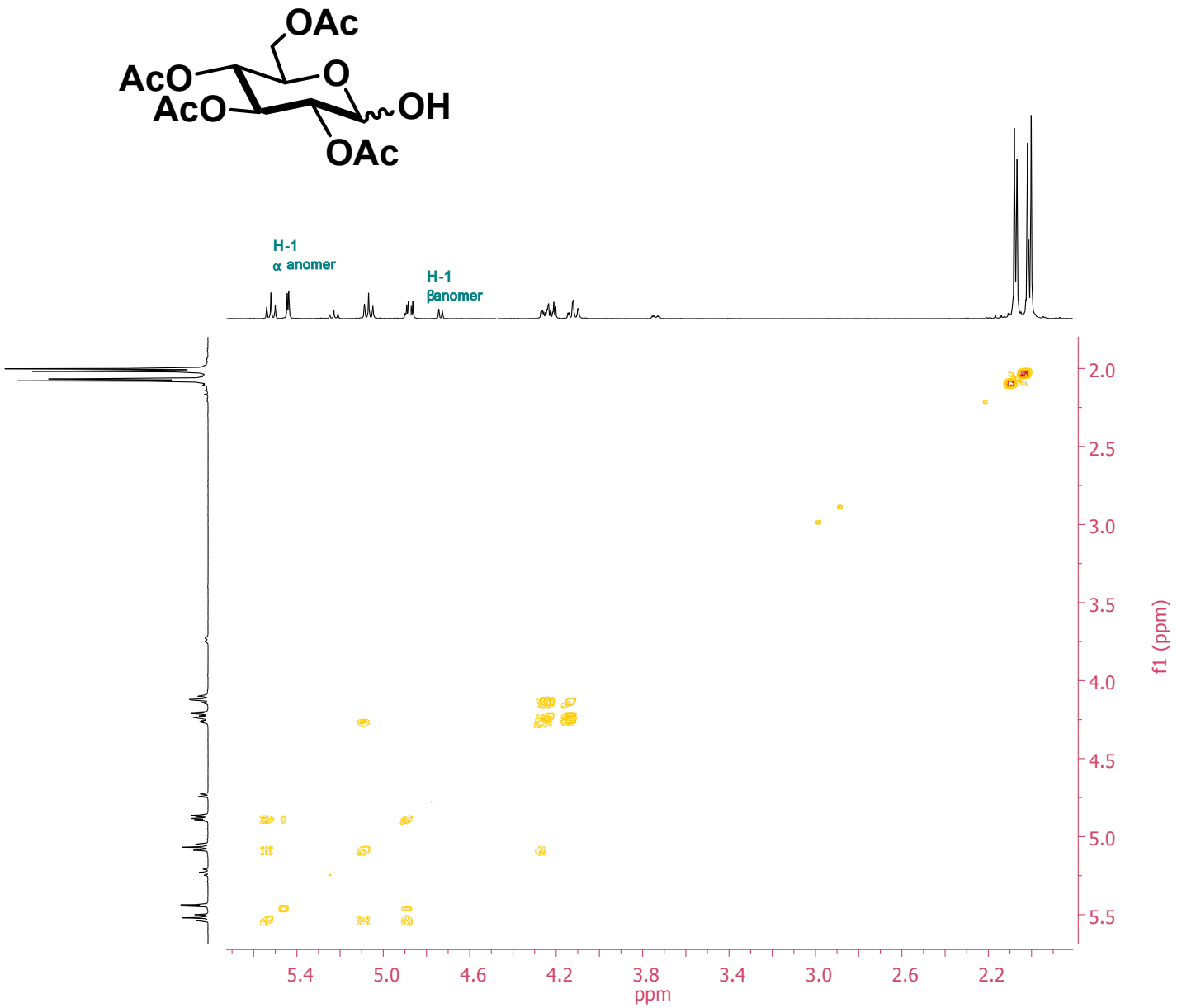
HSQC 2D NMR experiment of ξ-D-glucopyranose pentaacetate (**4**, $CDCl_3$).



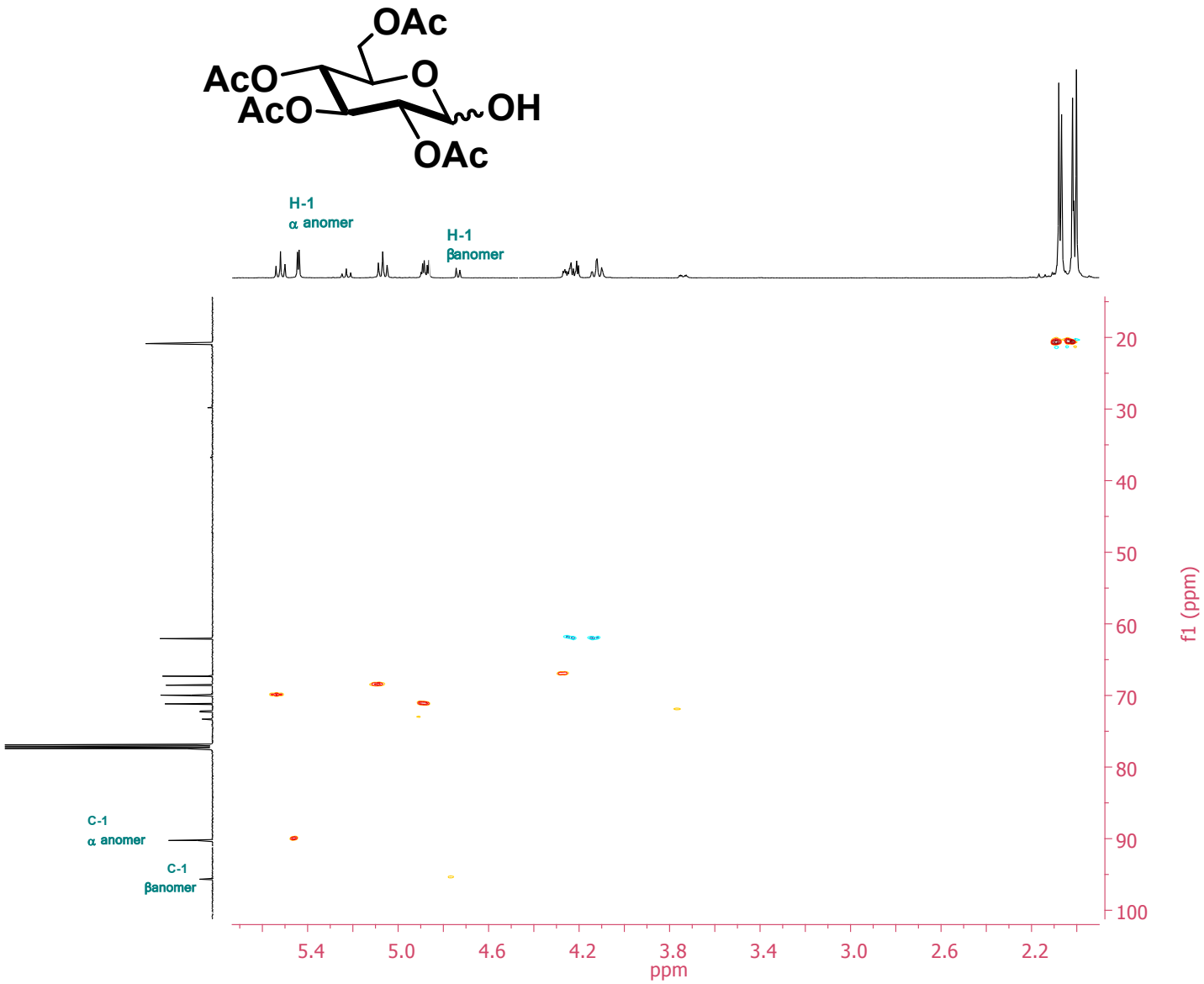
^{1}H NMR Spectrum of ξ-D-glucopyranose tetraacetate (**5**, 500 MHz, $CDCl_3$).



^{13}C NMR Spectrum of ξ-D-glucopyranose tretraacetate (**4**, 125 MHz, $CDCl_3$).



COSY 2D NMR experiment of ξ-D-glucopyranose tetraaacetate (**4**, $CDCl_3$).



HSQC 2D NMR experiment of ξ-D-glucopyranose tetraaacetate (**4**, $CDCl_3$).