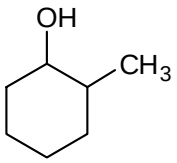
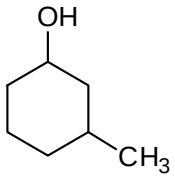
Chemistry 4361/8361 Monday, October 15

In-Class Exercise:
Heteronuclear Correlations in HMQC

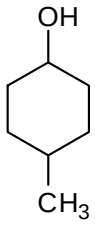
The ^{1}H, ^{13}C, ^{1}H-^{1}H COSY, and ^{1}H-decoupled HMQC spectra shown on the following pages are of a methylcyclohexanol. The primary goal of this problem is to determine which methylcyclohexanol structure matches the spectra.



2-methyl-cyclohexanol



3-methyl-cyclohexanol



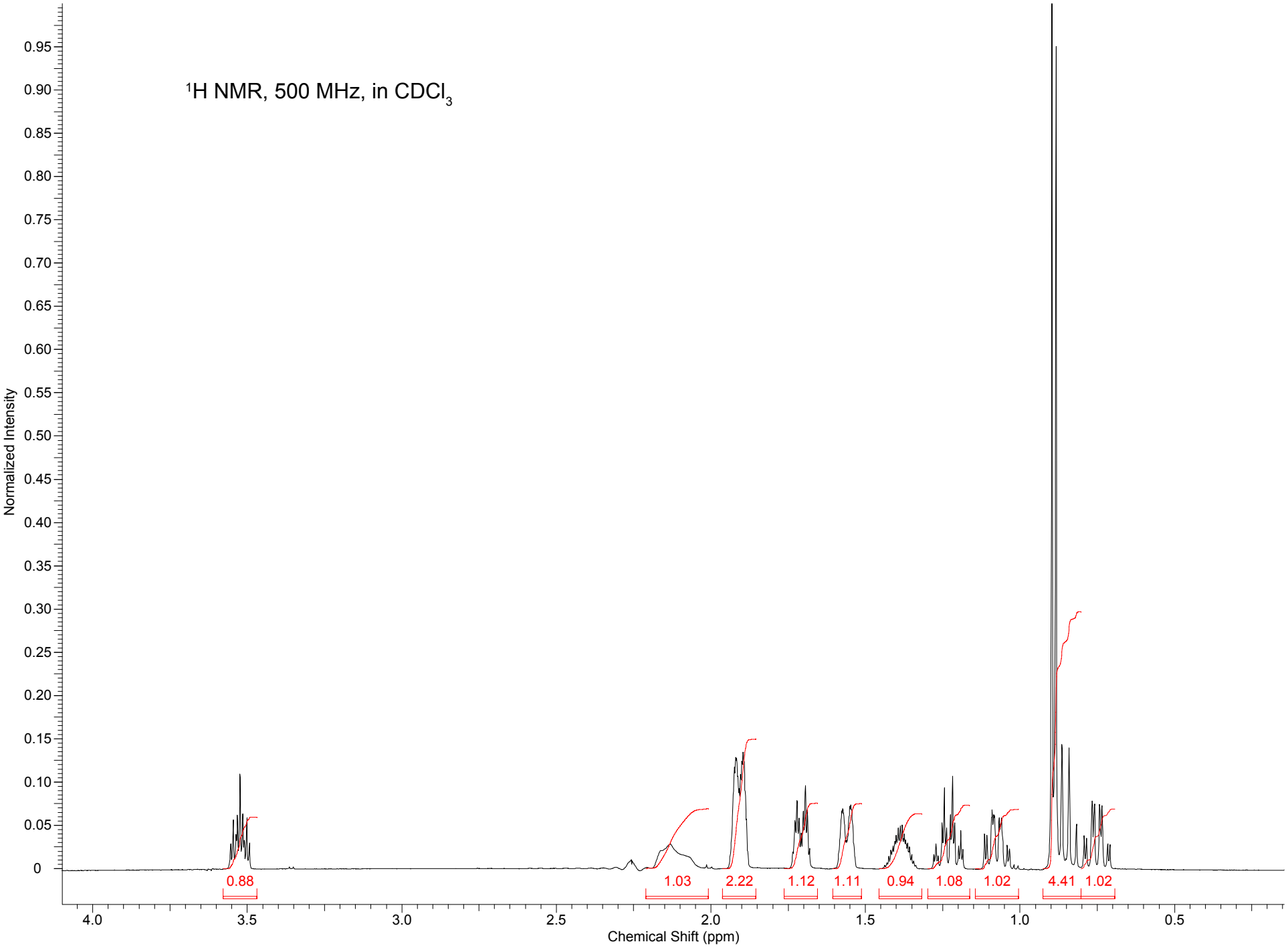
4-methyl-cyclohexanol

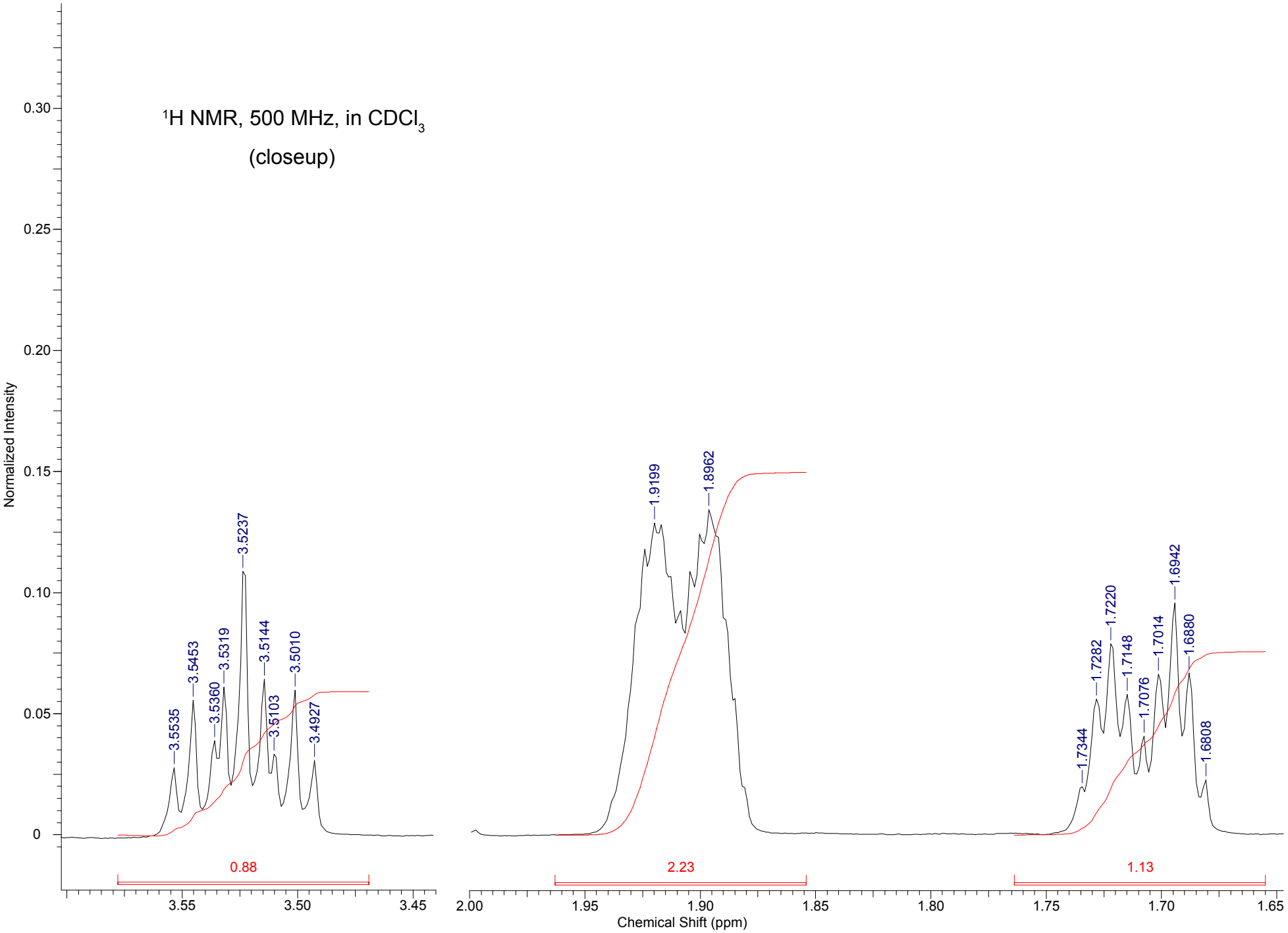
a. Assign chemical shifts to each of the protons and carbons in one of the three structures shown above. Do not worry about assigning *cis*- or *trans*- to the two substituents just yet; simply locate the substituents on the ring. Also, in cases where two protons are attached to the same carbon, don't worry about which one is which (axial vs. equatorial, up vs. down) in this part of the problem.

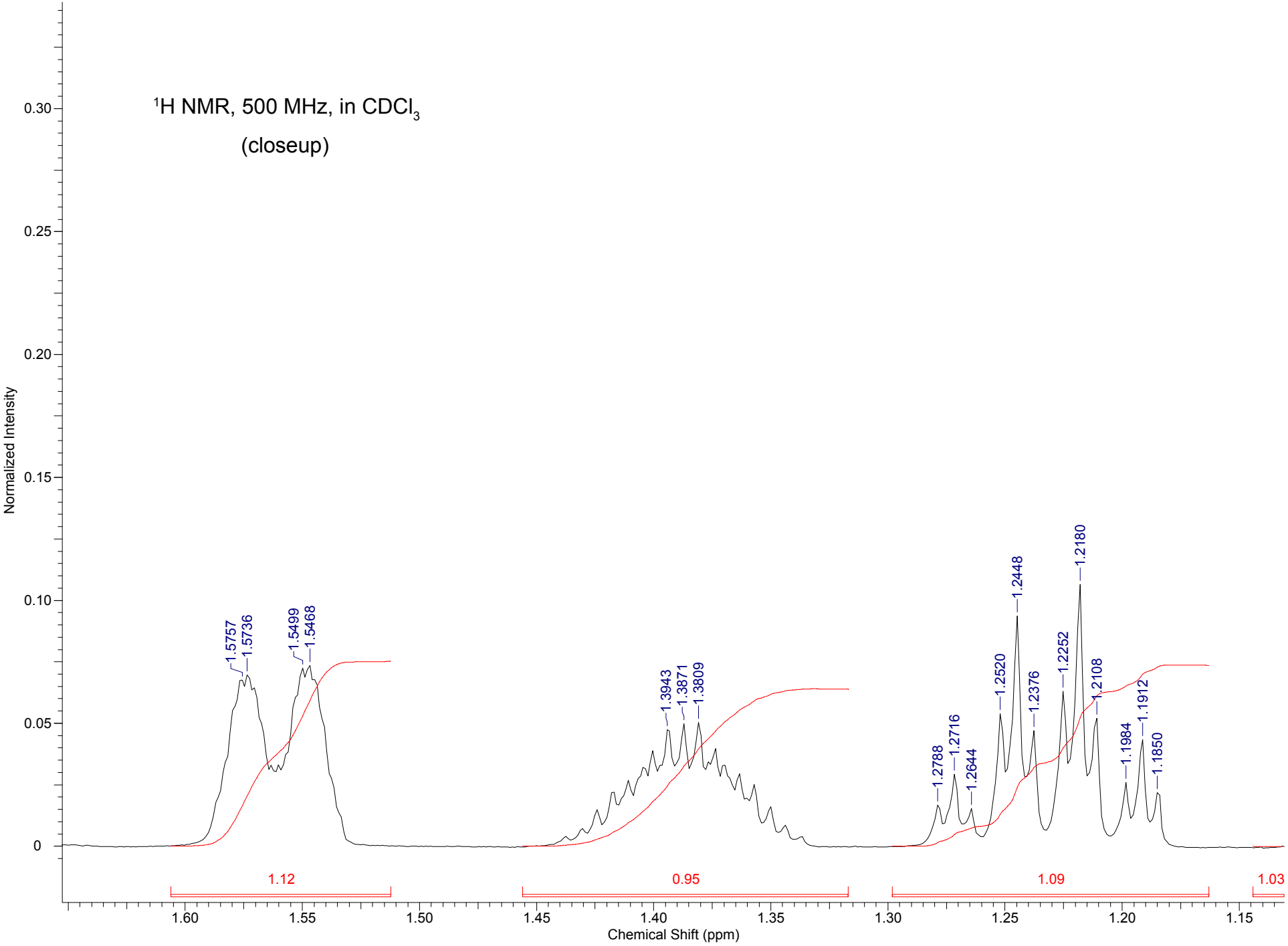
   Notes: It looks as though one ^{1}H-^{13}C correlation is missing from the HMQC; I think you can deduce which one is missing, and what H and C it belongs to.

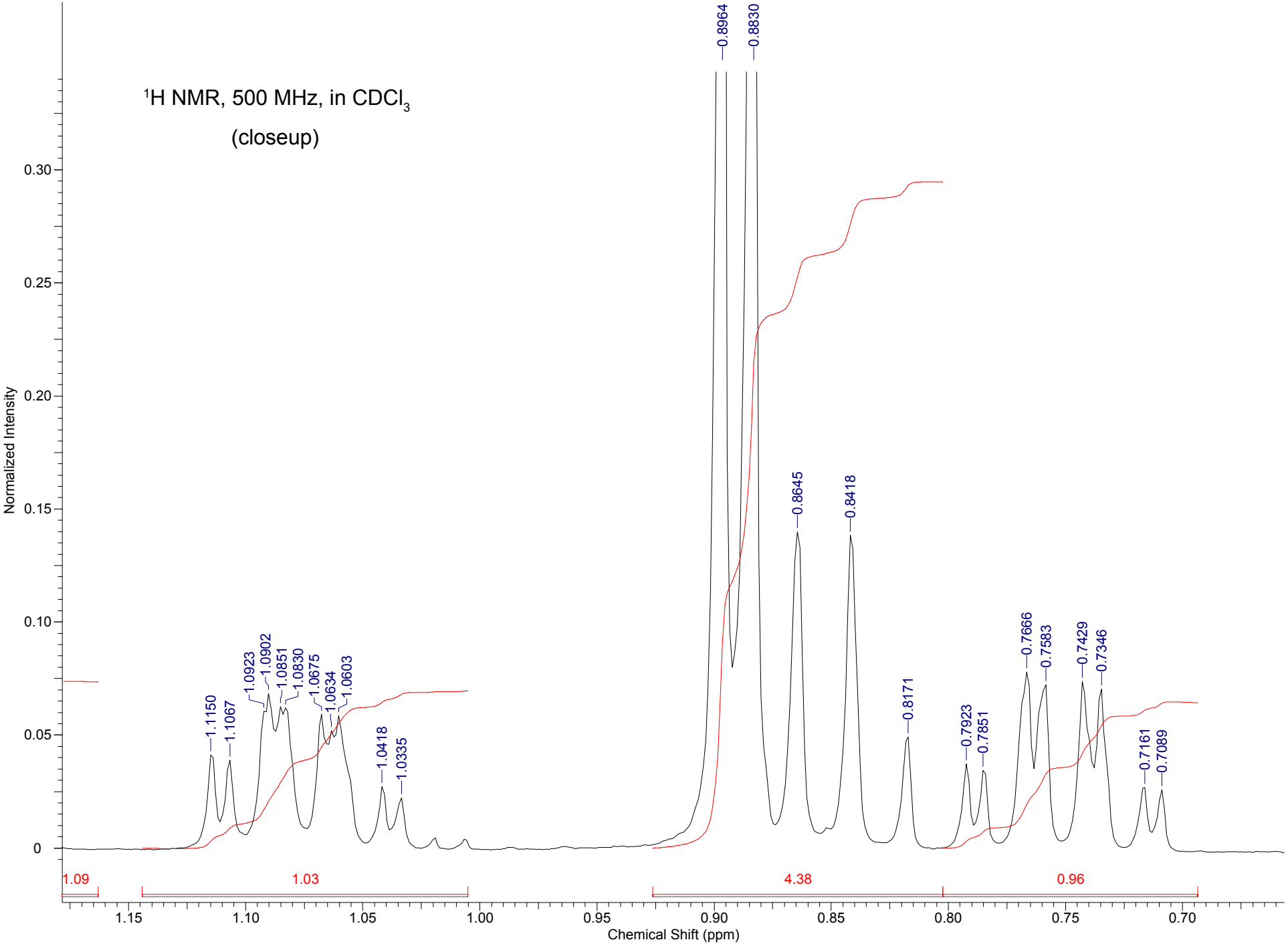
   The large cross-shaped peak in the center of the HMQC spectrum is an experimental artifact. Please ignore it.

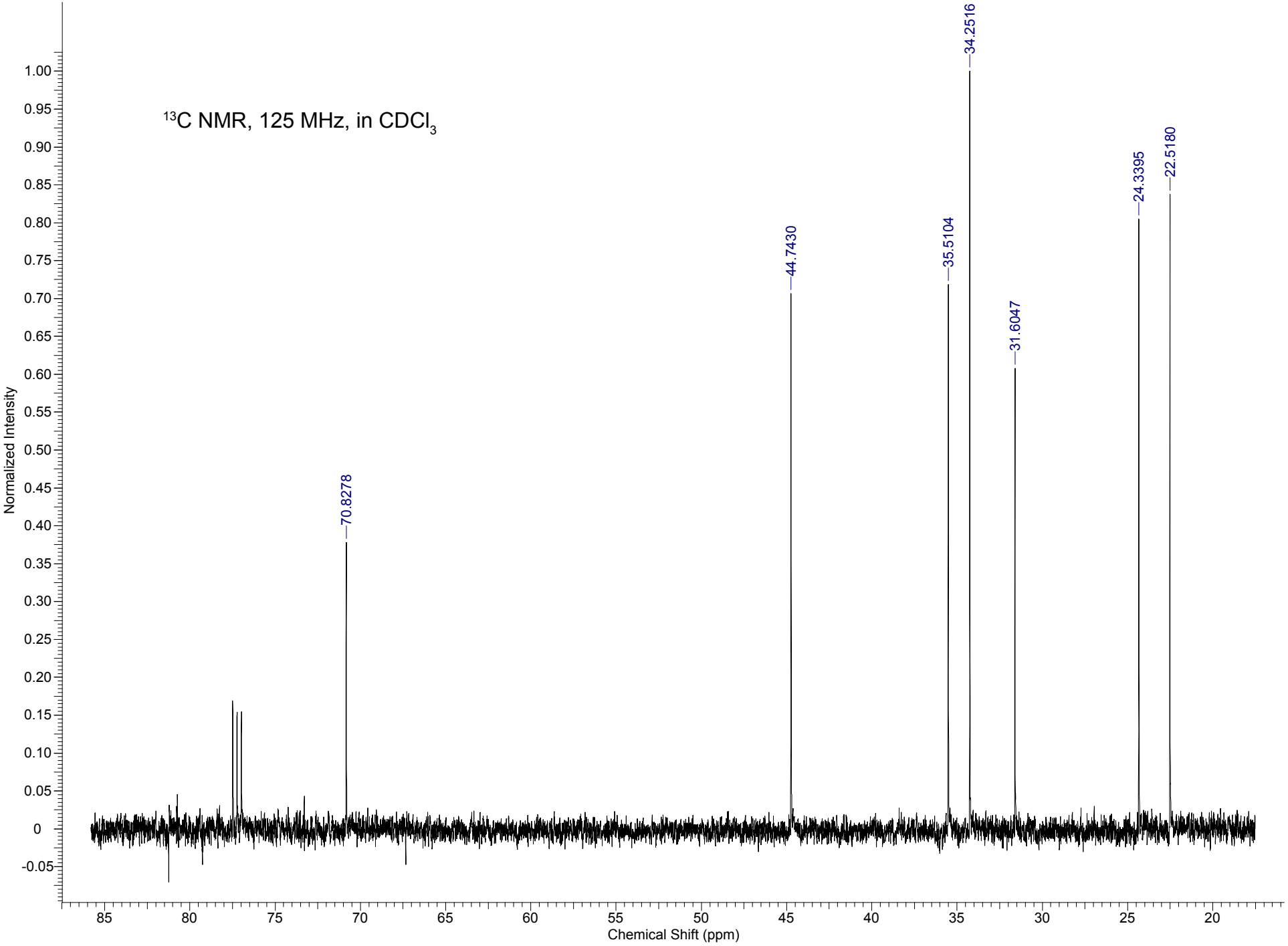
b. Is the molecule *cis*- or *trans*-disubstituted? Can you tell?


^{1}H NMR, 500 MHz, in $CDCl_3$
Normalized Intensity
0.95
0.90
0.85
0.80
0.75
0.70
0.65
0.60
0.55
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0
0.88
1.03
2.22
1.12
1.11
0.94
1.08
1.02
4.41
1.02
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
Chemical Shift (ppm)

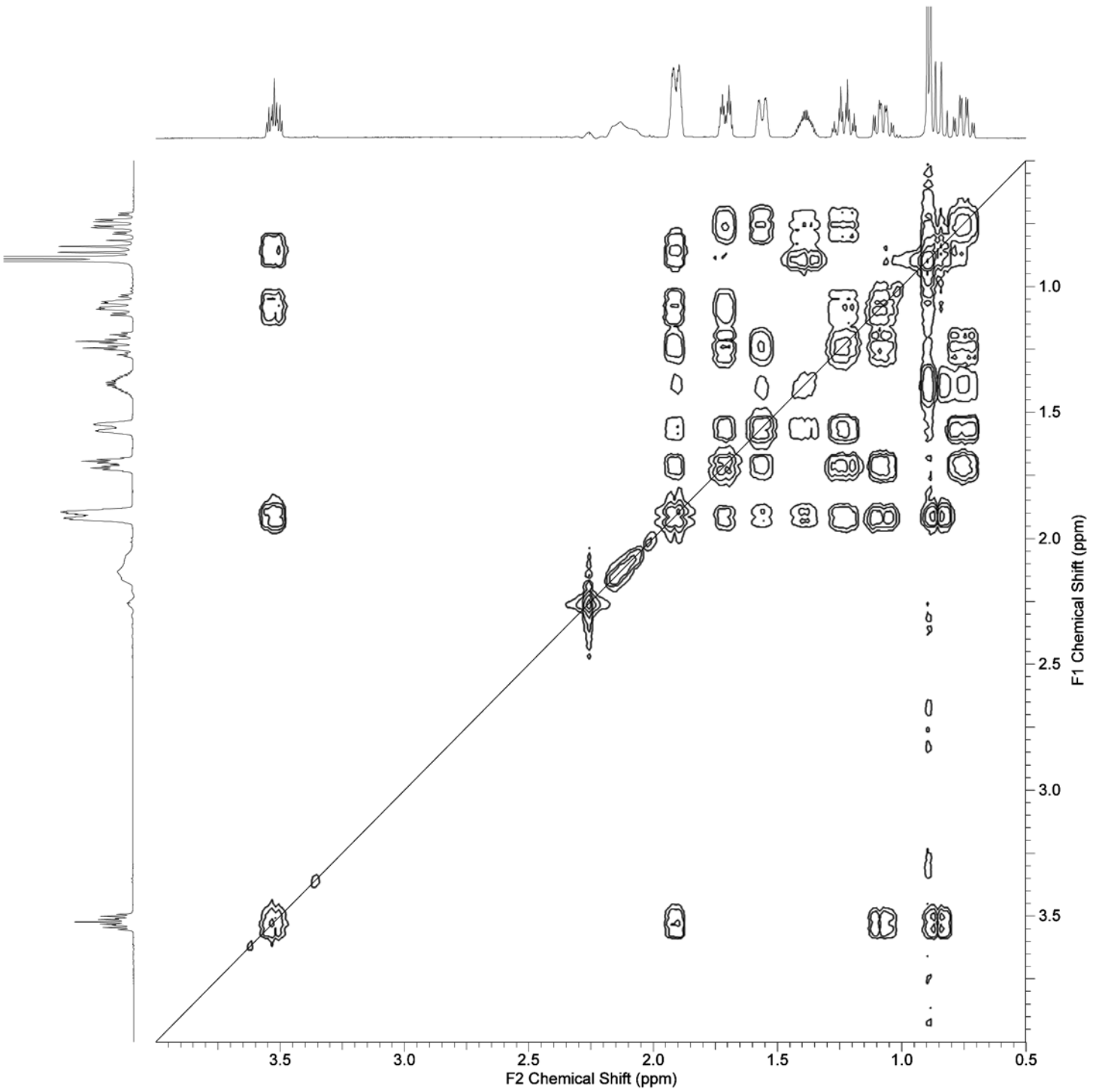

^{1}H NMR, 500 MHz, in $CDCl_3$
(closeup)
Normalized Intensity
0.30
0.25
0.20
0.15
0.10
0.05
0
3.5535
3.5453
3.5360
3.5319
3.5237
3.5144
3.5103
3.5010
3.4927
0.88
3.55
3.50
3.45
1.9199
1.8962
2.23
2.00
1.95
1.90
1.85
1.80
1.7344
1.7282
1.7220
1.7148
1.7076
1.7014
1.6942
1.6880
1.6808
1.13
1.75
1.70
1.65
Chemical Shift (ppm)


^{1}H NMR, 500 MHz, in $CDCl_3$
(closeup)
Normalized Intensity
0.30
0.25
0.20
0.15
0.10
0.05
0
1.5757
1.5736
1.5499
1.5468
1.3943
1.3871
1.3809
1.2788
1.2716
1.2644
1.2520
1.2448
1.2376
1.2252
1.2180
1.2108
1.1984
1.1912
1.1850
1.12
0.95
1.09
1.03
1.60
1.55
1.50
1.45
1.40
1.35
1.30
1.25
1.20
1.15
Chemical Shift (ppm)


^{1}H NMR, 500 MHz, in $CDCl_3$
(closeup)
Normalized Intensity
Chemical Shift (ppm)
0.8964
0.8830
0.8645
0.8418
0.8171
1.1150
1.1067
1.0923
1.0902
1.0851
1.0830
1.0675
1.0634
1.0603
1.0418
1.0335
0.7923
0.7851
0.7666
0.7583
0.7429
0.7346
0.7161
0.7089
1.09
1.03
4.38
0.96


^{13}C NMR, 125 MHz, in $CDCl_3$
Normalized Intensity
Chemical Shift (ppm)
70.8278
44.7430
35.5104
34.2516
31.6047
24.3395
22.5180

^{1}H-^{1}H COSY, 500 MHz, in $CDCl_3$



^{1}H-^{13}C HMQC, 500 MHz, in $CDCl_3$