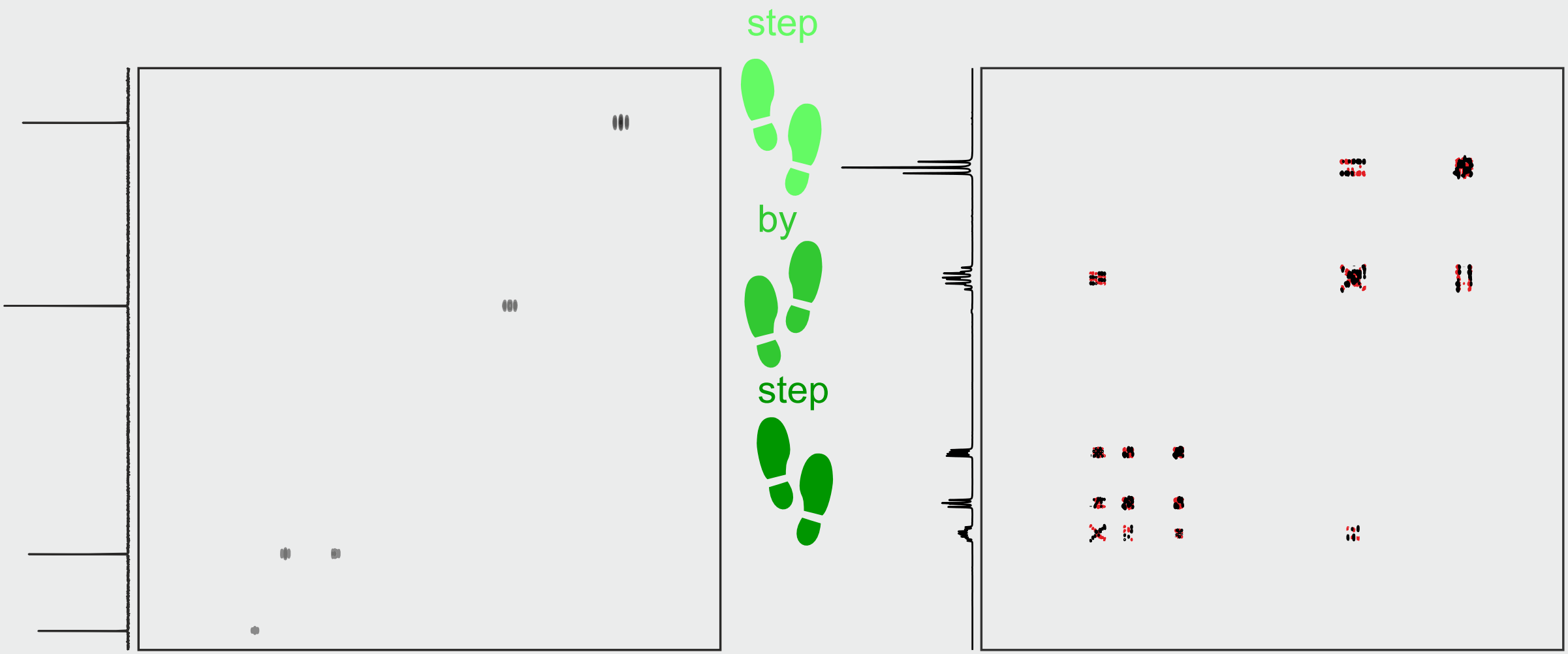
Exercise plus Solution – Quick overview

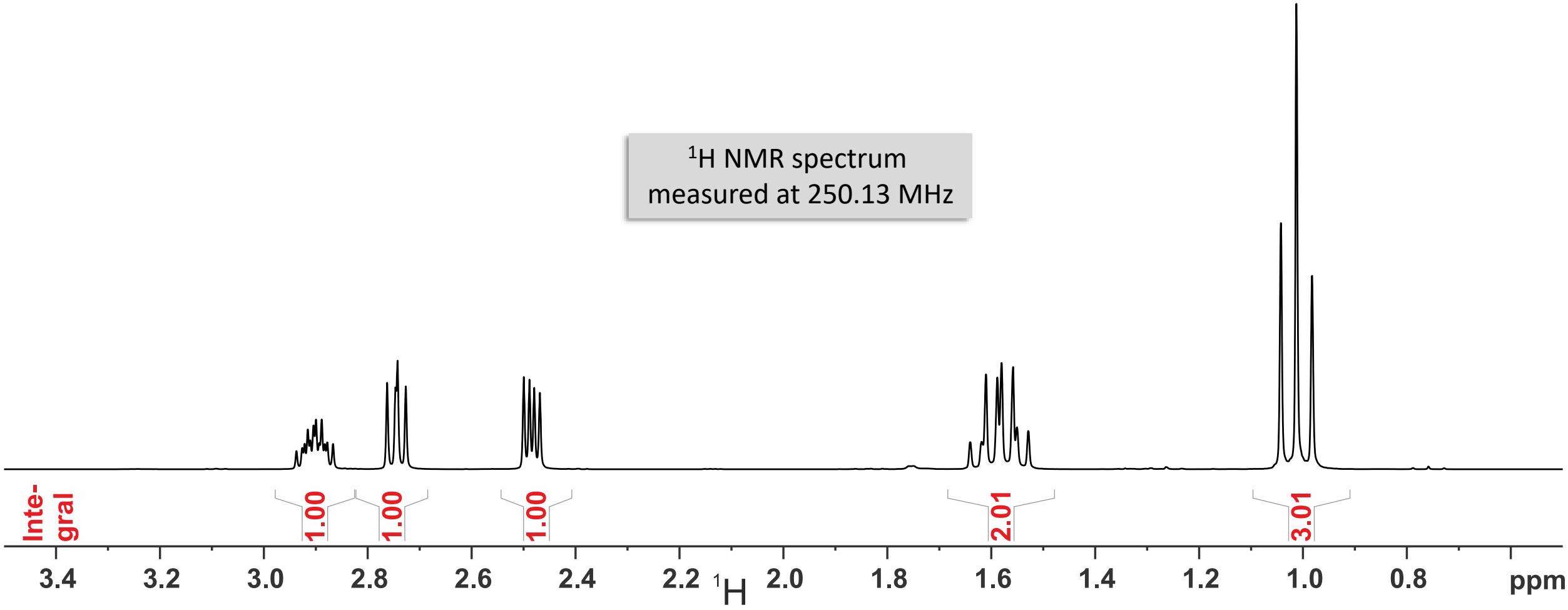
It is recommended to use this version only for a quick overview of the NMR challenge. All animations of the PowerPoint version are missing, under certain circumstances quality deficiencies may also occur.
The higher quality PowerPoint files are freely available for download at any time.



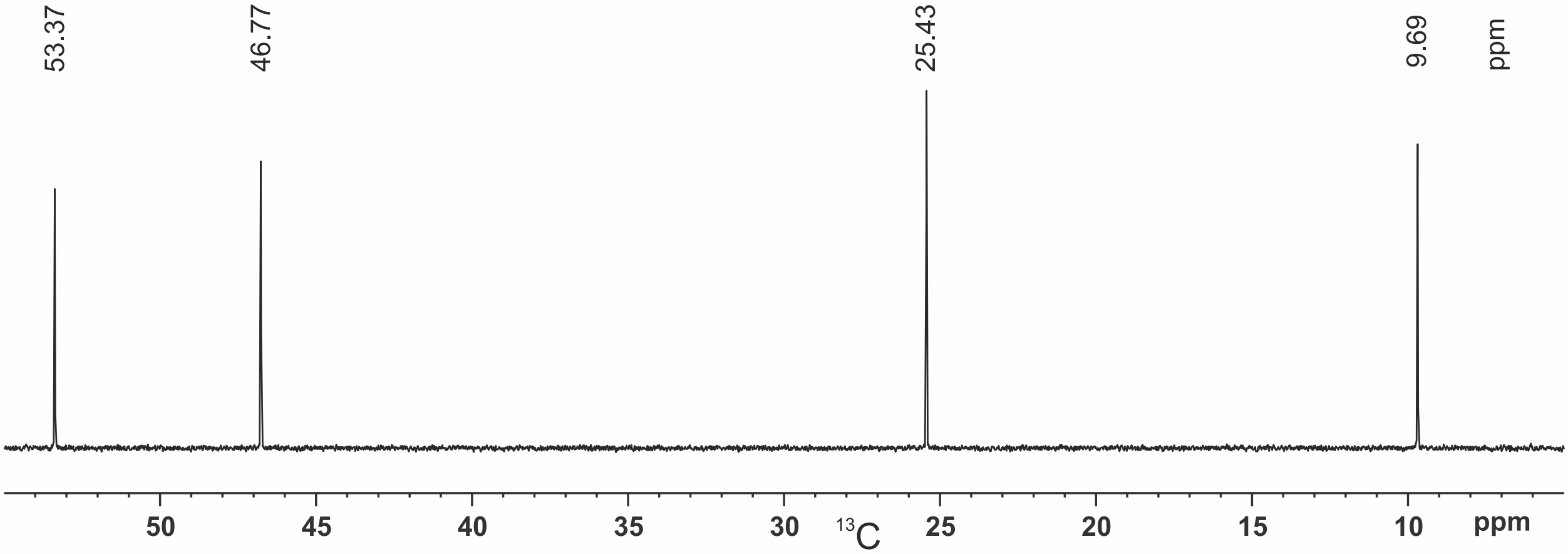
C_4H_8O measured in $CDCl_3$

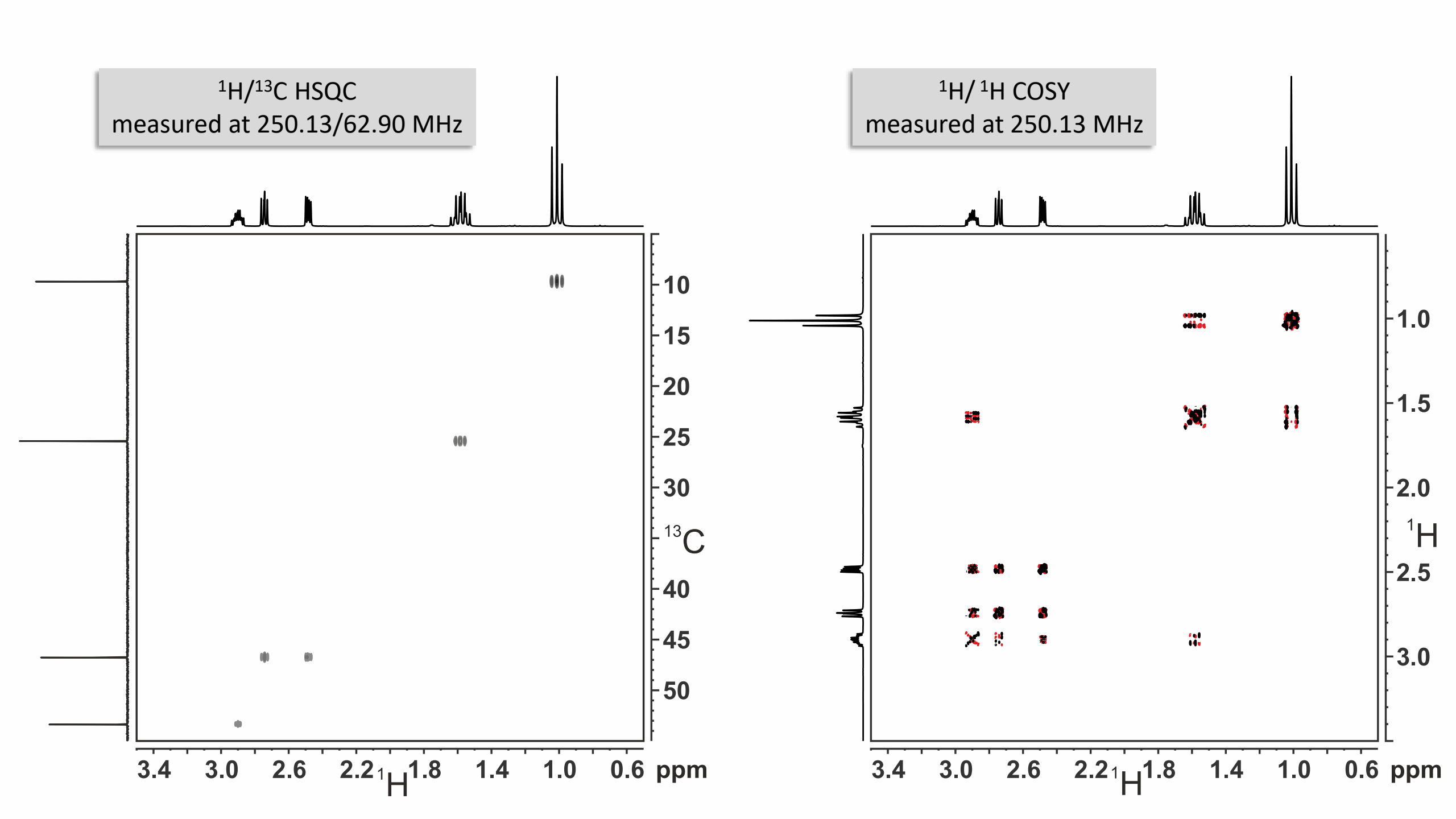
Deduce the structure without worrying care about the coupling patterns in the proton spectrum!

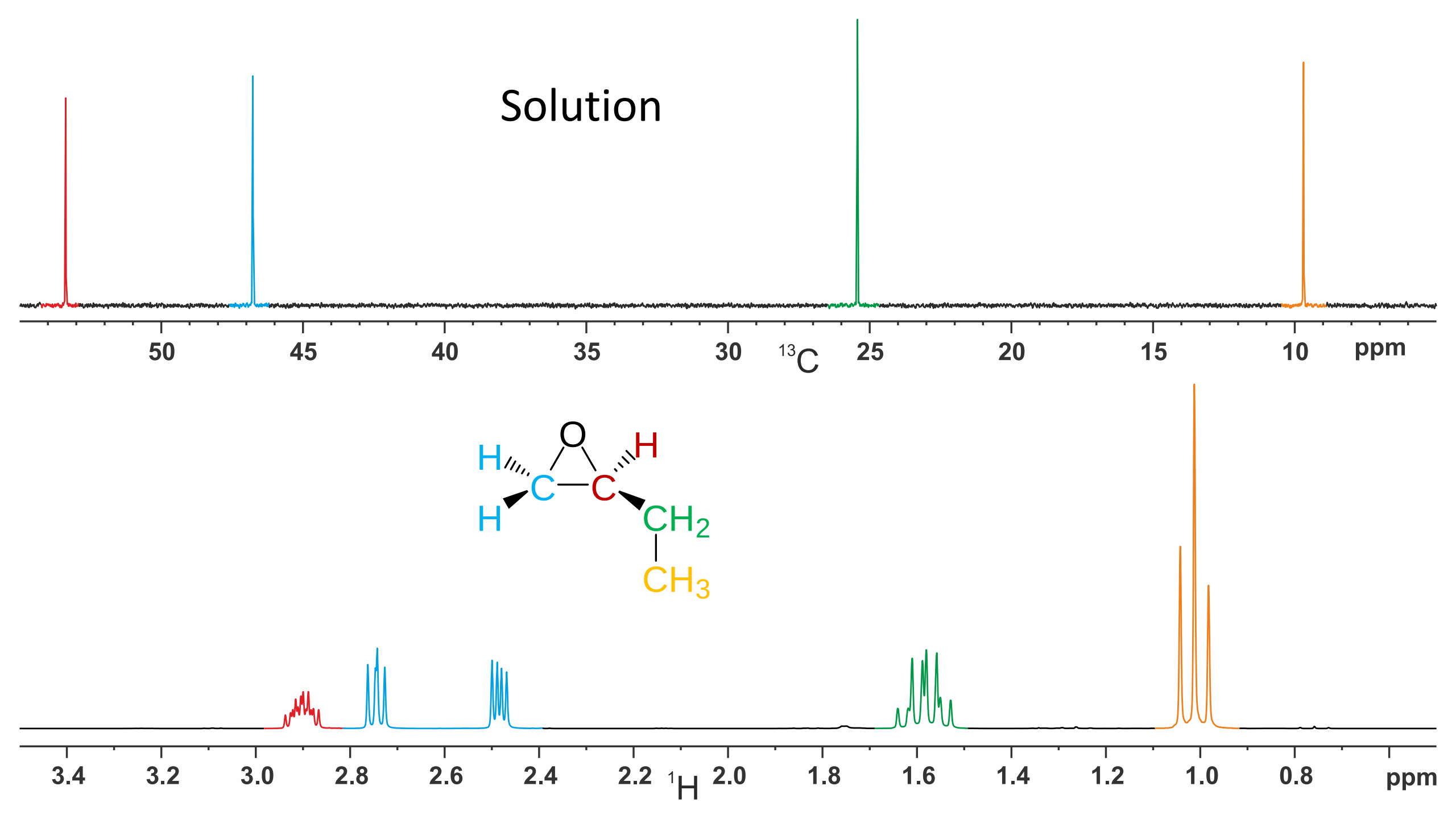
^{1}H NMR spectrum measured at 250.13 MHz



$^{13}C\{^{1}H\}$ NMR spectrum
measured at 62.90{250.13} MHz



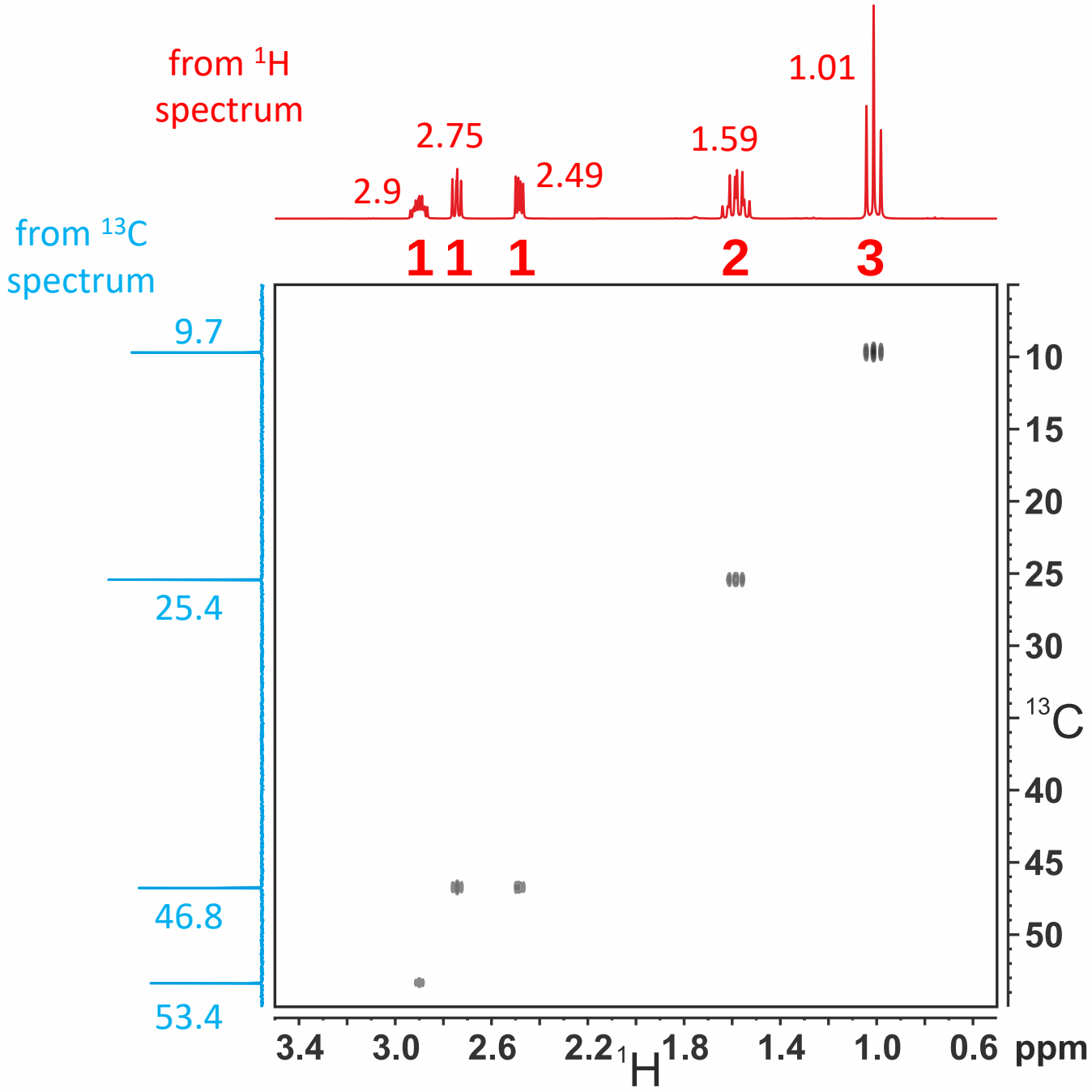

^{1}H/^{13}C HSQC
measured at 250.13/62.90 MHz
^{1}H/ ^{1}H COSY
measured at 250.13 MHz
10
15
20
25
30
^{13}C
40
45
50
3.4
3.0
2.6
2.2
^{1}H
1.8
1.4
1.0
0.6
ppm
1.0
1.5
2.0
^{1}H
2.5
3.0


Solution
50
45
40
35
30
^{13}C
25
20
15
10
ppm
O
H
H
C
C
H
H
CH_2
CH_3
3.4
3.2
3.0
2.8
2.6
2.4
2.2
^{1}H
2.0
1.8
1.6
1.4
1.2
1.0
0.8
ppm

Step-by-Step

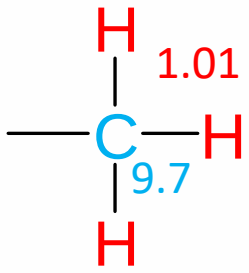
If available, an HSQC is nearly always the best starting point for a structure elucidation.

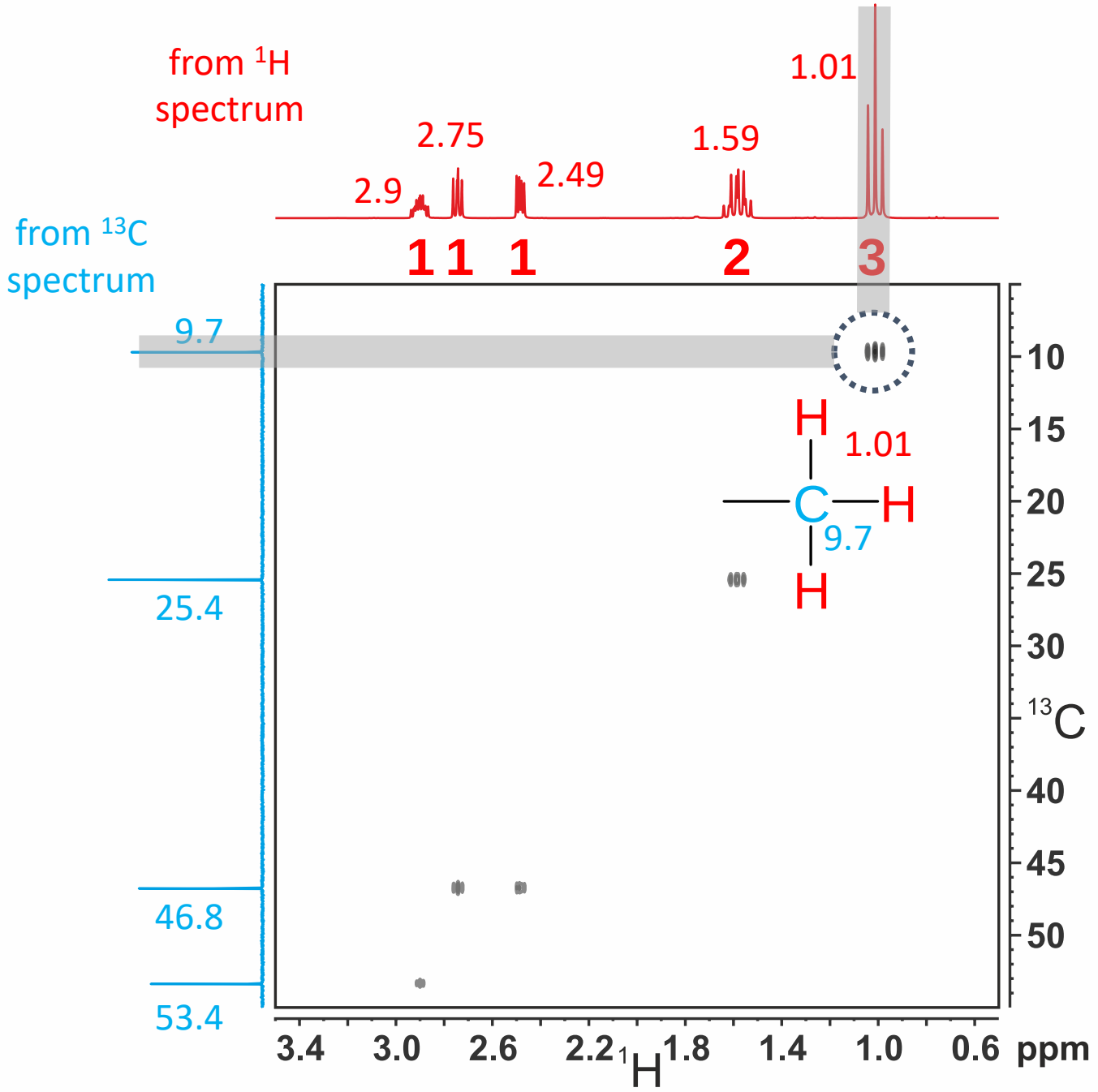
As a first step it is recommended to collect the integrals and chemical shifts from the one-dimensional spectra.



Now one can determine the CH_n fragments from the cross peaks and the integrals one after the other.

For the moment this results in a disordered collection of building blocks.





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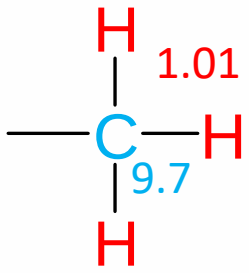

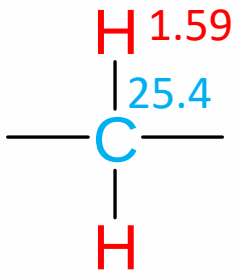

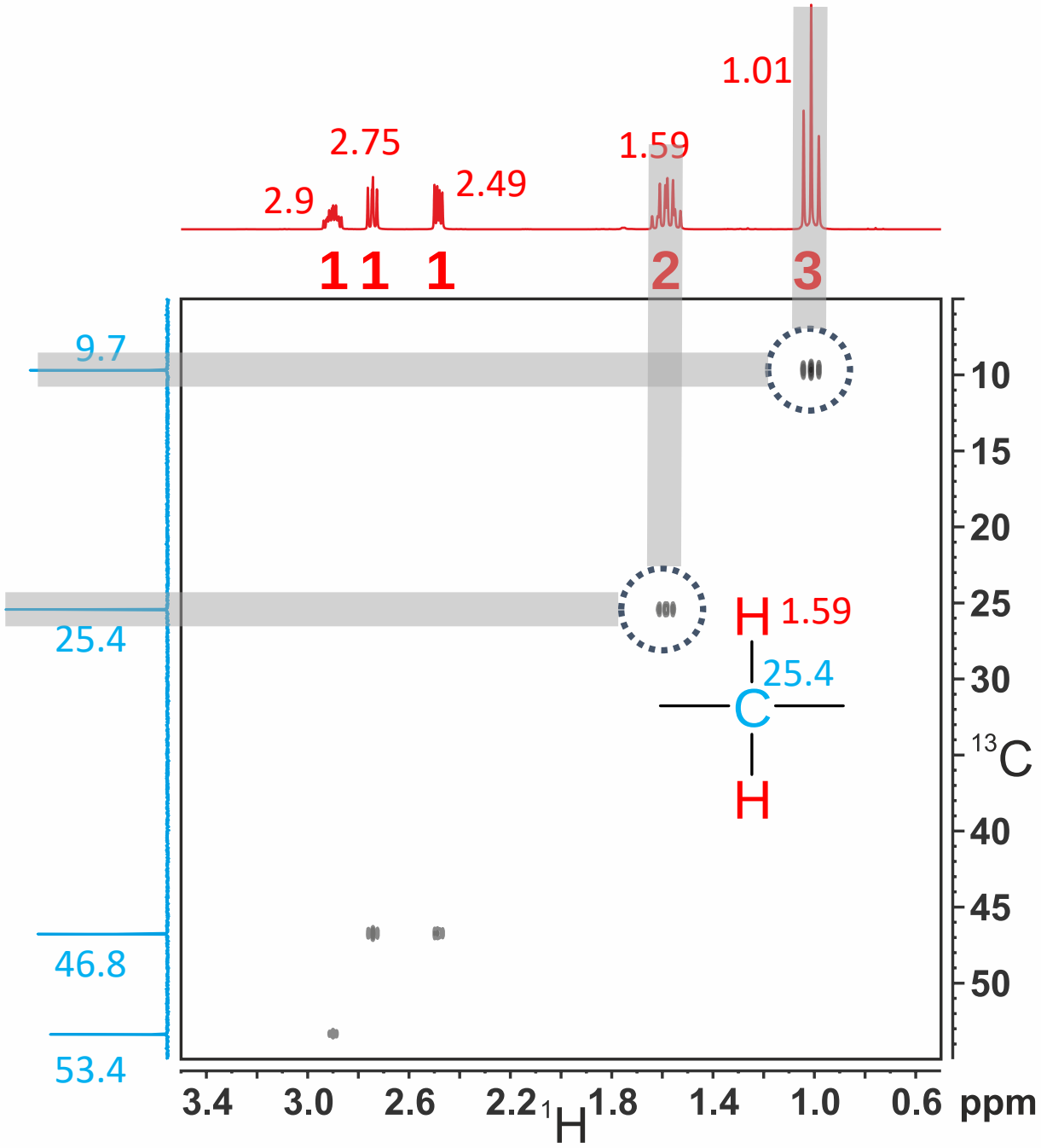

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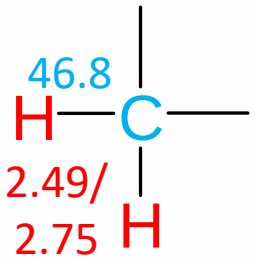

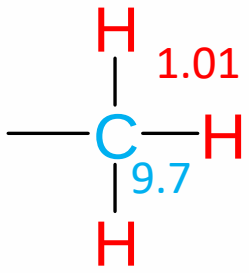

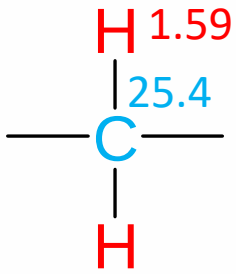

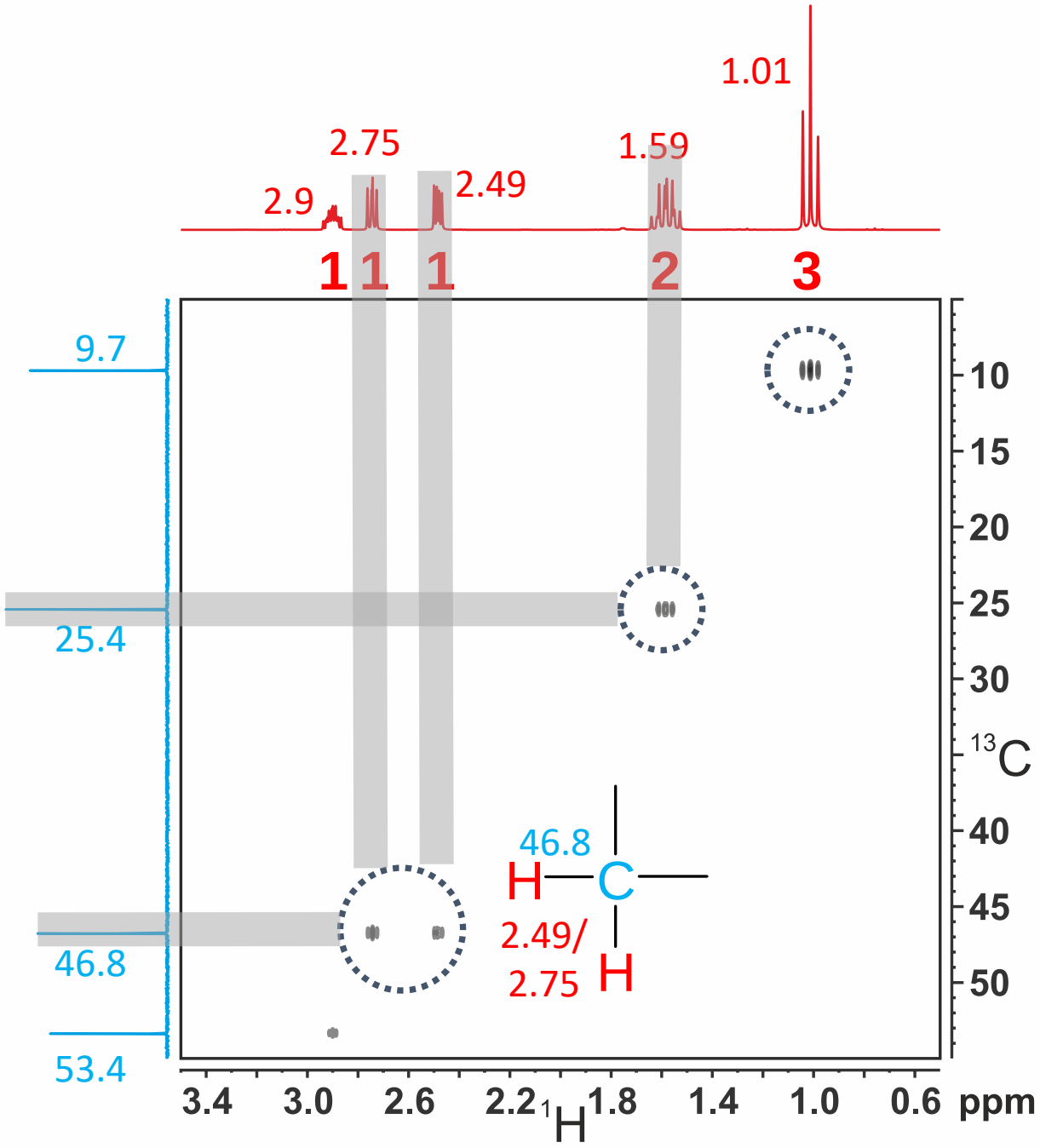

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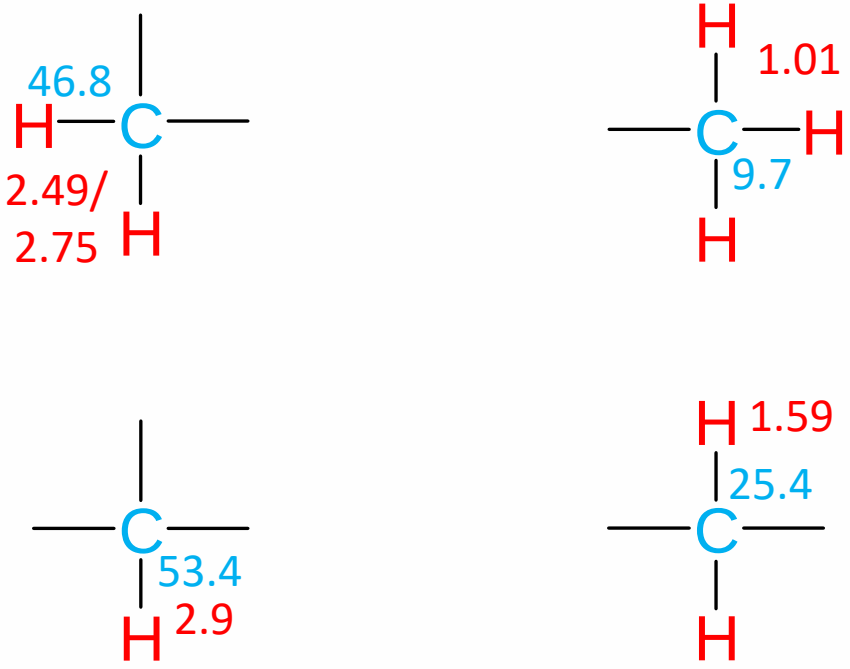
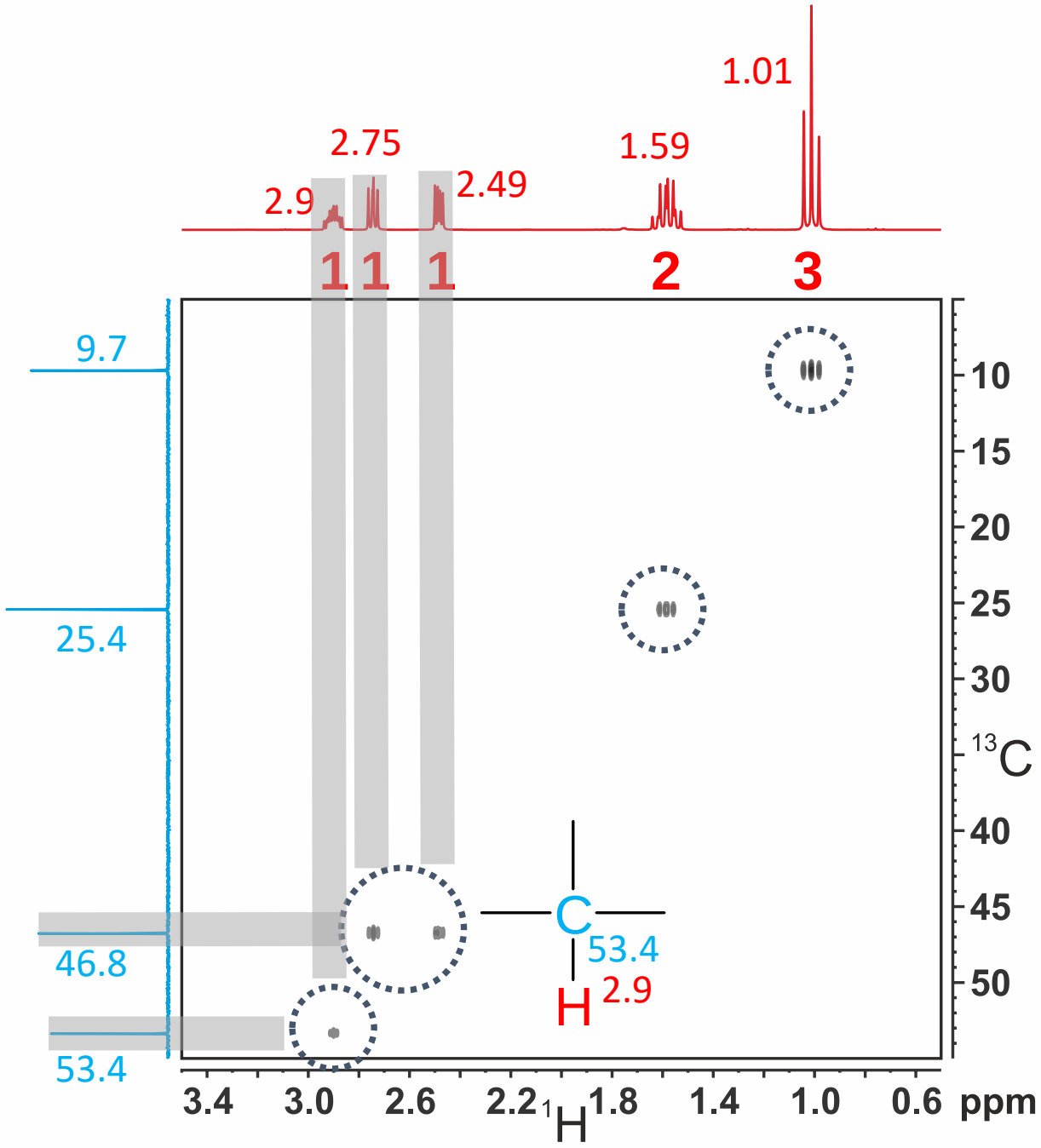
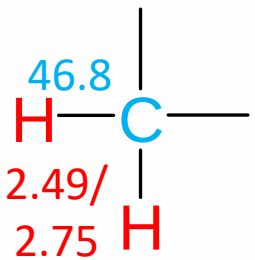
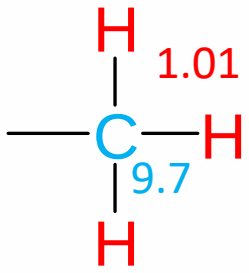


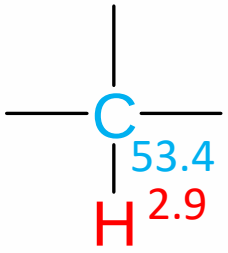


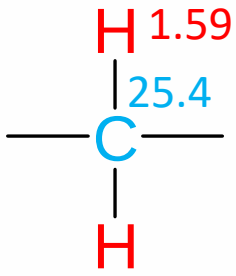
Let us switch to the COSY now and combine the building blocks using their proton chemical shifts.

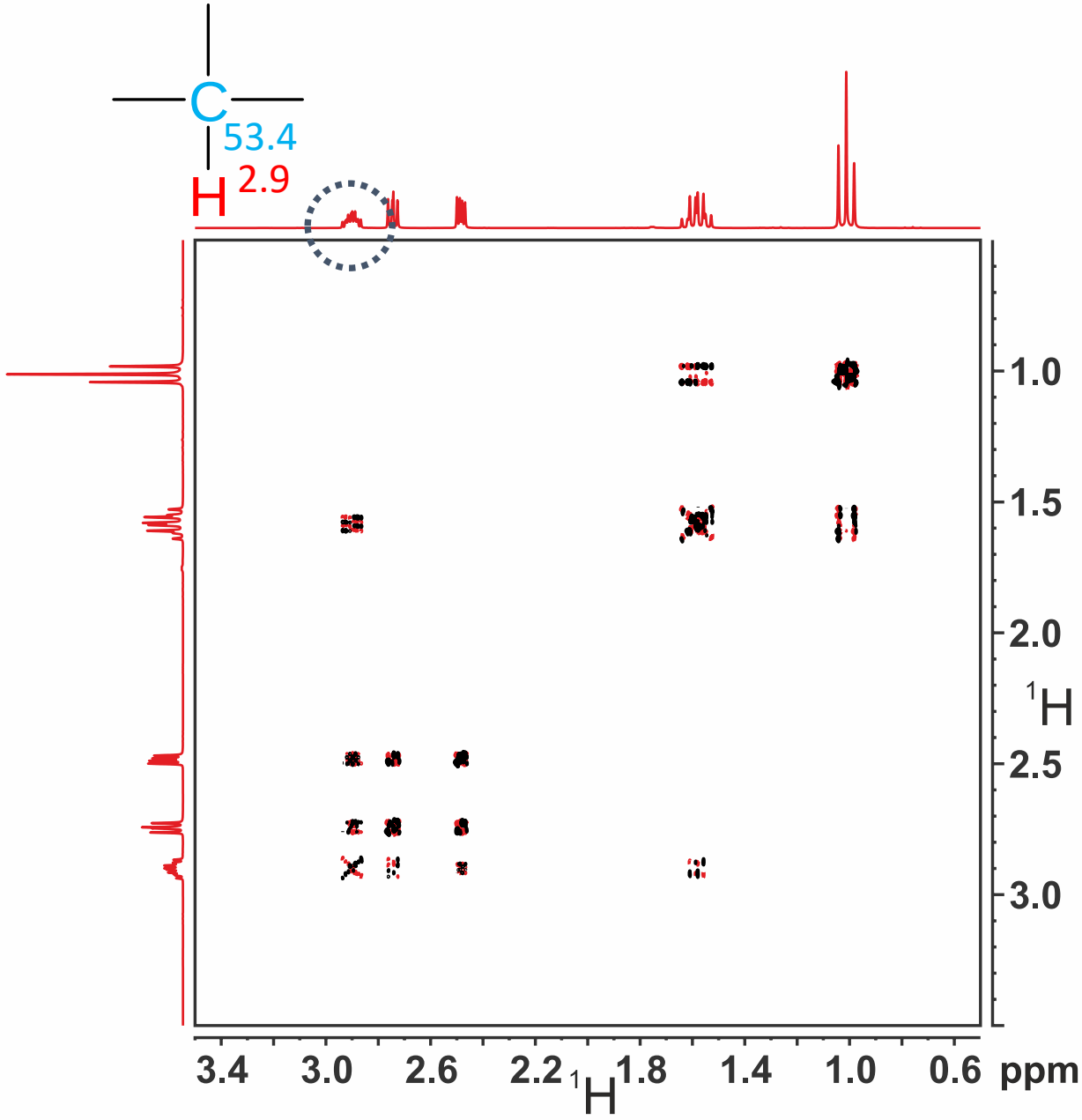
As a first step let us reorder the building blocks by their proton chemical shifts and put them close to the corresponding proton signals in the projection.











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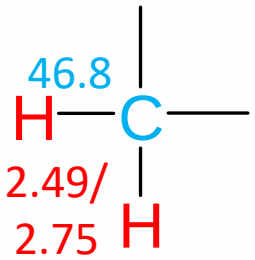

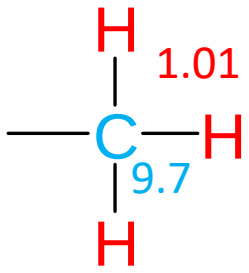

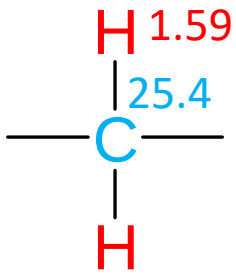

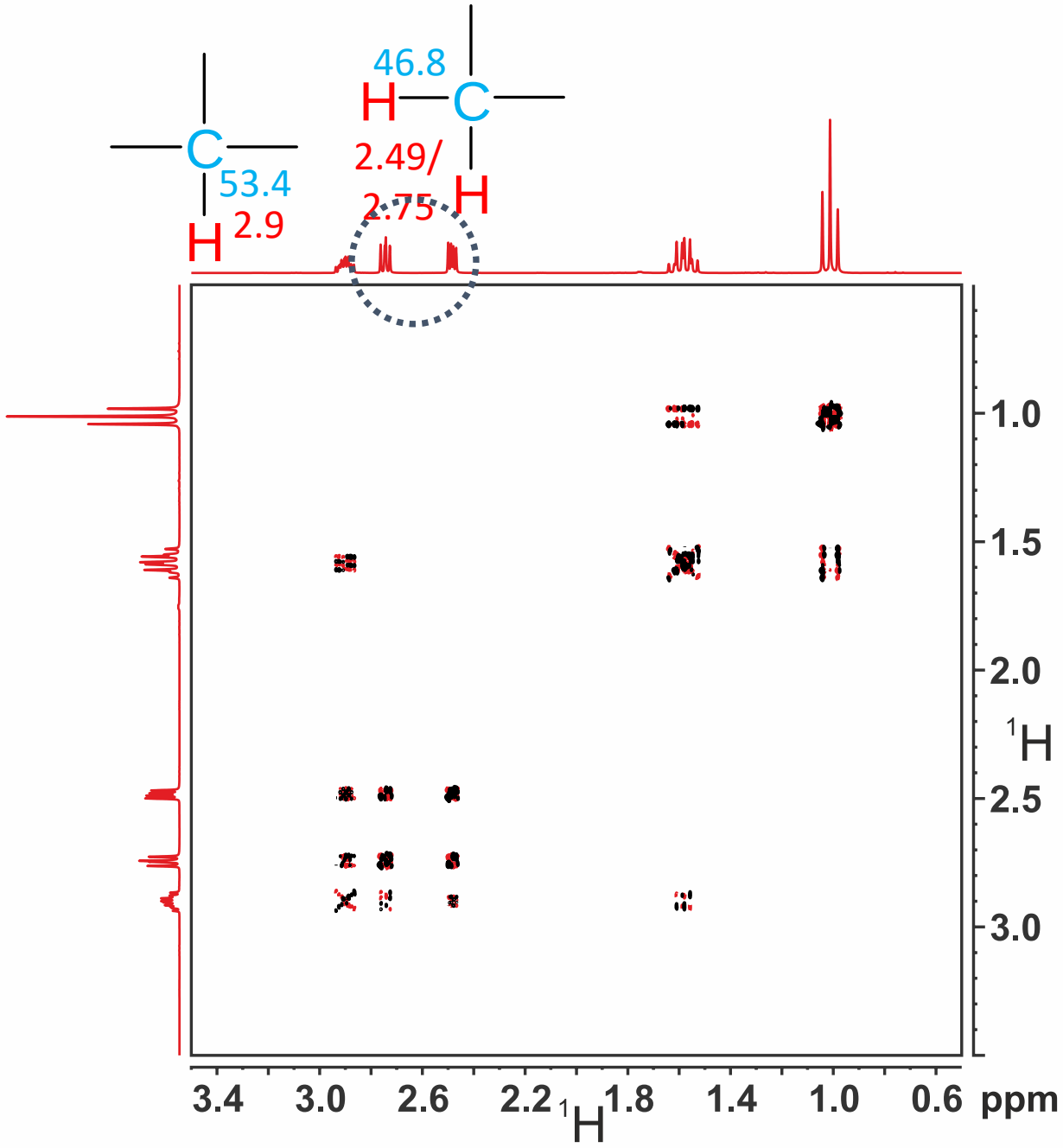

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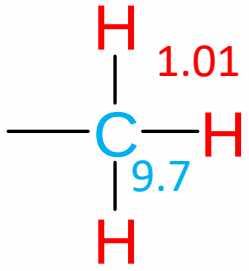

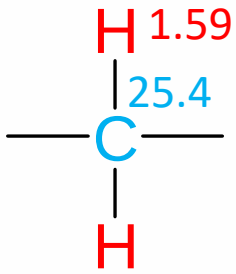

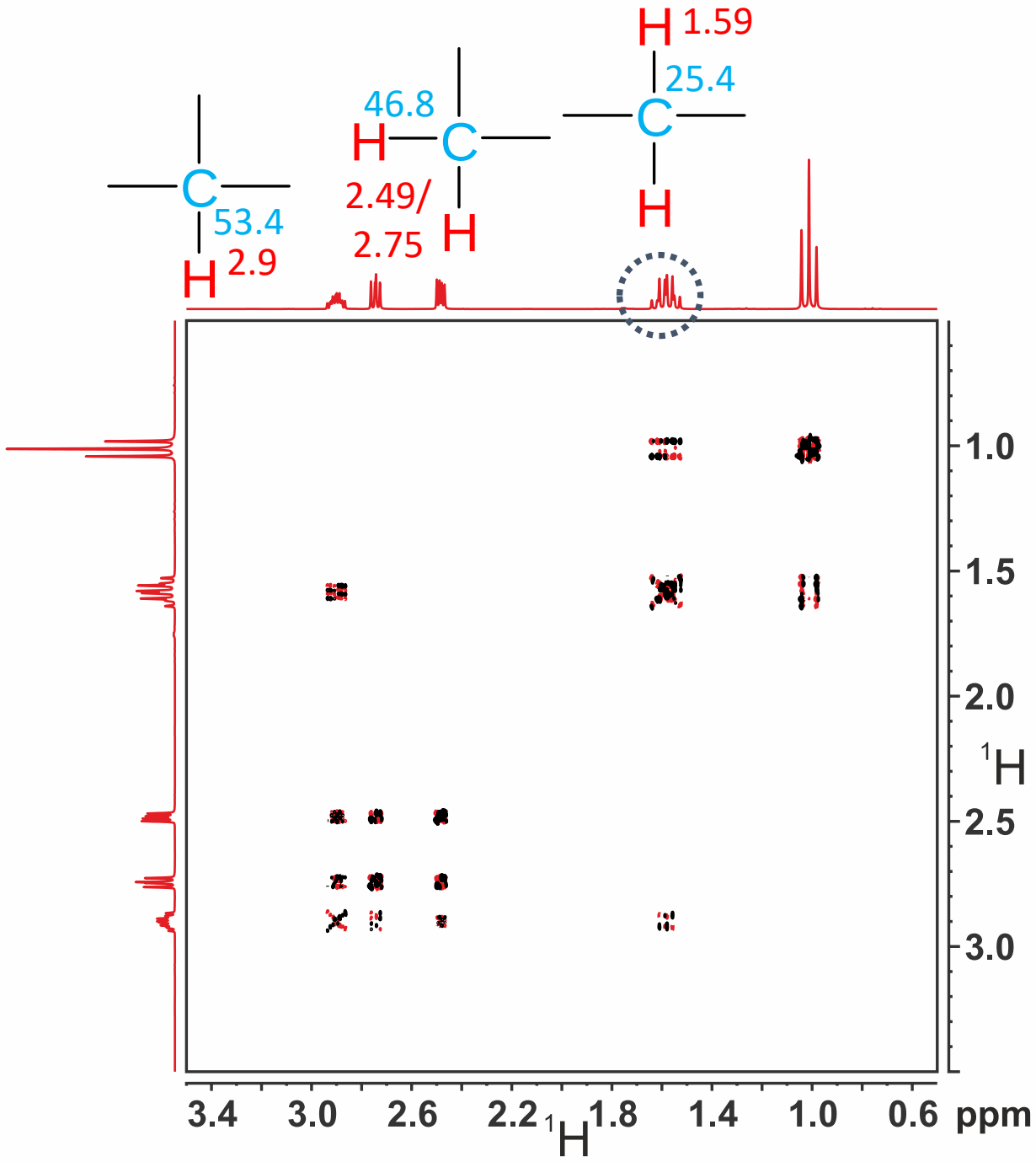

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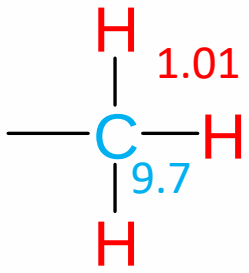

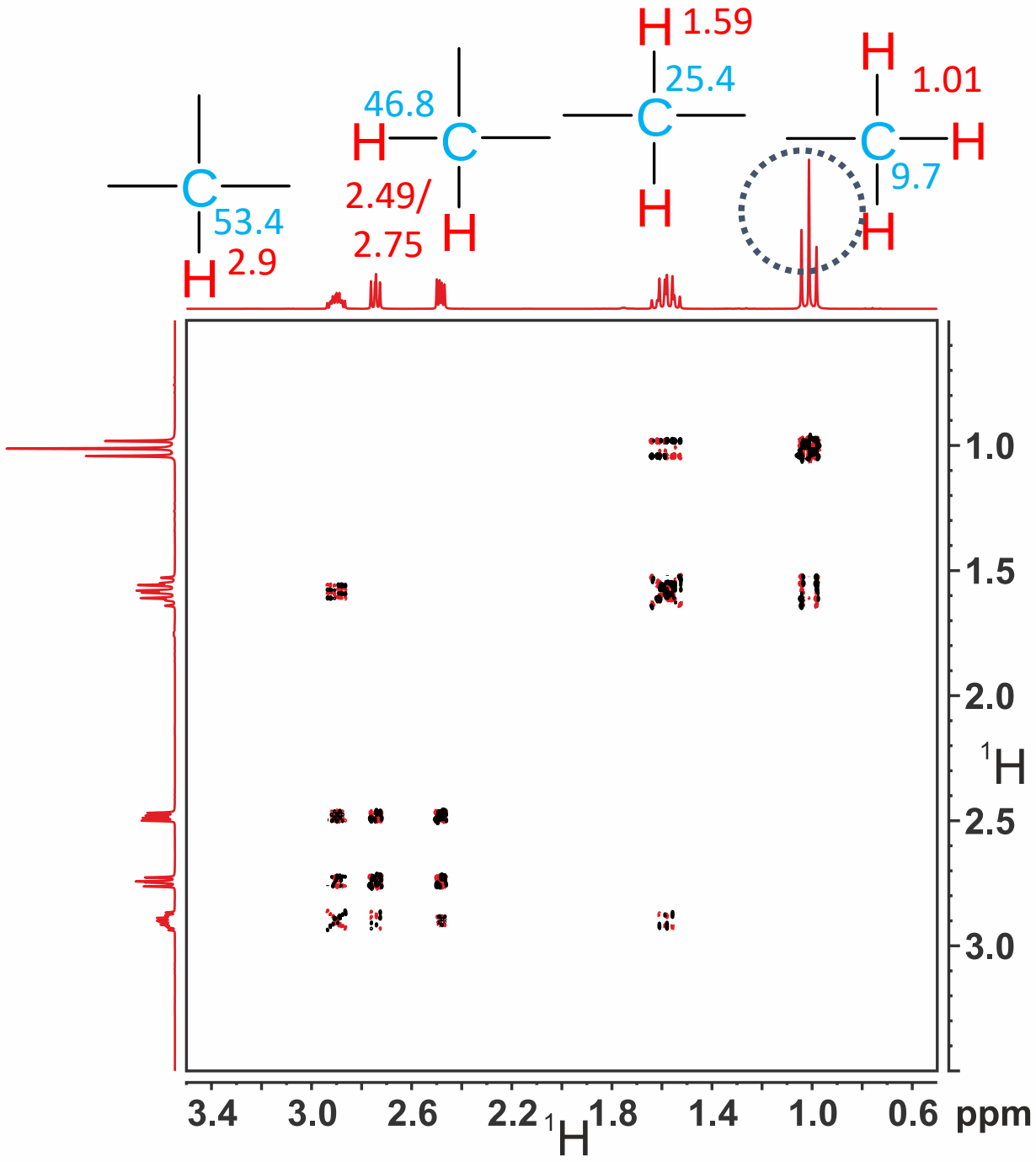

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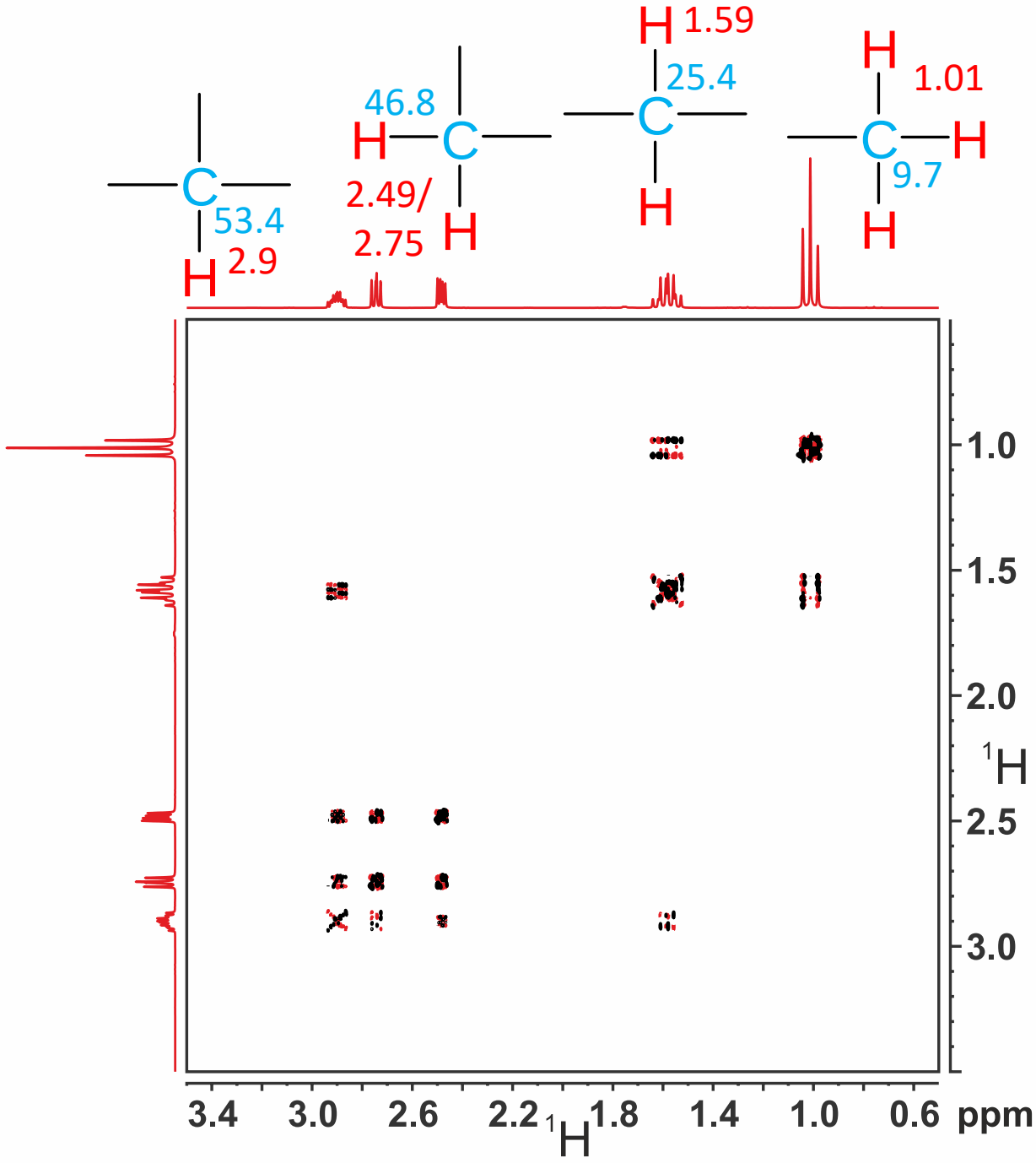
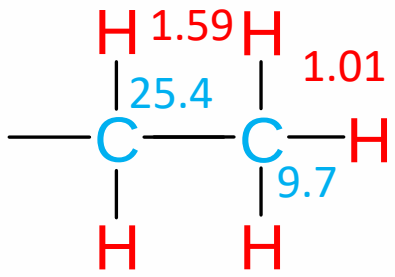
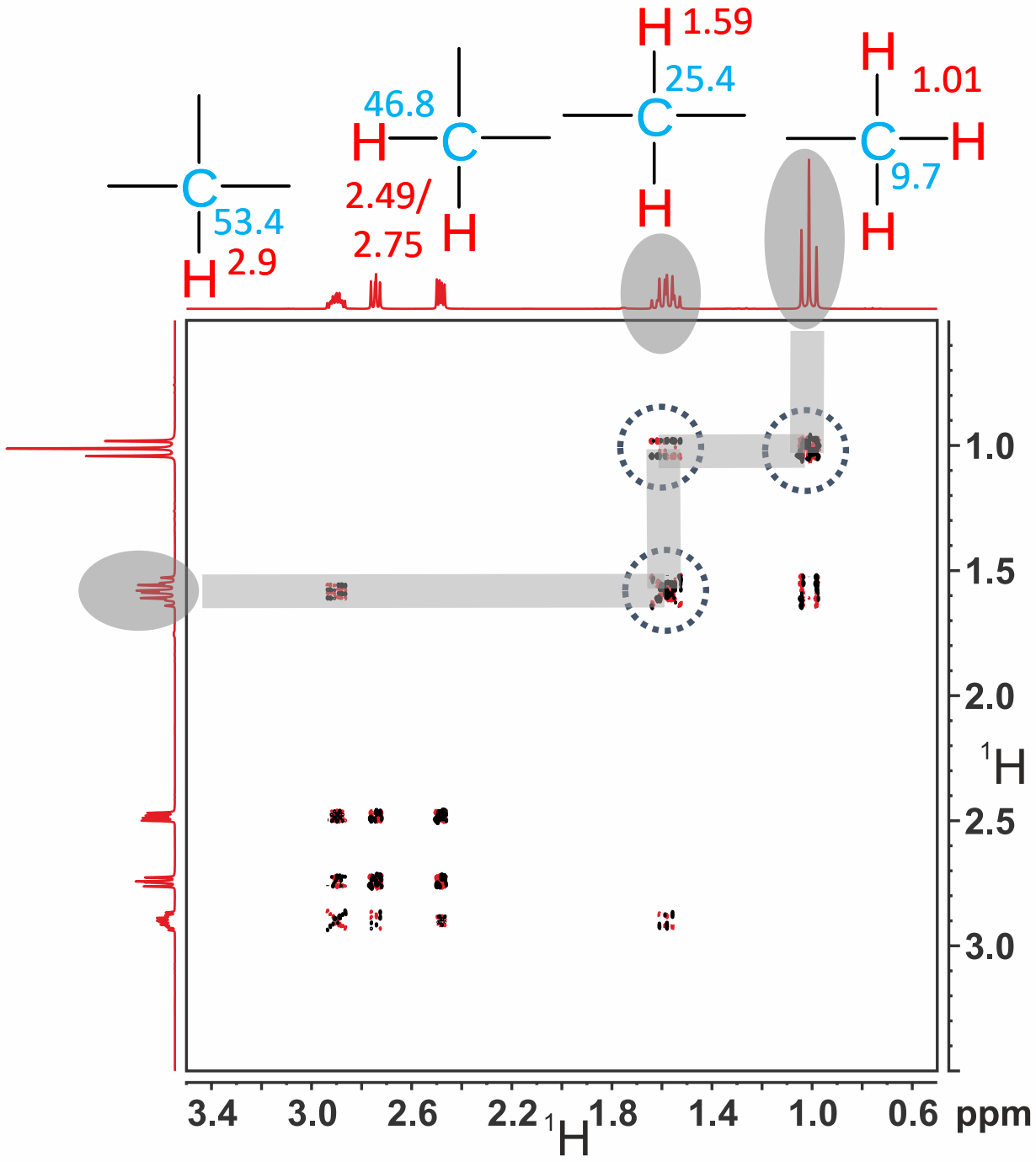


Let us switch to the COSY now and combine the building blocks using their proton chemical shifts.

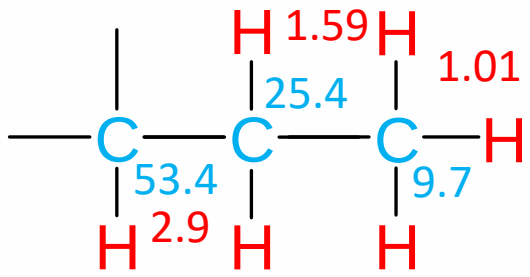
The methyl group clearly is one end of the molecule. We use the methyl group to start the sequential ordering of the building blocks.

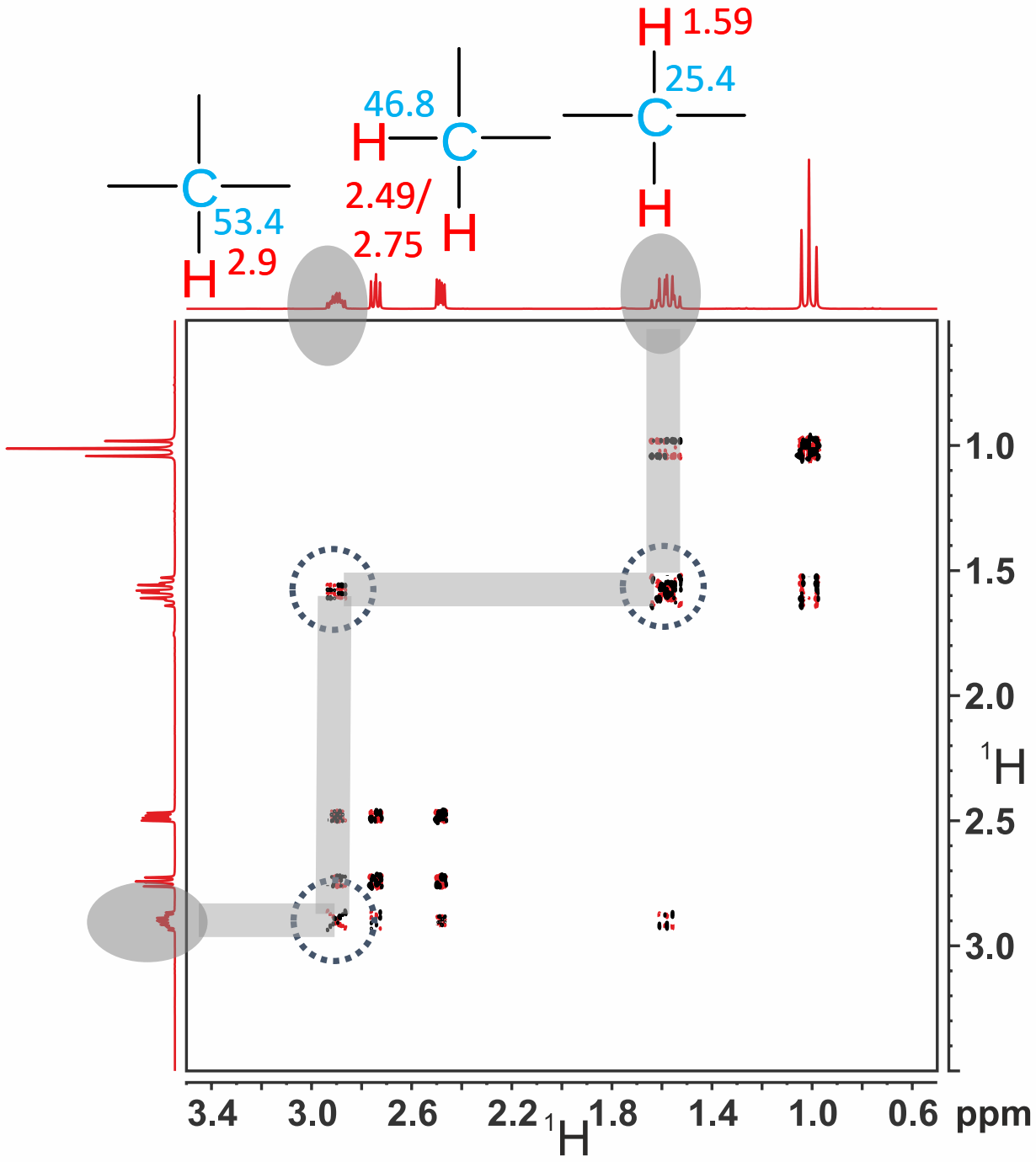




Let us switch to the COSY now and combine the building blocks using their proton chemical shifts.

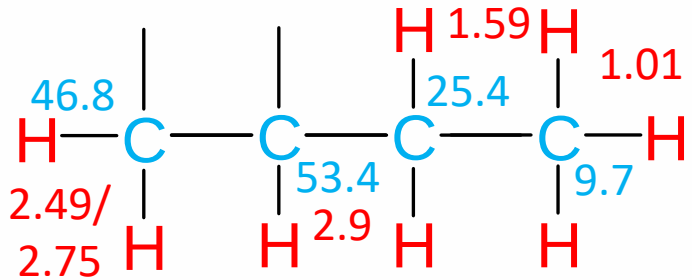
The next building block has to be attached to the ethyl group found so far.

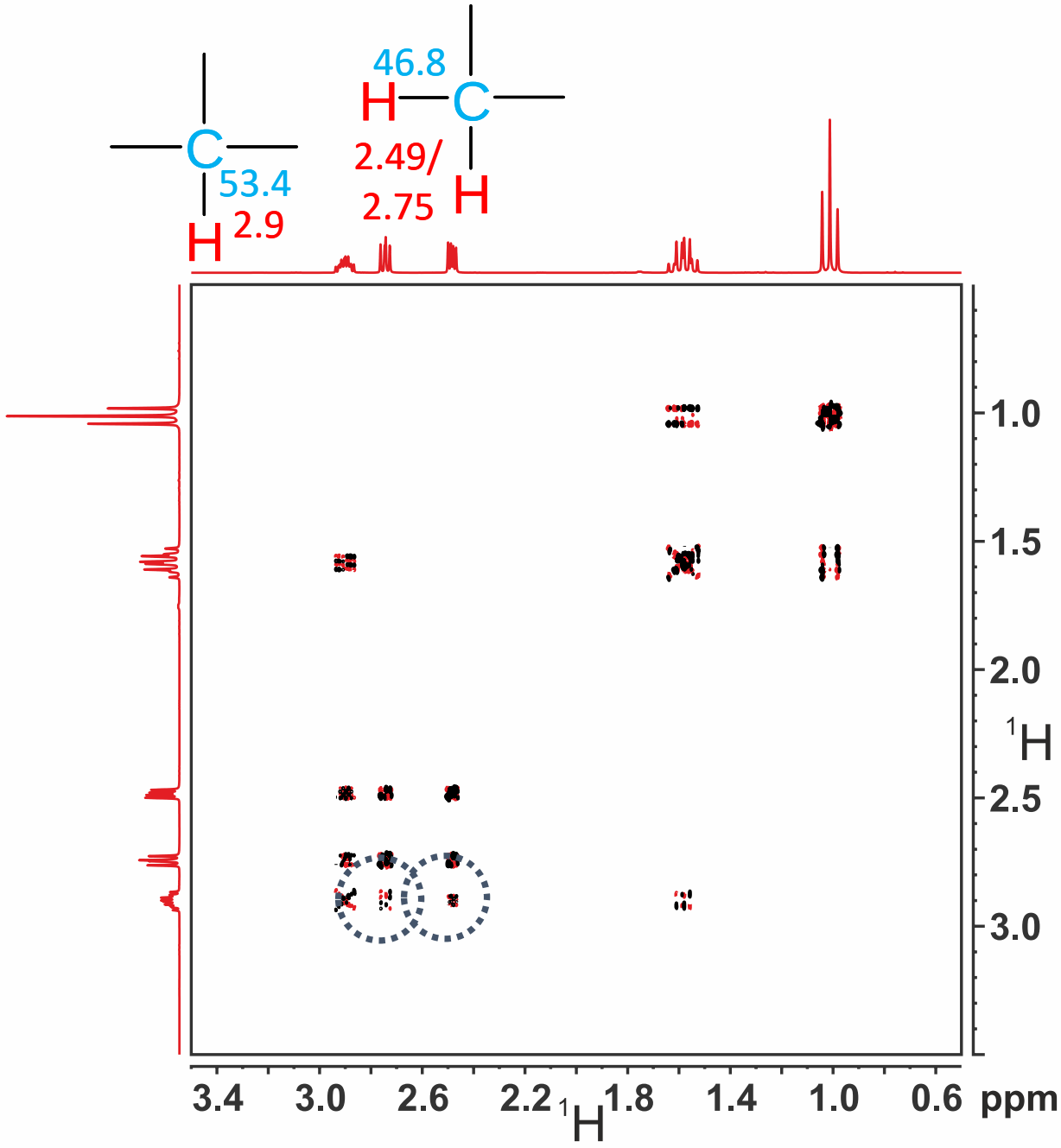




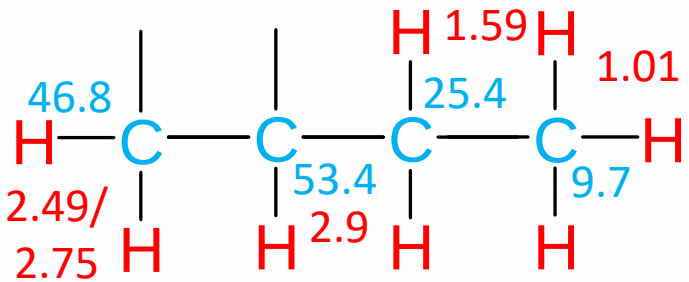
Let us switch to the COSY now and combine the building blocks using their proton chemical shifts.

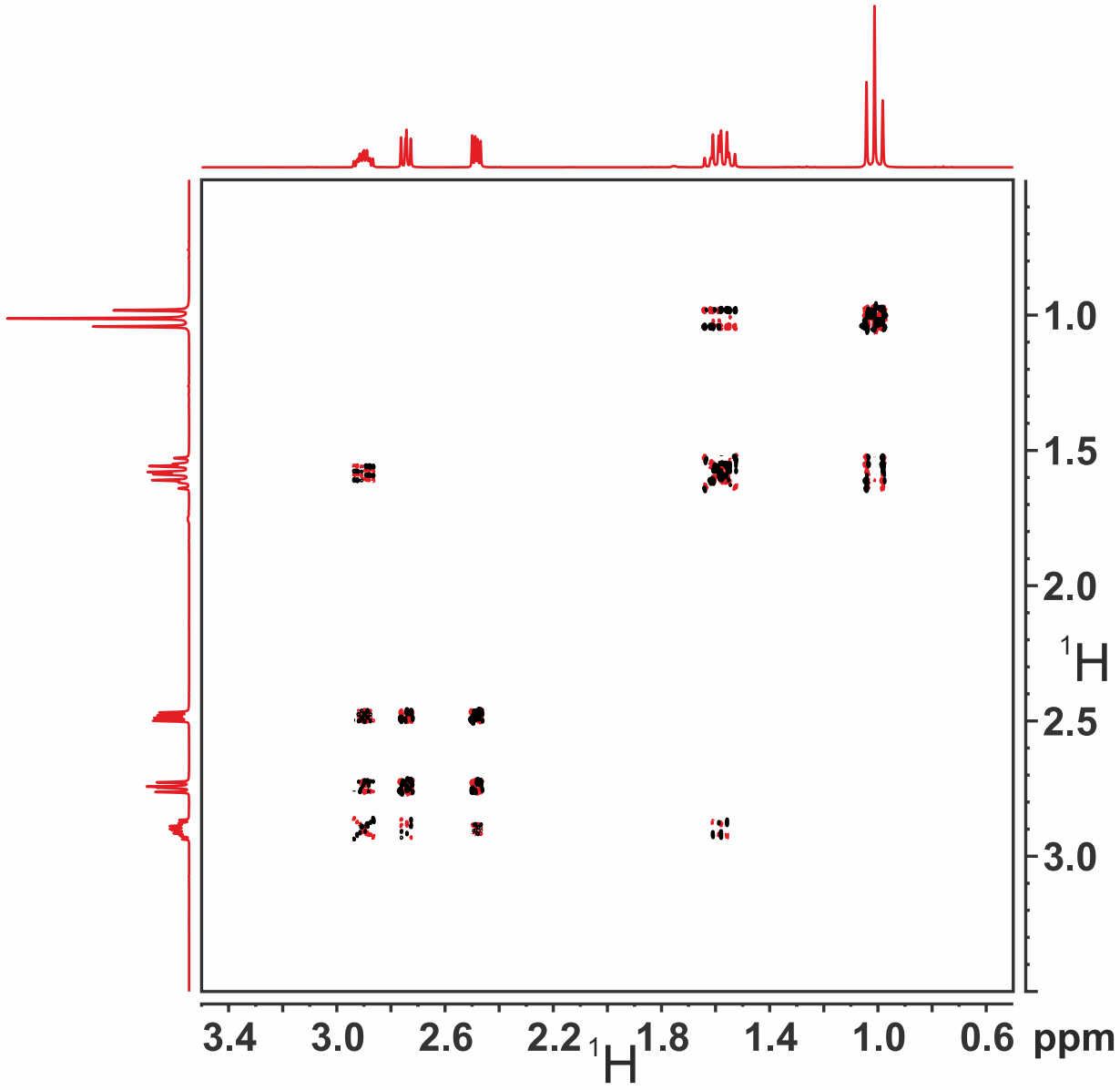
There is only one way to attach the final methylene group. Because this group contains two diastereotopic protons, we observe two cross peaks in the COSY.

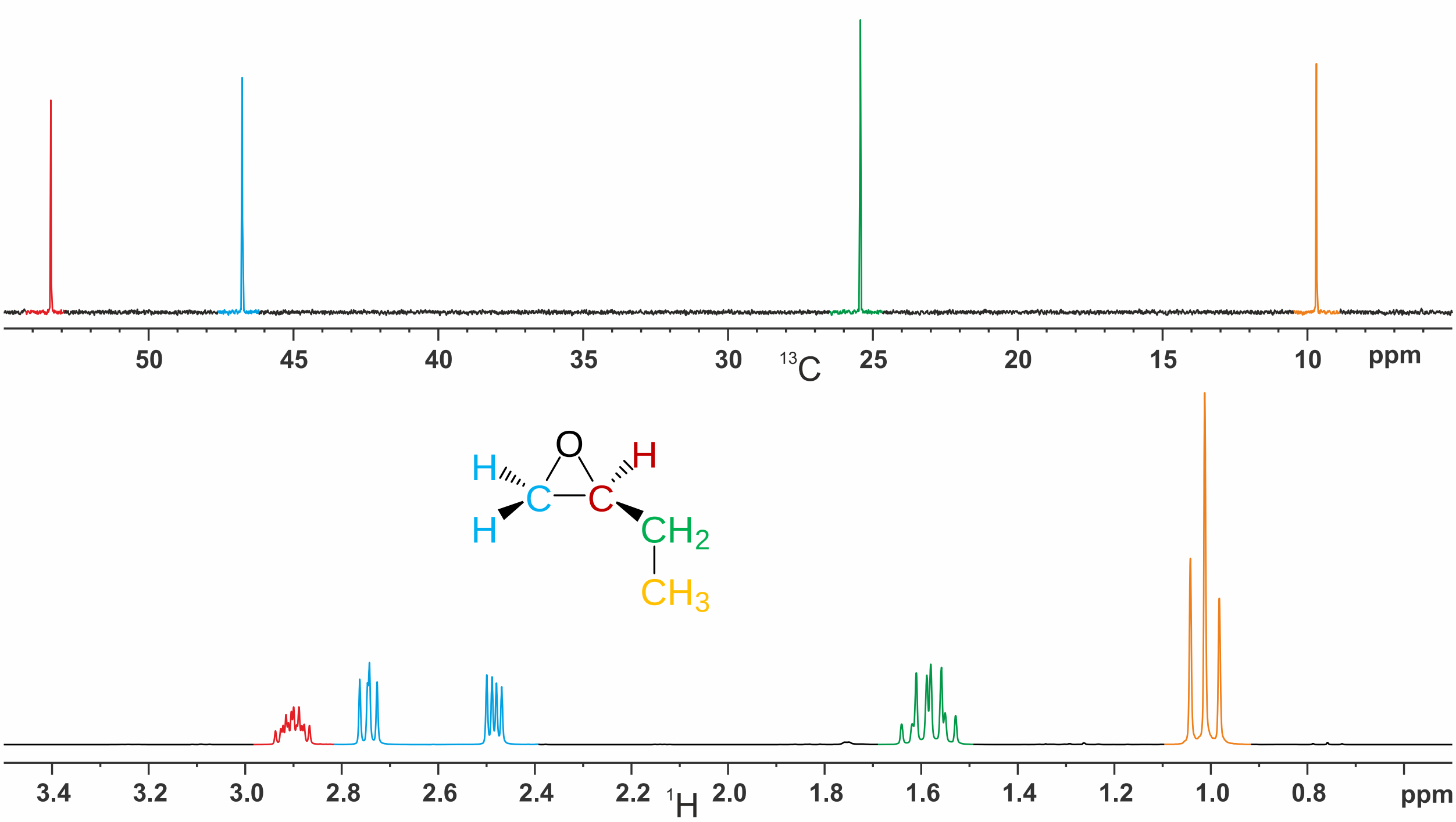




We are left with one unassigned oxygen and two open bonds, both of which we can accommodate in …



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3.4
3.2
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2.6
2.4
2.2
^{1}H
2.0
1.8
1.6
1.4
1.2
1.0
0.8
ppm

Contributions

Spectrometer time

TU Munich

Measurements

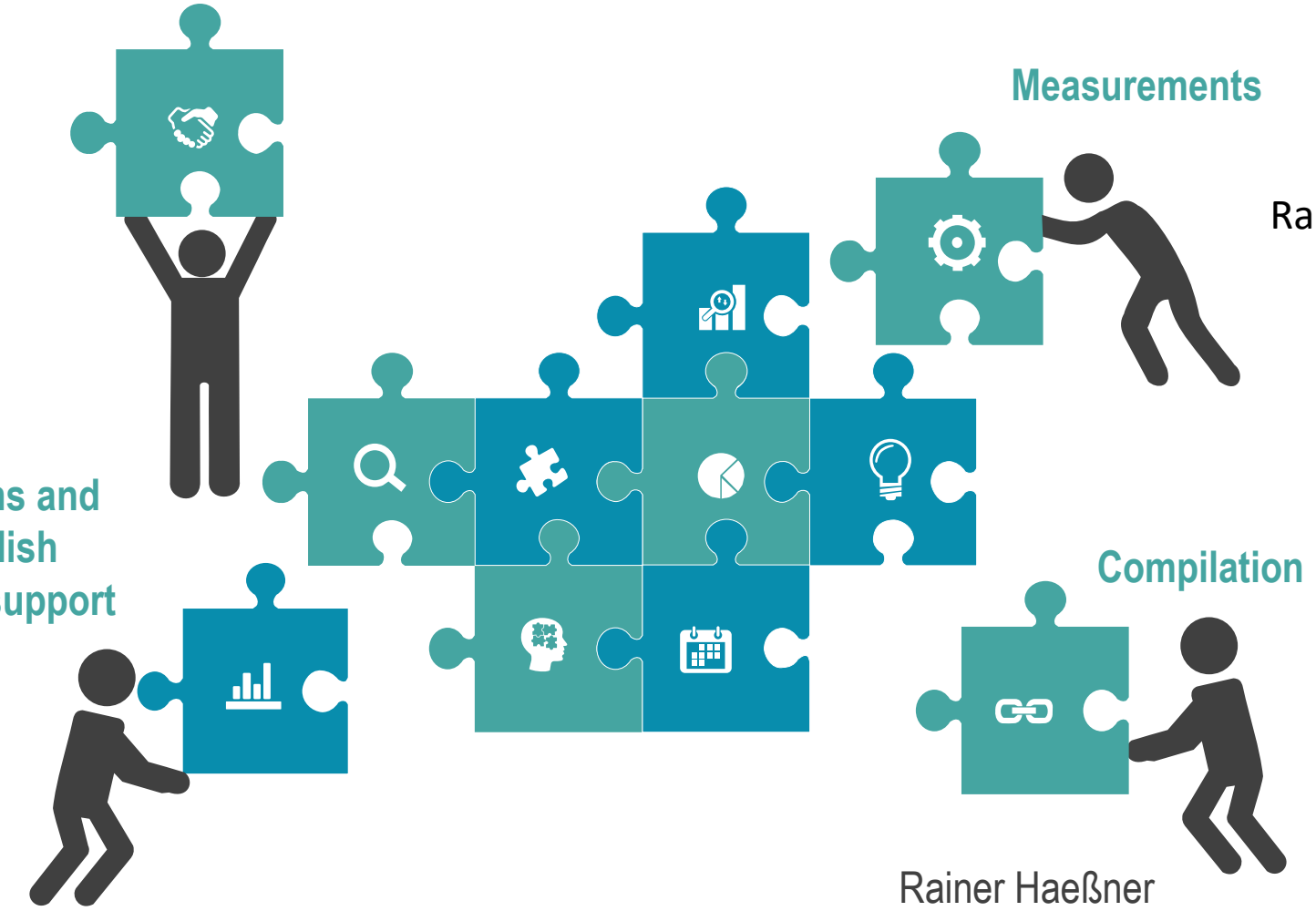
Rainer Haeßner

Discussions and native English language support

Alan Kenwright

Compilation

Rainer Haeßner



More exercises …