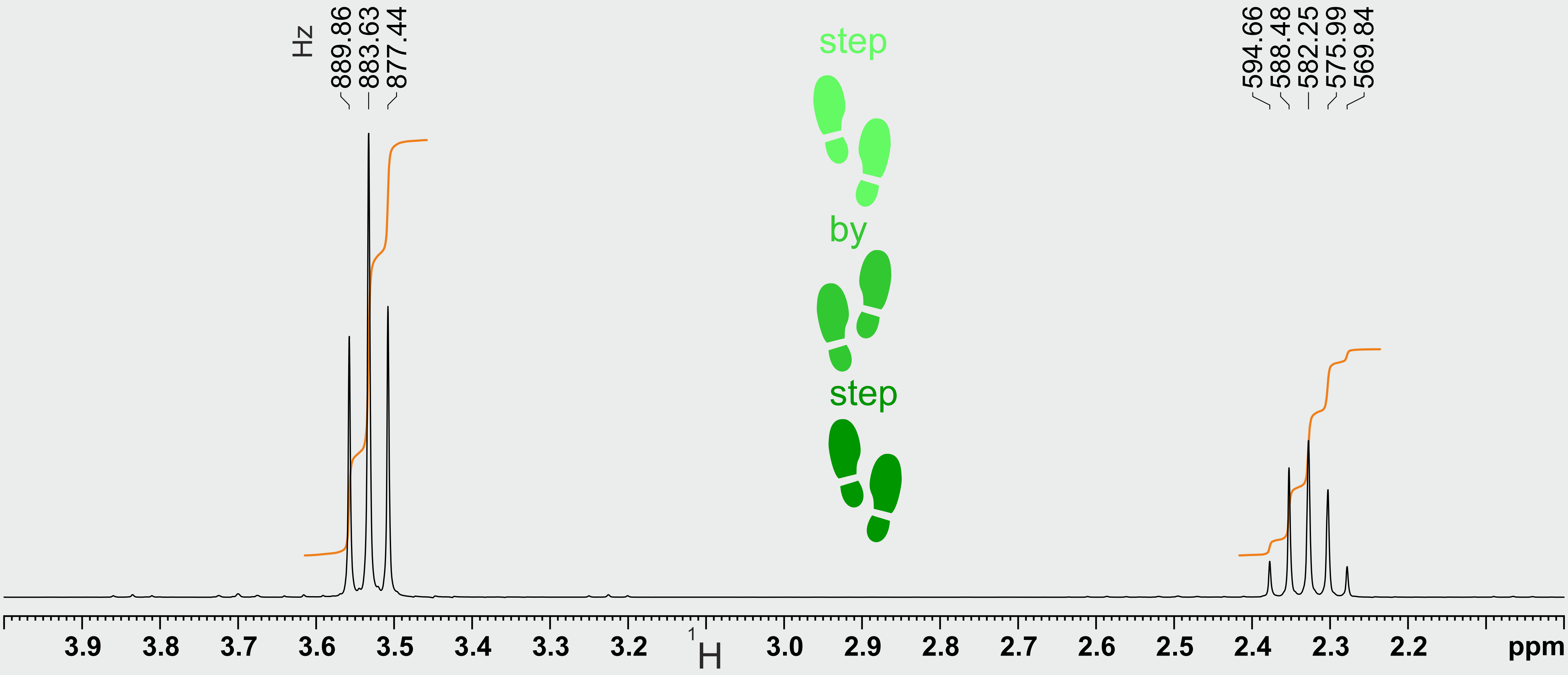
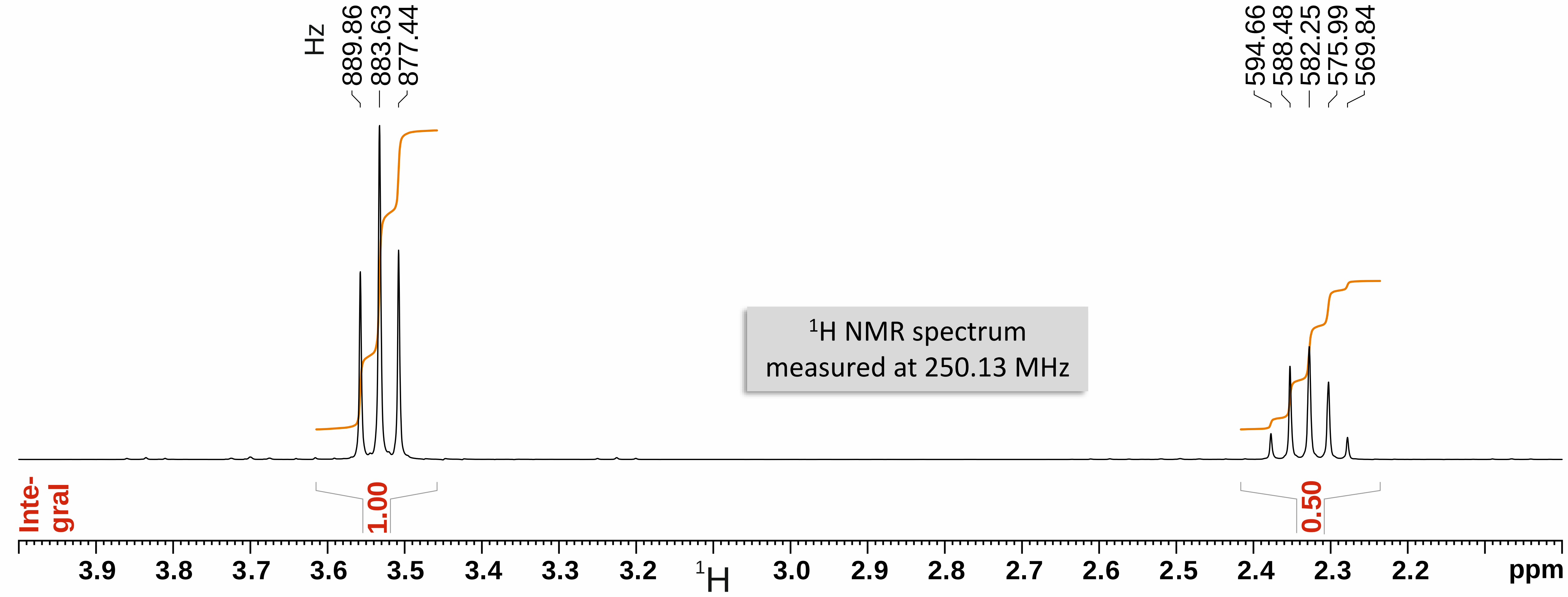
Exercise plus Solution – Quick PDF overview

It is recommended to use this PDF 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.



$C_3H_6Br_2$ measured in $CDCl_3$

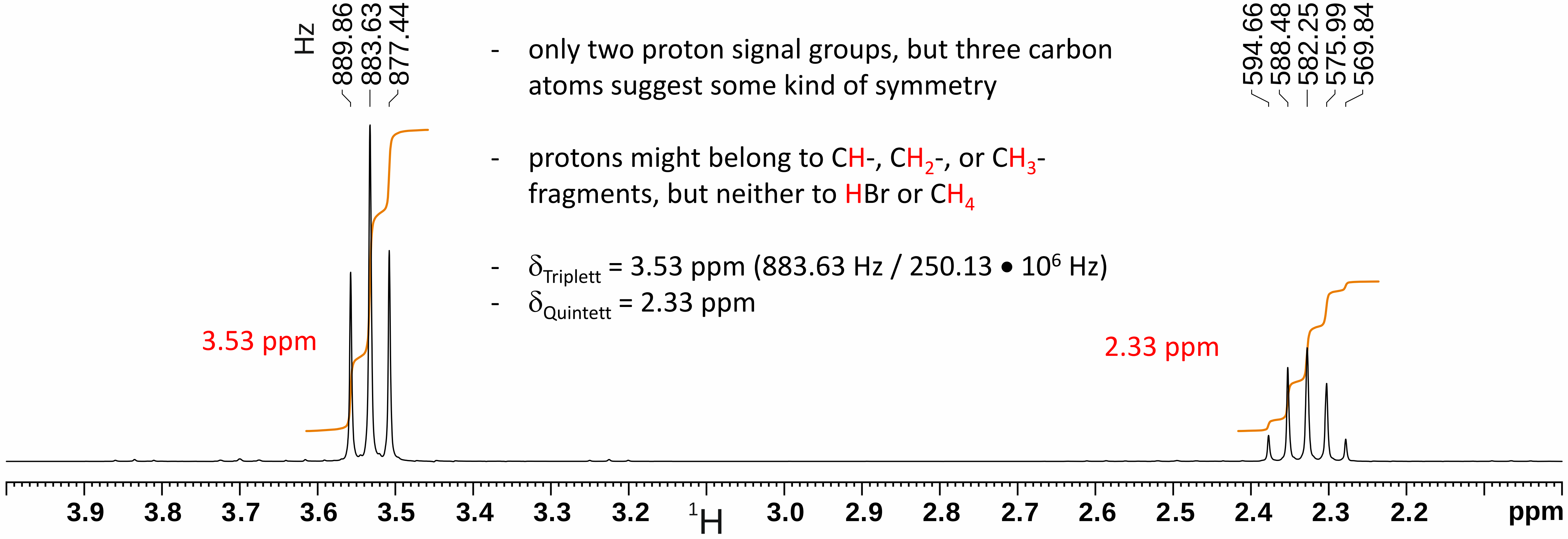
Deduce the structure!



Solution

Part 1 – First considerations

- no double bond equivalents
- only two proton signal groups, but three carbon atoms suggest some kind of symmetry
- protons might belong to CH-, CH_2-, or CH_3-fragments, but neither to HBr or CH_4
- $\delta_{Triplett}$ = 3.53 ppm (883.63 Hz / 250.13 • 10^6 Hz)
- $\delta_{Quintett}$ = 2.33 ppm



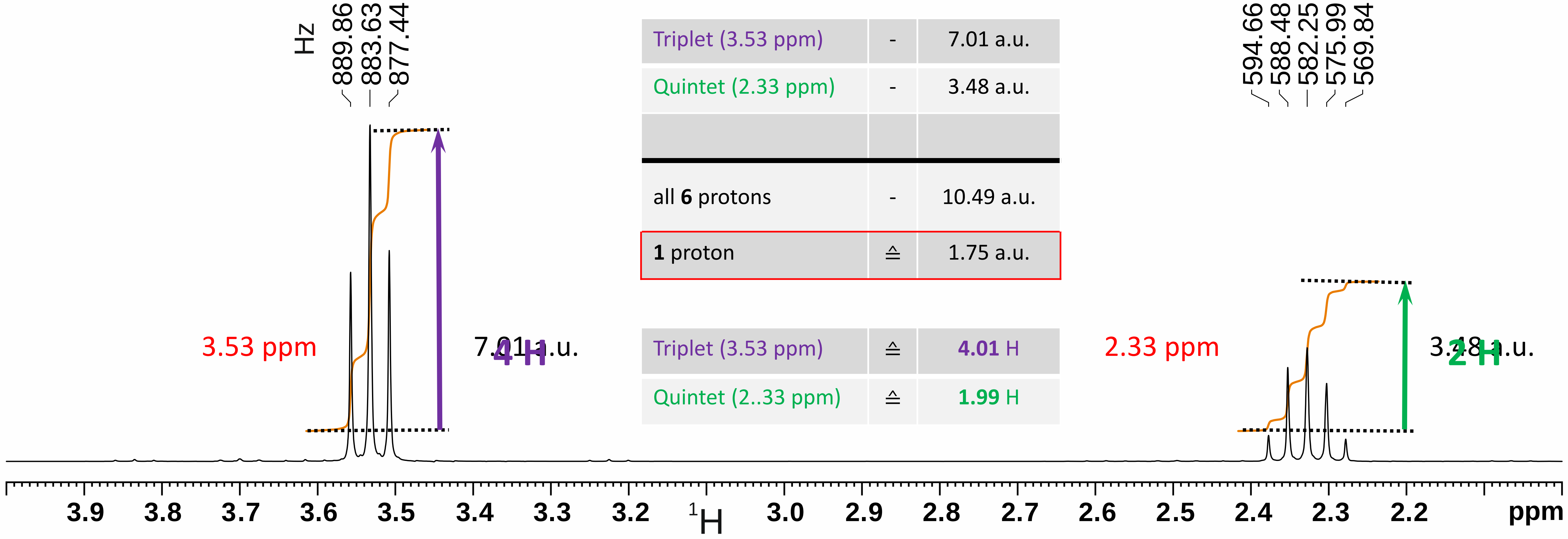
Solution

Part 2 - Integration

a.u. ???

arbitrary units

This depends on the output device and your home country.
For example, using a tablet in Europe, you could think about "**centimeters**".



Triplet (3.53 ppm)	-	7.01 a.u.
Quintet (2.33 ppm)	-	3.48 a.u.
all **6** protons	-	10.49 a.u.
1 proton	≙	1.75 a.u.
Triplet (3.53 ppm)	≙	**4.01** H
Quintet (2..33 ppm)	≙	**1.99** H

Solution

Part 3 – Building blocks

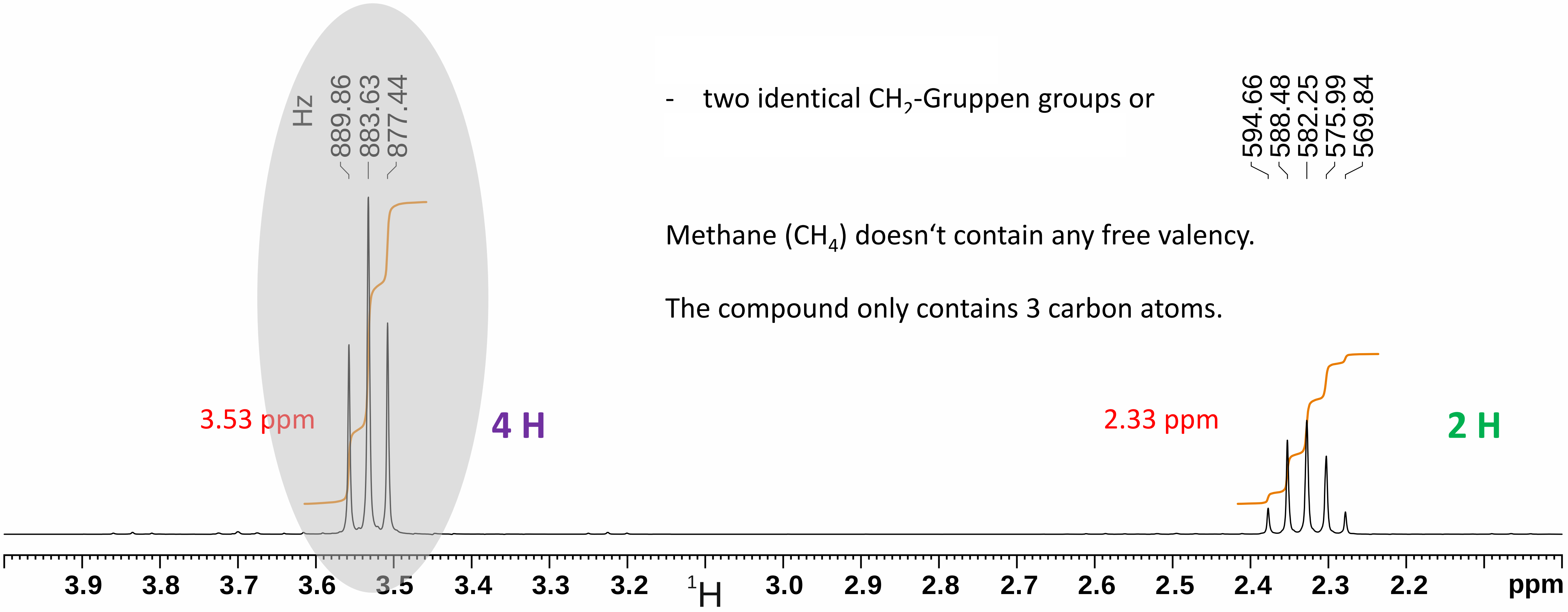
There is no double bond equivalent and only three types of atoms (C, H, and Br).

Using these pieces of information, a multiplet containing 4 protons only might be constructed from

- two identical CH_2-Gruppen groups or

Methane (CH_4) doesn‘t contain any free valency.

The compound only contains 3 carbon atoms.



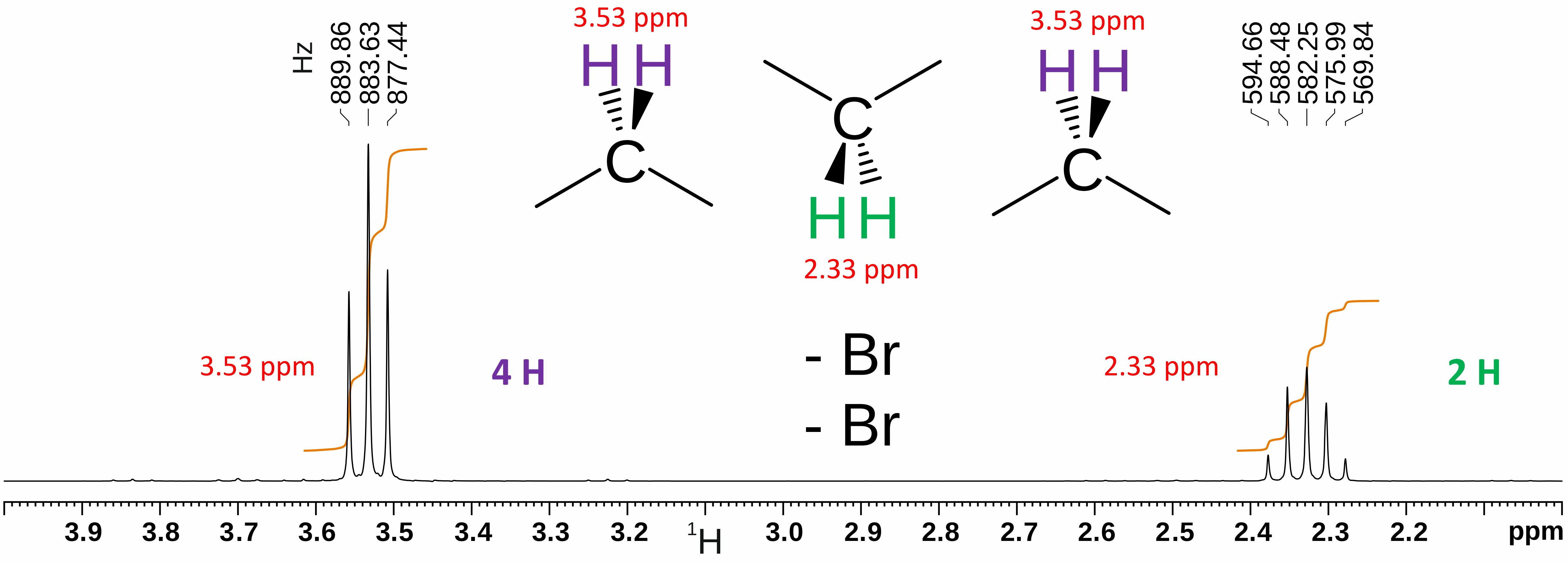
Solution

Part 3 – Building blocks

molecular formula - $C_3H_6Br_2$

already assigned - C_2H_4
still to assign - CH_2Br_2

HBr – as finished molecule – is excluded



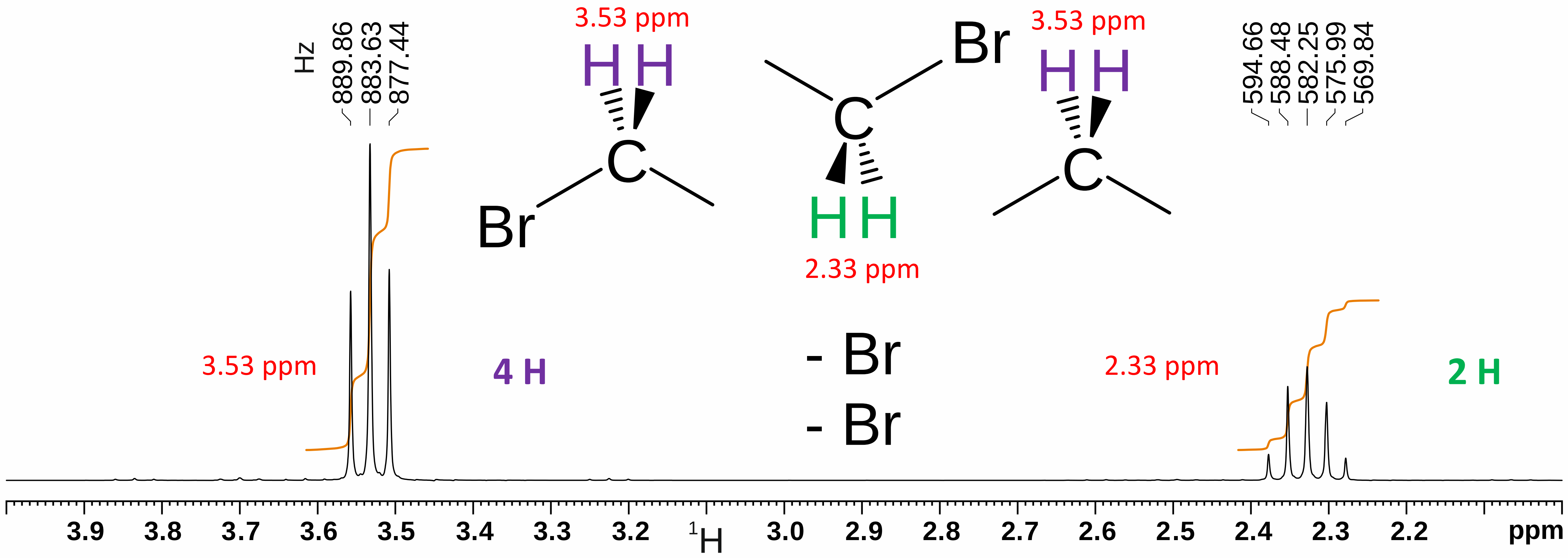
Solution

Part 4 – Insert -Br

Attaching both Br to the same carbon atom would result in CH_2Br_2, which has no free valency (like methane).

Attaching one Br to the methylene group with proton chemical shifts of 3.53 ppm and the other Br to the methylene group with proton chemical shifts of 2.33 ppm would result in three different signal groups.

There is only one remaining possibility …

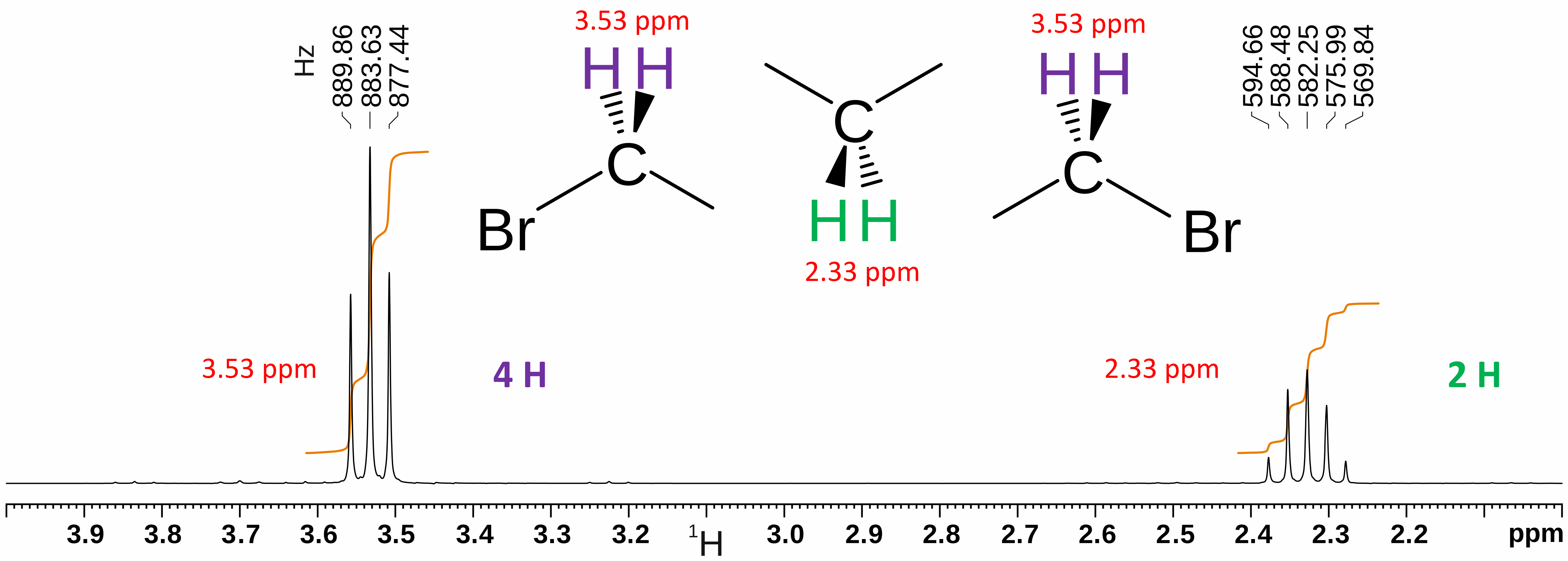


Solution

Part 4 – Insert -Br

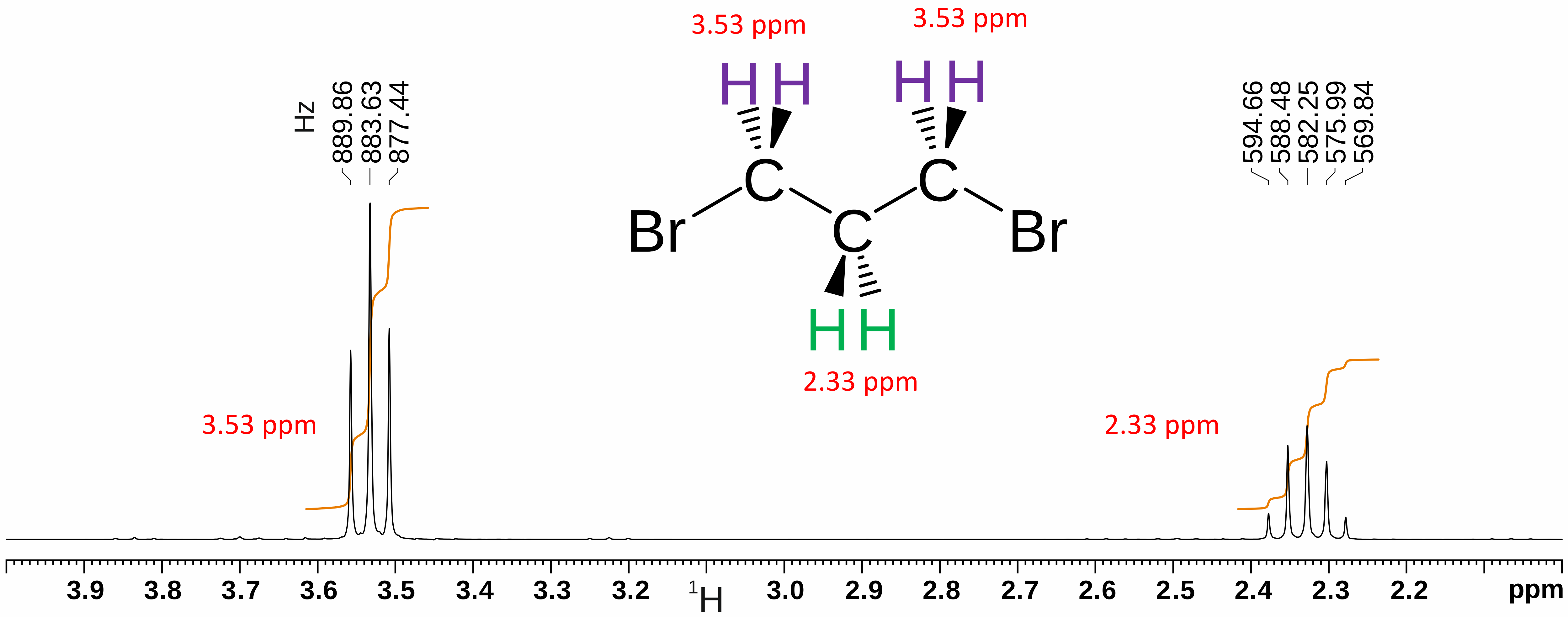
Now the CH_2 groups with proton chemical shifts of 3.53 ppm stay symmetric.

There is only one way to put it all together.



Solution

Part 4 – Insert -Br

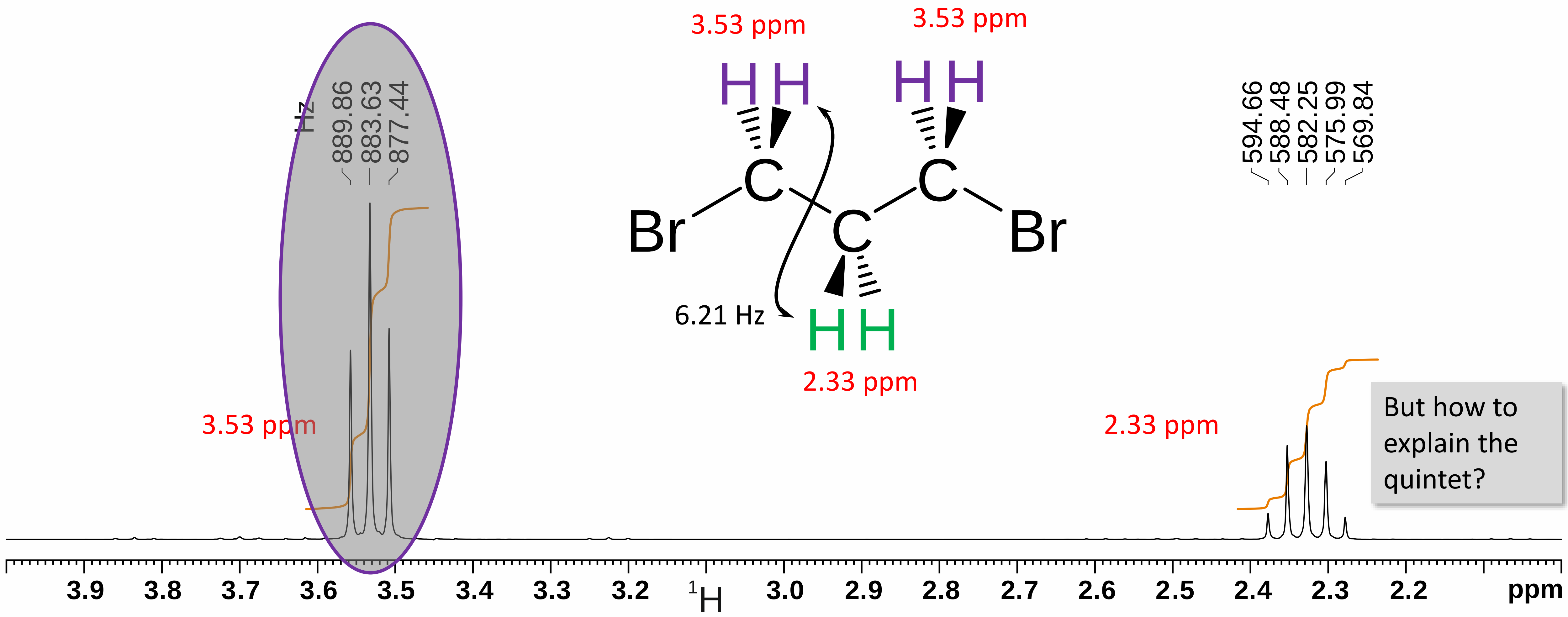


Solution

Part 5 – Multiplets and coupling constants

The explanation for the triplet at 3.53 ppm ist easy. There are two equivalent neighbour protons. This is valid for the CH_2 groups both in 1- and 3-position of the molecule.

From the triplet you can calculate the vicinal coupling constant
((889.86 Hz – 877.44 Hz) / 2)



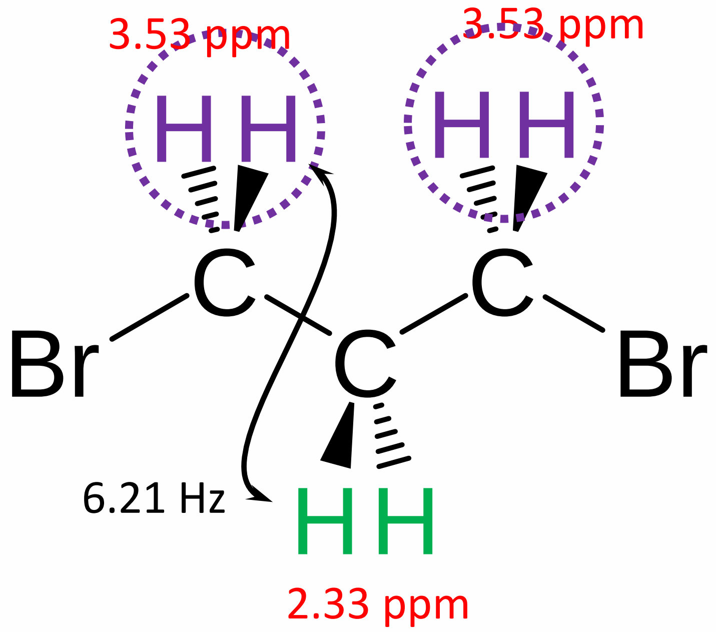
But how to explain the quintet?

Solution

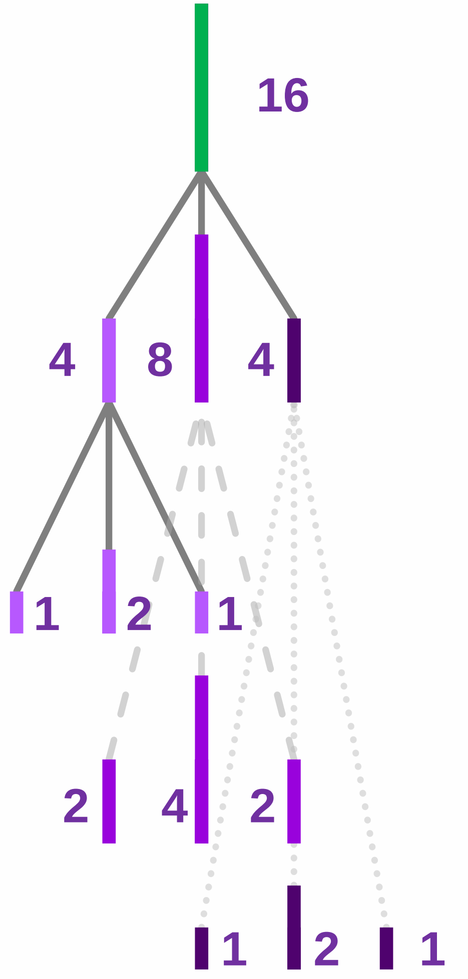
Part 5 – Multiplets and coupling constants

There are four equivalent neighbouring protons to the methylene group protons at 2.33 ppm. As the resulting coupling pattern we expect a quintet.
The neighbouring protons do not necessarily have to be bound to the same carbon atom.

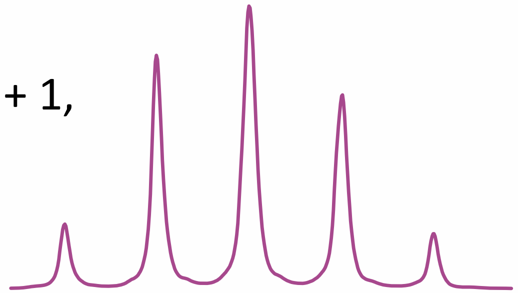
If this view initially causes difficulties, one can also think of the multiplet as a triplet of triplets with identical coupling constants. Let us inspect the coupling with the methylene group protons at the left side, neglecting the methylene group protons at the right side.



If we now take the methylene group protons at the right side into account, each line separately splits into a triplet.

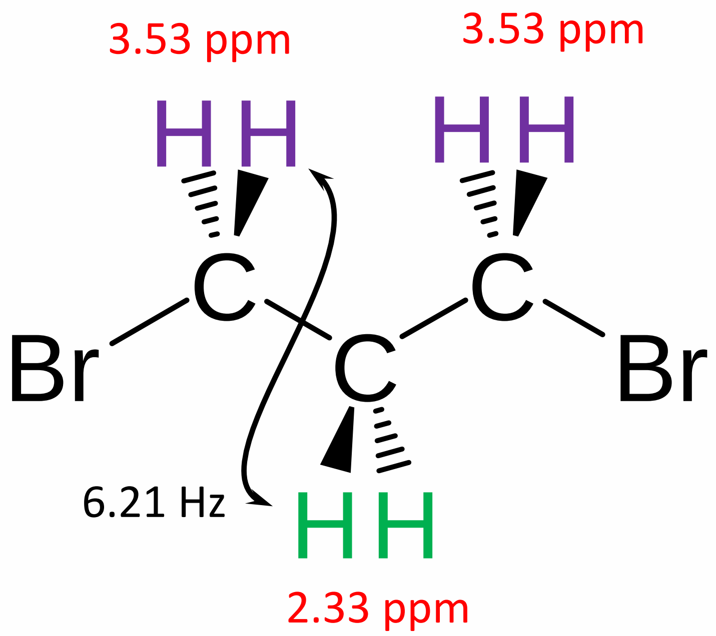


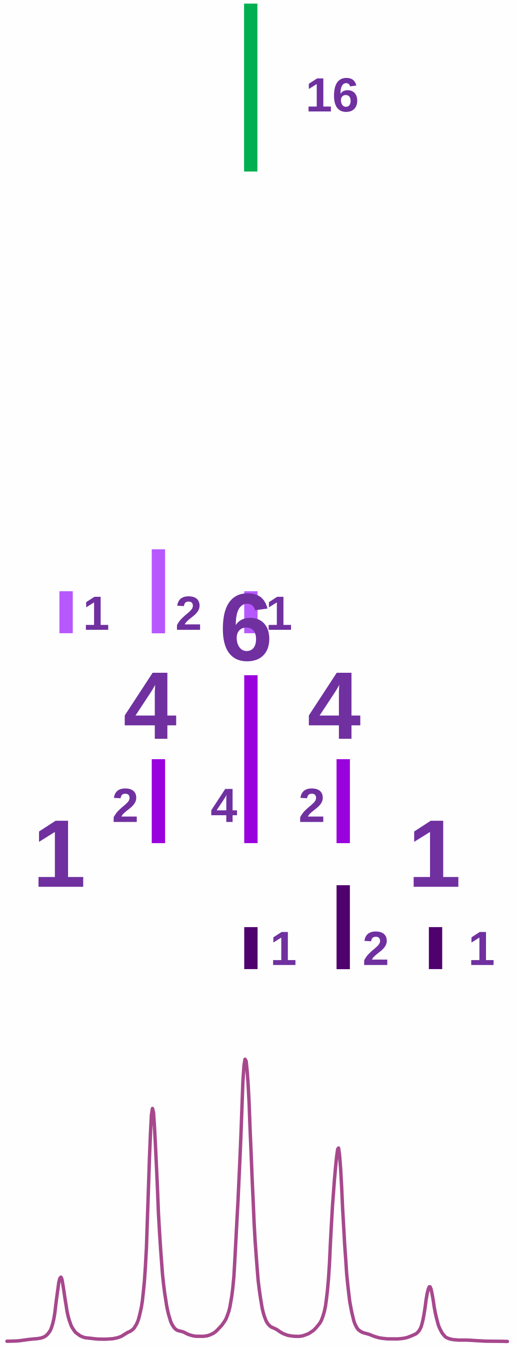
And now we have to add 1 + 1, etc



Solution

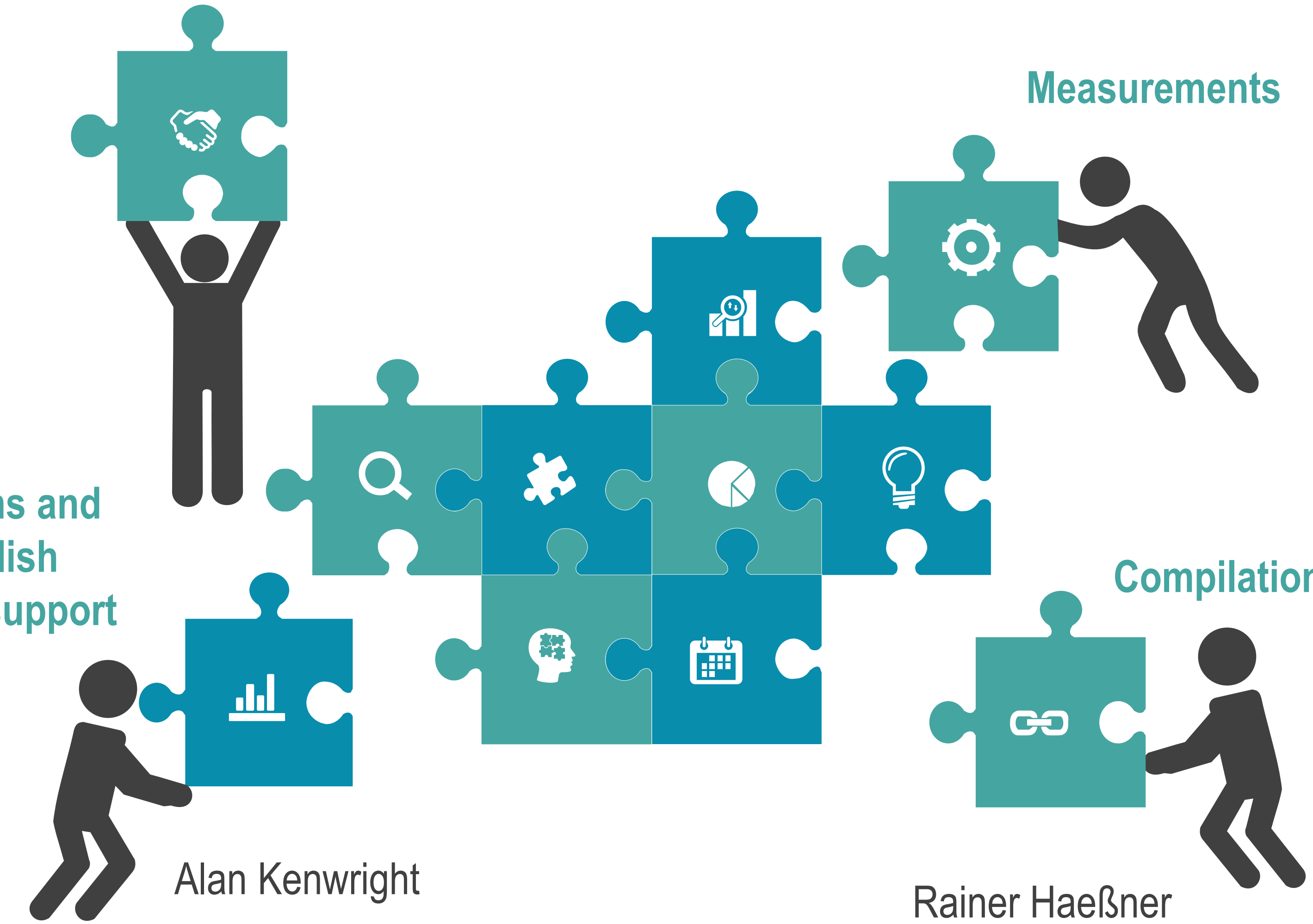
Part 5 – Multiplets and coupling constants





Due to the roofing effect the intensity ratio of the measured quintet differs a little bit from the ideal quintet.

Contributions



Spectrometer time

TU Munich

Measurements

Rainer Haeßner

Discussions and native English language support

Alan Kenwright

Compilation

Rainer Haeßner

More exercises …