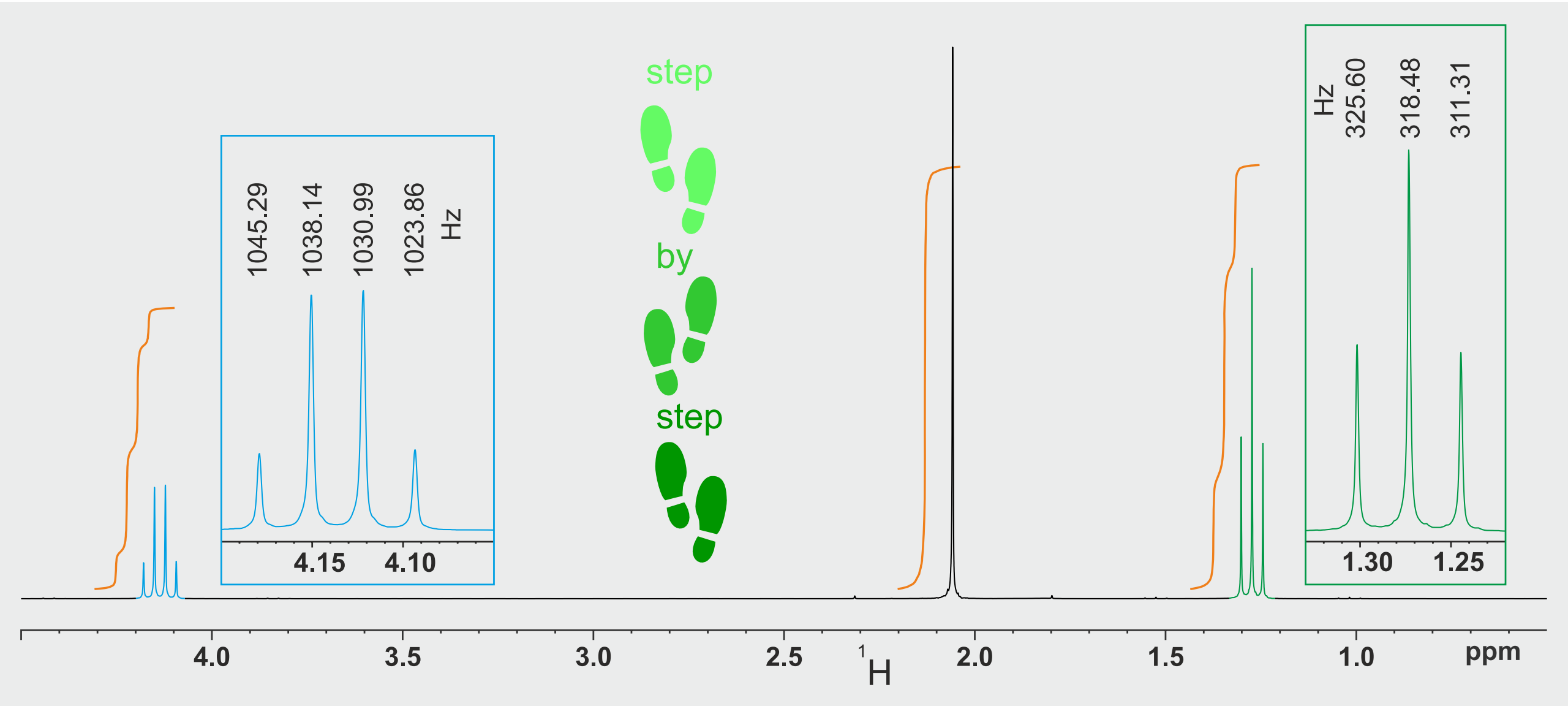
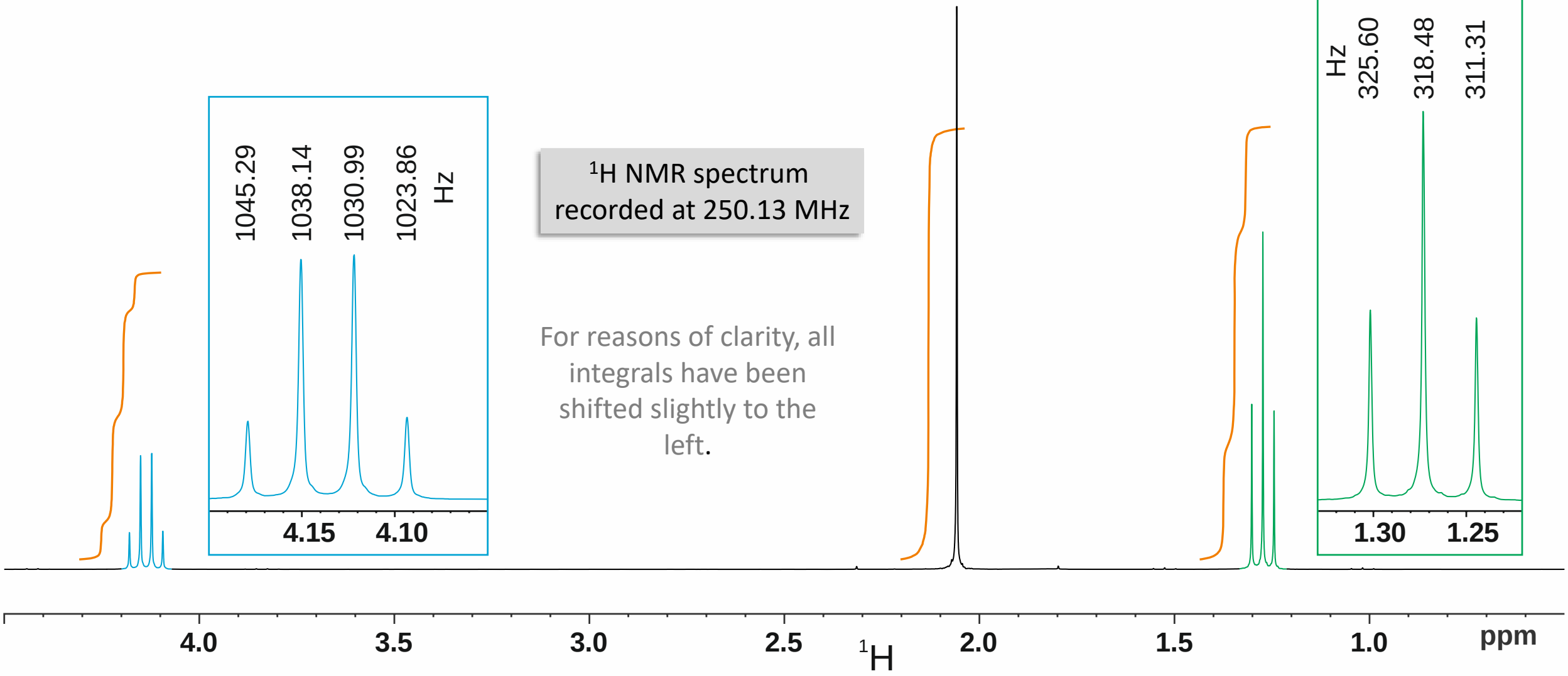
Exercise plus Solution – Quick overview

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.



$C_4H_8O_2$ measured in $CDCl_3$

Deduce the structure and measure the coupling constant!



^{1}H NMR spectrum recorded at 250.13 MHz

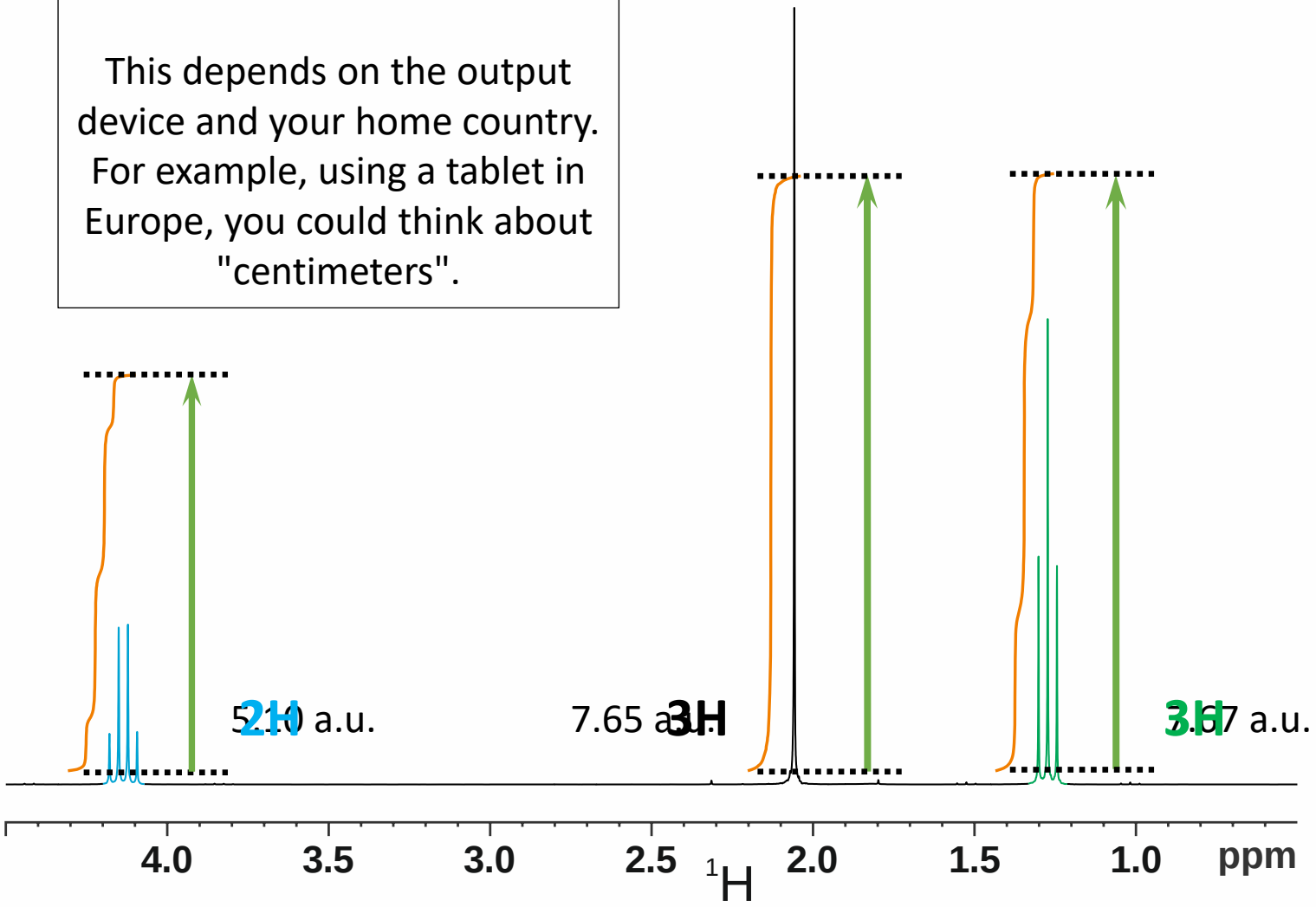
For reasons of clarity, all integrals have been shifted slightly to the left.

Solution

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".

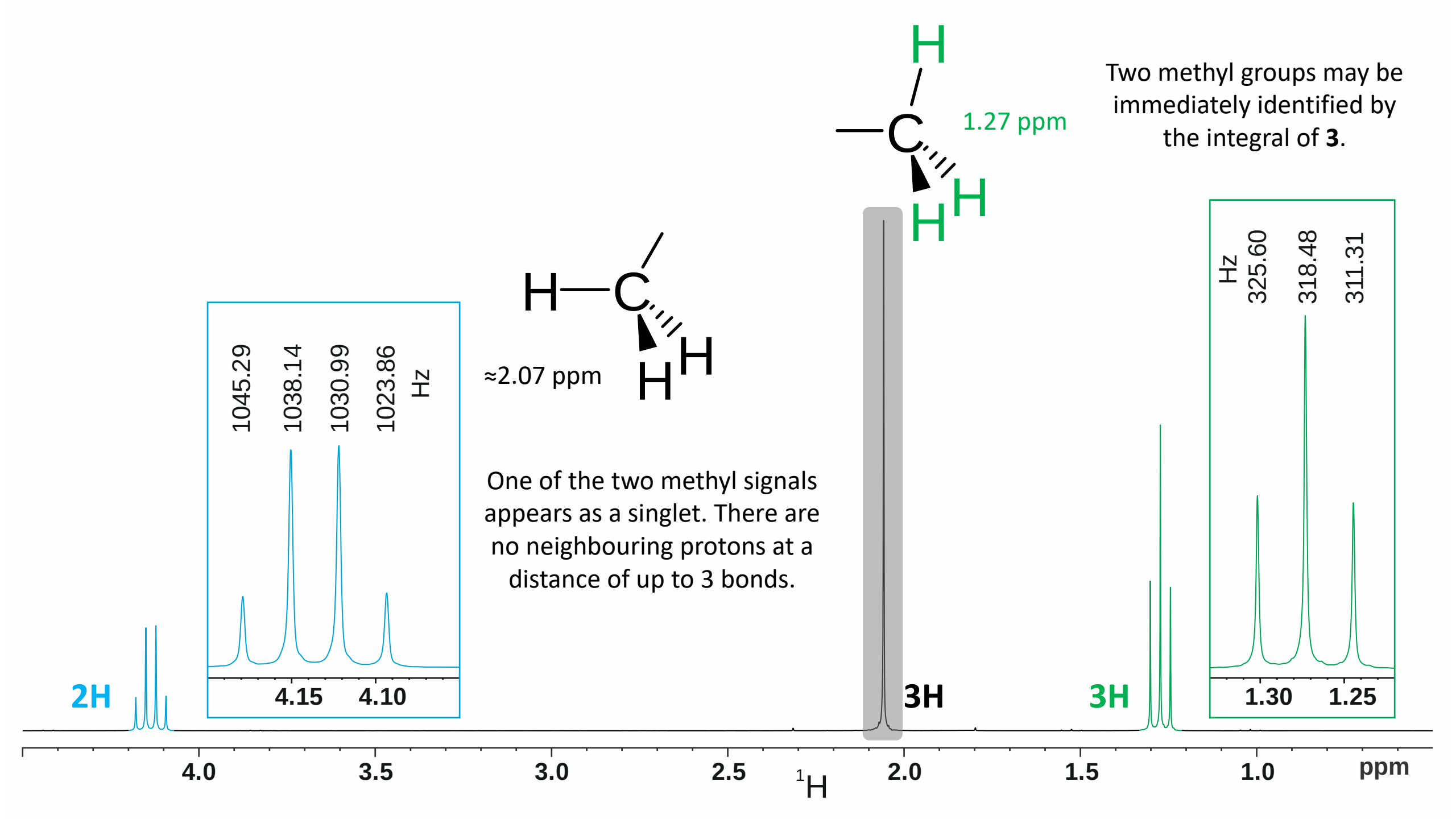


$C_4H_8O_2$

- one degree of unsaturation (double bond equivalent)
- three signal groups
- Integration:

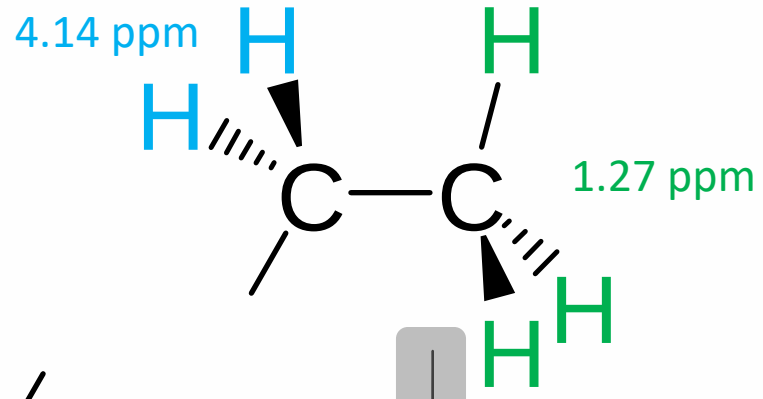
low field multiplet	-	5.10 a.u.
singlet at 2.1 ppm	-	7.65 a.u.
high field multiplet	-	7.67 a.u.
all **8** protons	-	20.42 a.u.
1 proton	≙	2.55 a.u.

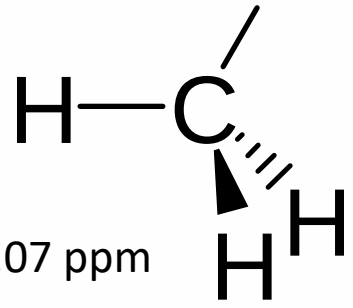
low field multiplet	≙	**2.00** H
singlet at 2.1 ppm	≙	**2.99** H
high field multiplet	≙	**3.01** H


Two methyl groups may be immediately identified by the integral of **3**.
1.27 ppm
≈2.07 ppm
One of the two methyl signals appears as a singlet. There are no neighbouring protons at a distance of up to 3 bonds.
1045.29
1038.14
1030.99
1023.86
Hz
4.15
4.10
325.60
318.48
311.31
Hz
1.30
1.25
2H
3H
3H
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
^{1}H

According to the n-1 rule, there are two equivalent protons in the vicinity (up to three bonds) of this methyl group.

These protons must be on a methylene group, because there can only be one neighbouring group.

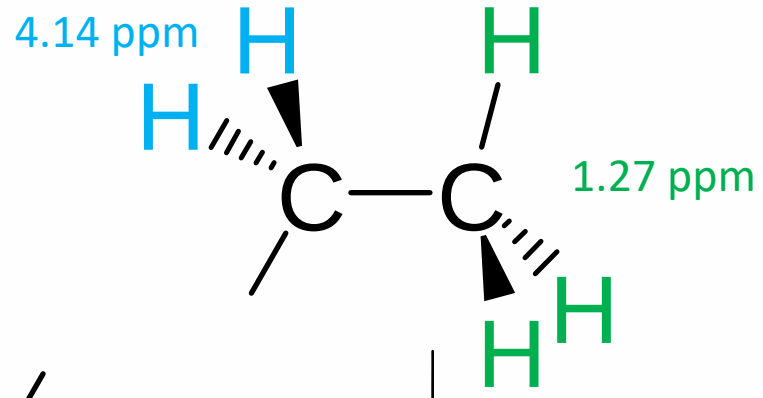


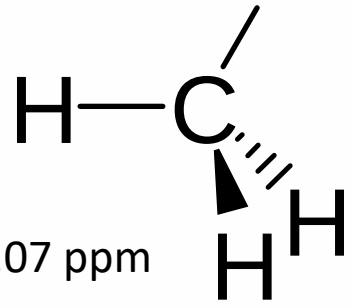


One of the two methyl signals appears as a singlet. There are no neighbouring protons at a distance of up to 3 bonds.

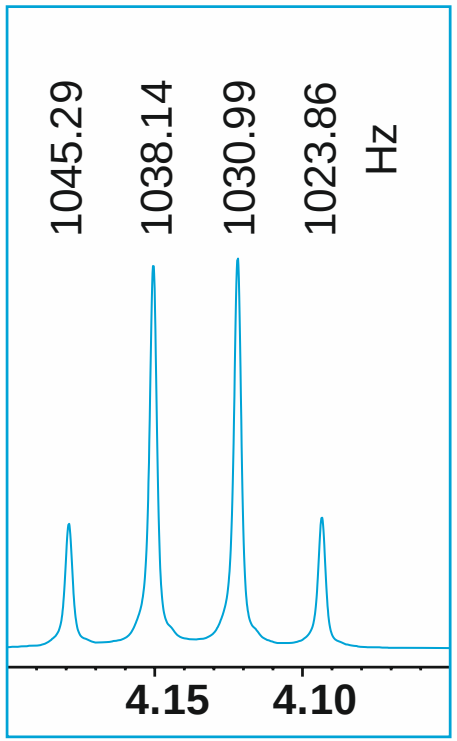
According to the n-1 rule, there are two equivalent protons in the vicinity (up to three bonds) of this methyl group.

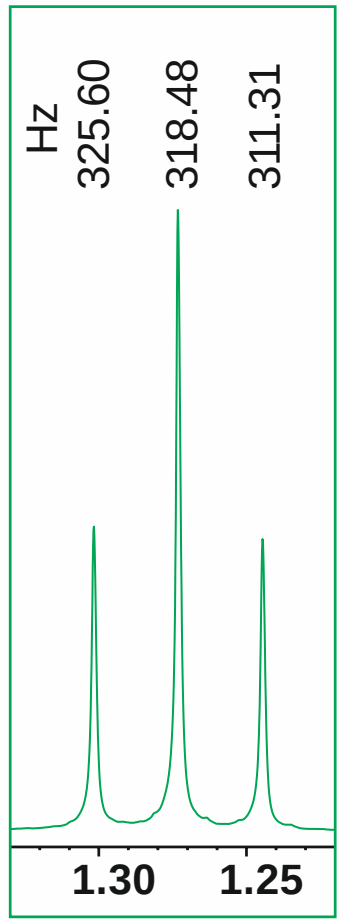
These protons must be on a methylene group, because there can only be one neighbouring group.

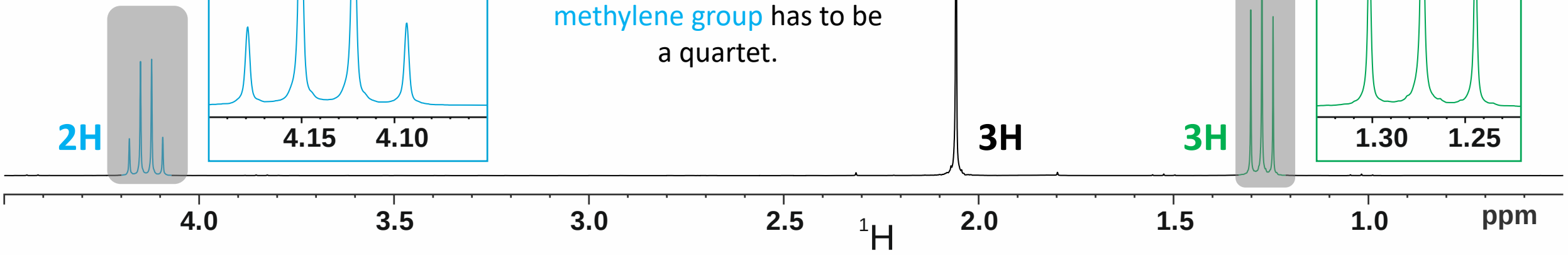


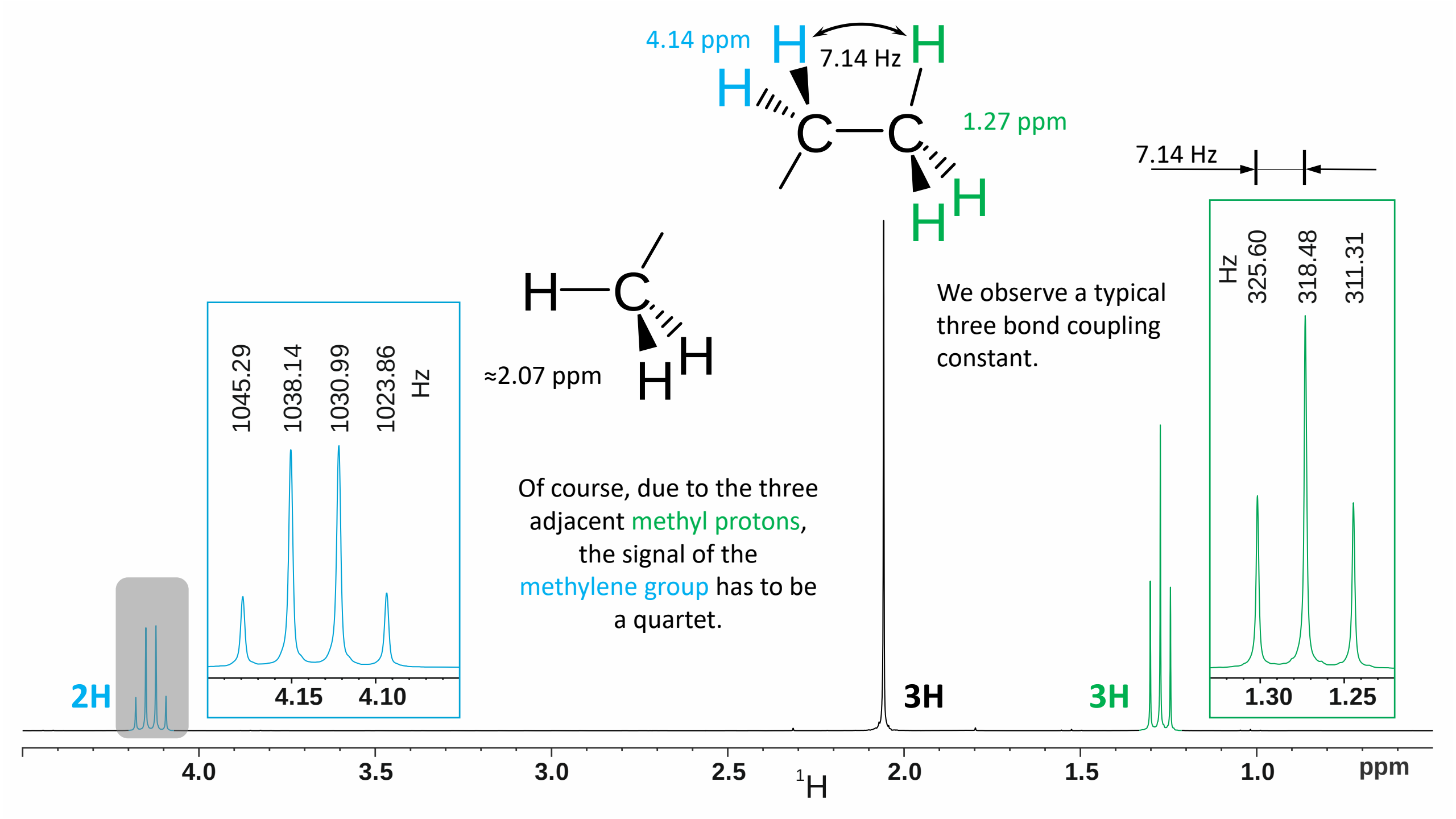


Of course, due to the three adjacent methyl protons, the signal of the methylene group has to be a quartet.

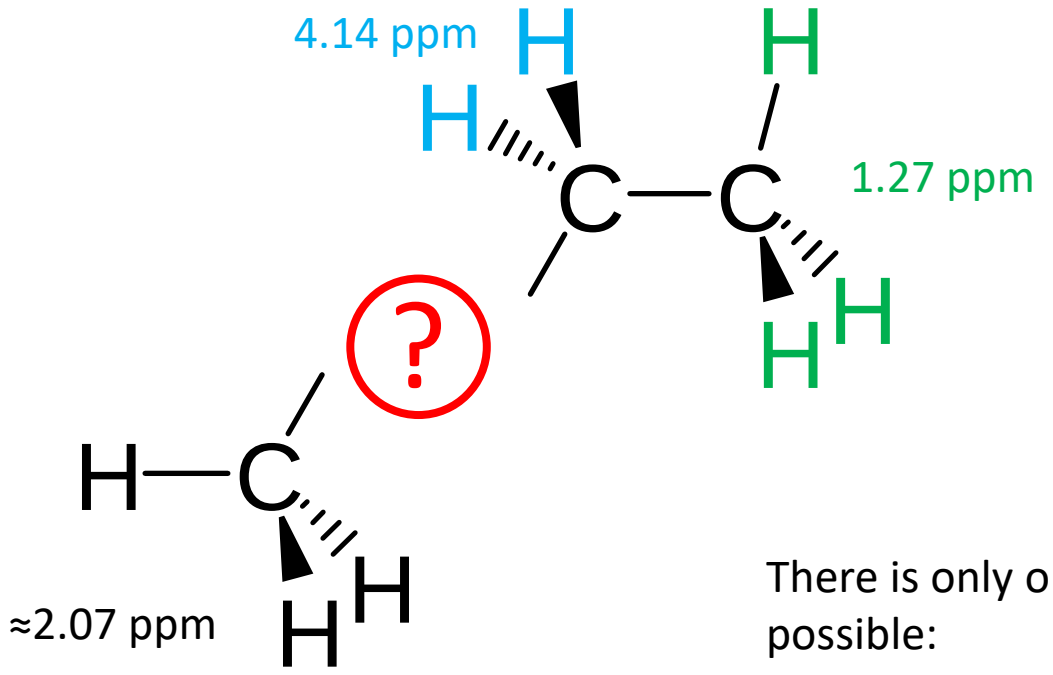




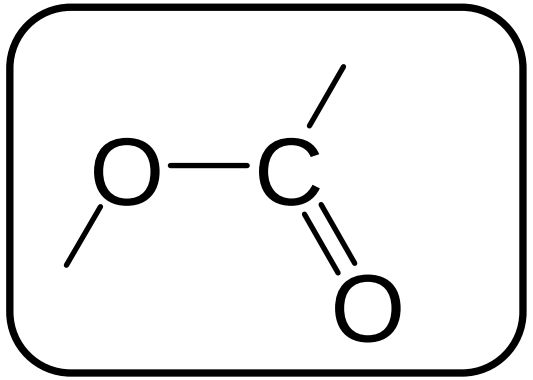



4.14 ppm
7.14 Hz
1.27 ppm
≈2.07 ppm
7.14 Hz
Hz
325.60
318.48
311.31
1045.29
1038.14
1030.99
1023.86
Hz
We observe a typical three bond coupling constant.
Of course, due to the three adjacent methyl protons, the signal of the methylene group has to be a quartet.
2H
3H
3H
4.15
4.10
1.30
1.25
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
^{1}H

There are still one carbon atom, two oxygen atoms and a double bond equivalent to be placed between the two known fragments.



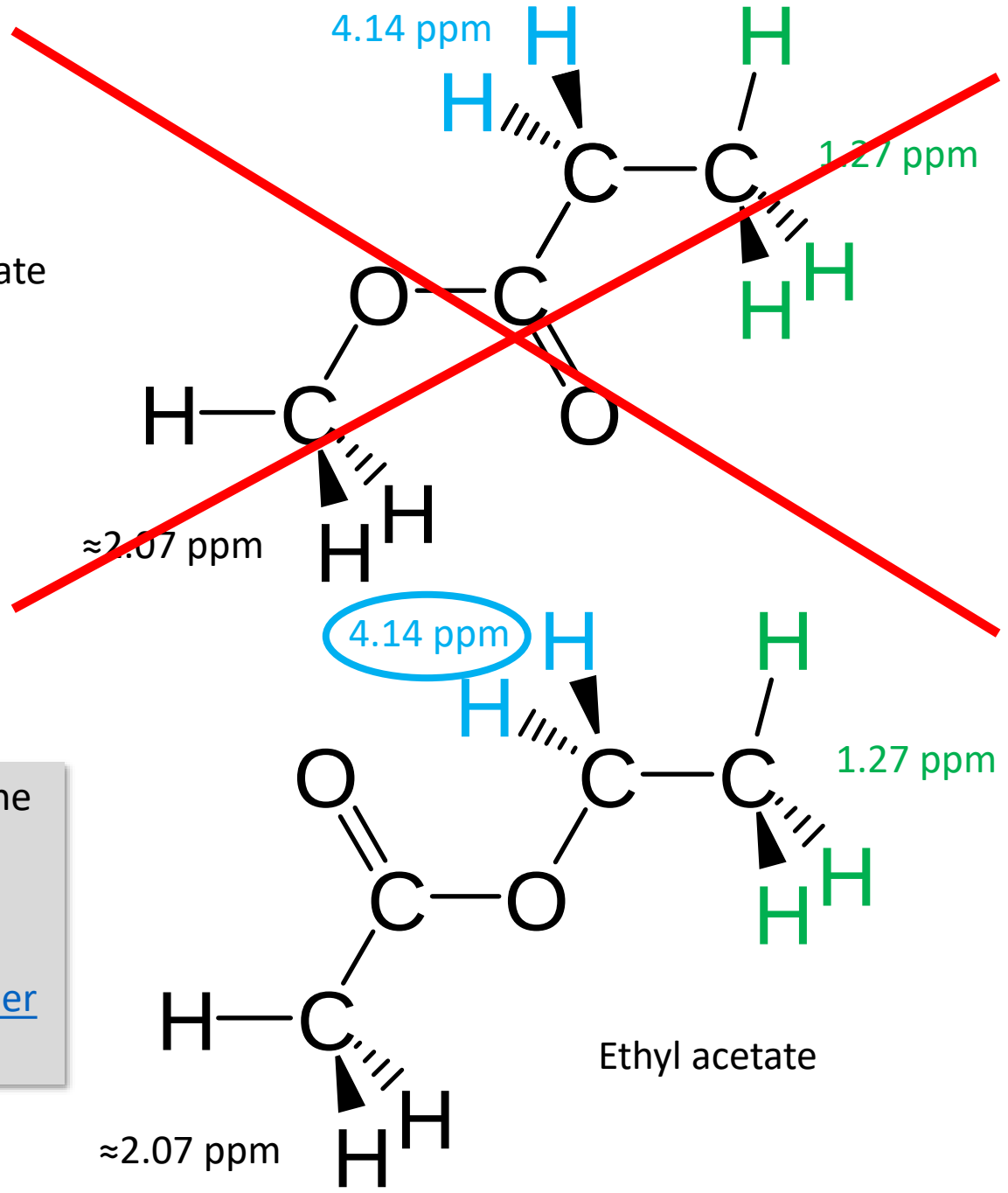
There is only one fragment possible:



But how should the fragment be placed between the two free valences?

There are two possibilities.

Methyl propionate



base	0,23
-CH_3	0,47
-COOR	1,55
sum	**2,25**

or

Ethyl acetate

base	0,23
-CH_3	0,47
-OCOR	3,13
sum	**3,83**

It is worth attempting to estimate the chemical shift of the methylene protons using the simple Schoolery rules.
https://en.wikipedia.org/wiki/Shoolery%27s_rule

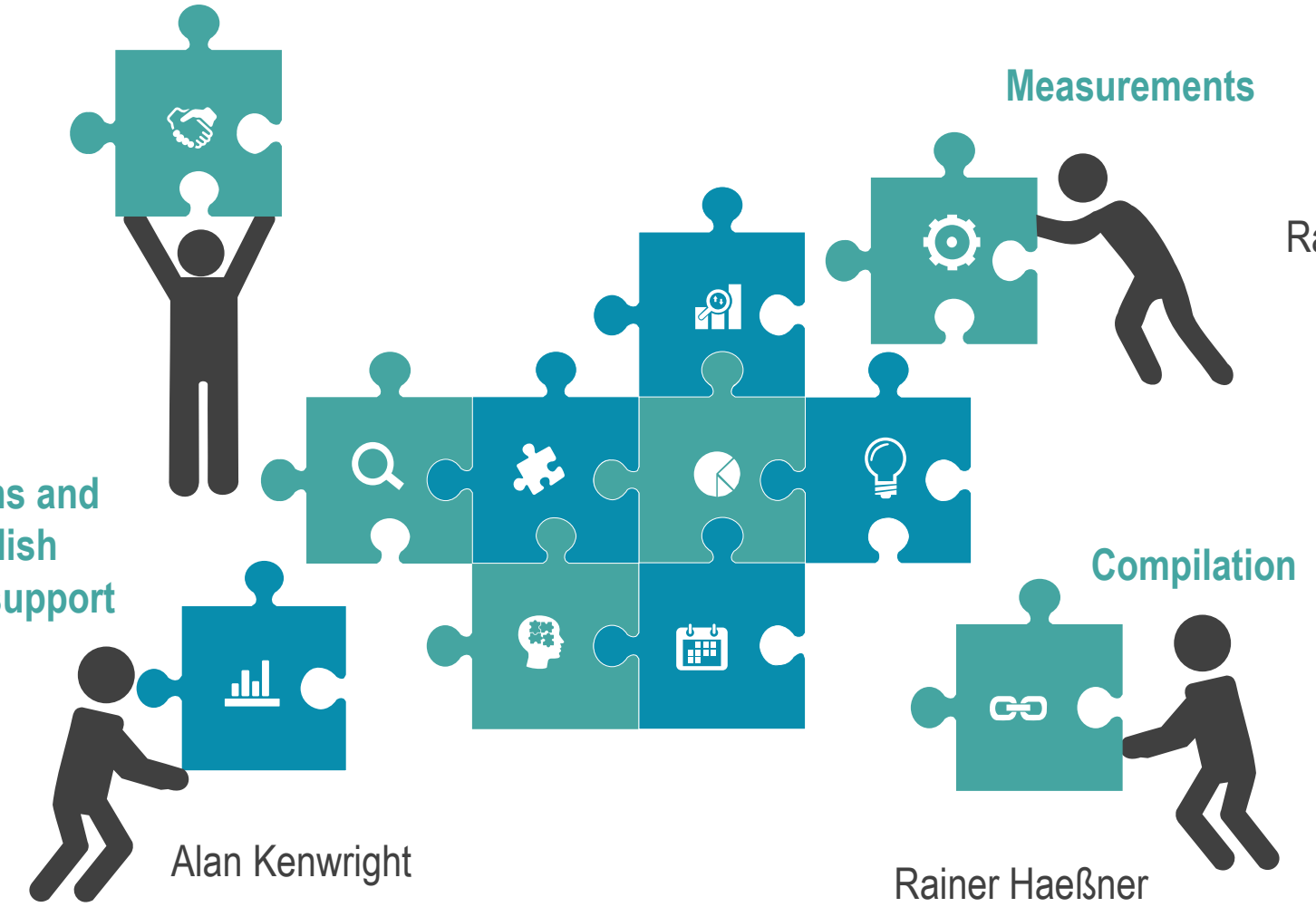
Contributions

Spectrometer time

TU Munich

Measurements

Rainer Haeßner



Discussions and native English language support

Alan Kenwright

Compilation

Rainer Haeßner

More exercises …