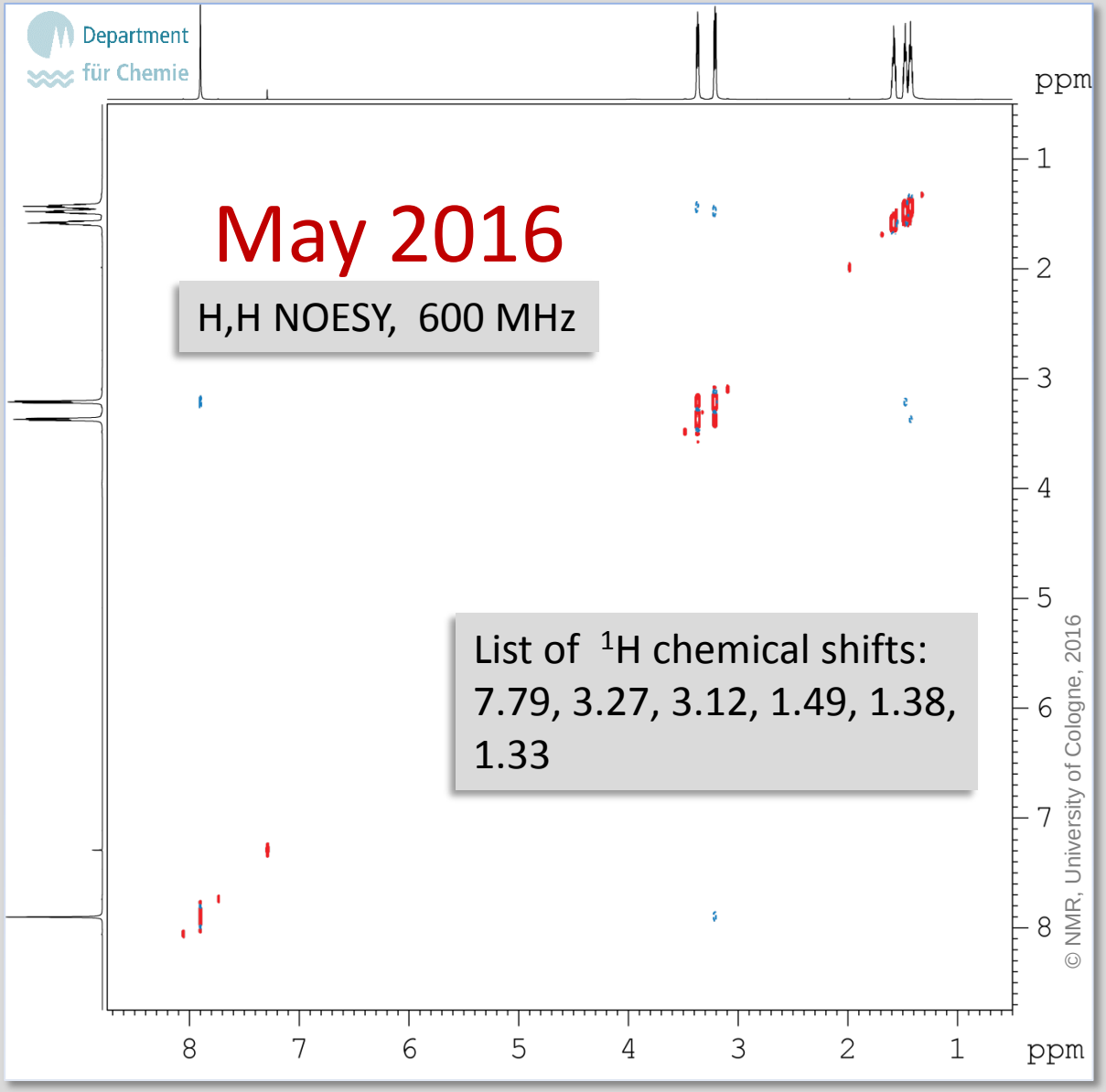
Problem of the Month:



Problem of the Month:
May 2016

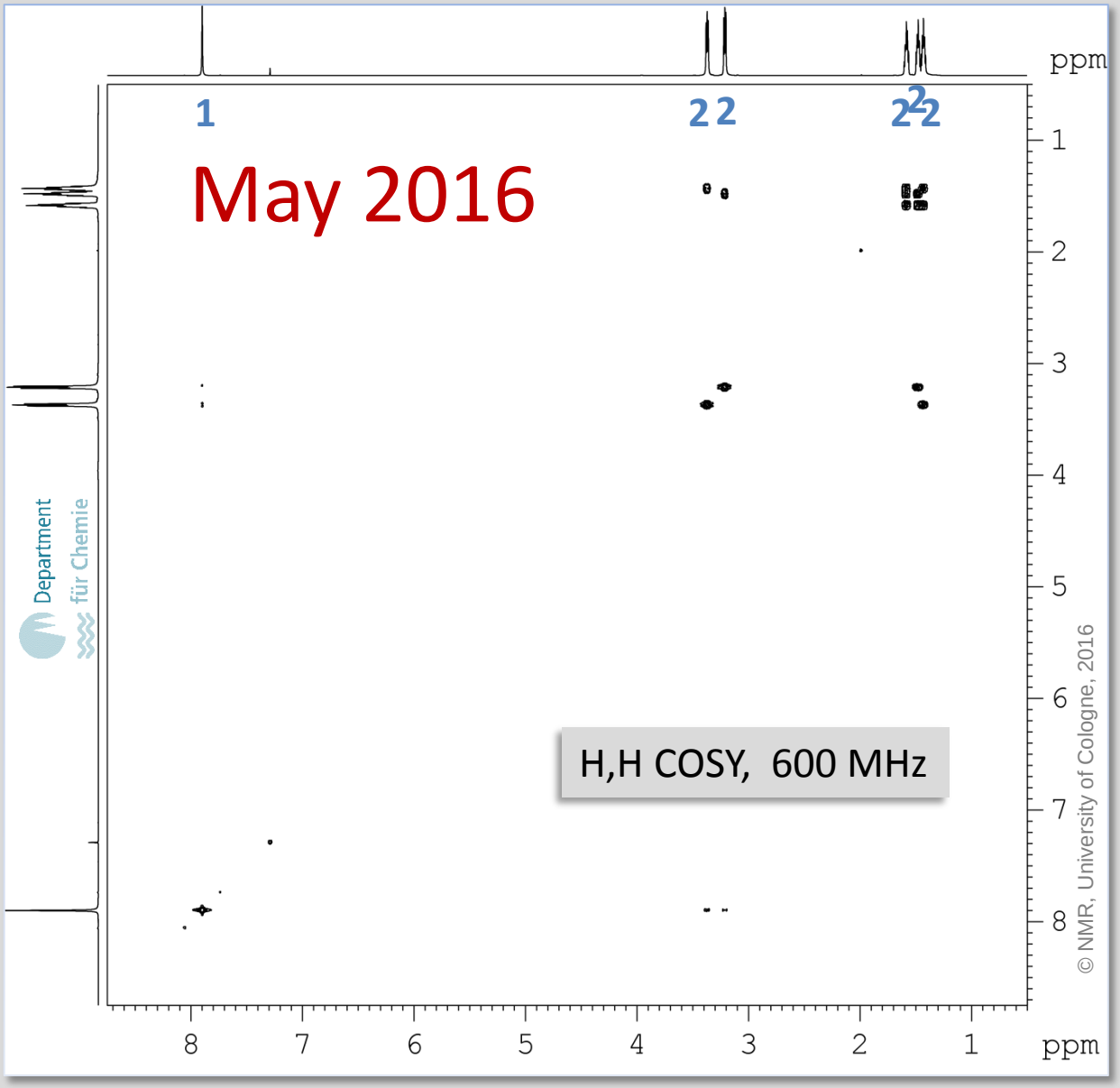
Strategy

(1) In the H,H NOESY experiment (NOE = nuclear Overhauser effect) shown on the title page, protons which are apart from each other no more than a distance of 4Å, show cross peaks. Here, through-space, as well as chemical exchange correlations can be analyzed.

(2) This experiment is phase-sensitive, i.e. cross peaks that are of the opposite phase (blue) as the diagonal peaks (red) stem from NOE. Signals, which are of the same phase as diagonal peaks (red) originate from chemical exchange.

(3) The molecular formula of this month's molecule is $C_6H_{11}NO$. What does this tell you about the number of DBE?

Problem of the Month:



Problem of the Month:
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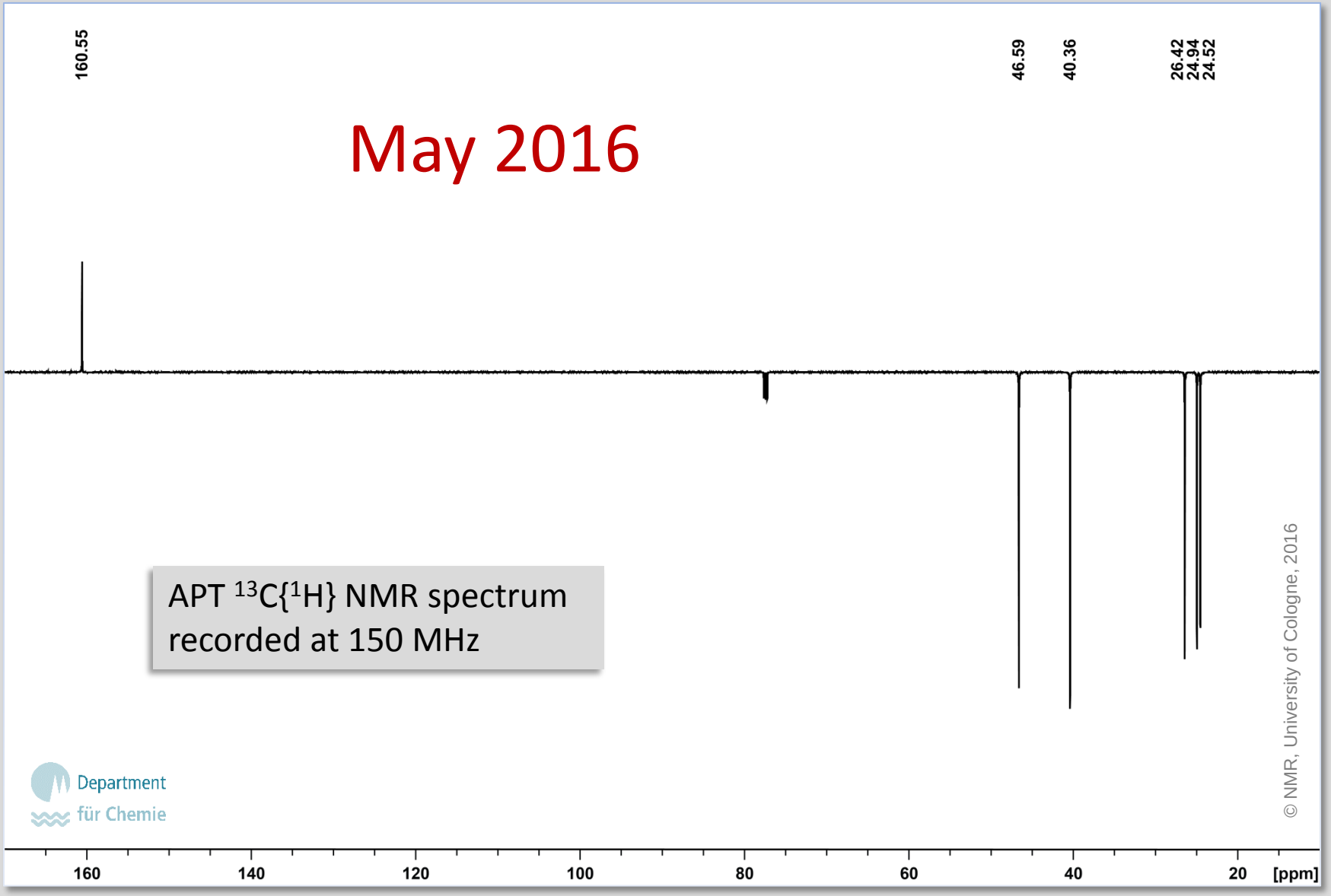
Hints

(1) On the previous page, H-H correlations through bonds can be inspected in the COSY experiment. Here, long-range couplings (> $^{3}J_{HH}$) are visible in two cases. The absolute number of protons is printed in blue. Carefully compare the cross peaks with the ones visible in the NOESY spectrum.

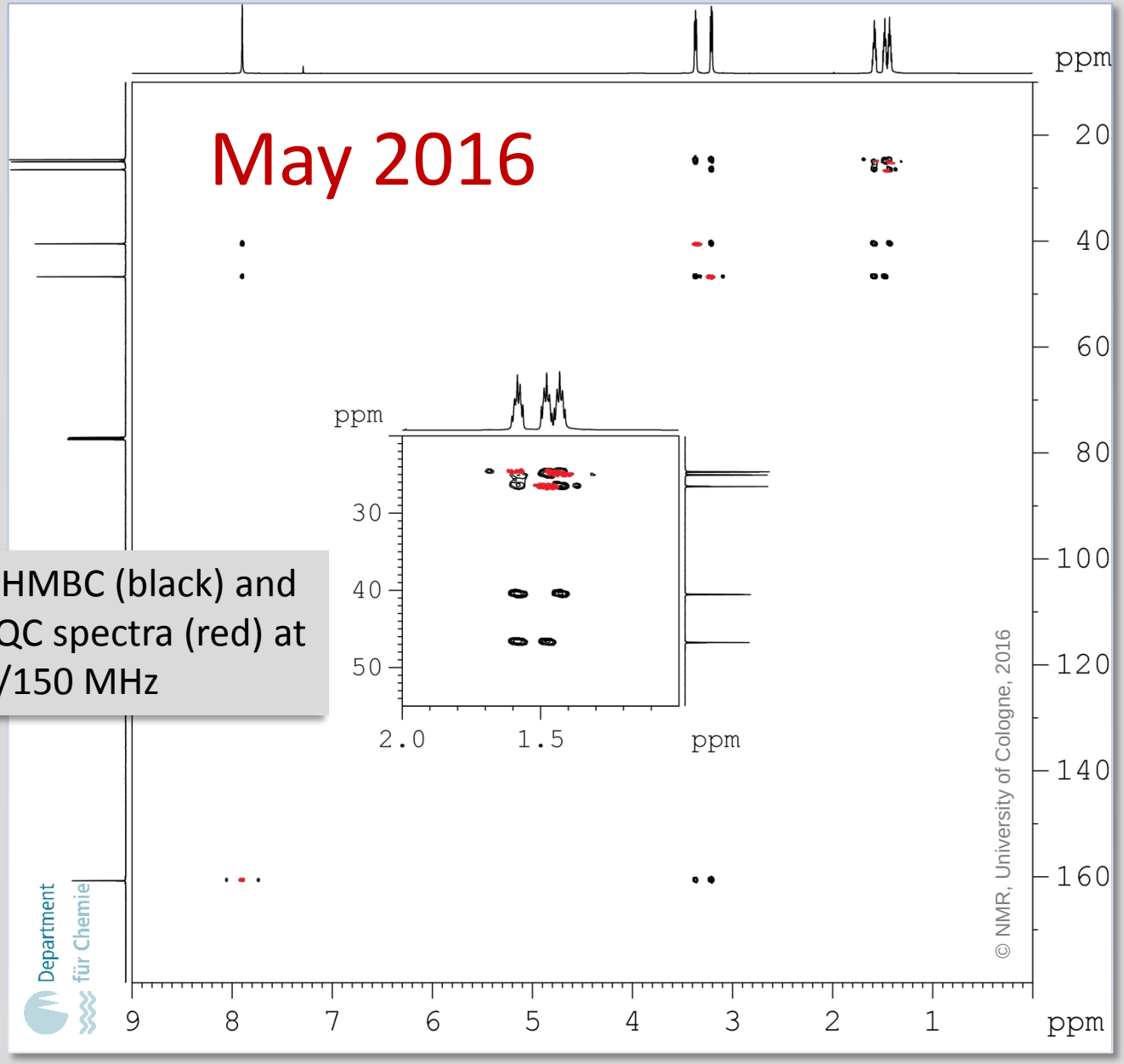
(2) Take a look at the chemical shifts in the multiplicity-edited APT spectrum (next page). Here, a C-H carbon stands out due its atypical shift. If you do a subspectrum search for a doublet (D) at this frequency, the type of carbons in question can be narrowed down. It might be interesting to search for the group of sp^3 hybridized carbon signals, as well.

(3) To elucidate the 'skeleton' of the compound, there are H-C correlation spectra on the page after the next page. $^{1}J_{CH}$ correlations from the HMQC experiment are labelled in red.

Problem of the Month:



Problem of the Month:



H,C HMBC (black) and HMQC spectra (red) at 600/150 MHz

Problem of the Month:
May 2016

Solution

(1) There are six non-equivalent carbons in the target molecule‘s structure - the same number as in the molecular formula. Also, there are - with one exception - no sp^2 hybridized carbons , which excludes the possibility of an aromatic /olefinic molecule. Calculation of DBE yields two.

(2) Connections between the CH_2 units are straight forward from the COSY spectrum: One coupled spin system consisting of 3.12-1.38-1.49-1.33-3.27 can be identified, additionally, long range correlations from 7.79 to 3.12 and 3.27 are present.

(3) From the NOESY experiment, exchange can be detected at least between 3.12/3.27. Also, there is only a NOE contact from 7.79 to 3.27, not to 3.12 - why? If you are still in doubt - enter the chemical shifts of the ^{13}C signals in nmrshiftdb2 as a „spectrum search“ (option „complete“).