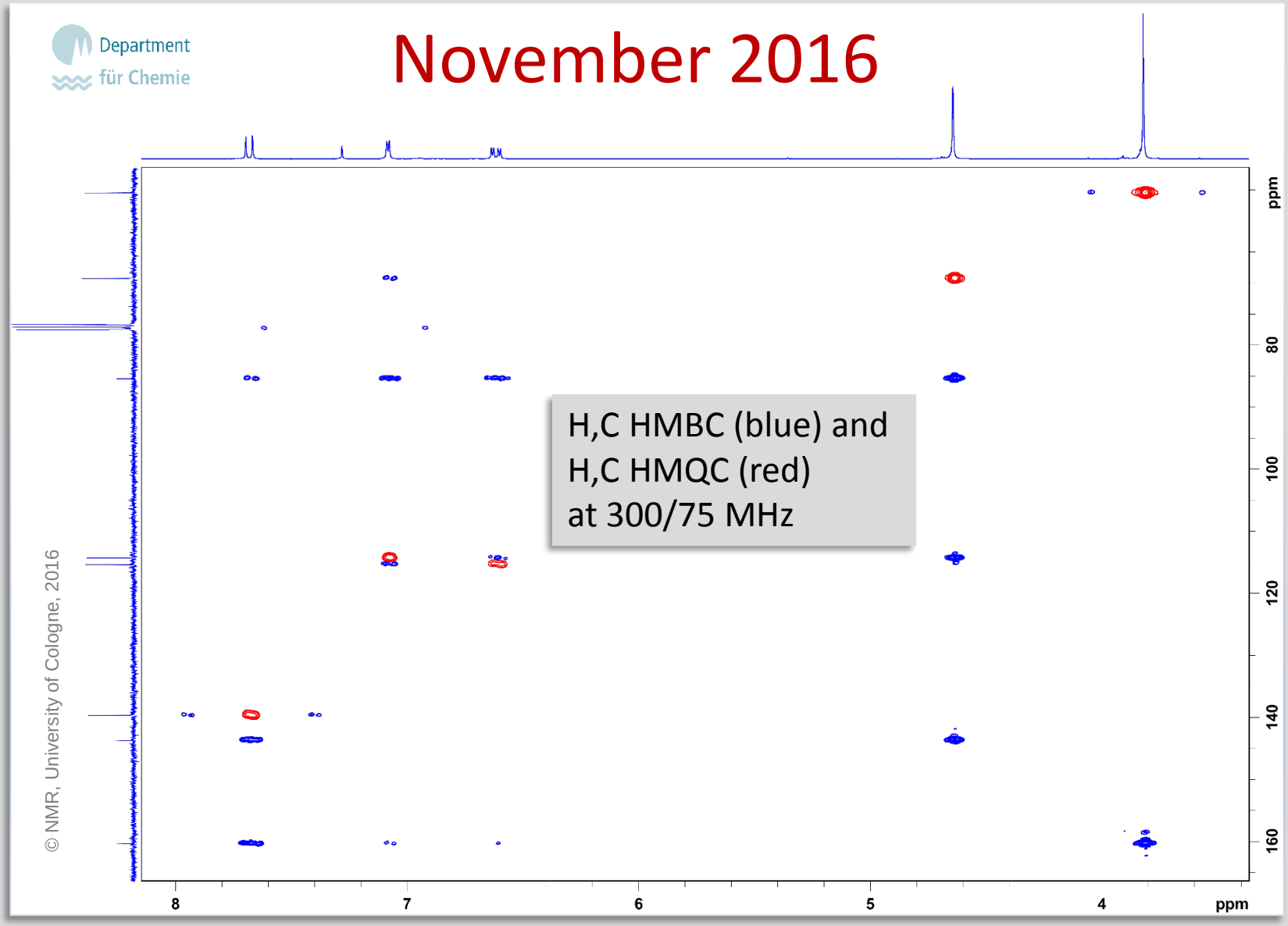
Problem of the Month:

November 2016



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Strategy

(1) The spectra for structure elucidation presented this month are a good example to show, how the QuickCheck tool might assist you. However, there is a catch in it!

(2) The experiments shown on the first page derive the C-H connectivities in the molecule: HMQC with cross peaks for $^{1}J_{CH}$ are visible in red, while HMBC peaks for long range correlations ($^{2}J_{CH}$, $^{3}J_{CH}$ and some dublets for $^{1}J_{CH}$). In aromatic systems, the intensity of $^{2}J_{CH}$ cross peaks is weaker than the one of $^{3}J_{CH}$ cross peaks.

(3) The molecular formula of this month‘s molecule is $C_8H_9IO_2$. First, derive the number of DBE (degree of unsaturation).

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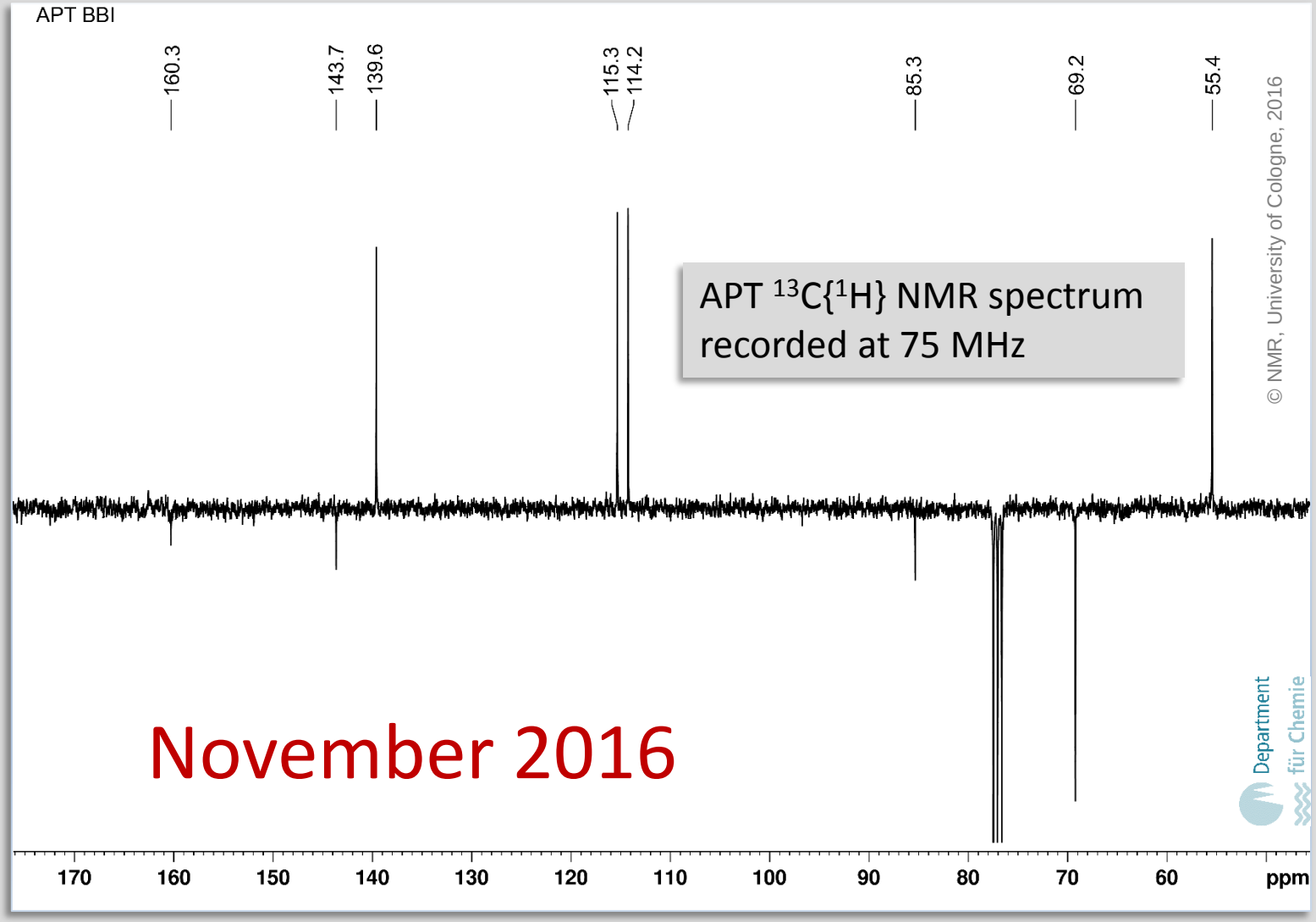

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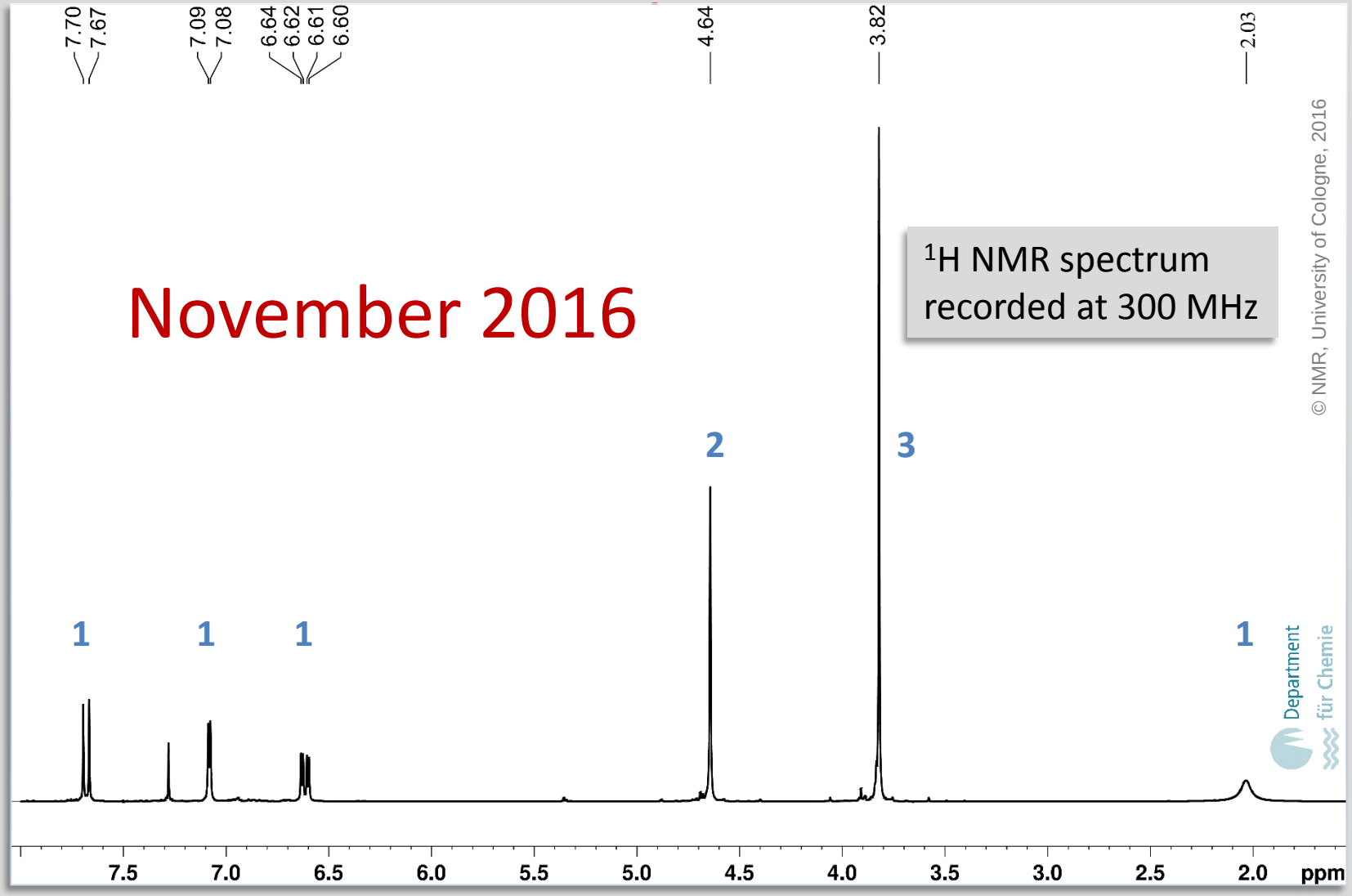

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Hints

(1) The previous two experiments allow to evaluate the compound by chemical shifts, multiplicity of carbons (APT), signal intensity (integrals ^{1}H) and by analysis of the H-H coupling patterns. The absolute number of protons is printed in blue.

(2) There is one signal the ^{1}H NMR which differs from the other ones in its broad lineshape. Also, there is no correlation in the HMQC with this proton – that is why this area is not shown on the first page.

(3) After identification of the aromatic spin system and collecting information about individual fragments, try to identify which substituent is bound to which position by carefully re-investigating the HMBC correlations.

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Solution

(1) There are only non-equivalent carbons in the target molecule‘s structure - which means that there is no higher symmetry. For the chemical shifts of proton signals in the left part, there must be aromatic moiety, which is supported by the degree of unsaturation (DBE = 4).

(2) Four spin systems exist in ^{1}H: One O-CH_3 unit (3.82), one O-CH_2 (4.64) unit, an „exchanging“ OH proton (2.03) and an aromatic AMX spin system. The latter consists of two protons that are three bonds a part (7.68 and 6.61) and a third proton (7.08) that shares long-range coupling to the one of them (6.61).

(3) The substitution pattern of the aromat can be derived through the $^{3}J_{CH}$ cross peaks. The location of iodine is easy to spot for the ^{13}C chemical shift. If you are still in doubt - enter the chemical shifts of the ^{13}C and ^{1}H signals with your suggested structure in nmrshiftdb2‘s QuickCheck. The solution can be found here.