Determination of aspirin and caffeine

ASSIGN BUSTER

Results:

Identify and mark the signals in the spectra and note the chemical shift values of the methyl resonances in aspirin and caffeine and the methylene resonance in s-trioxane.

Using above expression, calculate the weight of aspirin and caffeine in one tablet, and the percentage w/w of each component in one analgesic tablet.

Whole tablet weighed = 0. 501

Half tablet weighed = 0. 270

Mass of s-trioxane = 0. 05

RMM of aspirin = 180

RMM of caffeine = 194

RMM of s-trioxane = 90

No. of moles of components = component integral/no. of protons giving signal

No. of moles of standard standard integral/ no. of protons giving signal

To find out the weight and percentage w/w of Aspirin and Caffience following calculations were made:

Aspirin:

No of moles of components (x) = ?

No of moles of standard = mass of s-trioxane / RMM of s-trioxane

= 0. 05 / 90 = 0. 000555

Component integral = 202. 72

No of protons giving signal = 3

Standard integral = 200

No of protons giving signal = 6

Putting values in the above eq. 1:

x / 0. 000555 = (202. 72/3) / (200/6)

x/0. 000555 = 67. 57 / 33. 33

x = 67. 57 x 0. 000555 / 33. 33

x = 0. 00113 moles

Mass of aspirin = moles x RMM

= 0. 00113 x 180 = 0. 203g = 203mg

% w/w of aspirin in the tablet = mass of aspirin / mass of the tablet x 100

= 0. 203 / 0. 501 x 100

= 40. 5%

Caffeine:

No of moles of components (x) = ?

No of moles of standard = mass of s-trioxane / RMM of s-trioxane

= 0. 05 / 90 = 0. 000555

Component integral = 14. 0

No of protons giving signal = 3

Standard integral = 200

No of protons giving signal = 6

Putting values in the above eq. 1:

x / 0. 000555 = (14. 0/3) / (200/6)

x/0. 000555 = 4. 66 / 33. 33

x = 4. 66 x 0. 000555 / 33. 33

x = 0. 0000775 moles

Mass of caffeine = moles x RMM

= 0. 000075 x 194 = 0. 0145g = 14. 5 mg

% w/w of aspirin in the tablet = mass of aspirin / mass of the tablet x 100

= 0. 0145 / 0. 501 x 100

= 2. 89%

Discussion:

1. Comment on the chemical shift positions of the methyl groups in aspirin and caffeine.

2. Aspirin shows about 6 singlets in the spectrum, all in different environment. It has got one methyl group which gives rise to a singlet at ? 2. 3498 as there are no neighbours and the n+1 rule is followed. It has integral of 3 as three protons are giving rise to the chemical shift at ? 2. 3498. The four singlets between ? 7. 1292 – ? 8. 1123 correspond to benzene ring protons. In aspirin there is a very broad singlet at ? 11. 0082 due to the carbonyl next to hydroxyl proton which shifts it towards the left hand side.

Caffeine has got three methyl groups which give rise to three singlets as all the three methyl groups are in different environments to each other. All the three peaks have integral of 3 which arises due to the three protons on each methyl groups. The first singlet at ? 3. 4133 is due to the protons (a) next to nitrogen with single bond. The second singlet is seen at ? 3. 5910 corresponding to protons(c) next to double bonded carbon and oxygen and the last methyl singlet (b) at ? 4. 004 is due to the protons next to two double bonded oxygens attached to two carbons. There is also a singlet seen at ? 7. 5172 that arises due to a single proton CH between two nitrogens.

3. Compare your results to the contents claimed by the manufacturer and discuss any differences observed.
4. How does this method compare with determinations by UV absorbance and HPLC. What are the NMR method’s limitations?
5. UV techniques are simple and rapid. It can be used for the quantitative determination of highly conjugated compounds and metal ions. Metal ions can be coloured and determined by UV. HPLC is a separation techniques used for compounds on basis of their rate of elution and

can separate complex mixtures. HPLC analysis is very quick with high resolution. The stationary column can be used repeatedly for number of times. In HPLC analysis, automated instrumentation and quantitation can be used. It also has low sensitivity and accuracy. NMR is an expensive technique. Compared to UV and HPLC the instrumentation is more costly. The sample to be analyzed has to be free of any contaminants. It takes longer time as compared to the other techniques mentioned. In NMR the chemical shift corresponds to the structure of the molecule being analysed so for compounds with similar structures it is difficult to separate the signals. Also it is an insensitive technique.

References:

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