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ANALYTICAL CHALLENGE

Solution to NMR hide-and-seek challenge

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The winner of the NMR hide-and-seek challenge (published in volume 408 issue 27) is:

John Warren, LGC, Teddington, UK.

The award entitles the winner to select a Springer book of his choice up to a value of €100.

Our congratulations!

Solution

The substance described in the NMR hide-and-seek challenge [1] is histamine (Fig. 1).

In the realm of natural products, histamine is one of the simplest nitrogen-containing compounds. The colourless hygroscopic crystals melt at 83.5 °C and are easily dissolved in water. In aqueous solution histamine exists in two tautomeric forms (Fig. 2).

Histamine plays an important role in the human body: it is involved in several physiological functions as in immune responses and it acts as a neurotransmitter. The compound was synthesized first by Windaus and Vogt in 1907 via Curtius degradation of imidazole propionic acid hydrazide. They noted: "we have not yet fully investigated this peculiar substance. The investigation progresses slowly because of the difficult accessibility of the starting material. Nevertheless we report the previous results now, since others are working on the synthesis of histidine as well" [2]. Three years later Dale and Laidlaw [3] fully described many of the physiological effects of this substance.The name "histamine" was derived from "histo" and "amine", yielding "tissue amine". Occasionally in the medical literature the term "H substance" is used for histamine or a hypothetical histamine-like diffusible substance released in allergic reactions of skin and in the responses of tissue to inflammation. However, the histamine content of a single nettle hair was found to be only 6 ng [4].

For structural determination the ^{1}H–^{13}C heteronuclear multiple bond correlation spectrum is given with the complete assignment of all carbon and hydrogen atoms that are detectable in dimethyl-d_6 sulfoxide solution of histamine dihydrochloride (Fig. 3).

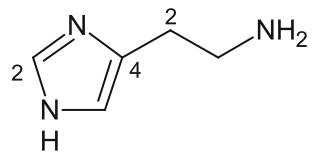

Fig. 1 Histamine ($C_5H_9N_3$, $M = 111.15$ g/mol). IUPAC name 2-(1*H*-imidazol-4-yl)ethanamine

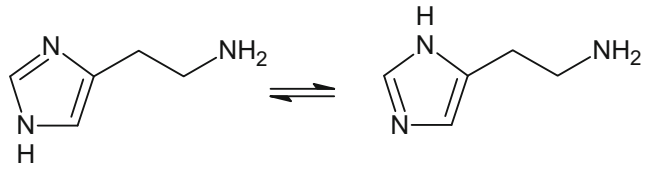

Fig. 2 The tautomeric forms of histamine

This article is the solution to the Analytical Challenge to be found at http://dx.doi.org/10.1007/s00216-016-9860-x

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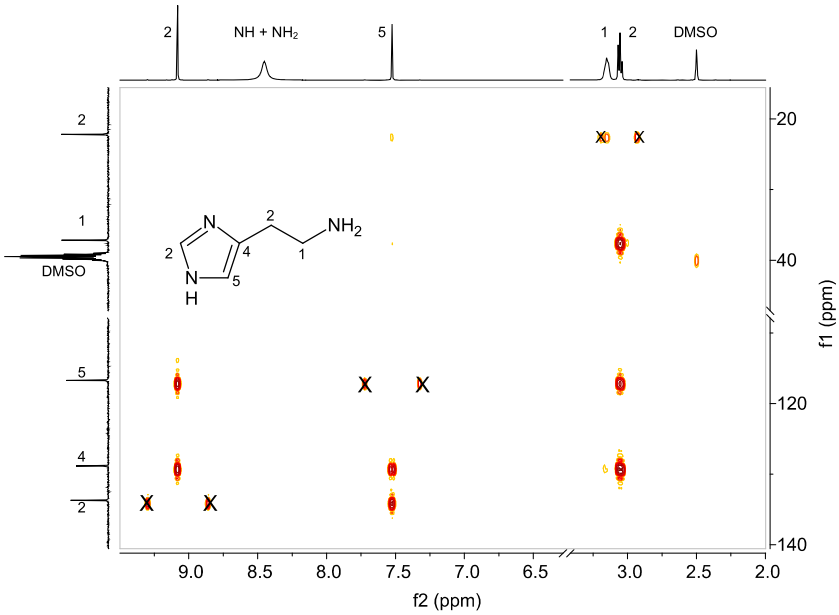

Fig. 3 Heteronuclear multiple bond correlation spectrum of histamine [500 MHz, dimethyl-d_6 sulfoxide (*DMSO*)]. The symmetrical satellite signals are residual single quantum coherence signals (so-called heteronuclear single quantum coherence artefacts) and are marked by *X*

References

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