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ANALYTICAL CHALLENGE

Wing-giving challenge

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We would like to invite you to participate in the Analytical Challenge, a series of puzzles to entertain and challenge our readers. This special feature of "Analytical and Bioanalytical Chemistry" has established itself as a truly unique quiz series, with a new scientific puzzle published every three months. Readers can access the complete collection of published problems with their solutions on the ABC homepage at http://www.springer.com/abc. *Test your knowledge and tease your wits in diverse areas of analytical and bioanalytical chemistry by viewing this collection.*

In the present challenge, chemical substances are the topic. And please note that there is a prize to be won (a Springer book of your choice up to a value of €100,- given to one winner selected randomly). Please read on…

Meet the challenge

The chemical substance we are looking to identify is widely found in animal and human tissues alike. It was first isolated by two German scientists from a bile of an animal nearly a century ago and was named after the Greek name of this animal. Some twenty years later this substance was also discovered in the human body where it accounts for up to 0.1% of total human body weight and plays an important role in several essential biological processes. Despite its importance, this substance is not an essential nutrient for humans as the body produces it itself. Nevertheless, it is found in various dietary supplements aimed towards athletes and in drinks marketed to provide mental and physical stimulation. A 2008 review found no documented reports of either positive or negative health effects associated with this substance. Nevertheless, the global market of this substance is estimated to exceed US$ 200 million in 2020 with no signs of fading away.

The challenge

Figures 1 and 2 show the high-resolution ESI–MS spectrum and the ATR-IR spectrum of the pure compound. Figure 3 shows the ^{1}H-NMR spectrum of the compound dissolved in deuterated DMSO (top). The lower spectrum was cleaned from residual proton signals of the solvent DMSO (at 2.50 ppm) and of residual water (at 3.32 ppm). Only the three peaks arising from our substance were integrated; a broad signal at 7.65 ppm and two multiplets, one at 2.72 ppm and other at 3.05 ppm. The smallest integer ratio of the three integral values is 3:2:2. The ^{13}C-NMR spectrum of the substance was also obtained and is shown as a F1 projection in the HSQC and HMBC spectra (Fig. 4). In the heteronuclear single-quantum correlation (HSQC) spectrum, the direct spin couplings ($^{1}J_{H–C}$) are shown. In HMBC spectra, couplings over two or three covalent bonds (H–C–C and/or H–C–C–C) may be detected and we observe spin couplings over two covalent bonds ($^{2}J_{H–C–C}$). The ^{15}N-HMBC spectrum is also given (Fig. 4). All two-dimensional NMR spectra are pictured in ^{1}H chemical shift range from 2 to 8 ppm and note the larger linewidth of the proton signal at 7.65 ppm and the absence of any couplings with this resonance.

Can you identify this mysterious substance? Assign both triplets observed in the ^{1}H-NMR and find at least one online spectrum that is labeled differently than your answer.

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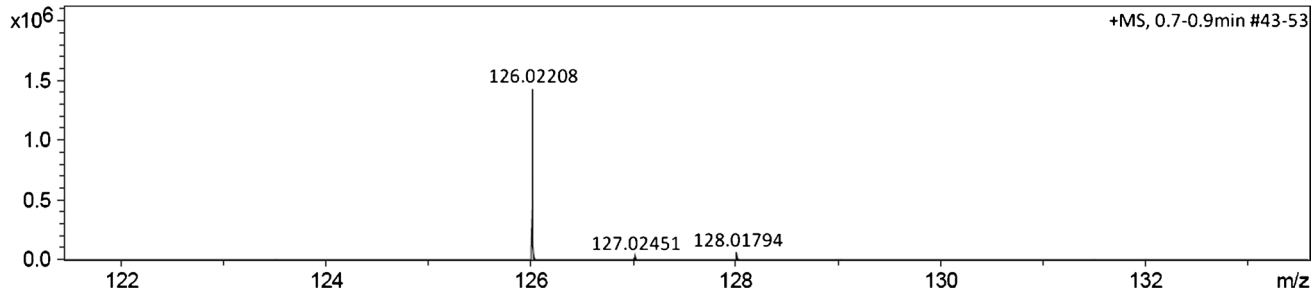

Fig. 1 The high-resolution electrospray ionization mass (ESI–MS) spectrum of the pure compound. Note that base peak observed here at 126.022 m/z is created by the addition of a hydrogen cation $[\mathbf{M}+\mathrm{H}]^+$

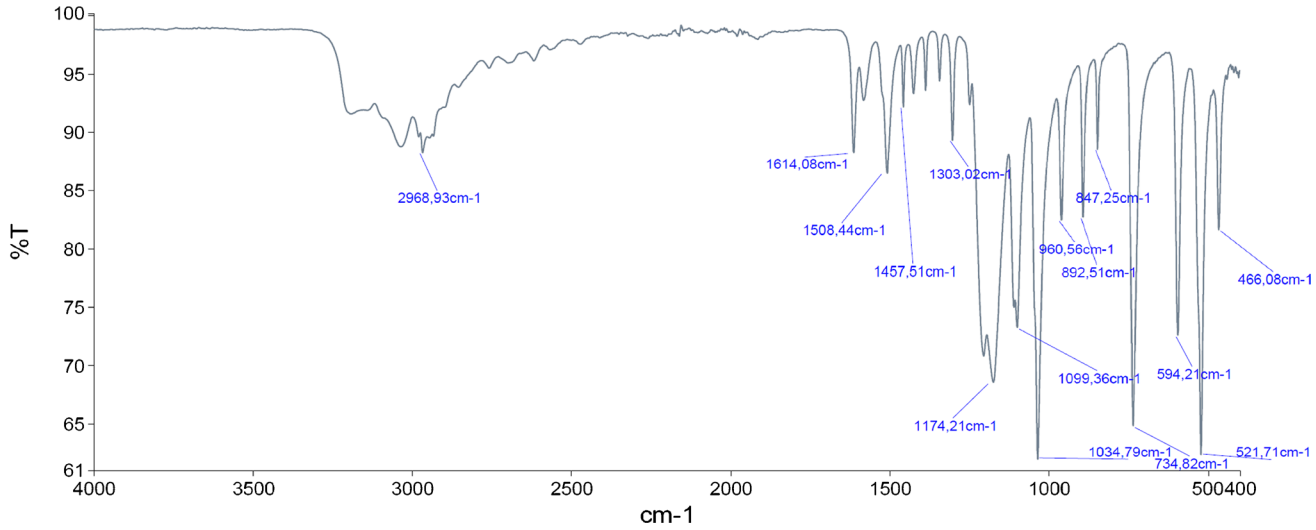

Fig. 2 The attenuated total reflection infrared (ATR-IR) spectrum of the pure solid compound

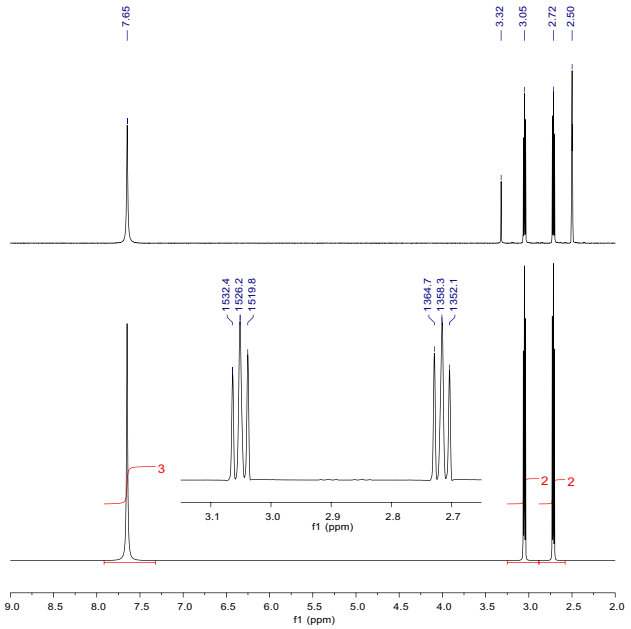

Fig. 3 500 MHz ^{1}H-NMR of the compound, measured in DMSO-D6 (top) and the same spectrum without solvent and water signals (bottom). The solvent signal at 2.5 ppm was used as reference. Only the three substance signals were integrated

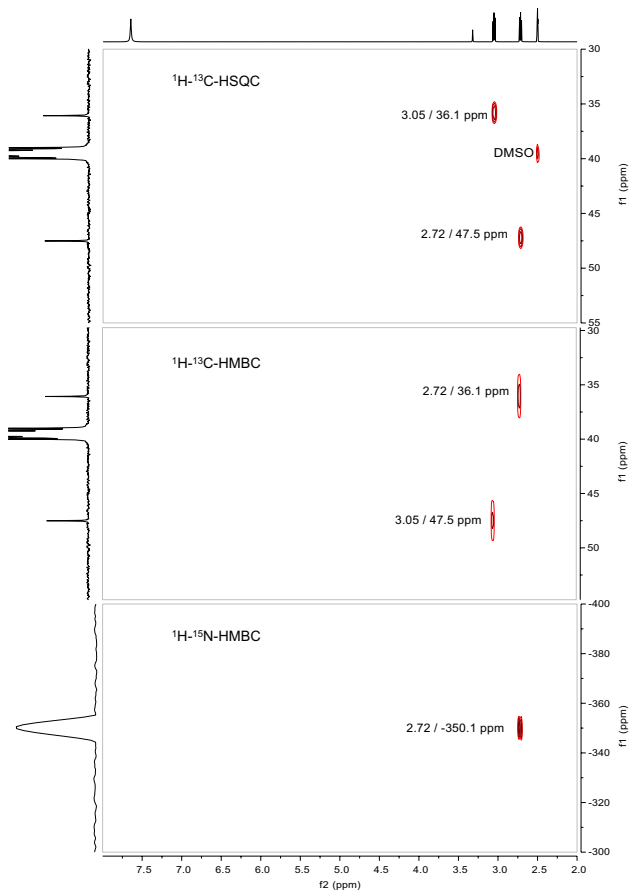

Fig. 4 Three two-dimensional shift correlated spectra. Top: the heteronuclear single-quantum correlation ^{1}H-^{13}C-HSQC spectrum; middle: the heteronuclear multiple-bond correlation ^{1}H-^{13}C-HMBC spectrum; and bottom: the ^{1}H-^{15}N-HMBC spectrum. DMSO and nitromethane were used as references for ^{13}C and ^{15}N chemical shifts, respectively. All spectra are pictured with same ^{1}H chemical shift range from 2 to 8 ppm in the horizontal dimension

We invite our readers to participate in the Analytical Challenge by solving the puzzle above. Please send the correct solution to abc@springer.com by July 1, 2023. *Make sure you enter "Wing-giving challenge" in the subject line of your e-mail. The winner will be notified by e-mail and their name will be published on the "Analytical and Bioanalytical Chemistry" homepage at* http://www.springer.com/abc *and in the journal (volume 415/issue 24) where readers will find the solution and a short explanation.*

The next Analytical Challenge will be published in 415/16, July 2023. If you have enjoyed solving this Analytical Challenge you are invited to try the previous puzzles on the ABC homepage.

Conflict of Interest The author declares no competing interests.