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ANALYTICAL CHALLENGE

Elemental analysis 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, elemental analysis is 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

Many chemistry journals require results of elemental analysis as supporting evidence for the identity of organic substances. A common journal requirement is that the data should be provided to an accuracy of ± 0.4% which presumably means that the elemental analysis results for carbon, hydrogen, and nitrogen should agree with the postulated structure to within 0.4%.

Discussions about elemental analysis measurements could benefit from additional metrological clarity about the quantity in question and the nature of the bias. First, we are talking about the mass fraction of the elements in the analyzed substances. Second, we are talking about the absolute deviation of the mass fraction results expressed in cg/g, not the relative deviation. Third, it remains unclear as to what level of confidence should be attached to this value.

In any case, the results of elemental analysis have been critically evaluated on many occasions. Recently, Kandioller et al. [1] conclude that "more and more often the elemental analysis values in submitted manuscripts or already accepted publications are doubtful." To this end, Kuveke et al. sought to assess the variability of the elemental analysis results for C, H, and N in a large-scale interlaboratory study involving academic institutions and commercial services, with each participant analyzing the same five substances [2, 3]. This study showed that the variability of carbon and nitrogen results exceeded the recommended threshold of 0.4% by a factor of two. Clearly, the measurement science needs to improve in order to provide elemental analysis results that meet the requirements of many chemistry journals. However, the subject of this challenge is not the elemental analysis measurement, but rather the ± 0.4% journal requirement itself [4].

The challenge

Consider one of the compounds used in the interlaboratory study by Kuveke et al. [2], DL-tryptophan ($C_{11}H_{12}N_2O_2$). Since this is a computational exercise, let us enjoy a perfect trifecta of circumstances. First, we have a sample of pure DL-tryptophan at our disposal without any impurities or co-crystalized water. Second, reliable elemental composition measurements of C, H, and N are performed with associated measurement uncertainties that meet journal requirements. Third, we know that our substance contains only C, N, H, and O, and we also know that it has two nitrogen atoms in each molecule.

Thus, the average measurement results for these three elements agree with the expected (theoretical) values: the measured mass fraction of carbon is $w_C = 64.69$ cg/g (commonly written as 64.69%) with the expanded uncertainty $U(w_C) = 0.40$ cg/g (commonly written as 0.4%) at 95% confidence. Similarly, the measured mass fraction of nitrogen is $w_N = 13.72$ cg/g with the expanded uncertainty $U(w_N) = 0.40$ cg/g, and the measured mass fraction of hydrogen is $w_H = 5.92$ cg/g with the expanded uncertainty $U(w_O) = 0.40$ cg/g. We shall interpret the aforementioned measurement uncertainties of C, N, and H using the Gaussian distribution. For example, the results of carbon can be

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modeled using a Gaussian distribution with the mean 64.69 cg/g and standard deviation (0.40/2) cg/g = 0.20 cg/g.

The measurements described here represent a utopian best-case scenario so they will serve to evaluate the journal requirements themselves!

What is the probability of obtaining the correct molecular formula of tryptophan from these measurement results?

Declarations

Conflict of interest The author declares no competing interests.

References

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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 April 1, 2024. Make sure you enter "Elemental analysis 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 416/issue 16) where readers will find the solution and a short explanation.*

The next Analytical Challenge will be published in 416/9, April 2024. If you have enjoyed solving this Analytical Challenge you are invited to try the previous puzzles on the ABC homepage.