

Online Resource 1. Examples of mistakes and inconsistencies

Article title: Comments on “Boron and lithium isotopic signatures of nanometer-sized smectite-rich mixed-layers of bentonite beds from Campos Basin (Brazil)” by N. Clauer, L. B. Williams, and I. T. Uysal

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Supplementary information (Online Resource 1) for “Comments on ‘Boron and lithium isotopic signatures of nanometer-sized smectite-rich mixed-layers of bentonite beds from Campos Basin (Brazil)’ by N. Clauer, L. B. Williams, and I. T. Uysal,” by J. M. Wampler

Examples of Mistakes and Inconsistencies

Many statements by Clauer et al. (2022) are inconsistent, at least in part, with their tabulated analytical results. Here, examples of such statements are quoted, and each quotation is followed by an explanation of what is wrong. The last three examples are from the Conclusions section, from which propagation of misinformation is more likely than from the main text of the article.

“Traces of illite were also identified in the fractions, especially in the two coarser A1 ones, ...” (p. 75)

To call the amount of illite in the coarsest fraction of sample A1 a trace is inconsistent with the data of Table 1, where the amount of discrete illite is represented by xx. There is no established convention for what x, xx, etc. mean as estimates of mineral abundance based on X-ray powder diffractometry, so when these symbols are used it is usual for them to be defined. Clauer et al. (2020), who presented the diffraction patterns on which the data of Table 1 are based, used Tr, X, and Xx without definition to represent their estimates of the abundance of discrete illite in the A1 size fractions. Inspection of their diffraction patterns and K₂O-content values for the A1 subfractions shows that they did not use Xx to stand for just a trace of discrete illite in the 50–100 nm fraction.

“Its [sample B1’s] $\delta^7\text{Li}$ remained constant within analytical error at $11.6\pm 3.4\text{‰}$ with contents increasing from 87 to 104 $\mu\text{g/g}$ in the two coarser size fractions (Table 2; Fig. 3).” (p. 75)

The average value for $\delta^7\text{Li}$ in the three B1 subfractions is $11.4\pm 2.4\text{‰}$, not $11.6\pm 3.4\text{‰}$. [The uncertainty value, 2.4‰ is twice the standard error of the mean using the sample standard deviation of the individual $\delta^7\text{Li}$ values (each of which was the average $\delta^7\text{Li}$ from three analyses).] The value $11.6\pm 3.4\text{‰}$ is an individual value for the 50–100 nm fraction, where the uncertainty is twice the standard error of the mean using the sample standard deviation of the three Li isotopic analyses of that subfraction.

“The $\delta^{11}\text{B}$ and B contents of sample A1 are correlated with the K contents ...” (p. 75)

For the sample A1 size fractions, B content is positively correlated with K content, but $\delta^{11}\text{B}$ is not.

“The smectite-rich A1 fractions, for which the K content is very low and narrowly spread, ...” (p. 80)

It is the B1 fractions, not the A1 fractions, for which the K content was very low and narrowly spread.

“In addition, the $\delta^{11}\text{B}$ and B contents of that [the B1] coarser fraction are closer to those of the intermediate size fraction than to those of the finest fraction. They are also close to those of sample A1, ...” (p. 81)

The B content of the coarsest B1 fraction is close to that of the two coarser A1 fractions, but its $\delta^{11}\text{B}$ value is not close to that of any A1 fraction.

“In this context, the $\delta^7\text{Li}$ values that are quite distinct from each other, may relate to the overwhelming occurrence of smectite, as are the $\delta^7\text{Li}$ values relative to the K contents.” (p. 81)

No subset of the six studied size fractions had $\delta^7\text{Li}$ values that are quite distinct from each other. The $\delta^7\text{Li}$ values of the six size fractions are all the same within the 2σ analytical uncertainty, provided that such uncertainty is based on the overall precision shown in Table 2, not on the individual 2σ values shown in the table. Despite a statement on p. 74 that “... each aliquot was analyzed three times, or more, with an overall analytical precision of $\pm 2\sigma$ (Table 1),” the 2σ values in Table 2 [not Table 1] for the average $\delta^7\text{Li}$ [not $\delta^{14}\text{Li}$] of each size fraction are not values of the overall precision for $\delta^7\text{Li}$ —they are values for twice the standard error of the mean based on the individual values of sample standard deviation, two of which are fortuitously small. The average of the individual 2σ values for $\delta^7\text{Li}$ is $\pm 2.2\text{‰}$ (an overall value typical of the variability of the results obtained from three analyses of each size fraction). If the uncertainty of each measurement is taken as $\pm 2.2\text{‰}$, the six $\delta^7\text{Li}$ values do not vary outside the analytical uncertainty.

“For Li, the finest and the coarsest B1 fractions yield heavier $\delta^7\text{Li}$ values than the three separates of sample A1 with only a lower Li content for the finest fraction.” (p. 81)

The $\delta^7\text{Li}$ value of the coarsest B1 fraction does not differ beyond analytical uncertainty from those of the three separates of sample A1, if analytical uncertainty is considered to be given by the 2σ values of Table 2. The 2σ value for $\delta^7\text{Li}$ of the finest B1 fraction is fortuitously small, so its Li would appear to be heavier than that of the A1 fractions unless one recognizes that when $n=3$ a sample standard deviation is commonly far from the population standard deviation. In such a case, estimates of analytical uncertainty should be based on typical values of sample standard deviation, not on individual values. Under that approach, the $\delta^7\text{Li}$ value of the finest B1 fraction also does not differ beyond analytical uncertainty from those of the three separates of sample A1.

“The $\delta^{11}\text{B}$, $\delta^7\text{Li}$, and B and Li contents of the three size fractions from the two studied samples are quite varied.” (pp. 81-82)

The $\delta^7\text{Li}$ values of the six size fractions studied are not quite varied. As noted above, they are all the same within the 2σ analytical uncertainty, provided that such uncertainty is based on the overall precision shown in Table 2, not on the individual 2σ values shown in the table.

“In the case of sample A1 ..., the $\delta^{11}\text{B}$ is constant relative to the size fractions, while its $\delta^7\text{Li}$ is widely spread, as are the contents of both B and Li.” (p. 82)

The $\delta^7\text{Li}$ values for sample A1 are not widely spread; they are the same within analytical uncertainty.

“Conversely, the repartitions in the coarser size separate of the second B1 sample are different than in the two finer fractions that yield similar B and Li contents, ...” (p. 82)

The two coarser size fractions of the B1 sample were closely similar in B and Li contents, The two finer size fractions were not similar in B content and not closely similar in Li content.

REFERENCES

- Clauer, N., Środoń, J., Aubert, A., Uysal, I. T., & Toulkeridis, T. (2020). K-Ar and Rb-Sr dating of nanometer-sized smectite-rich mixed layers from bentonite beds of the Campos Basin (Rio de Janeiro State, Brazil). *Clays and Clay Minerals*, 68, 446–464.
- Clauer, N., Williams, L. B., & Uysal, I. T. (2022). Boron and lithium isotopic signatures of nanometer-sized smectite-rich mixed-layers of bentonite beds from Campos Basin (Brazil). *Clays and Clay Minerals*, 70, 72–83.