

Interactive comment on “Measurement of geologic nitrogen using mass spectrometry, colourimetry, and a newly adapted fluorometry technique” by Benjamin W. Johnson et al.

Anonymous Referee #2

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This paper compares several techniques to quantify ppm-level of nitrogen trapped in geological samples. Authors are particularly interested in crustal rocks, where a large portion of nitrogen is believed to be trapped in the ammonium form. As authors point out, understanding the behaviors of these type of nitrogen could lead to answering to the question how nitrogen cycled within the surface region of Earth that consist of atmosphere, biosphere and crusts, and also between the surface region and the mantle.

I agree with the most general authors' point that nitrogen study in various crustal rocks bears substantial scientific significance. However, I am not always convinced with the authors' strategy obtaining large numbers of plain concentration data for the ammo-

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mium form nitrogen in crustal rocks, aiming at a novel understanding regarding the nitrogen behavior in the crusts. Nitrogen quantification in rocks itself has been done since several decades ago by several techniques. What kind of a new finding they expect by now? The example they introduce in 4.4 essentially says that they can roughly confirm the estimation of nitrogen budget in crustal rocks, which was already done several decades ago.

Many of the important references are missing in this paper. I don't request authors to do a thorough review of the previous techniques, but they should at least describe in the paper what is the standard techniques used for nitrogen study in rock samples. One of the technique is mass-spectrometry. Indeed, authors introduce one example of mass-spectrometry. However, the technique they refer to is not the one normally used for rock studies. Putting samples in a Tin-capsule and heating it to 1000oC is a technique to be applied to biological (easier-to-combust and with large N concentration) samples. See numbers of papers, for instance, by S. R. Boyd, S. E. Bebout, D. Haendel or D. L. Pinti, on ammonium form nitrogen trapped in sedimentary or crustal rocks. They all care for the nitrogen extraction problem or contamination issue, and present reasonable solutions. I understand well that authors would like to sell the fluorometry technique, but the comparison with other techniques must be done in a fair manner. The other technique I realized missing in this paper is the Kjeldahl method. This is a well-established chemical technique to extract and quantify ammonium nitrogen, therefore, must be directly compared with the two chemical techniques introduced in the paper. For example, Honma and Itihara, GCA (1981) measured numbers of crustal rocks by this method. Essentially what is the new selling point in the fluorometry technique? Isn't it just a variation of the classical Kjeldahl method?

In summary, I consider that references to other techniques are absolutely missing or poorly described in this paper, which prevents readers from reasonably understanding the pros and cons of the techniques.

Misc: 1. Briefly summarize the principle of techniques in the introduction, rather than

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to just say read this paper. 2. Table 3. I don't understand why the concentration of ammonium "in the sample" change after the distillation process. Distillation means just vaporizing water, isn't it? Why does the differences of concentration before and after the distillation differ between samples (e.g., among BCR-1/2, BHVO-2 and G-2). I am a bit worried to find that the post-distillation concentrations are curiously similar among these three.

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