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

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Referee Comment of: Measurement of geologic nitrogen using mass spectrometry, colourimetry, and a newly adapted fluorometry technique, by Johnson and Coauthors.

In the manuscript the authors measured a number of geochemical rock standards using 3 different techniques: mass spectrometry, colourimetry, and fluorometry. The fluorometry approach is a novel adaptation of a technique commonly used in biologic science, and is applied in the paper to assess geologic NH4+. This work is important and informative for the geochemical community that is broadly interested in volatile cycling and N speciation in the Earth. The results presented here represent a valuable first step towards producing an internationally recognized standard for N in solid materials.

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The discussion of the different techniques is also quite valuable, and the authors have thought outside of the box on how to make N measurements more efficiently. However, more emphasis should be placed on the utility of obtaining N isotopes, which is only possible using the mass spec technique. The results of the study are not fully satisfying (some lack of agreement), however they represent an important step forward and should be published in Solid Earth.

Comments:

Abstract: I agree that measuring NH4 is extremely important, especially for understanding the N composition of Earth's crust. The fluorometry technique sounds promising, but as the paper reveals has it's shortcomings as well (i.e., it's still a work in progress). Furthermore, the development of N standards for rocks is much welcomed and it would be good to determine the N isotopic ratio of such standards in many of the labs worldwide (i.e., Tokyo, Nancy, Scripps).

Introduction: The authors are correct to point out the difficulty of measuring N in rocks and that very few labs do this, thus I agree that a new and cheaper technique would greatly benefit the community at large. But it needs to be highlighted that these alternative techniques (colourimetry and fluorometry) are also time consuming – requiring chemical digestions and irradiation. They also cannot be used to determine N isotopes, which in my opinion is a major shortcoming.

Line by line comments:

Pg 2 l 23 – does it also work for other species of N? What exactly is mean by 'straightforward'? Has this technique been compared with standard techniques to show that there is no fractionation?

Pg 4 l 83 – is this a step that the authors are taking (i.e., distributing the samples to be analyzed isotopically by the larger community in order to test the reproducibility of these standards)?

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Pg 5 l 40 – you describe the distillation process in detail, but how much time is needed to complete this work? It seems like it will take a large amount of time (days to weeks) in which case it becomes more difficult to argue that this technique is more efficient than standard techniques.

L 58 – should this reference be in brackets?

Pg. 6 table 2 – why do the uncertainties on the isotopic values vary so much? From 0.1 to 1.3?

Table 4 – explicitly state that these are in units of ppm

Pg 7 figure 1 – can you explain exactly what absorbance means?

Pg 8 l 14 – but importantly the mass spec work will also provide invaluable isotopic information

Pg 10 figure 4 – how does this correction work over 0.35 KOH %?

Table 5 – can this table be combined with table 3? Seems to be much of the same info. . .

Pg 13 l 4 – a more detailed description of the rocks would be welcomed

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