

## Referee Report

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### General comments:

This is the second round of revisions. In the first round, the reviewers suggested several additional tests and requested more detailed information regarding the samples. However, these points have not been fully addressed in the revised manuscript. I understand that the authors are following the Associate Editor's suggestion to list the additional tests as future development. However, I strongly encourage the authors to conduct some of these tests prior to publication. Specifically, acquiring ages for washed goethite grains, heating grains to higher temperatures to ensure complete degassing, and measuring U and Th on unheated samples should be performed. Additionally, SEM images of the different types of goethite grains should be included in the supplementary information.

If the Associate Editor considers it sufficient to list the additional tests as future work, please disregard my request and proceed with publishing the manuscript following the implementation of minor revisions.

### Specific comments:

Line 16: Replace "Fe- and Mn-oxide mineralization rocks" with "Fe- and Mn-oxide cemented rocks"

Line 20: My concern is that the authors generalize their observations from the fine-grained, mixed-phase, goethite-rich material to well-crystallized, massive goethite samples, which are commonly targeted in geochronology studies. Adsorbed U will be much less prominent on well-crystallized, massive goethite samples.

Line 20: Does "*multi-mineral grains*" mean "impure goethite grains"?

Lines 31-33: Farley et al (2024) show that goethite breaks down during vacuum step-heating experiments, precluding calculation of Helium diffusion parameters.

Line 59: Replace "fragments of Fe- and Mn-oxide mineralization" with "fragments of Fe- and Mn-oxide mineralized rocks"

Figure 2: Identify all peaks. Also, replace "Iron Oxide Hydroxides" labels with "Goethite", "Feroxyhyte", or "Ferrihydrite".

Lines 106-107: As far as can tell, there is no information on "standard material" in Flowers et al. (2022). The sample analyzed by Yakubovich et al. does not represent a standard material used in (U-Th)/He geochronology.

Lines 114, 117: Clarify the phrase "without extra cooling" – what does it mean?

Line 190: Replace “thus the quantification of the number of floating particles in the solution” with “thus determining the concentration of Mn and Fe in the solutions was not possible.”

Line 191: From Table 3 - dark grain-1 (Th/U: 0.26, 0.06, and 0.32) and dark grain-2 (Th/U: 0.6, 1.3); vein grain-1 (Th/U: 1.4, 0.6, and 2.5) and vein grain-2 (Th/U: 1.7, 3.0). Interestingly, the observed Th/U ratios for grains that were not cleaned and those that were fall within similar ranges – dated yellow goethite grains Th/U: 2.4, 2.7 and yellow goethite grains used in the leaching experiment Th/U: 2.5, 3.0; dated dark grains Th/U: 0.3-0.8 and dark grains used in the leaching experiments Th/U: 0.3-1.3.

Line 287: “*The He loss from goethite is strongly controlled by radiation damage.*” – What is the basis for this statement?

Lines 338-339: Delete the phrase: “*Alternatively, the small number of dated samples and distribution of samples may preclude being able to detect continuous Neogene mineralization throughout the region.*”

Line 375-379: Reference appears in duplicate. Please correct it.