

Response letter – Referee #2 (Philippe Claeys)

Comments of reviewers in black, author responses in blue

This manuscript addresses the challenge of uncertainty in U-Th dating due to high, complex initial thorium contamination in stalagmites. By integrating isochron analysis, stratigraphic constraints, and annual layer counting to determine the initial $^{230}\text{Th}/^{232}\text{Th}$ ratio successfully, and by establishing multiple age models to construct robust chronologies over the past 2.7 kyr in the Áaktun Kóopo cave in Yucatan, it provides a crucial chronological foundation for studying hydrological and environmental changes during the Mayan social upheaval. The study indicates that assuming a fixed initial thorium concentration may lead to significant dating biases, with important methodological implications for high-precision stalagmite chronology research.

Dear Philippe Claeys, we thank for the time to review this manuscript, and the generally positive feedback.

Based on the titles and content, this manuscript is a methodological study to constrain initial thorium concentrations through several approaches and to determine a robust speleothem Age-depth model, which is then used to reconstruct hydrological change in the Yucatán Peninsula, Mexico. The emphasis should be placed more strongly on the process of method development, with the hydroclimatic reconstruction serving merely as an illustrative application. Currently, the organization/content of the abstract appears a bit unbalanced, with a relatively greater focus on presenting the paleoclimate results compared to the methodology used.

We thank for this suggestions, and we agree with the reviewer. We will revise the abstract accordingly to put the emphasis more on the methodology:

“Speleothems, such as stalagmites and flowstones, are invaluable archives of past environmental and climatic conditions due to their layered growth and suitability for precise $^{230}\text{Th}/\text{U}$ dating. In tropical karst settings, however, elevated and variable detrital thorium contamination can severely compromise age accuracy, especially for young speleothems. Here, a suite of stalagmites from Áaktun Kóopo’ Cave from the Yucatán Peninsula is used as a case study to develop and evaluate a multi method framework for constraining initial thorium and constructing robust age–depth models. We combine extensive $^{230}\text{Th}/\text{U}$ dating with local isochron analysis, stratigraphic approaches, and annual Sr/Ca layer counting to constrain elevated and initial ($^{230}\text{Th}/^{232}\text{Th}$) activity ratios and resolve pronounced chronological inversions. High uranium concentrations in the speleothems (average c. 1ppm) allow precise measurements, yet we infer a high and largely unsystematic variability of high initial ($^{230}\text{Th}/^{232}\text{Th}$) activity ratios in space and time, with values spanning between 4 and 68, thus far exceeding standard bulk Earth values. This variability demonstrates that elevated and temporally variable initial Th may be common in tropical karst settings and that multiple, independent constraints on initial ($^{230}\text{Th}/^{232}\text{Th}$) activity ratios are essential for reliable chronologies in such environments. Applying this framework to Áaktun Kóopo’ cave stalagmites yields internally consistent stalagmite age–depth models spanning the past 2.7 kyr, as well as evidence for earlier glacial and interglacial growth phases. These chronologies provide the basis for high resolution, multi proxy reconstructions of hydroclimate and vegetation change in the northeastern Yucatán Peninsula during the entire era of Maya cultural evolution, and they illustrate how robust speleothem chronologies can be obtained even in cave systems affected by elevated and highly variable initial thorium.”

Furthermore, although the introduction highlights the importance of constraining initial thorium for U-Th dating through a progressive line of reasoning, it should more clearly address the specific challenges posed by initial Th uncertainty in U-Th dating, as well as the limitations of the existing approaches to correct for initial thorium.

Thank you, we will revise the introduction accordingly. For example, we will adjust L73ff to:

“These observations thus demonstrate a vast range of initial Th variability, also within single cave sites. However, large (and poorly constrained) variations in R02i directly translate into additional, often dominant, age uncertainties, particularly for young and/or strongly contaminated speleothems. Classical correction schemes based on a single bulk Earth detrital ratio can therefore produce stratigraphic age inversions and biased chronologies when applied in such settings.”

In addition, we will change L87ff to:

“While monitoring detrital activity ratios in ZAC and drip waters provides useful information about initial activity ratios, it offers little insight into whether and how these ratios change over time. Overall, most studies still rely on only one of these methods, and the resulting uncertainty in initial Th variability is often poorly quantified. This highlights the need for more systematic assessments of initial Th when correcting 230Th/U ages from such environments.”

2. References about the Isochron method should be added (line 78). This is important.

We will add relevant references, e.g., Kaufman (1993); Richards and Dorale (2003); Frank et al. (2002); Ludwig and Titterton (1994).

3. In the introduction or study site part, there should be more details about the climatic, geological, and regional hydrological conditions of the study area. The initial ($^{230}\text{Th}/^{232}\text{Th}$) activity ratio of detrital material can vary with the characteristics of the host rock, soil, and chemical complexing agents during the aqueous transport of Th. Thus, it is better to give a clear description of the climatic, geological, and regional hydrological background.

We will add the additional information.

For example, we will add the climatological information at the beginning of section 2.1:

“This region experiences a warm, humid tropical climate with highly seasonal rainfall (wet season June–November), high year round humidity and frequent tropical storms and hurricanes (Romero and Alfaro, 2024; Medina-Elizalde et al., 2016a) and the soils are typically thin and variable, with pockets rarely reaching 0.5 m between areas of exposed bedrock (Lases-Hernandez et al., 2019).” (L115ff)

In addition, we will specify the geological setting as:

“Áaktun Kóopo’ Cave is developed within the low relief Yucatán carbonate platform, which consists of thick Cretaceous and Tertiary shallow marine limestones, forming a great limestone plain with a shallow water table and almost no surface rivers (Ward et al., 1995; Perry et al., 2009; Smart et al., 2006).” (L120ff)

4. The relative height or elevation information of the cave samples should be discussed because flood events can disrupt the stability of cave environments through clay input and sedimentary discontinuities.

We will add an according statement in section 4.2.2:

“One prominent flooding layer (~ 1 cm thick) appears in stalagmites E0-C, E1, and E23-3, which were all collected from the same cave chamber at the same elevation. This layer likely represents [...]”.

As well as

“Thinner layers (0.5–1 mm) in stalagmites E1 and E8, which also stems from the same area in the cave, correspond to a shorter interruption [...]”.

5. The clarity and quality of Figure 1 both need to be significantly improved. Also, Figure 1b seems unnecessary, since this information is already shown in Figure 1a. It is recommended to provide more detailed information on the cave environment itself, and, in particular, the places where the walls were modified. see P. 5

We will revise Figure 1, and remove Fig. 1b. In addition, we will highlight the information about the location of Mayan walls in the caption.

6. Methodology: Since this manuscript addresses the challenges of the initial Th, it is recommended to add more details about the U and Th extraction processes used in this study. see P. 8 205-215.

We will add more details about the protocol and extraction process in the according section, e.g.,

“For the analysis, small sample plates weighing approximately 100 mg were cut perpendicular to their growth direction using a diamond wire saw and subsequently sample leaching in weak acid, weighing, dissolution in ultra-clean nitric acid and spiking with our in-house triple-spike solution (‘TriSpike’, ^{233}U , ^{236}U and ^{229}Th) (Kerber et al., 2023). For drip water analysis, about 15 to 30ml of sample was weighed and spiked with ‘TriSpike’, acidified with ultra-clean nitric acid, subsequently dried down and re-dissolved. Overall, the preparation involving sample leaching, dissolution, spiking, and wet column extraction chromatography using Eichrom UTEVA resin, followed established protocols (Wefing et al., 2017; Douville et al., 2010; Matos et al., 2015). Chemical blanks are regularly monitored and commonly below 0.4 fg for ^{234}U and 0.04 fg for ^{230}Th (Kerber et al., 2023; Kerber et al., 2025). Chemical blanks are regularly monitored and commonly below 0.4 fg for ^{234}U and 0.04 fg for ^{230}Th (Kerber et al., 2023; Kerber et al., 2025).” (L218ff)

Also, the methodology used to measure drip water is not provided; see Table S2.

Drip water and speleothem samples were analyzed with the same methodology. We will add the according information:

“A total of 157 samples from Áaktun Kóopo’ speleothems as well as the three drip water samples were analyzed using high-precision $^{230}\text{Th}/\text{U}$ dating performed with a multi-collector inductively coupled plasma source mass spectrometer (MC-ICP-MS, Thermo Fisher Neptune plus) at the Institute of Environmental Physics, Heidelberg University. [...] For drip water analysis, about 15 to 30ml of sample was weighed and spiked with ‘TriSpike’, acidified with ultra-clean nitric acid, and subsequently dried down and re-dissolved.”

Specific half-lives should also be provided, rather than just citing Cheng et al. (2013).

We will add the following more specific statement:

“Ages were calculated using the half-lives provided by Cheng et al. (2013) for ^{230}Th and ^{234}U as well as by Jaffey et al. (1971) for ^{238}U .” (L230ff)

Questions:

- This ms focuses on the impact of high detrital Th on U-Th dating errors and emphasizes the importance of initial Th correction. However, it attributes anomalous fluctuations in high detrital Th concentrations in tropical regions as the rule, without delving into the controlling mechanisms. Does this fundamentally undermine the logical basis of the initial Th correction? In other words, if the specific processes and driving factors behind anomalous Th signals cannot be elucidated, will the effectiveness and applicability of isochronous ages calculated based on the assumptions of "uniform initial ratios" or "statistical averages" necessarily face significant uncertainties?

We thank Referee #2 for raising this important conceptual point. We fully agree that the processes and sources controlling detrital Th transport and initial $^{230}\text{Th}/^{232}\text{Th}$ in tropical karst systems are complex and not yet well constrained. This is precisely why assuming a single, fixed “bulk Earth” initial ratio can be problematic and may bias ages, especially in young, highly contaminated speleothems.

This complexity does not, however, invalidate the logic of applying initial Th corrections in general. Rather, it means that such corrections must be treated as approximations with explicit uncertainty, and that they should be constrained as far as possible by independent information. In our study, we therefore combine and compare several approaches to estimate R02i.

Of course, in an ideal case one would work only with material that has very low detrital Th and minimal initial-ratio complexity. In Áaktun Kóopo and similar sites of high archaeological and paleoclimate interest, such “ideal” speleothems are not always available, so dealing with significantly Th-contaminated material is unavoidable if one wishes to obtain any chronological information at all. In this context, independent constraints such as layer counting do not make Th corrections obsolete, but they can reduce the degrees of freedom in choosing R02i and provide a powerful test of whether a given correction scheme yields realistic ages. Our main conclusion is therefore not that anomalous Th behavior removes the usefulness of initial Th corrections, but that it necessitates (i) moving beyond a single fixed initial ratio, (ii) using multiple, independent constraints whenever possible, and (iii) explicitly propagating the remaining uncertainty into age–depth models.

To make this reasoning also clearer in the revised manuscript, we will add a statement to section 4.1.2:

“In such settings, initial Th corrections are best treated as approximations with explicit uncertainty, constrained where possible by multiple, independent estimates of R02i.” (L395ff)

As well as to the conclusions:

“While low-Th speleothems with simple initial ratios would be preferable, such material is not always available at sites of high archaeological and palaeoclimate interest, so working with variable Th-contaminated samples is sometimes unavoidable if any chronological information is to be obtained.” (L555ff)

- The validity of the isochron approach fundamentally relies on homogeneous initial thorium isotopic compositions among co-genetic subsamples. However, the manuscript explicitly reports highly variable and largely unsystematic initial ($^{230}\text{Th}/^{232}\text{Th}$) activity ratios. How can the authors justify the assumption of isotopic homogeneity required for meaningful isochron construction under such strongly heterogeneous conditions?

We thank Referee #2 for discussing this important point. We fully agree that the classical isochron approach is formally valid only when co-genetic subsamples share a common initial Th isotopic composition. In *Áaktun Kóopo'*, we therefore use isochrons only as local, first-order approximations applied to closely spaced subsamples within limited depth intervals, where the assumption of similar initial Th is most likely to hold. The resulting R02i values are treated as approximate rather than exact and are not assumed to represent the entire cave system.

In practice, these local isochron estimates are used alongside other approaches (e.g. stratigraphic reasoning and, where available, Sr/Ca layer counting) to narrow the plausible range of R02i and to quantify the associated age uncertainty. Our intention is not to claim perfectly homogeneous initial Th, but to show that, even under heterogeneous conditions, the isochron approach can still provide useful first-order constraints on R02i when interpreted cautiously and in combination with independent information.

To acknowledge this simplification, we will change the first paragraph of section 4.1.1 to

“Dense sampling in some stalagmites allows for first-order linear two-endmember mixing regressions (Osmond type 1- like ‘isochrons’). In this study, such ‘isochrons’ are applied only to closely spaced, co-genetic subsamples and are interpreted as local, first-order estimates of R02i, similar to the approaches of Warken et al. (2020) or Stinnesbeck et al. (2020). The ‘isochron’ approach using ISOPLOT (Ludwig and Titterton, 1994; Ludwig, 2008) (Vermeesch, 2018), produced five successful ‘isochrons’ from three stalagmites (E0-C, E4, E8), which yielded R02i ranging from 6.4 ± 2.4 to as large as 19 ± 10 .” (L353ff)

In addition, we will modify the conclusion section to:

“To determine R02i and resolve chronological inversions, we successfully employed a multi-method approach, combining isochron methods, stratigraphic constraints, and relative seasonal layer counting (Sr/Ca). We note, however, that R02i constrained by local ‘isochrons’ assuming binary-mixing relationships should be regarded as helpful local, first-order approximations to delimit the plausible range of initial Th activity ratios, rather than as exact descriptions of the underlying Th sources.” (L574ff)

- The authors relied on multiple verifications based on the isochronous method to correct the initial Th. It is known that young, low-thorium samples are highly sensitive to scattering caused by blank errors in the $^{230}\text{Th}/^{232}\text{Th}$ ratios. Still, the manuscript lacks a

good discussion of the contribution of procedural blanks and the propagation of uncertainty. How do the authors handle the blank correction propagating into the uncertainty of the final isochronous line?

We thank Referee #2 for highlighting the importance of blank corrections, especially for young, low-Th samples. We agree that such samples are particularly sensitive to scattering in measured $^{230}\text{Th}/^{232}\text{Th}$ ratios in general, but possibly also arising from procedural blanks. In the revised manuscript we now report the levels of our procedural blanks for Th and U and add a statement that all ages and activity ratios presented in the paper are corrected for these blanks:

“Chemical blanks are regularly monitored and commonly below 0.4 fg for ^{234}U and 0.04 fg for ^{230}Th (Kerber et al., 2023; Kerber et al., 2025).” (L225)

Notably, the individual procedural blanks analyzed along with the Áaktun Kóopo' series agree with these long-term observations (Kerber et al., 2023; Kerber et al., 2025), with values of 0.04 ± 0.03 (MAD) fg for ^{230}Th , 0.002 ± 0.001 ng for ^{232}Th , and 0.4 ± 0.2 (MAD) fg for ^{234}U . The Áaktun Kóopo speleothems generally have moderate to high U, so the signal-to-noise ratio for most Th measurements is favorable and blank contributions are small compared to the total Th signal. Only for the very youngest samples does the signal-to-noise ratio become poorer and the relative influence of the blank correction increase. For such samples, this contribution can be non-negligible and may increase the scatter in $^{230}\text{Th}/^{232}\text{Th}$ ratios.

However, for most samples, the influence of procedural blank variability is negligible for the inferred R02i spread. For example, samples E0-C-69 (#10975) and E0-C-73 (#11237) have relatively high S/N ratios but require R02i correction ratios of 11.5 and 41.5 respectively. By artificially doubling the original procedural blank correction, the required R02i to get the same corrected ages would be in the range of 10 and 37, respectively. In summary, while we cannot rule out that undetected outliers in individual chemistry blanks have contributed to some of the data points, and increase the variability in the data set, several lines of evidence suggest that the main patterns in our isochrons and in the inferred, large range of R02i are robust.

- Since not all speleothem samples have clear annual layers, how are the initial $^{230}\text{Th}/^{232}\text{Th}$ ratios determined for samples lacking chronological striations?

We thank Referee #2 for this question. In our study, annual Sr/Ca layer counting is only one of several possible constraints on initial $^{230}\text{Th}/^{232}\text{Th}$ ratios and is explicitly used only where clear, laterally continuous cycles are present (i.e. for selected intervals of E1, E8, and E23-3). For samples or stalagmite sections lacking such chronological laminae, R02i is instead constrained using a combination of (i) local isochrons based on groups of closely spaced subsamples, (ii) stratigraphic reasoning to enforce monotonically increasing ages with depth, and (iii) in a few cases, information from modern carbonate and drip-water activity ratios.

In other words, layer counting is not a prerequisite for determining initial $^{230}\text{Th}/^{232}\text{Th}$; it provides an independent check and local constraint where available. In sections without annual layers, we rely on isochrons and stratigraphic constraints alone, and the associated uncertainty in R02i (and thus in the corrected ages) is explicitly propagated into the final age–depth models.

- The manuscript applies two-endmember mixing regressions while simultaneously arguing for highly variable and unsystematic initial thorium compositions. How do the

authors reconcile the use of a binary mixing framework with evidence suggesting multiple temporally and spatially variable thorium sources?

We thank Referee #2 for this thoughtful question. We fully agree that, at the cave scale and over longer time spans, Áaktun Kóopo'. speleothems likely receive Th from multiple sources whose contributions vary in space and time. In this context, fitting two-endmember mixing regressions should be understood as a local, first-order approximation applied to subsets of samples that behave approximately linearly in $^{230}\text{Th}/^{238}\text{U}$ - $^{232}\text{Th}/^{238}\text{U}$ space, rather than as a literal statement that the entire cave system is governed by only two fixed Th reservoirs.

In practice, the binary-mixing fits are used to obtain approximate R02i values for restricted depth intervals where the data define a reasonably coherent trend, and these estimates are then considered alongside other constraints. In several key intervals, the resulting age–depth models are consistent with independent proxy comparisons, indicating that the two-endmember framework, although undoubtedly simplified, is sufficient as a first-order tool to recover robust chronologies. Our intention is not to deny the existence of multiple Th sources, but to show that, when applied locally and interpreted cautiously, binary mixing still provides useful constraints on initial Th under such heterogeneous conditions.

- The manuscript addresses the limitations of fixed initial thorium corrections under highly “detrital-contaminated” conditions. However, the exclusion of samples with $^{230}\text{Th}/^{232}\text{Th}$ ratios < 200 raises an important methodological question regarding the practical applicability of the proposed approach. If the most strongly contaminated samples still need to be discarded to achieve robust chronologies, to what extent does the proposed framework truly overcome the limitations associated with high detrital thorium contamination? The current workflow appears to improve uncertainty treatment primarily for moderately contaminated samples rather than resolving the challenges posed by the most problematic end-members.

We thank Referee #2 for this comment and agree that our current framework does not fully resolve the challenges posed by the most strongly detrital-contaminated samples. In this study, we deliberately apply first-order approximations, which work best at improving the treatment of initial Th uncertainty for moderately contaminated samples. Such approaches may help to keep analytical effort and laboratory costs at a feasible level especially for future studies in similar environments. We see our approach rather as a pragmatic compromise than a final solution, and we expect that future studies building on this framework may eventually help to better understand the underlying processes and thus allow more rigorous correction – and thus wider use – of strongly contaminated material as well.

- The manuscript primarily focuses on the propagation of age uncertainties associated with initial thorium contamination. However, potential variability in uranium concentrations may also significantly affect the reliability of U–Th ages, particularly under prolonged flooding or submergence. Although petrographic observations suggest no obvious recrystallization or diagenetic alteration, preservation of primary fabrics alone may not fully exclude subtle uranium remobilization or cryptic open-system behavior. Given the hydrologically dynamic cave environment and evidence for flooding events, how do the authors demonstrate that uranium concentrations and isotopic compositions were not modified post-depositionally?

We thank Referee #2 for this important comment. In principle, prolonged flooding or submergence could promote subtle U remobilization and open-system behavior, but in our case we consider this scenario unlikely for most of the dated material. The majority of stalagmites analyzed in this study come from cave sections that are not regularly flooded and show no evidence for long-lasting inundation. Also, petrographic inspection revealed well-preserved primary fabrics without recrystallization or secondary calcite cements. Along individual growth axes, U concentrations and $\delta^{234}\text{U}$ values vary largely consistent, which argues against major post-depositional modification of uranium contents or isotopic compositions. However, we acknowledge that this does of course not exclude the possibility of cryptic open-system effects on the more localized scale. However, we interpret the remaining, age inversions as being likely related to several possible effects. In addition to possible post-depositional modification on the microscale, undetected micro-hiatuses or uncertainties in initial Th correction (e.g., related to a more complex (isotopic) composition of the endmember(s)) could be possible reasons.

We will add a more explicit statement in section 4.1 of the discussion:

“Petrographic observations suggest no obvious recrystallization or diagenetic alteration; the stalagmites show well preserved primary fabrics and display largely coherent U concentrations and $\delta^{234}\text{U}$ values along their growth axes. Therefore, we consider wide-spread post depositional U remobilization to play a negligible role for our dated material.” (L326ff)

- Human activity increases the uncertainty of cave research. Did the Maya people's modification of the cave walls affect the cave's sedimentary environment? How to identify non-climate impacts of human activity changes?

We thank Referee #2 for this thoughtful question. Archaeological work in Áaktun Kóopo' has clearly documented that the Maya modified parts of the cave (e.g. low walls and partitions), but these studies have focused on the spatial organization and ritual use of the cave rather than on a detailed reconstruction of its sedimentological evolution. At present there is therefore no quantitative evidence that the Maya constructions systematically altered the large-scale sedimentary environment or hydrology of the chambers from which our stalagmites were collected.

Most stalagmites that contain distinct flooding layers come from a chamber that lies higher than areas that are more regularly flooded (e.g., the mortuary chamber), so inundation of these parts of the cave is expected to reflect comparatively rare, high-water events rather than day-to-day human activity at the cave floor. Nonetheless, human influences on the cave and the overlying soil–vegetation system cannot be ruled out, and non-climatic impacts are difficult to isolate with the available data.

- The manuscript emphasizes that dry conditions have been a major driver of social and environmental change among the Maya. However, much of the subsequent discussion focuses on flood events, inundation, and enhanced clay transport within the cave system. This raises an important question: the relationship between long-term hydroclimatic trends and short-term extreme hydrological events. Can the authors clarify whether the identified flood events are transient, superimposed on an overall drought trend, or reflect a trend of increasing hydroclimatic variability? Which factors contribute to the profound social and political upheaval marked by widespread population decline and the abandonment of many urban centers? A clearer distinction

between mean drought conditions and sudden flood dynamics will help strengthen paleoclimate interpretation and improve the coherence of climate narratives.

We thank Referee #2 for this comment and agree that the relationship between long-term hydroclimatic trends, short-term extremes, and Maya societal change is an important topic that has been discussed in literature since a long time. We would like to clarify, however, that the present manuscript does not suggest any causal link between drought conditions and social or environmental change. Where such links are mentioned (e.g., L97ff in the introduction) we explicitly refer to conclusions drawn in previous studies rather than to new evidence from Áaktun Kóopo'. We are convinced that the motivation for this study is phrased sufficiently clear with:

“This study focuses on constructing precise $^{230}\text{Th}/\text{U}$ chronologies for stalagmites from Áaktun Kóopo' Cave, [...] These chronologies will allow to fill a notable gap in high-resolution paleoclimate records for the region and provide a foundation for future investigations into hydroclimatic and environmental changes, particularly during periods of profound Maya societal changes.” (L102ff)

In principle, combining the Áaktun Kóopo' flood layers with the stable isotope and trace-element records could help to explore how short-term extreme events relate to longer-term hydroclimatic variability. However, a rigorous treatment of this question would require a dedicated analysis of proxy sensitivities (e.g. the drivers of $\delta^{18}\text{O}$) and a more extensive discussion than is possible within the scope of this methods-oriented study. We therefore refrain from drawing firm conclusions here about whether the identified flood events are transient features superimposed on an overall trend or reflect changes in hydroclimatic variability itself. Instead, we now explicitly note in the Conclusions that the chronologies and proxy records established in this work provide a framework for future studies that will address the relationship between long-term hydroclimate trends, short-term extremes, and their potential relevance for Maya cultural history in more detail.

The last section of the conclusions will be changed to:

“Ultimately, the improved chronologies and accompanying coherent proxy records from Áaktun Kóopo' Cave now provide a robust foundational framework for future high-resolution reconstructions of hydroclimate and environmental change throughout the timeline of Maya cultural evolution on the YP, e.g., how long-term trends and short-term extreme events, such as the identified flood layers, are related and how they may have intersected with phases of cultural change.”

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