

# Reviewer 1

Comments:

Line 148-149: The role of the second oven is not sufficiently explained here.

- 5 Re: We thank the reviewer for this constructive comment. We agree that the function of the second oven was not clearly described in the original text. The revised sentence reads:

*“...while the second oven is maintained at constant temperature and contains catalytic material to ensure complete oxidation and removal of non-carbon species.”*

- 10 Line 159-160: Strictly speaking, comparability across temperature windows requires that all experimental conditions are consistent, including carrier gas flow, heating rate, oxygen availability, and other operational parameters. If these differ between the applied techniques, then the same nominal temperature intervals may not correspond to equivalent processes or OM fractions. I therefore suggest that the authors explicitly clarify a bit here.

- 15 Re: We thank the reviewer for this important comment and agree that direct comparability across temperature windows requires consistent experimental conditions, which is not the case for the applied techniques. We have revised the sentence to clarify that the predefined temperature windows serve as a conceptual framework for comparison rather than representing directly equivalent OM fractions across methods. The revised sentence reads:

- 20 *“...selected to provide a consistent temperature framework for comparison with the SoliTOC decomposition scheme. While this alignment facilitates cross-method interpretation, it does not imply direct equivalence of organic matter fractions, given the differing analytical conditions and reaction pathways involved.”*

- 25 Line 187-190: Similar to Comment 2, the applied techniques rely on fundamentally different processes. Ts-Py-GCMS is a pyrolysis-based method, whereas ORO-AMS and SoliTOC are based on combustion.

Therefore, signals within the same temperature range may not represent comparable OM fractions, which should be clarified.

30 Re: We agree with the reviewer and thank them for this comment. The step-wise temperature approach is used intentionally to provide a consistent first-order framework that allows comparison of thermal trends across methods. We have revised the text to clarify that:

*“...While the underlying processes differ (pyrolytic decomposition for Ts-Py-GCMS versus oxidative combustion for ORO–AMS and SoliTOC), the use of a common temperature framework provides a reference for comparison of thermal trends across methods, not direct equivalence of OM fractions.”*

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Line 256-257: I saw higher ROC/TOC by ORO.

Re: We thank the reviewer for pointing this out. Upon revisiting the data, we confirm that ORO–AMS yields systematically higher ROC/TOC values compared to SoliTOC (Fig. S2). The original wording was incorrect and has been revised accordingly. The revised text now reads:

40 *“SoliTOC- and ORO–AMS–derived ROC/TOC ratios show consistent patterns across sample types, with higher values in PF, DB, and RU (ROC/TOC  $\approx$  0.25–0.45) and substantially lower values in AL (typically  $<0.15$ ). However, ORO–AMS systematically yields higher absolute ROC/TOC values compared to SoliTOC (Fig. S2).”*

45 Line 286-287: HO2 also shows a rebound in carbon age.

Re: We thank the reviewer for highlighting this point. While the rebound in the HO2 profile is described in the text, we agree that it was not sufficiently reflected in the overall summary. The revised sentence reads:

50 *“...Together, these patterns show that  $F^{14}C$  generally decreases with increasing thermal resistance across most features, with the exception of the AL and a partial rebound observed in the HO2 profile.*

Figure 4: The rationale for comparing  $^{14}C$  results from ORO with pyrolysis-based molecular fingerprints is not entirely clear.

Re: We thank the reviewer for this constructive comment. We agree that the rationale for combining  
55 ORO–AMS radiocarbon data with Ts-Py-GCMS molecular fingerprints was not sufficiently explained in  
the original manuscript.

We have revised both the figure caption and the main text to clarify that the combined presentation is  
intended to relate molecular composition to thermal stability and radiocarbon age, rather than to imply  
60 direct equivalence between fractions derived from pyrolysis and combustion-based methods. The revised  
text now reads:

*Caption Figure 4 Line 311 “The combined presentation is intended to relate molecular composition to  
thermal stability and radiocarbon age, rather than to imply direct equivalence between fractions obtained  
by pyrolysis and combustion-based methods.”*

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*And main text line 321 “To facilitate interpretation across methods, molecular compound-class  
distributions are presented alongside  $F^{14}C$  values derived from ORO–AMS for corresponding  
temperature intervals, providing a combined view of organic matter composition, thermal stability, and  
radiocarbon age.”*

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**Supplementary Material - Line 58: “green triangles”**

Re: We thank the reviewer for pointing this out. The figure description has been corrected to ensure  
consistency between the text and the plotted symbols. The revised sentence now reads:

*“...samples from FM2 (green triangles) and FM3 (red circles).”*

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**Supplementary Material - Line 59: “with ORO–AMS reporting slightly higher ROC/TOC values”**

Re: We thank the reviewer for pointing this out. The figure description has been corrected accordingly.  
The revised sentence now reads:

*“...with ORO–AMS reporting systematically higher ROC/TOC values (slope = 0.71,  $R^2 = 0.76$ , bias  $\approx$   
80 0.12),...”*

Supplementary Figure S2 Line 67-74 – “...Differences in absolute ROC/TOC ratios between SoliTOC and ORO–AMS are largely methodological. The continuous ramping approach of ORO–AMS and the discrete temperature steps of SoliTOC partition intermediate thermal fractions differently, leading to systematic offsets in ROC/TOC values. In particular, intermediate-temperature carbon fractions may be assigned differently between methods, resulting in higher apparent ROC/TOC values in ORO–AMS compared to SoliTOC. This offset (~0.2 on average in our dataset) is consistent with inter-method differences reported in similar comparative studies. These method-inherent biases highlight the value of using multiple thermal approaches to constrain OM reactivity and underscore the complementarity of SoliTOC and ORO–AMS.”

Supplementary Material - Line 82: “yield lower TIC/TC values than SoliTOC”

Re: We thank the reviewer for pointing this out. The figure description has been corrected accordingly. The revised sentence now reads:

“...with a systematic tendency for ORO to yield lower TIC/TC values than SoliTOC.”

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## Reviewer 2

We thank the reviewer for the positive and constructive evaluation of our manuscript and for recognizing the contribution of the study to permafrost carbon research. We appreciate the reviewer's suggestions, which helped us improve the methodological clarity and broaden the discussion of organic matter fate following thaw-slump mobilisation. Below, we address each point in detail.

### Comments:

1. Methodological clarification: The Ramped Oxidation technique is gaining traction in the community with ever-expanding applications. However, one thing that I am concerned about is the lack of homogeneity in step 1 of the process, i.e., the rate of heating and flow rate. This study uses 5 degree C/min. This is not the same across various other studies using this technique e.g., Garnett et al., or Stoner et al, (both of which haven't been cited here). A parallel can be drawn with something similar used to study e.g., atmospheric aerosol speciation (see Table 1 in Dasari et al., 2022 Front. Env. Sci. <https://www.frontiersin.org/journals/environmental-science/articles/10.3389/fenvs.2022.907467/full>).

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I believe these set of authors are perhaps the most well placed to discuss /comment on this aspect in detail in this manuscript: 'How does decreasing or increasing this rate of heating (and flow rate) affect the thermograms and thereby the findings/implications?' I suggest that the authors add a section or a paragraph regarding this in the methods or discussion, where they categorically discuss the lack of homogeneity (intentional or unintentional) of the heating /flow rates across the usage of RPO technique and its potential implications on the analysis (on this dataset) and in general. If possible, perhaps show in the Supp.info thermogram (s) using different heating /flow rates for such exotic samples if available, or for an equally interesting sample material.

25 A reader would want to know how and why this rate was chosen. Also, why didn't the authors consider heating ramp to 900 °C as in other studies using similar technique?

Re: We agree with the reviewer that ramp rate, flow rate, gas composition, and system configuration are important operational parameters in RPO/ORO analyses, which have been extensively tested in Hemingway et al. (2017) and, for the current setup, in Bolandini et al. (2025). Rather than repeating these methodological discussions in full, we revised the Methods section to clarify that the carrier-gas flow was selected based on previous optimisation of the ORO–AMS setup (Bolandini et al., 2025). We also clarify that the influence of ramp rate on thermogram shape and activation-energy estimates is explicitly treated in the kinetic framework used here (Hemingway et al., 2017). Finally, we added broader methodological context noting that comparisons among RPO/ORO studies should consider differences in operational settings and system configurations (Dasari and Widory, 2022; Garnett et al., 2023; Stoner et al., 2023). We also clarified that the samples were ramped to 900 °C, but thermograms are shown only up to 800 °C because only minimal additional CO<sub>2</sub> was released above this temperature. The added paragraph reads:

*“The carrier-gas flow of 90 mL min<sup>-1</sup> was selected based on previous optimisation of the ORO–AMS setup, where this setting provided stable gas transport, limited reflux or back-mixing, and ensured reproducible CO<sub>2</sub> transfer to the trapping interface (Bolandini et al., 2025). A heating rate of 5 °C min<sup>-1</sup> was used to provide sufficient thermal resolution while maintaining adequate CO<sub>2</sub> yield per temperature interval for AMS analysis. The influence of ramp rate on thermogram shape and activation-energy estimates is explicitly treated in the kinetic framework used here (Hemingway et al., 2017). Nevertheless, because ramp rate, gas composition, flow rate, and system configuration can influence thermogram shape and apparent thermal metrics, comparisons among RPO/ORO studies should consider operational differences, as also noted for other ramped oxidation and thermal–radiocarbon approaches (Dasari and Widory, 2022; Garnett et al., 2023; Stoner et al., 2023).”*

and in the text (line 187-188): *“Samples were ramped to 900 °C, but thermograms are displayed only up to 800 °C because only minimal additional CO<sub>2</sub> was released above this temperature.”*

2. Charring issue: I believe any organic material can char after a certain temp range. How did the authors address this issue in this dataset? Please provide clarification in the manuscript.

Re: We thank the reviewer for this important comment. We agree that charring and secondary pyrolysis  
55 reactions may occur during thermal decomposition, particularly at higher temperatures, and that this needs  
to be explicitly considered when interpreting Ts-Py-GCMS results. We have clarified this point in two  
places. First, in the ORO–AMS Methods section, we now specify that the oxygen-rich carrier gas was  
used to favour oxidative decomposition and minimise charring during ramped oxidation. Second, in the  
Ts-Py-GCMS Methods section, we clarify that pyrolysis-derived compounds are interpreted as  
60 operational pyrolysis products rather than direct molecular inventories of the original OM. High-  
temperature fractions, especially aromatic and condensed compounds, are therefore interpreted cautiously  
because they may include secondary products formed during pyrolysis, including char-derived structures.  
The revised text reads (line 172-173):

*“This oxygen-rich carrier gas was used to promote complete oxidative decomposition of the sample and  
65 minimise charring during ORO–AMS analysis.”*

(line 222-225):

*“While the underlying processes differ (pyrolytic decomposition for Ts-Py-GCMS versus oxidative  
combustion for ORO–AMS and SoliTOC), the use of a common temperature framework provides a  
reference for comparison of trends across thermal windows, without implying direct equivalence of OM  
70 fractions.”*

(line 246-248):

*“Because Ts-Py-GCMS involves thermal decomposition under oxygen-free conditions, charring and  
secondary pyrolysis reactions may occur, particularly at higher temperatures. Therefore, identified  
compounds are interpreted as operational pyrolysis products rather than direct molecular inventories of  
75 the original OM.”*

3. Discussion: While the authors have touched a bit on the 'fate' of OM. I believe the run-off samples are  
super interesting and can be used to expand the discussion towards whether this thermally stable OM is  
expected to be a sink of C to the Arctic ocean or river sediment OR further in the river system, contribute  
80 to the release of 'aged' C as GHGs (see Dasari et al., 2024 PNAS Nexus) from the river system? I think  
it's important to at least comment on it in the discussion.

Re: We thank the reviewer for this constructive suggestion. We have expanded the discussion to clarify that runoff samples represent a key transitional pool between terrestrial mobilisation and downstream processing. We now discuss two non-exclusive pathways: downstream transport and sedimentary storage of thermally stable particulate OM, and potential delayed transformation into dissolved or gaseous carbon during riverine processing. We added the following paragraph (line 475):

*“The RU samples therefore represent an important transitional pool linking mobilisation of terrestrial material to riverine export and potential in-stream processing. Their similarity to PF and DB material indicates that runoff exports old, thermally stable particulate OM with limited alteration during early mobilisation. This material may be transported downstream and deposited in riverine, deltaic, or coastal sediments, acting as a transient or longer-term particulate carbon sink. However, river systems are not passive conduits: hydrodynamic sorting, oxygen exposure, changes in mineral association, and microbial processing may alter OM reactivity during transport. Under such variable conditions, parts of this thermally stable, aged carbon could still be transformed into dissolved or gaseous forms and contribute to a translocated, delayed greenhouse-gas release, as recently suggested for aged carbon leakage from the Mackenzie River system (Dasari et al., 2024).”*

4. Minor issues: I have found in several places across the ms the statements are not followed by the Figure numbers. Please fix this to improve readability.

Re: We thank the reviewer for pointing this out. We have revised the manuscript to ensure that relevant statements are consistently accompanied by the appropriate figure references, improving readability and guiding the reader more clearly through the methods, results, and discussion. The main changes are listed below.

Correction for Table 1 clarity:

Site	Coordinates	Elevation (m)	Active-layer depth (cm)	Headwall height (m)	Scar-zone area (ha)
CB	67.182° N, 135.732° W	576	46	5.8	3.4
SF	67.183° N, 135.811° W	720	56	7.6	< 1
FM2	67.257° N, 135.236° W	338	23	24.2	48

Site	Coordinates	Elevation (m)	Active-layer depth (cm)	Headwall height (m)	Scar-zone area (ha)
FM3	67.253° N, 135.273° W	391	65	9.8	10

Corrected text for readability:

line 124 *“from the headwall”*

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line 144 and 163 added *“(Mittelbach et al., 2025)”*

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line 166-168 modified *“The ROC/TOC ratio, first introduced as an operational index of recalcitrance by Mittelbach et al. (2025), may therefore serve as a proxy for intrinsic oxidation resistance but is not necessarily equivalent to biological lability (see Results and Discussion for an assessment of methodological limitations).”*

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line 407-408 *“Thermal behaviour, radiocarbon patterns, and molecular compositions together show that fundamentally different OM pools are present within the different geomorphic features of the four slumps (Figs. 2–4).”*

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line 418-419 *“Py-GCMS molecular fingerprints provide a compositional context for these contrasting thermogram shapes when evaluated within the common temperature framework (Fig. 4).”*

line 429 *“Radiocarbon profiles across thermal windows reinforce this interpretation (Fig. 3b,d).”*

line 450 *“Activation energy-resolved  $F^{14}C$  spectra further support these trends in energy space (Figs. S17–S18).”*

130 line 460 ~~“AL samples consistently occupy a low  $\mu_E$ , high  $F^{14}C$  domain, whereas PF, DB and RU plot towards higher  $\mu_E$  and lower  $F^{14}C$ .”~~ This sentence has been removed for readability.

line 463-464 ~~“—likely reflect the presence of protected or mineral-associated OM within active-layer and upper-permafrost horizons.”~~

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line 469-470 ~~“above, individual AL samples can host young and old carbon across overlapping activation-energy ranges and thus do not follow a strict “young = low-T / old = high-T” rule. Instead, these broader  $\mu_E$ - $F^{14}C$  and  $\mu_E$ -ROC/TOC trends emerge when ...”~~

140 line 500 ~~“Across the Peel Plateau sites, ROC/TOC ratios and bulk  $F^{14}C$  patterns reveal... (Fig. 5b).”~~

line 524 ~~“... deposits (Kokelj et al., 2017; Zolkos et al., 2018).”~~

145 line 529-530 ~~“Across FM2, FM3, CB and SF, deeper PF—as well as mobilised components DB and RU—consistently share low bulk  $F^{14}C$ , high ROC/TOC and high  $\mu_E$  (Fig. 5; Figs. S17–S19),...”~~

150 line 535 ~~“Importantly, the uniformly low TOC contents in PF, HO, DB and RU across all slumps further show that neither FM2/FM3 nor CB/SF host significant extents of peat rich or historically productive ecosystems—consistent with a glacial moraine, ice-rich substrate.”~~ This sentence has been removed for readability.

line 548-549 ~~“These systems are characterised by relatively low TOC and strong mineral control on OM stabilisation, closely matching the geomorphic and ...”~~

155 line 566-567 ~~“Mapping studies show that the vast majority of Yedoma occurs in North Asia, whereas the Canadian share is small and spatially restricted”~~—this sentence has been removed for readability.

line 579-581 *“In contrast, permafrost material is uniformly radiocarbon-depleted, thermally stable, and compositionally resistant. The similarity to debris and runoff indicates largely downslope mobilisation of permafrost-derived OM rather than in situ decomposition.”*

line 590-593 we modified *“... characteristics of PF-derived particulate OM. Instead, slumping primarily redistributes compositionally resistant, ancient carbon downslope with little evidence for rapid transformation, consistent with observations from other mass-wasting-dominated systems with similar geological settings.”*

line 626-628 we modified *“... developed in such substrates therefore likely mobilise a fundamentally different permafrost carbon pool than Yedoma-derived slumps. Predicting the fate of thaw-mobilised permafrost carbon thus requires integrating OM age, composition, and energetic stability within geomorphic context.”*

Conclusion section:

In the funding section, we added *“J.D.H. acknowledges funding from the European Research Council (ERC) under the European Union’s Horizon 2020 research and innovation program (Grant agreement No 946150).”* and corrected the names *“J. E. Vonk acknowledges funding from a European Research Council (ERC) Starting Grant (THAWSOME, grant no. 676982). K. Keskitalo further acknowledges funding from the Nederlandse Organisatie voor Wetenschappelijk Onderzoek (NWO, Dutch Research Council; Rubicon grant no. 019.212EN.033).”*

Additional minor corrections to spacing, grammar, punctuation, and terminological consistency (e.g., use of “OM” versus “organic matter”) were implemented throughout the manuscript to improve clarity and readability.

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