This revision of the MS very successfully clarifies that the MS is about carbonate removal methods prior to RPO analysis. The text is much clearer and reads well with some editorial changes needed. Specific suggestions are included below.

Late in my review I read the response letter and learned that the TOC and carbon isotope data might be published later. That is great.

I have an issue with Line 52 of the MS which states "Using RPO analyses supplemented with bulk measurements (TOC and δ13Corg), we assess the potential…" – BUT the only actual mention of carbon isotope data is in Table S2. There is no discussion of the carbon isotopic data in the manuscript. The issue can be solved by REMOVING "supplemented with bulk measurements (TOC and δ13Corg)," from Line 52. If the portion of the above sentence is removed, the data can be left in the MS even though it is not discussed.

In a future paper I would like to see the statement seen in the original MS Conclusions that rinsing of high carbonate samples with liquid acid was the best pretreatment for carbon isotopic analysis of organic carbon. I actually referred people to the pre-print with this in mind. I would like a paper to easily refer people to in the future.

Jason Curtis, Sept 17, 2025

Line 7 - Remove "The" – very first word in the abstract

Line 8 – Be clear as to what "characterizing sedimentary organic carbon" means. Concentration? Labile versus refractory? Isotopically? Etc.

Line 8 – Same as above. "bulk carbon and molecular-level analyses" Bulk what, molecular what analysis. See similar comment for line 101.

Line 15 – "Notably" not quite the right word. Maybe "Generally, results from…"

Line 37 Remove - "Moreover"

Line 68 – "additionally" should be removed

Line 69 needs work "and temperatures, the potential influence of heating" Maybe "…and drying temperature to understand potential influence…"

Line 70 "furthermore" is not the right word. Use "Additionally" or similar

Line 79 – "to be neutralized" should be "until neutralized"

Line 80 – what mass of sample?

Line 83 remove “beneath”

Line 84 “This is based on the former practice that” - should be “this is because previous studies have shown …”

Line 88 and further – Comment – I still contend that freeze drying, air drying, or oven drying fumigated samples does not remove the HCl. It simply removes the water from the sample. It leaves residual HCl. And this residual HCl is strong. This is rarely noted in papers.

Line 94 – “Aside” is awkward. Maybe “alongside” but this is not perfect either.

Line 98 – What does the “**§**” symbol refer to?

Line 98 – remove “to be” – not needed

Line 101 – What does “bulk” analysis refer to? Bulk %CaCO3?, bulk total lipids, bulk cholesterol? I think that it means bulk d13C but this needs to be clear

Line 102 – Again, what does “bulk carbon measurement” mean. Should be “bulk carbon isotopic measurement”.

Line 102 – change “placed” to “placed into”

Line 104 – IRMS should probably be a Thermo Electron. Corporation name in China might be different than I am used to. Check this.

Line 105 – Call for table 2 should be Table S2 – CHECK calls for tables throughout.

Line 106 – change to “measurement of usgs40”

Line 110 – “with thermocouples mounted”. “mounted” seems unnecessary.

Line 115. If the 5% O2 is 95% Helium and 5% O2 just write this. Don’t simply use “diluted” which does not tell anything about what it was diluted with. I later learn from the Sup info and the diagram that it is O2 mixed with He.

Line 115 continued – I don’t see how the gas inputs in the text match up with the gas inputs in Fig S1.

Line 116 – define “sub-oxidation mode” – probably what was described before but not clear enough

Line 116 – “adopted” maybe should be “used”

Line 122 – “Notably, the residual chloride in sediments” should be “Residual chloride in acidified sediment samples”

Line 124 – “This consideration was of no concern to” should be “Residual chloride was of no concern for because it was already removed”

Line 124 “rinsing” should be “rinsed”

Line 125 – “counterparts” should be “samples”

Line 125 – “surged the risk” – should be “increased the possibility of damage to catalytic wires”

Line 126 “as the standard” probably not needed

Line 130 – “the in-house standard (Irati T2)” should be “Irati T2”. End the sentence here

Line 130 – Start a New sentence. Change “…by assuming that chlorine gas is generated…” to “We assume that chlorine gas generated…” (no need for “is”)

Line 138 – “Based on aforementioned” should be “Using the aforementioned”

Line 158 – “gradient of HCl concentrations” should be “gradient of HCl concentrations used for sample acidification”

Whole sentence from Line 156 to line 158 – “Although variations” Remove “Although” - Then later – “and S4), thermographic patterns” should be “and S4) PERIOD then start a new sentence. “Whereas thermographic patterns”. (split sentence into two. This will help to emphasis the second sentence.)

Line 159 – “To illustrate the inherent consistency between thermograms and HCl concentrations being applied” remove “inherent” and change “being applied” to “HCl Conc used to acidify samples”

Line 169 “In contrary” should be “In contrast”

Line 170 “has been” should be “was”

Line 183 – Use “as aggregates”

Line 190 – Include which metal are isotopically fractionated.

Line 215 – “forms” should be “formed”

Fig 2 – “(a), (b), (c) and (d) represent subsamples” Maybe change to “Panels (a), (b), (c) and (d) are subsamples…”

Line 224 – “Powders” adds confusion. I suggest using “Samples”

Line 241 – “other” should be “rather”

Line 259 – “held by” should be “found in”

Line 264 “Former studies” should be “Previous studies”

Line 268 “overlain together” is repetitive. “Overlain” is sufficient.

Supplementary

Fig S1: “The” schematic – remove “The”

Fig S1 “and the corresponding thermogram” should be “and a representative thermogram”

Fig S2: Suggested changes “Normalized thermograms of the standard sample (Irati T2) analyzed before and after analyses of acid-fumigated subsamples. No obvious shifts are observed when comparing the thermograms.”

Fig S4 – Initially I had no idea what FD means. Determined that it is Freeze dried. No clue what heat is if it is not oven dried. Define FD, OD, and heat in the caption.

Fig S5: The “shot” should be the “Image” or the “photograph”

Fig S5 Continued – “The black remnants are likely sourced from the incomplete” should be “The black remnants are likely a result of incomplete”

Fig S6 – I suggest using “Raw sample” or “original unmodified sample” instead of “raw aliquot”

Table S2 – I would suggest adding to the caption something like “See Table S1 for definitions of different experiment conditions (EC numbers)”