

The authors would like to thank the Anonymous Referee for his/her attentive reading and his/her useful and constructive comments. We agree on most of them, see our detailed response below. Please note that the reviewer comments are in bold and that the line numbers correspond to the ones of the Ms with track change. The sentences or the quotations in italics are the modified parts of the Ms.

L.33 A final concluding sentence may be beneficial e.g., "This work is a methodological step to measure SIC, SOC and some of its properties in a single analytical run. More work is required (1) on a wider range of soil samples with differing land use other forms of carbonates and sampling depths and (2) to explore if the mathematical correction is also valid for the mentioned various conditions"

Thank you for this suggestion. In the conclusion of the paper (l.487-490), we mentioned that avoiding the statistical corrections should be the next step to improve SOC and SIC quantification by RE. So, we adjusted your sentence as follow to be consistent with the conclusion: *"This work is a methodological step to measure SOC and SIC contents in a single analytical run on a non-pretreated aliquot. More work is needed i) on a wider range of soil samples with differing land use and other forms of carbonate mineral and sampling depths and ii) to avoid the use of statistical corrections of the TOC and MinC parameters"* (l.34-36)

L.46 Do you mean "forming complexes with calcium ions"??

The calcium ion forms complexes with the organic matter and stabilizes the soil aggregates. The sentence was confusing so we modified it as follow: *"SIC can act as a sink of atmospheric C (Buglio et al., 2016; Cailleau et al., 2011; Gao et al., 2017; Vicca et al., 2022) and, thanks to the calcium ions associated with carbonate, SIC improves SOC stability by stabilizing soil aggregates and by forming complexes with organic matter (Rowley et al., 2018; Shabtai et al., 2023). Nevertheless, SIC can also act as a source of atmospheric C (Chevallier et al., 2016; Cardinael et al., 2019; Zamanian et al., 2021).* (l.48-52)

L.74 Thermograms do not actively measure anything, they are a graphical representation of the results of measurement. Please re-phrase

We rephrased: *"Thermograms plot a property against time and temperature and provide a rapid characterization of C associations in the sample, based on a single aliquot."* (l.79-81)

L.106 Please add 1 to max 3 concise hypotheses here.

We reformulated the end of the Introduction part: *"This study proposes to adapt the RE thermal analysis protocol to optimize the quantification of SOC and SIC in soil samples. Comparisons between SOC and SIC quantifications by EA (after decarbonation, noted EA_{HCl} , and after decarbonation, noted $EA_{550^{\circ}C}$, respectively) and by RE (with and without statistical corrections of the standard TOC and MinC parameters) were carried out on 30 different agricultural topsoils with a wide range of SOC and SIC contents and on three geostandard materials and a calcite sample. We hypothesised that i) statistical corrections on TOC and MinC parameters improve the estimation of SOC and low SIC contents, respectively, and ii) the*

standard cycle analysis needs to be adjusted to improve the estimation of high SIC contents by the corrected MinC parameters.” (l.105-112)

L.113 Please look up standard nomenclature for the journal and use throughout.

We changed by: “g SOC kg⁻¹”, “g SIC kg⁻¹” and, “g TC kg⁻¹” in all the Ms to avoid any confusion.

L.113 I do not understand this part:

1) You measured 3 samples (EA_{550°C}) with contents < 0.1 gC.kg⁻¹soil

2) You state that the EA analytical error is 2.0 gC.kg⁻¹soil

The two statements do not fit. Please clarify.

Below, calcite measurement has an error of 0.29 gC.kg⁻¹soil (EA). So, is the error in 2) for EA_{550°C}, not EA? If so, how can statement 1) be valid? Contents are also > 2.0 gC.kg⁻¹soil

You are right, “analytical error” is not the right term, and the explanation is unclear. As mentioned in the *Data analysis* section (l.321-323), the analytical error of EA is 5% according to the norm ISO (1995) and 2% for the RE. We chose the 2.0 gC.kg⁻¹soil limit because we did not see any SIC-specific RE signals, or very low ones, for soil samples with such low SIC contents. We modified the explanation in the *Material* section: “Among these 30 soils, four were considered noncalcareous because they had no or very low Rock-Eva® (RE) signals specific to SIC (Table 1). These four noncalcareous soils were analysed by EA and RE (Supplementary materials, SM 1), similar to the other soils.” (l.118-122) and in the *Corrections of the standard parameters* section: “For noncalcareous soils, the signals associated with the MinC parameter (SM 1) are then integrated into the correction of the TOC parameter, and the corrected MinC parameter is set to 0.” (l.238-239). We also modified the sentence on non-calcareous soils in the Discussion of the Figure 5: “For the four soil samples considered noncalcareous soils, the SIC contents assessed by EA_{550°C} averaged 0.5 ± 0.8 g SIC kg⁻¹, and the SIC contents assessed by the uncorrected MinC parameter averaged 1.07 ± 0.26 g SIC kg⁻¹, which is consistent with very low RE signals specific to SIC observed in these samples. Thus, we consider a soil sample as noncalcareous when the measured SIC content is lower than 2.0 g SIC kg⁻¹.” (l.360-364)

L.116 It is common to correct for residual moisture (measured on another aliquot) at 105°C. Please state if this was done. Residual moisture can be up to 10% of sample weight, depending on clay and SOC content.

We agree, the residual moisture correction is essential to quantify C variations. We did not perform this correction. The correction factor would have been applied in the same way on the two data sets because we compared two methods on the same soil samples. We specified it in the *Material* section: “No correction for the residual moisture at 105 °C was performed for either the RE or EA analysis.” (l.123-124)

L.130 In the WRB the entirely correct term is "Reference Soil Group (RSG)".

Please add a citation of the WRB somewhere. It is not that important for this manuscript, however, soil classification changes over time and it may be hard for coming generations of soil scientists to reconstruct the used system.

Recommended citation:

IUSS Working Group WRB. 2015. World Reference Base for Soil Resources 2014, update 2015 International soil classification system for naming soils and creating legends for soil maps. World Soil Resources Reports No. 106. FAO, Rome

Thank you for pointing out this oversight. We modified "Soil type" by "Reference Soil Group" in the Table 1 (l.135) caption and column name and we added the citation of the WRB.

L.132 Analytical error was given as 2.0 gC.kg⁻¹soil. If values are lower, please give the value as < 2.0

We added a * on the SIC content of the samples considered as non-calcareous and added the meaning at the end of the Table 1: "**soil samples considered noncalcareous because they had no or very low RE signals specific to SIC*" (l.139).

L.137 Please check nomenclature of units throughout. Sometimes there is a space, sometimes not

We modified e.g., "*The capsules were then dried at 60 °C for 48 h*" (l.149).

L.163 Not sure about that one. Is this purified air with standard atmospheric composition? Or pure oxygen? The latter would make more sense

We added more details on the gases used for the RE analysis: "*pyrolysis of the sample under an inert dinitrogen atmosphere (N₂, purity = 99.999%) and oxidation of the residue under synthetic air (80% N₂ and 20% O₂, purity = 99.999%).*" (l.159-161).

L.200 Thank you for this clear explanation!

Note: may this cause uncertainty? It is stated that "half" of the amount is from carbonate. Would that depend on the total amount of carbonate?

The ½ coefficient is a stoichiometric coefficient that depends on the amount of thermoresistant organic matter rather than the carbonate amount. The CO evolved between 550°C and 650°C is assumed to only come from the Boudouard's reaction (CO₂ + C → 2CO) implying that among the two CO molecules produced, one is coming from inorganic carbon (the CO₂ evolved by SIC thermal breakdown), and one is coming from the residual organic C. Thus, half of the S3'CO signal should be integrated in the TOC parameter calculation and the other half should be integrated in the MinC parameter calculation whatever the carbonate amount. However, we know that a part of thermoresistant OM can decomposed after 550 °C (SOTHIS correction). Thus, a minor part of the CO₂ involved in the Boudouard's reaction could also come from thermoresistant OM decomposition. As the pyrolytic CO signal is very low and is one of the lowest contributors to the TOC and MinC parameters (< 1%, Table 3), we assumed this uncertainty negligible.

L.264 This means that RE systematically underestimates TOC when fresh organic matter is present? Maybe make this a bit clearer.

We agree, we clarified the sentence: “As the studied soils were collected in agricultural topsoils (soil depth < 15 cm), they also contain fresh organic matter. Thus, in this study, the TOC parameter corrected using SOTHS was also corrected with the second correction of Disnar et al. (2003), i.e., by adding 6.8% of the corrected TOC (Figure 2).” (l.247-251)

L.270 Please check this equation! You calculated the "amount of correction", not the "corrected amount".

You are right! Thank you very much for noticing this mistake. We corrected: “Corrected TOC = (TOC+MinC) × 1.068” (l.257).

L.362 If slope is not significantly different to 1, please state the actual p-value

We modified the Figure 4 (l.331) and 5 (l.351).

L.377 Please state the p-value

We modified e.g., “Wilcoxon test: $P = 0.18$ ” (l.341) and “ 0.98 ± 0.01 , Figure 4, Student test: $P = 0.17$ ” (l.344).

L.384 Please check colors and possibly adjust: <https://www.color-blindness.com/coblis-color-blindness-simulator/>

The viridis color palette is often a good choice for printing b/w and colorblindness

We changed the colors of the Figures.

L.384 I am confused about the p-values.

1) I assume they refer to the slope

2) They should show the difference to 1?

3) Please state the actual p-value if not significantly different to 1

Yes, they refer to the significance of the difference between the slope and 1. We specified in the caption of the Figure 4 (l.331) and 5 (l.351): “The p-values presented on the graph indicate the significance of the difference between the slope and 1.”.

L.384 There is quite a difference to the Discussion paper

Exact. Because many corrections have been asked on the Discussion paper, we took the opportunity to add 6 soils to the initial soil panel and reanalysed the soil samples with the same RE apparatus of IFPEN. In the Discussion paper, the first panel was analysed with the RE apparatus of Unil and the second panel was analysed with the RE apparatus of IFPEN. The use of two different apparatus has been criticised by one reviewer. Thus, no more results given by the Unil RE apparatus are presented in the new Ms. The underestimation of SIC content higher than 62.5 g SIC kg⁻¹ is more pronounced because we fixed the amount of sample at 70 ± 1 mg. The Unil RE data had been obtained on sample amount between 60 and 70 mg.

L.411 This value will depend on the absolute amount of SIC in the crucible, right? So, for the actual settings and sample amount (70 mg) the content is true. Using smaller amounts will solve this problem / change the content, right?

Exactly. The 62.5 g SIC kg⁻¹ value has been assessed on the graph (this is why we specified " a value of approximately ..."), we took the value just behind the first underestimated sample. This value is consistent with the limit of SIC amount in the crucible found at 4 mg afterward. Having 4 mg of SIC in the 70 mg of sample contained in the RE crucible means that the sample has a SIC content of 57,14 g SIC kg⁻¹; a SIC content of 62,5 g SIC kg⁻¹ corresponds to 4,37 mg of SIC in 70 mg of sample (l.395).

L.413 But still very good. The significance comes from the high precision - just as a side note

We agree, the statistical correction is usually lower for the MinC parameter than for the TOC parameter.

L.501 However, especially in soils with high proportions of stable organic carbon (e.g., pyrogenic carbon) care must be taken with the RE method.

We did not understand your comment. Did you mean with the EA method? Here we explain that the systematic error observed for this soil is probably due to an incomplete combustion of the organic matter during the heating pretreatment before EA. We agree, this error is likely linked to the proportion of thermoresistant organic matter in the sample, possibly pyrogenic carbon but we cannot know for sure. As explained in the *Corrections of the standard parameters* section, the purpose of the SOTHIS correction is to correct the accounting of a part of thermoresistant organic matter in the MinC parameter. The coefficient used for this correction also depends on the proportion of thermoresistant OM and leads uncertainties. We specified it in the conclusion: *"To be independent of statistical corrections, which could depend on the SOC and SIC forms in the soil sample, further studies on a large panel of soils should focus on the distinction between the signals from the pyrolytic cracking and oxidative combustion of SOC and the signals from the SIC pyrolytic and oxidative thermal breakdown."* (l.487-490).