

Please note that our answers are in bold and that the line numbers correspond to the ones of the Ms with track change. The quotations in italics are the modified parts of the Ms.

This is a very nice and timely study. The idea to lower the pyrolysis temperature to 520°C is simple but brilliant, effectively solving the long-standing problem of signal overlap between SOC and SIC at the end of the standard pyrolysis. The manuscript is well-written, the dataset is extensive, and the results are convincing. This work has significant practical implications for improving Rock-Eval standard procedures.

The authors would like to thank the Anonymous Referee #2 for his/her attentive reading, his/her compliments on our study and his/her constructive comments. Indeed, simplest solutions are sometimes the best. We agree on all your comments, see our detailed response below.

However, a few points require clarification and are listed below:

Major Comments:

Did you test other temperatures (e.g., 515°C, 525°C)? A graph showing the SIC signal (from the oxidation phase) versus final pyrolysis temperature for a few samples would be very helpful to prove that 520°C is the optimal cutoff. Please provide a more detailed discussion on the decomposition temperatures of different carbonate minerals (calcite, dolomite, siderite) to better explain why 520°C is the chosen "practical compromise."

We tested a final temperature pyrolysis of 550 °C on few soil samples and the reference materials and concluded that 550 °C was not low enough to avoid SIC thermal breakdown during the pyrolysis phase. We decided to not show these results to shorten the article. The choice of 520 °C was rather motivated by the previous use of this temperature boundary in RE literature on soil so we specified it in the 1. Introduction section: "*With this standard pyrolysis, the temperature boundary of 520 °C is often used in soil literature to distinguish the HC signal emitted by refractory OM from the HC signal emitted by highly refractory OM (Malou et al., 2020; Deluz et al., 2024).*" (L96-98). Using a lower temperature boundary than 520 °C could slightly improve the separation of the SOC and SIC pyrolysis signals but will lead to a loss of meaningful HC signal for OM characterization. The decomposition temperature of calcite and dolomite is given in the 1. Introduction section. To avoid any confusion, we preferred to remove the mention of siderite in the 1. Introduction section and specify clearly that calcite and dolomite are the most common soil carbonates and that reducing the temperature at 520 °C prevent the decomposition of these carbonates only: "*During pyrolysis, the thermal decomposition of common soil carbonates (e.g., calcite, dolomite) begins around 550 °C (Hazera et al., 2023). Reducing the final temperature of pyrolysis to 520 °C offers a practical compromise: it prevents the decomposition of calcite and dolomite during pyrolysis while still preserving a meaningful HC signal for characterizing soil OM.*" (L103-109). We specified in the 3.1 section that no siderite was detected by XRD (see answer of your next comment) in the soil samples and that this type of carbonate might impact the SOC and SIC quantification by RE because of their lower thermal breakdown temperature: "*MINC₅₂₀ estimated properly SIC_{550°C+EA} up to 100 g C kg⁻¹ for the samples of this study containing exclusively calcite and dolomite. No siderite or oxalate carbonates were detected by the X-ray diffraction analyses on the samples used in this study (Table S1). The thermal decomposition of siderite*

or oxalate carbonates can start before 400 °C during the pyrolysis phase (Ferro, 2012; Sebag et al., 2018) and thus might impact the SOC and SIC quantification by RE.” (L309-312). Then, we added as a perspective in the 4. Conclusions section the need for specific studies for samples containing other carbonates than calcite or dolomite: “Even if calcite, and dolomite are the main carbonates in soil, further investigations are needed on soil samples containing carbonates with a thermal breakdown beginning before 520 °C, e.g. siderite and oxalate, to assess the influence of these carbonates on the SIC quantification by RE.” (L441-444). We hope these changes clarify the choice of 520 °C and the applications of this method.

The type of carbonate (e.g., calcite vs. dolomite) and its association with organic matter could affect its decomposition temperature. I recommend adding mineralogical data (e.g., XRD analysis) for a subset of samples to characterize the dominant carbonate phases.

We hoped the revision would have been quicker but unfortunately the Table S1 is still under revision. We apologize for this inconvenience. We asked a private link that you can check the Table S1 before its publication: <https://entrepot.recherche.data.gouv.fr/privateurl.xhtml?token=36591ec5-cc13-4f84-9fb5-a0d249c917cd>. As mentioned above, no siderite or other “rare” carbonates were detected by XRD. We specified it in the 2.1 Materials section: “*The X-ray diffraction analyses detected quartz, phyllosilicates, and carbonates. Carbonates were mainly calcite and, to a lesser extent, dolomite (Table S1).*” (L127-129).

Please discuss if the diagenetic carbonates in rocks behaved differently from the pedogenic carbonates in soils in your analyses. This will help clarify the applicability of your method to different sample types.

We did not discuss the carbonate forms in this study to shorten the article. The isotopic signatures ($\delta^{13}\text{C}$) of the SIC of the soil samples will be add in the Table S1. $\delta^{13}\text{C}_{\text{SIC}}$ were variable enough (between 0 ‰ and -15 ‰) to assume the presence of lithogenic and pedogenic carbonates in our soil samples. Moreover, several reference materials were rock or sediment samples. We reminded this at the end of the 3.1 section: “*The TOC_{520} and MINC_{520} parameters were reproducible and accurate estimators of the $\text{SOC}_{\text{HCl+EA}}$ and $\text{SIC}_{550^\circ\text{C+EA}}$ values for the soil samples as well as for the reference materials including rock and sediment samples.*” (L344-346). However, the PYRO520 is recommended mainly for soil applications because rocks and sediments can contain OM that are more thermos-stable than OM of soils. Thus, even if the organic and inorganic carbon quantification will be correct, the OM characterization of rocks and sediments could be affected by the reduction of the pyrolysis phase. This is why the initial end-of-pyrolysis temperature was set at 650 °C as mentioned in the 1. Introduction section (L94-96).

Minor Comments:

The larger errors for samples with $\text{SOC} \geq 40 \text{ g C kg}^{-1}$ are noted. Please add a brief hypothesis for this observation (e.g., related to more refractory organic compounds like pyrogenic carbon?).

We compared the $\text{SOC}_{\text{HCl+EA}}$ values and the SOC contents estimated as $\text{TC}_{\text{EA}} - \text{SIC}_{550^\circ\text{C+EA}}$ to discuss the performance of the PYRO520 cycle for SOC quantification. As expected, both ways to estimate SOC content by EA are not consistent. We added a discussion in the

3.1 section on the validity of the standard procedure itself: " The larger mean of absolute deviations between TOC_{520} et SOC_{HCl+EA} for soil samples rich in SOC was consistent with the results of Disnar et al. (2003) who reported particularly large deviations between the RE and EA estimations for "biopolymer-rich samples". However, the high SOC content estimations by EA showed also inconsistencies. The mean of the absolute deviation between SOC_{HCl+EA} and the SOC content estimated as $TC_{EA} - SIC_{550^{\circ}C+EA}$ was larger for samples with SOC content $\geq 40 \text{ g C kg}^{-1}$ ($8.53 \pm 7.71 \text{ g C kg}^{-1}$, $n = 11$) than for samples with SOC content $< 40 \text{ g C kg}^{-1}$ ($2.00 \pm 2.22 \text{ g C kg}^{-1}$; Table S1). Several studies reported effects of acid pretreatment on the OM and/or incomplete SIC removal by acid pretreatment that can lead to substantial errors in SOC quantification (Schlacher and Connolly, 2014; Apesteguia et al., 2018; He et al., 2025). Moreover, Apesteguia et al. (2018) pointed out that subtracting carbon contents estimated on different aliquots can lead to inconsistencies in the results. Thus, the remaining discrepancies between SOC_{HCl+EA} and TOC_{520} could be due to underestimation of high SOC contents by RE or analytical errors in the standard procedure itself." (L296-306). We summarized and added perspectives on this discussion in the 4. Conclusions section: "Discrepancies between the TOC_{520} parameter and the SOC_{HCl+EA} values persisted for samples rich in SOC ($\geq 40 \text{ g C kg}^{-1}$). However, it is not straightforward to attribute these discrepancies to either RE analysis or EA analysis on pretreated aliquots. Specific studies should focus on non-calcareous soils rich in SOC to conclude on the RE ability to estimate high SOC content." (L444-447).

The correlation between Cs650 and Cs520 is excellent. A short comment on the model's transferability to the new PYRO520 thermal program would be useful for readers.

The correlation is excellent, but it is not straightforward to understand why the deviation differ depending on the proportion estimated. Actually, we are not sure that the correlation between these predicted indices can be used like the correlations with the calculated indices thus we preferred to remove Cs proportions of the last sentence and to add two sentences on what can be done concerning the Cs proportions: "Since the HI, OIRE6, I, and R indices obtained with the two cycles were linearly correlated, the indices obtained with the PYRO650 cycle could be converted to be compared with those obtained with the PYRO520 cycle. As the PartySOC algorithm is a machine learning model, a specific study should focus on the validation of the correlation presented in this article. Ideally, the PartySOC model could be recalibrated with data obtained with the PYRO520 cycle." (L452-455).