Comments specific to chapter 3.1 (µ-XRF part)

There are multiple major issues here:

- In figure 1 and 2, you did not state the criteria which decide if a element is 'present' or 'absent'.
- The selection of elements spanning the x-axis does not match the collection of maps in the supplement. In addition, the collection of maps is also not consistent over all samples. And the maps itself do not back the present/absend matrices
- The selection of elements seems random. Are those the elements which were presented as most likely matches by the software? However, this selection is not backed by geochemical considerations. In the revision, please include due to their ion radius likely elements in feldspars, like strontium and rubidium while excluding unlikely elements like for example copper
- The selections of elements in figure 1 and 2 do not match.
- The µ-XRF maps in the supplement lack units for their x-, y-axis and scales
- You did not state how the pixel values of the element maps were calculated from the XRF-spectra, please clarify this. I guess the software did not perform a spectrum deconvolution, which likely would have required a set of expected elements, but did return just the signal intensities at the dominating K-alpha or L-alpha lines of the specific elements. However, this approach can lead to severe misinterpretations if the data is not reviewed carefully.
- This considered, the uranium map shown in figure S3 and the uranium presence shown in figure 1 are probably not valid. An 10 keV synchrotron beam can only exicate the M shell of uranium, which by the way is indicated by the small plot title "U_M" in figure S3. However, the sensitivity of M-alpha is very weak. But the photon energy of uraniums M-alpha is very close to the K-alpha line of potassium. Thus, I'm very sure your uranium signals are ghosts, arising from the strong potassium signals. If you compare the "K_K" map with the "U_M" map in S3, this is also very obvious
- There also other possible ghost signals in the data sets: For example, K-alpha of titatinum has nearly the same photon energy as L-alpha of barium. In order to distinguish between them you have to look also at L-beta of barium. Other example: K-beta of Fe overlaps K-alpha of Co. Thus, strong iron signals may lead to false-positive detection of cobalt.

All this considered, the µ-XRF data needs to be re-analysed and the maps and figures reworked. Please consider the data analysis approach used in your own workgroup previously, see Sontag-Gonzales et al. (2024). In addition, I recommend to read through Riedesel et al. (2026), DOI:10.1007/s00269-026-01345-z. Especially their figure 2 shows, how geochemistry correlations of feldspars can also be resolved. Regarding your selection of elements, I recommend to go just with the elements used by Riedesel et al.

(2026) and those listed by the reference compositions stated in table S1. Taken the limitations arising from your µ-XRF set-up into account, this leaves you with Ca, K, Fe, Mn and *maybe* Ba and Ti, if you can ensure they are spectrally resolved. In case you switch from your yes/no matrices to an more quantitative display, you may also add Si and Al.

Regarding the supplement. Please ensure that the µ-XRF maps meet basic scientific standards and correlate to the findings and figures in the manuscript. In addition, I recommend to add one full set of maps for one aliquot of X7363 and X7368 each to the supplement. Also, please add some example XRF spectra of points marked in the maps to the supplement.

Specific/technical comments

Line 41: Please refer also to the photon energy of the 880 nm emission

Line 50: You refer to the 'natural' RF curve but you did not define beforehand that (1) returns you the natural RF curve

Line 54: I guess you referred to Frouin et al. (2015) instead of Frouin et al. (2017)

Line 60: Please add a single sentence of the origin of the IR-RF signal according to the band model. Something like: "According to band model used in the early studies, the IR-RF signal arises from

Line 60/61: The sentence does not sound scientific. I suggest something like: "At the onset of the DRC, the signal intensity is at maximum … etc."

Line 108: Please refer to the SBU albite the same style as the other samples, by writing for example "Albite SBU Amelia". Also, is there a reference to this sample?

Line 117: Again, please keep the style consistent, by writing for example: "… sanidine FCs (An1Ab24Or2; Zimmerman and McIntosh 2011) from Fish Canyon Tuff (Cebula et al. 1986)"

Line 129: Small writing error: "… significance to Pleistocence …"

Line 143: "… they targeted …". To which work do you refer? Sontag-Gonzales et al. or Nazaretzki et al.? Please clarify in the text.

Line 153: Please put "at excitation energies of" before "10 keV" to prevent a misinterpretation that you mapped the 10 keV fluorescence

Line 174: I suggest to add the preferred wavelength ranges of the PMTs

Line 175: Did you mean IRSL instead of IRPL? If not, aren't the RF PMT better suited to the NIR emission of IRPL?

Line 179/180: "…, which has been reported to … light sensitivity". Please, either provide a reference or remove this half-sentence. I suggest to remove it, as this statement should be part of either the introduction or discussion.

Line 192 – 194: I would not list the used lenses here as this is usualy not done in publications like these. Is this the same optic as used in Kumar et al. (2026)? However, you could add the lens information to the caption of supplement figures S1 along with a statement that "reflection and transmission loses caused by the lenses are not taken inte account"

Line 201: Not an issue with this manuscript but a technical recommendation: Those cameras support lower readout rates with lower readout noise. The frame-transfer will be slower, but given you channel time of 5 sec, there is sufficient time available to improve the signal-to-noise ratio.

Line 267: So, the measurements were done after bleaching with settings like described in Frouin et al. (2017)? And figure 4 displays RF_reg curves of single grains measured with the NIR-sensitive PMT? And did you use the SBU or the Oxford device? Please clarify in the text.

Figure 4: The curve describtions you did in line 273 to 284 are hard to follow when looking at the graphs. A lin-log or log-log scaling of the axes would be better for this purpose. However, I admit that changing the axes of fig. 4 and 5 would make the diagrams styles worse and harder to grasp. Please consider to add such figures to the supplement.

Figure 4: The y-axis title states a bin width of 2 Gy, which is a channel time of 10 sec if this is the SBU device. Is this correct? This info don't has to be part of the caption but should be mentioned in line 267.

Figure 4: Please add more information to the caption (bleached, single-grain, PMT, RF70 protocol) and add the bandpass width of the filters (850/30 instead just 850).

Line 301: The behaviour of which particular curves do you mean? 850 nm of sanidine? Please clarify.

Line 303 – 311: You did already a curve discussion in lines 273 to 284. It would serve the readability and your argumentation string (line 312 builds upon line 302) if you in incoperate this paragraph into the previous curve discussion in a brief manner.

Line 315: Please remove "all"

Figure 5 : Please add the background curve if available or a short statement that no background correction was done. Please state also, that these are single-grain measurements.

Line 320: Please correct meaning regarding "850-880nm"

Line 327: Please write “K, Na and ..” and “ ... and lead (Pb)”.

By the way, as you could not detect Sr and Pb appearance (and others) due to the too low excitation energy of your µ-XRF setting, you may discuss this briefly in the discussion.

Line 329: Please provide a reference about the effects of inclusions in volcanic feldspars.

Line 336: Please write “decreasging-signal grain population”.

Line 351: Interesting idea! Please add the equation from Erfurt and Krbetschek (2003) here. Please add also the actual equation of how to calculate D95 from the fitting parameters of the Erfurt-Krbetscheck equation.

Supplement figure S8: I recommend to print the fitting parameters as well as R^2 and D95 to this figure. In addition, I recommend to add the same fitting diagrams for the other two samples shown in figure 6 to S8.

Line 373: Which SLS settings did you use? If you used those of Frouin et al. (2017), why the longer bleaching?

Figure 7: Please add to caption that this are IR-RF measurements with the 850/30 (?) filter and that the grain was bleached for 6h between each cycle.

Figure 8: It would be nice to have D95 points plotted in this figure. Please add also a short information about IR-RF with 850/30 filter and no backround correction to the caption.

Line 404: I wonder if stretched exponential fitting is still suitable for the microline + three-sanidine aliquot? The curve goes up after ~ 1500 Gy. This can not be modelled by a stretched exponential. How does this effect the D95 value? Please elaborate.

Line 441: "X7468" is probably a typo for "X7368"

Line 452: I suggest to include figure S11 in the main text. The figure is too interesting to be hidden in the supplement. I recommend to also plot D95 points here. If you decide against this suggestion, some other figure, for example a D95 distribution, would be nice to underpin your main conclusion.

Line 462: I already wondered, figure 3 appeared misplaced earlier in the text. Just a suggestion: Maybe it fits better here?

Line 478/479: But why could red TL be an explanation for sanidines IR-RF behaviour? I do not see the phyisical link here. Please explain closer or remove this sentence.

Line 501: There is no Fig. 08 or is Fig. 3 meant? However, sorting the (newly analyzed) µ-XRF occurences into the IR-RF curve categories is a good idea.

Line 520: You conclusion about the value of D95 is reasonable. But D95 depends on the validity of the fitting model. A stretched exponential might not be a good representation of an IR-RF curve in all cases. And in cases were it is not, D95 would contain a large systematic error. Thus, in order to be statistically valuable, D95 should come with a statistical measure of likelihood, like a p-value, a Chi^2 or a residual sign test, I don't know what suits best. It is not necessary to provide such a measure in *this* manuscript, however you should at least discuss it in one or two sentences.

Line 534: Please make clear, that the conclusions in this paragraph are only valid for SR IR-RF measurements and not for multi-grain PMT measurements. Even more, your conclusions are excellent arguments in favor of SR IR-RF.

Line 551: You *did* develop a "quantitative, transferable screening criterion" with D95. Please consider to rephrase this sentence.
