

The Forensics of the Dutch Apollo 11 Goodwill Moon Rocks Using Advanced X-ray Imaging and Spectroscopy

Corresponding Author: Dr Thomas Zillhardt

This file contains all editorial decision letters in order by version, followed by all author rebuttals in order by version.

Attachments originally included by the reviewers as part of their assessment can be found at the end of this file.

Version 0:

Decision Letter:

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Dear Dr Zillhardt,

Please allow us to sincerely apologise for the long delay in sending a decision on your manuscript titled "The Forensics of the Dutch Apollo 11 Goodwill Moon Rocks: how correlative high-resolution CT and multi-spectral imaging helped solve a mystery". It has now been seen by 2 reviewers, and we include their comments at the end of this message. They find your work of interest, but some important points are raised. We are interested in the possibility of publishing your study in Communications Earth & Environment, but would like to consider your responses to these concerns and assess a revised manuscript before we make a final decision on publication.

We therefore invite you to revise and resubmit your manuscript, along with a point-by-point response that takes into account the points raised. Please highlight all changes in the manuscript text file.

We are committed to providing a fair and constructive peer-review process. Please don't hesitate to contact us if you wish to discuss the revision in more detail.

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We hope to receive your revised paper within six weeks; please let us know if you aren't able to submit it within this time so that we can discuss how best to proceed. If we don't hear from you, and the revision process takes significantly longer, we may close your file. In this event, we will still be happy to reconsider your paper at a later date, as long as nothing similar has been accepted for publication at Communications Earth & Environment or published elsewhere in the meantime.

Please do not hesitate to contact us if you have any questions or would like to discuss these revisions further. We look forward to seeing the revised manuscript and thank you for the opportunity to review your work.

Best regards,

Joe Aslin

Deputy Editor,
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- Life sciences

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In particular, the Data availability statement should include:

- Unique identifiers (such as DOIs and hyperlinks for datasets in public repositories)
- Accession codes where appropriate
- If applicable, a statement regarding data available with restrictions
- If a dataset has a Digital Object Identifier (DOI) as its unique identifier, we strongly encourage including this in the Reference list and citing the dataset in the Data Availability Statement.

DATA SOURCES: All new data associated with the paper should be placed in a persistent repository where they can be freely and enduringly accessed. We recommend submitting the data to discipline-specific, community-recognized repositories, where possible and a list of recommended repositories is provided at <http://www.nature.com/sdata/policies/repositories>.

If a community resource is unavailable, data can be submitted to generalist repositories such as [figshare](https://figshare.com/) or [Dryad Digital Repository](http://datadryad.org/). Please provide a unique identifier for the data (for example a DOI or a permanent URL) in the data availability statement, if possible. If the repository does not provide identifiers, we encourage authors to supply the search terms that will return the data. For data that have been obtained from publically available sources, please provide a URL and the specific data product name in the data availability statement. Data with a DOI should be further cited in the methods reference section.

Please refer to our data policies at <http://www.nature.com/authors/policies/availability.html>.

REVIEWER COMMENTS:

Reviewer #1 (Remarks to the Author):

Nicely done study and very interesting topic. There are a few minor typos here and there but overall, nicely written and well-organized. I would agree with the conclusions that the Dutch goodwill samples are indeed of lunar origin and all data points to alignment with the Apollo 11 VHT basalts.

One aspect that puzzled me was the inclusion of the section on digital tomosynthesis. It is interesting to try it out for this application, but the results from that are not conclusive and not close to the resolution or quality as obtained by x-ray CT.

Worth giving it a shot, but I'd say since you have more complete data with the CT, I'd recommend just eliminating that section.

I also recommend removing or revising the first paragraph of the Discussion section (chapter 3). That paragraph stood out to me as more of a commentary on geopolitical topics rather than the science of your work.

Overall nice work and hope your department of state is appreciative that you confirmed the rocks are legitimate!

Reviewer #2 (Remarks to the Author):

The article needs major revisions in the methodology and result interpretation part. Detailed review is attached in .pdf file.

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Version 1:

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Dear Dr Zillhardt,

Your manuscript titled "The Forensics of the Dutch Apollo 11 Goodwill Moon Rocks: how correlative high-resolution CT and multi-spectral imaging helped solve a mystery" has now been seen again by Reviewer #2, and we include their comments at the end of this message. They find your work of interest, but retain some important points. We remain interested in the possibility of publishing your study in Communications Earth & Environment, but would like to consider your responses to these concerns and assess another revised manuscript before we make a final decision on publication.

In particular, we will need to see that any revised manuscript meets the following editorial thresholds:

* Present a compelling case that the interpretation of your data are robust, including:

- 1) clarification of the handling of radiography projection data
- 2) clearly accounting for the potential for movement artifacts in your conclusions
- 3) the inclusion of technical information in the main manuscript and
- 4) the removal of interpretations based on the stone morphologies from tomography data

We therefore invite you to revise and resubmit your manuscript, along with a point-by-point response that takes into account the points raised. Please highlight all changes in the manuscript text file.

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Important: The response to reviewers must not include any figures, tables or graphs. If you wish to respond to the reviewer reports with additional data in one of these formats, please add them to the main article or Supplementary Information, and refer to them in the rebuttal. Due to current technical limitations, any figures, tables, or graphs embedded in your rebuttal will not be included in the peer review file, if published.

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REVIEWER COMMENTS:

Reviewer #2 (Remarks to the Author):

I recommend the manuscript for publication after minor revisions.

The authors reflected on some of the points raised by the reviewers and improved the quality of the text. Several questions were solved by removal of technical details instead of corrections which I do not consider to be a suitable solution.

Nevertheless, technical data can be found in references and partially in supplementary materials.

I still think that the statement about clear observation of edges of stones from tomosynthesis measurement is too bold and should be removed (page 11).

Figure 5b: the caption is misleading. The image on the left presents transmission image recorded by the detector. Used terminology do not explain data manipulation (inversion) as absorption is just combined effect of attenuation and scattering. Supplementary materials refer to Fig. 5 as absorption to transmission conversion based on Beer-Lambert law. I think that the opposite is true as transmission recorded by the detector is converted to something else.

Supplementary materials:

The part with elastic bands holding the sample raises question about the achievable resolution of 2.37 μm . Moreover, the presented slices exhibit movement artefacts that might lead to wrong conclusions. Even advanced movement correction algorithms would struggle with random sample movement.

Added table with no caption and reference – last line states Exposure time - check – check

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Dear Dr Zillhardt,

Your manuscript titled "The Forensics of the Dutch Apollo 11 Goodwill Moon Rocks Using Advanced X-ray Imaging and

Spectroscopy" has now been seen by our reviewers, whose comments appear below. In light of their advice we are delighted to say that we are happy, in principle, to publish a suitably revised version in Communications Earth & Environment.

We therefore invite you to edit your manuscript to comply with our format requirements and to maximise the accessibility and therefore the impact of your work.

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REVIEWERS' COMMENTS:

Reviewer #2 (Remarks to the Author):

I recommend the manuscript for publication.

The authors reflected on the points raised by the reviewers and improved the quality of the text.

The supplementary materials are in agreement with the statements in the manuscript.

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The investigated GLSD sample is extremely interesting piece of history, and I would like to split the review into two parts as the article showed fingerprints of several authors. The article needs major revisions in the methodology and result interpretation part.

The claims in the parts dealing with the origin and mineralogy of the sample are convincing and properly referenced.

Questions and comments:

121 – Both UP and PMMA ... causing yellowing minimal at radiation ... the sentence should be rephrased.

Figure 2: The labels of the axes are truncated, Y axis changes from normal to log, Wavelength (keV) should be replaced by Energy (keV) in the whole paper. The plot is unfortunate choice as it makes an impression of measured spectrum of dust batch n°100085,11 with ideal detector (no energy spread). A table would provide all necessary information here.

The second part relates to the methodology and result interpretation. This part is supported with supplementary materials document. Three methods were used for the inspection of the sample – PDT, radiography/CT and XHI. Overall assessment of techniques is followed by few specific questions and comments.

PDT

Data quality provided by PDT is quite low and the statements like “The edges are sharp, testifying a legacy of catastrophic fracture and a lack of erosion” are too bold. The use of PDT seems redundant here as radiography presented later provides better resolution and just few projections give similar idea of the distribution of 4 samples. Conclusions about samples should be based on micro-CT.

Macro and micro tomography

The results of macro tomography show basic internal structure of the samples. Some details about segmentation techniques used should be beneficial as it is not clear whether it was manual or automatic. The presented micro-CT slices are quite small but the data seems to suffer from

reconstruction artefacts (blurred edges, duplicated structures). This might be caused by problems with determining the imaging geometry or by sample movement during CT measurement. Elastic bands were mentioned in supplementary document – dedicated custom-made mounting plate screwed to rotation table is quite common way of solving such challenges.

The resolution of macro and micro CT should be clearly stated to be able to verify claims about sizes of structures and pores.

Questions and comments:

Figure 5: absorption -> attenuation – what transformation has been used?

386 - It is not clear if mentioned 22,7 and 2,37 μm pixel size is before or after binning. How large are voxels after reconstruction?

XHI

The XHI on Timepix3 data shows results of PCA. Better description of the methodology would help with the clarity of the text. The plot in Fig. 8 should be clear evidence, that spectral response at selected locations is different. It should be better explained how the data was processed – raw intensity data -> attenuation and how it was normalised (it seems that background was used but normalised background for energies up to 10 keV are not close to 1).

Questions and comments:

The information about spectra granularity should be consistent:

298 - energy precision of about 2 keV

395 – step size 0.25 keV and an energy spread 2.5 keV.

Supplementary 68 - step size of 0.5 keV and a spread of 2 keV

Figure 8: A and B images are swapped.

Supplementary materials:

Supplementary materials should help with clarification of the text. There are several statements and values contrary to what is stated in the text. Provided detailed descriptions of data manipulation did not answer many question.

Questions and comments:

19 – parameters are in table 5 – no table 5

39 – the sensor is equally eddicient across... (efficient?)

40 - Forward Width Half Maximum (FWHM) -> Full Width Half Maximum

43 – Time-overThreshold -> time-over-threshold (ToT) -> term and its abbreviation repeats (66)

44 – small size (10x10 mm) -> 14x14 mm,

46 – in close proximity to the sample -> good spectral sensitivity... How is spectral sensitivity connected to distance? Good detection efficiency should be here.

this also rises the question about measurement geometry – 70 mm SDD, what was SOD and how large are then structures visible in Fig. 8.

72 - The higher energy signals (about 60 keV) originate from the source setting but the lower energy end of the spectra (3 keV) is a hard lower limit due to charge pile-up.

- X-ray tube was operated at 80 kVp – anything up to 80 keV is OK, anything above should be pile-ups.

- The low energy limit of the detector is set above electronic noise of the device (2.5 – 4 keV for most TPX3 devices).

- I am not sure what charge pile-up means here.

74 – low energy part of X-ray tube spectrum is always dominant. It can be modulated by using some filters. Also tube can be operated at higher current if needed for stability and combined with filter to decrease intensity.

The Forensics of the Dutch Apollo 11 Goodwill Moon Rocks Using Advanced X-ray Imaging and Spectroscopy

FIRST REVISION

COMMSENV-24-0139-T

1 ANSWER TO REVIEWERS

Reviewer 1

The authors would like to thank Reviewer 1 for their kind words of support and are grateful for the recommendations provided. The manuscript has been reviewed with consideration of these recommendations.

Reviewer 2

The authors appreciate the diligent, meticulous, and detailed proof-reading work done by Reviewer 2. Most of the large number of recommended amendments have been implemented and this has certainly improved the quality of the manuscript. Some suggestions however seemed to stem from a lack of understanding, most likely due to our poor description of some of the techniques used, and the authors have addressed these instead by providing better explanations of what we did in the revised text.

2 LIST OF CHANGES

Point	Change	Comment
Reviewer 1		
Minor typos	Additional proof-reading	
Remove tomosynthesis	Reformatted	We believe these datasets bring valuable information to the study.
Remove first paragraph discussion	Removed	
Reviewer 2		
Rephrase sentence starting “Both UP and PMMA”	Rephrased	Does not change the meaning, but aimed at making it easier to understand
Fig 2: labels truncated	No change	Labels are all visible
Fig 2: Y-axis normal/log	Caption changed to help understanding	The left graph is a zoom of the right graph, and the fact the scale is log on the right an linear on the left is added to the caption.
Axis label	Refreshed	
Fig 2: replace with table	No change	We believe that a graph conveys the message in a much clearer way and as it is displayed in the introduction it is not misleading
PDT redundant, low quality, conclusions too bold	Reformatted	We believe these datasets bring valuable information to the study.

Add details about segmentation techniques	Added some details about the segmentation	see supplementary
Data suffers from reconstruction artefacts (blurred edges, duplicated structures)	No change	Due to preservation requirements, mounting options were restricted and the sample moved during scanning. As mentioned, advanced reconstruction techniques
Recommendation on dedicated mounting plates	No change	Noted for future improvement of the analytical setup – it was not possible to do this for the object that is discussed in our paper.
Macro-micro-CT resolution clearly stated	Added to supplementary materials as well	There are scale bars on every figure. Pixel size is also mentioned in the methods section.
Figure 5: absorption -> attenuation – what transformation has been used?	Added to supplementary materials	
386 - It is not clear if mentioned 22,7 and 2,37 um pixel size is before or after binning. How large are voxels after reconstruction?	Specified	
Better description of PCA results needed	See below	
The plot in Fig. 8 should be clear evidence, that spectral response at selected locations is different.	No change	That is the case.

It should be better explained how the data was processed – raw intensity data -> attenuation and how it was normalised (it seems that background was used but normalised background for energies up to 10 keV are not close to 1).	Information added in the supplementary.	Data 4<x<6keV if affected by noise and poor statistics and not directly usable. Data <4keV should be ignored as the detector has poor
The information about spectra granularity should be consistent:	Corrected	
298 - energy precision of about 2 keV	No change	
395 – step size 0.25 keV and an energy spread 2.5 keV.	No change	Energy spread is not the same as energy precision
Figure 8: A and B images are swapped.	Corrected	
Reviewer 2: supplementary		
Supplementary 68 - step size of 0.5 keV and a spread of 2 keV	Corrected	
Supplementary materials should help with clarification of the text. There are several statements and values contrary to what is stated in the text. Provided detailed descriptions of data manipulation did not answer many question.	Corrected	
19 – parameters are in table 5 – no table 5	Added	
39 – the sensor is equally eddicient across... (efficient?)	Corrected	

40 - Forward Width Half Maximum (FWHM) -> Full Width Half Maximum	Corrected	
43 – Time-overThreshold -> time-over-threshold (ToT) -> term and its abbreviation repeats (66)	Changed with abbreviation	
44 – small size (10x10 mm) -> 14x14 mm,	Corrected	
46 – in close proximity to the sample -> good spectral sensitivity... How is spectral sensitivity connected to distance? Good detection efficiency should be here.	Changed	Similar meaning
this also rises the question about measurement geometry – 70 mm SDD, what was SOD and how large are then structures visible in Fig. 8.	No change	There is a scalebar on the figures and the rock dimensions are specified in the text. SOD is not relevant in this setup and was not measured.
72 - The higher energy signals (about 60 keV) originate from the source setting but the lower energy end of the spectra (3 keV) is a hard lower limit due to charge pile-up.	Section removed as confusing and not bringing any useful information after manuscript revision	
- X-ray tube was operated at 80 kVp – anything up to 80 keV is OK, anything above should be pile-ups.	See above	
- The low energy limit of the detector is set above electronic noise of the device (2.5 – 4 keV for most TPX3 devices).	See above	
- I am not sure what	See above	

charge pile-up means here.		
74 – low energy part of X-ray tube spectrum is always dominant. It can be modulated by using some filters. Also tube can be operated at higher current if needed for stability and combined with filter to decrease intensity.	See above	

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FIRST REVISION

COMMSENV-24-0139-T

1 ANSWER TO REVIEWERS

Reviewer 2

We are thankful to Reviewer 2 for their additional recommendations. We have addressed the new comments as detailed below.

2 LIST OF CHANGES

Point	Change	Comment
Reviewer 2		
Criticism of some removal of technical detail in revised version compared to original manuscript	No further change.	We followed the advice of the initial review of our original manuscript where it was found that we had added unnecessary information that did not help understand the methods or the experiment we performed and was instead cluttering the paper. As such we have removed these details which are not critical to the understanding of the work. .
Request removal of tomosynthesis statement	Rephrased.	We do not claim anymore in the new revision that edges are clear, we simply infer a partial conclusion from the DT observations. We hope this is acceptable as it was a useful step in our procedures and we think that others may find it useful although we admit it does not so smoothly integrate into the narrative.
Figure 5b caption considered “misleading”	Caption and supplementary wording inverted.	We inadvertently inverted the caption with supplementary wording during our initial revision and missed this. Thank you for noticing; this has now been corrected.
Supplementary material: comments regarding sample holding and sample movement	No change made	We disagree with the reviewer here. The pixel size quoted is not the same as the image spatial resolution. In cone beam geometry, pixel size is a precise value calculated from the actual physical dimensions of a pixel on the detector sensor and the magnification achieved by the lens + source/sample/detector

		distances. We do not make any claim regarding image spatial resolution in our paper and instead have used scale bars on all figures to help define what features can be resolved. The motion correction employed here uses an image registration technique with sub-pixel accuracy. This compensates for random movements very effectively, and when realigned, it is virtually impossible to differentiate from a dataset without movement. As observed on the radiographies, image blurring is negligible as the exposure time was much faster than the stage rotation speed. Note that all stages and sample holders will introduce vibrations, and it is good practice to use a correction algorithm in every case. The only possible downside is reduced field-of-view at the edges due to realignment, but in this particular study the sample fits well within the field of view. We would be happy to provide a video comparison as well as more details regarding the correction, but we feel that this approach is conventional (used in many other experiments) and falls outside the scope of this paper (see https://doi.org/10.1098/rsta.2020.0192, .
Supplementary: new table – caption missing	Caption added and table filled.	We are sorry that this was not picked up during our revision due to file sharing and file availability issues; we have now added the caption and filled out the table.