Results from analysis of sandstone fragment

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Summary of results

- X-ray fluorescence results are consistent with the sandstone fragment being derived from the Stone of Destiny.
- Colour analysis results are consistent with the sandstone fragment being derived from the Stone of Destiny.
- Scanning electron microscopy results are consistent with the sandstone fragment being derived from the Stone of Destiny.
- Microscopic examination results are consistent with the sandstone fragment being derived from the Stone of Destiny.

1 Introduction

The purpose of the analyses reported here was to investigate the characteristics of a sandstone fragment and compare it to previous analyses of the Stone of Destiny.

Previous analyses used in this comparison were non-destructive scientific investigations of the Stone of Destiny by Historic Environment Scotland's (HES) Science Team in 2023 (Report XRF090/MEY/2306 "XRF090 2023 Stone of Destiny") and a published paper on the composition of the Stone of Destiny with respect to local Perthshire sandstones by Fortey et al., 1998[1].

2 Methodology

2.1 Portable X-ray fluorescence

Portable X-ray fluorescence (pXRF) equipment was a Bruker Tracer 5g. Analysis was non-destructive.

XRF analysis from ten locations on the fragment was carried out using Bruker's GeoExploration calibration application, method "Oxide3phase". The analysis period was 90 seconds (30 seconds each at the following three settings):

• 30 kV, 13.8 μA	Ti 25 μm + Al 300 μm filter	8 mm collimator
• 50 kV, 23.4 μA	Cu 75 μm + Ti 25 μm + Al 200 μm filter	8 mm collimator
• 15 kV, 11.55 μA	no filter	8 mm collimator

[1] Fortey, Phillips, McMillan and Browne, 1998. A geological perspective on the Stone of Destiny. Scottish Journal of Geology, 34(2), 145-52. Fragments from the Stone of Destiny underwent detailed examination by the British Geological Survey in 1998, two years after the Stone returned to Scotland. This work identified the Stone as being indistinguishable from sandstones of the Scone Sandstone Formation, which outcrop in the area around Scone Palace, near Perth.

The GeoExploration calibration results were further processed using pXRF results from analysis of thirteen powdered calibration standards[2] to check and amend the accuracy of coefficients in the GeoExploration calibration.

The same methodology was used in analysis of the Stone of Destiny in January and November 2023.

The composition of the Stone of Destiny is based on 35 individual analyses at various locations on its surface.

The composition of the fragment is based on eight individual analyses at various locations on its surface. Only eight analyses were possible due to the small size of the fragment (<3 cm in length). Consequently, the analysed composition of the fragment may not be consistently representative of the stone from which it was derived.

Key to chemical elements

Al: aluminium	As: arsenic	Ba: barium	Ca: calcium
Cl: chlorine	Cr: chromium	Cu: copper	Fe: iron
K: potassium	Mg: magnesium	Mn: manganese	Ni: nickel
O: oxygen	P: phosphorus	Pb: lead	Rb: rubidium
Si: silicon	Sr: strontium	Ti: titanium	V: vanadium
Zn: zinc	Zr: zirconium		

2.2 Colour analysis

Colour analysis was carried out using a Konica Minolta chroma meter CR-400. Illuminant D65. Colour system L*a*b*. Measured area 11 mm diameter.

2.3 Scanning electron microscope (SEM) analysis

Analysis by scanning electron microscopy (SEM) provides high resolution microscopic analysis of the fragment. Quantitative analytical data on chemical elements in the fragment are compared with data obtained from pXRF analysis.

Analysis was carried out using a JEOL JSM IT200 floor standing scanning electron microscope equipped with a backscatter electron detector (BSED), secondary electron detector (SED) and integrated JEOL energy dispersive spectroscopy detector (EDS).

The EDS detector is sensitive to elements from beryllium (Be) to uranium (U).

The fragment was positioned within its original packaging foam and was connected to a large aluminium SEM sample holder using carbon tape. To assist in the dissipation of charged electrons from the fragment, copper tape was used to connect the sample surface to the sample holder. The fragment was uncoated.

[2] SIEM-10: Kupferscheiefer, SIEM-02: Cordierite Gneiss, SIEM-06: Gabbro, SIEM-01: Granite, SIEM-05: Greywacke, SIEM-08: Melilite Basalt, SIEM-04: Nosean Phonolite, SIEM-03: Peridotite, SIEM-09: Siltstone, SIEM-07: Tonolite, GBW07103: Biotite granite (GSR-1), GBW07406: Soil (GSS-6), CS-M2: Bruker GeoExploration standard.

Analysis was carried out under low vacuum (30 Pa) at the following energy settings:

- Accelerating voltage: 15kV
- Probe current: 60 μA
- Working distance: 9.2 mm – 12.7 mm
- Image detector: Backscatter electron (composition mode)

2.4 Stereo microscope analysis

The fragment was examined using an Olympus SZ61 binocular microscope, at magnifications up to 45 times. The microscope was attached to an Olympus SC30 camera for photography of the fragment's grain structure.

2.5 USB microscope analysis

Non-destructive microscopic analysis provides information on the fragment mineralogy and texture, particularly grain size, shape and compaction. It has been used to compare data from the fragment with the Stone of Destiny.

A Dino-Lite Edge AM7915MZT USB microscope attached to a laptop was used to take high magnification images of the fragment. The microscope was calibrated using a supplied scale card prior to analysis.

The same methodology was used in analysis of the Stone of Destiny in 2023.

3 Results

3.1 Portable X-ray fluorescence (pXRF)

Bulk chemical analysis of the Stone of Destiny and the sandstone fragment is shown in Table 1.

The amounts of 21 chemical elements in the fragment were compared to those in the Stone of Destiny (Appendix A). The aim was to determine whether the amounts of each chemical element are consistent or inconsistent with the fragment having been derived from the Stone of Destiny.

For most chemical elements the mean composition of the fragment was consistent with analysis of the composition of the Stone of Destiny, i.e. within standard deviations for the Stone of Destiny results. This applies to Al, As, Ba, Cl, Fe, Mg, P, Pb, Rb, Sr, V and Zr (see Table 1).

For chemical elements where the mean composition of the fragment was not within the standard deviation of the Stone of Destiny results (see Table 1), it should be noted that, due to its small size (< 3 cm in length), the analysed composition of the fragment may not be consistently representative of the stone from which it was derived. This is especially the case for trace elements, where amounts detected may be strongly influenced by the composition of individual grains within the stone.

The following elements' results were not within standard deviations for the Stone of Destiny results:

- Ca (as CaO): The fragment contained higher levels than were calculated for the Stone of Destiny. However, it should be noted that the surface of the Stone of Destiny contained varying amounts of gypsum ($CaSO_4.2H_2O$) due to surface contamination from an unknown source. Consequently, the expected, natural CaO content of the Stone of Destiny is based on a theoretical calculation, removing gypsum from the analyses results. This introduces inaccuracy to the calculated CaO content of the Stone of Destiny, making comparative analysis of CaO difficult. Additionally, it is possible that soluble calcium compounds on, or near, the Stone of Destiny surface have been lost by dissolution, which would not necessarily be expected to affect stone from the interior (which the fragment may be).
- Cr: Trace element. The fragment contained slightly higher levels than the Stone of Destiny. Compositional difference likely due to natural variation in the stone.
- Cu: Trace element. The fragment contained higher levels than the Stone of Destiny. Compositional difference likely due to natural variation in the stone.
- K (as K_2O): The fragment contained slightly lower levels than the Stone of Destiny. The amount of K_2O in samples from the Stone of Destiny was highly variable. Results from the fragment are consistent with being within this natural variation.
- Mn (as MnO): The fragment contained higher levels than the Stone of Destiny. This may be caused by natural, localised variation in stone composition.

- Ni: Trace element. The fragment contained higher levels than the Stone of Destiny. This difference can be caused by natural, localised variation in stone composition.
- Si (as SiO_2): The fragment contained slightly lower levels than the Stone of Destiny. The amount of SiO_2 in samples from the Stone of Destiny was highly variable. Results from the fragment are consistent with being within this natural variation.
- Ti (as TiO_2): The fragment contained slightly higher levels than the Stone of Destiny. The amount of TiO_2 in samples from the Stone of Destiny was highly variable. Results from the fragment are consistent with being within this natural variation.
- Zn: Trace element. The fragment contained slightly lower levels than the Stone of Destiny. The amount of Zn in samples from the Stone of Destiny was highly variable. Compositional difference likely due to natural variation in the stone.

Taking all pXRF results into consideration, it is concluded that the results over all are consistent with the sandstone fragment being derived from the Stone of Destiny.

Table 1. Normalised bulk chemical analysis of the Stone of Destiny and the fragment. SEM EDS data (not normalised) are described in Section 3.3 (calcium-rich minerals and inclusions have been excluded from this data). ND = not detected, NA = not analysed. Major oxides are the main constituents of the stone, trace elements are present in very small amounts, generally <0.02%.

	Stone of Destiny (pXRF)		**Fragment (pXRF)**		**Fragment (SEM EDS)**	
Major oxides	**Mean (%)**	***Standard deviation***	**Mean (%)**	***Standard deviation***	**Mean (%)**	***Standard deviation***
MgO	6.1	*1.3*	7.3	*0.7*	6.2	*1.1*
Al_2O_3	13.0	*1.4*	12.8	*0.8*	13.7	*1.6*
SiO_2	73.8	*2.9*	70.4	*2.5*	60.7	*6.2*
P_2O_5	0.05	*0.05*	0.003	*0.006*	ND	
Cl	0.2	*0.1*	0.3	*0.09*	0.8	*0.3*
K_2O	2.1	*0.3*	1.8	*0.1*	2.9	*0.5*
CaO	0.3	*0.1*	1.4	*1.0*	2.3	*1.8*
TiO_2	0.5	*0.1*	0.6	*0.08*	0.9	*0.3*
MnO	0.05	*0.01*	0.12	*0.01*	NA	-
Fe_2O_3	3.8	*0.9*	4.6	*0.3*	6.3	*1.3*
Trace elements	**Mean (ppm)**	***Standard deviation***	**Mean (ppm)**	***Standard deviation***	**Mean (ppm)**	***Standard deviation***
V	147	*33*	132	*0*	NA	-
Cr	108	*88*	200	*90*	NA	-
Ni	34	*11*	80	*9*	NA	-
Cu	65	*30*	142	*9*	ND	-
Zn	151	*57*	67	*7*	NA	-
As	22	*22*	8	*1*	NA	-
Rb	59	*8*	55	*3*	NA	-
Sr	143	*25*	151	*19*	NA	-
Zr	161	*43*	160	*38*	NA	-
Ba	662	*105*	618	*52*	NA	-
Pb	152	*170*	82	*35*	NA	-

3.2 Colour analysis

Colour analysis at six locations on the sandstone fragment (areas with no visible soiling) were compared to data from the Stone of Destiny (Figure 1). The aim was to determine whether the colour is consistent or inconsistent with the fragment having been derived from the Stone of Destiny.

As fragment colour data plot within the spread of data from the Stone of Destiny, the results are consistent with the sandstone fragment being derived from the Stone of Destiny.

Figure 1a. Colour analysis of the fragment and Stone of Destiny. The x-axis shows lightness (L) and the y-axis shows green-red coloration. The data are consistent with the fragment being derived from the Stone of Destiny.*

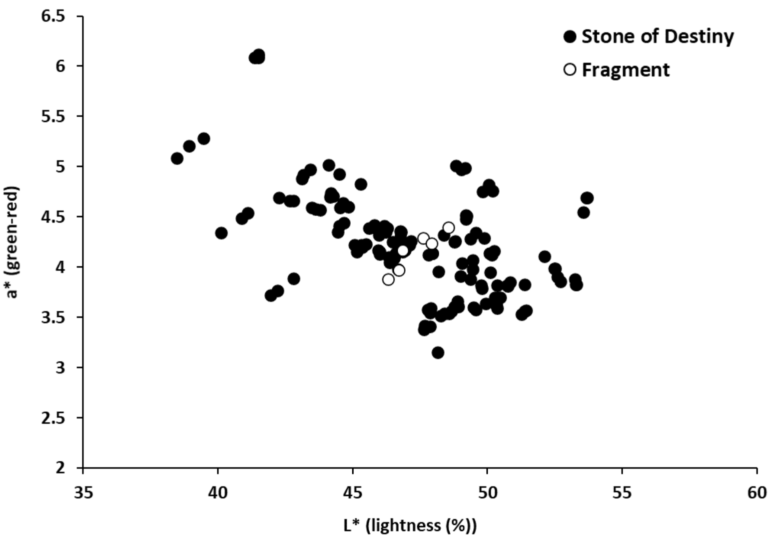
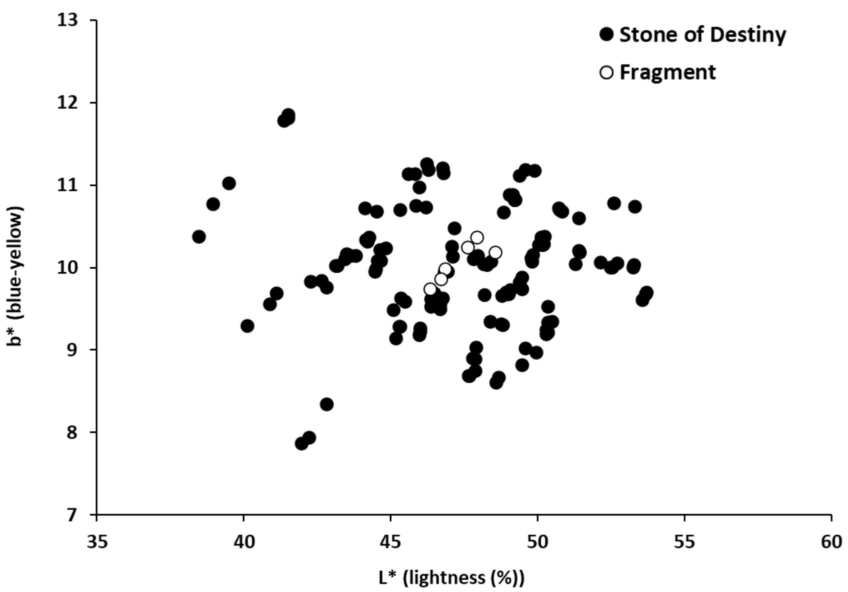


Figure 1b. Colour analysis of the fragment and Stone of Destiny. The x-axis shows lightness (L) and the y-axis shows blue-yellow coloration. The data are consistent with the fragment being derived from the Stone of Destiny.*



3.3 Scanning electron microscope (SEM) analysis

SEM analysis, using energy dispersive spectrometry (EDS) mapping, provides information on the distribution of chemical elements within the sandstone fragment (Appendix B1 to B4). This provides additional chemical data for comparison with pXRF results.

Minerals identified by SEM and EDS mapping include quartz, mica, clays, Na-feldspar (albite), K-feldspar and magnesium aluminosilicate minerals (e.g. pyroxene, amphibole or other mafic minerals). These results are consistent with observations on fragments from the Stone of Destiny by Fortey et al, 1998[3] (historic samples from the Stone of Destiny held by British Geological Survey (BGS)).

SEM analysis indicates the presence of silicious overgrowth cements (probably quartz and feldspar), clay and calcium-rich intergranular material (Appendix B5 to B7). The presence of intergranular clay is consistent with observations by Fortey et al., 1998. While the presence of calcium-rich intergranular material may be consistent with the presence of calcium carbonate cement, there is no evidence to suggest that the fragment is significantly calcareous.

EDS data from 10 regions on Sides A and B of the fragment (Appendices B and C) are consistent with pXRF analysis of the bulk chemistry of both the Stone of Destiny and the fragment (see Section 3.1, Table 1).

Variations between SEM EDS and pXRF data may be influenced by several factors, including:

- Difference in the size of the analysis area: the largest EDS mapping area was approximately 9 mm^2, the analysed area with the pXRF was approximately 50 mm^2.
- SEM EDS and pXRF analysis were undertaken at different energy settings, detecting a different range of elements.
- SEM EDS analysis was undertaken in non-ideal conditions: non-coated samples under low vacuum at varying working distances (occasionally at non-optimal settings).

Results are consistent with the sandstone fragment being derived from the Stone of Destiny.

[3] Fortey, Phillips McMillan and Browne, 1998. A geological perspective on the Stone of Destiny. Scottish Journal of Geology, 34(2), 145-52.

3.4 USB and stereo microscope analyses

The sandstone fragment is fine to medium-grained and appears relatively well graded over the size of the fragment. It has a similar grain size to the Stone of Destiny (fine to medium-grained) with a mean grain size of approximately 0.27–0.28 mm, ranging from 0.14 mm to 0.47 mm (Figures 2 and 3). This is consistent with observations of the Stone of Destiny by Fortey et al., 1998 (mean grain size 0.2-0.3 mm).

The fragment stone appears relatively well compacted. It is both grain and matrix supported, containing both point and line grain contacts, with indiscernible intergranular cement which varies from white to light grey in colour. Individual grains appear spherical to elongate and sub-angular to sub-rounded in shape. These textural observations are consistent with the Stone of Destiny.

Optical microscopy indicates that the fragment has a mineralogy consistent with the Stone of Destiny. It is characterised by pinkish and iron-stained, grey and glassy quartz and feldspar grains (SEM analysis indicating that the feldspar is Na and K-bearing), mica (tentatively identified in the forms of biotite, phlogopite and muscovite), and dark, indiscernible grains or lithic fragments. A large patch of orange-brown staining on Side B of the fragment (Figure 4 and Appendix C, Figure C2) is consistent with similar observations (presumed to be iron staining) from the Stone of Destiny.

Both the analysed fragment and the Stone of Destiny show consistency in their grain size, shape, compaction and visual mineralogy.

Both the fragment and the Stone of Destiny contain consistently sized, shaped, compacted and coloured quartz and feldspar grains, mica flakes and dark indiscernible grains or lithic fragments.

The fragment is visually consistent with the Stone of Destiny in terms of mineralogy, grain colour, grain size, grain shape, grain compaction – including consistency in the intergranular material in detrital grains.

Results are consistent with the sandstone fragment being derived from the Stone of Destiny.

Figure 2a. Stone of Destiny, Top surface (sample area 1173).

Figure 2b. Fragment (Side A, image 1). Both images were taken under similar magnifications and conditions. Images taken with Dino-Lite USB microscope.

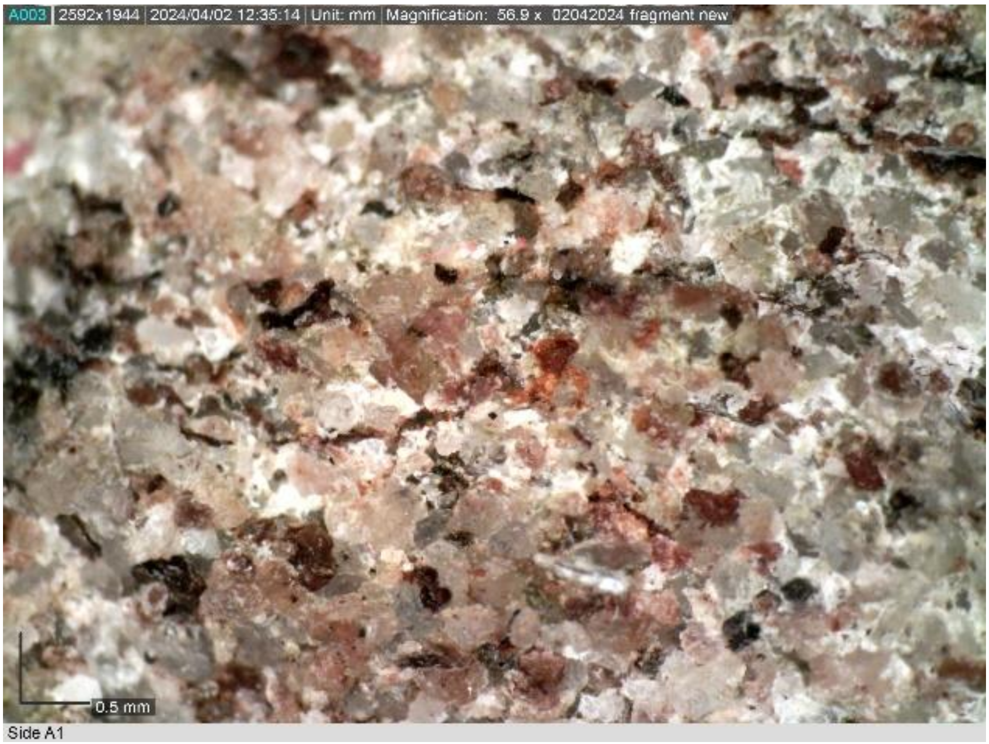


Figure 3a. Stone of Destiny, Top surface (sample area 1176).

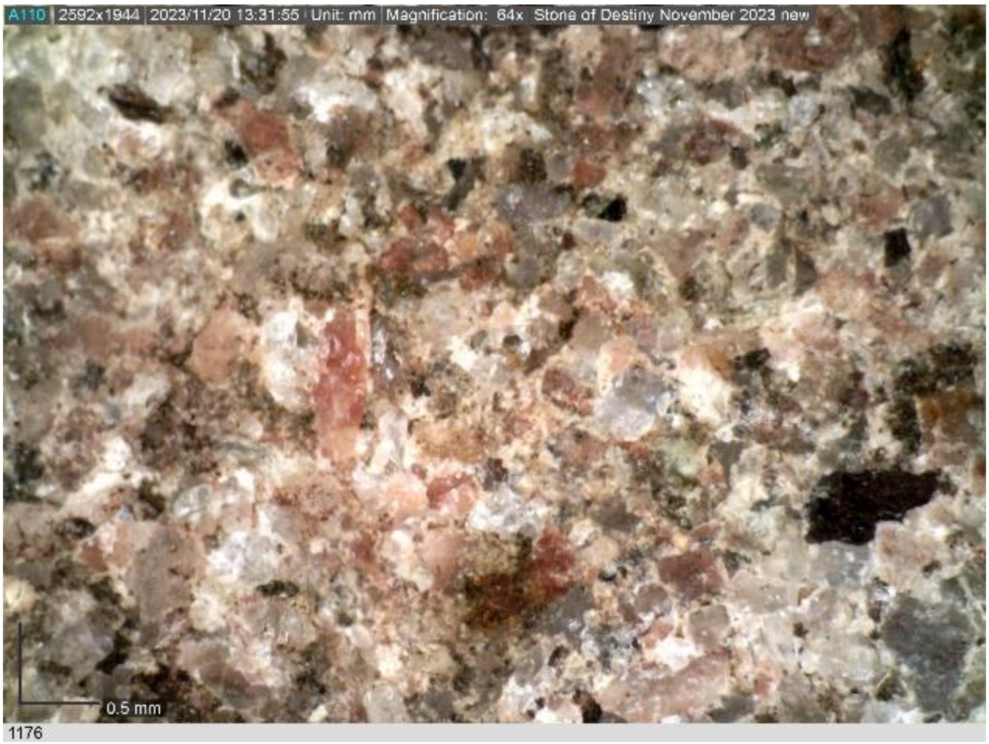


Figure 3b. Fragment (Side A, image 2). Both images were taken under similar magnifications and conditions. Images taken with Dino-Lite USB microscope.

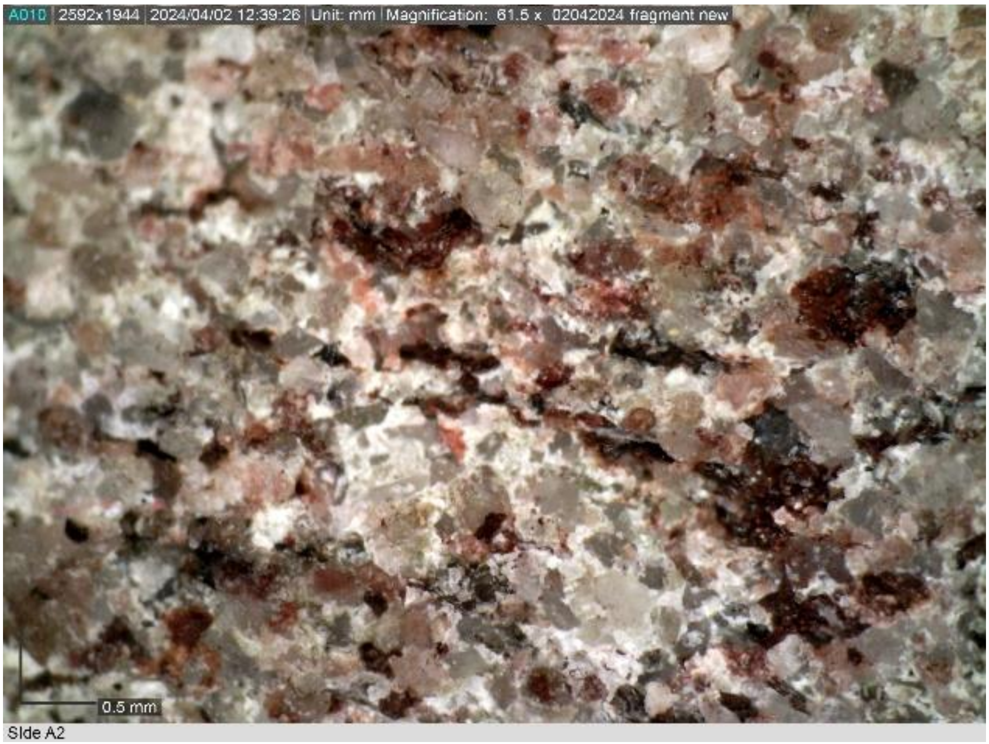


Figure 4. Stereo microscope image of fragment. The orange-brown patch (lower centre) is an area of probable iron oxide staining on the fragment.

Appendix A. Portable X-ray fluorescence data comparing composition of the fragment with the Stone of Destiny

Figure A1. Comparison of amount of aluminium oxide (as Al_2O_3) in Stone of Destiny and fragment.

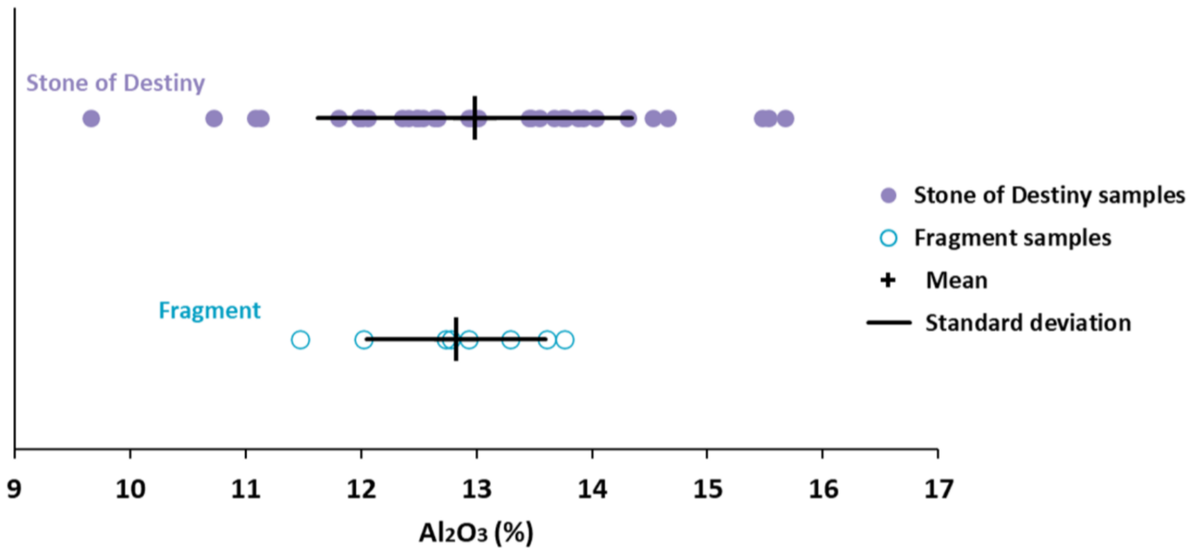


Figure A2. Comparison of amount of arsenic (As) in Stone of Destiny and fragment.

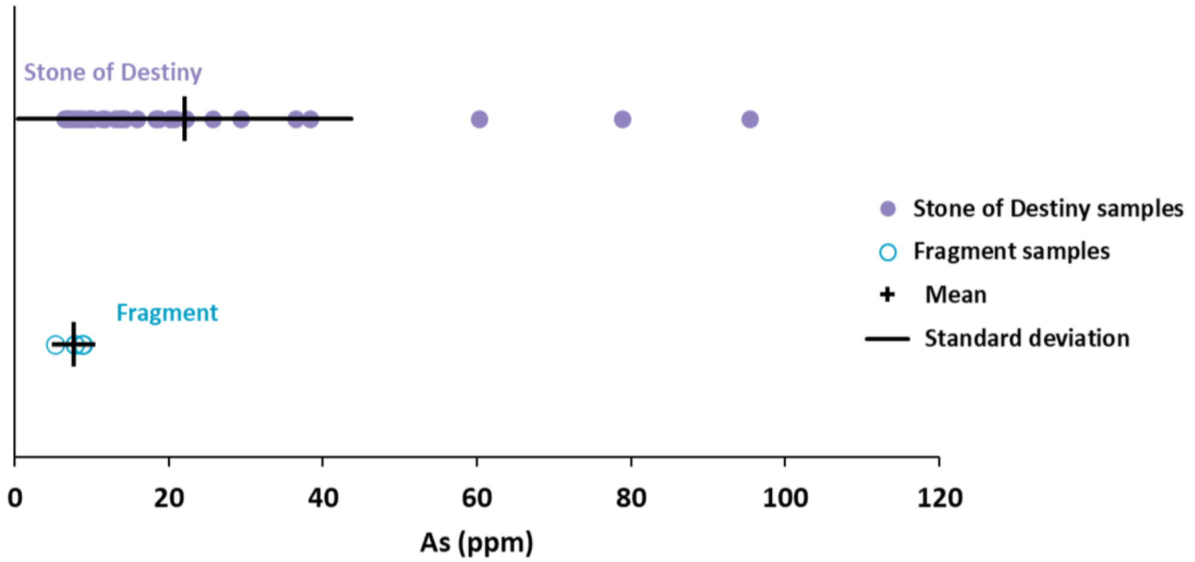


Figure A3. Comparison of amount of barium (Ba) in Stone of Destiny and fragment.

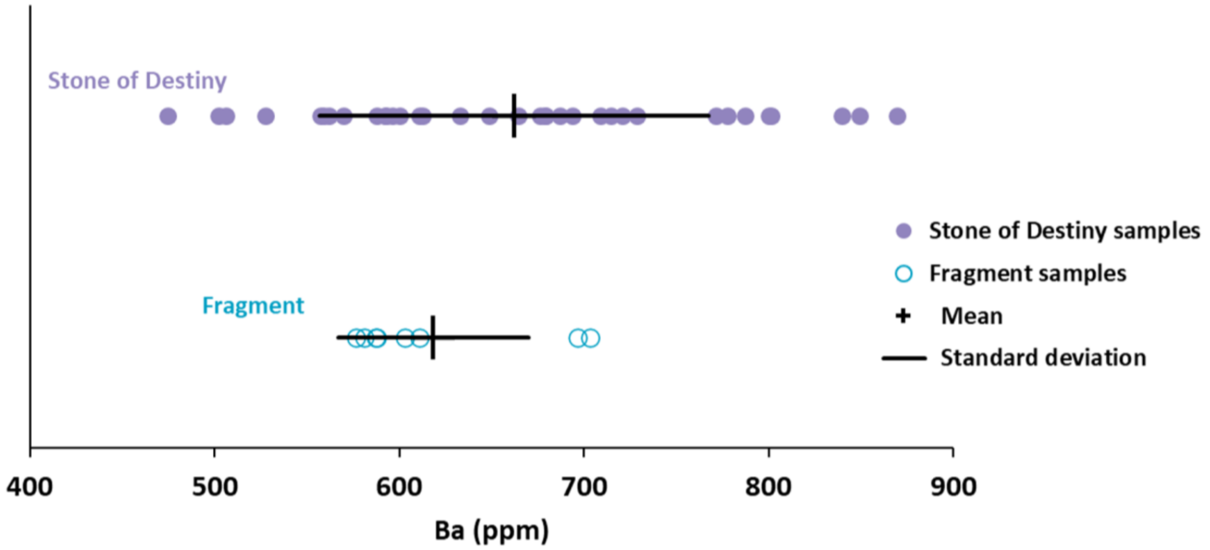


Figure A4. Comparison of amount of calcium (as CaO) in Stone of Destiny and fragment.

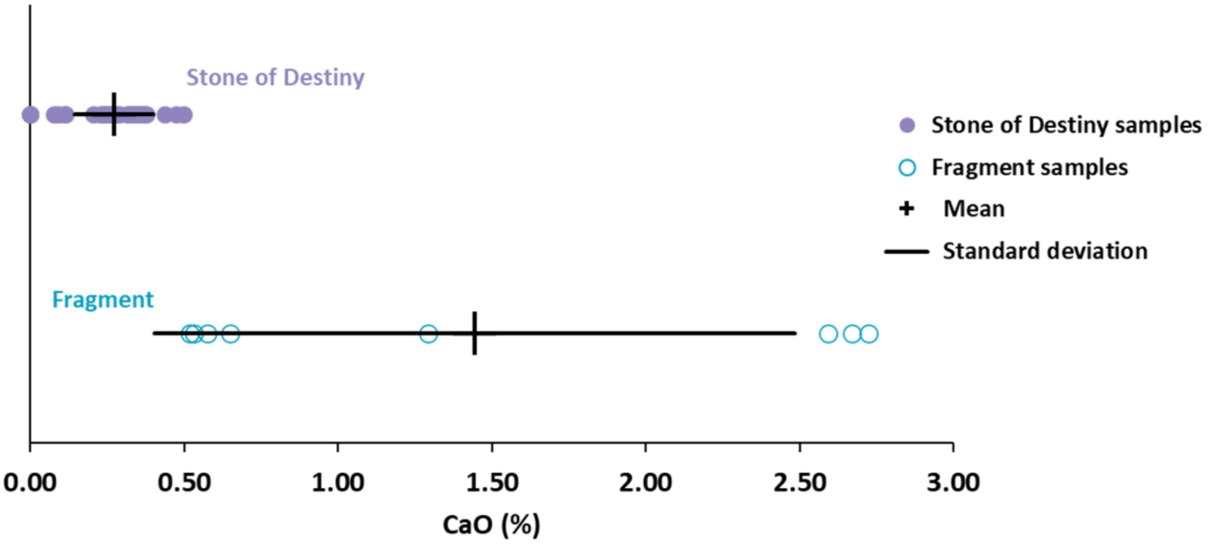


Figure A5. Comparison of amount of chlorine (Cl) in Stone of Destiny and fragment.

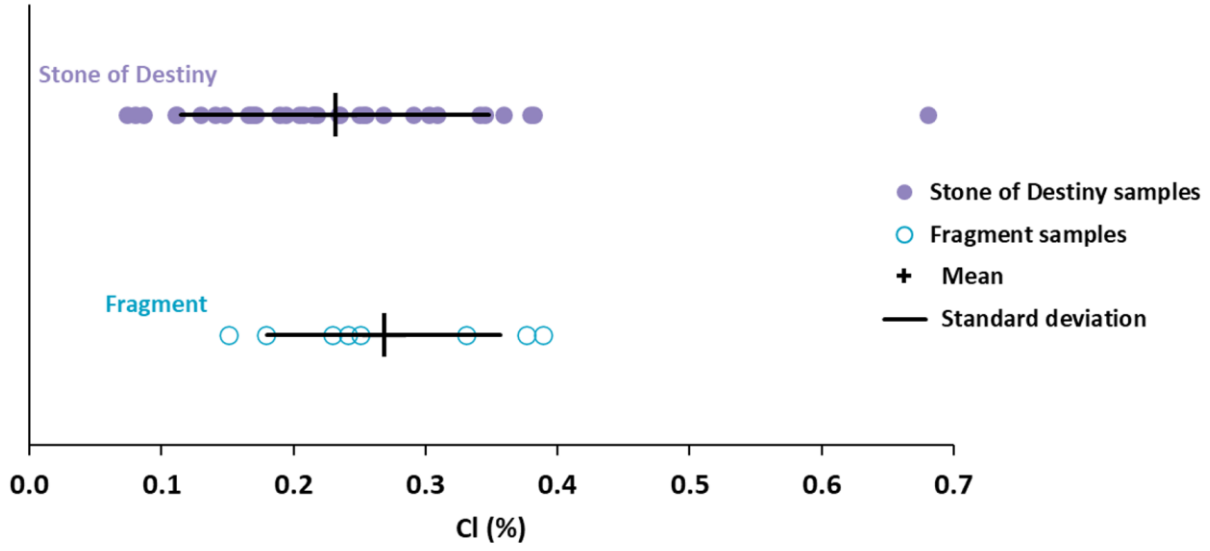


Figure A6. Comparison of amount of chromium (Cr) in Stone of Destiny and fragment.

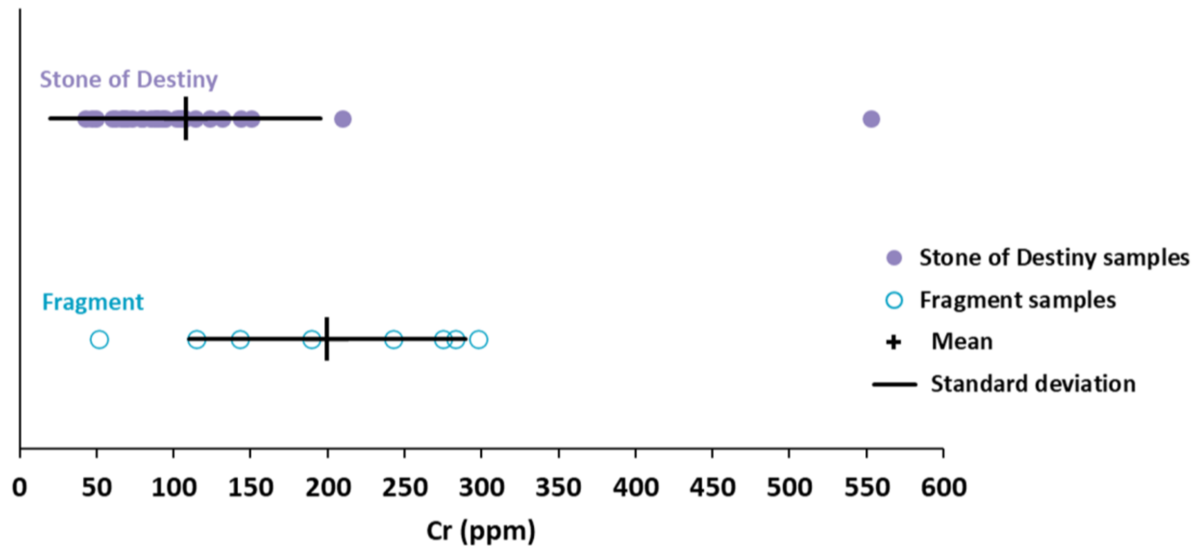


Figure A7. Comparison of amount of copper (Cu) in Stone of Destiny and fragment.

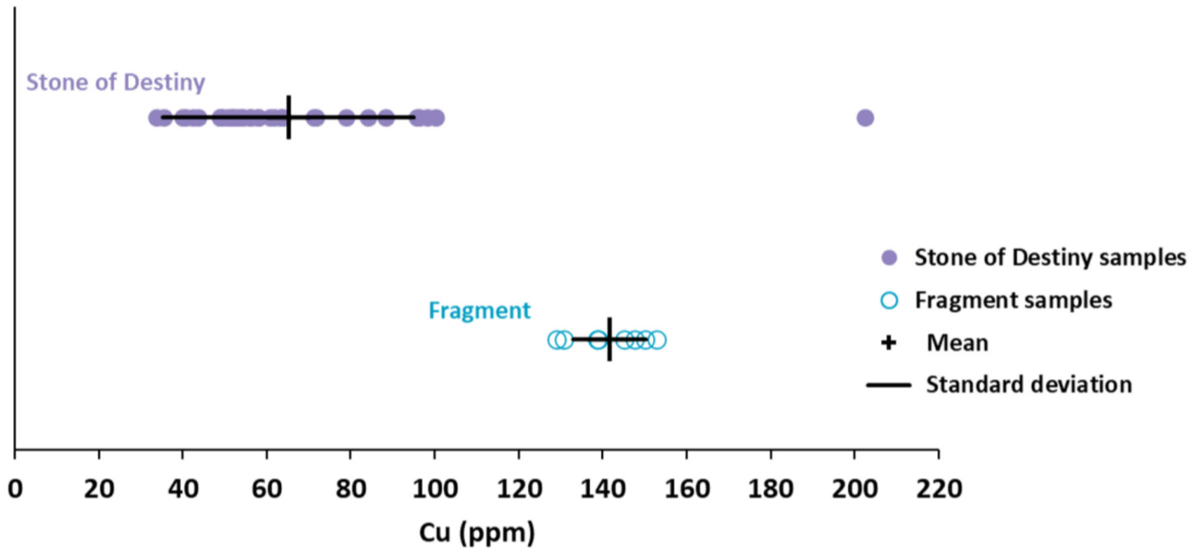


Figure A8. Comparison of amount of iron (as Fe_2O_3) in Stone of Destiny and fragment.

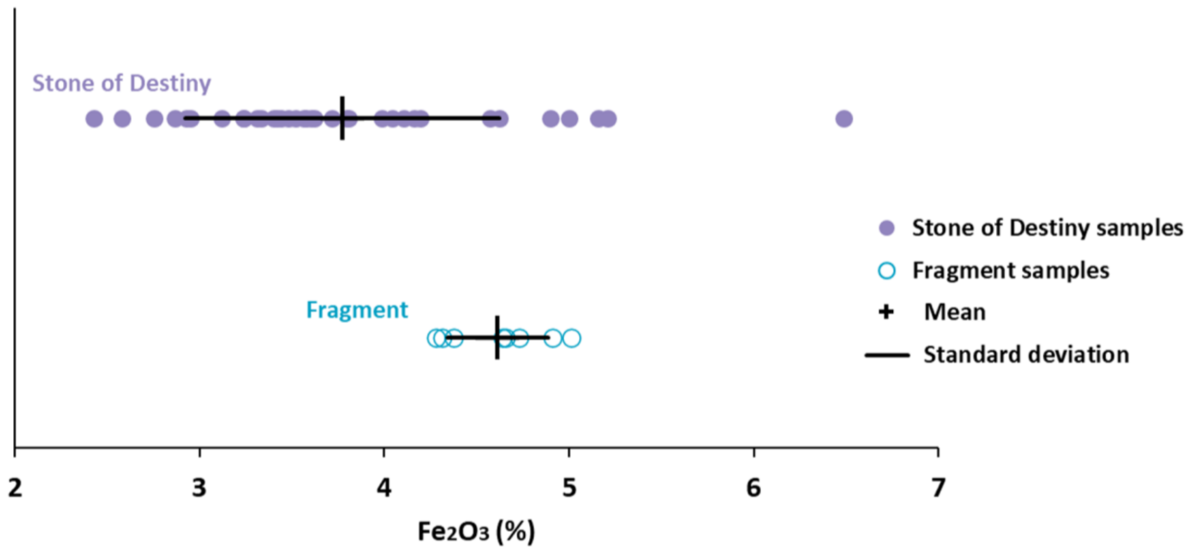


Figure A9. Comparison of amount of potassium (as K_2O) in Stone of Destiny and fragment.

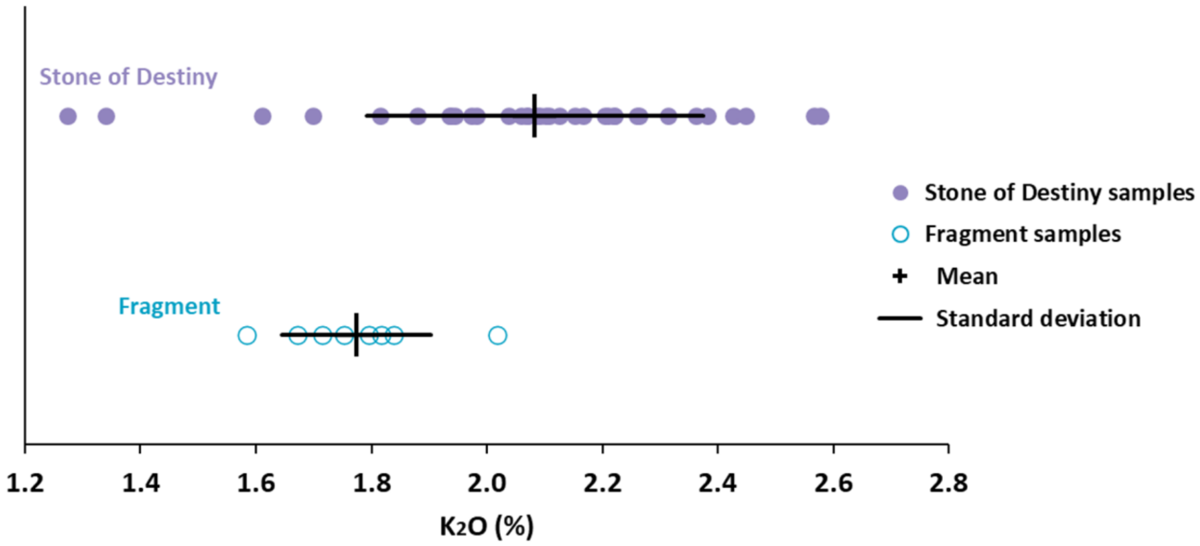


Figure A10. Comparison of amount of magnesium (as MgO) in Stone of Destiny and fragment.

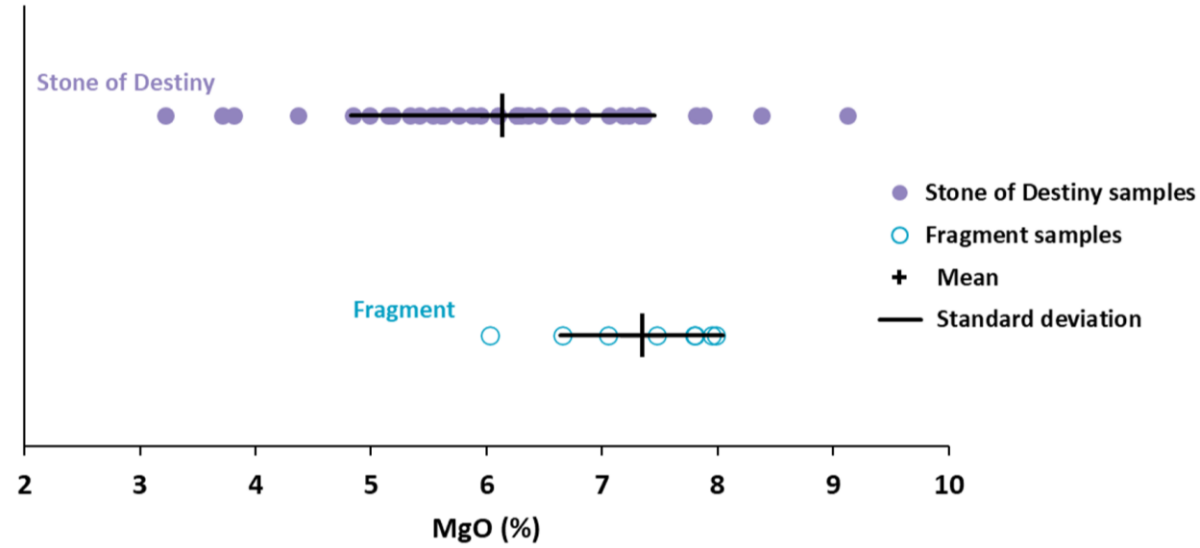


Figure A11. Comparison of amount of manganese (as MnO) in Stone of Destiny and fragment.

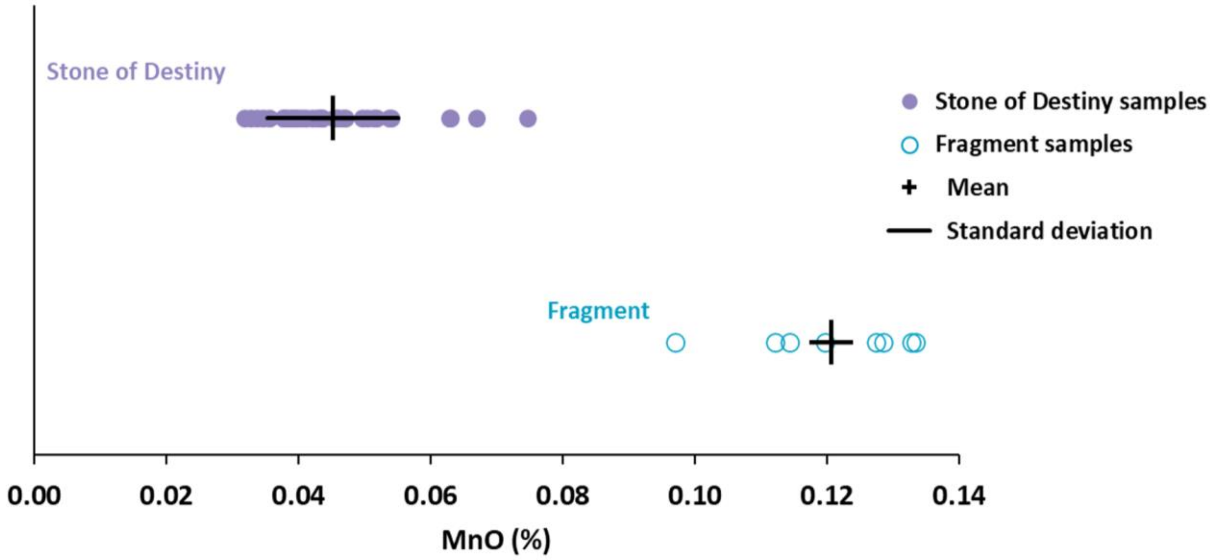


Figure A12. Comparison of amount of nickel (Ni) in Stone of Destiny and fragment.

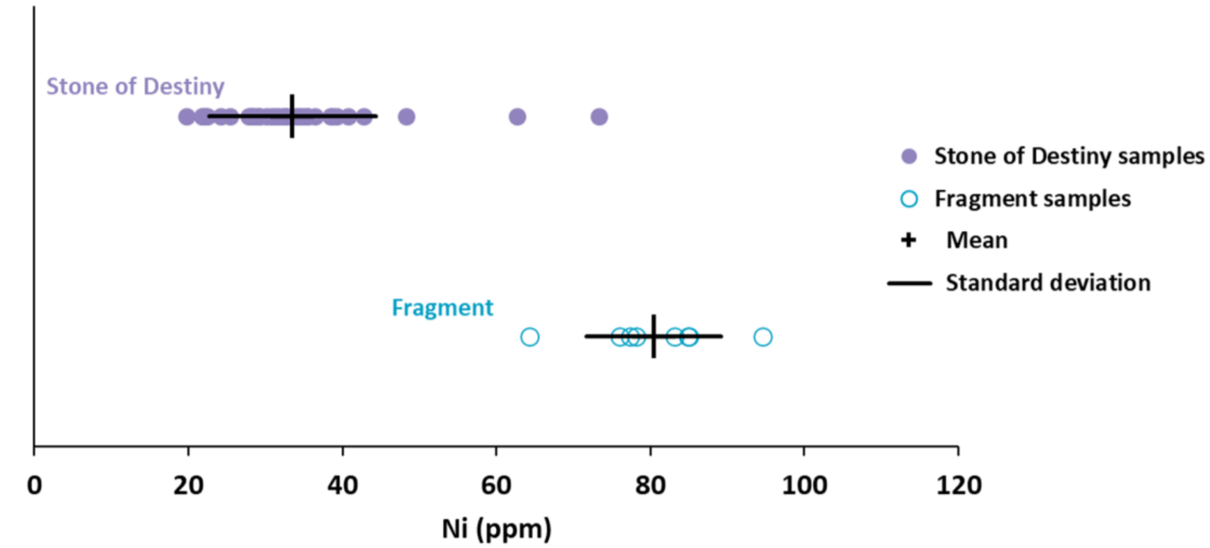


Figure A13. Comparison of amount of phosphorus (as P_2O_5) in Stone of Destiny and fragment.

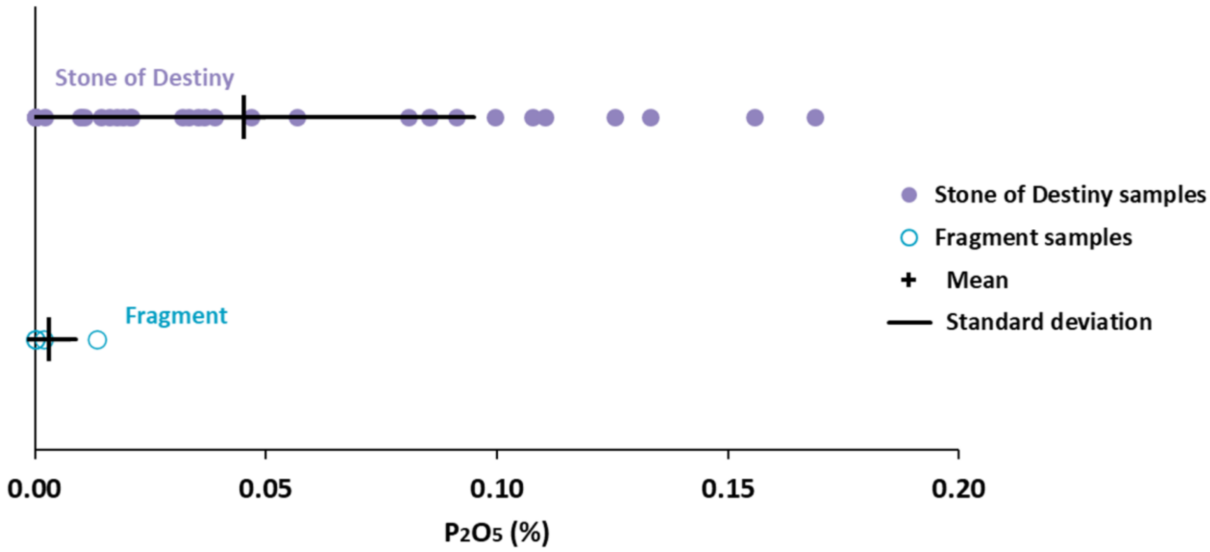


Figure A14. Comparison of amount of lead (Pb) in Stone of Destiny and fragment.

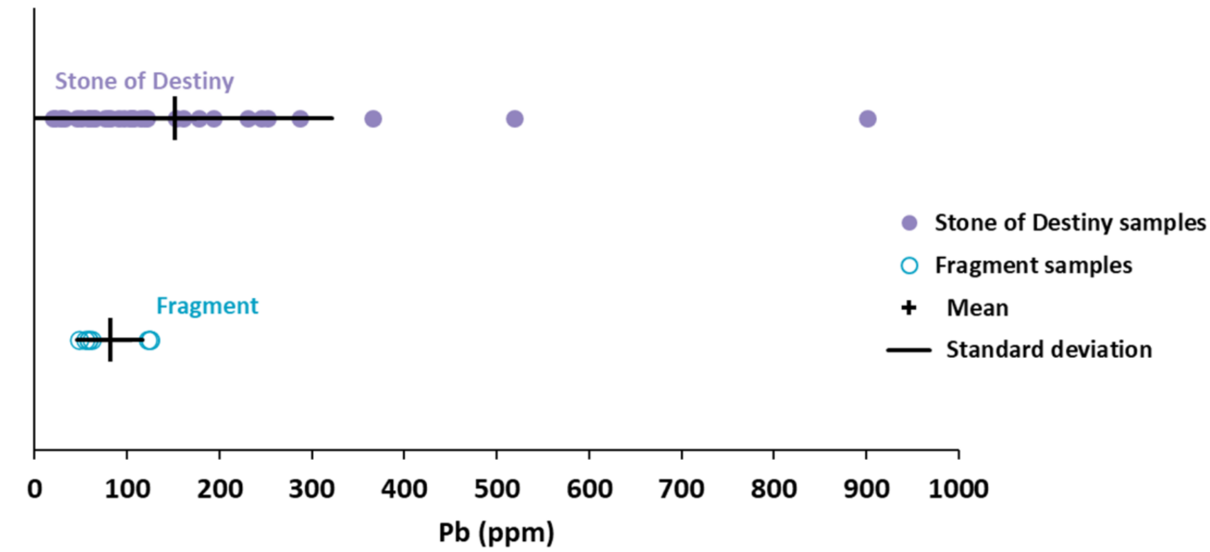


Figure A15. Comparison of amount of rubidium (Rb) in Stone of Destiny and fragment.

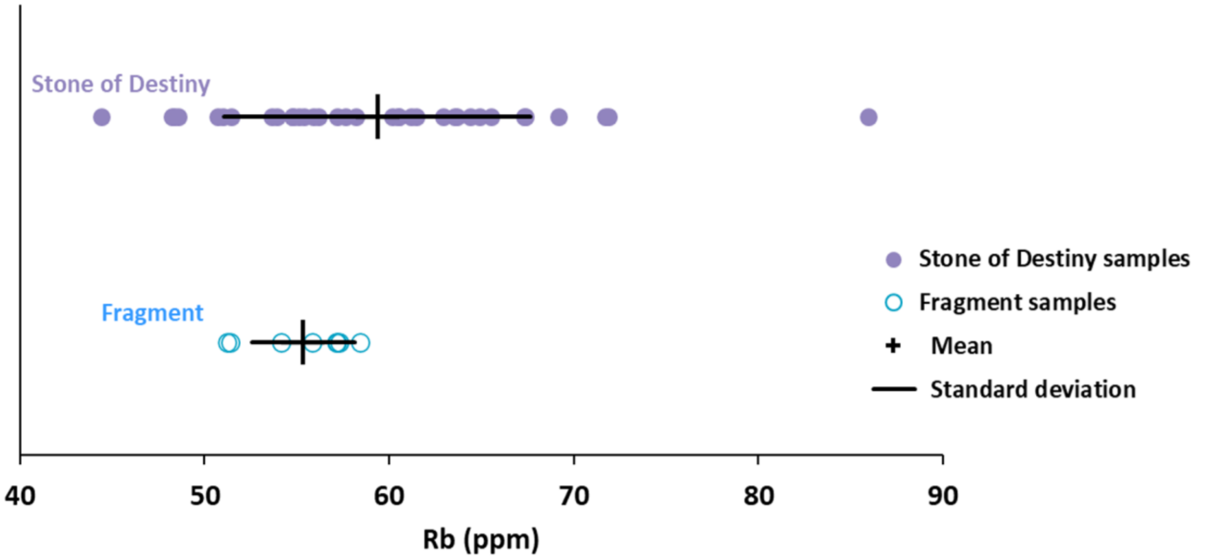


Figure A16. Comparison of amount of silicon (as SiO_2) in Stone of Destiny and fragment.

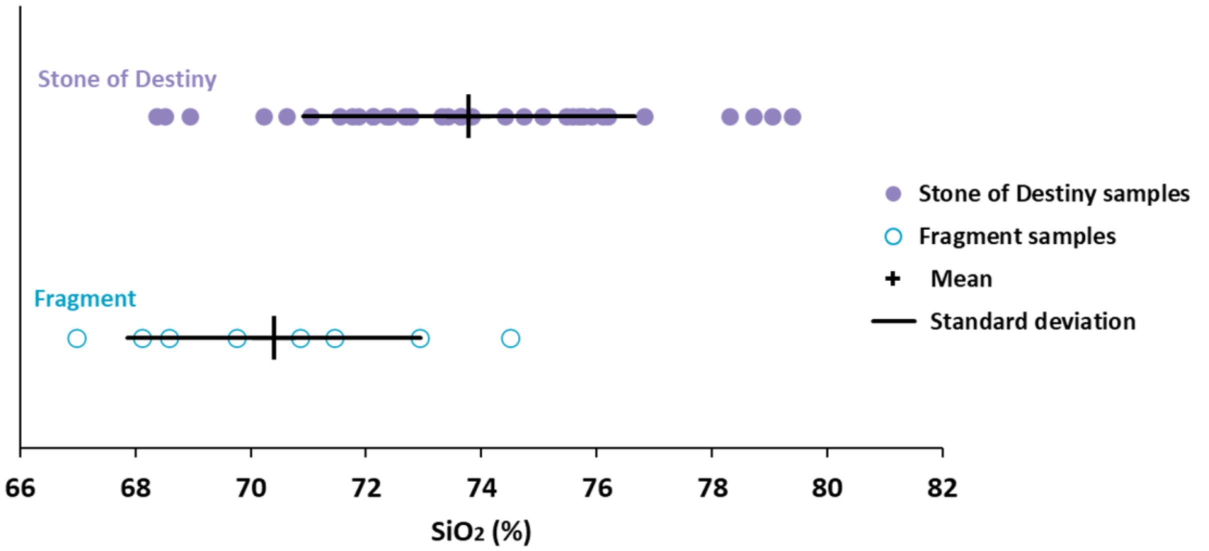


Figure A17. Comparison of amount of strontium (Sr) in Stone of Destiny and fragment.

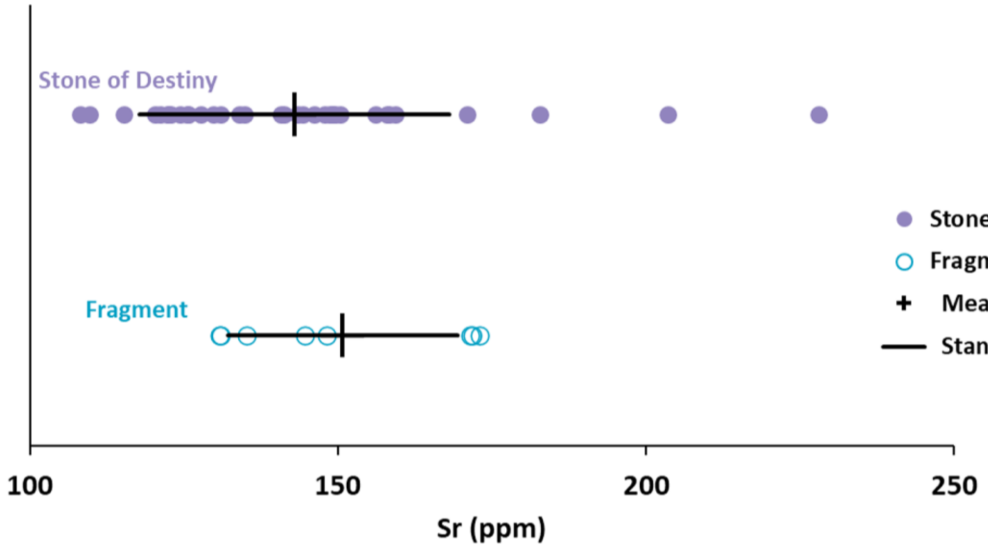


Figure A18. Comparison of amount of titanium (as TiO_2) in Stone of Destiny and fragment.

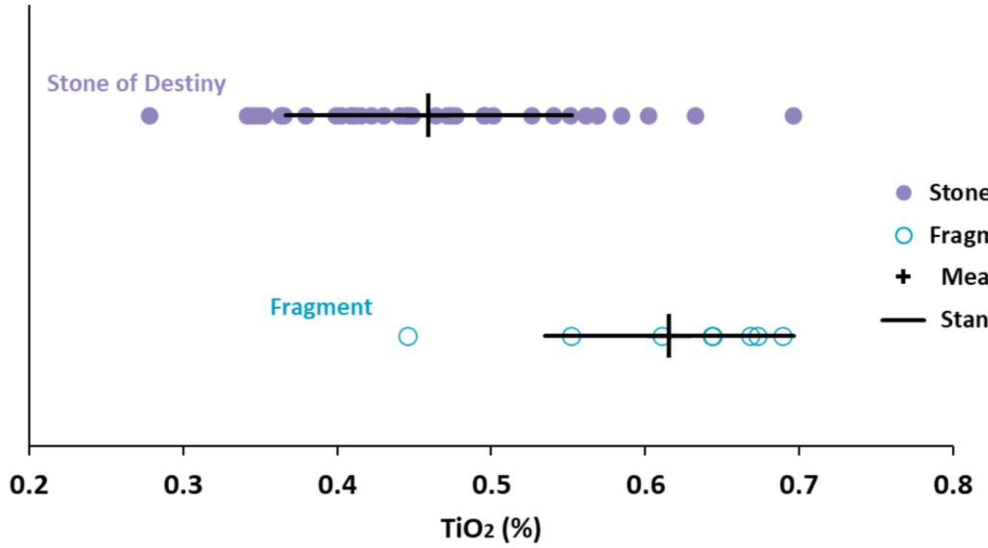


Figure A19. Comparison of amount of vanadium (V) in Stone of Destiny and fragment.

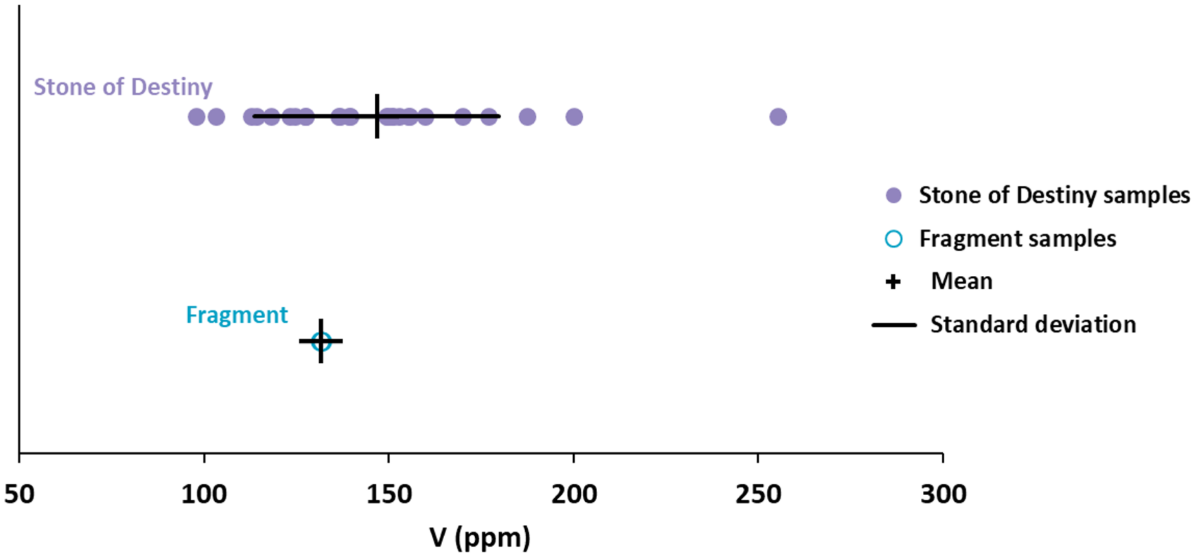


Figure A20. Comparison of amount of zinc (Zn) in Stone of Destiny and fragment.

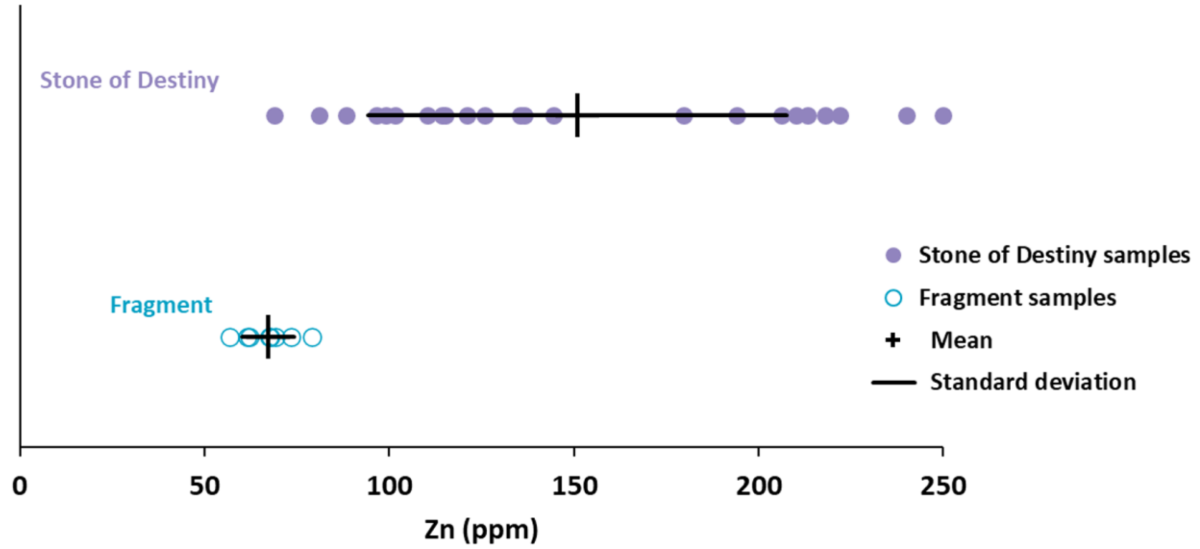
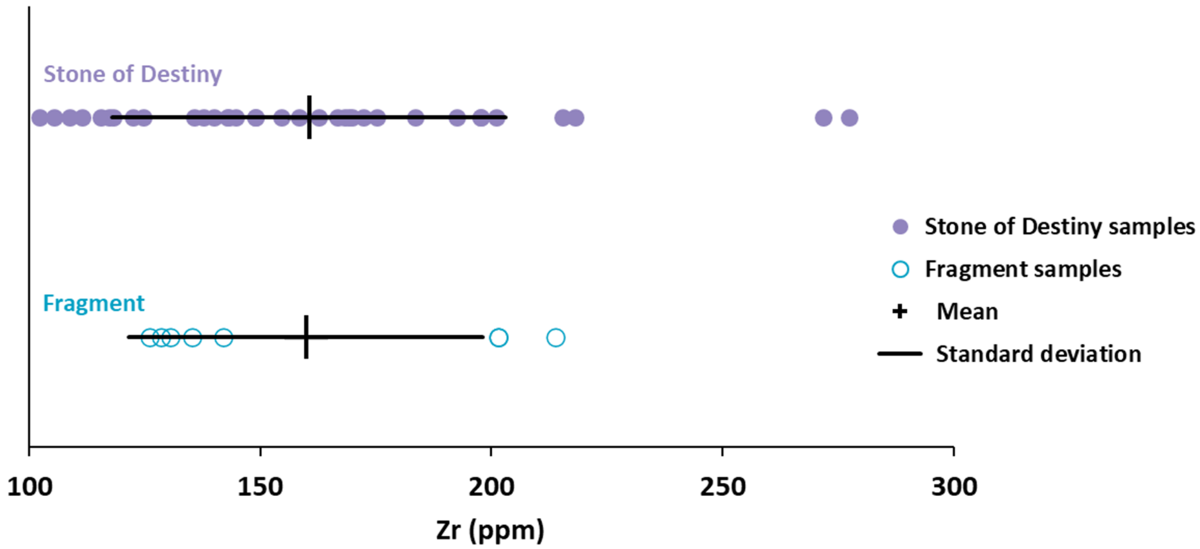


Figure A21. Comparison of amount of zirconium (Zr) in Stone of Destiny and fragment.



Appendix B. SEM energy dispersive spectrometry (EDS) chemical element distribution maps

Figure B1. Map area 001.
Yellow arrows indicate overlap between sodium (Na) and aluminium (Al) regions - probably indicating an Na-feldspar/albite mineral.
Red arrows indicate overlap between potassium (K) and aluminium (Al) regions - probably indicating a K-feldspar mineral.
White arrows indicate overlap between magnesium (Mg), aluminium (Al), and silicon (Si) regions - potentially pyroxene, amphibole or other mafic minerals).

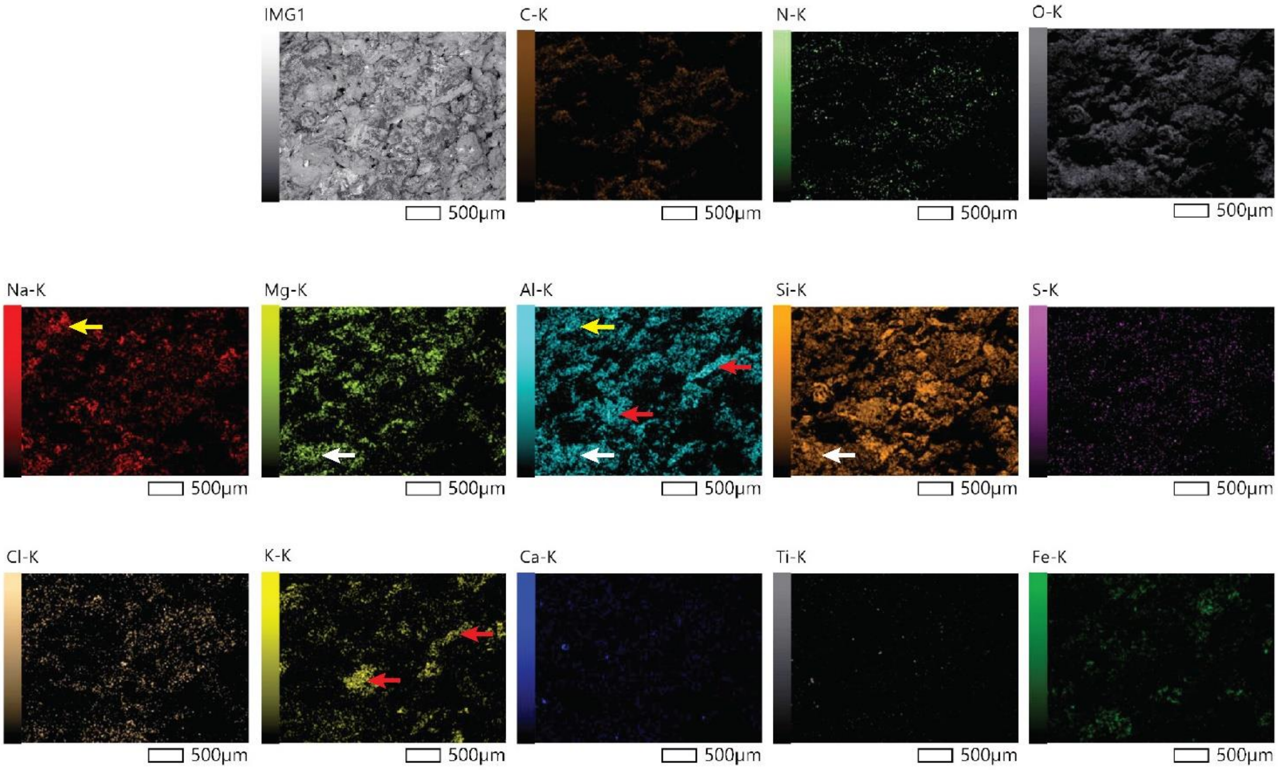


Figure B2. EDS spectrum for Map area 001. Peaks indicate detection of various chemical elements.

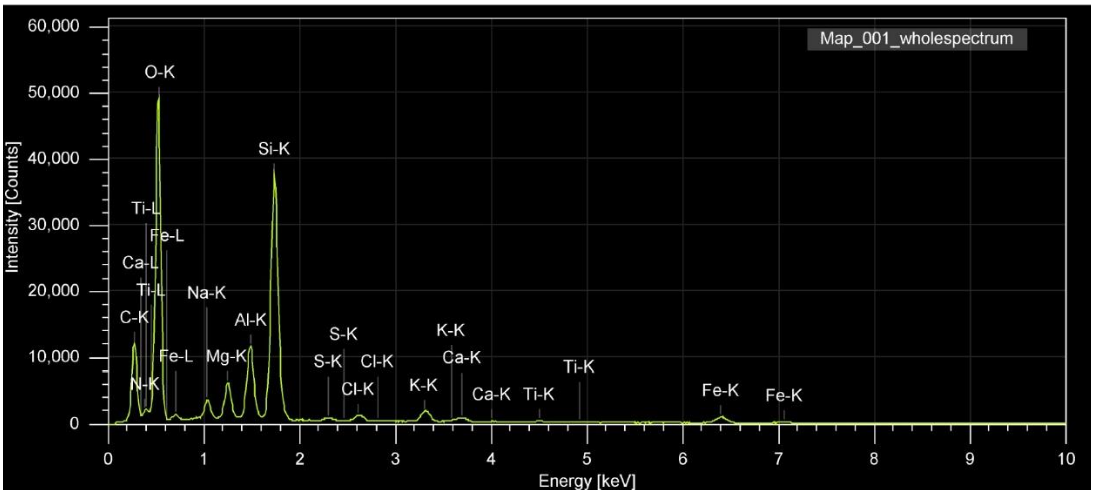


Figure B3. EDS element distribution maps of Map area 006. Showing the distribution of calcium (Ca) corresponding to the region encapsulating three quartz grains (indicated by silicon (Si) distribution).

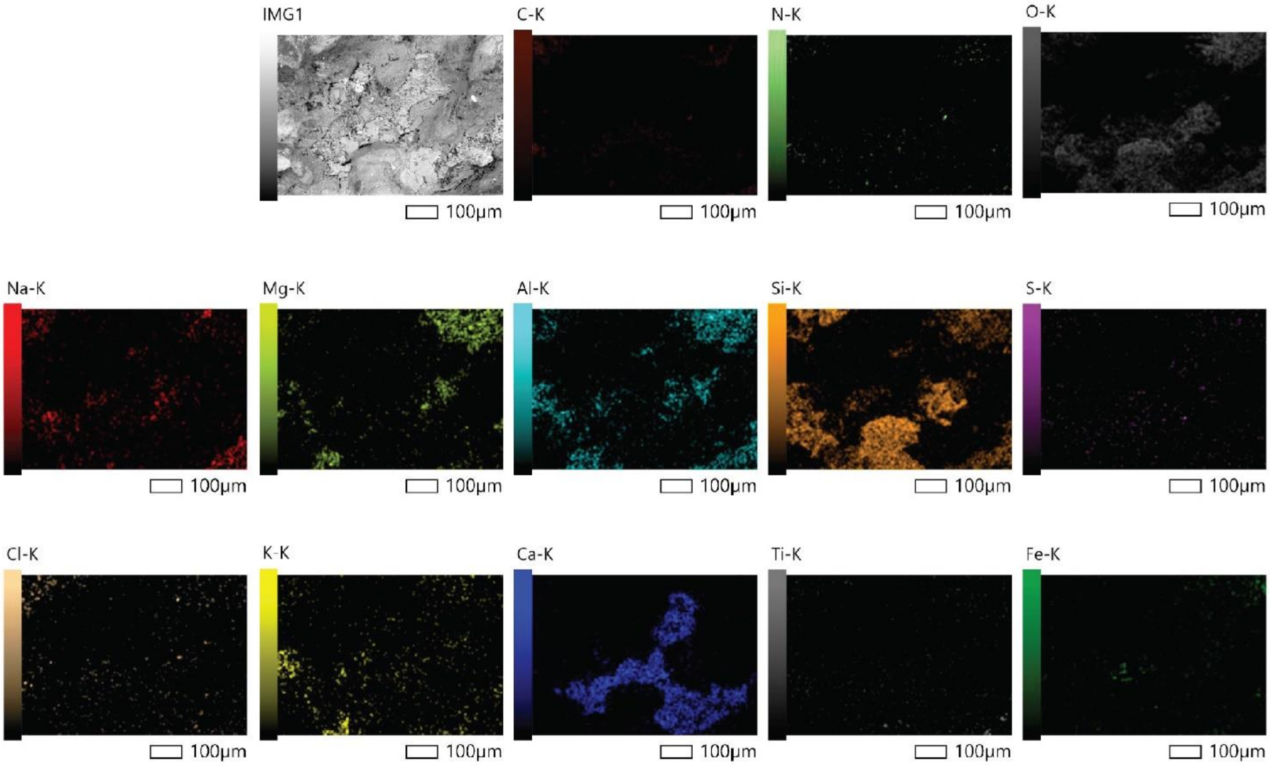


Figure B4. EDS spectrum for Map area 006. Peaks indicate detection of various chemical elements.

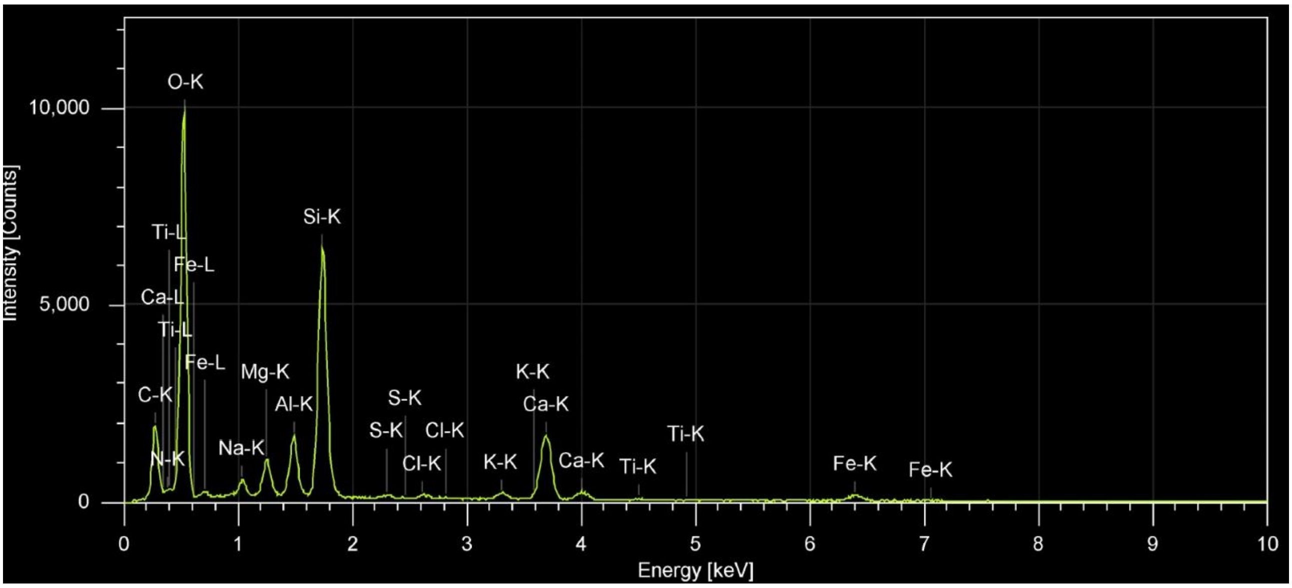


Figure B5. Backscatter electron microscope image of Side A of the fragment. Silica overgrowth cements are indicated by their sharp crystal faces (arrowed). Grey scale colours correspond to the mean atomic weight of each phase: higher atomic weights being lighter in colour and lower atomic weights, darker.

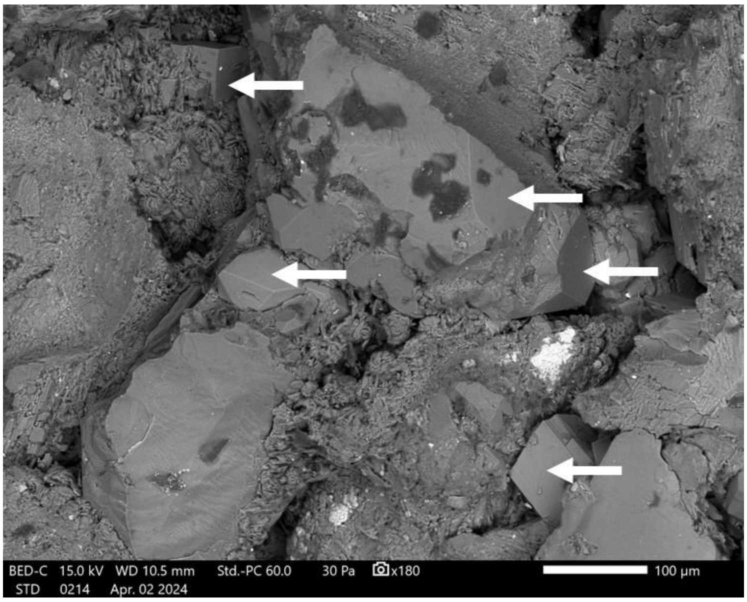


Figure B6. Backscatter electron microscope image of Side A of the fragment. Platy textures consistent with clays are evident throughout the stone, located in regions between detrital grains. Grey scale colours correspond to the mean atomic weight of each phase: higher atomic weights being lighter in colour and lower atomic weights, darker.

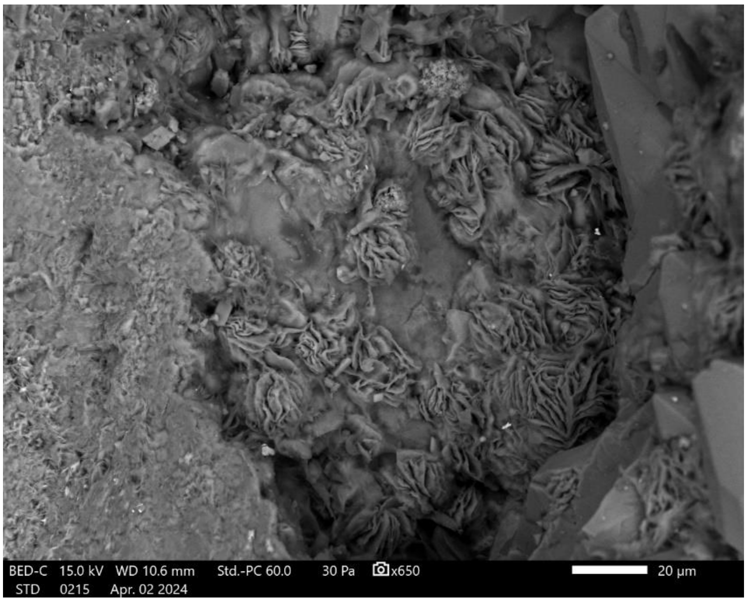
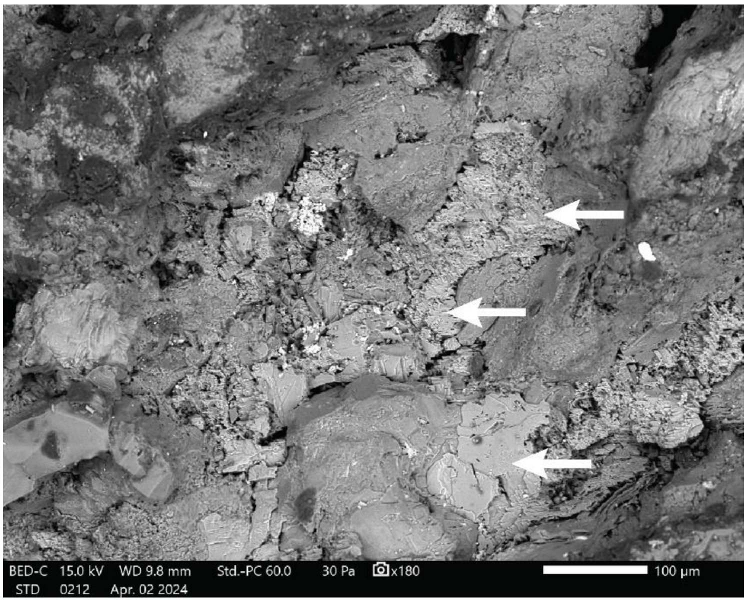


Figure B7. Backscatter electron microscope image of Side A of the fragment (Map area 006). This is a highly compacted area with calcium-rich intergranular material (arrowed) between quartz grains. Grey scale colours correspond to the mean atomic weight of each phase: higher atomic weights (e.g. Ca) being lighter in colour and lower atomic weights (e.g. Si), darker.



Appendix C. Photographs of the fragment

Figure C1. Photograph of Side A of the fragment.

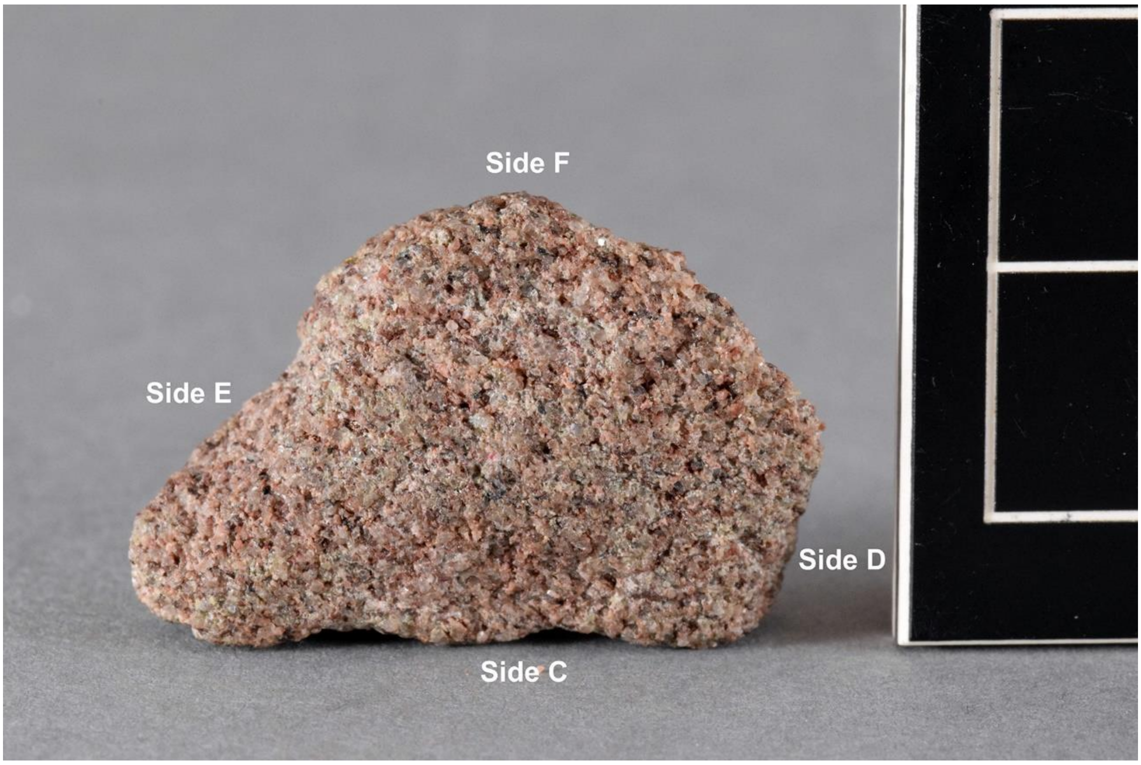


Figure C2. Photograph of Side B of the fragment.

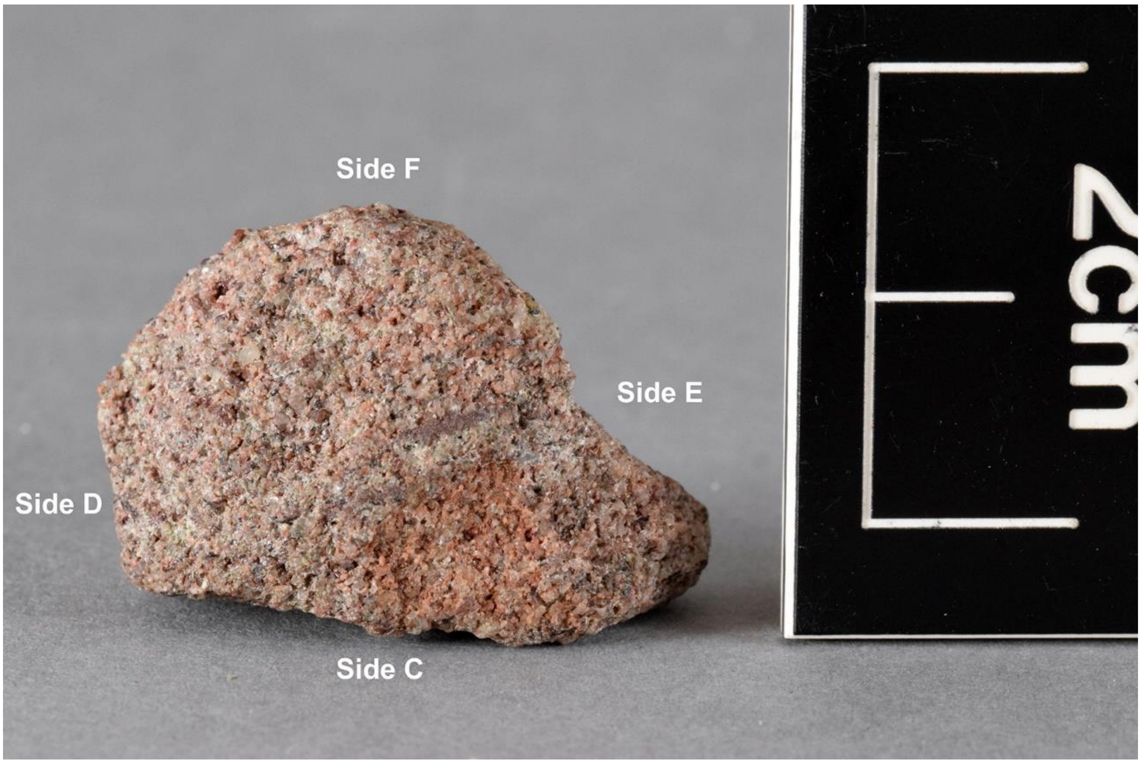


Figure C3. Photograph of Side C of the fragment.

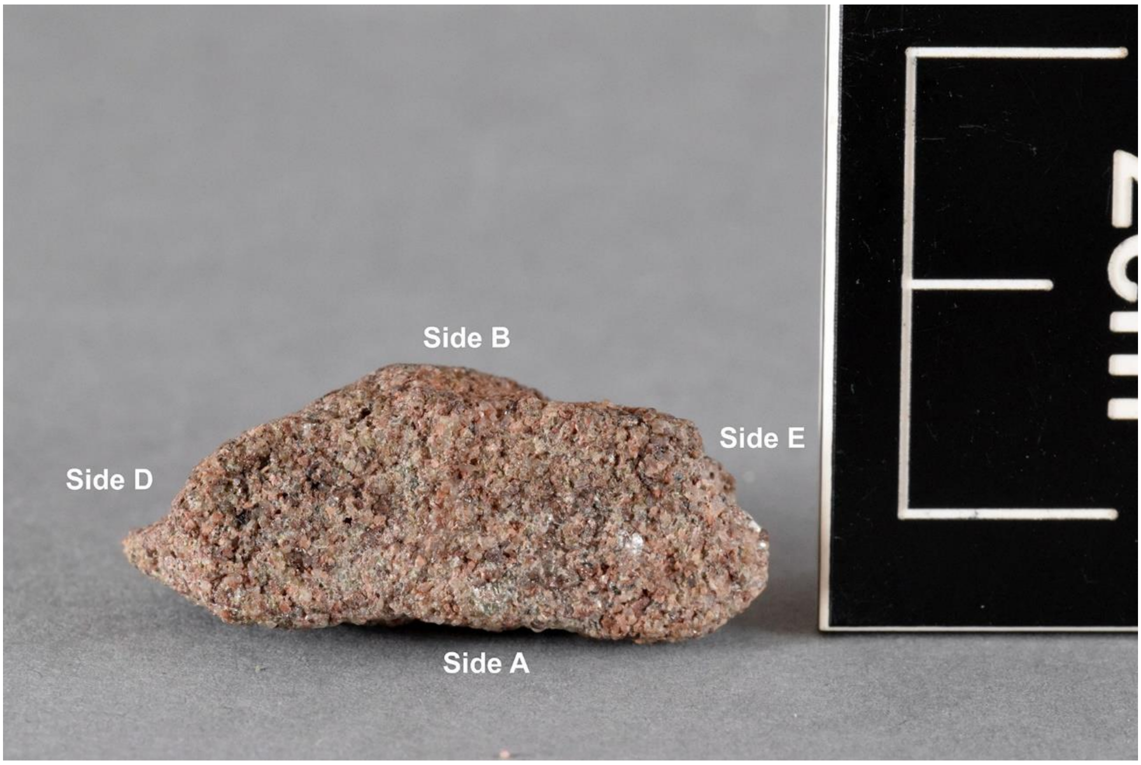


Figure C4. Photograph of Side D of the fragment.

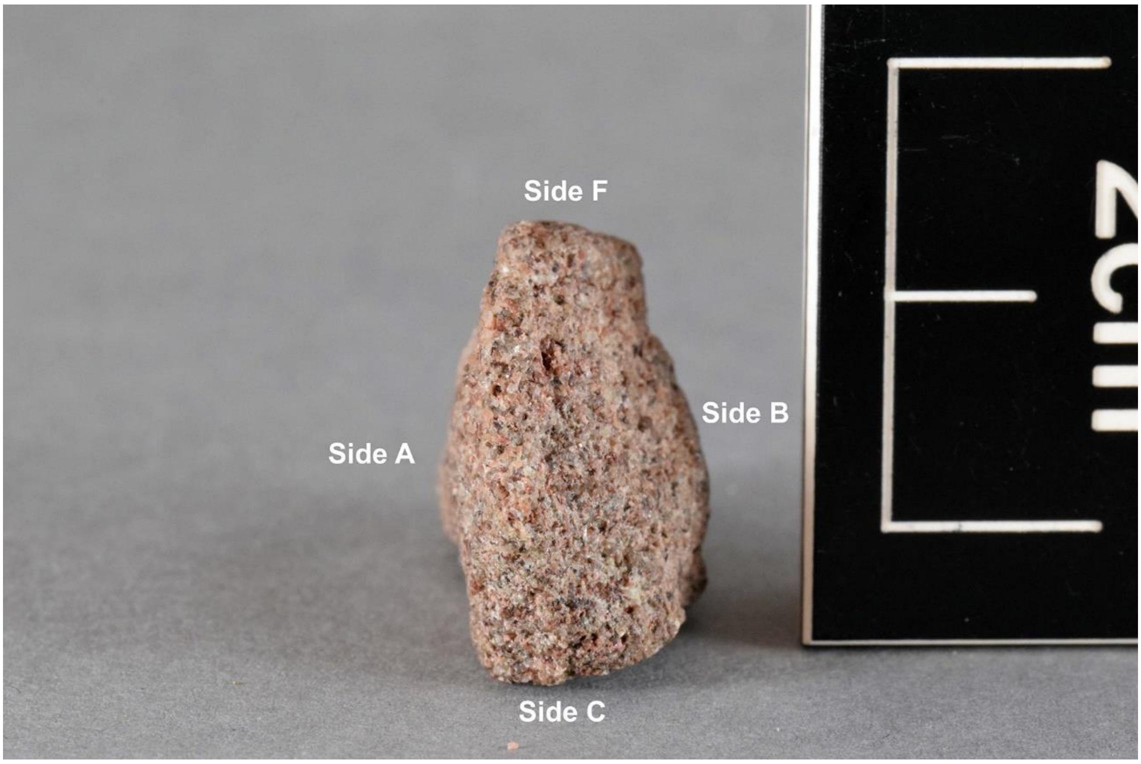


Figure C5. Photograph of Side E of the fragment.

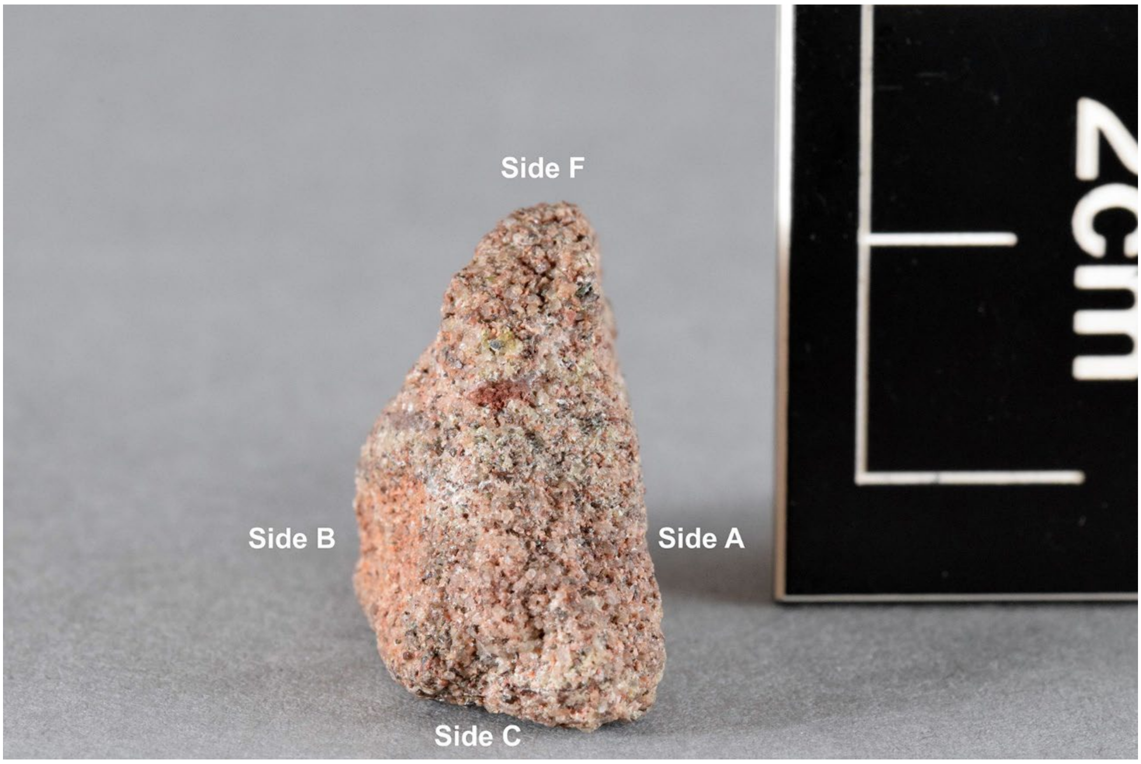


Figure C6. Photograph of Side F of the fragment.