

# **Analysis of Mortar from the South Portico of Wren's Cathedral, St Paul's Cathedral, London**

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Repair to the steps of the South Portico at St Paul's Cathedral led to the exposure of the original mortar raft, constructed between c.1677 and 1681 and the overlying Irish black marble steps laid in 1698. Samples of the hard mortar raft and the setting for the marble steps were taken and an intervening layer of soft, wet mortar was also sampled. Thin sections were taken of one sample of each layer. They were prepared by Steve Caldwell, University of Manchester, and stained using Dickson's methods (Dickson 1965). Chemical analysis was undertaken of all of the samples using Inductively-Coupled Plasma Spectroscopy, which was carried out at Royal Holloway College, London, under the supervision of Dr J N Walsh.

## **Methodology**

All the samples were washed, to remove contaminating soil, and air-dried. The outer surfaces of the ICPS samples were mechanically removed and the resulting block, weighing 3-4 gm, was crushed to a fine powder. The analysis measured the frequency of a range of major inclusions as percent oxides (App 1) and of a series of minor and trace elements as parts per million (App 2).

The thin sections were examined using a petrological microscopy and semi-quantitative data on the inclusion types present was recorded.

The ICPS data were examined using various statistical techniques, primarily Factor Analysis, in which an algorithm is applied which attempts to replace the N values measured with a smaller number of Factors. The relationship of the measured element values to the Factors is also provided in the form of weightings to be applied to each element and the proportion of the variability in the dataset "explained" by the factors is also given, as a percentage.

## **Thin Section Analysis**

### **Mortar Raft (Context 6)**

#### **Description**

The following inclusion types were noted in the sample from the mortar raft, V4530:

- Quartz. Moderate rounded and angular grains. The rounded grains are mainly c.0.3mm to 0.5mm across and have brown iron-stained veins with no trace of an

external iron cement. The Angular grains are mainly c.0.1mm to 0.2mm across and occur in lenses, often associated with dark staining of the groundmass.

- Oolitic limestone. Two large rounded grains of a pelletal or oolitic limestone. The ooliths are mostly well sorted, c.0.4mm across, and composed entirely of non-ferroan micrite, sometimes with an angular quartz grain or patch of sparry calcite at the centre. They have a single outer crust which has a slightly higher stain than the remainder and the cement consists of micrite of similar colour and texture to the ooliths, with rare areas of coarser sparry non-ferroan calcite infill.
- Opaques. Sparse rounded grains, some translucent dark brown but mostly completely opaque, up to 0.4mm across.
- Voids/Phosphate. Several amorphous voids were present, lined with phosphate. This is presumably post-burial infill of pores in the mortar.
- Ferroan calcite. Sparse sparry fragments c.0.1mm to 0.3mm across.
- Glauconite. Sparse round grains c.0.3mm across.
- Altered glauconite. Sparse round grains with a deep red colour, c.0.3mm across.
- Shell. Rare thin-walled shell, composed of non-ferroan calcite, probably land or freshwater mollusca.
- Micrite. Rare rounded fragments of a coarse-grained micrite composed of non-ferroan calcite and rounded dark brown grains less than 0.1mm across.
- Flint. Sparse rounded brown-stained grains up to 0.5mm across.
- Volcanic rock. A single subrounded grain of a volcanic rock, consisting of crystals of unidentified minerals in a glassy groundmass.

The groundmass consists mainly of fine-grained non-ferroan calcite with areas of dark staining, usually isotropic.

### Interpretation

Most of the inclusions were probably present in a quartzose sand added to the mortar during manufacture. Some of the darker stained areas probably reflect the mixture of soil, ultimately derived from the local brickearth. This might have taken place during manufacture or through subsequent contamination, even though these areas now have a calcareous groundmass.

Three inclusion types require special note. Firstly, the oolitic limestone. These fragments appear to be rounded grains and are probably detrital and present in the added sand. However, they might also be relicts of the limestone used to manufacture the lime, although whether these would appear rounded is unlikely. Similarly, they might be derived from oolitic limestone either used in the construction of St Paul's or its predecessors or the earlier

medieval or Roman structures. On balance, the rounding suggests that they are indeed detrital. Similar oolitic limestones outcrop in the middle and upper Jurassic of Oxfordshire and might be expected in calcareous sands and gravels in the Lower Thames valley.

Secondly, the ferroan limestone fragments are similar to the fragments of Irish black marble found in the other two samples. However, none of the fragments contain the rare but distinctive microfossils seen in that rock and on balance these fragments too are probably detrital Jurassic limestone.

Thirdly, the volcanic rock fragment is clearly part of the "trass" found in the setting for the marble steps. However, it is not clear from its position in the section whether it was part of the original mortar raft or has been intruded subsequently and then concreted with lime mortar *in situ*. On balance the evidence is too slight to show that the Trass was present on the site when the mortar raft was being constructed.

The conclusion is therefore that this mortar raft was made from lime mortar mixed with a local, Thames valley quartz sand and that the sample has been contaminated after burial both with calcium phosphate and local sediment.

### **Intermediate Mortar (Context 5)**

#### Description

The following inclusion types were noted in thin section (Sample V4526):

- Quartz. Abundant grains ranging from c.0.2mm to 0.5mm. The larger grains are rounded and similar to those in the mortar raft but the grain size distribution and roundness indicates that this is a different sand.
- Glauconite. Sparse subangular fragments up to 0.3mm across
- Altered Glauconite. Sparse subangular fragments up to 0.3mm across
- Flint. Rare rounded brown-stained and unstained fragments. A single rounded unstained grain 1.0mm across was present.
- Ferroan calcite. Rare rounded sparry ferroan calcite c.0.2mm across.
- Limestone. Rare angular fragments of limestone containing abundant fine-grained fossil fragments, composed of a mixture of dolomite and ferroan calcite, sometimes with ferroan calcite filling of dolomite tests, up to 0.2mm across, also rare angular quartz grains up to 0.3mm across. The groundmass is opaque. Comparison with the more abundant and larger fragments in the step bedding indicates that this is the Irish Black Marble used to construct the steps. This is a fossiliferous limestone of Carboniferous age from southeast Ireland. Some of these Irish ornamental limestones are crinoidal but no crinoid fragments are present in this rock fragments in this section, nor in the step bedding.

- Voids. Extensive amorphous voids lined or completely filled with calcium phosphate.

The groundmass consists of non-ferroan calcite micrite.

### Interpretation

This is another lime mortar, containing a higher proportion of quartz sand than the raft. The sand is, however, probably of local origin. Sparse chips of Irish Black Marble are present, indicating that the steps were on site and being prepared when this layer was laid down. The extensive replacement by phosphate is perhaps due to acidic groundwater being trapped in this layer between the two less permeable mortars above and below.

### The Step Setting (Context 3)

#### Description

The following inclusion types were noted in the thin section of the mortar base for the steps (V4522):

- Flint. Abundant angular fragments up to 1.5mm across. A few show signs of having been struck from a flint with a brown-stained cortex. Some extremely angular flakes are present and none of the angles of the fragments have been weathered. The flint is in the main unstained and contains sparse microfossils and some voids filled with chalcedony or coarser-textured silica.
- Limestone. Angular fragments of a fossiliferous limestone containing some recognisable microfossil fragments (ostracods) amongst a mass of unidentifiable material, less than 0.2mm across. The fossils are composed of dolomite with ferroan calcite infill. Sparse angular quartz up to 0.2mm across is present and the groundmass is opaque and black in reflected light.
- Chalk. Rare rounded fragments of micrite of similar texture to chalk. However, no microfossils are present to confirm the identification as chalk.
- Volcanic rocks. Moderate angular, subangular and sub-rounded fragments of volcanic rock ranging from c.0.2mm to 1.0mm across. The rocks vary from crystalline rocks with abundant feldspar crystals in a dark microcrystalline matrix, green pleiochroic pyroxene crystals up to 0.5mm across, colourless glass and vesicular lava.
- Quartz. Sparse rounded grains c.0.2-0.4mm across. Some of these have iron-stained veins similar to those in the other two mortars.
- Iron-cemented sandstone. A single fragment of sandstone, consisting of rounded quartz grains with a dark brown to opaque cement.
- Microcline Feldspar. Rare subangular fragments up to 0.5mm across.

- Opaques. Rare angular fragments, some vesicular.
- Glauconite. Sparse rounded grains up to 0.2mm.

The groundmass consists of a colourless isotropic material with abundant unidentified microcrystalline inclusions less than 0.1mm across. Several of the inclusion types (e.g. the flint, quartz and volcanic rocks) are surrounded by reaction rims.

### Interpretation

This material is not a lime mortar but a hydraulic cement (Anon 2004-2007). This is formed by the action of a fine-grained material rich in silica and aluminium with slaked lime. The resulting compound will set under water and is resistant to sulphate corrosion.

The materials used in this case are volcanic tuff, crushed flint and a small admixture of Thames valley sand.

The exact nature of the volcanic ash which was used is not clear. Clearly, the material originated in a volcanic ash fall, but there is no sign of fusion of the ash into a breccia or tuff and it may therefore have been an uncemented ash, a weathering deposit or a detrital deposit. Several of the ash fragments are rounded, and the lack of well-rounded grains is partly due to the crystalline structure of most of the fragments, which are more likely to fragment than erode. Furthermore, there are also no very sharp angled fragments which would be present if the material had been milled. Most likely, the material came from a detrital deposit. The lack of alteration to the volcanic glass indicates a Tertiary or recent date. These characteristics limit the potential sources in the old world to the Rhineland, Iceland, southern Italy and Greece.

The flint retains some cortex and is therefore not from a secondary source in the Thames basin but comes direct from the Upper Chalk. It shows no sign of rounding and some of the inclusions are delicate flakes which would have been broken if the material had come from a detrital or erosional deposit. Also, there is no sign of weathering of any of the angular flint surfaces, only of the brown-stained cortex. The most likely source of this flint is therefore a deposit of clay-with-flints or head and the material has probably been milled.

The Thames valley sand is too rare to be compared trait for trait with those in the other two mortars. However, the lack of angular, fine-sand-grade grains and the small size of the glauconite grains shows a greater similarity to the intermediate mortar than the mortar raft.

### Chemical analysis

Twelve samples of mortar were taken for chemical analysis, four from each context. The silica content, which was not measured, was estimated by subtraction of the total measured oxides from 100%.

All three mortars have a similar silica content (Fig 1) with the mortar raft (hard mortar in Fig 1) having a wider spread of values than the other two.

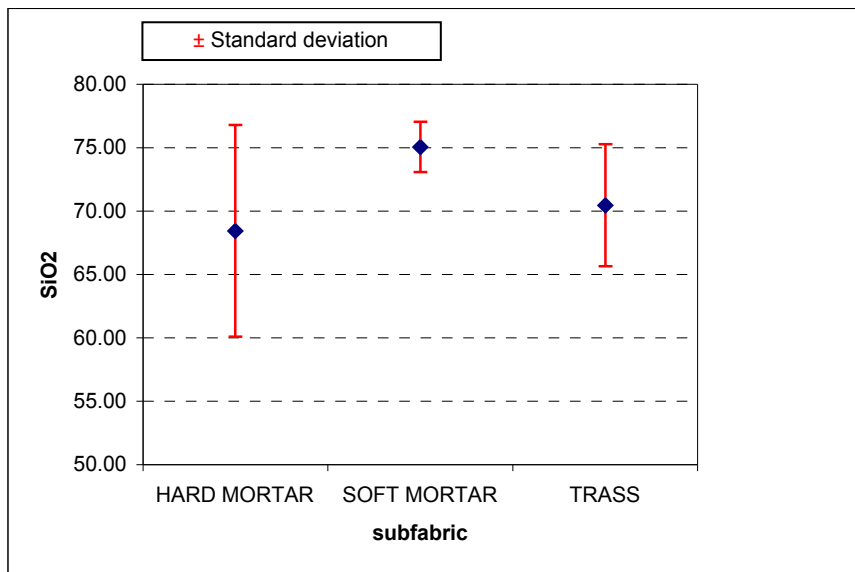


Figure 1

Five elements show distinct, non-overlapping, ranges for the three mortars.

These consist of aluminium, potassium, titanium, chromium and nickel (Table 1)

Table 1

Element	Context 6	Context 5	Context 3
Al <sub>2</sub> O <sub>3</sub>	0.61+/- 0.03	1.22+/-0.38	3.07+/-0.23
K <sub>2</sub> O	0.07+/-0.01	0.31+/-0.05	0.15+/-0.04
TiO	0.03+/-0.01	0.05+/-0.01	0.16+/-0.01
Cr	7.98+/-0.55	10.45+/-1.10	27.50+/-0.90
Ni	7.00+/-2.16	10.25+/-0.50	19.00+/-1.63

Other elements are present in similar frequencies in the two lime mortars (contexts 5 and 6) but in different frequencies, usually higher, in the trass mortar (context 3).

These include iron, scandium, vanadium, ytterbium, samarium, europium, dysprosium, yttrium and cobalt.

Since flint is almost pure silica and adds little else to the composition of the mortars and it is unlikely that the black marble fragments are present in sufficient quantities in the same to add more than calcium and strontium, we can assume that all elements which are elevated in the trass mortar are present as a result of the volcanic inclusions. By subtracting the mean

values for these elements determined from the analyses of the lime mortars we can even obtain an estimate of the relative frequency of these elements in the volcanic ash.

Elements which are present in higher or similar frequencies in the lime mortars are likely to have been present in the lime itself or in the sand aggregate. These consist of silica (estimated); magnesium, calcium, sodium (higher mean, but one high measurement from context 5), potassium, potassium, phosphorus, manganese, strontium,

Barium, copper and zirconium are mostly higher in the trass mortar but with anomalously low measurements in single samples. These suggest that these elements are present in either the black marble or trass.

Lead values include one anomalously high measurement from one of the trass mortar samples and are all quite high, when compared with local unglazed pottery. This suggests either contamination from groundwater (derived perhaps from corroding leadwork) or that the lime production process contaminated the lime.

### Source of Lime

Fig 2 plots the frequency of calcium (in percent oxide) against strontium (in ppm). It confirms that there is a correlation between the two elements and shows that the trass mortar has a lower ratio of strontium to calcium than the two lime mortars. It is possible that during the chemical reaction in which the hydraulic mortar is formed strontium is released but it seems more likely that these ratios reflect differences in the strontium content of the lime. If so, this would imply that the lime used in the trass mortar was from a different source than that in the lime mortars.

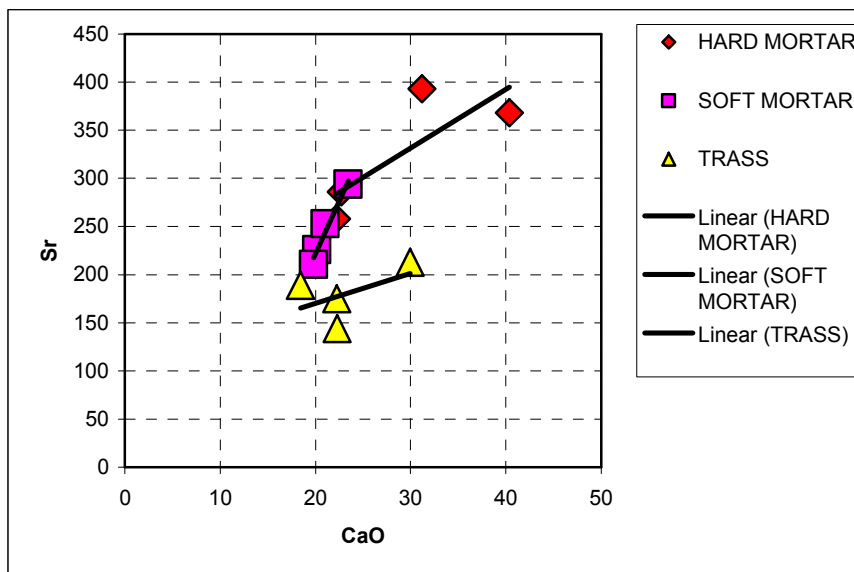


Figure 2

### Source of Sand

Those elements with the least correlation with the volcanic ash are probably in the main either present in the quartzose sand or soil contamination. To judge by the thin section, such contamination is highest in the soft mortar, context 5, and therefore potassium, which is highest in those samples is possibly a contaminant. The remaining elements are probably present in the quartzose sand, which includes opaques, glauconite and altered glauconite.

Twelve elements were chosen for their lack of correlation with nickel, one of the elements most strongly linked to the volcanic ash: Ce, Cu, Eu, Fe<sub>2</sub>O<sub>3</sub>, K<sub>2</sub>O, La, MgO, MnO, Nd, P<sub>2</sub>O<sub>5</sub>, SiO<sub>2</sub> and Zn. Factor analysis of the data for these elements reveals three factors and the contribution of elements to the first two factors is shown in Fig 3. A plot of F1 against F2 scores shows that most of the rare earth elements (Nd, La, Eu) and copper and zinc have similar weightings contributing to a high F1 score and in Fig 4 the trass mortar samples all have high F2 scores. All four of the trass mortar samples and one of the soft mortar (context 5) samples have high F1 scores. F2 scores distinguish the hard mortar (context 6) from the soft mortar (context 5) and this difference, seems to be due almost entirely to the higher silica and potassium content in the soft mortar and a higher manganese content in the hard mortar (context 6). The phosphorus weighting suggests that it is present in the lime mortars with no preference for one over the other and the magnesium weighting suggests that it is present in both the trass mortar and the soft mortar more than in the hard mortar.

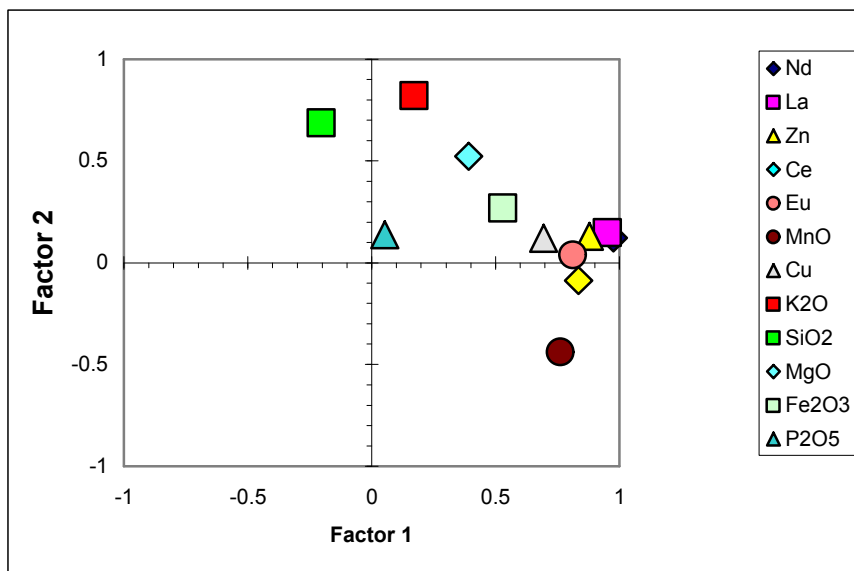


Figure 3



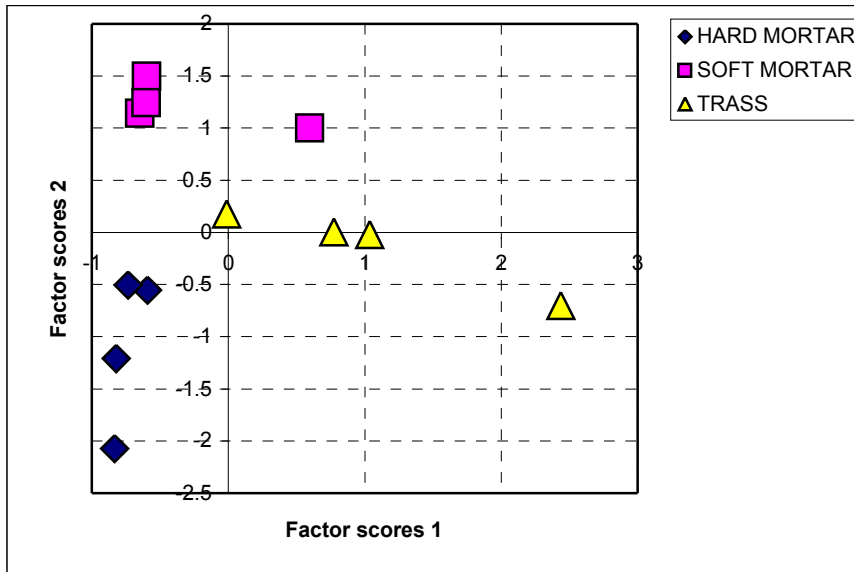


Figure 4

### Conclusions

The thin section and chemical analysis indicate that the hard mortar used as the raft upon which the south portico was built, constructed between c.1677 and 1681, was a lime mortar using local quartzose sand. There is no evidence that either the black marble used for the steps or the volcanic ash used to make the hydraulic mortar they were set in was present at that time.

The setting for the steps contains chips of black limestone which indicate that the steps were being prepared on site and the chippings used as aggregate in the mortar. The petrology of these chips, which are present in both the hydraulic mortar setting, context 3, and in a layer of soft lime mortar below, context 5, is consistent with Carboniferous limestone although the fragments in thin section lack the crinoids which are a noted feature of some of the Irish black marbles.

The setting for the steps also contains a mixture of volcanic sand and crushed flint. The volcanic sand is probably from the Eifel region of Germany, which is a known source of trass. The flint is probably from a deposit of chalk-with-flints or head from the Chilterns or north downs. There are, however, no distinctive features in thin section which could confirm this source, as opposed, say, to a Northern French source. It depends, therefore, on whether the flint was added to the mortar as a deliberate ingredient or whether the trass, as supplied, was deliberately “cut” with crushed flint because of the similarity in colour and texture and because crushed flint is a much cheaper commodity to obtain. Whether deliberate or not, the thin section shows that a similar reaction between the flint and the hydraulic mortar took place as that between the volcanic ash and the mortar.

One of the suggested reasons why volcanic ash is said to be so efficient in the production of hydraulic mortar is that the vesicular nature of the ash, and the crypto-crystalline groundmass, provide a large surface area in contact with the lime and it may be that the flint, which shares that crypto-crystalline nature but lacks the aluminium content, worked acceptably as a substitute.

The strontium content of the samples suggests that a different source of lime might have been used for the hydraulic mortar whilst the thin sections suggest that local Thames valley sand was used in all three mortars, varying in quantity, but that differences in composition and texture suggest a different source, within the Thames valley, for the sand used in the mortar raft and that used in the late lime and hydraulic mortars.

### Bibliography

Anon (2004-2007) Pozzolana. <http://en.wikipedia.org/wiki/Pozzolana>

Dickson, J. A. D. (1965) "A modified staining technique for carbonates in thin section." *Nature*, 205, 587

**Appendix 1: ICPS Data for Context 6 Major elements (percent oxides)**

TSNO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	MnO
V4530	66.47	0.57	1.23	0.25	31.20	0.03	0.06	0.02	0.13	0.04
V4531	75.26	0.60	1.37	0.26	22.19	0.04	0.07	0.02	0.15	0.04
V4532	57.46	0.61	0.89	0.38	40.37	0.04	0.06	0.04	0.10	0.05
V4533	74.61	0.64	1.47	0.26	22.71	0.03	0.07	0.03	0.14	0.04
Mean	68.45	0.61	1.24	0.29	29.12	0.04	0.07	0.03	0.13	0.04
SD	8.34	0.03	0.25	0.06	8.56	0.01	0.01	0.01	0.02	0.00

**Appendix 2: ICPS Data for Context 6 Minor elements (parts per million)**

TSNO	Ba	Cr	Cu	Li	Ni	Sc	Sr	V	Y	Zr*	La	Ce	Nd	Sm	Eu	Dy	Yb	Pb	Zn	Co
V4530	35.00	7.70	18.00	3.00	7.00	1.00	393.00	17.00	7.00	9.00	7.00	8.00	7.43	0.80	0.21	0.90	0.60	504.40	29.00	3.00
V4531	33.00	7.70	15.00	2.00	8.00	1.00	258.00	24.00	8.00	10.00	7.00	13.00	7.52	0.70	0.30	1.00	0.50	210.60	33.00	2.00
V4532	24.00	7.70	11.00	1.00	4.00	1.00	368.00	17.00	7.00	9.00	6.00	15.00	6.58	0.60	0.24	1.00	0.60	53.30	23.00	2.00
V4533	51.00	8.80	16.00	3.00	9.00	1.00	286.00	24.00	7.00	11.00	8.00	10.00	8.37	1.10	0.20	0.90	0.60	143.00	31.00	3.00
Mean	35.75	7.98	15.00	2.25	7.00	1.00	326.25	20.50	7.25	9.75	7.00	11.50	7.47	0.80	0.24	0.95	0.58	227.83	29.00	2.50
SD	11.24	0.55	2.94	0.96	2.16	-	64.49	4.04	0.50	0.96	0.82	3.11	0.73	0.22	0.05	0.06	0.05	195.32	4.32	0.58

**Appendix 3: ICPS Data for Context 5 Major elements (percent oxides)**

TSNO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	MnO
V4528	75.51	0.93	1.43	0.51	21.01	0.10	0.28	0.05	0.15	0.03
V4527	76.57	1.02	1.45	0.53	19.80	0.10	0.29	0.05	0.16	0.03
V4529	75.96	1.14	1.42	0.74	20.15	0.10	0.29	0.05	0.12	0.03

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V4526	72.14	1.77	1.43	0.47	23.45	0.13	0.38	0.06	0.13	0.04
Mean	75.04	1.22	1.43	0.56	21.10	0.11	0.31	0.05	0.14	0.03
SD	1.99	0.38	0.01	0.12	1.65	0.02	0.05	0.01	0.02	0.01

*Appendix4: ICPS Data for Context 5 Minor elements (parts per million)*

TSNO	Ba	Cr	Cu	Li	Ni	Sc	Sr	V	Y	Zr*	La	Ce	Nd	Sm	Eu	Dy	Yb	Pb	Zn	Co
V4528	76.00	9.90	20.00	7.00	11.00	1.00	253.00	18.00	7.00	8.00	8.00	8.00	8.37	0.40	0.30	0.90	0.70	94.90	28.00	2.00
V4527	69.00	9.90	17.00	6.00	10.00	1.00	211.00	20.00	7.00	11.00	7.00	14.00	7.61	0.80	0.20	1.10	0.70	152.10	32.00	3.00
V4529	73.00	9.90	16.00	8.00	10.00	1.00	226.00	18.00	7.00	11.00	8.00	11.00	8.37	1.00	0.30	0.90	0.60	123.50	29.00	3.00
V4526	98.00	12.10	15.00	7.00	10.00	2.00	294.00	23.00	7.00	23.00	14.00	17.00	14.19	0.60	0.30	1.10	0.80	193.70	37.00	3.00
Mean	79.00	10.45	17.00	7.00	10.25	1.25	246.00	19.75	7.00	13.25	9.25	12.50	9.64	0.70	0.27	1.00	0.70	141.05	31.50	2.75
SD	12.99	1.10	2.16	0.82	0.50	0.50	36.41	2.36	-	6.65	3.20	3.87	3.06	0.26	0.05	0.12	0.08	42.16	4.04	0.50

*Appendix5: ICPS Data for Context 3 Major elements (percent oxides)*

TSNO	SiO2	Al2O3	Fe2O3	MgO	CaO	Na2O	K2O	TiO2	P2O5	MnO
V4522	75.47	2.74	2.37	0.44	18.42	0.13	0.16	0.14	0.09	0.04
V4525	71.38	3.08	2.28	0.49	22.25	0.10	0.12	0.16	0.10	0.04
V4523	63.91	3.15	1.66	0.60	29.94	0.10	0.20	0.17	0.17	0.10
V4524	71.14	3.29	2.21	0.52	22.28	0.12	0.13	0.17	0.10	0.04
Mean	70.47	3.07	2.13	0.51	23.22	0.11	0.15	0.16	0.12	0.06
SD	4.80	0.23	0.32	0.07	4.83	0.01	0.04	0.01	0.04	0.03

*Appendix6: ICPS Data for Context 3 Minor elements (parts per million)*

TSNO	Ba	Cr	Cu	Li	Ni	Sc	Sr	V	Y	Zr*	La	Ce	Nd	Sm	Eu	Dy	Yb	Pb	Zn	Co
V4522	106.00	27.50	17.00	10.00	19.00	3.00	189.00	41.00	10.00	20.00	10.00	16.00	10.62	1.20	0.43	1.30	0.90	111.80	29.00	5.00

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V4525	137.00	27.50	28.00	7.00	19.00	4.00	175.00	45.00	12.00	31.00	12.00	19.00	12.78	1.40	0.54	1.60	1.20	232.70	40.00	5.00
V4523	58.00	28.60	25.00	6.00	21.00	4.00	213.00	34.00	14.00	51.00	16.00	22.00	17.01	1.70	0.48	2.10	1.10	2,919.80	44.00	6.00
V4524	130.00	26.40	22.00	7.00	17.00	4.00	144.00	45.00	12.00	31.00	12.00	18.00	12.69	1.50	0.45	1.50	1.10	234.00	39.00	5.00
Mean	107.75	27.50	23.00	7.50	19.00	3.75	180.25	41.25	12.00	33.25	12.50	18.75	13.28	1.45	0.48	1.63	1.08	874.58	38.00	5.25
SD	35.72	0.90	4.69	1.73	1.63	0.50	28.81	5.19	1.63	12.92	2.52	2.50	2.68	0.21	0.05	0.34	0.13	1,364.69	6.38	0.50