APPENDIX 3

AN INVESTIGATION INTO ANCIENT MORTARS

by

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Beginning in 1993, the Swedish Institute in Rome and stone conservation specialist Marie Klingspor Rotstein started a collaboration project in order to examine construction techniques used in Etruscan stone walls in San Giovenale, specifically in the stone walls of the housing on the Borgo. The aim was to examine if there was any evidence of lime grout or mortar usage in the walls, and if there were traces of plaster on the surfaces. If so, it would prove the Etruscans used advanced techniques. Professor Carl Nylander, architect Börje Blomé and Marie Klingspor Rotstein conducted a visual survey that year. Samples were taken for analysis. Complementary *in situ* research was done in the spring of 1994 (*Fig. 136*).

The walls had been thoroughly cleaned during excavation. By foresight, soil baulks had been left intact in certain places, close to some of the walls, thus consisting of untouched material (*Fig. 137*). Samples were taken from this soil by inserting steel rods, which then came to contain material from outside the walls and from fillings between blocks (*Fig. 138*). In addition, samples were taken from flooring material and layers (*Fig. 139*), and from a section which presumably could have been used as a hearth. The authors have later, together with the Copernicus University in Poland, undertaken petrographical (lithological) analyses, supported by X-ray diffraction.

THE INVESTIGATION[232]

Altogether close to 50 samples were taken from joints, plaster, flooring and a hearth. Some of these were selected for analysis. Below follows an account of the analysed material, which forms the basis for the conclusions.

ANALYSIS OF SAMPLES FROM BUILDING MATERIALS

Altogether 14 samples were analysed. Of these samples, twelve were chosen for microscopic analysis (planimetric analysis under polarizing microscope) and eight were chosen for X-ray examination (X-ray diffraction). Altogether 20 analyses were thus made. The following samples were chosen for petrographic analysis:

- saved soil in the baulk by the walls: one sample: no. 0A
- stone block: one sample: no. 0 (*Figs. 140–141*)
- mortar from the joint: three samples: nos. 3 (*Fig. 142*), 5 (*Fig. 143*) and 10B (*Fig. 144*)
- material on the walls—“plaster”: two samples: nos. 11 and 14
- material in “floor”: five samples: nos. CN1, CN3, CN16, CN17 and CN19
- material in “hearth”: two samples: nos. 21A and 22.

MACRO- AND MICROSCOPIC DESCRIPTION OF SAMPLES

Sample from soil (*Fig. 145*)

Sample no. 0A is from the saved, untouched soil baulk adjoining the walls. It is a porous formation of fragments of vulcanite with grains of dark glass, for example obsidian; fragments of rock with white mineral substance; non-crystalline silicon dioxide (perhaps including chabazite); with a presence of detritric quartz (perhaps not in large quantities but clearly present); also detritic or uncrystallized feldspar—orthoclase and decomposed biotite.

Sample from block

Sample no. 0 is a largish rock fragment, somewhat compounded; can be crumbled with ease.

A slight porosity is evident in the sample. The colour of the rock: greyish rusty with a very slightly blotchy appearance, derived from the presence of integral elements of a differing colour. Some of these were white crumbling clumps, with a circumference

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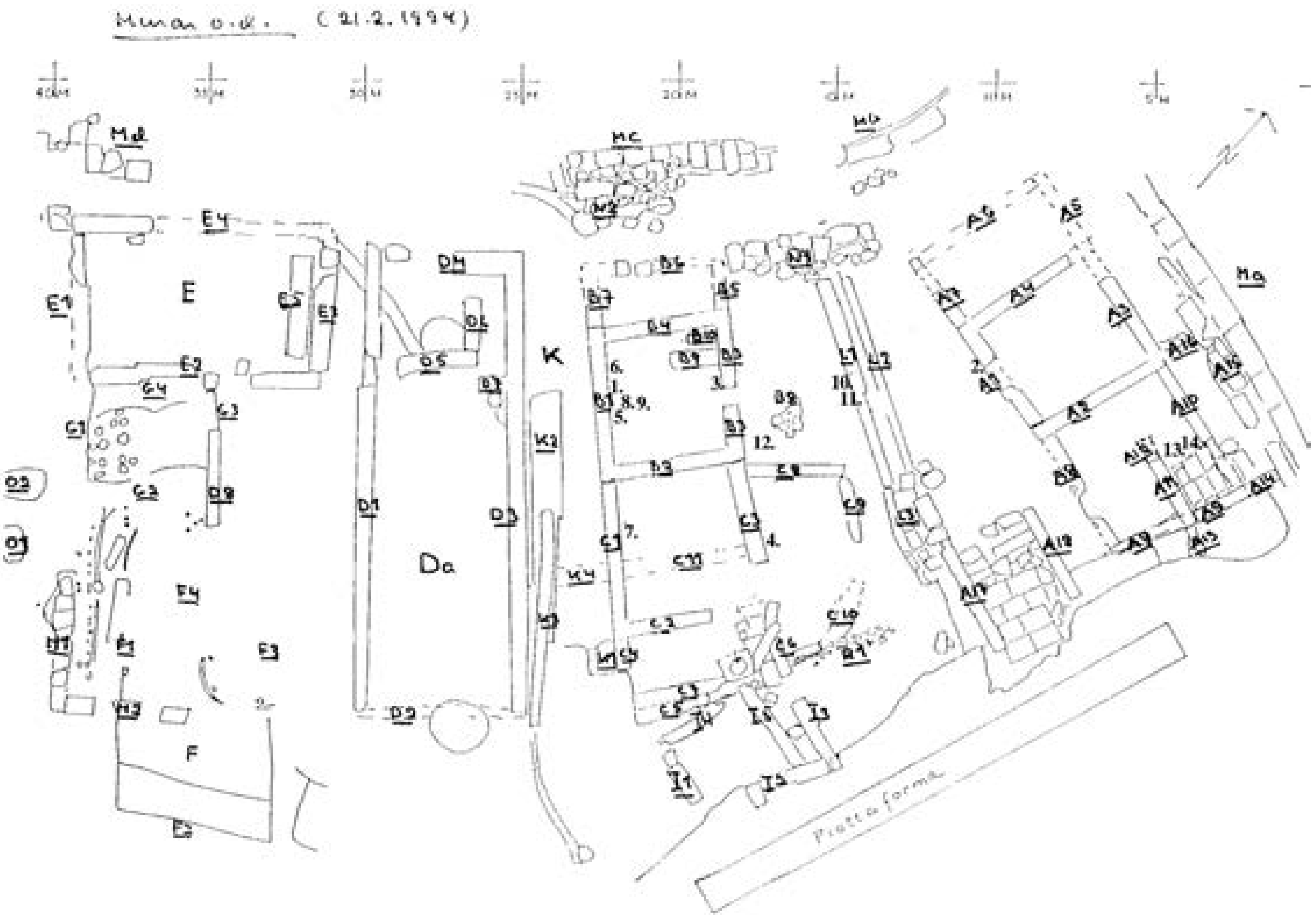

1. Sample 0A soil
2. Sample 0 block
3. Sample 3 joint
4. Sample 5 joint
5. Sample 10B joint
6. Sample 11 plaster
7. Sample 14 plaster
8. Sample CN1 floor
9. Sample CN3 floor
10. Sample CN16 floor
11. Sample CN17 floor
12. Sample CN19 floor
13. Sample 22 hearth
14. Sample 21A hearth (slag)

Fig. 136. Drawing showing spots where samples described in the text were taken.

of up to 0.5 cm, and some dark grey, up to 2 cm in circumference. These components are volcanic lava with a fibrous structure. In addition, there are black and dark brown lava fragments, but the latter are less hard and disintegrate easily under pressure. Also seen under a microscope are black grainy shapes, usually flattened or elongated. Thin scale-like shapes (most likely mica) can be split from these. Colourless, flat, angular, glass like, transparent minerals are also sporadically in evidence.

Sample from mortar in joint

The samples are loosely bound and fragment into clumps of differing sizes and have a greyish rusty colour. Sometimes there are white clumps crumbling into powder, as well as white, more compounded and crusty shapes or mineral clusters in the basic paste. Sporadically, grains of detritric quartz (crushed), small scale-like mica shapes, and flat, transparent, glass-like shapes, that are not quartz crystals, are evident. There are fragments of volcanic lava in the various samples.

There are also grey, bigger clumps of volcanic lava fragments (represented by differing shapes depending on the samples). As in the previous sample, small, dark, roundish grains can also be seen. This material has been isolated from its natural context, and a forced phase of change, due to weathering, diagenesis and the arrival of secondary minerals, richly represented in this sample, is the result.

Interestingly, one of the samples sported organic shapes: an unknown insect, perhaps not surprising if an insect had hidden in a crack in the rock. Vegetation, in the shape of nutshells, is another surprising finding.

The samples from soil, block and joint have different colours. Soil is greyish brown, joint is brown, and block also seems to be brown, but when crushed, the fresh fracture is rust- and grey coloured with a clear glass sheen and thus has a more crystalline shape.

The samples also show differences in density. The sample from soil is loosely bound with a presence of somewhat more contained clumps. Joint is denser, but crumbles despite this fairly easily, as does most mortar. Block is the densest, hardest, and difficult to crush—as for example weathered rock.

Fig. 137. Area Bc between walls C8 and B3, with saved untouched soil in the baulk. Photograph by M. Klingspor Rotstein.

Fig. 138. Filling between the blocks. The filling differs visibly from the surrounding tufa blocks. Photograph by M. Klingspor Rotstein.

Samples from "plaster"

Sample no. 11. The outer surface of the sample has a greyish rusty colour and two layers can be seen on the fresh fracture; the first, thicker, layer is lighter in tone and homogeneous, meaning without any clear presence of other, coloured, mineral grains. However, laminates are evident in places. The second layer is thinner and darker, and has the same tone as the outer surface. In this area there are crumbled grains of mineral and "microscopic rock fragments" of different mineral collections.

Sample no. 14 (*Fig. 146*). The outer surface of this sample has a light brown colour; this tone is similar but not homogeneous on the freshly produced surfaces. There are lighter and darker areas and stains. Even traces of lamination can be seen. Also, crushed grains are evident. There is a very thin fragment in the lower part of the sample, looking like a separate, darker, layer; this occurs only occasionally, corresponding with the darker layer in sample 11. In the planimetric analysis, these fragments were classed as specific results of layering.

Samples from "floor"

Sample no. CN1 is relatively compounded, in parts fragile however, with a tendency to fall apart. The colour is light brown and the outer surface (the flooring) is uneven, not levelled, and may be corroded, evidence of which is its brownish grey colour. Fresh cuts have a light brown colour, with somewhat stained, partly greyish brown mineral additions with small white and black spots. When magnified, the sample looks like a compact mass, although somewhat porous, with visible, slightly dark fragments—lava fragments and very small or uncoloured, glass-like grains of mineral.

Sample no. CN3 has a greyish, non-homogeneous tone, brown in places. Other features are as in CN1.

Sample no. CN16 (*Fig. 147*) has a light brown colour, occasionally somewhat rusty. It is generally heterogeneous and varied, with black lithological elements (lava). This sample is relatively frail. The other properties of this sample are analogical with earlier floor samples.

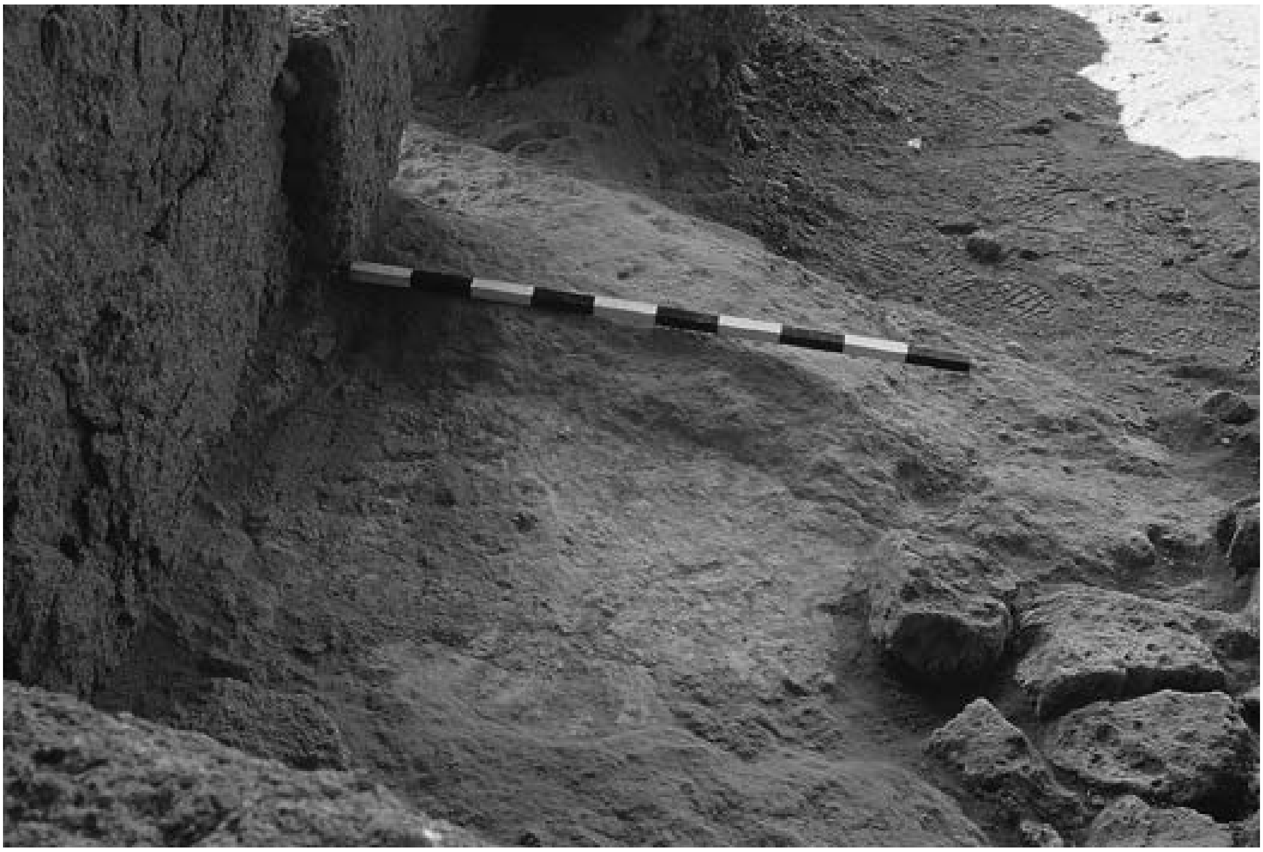

Fig. 139. Area Bc between walls C8 and B3, freshly excavated and swept. The ground appears to consist of light, levelled clay. Photograph by M. Klingspor Rotstein.

Fig. 140. Yard Ad, wall A1. Some of the samples were taken from the untouched filling material of the saved soil of the baulk, using tubes. Left of this sample is the spot where a tufa sample 0, block was taken. Photograph by M. Klingspor Rotstein.

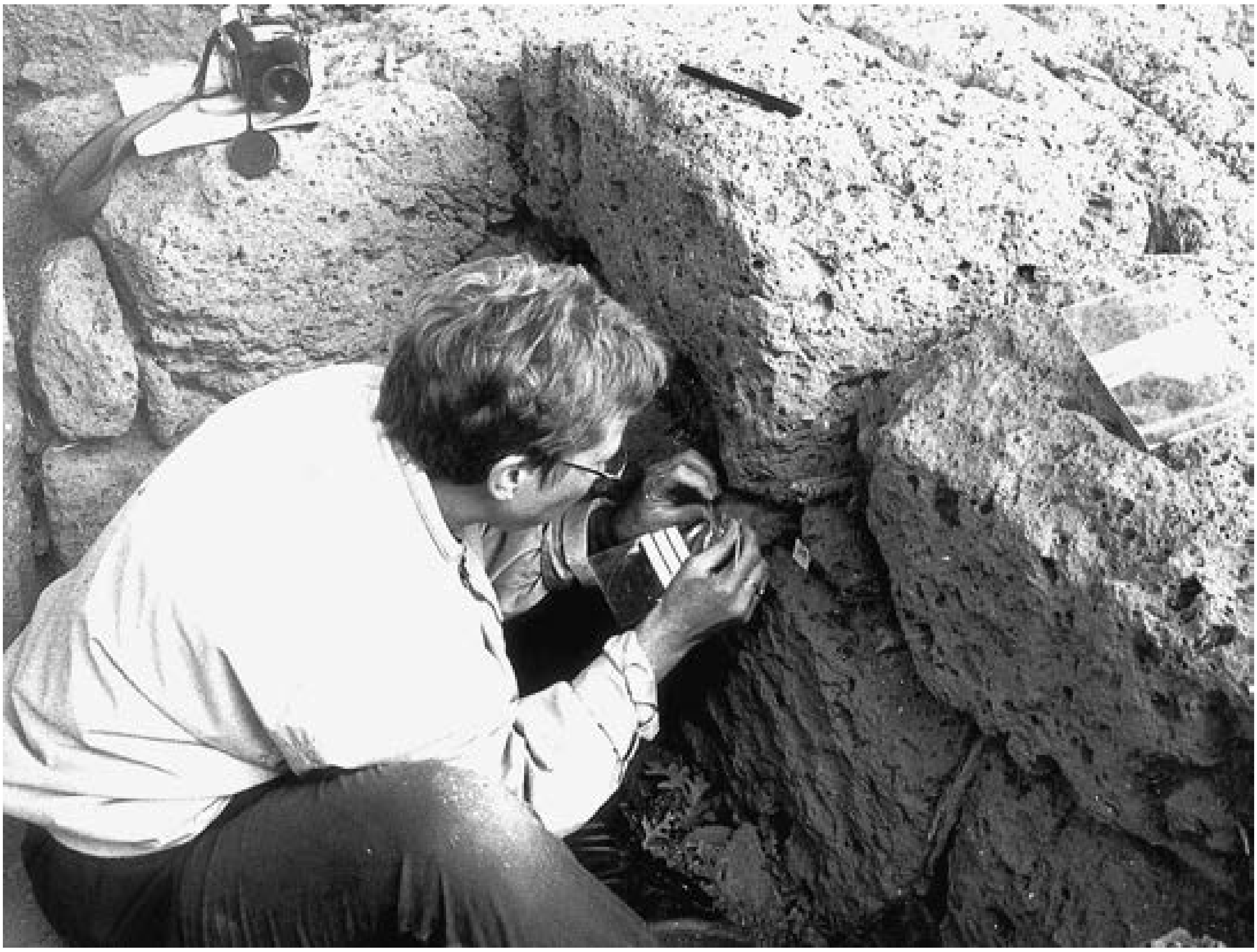

Fig. 141. It was quite difficult to extract a big enough piece to be suitable for analysis, from the frail material in the joints. Photograph by B. Blomé.

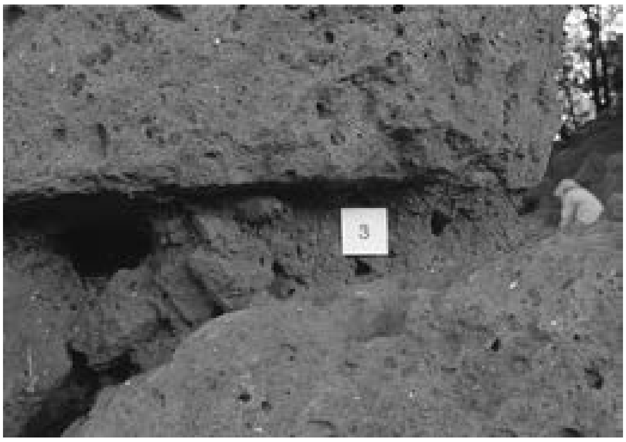

Fig. 142. Room Ba, wall B3. Sample 3 from joint consisted of quite solid larger pieces. Photograph by M. Klingspor Rotstein.

Sample no. CN17 (*Fig. 147*) has a greyish brown colour with a clear porosity, but is, despite this, quite compounded. The contents correspond with those of earlier samples. It differs only by a smaller amount of glass-like uncoloured minerals, and a larger amount of white, compounded minerals (crust-like).

Sample no. CN19 (*Fig. 148*) is a completely different looking sample. The overwhelming colour is light, almost white, with a tendency towards yellow (orange). Weathering has given the multicoloured material on the surface a greyish tone, which gives an impression of silt. The sample is very frail, loosely compounded. It disintegrates into flat laminates, parallel with the surface level. Some areas are clearly free from iron. Fragments of darker lava can be seen sporadically, and there are brownish centres, resulting from water filtration. There is no evidence of minerals (secondary contents) in the sample, but areas of iron like infiltrations are visible.

Samples from “hearth” (*Fig. 149a*)

The first sample, no. 21A (*Fig. 149b*), differs completely from the above descriptions. It consists of fragments of differing sizes, generally flat, with clear composition of the plates as in “slip form”. Some pieces look like slag. They are made up of a greyish, finely porous mass, or have a light fibrous structure.

Small white clumps of mineral are sporadically visible. The surfaces are either rust coloured or reddish rusty (haematite) or then completely metallic (iron) with characteristic depressions (canals) on the surface in the shape of clear lines, arranged in concentric ellipses or similar markings. Here and there the depressions are covered by collections of clearly formed holes

Fig. 143. Area Cc, wall C3. Sample 5, from joint. Photograph by M. Klingspor Rotstein.

(possibly from air bubbles). The third shape in this sample consists of small black and metallic shiny grains. The next shape in the sample consists of flat pieces, neither covered by haematite, nor by canal systems, but covered with a mineral substance, reflecting either mica or coal. The last shape is made of small greyish grains, which look like they might derive from solidified ash from a fireplace, i.e. diagenesis.

The second sample, no. 22, is made up of two small pieces. Both pieces are rather compounded and show a certain separation into layers. The bigger piece shows an infiltration of a white mineral substance. The sample has a light brown, occasionally greyish, colour. There are white mineral clusters in the light brown areas. There is graininess in the grey areas. The grains differ in size and colour. Also evident, are bigger elements, in the shape of black grains, or uncoloured, glass-like mineral shapes, commonly found in the earlier samples. This sample is not typical of a hearth.

PLANIMETRIC ANALYSIS OF THE SAMPLES

During planimetric analysis, 15 components were discernible. All of them do not occur simultaneously in one sample. The main ingredient is so-called volcanic ash. In this context it is a fairly vague concept, as, at this stage it is not a question of pure ash anymore. It is probable that this ash in its original form, together with other lithological ingredients, was ejected from the volcanic crater and then carried by the wind, or by water, to distant areas. Thereafter it was, through different geological weathering processes, enriched with clay minerals (illite, montmorrilonite, caolinite) and very often with different iron compounds. Under the polarizing microscope, if not masked or torn into smaller or larger monomineral pieces, this ash is practically indiscernible in such a lithological collection. Certain amounts of iron, as a result of diagenetic processes masked into clear clumps, form a special component, visible in the tables. The brown colour of analysed pyroclastic sediments shows the amount of evenly distributed trivalent iron. Due to the fact that we here deal with a mixture of volcanic glass, and as a main ingredient, volcanic ash, as well as clay minerals and iron oxide combinations, and because these cannot be seen separately under the polarizing microscope, we have given this mixture the working name ash. Some fragments in this ash mixture show a weak reaction to the polarized light. The micro-section of the sample looks like abstract mosaic under the microscope: dark, uneven, wormlike mineral forms (consisting of a large amount of iron) penetrate and disappear into the light background which consists of clay minerals or glaze of volcanic ash (non-crystalline or partly crystallized). There are also fragments of volcanic lava in this white-grey-brown mosaic, as well as fragments of porphyry and various particles or crystalline minerals.

The second component is fragments of volcanic rock, pieces of petrified lava. These particles derive from different kinds of

Fig. 144. House B, saved soil B1. Sample 10B was taken from the joint behind the saved untouched soil of the baulk. Photograph by M. Klingspor Rotstein.

Fig. 145. House B, wall B1, saved soil from the baulk. Samples floor CN1 and CN3 are described in the text. Sample 0A (zero A) soil was also taken from this piece of soil as comparison. Sample 10B was likewise taken from this place. Photograph by M. Klingspor Rotstein.

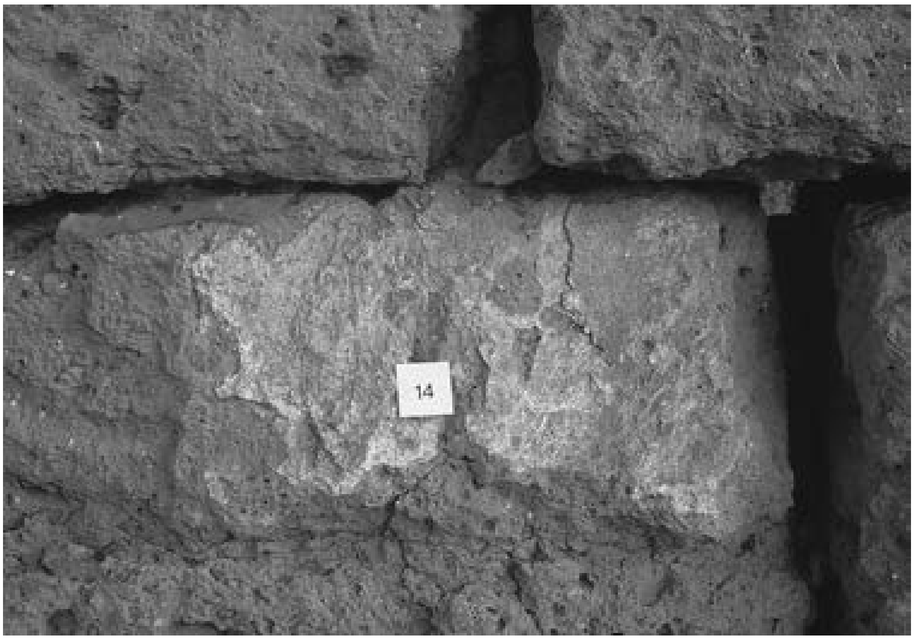

Fig. 146. House C, wall C1. Sample 14, "plaster". Similar white hard layers were found on a number of places on the blocks. Photograph by M. Klingspor Rotstein.

lava. The most common is "fibrous lava", where the volcanic glass, crystallizing when cooling, developed oblong shapes, i.e. rather loosely entwined long fibres. The second shape consists of fragments or grains of "porous" lava. These are greyish brown, non-transparent, usually rounded particles, with a large amount of pores, likewise round. Some are filled with a non-crystalline mass. In addition, there are small fragments of black or dark brown lava, sometimes with only the occasional pore—this is obsidian. The other lava varieties, found in the samples in smaller amounts, have been sorted under "other". Number one among the mineral shapes is chabazite, most likely followed by the related mineral heulandite, and the non-zeolitic mineral hauyn. These minerals are very similar and difficult to distinguish—this is the reason they have been joined together in the planimetric analysis under the name of the main mineral, chabazite.

A mineral from the pyroxene group is also clearly discernible. Non-quartz porphyry fragments and oxides and iron hydroxides in the shape of small brown, non-transparent clumps are sporadically visible.

Feldspar and detritic quartz from other kinds of rock can also sporadically be seen—additions found only in mineral material that have been treated by humans (e.g. mortar).

Fig. 147. Area Bc, saved soil in a baulk by Drain L. Samples CN16 and CN17 are described in the text. Photograph by M. Klingspor Rotstein.

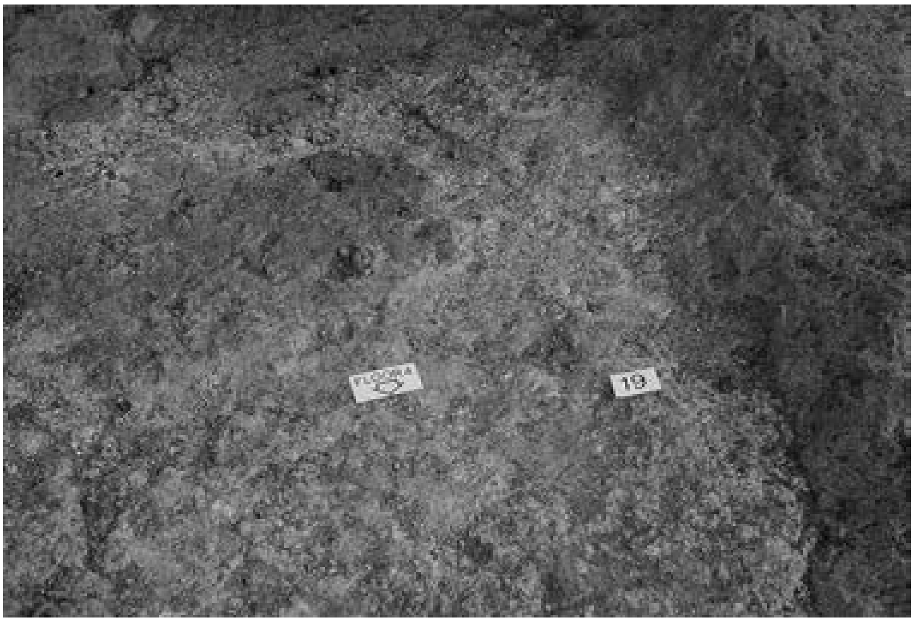

Fig. 148. Area Bc, outside House B, by wall B3 where sample CN19 floor was taken. Photograph by M. Klingspor Rotstein.

Non-crystalline mineral shapes, so called "iron infiltrations", found only in samples from joint and floor, constitute a separate problem.

X-RAY ANALYSIS

X-ray Diffraction was done in order to confirm and define the result of the microscopic analysis.

Samples from the soil by the wall, from block and joint

A clay mineral, in the shape of a mixture, with illite and montmorillonite as a product of transformation of other minerals, was found in the diffractogrammes of samples taken from walls (blocks and joints) and adjoining soil.

The zeolite mineral, chabazite, is also very much in evidence. Chabazite and the very similar heulandite, are products of hydrothermal conversion. They are also found as components in vulcanites. As converted minerals, they fill up voids, i.e. empty

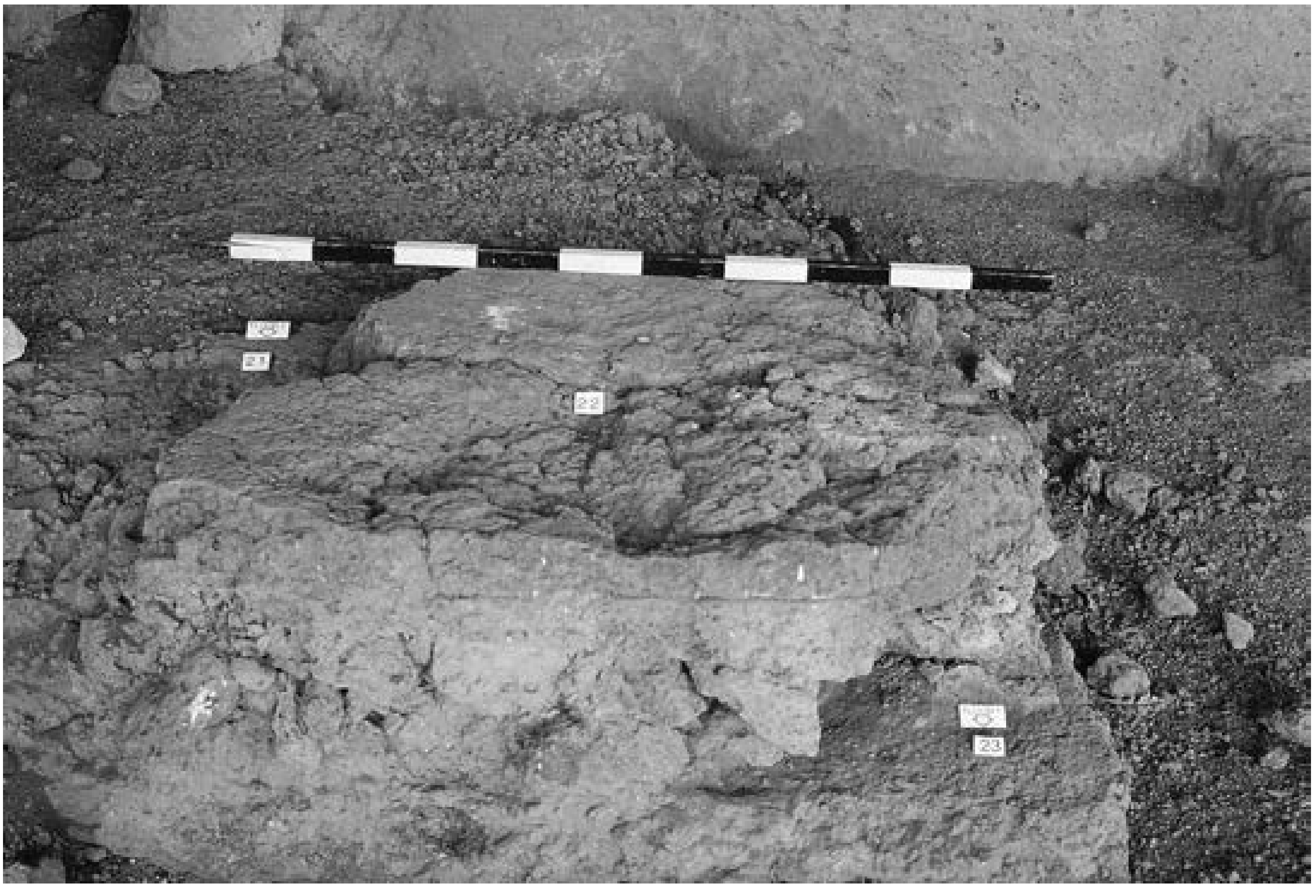

Fig. 149a. Work area Ac, an area burnt red. Sample 22 was taken here. Sample 21A comes from the side of the hearth from what was presumed to be slag. Photograph by M. Klingspor Rotstein.

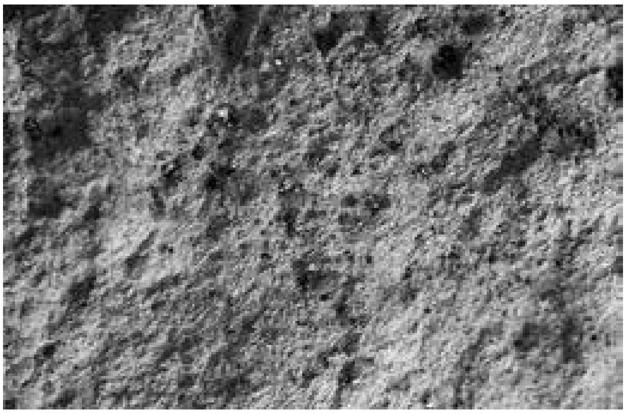

Fig. 149b. Sample 21A. Photograph by M. Klingspor Rotstein.

areas (pores, cracks) in the rock. Some of these minerals have converted into hauyn and nepheline through pseudo-morphosis; this is a very important finding. Because of this, they can be seen in some samples, but are absent in others.

The white fragments or grains, occurring in pure or laminated form, could be precisely this rock background made of chabazite or rock containing some of this mineral. The white fragments are found in all the samples. The presence is slight in the samples taken from the soil, somewhat larger in samples from the joints, and considerable in samples from the block. There is evidence of a certain amount of detritic quartz from other kinds of rock in the samples taken from the soil. The same, in a small amount, is also present in the samples from joint, but not in the sample from the block—this, despite the fact that the diffractogramme shows a presence of silicone dioxide. This presence does not derive from detritic quartz but from silicon dioxide, which has recently crystallized but not yet formed larger crystals.

Samples from “plaster”

X-ray analysis of samples show a presence of illite, montmorillonite, and chabasite as well as leucite and crystalline silicone dioxide, the latter probably a result of re-crystallizing volcanic components.

Samples from “floor”

Clay minerals (montmorillonite and illite) and chabazite prevail in the samples taken from the floor. Other minerals were also identified: sodalite (belonging to the same group as hauyn) and leucite (feldspar). Surprisingly, one of the samples (CN3) showed a presence of mullite—a mineral found in burnt clay (mudbrick production), and of tridymite—a kind of quartz, also produced under high temperatures. This confirms that the samples were taken from the floor by the hearth.

Samples from “hearth”

The analysis of samples from the “hearth” gave very interesting results. The following were identified: illite, chabazite, montmorillonite, but also mullite and tridymite, the same minerals as in one of the samples from the floor, but here in much larger quantities. Small amounts of montmorillonite in samples show that the mineral has turned into mullite under high temperatures (950–1300°C). Tridymite is polymorphic quartz, which also stabilizes at very high temperatures (870–1470°C).

CONCLUSIONS

The results indicate that a lime-free mortar was used to join the blocks.

The analysis confirms that the walls were built of natural stone blocks made from a volcanic rock, the tufa. The material between the stone blocks can be classified as a purposely used mortar, joining together the blocks. In addition to volcanic material and clay mineral (clay), grains of quartz are also in evidence. Volcanic tufa does not contain quartz, so these grains must have been added to the mortar during production. At this point the question arose whether here was a specific kind of (hydraulic) fixing agent—volcanic *pozzolana* and clay mineral—or a primitive agent in the shape of ordinary clay with volcanic tufa particles as ballast.

Further analysis of the samples from the joints confirmed that the material is a purposely used mortar. The occurring quartz is not ballast, but the percentual content of this material is incidental. However, the presence of quartz in all the samples is proof that the material in the joints is not weathered matter from the tufa blocks, but a specially added mixture, “mortar”. There is no evidence that the volcanic material was added as ballast or as a hydraulic extra. There is no proof that it is there by coincidence either, as a contamination of the clay which derives from volcanic terrain. The large amount of this material in the samples is, however, an indication that it could have been specially added. The research did not allow us to conclude that here is an advanced mortar, but showed instead that it is a more primitive mixture of clay and volcanic material. All the examined joints have clear marks of water usage during production.

The white “plaster” strata were examined. *In situ* they look as if they were specially added to certain stone surfaces as they, with their almost white colour and dense structure, differ from the brownish and very porous tufa blocks. Petrographic research shows that one of the samples was made up of several layers, laminates, which is not a naturally occurring phenomenon. Despite this, the analysis did not yield any evidence that allowed the white layers to be interpreted as plaster, because they did not contain anything new or foreign compared with the underlying tufa.

The specially treated floors were also examined. The samples show that the different levels (floor) in the saved soil adjoining the wall contain clay minerals and volcanic material, in other words similar material as in the joints. However, only one of the analysed layers, the one in area Bc outside House B, wall B3, showed clear marks of having been prepared with water. This layer also looked different from the others. It had a much decomposed surface layer which points to a long period of wear and tear, water etc. and can be classified as a floor.

When samples were taken from the excavated hearth interesting discoveries were made. The research shows that the hearth itself had been heated to very high temperatures. The existence of minerals that develop during burning in temperatures between 1000–1500°C indicates this. In addition, “slag” by the hearth turned out to be iron compounds like haematite, pure metallic iron and slag. Consequently, this is evidence that iron was handled and worked in San Giovenale. It is possible that the nearby basin was used in this connection.

Table 1. Sample from block: summary of lithological composition (volume percentage).

Sample	*Volcanic ash*	*Fibrous lava*	*Black lava*	*Porous lava*	*Other kind of lava*	*Porphyry*	*Chabasite*	*Iron compounds*	*Pyroxenes*	*Feldspars*
0	62,7	10,2	3,0	10,3	2,4	1,1	2,9	2,6	4,2	0,6

Table 2. Samples from joints: summary of lithological composition (volume percentage).

Sample	*Volcanic ash*	*Fibrous lava*	*Black lava*	*Porous lava*	*Other kind of lava*	*Porphyry*	*Chabasite*	*Iron infiltrations*	*Iron compounds*	*Pyroxenes*	*Feldspars*	*Quartz*
3	56,0	12,6	6,6	3,4	3,1	4,8	4,2	2,3	3,0	1,9	1,2	0,9
5	56,8	13,0	6,2	4,8	2,1	2,1	4,8	4,5	1,9	2,2	1,3	0,3
10B	55,4	7,9	5,0	5,9	1,7	4,8	3,9	10,4	2,9	2,1	–	–

Table 3. Samples from “plaster”: summary of lithological composition (volume percentage).

Sample	*Volcanic ash in “plaster”*	*Lava fragments in “plaster”*	*Chabasite in “plaster”*	*Pores in “plaster”*	*Crust in “plaster”*	*Volcanic glaze in “plaster”*	*Volcanic ash in block under “plaster”*	*Lava fragments in block under “plaster”*	*Chabasite in block under “plaster”*	*Volcanic glaze in block under “plaster”*
11	66,9	3,6	–	24,3	5,2	–	47,6	42,9	9,5	–
14	71,9	5,7	1,2	17,4	–	3,8	51,4	37,2	–	11,4

Table 4. Samples from “floor”: summary of lithological composition (volume percentage).

Sample	*Volcanic ash*	*Black lava*	*Porous lava*	*Fibrous lava*	*Other kind of lava*	*Chabasite*	*Iron compounds*	*Pyroxenes*	*Porphyry*	*Feldspars*	*Volcanic glaze*	*Iron infiltration*
CN1	55,6	1,6	33,1	4,5	1,4	0,9	0,8	1,1	1,0	–	–	–
CN3	75,1	4,9	7,2	3,9	1,6	1,6	1,1	1,2	1,8	–	1,6	–
CN16	76,2	3,2	0,7	6,6	4,4	2,8	1,5	0,9	2,6	1,1	–	–
CN17	75,7	4,8	4,6	5,2	3,5	1,8	1,4	1,1	1,3	1,3	–	–
CN19	78,3	1,1	0,9	1,6	1,3	0,8	–	–	0,4	–	2,6	13,0