## Włodzimierz Godlewski

# Old Dongola: Kom A (Acropolis), 2003

Polish Archaeology in the Mediterranean 15, 193-215

2004

Artykuł został opracowany do udostępnienia w internecie przez Muzeum Historii Polski w ramach prac podejmowanych na rzecz zapewnienia otwartego, powszechnego i trwałego dostępu do polskiego dorobku naukowego i kulturalnego. Artykuł jest umieszczony w kolekcji cyfrowej bazhum.muzhp.pl, gromadzącej zawartość polskich czasopism humanistycznych i społecznych.

Tekst jest udostępniony do wykorzystania w ramach dozwolonego użytku.



# OLD DONGOLA

## KOM A (ACROPOLIS), 2003

#### Włodzimierz Godlewski

The team from the Polish Center of Mediterranean Archaeology of Warsaw University continued excavations on Kom A in the southwestern, fortified part of the capital city of Dongola between January 17 and March 6, 2003. Previous fieldwork on Site SWN in 1999 and 2001 had covered the Cruciform Building (B.III), Palace Building (B.1) and riverside fortifications, and had extended the area under investigation to the east and north where residential architecture of the last phase of settlement was unearthed (H.1-H.4). The oldest known building in Dongola, B.IV, was traced between B.I and B.III, which used it as foundations. The key task of the season was to establish the maximum extent of the palace (B.I) and to clear in its entirety and protect the Cruciform Building (B.III.1-2) (Fig. 1). Exploration of the pottery deposit found in room B.I.15 provided important data for the chronology of the earliest stages of palace existence.

Studies focused on the collections of imported amphorae from B.I.15 (D. Bagińska), mud stoppers (A. Jaklewicz), mural painting from the Cruciform Building (D. Zielińska) and the osteological material from B.I.15 and the fill between the Cruciform Building and Palace (M. Osypińska). Protection and conservation activities in the Cruciform Building were charged to Haidar Hamid. Dr. Adam Łajtar of the SDRS expedition at Banganarti kindly consulted the reading of the epigraphical finds.

- 1) The team directed by Prof. Dr. W. Godlewski included Mr. Haidar Hamid, restorer and NCAM representative; Ms Dobiesława Bagińska, ceramologist; Ms Dobrochna Zielińska, Mr. Artur Obłuski, archeologists; Ms Anna Jaklewicz, student of archeology. The animal osteological material from site SWN was examined by archeozoologist Mrs. M. Gauza-Osypińska.
- 2) W. Godlewski, PAM XI, Reports 1999 (2000), 205.
- 3) Id., PAM XIII, Reports 2001 (2002), 203-214.

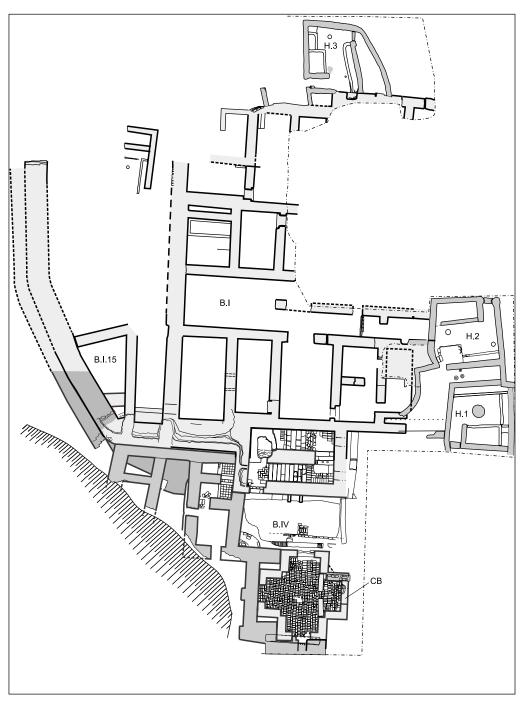


Fig. 1. Citadel. Site SWN, plan after the 2003 field season (Drawing W. Godlewski, M. Puszkarski, D. Zielińska)

## RELATIVE CHRONOLOGY OF THE ARCHITECTURE ON KOM A

A relative chronology of the architecture in the southwestern part of the ancient town (SWN) was established in the course of the fieldwork. The sequence starts at the turn of the 5th century and can be traced through the 17th century as follows:

- Building IV and the riverside fortifications
- 2. Palace (B.I) and Cruciform Building (B.III.1)
- 3. Building II
- 4. Cruciform Church (B.III.2)
- 5. Dwellings (H.1-H.4)

It is the second such chronological laddering of the architecture inside the fortifications on Kom A, the first having been established for the structures in the northwestern corner of the defense walls (NW). The latter also covered a period from the end of the 5th through the 17th century. The earliest stage, that is, the construction of the fortifications was followed by some early architecture identified only in test pits (A.111), and successively by House A.106 (House of the Ecclesiastics) which was contemporary with the Palace (B.I). After the house was abandoned, the area was long used as a place for storing goods in silos and, in the latest phase, for some kind of economic activities <sup>4)</sup>

#### **FORTIFICATIONS**

The defense wall uncovered in 2001 now continued to be excavated in a northerly direction. Its destruction at this point presumably dates to Post-Makurian times (17th century), after which the ruins were further washed out by rain water rushing down the slopes of the kom towards the river valley. The examined section was 32 m long and turned out to be homoge-

neous in construction, measuring a regular 2.70 m in width. The bedrock on which it was founded fell away rather steeply to the north, more than a meter over a distance of 30 m. No new dating evidence was unearthed, but there is no doubt that the defenses preceded the Palace (B.I), which was built against the inside face of the wall.

#### **BUILDING B.IV**

The remains of the southwestern corner of this structure, dismantled down to the floors, were found between the Palace (B.I) and Cruciform Building (B.III.1). Both the later structures were founded on bedrock, disturbing in the process the pavement and south wall of Building IV (*Fig. 2*). A section of the pavement of this earlier

building was identified also inside the staircase B.I.1.

The south wall of B.IV had rested on bedrock. It was erected of baked red brick, fairly soft and measuring at least +31.0 x 17.0 x 7.5 cm. One course of headers set on edge, which had been used as a foundation, was discovered to run for a distance of

more than 4.50 m. The southern face of this wall, which had an overall thickness of c. 70 cm, was damaged presumably during the building of B.III. The paving of B.IV consisted of baked bricks, 32.5 x 15.5 x 7.5 cm, set headers on edge on a regular bedding that looked as if it were a woven mat. The floor was cleared over a distance of over 7 m, the wall obviously taking its course to the east, under an artificial embankment; at the western end it was destroyed by the riverside defenses of the

Palace and structure B.III erected in the late 13th century (B.II).

Building IV was undoubtedly the oldest structure in this part of the town. It was quite extensive, drawn out along an E-W axis and well constructed. Its relation to the city walls in the riverside section was not determined and there was no ceramic dating material from under its floors. As it is earlier than B.I, it can be dated provisionally to the 6th century.



Fig. 2. Citadel. Site SWN, Building IV, surviving brick pavement (Photo W. Godlewski)

### PALACE BUILDING (B.1)

The objective of tracing at least the outer walls of the building on the town side and determining its size overall was not achieved despite efforts in this direction. Clearing of the layers of upper fill uncovered the top of the northern facade, but the same could not be done for the eastern, much more severely destroyed part of the building. Post-Makurian architecture, presumably of the 16th-17th

century, obscured this section, e.g. the two houses H.1 and H.2 excavated in the southeastern corner of B.I.

A projection 1.30 m deep extended beyond the line of the northern palace facade and it is to its face that the Post-Makurian house H.3 was attached. The mud brick and baked brick bond of the northern facade on the outside is identical with that in the southern facade of the



Fig. 3. Site SWN. Building B.I, south wall (staircase façade) (Photo W. Godlewski)

staircase (B.I.1). Assuming the same foundation level for the structure as a whole, this northern facade should be seen as standing over 6 m high. The utilitarian space inside the Building reached about 1000 m<sup>2</sup> on one level and at least one floor is largely preserved. Thus, the plan (cf. Fig. 1) registers walls of both the ground floor and upper floor, making it difficult to discern the room layout inside the structure. One thing is certain: the building had been meticulously planned inside and finely finished. Since the southern staircase (B.I.1) led directly to the upper floor, another entrance should be assumed as leading from the town, also

facilitating communication between the floors. The southern staircase facade has now been cleared in its entirety (section 8.75 m long) down to the foundations. The bottom parts of the walls were constructed of gray sandstone (Fig. 3). The stone wall is 1.15 m high overall, except for the section the southern entrance in southwestern corner, where it is even with the door jambs supporting a stone arch of voussoirs above the doorway.<sup>5)</sup> To the east of the entrance two stone blocks decorated with lion protomas projected from the stone base of the wall. The top back part of these stone blocks was squared and presumably served to support a horizontal

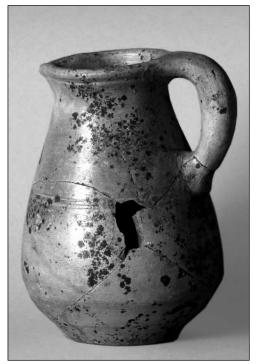


Fig. 4. Jug (Add.03.058), White Ware (Photo A. Obłuski)



Fig. 5. Lamp (Add.03.088), Red Ware (Photo A. Obłuski)

5) Cf. PAM XIII, op. cit., Fig. 3 on p. 207.

stone ledge. The structure appears to have been some kind of bench. The upper sections of the south facade were erected of baked brick on the outside and mud brick on the inside, forming a uniform construction. Windows lighting up the staircase were pierced only through the east wall.

The clearing of the pottery deposit in unit B.I.15, which was a depository of toilet facilities on the upper floor, was completed, providing important dating evidence for the early stages in the existence of the building. The assemblage of tableware and local cooking pots, as well



Fig. 6. Mud stopper with round stamp (Add.03.120) (Photo A. Obluski)

as imports from Egypt (e.g. toilet vases) includes red- and white-ware products (Figs. 4,5) - mainly small hemispherical bowls, plates and basins. A small percentage of the vessels features painted decoration in the form of concentric bands on the floors of small bowls or straight and wavy lines on the ledge rims of large plates. Amphorae imported from Egypt constitute an important group. More examples of storage vessels from Aswan, Middle Egypt (LR.7) and the Mareotis were identified. 6) Previously unnoted Palestinian amphorae were represented by the upper part of a vessel with vertical rim and bag-shaped body with ribbing and painted white decoration on the body (Peacock-Williams class 46).7)

The deposit also contained numerous amphorae mud sealings with inscribed round and elongated stamps (*Fig.* 6) – over 50 examples altogether, providing good evidence for the actual number of amphorae, presumably only of the LR.7 type, supplied to the residents of the building.

Glass products from the same deposit included interesting evidence of fragmentarily preserved wineglasses and lamps used in *polykandela*. The most surprising find was a broken lump of glass of satisfactory pureness, which must be seen as evidence of glass processing taking place in the Dongola workshops. It is a separate issue where such good-quality glass could have come from. It is quite possible that blocks of glass were also imported from Egypt as raw material for processing on the spot.

<sup>6)</sup> Their presence had been noted already during the explorations in 2001, cf. id., PAM XIII, op. cit., 208-210.

<sup>7)</sup> Add.03.328: H. +32.0 cm; Dia. rim 10.5 cm; Dia. max. 35.5 cm; orange fabric; handles missing. D.P.S. Peacock & D.F. Williams, Amphorae and the Roman Economy. An Introductory Guide (London-New York 1986); E. Alliata, Nuovo settore del monastero al Monte Nebo-Siyagha, Christian Archaeology in the Holy Land. New Discoveries, ed. G.C. Bottini, L. Di Segni, E. Alliata (Jerusalem 1990), 427-466, figs. 18-19.

#### CRUCIFORM BUILDING (B.III)

The entire interior of this small structure, which started to be explored in 2001, was now cleared, as well as the northern façade. Two distinct periods of occupation were observed: initially, the building had been accessible through all four arms (B.III.1); then it was rebuilt as a small church with only one entrance from the south (B.III.2).

#### BUILDING B.III.1

Erected as a small freestanding structure, it stood south of the palace staircase (B.I), just 4 m away from the entrance facade, on open ground lying just south of the oldest structure in this area, that is, Building B.IV. The bedrock here fell away gently to the south and was already covered with an

occupational layer of varying thickness at the time that the construction took place. The red bricks used in it measured 31.0-32.0 x 17.0 x 7.5-8.0 cm and were bonded in mud mortar, the joints being c. 1.0-1.5 cm thick. Wall structure was regular with alternating layers of headers and stretchers in the face; this corresponded to courses of two rows of bricks set crosswise to the run of the wall and courses of single bricks set crosswise between two outer rows of bricks lining the edge of the wall. The walls were 63-65 cm thick. The narrow walls closing the arms with the entrance arcades were 50 cm thick and featured a modified brick arrangement: one row lining the edge of the wall and the other row with

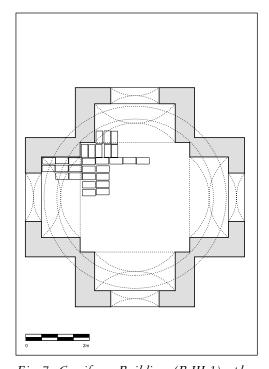


Fig. 7. Cruciform Building (B.III.1), plan (north is at the top) (Drawing W. Godlewski, D. Zielińska)

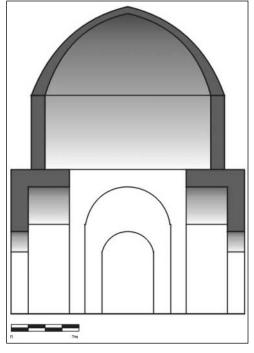


Fig. 8. Reconstruction of the Cruciform Building (B.III.1) (Rendering D. Zielińska)

bricks laid crosswise, alternating in position in successive courses, which resulted in the alternating header and stretcher courses observed in the wall faces.

The foundation of the walls in the north arm, made of headers set on edge, was on the same level at either end. A foundation trench had been cut, partly damaging the south wall of the older building B.IV; the foundation bricks were laid on a layer of fill, c. 15 cm thick, deposited on bedrock.

The outer dimensions of the building were 6.80 x 6.80 m (*Fig.* 7). Inside, the square central space measured 3.30 x 3.30 m. The four arcades opening of this center-square were each 1.20 m deep and 3.35 m wide. The entrances in the end walls of each of the four arms also had an

arcade that was 1.20 m wide and 2.50 m high. The highest that the walls of this building have been preserved was in the northeastern corner (up to 3.20 m above the floor), the lowest in the west and south (not exceeding 1.20 m in height).

The eastern arm, which is in the best condition, measures 1.20 m in length, 2.27 m in width at the outside, broadening to 3.25 m in two recessed steps toward the center of the structure. The eastern entrance in the central part of the arm, 1.22 m between the jambs, was blocked with a red-brick wall. A barrel vault covered the arm, the springing of the vault preserved on the south wall being c. 2.70 m above the floor. The topmost point of the vault inside was most probably 3.85 m above the floor. The arcade on the



Fig. 9. Cruciform Building (B.III.1). Pendentive of the dome in the outer northeastern corner of the structure (Photo W. Godlewski)

inside must have been higher, but how much higher cannot be judged on the grounds of the present evidence.

In the outer northeastern corner, where the northern and eastern arms meet, a block of sandstone was preserved, 14 cm thick and c. 30 cm wide, squared and laid along the diagonal of the two joining walls, 3.34 m above the foundation level. It was structurally one with both walls and plastered on the outside like the entire structure. It must have been a kind of external pendentive under the circular drum of the dome (*Fig.* 9).

Based on the evidence of the full plan, the walls with the arcaded entrances in the northern and western wings, the spring of the vaults and the stone pendentive described above, a reconstruction of the building has been attempted (*Fig. 8*). This small cruciform structure obviously had projecting vaulted arms and a central dome supported on a drum. The assumed total height of the structure was c. 9.20 m.

The entire building was plastered on the outside, but the plastering is best preserved in the western part, especially on the north and south walls of the western arm, which were protected by screening walls with sand filling the gap. The lime plaster<sup>8)</sup> with coarse sand added as temper formed a thick coating, the surface was smoothened. In some sections, the plaster is peeling, revealing the presence of three

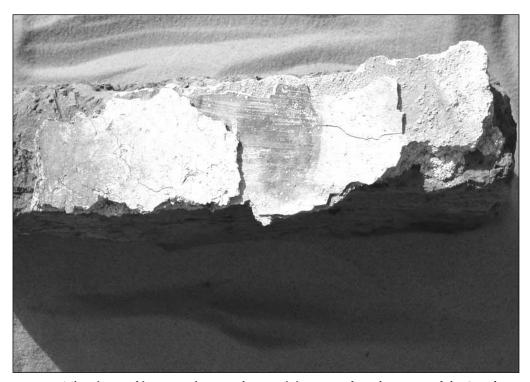


Fig. 10. Three layers of limestone plaster with painted decoration, from the interior of the Cruciform Building (B.III.1) (Photo W. Godlewski)

<sup>8)</sup> For a chemical analysis of plaster samples, see the appendices at the back of this report.

coats (by the western entrance) which would suggest successive renovations of the outside form, especially in the vicinity of the entrances.

On the inside walls, three successive coats of lime plaster were noted, corresponding to successive renovations of the interior (Fig. 10). Evidence of renovations has been preserved in the bottom parts of the walls, just above the original pavement and below the level of the late floor. It is not clear whether successive coats of plaster covered the entire interior or only the bottom parts of walls that were most exposed to damages in an open interior. The second coat of plaster was especially fine and very smooth. The entire interior was decorated with wall paintings, executed obviously by master painters in the tempera technique. On the vault of the eastern arm of the building, the first coat of plaster preserved a representation of Nubian warriors standing in a heavenly meadow.9) The bottom parts of the walls presumably had decoration imitating marble revetment and stuccowork emphasizing the tectonics of the interior.

The original paving, obscured by a later cement floor, was cleared in the western and northern arms of the structure and partly in the center (cf. Fig. 7). It consisted of bricks (33.0 x 16.0 and 36.0 x 17.0 cm, thickness as yet unknown) laid flat in a regular arrangement. From what can be said after fieldwork in 2003, the bricks in the northern arm were arranged longitudinally, that is, parallel to the lateral walls. The southern arm presumably mirrored this arrangement. In the center and in the western arm, the bricks ran parallel to the side walls in this arm, that is, following an E-W orientation. This was presumably

continued in the eastern arm. Thus, it seems that the bricks in the pavement of the original structure formed a cross, emphasizing the transverse arm with a continuous E-W alignment.

A coarse (sand with lime) cement floor, c. 2.0 cm thick, was introduced on top of the pavement. The surface was smoothened and dark red in color. Two layers are in evidence, laid rather quickly one after the other, and the first layer was not smoothened.

Between the north facade of the building and the palace there was the court with reused paving from the B.IV structure. As there was a difference of 35 cm between the levels of the Cruciform Building and the court, and the jagged edges of the south wall of B.IV were partly visible above the building's floor, it looks as if the Cruciform Building had been surrounded by a bench, now cleared along the northern façade, but presumably running all around the building. This bench was 114 cm wide, built of three courses of red bricks with a rubble, sand and stone core. In line with the entrance, on the outside of the bench, a step was discovered, also made of red bricks and measuring 85 by at least 85 cm (it must have been equal to the width of the entrance). This step facilitated climbing up onto the bench.

#### DATING AND FUNCTION OF B.III.1

No foundation stela has been discovered and there is no relatively well-dated foundation or under-floor pottery deposit to provide an independent dating for the structure. It was erected after B.IV had been leveled to the floor, presumably shortly after B.I had been built. The suggested dating to the second half of the 7th century is very likely.

<sup>9)</sup> Godlewski, PAM XIII, op. cit., 211, Fig. 6. Also see contribution by D. Zielińska in this volume.

Technological features, such as red brick used in its construction and the external plastering resemble materials used in the third Cathedral (RC), which is currently dated to the second half of the 7th century.

The importance of B.III.1 follows from its specific location: at the edge of a rocky elevation on the riverbank, above what is believed to have been a private river harbor and in front of the entrance façade to the palace B.I. The open character of the structure with four arcades in the arms and the absence of any internal furnishings rather settles the issue against its identification as a strictly sacral structure. The small size, on the other hand, and the cruciform plan suggest a funerary purpose, but as there is no recognized tomb and no trace of a sarcophagus possibly standing in the center space, this hypothesis has to be discarded. It may well be that the entrance to the crypt had been excavated in bedrock on the west, below the foundation level of B.III, and is now concealed under the 13th century screening wall, but this idea remains to be tested.

Another possibility is to consider the small domed structure on a cruciform plan as a commemorative building marking an event of importance for the city and the residents of the palace. The presence of representations of Nubian warriors in the vault of the eastern arm and the fine interior finishing, as well as systematic renovations could suggest that we are dealing with a commemoration of a military victory of some kind. The date for the erection of the building, set in the second half of the 7th century, links the structure with the siege laid to Dongola by Abdullahi abu Sarh in

652, after which a *baqt* treaty was signed with Egypt. Thus, B.III in its initial stage could have been a monument erected in honor of the citadel's defenders and to commemorate the signing of the treaty. It is noteworthy that after renegotiating the treaty with the caliphs in Baghdad in 836, King Georgios of Makuria raised a building commemorating his successful visit – most likely, the Cruciform Church (CC).<sup>10)</sup> Its monumental form of a cross with free, projecting arms would have thus referred to its ancient predecessor, the monument B.III.1.

#### B.III.2

During Makuria's wars with the Mamluk sultans Baybar and Qalawun in the second half of the 13th century, the Cruciform Building was protected behind a screening wall erected against its southern and western facades. It was subsequently severely damaged during the siege laid to the city. The area between B.I and B.III, in the court (SWN.8) and inside the staircase (B.I.1), was filled with a thick deposit containing enormous quantities of animal bones. 11)

After the situation had stabilized, presumably in the first half of the 14th century, the Cruciform Building was transformed into a small church (Fig. 11). The entrances in the eastern, northern and western arms were blocked, leaving only one door from the south. The floor level was raised by 40 cm and a new brick paving was introduced. Just in front of the passage from the central space to the eastern arcade a high altar screen was constructed, providing only a narrow entrance to the sanctuary. The interior was coated with a new mud plaster and

<sup>10)</sup> W. Godlewski, "The Cruciform Church at Old Dongola (Sudan). Some Comments", in: *Coptic Studies*, ed. W. Godlewski (Warsaw 1990), 127-137.

<sup>11)</sup> For a summary report on this and other osteological remains from the site, cf. contribution by M. Osypińska in this volume.

paintings typical of church decoration: a fragmentary college of apostles has been preserved in the sanctuary, including a portrait depiction of a bishop of Dongola, and to the north of the altar screen, severely damaged remnants of a Nativity scene. <sup>12)</sup>

The church remained in use presumably until the fall of Christianity at Dongola. After a while, the floor was covered with a thick layer of windblown sand and the interior used by the later inhabitants of the citadel as storage and household space.

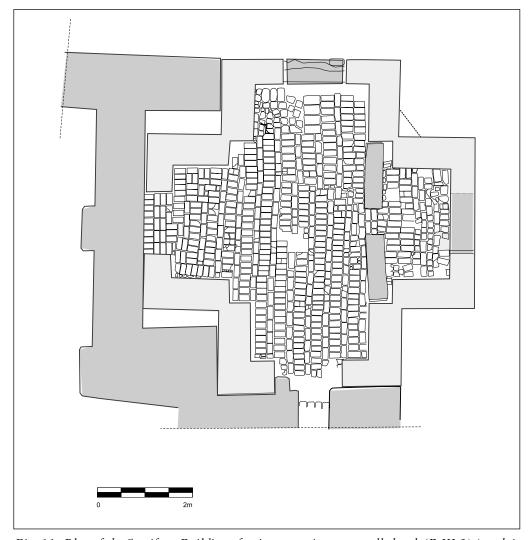


Fig. 11. Plan of the Cruciform Building after its restoration as a small church (B.III.2) (north is at the top) (Drawing W. Godlewski, D. Zielińska)

12) Also discussed by D. Zielińska in her contribution to this volume.

# "THE KINGDOM OF THE POLIS OF DONGOLA" POST-MAKURIAN TIMES (15TH-17TH CENTURIES)

After the royal court left Dongola in the early second half of the 14th century and an influx of new inhabitants occurred, there was a resurgence of architecture in the partly ruined town, on the citadel as well as in the abandoned buildings of the Cathedral and Cruciform Church. The empty and somewhat damaged palace structure was also reoccupied with dwellings being built among the ruins. During the past season, four such houses

were explored in the eastern and northern parts of the palace and in the adjoining area level with the filled in ground floor of the building.

#### SWN.H.3

A house composed of traditionally two rooms covered with a flat roof and preceded by a courtyard in front of the entrance on the west was built onto the northern facade of Building B.I. The main

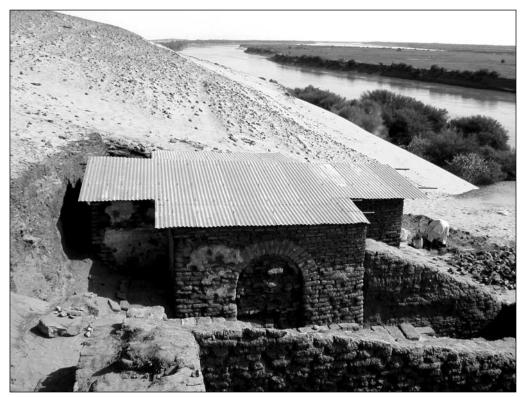


Fig. 12. Shelter built over the Cruciform Building (B.III) for protection of the murals inside it (Photo W. Godlewski)

13) The name of the kingdom survives in graffiti recently discovered at the complex in Banganarti and studied by Dr. Adam Łajtar (see Łajtar, *JJP* 33 (2003), 137-159). Cf. also A. Łajtar's report on the Banganarti inscriptions from the 2003 season in the present volume.

room of this house (H.3.1) was cleared down to the mud floor; the other room, which was much smaller, presumably served storage purposes. The walls of the main dwelling quarters were whitewashed repeatedly. The furnishings included a bench by the east wall and a large rectangular platform by the west wall, most likely a bed; two similar platforms stood in the northwestern corners of the room. A series of storage jars and cooking pots was uncovered in room H.3.1, as well as two terracotta boxes, finely painted on the outside (*Fig. 13*), intended for personal belongings or for household bric-a-brac.

#### HOUSES SWN.H.1 AND H.2

The two houses located next to one another in the eastern part of the excavated area are not contemporary. House SWN.H.1 is un-

doubtedly later. Its one square room was accessible from the south through an entrance with an inner wall around it; the flat ceiling was supported on a massive circular pillar standing in the middle of the room. Low benches lined the walls. House SWN.H.2 was entered from the north and was composed of two rooms (cleared only down to the upper mud floor this season). The three storage jars excavated in the long narrow room H.2.2 confirmed a household function for the area.

The dating of particular houses is not very precise. Locally made furnishings, chiefly pottery, occur over a long period of time. The presence of glazed ceramics and clay pipe stems imported from Ottoman Egypt suggests that the last period of occupation should be linked with the turn of the 17th century.



Fig. 13. Painted clay box from the late house H.3 (Add.03.412) (Photo A. Obłuski)

#### APPENDICES

The following are reports on three separate analyses of samples of plaster, pigment and binder from a building discovered in 2001 and excavated in 2003 on Kom A in Old Dongola. Lab analyses were carried out in Poland.

Sample no. 1 came from the three coatings of plaster found on the building exterior. Samples 2-4 represent the three successive coatings of plaster belonging to the first phase in the existence of the structure (B.III.1), starting from the innermost (no. 2) and ending in the

thickest, third layer with incised modeling of details (no. 4). Sample no. 5 originated from a fourth coating of plaster, which was laid once the building was reconstructed as a small church (B.III.2).

Four samples were submitted for pigment and binder analyses: samples nos. 9-11 from the first layer of the plaster, with blue and red pigments being analyzed in sample no. 9, black in sample no. 10, dark and light green in sample no. 11 and yellow and orange in sample no. 12.

#### APPENDIX 1

## ANALYSES OF SAMPLES OF PLASTER FROM THE CRUCIFORM BUILDING (B.III) IN DONGOLA

## Irena Koss Academy of Fine Arts, Warsaw

The plaster samples were analyzed with the microchemical method using a stereoscope microscope Mst-130 (100x) and a Zeiss Biological Microscope for transmitted light (680x). Thin sections cut for samples nos. 2, 3 and 4 were examined under an OPTON polarizing microscope in polarized transmitted light, parallel and crossed nicols (480x).

Sample no. 1 (from the exterior plasterwork near the entrance from the west, encompassing all three layers of the plaster)

*Macroscopic description:* The sample contains three layers of plaster. The outer ones contain quartz grains, the middle one is microcrystalline. All layers are creamish-white, contaminated with brown clay.

(1) Outer (top) layer: White, microcrystalline binder, quartz filler.

No reaction in water. Binder dissolves in 3M HCl, leaving poorly selected quartz, with a varying degree of translucency and mostly well worn. Part of the quartz is contaminated with a russet-colored substance. Rock detritus, presumably igneous, is present. Heating a drop leads to recrystallization of gypsum at levels of considerable intensity. Positive reaction for ferric and silicate compounds, characterized by considerable intensity. Negative reaction with Lugola liquid and ninhydrin.

Conclusion: The sample in question is lime plaster containing very poorly selected quartz filler, mostly well worn. The small quantity of gypsum is due to weathering. The presence of ferric compounds and clay should be attributed to contamination with a loamy substance. No starch gum (dextrin) or protein substances were observed.

(II) Middle layer: 4-5 mm thick. White, microcrystalline. Binder dissolves in 3M HCl, releasing gas. Very fine quartz grains remain, as well as a small amount of microcrystalline suspension. No recrystallization of gypsum was observed. Medium-intensive, positive reactions to the presence of Fe<sup>3+</sup> ions and silicates.

Conclusion: This layer corresponds to a lime whitewash containing very fine quartz, falling within the lower limits for the sand fraction. Ferric compounds and clay are due to contamination with a loamy substance. No dextrin or protein substances present.

(III) Innermost layer: Also contains quartz filler. Binder dissolves completely in a reaction releasing gas. No gypsum recrystallization after heating. Considerable intensity of reaction for ferric compounds. Positive reaction for silicates. Negative reaction with Lugola liquid and ninhydrin.

Conclusion: Lime plaster with quartz filler. No gypsum. This is presumably an undercoating with more difficult access of sulfur compounds. Ferric compounds and clay are due to contamination with a loamy substance. No dextrin or protein substances present.

Sample no. 2 (interior plastering, top layer)

*Macroscopic description*: Beige, very brittle, considerable quantities of organic filler.

*Microscopic description*: Microcrystalline binder. Quartz grains visible, as well as plant fibers and boneblack.

Microchemical reactions: Plaster was not softened in water, but the fibers clearly were. Binder dissolves in 3M HCl, causing strong release of CO<sub>2</sub> ("foaming"). Considerable amounts of air bubbles remain, drop is yellow in color. After heating, very clear recrystallization of gypsum. Very clear reaction for Fe<sup>3+</sup> ions and silicates. In view of the clay content, boneblack cannot be determined by microchemical analyses. The presence of charcoal cannot be excluded. Clear purple tint in reaction with Lugola liquid. Positive biuret reaction for protein presence.

Thin section: Plaster with clear domination of the binder visible. Microcrystalline binder of the filling kind. Considerable amounts of underburnt lime. Poorly selected and well worn quartz is the chief mineral in the filler. Only the smallest grains are angular. Grain size: 0.9; 0.7; 0.5; 0.4; 0.2 mm. Gradual transition from the largest to the smallest grains with the larger grains predominating. Single grits of sandstone, one of feldspar and one of siliceous rock, two weathered igneous grits. Considerable quantities of organic fibers observed, presumably charred. In a few cases, internal structure discernible – fragments of cereal or plant stems. Gypsum and calcite has crystallized on some of the fibers, preserving the original structure. Significant accumulation of ferric compounds in the plaster. A few grains of a strongly red substance observed in both parallel and crossed nicols. Heavily porous texture.

Quantitative make-up measured in the thin section, in % (total 100%): quartz 15.0; binder 62.3; rock grits 0.6; underburnt lime 13.2; organic fibers 8.9. Quantitative ratio of binder to filler based on thin-section measurements: 1.7:1.

Conclusion: Lime plaster. Microcrystalline binder of the filling type. Gypsum of uncertain origin present, presumably product of weathering, but if dextrin present concurrently, then it could have been added on purpose. No evident concentrations of gypsum were observed. Quartz filler characterized by a varying degree of selection and fairly well worn. Sporadic sandstone grits, feldspar, weathered igneous rock. Contamination with a loamy substance. Significant amounts of charred organic fibers. Protein presence uncertain; it could be linked to organic fibers or microorganism. Quantitative ratio of binder to filler: 1.7:1.

Sample no. 3 (interior plastering, middle layer)

*Macroscopic description*: Gray-white plaster, flat. Polychromy on one surface.

*Microscopic description*: Plaster with microcrystalline binder. Considerable numbers of closed voids. Fibrous substance of white color, insignificant amount of quartz, visible grains of boneblack.

*Microchemical reactions*: Slight dispersion of the sample in water. Only part of the binder dissolves in 3M HCl. Very intensive recrystallization of gypsum after heating. Also very intensive positive reaction for Fe<sup>3+</sup> ions. Positive reaction for silicates, with Lugola liquid for dextrin and starch, and biuret reaction for proteins.

Thin section: Plaster with microcrystalline filler visible. Main filler ingredient consists of fibrous gypsum grits of varying length (4.9; 1.8; 1.0; 0.2 mm) and thickness (0.4; 0.2; 0.07 mm). Quartz grains present (5-6 in the field of vision) – of varying size 0.4; 0.07 mm. Also observed grits of well-crystallized calcite with a characteristic rhombohedral cleavage. Organic fibers present. Very strong gathering of ferric compounds in the surface layer. A few strongly red grains were also noted.

Quantitative make-up of the plaster as measured in the thin section, in % (total 100%): quartz 0.9; binder 63.8; gypsum grits 33.2; organic fibers 2.1. Quantitative ratio of binder to filler based on thinsection measurements: 1.8:1.

Conclusion: Plaster containing calcite and gypsum as elements of the microcrystalline binder. Mutual proportions impossible to determine in this study. The white fibrous substance is gypsum with this form of crystallization. Small amount of quartz filler (0.9%) and dextrin and protein added. Contamination with a loamy substance or some clay or silt. Charcoal or other vegetal black present. Quantitative ratio of binder to filler: 1.8:1.

Sample no. 4 (interior plastering, third layer, thick plaster with traces of modeling))

Macroscopic description: White, porous, brittle. Microscopic description: Microcrystalline structure, white in color, very soft, cracked.

Microchemical reactions: No visible reactions in water. Sample partly dissolves in 3M HCl. Drop remains cloudy. Very strong recrystallization of gypsum after heating. Gypsum very well crystallized in the form of long fibrous forms. Medium intensity of reaction for ferric ions. Very evident reaction for silicates. Negative reaction with Lugola liquid; biuret reaction for protein uncertain.

Thin section: Shows plaster containing gypsum grits and a domination of gypsum binder. Gypsum and lime binder unevenly mixed. Significant quantities of microcrystalline grits, presumably marl limestone, marl or mudstone grits of different size, e.g. 1.2; 0.7; 0.4; 0.2 mm. Also present are minor crystals of quartz and quartz sandstone of different

size and varying degree of wear (prevailing small size – 0.2; 0.07 mm). Fibrous gypsum crystals. Charcoal present, as well as strongly weathered single aluminosilicate grits and russet-colored concentrations of ferric compounds.

Quantitative make-up of plaster measured in the thin section, in % (total 100%): quartz 2.1; binder 75.2; rock grits 15.5; gypsum grits 4.5; organic fibers 2.7.

Considerable quantities of very fine fiber pieces and charcoal in the binder hinder counting.

Quantitative ratio of filler to binder based on thin-section measurements: 3:1.

Conclusion: Lime or lime-gypsum mortar with quartz filler. Very well crystallized gypsum occurs and quartz in small quantities 2.1%. No dextrin present, protein presence uncertain. Considerable presence of silicates, presumably due to an addition of clay or silt. Russet-colored grits of microcrystalline structure, poorly worn, were observed, as were considerable quantities of vegetal black. Certain identification of rock grits impossible – presumably marl limestone, marl or mudstone. There is three times more binder than filler.

**Sample no.** 5 (interior plastering, fourth layer = B.III.2)

*Macroscopic description:* Consistently beige color, brittle, powdery.

Microscopic description: Microcrystalline substance of beige color.

Microchemical reactions: Crumbles in water, leaving significant amounts of quartz grains, characterized by varying degrees of selection and wear. Residual dissolving reaction with release of gas in 3m HCl, concerns 2-3 grits in the field of vision. No gypsum recrystallization after heating. Medium intensity of reaction for ferric compounds. Very strong reaction for silicates. Biuret reaction for protein negative. Also negative reaction with Lugola liquid, although the natural beige color of the sample hinders proper assessment.

Conclusion: Mud plaster.

#### APPENDIX 2

## ANALYSES OF PIGMENTS FROM THE CRUCIFORM BUILDING (B.III) IN DONGOLA

### Anna Nowicka Academy of Fine Arts, Warsaw

The pigments were identified based on the following:

- microscopic observation of samples in reflected and transmitted light (water smears):
- sensitivity to acids and alkalis;
- anion and cation determination in the following microcrystaloscopic reactions:
- PO<sub>4</sub><sup>3-</sup> anion determination. Reaction with ammonium molybdate, (NH<sub>4</sub>)<sub>2</sub>MoO<sub>4</sub>, in HNO<sub>3</sub> medium (after dissolving sample in concentrated HNO<sub>3</sub> and evaporation). Product of the reaction: characteristic yellow deposit of ammonium molybdate-phosphate, (NH<sub>4</sub>)<sub>3</sub>[P(Mo<sub>3</sub>O<sub>10</sub>)<sub>4</sub>].
- Pb<sup>2+</sup> cation determination. 1) Reaction with potassium iodide, KJ (solution with concentration O, 1M) in diluted CH<sub>3</sub>COOH medium (after dissolving sample in concentrated HNO<sub>3</sub> and evaporation). Product of the reaction: characteristic yellow deposit of lead iodide, PbI<sub>2</sub>; 2) Reaction with four-ingredient mixture after first dissolving the sample in HNO<sub>3</sub> and evaporation. Mixture composition: saturated solutions of CH<sub>3</sub>COONH<sub>4</sub>, KNO<sub>2</sub>, Cu(CH<sub>3</sub>COO)<sub>2</sub> and 30% CH<sub>3</sub>COOH mixed in 1:1:1:1 proportions. Product of the reaction: regular black crystals of K<sub>2</sub>Pb[Cu(NO<sub>2</sub>)<sub>6</sub>].
- Fe<sup>3+</sup> cation determination. Reaction with potassium ferrocyanide, K<sub>4</sub>[Fe(CN)<sub>6</sub>], in HCl medium (after partial dissolution of sample in concentrated HCl and evaporation). Product of the reaction: blue fine-grained deposit of iron ferrocyanide, Fe<sub>4</sub>[Fe(CN)<sub>6</sub>].
- Fe<sup>3+</sup> cation determination. Reaction with potassium thiocyanate, KSCN, in concentrated HCl medium, after first dissolving sample in 4M NaOH and evaporation. Product of the reaction: red deposit of iron thiocyanate, Fe(SCN)<sub>3</sub>.

- Fe<sup>2+</sup> cation determination. Reaction with  $\alpha,\alpha'$ -bipiridyl, after dissolving sample in HCl and evaporation.
- Cu<sup>2+</sup> cation determination. Reaction with potasium ferrocyanide, K<sub>4</sub>[Fe(CN)<sub>6</sub>], in HCl medium. Product of the reaction: red-brown deposit of copper ferrocyanide, Cu<sub>2</sub>[Fe(CN)<sub>6</sub>].
- S<sup>2-</sup> anion determination: Reaction with azide reagent. In the presence of sulfides, gas bubbles are released and the reagent is discolored.

## Sample no. 9: a) blue and b) red (interior plastering, first layer)

*a) Microscopic observation in reflected light* (x 48): Painted layer is light blue with apparent darker crystals of turquoise shade.

Microscopic observation of water smear in transmitted light (x 640): Observed blue grains, color intensity differing for various grain sizes (the bigger the grain, the darker it is); pigment grains translucent, angular breaks; size reaches up to 0.07 mm;

Solubility in acids and alkalines: In 3M HCl, conc. HNO<sub>3</sub> and 4M NaOH, the pigment does not change either in cold acid or after heating.

Cu<sup>2+</sup> cation determination: negative result.

# Conclusion: The sample represents Egyptian blue, $CaCu[Si_4O_{10}]$ .

b) Microscopic observation in reflected light (x 48): The surface of the painted layer observed in side light looks like very fine, delicate brocade; it is not mat. After the grayish-white film was removed from the surface, the layer proved to be an intensive red, dark with a brownish shade.

Microscopic observation of water smear in transmitted light (x 640): Observed grains include dark red, of bigger size, angular, semi-translucent, some almost black; size – maximum 0.0045 mm.

Solubility in acids and alkalines: 3M HC1, conc. HNO<sub>3</sub>, NaOH – no apparent changes in the sample; conc. HCl – after heating, pigment is partly dissolved, after evaporation of the drop a yellowish ring remains.

Fe<sup>3+</sup> cation determination: positive.

Conclusion: The sample contains iron oxide red. Microscopic appearance in reflected and transmitted light indicates that it is powdered hematite (Fe<sub>2</sub>O<sub>3</sub>).

Sample no. 10: black (tested) and yellow (interior plastering, first layer)

Microscopic observation in reflected light (x 48): After grayish-white film was removed, the painting layer proved to be intensively black and matt. Pigment sampled from the painting layer floats after treatment with a drop of water.

Microscopic observation of water smear in transmitted light (x 640): Observed black grains, fine and very fine, most under 0.003 mm, biggest c. 0.003 mm innumerous grains of charcoal black, 0.015-0.48 mm;

Solubility in acids and alkalines:  $3M\ HCl-no$  apparent changes in the sample; conc.  $HNO_3-brownish$  ring on edges of drop, but the black pigment is not dissolved;  $4M\ NaOH-no$  apparent changes.

*Sensitivity to turpentine oil:* Pigment is not dissolved either cold or after heating.

 ${\rm PO_4^{\; 3^-}}$  anion determination: negative;  ${\rm S^{2^-}}$  anion determination: negative.

Conclusion: The black pigment is lampblack mixed with charcoal black. Brown ring on drop of conc. HNO<sub>3</sub> suggests the presence of asphalt, which is not confirmed, however, by the reaction to NaOH and turpentine oil (asphalt is sensitive to these substances). Consequently, the binder must be responsible for the ring. The negative result of phosphate anion determination excludes boneblack. The negative result of sulfide determination excludes the presence of the mineral galena.

Sample no. 11: a) dark emerald green, b) light olive-green, c) red (interior plastering, first layer)

a) Microscopic observation in reflected light (x48): The surface of the painting layer is heterogeneous - there are lighter spots, darker ones of colder or warmer shade.

Microscopic observation of water smear in transmitted light (x 640): Apparent grains are green, irregular in form, rounded, differentiated, semi-translucent, some of a warmer shade (russet or yellowish); size most frequently 0.006-0.01 mm, maximum size 0.018 mm; colorless, angular, translucent, size 0.015-0.03 mm; most likely quartz.

*Solubility in acids*: 3M HCl – no apparent change in cold acid, after evaporation yellow ring appears; conc. HNO<sub>3</sub> – dissolves in part; conc. HCl – heated, the pigment dissolves in part.

Fe<sup>3+</sup> and Fe<sup>2+</sup> cation determination: positive; Cu<sup>2+</sup> cation determination: negative.

*Conclusion:* The pigment sample is **earth green** (hydrated aluminosilicates: Fe<sup>2+</sup>, Fe<sup>3+</sup>, magnesium and potassium).

b) Microscopic observation in reflected light (x 48): The painting layer is very thin, fairly compact but brittle. Orange-yellow spots visible.

Microscopic observation of water smear in transmitted light (x 640): Apparent numerous yellow-green, transparent grains in the form of hexagonal plates (more or less regular), size 0.009-0.024 mm; yellow-green, semitransparent grains, rounded, size 0.006-0.015 mm; yellow-green grains with angular edges, transparent and semi-transparent, irregular in form, some rusty, size 0.015-0.03 mm; blue-green, light-colored, semi-transparent grains, rounded, size 0.006-0.015 mm; few rusty-red, semi-transparent grains, rounded, size 0.006-0.008 mm; very few yellow, semi-transparent grains, rounded, size 0.015 mm.

Solubility in acids: 3M HCl and conc. HCL – no apparent change in cold acid; heated, the pigment dissolves in part; conc. HNO<sub>3</sub> – dissolves in part; 4M NaOH - sample grays slightly; heated, changes color to rusty.

 $Fe^{3+}$  and  $Fe^{2+}$  cation determination: positive;  $Cu^{2+}$  cation determination: negative.

Conclusion: The pigment sample is earth green (hydrated aluminosilicates: Fe<sup>2+</sup>, Fe<sup>3+</sup>, magnesium and potassium) containing a large amount of a greenish-yellow mineral (probably aluminosilicate characterized by monoclinic crystallization) appearing in the form of plates, resembling the pieces of yellow in sample no. 12.

Sample no. 12: a) yellow, b) orange, c) red (interior plastering, second layer)

a) Microscopic observation in reflected light (x 48):
 Yellow-colored layer, glimmering strongly (brocade effect).

Microscopic observation of water smear in transmitted light (x 640): Observed grains – numerous yellow, light-colored, translucent, in the form of hexagonal plates (more or less regular), size 0.003-0.015 mm; yellow, semi-translucent, rounded, size c. 0.003-0.006 mm.

Solubility in acids and alkalines: 3M HCl – no evident reaction in cold acid, pale yellow residue left after evaporating the sample; conc. HCl – partly dissolves when hot; 4M NaOH – no evident reaction.

Fe<sup>3+</sup> cation determination: positive.

Conclusion: The yellow layer is composed of mainly a pulverized yellow mineral – aluminosilicate characterized by monoclinic crystallization. The sample also contains some iron oxide yellow. Instrumental analysis is required to determine the exact kind of aluminosilicate.

b) Microscopic observation in reflected light (x 48): Heterogeneous surface of painting layer – apparent red, yellow spots, innumerous grains shining like microscopic mirrors.

Microscopic observation of water smear in transmitted light (x 640): Observed grains – red of brown and orange shade, semi-translucent, differentiated, size 0.7-0.012 mm; yellow, differentiated, rounded, semi-translucent, size most frequently c. 0.003 mm, maximum 0.009 mm; colorless, clear, angular breaks, size 0.01-0.03 mm (most likely quartz); innumerous hexagonal plates, clear, yellowish, size c. 0.004 mm (presumably responsible for the microscopic shining spots described above);

Solubility in acids and alkalines:  $3M\ HCl-most$  of the sample is not dissolved; conc.  $HNO_3-mot$  dissolved;  $4M\ NaOH-mot$  evident changes in the sample; conc. HCl-mot dissolves when hot.

Pb<sup>2+</sup> cation determination: negative; Fe<sup>3+</sup> cation determination: positive.

Conclusion: Painting layer contains a mixture of iron oxide red (Fe<sub>2</sub>O<sub>3</sub>) and iron oxide yellow (Fe<sub>2</sub>O<sub>3</sub> · nH<sub>2</sub>O). Negative result of lead cation determination excludes addition of lead yellow. The layer also contains an insignificant amount of a mineral, possibly the same aluminosilicate characterized by monoclinic crystallization that was found in the yellow layer of the sample (see above).

#### APPENDIX 3

## ANALYSES OF BINDERS IN THE PAINTING LAYER FROM THE CRUCIFORM BUILDING (B.III) IN DONGOLA

### Irmina Zadrożna

Warsaw University of Technology

The analysis of binders used in the painting layer was carried out on samples taken from different places in the object under conservation-laboratory conditions. Two independent spectroscopic techniques were employed to obtain information on the different ingredients of the binders: Gas Chromatography-Mass Spectrometry (GC-MS) and for a few samples the Fourier Transformation Infrared Spectroscopy (FTIR).

GC-MS is the best method for analyzing complex mixtures as up to hundreds of individual elements making up a mixture can be separated out in the capillary columns and then identified explicitly by the spectrometer. The technique also permits a quantitative estimate of particular ingredients. It however, suitable requires, preparation, as the compounds used as binders belong to different classes and are frequently used concurrently in the painting technique. The FTIR technique complements gas chromatography-mass spectrometry, identifying the binders as belonging to specific classes of compounds. In this case, the FTIR results served to confirm GC-MS data.

For chromatographic analysis acidolysis of the samples had to be carried out. The product of acid hydrolysis was subjected to derivatization (silylation) using BSA (N, Obistri-methylsilyloamide acetic acid) and

analyzed chromatographically. The GC-MS analysis was carried out using an Ultra 2 capillary column and a Hewlett-Packard HP-5890II gas chromatograph coupled with HP-5971A mass spectrometer. Chromatography conditions: carrier gas helium 1 ml/min, dosing temperature 270°C for the samples, initial temperature 50°C for 5 min, 5°C/min up to 300°C for 5 min. Samples containing a minimum of a few dozen micrograms of the organic material dosed out by the splitless technique. Identification of simple compounds was carried out most frequently through comparison of recorded spectrum with the NIST Library computer spectra. Model analyses of binders of known composition were also carried out to provide comparative material. Estimation of relative quantity of selected compounds was made by measuring the area of the signals in the chromatogram.

The FTIR spectra were achieved on a Perkin-Elmer spectrophotometer in a KBr medium.

#### Sample no. 9 (first layer of plaster)

Sample was subjected to acidolysis. The chromatogram contains insignificant amounts of amino acids: glycine, alanine, proline, glutamic acid and aspartic acid, characteristic of a protein binder – egg yolk. Insignificant amounts of mono- and dicarboxyl acids also occur, including

palmitic (P) and stearic (S) in a ratio typical of nut oil (P / S)  $\cong$  3.

An infrared spectrum for this sample identified amide bands at 3400, 1624, 1532 cm<sup>-1</sup> (a strong carbonate-related band partly superimposed on it) indicating the presence of a protein binder, and a band at 1736 cm<sup>-1</sup> characteristic of the oil ester group.

Binder composition: egg yolk and nut oil.

#### Sample no. 10 (first layer of plaster)

Infrared spectrum for the sample yielded identical results as in the case of sample no. 9, indicating the same make-up of the binder.

#### Sample no. 11 (first layer of plaster)

Sample was subjected to acidolysis. The chromatogram contains insignificant amounts of amino acids: glycine, alanine, proline, glutamic acid and aspartic acid, characteristic of a protein binder – egg yolk. Insignificant amounts of mono- and dicarboxyl acids also occur, including palmitic (P) and stearic (S) in a ratio typical of nut oil (P / S)  $\cong$  3.

An infrared spectrum for this sample identified amide bands at 3396 cm<sup>-1</sup>,

1624 cm<sup>-1</sup> and 1560 cm<sup>-1</sup> indicating the presence of a protein binder.

**Binder composition:** egg yolk with an insignificant amount of nut oil.

#### Sample no. 12 (second layer of plaster)

The chromatogram made for the product of acid hydrolysis revealed the presence of amino acids: alanine, proline, aspartic acid, glutamic acid and others testifying to the use of a protein binder. Comparison with a model spectrum for egg yolk suggests its presence. Also observed are mono- and dicarboxyl acids, including palmitic (P) (retention time 33.63 min) and stearic (S) (retention time 37.07 min) in a ratio typical of nut oil (P / S  $\cong$  3).

The infrared spectrum for this sample revealed bands representing amide bonding at 3408 cm<sup>-1</sup> and at 1624 cm<sup>-1</sup> and 1576 cm<sup>-1</sup> (strong carbonate-related band partly superimposed on the last). Also present are bands at 1736 cm<sup>-1</sup>, characteristic of the oil ester group, and a band at 2516 cm<sup>-1</sup>, originating from the clay in the plaster.

*Binder composition:* egg yolk, nut oil (in considerable excess).