TECHNICAL ANALYSIS REPORT

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"Conservation of Roman Wall Paintings In Luxor Temple: Study of the constitutive materials and of the techniques employed for the realization of the different pictorial phases".

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A R C E AMERICAN RESEARCH CENTER IN EGYPT

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Conservation of Roman Wall Paintings in Luxor Temple

Study of the constitutive materials and of the techniques employed for the realization of the different pictorial phases.

Fall 2006 Campaign

Submitted by:

Artelab s.r.l. – Via dei Pettinari n. 73, Roma

March 2007

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1. Materials and methods

Laboratory tests were carried out on the samples taken by restores to study the materials and the execution technique. The following methods were used to study the samples:

- preliminary examination of all samples by stereomicroscope, with microchemical tests and production of photographic documentation;
- preparation and study of stratigraphic sections under reflected light, with microchemical and histochemical tests;
- optical mineralogical analysis finalized to the identification of the pigments;
- microchemical tests aimed at identifying the class to which the binding and fixative – protective agents belong;
- infrared spectrophotometric analysis (FT-IR: *Fourier Trasformated Infrared*).

The main aims of the investigations during this phase of the study, though within the limits of the number and type of carried out examinations, are the following:

- to define the prevailing pictorial technique used for the realization of the paintings;
- to determine the stratigraphic sequence (rendering, preparation, etc.);
- to correlate different portions of the paintings, aiming to acquire data confirming or not their belonging to a same executive phase.
- to advance a reconstructive hypothesis of the chronological succession of the layers found by the surveys and their execution phases.

The tests were carried out in compliance with the UNI – Normal recommendations, where possible, and with the indications provided by the scientific publications published by national and international institutes that operate in the field of cultural heritage conservation: ICR, ICCROM, ICOM, ASTM.

Roma 26 - 03 – 2007 Dott. Domenico Poggi

2. Processing and interpretation of analytical results

Following are briefly presented the outcomes of the analysis, their interpretation and the correlation between the data altogether obtained. In order to facilitate the comprehension, the outcomes are explained separately, according to the kind of material and execution technique. Finally, in a conclusive paragraph, is presented a reconstructive hypothesis of the chronological succession of the layers identified by means the analysis and, therefore, of the respective execution phases.

2.1. Stony material

Residuals of the stony material of the support masonry were found only in the sample n. 2, at the base of the "*arriccio*" mortar layer

According to the outcomes of the stereomicroscope observation and within the limits set by observation under the stereomicroscope, it is safe to assume that the sandstone is attributable to a litotype of the "Nubian Sandstone" kind, deriving from the "Qoseir – formation. (1). This formation, outcropping in the Gebel el-Silsila area, provided a large part of the tombstone materials used to build the Pharaonic temples of Luxor and also the replacement blocks still come from this outcrop.

The brown-reddish colouration of the stone derives from some haematitic - limonitic dispersions.

2.2. The support plasters and their stratigraphic relations

The analysis of the sampled specimens allowed to identify some fragments of plasters made up of two typologies of mortar; in particular it was possible to identify:

1. Mortars realized with lime, vegetable fibres and a siliceous sand with a mainly quartz formation. This kind of mortar was used for the creation of the bottom layers (`*arriccio*');

2. Mortars realized with lime and siliceous sand with a composition very similar to the one used for the "*arriccio*" layers. This second type of mortar was used for the plaster layers, characterized by relatively regular thickness, generally ranging between 1.5 and 2,5 millimetres.

Both kinds of mortar were realized with a sand of the same nature, attributable to the desert sand emerging in the area of Luxor.

In all sampled specimens no fragment including the "*arriccio*" drawing overlapping the plaster was found . This proves the insufficient adhesion between the two mortar layers.

The basal "face" of some plaster fragments present in the specimens is characterized by a smooth surface. It sometimes presents a slight whitish lime layer that probably was applied as primer directly on the "*arriccio*" (see the sheets of specimens n. 3 and 5). Also the plasters surface is generally flat and smooth as if it was the result of a work with a plaster trowel or a similar tool.

[1] Concerning this, see the article written by B. Fitzner et Alii (2003). Weathering damage on Pharaonic sandstone monuments in Luxor – Egypt. In Building and Environment, Vol. 38, N. 9, Spt. 2003, pp. 1089 – 1103. Ed Elselvier.

2.3 Pictorial film: its structure and relations with the plaster layer

The study of the *cross sections* stated that the pictorial film is nearly always composed by a single layer, more or less dense and containing calcium carbonate from lime. Thanks to its relations with the support plaster and according to the results of the FT-IR analyses and the microchemical and istochemical tests, it is safe to assume that most layers were applied with a "*mezzo fresco*" technique. Some layers seem to have been applied "*a secco*" only in the specimen n. 9 and maybe in the n. 10 and 11 ones.

The stratigraphy of the specimen n. 9 can be an illustrative example of the execution technique used for all analysed specimens. In fact, the cross section of such specimen presents a thin whitish drawing over the lime and siliceous sand plaster with a mainly quartz formation. The latter could derive from a superficial accumulation of lime, caused by the smoothing of the plaster or by the application of a very diluted drawing of whitewash. Following: the pictorial film, formed by a pale red layer with light violaceous tones and a green drawing.

The first layer is made up of calcium carbonate, haematite (Fe_2O_3) and a small amount of Egyptian blue. It is a mixture of silicates, with different degrees of crystallinity, including quartz and cuprorivaite ($CaCuSi_4O_{10}$). It was probably applied with a "*mezzo fresco*" technique; in fact the drawing insinuates inside the fissures of the below plaster, thus indicating it was applied on a drying plaster.

Instead, the green pictorial layer, which was pigmented with "Green earth", little green "*fritta egizia*" and rare Egyptian blue, was applied with a "*secco"* technique, dispersing the pigments inside a proteic binder, probably animal glue.

In specimens n.10 and 11, both taken in correspondence of some blue-azure paintings realized with Egyptian blue, the pictorial drawing is in direct contact with the lime and sand plaster (in the specimen n.10 there are traces of a thin level of haematite, referable to a preparatory design, between the two drawings). At present, the blue-azure pictorial layer is nearly lacking in calcite. Therefore it seems to derive from a layer applied with a "*secco*" technique. However, neither micro nor istochemical tests could define which kind of binder was used. This could be due to the strong alteration and mineralization in oxalates (weddellite, whewellite or mooloite) of the binder itself, favoured by the presence of the copper present in the blue pigment based on cuprorivaite-: $CaCuSi_4O_{10.}$ The FT-IR test revealed small quantities of oxalates.

Pigments used for the other paintings, applied a "*fresco*" or with a "*mezzo fresco*" technique, are jarosite [$KFe_3(SO_4)_2(OH)_6$] for the yellows (sample n. 4) and haematite for the reds (sample n. 5) or pinks (sample n. 12), where it is diluted in the lime.

2.4 The ivory "Scialbo" and its relations with the other layers (plasters, pictorial film)

Micro-stratigraphic analysis, microchemical and histochemical tests and spectrophotometric examinations (FT-IR) proved that the "*scialbo*" was realized with lime, gypsum and a proteic substance referable, as first approximation, to animal glue or, less probably, to gelatine (2). At the base of the scialbo, in only one sample (sample n. 7), a blue-azure pictorial layer is present, composed of Egyptian Blue, attributable to the roman phase. In some points, between the two layers it is possible to observe traces of a brown layer referable, in relation to only the microscopic "appearance", to residues of gum arabic and particled matter (a hypothesis which would be interesting to verify by micro-FT-IR or micro-Raman).

In none of the samples comprehending the scialbo was noticed the presence of fragments of plaster or arriccio.

In almost all of the samples, over the ivory scialbo there is a thin yellow-brownish film composed of gypsum and some proteic substances (see the sheet of the sample n. 1). In sample n. 7, above this film, a thin and discontinuous brown layer is also present, which is attributable to a gum arabic based adhesive or to a protective (see the sheet of sample n. 7, in which microchemical tests evidenced the presence of polysaccharides).

2.5 Superficial brownish - grey or brown layers

In all specimens over the pictorial film or in direct contact with the support layers (plasters and scialbo) was found a more or less continuous and dense drawing of a variable colour ranging from brownish-grey to brown. In most cases analyses stated that such layer derives from a repainting applied during a relatively recent maintenance and realized with gypsum, a small amount of carbon, "Earths" and, maybe, some proteic binders (specimen n. 12). In some cases (specimen n.2 and 5) it seems that the layer pigmented with "Earths" derives from a repainting mixed with earthy particles (3).

Moreover, microchemical tests aimed at identifying polysaccharides excluded that the superficial brownish-grey layers could represent an altered fixative based on gum Arabic.

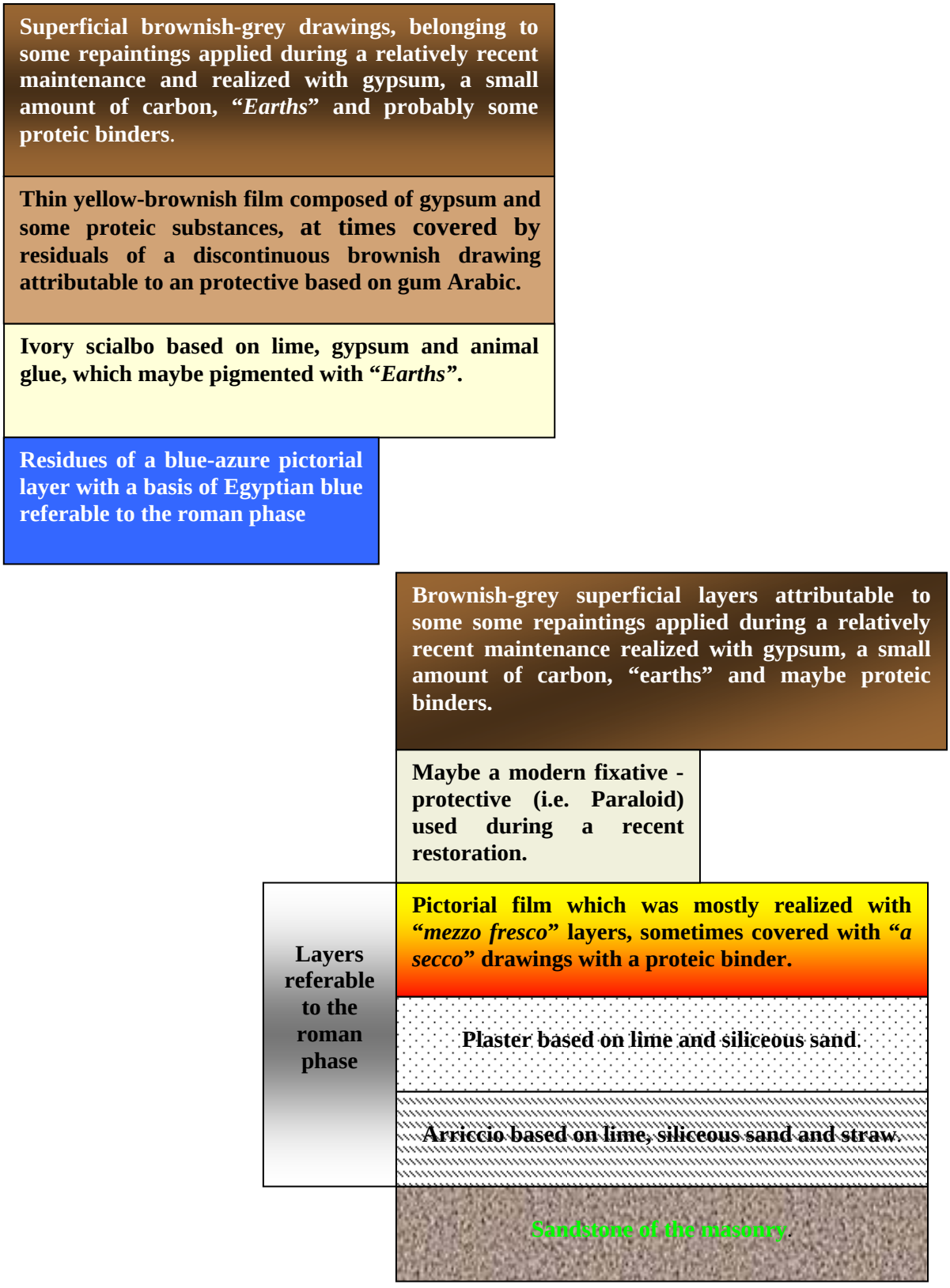
Some borders of a white-greyish drawing, with optical - morphologic features similar to those characterizing the fixatives - protectives used during recent restorations were found in the specimen n.9, between the brownish-grey layer based on gypsum and the pictorial film. This hypothesis, based only on the characteristics resulting under the reflected light test, should be confirmed with targeted analysis such as the IR

spectrophotometry (FT-IR, micro-FT-IR) and the Scanning Electron Microscope (SEM - EDS) microanalysis.

2 A certain identification of the proteic binder used is possible using finest techniques such as HPLC (High Pression Liquid Cromatography)

3 The latter could be mistaken for the "earths" used as pigment, if analyzed only with optical microscope in reflected light.

2.6 Reconstructive hypothesis of the chronological succession of the layers found by the analysis and their execution phases



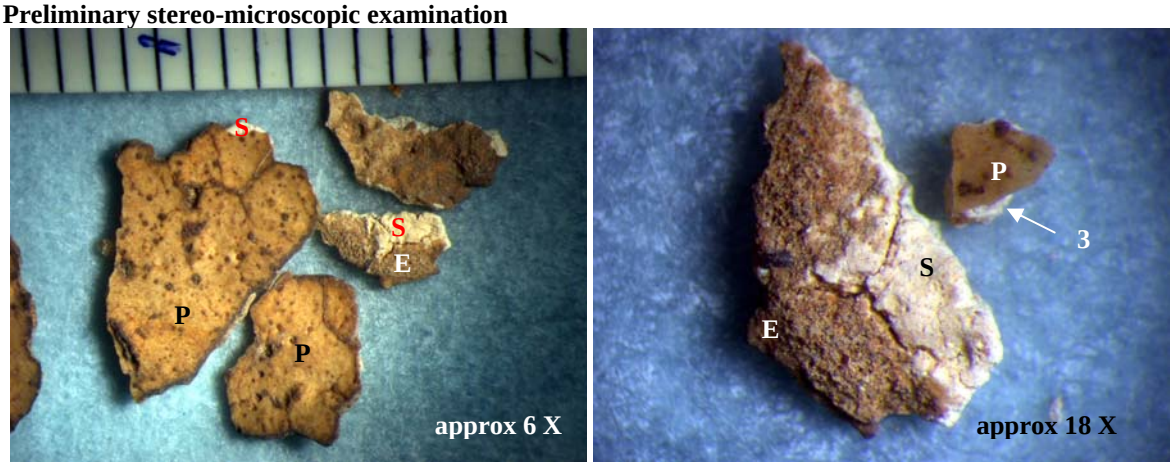
3. Analytical Sheets

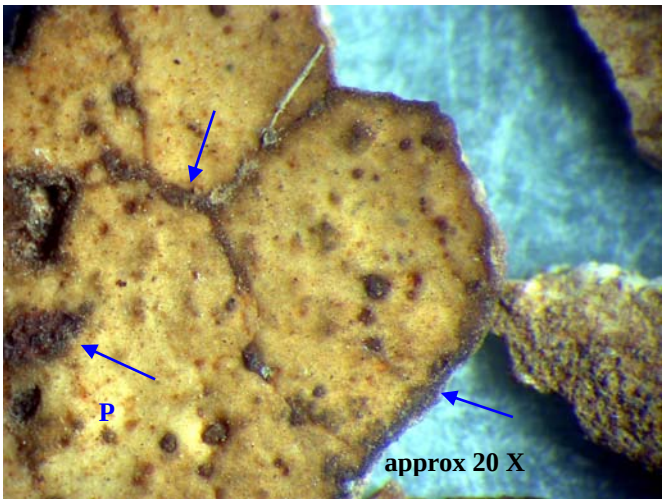
Sample no. 1

Information on the sampling area and description accompanying the sample

"East wall, yellow scialbo."

Preliminary stereo-microscopic examination

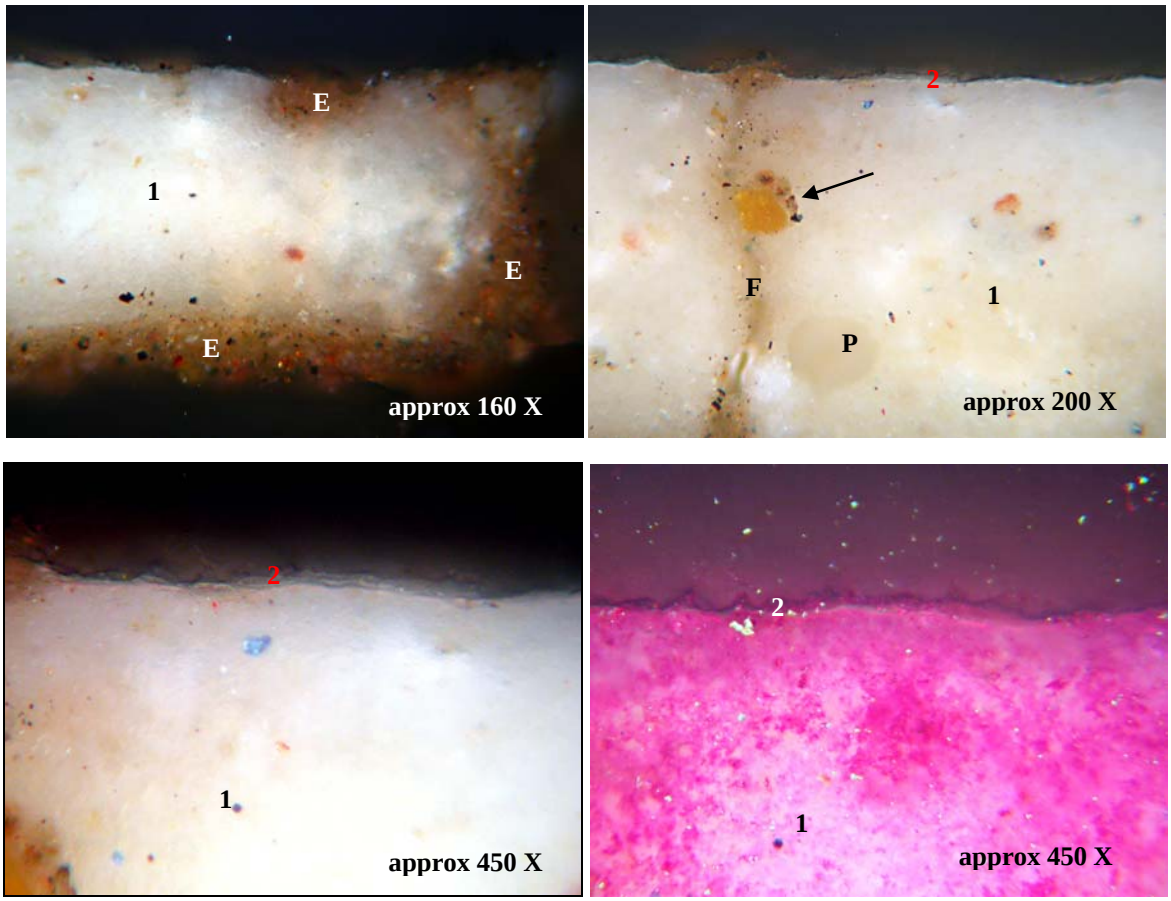




Stereo-microscope, reflected light.

The upper left photograph describes some of the millimetric scales present in the specimen. They are formed by the ivory "scialbo" (S) covered by a thin yellow-brownish film (P) and some residuals of a dark-brown earth-like layer (E) present at the base of the "scialbo". The latter is well shown in the upper right picture, at a higher magnification.
The picture below represents instead the surface of the "scialbo", darkened by the "thin yellow-brown film" thickening near fissures edges or over small cavities (arrows).
In the following pages are presented the results of the microstratigraphic analysis on polished cross section, of the micro and histochemical tests and of the chemical-mineralogical tests carried out with infrared spectrophotometry (FT-IR).

Microstratigraphyc analyses on cross section with microchemical and histochemical tests
Analytical sheet written according to the Doc Normal 12/83



Cross section, reflected light

The upper left photograph, at a relatively low magnification, shows a large part of the section describing a lateral portion of the scialbo (n. 1) and the surrounding earth-like deposit (E). The tests and the spectrophotometric analysis results (see below) proved that the scialbo was made with gypsum, calcium carbonate and a proteic medium (probably animal glue).

The upper right photograph shows a portion of the surface of the scialbo affected by a transversal fissure (F) which is partially filled up with an earth-like deposit. The picture also shows a perfectly globular pore (P) and a yellow ochre grain (arrow) that perhaps was used as pigment during the realization of the scialbo. On the upper part, the thin brownish film (n. 3) is hardly visible but, however, it is better illustrated in the two photographs below, taken at a higher magnification.

The bottom right photograph was taken after the histochemical test with fuchsine which revealed the presence of a proteic medium both in the scialbo (n. 1) and in the above film (n. 2). The film does not contain low-fusing substances since it does not melt (i.e. as waxes do) if it stays for a long time under an IR warm light lamp. It can't be corroded by hydrochloric acid and also contains gypsum, as the microscopical analysis with transmitted polarized light proved.

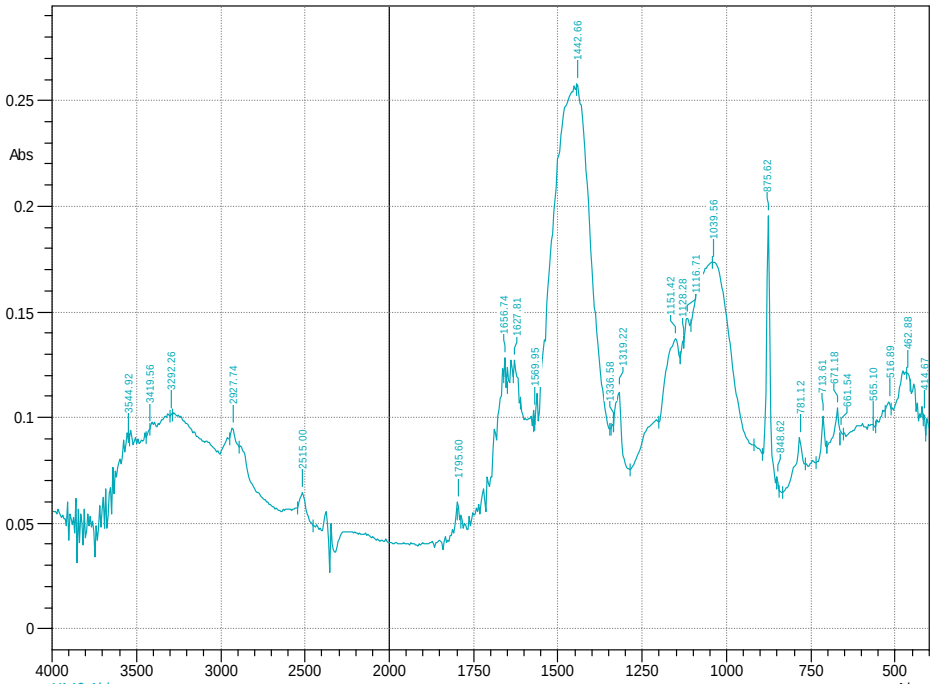
Infrared spectrophotometric analysis (FT-IR)

The sample, including some sub-millimetric scales of the ivory scialbo, have been accurately grounded with an agate mortar together with KBr
The material has been analysed in transmittance by DSR cell.
The interpretation of the FT-IR spectrum obtained by the analysis has been carried out by comparison with the laboratory data base and with the one reported by various scientific publications. In particular, assignment was made on the basis of the vibration frequencies, recorded under the same test conditions, of reference standards, either pure or mixed with specific matrixes (calcite, calcite and gypsum, etc.).

Results

Study of the IR spectrum obtained from the analysis (see following page) establishes that the sample is essentially formed from the following components listed in order of relative abundance:
- *Calcite* ($CaCO_3$);
- *Gypsum* ($CaSO_4.2H_2O$);
- *Silicates* probably including *layered silicates (minerals e/o clay minerals: calcium, sodium, potassium and other ions silicon aluminate hydrate);*
- *Calcium oxalate bihydrate* (*weddellite*: $CaC_2O_4.2H_2O$);
- *Organic substances* that may be refereable to proteins.

The IR spectrum does not reveal the presence of other organic binders as polisaccharides, natural resins, oils and waxes.
Also the negative results of the micro-chemical tests confirm the absence of polysaccharides (such as gum Arabic, starch, honey, etc) and natural resins (colophony, dammar, etc)([4]).



Spectrum FT-IR.

[4] Spot tests have been carried out on few tens milligrams of the specimen including both the scialbo and the superficial layer. The analyses were carried out in compliance with the indications contained in *DIMOS: corso sulla manutenzione di Dipinti murali – Mosaici – Stucchi; Parte I: Tecniche di Esecuzione – Materiali Costitutivi, Modulo 3, Leganti, Fissativi, Pigmenti: Metodi di Riconoscimento. Istituto Centrale del Restauro, 1978 Roma...*

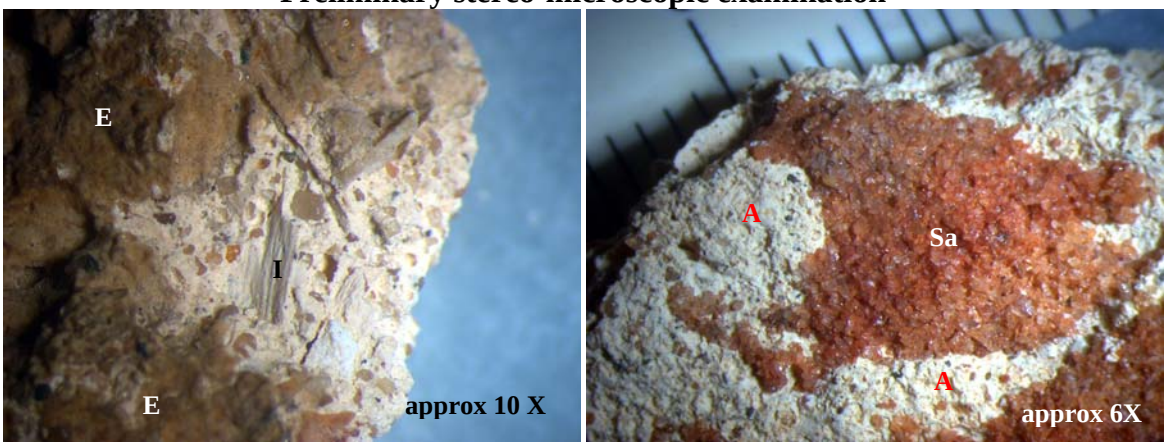
Sample no. 2

Information on the sampling area and description accompanying the sample

"East Wall, fragment including the stone material of the masonry and the first plaster layer"

Macrophotography

Preliminary stereo-microscopic examination



Stereo-microscope, reflected light

The two above photographs represent respectively the surface (on the left) and the inner face of the sampled fragment. The surface presents a thin dark-brown layer (E) that irregularly covers the mortar. The mortar, entirely similar to that contained in specimen n. 5 (see its sheet) and composed by lime, siliceous sand, vegetable fibres, is attributable to an "arriccio" (A).
The white arrow indicates the traces of the vegetable fibres visible in the mortar.
The upper right image shows some flaps of the reddish-brown sandstone of the masonry (SA), "pulled away" during sampling operations, together with the mortar (A). Within the limits set by observations under the stereo microscope, it is safe to assume that the sandstone is attributable to a litotype of the "Nubian Sandstone" kind, which derived from the "Qoseir - formation „(5). This formation, outcropping in the Gebel el-Silsila, provided a large part of the tombstone materials used to build the Pharaonic temples of Luxor and also the replacement blocks still come from this outcrop. The two photographs below, taken through the stereomicroscope, describe the details of the surface and of the lower part of the fragment. The letter I indicate the print of a vegetable fibre present in the "arriccio" mortar. In the next pages are presented the results of the chemical-mineralogical analysis by infrared spectrophotometry (FT-IR) carried out on a micro-specimen selectively removed from the superficial dark-brown layer.

[5] Concerning this, see the article written by B. Fitzner et Alii (2003). Weathering damage on Pharaonic sandstone monuments in Luxor – Egypt. In Building and Environment, Vol. 38, N. 9, Spt. 2003, pp. 1089 – 1103. Ed Elselvier.

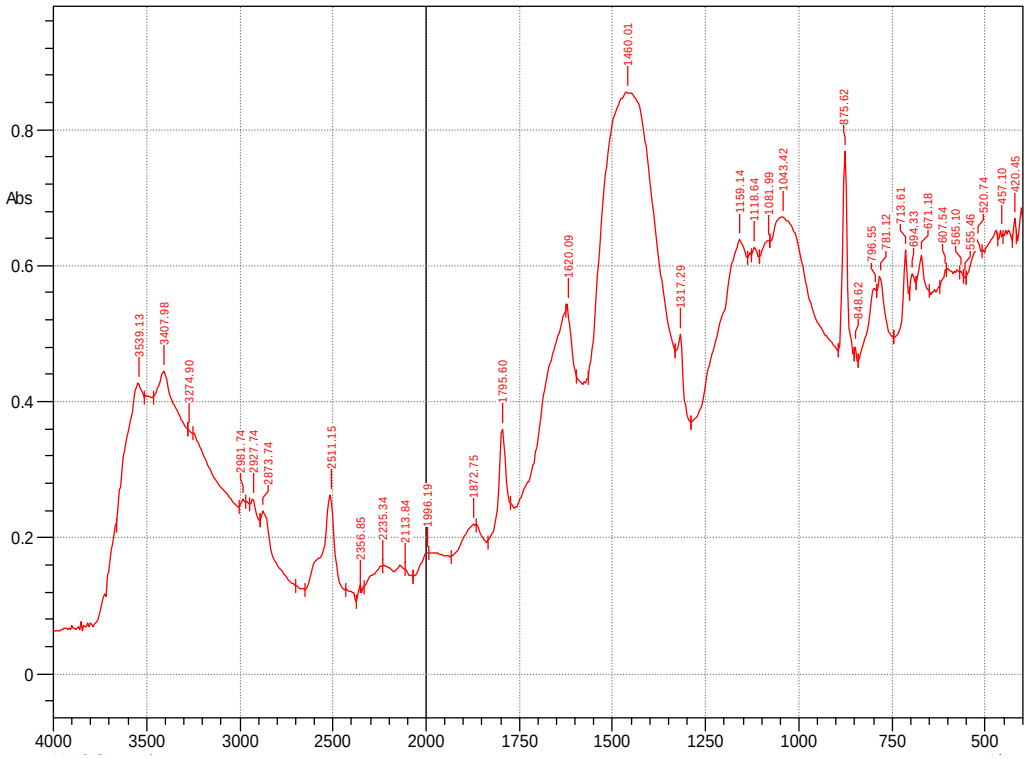
Infrared spectrophotometric analysis (FT-IR)

The micro-specimen, including the thin dark-brown layer and a part of the lower *arriccio*, have been accurately grounded with an agate mortar together with KBr
The material has been analysed in transmittance by DSR cell.
The interpretation of the FT-IR spectrum obtained by the analysis has been carried out by comparison with the laboratory data base and with the one reported by various scientific publications. In particular, assignment was made on the basis of the vibration frequencies, recorded under the same test conditions, of reference standards, either pure or mixed with specific matrixes (calcite, calcite and gypsum, etc.).

Results

Study of the IR spectrum obtained from the analysis (see following page) establishes that the sample is essentially formed from the following components listed in order of relative abundance:

- *Gypsum* ($CaSO_4.2H_2O$);
- *Calcite* ($CaCO_3$);
- *Quartz* (SiO_2) attributable to the plaster support aggregate;
- *Silicate minerals* probably including *feldspars (composed of silica, aluminium, potassium, calcium and sodium)* e/o *layered silicates (micas* e/o *clay minerals*) that can be attributed both to the dark-brown layer and to the lower arriccio,
- *Soda nitre* ($NaNO_3$);
- *Calcium oxalate:* (*whewellite*: $CaC_2O_4.0.5H_2O$);
- *Organic substances*;



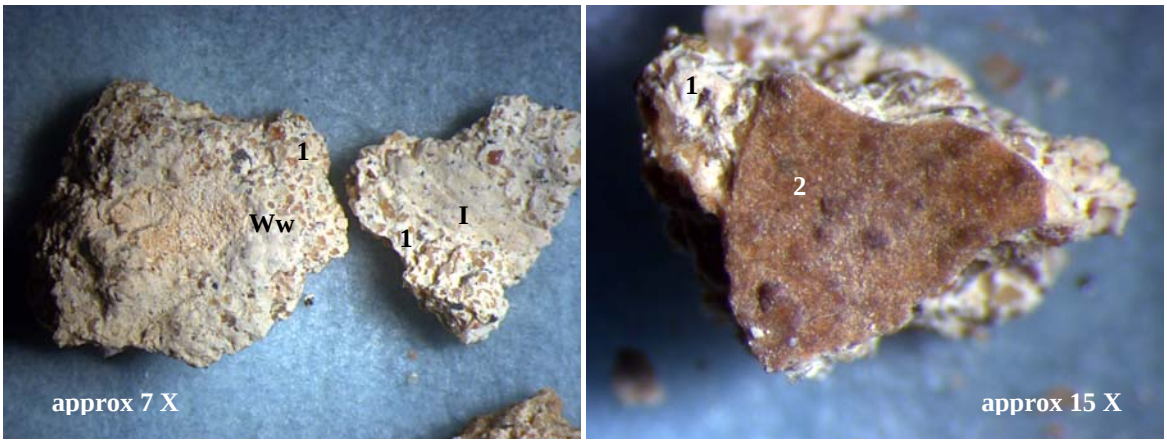
Spectrum FT-IR.

Sample no. 3

Information on the sampling area and description accompanying the sample

"West wall, light rendering of the first level

Preliminary stereo-microscopic examination



Stereo-microscope, reflected light.

The photograph on the left represents the inner face of two of the sampled flakes. In the photograph, along with the lime and the siliceous sand plaster (n. 1), a discontinuous whitish layer can be observed (Ww), attributable to a whitewash layer that was applied as primer probably over an "arriccio" made with lime, sand and vegetable fibres. Thanks to the observations of the other samples (for instance, see the sheet of the specimen n.5) and to the finding of a vegetable fibre print at the base of the plaster (I) it is safe to assume the presence of an "arriccio". The plaster has a relatively regular thickness, around 2-3 millimetres .

The imagine on the right illustrates the surface of one of the flakes shown in `the previous photograph'. This photograph shows a compact ochreous layer with a film aspect (n. 2). A micro specimen was selectively removed from this layer and analyzed by FT-IR (see next pages).

Infrared spectrophotometric analysis (FT-IR)

The micro-specimen, selectively removed from the superficial layer, have been accurately grounded with an agate mortar together with KBr.
The material has been analysed in transmittance by DSR cell.
The interpretation of the FT-IR spectrum obtained by the analysis has been carried out by comparison with the laboratory data base and with the one reported by various scientific publications. In particular, assignment was made on the basis of the vibration frequencies, recorded under the same test conditions, of reference standards, either pure or mixed with specific matrixes (calcite, calcite and gypsum, etc.).

Results

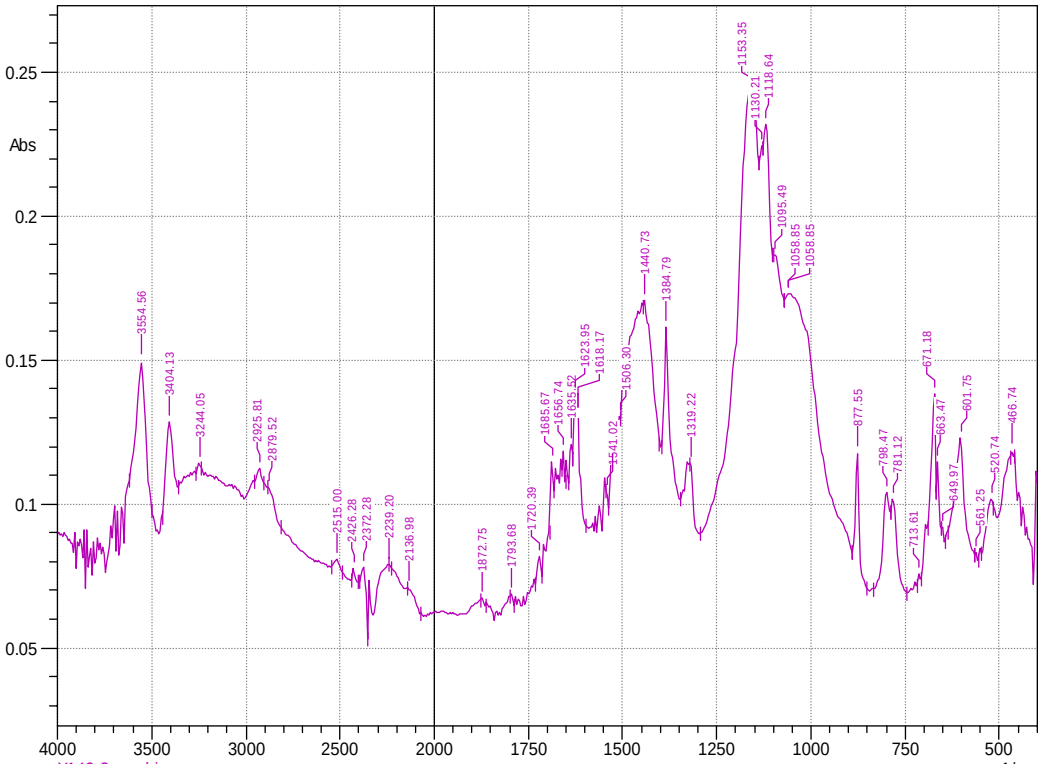
Study of the IR spectrum obtained from the analysis (see following page) establishes that the sample is essentially formed from the following components listed in order of relative abundance:

- *Gypsum* ($CaSO_4.2H_2O$);
- *Calcite* ($CaCO_3$);
- *Quartz* (SiO_2) attributable to the plaster support aggregate;
- *Silicate minerals* probably including *feldspares (composed of silica, aluminium, potassium, calcium and sodium)* e/o *layered silicates (micas* e/*o clay minerals)* that can be attributed both to the ochre layer and to the lower plaster;
- *Soda nitre* ($NaNO_3$);
- *Calcium oxalate:* maybe present both *whewellite* ($CaC_2O_4.0.5H_2O$) and *weddellite* ($CaC_2O_4.2H_2O$);
- *Organic substances* that may be refereable to proteins;

The IR spectrum does not reveal the presence of other organic binders as polisaccharides, natural resins, oils and waxes.

Also the negative results of the micro-chemical tests confirm the absence of polysaccharides (such as gum Arabic, starch, honey, etc) and natural resins (colophony, dammar, etc)([6])..

[6] Spot tests have been carried out on few tens milligrams of the specimen including both the scialbo and the superficial layer. The analyses were carried out in compliance with the indications contained in *DIMOS: corso sulla manutenzione di Dipinti murali – Mosaici – Stucchi; Parte I: Tecniche di Esecuzione – Materiali Costitutivi, Modulo 3, Leganti, Fissativi, Pigmenti: Metodi di Riconoscimento. Istituto Centrale del Restauro, 1978 Roma...*



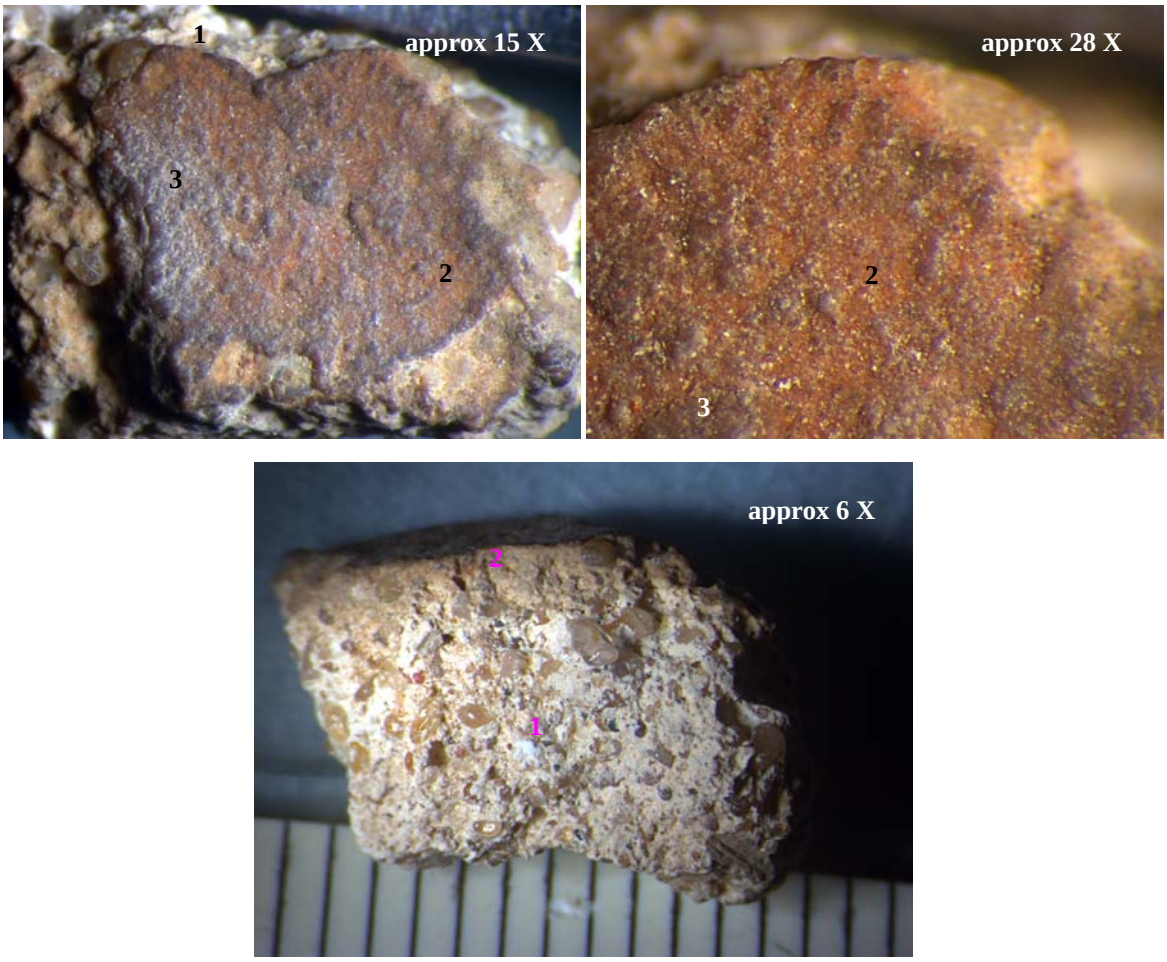
Spectrum FT-IR

Sample no. 4

Information on the sampling area and description accompanying the sample

"West Wall, top level, plaster with pictorial film"

Preliminary stereo-microscopic examination



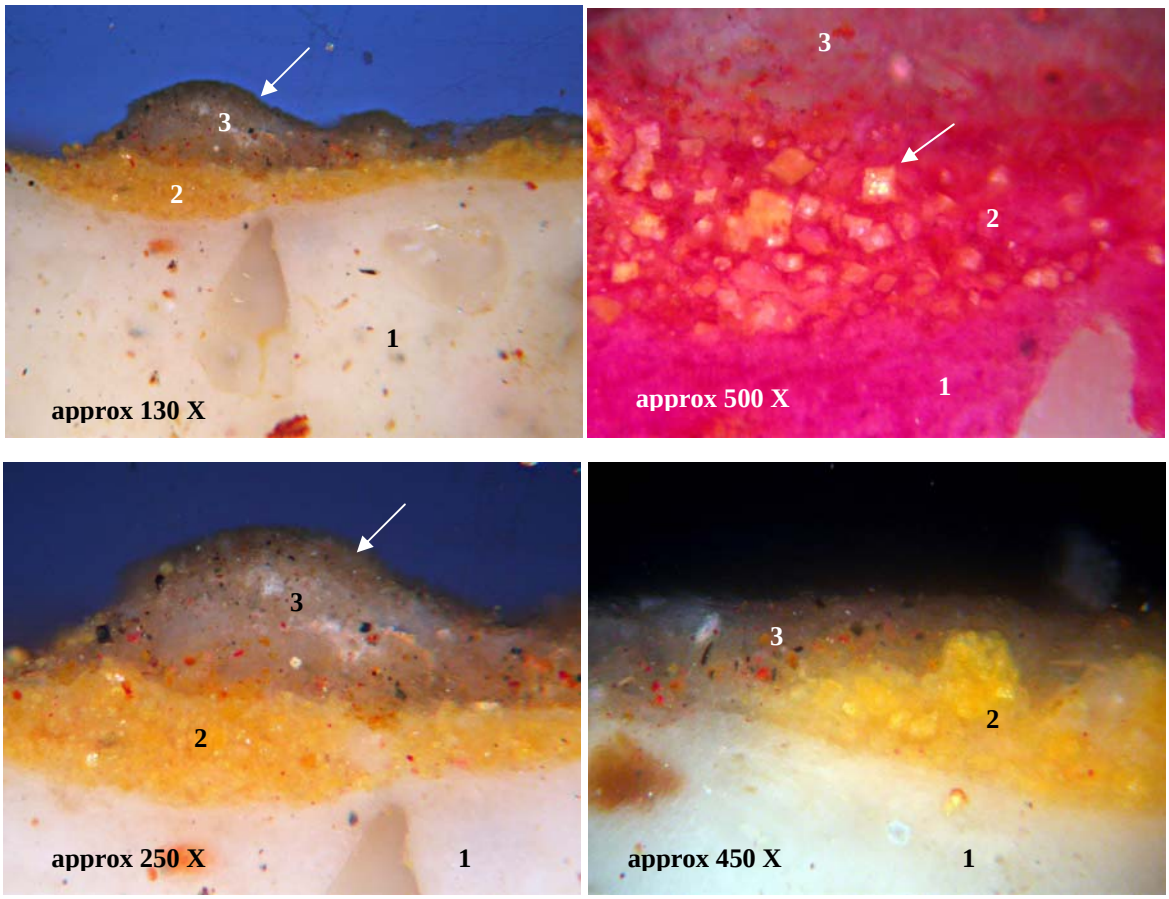
Stereo-microscope, reflected light.

The photographs at the top show two details of the surface of the sampled fragment at different magnification. This surface is characterized by the presence of a compact ochreous layer and it is apparently similar to the one of the specimen n. 3, which is covered with a discontinuous brown drawing. Instead, the below picture aims at representing, in cross section, both the structure of the plaster made up of lime and siliceous sand (n. 1) and the overlying ochreous layer (n. 2).

The FT-IR analysis was carried out on several tens milligrams of dust selected from the superficial layer. It indicated the presence of gypsum, calcite, nitrates, silicates and traces of organic substances in the two upper layers (see the next page).

Microstratigraphyc analyses on cross section with microchemical and histochemical tests

Analytical sheet written according to the Doc Normal 12/83



Cross section, reflected light

The four photographs describe the details of the cross section and illustrate the entire sequence of the layings and their stratigraphic relations. The upper right picture, taken after the fuchsine test, shows the morphology of the jarosite grains, the pigment used for the paint layer (n. 2). Here the strong fuchsia colouring does not derive from the chemical reaction between the protein binder and the fuchsine, but from the mere absorption of fuchsine caused by the porosity of the plaster and its overlaid layers. The resin used for the preparation of the *cross section* couldn't in fact consolidate completely the specimen.

The succession of layers is described in detail on the following: 1) plaster consisting of lime and siliceous sand with a mainly quartz formation; 2) discontinuous yellow pictorial layer, containing jarosite and dispersed in a calcium carbonate binder; 3) grey layer with light violet tones attributable to a drawing containing gypsum, ochres, ores (presumably including iron sulphides) and a small amount of carbon. The upper portion of such layer, for a thickness about some micrometers, seems slightly darker, probably because of the presence of fine carbon particles inside it (arrows). Such portion reacts weakly to the fuchsine test aimed at identifying proteins.

Infrared spectrophotometric analysis (FT-IR)

The micro-specimen, selectively removed from the only superficial layers, have been accurately grounded with an agate mortar together with KBr.
The material has been analysed in transmittance by DSR cell.
The interpretation of the FT-IR spectrum obtained by the analysis has been carried out by comparison with the laboratory data base and with the one reported by various scientific publications. In particular, assignment was made on the basis of the vibration frequencies, recorded under the same test conditions, of reference standards, either pure or mixed with specific matrixes (calcite, calcite and gypsum, etc.).

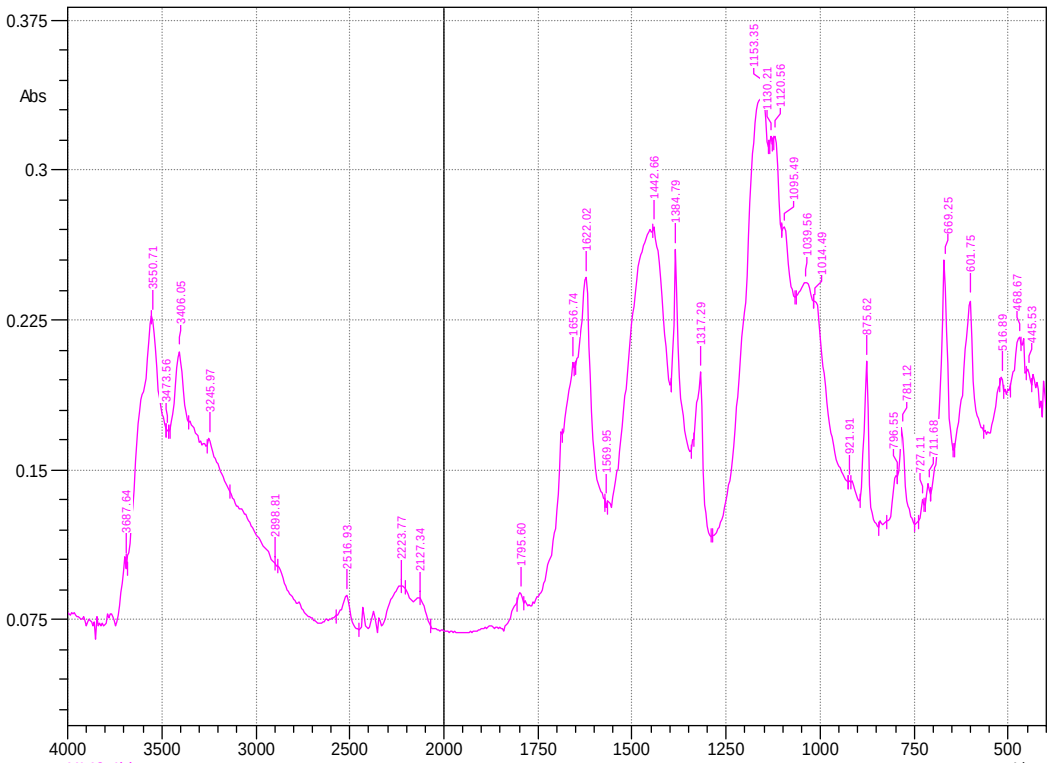
Results

Study of the IR spectrum obtained from the analysis (see following page) establishes that the sample is essentially formed from the following components listed in order of relative abundance:
- *Gypsum* ($CaSO_4.2H_2O$);
- *Calcite* ($CaCO_3$);
- *Silicate minerals* probably including *feldspars (composed of silica, aluminium, potassium, calcium and sodium)* e/o *layered silicates (micas* e/*o clay minerals)* that can be attributed both to the upper layers and to the lower plaster;
- *Soda nitre* ($NaNO_3$);
- *Calcium oxalate:* both *whewellite* ($CaC_2O_4.0.5H_2O$) and *weddellite*: ($CaC_2O_4.2H_2O$);
- *Organic substances:* present in traces;

The IR spectrum does not reveal the presence of other organic binders as polisaccharides, natural resins, oils and waxes.

Also the negative results of the micro-chemical tests confirm the absence of polysaccharides (such as gum Arabic, starch, honey, etc) and natural resins (colophony, dammar, etc)([7])..

[7] Spot tests have been carried out on few tens milligrams of the specimen including both the scialbo and the superficial layer. The analyses were carried out in compliance with the indications contained in *DIMOS: corso sulla manutenzione di Dipinti murali – Mosaici – Stucchi; Parte I: Tecniche di Esecuzione – Materiali Costitutivi, Modulo 3, Leganti, Fissativi, Pigmenti: Metodi di Riconoscimento. Istituto Centrale del Restauro, 1978 Roma...*



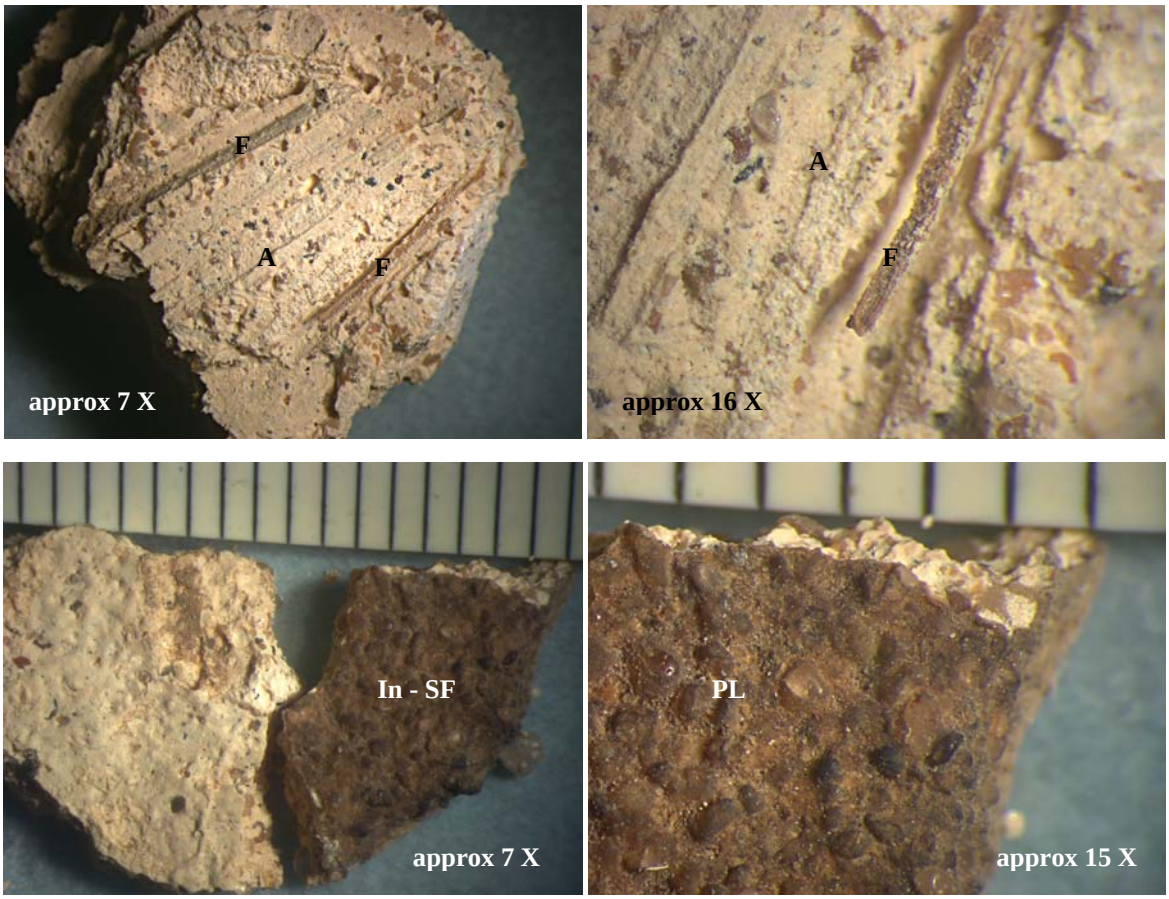
Spectrum FT-IR

Sample no. 5

Information on the sampling area and description accompanying the sample

"East Wall, fragment comprising two plaster layers and the pictorial film. The lower layer contains straw".

Preliminary stereo-microscopic examination



Stereo-microscope, reflected light.

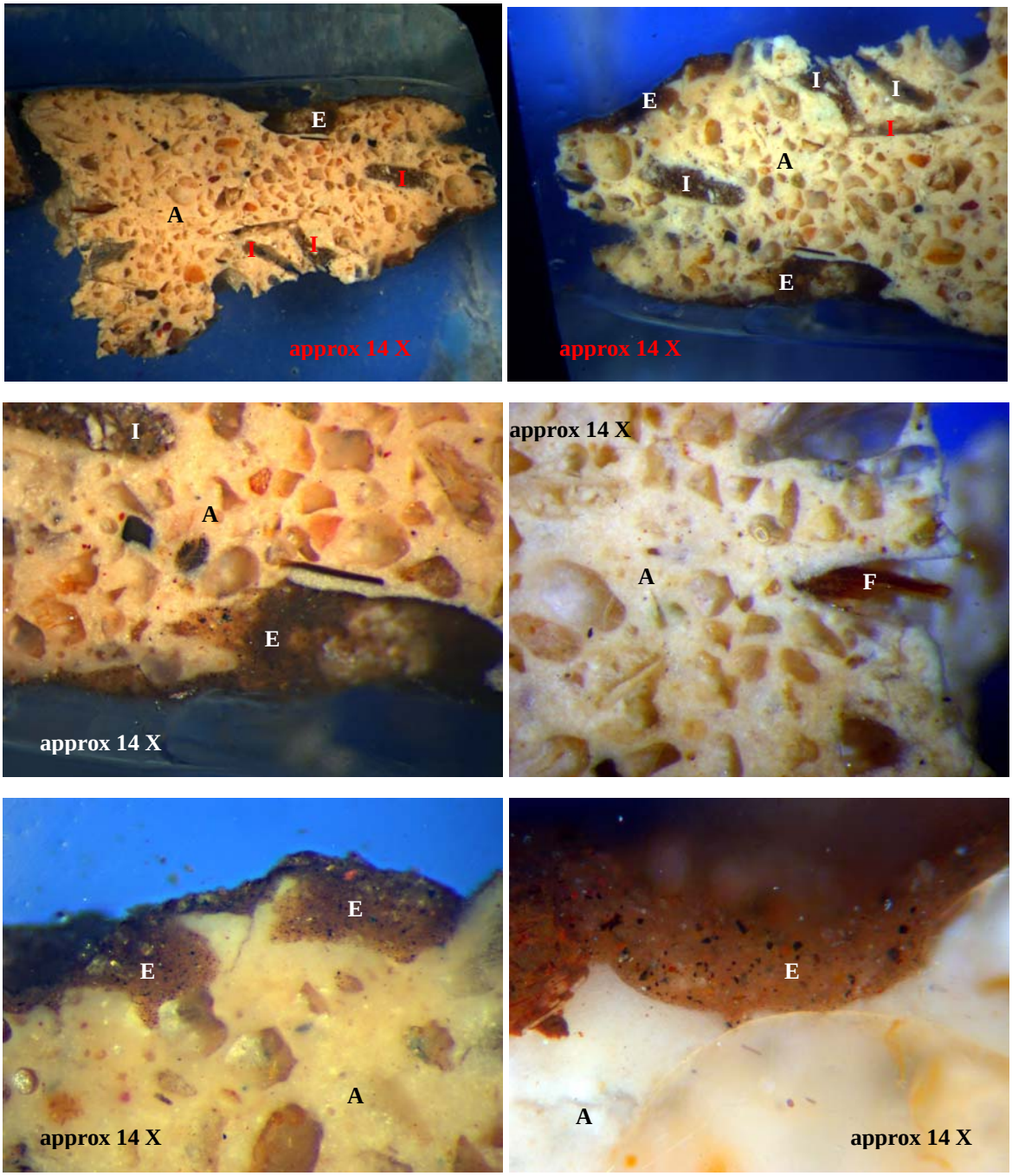
The images describe the structure of the most representative fragments of the specimen. In particular, the photographs at the top illustrate two aspects of a mortar composed by lime, siliceous sand and vegetable fibres (F), attributable to a layer of "arriccio" (A). The two pictures below represent plaster fragments (In) with a regular thickness.
The plaster surface (In - SF) and also the arriccio one (see next pages) are covered by a dark-brown drawing attributable to a layer of gypsum pigmented with "earth pigments" and carbon (PL) probably mixed with dust (compare with the sample n. 4). The microchemical tests (8) indicate the absence of polysaccharides (such as gum Arabic, starch, honey, etc) and natural resins (colophony, dammar, etc).

The images taken in polished cross-section are presented in the following pages.

[8] Spot tests have been carried out on few tens milligrams of the specimen including both the scialbo and the superficial drawing. The analyses were carried out in compliance with the indications contained in *DIMOS: corso sulla manutenzione di Dipinti murali – Mosaici – Stucchi; Parte I: Tecniche di Esecuzione – Materiali Costitutivi, Modulo 3, Leganti, Fissativi, Pigmenti: Metodi di Riconoscimento. Istituto Centrale del Restauro, 1978 Roma..*

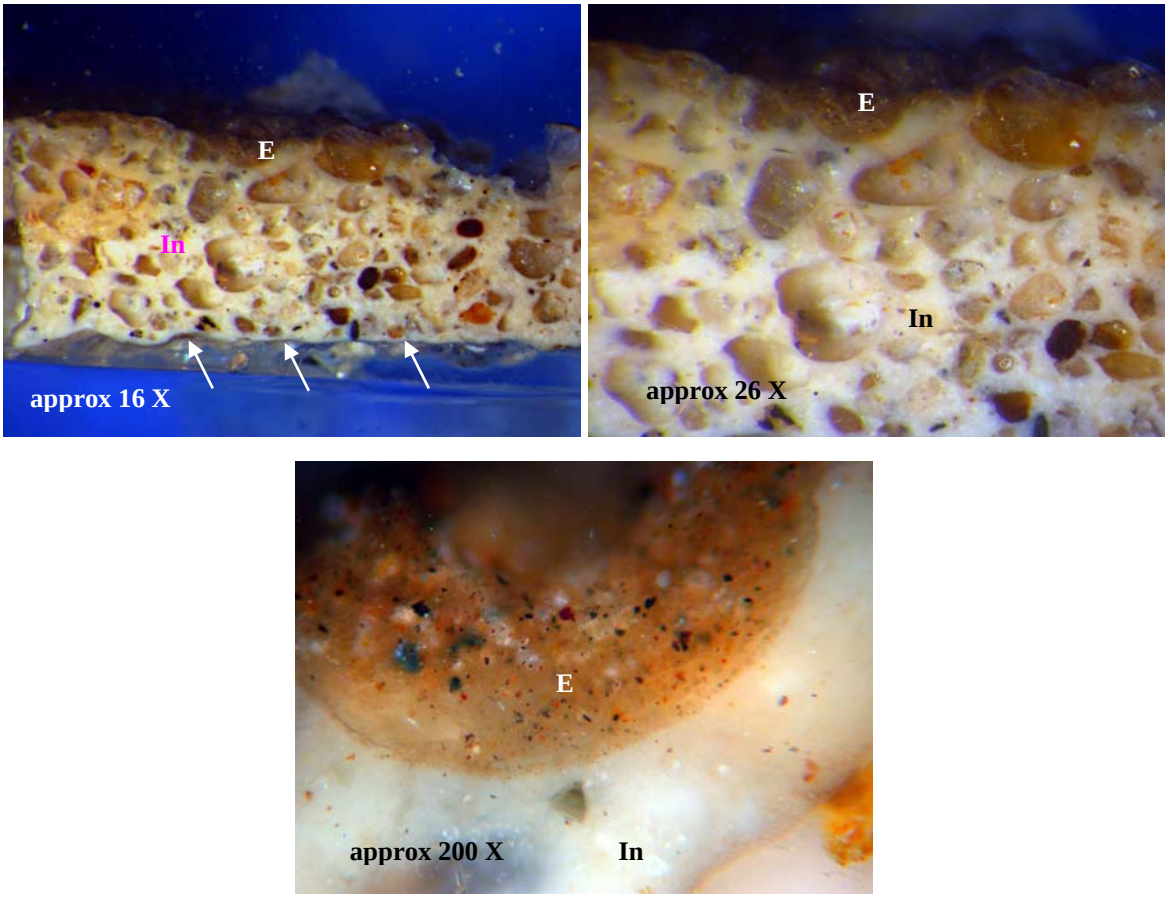
Microstratigraphyc analyses on cross section with microchemical and histochemical tests

Analytical sheet written according to the Doc Normal 12/83



Cross section, reflected light

The pictures describe different aspects of the mortar, attributable to the “arriccio” layer (A); it is composed by lime, siliceous sand with a mainly quartz formation and vegetable fibres. At the mortar’s edges there are: earthy incrustations and/or residuals of a gypsum and “earth pigments” based (E) scialbo. Inside, beyond many sand grains, there are several vegetable fibres prints filled up with an earthy deposit (I). The middle right picture shows a real fibre (F). The `photographs' below show two aspects of the dark - brown layer attributable to a gypsum drawing pigmented with “earth pigments” and carbon (E) and probably mixed with earthy particles (E).The plaster is described in the next page.



Cross section, reflected light

The three photographs describe the mortar composed by lime and siliceous sand, with a mainly quartz formation, attributable to the plaster layer (In). The absence of plant fibres in the mortar, the regular thickness and the morphology of the basal portion of the layer (arrow) prove that the mortar belongs to the plaster drawing laid upon the arriccio (see previous pages).

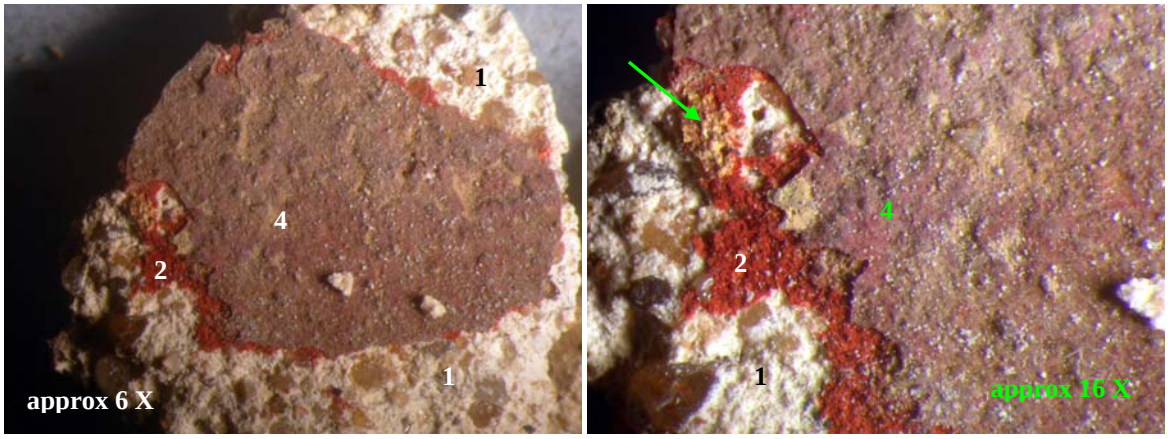
On the surface there is a dark - brown drawing attributable to a gypsum layer pigmented with “earth pigments“and carbon probably mixed with earthy particulate (E) and very similar to the one found on the arriccio fragments.

Sample no. 6

Information on the sampling area and description accompanying the sample

"South Wall at a height of approximately 3.5 - 4,0 m from the pavement. Plaster fragment with red pictorial film".

Preliminary stereo-microscopic examination



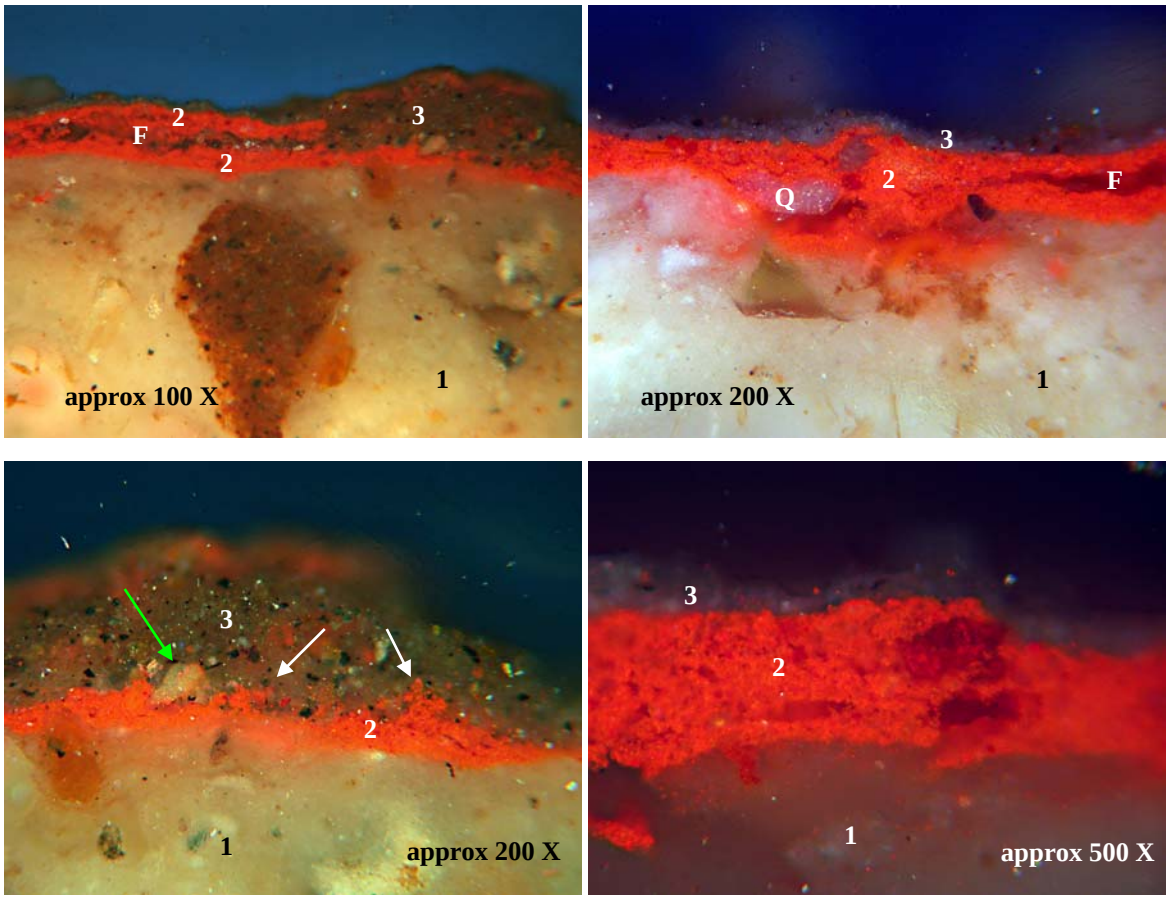
Stereo-microscope, reflected light.

The photographs describe two aspects of the surface of the sampled fragment. Over the lime and siliceous sand plaster (n. 1) there is a dark red pictorial layer composed of haematite (n. 2), as microstratigraphical analysis and microchemical tests proved. On the upper part there are traces of a yellow layer, probably composed of jarosite (n. 3) and a thin but continuous greyish level with a translucent aspect (n. 4). In the following pages the outcomes and the photographs of the polished cross-section analysis are presented.

A microchemical test aimed at identifying the polysaccharides (3) was carried out on several tens milligrams of dust selectively removed from the surface of the pictorial film(9).
Thanks to the negative outcome of the test it is possible to exclude that the superficial greyish layer could represent a deteriorated gum Arabic – based fixative agent. Moreover the absence of polysaccharides indicates that such kind of substance was not used as medium in the pictorial layers, at least in the area in which the specimen was sampled.

[9] Polysaccharides are typical of some plant gums, but they are also in starches and honey .

Microstratigraphyc analyses on cross section with microchemical and Histochemical tests
Analytical sheet written according to the Doc Normal 12/83



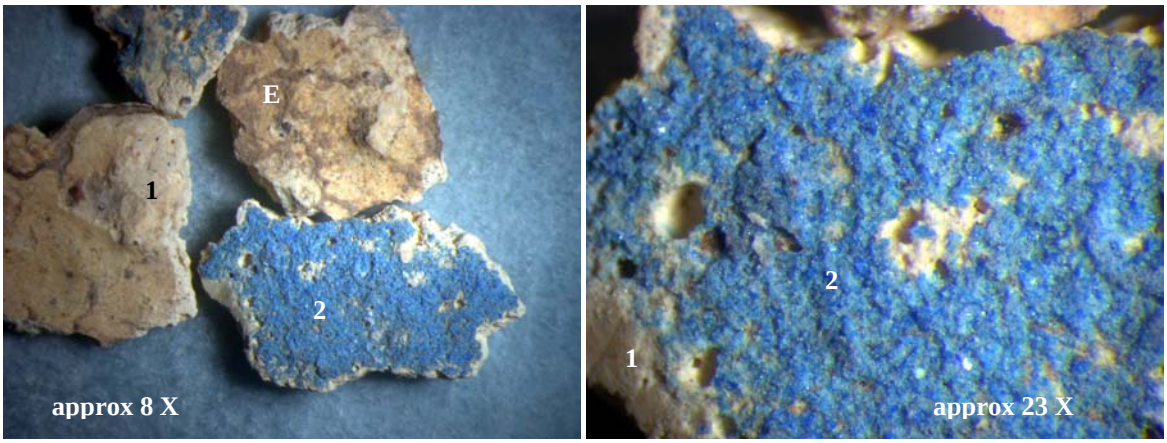
Cross section, reflected light

The four photographs illustrate the plaster surface (n. 1) and its relations with the red haematite (Fe_2O_3) based pictorial film (n. 2) that was applied "*a fresco*". This one shows a mediocre state with erosions (bottom left "photographs": arrows) and partial detachments caused by the presence of some longitudinal fissures (F). Between the red layer and the upper grey level there is a single yellow grain (green arrow) attributable to jarosite, which could indicate the original presence of a yellow pictorial drawing now almost completely eroded. Over the pictorial layer there is a brownish-grey drawing (n. 3) attributable to the layer of a scialbo made up of gypsum, a small amount of carbon and "earth pigments".

Sample no. 7

Information on the sampling area and description accompanying the sample

"East Wall, blue laid on the scialbo".



Stereo-microscope, reflected light.

The picture on the left illustrates some of the sampled flakes, comprehending the ivory scialbo and residues of the underlying blue-azure pictorial layer, 'pulled away' during the collecting together with the scialbo itself. The latter, with a regular millimetric thickness (about 1.5 – 2.0 mm), is composed of gypsum, calcium carbonate and proteinic substances (presumably animal glue). It is the ivory "scialbo" that was also noticed in samples n. 1 and 8. The blue-azure coloured pictorial layer visible at the base of the "scialbo", composed of Egyptian blue, is referable to the roman age pictorial phase.
On the upper face of the ivory "scialbo" are traces of a 'earthy' looking deposit (E) and of a brown resinous substance attributable to a protective 'layer' or to an adhesive with a basis of gum arabic.
The image on the right, more 'magnified', points out the structure of the pictorial layer realized with Egyptian blue.

Qualitative tests aimed at identifying the class to which the binders and organic fixative agents belong

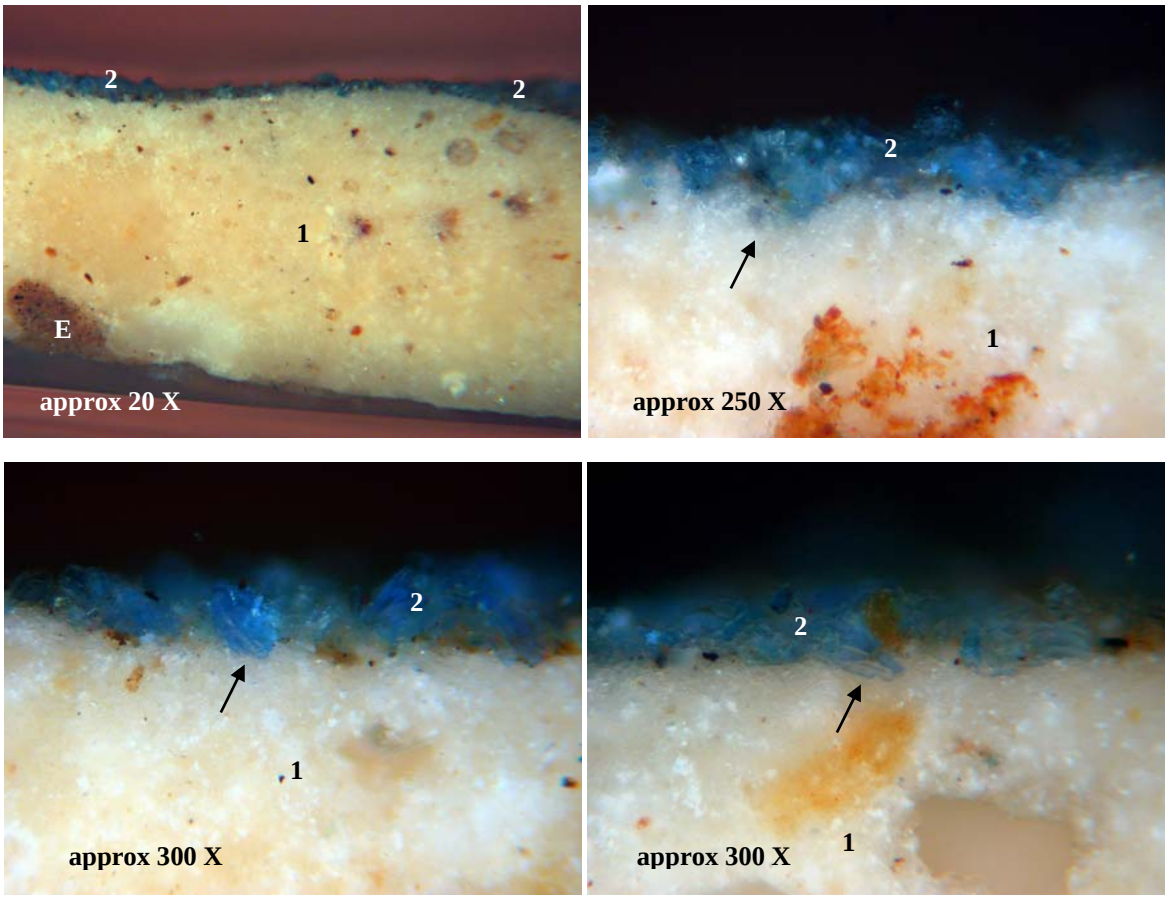
Method

Tests have been carried out according to the guidelines of the DIMOS: *corso sulla manutenzione di Dipinti murali – Mosaici – Stucchi; Parte I: Tecniche di Esecuzione – Materiali Costitutivi, Modulo 3, Leganti, Fissativi, Pigmenti: Metodi di Riconoscimento. Istituto Centrale del Restauro, 1978 Roma*

Results

Qualitative analysis of the organic substances		
Layer or layers analysed	*Carried out test*	Results
White scialbo + pictorial blue-azure layer containing Egyptian blue	Proteic substances (egg, animal glue, casein, etc.)	++
	Polysaccharides (gum Arabic, honey, starch, etc.)	-
	Natural resins (colophony, dammar, etc.)	-
Residuals of a brown layer present at the base of the ivory scialbo.	Proteinic substances (egg, animal glue, casein, etc.)	-
	Polysaccharides (gum Arabic, honey, starch, etc.)	++
	Natural resins (colophony, dammar, etc.)	-
KEY ++: substance present in large quantity; +: s. present; +/-: s. may be present in traces; - s. absent.		

Microstratigraphyc analyses on cross section with microchemical and histochemical tests
Analytical sheet written according to the Doc Normal 12/83



Cross section, reflected light.

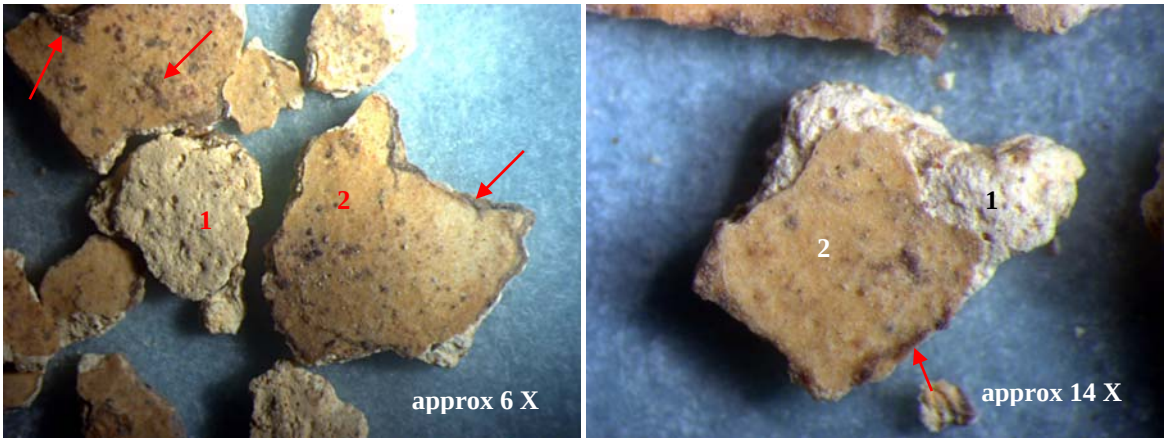
The image up on the left, shot at a low 'magnified', describes most of the section. Evident are: the "scialbo" (n. 2) composed of gypsum, calcium carbonate and a proteinic medium (maybe animal glue) and the underlying blue-azure coloured pictorial layer (n. 1), with a basis of Egyptian blue, referable to the roman phase. The other images, greatly 'magnified', illustrate the relations between the two layers which, at times, tend to compenetrate each other (arrow). Between the blue-azure layer and the scialbo are traces of a brown layer referable, in relation to only the microscopic "appearance", to residues of gum arabic and particled matter (a hypothesis which would be interesting to verify by micro-FT-IR or micro-Raman).

On the basis of the acquired information it is difficult to comprehend whether the calcium carbonate present in the scialbo may derive by the use of a really fine "inert" (carbonate lime 'in lumps', S.Giovanni white type) or of actual lime mixed with the other two mediums (gypsum and proteinic substance).

Sample no 8

Information on the sampling area and description accompanying the sample

"East Wall, scialbo."



Stereo-microscope, reflected light.

The picture on the left describes some of the millimetric flakes present in the specimen. They are formed by an ivory "scialbo" (n. 1) very similar to that of the specimen n. 1. Over the scialbo there is a thin yellow-brown film (n. 2), probably composed of gypsum and a proteic medium (compare with the specimen n. 1). Such film thickens near the fissures edges or over small cavities (arrows), where some residuals of an earthy deposit are overlaid on it.

Qualitative tests aimed at identifying the class to which the binders and organic fixative agents belong

Method

Tests have been carried out according to the guidelines of the DIMOS: *corso sulla manutenzione di Dipinti murali – Mosaici – Stucchi; Parte I: Tecniche di Esecuzione – Materiali Costitutivi, Modulo 3, Leganti, Fissativi, Pigmenti: Metodi di Riconoscimento. Istituto Centrale del Restauro, 1978 Roma*

These tests have been carried out on some milligrams of material including both the scialbo and the overlaid yellow-brown film

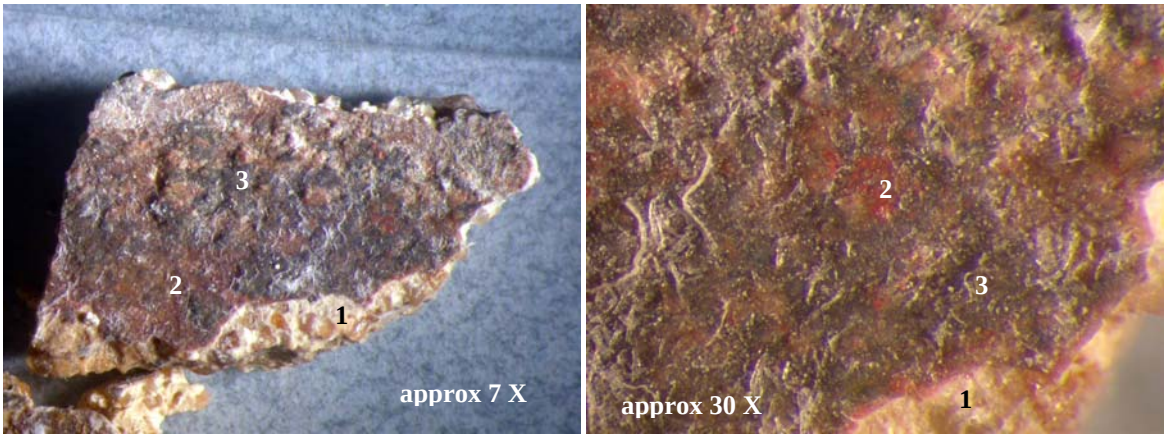
Results

Qualitative analysis of the organic substances	
Proteinic substances (egg, animal glue, casein, etc.)	++
Polysaccharides (gum Arabic, honey, starch, etc.)	-
Natural resins (colophony,, dammar, ecc.)	-
KEY ++: substance present in large quantity; +: s. present; +/-: s. may be present in traces; - s. absent.	

Sample no. 9

Information on the sampling area and description accompanying the sample

"East Wall, green shield behind a horse"



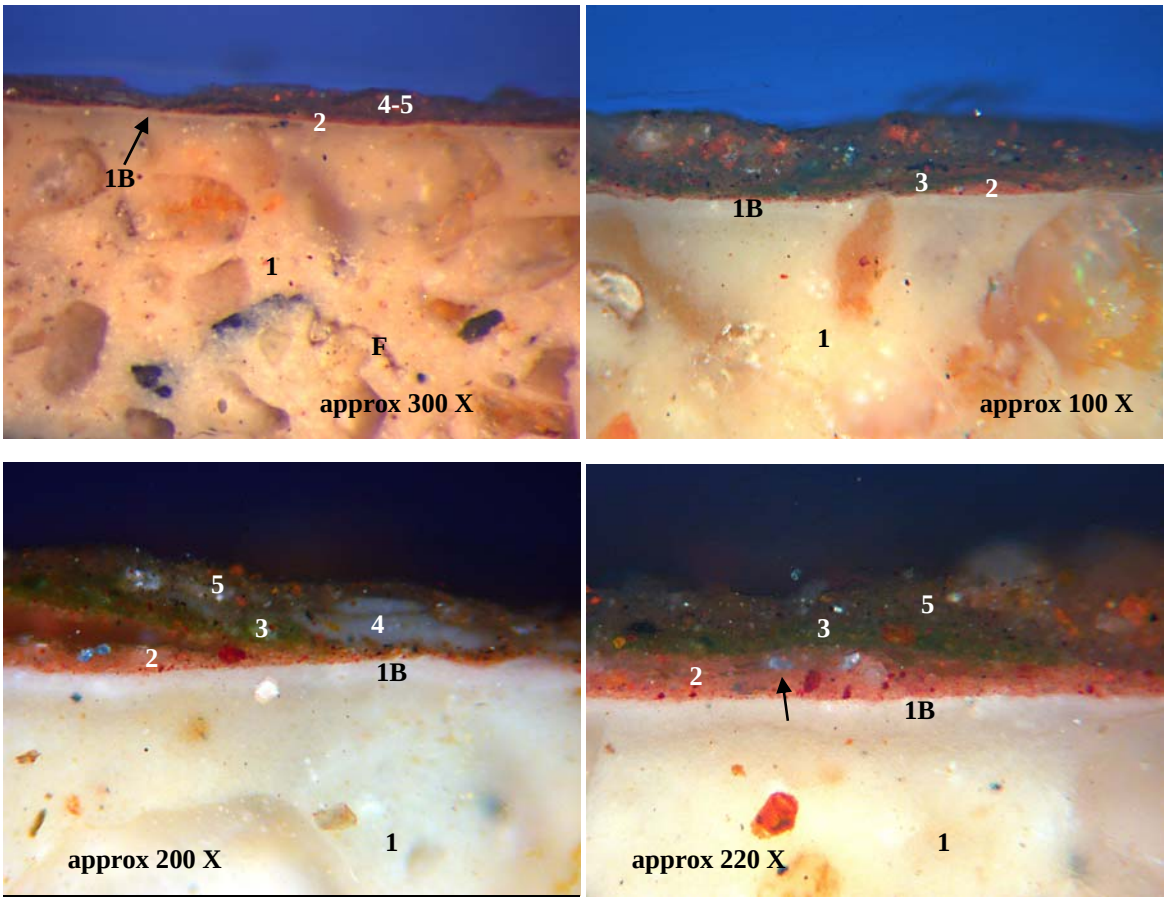
Stereo-microscope, reflected light.

The two photographs, taken at different magnification, show the surface of the fragment used for the polished cross-section prepared for the microstratigraphic study. The fragment includes a layer of plaster composed of lime and siliceous sand (n. 1), a thin red drawing (n. 2) and a discontinuous and irregular brownish - grey layer (n. 3)
The examination of the polished cross-section pointed out the presence of another green pictorial layer.

A microchemical test aimed at identifying the polysaccharides (3) was carried out on several tens milligrams of dust selectively removed from the surface of the pictorial film(10)
Thanks to the negative outcome of the test it is possible to exclude that the superficial greyish layer could represent a deteriorated gum Arabic – based fixative agent. Moreover, the absence of polysaccharides indicates that such kind of substance was not used as medium in the pictorial layers, at least in the area in which the specimen was sampled.

[10] Polysaccharides are typical of some plant gums, but they are also in starches and honey .

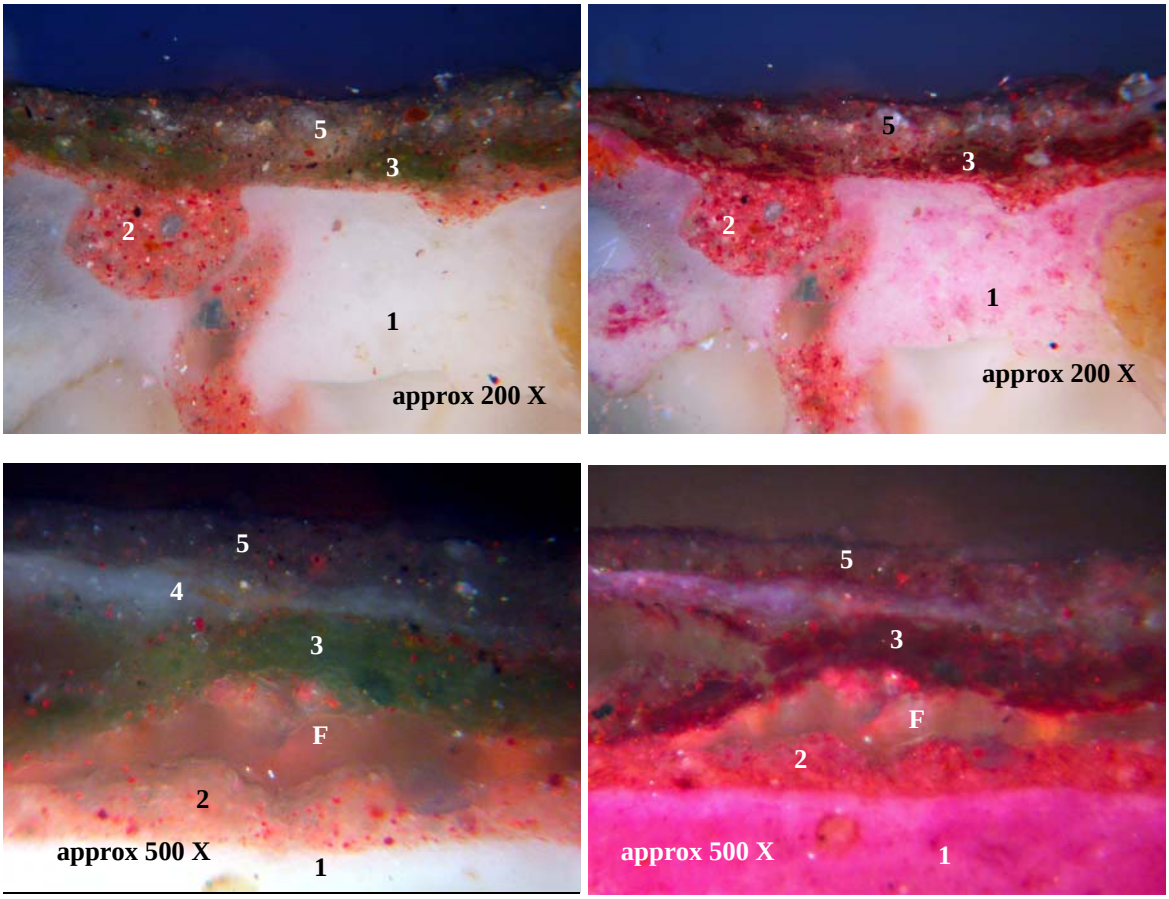
Microstratigraphyc analyses on cross section with microchemical and histochemical tests
Analytical sheet written according to the Doc Normal 12/83



Cross section, reflected light.

The upper left photograph, taken at a relatively low magnification, shows the structure of the plaster realized with lime and siliceous sand with a mainly quartz formation (n.1). The photograph also illustrates the presence of a thin whitish level (n. 1B) which could derive from a superficial accumulation of lime, produced by finishing the plaster or by the application of a very diluted layer of whitewash. Following: the pictorial film, composed of two drawings of colour, respectively pale red (n. 2) and green (n. 3); two other layers (n. ri 4 and 5), better visible in the below pictures and in the next page. The letter F indicates a fissure produced by the drying process regarding the lime present in the binder.
The optic and morphologic features of the drawing n. 4, that is very discontinuous and of a white - greyish colour, are similar to those characterizing the fixatives - protectives used during recent restorations ([11]).. The overall colour of the upper layer is grey with violaceous tones (n. 5) and is composed of iron oxides, carbon particles, perhaps ores and pigment granules that were accidentally englobed by the surface of the lower layers
This layer can be attributed to a pictorial drawing applied during recent restoration. The black arrow in the bottom right picture indicates some grains of Egyptian blue present within the layer n. 2 that, therefore, should take on a light violaceous tonality. (As follows)

[11] Such hypothesis is formulated on the base of the detectable characteristics under reflected light test and it should be confirmed with targeted analysis, such as IR spectrophotometry (FT-IR, micro-FT-IR) and microanalysis with a Scanning Electron Microscope (SEM - EDS).



Cross section, reflected light.

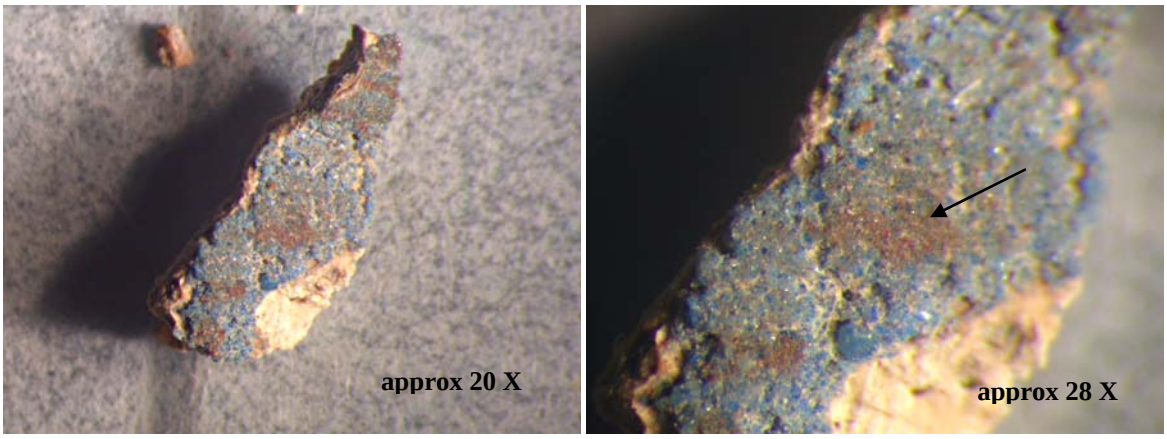
The four photographs describe two details of the cross section before and after the fuchsine test aimed at identifying and localizing all proteic substances. It is sure that such substances are present in the green pictorial drawing that is composed of Green Earth, few green "*fritta egizia*" and rare Egyptian blue (n. 3). It is less probable that the red layer with violaceous tones (n. 2) holds them, but they could be found in the repainting (n. 5).

In the above photographs the red layer with violaceous tones (n. 2) penetrate into the fissures of the below plaster. This indicates that the pictorial film was applied on the plaster when it was already drying up. Therefore the first layer of colour (n. 2), composed of calcium carbonate, was applied with the `*mezzo fresco*' technique.

Sample no 10

Information on the sampling area and description accompanying the sample

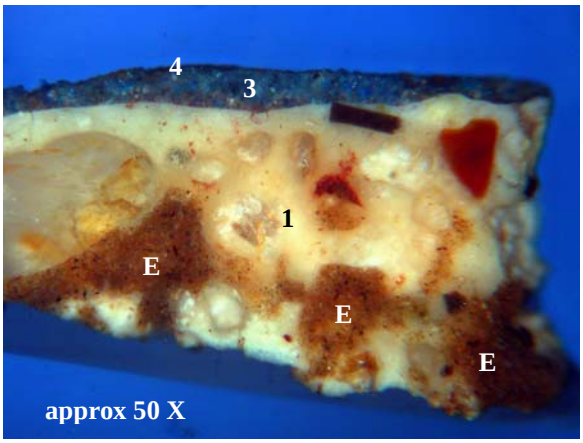
"East Wall, blue shield".

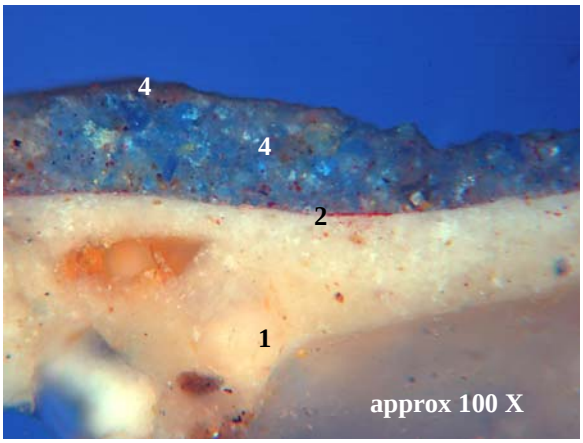


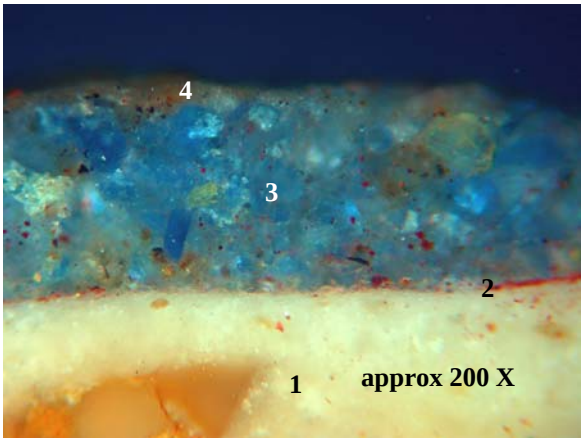
Stereo-microscope, reflected light

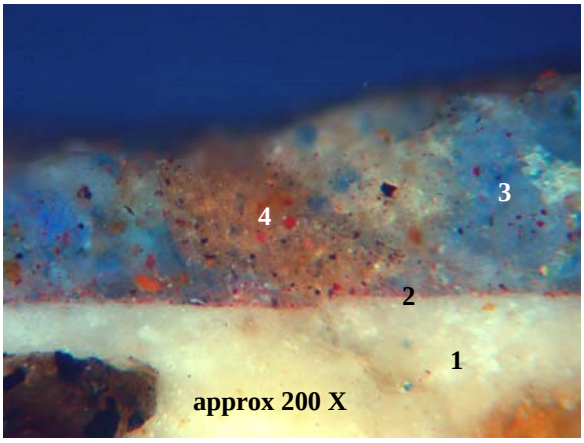
The two photographs, taken at different magnification, illustrate the surface of the fragment used for the polished cross-section prepared for the microstratigraphic study. The fragment includes a blue - azure Egyptian blue based (n. 2) and traces of a grey-violaceous drawing (arrow).
At the base of the plaster (not visible in the photographs) there is a discontinuous earthy deposit (see also the photos of the cross section shown in the following page).

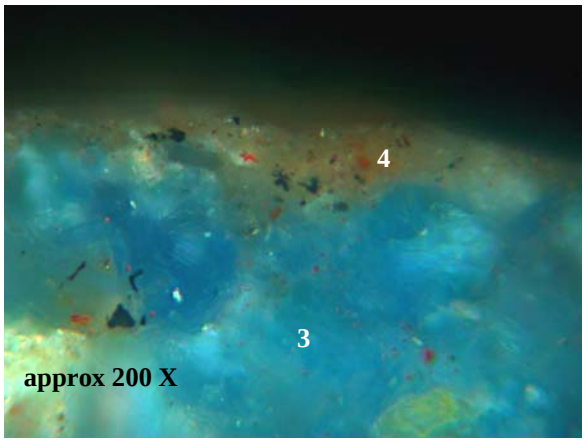
Microstratigraphyc analyses on cross section with microchemical and histochemical tests
Analytical sheet written according to the Doc Normal 12/83











Cross section, reflected light.
The upper left photograph, at a relatively low magnification, shows nearly the entire polished cross-section. At the base of the lime and siliceous sand plaster (n.1) there is an earthy deposit (E) penetrated in the fissures of the mortar. Over the plaster drawing, separated by a slight and discontinuous level of haematite (n. 2, not visible in the 'photograph'), there are: a thick blue-azure pictorial layer made up of Egyptian blue (n.3) and a thin and discontinuous grey - violaceous level(n. 4).

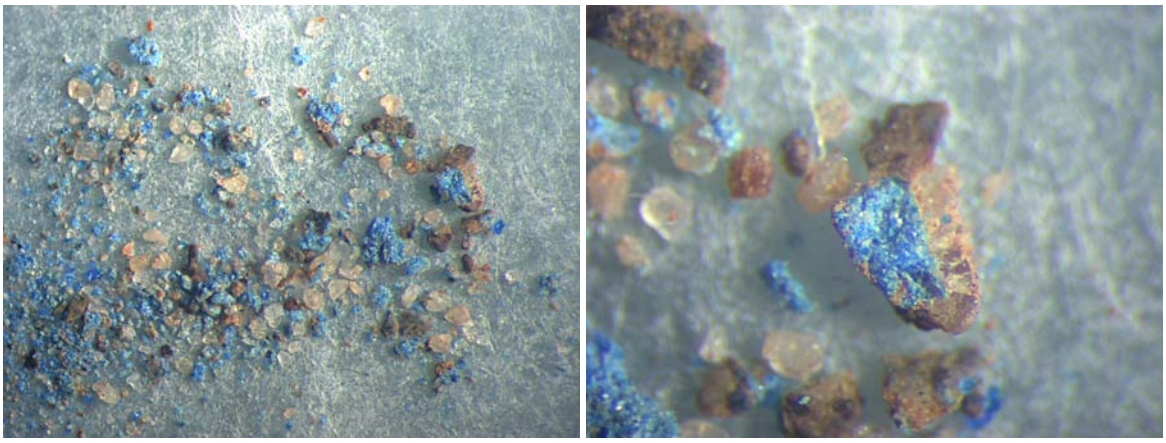
The other photographs, at higher magnification, describe in detail all layers. In particular, the bottom left picture shows the thin and discontinuous superficial layer of a violaceous-grey colour (n. 4) that probably was applied during a recent restoration. The thin and discontinuous red level containing haematite (n. 2) probably represents the preparatory drawing.

At the moment, the blue pictorial layer is nearly lacking in calcite; therefore it seems to derive from a "*stesura a secco*". Nevertheless micro and histochemical tests did not determine which kind of binder was used, probably owing to the strong alteration and mineralization in oxalates of the binder itself. It was favoured by the presence of the copper contained in the Egyptian blue, that is cuprorivaite ($CaCuSi_4O_{10}$) based.

Sample no 11

Information on the sampling area and description accompanying the sample

"North Wall, wall relief, dust of"fritta egizia."



Stereo-microscope, reflected light.

The two photographs describe part of the sampled dust. In particular, the one on the right shows the blue-azure pictorial layer placed on the lime and siliceous sand plaster. Over the pictorial film there is also a greyish layer.

Infrared spectrophotometric analysis (FT-IR)

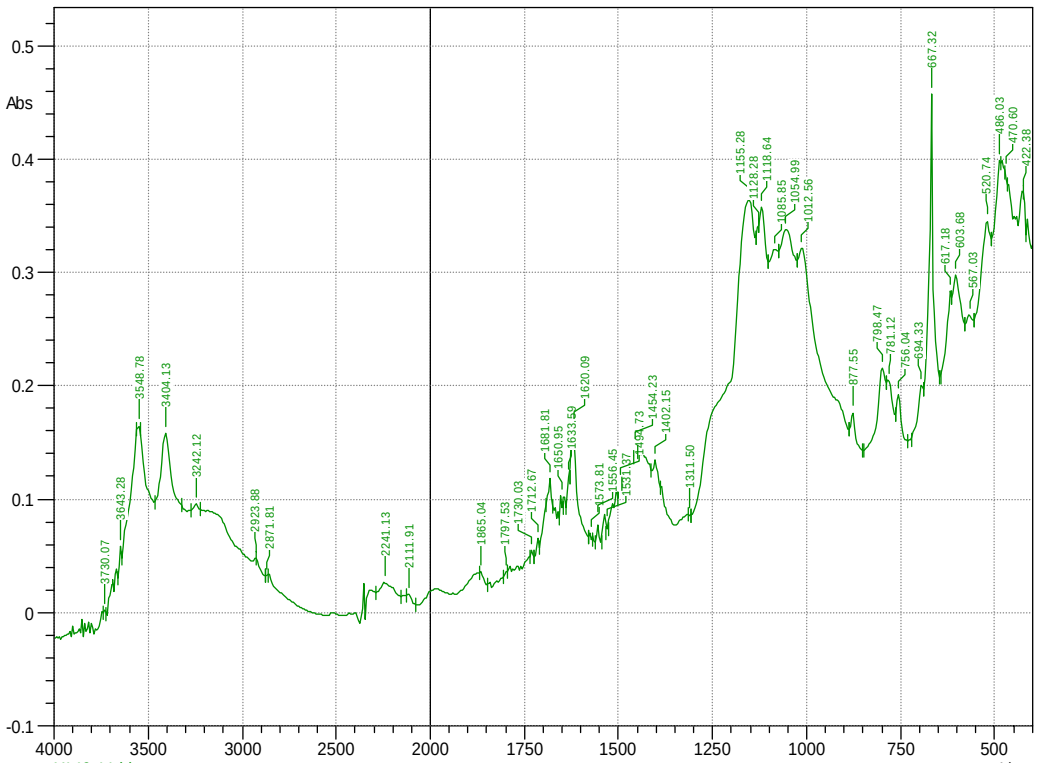
The micro-specimen, including the blue-azure pictorial film and the overlaid greyish layer, have been accurately grounded with an agate mortar together with KBr.
The material has been analysed in transmittance by DSR cell.
The interpretation of the FT-IR spectrum obtained by the analysis has been carried out by comparison with the laboratory data base and with the one reported by various scientific publications. In particular, assignment was made on the basis of the vibration frequencies, recorded under the same test conditions, of reference standards, either pure or mixed with specific matrixes (calcite, calcite and gypsum, etc.).

Results

Study of the IR spectrum obtained from the analysis (see following page) establishes that the sample is essentially formed from the following components listed in order of relative abundance:
- *Silicate mineral* attributable to *cuprorivaite* ($CaCuSi_4O_{10}$);
- *Gypsum* ($CaSO_4.2H_2O$);
- *Quartz* (SiO_2)

There are present small amounts e/o in traces, also: *Calcite* ($CaCO_3$); *Calcium oxalate* both *whewellite* ($CaC_2O_4.0.5H_2O$) and *weddellite* ($CaC_2O_4.2H_2O$) or copper (*mooloite*, $CuC_2O_4.H_2O$) and *Organic substances.*The IR spectrum does not reveal the presence of other organic binders as polisaccharides, natural resins, oils and waxes.

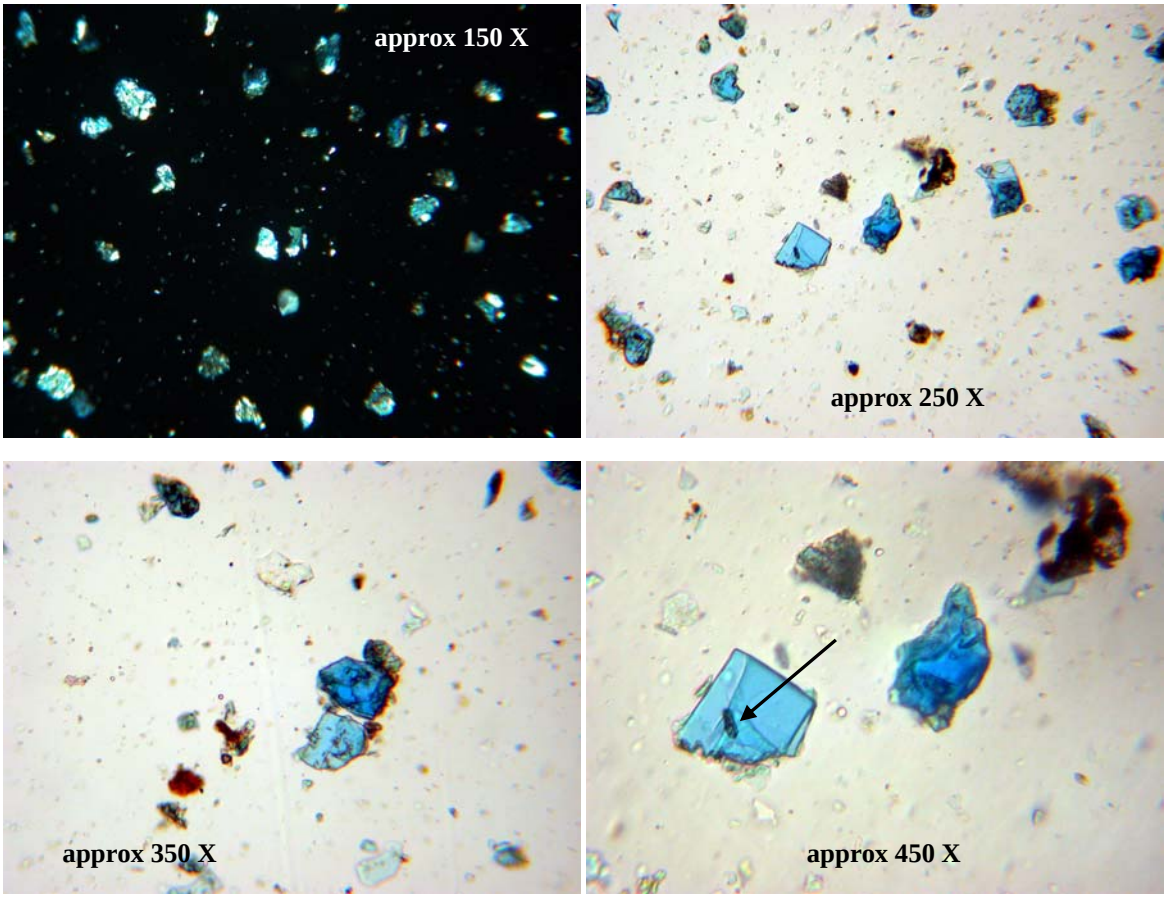
Also the negative results of the micro-chemical tests confirm the absence of polysaccharides (gum Arabic, starch, honey, etc) and natural resins (colophony, dammar, etc)([12])..



Spectrum FT-IR.

[12] Spot tests have been carried out on few tens milligrams of the specimen including both the scialbo and the superficial layer. The analyses were carried out in compliance with the indications contained in *DIMOS: corso sulla manutenzione di Dipinti murali – Mosaici – Stucchi; Parte I: Tecniche di Esecuzione – Materiali Costitutivi, Modulo 3, Leganti, Fissativi, Pigmenti: Metodi di Riconoscimento. Istituto Centrale del Restauro, 1978 Roma...*

Optical mineralogical and microchemical analysis aimed at identifying the blue-azure pigment



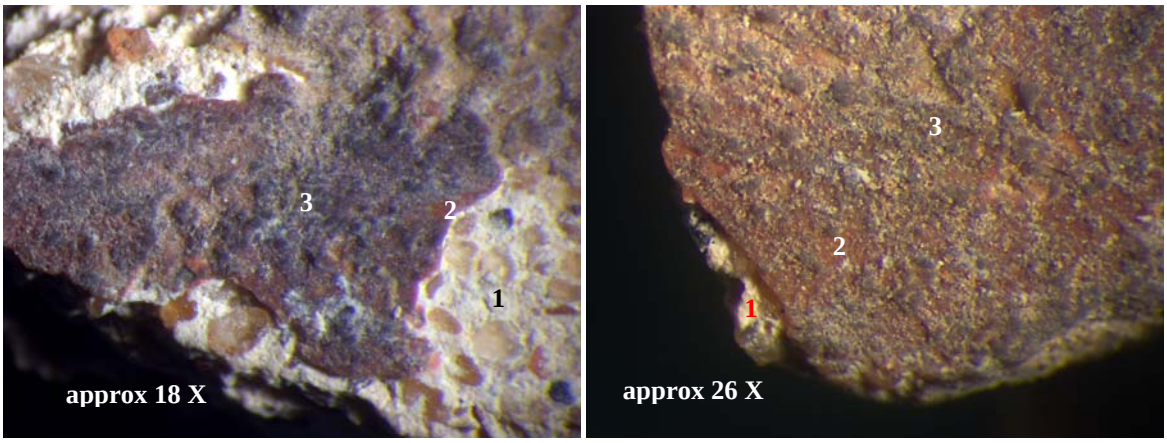
"Preparation", polarizing microscope, transmitted light. Upper left picture: Nicols +; other pictures: only polarizing

The four photographs, illustrate different Egyptian blue granules characterized by the typical colouration ranging between azure and blue and, above all, by the presence of some prismatic inclusions inside it (arrow).
The upper left photograph shows that most of the blue Egyptian granules are birefringent with interference colours of the first class blue grey type (luminous granules).

Sample no 12

Information on the sampling area and description accompanying the sample

"East Wall, brown – orange "tondo" behind green shield".



Stereo-microscope, reflected light.

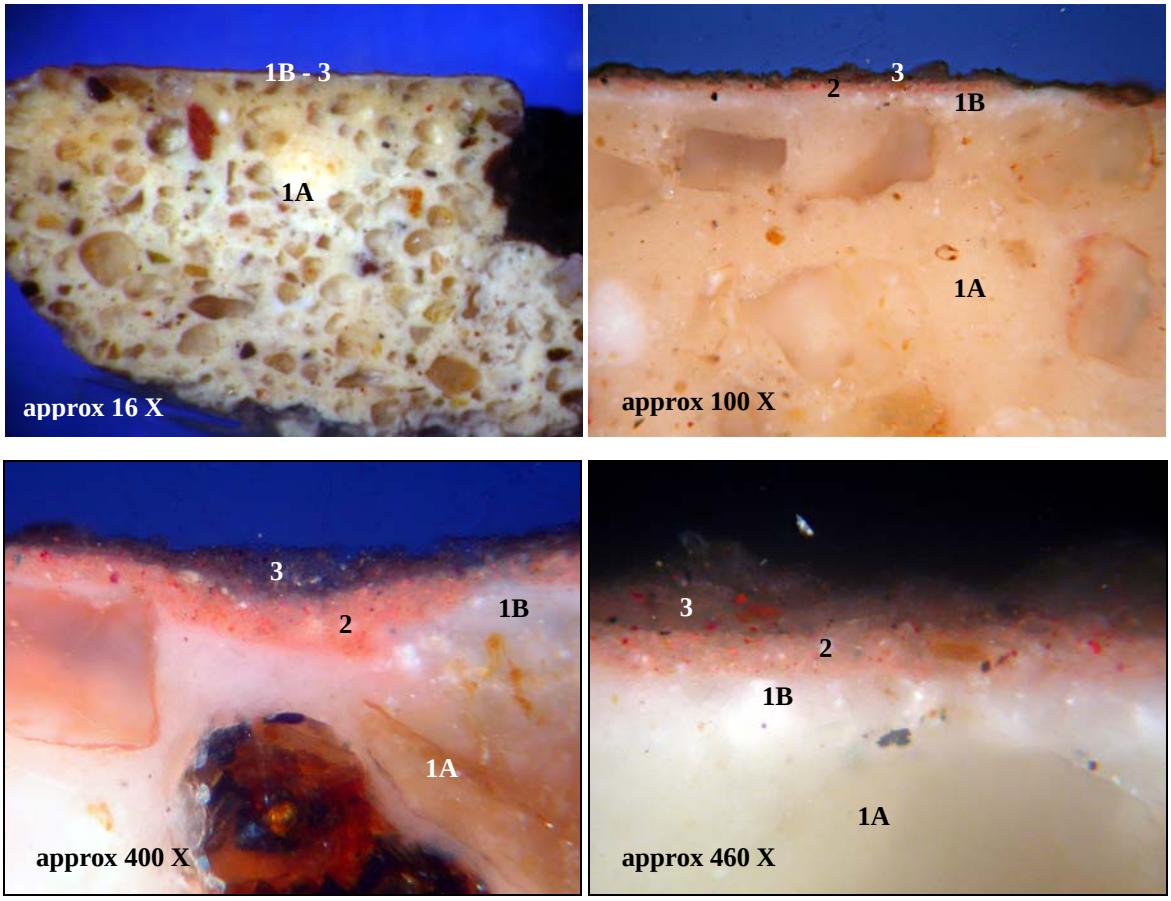
The two photographs, at different magnification, show the surface of the sampled fragment. This includes a layer of lime and siliceous sand plaster with a mainly quartz formation (n. 1), a reddish drawing (n. 2) and a discontinuous blackish – grey layer (n. 3).

A microchemical test aimed at identifying the polysaccharides (3) was carried out on several tens milligrams of dust selectively removed from the surface of the pictorial film. (13)The negative outcome of the test it is safe to exclude that the superficial greyish layer could represent a deteriorated gum Arabic – based fixative agent. Furthermore the absence of polysaccharides indicates that such substances were not employed as medium for the pictorial layer, at least where the specimen was sampled.

In the following page the outcomes of the microstratigraphic analyses in polished cross-section are presented.

[13] Polysaccharides are typical of some plant gums, but they are also in starches and honey .

Microstratigraphyc analyses on cross section with microchemical and histochemical tests
Analytical sheet written according to the Doc Normal 12/83



Cross section, reflected light.

The upper left photograph, at a relatively low magnification, shows the structure of the plaster, made with lime and siliceous sand with a mainly quartz formation (n.1A). In the photograph it is possible to note also a thin whitish level (n. 1B) over the plaster. It probably derives from a superficial accumulation of lime, created by the smoothing of the plaster or by the application of a very diluted layer of whitewash.

Following there is an rose pictorial layer, composed of haematite finely dispersed in a calcium carbonate binder. Therefore the layer was applied with the "*buon fresco*" technique or, more probably, with a "*mezzo fresco*" one.
The layer n. 3, of a general grey colour with violaceous tones, is composed by gypsum, proteic substances, iron oxides and probably carbon particles. This painting was applied during a "restoration".