

Mammuthus meridionalis from Madonna della Strada (Scoppito, L'Aquila): diagnostics and restoration

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SUPPLEMENTARY ONLINE MATERIAL

METHODS

I - Mapping

A total of 156 photographs (size 7360×4912 pixel, 300 dpi) were obtained using a Nikon D800 digital camera and Nikon Nikkor AF-S 24-70 mm f/2.8G Ed lens (used for most photos). Of these, 113 were taken in RGB at from ISO100 to ISO125 and 43 images were taken in UV light at ISO400. The latter were obtained by applying a Kodak Wratten 2a filter on the lens, and using a UV radiation source with a 400 watt gas-discharge lamp with an emission peak of about 360 nm (Fig. S1). The x-ray

plates consist of 45 impressed images showing the inner trabecular structure of the bones and where we assumed that armatures, pins and metal structures had been inserted. The x-ray plates were obtained using an x-ray tube with a maximum capacity of 300 KV. The rays were impressed on phosphor film inserted in a lightproof cassette. Exposure times were calibrated according to the osteological structures. The resulting plates were digitalized with a high resolution scanner and the grey scale was corrected to improve the visibility of details.

Mapping was performed using AutoCAD-LT-2013, with homologous overlapped RGB and UV photos and

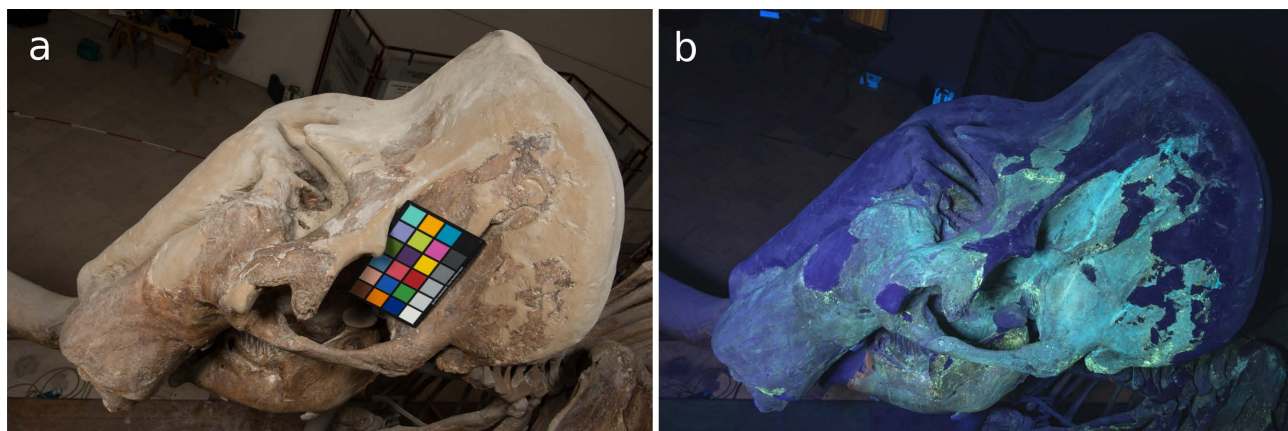


Fig. S1 - Lateral-dorsal view of the skull in (a) RGB with colour calibration target (x-Rite release) and (b) UV. In b), the reconstructed portions in dark-blue and consolidants or glues along the temporal fossae (cyan) are clearly evident.

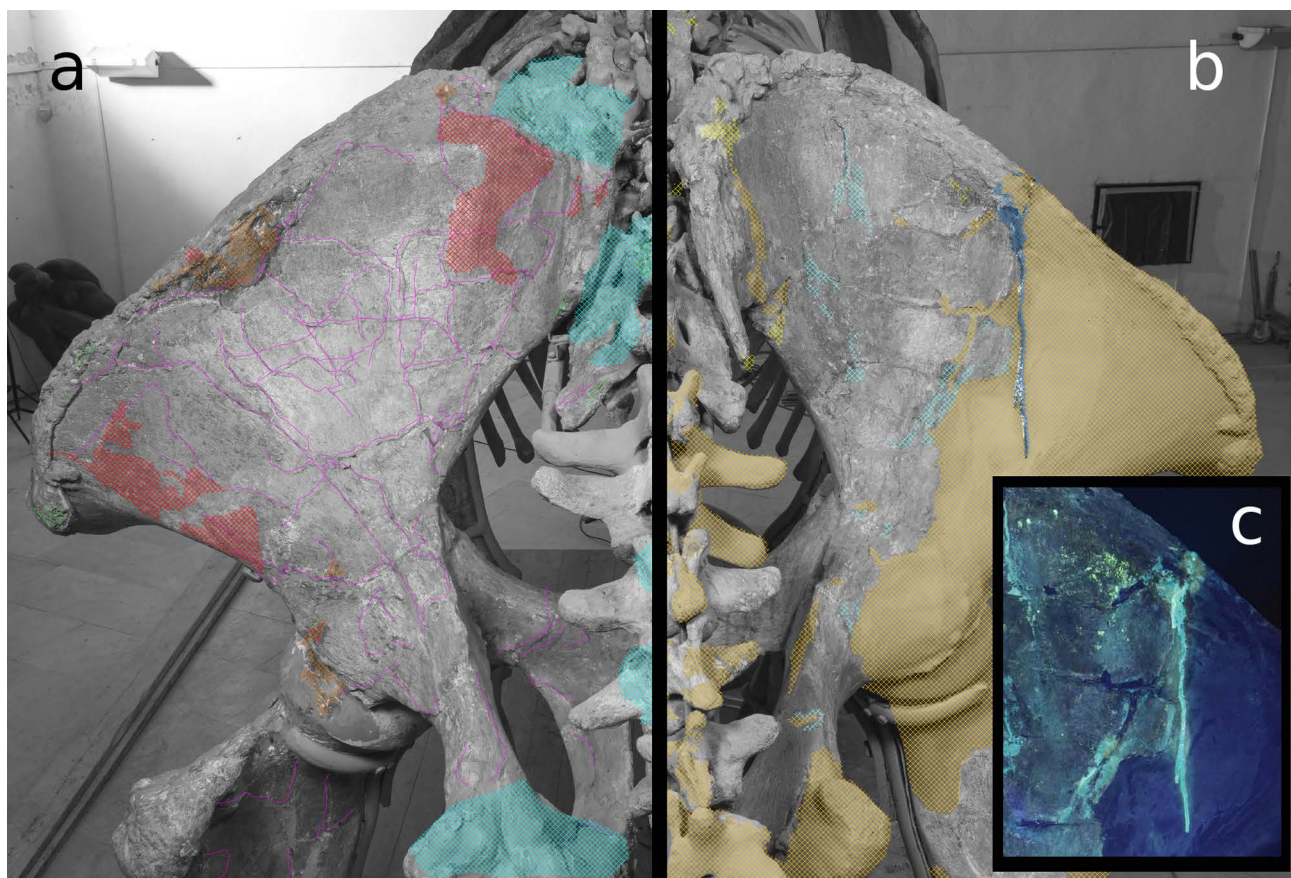


Fig. S2 - Mapping of the (a) left and (b) right ilia in distal view. a) For definition of the classifications, see Tab. S1; b) reconstructions (beige) and glues and consolidants (blue and fluorescent blue); c) detail of previously consolidated right ilium in UV light.

x-ray plates where available. The vector images were exported as .dwg and .pdf files, for a total number of 116 images. Different types of information were recorded on different layers in the form of polygons divided into four sets: surface characterization, considering the initial status of the bones and their taphonomy; reconstructions and paint from previous restorations; areas used as preliminary markers (x-ray, chemical samples), and emergent materials identified in relation to a specific chromatic response to UV light (Tab. S1, Fig. S2).

2 - Compositional and textural investigations

The analyses were performed on small samples measuring about 0.5 mm³. In just a few selected cases, thin sections were prepared from samples generally measuring about 1.0-1.5 cm. Preliminary stereomicroscopic study of the samples was followed by detailed study by optical microscopy (OM), with observations in polarised transmitted and reflected light. Based on these observations, studies were then performed using complementary diffractometric and spectrometric techniques: X-ray powder diffraction (XRPD) to identify the mineralogical and crystallographic characteristics of the crystal phases present and infrared (FTIR and μ -FTIR) and μ -Raman spectroscopy to characterise the inorganic and organic phases. Elemental analysis was carried out by X spectroscopy using a scanning electron microscope (SEM) coupled to an energy dispersive spectrometer (EDS) (Fig. S3). Textural analyses, particularly important

in light of the project's objectives, were carried out with both OM and SEM.

During the first sampling phase, fragments of various compositions were taken from the surfaces of the artefact (6 representative samples of the ulna, rib, radius and humerus); tusk (2 samples, internal and external); reconstructions (4 samples); surface patinas (2 samples); resins (5 samples) and paints (4 samples).

For the second phase involving 3D study of the long bones, minimal sized core samples were taken from the epiphyses of the right ulna, right tibia and left humerus. Polished thin sections were obtained from the median-longitudinal plane of the core samples for textural and spectroscopic study (OM, SEM-EDS, FTIR and μ -FTIR and μ -Raman); the lateral portions provided the powder for the XRPD study.

3 - The dynamic behaviour of the skeleton

The Finite Elements model of the mammoth was constructed using the 3D geometric model obtained with the laser scanner technique. The geometric "shell" elements externally defining the overall geometry were converted into plate finite elements by automatic refinement to obtain a more precise and regular mesh. The structure thus defined was corrected manually to remove all the defects of shape (overlapping faces, multiple faces, etc.), rendering the model suitable for transformation into a solid mesh. The 2D plate elements were then transformed into tetrahedral solid elements by automatically filling

1) SURFACE CHARACTERIZATION	
includes everything noted on the bone surface before conservative restoration	
<i>brown colour</i>	Dark brown colour of the bones. Area sampled for chemical and mineral analyses
<i>red colour</i>	Red colour of the bones. Area sampled for chemical and mineral analyses
<i>hard dusty coat (light blue)</i>	Dusty substance distributed over the bone surface and adhering to the periosteum
<i>fractures/cracks</i>	Areas with visible cracks as the consequence of peri-depositional events (presumably stable) or post-recovery events deriving from time decay, sudden movements or overload stress. Some cracks had been partially filled during previous restorations.
<i>microholes</i>	The presence of holes was limited to the anterior diaphysis of the right femur
<i>trabecular bone</i>	Inner trabecular structures or areas without periosteum
2) PREVIOUS CONSERVATIVE RESTORATIONS	
(paints - reconstructions): includes everything added after recovery of the skeleton	
<i>light brown paint</i>	Area with light brown paint
<i>dark brown paint</i>	Area with dark brown paint
<i>metal pins</i>	The position of the pins was obtained from the x-ray plates
3) DIAGNOSTICS	
identification of bone microparts for preliminary analysis	
<i>x-ray</i>	Identification of squares for x-ray study
<i>first sampling</i>	Sampling for first diagnostics phase (17/7/2013).
4) SUPERIMPOSED SUBSTANCES	
areas identified by the different chromatic response of the surface in UV photos	
<i>bright light blue</i>	Probable consolidant from fractures and broken parts
<i>purplish blue response</i>	Corresponding to reconstructed and painted parts
<i>red response</i>	Red line painted to define the boundary between original bones and reconstructions during the first restorations
<i>light blue response</i>	Area with a light blue response to UV
<i>fluorescent yellow response</i>	Plaster/reconstructions not painted, or with paint removed subsequently

Tab. S1 - Legend of pre-restoration mapping of the periosteum. The information on the areas was identified and determined by RGB and UV photos and x-rays.

the cavities in the model to obtain a completely solid 3D model (Fig. S4).

In a second phase, the steel frame was manually shaped using beam-type one-dimensional elements. The model of the frame is independent from the structure of the mammoth and linked at points corresponding to the beam elements distributed along the frame (Fig. S5). This modelling strategy was functional to correct transfer of the entire weight of the bones to the frame.

Knowledge of the weight of the skull led to the following properties being used for the bones:

Mass density: 13000 N/m³

As regards the elastic modulus, a relatively low value was used (elastic modulus = 2.58e + 008 N / m²) as a lower

skeleton stiffness enables the frame to support the entire mass resulting from the mammoth skeleton. Type S235 steel was used for the frame structure.

The FE model was calibrated with the results of the dynamic tests. Four/five acquisition setups were employed for the dynamic identification, with 8 to 12 seismic high resolution piezoelectric accelerometers fixed at different levels on the frame (Figs S6, S7). The dynamic identification tests clearly identified the first two frequencies (0.56 Hz, 0.75Hz), representing the first vibration modes in the transverse and longitudinal directions.

Linear static analysis was carried out in order to assess the stress levels in the elements of the metal frame. The only force considered for this analysis was self-weight.

4 - The restoration

In chronological order, the operations were as follows: 1. disassembly; 2. removal of paint from the surfaces of the reconstructions and bones; 3. cleaning of the original parts with removal of the previous protective agent from the surface; 4. removal of plaster and mastic residues using a precision micro-sandblaster; 5. removal of excess filler and identification of the contact surfaces between the original elements and reconstructions; 6. pre-consolidation of a number of particularly fragile portions and consolidation by immersion; 7. structural consolidation with resin

Mode nr.	Frequency (Hz)	Mass X (%)	Mass Y (%)
1 st mode	0.547	84.2	0.76
2 nd mode	0.793	0.55	35.56
3 rd mode	1.036	0.20	46.56
4 th mode	2.060	0.01	0.00
5 th mode	2.399	0.00	0.05

Tab. S2 - Frequencies and participant mass related to the first 5 mode shapes.

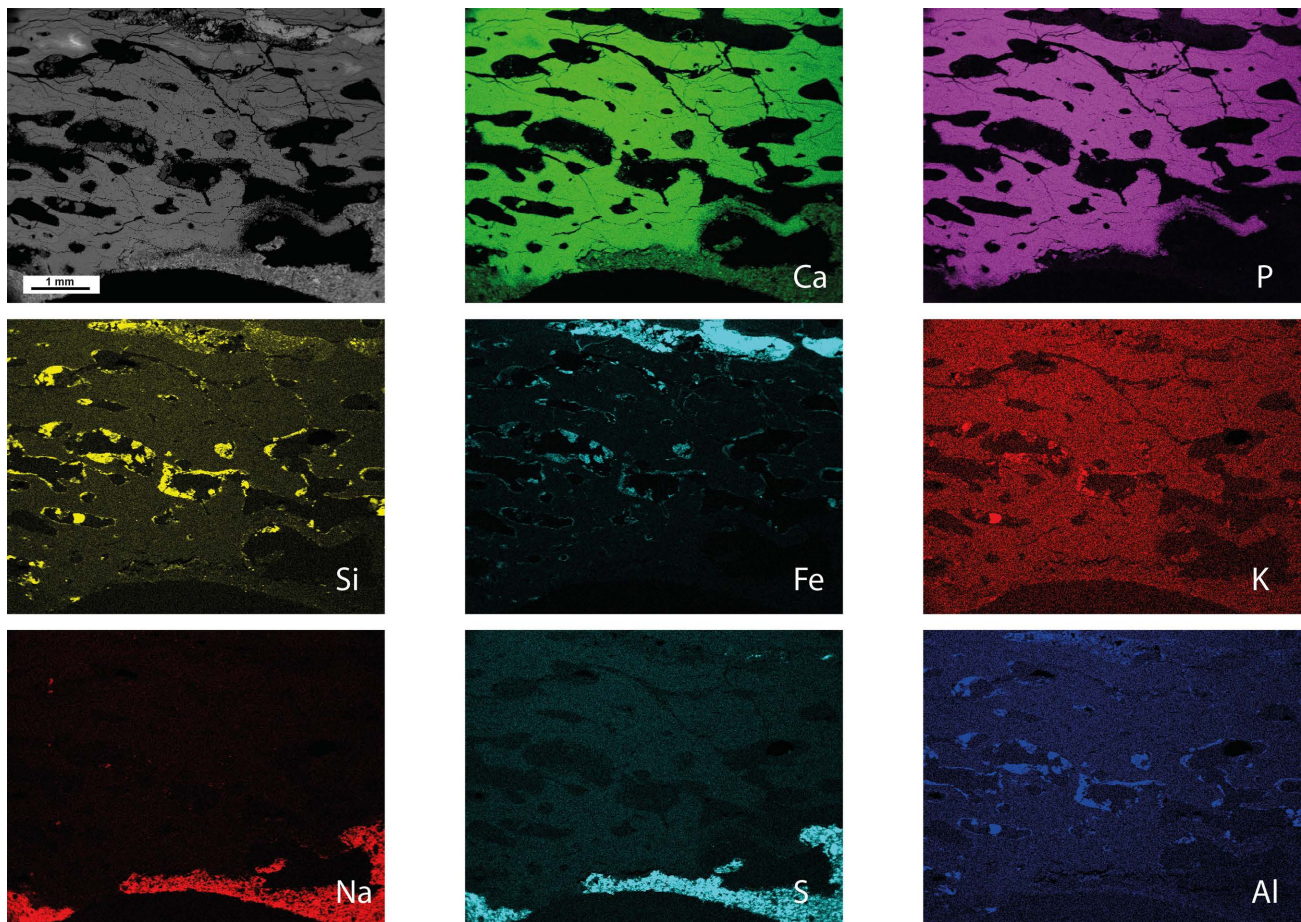


Fig. S3 - Chemical maps based on SEM-EDS analyses of a thin section of a bone sample of the right tibia.

infiltrations and gluing; 8. reconstructions; 9. filling; 10. colouring of the reconstructed portions.

1. The bone elements were disassembled in three different phases, taking into account the sequence of operations in order to rationalise and reduce handling of the bone elements. This operation was supported on site by wheeled multi-shelved tables where the removed bones were placed after cataloguing. To improve safety, before

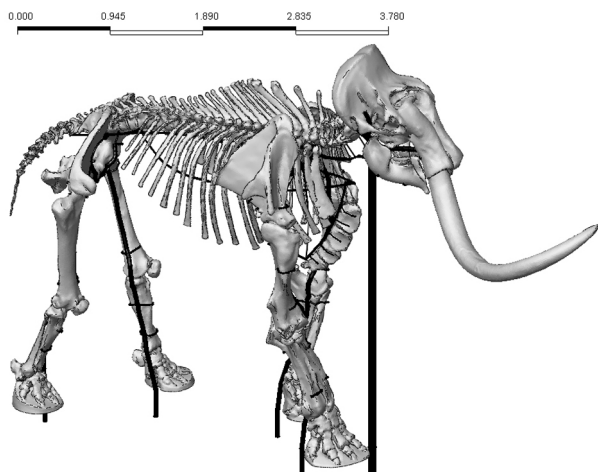


Fig. S4 - The Finite Element model of the *Mammuthus meridionalis*.

dismantling, a protective net was mounted under the skeleton. In the case of the largest bones, suitable lift and support systems were designed and modulated according to the anatomical characteristics and fragility of the various elements. The most complex movements involved the pelvis and skull which required specific protection and lifting simulations (Fig. S8). Modular systems were used for the limbs, with specific structures for the skull, pelvis and scapulae (Fig. S9). The supports on which the various elements rested transmitted the weights evenly, avoiding dangerous load concentrations.

Before removing the pelvis, a safety bandage with Paraloid B72 was applied, as the 160 kg weight, considerable size, minimal thickness of the ilium wings and sponginess of the ilium and pubis placed this anatomical element at considerable risk. After protection of the surfaces with cyclododecane in mineral solvent, a protective layer of Japanese tissue was applied and plaster was poured into the two ischiopubic openings to improve stability during handling. A metal stabiliser tie rod was inserted into the central cavity. Weighing about 500 kg, the skull was moved with the help of specifically designed and constructed systems consisting of metal tie rods, straps and manual hoists, with the insertion of rubber protection. Use of the top metal crossbeam of the scaffolding and lift platform was fundamental.

2. The light brown paint was removed from the surfaces of the reconstructions and bone material with solvent (acetone) applied on pads and probes.



Fig. S5 - The Finite Element model of the metal structure supporting the mammoth.

3. Before cleaning, the pure solvents and solvent mixtures were tested. The efficacy of cleaning and their actual capacity to remove the PVAc residues from the bones were verified by analyses performed on microsamples before and after cleaning and on powder residues. The solvent tests were carried out using acetone, butyl acetate, dimethyl sulphoxide and Dowanol, pure and mixed in various percentages.

The analytical results (FTIR) showed that cleaning with acetone completely removed the PVAc present in considerable quantities on the bone surfaces, without signs of “transport” dynamics in depth. The volatility of the acetone was also a further advantage for cleaning. Small bones were cleaned by partial controlled immersion in suitable tanks containing the solvent, using soft bristle brushes to completely remove the PVAc residues. In the



Fig. S6 - View of some of the acceleration sensors.

case of large bones, the acetone was applied by brush and poultice.

4. The presence on the bones of widespread plaster residues not removed after recovery of the skeleton in 1955 and mastic residues left from previous restorations necessitated the use of a precision micro-sandblaster. The inert substance used was Hydrosift HDO micronised calcium carbonate with a grain size of 140 μm and a rounded shape, allowing the action to be precisely targeted by the operator (Fig. S10).

5. Lowering the edges of the reconstructions covering extensive portions of bone enabled the contact surfaces between the original bone and reconstructions to be identified and the diffusion and penetration paths to be improved for more effective tissue consolidation.

6. The consolidant was chosen bearing in mind that the bones had already been treated previously. It was decided not to experiment with either nanotechnologies or ethyl silicate as there was a lack of supporting literature. The choice therefore fell on suitable acrylic products (Keene, 1987; Shelton & Chaney, 1994; Linares Soriano & Carrascosa Moliner, 2016) with the following characteristics: low specific weight, minimum molecule

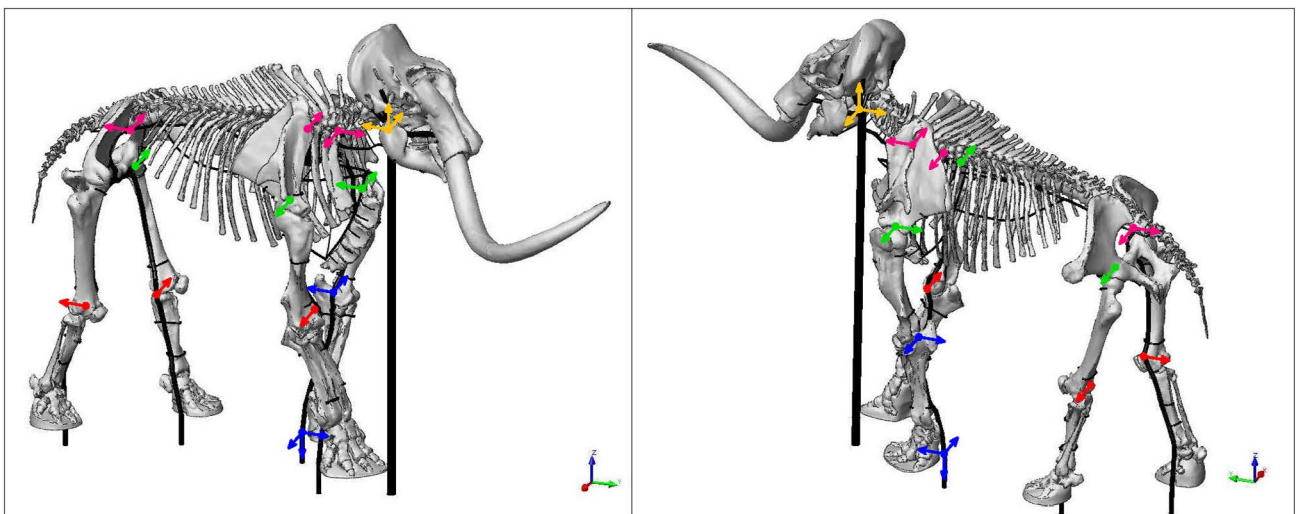


Fig. S7 - Points of application of the acceleration sensors.



Fig. S8 - The movement of the skull required specific protection and suitable lifting equipment.

size, low viscosity and high adhesive capacity. After specific research, the consolidant chosen was Degalan LP 64/12, an acrylic polymer which, unlike the more common Paraloid B72, had all the above characteristics. The decision was made after testing the two consolidants dissolved in different solvents (acetone, butyl acetate and methyl ketone) at concentrations of 5%, 7% and 10%. Butyl acetate solvent was preferred to acetone for its lower volatility and application by immersion was chosen as the most suitable procedure.

Chemical stratigraphic and micro-FTIR analyses were performed on polished sections of small core samples taken from the bone elements treated with immersion in Paraloid B72 and Degalan LP 64/12, both in a 5% solution in butyl acetate. With the sample treated with Degalan LP 64/12, penetration reached about 20 mm, better than the 15 mm penetration achieved in the sample treated with Paraloid B72.

Resistograph study of five points on three treated bone elements (2 points on the phalanx, 2 on the radius and 1 on the vertebra) showed good reinforcement of the spongy tissue trabeculae. After further testing on site, the immersions were performed in a 10% solution of Degalan LP 64/12 in butyl acetate for a period of between 2 and 4 hours depending on the size of the anatomical element (Fig. S11).

Particularly fragile anatomical elements, or parts of these, were consolidated with localised injections of 10% Degalan LP 64/12 in butyl acetate. Before consolidation, nylon-lined boxes of a size and shape corresponding to those of the bones were prepared, with the aim of optimising and limiting as far as possible the quantities of solvent used for the immersions. The solutions were used for a number of cycles as long as the liquid remained sufficiently clear.

After the consolidant bath, the bones were dried slowly for different periods of time according to the dimensions



Fig. S9 - Support systems with specific structures were used for the skull and pelvis.



Fig. S10 - The restorer using a micro-sandblaster on the skull to remove residues of plaster and mastic left from previous restorations.



Fig. S11 - Handling of a humerus after immersion in consolidant solution.

and characteristics of the individual bones, from 3-4 weeks for the smallest to more than 2 months for the skull and pelvis.

Immersion of the skull was particularly complex due to its size and the 700 litres of solvent used. For this purpose, a box was constructed around the bone element and its support.

7. For all moderately significant lesions, following containment micro-filling to avoid leaks of the product, structural consolidation was performed by injections of a 10% solution of the consolidant Degalan LP 64/12 in butyl acetate, or a liquid two-component epoxy resin. Cracked or very porous mobile or detached parts were first consolidated, then glued using a two-component epoxy resin, with insertion of fibreglass bars where necessary.

8. It was decided to modify a number of the anatomical parts reconstructed during previous restorations as these did not perfectly correspond to the anatomy of the animal. For these new reconstructions, a light two-component epoxy-based putty, reversible in polar solvents, commercially known as Balsite was used. This product was chosen for its positive characteristics, including ease of working, good adhesion to the fossil surfaces and elasticity (Ciocchetti & Munzi, 2007). This epoxy resin is very light and tends to adhere to the tools used for shaping and the nitrile gloves. Shaping could therefore only be performed correctly by moistening the tools and gloves with absolute alcohol. Thanks to its particular formula, it responds coherently with the support to the stresses generated by thermohygro-metric variations and it is therefore indicated

for reconstruction of the missing parts of fragile specimens (Ciocchetti & Munzi, 2007). Balsite has already been used successfully in the restoration of other fossil vertebrates (Agostini et al., 2010; Reggiani & Ghezzi, 2015)

9. The same product was also used as a filler. Balsite was found to have the best characteristics in terms of appearance, workability and speed of execution. Use of Balsite with the addition of 5% weight of a mixture of natural coloured earths, and walnut husk to obtain a stippled effect proved suitable for small and medium reconstructions of all bones. For larger reconstructions, only the junction with the bone was restored.

10. After specific testing, the reconstructions were integrated chromatically. The choice of the method respects the basic restoration principle according to which the chromatic effect must be in tone with the original material, but without falsification. Maimeri paints diluted in turpentine oil were used, giving a stippled chromatic effect very similar to the effect obtained in the reconstructions made from additioned Balsite. For small surfaces, the stippled effect was achieved using the point of a brush, while for larger surfaces it was achieved by spattering using a round brush with hard bristles soaked in the paint.

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