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Writing that cannot be erased: investigations of a box of pigmented inlays from the tomb chapel of an Old Kingdom noble

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Summary The British Museum holds in its collections a box of pigmented inlays from the tomb of Nefermaat, a senior official of the Egyptian Old Kingdom (early Fourth Dynasty, c.2600 BC), collected by Sir Flinders Petrie in the late nineteenth century. The pieces relate to an early, and apparently rapidly abandoned, style of tomb decoration where shapes were cut into limestone slabs and the resultant hollows filled with coloured pastes. This box of inlays thus represents some of the earliest evidence for tomb decoration available for study and reflects a very different form of decoration from the tempera painting generally found in Egyptian tombs. In terms of the pigment component, the most unusual feature was the definite presence of malachite and the apparent absence of either Egyptian blue or green frit, the two frit colours which so dominate the conventional Egyptian palette. The findings for the organic fractions were even more unexpected. Analysis of solvent extracts by gas chromatography-mass spectrometry (GC-MS) showed the presence of fatty acids and diacids that are typical of degraded oils. Subsequent analysis by pyrolysis-GC-MS yielded mostly straight-chain (alkane/alkene) pyrolysis products, which are consistent with a polymerized lipid; this apparently extensive polymerization implies the use of a drying oil. Linseed is the only source of drying oil known to have been available in ancient Egypt, although a number of semi-drying types, such as safflower or poppy oil, may also have been available. The presence of such a medium is potentially extremely significant, as the use of oil binders in dynastic Egyptian painting is virtually unknown. The use of an oil binder is unprecedented for such ancient material and may demonstrate a previously unsuspected technology.

INTRODUCTION

One of the most immediate images brought to mind by the mention of ancient Egypt is of vividly coloured tomb paintings, depicting scenes from life in both this world and the anticipated next. These paintings are among the glories of the ancient world and the source of much information on all aspects of Egyptian life and belief. However, in our current state of knowledge, the methods used in their production seem to emerge almost fully developed at the beginning of the Old Kingdom, the first period in which the distinctive material culture of dynastic Egypt was fully established. Artistic styles and conventions are known to have changed over time, but the technical aspects of both the painting method (tempera on stone or dry plaster), and the (very limited) palette seem to have been established early and to have undergone remarkably few changes until the influence of the Greek/Roman world grew during the Late Period and succeeding eras [1]. This apparently sudden development

is almost certainly illusory, resulting from the destruction of earlier examples of the evolving decoration of tombs [2; p. 55]. This study presents the results of examination for the earliest wall painting held in the collections of the British Museum, conducted in an attempt to establish any precursors of these fixed conventions.

THE TOMB CHAPELS OF NEFERMAAT AND ITET AT MEIDUM

Nefermaat and his wife Itet lived during the reign of Sneferu (c.2613–2589 BC), the first king of the Fourth Dynasty. Nefermaat was the son of an unnamed king, perhaps Sneferu himself, and held the titles Vizier and Overseer of all the King's Works. The large tomb shared by him and his wife consisted of two undecorated subterranean burial chambers with a common mudbrick superstructure above

in the form known as a *mastaba* (a rectangular, flat-topped building with sides inclined at a slight angle). Structures of this type had first appeared in the early dynastic period (First and Second Dynasties, c.3100–2686 BC), and it was the preferred form for elite tombs during the Old Kingdom. A cult place for the dead gradually came to be incorporated into the *mastaba*, evolving from a shallow niche cut in the eastern face. At the beginning of the Old Kingdom, the niche began to be extended into the mass of the superstructure to create a narrow chamber which served as the chapel for the cult. Later, the number of chambers multiplied until, by around 2300 BC, they sometimes occupied the entire superstructure. The eastern face of the *mastaba* of Nefermaat and Itet contained two separate chapels, the southern for the husband and the northern for the wife. Both are among the earliest surviving examples of tomb chapels containing internal decoration, here executed on the limestone slabs that lined the mudbrick walls.

In each chapel (as in those of another high-ranking pair, Rahotep and Nofret, at the same site) the western wall contained the false door, the portal through which the deceased could enter the chapel in spirit to partake of offerings which priests or relatives would present there to sustain the person in the afterlife. A stela with a figure of the deceased seated before a table of offerings was incorporated into this wall. The other walls of the chapels and their façades were covered with images belonging to the genre now known as ‘scenes of everyday life’. They represent chiefly the procuring of foodstuffs – hunting desert animals, catching birds and fish, butchering cattle, ploughing – and also depictions of the deceased with family members and rows of servants representing estates from which they were entitled to profit. These scenes reflected the earthly life that Nefermaat and Itet would have known, confirming their high status as master and mistress of productive land and servants, while also magically providing them with an eternal supply of sustenance, a safeguard against the possible failure of the offering cult. These scenes were repeated and amplified in thousands of elite tombs throughout the next two millennia. The chapels of Nefermaat and Itet are historically important as they contain the earliest surviving examples of such scenes found in their original context [2; p. 55]. They were, however, accessible only for a short time, as the tomb underwent several phases of construction, and before its final completion the decorated chapels had been sealed and hidden as the *mastaba* was extended, while new cult places were constructed against the external façade [2; pp. 38–41; pp. 43–44].

Several different methods were employed at Meidum to create the images on the tomb-chapel walls. In the chapels of Rahotep and Nofret the scenes were carved in relief and painted. In Itet’s chapel some surfaces were decorated in paint alone; two fragments of this decoration from her chapel are held by the British Museum (EA 69014 and 69015) and have been the subject of a previous article [3]. But in other parts of the chapel of Itet, and in that of Nefermaat, the craftsmen took the unusual step of creating images by applying coloured pastes into cells cut into the limestone.

Fine details were then added either by painting onto the surface of the paste, or by inlaying a second layer of differently coloured pastes into the first. Unusually, an inscription in the chapel of Itet makes a direct reference to Nefermaat’s paste-inlays, stating: ‘He is one who fashions his representations in writing that cannot be erased’ [2; p. 84; p. 167]. This suggests that the paste-inlay technique was an innovation at the time, and that it had perhaps been selected at Nefermaat’s personal instigation. No other examples of this technique have ever been identified in an ancient Egyptian tomb, and for this reason it is often supposed that it was abandoned at an early date, having been found to be unsuccessful. However, the paste-inlays were largely intact at the time of the tomb’s investigation by Auguste Mariette in 1872, and their subsequent deterioration may be attributed to poor handling after excavation rather than to a fundamental technical weakness [2; p. 165]. Since the tomb chapels of Nefermaat and Itet were sealed shortly after their completion, there would have been little opportunity for craftsmen to assess the effectiveness of the technique, and it appears more likely that the practice was discontinued because it was, in evolutionary terms, a retrograde step, a labour-intensive and time-consuming process that yielded a result inferior to that of relief carving or flat painting [2; p. 38; p. 165].

THE INLAY BOX

This contribution discusses a box of pigmented inlays (EA 69384, Figure 1) from Nefermaat’s tomb chapel. The tomb was originally excavated by Auguste Mariette in 1872 and later re-examined by Flinders Petrie in 1890–1891 [2; pp. 10–16, 4]. When first excavated, the inlays seem to have been in good condition, but by the time of Petrie’s examination, the chapel of Nefermaat had suffered considerable damage and many of the inlays had been lost, perhaps due to the effects of temperature changes or to chemical deterioration of the inlays themselves. Petrie collected a number of samples of the remaining inlays. Although these can now be found scattered across museums worldwide, we know of no attempts to use modern analysis to examine the inlays themselves, some work having been carried out on the plasters between 1986 and 1988 by the Polish-Egyptian Restoration Mission [5]. The pigmented inlays in the British Museum box (EA 69384) discussed below were purchased from Petrie by the Victoria and Albert Museum in 1891, before being transferred to the collections of the British Museum in 1982. There was considerable interest in the nature of the pigments used in ancient Egypt even at the time of Petrie’s investigations, and the paints in both the wall paintings and the inlays from this tomb were chemically analysed by Petrie’s associate, Flaxman Spurrell [4; p. 29; p. 50], with notable success, as will be discussed below. The results of Spurrell’s examinations are included in the descriptions of the inlays written inside the British Museum box, Figure 1.



FIGURE 1. The British Museum box of inlays from the *mastaba* of Nefermaat (EA 69384)

INVESTIGATION OF THE INLAYS

Macroscopic examination

The box contains 12 pieces of coloured inlay mounted in six compartments divided by colour, Figure 1. Hand written inscriptions describe these as ‘yellow’, ‘olive-yellow’, ‘green’, ‘red’ and ‘black’, ‘red-brown’ and ‘red’, although in some cases, two colour layers are present (see the discussion of the pigments below). The sizes of the pieces vary considerably from around 60 × 70 mm to 5 mm square. There are two basic thicknesses (c.5 and c.10 mm). No blue is present in this box, and Petrie found no blue inlays, although he did record the presence of blue in the conventional paintings of Itet’s tomb. Unfortunately, no blue is present on the painted fragments from Itet’s chapel held by the British Museum, so it has not yet been possible to undertake direct analysis, although it is hoped to source such material in the future.

Microscopic examination

Small samples taken from the blocks and subjected to microscopic examination show the inlays to be brittle

masses of coloured crystals. All are mixed with a high proportion of small (0.1–1.0 mm) brownish, translucent globules, apparently of an organic material; Figure 2 shows a section through the ‘green’ block. Spurrell had a number of theories to account for the globular nature of the binder, including the accumulation of organic ‘polish’ into air bubble spaces at the surface of the inlay and segregation of binder when mixed with the paste. By whatever means it was incorporated into the mass, this organic matter seems to be the principal binder holding the pastes together. In some of the blocks, crystals of calcium sulphate are clearly visible and probably aid in consolidation, but others (such as the ‘brick red’ block) seem to consist almost entirely of pigment grains, which would have little inherent integrity. The further analysis carried out on the various constituents of the blocks is described below.

Pigment content

The pigment content of the pieces was investigated using Raman spectroscopy, X-ray fluorescence (XRF) analysis and optical microscopy; experimental details are given in the experimental appendix.



FIGURE 2. Cross-section of a sample from the 'green' block, clearly showing the mixture of organic and inorganic materials

The inorganic fraction of the 'yellow' block was found to consist of reasonably pure 'jarosite' (strictly a mixture of jarosite and natrojarosite). Virtually no calcium sulphate was found to be present and the coherence of the block was presumably maintained by its organic component. The jarosite group consists of naturally occurring minerals formed by the alteration of iron oxides or pyrites. The group includes jarosite itself (hydrated potassium iron(III) sulphate $KFe_3(SO_4)_2(OH)_6$), natrojarosite ($NaFe_3(SO_4)_2(OH)_6$) and a number of other variants, although only jarosite and natrojarosite seem to have been recorded in ancient Egyptian contexts. Jarosite was first recorded as an Egyptian pigment on decorated pottery of the Eleventh Dynasty from El Tarif, but it was later suggested that the jarosite found in Egypt was not used as a pigment in its own right, but was instead a degradation product, present as the result of the chemical decomposition of an iron-rich frit pigment, originally red-brown or green in colour [6]. However, jarosite has since been found in a large number of other contexts from the Old Kingdom onwards (including on the conventional wall paintings from Itet's tomb chapel [3]) and strong arguments have been made against this interpretation based on chemical, historic and stylistic grounds [3, 7, 8]. On this basis jarosite can now be accepted as one of the three yellows which form part of the known Egyptian palette (these are yellow ochre, the most common, jarosite and orpiment (As_2S_3), the last apparently being a late addition, first appearing in the Middle Kingdom period). The 'olive-yellow' block is similar to the 'yellow' but contains a mixture of jarosite, calcium sulphate (present largely as anhydrite) and black carbon particles, presumably added to the mix to temper the colour. The carbon particles were sufficiently large and well separated that at high magnification some cellular structure could be observed, confirming the source to be crushed charcoal, rather than lamp black (soot). These yellows are the only material where Spurrell's early identifications can be questioned; he defines them as 'ochreous clays', but misses the potassium/sodium and sulphur substi-

tutions, something for which he can hardly be faulted given the methods available to him.

The 'green' pigment block is bicoloured, with a malachite-containing layer overlying one of jarosite similar to the material in the 'yellow' block. This layering may relate to construction methods – Petrie refers to the production of complex features, such as feathers, by cutting into a base inlay and refilling with a different colour [4; p. 25] – or to economy; as discussed below it is likely that malachite was an expensive pigment. This use of malachite is the one point where this set of pigments differs from the conventional Egyptian palette. The nature of the greens used in ancient Egyptian art has long been a subject of debate. It now seems clear that green frit (Egyptian green, a glassy pigment consisting chiefly of copper wollastonite) was the main, perhaps only, green pigment in use from the First Intermediate period onwards [9], but prior to this the picture is less clear. It is certain that, with the exception of this sample, there are very few reliable identifications of malachite used as a true paint, although it can occur as a degradation product of the two frit colours, Egyptian blue and green frit. In addition, the most comprehensive survey of Egyptian pigments undertaken in modern times (by the Max Planck Institute) found no malachite [10]. In contrast, it is known that malachite was widely used as a cosmetic throughout the dynastic period. One possible explanation for this differentiation is that malachite was too expensive for widespread use as a pigment, as may be suggested by its use only as the top coating in this block. Its appearance in this very early context may well relate to the lack of availability of alternatives; the earliest known appearance of frit colours is the use of Egyptian blue in the Fourth Dynasty. Green also occurs in the conventional paintings of Itet's tomb chapel but this has so far proved impossible to identify, given the constraints of permissible sample size [3], but it is hoped that this will be revisited in future work.

The 'black' block (more a dull grey) consists mainly of carbon combined with a mixture of calcite and anhydrite. The preponderance of black material made it difficult to distinguish individual particles, and it was not possible to determine if any came from crushed charcoal as in the 'olive-yellow' material. Again carbon is by far the most common black pigment of the Egyptian palette, although a few instances of the use of manganese blacks have been recorded [11].

Finally, three blocks of red material are included in the box, probably reflecting the predilection for the use of red that occurs in tomb paintings throughout the dynastic period. Of these, the one termed 'red-brown' consists largely of hematite ($\alpha-Fe_2O_3$) of varying degrees of crystallinity, with a small proportion of quartz, and is clearly a block of virtually pure red ochre. The use of red ochre is in keeping with the other evidence from other Egyptian contexts, where it is by far the most commonly reported red pigment [1]. In the other two blocks the red colour is lighter due to hematite crystals sparsely distributed in a white calcium sulphate (mixed gypsum and anhydrite)

matrix. While one of the blocks is of pure red, the other is supported on a white block of gypsum/anhydrite. As with the green material discussed above, this may relate either to production methods or to economy, but given the ready availability of red ochre throughout Egypt, technique seems to be the most likely explanation in this case.

Organic content

The inlays are bound, at least in part, with an organic medium visible as yellow/brown translucent globules described above and seen in Figure 2. Spurrell’s investigations of these inclusions (solubility tests and burning) concluded that they were a resinous material, closest to ‘mastic’ [4; p. 29; p. 50]. Spurrell also attributed apparent hardness, darkening and, in some cases, lamination at the surface of the inlays to the application of an organic ‘polish’, such as oil or turpentine, or to the addition of an animal matter, possibly gelatine. However, apart from these physical observations it is unclear on what evidence these identifications were based.

The composition of the organic components is now being reinvestigated using gas chromatography-mass spectrometry (GC-MS) and pyrolysis-GC-MS (py-GC-MS). Preliminary results from the globular inclusions indicate that they are composed of plant-derived oil rather than resin. Analysis of solvent extracts by GC-MS has shown the presence of fatty acids and diacids that are typical of degraded oils [12, 13], although a substantial fraction of the globular organic inclusions proved to be insoluble. Subsequent analysis by py-GC-MS yielded mostly straight-chain (alkane/alkene) pyrolysis products, which are consistent with a polymerized lipid [14]; this apparently extensive polymerization implies the use of a drying oil. Linseed is the only source of drying oil known to have been available in ancient Egypt although a number of semi-drying types, such as safflower or poppy oil, may also have been available [15]. The presence of such a medium is potentially extremely significant as the use of oil binders in dynastic Egyptian painting is virtually unknown.

CONCLUSIONS

This box of inlays represents a very different form of decoration from the tempera painting generally found in Egyptian tombs. In terms of the pigment component, the most unusual feature is the definite presence of malachite. It is also noticeable that no blues were recorded in any of the inlayed blocks found in the tomb, and features, such as hieroglyphs, which would be expected to be shown in blue in later convention are here filled with green. The blue that occurs in the conventional paintings in Itet’s tomb was described by Spurrell as a “blue verditer tint, apparently derived from an impure earthy blue carbonate of copper”; this is presum-

ably azurite – he uses the term chessylite, an early synonym. Spurrell also clearly states that “no traces of green or blue frits have been found” in either of the tombs. Admittedly he had only limited testing facilities available to him, but the analytical work described here has confirmed the remarkable accuracy of his work, and this comment shows that he was specifically searching for evidence of frit colours. It could be that these two tomb chapels reflect decoration produced before the frits that so dominate the conventional Egyptian palette became available, thus enforcing the use of natural carbonates. Given the extravagant use made of pigments in the coloured blocks – the amount of raw material used in any one of the blocks in this box would have covered a substantial area of wall if used as a simple paint – it is unlikely that cost was a factor.

The findings for the organic fractions are even more unexpected and further work on this material and on other pieces from the tomb is now underway in an attempt to establish the type of oil and to explore the method of incorporation into the inlays. It is hoped that the globular nature of the particles may prove a useful clue as to the processes involved. The use of such an oil binder is unprecedented for such ancient material and may demonstrate a previously unsuspected technology.

EXPERIMENTAL APPENDIX

Analysis of the inorganic materials was carried out mainly by Raman spectroscopy, using a Jobin Yvon Infinity spectrometer with green (532 nm) and near infrared (785 nm) lasers, with maximum powers of 2.4 and 4 mW at the sample respectively. The spectra were identified by comparison with the British Museum in-house reference database and with published data. These results were confirmed by the elemental data produced using a Bruker Artax XRF

TABLE 1. Analytical results for the inorganic fractions of the blocks

Description	Visual appearance	Microscopic appearance	Identification
Yellow	pale yellow	yellow crystals	jarosite
Olive-yellow	darker yellow	yellow crystals with sparse white and black crystals	jarosite anhydrite carbon (charcoal)
Green	bright green mass on yellow support	green crystals yellow crystals	malachite jarosite
Black	dark grey	black particles in yellow and white mass	carbon calcite anhydrite
Red-brown	red	fine red crystals	hematite
Red 1	red on white support	sparse red crystals in white mass white crystals	hematite gypsum/anhydrite gypsum/anhydrite
Red 2	red	sparse red crystals in white mass	hematite gypsum/anhydrite

spectrometer [16]. All the analyses were carried out directly on the surface of the samples without any preparatory cleaning. The two sets of results were in full accord, and the resultant identifications are given in Table 1.

Individual organic binder inclusions were removed with tweezers from the broken edges of the inlays and crushed to fine powder in conical microvials. These were solvent extracted with 100 μ L of dichloromethane, transferred to fresh vials, then dried under nitrogen and, prior to analysis, derivatized using bis(trimethylsilyl)trifluoroacetamide (BSTFA) + 1% trimethylchlorosilane (TMCS) to form trimethylsilyl derivatives. Additional unextracted samples were taken for direct analysis by py-GC-MS. The samples were analysed using an Agilent 6890N gas chromatograph coupled to an Agilent 5973N mass spectrometer. Solvent-extracted samples were injected in on-column mode with the inlet temperature programmed to follow the oven cycle. Pyrolysis of unextracted samples was undertaken using a CDS Pyroprobe 1000 with a probe temperature of 750°C (15 seconds) and interface temperature of 280°C. These were introduced to the GC in split (10:1) mode at 280°C. All the analyses used an Agilent HP5-MS, 30 m \times 0.25 mm column with a film thickness of 0.25 μ m, fitted with 1 m \times 0.53 mm retention gap. The carrier gas was helium in constant flow mode at 1.5 mL per minute. After a one minute isothermal hold at 50°C, the oven was temperature programmed to 330°C at 10°C per minute with the final temperature held for 15 minutes. The MS interface temperature was 280°C. Acquisition was in scan mode (50–600 amu per second) after a solvent delay of five minutes. Mass spectral data were interpreted manually with the aid of the NIST/EPA/NIH Mass Spectral Library version 2.0 and comparison with published data.

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