

Chapter 5

Egyptian Eye Cosmetics (“Kohls”): Past and Present

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Abstract

The published literature was summarised and reviewed for historical/archaeological data on the usage and chemical composition of Egyptian eye cosmetics (“kohls”). A total of 27 kohl samples were purchased in modern-day Egypt; 18 in Cairo, 4 in Aswan and 5 in Luxor. Also, very small amounts of material were carefully removed from inside six Pharaonic kohl pots (held in the Royal Albert Memorial Museum and Art Gallery, Exeter, UK). These pots were dated to either Middle or New Kingdom (*i.e.* between c. 2040 BC to c. 1070 BC). Each of the 33 samples was analysed by one or more of the following techniques: X-ray powder diffraction (XRPD), low vacuum scanning electron microscopy (LV SEM), infrared spectroscopy (IR) and the relatively new technique of quantitative scanning electron microscopy (QEMSCAN). For the 27 modern-day samples, it was found that the main component for six was galena (PbS) and for the remaining samples one of the following: amorphous carbon (6), calcite (CaCO₃) (6), elemental silicon (1), talc (Mg₃Si₄O₁₀(OH)₂) (2), cuprite (Cu₂O) (1), goethite (FeO(OH)) (1), barite (BaSO₄) (1), halite (NaCl) (1), and for two samples, an unknown amorphous organic compound. Three of the galena-based samples also had their (average) particle sizes determined from electron micrographs. Five of the six Pharaonic kohl pots contained lead-based compounds (one being empty of kohl). Two were black and so were most likely galena, whilst the other three were white and so were one or more of several possible “made” or natural lead compounds. The composition of the six Pharaonic kohl pots was also studied (only using the LV SEM and QEMSCAN analytical techniques) and the results compared to the compositions expected from previous visual inspections. Whilst only about a quarter (22%) of the modern-day Egyptian kohl samples contained a lead compound as a major component; it was seen, both from our results and those reviewed, that in the Pharaonic past this percentage was much higher.

Keywords: Pharaonic Egypt, eye cosmetics, kohl, X-ray powder diffraction, low vacuum scanning electron microscopy, quantitative scanning electron microscopy.

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1. INTRODUCTION

In ancient Egypt, even sacrificial cows had eye make-up applied before their ritual slaughter. A relief in the fifth dynasty (*i.e.* c. 2400 BC) temple of King Sahure shows such cows being tended to by female temple personnel [1]. As for the details of the usage of eye make-up/eye-paint (“kohl”) by the human population of ancient Egypt, there is sometimes a degree of uncertainty with respect to the dates of usage, exact original composition of the kohls and their reason(s) for usage (*i.e.* if cosmetic and/or medicinal and/or magico-religious and if used as cosmetics, were they used on the eye and/or face). However, all the authors agree that there was green eye-paint initially and “a little later” black eye-paint and that it was used for one or more of the above reasons by men, women and children from all social levels.

The green eye-paint was usually the ore malachite, basic copper carbonate, which was mined in Sinai and the Eastern desert. It has been found in Badarian/pre-dynastic (*i.e.* 5000–3000 BC) tombs (Ref. [2]; an unusual example, involving the burial of an elephant) and until recently was assumed to have been used until at least the nineteenth dynasty (*i.e.* c. 1250 BC) [3,4]. However, a more recent publication [5] states that the green (malachite) eye-paint “... seems to have been used only until the middle of the old kingdom, when it was replaced by the black galena-based form ...”. The middle of the old kingdom was c. 2500 BC.

Black eye-paint has been found at least once in a Badarian period tomb, occurs more often in later tombs and continued to be in use until the Coptic period (*i.e.* c. 395–640 AD). The term “msdmt”/“mesdemet” was used for eye-paint in general and for black eye-paint in particular; whereas the green eye-paint was referred to by the term “wadju”/“udju”/“ouadjou”. Results of many analyses of tomb funerary gifts of black/grey-black eye-paint show that often the main component was the ore galena (lead sulphide). This ore was mined in several localities in Upper Egypt. Sometimes, malachite and galena were also imported, often from Arabia *via* “Punt” (which was probably the present-day Eritrea/Somalia). Both eye-paints occur, either individually or together, in several of the listed (papyri) therapeutic “recipes” used as eye salves by the ancient Egyptians [6]. For those readers interested in

the details of ancient Egyptian medicine, which is beyond the scope of this chapter, there are several books/reviews/articles etc. available in the published literature [7–9]. Also, for an overview of eye diseases in ancient Egypt, see Ref. [10].

“Cleopatra experimented with eye colours to effect, using black galena on the eyelid and to delineate her brows, and painting the upper lids deep blue, and the lower lids bright green” [11]

There are “statements”, such as the one above, in the published literature of Cleopatra’s usage of a *blue* eye cosmetic. Sometimes it is merely stated that the blue eye cosmetic was used by (Pharaonic) “Egyptian ladies” and sometimes, additionally, that it was lapis (lazuli) [12]. No source reference(s) for any of these statements have ever been seen by these authors.

Cleopatra VII (69–30 BC) may well have used blue eye cosmetic to entrance Mark Antony, but stating that she used lapis lazuli for this effect is pure speculation. Lapis lazuli was used for small objects (*e.g.* beads, amulets, scarabs, etc.) from predynastic times in Egypt and most probably it came from the remote mines of Badakshan (north-east Afghanistan). It was also used for inlay jewellery from predynastic times. The inlaid eyelids and eyebrows of Tutankhamun’s famous gold mask are made of lapis lazuli. However, such usage should not be taken to mean that the material was used as an ingredient for eye cosmetics. It was also used as a paint pigment (in ultramarine), but much later (*c.* eleventh century AD) [3]. It is also occasionally listed in (papyrus) recipes for eye diseases/infections [6].

We have come across only a few publications in English which give data on the numbers, provenance (*i.e.* data on the date/period and name of the site of excavation) and compositions from chemical analytical studies, of the different eye-paint/makeup/cosmetics found in ancient Egyptian tombs. One such reference (Ref. [3]) summarises 74 such samples, from analytical work done by both himself (Lucas) and several prior authors. Some analytical results (*i.e.* those of Ref. [13]) were deliberately excluded by these authors for a variety of reasons (such as: missing dates and origins, and missing numbers of the types of particular samples studied). Also, whilst 73 of the 74 samples summarised were free from resin, some of the excluded samples did contain such material and so were regarded by these authors as not being cosmetic samples. Wax and/or fatty matter was found in a few of the 74 summarised samples and these were regarded as probably being cosmetics of some sort.

The above 73 results are given in Table 1. We have excluded, from the original 74 summarised samples, the one sample whose analysis was listed as “uncertain”. It can be seen from this table that the majority of the grey/grey-black/ black samples are galena-based; that is 44 of 58, *i.e.* 76%. Also, the only antimony (tri) sulphide-based (*i.e.* the ore stibnite as the main component) sample found was dated to the nineteenth dynasty, whilst the 44 galena-based samples had a wide variety of dates. Thus Table 1 represents a summary of the analyses on cosmetics (assumed by us to be mostly kohls/eye-paints) samples carried out (by a variety of authors) from the 1880s to the early 1960s; where the analyses were mostly performed using “wet chemistry” analytical techniques.

In contrast, using modern analytical (*i.e.* synchrotron, spectroscopic) equipment, it has recently been shown by Louvre-based researchers [14–17] that several *synthesised* lead compounds (phosgenite ($\text{Pb}_2\text{Cl}_2\text{CO}_3$) and laurionite (PbOHCl)) were deliberately made and then added to usually galena-based cosmetics for either their (supposed) therapeutic

Table 1. Composition of eye-paints (“kohls”) from ancient Egypt (1)^a

Number	Main component	Additional components
44	Galena (PbS)	1 with Gypsum (CaSO ₄ .2H ₂ O); 5 with Carbon; 2 with trace of Antimony Sulphide (Sb ₂ S ₃)
2	Carbonate of lead (PbCO ₃)	1 with trace of Sb ₂ S ₃
6	Brown Ochre (Fe ₂ O ₃ .nH ₂ O)	None
1	Limonite (Iron oxide/hydroxide mixture)	None
2	Magnetic oxide of iron (Fe ₃ O ₄)	1 mixed with earthy matter
10	Oxide of manganese (MnO ₂)	None
1	Black oxide of copper (CuO)	None
1	Sulphide of antimony (Sb ₂ S ₃)	None
5	Malachite (Cu(OH) ₂ .CuCO ₃)	1 mixed with resin
1	Chrysocolla (CuSiO ₃ .2H ₂ O ^b)	None

^a From Ref. [3].

^b One formula for this mineral, others exist.

effects or possibly to give varying shades of colour to them for use as eye/face cosmetics. The latter effect was also achieved by adding a (white) available lead ore – cerussite (lead carbonate). It is thought that these light grey or white mixtures may have been used as face cosmetics or even as foundation creams. Some of their analytical results are summarised in Table 2. In this table we have included only those published results where both complete (*i.e.* the percentages quoted do sum to 100%) compositional and time period data have been given for the samples studied. Many of these results show that “fatty acids of animal provenance” *are* also present and hence there is some discussion as to their exact usage by the ancient Egyptians (see below). These “made additions” appear to have been initiated at around 2000 BC and to have continued to at least c. 1200 BC.

As for any analyses of ancient Egyptian kohls from the early 1960s (*i.e.* after the analyses summarised by Lucas and Harris [3]) to the present (but apart from the above mentioned Louvre-based work) – we have found no publications describing any such analyses. However, there have been several papers published where kohls were one of several lead-based ancient Egyptian artefacts subjected to lead isotopic analysis (LIA). All the kohls (29 samples) so studied contained lead to varying degrees and all are stated to contain galena. Seventeen kohls (1 lump and 16 powders, variously dated from the sixth to eighteenth dynasties) were studied by Brill *et al.* [18]; eleven (mostly powders, and all pre-/protodynasty dated) by Stos-Gale and Gale [19] and one (“clots of galena” found at their excavation site and radiocarbon dated to 3080 ± 110 BC) by Hassan and Hassan [20]. No details of any chemical analyses undertaken on the kohl samples are given in any of these three publications. As almost all the above samples were obtained from UK museums,

Table 2. Composition of eye-makeup ("kohls") from ancient Egypt (2)^a

Sample number	Pharaonic period	Major component (%)	Minor components (%)	Reference
N811d	New Kingdom	Galena (PbS) (75)	Cerussite (PbCO ₃) (14); Phosgenite (Pb ₂ Cl ₂ CO ₃) (11)	[14]
N811g	New Kingdom	Galena (75)	Cerussite (14); Phosgenite (11)	[14]
N1332	New Kingdom	Galena (89)	Phosgenite (6); Cerussite (3); Laurionite (PbOHCl) (2)	[15]
N1367d	New Kingdom	Galena (100)	None	[15]
N1367g	New Kingdom	Galena (100)	None	[15]
AF167	New Kingdom	Galena (100)	None	[16] ^b
AF6772	New Kingdom	Galena (62)	Cerussite (28); Laurionite (10)	[16] ^b
E11047	New Kingdom	Galena (41)	Cerussite (41); Phosgenite (18)	[14]
E11048b	New Kingdom	Galena (50)	Phosgenite (37); Cerussite (13)	[16] ^b
E11048c	New Kingdom	Cerussite (42)	Phosgenite (29); Galena (20); Anglesite (PbSO ₄) (9)	[15]
E11048d	New Kingdom	Cerussite (47)	Galena (28); Phosgenite (25)	[15]
E11048e	New Kingdom	Galena (40)	Phosgenite (28); Cerussite (26); Laurionite (3); Anglesite (3)	[15]
E14455	New Kingdom	Galena (70)	Zinc-based cpds. (20); Anglesite (10)	[14]

Continued

Table 2. Continued

Sample number	Pharaonic period	Major component (%)	Minor components (%)	Reference
E14569	New Kingdom	Laurionite (35)	Cerussite (25); Galena (24); Phosgenite (16)	[16] ^b
E20514	New Kingdom	Galena (72)	Phosgenite (9); Sphalerite (ZnS) (9); Anglesite (4); Cerussite (3); Smithsonite (ZnCO ₃) (2); Laurionite (1)	[17] ^b ; [15]
E21562	Middle or New Kingdom	Phosgenite (32)	Cerussite (25); Laurionite (19); Galena (15); Anglesite (9);	[14]
N1209	Middle Kingdom	Galena (44)	Anglesite (21); Phosgenite (10); Cerussite (13); Laurionite (12)	[15]
E23100	Middle Kingdom	Galena (58)	Anglesite (19); Cerussite (19); Laurionite (4)	[14]
E23105	Middle Kingdom	Galena (44)	Quartz (SiO ₂) (38); Calcite (CaCO ₃) (18)	[14]

^a Based on various published papers, all of which were supported by L'Oreal Research and the Louvre Museum, France.

^b The references where the finding of "fatty acids" in the samples is mentioned.

it is possible that some have been previously analysed and are amongst those summarised by Lucas and Harris [3].

Both ancient Egyptian eye-paints, green and black, were initially ground to a powder and then applied dry or were mixed with water or a water-soluble gum to give a paste which was then applied with a finger or, at later dates, by means of a kohl-stick. This stick was made of stone (*e.g.* black haematite), bone, wood or ivory. It is still a matter of some discussion if fatty-matter/oil/wax, or even possibly resin, was separately used for applying the kohl or if the presence of this additional material, when actually within the ancient funerary containers, meant that it was another form of cosmetic (*i.e.* for the face, the fats etc. giving good coverage/spreadability) or if it was to be used as an eye salve in the after-life.

In the "Sayings of the Prophet" (PBUH) Muslims are advised to use kohl made from "ithmid" (or sometimes, "ethmid"/"athmid"). In Hadith number 1, it is stated " ... 'Use kuhl made from ithmid on the eye; it brightens eyesight and strengthens the growth of the eye lashes' ... " (www.ummah.net/moa-on-line/hadith/shumaail/st7.html). "Ithmid" was described as being a dark, reddish-black, shiny stone and has always been assumed to be the ore stibnite. Possibly, over time, the ore galena was substituted; as galena was not as uncommon, looked and felt much the same and was much less expensive to obtain (also see in Section 4.1).

Some of the early Muslim medical authors made a clear distinction between two types of eye powders/pastes: "siyaf" powders and pastes which were for cosmetic purposes and which were usually made from soot (*i.e.* amorphous carbon), whilst "kuhl" (kohl) powders and pastes were used to treat various conditions and diseases of the eye. The latter were, and still are, made in southern Oman from the charcoal obtained from the root of the local "ra" (*Aerva javanica*) plant, plus small amounts of mother-of-pearl and frankincense and, most importantly, some antimony/lead sulphide (*i.e.* the "ithmid"; this being perceived as being the "active ingredient") [21].

This distinction, of two types of eye powders/pastes, was still being observed in Egypt of the 1820s and 1830s. It was reported that cosmetic kohl was obtained from burning an aromatic resin ("liban", a species of frankincense, see later) or shells of almonds; but that kohl with medicinal properties was made by mixing galena powder with Sarcocolla (mother-of-pearl), long pepper (a particularly potent variety of pepper), sugar candy, fine dust of Venetian sequin (an old gold coin), and sometimes powdered pearl [20,22].

In modern-day Egypt, kohls are used only by females, and their children, from all socio-economic classes, for both beautification and as an ethnic remedy, *i.e.* to relieve eyestrain, pain or soreness. Anecdotal evidence (Hardy; personal communications, 2001) suggests that there is more use of kohl in rural than urban areas of Egypt, and that middle/upper class ladies in urban areas such as Cairo are now-a-days more likely to use the modern eye pencils for reasons of convenience of purchase, less messy in usage and because they are "known to be good". Also, "traditional" kohls are perceived as "old fashioned", especially by the younger student generation.

One "traditional" recipe for making home-made kohl in modern-day Cairo is (Hardy; personal communications, 2001) (extra data has sometimes been added, in *italics*): " ... add olive oil to "leban dakar" (*this is the Egyptian vernacular name for gum-resin in general and for frankincense in particular; see above for its usage in Egypt of the 1800s*) until it is covered (in a saucer). Leave for 2 days and then transfer the contents to a plate. Add several small pieces of cotton and then light them; as it burns, place a pottery bowl over it to collect the

smoke residue (*soot*). Leave for an hour. Remove the smoke residue from the bowl with a feather and place in a kohl jar". It is then applied daily using an applicator stick, which is usually made of glass or plastic. Sometimes olive oil is used for ease of actual application of the kohl. This "recipe" can be compared to another reported method of preparing home-made kohl in modern-day Egypt [23]: "... burn the following ingredients together – sumach (*a shrub with green flowers and red hairy fruits*), nutmeg tree, extracts from previously burnt frankincense, sugar crystals and perfumed cherry". The resulting ash is then used as the kohl. Both of these "recipes" give essentially the same end product – innocuous amorphous carbon.

2. MATERIALS AND METHODS

2.1. Modern-day samples

A total of 18 samples were obtained from the city markets (souks) of Cairo. The cost of these kohls varied between 2.5 and 10 Egyptian pounds, where one GBP was then 5.5 Egyptian pounds. Later, nine additional samples of mostly *coloured* kohls were purchased in the souks of Aswan (4 samples) and Luxor (5 samples). Their price varied between 1 and 10 Egyptian pounds. Because of the brief duration of these later visits to Aswan and Luxor, it was decided beforehand to look mostly for and purchase "unusual" (*i.e.* coloured) kohl samples. All the (few) available green, blue or red samples seen were purchased. Figure 1 shows how these 27 kohls are distributed by the countries where they were made.

All these kohl samples were examined by the analytical techniques of X-ray powder diffraction (XRPD) and scanning electron microscopy (SEM) with an attached energy dispersive X-ray microanalyser. Additionally, ten of the (artificially) coloured samples were examined by infrared (IR) spectroscopy.

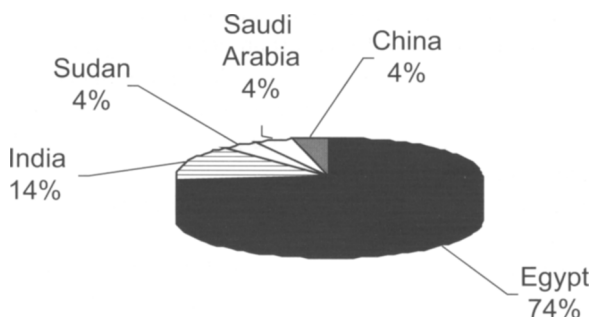


Fig. 1. Origins of modern-day Egyptian kohl samples studied.

2.1.1. X-ray diffraction

The kohl samples were, where necessary, ground to a powder and then mounted in an aluminium holder. The Cairo samples were analysed using a Bruker AXS D8 Advance diffractometer. Diffraction data was collected with generator settings of 40 kV and 40 mA. A monochromator and automatic divergence slits were used with the $\text{CuK}\alpha$ X-ray radiation. One data scan was performed for each sample, using a step scan of size 0.02° and a time of 2 s/step over a $2\text{-}\theta$ range of $2\text{--}80^\circ$. The Aswan and Luxor (powder) samples were analysed using a Siemens D5000 diffractometer, using $\text{CuK}\alpha$ X-ray radiation. One data scan was performed for each sample, using a step scan size of 0.0256° and a time of 1 s/step over a $2\text{-}\theta$ range of $8\text{--}70^\circ$.

These scans were used to determine the major and minor components (phases) present in the samples by comparing the obtained data to the reference data in the 2000 JCPDS (Joint Committee for Powder Diffraction Standards) database [24]. The major phase was defined to be that phase which had a presence of $>80\%$ of the sample. Semi-quantitative results were obtained using this analytical method; any percentages quoted are estimates only. The minor phases given in brackets in Tables 3 and 4 are those estimated to be $\leq 5\%$ of the sample. Also, each group of minor phases are listed in decreasing order of their estimated percentage presence.

2.1.2. X-ray microanalyser

Each of the kohl (powder) samples was mounted on an aluminium stub using an adhesive carbon tab. They were then examined in a JEOL JSM 5300 LV SEM with a Rontec energy dispersive X-ray (EDX) microanalyser attached.

The LV (low vacuum) SEM is designed so that its electron gun and electron optical system are kept under high vacuum (typically about a millionth of a torr), whilst the specimen chamber is differentially evacuated to low vacuum (typically a few torr) by another pumping system. The gas molecules surrounding the electron beam are ionised and the electric charge on the specimen is neutralised, thus allowing non-conductive specimens to be studied without coating.

Elements lighter than carbon (*i.e.* $Z \leq 5$) cannot be detected using the above equipment. Detection was qualitative and the element peaks that were only just above the background are given in brackets in Tables 3 and 4. Three Cairo samples (nos. 1, 3 and 6 in Table 3) were imaged at various magnifications in back-scattered mode, so that an estimate could be made of the average particle size of the galena cubes in each sample.

2.1.3. Infrared spectroscopy

Infrared spectra were collected on ten of the kohl samples which were artificially coloured, *i.e.* those coloured with a small amount of a (probably synthetic) colourant and not from the colour of the major phase (or even one of the listed minor phases). These were the 7 (of the 9, that is all of them except L4 and L5) samples from Aswan and Luxor (see Table 4) and three samples from Cairo (nos. 12, 13 and 14 in Table 3). The spectra were collected using a Perkin Elmer IR spectrometer, with each sample made into pellet form after mixing with potassium bromide.

2.2. Pharaonic samples

Recently six Pharaonic kohl pots became available for study by us. Small amounts (“scrapings”) were carefully removed from all six pots, both from the inside (for the kohl; sample nos. M1 to M6) and the outside (for pot material; sample nos. MP1 to MP6). Table 6 summarises the data (*e.g.* Museum catalogue numbers, approximate age, possible provenance, height, texture, colour and initial analytical results) on the pots and their contents. Figure 4 shows all six pots with a centimetre scale (with a colour picture of them on the book’s front cover).

All the pots belong to the Royal Albert Memorial Museum (Exeter, UK) and the Museum’s knowledge of their provenance etc. is unfortunately limited by the paucity of the data provided by the original donors. However, we have been able to add a little more information (Morkot; personal communication, 2005; see Table 6). Namely that pots MP1, MP2, MP4 and MP5 are probably of Middle Kingdom date, whilst MP3 is probably of New Kingdom date. Pot MP6 can be dated with near certainty to the Middle Kingdom because of its obvious “Blue marble” composition. Also, the compositions of MP4 and MP5 can also be stated, with near certainty, to be “Egyptian alabaster”. However, the listed compositions for MP1, MP2 (both as travertine) and for MP3 (as “Egyptian ceramic”) are tentative. Pots MP1, MP2 and MP3 are part of the Montague collection in the Museum, whilst pots MP4, MP5 and MP6 are supposed to have been loaned to them from the Petrie Museum of Egyptian Archaeology (London, UK) some decades ago.

The samples were examined by the analytical techniques of SEM with an attached energy dispersive X-ray microanalyser and by automated scanning electron microscopy with linked energy dispersive spectrometers (QEMSCAN).

2.2.1. X-ray microanalyser

Nine of the twelve Pharaonic samples were mounted on individual aluminium stubs using adhesive carbon tabs. They were then examined in a JEOL JSM 5300 LV SEM with a Rontec energy dispersive X-ray (EDX) microanalyser attached, as for the modern-day samples above.

Element detection was both qualitative and quantitative and the elements found are given, in decreasing order of their weight percent, in Table 6. The elements that were at less than 1% each are given in brackets. As the amounts of samples MP1, MP2 and MP3 were extremely limited, it was decided to do QEMSCAN analyses only on them (see below).

2.2.2. QEMSCAN (*quantitative scanning electron microscopy*)

QEMSCAN is an automated scanning electron microscope, which provides particle-by-particle quantitative mineral data [25–27].

The QEMSCAN operating system comprises a scanning electron microscope coupled with four energy dispersive spectrometers arranged at approximately 90° intervals around the sample chamber.

The particle mineralogical analysis (PMA) mode of operation was used for the analyses of these 12 samples. This mode of analysis is conducted where the particles are automatically located, using the contrast in backscatter coefficient (which is proportional to the

mean atomic mass of the material) between the particle and the mounting substrate (which was epoxy resin for all these samples). Once located, the electron beam is rastered across the particle at a user-defined stepping interval (pixel spacing). Here, for the pot contents (*i.e.* the kohls; sample nos. M1 to M6), two particle size fractions were analysed. Particles between 1 and 25 μm in size were analysed using a pixel spacing of 0.5 μm and particles between 25 and 200 μm in size were analysed using a pixel spacing of 2 μm (see Table 7). For the pot materials themselves (*i.e.* samples nos. MP1 to MP6), analysis was done on particles of between 25 and 200 μm in size at a pixel spacing of 2 μm (see Table 8).

At each pixel spacing, an X-ray energy spectrum was rapidly acquired and compared with a look-up table of known chemical composition minerals (the Species Identification Protocol (SIP)), then a mineral identification is made, and its weight percent is subsequently calculated. Individual identifications are typically made online in under 1 ms, which equates to in excess of 250 000 individual analyses points per hour (taking into account the time needed to move the sample stage and electron beam). All analytical measurements are stored, thus allowing the sample to be re-interpreted subsequently off-line.

As only small/very small amounts of material were available for QEMSCAN analysis, the individual samples were prepared as "sprinkle particle mounts". This involved collecting a tiny amount of sample on the end of a clean cocktail stick and dispersing the particles in a drop of methanol on a polished blank epoxy resin block. The methanol was allowed to flash off and the sample was coated with carbon to a thickness of 250 Å.

3. RESULTS

3.1. Modern-day samples

Table 3 lists the results obtained, for the 18 modern-day Cairo samples, in the order: (first) lead-based, amorphous carbon/carbon-based (in an amorphous organic compound), calcium-based, copper-based, iron-based and (last) silicon-based (for the main element of the major phase present) samples.

Six of these eighteen samples studied were lead-based; the four purchased as powders were black or grey-black in colour whilst the two lumps were both silver-grey in colour. The major phase was always found to be galena (PbS) and all the powders contained minor phases of anglesite (PbSO_4) and cerussite (PbCO_3). All except one of these six samples have an estimated percentage for galena of $\geq 95\%$. The exception, sample no. 2, contains about 10% of camphor ($\text{C}_{10}\text{H}_{16}\text{O}$) and about 3% of zincite (ZnO) in addition to the above mentioned minor phases of anglesite and cerussite (each about 1%). It thus contains only about 85% of galena. Of these six lead-based samples, two originated in India and four in Egypt. Two of the latter four were purchased as lumps ("kohl hagar", that is "kohl stone") and both contained very small amounts (*i.e.* $\leq 1\%$) of anglesite. One of these two (sample no. 3), obtained in a well-established shop in the main souk (Khan Al-Khalili) of Cairo, was insisted to be (by the shop-keeper), *wrongly*, the ore stibnite (that is antimony trisulphide, Sb_2S_3). This again highlights the very similar appearance (metallic lustre), colour (silver-grey/grey-black) and feel (relatively soft) of the ores galena and stibnite, especially when they are in massive states.

Table 3. Analysis of kohl samples from modern-day Cairo

Sample no.	Texture	Colour	Made in	Purchased in	XRPD major phase	XRPD minor phase(s) ^d	SEM ^e
1	Powder	Black	India (Bombay)	Cairo main souk ^a	Galena (PbS)	(Anglesite (PbSO ₄)) (Cerussite (PbCO ₃))	Pb, S, C, O
2	Powder	Grey-black	India (Bombay)	Cairo main souk	Galena	Camphor (C ₁₀ H ₁₆ O) (Zincite (ZnO)) (Anglesite) (Cerussite)	Pb, S, C, O, Zn
3	Lump	Silver-grey	Egypt	Cairo main souk	Galena	(Anglesite)	Pb, S, C, O
4	Lump	Silver-grey	Egypt	A Cairo souk (Shubra)	Galena	(Anglesite)	Pb, S, C, O
5	Powder	Grey-black	Egypt	Cairo main souk	Galena	(Anglesite) (Cerussite)	Pb, S, C, O
6	Powder	Grey-black	Egypt	Cairo main souk	Galena	(Anglesite) (Cerussite)	Pb, S, C, O
7	Powder	Black	Egypt	Cairo main souk	Amorphous carbon	None	C, O, S
8	Powder	Black	China	A Cairo souk (Shubra)	Amorphous carbon	None	C, O, N (S)
9	Powder	Black	India (Bombay)	Cairo main souk	Amorphous carbon	(Talc (Mg ₃ Si ₄ O ₁₀ (OH) ₂)) (Quartz (SiO ₂))	C, Si, Mg, O, Cl (S)

10	Powder	Black	Egypt	Cairo main souk	Amorphous carbon	None	C, S, O
11	Powder	Black	India	A Cairo souk (Shubra)	Amorphous carbon	(Zincite)	C, S, O, Zn
12	Powder	Blue	Egypt	Cairo main souk	An unidentified amorphous cpd	Unknown	C, Cl, S, O (N, Si)
13	Powder	Green	Egypt	Cairo main souk	An unidentified amorphous cpd	Unknown	C, S, O, N
14	Powder	Purple	Egypt	Cairo main souk	Calcite (CaCO ₃)	Talc and unidentified	Ca, O, Si, C, S, Na, Mg, Ti, Fe (Zn, Cu, N)
15	Lump	Grey-black ^b	Sudan	A Cairo souk (Shubra)	Cuprite (Cu ₂ O)	None	Cu, O, C
16	Powder	Yellow-brown	Egypt	Cairo main souk	Goethite (FeO(OH))	Unidentified	Fe, O, C, S, Cu (Si)
17	Lump	Light grey	Saudi Arabia	Cairo main souk	Silicon	Iron di-silicide (FeSi ₂)	Si, Fe (C)
18	Powder	Grey-white	Egypt	Cairo main souk	Talc	Unidentified	Si, Cu, Mg, O, C, Zn

^a Khan Al-Khalili.

^b When ground, a red powder was obtained.

^c Indicates that the peaks in brackets are only just above background.

^d Each of the minor phases given in brackets were estimated to be less than 5% level in the sample.

Three of the above lead sulphide-based samples (nos. 1, 3 and 6), had their particle sizes (*i.e.* length of the edge of the cube) visually estimated from SEM images of the galena particles (cubes). Sample no. 1 (a matt black powder) had a size range of only 3–10 μm , and an estimated average size of 5 μm . Sample no. 3 (a silver-grey lump, hand ground to a highly iridescent grey-black powder) had a larger size range, 22–135 μm , and an estimated average size of 69 μm . Sample no. 6 (a slightly iridescent grey-black powder) had a size range of 6–24 μm , and an estimated average size of 11 μm .

Seven of the samples were based on amorphous carbon (5) or on carbon in a coloured amorphous organic compound (2): five were black; one, bright green and one bright blue in colour. Of the other five (black) samples, only two had minor phases; zincite in sample no. 11 and talc ($\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$) and quartz (SiO_2) in sample no. 9; both samples originating in India. One of the other black samples originated in China and the remaining two in Egypt. The two brightly coloured samples (nos. 12 and 13) mentioned above were also made in Egypt, had the name “Nefertiti” on their containers and were readily available in the tourist areas of the main souk of Cairo.

One sample (no. 14), also labelled “Nefertiti”, was based on calcite (CaCO_3) and had a known minor phase of talc (at about 10%). As its colour was purple, it was assumed to have a small amount of an unknown colourant present. The sample (no. 16) based on goethite ($\text{FeO}(\text{OH})$) and the one (sample no.18) based on talc were yellow-brown and grey-white in colour respectively. Both contain small amounts of at least one minor phase each, the nature of which are currently unknown. Their colours correspond to the natural colours of the two major phases found. Their containers, the prices and the shop packaging were all almost identical to the previously mentioned samples that were labelled “Nefertiti”. These five samples (nos. 12, 13, 14, 16 and 18), which were all made in Egypt, were regarded by some of the main Cairo souk shopkeepers as being the semi-official kohl and so were the ones most usually offered to tourists.

The IR spectrum of sample 14, purple in colour, showed the peaks for calcite and talc (major and minor phases respectively) and unfortunately these peaks “blotted out” any peaks arising from the small amount of colourant present. However, for samples 12 and 13 (blue and green, respectively, and no phases were identified from the XRPD, as both were totally amorphous), there were good fits for a benzo-sulphonamide compound, possibly with a nitro-group situated “para-” to the sulphonamide group on the benzene ring. These results are supported by the SEM results for these two samples (see Table 3). It is currently unclear if the above sulphonamide compound(s) give the blue and green colours seen for these two samples.

The last two Cairo samples (nos. 15 and 17) were both purchased as lumps, were grey-black and light grey in colour and originated in the Sudan and in Saudi Arabia, respectively. Sample no. 15 gave a red powder on grinding and was found to be pure cuprite (Cu_2O) on analysis. This substance has been observed before once before by us, in the context of ethnic remedies/cosmetics of the Middle East; under the name “seika” it was bought in Dubai main souk, where it was sold as a *face* cosmetic [28]. Regarding the other sample (no. 17), it proved to be difficult to grind to a powder and when analysed, the major phase was found to be elemental silicon, with a minor phase of iron di-silicide (FeSi_2 , at approximately 10%). Such a mixture does *not* occur naturally and how such an unusual, and obviously man-made, material came to be available as a “kohl hagar” in a side-alley of the main souk in Cairo is unclear to us.

Table 4 lists the results of the analyses of the nine kohl samples from modern-day Aswan and Luxor. They are listed in the order of purchase and overall five are based on calcite, one on talc, one on barite (BaSO_4), one on halite (NaCl) and one on amorphous carbon. All the calcite-based samples contain approximately 2% quartz each and one (A2) additionally contains approximately 12% talc. The talc-based sample (A1) additionally contains some quartz (11%) and calcite (2%). The barite-based sample (A4) also contains some quartz (2%). Also, 8 of the 9 samples contain a small percentage (1–2%) of a (probably organic) colourant whose exact chemical nature is currently unknown. The IR spectra obtained for 7 (*i.e.* all except L4 and L5) samples had peaks for the major phases (calcite, talc and barite) and, as before for sample 14, these peaks effectively overlapped with any colourant peaks.

3.2. Pharaonic samples

Table 6 lists the quantitative elemental data obtained from the LV SEM analyses on nine of the twelve samples (three of the pot-only samples and all six of the internal samples). The last column in the table lists the “Probable main component(s)” for both the eye cosmetic (kohl) contents and the pots themselves; these being based on past experience, colour, the LV SEM results and (for the pots) on the experience of a local archaeologist (Dr. R.G. Morkot; personal communication, 2005).

Pot samples MP1, MP2 and MP3 did not have enough material available to do LV SEM and so their expected chemical compositions of travertine (a naturally occurring calcium carbonate deposit) for MP1 and MP2, and of “Egyptian ceramic” (also known as “Nile mud”, mostly a mixture of silicates) for MP3 had to await QEMSCAN analyses (see below). However, for the other three pots, LV SEM was done and its quantitative results (specifically, the first three elements found in each case) supports the listed expected main component in all three cases. That is “Egyptian alabaster” (calcite, CaCO_3) for MP4 and MP5, and “Blue marble” (anhydrite, CaSO_4) for MP6.

The kohl contents of the six pots show the presence of lead, in significant amounts, in five cases. The one exception, sample M3, showed only a very small amount of lead (less than 1%). Thus pot MP3 was assumed to be empty of kohl and the sample analysed to have been of the pot itself. Two of the pot contents were black (M4 and M5) and this colour plus the presence of both lead and sulphur in significant amounts indicates lead sulphide (the ore galena) to be probably present. The other three analysed contents (M1, M2 and M6) were all white/light brown in colour and were all found to have lead, carbon and oxygen present in significant amounts. Additionally, M1 and M6 each contained chlorine. This indicates that M1 and M6 could contain significant amounts of one or more of the following lead compounds: laurionite ($\text{Pb}(\text{OH})\text{Cl}$), phosgenite ($\text{Pb}_2(\text{CO}_3)\text{Cl}_2$), cerussite (PbCO_3), and hydro-cerussite ($2\text{PbCO}_3 \cdot \text{Pb}(\text{OH})_2$); while sample M2 (which had no chlorine) could contain significant amounts of only the last two lead compounds mentioned.

Table 7 gives all the QEMSCAN results on the pot contents (samples M1 to M6). The major components for M3 were found to be various silicates (mostly calcium–aluminium silicates), calcite and quartz. Only a very small amount of lead compounds (phases) was found (0.2/0.1%). This confirms the above assumption that pot MP3 is empty of kohl. The other five content samples all have large (*i.e.* >75%) amounts of lead phases present.

Table 4. Analysis of kohl samples from modern-day Aswan and Luxor

Sample no.	Texture	Colour	Made in	Purchased	XRPD major phase	XRPD minor phase(s) ^a	SEM ^b
A1	Powder	Light blue	Egypt	Aswan (souk)	Talc (Mg ₃ Si ₄ O ₁₀ (OH) ₂)	Quartz (SiO ₂) (Calcite (CaCO ₃)) (Colourant*)	Si, Mg, O, C, (Fe, Ca)
A2	Powder	Light blue-green	Egypt	Aswan (souk)	Calcite	Talc (Quartz) (Colourant*)	Ca, C, Si, O, Mg
A3	Powder	Blue	Egypt	Aswan (souk)	Calcite	(Quartz) (Colourant*)	Ca, C, O (Na, S)
A4	Powder	Dark green	Egypt	Aswan (souk)	Barite (BaSO ₄)	(Graphite) (Quartz) (Colourant*)	Cl, Ba, S, C, Cu, O
L1	Powder	Green	Egypt	Luxor (main tourist souk)	Calcite	(Quartz) (Colourant*)	Ca, C, O (Cl)
L2	Powder	Dark blue	Egypt	Luxor (main tourist souk)	Calcite	(Quartz) (Colourant*)	Ca, C, O
L3	Powder	Dark green	Egypt	Luxor (main tourist souk)	Calcite	(Quartz) (Colourant*)	Ca, C, O (Cl)
L4	Powder	Red	Egypt	Luxor (main tourist souk)	Halite (NaCl)	(Colourant*)	Cl, Na, S, O, C, Fe, K
L5	Powder	Black	Egypt	Luxor (main tourist souk)	Amorphous Carbon	None	C, Ca, S, Si, O

^a The minor phases given in brackets were those estimated to be at less than 5% level in the sample.

^b The elements given in brackets are those whose peaks are only just above background.

* One or more (synthetic) organic colourants, whose exact nature is currently unknown.

Table 5. Summary of Egyptian modern-day kohl sample names

Sample no.	Sample name ^a	Data on contents?	Data on medicinal effects?	Contains lead?
(A) Cairo samples:				
1	Khojati Surma Sada	Y	N	Y
2	Khojati Surma No. 9	Y	Y	Y
5	CHOL NORHAN (cold) ^b	N	N	Y
6	CHOL NORHAN (hot) ^b	N	N	Y
7	CHOL NORHAN	N	N	N
8	Kohl Noori ^b	N	N	N
9	Hind Ka Noor eye liner	N	Y ^b	N
10	LUX No. 1	N	N	N
11	Black Shahrazad	N	N	N
12	Nefertiti	N	N	N
13	Nefertiti	N	N	N
14	Nefertiti	N	N	N
(B) Aswan and Luxor samples:				
A2	Kohl Shahrazad	N	N	N
A3	Cleopatra Super	N	N	N
A4	Cleopatra	N	N	N
L1	Kamal Cleopatra ^b	N	N	N
L5	CHOL MOHGA	N	N	N

^a The English name on the label of the container or on the leaflet inside the container, unless translated.

^b Translated from Arabic.

Unfortunately, at the present time, this analytical technique cannot distinguish between the various lead phases thought to be present (see above). Further work is in progress, using single-element wavelength dispersive spectrometry, and we hope to publish a later paper on the results. Other compounds sometimes found to be present in these five samples, at >2%, were: calcite, gypsum/anhydrite, quartz, iron compounds and various silicates. Very small amounts of (*i.e.* down to 0.1%) of sphalerite, copper/nickel/silver phases, ilmenite, rutile, sphene, and apatite were also sometimes found. The percentage of the non-identified phases was never >2% and was usually much less than this amount.

Table 8 gives the QEMSCAN results on the pot samples (*i.e.* MP1 to MP6). The presence of relatively high percentages (up to 13.2%) of lead compounds in three of the samples (MP2, MP4 and M6) was unexpected and is currently being further investigated. The major components for both MP1 and MP2 were various silicates (86.0 and 70.4%, respectively); for MP3, MP4 and MP5 it was calcite (54.5, 59.0 and 93.1%, respectively) and for MP6, it was gypsum/anhydrite (88.0%). Other significant amounts (*i.e.* >10%) of

Table 6. Information on six (Pharaonic) Egyptian kohl samples (and their containers)^g plus initial analytical results

Sample no. (approx. period ^a) [Pot no.] [Museum cat. No.]	Texture of contents	Colour of contents [Pot colour] [Pot height]	LV SEM ^c on contents (in decreasing order of wt. % ^b) [LV SEM on the pots ^e]	Probable main component(s) of the contents (kohl) [Probable main component(s) of the pot]
M1 (Middle Kingdom?) [MP1] [5/1946.771]	Powder	White/V. light brown [Grey-Brown] [3.1 cm]	Pb, O, C, Cl, Zn (Si, Ca, Fe, Al) [Not done ^f]	Lead carbonate/chloride/hydroxide [Travertine? ^a (a form of CaCO ₃)]
M2 (Middle Kingdom?) [MP2] [5/1946.769]	Powder	White/V. light brown [Grey-White] [3.8 cm]	Pb, O, C, Fe, Si, Al (Na, Mg Ca) [Not done ^f]	Lead carbonate/hydroxide [Travertine? ^a]
M3 (New Kingdom?) [MP3] [5/1946.772]	Powder	Dark brown/Black [Red-Brown] [5.9 cm]	O, C, Ca, Si, Fe, Al, K, Mo (Cl, Na, Mg, Ti, Pb) [Not done ^f]	None (pot thought to be empty) [“Egyptian ceramic” ^a (sometimes “Nile mud”)]
M4 (Middle Kingdom?) [MP4] [357/1974/5]	Powder	Black [Light Brown] [6.5 cm]	C, O, Pb, S, Ca, Fe, Zn, Cl (Si, K, Al) [O, C, Ca, Cu, Si (S, Mg)]	Lead sulphide [“Egyptian alabaster” ^a (that is Calcite)]

M5 (Middle Kingdom?) [MP5] [64 1919]	Powder	Dark brown/Black [<i>Light Brown</i>] [4.5 cm]	O, C, Pb, Ca, S, Cl, Al (Si, Fe, Cu, Na) [O, Ca, C (Si, Mg)]	Lead sulphide [“ <i>Egyptian alabaster</i> ” ^a]
M6 (Middle Kingdom) [MP6] [“Abydog 1922”] ^d	Powder	White/Light brown [<i>Blue-White</i>] [4.2 cm]	Pb, C, O, Cl, S, Fe, Cu, Zn (Ca, K, Si, Na, Al) [O, Ca, S, Si, Fe (K, Na, Al, Mg, C)]	Lead carbonate/chloride/hydroxide [“ <i>Blue marble</i> ” ^a (<i>that is Anhydrite</i>)]

^a Done by a local archaeologist (Dr. R.G. Morkot), on the basis of pot shape, size and colour, etc. (a “?” indicates that the approximate period/composition given is tentative).

^b The elements in brackets are at less than 1% level each.

^c For details of this technique see text.

^d This pot is thought to have come from the excavation of Abydos in 1922 by W.M.F. Petrie.

^e Done from *external* “scrapings” from each pot (pot data is given in *italics* in [...]).

^f Insufficient material to do LV SEM.

^g From the Royal Albert Memorial Museum (Exeter, UK).

compounds present were (apart from the lead compounds already mentioned above): gypsum/anhydrite (MP3 and MP4) and various silicates (MP3).

4. DISCUSSION

4.1. Comparison of past and present origins/compositions

The origins of the modern-day Egyptian kohl samples analysed are shown in Fig. 1 and it can be seen that almost three-quarters of them originate in Egypt. However, for the ancient samples analysed by others (see Tables 1 and 2) and by ourselves (see Tables 6–8), we can only speculate on their origins. It is known that almost all of the main components listed in Table 1 were available within Egypt itself; the one exception being stibnite, which probably came from Asia Minor (Turkey), certain of the Greek islands or possibly Arabia. It is also known that some of the eye-paints were imported from Naharin (in western Asia) and from Punt (Eritrea/Somalia) – the latter presumably being only a staging post for material that originated in Arabia. Both galena and malachite still occur in various parts of Arabia [3,29].

Of the 18 Cairo-purchased modern-day samples, 6 were found to be lead-based (*i.e.* 33%). If the 9 samples purchased in Luxor and Aswan are included, then this percentage falls to 22% (Fig. 2). This compares very favourably with the value of 63% for the lead-based samples in antiquity (Fig. 3; based solely on the data given in Table 1). Unfortunately the Louvre-based authors have so far *only* published analytical data on lead-based samples (for instance, given in Table 2) and thus their data were not included in the preparation of Fig. 3. Also, of the six Pharaonic kohl pots studied by us, high percentages (>75%) of lead phases were found in five of the pots (one was assumed to be empty of kohl as essentially no lead phases were found) (see Tables 6–8). The other phases listed in Table 7 are either contamination from the environment (*e.g.* quartz from ubiquitous sand), from the original lead ores (*e.g.* the silver phases) or from the pot itself (*e.g.* the various calcium compounds found). Also, some degree of mineralogical alteration is likely to have occurred as the pots have been open for an unknown period of time. However, regarding the lead

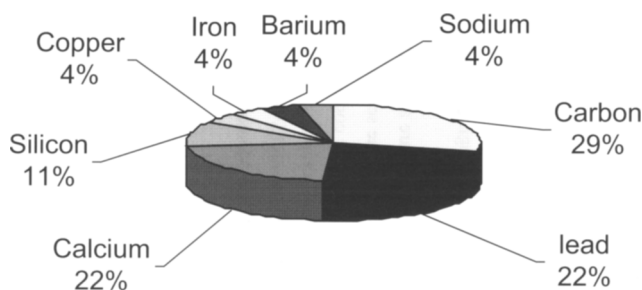


Fig. 2. Distribution of the main element of the major phase in the modern-day Egyptian kohl samples studied.

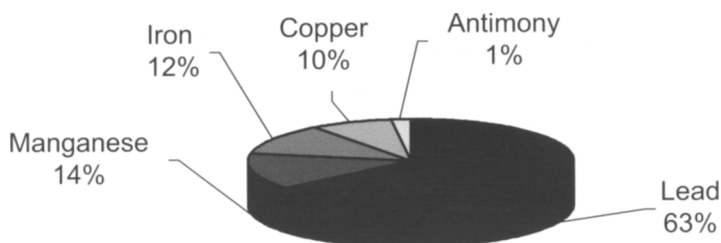


Fig. 3. Distribution of the main element of the major phase in ancient Egyptian kohl samples (Ref. [3]; Table 1 only).

phases, as previously stated we cannot distinguish definitely between the various lead phases that are present in these samples. Further work on this is in progress and will appear in a later publication. The other major differences between past and present compositions are: the presence of manganese and (once) antimony compounds only in ancient samples; the absence of any analysed ancient samples that consist mainly of carbon; and the presence of barium, silicon and calcium compounds, as the major components, in only modern-day samples. Also, in the past, the colour of the sample was due to the main component, though various “whitening” materials were added sometimes (see next section), as compared to the present-day, when the colour is given by a small percentage (1–2%) of a (probably synthetic and organic) colourant added to the (white, inexpensive and readily available) main component (such as calcite or talc) (see for eight of the nine samples in Table 4 and for three of the 18 samples in Table 3).

As already mentioned, only one “old” (analysed) sample, from the nineteenth dynasty, has been found to be an antimony compound (stibnite, Sb_2S_3) (see Table 1). The reason for the occasional statement that antimony/antimony (tri)sulphide/stibnite was used as an eye cosmetic in ancient Egypt (*e.g.* Refs. [30,31]) is mainly one of philology. The ancient Egyptian word for eye-paint in general and the black form in particular was “msdmt” (mesdemet) and it became “cthm” (stem) in Coptic, then “stimmi” in Greek and finally “stibium” in (Roman) Latin. This last word was later used for the element antimony, and stibnite for its sulphide ore. Also, as already mentioned, in their massive states the two ores stibnite and galena look very similar. Stibnite is rarer than the more common (and cheaper) galena and so the temptation to replace the former with the latter would have been an “economic incentive” from the earliest times.

Also, one of the authors can personally testify to being offered “ithmid”/“ethmid”/“athmid” (that is the eye cosmetic of Islam, which has generally been assumed to be the ore stibnite) in various souks of modern-day Arabia and subsequently finding them all to be the ore galena. To our knowledge no early-Islam sample, from a reputed museum and of known provenance, has yet been chemically analysed.

As to the speculated usage of lapis lazuli as a blue eye cosmetic by Cleopatra VII (see Introduction), there is presently no analytical published data showing that lapis lazuli was used as an eye cosmetic in ancient Egypt. Funerary cosmetic items from Pharaonic Egypt, that have been subjected to detailed chemical analytical study and subsequently published in peer reviewed and abstracted journals, do not currently include any definitive

blue material. Chrysocolla (a hydrated copper silicate) has been found once, but its colour can vary from blue-green to green. However, a modern-day study of a North African recipe for blue eye-shadow showed that by subjecting natural galena to “heat treatment”, a blue-appearing material is produced. The blue colour is in fact an optical interference effect resulting from the formation of layers of anglesite (PbSO_4) and then of lanarkite ($\text{PbO}\cdot\text{PbSO}_4$) on the original galena [32]. It is possible that these materials, and their effect, were produced accidentally in ancient Egypt. However, the recent analyses of many eye cosmetics stored in the Louvre have found anglesite, but not lanarkite, to be present sometimes (*e.g.* see Table 2).

4.2. Toxicology of lead

Lead compounds are toxic by ingestion, inhalation and by skin exposure. Children are more susceptible than adults to lead intoxication. The toxic effects of lead form a continuum from clinical or overt effects to more subtle ones [33]. The critical effects in infants and children involve the nervous system. Blood lead levels once thought to be safe have been shown to be associated with intelligence quotient deficits, behavioural disorders, slowed growth and impaired hearing [34,35]. Blood lead levels in children that are greater than $10\ \mu\text{g}/\text{dl}$ are now considered abnormal [36], and recently it has been shown that significant intellectual impairment occurs in young children who have blood lead levels below $10\ \mu\text{g}/\text{dl}$ [37]. Severe lead poisoning, resulting in encephalopathy, can result when the blood lead levels are greater than $70\ \mu\text{g}/\text{dl}$. A recent report has demonstrated that young infants exposed to lower levels of lead following the use of traditional medicines can also present with encephalopathy [38]. Reported cases of acute encephalopathy in infants that are directly linked to excessive usage of a lead-based kohl are now fewer than several decades ago, but unfortunately still do occur [39].

Frequently, mothers apply kohl to infants and children as a traditional measure to beautify and protect the child from the “evil eye”. Lead-based kohls can be easily ingested by these infants who may wipe their eyes and face and subsequently lick their fingers; earlier (animal) studies [40] have shown that transcorneal transport is not a significant contributory mechanism for absorption of lead from lead-based eye cosmetics. It is worth noting that adults absorb 5–15% of ingested lead while children can absorb as much as 41% of ingested lead.

Solubility studies have shown that the particle size of the ground galena is directly related to its rate of dissolution (*i.e.* conversion to the more soluble, and hence more readily absorbed, chloride forms) in gastric fluid. An increase in the rate of dissolution, by a factor of approximately two, was found for galena of mean particle size $30\ \mu\text{m}$ as compared to galena with a mean particle size of $100\ \mu\text{m}$ [40]. As a result of this effect, larger sized particles of galena could well pass through the GI (gastro-intestinal) tract before it is converted to a more readily absorbed form. This particle-size effect could well explain the varying degrees to which galena has been reported to be absorbed in the gut. As the galena powder is ground it loses its initial high iridescence to become progressively more matt in appearance, becoming totally matt at a mean particle size of about $\leq 10\ \mu\text{m}$. In this study, the two galena-based Indian-made kohl powders (sample nos. 1 and 2; with sample no.1

found to have an estimated average particle size of 5 μm) are totally matt in appearance; the two galena-based samples made in Egypt (sample nos. 5 and 6; with sample no. 6 found to have an estimated average particle size of 11 μm) are mostly matt; and the two hand-ground samples (sample nos. 3 and 4; with sample no. 3 found to have an estimated average particle size of 69 μm) are highly iridescent. Thus if these six galena-based samples were equally ingested there would be a range in the rates of their dissolution in the stomach's gastric fluids, with the two Indian-made samples having the highest rates of dissolution, and hence absorption.

As the present, so the past. A range of powder appearances (from wholly matt to wholly highly iridescent), and hence a range of particle sizes, have been found for the galena in some of the ancient Egyptian kohl samples in the Louvre. Using data from several techniques (SEM and Transmission electron microscopy (TEM) images for particle sizes, and peak profile analysis of synchrotron XRD data for crystallite sizes), it has recently been suggested [14] that there were four or five “manufacturing procedures” used by the ancient Egyptians for making kohl. These are: (1) gentle crushing, (2) gentle crushing and sieving, (3) crushing, (4) crushing and sieving and perhaps (5) crushing and heating (to 200–300°C). Kohls made by “procedures” (1) and (2) (and (5), if used) are iridescent and those from the two other “procedures” are mainly matt. Possibly this was done in response to a demand for differing styles of eye cosmetic. These variously made powders would then perhaps have been mixed with a naturally occurring white dilutant (such as cerussite) to give shades of grey cosmetics and/or mixed with one or both of the “made” lead compounds (phosgenite and laurionite) to give eye salves or possibly face cosmetics. These two “made” compounds have been found to have smaller particle sizes (down to 1 μm).

More than 90% of lead in blood resides in the red blood cells. The total body burden of lead can be divided into two kinetic pools, which have different rates of turnover. The largest pool is in the skeleton, which has a very slow turnover (a half life of more than 20 years) [36]. The other pool is in the soft tissue, where it is much more labile. Lead in the trabecular bone is more labile than in the cortical bone, and trabecular bone has a shorter turnover time. Lead in bone may contribute up to 50% of blood lead. During pregnancy and lactation, mobilisation of lead from maternal bone is a cause for concern. Strong correlations between maternal and umbilical cord blood lead levels demonstrate that lead is transferred from the mother to the foetus [41,42]. Cumulative effects of low levels of lead exposure *in utero* and after birth can have similar detrimental effects. An increase in maternal-blood lead level may contribute to a reduction in gestation period and low birthweight. The foetal brain may also be particularly sensitive to the toxic effects of lead because of the immaturity of the blood–brain barrier.

In an adult population the most critical adverse effect of lead is probably hypertension. Other toxic effects of concern are peripheral neuropathy, lead-induced anaemia and lead nephropathy.

In view of the above mentioned toxic effects and the still widespread use of kohls in the present-day Middle East, it follows that children who have a lead-based kohl regularly applied to them are at risk of serious and fatal toxicities of the nervous system and also to more subtle, subclinical, long-term effects.

4.3. Written information on container/packaging

In ancient (Pharaonic) Egypt, the funerary containers used for eye cosmetics and/or eye salve were made of materials such as: glass (New Kingdom onwards), wood, reed, bone, steatite (also known as soapstone, a massive form of talc), serpentine (a magnesium silicate), ivory and/or ebony, obsidian (a glassy volcanic rock), rock crystal (a form of quartz), alabaster (a fine-grained massive form of gypsum, that is hydrated calcium sulphate), “Egyptian alabaster” (which is in fact calcite, calcium carbonate), anhydrite (the anhydrous form of calcium sulphate, the so-called “blue marble” of ancient Egypt) or “Egyptian ceramic” (also known as “Nile mud”; mostly a mixture of silicates). Also, multiple containers (*i.e.* two, three or four joined containers) are known and it is assumed that these were for kohl to be used in different seasons and/or contained kohl of different colours. On some containers, both single and multiple, are written, in hieroglyphs, comments such as: “Genuine, very excellent kohl”; “Opens vision” (*i.e.* an eye salve/solution); “Repels blood” (*i.e.* checks bleeding) [1] and “Good for the sight”; “To cause tears”; “For daily use” [43].

The Pharaonic kohl pots studied by us (see Fig. 4) were originally thought to be composed of the materials listed in Table 6 (under “*Probable main component(s) of the*



Fig. 4. The six Pharaonic kohl pots studied. Pot nos. (from L to R and back row first): MP4, MP5, MP6, MP3, MP1 and MP2 (see Tables 6, 7 and 8) (© Royal Albert Memorial Museum and Art Gallery, Exeter, UK).

pot itself”). The analytical results in Tables 6 and 8 show that these were correct for pots MP5 and MP6; *i.e.* “Egyptian Alabaster” (calcite) for MP5 (at 93.1%) and “blue marble” (anhydrite) for MP6 (at 88.0% for gypsum/anhydrite in Table 8 and anhydrite rather than gypsum from Table 6). However for MP1 and MP2, the expected travertine (a naturally occurring calcium carbonate deposit) was in fact found to be incorrect and their main components (at 86.0 and 70.4%, respectively) were “various silicates” (mostly calcium aluminium silicates). This indicates that these pots are probably made of “Egyptian ceramic” (see above). For MP4, a significant amount (59.0%) of calcite was found, but additionally significant amounts of gypsum/anhydrite (at 26.2%) were also found. Also, for pot MP3, high percentages of calcite (54.5%) and gypsum/anhydrite (26.2%) were found. If these pots had been made from “Egyptian ceramic”, then significant amounts of silicates would have been expected. This indicates that the pots MP3 and MP4 were possibly made from a mixture of “Egyptian alabaster” and alabaster (that was then a naturally occurring sedimentary deposit).

As regards the contents of the containers, the hieroglyph for “msdm” is sometimes seen and in one case the hieroglyph for “high quality”, repeated three times, is placed before it [17]. As already stated, the word “msdm” is used for eye-paint in general and the black form in particular. When this hieroglyph was observed on one sample of a nineteenth dynasty funerary deposit it was found to be galena (lead sulphide), whilst other samples from the same funerary deposit but with hieroglyphs saying “eye lotion to be dispersed, good for eyesight”, were found to be mixtures of lead chloride and lead carbonate [16]. This provides evidence that the ancient Egyptians (here, New Kingdom) had a knowledge of “wet chemistry” and made these lead compounds with the definite intention of using them for therapeutic purposes.

Out of the 18 modern-day Cairo samples studied, 12 had a name written on the container and/or on the packaging. Of the 9 samples from Aswan and Luxor, only 5 had a name on the container. Table 5 lists the names as found on the labels/packaging. These names are usually in English, but in two cases no English name was found and the translation of the name (from Arabic) is given. Also given in this table, on a simple Y/N basis, is whether any information is given on the contents, on the medicinal effects of using the kohl and whether the sample was found to contain lead.

Regarding the data on the contents, only 2 samples (nos. 1 and 2) gave definite *quantitative* “contents formulas” on enclosed leaflets (as percentages). Sample no. 1 has “Asmad” given as 100% of its contents and this is often found to be lead sulphide (as here). Sample no. 2 gives the following contents data (with the percentages given in brackets): Asmad (70%); Bh. Kafoor (28.5%); Sadaf Softi (Mori) (0.70%); Ark Phudina (0.30%); Ph. Ph. Safed (0.25%) and Hab-El-Arus (0.25%). On translation/interpretation these substances are: lead sulphide, camphor, “pearl” (*i.e.* probably the mineral aragonite, or possibly calcite – both being forms of calcium carbonate); extract of mint; “white potassium?” (the exact chemical name is currently unknown) and “Bean of the bride-groom” (Java pepper) respectively. As given in Table 3, both these samples contain lead sulphide as the major phase and for sample no. 2 camphor is also found. Additionally, both these samples contain small amounts of anglesite and cerussite, with sample no. 2 also having a small amount of zincite. No other labelled sample gave any contents data on its container or on its packaging.

As regards the data on medicinal effects, it was found that only two samples (sample nos. 2 and 9) gave this information – in an enclosed leaflet in each case. Both samples were made in Bombay (India), but not by the same company. For sample no. 9 (Hind Ka Noor eye liner), the data was written in colloquial Arabic and (after translation) it stated that “it was good for”: “Reducing cold”, “Eye ache”, “All diseases of the eye”, “Heat in the eye”, and “Improves eye-sight and strengthens vision”. It also stated that “Used on a daily or weekly basis it will protect you always against diseases of the eye” and “can be used by adults or children”. This particular kohl has been seen before, in the souks of: Amman (Jordan) (unpublished data), Abu Dhabi city (Ref. [44]; sample no. 11), Bahrain and Oman (Ref. [45]; sample nos. 27 and 28 for Oman and no. 31 for Bahrain). The major phase is always amorphous carbon and the minor phase(s) are one or more of the following: dolomite ((Mg,Ca)(CO₃)₂), graphite, quartz and talc. All these substances are unlikely to give rise to adverse medical conditions when used externally on the eye; however, if used internally (e.g. on the conjunctiva surface) then abrasions could well occur, especially from the harder substances present (i.e. quartz and, to a lesser extent, dolomite and graphite). Also, the larger and more irregular the compound’s particles, then greater will be the potential for eye abrasions.

Sample no. 2 (Khojati Surma No. 9) has the written information (in English): “It is cooler than Khojati Surma No. 13 and of better quality. Its regular use keeps the eyes clean, healthy and reduces the adverse effects caused by heat”. Khojati Surma No. 13 was one of the samples purchased in Abu Dhabi city souk [44] and was found to contain calcium carbonate as the major phase and with camphor, kaolinite, iron silicate hydrate and graphite as the minor phases. As stated before, these substances are unlikely to give toxicity; however, this is not the case for our sample no. 2, where the major phase is a lead compound (galena). Thus, the use of this kohl could give rise to lead toxicity and its quoted medicinal effects are questionable at best and dangerous at worst.

Additionally, found only in the enclosed leaflet of sample no. 1 (Khojati Surma Sada; also sample no. 1 in our Abu Dhabi city study), is the following statement (in English): “This is the purest form of Surma scientifically ground in different extracts. As there is no addition of any other medications, it does not make the eye water *and may be used for children below the age of 8 years*” (our addition in *italics*). This is identical to the wording found along with the sample purchased in Abu Dhabi and as then, we can only repeat our view that such advice, especially with regard to young children, is both dangerous and outrageous as the major phase is (as before) lead sulphide *and* is of such a small particle size that it will be readily absorbed by the body’s gastric juices.

Samples nos. 1 and 2 are also the only ones to have written data on how to apply the kohl to the eye. Both contain a plastic applicator rod in the sample box; ten other samples (nos. 5, 6, 7, 10, 12, 13, A2, A3, A4 and L5) have an applicator rod actually in the container with the kohl powder. The written data for samples 1 and 2 states (in English): “Apply with a clean and dry salai (applicator) a minimum quantity of surma in the morning and half an hour before going to bed”. Sometimes, in present-day Egypt, the powder is applied as is (i.e. dry), sometimes after mixing with water to make a paste and sometimes after mixing with “an oil” (often olive oil). The Egyptian Bedouin are known to use the juice of chopped onions on the applicator before placing it in a kohl powder and then on/in/around the eye. The resulting “washing of the eye” is regarded as a beneficial side-effect of using the kohl. It is also reported

that in modern-day Upper Egypt (black) kohl, which has had both lemon juice and onion juice added to it *via* the use of an applicator, is placed in/on/around a new-born baby’s eyes once a week for the first 40 days of its life. This is done for two reasons; to kill germs (the juices) and to take away the “evil eye” (the kohl) (Hardy; personal communications, 2001).

Of the 17 labelled samples, four were found to contain lead. Two have already been discussed (sample nos. 1 and 2). The other two were sample nos. 5 and 6; both of these samples give no mention of containing lead on the label, but in addition to the name (CHOL NORHAN) there was a “cold” (Arabic) symbol on the label of sample no. 5 and a “hot” (Arabic) symbol on the label of sample no. 6. A “hot” kohl sample is often used as an eye medicine and so is sometimes placed *inside* the eye, whilst a “cold” kohl sample is used solely for beautification and is often only used on the outside of the eye. In Oman, a kohl used as a medicine (*i.e.* “hot”) often contained, or was supposed to contain, an “active ingredient” of a lead or antimony compound [21]. Here, *both* “hot” and “cold” labelled kohl samples have lead sulphide as the major phase; but the third CHOL NORHAN sample (no. 7) purchased, which had no such “hot”/“cold” label, but was designated “cold” by the shopkeeper, consists solely of amorphous carbon.

5. CONCLUSIONS

As has been stated before, lead is of no known biological value, is not an essential element and when present in the blood can give rise to toxicity. In this study, lead was found to be present in almost one-quarter of the Egyptian modern-day samples studied. Six samples were based on galena, six on amorphous carbon, three on silicon/silicon-based compound (talc), six on calcite, one on cuprite, one on goethite, one on barite, one on halite and two on unknown (but assumed to be carbon-based) amorphous compounds. If the other “heavy metals” (copper and iron, *i.e.* those metals with a density ≥ 5 gms/c.c.) found as a major phase element in these samples are included, then the above percentage rises from 22% (only lead-containing) to 30% (copper, iron and lead-containing). In the (Pharaonic) past a higher percentage (63%, using the Table 1 data only) of the samples were lead-based. This was confirmed by our findings; five of the six Pharaonic kohls studied by us had lead-based contents (one was empty). Two were black in colour and so probably contained lead sulphide, whilst the other three were white in colour and probably consisted of a mixture of various natural/“made” lead compounds/minerals (such as cerussite, laurionite, phosgenite, hydro-cerussite and anglesite). It appears unlikely that the ancient Egyptians understood lead’s toxic nature as they deliberately used the black eye-paint, and other “made” lead compounds, in the treatment of eye diseases/infections.

Now and then, young children are especially vulnerable to lead toxicity; with the level of lead in blood that can cause long-term damage being continuously revised downwards. It now appears that there may be no lower limit for adverse effects from lead exposure to occur and that once the impairments have occurred they may well be both persistent and irreversible. Thus it is essential that the initiatives started by the Egyptian Government/USAID/CDC be continued and that there should continue to be a reduction in the availability of lead-containing eye cosmetics in modern-day Egypt.

Table 7. QEMSCAN results (wt. %s)^a on the contents of six (Pharaonic) Egyptian kohl pots

Mineral	M1 (%)	M2 (%)	M3 (%)	M4 (%)	M5 (%)	M6 (%)
Lead phases ^b	91.3 (94.0)	87.4 (90.9)	0.2 (0.1)	79.5 (81.4)	78.4 (80.4)	91.5 (92.3)
Calcite (CaCO ₃)	1.4 (0.7)	1.8 (0.8)	27.9 (18.2)	6.5 (4.7)	11.0 (8.5)	0.3 (0.2)
Gypsum/Anhydrite ^b (CaSO ₄ ·2H ₂ O/ CaSO ₄)	0.3 (0.4)	3.3 (4.3)	0.9 (1.9)	3.8 (5.3)	4.1 (5.4)	0.4 (0.5)
Quartz (SiO ₂)	3.7 (1.1)	1.0 (1.0)	17.7 (12.3)	3.7 (2.6)	0.9 (0.7)	1.6 (0.8)
Iron phases	1.3 (1.0)	3.7 (1.5)	1.2 (0.2)	3.0 (2.4)	3.1 (3.0)	2.1 (1.8)
Sphalerite (ZnS)	0.6 (1.3)	0.0 (0.0)	0.0 (0.0)	0.5 (0.8)	0.5 (0.6)	0.4 (0.4)
Copper phases	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.7 (1.0)
Silver phases	0.0 (0.0)	0.2 (0.0)	0.0 (0.0)	0.0 (0.5)	0.0 (0.0)	0.0 (1.6)
Nickel phases	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.2 (0.1)	0.6 (0.5)	0.0 (0.0)
Ilmenite/Rutile (FeTiO ₃ /TiO ₂)	0.0/0.0 (0.0/0.0)	0.0/0.0 (0.0/0.1)	0.5/0.2 (0.7/1.0)	0.0/0.1 (0.2/0.1)	0.2/0.0 (0.2/0.0)	0.0/0.1 (0.1/0.0)
Sphene/Apatite (MgAl ₂ O ₄ / Ca ₅ (PO ₄) ₃ (F, Cl, OH))	0.0/0.0 (0.0/0.0)	0.0/0.0 (0.0/0.0)	0.1/0.9 (0.4/0.6)	0.0/0.1 (0.0/0.0)	0.0/0.0 (0.0/0.0)	0.0/0.0 (0.0/0.0)
Various Silicates	1.4 (1.5)	1.6 (1.1)	50.1 (64.6)	2.2 (1.8)	0.9 (0.7)	0.9 (0.7)
Non-identified phases	0.0 (0.0)	1.0 (0.3)	0.3 (0.0)	0.4 (0.1)	0.3 (0.0)	2.0 (0.6)
Number of particles analysed	10 050 (5019)	10 012 (661)	10 225 (2706)	10 132 (5012)	10 106 (5107)	10 012 (4027)

^a Two size fractions of the kohl pots were analysed. Particles between 1 and 25 µm; and the values in brackets are for particles between 25 and 200 µm (see Experimental section for details).

^b In this technique, energy dispersive spectrometry cannot currently distinguish between the various lead phases present, and also between Gypsum and Anhydrite.

Table 8. QEMSCAN results (wt. %s) on the composition of six Pharaonic kohl pots

Mineral	MP1 (%)	MP2 (%)	MP3 (%)	MP4 (%)	MP5 (%)	MP6 (%)
Lead phases	0.5	13.0	0.5	13.2	1.9	8.3
Calcite	0.8	1.5	54.5	59.0	93.1	0.2
Gypsum/Anhydrite	1.3	3.5	26.2	21.5	1.3	88.0
Quartz	7.3	9.1	1.9	1.0	0.4	0.5
Iron phases	0.3	0.4	0.4	0.6	0.0	0.1
Sphalerite	0.0	0.0	0.0	0.2	0.0	0.1
Copper phases	0.0	0.0	0.0	0.8	0.0	0.0
Ilmenite/Rutile	0.1/0.1	0.0/0.2	0.2/0.0	0.6/0.0	0.0/0.0	0.0/0.0
Sphene/Apatite	0.1/0.1	0.1/0.1	0.0/0.2	0.0/0.0	0.0/0.0	0.0/0.0
Nickel phases ^a	3.1	1.4	0.0	1.2	2.8	0.4
Various Silicates	86.0	70.4	16.1	1.8	0.5	2.3
Non-identified phases	0.3	0.3	0.0	0.1	0.0	0.1
Number of particles analysed	5065	5053	5019	4226	5044	5074

^a These percentages are a summation for Nickel sulphides and elemental Nickel (which is assumed to have come from the sampling spatula).

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