CCCXLII.—*Examination of an Ancient Egyptian (Tut-ankh-Amen) Cosmetic.*

By A. Chaston Chapman and H. J. Plenderleith.

The discovery by Dr. Howard Carter of a sealed cosmetic jar of calcite among the many objects of interest in the tomb of Tut-ankh-Amen at Luxor presented a unique opportunity for the examination of an ancient fatty substance which had not entirely decomposed in the process of time. The tomb itself had remained sealed from the outside air for more than 3300 years, and when opened in 1922 the introduction of sterile swabs failed to indicate the presence of any bacteria. It was therefore of much interest to examine this ancient fatty material with a view to ascertain what changes it had undergone in this sterile environment and, if possible, to collect data which might shed some light upon its probable source.

The jar had been sealed by natural agency, the changes in temperature and moisture having caused certain salts, nearly always associated with Egyptian objects of antiquity, to crystallise round the lid and so to form a hard, protective incrustation.

When the jar was opened by Dr. Carter a considerable quantity of cosmetic was found inside (about 450 g.). This was rather sticky, and presented the appearance of a heterogeneous mixture consisting of yellow nodules together with a chocolate-coloured substance. It melted, in part at least, at the heat of the hand, emitting a faint yet distinctive odour. This odour was to us at first suggestive of that of coconut, but has been variously described since either as resembling that of the flowers of broom, or as being rather valerianaceous in character. The cosmetic had a decidedly fatty smell, but certainly not that characteristic of advanced rancidity. A careful microscopical examination failed to reveal any traces of vegetable fibre or other organised structures.

It is of interest in this connexion that a fat found in Egyptian jars protected by thick mud layers has been described by Petrie and Quibell (" Naquada and Ballas," 1895, p. 39) as dating from a much earlier period of Egyptian history and possessing the external characters at least of the specimen under discussion. The Naquada fat, however, was found to contain vegetable fibre when examined under the microscope. Although the histological examination of the debris proved wholly inconclusive in the hands of Dr. Thistelton-Dyer at Kew, these investigators seem convinced that the presence of fibre in the fat is indicative of its vegetable origin—a conclusion which is not beyond question, having regard to the fact that fats are stated on good authority to have been preserved by the Egyptians by rolling them in vegetable fibre. While Petrie and Quibell state that the nature of their specimen is not yet certain, they conclude that the only source known for it would be one of the vegetable butters, *e.g.*, from Bassia (Shea butter) or the oil palm. It seems unlikely, however, as pointed out later, that the odorous constituents of such fats would be stable enough to withstand the action of oxygen and moisture for such a prolonged period. It should be mentioned that excavators are decided in stating that this smell of coconut is often noticeable in specimens freshly excavated, and this suggests that it might be due to an odorous product of decomposition of such substances as fats, resins, or balsams.

The Tut-ankh-Amen specimen was largely organic in nature but left a decided residue on ignition consisting mainly of calcium carbonate. Neither nitrogen nor sulphur was present.

General Proximate Analysis.

Fatty matters soluble both in light petroleum and in ether	59·1%
Fatty matters soluble in ether, but practically insoluble in light petroleum	28·6
Matters soluble in alcohol, but insoluble in ether	0·9
Matters soluble in water, but insoluble in alcohol	4·4
Matters insoluble in any of the foregoing solvents	4·7
Moisture (loss on drying at 105°)	2·3
	100·0

The total fatty matters extracted by ether, and amounting to 87·7%, had an acid value of 252·6, an iodine value of 4·5, contained only a trace of unsaponifiable matter, and left 0·4% of ash on ignition. There was also present in it 5·46% of free and combined glycerol, which is equivalent to 4·8% on the cosmetic itself. The cosmetic left on ignition 2·04% of ash.

Fatty Matters Soluble in Ether and Light Petroleum.—The portion of the fatty matter which was soluble both in ether and in light petroleum consisted of a white, crystalline solid, having about the same consistency as palmitic and stearic acids and resembling them in appearance. This retained the odour of the original cosmetic, which now resembled still more closely that of coconut. A portion of this fat gave the following results : Reichert value, 0·9 ; Polenske value, 0·5 ; acid value, 214·4.

Fatty Matters Soluble in Ether, but Insoluble in Light Petroleum.—This portion was softer than the preceding portion, showed but little tendency to crystallise, and had a distinct yellow colour. It had an acid value of 331·5 and had only a very slight coconut odour.

Matters Soluble in Alcohol, but Insoluble in Ether or Light Petroleum.—These consisted of a reddish-brown, viscous material, possessing no distinctive odour. The amount was so small that a separate examination was impossible.

Matters Soluble in Water.—This portion was left on evaporation of the water as a translucent, brown, resinous substance, resembling shellac in appearance, and consisted chiefly of calcium salts. On the addition of dilute hydrochloric acid to its aqueous solution, a precipitate was formed which gradually coalesced, forming a sticky, brown mass. This was readily soluble in alcohol, but almost completely insoluble in ether.

Matters Insoluble in Ether, Alcohol, or Water.—This fraction, when dried, was a brown, amorphous powder, free from odour; on ignition it left 31% of mineral matter, consisting almost entirely of calcium carbonate. When it was treated with dilute acid there was no decided effervescence, but on warming, decomposition apparently occurred, and an acidic substance was formed which was insoluble in water and softened on warming. This acid was insoluble in ether, but was fairly readily dissolved by hot alcohol, separating on cooling as a brown sludge.

A consideration of the above results appears to indicate that the constituents of the cosmetic may be roughly divided into two classes: (*a*) the fatty matters soluble in ether, and (*b*) matters of a resinous character.

It will be seen from the iodine number that the fatty acids are, as might have been anticipated, almost entirely saturated in character. The examination of the fatty matter shows it to contain fatty acids having a high neutralisation value, *i.e.*, the acids are either polybasic or of low molecular weight.

The glycerol is largely, if not entirely, in the free condition, and doubtless this accounts to some extent for the moist character of the cosmetic.

The portion of the material which, although soluble in ether, is almost insoluble in light petroleum, appears to consist of "oxidised" acids, *i.e.*, acids containing two or more hydroxyl groups in the molecule. These are characterised by their relative insolubility in light petroleum, their high neutralisation value, and their relatively great solubility in water.

It is highly probable that these acids have been formed in process of time by oxidation of unsaturated acids present as glycerides in the original fat. Such substances are, in fact, known to be formed when, *e.g.*, oleic acid is oxidised by means of air, and this action would no doubt account for the absence of unsaturated products and for the high acidity. The assumption, which seems probable, that the whole of these acids has been formed in this manner would point to the presence in the original cosmetic of about 25—30% of olein or other unsaturated glycerides. It may be mentioned in this connexion that the adipocere formed in the case of animal bodies which have been buried for a considerable time has been found to consist largely of palmitic and stearic acids, together with oxidised acids, oleic acid being absent. The Reichert and Polenske figures (*vide supra*) indicate that the amount of steam-distillable, low molecular-weight, fatty acids is so small as to preclude the presence of any appreciable quantity of a fat such as coconut fat or palm-kernel oil. It is, of course, just possible that such acids may have been decomposed in the course of time, but having regard to their known stability, and to the persistence of the glycerol, this seems very unlikely.

Further evidence of the presence of oxidised acids is to be found in the fact that the so-called " saponification value " of the separated fatty matters is very considerably in excess of the acid value, *i.e.*, the number representing the amount of alkali required for the neutralisation of the acids actually present as such. Inasmuch as the greater part, at least, of the glycerol would appear to be in the free condition, this difference can only be accounted for by the presence of a considerable proportion of acid anhydrides. Lewkowitsch (*Analyst*, 1902, **27**, 139) and other observers have directed attention to this point, and in the case of the oxidised acids from blown oils, such as rape, cotton-seed, maize, etc., differences of from 15 to more than 40 units have been recorded, and this in the acids when freshly formed. There can be little doubt that over so long a period of time the difference in the case of a material such as this cosmetic would be even greater.

In the next place, the cosmetic was very carefully examined with the object of ascertaining the nature of any unsaponifiable matter present. It is, of course, well known that the presence solely of cholesterol in a fat or oil is very generally regarded as indicating its animal origin, whilst the presence of other sterols, chiefly phytosterol, is indicative of a vegetable product. As large a proportion of the fat as possible was saponified, and the very small amount of unsaponifiable matters extracted with great care, but this was found to be entirely free from cholesterol, phytosterol, or any other similar crystalline substance. This is not altogether surprising, since it is known that these sterols undergo decomposition and tend to disappear in process of time. It is, however, disappointing inasmuch as a definite result would, of course, have gone a long way towards deciding whether the fat originally present in the cosmetic was of animal or vegetable origin. Incidentally, it may be observed that the very small amount of unsaponifiable matter obtained showed the absence of matters of a waxy nature, since the unsaponifiable matters in the waxes are, generally speaking, of a very stable character. An attempt was next made to obtain some information as to the identity of the saturated fatty acids present in the cosmetic.

A portion of the original material (70 g.) was esterified with methyl alcohol, and the resulting methyl esters (60 g.) were fractionally distilled at 15 mm., portions being collected as follows :

Fraction 1.	Temp. 115—128°	2·49 g.	57·97 g.
„ 2.	„ 128—154	4·93	
„ 3.	„ 154—182	10·72	
„ 4.	„ 182—205	17·72	
„ 5.	„ 205—220	17·52	
Residual tarry matter		4·59	

Fractions 4 and 5 were redistilled, portions being collected at 195—205° and 210—220°, respectively, and all the fractions were then hydrolysed by caustic soda, and the resulting sodium salts decomposed with hydrochloric acid. The free acids so obtained were crystallised from ethyl alcohol repeatedly until sharp melting points were obtained where possible. It was found that the melting points of the acids obtained from fractions 5, 4, and 3 corresponded with those required for stearic, palmitic, and myristic acids, respectively, whilst the melting points for the acids obtained from fractions 2 and 1 could not be rendered sharp by repeated crystallisation, but were located over a range higher than the melting points of lauric and capric acids, respectively. The coconut odour persisted and was decided in the case of the acids from fractions 2 and 3.

The above results do not, unfortunately, enable us to form a definite opinion as to the nature of the fat or fats originally employed, or even to say with certainty whether they were of animal or of vegetable origin. On the whole, we are inclined to regard the chemical evidence as supporting the view that the fat was of animal character, since it does not seem probable that any vegetable fat with the small proportion of olein this appears originally to have contained would have been available. The chemical evidence, generally, would seem to exclude coconut or palm-kernel oils.

Having regard to all the results, it appears probable that the cosmetic consisted of about 90% of a neutral animal fat with about 10% of some resin or balsam.

The large proportion of calcium present is evidently due to the action of the liberated acids on the walls of the containing vase. The smell of the material, which is one of its most characteristic features, is very probably due to odorous substances formed in process of time from the resins or balsams employed. The odorous constituents of coconut, so far as these are known, are of a kind which would scarcely be likely to have resisted the destructive effect of oxygen and moisture over so long a period, and it is much more likely that the substance or substances formed as the result of oxidation changes would have greater stability.

In conclusion, we desire to express our warm thanks to Dr. Howard Carter for the privilege of examining this specimen, and to Dr. Alexander Scott for his kind assistance in furthering the investigation.

Chemical Laboratories,
8, Duke Street, E.C. 3.

British Museum Laboratory,
39, Russell Square, W.C. 1.

[*Received, August* 14*th*, 1926.]