

**THE INVESTIGATION OF ANCIENT ORIENTAL MATERIALS AND ARTIFACTS  
BY THERMAL ANALYSIS**

HANS G. WIEDEMANN, METTLER INSTRUMENTE AG  
CH-8606 GREIFENSEE, SWITZERLAND

**ABSTRACT**

The application of thermoanalytical techniques in the investigation of ancient materials is illustrated with several examples: incense from the myrrh trees of the Queen Hathshepsut; barley seeds used for weight determinations; silver, gold and bronze coins and weights; ancient seals made of limestone or talc. Modern thermoanalytical methods have the advantage of being fast, sensitive and that they usually require only minute samples of such valuable objects.

**INTRODUCTION**

Ancient materials and artifacts carry messages which help to identify the age of formation, the typical range of components and the morphology of appearance. The results depend on the methods used. Thermal analysis is just one of the many different methods of investigation. This technique is especially suitable, not only because it analyzes small samples, but also for the information it yields on the reproduction of the synthesis of the material. Some of these thermal methods may also be nondestructive.

The first topic of this study was the age determination of the different trees which in ancient times surrounded the Amun Temple in Qurna, Deir el Bahari, Egypt. Today only some stubs of the trees are still extant. Queen Hatshepsut built this monument to honor her father, the god Amun, and decorated the temple with imported myrrh trees. The age of the trees is related to the changes in the cellu-

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Thermal Analysis Highlights, 9th ICTA, Jerusalem, Israel, 21-25 August 1988.

lose and lignin content. Since only the myrrh trees contain resin in form of incense, the thermal behaviour of this important material was of interest, too.

A further investigation refers to the time when the names of weights gradually changed to the names of the money. The weight stones were calibrated all over the ancient world with seeds, which can be correlated by using both husked and unhusked seeds (as opposed to the use of seeds from hybridized grain species, whose weight has changed over the years).

The last topic was the investigation from ancient seals of Palestine and other areas by thermoanalytical methods. Also some aspects of the translated text on the seals is correlated with the results, which can also be confirmed by a papyrus from the Brookline museum, New York.

#### **EXPERIMENTAL TECHNIQUE**

For experimental investigations on ancient materials destructive methods can be tolerated only if they are sensitive and therefore require very small amounts of material. This is the case for X-ray and MS but also for the special thermoanalytical instrumentation (Mettler TA 3000) which was used in the present studies. This new measuring and evaluation system is composed of DSC, TG and TMA. The data acquired during these thermal analyses are saved in a working storage which is accessible for the subsequent evaluation.

The various materials were analyzed by X-ray powder techniques (Guinier, de Wolff camera with  $\text{CuK}_\alpha$ - radiation). The SEM pictures were taken with a Cambridge electron microscope. For chemical analysis the reaction products and the original material were analyzed by means of EDAX (energy dispersive analysis X-ray).

**THE TREES OF QUEEN HASHEPSUT [1-4]**

Queen Hatshepsut was the only female pharaoh in ancient Egypt (18th dynasty, 1505-1449 B.C.). She ruled successfully more than 25 years and was a popular sovereign. According to the Old Testament she also was responsible for the education of Moses [1]. However this statement has not been proved historically.

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Exodus, Mose 5, 2, 2

- 5 And the daughter of Pa'raoh came down to wash herself at the river; and her maidens walked along by the river's side; and when she saw the ark among the rushes, she sent her maid to fetch it.
- 6 And when she had opened it, she saw the child and, behold, the babe wept. And she had compassion on him, and said, This is one of the He'brews' children.
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Figure 1 shows the head of Queen Hatshepsut, which represents a part of a sphinx head.

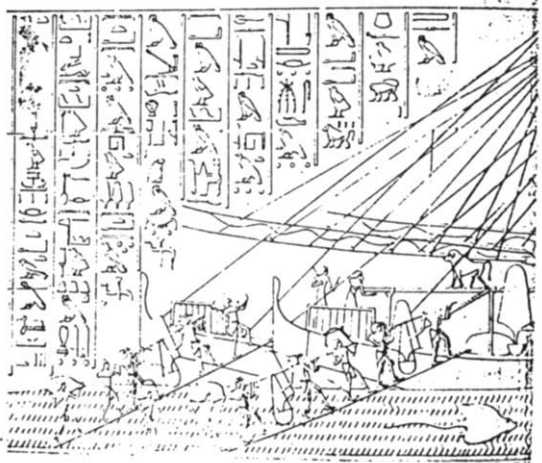


Fig.1 Queen Hatshepsut, sphinx head sculpture (1490-1468 B.C.)      Fig.2 Loading of myrrh trees in Punt

Hatshepsut built the temple of Deir el Bahari in Thebes to honor her father, the god Amun. This temple, which exists today and has been restored by Polish archaeologists, was the starting point of these present investigations.

The inscriptions which are still preserved on the temple walls, tell about an expedition to Punt in Ethiopia. The purpose of this travel was to visit foreign countries in order to establish trade by bartering. Hatshepsut wanted to get some myrrh trees to decorate the temple of Amun. The pictures on the temple walls show the loading of the trees aboard the boats (Fig. 2).

It was possible to get small amounts of the still existing myrrh trees stubs for investigations. The TG- and DTG-curves of these samples were recorded in oxidizing atmosphere (Fig. 3a).

They show the weight losses and the different thermal stability of cellulose at 180 to 380°C and of lignin at 380 to 500°C. Isotope

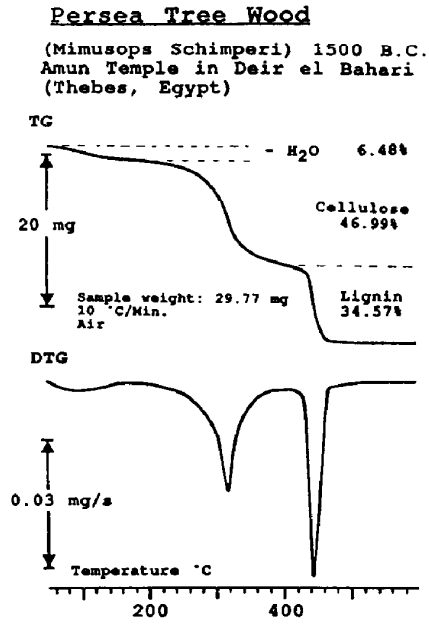
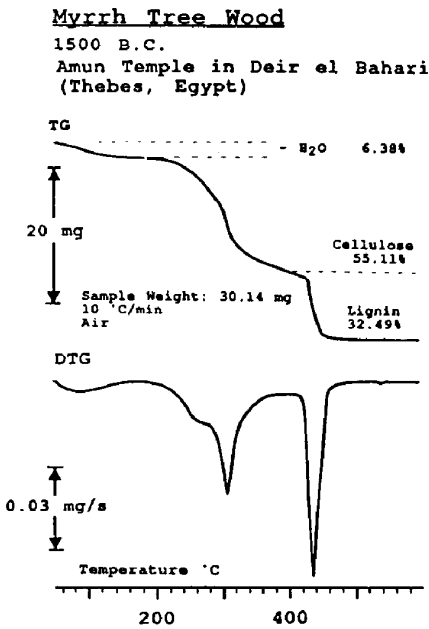


Fig.3a TG- and DTG-curves of myrrh tree wood

Fig.3b TG- and DTG-curves of persea wood

dating with C14 of the same tree stubs gave  $2700 \pm 110$  years. The difference to the times of Hatshepsut may be due to the position from which the samples have been taken: in the case of the C14-method it was from the outside of a stub of 25 cm in diameter. The growth time of the trees in order to reach this diameter however is about 600 years.

Results of analytical pyrolysis with a linear mass spectrometer proved that also in the case of these myrrh tree stubs the dehydration process of cellulose goes from levoglucosan (mass 162) to levoglucosenon (mass 126), similarly as was found for ancient papyri from the same era [4]. Cross sections of the sample definitely proved that the wood is from tree stubs, and that the contained resin corresponds to incense.

Further tree stubs have been found outside the entrance to the temple of Deir el Bahari, which belong to the species *Persea*, *Mimusops Schimperii*. This tree produces very tasty fruit, which may have served in ancient times as some kind of refreshment to visitors to the temple. The wood of this tree, which has a high SiO<sub>2</sub>-content, is relatively hard and therefore was used for making sculptures. The age determination of these stubs by means of an acceleration mass spectrometer also leads to about 2700 years. Since the samples were collected from the outside of the tree stub, which showed 800 annual rings, the age agrees very well, too, with the time at which the temple was built. The difference between the Myrrh tree stubs from the temple area and the *persea* tree stubs at the entrance can be seen in the quite different burning profiles (see TG-/DTG-curves, Fig. 3b) of these materials.

#### INCENSE [7]

A comparison of ancient incense and the incense used today shows great similarity. Incense was used in ancient times during religious rituals and divine services. For this purpose the material was vaporized in little furnaces during the ceremonies. The thermal behavior of incense is shown in form of a DSC curve (Fig. 4). The two first peaks correspond to the vaporization of the volatile components and the large peak to the pyrolysis of the residue.

Figure 5 shows two of the decomposition products of incense, Olive - tol (I) and Verbenol (II) and the possible formation of Tetrahydro cannabinol (III). The effect of vaporized incense at religious meetings can be explained as follows:

- 1st) Creating a specific atmosphere.
- 2nd) The phenols as decomposition products had an antiseptic action, important at a gathering of so many people.
- 3rd) The pyrolytically generated cannabinoids induced psychoactive stimulation.

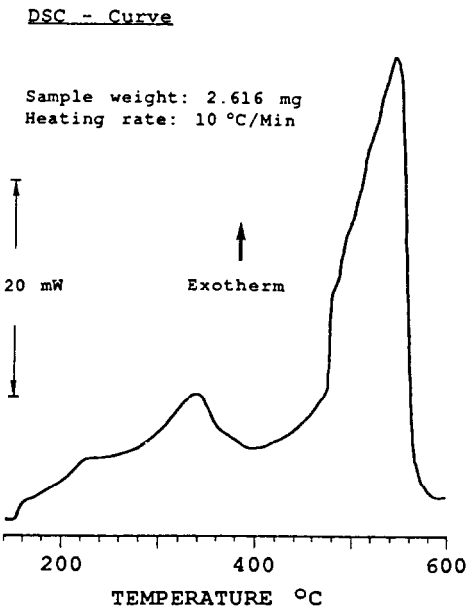


Fig.4 DSC curve of incense

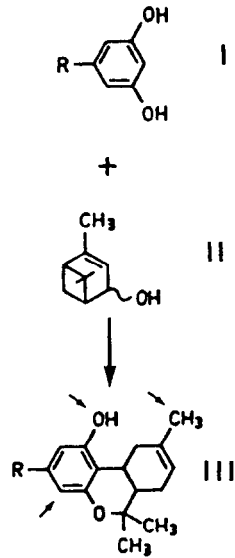












Fig.5 Formation of Tetrahydro cannabinol during burning incense

**SEEDS- WEIGHTS- MONEY [5-12]**

We find a good example of a measuring system in ancient Egypt whose use even extends up until today. The basis for the linear measure systems was the measurement from the elbow to the index finger, i.e. the cubit (52.5 cm). A fragment of an original cubit is shown in Figure 6. The cubit is divided into seven hand breadths (i.e. 28 finger

**Table of Length and Fractions of a Cubit**

Royal Cubit		= 28 Fingers / 52.64 cm
Small Cubit		= 24 Fingers / 45.00 cm
Upper arm		= 20 Fingers / 37.50 cm
1/2 Royal Cubit		= 14 Fingers / 26.25 cm
1/2 Small Cubit		= 12 Fingers / 22.50 cm
2 Palms		= 8 Fingers / 15.00 cm
Fist		= 6 Fingers / 11.25 cm
Hand's breath		= 5 Fingers / 9.38 cm
Palm		= 4 Fingers / 7.50 cm
Finger width		= 1 Fingers / 1.88 cm

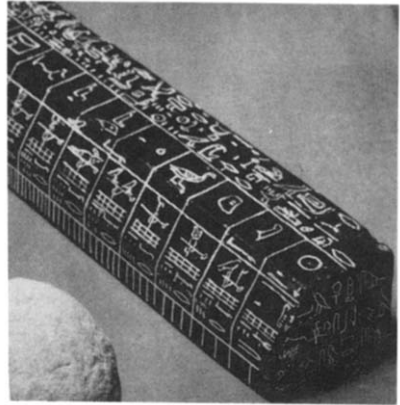


Table 1 Length and Fractions  
of a Cubit

Fig.6 Fragment of an ancient  
cubit of Osorkon, Museum Cairo

breadths) and also allows the measurement of small objects as well as long distances (Tab. 1). Fig. 7 shows the fundamental unit of the cubit which corresponds to the width of one finger or to the diameter of six grains of barley. Each one of the 28 fingers which make up the length of one cubit are correlated to deities of ancient Egypt. For example the measure of the bronze statuette of the God Osiris corresponds exactly to the length of five fingers, whereby the fifth

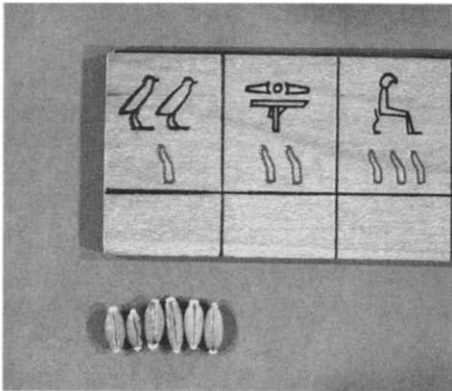


Fig.7 Six grains of barley compared to the width of one finger

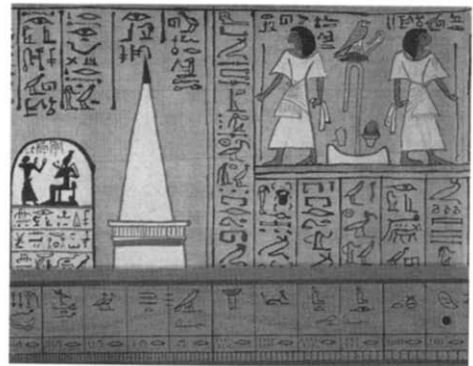


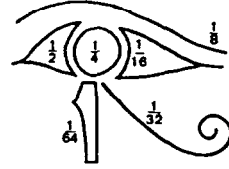
Fig.8 Column subdivision on the Papyrus Hunefer in finger widths

Remark: In Figures 7 and 8 a reproduction of a cubit in form of a today's ruler is used. (Reproduction by the Museum of Fine Arts, Boston, U.S.A.).

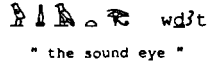
Measures in Fingers	ISRAELIAN Measures	EGYPTIAN Measures	BABYLONIAN Measures
28	ammah 49.5 cm	Royal cubit 52.5 cm	ammatu 49.5 cm
20	seraim 36.7 cm	upper arm 37.5 cm	brick 32.76 cm
14	serath 27.71 cm	.5 R.cubit 26.5 cm	foot 27.58 cm
12	tophah 24.75 cm	.5 s.cubit 22.5 cm	0.5 cubit 25.86 cm
4	tephach 8.25 cm	palm 7.5 cm	palm 6.9 cm
1	etzba 2.0g cm	finger 1.88 cm	

Babylonian measures are derived from the cubit of Nippur

Table 2 Comparison of Oriental Cubits and their Fractions



The symbols employed in this figure, are derived from the ancient myth of the falcon-god Horus:



These fractions together add up to 63/64; presumably the missing 1/64 was supplied magically by Thoth.

Fig.10 Horus eye as corn measure

finger carries his name. Another example is the subdivision on the Papyrus of Hunefer, a part of the Book of Death (ca. 1370 B.C., British Museum, London). The breadth of one column equals one finger, 18.8 mm (Fig. 8). All measures in temples, tombs and pyramids are based on cubits as a unit. Long distances were measured in cubits, too, e.g. the daily work of a towing crew is given as 10000 cubits, which is marked on the banks of the river Nile in the form of stelae. A comparison of linear measures used in ancient Egypt, Israel and Babylonia, is given in Table 2.

Capacity - Measures	Log(in cm <sup>3</sup> )	720 Log = Litres
Syrian-Babylonian	541	Homer 389.7
Phoenecian	508	Kor 365.7
Isrealite	525	Homer 389.7
Egypt	545	-

F.G. Skinner [12]

Table 3 Capacity-Measures

Fig.9 Capacity measure / copper (Amenophis III., 1417-1379 B.C.)





The square measure Aroura, originated in the cubit, served as land and field survey and corresponds to an area of 100 x 100 cubits. The daily measurement of food and commodities was carried out with help of hand or stand scales (see Fig. 11) or by dry or liquid measures (see Tab. 3 and Fig. 9).

One of the large units, the Hekat, was a half-peck dry measure for barley, wheat, corn and grain. For storage granaries a much larger unit was used like the "100 quadruple Hekat". One cubic cubit contains 30 Hekats of grain. The Henu, about one tenth of a Hekat, was a smaller unit for grain ( ~0.45 l ).

The smallest named unit for grain was the Ro, which was 1/320 part of the Hekat. The Ro was suitable in the making of mixtures of medicines and can be seen in the Papyrus Ebers which contains hundreds of such recipes. It corresponds to a today's tablespoon full of grains or other goods. The only fractions of a Hekat used were 1/2, 1/4, 1/8, 1/16, 1/32, 1/64, and these were written in a special way. They were called Horus-eye fractions, and were used solely for grain (see Fig. 10). Then 1/64 of a Hekat contained 5 Ro, and for any fraction of a Hekat less than 1/64, Ro and sometimes fractions of a Ro had to be used. The summation of the Horus eye fractions equals 63/64, the missing 1/64 belongs to the messenger of gods, Toth, in ancient Egypt [10].

Most civilizations took seed of barley, wheat, millet etc. to calibrate the scales, weights and dry measures respectively. In the Mediterranean barley and wheat was generally used, in China millet [12].

The seed's weight is very homogeneous and varies little even under very different climatic conditions. This is valid for the volumes of the grains, too.

Thus the weight units were compared with counted quantities of seed. Later, the weight units were replaced by solid weights which were also calibrated with counted seed. The weights were made out of different stones and minerals, e.g. basalt, agate, hematite, and later, depending on their size, out of metals like bronze, lead, silver, gold, etc.

The Babylonian weights which are considered the originals, probably

also had some importance as money. Gold and copper rings of defined weight were used on certain occasions, especially as payment for goods of higher value (Fig. 11). Stone weights were marked with their corresponding ring value. These rings represent the oldest known metal coins. The prices of articles were set by quoting their weight in copper or gold. In the highly developed ancient commerce, orders, accounts and receipts were written in form of documents, whereby the value of an article was added in terms of Deben (copper). The weight of one Deben corresponded to 91 grams. Fragments of such ancient business and accounting documents were found on the island Elephantine in the Nile. Originally gold and silver rings were used as jewelry having about the same value.

The larger weights used in the ancient world (ca. 1500 B.C.) had specific forms, e.g. sleeping ducks, hippopotamus, cows or simple shapes like cubes with rounded edges.

Also the smaller weights were made in the form of animals or in the shape of grains of barley, emmer or other seeds (Fig. 12). It is now an established fact that grains and seeds were used in ancient times not only as standard weight units, but also for calibrations of length and volume measures. Especially husked barley seeds were the standards for calibration.

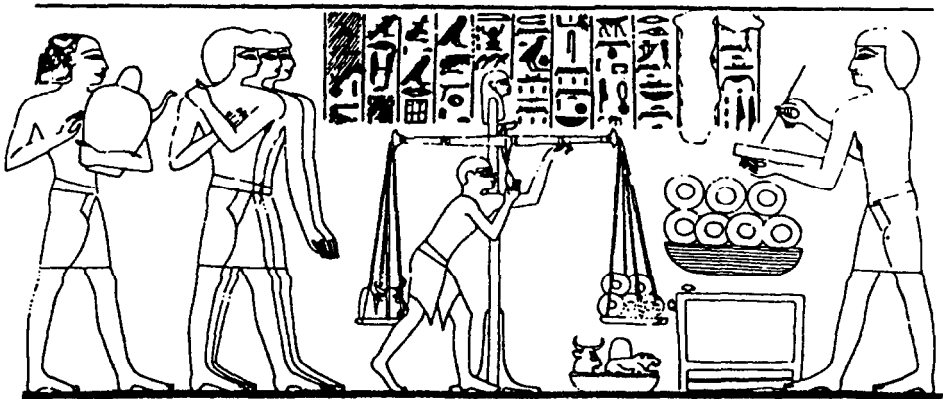
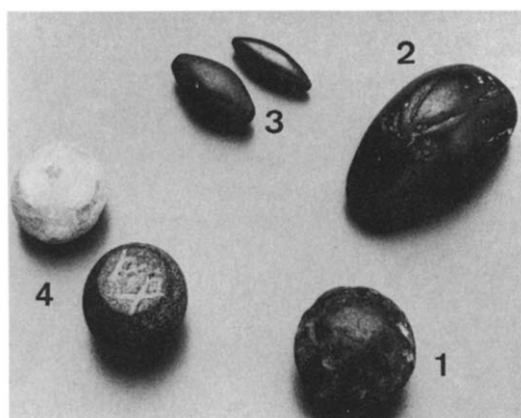


Fig.11 Weighing and issuing precious metals.



- 1) Kedet
- 2) Shekel
- 3) Staters
- 4) Ounces

Fig.12 Shapes of small ancient weights

In the Old Testament, two different weights are given for the same weight unit, the shekel. Hence, perhaps the Bible verse [1]:

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Deuteronomy, Mose 5, 25

13 Thou shalt not have in thy bag divers weights, a great and a small.

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The shekel was used as weight and also as coin. The gold shekel (royal shekel), however, was used as weight only. Its weight (16.37 g) corresponds exactly to 360 unhusked barley seeds as we found out (Fig. 13).

The other royal shekel was the silver shekel which was used as a coin and as a weight unit. Our investigations proved that its weight (14.86 g) corresponds to 360 husked barley seeds (Fig.13). These results may

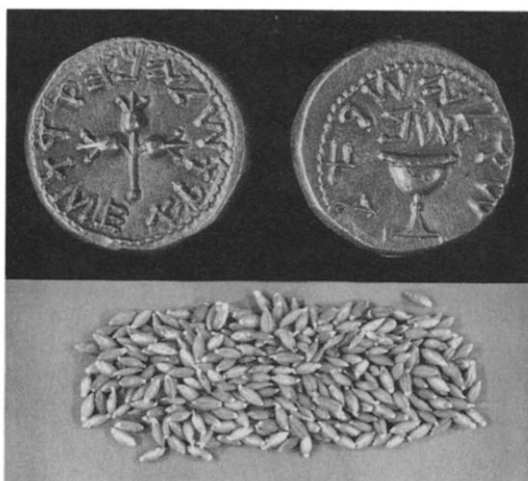


Fig.13 Silver shekel and its corresponding weight in barley

help to clarify some of the discrepancies which exist in the literature. The third kind of weight unit was the common shekel, made out of silver or bronze, which was used also as a coin. Its weight (7.3 g) corresponds to 180 husked barley seeds. Therefore it is equal to 1/2 shekel. There is also an interesting relation between the deben and the shekel. The weight of 1 deben (91 g) corresponds exactly to 12 common shekel. These and other interesting relationships between the weight units which were used in the ancient world are compiled in Table 4.

A further interesting relationship exists between the weight of the Roman bronze Ounce (2nd-3rd Century A.D.) and the ancient Egyptian Kedet (664-525 B.C.). The weight of the ounce corresponds precisely to that of 3 Kedet.

A selection of such ancient coins (Tetradrachme, Stater and Shekel) is shown in the photograph (Fig. 14). The nondestructive chemical analysis carried out with EDAX (Fig. 15) proved that the Shekel was made with pure silver (some impurities such as Cu, Si are present only on the surface). This is also true for the Tetradrachme, which shows some surface impurities (Ca, Si, Al, Mg, Fe). The Stater on the other hand, which was struck in Teos 310 B.C., consists of exceptionally pure gold. For the Egyptian Kedet the EDAX proved its very heterogeneous composition and iron as the main impurity in this ancient bronze coin (the segregations showed higher concentrations of lead (up to 2.1%) and tin (up to 12.1%).

In contrast to these findings the Roman bronze Ounce is rather homogeneous and corresponds almost to a technical bronze. Some small-scale segregation however was found also in this bronze, especially with high-lead content (up to 18.8%). The one century older Roman lead Ounce (1st-2nd century A.D., found by Xanten am Niederrhein), again is very heterogeneous and contains tin-rich segregations between 78.6% and 96.8% Cu and Pb-rich areas from 56.3% to 86.3% Pb. This coin was strongly corroded, the grayish-white surface layer consists of almost pure  $PbCO_3$ , as could be proved by X-ray diffraction analysis (Fig. 16), and by simultaneous TG/DTG (Fig. 17). The thermal decomposition of this corrosion layer is identical to that of pure, freshly precipitated  $PbCO_3$ . The chemical composition of the various weights and money which have been discussed, are summarized in Table 5.



Fig.14 Ancient coins:  
Tetradrachme,  
Gold Stater &  
Silver Shekel

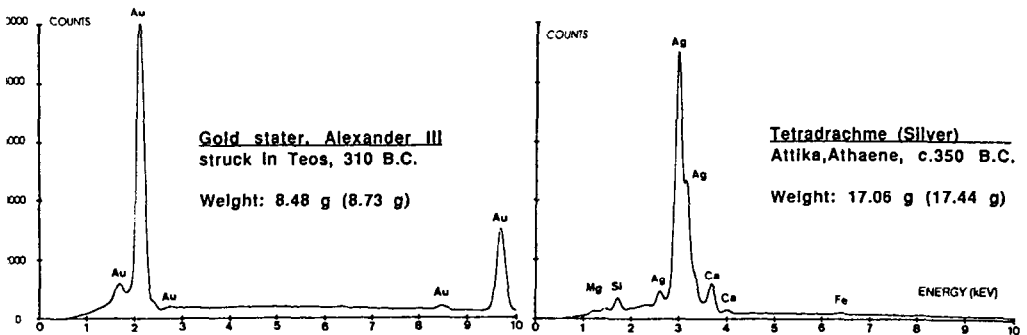


Fig.15 EDAX spectra of different coins.

### Israel/Babylonia

Talent	=	58.944 kg	=	60	Minas
Mina	=	0.9875 kg	=	60	Gold Shekel
1/3 Mina	=	0.3275 kg	=	20	Gold Shekel
Gold Shekel	=	16.37 g	=	360	Grains Barley (unhusked)
Silver Shekel	=	14.55 g	=	360	Grains Barley (husked)
Beka	=	8.188 g	=	1/2	Gold Shekel

Table 4 Comparison of Ancient Weight Units

**Composition of Ancient Weights and Money**

	EGYPT	ISRAEL ATTIC	GREECE CLASSIC	GREECE	ROMAN EMPIRE	
	Kedet	Sheckel	Drachme	Stater	Uncia (Bronze)	Uncia (Lead)
ELEMENTS    %						
Au				99.9		
Ag		99.2	98.0			
Cu	89.9 85.2*	0.2			93.2 74.0*	
Pb	0.5 2.0*				0.3 18.8*	99.4 2.7*
Sn	8.9 12.1*		11.2		6.5 7.2*	0.5 96.8*
Fe	0.7 0.6*		0.1			
Al			0.2			

\*) *Composition of Segregations*

Table 5    Composition of Ancient Weights and Money



a) precipitated  $PbCO_3$

b) Cerussite

c) surface corrosion layer (Roman ounce)

Fig.16 X-ray diffraction pattern of lead carbonates

## ANCIENT SEALS [13-18]

Seals are more or less among the oldest documents which carry written information. Therefore the identification of the materials from which the seals are made and their age determination is of special interest.

Another point is the translation of the written text. We investigated one seal/scarab which was made from silicified limestone (Fig. 18). The determination of the phase composition can be done by TG and DSC measurements. The limestone can be evaluated by decomposition, the  $\text{SiO}_2$  content by the  $\alpha$ - $\beta$  transformation; when the measurement is performed in  $\text{CO}_2$  atmosphere, this investigation is non-destructive. A triangular presentation of the compositions (Fig. 19) shows diffe-



Fig.18 Scarab of silicified limestone, Amarna 3370 B.C.

## Roman Ounce

2. Century A.D., Pb-Sn Alloy

Investigation of the surface corrosion layer

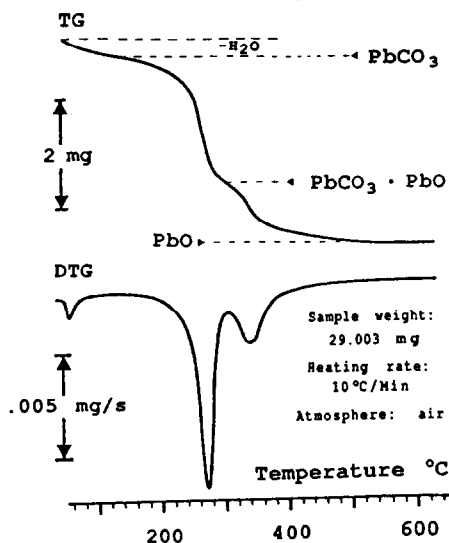


Fig.17 TG/DTG of a surface layer of a Roman lead Ounce and the precipitated  $\text{PbCO}_3$

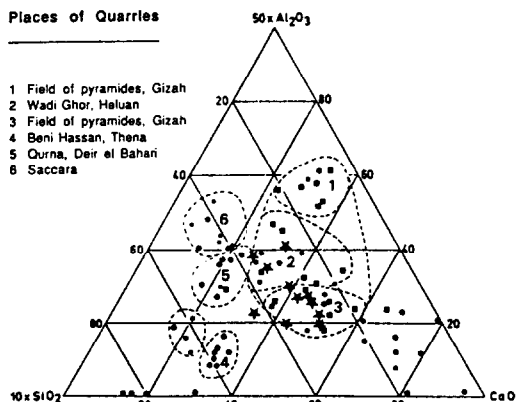


Fig.19 Triangular presentation of the composition of silicified limestones from different quarries in Egypt.

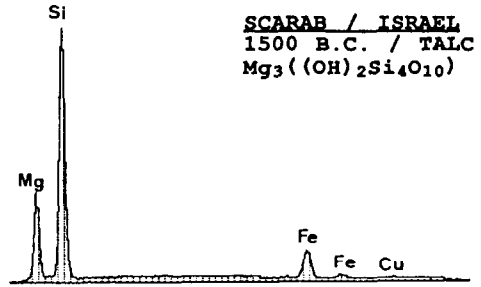
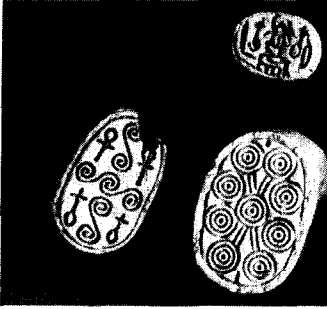


Fig.20 Canaan scarabs, talc Fig.21 Edax of scarabs (Israel), talc

rences between the silicified limestone from different places in Egypt which were used for buildings, sculptures and scarabs [17]. Our sample came from Amarna, Lower Egypt and could be identified with the material of this region.

Another kind of Scarab comes from Canaan. These were made from talc, a magnesium hydroxy silicate. Figure 20 shows two samples. The natural form of the material is relatively soft, so that forming and engraving is very simple to do. Only on heating the material does become hard. An EDAX analysis of these scarabs proved the composition of talc (Fig. 21), and in addition a small amount of iron which is incorporated either into the talc or into a secondary mineral phase, chlorite.

Figure 22 shows the TMA and TG curves of an original scarab from Israel. During the continuous release of adsorbed water up to about 400 °C the material shrinks and shows a weight loss of less than 1%. Above this temperature the normal, relatively high thermal expansion of enstatite can be seen from the strong upward drift of the TMA - curve. It should be mentioned here, and X-ray powder patterns have proved, that these ancient scarabs have been heat treated already and were transformed completely into orthorhombic enstatite. Figure 23 shows the X-ray powder pattern of the original scarab compared to talc. Therefore these scarabs have been hardened already in ancient times by heating to at least 900°C. The second slight weight loss around 800 - 900 °C in the original material cannot be explained as yet, since X-ray patterns do not show any trace of talc in the scarab sample. Besides it is highly unlikely that decomposed talc would rehydrate. Since the thermal decomposition of massive talc leads to a porous enstatite (unless the material is sintered at high temperature), the



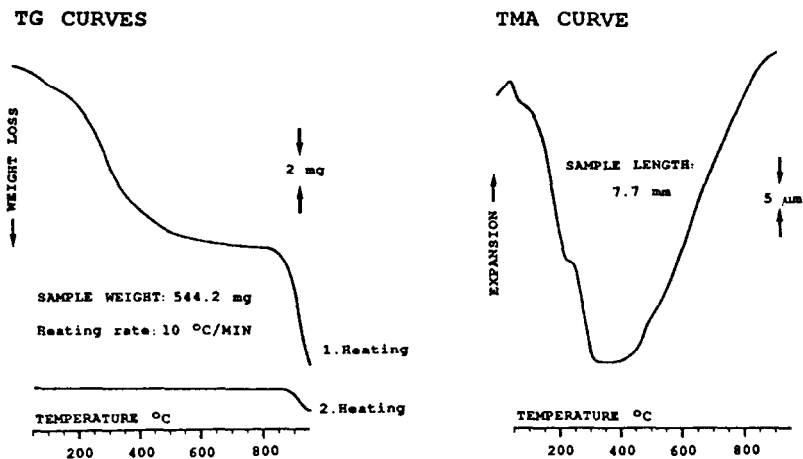


Fig.22 TG and TMA curves of a scarab from Israel

amount of adsorbed water may be related to the age of the scarabs. Further measurements on a larger number of samples are necessary however for making any definite statements.

Also the next two seals, of which the inscriptions are reproduced, (Fig. 24) were found in Israel [13-16]. The Hebron and Gaza regions are well known as sources for this kind of material. As was found out of the context, these items may belong to the XIIth or XIIIth

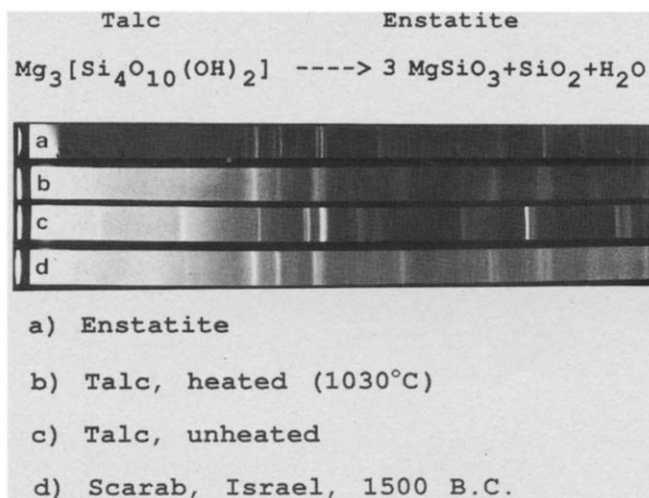


Fig.23 X-ray pattern of talc samples and an original scarab



Fig.24 Two reprints of ancient scarabs of Canaan

Dynasty or to the Hyksos period(1785-1580 B.C.). The private names of the bearer of offices are all Egyptian except the overseer of the treasury, Jacob. Among kings and princes Semitic names are much more common.

The reprint of the left scarab is divided into two parts, the right, comprising more than half, shows a historical scene, the left contains the inscription. The scene shows the ritual killing of the oryx. The king is holding the animal with his left hand and swings the mace with his right. He wears the Red Crown of Lower Egypt. Behind him there

	m()n-h-m-a	Munahhima	"God is merciful"
in Hebrew		menhem	"The one who alleviates grief"
	c-t-n-a		"My Lord is xy"
in Hebrew		aduni	"My Lord"
in Amoritic		adunijjah(u)	"My Lord is Jawhe"
		Aduni Ba <sup>c</sup> al	"My Lord is Baal"
	c()g-b-tu		"protect"
in Amoritic		Ya-ah-qu-ub-el	"God may protect"
in Hebrew	ja <sup>c</sup> aqob-el	Jacobel	"Jacob"
in Ugarit	Yaqub-ba al		"Baal may protect"

In the Papyrus 35.1446 of 95 registered names 82 were still preserved.  
51 were Asiats which means Semitic people

Table 6 Northwest-Semitic Names in a List of Egyptian Slaves [14,18]

is the sign "destroy" (sk), which spells out what the king is doing. On the left, at right angles to the scene there is a group of signs "Foremost of the Northland" (ḥ3t mḥw). This must be regarded as a new title of the kings of the Hyksos period.

On the right scarab within a frame consisting of a thin line there is the inscription: "The eldest king's son, Jacobam" (S3 nsw ṣmsw yḥkꜣm). Thus Yacobam could be Jacob of the Old Testament, the very same person before accession to the throne. Table 6 shows three selected names of a list of slaves from the contemporary papyrus No. 35.1446 (Brooklyn Museum, New York), where the name of Jacobel is also mentioned (Tab. 6). The translation of the name means probably "Baal may protect" [14, 18].

#### **CONCLUSION**

Experimental investigations on a variety of ancient artifacts and materials such as wood, incense, weights, money and seals proved that thermal analysis especially in combination with analytical pyrolysis, microscopy and X-ray are powerful methods in archaeometry.

Analysis of these precious objects can be carried out with minute amounts of samples and is even non-destructive in certain cases. Furthermore some interesting, mutual relationships between such ancient materials and their historical background may be derived from such investigations.

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